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THE COMPATIBILITY OF VARIOUS METALS WITH LIQUID FLUORINE

TECHNICAL DOCUMENTARY REPORT No. ASD-TDR-62-250

MARCH 1962

AIR FORCE MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Project No. 7312, Task No. 731202

Best Available Copy

(Prepared under Contract No. AF 33(616)-6515 by the
Air Products and Chemicals, Inc., Allentown, Pennsylvania;
Sidney Kleinberg and James F. Tompkins, authors)

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FOREWORD

This report was prepared by Air Products and Chemicals, Inc. under USAF Contract No. AF 33(616)-6515. This contract was initiated under Project No. 7312, "Finishes and Materials Preservation", Task No. 731202, "Corrosion and Deterioration Control". The work was administered under the direction of Air Force Materials Laboratory, Research and Technology Division, with Mr. O. O. Srp (MAMP) acting as project engineer.

This report covers work conducted from October 15, 1960 to October 15, 1961.

This report has been preceded by two summary technical reports; WADD TR 60-436, which described the work done from June 1959 to June 1960 and WADD TR 60-819, which described the work done from June 1960 to October 15, 1960. This report concludes work under this contract.

ABSTRACT

This report covers studies of liquid fluorine and its contaminants, reactions on metal surfaces, fluoride films, and immersion tests of tensile specimens. The solubilities of contaminants in liquid fluorine are: 40% CF_4 at -297° F, 4.5% CF_4 at -320° F, > 10% OF_2, < 0.5% HF, < 0.1% SiF_4 and SF_6 at -320° F. A method of preparing contaminant free fluorine and its infrared analysis is discussed.

No liquid phase reaction between fluorine and metals has been observed but gas phase reaction occurs during warmup. Small weight changes in metal specimens immersed in liquid fluorine for 15 and 75 minutes were noted. Increased corrosion of similar specimens in OF_2-contaminated fluorine is not conclusive. Exposure of hydrocarbon films on metals to F_2 leaves carbon deposits while exposure to ClF_3 or $ClF_3 + F_2$ leaves fluorocarbon films.

No detectable fluoride films formed on aluminum specimens using electron diffraction techniques. At low temperatures gaseous fluorine formed films of less than 200 Angstroms on metal powders with initially rapid film formation tapering off to a negligible rate after one day. Metal powders adsorb gaseous fluorine at -297° F.

Metal tensile specimens exposed to liquid fluorine for one year corroded < one mil. These specimens had essentially the same tensile properties as similar ones exposed to liquid nitrogen.

PUBLICATION REVIEW

This technical documentary report has been reviewed and is approved.

FOR THE COMMANDER:

O. O. SRP
Acting Chief, Physical Metallurgy Branch
Metals and Ceramics Division
AF Materials Laboratory

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THE COMPATIBILITY OF VARIOUS METALS WITH LIQUID FLUORINE

SUMMARY TECHNICAL REPORT

I. INTRODUCTION

The objective of the experimental work described in this report has been to broaden the existing knowledge of liquid fluorine handling. The work done under this project has resulted in a better understanding of the behavior of materials exposed to liquid and gaseous fluorine from a corrosion viewpoint. Air Products and Chemicals, Inc. was assigned this project in June 1959 by Materials Central of the Aeronautical Systems Division, under contract AF 33(616)-6515. This report, covering the period from October 15, 1960 to November 15, 1961, has been preceded by two summary technical reports covering the periods June 1959 to June 1960 and June 1960 to October 1960. (13, 14).

The work during the first period dealt with the determination of the corrosion rates of typical metals during static exposure to liquid fluorine for periods up to two weeks. Metals tested included several aluminum alloys, various stainless steels, titanium, magnesium, copper, brass, monel, and nickel. Amorphous and graphitic carbon were also tested. Specimens were tested in stressed and unstressed conditions. The impact-ignition characteristics of titanium and aluminum in liquid fluorine were studied.

During the second period, studies of contaminants in liquid fluorine, fluoride film studies, metal-liquid fluorine corrosion rate with time, metal ignition in liquid fluorine, and one-year exposure studies of tensile specimens were started.

During the present period the above work has been continued. The work for this year can be divided into the following general areas:

A. Fluorine Contaminants
B. Reactions of Fluorine on Metal Surfaces
C. Fluoride Film Studies
D. Properties of Metals After One-Year Exposure to Liquid Fluorine

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II. DISCUSSION OF EXPERIMENTAL WORK

A. FLUORINE CONTAMINANTS

The work involving fluorine contaminants consisted of the following studies:

1. Identification of contaminants in commercial fluorine cylinder gas.

2. Removal of observed contaminants from commercial fluorine cylinder gas.

3. Measurement of solubility of possible contaminants in liquid fluorine.

4. Development of infra-red analytical technique for gaseous fluorine.

Commercial fluorine is produced by the electrolysis of KF · 2HF (10). In this process, small quantities of contaminants are generated. Hydrogen fluoride (HF), present in greatest quantity, is removed by condensation and by passing fluorine gas through sodium fluoride absorption towers; however, it has been found that trace quantities of hydrogen fluoride remain in gaseous fluorine even after treatment (10). Other contaminants produced from moist or impure electrolyte or by reaction of gaseous fluorine with process equipment, are not removed. For most applications, the presence of small quantities of contaminant in gaseous fluorine presents no serious problems; however, when fluorine is liquefied for use as an oxidizer for missile fuels, the presence of contaminants is highly undesirable. The experimental work described below was designed to determine the effect of insolubility and corrosive action caused by the presence of fluorine contaminants.

1. Experimental Apparatus

A multi-purpose experimental apparatus was designed and built to be used for identification of contaminants in liquid fluorine, their removal from liquid fluorine, and for measuring solubilities.

A schematic diagram of the experimental apparatus is shown in Figure 1. In order to be able to work with small quantities of low pressure fluorine gas, a reservoir was connected to a fluorine cylinder located behind a steel barricade. The reservoir was filled to about 60 psig and isolated from the cylinder. The fluorine used in experiments was metered from the reservoir with a Hoke needle valve with a metal-to-metal seat. The system for contaminant studies included a sodium fluoride bed, in which hydrogen fluoride could be absorbed, and four condensing compartments. Three condensers were made of monel and the fourth was a stainless steel receiver for holding processed fluorine. Fluorine could be transferred from one condenser to any other. A Hoke, 5-12 micron filter was attached to one condenser, another was equipped with a cooling coil and the third was a simple cell. The cells were connected into the system with swedge-type connectors so that they could be easily removed from the system. When necessary, a glass condensing cell could be substituted for a monel cell for visual examination of liquid fluorine.

A series of manifolds were connected to the system in addition to the fluorine manifold. It was possible to draw vacuum, purge with nitrogen or helium, or dispose of the fluorine to a 20 foot deep pit filled with lime. Four Dewar vessels were arranged so that each condenser and its related apparatus could be submerged in the liquid nitrogen or liquid oxygen. (Because the boiling point of fluorine is -306° F at 1 atm and about -297° F at 12 psig, the gas could be liquefied in liquid nitrogen at atmospheric pressure or in liquid oxygen under moderate pressure.)

All work was carried out within 1/4" thick steel cabinets with valve stems projecting through the steel. The system could be examined using periscope mirrors (13). The cabinets were well ventilated at all times. All operations were conducted remotely to eliminate possible hazards of handling fluorine.

2. Identification of Contaminants

Since it was to be expected that the quantities and concentrations of contaminants in gaseous cylinder fluorine would vary among cylinders, several were sampled. Each time a different cylinder was placed on stream, the entire system was evacuated up to the cylinder to insure

that no contaminants could come from the residual gas in the equipment. The apparatus was then purged with nitrogen or helium and again evacuated before fluorine was introduced.

Since the quantity of contaminants in cylinder fluorine is often small, it was necessary to concentrate them to obtain sufficient quantities for good analytical results. Fluorine was condensed in a liquid oxygen bath at about 15 psig. The fluorine was then distilled by reducing the pressure and collecting it at liquid nitrogen temperatures in a second condenser. The residue from the distillation was a collection of contaminants less volatile than fluorine. With the fluorine gone, the contaminants were isolated, warmed, and allowed to vaporize. They were then sampled in an infra-red cell and analyzed.

The results of experiments for identification of contaminants in cylinder fluorine are presented in Table I. The following contaminants have been found: HF, OF_2, SiF_4, SF_6, CO_2, SO_2F_2, and CF_4. The presence of COF_2 is questionable, but may be present in cylinder fluorine and decompose to CO_2 and SiF_4 when handled in the experimental apparatus. Oxygen difluoride, OF_2, was found in only one sample, but the presence of this material is not unexpected as it may be generated in a fluorine cell when fresh electrolyte is used (10).

3. Purification of Cylinder Fluorine

The most troublesome contaminants in fluorine are expected to be those which are insoluble in the liquid and which would be present as solids which could plug transfer lines and valves. It was felt that filtration of liquefied fluorine would remove these contaminants. The fluorine could further be purified by distillation.

Filtration of liquid was accomplished by condensing the gas in a monel cell and forcing it through a Hoke 5-12 micron filter. The filtered fluorine was analyzed by taking a sample in an IR cell or by distilling the fluorine as described above and collecting the contaminants if any were present. In most instances, the fluorine was passed through a NaF bed to absorb troublesome HF before filtration.

To obtain fluorine of high purity, an all metal system must be used, as it is suspected that fluorine reacts with pyrex glass to form SiF_4 if glass is in the system. If all contaminants are not removed by passing fluorine through the NaF bed and filtering, the gas must be distilled. The distillate is analyzed by infra-red spectrophotometry. If contaminants remain, a fresh condenser is evacuated and the fluorine is again distilled. As long as contaminants appear in the distillate, another distillation is performed until contaminant-free fluorine is produced.

The major requirement for producing contaminant-free fluorine is an absolutely leak tight system. Even the smallest leaks allow contamination into the system. With a leak tight system, high purity fluorine may be routinely produced by passing the gas through a sodium fluoride scrubber, filtering the liquid, and distilling by a one-step technique.

Table I summarizes the runs made to produce contaminant-free fluorine. Fluorine with no identifiable contaminants was produced only after all leaks in the experimental apparatus and analytical equipment were eliminated. It was verified that passing gaseous fluorine through NaF does not remove last traces of HF. This may be because the NaF does not quantitatively remove HF or because there is imperfect contact of the gas with the NaF pellets in the absorber. Filtration alone is insufficient to remove all contaminants because some solid particles are small enough to pass through the filter. However, because of the low volatility of fluorine contaminants, single step distillation may be used for separating contaminants from fluorine. The distillation is a simple technique and may be used for purifying fluorine when high purity material is needed.

4. Solubility of Fluorine Contaminants

The method selected to determine the solubility of contaminants in liquid fluorine was to prepare mixtures of known quantities of contaminants in purified gaseous fluorine, condense the gas, and note whether solids were present in the liquid.

Pure fluorine was prepared as described above. The contaminant was mixed with the fluorine in the gaseous state. Assuming the ideal gas laws to hold at low pressures, the volume of a gas is proportional

to its partial pressure in a mixture. Purified fluorine gas was added to an evacuated cylinder to a predetermined pressure. Contaminant gas was added to the purified gas to increase the pressure by a predetermined value. The fraction of contaminant in the fluorine gas was then the ratio of the partial pressure of contaminant to the total pressure.

After the contaminated fluorine was prepared, it was condensed in a glass cell immersed in liquid nitrogen. The cell was observed through strategically located periscope mirrors so that both the top and sides of the cell could be examined. If solids appeared in the condensed fluorine, more contaminant had been mixed than was soluble. A new mix was prepared with less contaminant, and condensed. The solubility was determined to be the mix when solids did not appear in the liquid.

Table II summarizes runs made to determine the solubility of contaminants in liquid fluorine. At -297° F the solubility of CF_4 in fluorine is greater than 40 Mol%. At -320° F its solubility is 4.5%. At least 10% OF_2 is soluble in liquid fluorine at -320° F. Less than 0.2% SF_6 and SiF_4 are soluble at -320° F. The solubility of HF is less than 0.5%.

The limitation of this method is the accuracy of preparing mixes of contaminant in gaseous fluorine. The solubilities reported as less than a certain percentage are the smallest percent mixes that could be accurately prepared.

5. Infra-Red Analysis

The most important aspect of this work was to develop a rapid and simple analytical technique for analyzing fluorine. The method selected was infra-red spectrophotometry using a Beckman IR-4. Two types of sample cells were used, one with standard sodium chloride windows, the other with calcium fluoride windows.

Calcium fluoride windows were required for analysis of fluorine because they are inert to fluorine gas. The fluorine reacts with standard NaCl windows as follows:

$$2\,NaCl + F_2 = 2\,NaF + Cl_2$$

This is a quantitative reaction sometimes used in the analysis of fluorine, in which fluorine is reacted with NaCl and the chlorine gas is collected and analyzed.

The problem caused by using CaF_2 windows is that the CaF_2 absorbs light of wave lengths greater than 10 microns. In IR analysis, many important peaks are in the 10-15 micron range. Fortunately, many compounds associated with fluorine (HF, CF_4, and CO_2) do have absorption peaks below 10 microns from which they can be quantitatively identified (OF_2, SF_6, SO_2F_2, and SiF_4 are exceptions).

Whenever possible, NaCl windows are used. When fluorine is distilled from contaminants, the contaminants may be collected in the NaCl cells and the full range of the IR can be utilized.

The most serious problem in the use of IR cells is to get an absolutely leak tight seal between the windows and the metal cell body. Because of the corrosive nature of the fluorine, the seal supplied by the manufacturer often develops minute leaks. In many analyses, up to 1% HF was detected in fluorine using the manufacturers gaskets on the cells. (The HF comes from the reaction of atmospheric moisture with fluorine).

To improve the seal, various thin metal gaskets were tried, both amalgamated and unamalgamated, as a substitute for amalgamated lead, but without success. A considerable improvement in sealing the IR cells was made by increasing the thickness of the amalgamated lead gasket several-fold to 0.020 inches. The use of this gasket resulted in a seal which did not fail in 20 fillings with fluorine.

When leaks finally developed in the new gasket, it was replaced. A satisfactory seal was not obtained even after several gasket changes. It was then found that the seating surface of the IR cell had become rough. The seat was then carefully lapped smooth with lapping compound and the gasket was installed. This time the gasket was found to be leak tight.

Using the above technique the following possible contaminants in fluorine could be detected: CF_4, SF_6, SiF_4, HF, OF_2, COF_2, SO_2F_2, C_2F_6, CO_2. The major IR peaks occurred at the following wavelengths:

Contaminant	Major Infra-Red absorption peaks - Microns
HF	2.45 and 2.55
SF_6	10.55
SiF_4	9.7
OF_2	11.9, 12.1, and 12.25
SO_2F_2	11.4, 11.6, and 11.8
COF_2	8.1 and 8.2
CO	4.55 and 4.65
CO_2	4.22
CF_4	7.78
C_2F_6	7.98

REACTIONS OF FLUORINE ON METAL SURFACES

A series of experiments were conducted to study reactions of fluorine on metal surfaces. This work was designed to measure reactions between liquefied cylinder fluorine and metal specimens and then to study the effects of contaminants in the liquid or contaminants on the metal on these reactions. Specifically covered are:

1. Removal of traces of hydrocarbons from metal surfaces with gaseous fluorine, ClF_3, and mixtures of F_2 and ClF_3.

2. Short term immersion tests (15 and 75 minutes) to measure corrosion of metal specimens in liquefied cylinder fluorine.

3. Short term immersion tests (15 and 75 minutes) to study the effect of OF_2 in liquefied fluorine on corrosion of metal specimens immersed in the liquid.

4. Visual observation of the action of liquefied cylinder fluorine on metal surfaces.

1. Experimental Apparatus

A series of tests involving the reaction of liquid and gaseous fluorine on metal surfaces were conducted in an apparatus especially designed for these tests.

Several small monel cells (Figure 2) were built from 1-1/4 inch monel tubing with a 0.065 inch wall. A bottom blind flange was made of 1/4" monel sheet and drilled in the center for 1/4" tubing. A pair of matching flanges were made for the top of the cell and held together with eight stainless steel nuts and bolts. A rupture disc made of 0.005" copper sheet was bolted between the top flanges. The cell was drilled 1/4" from the top for 1/4" tubing. The top and bottom flanges and the tubing were all welded to the cell. The bottom-entering tube was used for introducing fluorine into the cell and the top tube was used as an outlet.

A series of these cells were set on a manifold so that fluorine could be introduced or removed, vacuum could be drawn, or nitrogen introduced for purging. Each cell was placed in a Dewar vessel which could be remotely filled with liquid nitrogen. All control was accomplished by valves with stems projecting through a 1/4" steel barricade.

Each cell could be divided into four compartments with a loose fitting cross-shaped insert. This enabled four small metal specimens to be simultaneously exposed and held erect in the cell.

Several cells could be used together for exposure tests, or run separately.

2. Removal of Traces of Hydrocarbons from Metal Surfaces

The purpose of these studies was to determine the efficiency of removal of liquid films of hexadecane from metal surfaces by exposing them to gaseous fluorine, chlorine trifluoride, or a mixture of both. It was expected that the hexadecane would be converted into CF_4 and HF, with the result that the metal would be cleaned and become protected by the formation of a fluoride film.

A series of small metal dishes 1" in diameter were made of brass, copper, aluminum, titanium, nickel, and monel in thicknesses of 0.001", 0.005", 0.010", and 0.062" and 0.125". Solutions of hexadecane in trichloroethylene were prepared in varying concentrations. Hexadecane was deposited on the metal surface by metering a small quantity of solution onto the dish and evaporating the trichloroethylene in a vacuum oven at 80° F. Oil films weighing from 1 to 60 milligrams and varying in thickness from 0.001 to 0.006 inches were deposited. The weight of oil deposited was determined by weighing the clean and dry dishes before and after deposition of the film.

The dishes were placed in the small monel cells described above, which were sealed and thoroughly evacuated. Fluorine, chlorine trifluoride, or mixtures of both were then introduced into the cell at pressures ranging from 1 to 5 atmospheres.

The extent of reaction of the hexadecane film with the gas was noted by pressure surges as the gas was added to the test cell. After a reasonable hold time, the gas was evacuated and the dish was weighed and inspected. In some instances, the dish was washed with known volumes of carbon tetrachloride, and the resulting solution was analyzed by infra-red techniques.

In general, removal of hexadecane films from metal surfaces was unsuccessful. Instead of the film being removed, most samples actually gained weight. Often a deposit of finely divided carbon was left on the surfaces when gaseous fluorine was exposed to the oil. Reaction was closest to completion at the high pressures when the thinnest metal dishes were used. The thin coupon would absorb the least amount of heat from the reaction of the gaseous fluorine with the hexadecane and the high pressure would favor a faster reaction.

When ClF_3 or mixtures of ClF_3 and F_2 were used, an oily deposit was observed on the metal dish. Infra-red analysis of this oil indicated its structure to be identical to Hooker Fluorolube oil MO-10. It was felt that the ClF_3 reacted with the hydrogen in the hydrocarbon to replace it with fluorine or chlorine, leaving the carbon skeleton intact. With fluorine gas, a waxy solid was

sometimes observed. On the thinner metal dishes, more reaction took place, with the degradation of the hydrocarbon to carbon, and with attack of the metal with the formation of metal fluorides. The infra-red analyses of the oily residues from these runs revealed that no hydrogen to carbon bonds remained.

The data are shown in Table III.

3. Short Term Immersion Tests

Previous results (13) from two-week and one-day immersion tests showed that weight changes of metals exposed to liquid fluorine are independent of immersion time. It was necessary to define a representative short exposure period during which the weight changes noted for metal specimens was the same as for longer periods. Experiments with titanium and magnesium (14) exposed to liquid fluorine for 15 minutes showed that titanium had a similar weight change in 15 minutes as in two weeks, while magnesium exhibited a varying weight change for the shorter exposure period. The current investigation was made to determine the behavior of aluminum, stainless steel, copper, and brass for the very short exposures.

Small metal strips of test materials were cut, polished, cleaned, dried, and weighed. The four compartment monel cell described above was passivated with gaseous fluorine. The test coupons were placed in the test cell which was then evacuated. Cylinder fluorine was condensed in the cells until the specimens were submerged in liquid. The specimens were exposed for either 15 or 75 minutes and the fluorine was evacuated. After purging the cell with dry nitrogen, the specimens were again weighed.

It was expected that the weight changes for exposure times of 15 minutes would be identical to weight changes for tests lasting 75 minutes, one day or two weeks. However, examination of results (Table IV) shows that many of the metals tested exhibited negative weight changes for the 15 minute exposure while the weight changes for 75 minute exposure were mostly positive and were similar to those reported for the one-day and two-week tests.

It must be noted that all weight changes are extremely small, the largest being 0.8 mg. Although the accuracy of an analytical balance is ± 0.1 mg, the "before" and "after" weighing had to be done on different days and this affected the accuracy of the weights. Also, most fluorides react with atmospheric humidity, and since the samples had to be exposed to the atmosphere for weighings, this may have affected the weights. This is the probable explanation for the small variations in weight changes for samples of the same material.

4. Effect of OF_2 in Liquid Fluorine on Corrosion

It was felt that certain contaminants in liquid fluorine could affect the corrosion rate of the liquid. The most reactive contaminant in liquid fluorine is oxygen difluoride, OF_2. It is miscible in liquid fluorine to the extent that at least 10% OF_2 in liquid fluorine forms a single phase at -320° F.

The tests to determine the effect of OF_2 were short term immersion tests identical to those just described, except that for the exposure tests purified fluorine contaminated with OF_2 was used instead of cylinder fluorine. The contaminated fluorine was prepared by adding OF_2 to purified fluorine in the gas phase as described previously by adjusting partial pressures of the two gases. Metals exposed to 1% OF_2 in fluorine included aluminum alloys, brass, copper, several types of stainless steel, monel, and nickel. Metals exposed to 10% OF_2 in liquid fluorine included brass, copper, type 304 stainless steel, and aluminum 6061.

Results of short term immersion tests for metals exposed to fluorine contaminated with 1% OF_2 are shown in Table V. The main differences between the results for samples immersed in liquefied cylinder fluorine and those in liquid fluorine contaminated with 1% OF_2 was that the weight changes of the samples were mostly negative for the samples in the contaminated fluorine both for the 15 and 75 minute exposure.

Careful comparison of data for short term exposure of metal specimens to both cylinder fluorine and fluorine contaminated with OF_2 indicates that weight changes for specimens exposed to fluorine contaminated with OF_2 are somewhat greater than weight changes for specimens exposed to cylinder fluorine. In most cases,

all weight changes border on the accuracy of weighings, but a definite trend is apparent. These weight changes represent corrosions of less than 0.1 mil of metal involved in the reaction. In some cases there are weight losses for immersion in cylinder fluorine and weight gains for immersion in OF_2-contaminated fluorine. For aluminum 2017 the reverse was true. In all cases, when specimens were immersed in fluorine contaminated with 10% OF_2, weight changes were positive. For copper and brass, these changes were much larger than for exposure to cylinder fluorine or fluorine contaminated with 1% OF_2.

5. Visual Observation of Action of Liquid Fluorine on Metals

As a continuation of work previously reported (14), samples of Aluminum 2017, Titanium A110AT, and yellow brass were placed in pyrex glass cells and immersed in liquid fluorine. Results from these tests were similar to earlier results. As long as the samples remained in the liquid fluorine, the surface appearance was bright and lusterous, indicating that little or no corrosion takes place in liquid fluorine. However, when the fluorine was being removed from the samples by evaporation, films could be seen forming when the evaporation was almost complete, and these reactions were always observed in the gas phase. White solids were present in liquid fluorine in all tests, but they did not appear to affect the corrosion behavior of the fluorine. The possibility exists that the solids deposit on the surface of the metal samples during evaporation and are somehow involved in the observed film formation.

C. FLUORIDE FILM STUDIES

The general non-reactivity of metals with liquid fluorine has been attributed to the formation of a protective metal fluoride film (4, 9, 11). The protective film is the important factor inhibiting corrosion at high temperatures (3, 6, 7), but its necessity at low temperatures, expecially at liquid fluorine temperatures, has not been demonstrated. Numerous tests have indicated that a protective film plays little or no part in the corrosion resistance of metals to liquid fluorine. It has been shown, experimentally, that film formation by gas exposure (passivation) prior to liquid exposure is not a requisite for liquid fluorine systems. Wire brushing of sample surfaces under liquid fluorine, tensile tear of specimens in liquid fluorine, and flexing and bending of metals in liquid fluorine all failed to produce any accelerated corrosion (13, 14).

The current experimental work was designed to approach the study of protective films from an analytical point of view. One approach was to detect the composition and thickness of fluoride films on corrosion samples by electron diffraction techniques. The second approach was the measurement of the gas-solid reaction between fluorine and metal powders to determine directly the quantity of fluorine tied up with a metal as a result of the passivation reaction.

1. Electron Diffraction Studies

A total of 36 samples of titanium and aluminum were exposed to fluorine under the following conditions: one hour gas exposure at ambient temperature and 1/2 atmospheric pressure; one hour gas exposure followed by six hour liquid fluorine exposure; six hours exposure to liquid without gas exposure. Twelve samples representative of each of the three treatments were prepared.

The surfaces of the samples were analyzed by electron diffraction using the reflection technique(1). From the "d" line spacings it was expected that both the composition and thickness of films would be obtained.

The first 18 samples were used to determine the optimum method of surface preparation prior to exposure. It was found that the surfaces of these samples were too smooth and that the formation of a characteristic pattern depends on a surface full of tiny ridges and valleys.

The second group consisted of aluminum metal strips polished with metallurgical paper by the proper technique, cleaned, dried, and stored in a desiccator prior to study. A portion of the samples were exposed to gaseous fluorine at room temperature, others were immersed only in liquid fluorine, while one was left in a desiccator as a blank and was not exposed to gaseous or liquid fluorine.

Results indicated that in all cases only diffraction patterns of $Al_2O_3 \cdot H_2O$ could be observed. No aluminum fluoride could be detected. It is possible that fluoride film whiskers could exist on metal but could be obscured by uniform oxide film over the whole metal surface.

Because of these inconclusive results, work was concentrated on the reaction of gaseous fluorine with metal powders.

2. Reaction of Gaseous Fluorine with Metal Powders

As a means of better understanding the reaction which takes place during the gas-phase passivation of metal surfaces, the reaction of gaseous fluorine with finely divided metals was studied. Experiments were designed to measure film thicknesses as small as 1 Angstrom and to follow film thickness formation as a function of time of exposure. The tests could be conducted at various temperatures and pressures with a variety of powders.

a. Experimental Apparatus

The experimental apparatus used for studying the reaction of metal powders with gaseous fluorine is shown in Figure 4. The important portion of the apparatus consisted of two volumes separated by a valve. The first volume was small compared to the second and consisted of copper tubing to which a pressure gauge was connected. The second volume included a copper coil and a monel sample holder. The first volume was manifolded to a line through which gaseous fluorine could be

introduced or removed. Included were provisions for a nitrogen purge and for evacuating the system. A 0-760 mm Hg absolute pressure gauge and a mercury manometer for calibrating the gauge were attached to the second volume. It was possible to isolate the gauge and/or the manometer from the system with valves.

For runs made above ambient temperatures, the system was immersed in a constant temperature bath equipped with a heater, temperature controller, and air agitator. Water was continually dripped into the bath and allowed to overflow so that the volume of the bath would not decrease by evaporation. A mercury-in-glass thermometer was used to measure the bath temperature.

For runs made below ambient temperature, the experimental apparatus was compacted to fit into a Dewar vessel. The coolant was either a dry ice-trichloroethylene mixture or liquid oxygen. Temperature was measured with a thermocouple. Temperature control was not necessary in this case,as the temperature of these two coolants is constant as long as there is sufficient dry ice or liquid oxygen in the Dewar.

b. Procedure

The system was designed so that the first volume could be brought to 50 psig pressure. When the valve separating the volumes was opened and the pressures were equalized, the pressure was slightly below 1 atmosphere absolute. The pressure in the system could be read to ± 1 mm Hg. When fluorine was used and a metal powder was present in the sample holder, reaction occurred,and the fluorine reacted with the powder to form a metal fluoride thus reducing the system pressure. The pressure reduction was converted to a fluoride film thickness by appropriate calculations.

The step by step procedure followed was as follows:

(1) The system was checked for leaks by pressurizing with nitrogen and soap checking all connections.

(2) The apparatus,less the metal powder sample, was passivated by filling with gaseous fluorine which was allowed to remain overnight.

(3) The system was evacuated and purged with dry nitrogen to remove last traces of fluorine.

(4) The sample holder was weighed, the metal powder added, and again weighed. It was then placed in the system, sealed, and leak checked by pressurizing with dry nitrogen.

(5) Vacuum was drawn on the system, and the sample holder was heated to about 450° F to drive off moisture.

(6) The system was placed in the constant temperature bath and "standardized". The standardization procedure involved running a blank test using nitrogen instead of fluorine. Nitrogen was introduced into the first volume to 50 psig and isolated from the manifold by closing a valve. The valve to the second volume was opened and the equalized pressure noted. This pressure was an indication of the "zero time" pressure to be expected in the system when fluorine was used. Knowing the liquid volume of the system, the weight (or moles) of gas in the system could be calculated. (The exact volume of gas is the liquid volume of the system less the volume of the powder in the sample holder.)

(7) The system was again evacuated.

(8) Fluorine was added to the system (same conditions as in step (6).

(9) Pressure readings were taken, and changes in system pressure with passage of time were noted.

(10) At the end of a run, the system was evacuated, and another 'nitrogen standard" was taken.

(11) The system was evacuated, and a "fluorine standard" was taken. If the system pressure remained constant, the equalized pressure was taken as the "zero time" pressure used in calculating film thicknesses. If pressure dropped, fluorine was allowed to remain in the system until no further pressure change could be noted, and the fluorine standardization was repeated.

(12) The system was evacuated and purged with nitrogen.

(13) The powder was removed and examined.

(14) The equipment was cleaned by dipping in dilute HCl, washing with water, and drying.

When runs were made at -297° F, slight modifications in procedure were necessary. The vapor pressure of fluorine at -297° F is approximately 12 psig, and when fluorine is introduced into the first volume of the apparatus at 50 psig, the fluorine condenses, and the quantity of material introduced cannot be calculated from the ideal gas laws. It was, therefore, necessary to introduce the initial fluorine charge into the liquid oxygen bath, allow 30 minutes for cooldown, and then open the valve between the volumes to equalize the pressure, which dropped to about 1/2 an atmosphere.

The quantity of fluorine consumed in the reactions can be calculated from the ideal gas law:

$$n = \frac{PV}{RT} \quad \text{gm-mol} \tag{1}$$

If the initial quantity of fluorine in the system is known, as well as the final quantity, the fluorine reacted is:

$$N_F = N_1 - N_2 = \frac{P_1 V_1}{Z_1 RT_1} - \frac{P_2 V_2}{Z_2 RT_2} \tag{2}$$

At low pressures, it may be assumed that $Z_1 = Z_2 = 1$. The system is held at constant temperature, so $T_1 = T_2$. Assuming that when fluorine reacts with a metal powder, the volume of the powder and the system do not increase; $V_1 = V_2$. The fluorine is further assumed to combine with the powder with the only result being a decrease in the system pressure, which is used to measure the quantity of fluorine reacted:

$$N_F = \frac{V_T (P_1 - P_2)}{R\,T} \tag{3}$$

The weight of fluorine reacted is: $W_F = N_F$ moles x 38 gm/mol

$$W_F = \frac{38\ V_T\ (P_1 - P_2)}{R\ T} \qquad (4)$$

where: N_F = gm-moles fluorine reacted

R = Universal gas constant

T = Absolute temperature

P = Pressure

V = Volume

W_F = Weight fluorine reacted

38 = Molecular weight of fluorine

At standard conditions, 38.0 grams of gaseous fluorine occupy 22,400 cc at 760 mm Hg absolute and 492° R so that:

$$R = \frac{(22,400)\ (760)}{(492)} \qquad (5)$$

Substituting in Equation (4)

$$W_F = \frac{(492)\ (38)\ (V_T)\ (P_1 - P_2)}{(22,400)\ (760)\ T} = 1.1 \times 10^{-3}\ \frac{V_T \Delta P}{T}\ \text{gm} \qquad (6)$$

where: V_T = Void volume of the system (water volume less the volume of the powder)

ΔP = Pressure reduction in the system caused by reaction; $P_1 - P_2$.

The film thickness in Angstroms may now be directly calculated:

$$d = \frac{M_{MF}}{M_F} \frac{10^8 \ \text{Å}/\text{cm}}{A\,\rho} (W_F) = \frac{(M_{MF})\,(W_F)\,(10^8)}{(M_F)\,(a)\,(W_s)\,(\rho)} \text{ Angstroms} \qquad (7)$$

where: d = Film thickness in Angstroms

M_{MF} = Molecular weight of metal fluoride formed by reaction.

M_F = Molecular weight of fluorine = 38 gm/gm-mol

A = Total area of the powder being tested in square meters = $a\,W_s$

a = Specific surface area square meters/gm

W_s = Weight of the powder sample - gms

ρ = Density of the metal fluoride gm/cc

The specific surface areas of the powders tested were determined by the B. E. T. method (2). The areas were very small and problems associated with the technique are discussed later.

Using monel powder as an example and substituting the following values directly into Equation (7).

a = 0.0165 M^2/gm

M_{MF} = (monel fluoride, 70% N_1F_2 - 30% CuF_2) = 98.1 gm/gm-mol (calculated)

ρ = 8.78 gm/cc

$$d\ \text{monel} = \frac{(98.1)\ (10^8)\ (W_F)}{(0.0165)\ (38.0)\ (8.78)\ (10^4\ \frac{cm^2}{M^2})\ (W_S)} =$$

$$9.4 \times 10^4\ \frac{W_F}{W_S}\ \text{Angstroms} \qquad (8)$$

Combining Equation (8) with Equation (6):

$$d\ \text{monel} = (9.4 \times 10^4)\ (1.1 \times 10^{-3})\ \frac{V_T\ \Delta P}{T\ \ W_S} = 103\ \frac{V_T \Delta P}{T\ \ W_S} \qquad (9)$$

Knowing V_T, T, and W_S, the film thickness may be calculated at any time during the reaction by noting the system pressure.

The sensitivity of any measurement depends on how well pressure is measured. The pressure gauge used was readable to ± 1 mm Hg and presented the limitation of measurement. Using 100 gms of monel as an example at a system of +86° F, with a system void volume of 130 cc, the sensitivity of measurement is:

$$d = \frac{(103)\ (130)\ (2)}{(546)\ \ (100)} = 0.49\ \text{Angstroms} \qquad (10)$$

The system is more sensitive at higher temperatures

at +183° F, the sensitivity is 0.42 Å

at -113° F, the sensitivity is 0.77 Å

at -297° F, the sensitivity is 1.97 Å

Based on the above sensitivities and other possible sources of experimental error, a run was considered reproducible if results agreed to within ± 1 Angstrom.

Several preparatory experiments were necessary to determine a convenient sample size and system volumes for these runs. Coils were introduced into the system to give appropriate volumes in the two parts of the system.

The accuracy of results depended on how well all of the terms in Equations (6), (7), and (8) could be measured. Molecular weights and densities could be gotten from standard handbooks and by appropriate calculations. Pressure, temperature, volume, and sample weights are measured by routine techniques. Measurement of surface area of powders was a more difficult problem.

The areas of the powders were measured by a standard technique called the B.E.T. method (2). It relies on the adsorption of molecular layers of gas on the powder surface. From the quantity of gas adsorbed and molecular thickness of the gas on the surface, the area of a sample can be calculated. The accuracy of results is improved as the surface area of the sample is increased. The lower limit of the method is accepted to be 1.0 M^2/gm. The areas of the samples used proved to be considerably smaller, and this makes the absolute values of the results subject to question.

It was desirable to check whether system pressure or sample size affected the results. All runs were made below 1 atm abs., so that should leaks develop, they would be into the system, rather than out. It was not known whether the fact that the pressure in the system was constantly decreasing during a run would affect the rate of film formation. To check this, several runs were made with monel powder at +86° F at system pressures ranging from 202 mm Hg abs to 541 mm Hg abs. Similarly, runs were made with samples of monel ranging from 54.2 gm to 137.0 gms to see if sample size affected results.

c. Results

Fifty-one runs were made to determine the fluoride film thicknesses on 10 metal powders at four temperatures. Metals studied included: aluminum, brass, copper, monel, nickel,

types 304, 316, 347, 310 stainless steels, and titanium. The temperatures investigated were +183° F (below boiling water), +86° F (above ambient), -113° F (dry ice in trichloroethylene), and -297° F (liquid oxygen). Film thicknesses were small and were in all cases less than 200 Å. Table VIII summarizes results of all runs made. Figures 6 through 10 compare film thicknesses on 10 metals at each of the temperatures. Figures 11 through 15 compare the films formed on each powder at different temperatures.

Results with monel powders indicated that the film thickness formed in one hour was approximately the same regardless of the temperature of the run. At initial system pressures of 541 mm Hg abs and 312 mm Hg abs, results were identical (Figure 1). At 202 mm Hg abs there was less reaction between the monel and gaseous fluorine than at higher pressures. Using samples weighing 81.25 gm to 137.0 gm, results were comparable, but with a 54.2 gm sample, the film formed appeared greater than for larger samples.

Tables IX and X summarize the films formed on the 10 metal powders in one hour, 2.5 hours, and at the end of the run for various temperatures.

D. EXTENDED IMMERSION OF TENSILE SPECIMENS IN LIQUID FLUORINE

Numerous tests have been conducted to determine the corrosion rate of metals in liquid fluorine and results indicated that the corrosion rate was extremely small. The next step was to determine the effect of liquid fluorine on the mechanical properties of metals. The experiments in this section were designed to compare the changes in mechanical properties of metals immersed in liquid fluorine for one year to similar specimens immersed in liquid nitrogen for the same period by tensile testing small samples after exposure.

1. Experimental Apparatus

An apparatus was designed to hold 60 tensile specimens under liquid fluorine for one year in a leak-tight system. The liquid fluorine container was made from 2-4" nominal diameter schedule 40 stainless steel pipe caps and a short length of pipe (Figure 19) welded together. A nozzle of 1" stainless steel pipe was welded to the top of the sample container. Two 1/4" monel valves with metal-to-metal

seals were "teed" together and connected to the nozzle with 1/4" stainless tubing welded to the side. These values isolated the container from a series of manifolds (Figure 20) which could be used for introducing fluorine, removing fluorine, introducing nitrogen or helium, or evacuating the container. The system was protected by a 400 psig copper rupture disc in the nozzle of the container in a line which exhausted to a 20 foot deep lime pit used for fluorine disposal.

Special tensile specimens were machined from 1/4" rod to fit into the fluorine container (Figure 21). A special rack was made to hold the specimens upright in the container (Figure 22). The holes in the rack were drilled so that the liquid fluorine could easily flow between the rack, container, and specimens. Each hole was numerically coded with hole no. 1 identified by a punch mark so that the specimens could be identified by their position in the rack.

In order that a large number of specimens could be tested in one relatively small apparatus requiring a nominal quantity of liquid fluorine, the specimens had to be made smaller than standard specimens. Special adapters made from 3/4" bolts were made to allow the special specimens to fit into a standard tensile test machine. Each specimen was necked down by 0.001 inch in the center so that it would break at this point (Figure 21).

The liquid fluorine container was placed in a large flanged Dewar vessel. The nozzle of the container was welded to the cover flange of the Dewar to hold it upright and centered in the Dewar. The Dewar was placed in a large box insulated with glass wool and located behind an oak barricade. The liquid level in the Dewar was measured and controlled by a differential pressure controller which opened a solenoid valve to introduce liquid nitrogen when the level dropped below the top of the specimens in the container. With liquid fluorine in the container and liquid nitrogen in the Dewar, the pressure in the container was the vapor pressure of liquid fluorine at -320°F.

During the test period, 60 tensile test specimens consisting of 5 samples of 12 metals were kept immersed in liquid fluorine in the container, and 60 were kept in liquid nitrogen in the Dewar. The twelve metals studied included: 304 and 410 annealed stainless steels, hi-strength steel-Armco Ph 15-7 Mo annealed, hard electrolytic copper, types 1100-1114 and 6061-T6 aluminum, nickel, monel, types A 110 AT and C 120 AV annealed titanium, and type AZ-31 and HK-31 magnesium.

2. Experimental Procedure

All components in the system, including the tensile specimens, were thoroughly degreased, cleaned, and dried. All but the specimens were passivated in gaseous fluorine before being exposed to liquid fluorine. The samples were carefully weighed and placed in assigned spaces in the specimen rack. They were finally placed in the container and the cap was welded to the vessel. The system and piping were assembled in place and all joints were leak tested. When no leaks could be detected, the Dewar cover flange was bolted down and the Dewar was insulated. From this point on, all operations took place from behind the barricade using "peep holes" and long stem valves for observation and control.

The entire system was thoroughly purged with dry nitrogen and finally evacuated. On August 29, 1960, the Dewar was filled with liquid nitrogen and five pounds of fluorine were slowly condensed in to the fluorine container. The fluorine cylinder from which the fluorine was taken was weighed before and after filling the container to determine the weight of fluorine added.

The liquid nitrogen level in the Dewar was checked daily and additional liquid was added to keep the Dewar full. A complete daily record was kept of the liquid level in the Dewar and the pressure in the fluorine container to assure that the rupture disc remained intact and that the system had not developed leaks. The one year test period passed without incident and with a minimum amount of attention.

On August 29, 1961, the anniversary of the initiation of the test, the liquid level in the nitrogen Dewar was allowed to drop. Dry nitrogen was blown through the liquid to hasten evaporation. As the liquid level dropped, the fluorine container began to warm and

the pressure slowly increased. When the pressure in the container reached 7 psig, the discharge valve to the lime pit was cracked open. A careful record was kept of the exact position of the discharge valve to maintain 7 psig in the fluorine container during the time of disposal.

After about 24 hours, the pressure in the container could no longer be held at 7 psig and the fluorine was assumed to have vaporized. The container was purged 5 times with dry nitrogen by pressurizing to 15 psig and bleeding off the pressure. Finally, vacuum was drawn on the system.

At this point, the rupture disc on the container failed. Water which had accumulated in the lime disposal pit after a rainfall was drawn into the container by the vacuum pump. As soon as this happened, the vacuum pump was turned off, the fluorine container was removed from the system, the container was opened, the samples were removed, dried, and weighed.

With the fluorine container removed from the system, a nitrogen line was tied to the valve through which the fluorine had been bled from the system. The valve was then calibrated by measuring the flow of dry gaseous nitrogen through the valve at different positions. The nitrogen calibration was used to calculate the total volume of fluorine that had been bled from the system. As well as could be determined by this measuring technique, sufficient fluorine was present in the system at the conclusion of the run to have immersed the specimens.

3. Results

The tensile specimens which were immersed in liquid fluorine were weighed in the "as-is" condition and again after thorough cleaning. These data are shown in Table XII for each specimen and summarized (by average) in Table XIII.

In all cases, the corrosion rate was less than 1 mil in one year (based on weight difference after cleaning). Foreign, hairlike growths were found on some specimens, probably caused by electrolytic action and these were cleaned from the specimens

with a toothbrush and soapy water before final weighing. The corrosion rates reported are only approximate as cleaning of the specimen threads was difficult and traces of matter may have remained in some cases. Magnesium AZ-31 corroded most. The corrosion was less than 0.01 mils in one year for 304 stainless steel, Armco PH 15-7 Mo, nickel, monel, and copper. These data indicate that there is negligible corrosion for the 12 metals tested in liquid fluorine.

The 120 tensile specimens were tested on a standard tensile test machine with a Tinius Olson Stress Strain Recorder by Lehigh University (Fritz Laboratory). There were no significant differences in mechanical properties between samples immersed in liquid nitrogen and liquid fluorine. Long term exposure to cold temperature (-320° F) appeared to improve tensile properties over the handbook values. The data showing tensile properties of each of 120 specimens as submitted by Lehigh University are presented in Table XIV. Results are summarized (by average) in Table X V.

III. DISCUSSION OF RESULTS

A. FLUORINE CONTAMINANTS

The contaminants present in fluorine include gaseous products generated in the electrolytic manufacture of fluorine and those resulting from the reaction of fluorine with various equipment and system contaminants. HF and OF_2 are produced from moisture, SiF_4 from glass, SF_6 and SO_2F_2 from impure hydrogen fluoride, CO_2 and COF_2 from impure electrolyte, and CF_4 from carbon electrodes. SiF_4 may have been generated in a glass collecting apparatus and when a monel cell was substituted for glass, the SiF_4 content of fluorine analyzed decreased sharply.

After some difficulty in eliminating leaks from the purification system and analytical apparatus, contaminant-free fluorine was prepared. It is now felt that on a large scale, hyper-pure fluorine may be routinely prepared. Gaseous fluorine should be passed through a sodium fluoride scrubber, condensed at liquid nitrogen temperatures, filtered, and collected. The distillation of the fluorine is necessary to remove soluble contaminants and non-condensables including nitrogen and oxygen if these are objectionable. It is of primary importance that the purification system be leak-free.

It is more practical to produce pure fluorine than to purify contaminated fluorine. This may be accomplished by using pure, dry electrolyte in the fluorine cell, adding pure HF to the electrolyte when make-up is required, and keeping the fluorine handling system clean.

Most contaminants are insoluble in liquid fluorine except for OF_2 and CF_4. Carbon tetrafluoride is an inert material but OF_2 is fairly corrosive and is a potential oxidizer. At the time of these tests, OF_2 was not commercially available so the material was prepared in the test units by the method of Lebeau and Damiens (7) and elaborated on by Ruff and Menzel (8). The material was of high purity but the exact analysis was not determined because a sample with known composition was not available for comparison. The material must be handled carefully, especially as a gas at high pressure (200 psi).

The solubility of HF in liquid fluorine was measured to be less than 0.5% by gas blending and condensation. However, it has been found that less than 0.2% HF in fluorine will precipitate from the liquid. In all probability, the solubility is much lower. Traces are found in almost all fluorine. It forms when traces of moisture contact gaseous fluorine. It is not corrosive in liquid fluorine or at temperatures where it is a solid (freezing point = -118°F), but in the liquid or gaseous state it causes severe corrosion. Most of the corrosive action of fluorine may be attributed to the presence of HF in gaseous fluorine, to moist surfaces contacted by gaseous fluorine, or gaseous fluorine allowed to contact atmospheric moisture.

B. REACTIONS WITH FLUORINE ON METAL SURFACES

Visual observation of metals in liquid fluorine indicated that the fluorine does not attack metal surfaces. The presence of solid contaminants also does not cause corrosion. However, when the fluorine is vaporized and the samples warm, the solids migrate to the metal surfaces and cling much as a ring forms around a bathtub. Once on the surface and warmed so that they vaporize, they seem to react with the metal to form a film. Corrosive materials, such as HF, would be most harmful. These observations show that metals do not react with liquid fluorine, even if contaminants are present, and explain why the corrosion rate of metals in liquid fluorine is extremely low, even after prolonged exposure. However, if a metal is subjected to cycles of liquid and gaseous fluorine, the gas phase corrosion could become a problem if contaminant-free fluorine is not used.

The removal of oily films from metal surfaces by exposing them to gaseous fluorine or ClF_3 is not a satisfactory means of passivating a dirty surface prior to exposing it to liquid fluorine. Reaction between the gas and oil is incomplete and traces of carbon or fluorinated oil remain. These may be relatively inert to gaseous fluorine but could react explosively with the liquid (13).

Short term immersion tests of metal samples in liquid fluorine indicate that, in most cases, the metals tested exhibited negative weight changes for very short immersion times, while for the longer periods of exposure weight changes were positive. A possible explanation for the weight loss

is the initial formation of a fluoride film accompanied by dissolution of this film in the liquid fluorine. Electrical conductivity measurements of liquid fluorine saturated with various fluoride salts showed a very low conductivity (13 & 14) indicating that either the fluorine solutions of fluoride salts are non-ionic, or, if solutions are partially or totally ionic, the number of ions present is small. If non-ionic solutions are formed, solubility will be expected to be low, as illustrated by Hildebrand's solution equation:

$$\ln a = \frac{H_f}{R}\left[\frac{1}{T_m} - \frac{1}{T}\right] \tag{11}$$

where:

a = mole fraction of salt in a saturated solution

H_f = heat of fusion of the salt

R = universal gas constant

T_m = melting point of the salt (abs units)

T = system temperature (abs units)

Typical values for these variables are:

H_f = 2000 cal/gm mol

R = 2 cal/gm mol deg K

T_m = 1300° K

T = 78° K

substituting in Equation (11)

$$\ln a = \frac{2000}{2}\left[\frac{1}{1300} - \frac{1}{78}\right] = -12.0641$$

$a = 5.78 \times 10^{-6}$ mole fraction of salt in a saturated liquid fluorine solution.

The dissolution of a surface film of metal fluoride would be halted in a short period of time due to saturation of the liquid fluorine. This would account for the negative weight changes observed for very short exposure times. When the fluorine became saturated, the protective film could grow to some limiting thickness which would account for positive weight changes observed for longer exposure times. The above calculated value of "a" may be compared to the experimental value by considering the case of a typical metal exposed to liquid fluorine for 15 minutes. A typical weight loss for a sample is -0.1 mg or 1×10^{-4} gm. The volume of liquid condensed in the cell is about 33 cc. The density of liquid fluorine is 1.5 gm/cc, so the weight of condensed fluorine is roughly 50 gm corresponding to 1.3 gm-moles. The number of gm-moles of metal dissolved is 2×10^{-6}. The mole fraction dissolved is $2 \times 10^{-6}/1.3 = 1.5 \times 10^{-6}$, which is the same order of magnitude as the above calculated value. Based on the limited accuracy of these sample calculations, it can be seen that trace quantities of metal fluoride may have dissolved in the liquid fluorine. However, the weight changes involved in these experiments are so small that it is difficult to determine whether the changes were caused by corrosion or by inaccuracies in weighing.

The increase in weight changes for metal samples exposed to OF_2 over the weight change for similar specimens exposed to cylinder fluorine suggests that OF_2 in liquid fluorine accelerates corrosion. This increase is very small and may have been caused by inaccuracy in weighing or other uncontrolled conditions rather than by corrosion. It is evident that short term immersion of specimens in fluorine contaminated with up to 10% of OF_2 will not present a serious corrosion problem. However, until exposure tests of longer duration are run to verify this observation, it is advisable to avoid the use of OF_2-contaminated fluorine.

C. FLUORIDE FILM STUDIES

1. Electron Diffraction Studies

The reason that electron diffraction techniques could not be used to measure fluoride film thicknesses is that the limit of this technique is about 30 Å. Fluoride films on aluminum are usually much smaller. The fact that an oxide film was observed instead of fluoride film

indicates that the "protective fluoride film" formed on metal surfaces, protects the metal from further attack by fluorine, but does not protect the surface from other agents. Metal fluorides tend to hydrolyze and absorb moisture. This action often converts the film to an oxide and liberates HF. The film is so thin that little water need be contacted with it to destroy it. Atmospheric humidity usually is adequate to do the damage as was demonstrated in tests with metal powders (discussed later) and the time of contact required is small. It becomes a necessity to measure fluoride films without allowing the atmosphere to contact the film which almost necessitates making measurements in situ in the apparatus where the film is formed. The problems involved in selecting an instrument to measure fluoride films from 1 to 200 Angstroms in situ are many. The studies of fluoride film formation on metal powders appeared to be the most practical approach to the problem.

2. Reaction of Metal Powders with Gaseous Fluorine

The primary observation from this study is the low thickness of the apparent fluoride films formed on metal powders. Most of the film formed very rapidly in the first few minutes of exposure. In one hour the reaction rate dropped to a small fraction of the rate of the first minutes. The rate thereafter remained almost constant for several hours and after one day was barely detectable.

The above behavior of metal powders was observed at temperatures of +183° F, +86° F, and -113° F. The film thickness calculation was based on pressure change with time as fluorine reacted with the metal. Because reaction between metal powders and gaseous fluorine is extremely rapid during the first seconds of exposure, the zero time pressure was determined at the end of a run, after reaction with the powders had ceased. The zero time pressure was called the "fluorine standard" and was obtained by exact duplication of conditions of the run, but without there being any reaction between the powder and the fluorine. "Nitrogen standards" were also taken before and after the passivation and agreed with the fluorine standard to within 1%.

The behavior of metal powders at -297° F was different than their behavior at higher temperatures. All evidence indicates that adsorption of gas on metal powder surfaces at -297° F takes place. When the gas standards were taken at -297° F, the system pressure dropped for about 30 minutes and remained constant. The pressure drop with time for the ten metals studied is presented in Figures 16 through 18.

It can be seen that the gas standardization steps and the reaction step are closely parallel curves. The system pressure drops as gas is adsorbed on the metal powder. The pressure drop caused by reaction between fluorine and metal powders may be calculated from the difference in pressure of the fluorine standard at any time and the corresponding pressure of the system during reaction. Film thicknesses at 15, 30, and 60 minutes have been calculated in this manner and are presented in Table XI. It can be seen that in some cases negative film thicknesses were calculated and in some cases the film thickness grew smaller with time. These results represent an unreal situation. They are caused for the following reasons:

a. The effect of adsorption is much larger than the effect of reaction and obscured the extent of reaction.

b. The results are based on small differences between large numbers which are variable and changing with time.

c. The sensitivity of measurement is low at low temperatures.

d. The adsorbed gas may not have been completely removed from the metal powders after the first adsorption.

e. Apparently "zero time" conditions were not duplicated with sufficient accuracy.

Based on results of experimental work, it may be concluded that if any reaction occurs between gaseous fluorine and metal powders at -297° F, it is very small and does not continue beyond the first 30 minutes of exposure.

It should be noted that in Run 64, titanium did react chemically with gaseous fluorine. When 40 gm of titanium powder were reacted with gaseous fluorine at -297° F and an initial pressure of 302 mm Hg abs, the pressure dropped to 2 mm Hg abs in seven minutes. This pressure drop was due to chemical reaction and not due to adsorption of gaseous fluorine on the metal surface.

The measurement of the surface areas of metal powders was, perhaps the most critical phase of these experiments. The Brunauer-Emmett-Teller or B.E.T. method (2) was used for this determination. Although this procedure is widely used for area determination, its useful range is 1 to 1000 square meters per gm of sample. The accuracy is greatest for largest areas.

In the case of metal powders used in these experiments, the measured areas were in all, but one case, less than 1 m^2/gm and in 2 cases less than 0.1 m^2/gm. In this range of areas, small inaccuracies appear magnified and the reproducibility of the results becomes questionable.

It was felt that measuring surface areas of metal powders before and after reaction with gaseous fluorine would give some indication of whether the surface had been affected by the fluorine. Results of these measurements indicated that there was a wide variation in the "before" and "after" exposure areas. Since the cause of this variation was not exactly known, it was decided to use a "standard" area for calculation and comparison purposes. These "standard" areas are listed in Table XVI and were determined from measurements made with fresh samples before exposure to fluorine. It must be noted that the areas are the result of a single measurement. In using these "standard" areas for calculations, it was assumed that (1) the measurements were correct, and (2) the samples taken were from the same container were uniform and representative of the entire contents of the container.

An independent study of the limitations of the B.E.T. method for area determinations indicated that measurements on metal powders would yield only order-of-magnitude results. The differences in "before" and "after" exposure areas probably occurred because the method was not sensitive enough to give accurate results. The experimental

results of area measurements to determine film thicknesses on metal powders supports this conclusion. The variation in "before" and "after" results was not caused by fluorine affecting the areas. The areas of two independent samples of monel powder from the same container were measured in the fresh condition. Results differed by 32%. Non-uniformity of the powder did not cause this variation. This was demonstrated in 8 runs made with monel powder. Using a "standard" area to calculate film thickness on monel, the 1 hour films were in most the same thickness even though the runs were made with different sample weights at different temperatures and pressures. These results could not have been achieved if the samples tested were not uniform. Area measurements on monel samples varied from -32% to 120% of the "standard" area. Using the "standard" area, results of film thickness measurements are comparable even if not precise.

A list of "standard" areas is presented in Table XVI and a list of "after exposure" areas is presented in Table XVII along with percent deviation from "standard" areas.

The appearance of metal powders after exposure to gaseous fluorine showed, upon careful examination under a microscope, that they were generally less light-reflective and darker than fresh samples. It was quite difficult to differentiate between exposed and fresh samples, even when they were placed side by side. Aluminum powders had a whitish tint after exposure which may have been due to fluoride or oxide films. Exposed copper samples were somewhat darker than fresh samples. Copper powder exposed at +183° F had a greenish tint. This sample was stored in a glass jar which later became badly etched, presumably by HF release from the powder. A copper powder exposed at -113° F turned purple. Some powders were caked upon removal from the sample cell. In some instances the powders had to be scraped from the cells and this usually removed metal fluorides which had formed on the walls of the monel sample holder.

In early runs, microscopic examination of selected powders removed from the sample cell after exposure to gaseous fluorine indicated the presence of large crystals assumed to be fluorides. These crystals were of various colors and did not necessarily correspond to the color of the fluoride of the metal being studied. Suspecting

system contamination to be present, a small micron stainless steel Hoke filter was installed just ahead of the sample holder. Subsequently, no crystals were found in the exposed powders indicating that fluorides formed in the system had fallen into the powder samples. There was never positive identification of fluoride crystals in the exposed metal powders, probably because the films formed were so thin that the crystals formed were too small for microscopic identification.

It has been noted that any significant reaction between gaseous fluorine and metal powders ceases after one or more days of exposure. Upon initial exposure to gaseous fluorine, reaction is too rapid to note the "zero time" system pressure. A "fluorine standard" was established to determine this pressure. The "standard" was obtained after a sample was exposed to fluorine for a day or more after which time the system pressure varied by 2 mm Hg or less per hour. The fluorine was evacuated from the system and the sample was re-exposed to the same quantity of fluorine which had been used in the previous reaction. This was done without opening or otherwise disturbing the system. The "fluorine standard" agreed within 1% of a "nitrogen standard".

Several runs were made to determine the durability of the protective fluoride film formed on metal powders after their exposure to gaseous fluorine. Samples which had been exposed to fluorine were removed from the sample holder for examination After about one hour of exposure to the atmosphere they were returned to the sample holder and again exposed to fluorine under the same conditions as previously. In all cases, these samples, which had been inert to the gaseous fluorine while taking the "fluorine standard" reacted with the fluorine. During the second reaction, less fluorine was consumed than during the first reaction, but the reaction was sufficiently large to indicate that the protective film was not protecting the powder. The quantity of fluorine consumed during first and second exposures of selected samples is compared in Table XVIII.

A possible explanation of the reaction of exposed samples with fluorine was that during the first exposure the fluorine gas was contacting only a portion of the powder and removal of the sample,

accompanied by agitation, exposed fresh surfaces. To determine whether agitation of the samples exposed fresh surfaces, the sample holder was removed from the system at the conclusion of a run and agitated without removing the sample. When the sample holder containing the powder was returned to the system and again exposed to fluorine, there was little reaction. This observation lead to the conclusion that agitation alone was not responsible for reaction of exposed powders with fluorine. The major factor causing the second reaction was expected to be atmospheric moisture reacting with the exposed powders when they were removed from the sample cell.

A test was made to determine the extent of moisture adsorption by metal fluorides. A metal fluoride powder, MgF_2, was selected for study. This material was expected to be inert to gaseous fluorine. By exposing it to fluorine, the moisture on the powder would react to passivate the powder. When reaction ceased, the MgF_2 could be exposed to the atmosphere and allowed to adsorb moisture. Re-exposure of the powder to fluorine would allow this moisture to react and the fluorine consumption would be a measure of the moisture pickup.

The MgF_2 used for this experiment had an initially high moisture content. The moisture was finally removed after heating the sample with an oxy-acetylene torch and repeatedly exposing it to fluorine when there was no reaction between the MgF_2 and fluorine, the powder was removed from the sample holder and exposed to the atmosphere in the same manner as had been done with metal powders. When the MgF_2 was again exposed to fluorine, several times volume of the system was required to passivate the sample, indicating that the MgF_2 had picked up a large quantity of atmospheric contamination.

A rough check on the water absorbing tendency of metal fluorides was made by heating samples of MgF_2, $CuF_2 \cdot 2H_2O$, and $NiF_2 \cdot 4H_2O$ to 500° F to remove moisture, weighing them, and leaving them exposed to the atmosphere overnight. The $CuF_2 \cdot 2H_2O$ gave off HF fumes upon heating and was converted to CuO. The other samples gained weight after overnight exposure to the atmosphere. The hygroscopic nature of metal fluorides was thus confirmed.

It was concluded from these observations that the fluoride film formed on metal powders is hydrolyzed and destroyed by exposure to atmospheric humidity. Re-exposure to fluorine results in reaction of the moisture with the fluorine and the formation of a new film. Since the damage to the film by humidity is not instantaneous, all of the film was not destroyed when the powders were examined and the quantity of fluorine reacted upon second exposure depended on the extent of deterioration of the film.

It has been reported earlier (13, 14) that moisture in a fluorine system accelerates corrosion. The fluorine reacts with water to produce HF, which is highly corrosive. When samples immersed in liquid fluorine are warmed to room temperature, there is a gas phase reaction which causes the shiny surface to become dull. This same reaction may occur if metal powders are exposed to gaseous fluorine which is contaminated by HF. The fluoride film formed would be caused by the reaction with HF rather than reaction with fluorine. At -297° F, HF is solid and does not react with metals. The temperature of -113° F is close to the freezing point of HF (-118° F) and little reaction is expected between the HF and metal. At +86° F and +183° F the temperature is above the boiling point of HF (67° F) and HF becomes a highly corrosive gas.

Moisture in the system comes from two sources, moist samples and leaks. To assure that the samples were dry, they were heated to 450° F while vacuum was being drawn on the system. The system was leak-checked before each run and it was usually possible to maintain the system at less than 100 microns when drawing vacuum. Nevertheless, there was the possibility that minute leaks were present or could develop over the period of the runs which lasted a day or more.

In several runs, analysis of residual gas in the system after a sample had been exposed to fluorine showed large percentages of HF to be present. The presence of HF may be explained by calculating the small quantities of water in the system which would be required for the production of HF. Since the apparatus is submerged in a water bath, water would enter the system if there were any leaks. Assume the following reaction:

$$F_2 + H_2O = 2HF + 1/2\ O_2 \qquad (12)$$

In a system with a void volume of 130 cc, an initial fluoride pressure of 600 mm at 86° F, the quantity of fluorine present is, from Equation (6):

$$W_F = \frac{(1.1 \times 10^3)\ (130)\ (600)}{546} = 0.157 \text{ gm } F_2 \qquad (13)$$

$$= 0.00413 \text{ gm moles } F_2$$

Based on Equation (6), let x = number of moles of fluorine which react with water. Assume all water is reacted to produce HF + O_2. On the basis of 1 mole of fluorine at the start, there are 1 - x moles of F_2 remaining after the reaction 2x moles of HF and 1/2x moles of O_2. The final quantity of gas is 1 + 1.5x moles of gas. The ratio of HF in the gas is 2 x / (1 + 1.5x). If this ratio is 10% (0.1) HF

$$\frac{2x}{1 + 1.5x} = 0.1 \quad \text{and} \quad x = 0.054 \qquad (14)$$

or 5.4% fluorine is required to react with water to produce 10% HF in the final gas. In most cases, 50% of the fluorine charged reacts with the metal powder. For the remaining gas to analyze 10% HF, the quantity of water necessary for reaction with fluorine is:

$$W_{H_2O} = (0.054)\ (0.5)\ (0.00413)\ (18) = 0.002 \text{ gm or ml} \qquad (15)$$

It is possible that this small quantity of water could enter the system during a run without being detected. The residual gas analysis noting the quantity of HF present is indicated in Table VIII. In many instances it was impossible to get residual gas analyses because the IR sample cells were being repaired to eliminate leaks.

The fact that little or no HF is present in the residual gas does not mean that HF was not produced during the reaction. It is possible that HF did form but reacted with the metal powder and was mostly consumed.

There may have been several simultaneous effects in the system which affected results:

a. Pressure drop due to reaction of fluorine with metal powders.

b. Pressure rise due to reaction of moisture with gaseous fluorine.

c. Pressure drop due to reaction of HF with metal powders (expected between stainless steels and HF).

It has been noted that the pressure drop in the system is most predominant. If reaction between water and fluorine takes place, the calculated film thickness is too small. Despite the possibility of side reactions, the effect of HF on the film thicknesses had to be neglected in the calculations because of the following reasons:
It was impossible to measure:

a. The rate of HF formation in the system

b. The extent of reaction of HF with the powders was unknown

c. The extent of reaction of HF with system components

d. The source of HF (i.e. whether it was formed prior to a run, during a run, or during sampling of residual gas for analysis).

There appears to be no correlation between the behavior of metal powders with gaseous fluorine at different temperatures and this may be attributed to the effects of HF on the experiments. The effect of HF on system pressure may be considered by re-examining Equation (14).

Let F = fraction of fluorine reacted to yield HF. Rewriting Equation (14)

$$\frac{2x}{1 + 1.5x} = F \qquad (16)$$

or

$$x = \frac{F}{2 - 1.5F}$$

From the percent HF in the residual gas, it is possible to calculate how much fluorine reacted to form HF. The effect on the system pressure is that the pressure rises by 1.5 times the fluorine consumed. Taking the example of 10% HF in residual gas, 5.4% of the fluorine reacted and the system pressure rose by 8.1%. In a system with 600 mm Hg abs initial pressure with 50% of the fluorine reacted with metal powder, the pressure change due to HF formation is

$$600 \times 0.5 \times 0.054 \times 1.5 = 24.3 \text{ mm Hg} \qquad (17)$$

From Equation (10), a 2 mm error in pressure reading resulted in an error of 0.49 angstrom. In this case the error caused by 10% HF in the residual gas is about 6 angstroms. The effect of HF depends on the quantity of fluorine reacted, the residual system pressure, the precent of HF in the residual gas, and the reaction temperature. While the comparison of films formed at different temperatures may not be good for the powders studied because of differences between numbers, the order of magnitude of results is certainly valid. It may be seen from Table XVIII that the films formed on the powders were of the same order of magnitude. There is more variation among the stainless steels as they are more susceptible to HF attack than the other metals studied. More precise results could be obtained if additional studies were made.

In general, all the metals studied are satisfactory in corrosion resistance to gaseous fluorine. Judgment of superior corrosion resistance cannot be based on these tests. The fact that one metal forms less film in one hour than another does not indicate superior corrosion resistance since the film may continue to form indefinitely. There is also no basis for making corrosion comparisons for materials where only a few surface molecules react with the corrosive medium.

In discussing the ten metal powders studied, it is convenient to group them according to their physical nature and point out similarities in their chemical behavior.

Aluminum and Titanium: These metals are useful because of their high strength to weight ratio.

At -113°, aluminum formed a film of 6.5 Å in one hour which increased to 163 Å after 118 hours. This unusually larger increase in film thickness after 5 days of exposure indicates that the film was not entirely protective and allowed the reaction to continue. This behavior at -113° F is similar to that for monel and nickel. At +183° F, the test apparatus developed a leak too small to detect by soap-testing or vacuum testing (by holding system vacuum). Yet, after the initial 101 minutes of exposure, the system pressure began to rise rather than fall. The residual gas contained 75% HF which could only have come from water leaking into the system. At 86° F. the one hour film of 4 Å increased to 139 Å after 4 days, contrasting the reaction at -113° F.

Titanium powder reacts violently with gaseous fluorine. While it has been demonstrated that solid metal test strips of titanium will ignite in liquid fluorine, the reaction is not self propagating. This is not the case with metal powders in gaseous fluorine. When reaction was started between titanium and gaseous fluorine at +86° F, -113° F, and -297° F, the reaction did not stop until most of the fluorine was consumed. The reaction at +86° F was explosive and at other temperatures less violent. Duplicate runs were made with titanium and gaseous fluorine at -113° F and -297° F using smaller samples than had been used in the cases where all the fluorine had been consumed. With small samples, titanium behaved much as other powders. This observation leads to the conclusion that with large samples, there is sufficient heat of reaction to cause the reaction to continue. With small samples, the cooling effect of the constant temperature bath removes most of the heat of reaction rapidly enough to slow the reaction. After the initial few seconds, a protective film has formed on the titanium which prevents further violent reaction. It is expected that the larger the area per unit volume of titanium and the poorer are conditions to draw away heat of reaction, the more likely is a sample to burn in gaseous fluorine. At these worst conditions, it is expected that the reaction between fluorine and metal will continue until either is consumed. It is not anticipated that this type of reaction could occur with ordinary structural members made of titanium because the area to volume ratio is small.

Brass and Copper: These metals are useful for cryogenic service, are inexpensive and readily available, and easy to join using ordinary soldering or brazing materials.

The behavior of brass is influenced by its copper content. Very small films formed on brass. The maximum one hour film was 5.6 Å at +86° F reaching 11 Å after three days. In a duplicate run at +86° F, the one hour film was 3.3 Å and 10 Å after two days, indicating that results with this powder are reproducible. Yellow brass was used for runs at -113° F and +86° F after which the supply was exhausted. Red brass was used for runs at +183° F and -297° F. Because of dissimilarities of the two samples and inaccuracies in area measurements, results with different samples are not comparable. However, the tendency of this metal to form very small one hour films which do not increase appreciably with time is evident from the results.

Copper showed similar behavior to brass when exposed to gaseous fluorine. The films formed on copper were thicker than those on brass. Runs were considered reproducible at +86° F with the curves showing film thickness formation with time almost parallel for four days of exposure. At all temperatures, the film thickness at the end of the runs were 2 to 4 times the one hour films indicating little appreciable increase in film after long exposure.

Both copper and brass have been successfully used in gaseous fluorine service. Their resistance to HF is good and an important consideration in their use.

Monel and Nickel: These metals have excellent corrosion resistant properties and are suited for use at cryogenic temperatures.

Monel powders were studied more than all other metal powders. Results were found reproducible in that the film thickness after three hours was 9 ± 1 Å at different temperatures, pressures, and sample sizes. The film thickness reached this value most rapidly at +183° F and most slowly at -113° F. At a reduced initial

pressure of fluorine gas of 202 mm Hg abs, less reaction occurred than at higher pressures. At 312 mm Hg abs reaction was the same as at higher pressures. The pressure where less reaction is first noted is probably about 1/3 atm abs. Using samples half the usual weight, there seemed to be more film formed. This small size seemed to be the minimum size samples which could be run without introducing appreciable error into results. At -113° F, the film at the end of 28 hours reached 167 Å which was a twenty fold increase over the film in 2.5 hours. The behavior of monel at this temperature was similar to that of nickel in that an appreciable increase in film thickness is noted after prolonged exposure at -113° F. At temperatures of +86° F and +183° F, the film was only 3 to 4 times the one hour thickness.

Nickel formed relatively thin films based on long exposures despite the fact that the one hour film of 10.3 Å at +86° F was the largest one hour film of all powders at that temperature. The film reached only 15.8 Å after 25 hours. A new batch of powder was used for runs at -113° F and -297° F making results at these temperatures and higher temperatures non-comparable because of uncertainties in area measurement. At -113° F there was more than a tenfold increase in film from the one hour value to after 25 hours; this result is similar to the one for monel at -113° F.

Monel and nickel have been consistently used in fluorine systems with much success and safety. Their resistance to HF is a great asset in their use with fluorine.

Stainless Steels: This group of alloys have similar compositions and chemical properties. Although generally used because of their corrosion resistance, they are susceptible to halide attack.

The chemical compositions of the four types of stainless steel studied are given in Table XIX. The corrosion resistant properties are dependent on the constituents of the metal although this relationship has not been studied in these experiments.

Type 304 stainless steel formed its most stable film at +86° F. The one hour film increased from 8 to 14Å in one day. The end-of-run films at other temperatures were higher than for other metals and this was a typical case for the four stainless steels. At +183° F the film increased from 12Å in one hour to 44Å after a day. At -113° F, 304 stainless steel reacted similarly to monel and nickel with the one hour film of 5Å increasing almost ninefold to 44Å overnight. It is hard to blame this behavior on the nickel content as this stainless steel had the lowest nickel content of the 3 nickel-containing stainless steels studied and was the only one to exhibit the large increase in film at -113° F after prolonged exposure.

Type 316 stainless steel formed films which did not increase much after continued exposure to gaseous fluorine. The greatest percent increase was from 5Å in one hour to 16.7Å in one day. It had the least film of the stainless steels at -113° F. At +183° F, the film increased least of all metals from 30.8Å in one hour to 35.4Å in 4 days. With all other stainless steels, the one hour film tripled at -113° F from the one hour value to the end-of-run value. It probably ranks high in its suitability for use in fluorine service compared to other stainless steels.

Type 347 stainless steel formed the least film of the stainless steels at +183° F and +86° F. The one hour film tripled at +183° F from 9.3Å to 27.8Å in 39 hours. At +86° F, the one hour film increased from 5.4Å to 11.4Å overnight. At -113° F, the one hour film tripled from 13 to 41Å in 28 hours. This metal ranks with 316 stainless steel for use in fluorine systems.

Type 410 stainless steel contains no nickel and formed the largest films of all stainless steels. At +183° F the one hour film increased from 29.3 to 90.7Å in a day. At +86° F the one hour film of 8.5Å reached 63.8Å in 3 days. The smallest increase of one hour film was at -113° F where the one hour value of 22.1Å reached only 25.9Å in a day. This small increase in film due to prolonged exposure at -113° F may be due to the absence of nickel in the alloy.

D. EXTENDED IMMERSION OF TENSILE SPECIMENS IN LIQUID FLUORINE

The one year exposure of metal specimens to liquid fluorine verified other results which indicated that there is little corrosion of metal specimens in liquid fluorine. The maximum corrosion of 0.7 mils in one year (average) was observed for Magnesium HM-31.

The significance of the observation that the tensile properties of metals were improved after immersion in a cold bath at -320° F should be confirmed with additional tests.

If metals do not show significant corrosion in liquid fluorine, it is to be expected that there would be no deterioration of tensile properties as was demonstrated in these studies. A decrease in mechanical strength is expected for these metals which corrode most. Magnesium HK-31 corroded 0.7 mils in liquid fluorine and the yield strength was 46,000 psi. The yield strength for samples in liquid nitrogen was 49,000 psi.

The percent elongation and reduction in area depend somewhat on the rate of applying load to the tensile specimens when they are "pulled" in the test machine. All specimens were pulled at 0.025 inches per minute until yield at 2% elongation and then at a rate of 0.10 inches per minute until rupture. Stress-strain curves were automatically plotted until the 2% elongation load was reached.

Although the specimens were necked down by 0.001 - 0.002 inches in center so that they would break at this point, many broke at the ends. These were the specimens which had ultimate strengths very close to the yield strength. Magnesium specimens had ultimate strengths of 50,000 psi and yield strengths of 49,000 psi.

IV. CONCLUSIONS and RECOMMENDATIONS

A. FLUORINE CONTAMINANTS

The contaminants less volatile than fluorine which are present in an average fluorine cylinder include: CO_2, CF_4, HF, SiF_4, SF_6, OF_2, and SO_2F_2. Of these, only CF_4 and OF_2 are soluble. The others, present in the quantities found in cylinder fluorine, form solids if the fluorine is liquefied. It is recommended that they be removed if

the fluorine is to be liquefied and used in a flow system as they may cause plugging of valves and small lines. OF_2 should be removed as it appears to increase the corrosion rate of metals in fluorine contaminated with it.

The recommended procedure for purifying fluorine to 100% purity is to:

1. Pass gaseous fluorine through a sodium fluoride scrubbing tower.

2. Liquefy.

3. Filter through a fine filter.

4. Distill in a small evaporator to remove residual contaminants.

The entire purification system must be perfectly leak-tight. The purified fluorine must be handled in evacuated and passivated equipment equally leak free.

The solubility of CF_4 at -297° F and -320° F is 40 mol percent and 4.5 mol percent respectively. At -320° F, OF_2 is soluble in quantities greater than 10%. Other contaminants are insoluble.

While the infra-red technique has been found to be highly satisfactory for the analysis of gaseous fluorine, other methods may be practical. Gas chromatography may be acceptable if suitable column and detection techniques are used.

B. REACTIONS ON METAL SURFACES

Visual observation of the action of liquid fluorine on metal surfaces indicates that little or no corrosion occurs in the liquid phase. Reaction was observed in the gaseous phase. It is concluded that the gas phase reaction is due to the presence of contaminants. It is recommended that gaseous fluorine be contaminant-free to reduce possible corrosion. The contaminants, except for OF_2, appear to be inert in the liquid. Liquefying and evaporating fluorine repeatedly in a metal container will probably increase the corrosion of the metal, but this has not been verified.

Traces of hydrocarbons cannot be removed from metal surfaces by exposing them to gaseous fluorine, ClF_3, or mixtures of F_2 and ClF_3. The hydrocarbons react with these gases but are not converted to CF_4 and HF which are inert to liquid fluorine. Traces of carbon and fluorinated oils are produced which could react explosively with liquid fluorine. It is recommended that all metal surfaces be meticulously cleaned before exposing them to liquid fluorine even if the surfaces are to be passivated in gaseous fluorine. This passivation technique is useless for removing the harmful effect of oils, even though it may remove traces of moisture. The technique for cleaning materials for oxygen service is probably satisfactory for materials to be used with fluorine and should be used.

Short term immersion tests indicate that almost all corrosion of metals in liquid fluorine occurs in the first minutes of exposure. Serious corrosion does not occur after this time and almost all metals suitable for cryogenic service can be used safely for liquid fluorine. Oxygen difluoride increases the corrosion rate of metals in liquid fluorine, but not sufficiently to create a safety problem. Nevertheless, it should not be present in liquid fluorine as no additional corrosion in liquid fluorine is desirable.

C. FLUORIDE FILM STUDIES

Gaseous fluorine is adsorbed on metal powders at -297° F. There appears to be little if any reaction between gaseous fluorine and metal powders at this temperature. The fluorine may be desorbed by heating the powder to room temperature and/or drawing vacuum on it.

Passivation of metal surfaces before exposure to liquid fluorine is necessary only if the surface is suspected of having traces of contaminants present. Passivation will not remove hydrocarbons or oils, but it may eliminate moisture adsorbed on the metal surfaces. Apparently, film formation is not a factor in corrosion resistance of metals exposed to fluorine at low temperatures ($<0°$ F). What little film forms is formed rapidly and in several minutes reaches more than half the

value attained in one hour. One hour fluoride films are usually less than 100 Å. If a passivation procedure is used, the time of exposure of fluorine required to give passivation protection is probably not more than one hour. Only clean systems should be passivated and these at a higher temperature and pressure than contemplated for service. Passivation should be accomplished as the prior step to fluorine service. Unless the passivated surface is kept in a dry, inert atmosphere, the benefit of passivation to form a protective fluoride film are lost. If the passivation has as its purpose to clean surfaces, and does so, the clean surface will be preserved better in a dry, inert atmosphere.

Care should be taken in using titanium in a fluorine system. The titanium may ignite and cause a violent reaction. However, if there is a small surface to volume ratio of metal, the heat of reaction may be conducted away from the point of ignition rapidly enough to prevent an explosion. Titanium may be used more safely in low temperature systems than in high temperature ones because of easier heat removal from a possible reaction site. Thus, the temperature, pressure, and service must be carefully evaluated before titanium is selected as a material of construction for fluorine service.

D. EXTENDED IMMERSION TESTS

Mechanical properties of tensile specimens immersed in liquid fluorine are essentially the same as mechanical properties of similar specimens immersed in liquid nitrogen for the same period. The mechanical strength of the metals exposed to low temperatures appeared to be superior to nominal handbook values. These experiments lead to the conclusion that all of the metals tested are satisfactory for liquid fluorine systems. The selection of materials should consider their resistance to HF, which is often associated with liquid fluorine. Also to be considered are cycling between gaseous and liquid service, expected system cleanliness, and temperature cycling of the system.

The corrosion rate of metals immersed in fluorine is extremely low, (less than one mil in one year). All the metals studied are recommended for liquid fluorine service based on static corrosion requirements.

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14. Sterner, C.J. and Singleton, A. H.: The Compatibility of Various Metals and Carbon with Liquid Fluorine, WADD Technical Report 600-819 (January 1961).

APPENDIX I

FIGURES

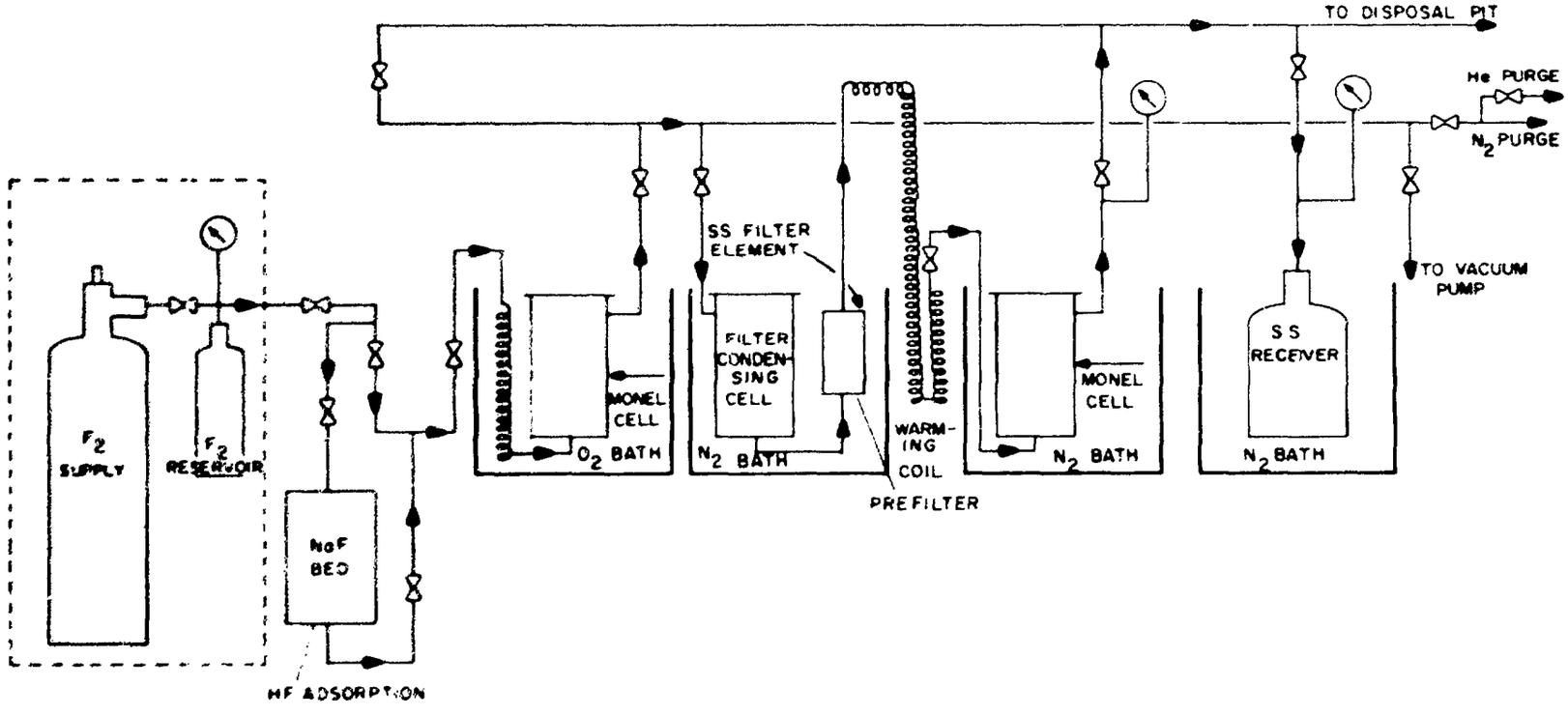


FIGURE I
APPARATUS FOR STUDYING FLUORINE CONTAMINANTS

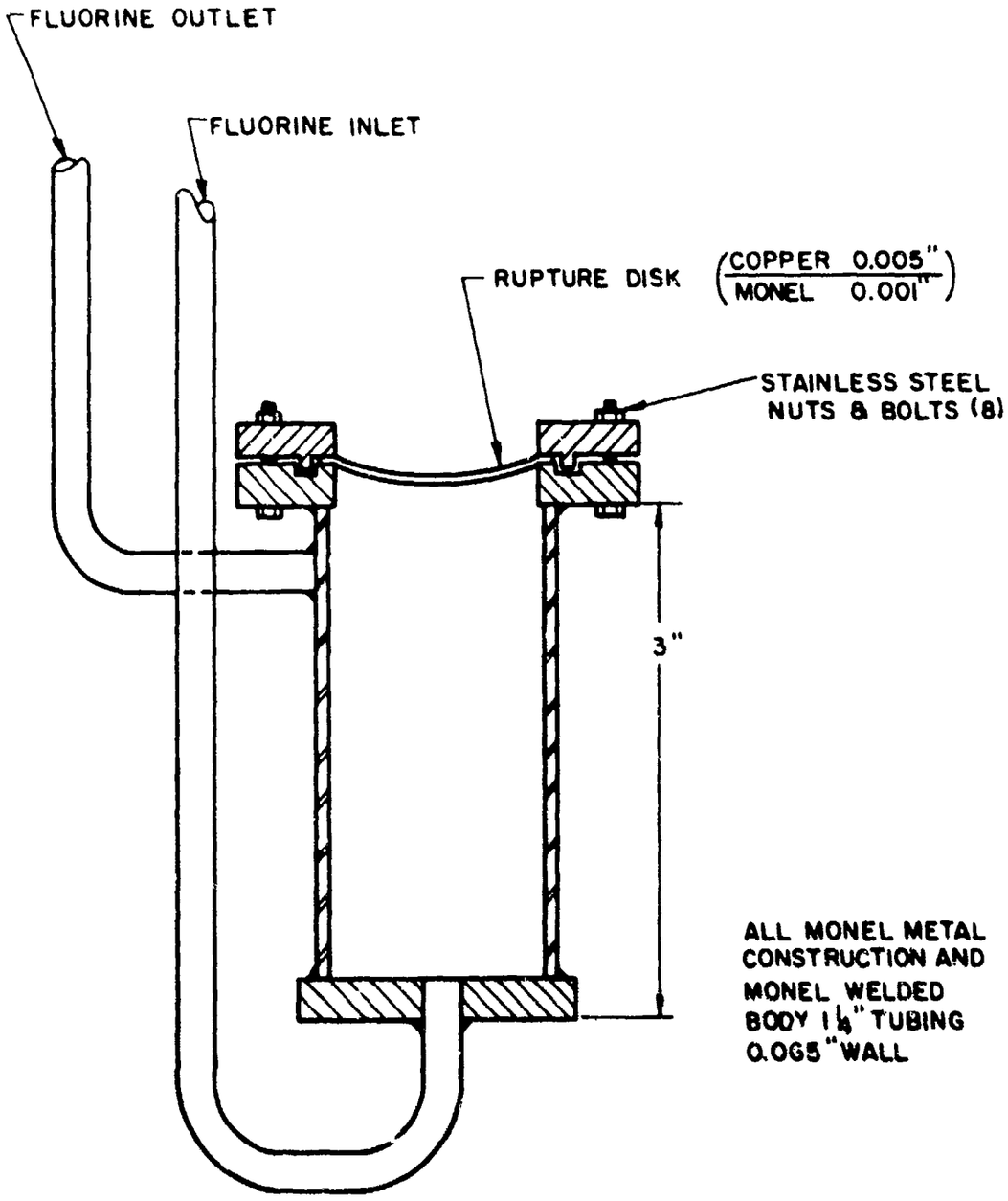


FIGURE 2

IMMERSION SAMPLE CELL

SCALE ____ 1"=1"

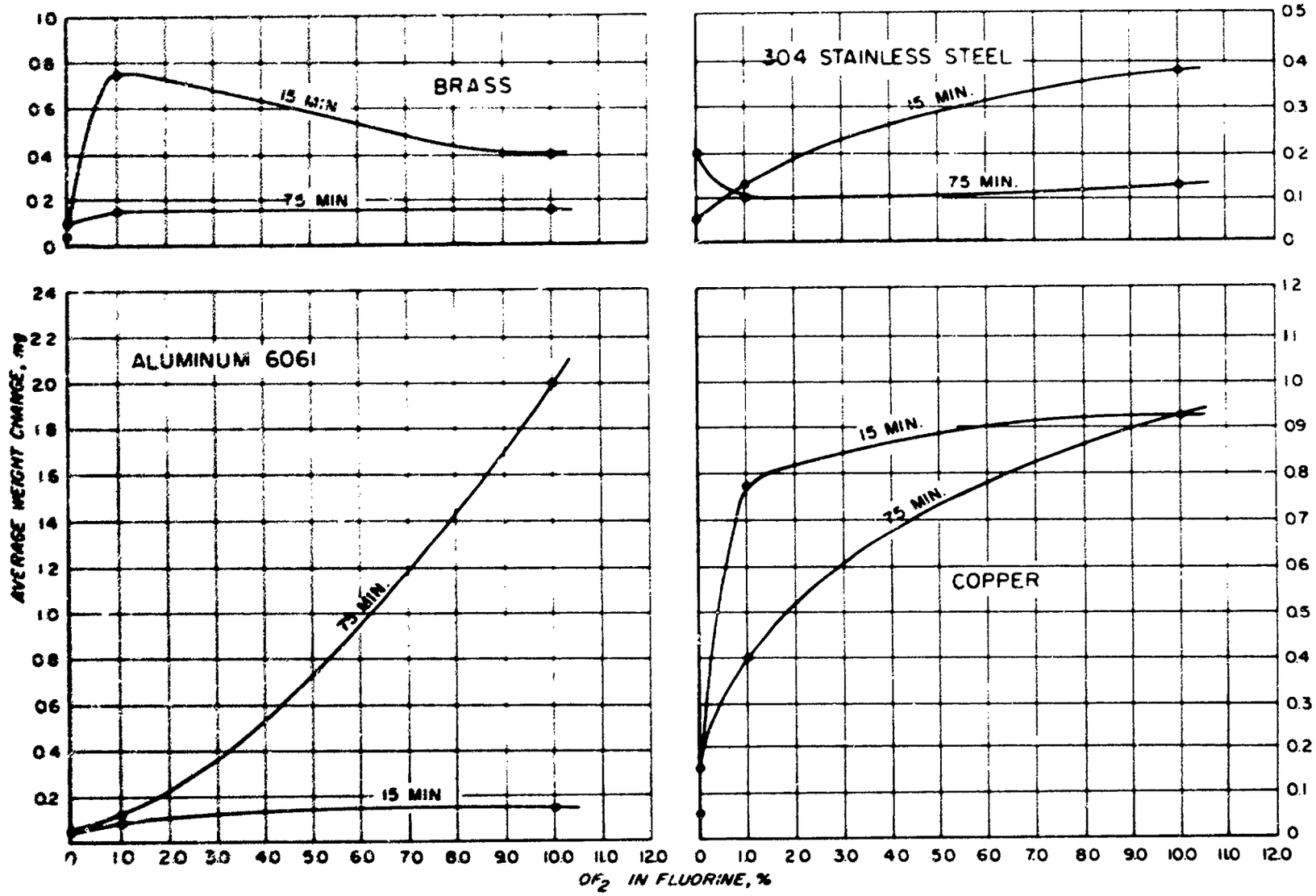


Figure 3. Effect of OF_2 Concentration in Liquid Fluorine on Average Absolute Weight Changes of Aluminum 6061, Brass, Copper and 304 Stainless Steel

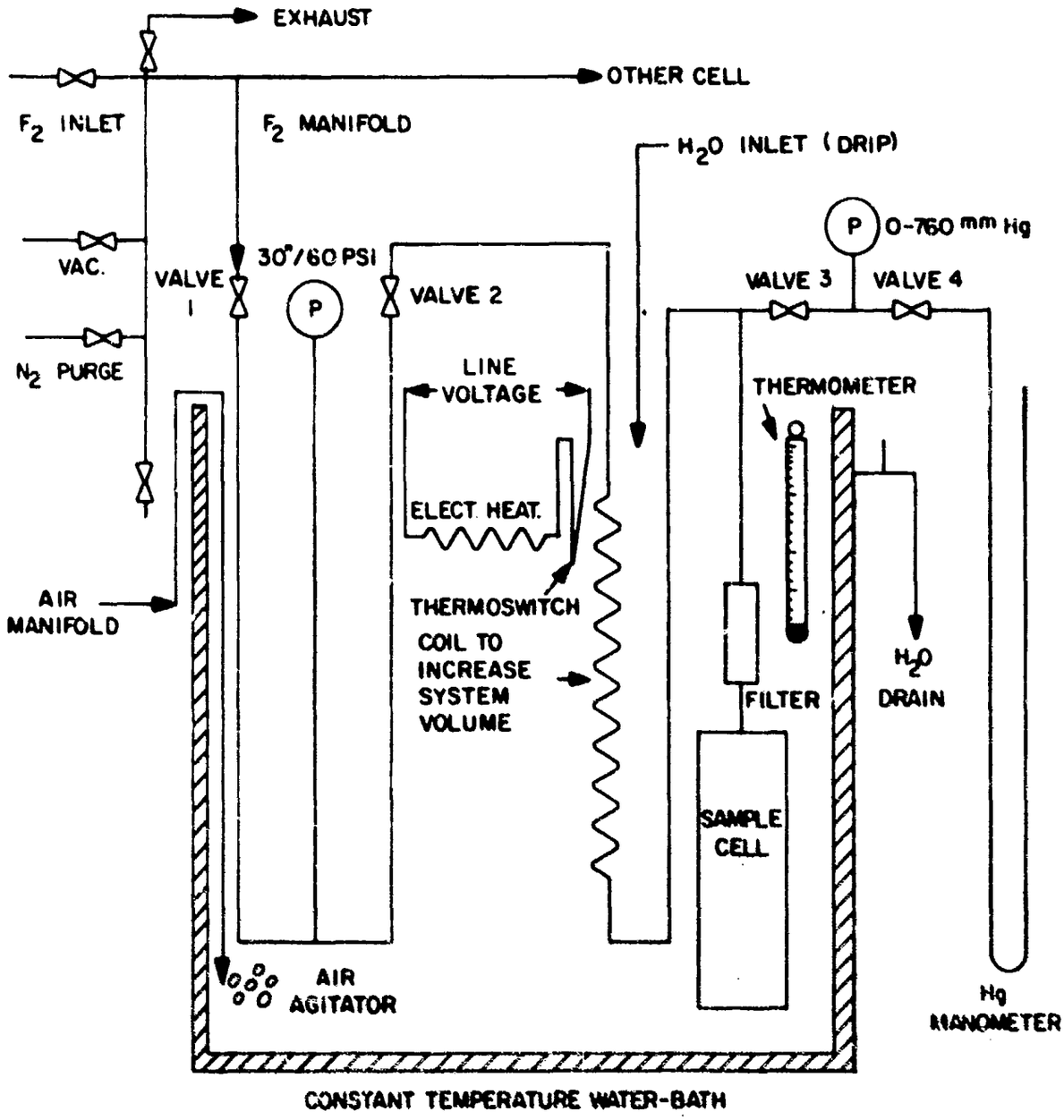


FIGURE 4

TEST APPARATUS FOR MEASUREMENT OF FLUORIDE FILM THICKNESS ON METAL POWDERS AT +86°F AND 183°F

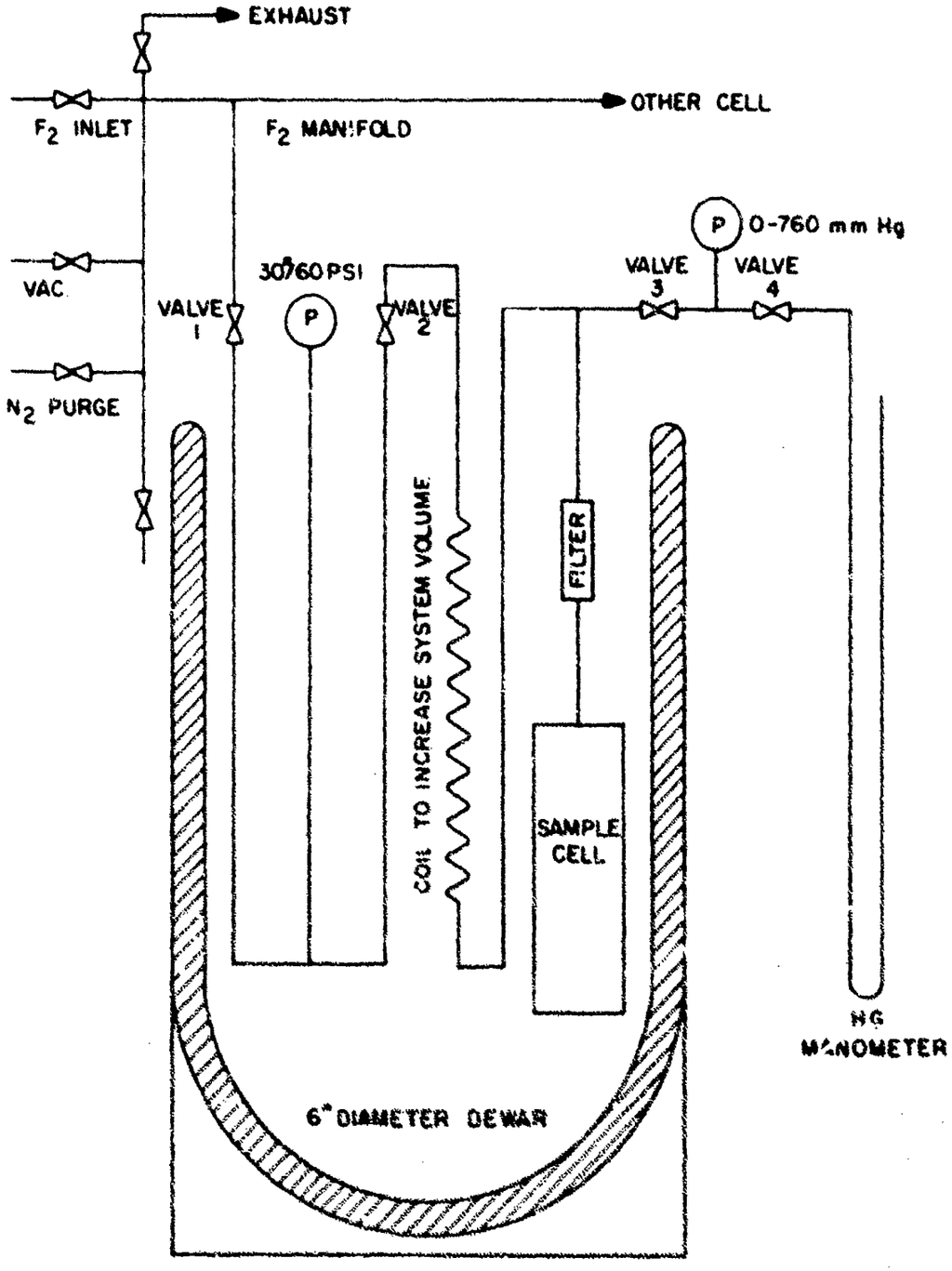


FIGURE 5
TEST APPARATUS FOR MEASUREMENT OF FLUORIDE FILM THICKNESS ON METAL POWDERS AT -113°F & -297°F

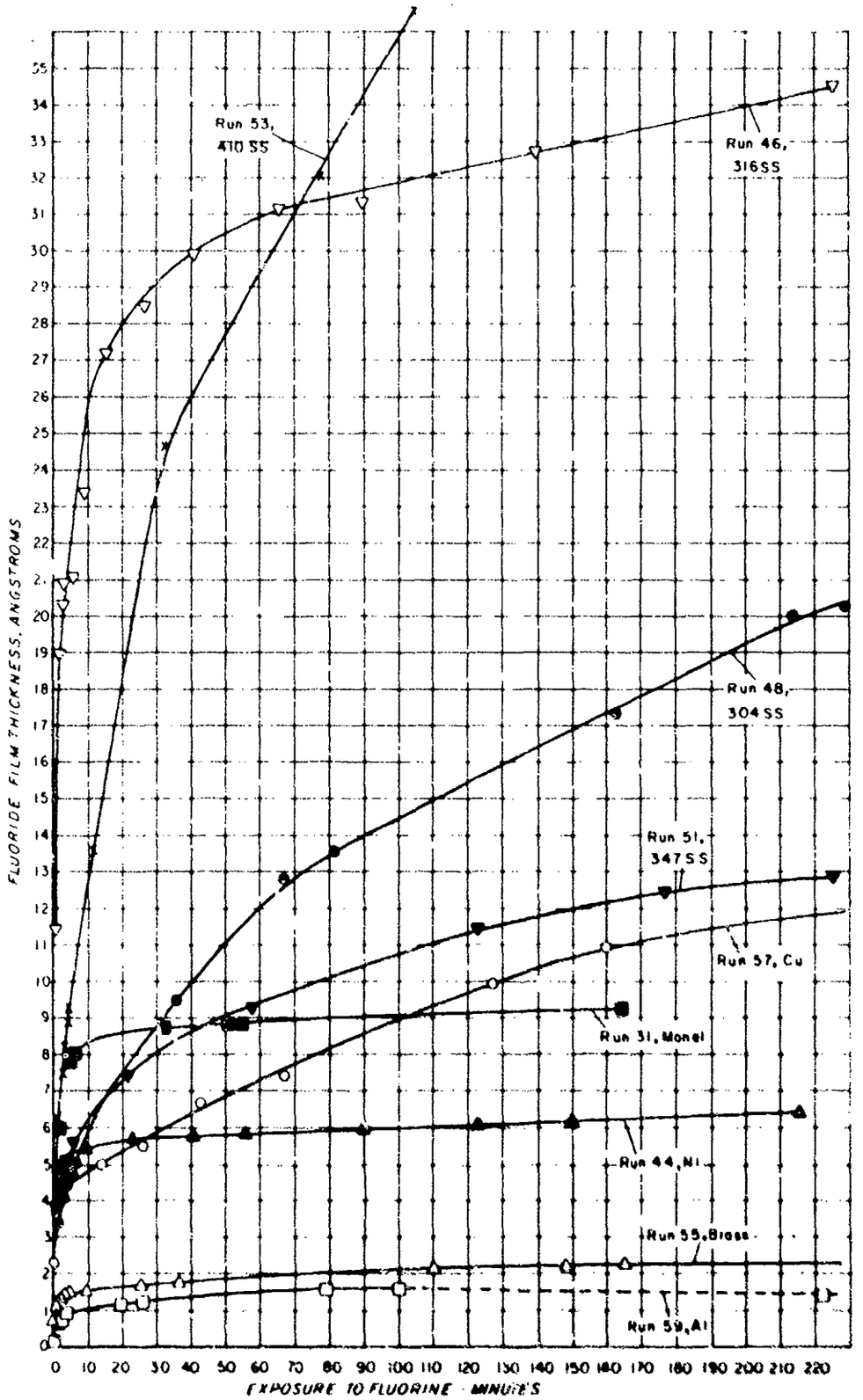


Figure 6 Variations of Film Thickness on Metal Powders at +183°F to Time of Exposure to Gaseous Fluorine

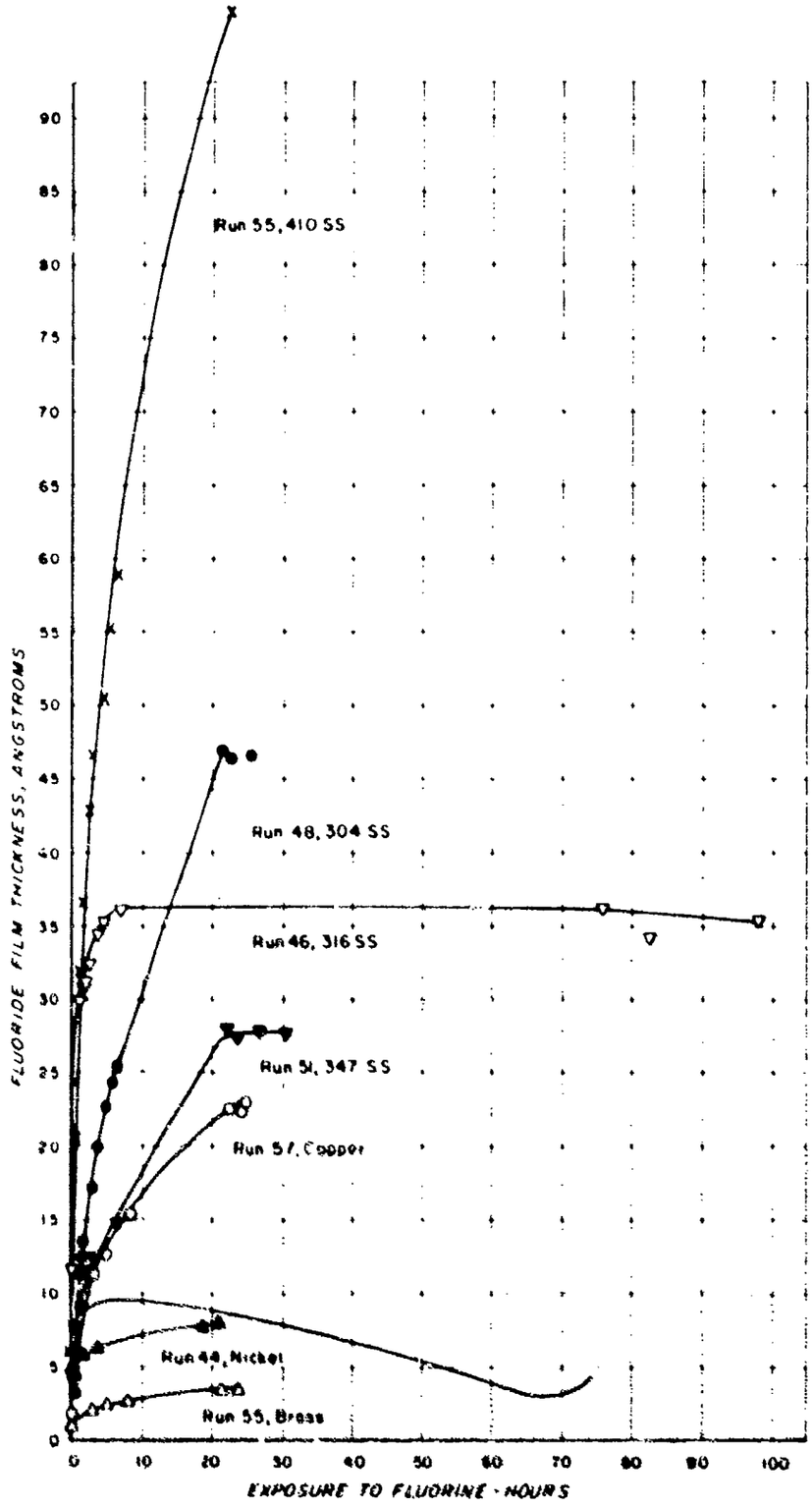


Figure 7 Variation of Film Thickness on Metal Powders at +183°F to Time of Exposure To Gaseous Fluorine - Total Run

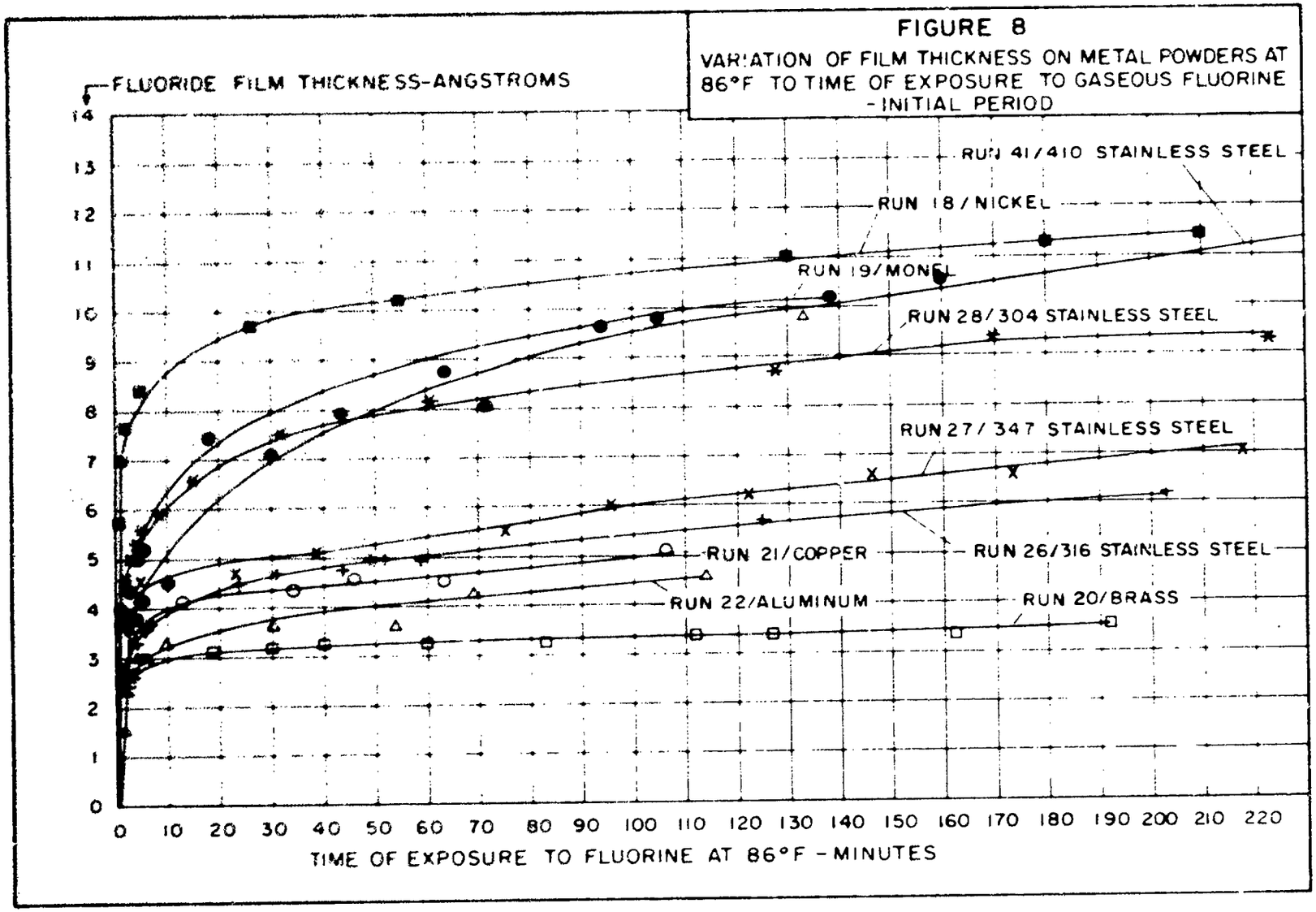


FIGURE 8

VARIATION OF FILM THICKNESS ON METAL POWDERS AT 86°F TO TIME OF EXPOSURE TO GASEOUS FLUORINE - INITIAL PERIOD

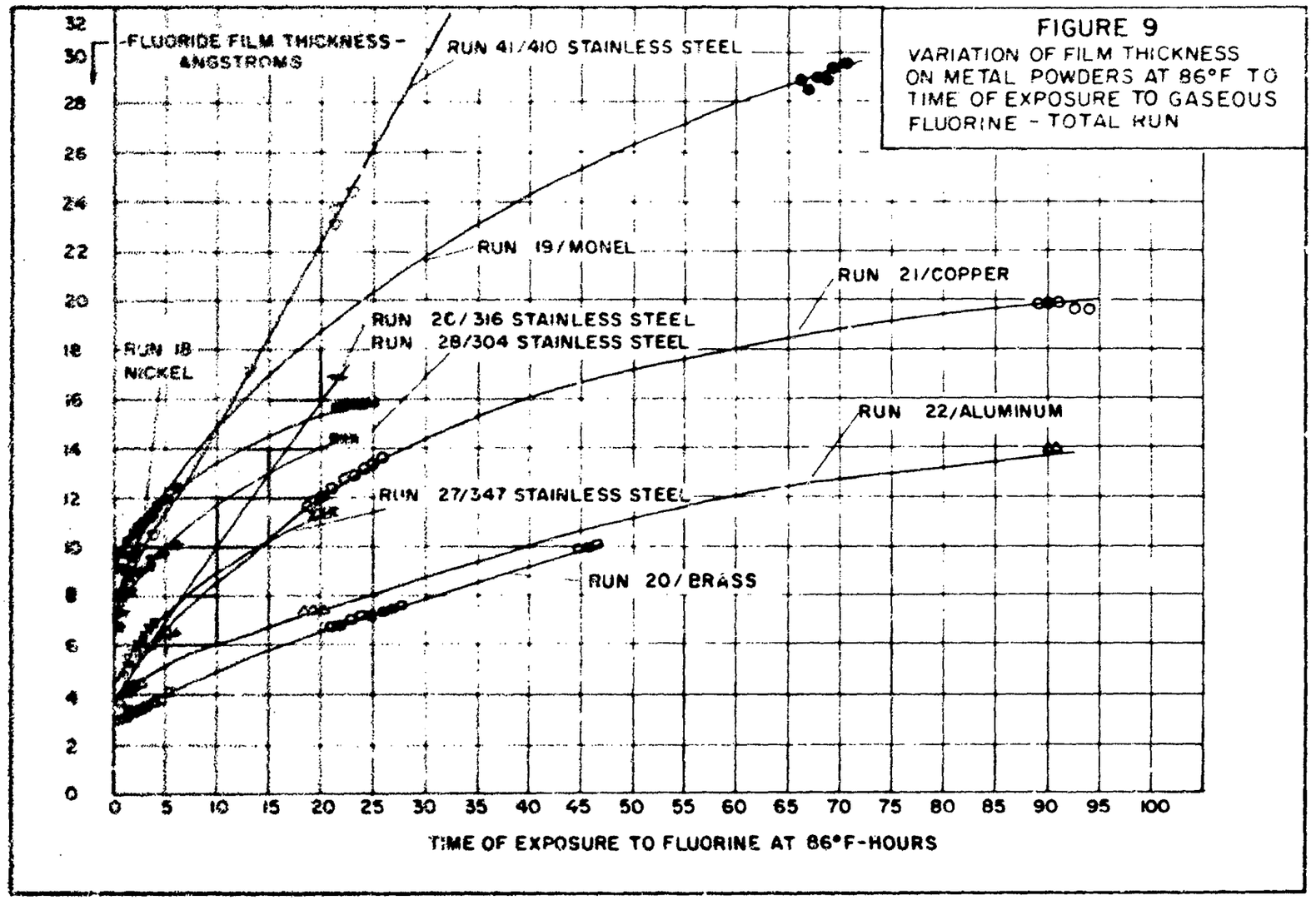


FIGURE 9
VARIATION OF FILM THICKNESS ON METAL POWDERS AT 86°F TO TIME OF EXPOSURE TO GASEOUS FLUORINE - TOTAL RUN

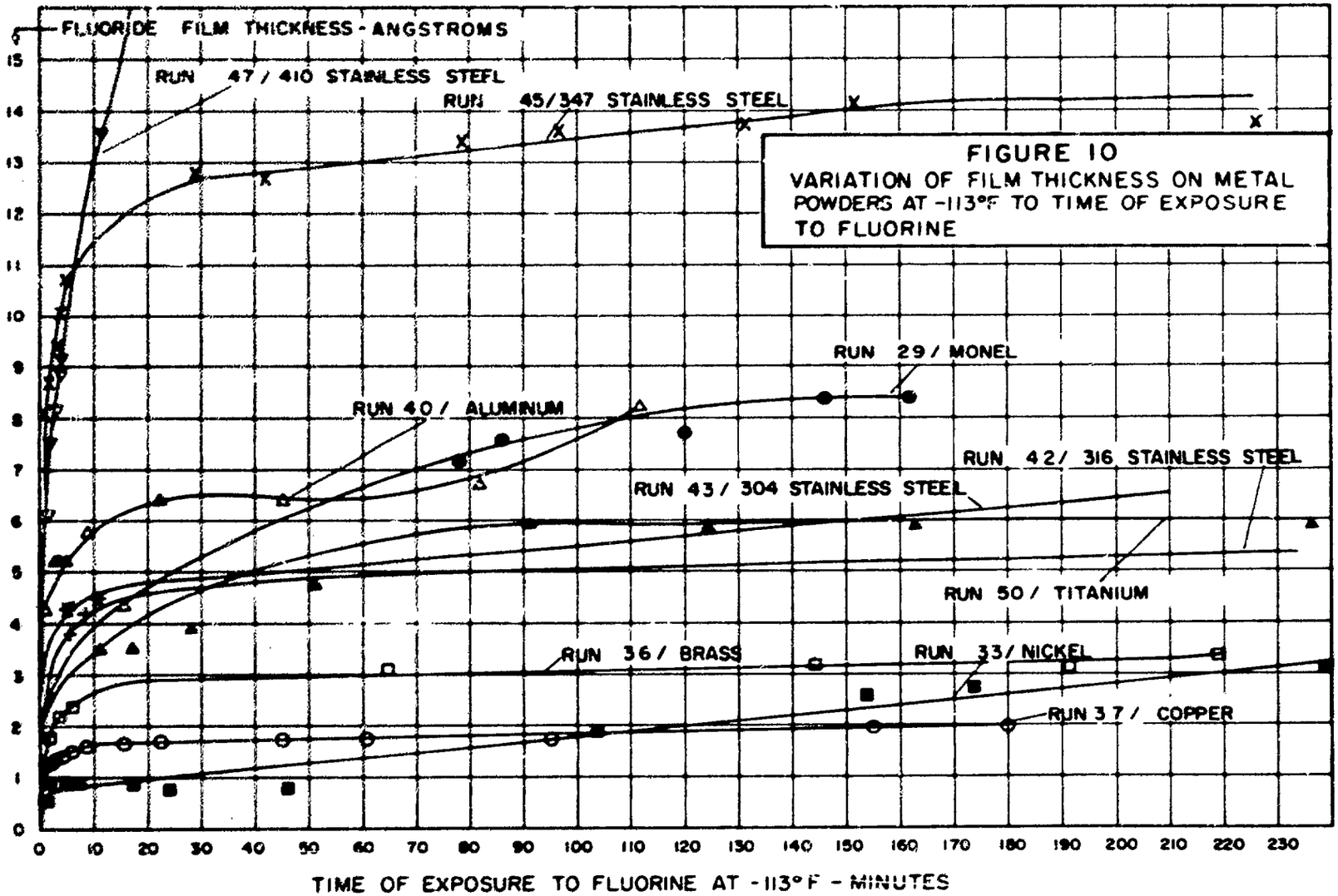
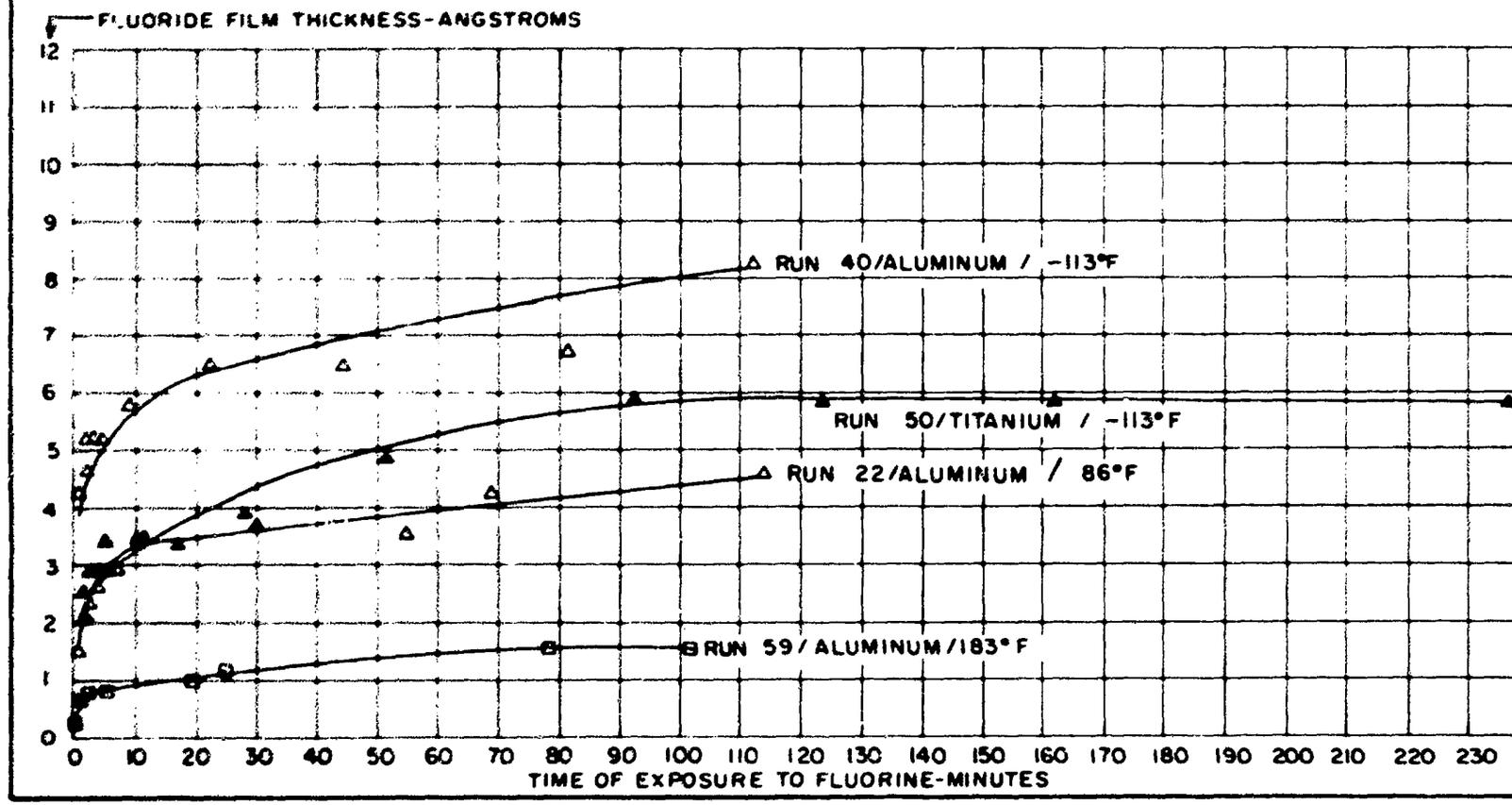


FIGURE 10
VARIATION OF FILM THICKNESS ON METAL POWDERS AT -113°F TO TIME OF EXPOSURE TO FLUORINE

FIGURE 11
VARIATION OF FILM THICKNESS ON ALUMINUM AND TITANIUM TO TIME OF EXPOSURE TO FLUORINE



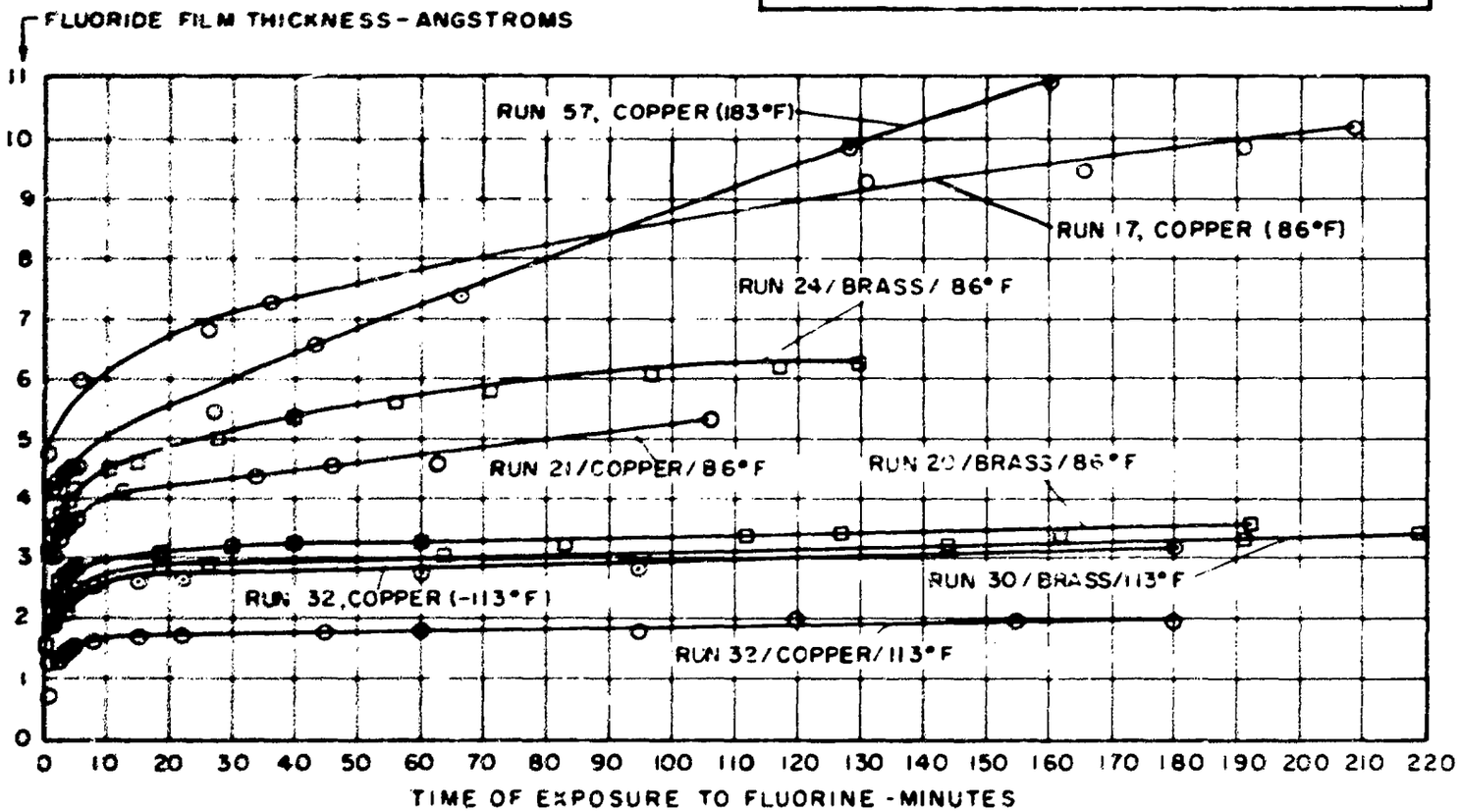


FIGURE 12
VARIATION OF FLUORIDE FILM THICKNESS ON COPPER AND BRASS POWDERS AT TWO DIFFERENT TEMPERATURE

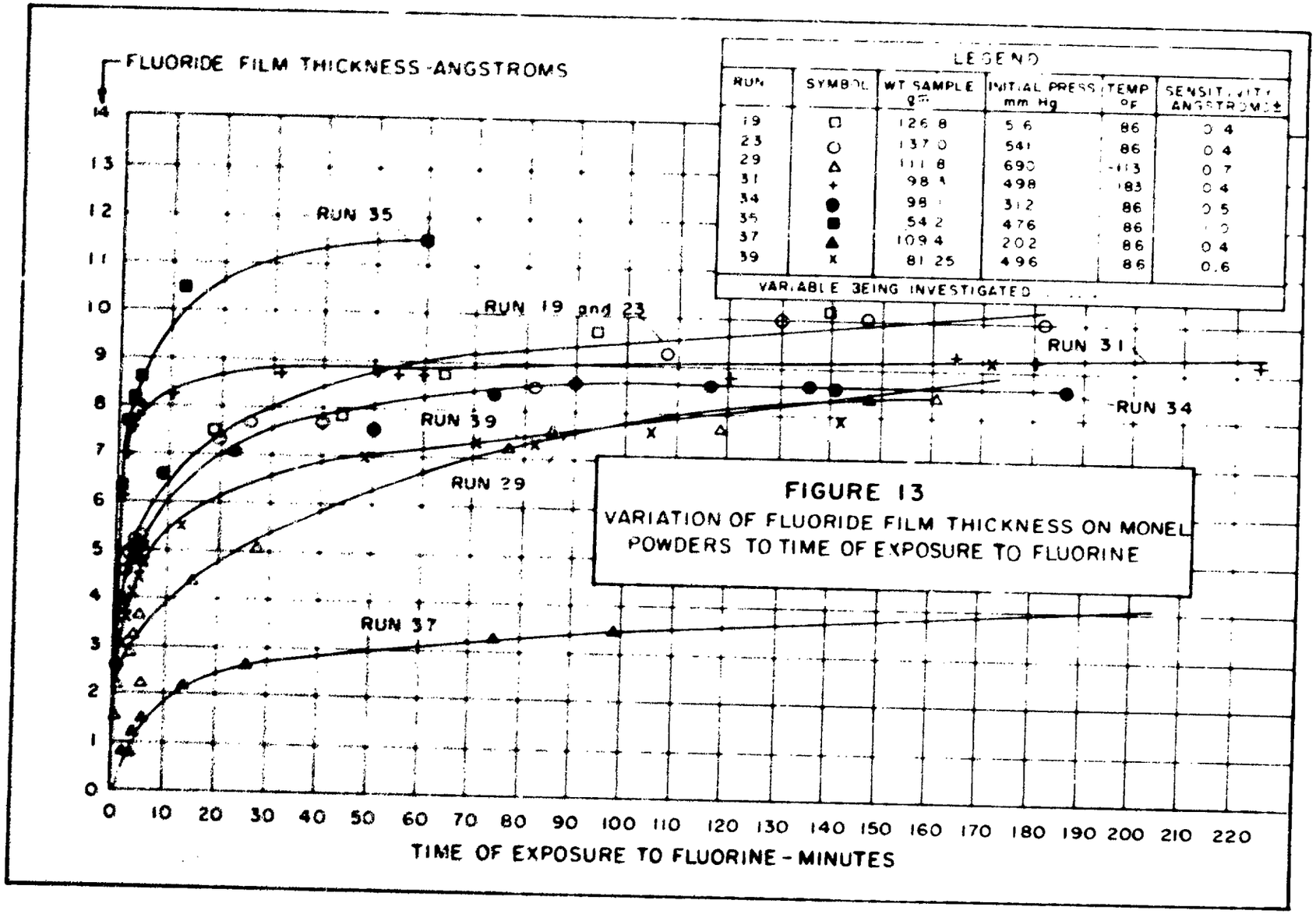


FIGURE 13
VARIATION OF FLUORIDE FILM THICKNESS ON MONEL POWDERS TO TIME OF EXPOSURE TO FLUORINE

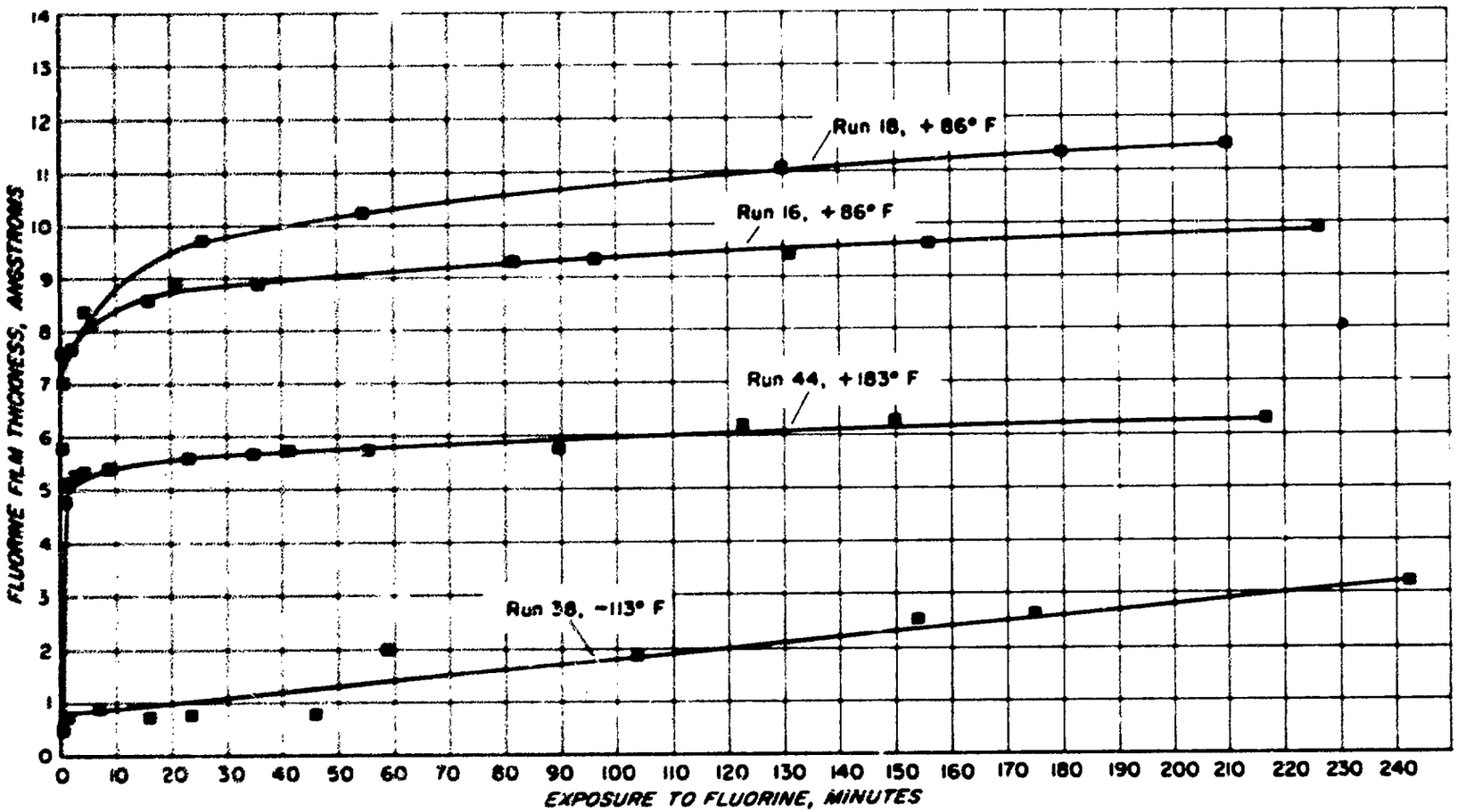


Figure 14. Variation of Film Thickness on Nickel Powders to Time of Exposure to Fluorine

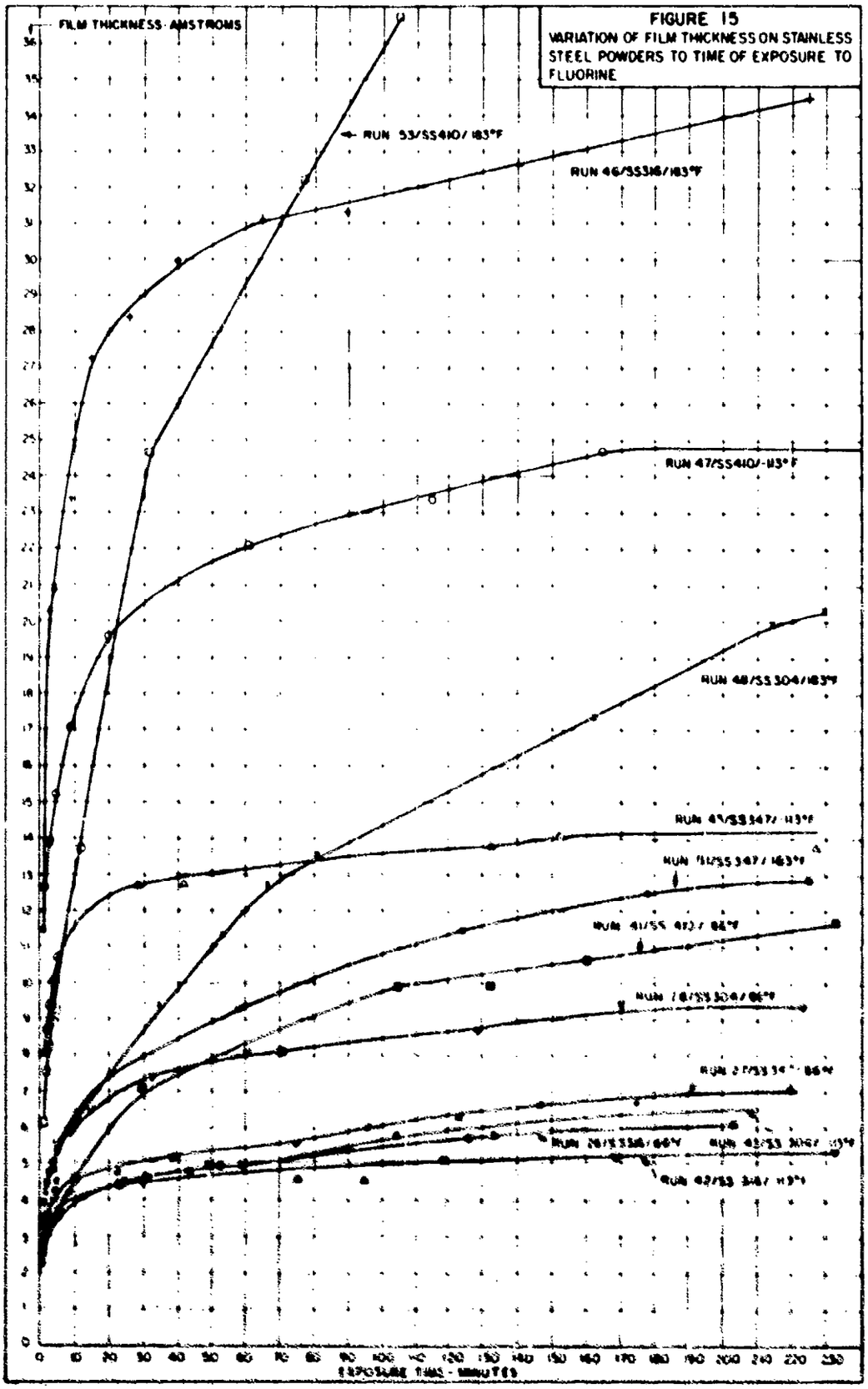
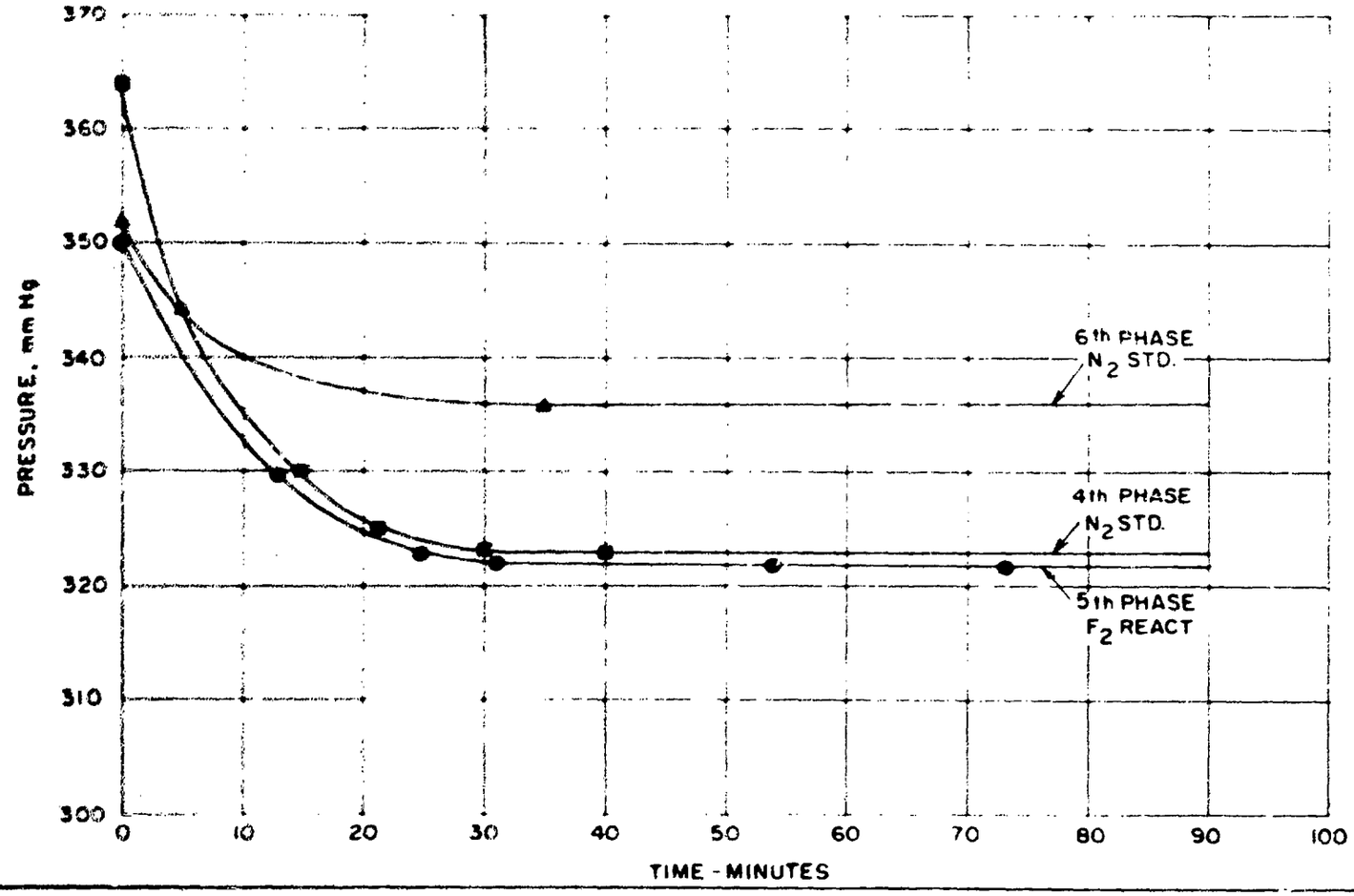


FIGURE 15
VARIATION OF FILM THICKNESS ON STAINLESS STEEL POWDERS TO TIME OF EXPOSURE TO FLUORINE

FIGURE 16a

RUN 58, EXPOSURE OF GASEOUS FLUORINE TO 304 STAINLESS STEEL POWDER AT -297°F



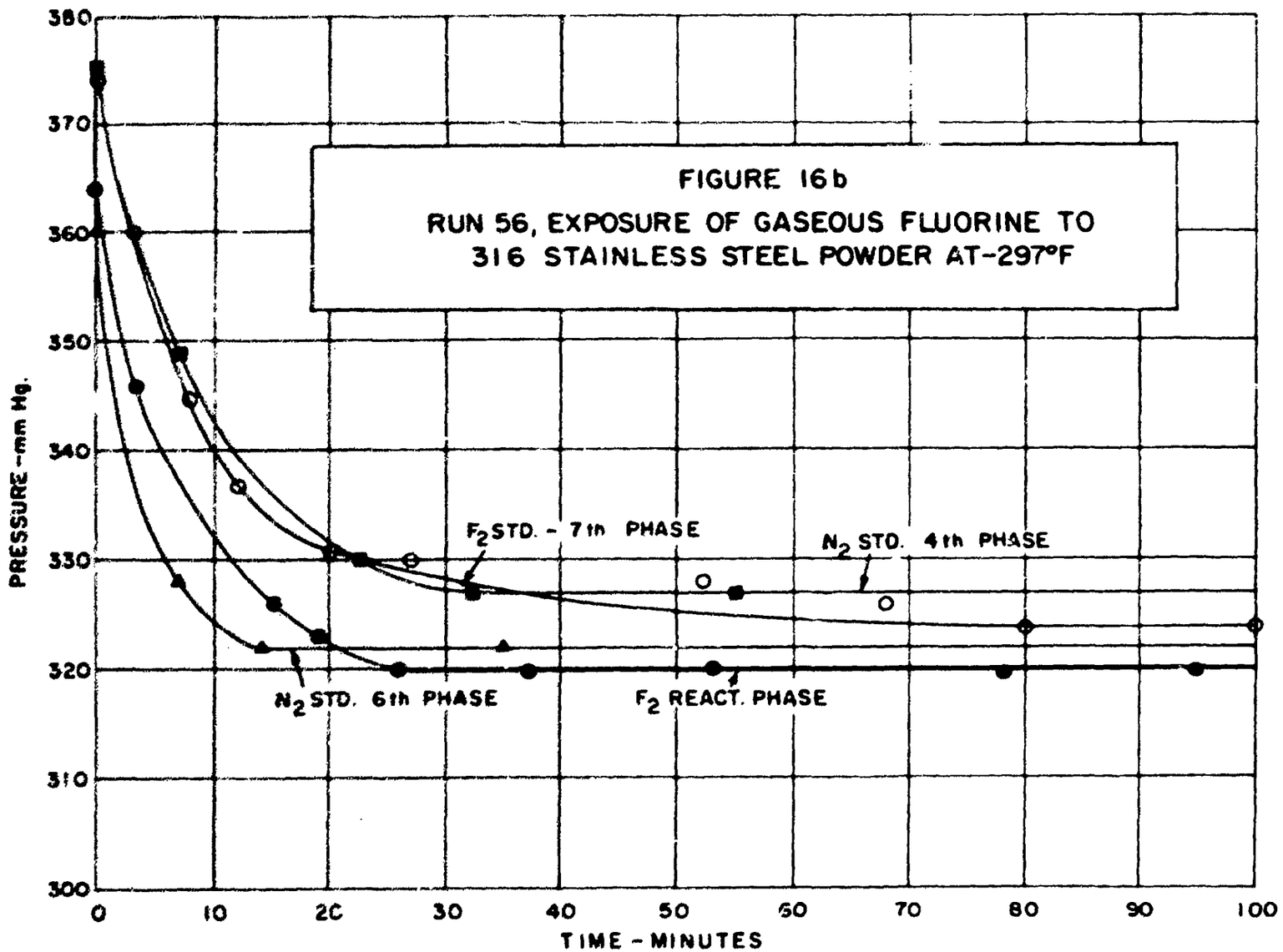
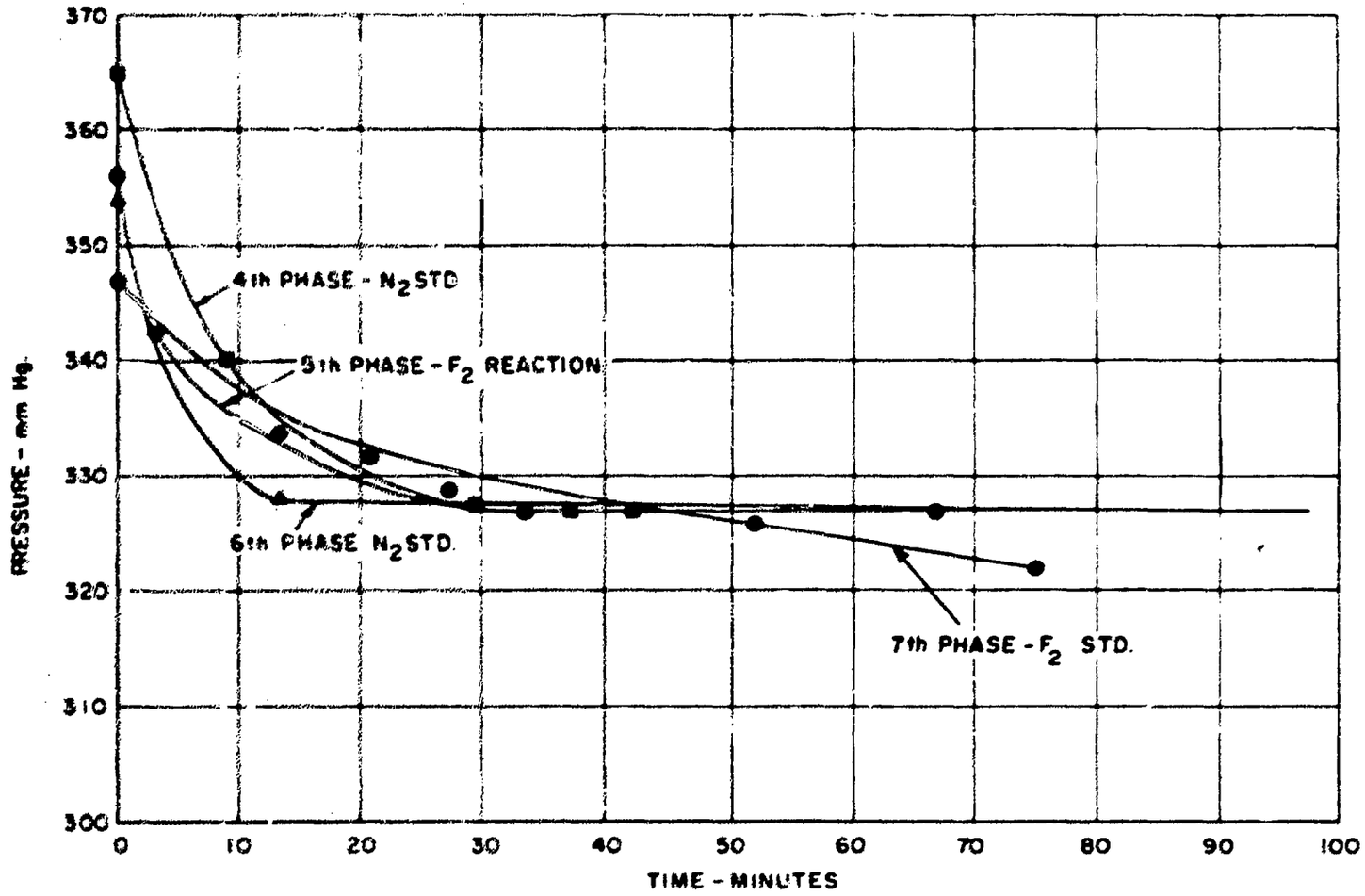


FIGURE 16b
RUN 56, EXPOSURE OF GASEOUS FLUORINE TO
316 STAINLESS STEEL POWDER AT-297°F
PRESSURE - mm Hg.
TIME - MINUTES
380
370
360
350
340
330
320
310
300
0
10
20
30
40
50
60
70
80
90
100
F_2 STD. - 7th PHASE
N_2 STD. 4th PHASE
N_2 STD. 6th PHASE
F_2 REACT. PHASE

FIGURE 16c

RUN 60, EXPOSURE OF GASEOUS FLUORINE TO 347 STAINLESS STEEL POWDER AT -297°F



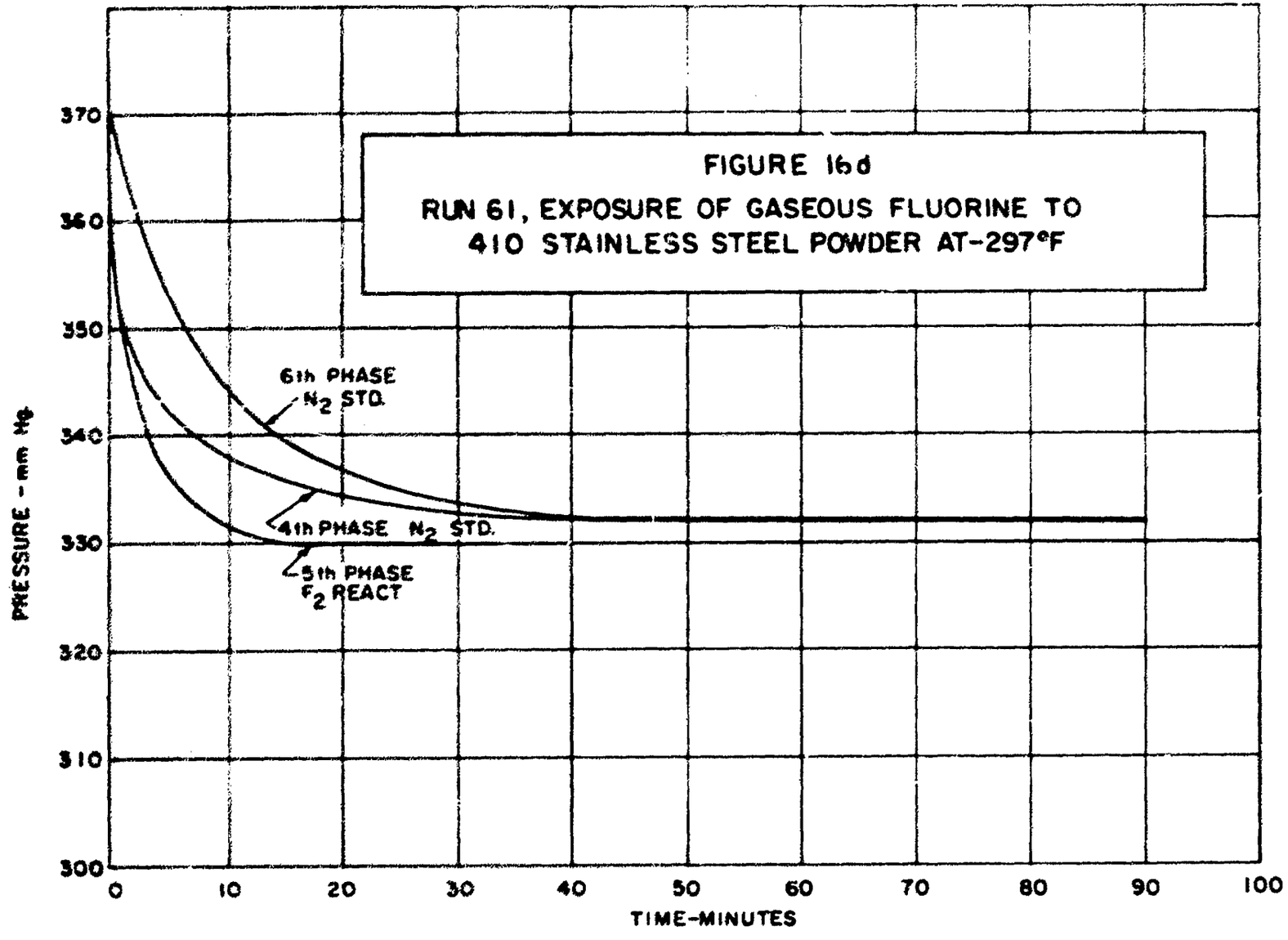


FIGURE 16d

RUN 61, EXPOSURE OF GASEOUS FLUORINE TO 410 STAINLESS STEEL POWDER AT-297°F

FIGURE 17a

RUN 66, EXPOSURE OF GASEOUS FLUORINE TO ALUMINUM POWDER AT - 297° F

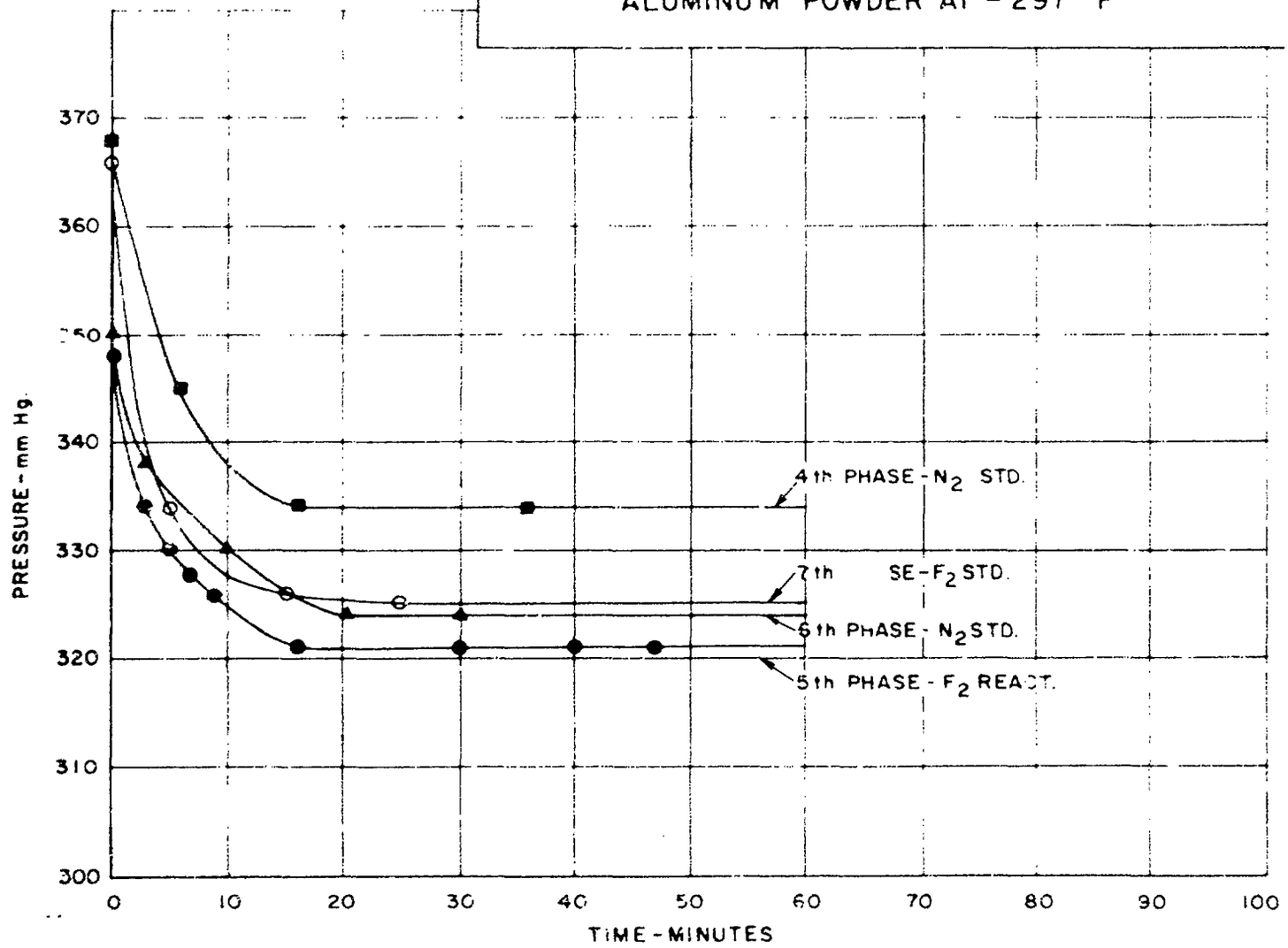


FIGURE 17b

RUN 62, EXPOSURE OF GASEOUS FLUORINE TO BRASS POWDER AT −297°F

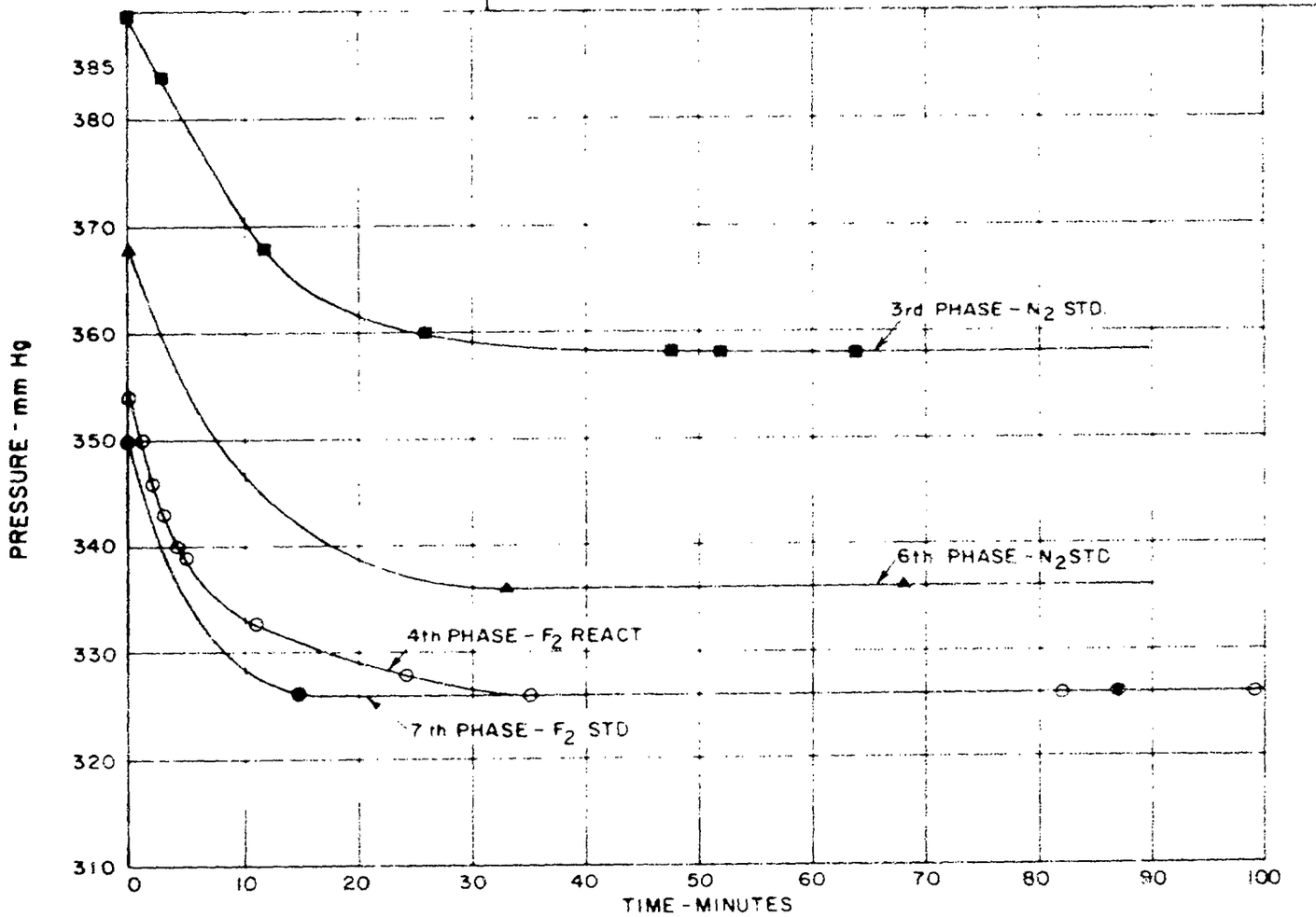


FIGURE 17c

RUN 63, EXPOSURE OF GASEOUS FLUORINE TO COPPER POWDER AT −297° F

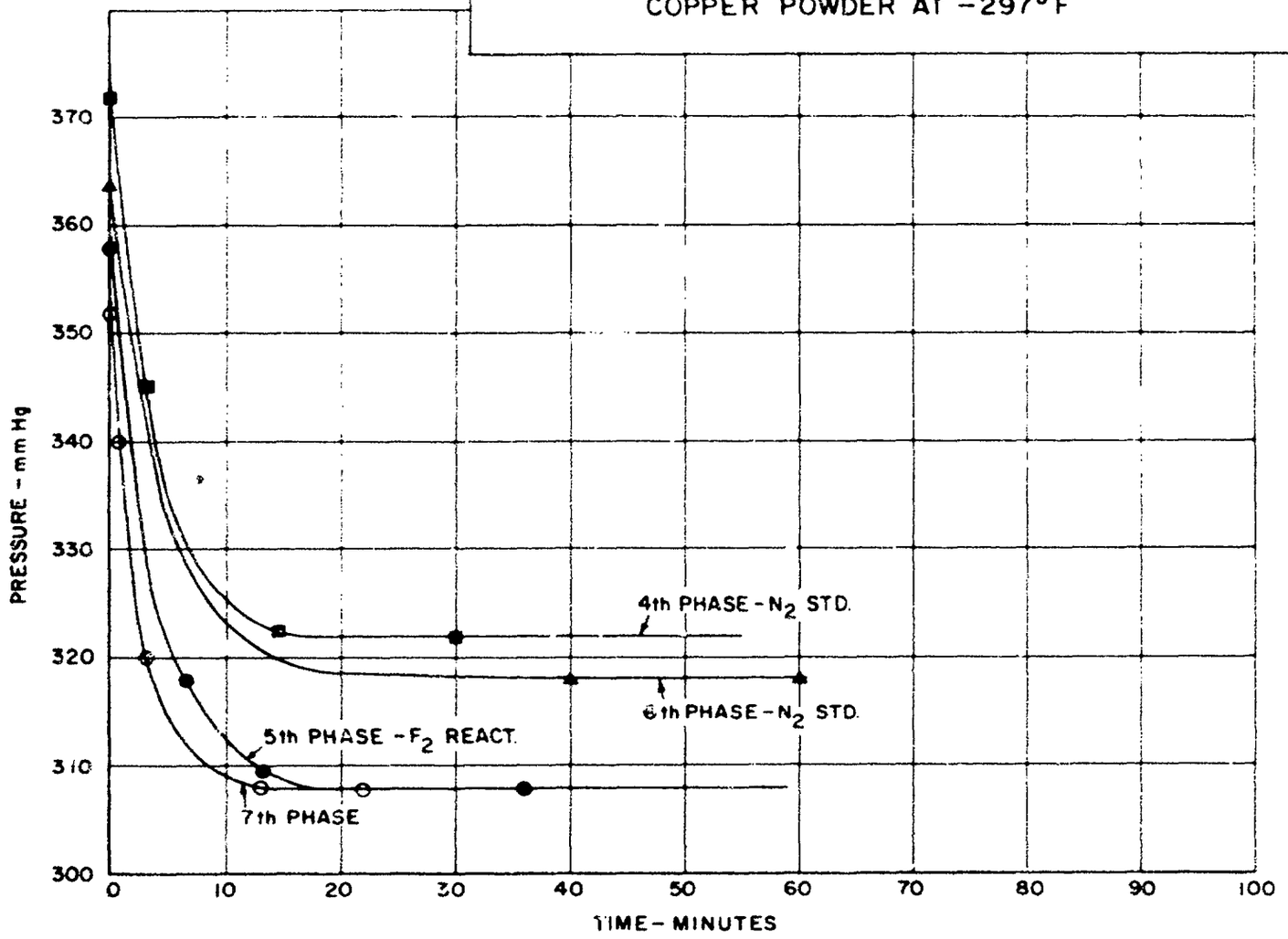


FIGURE 17d

RUN 52, EXPOSURE OF GASEOUS FLUORINE TO NICKEL POWDER AT −297°F

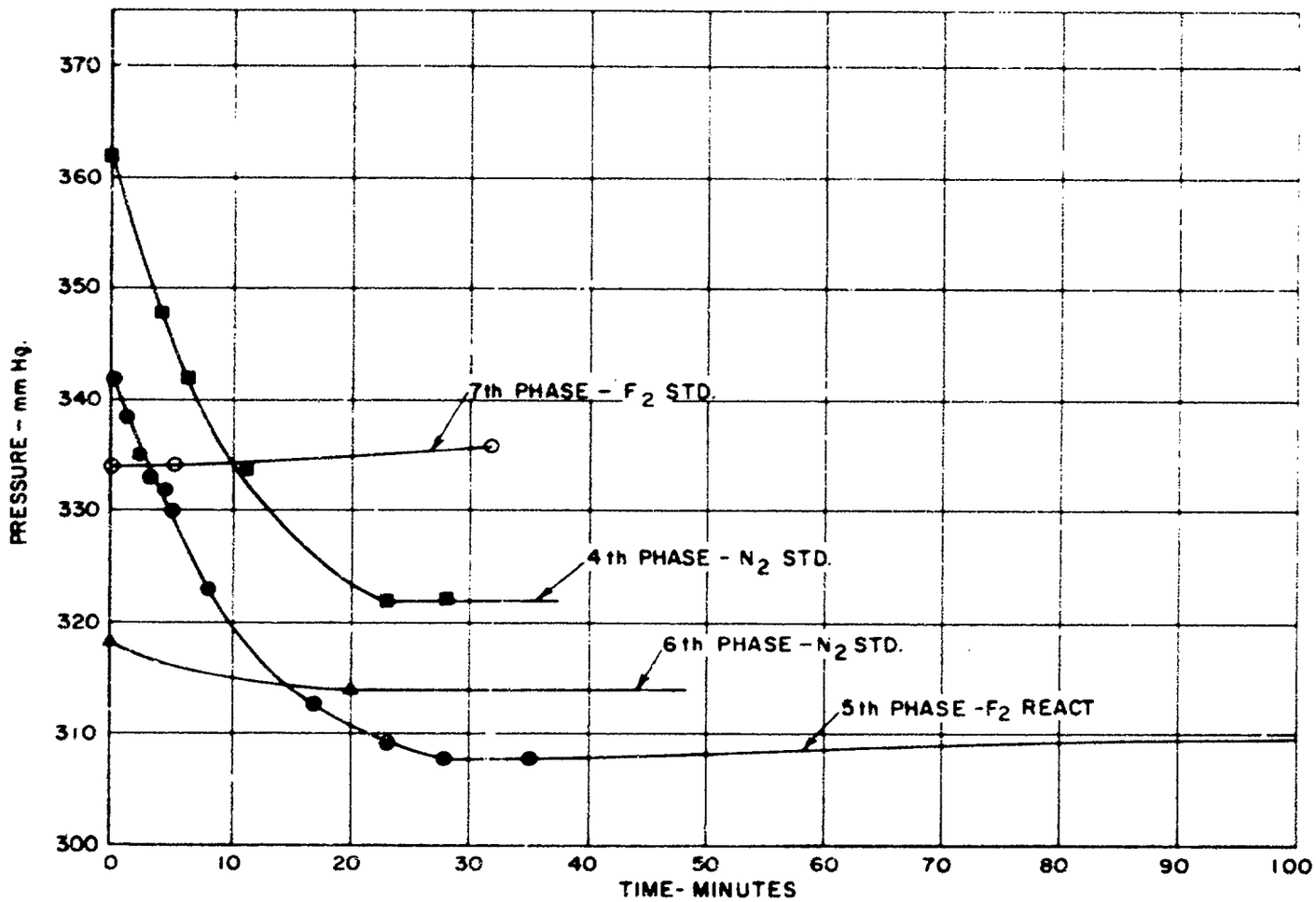


FIGURE 18a

RUN 19, EXPOSURE OF GASEOUS FLUORINE TO MONEL POWDER AT 86° F

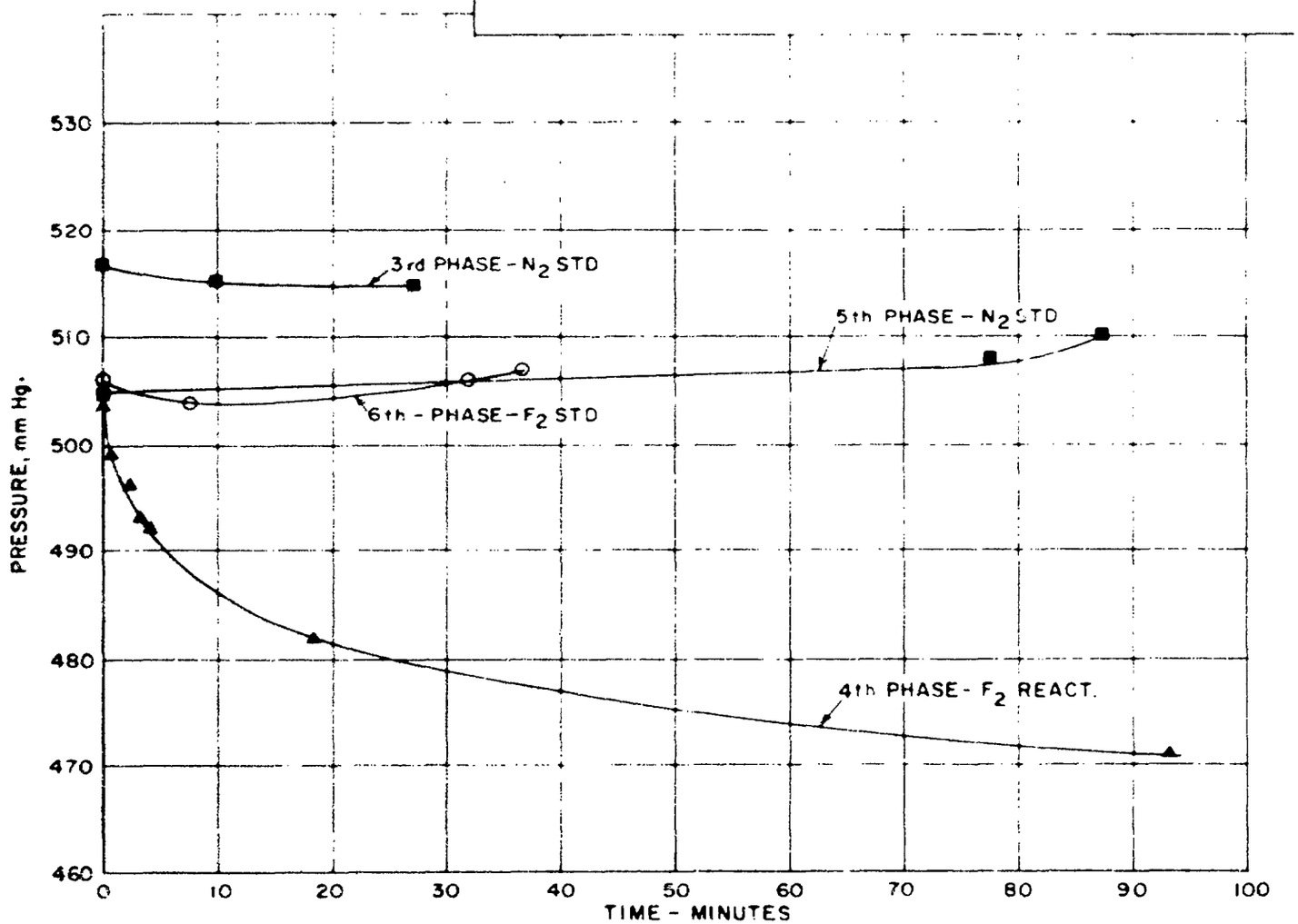


FIGURE 18b

RUN 29, EXPOSURE OF GASEOUS FLUORINE TO MONEL POWDER AT -113° F

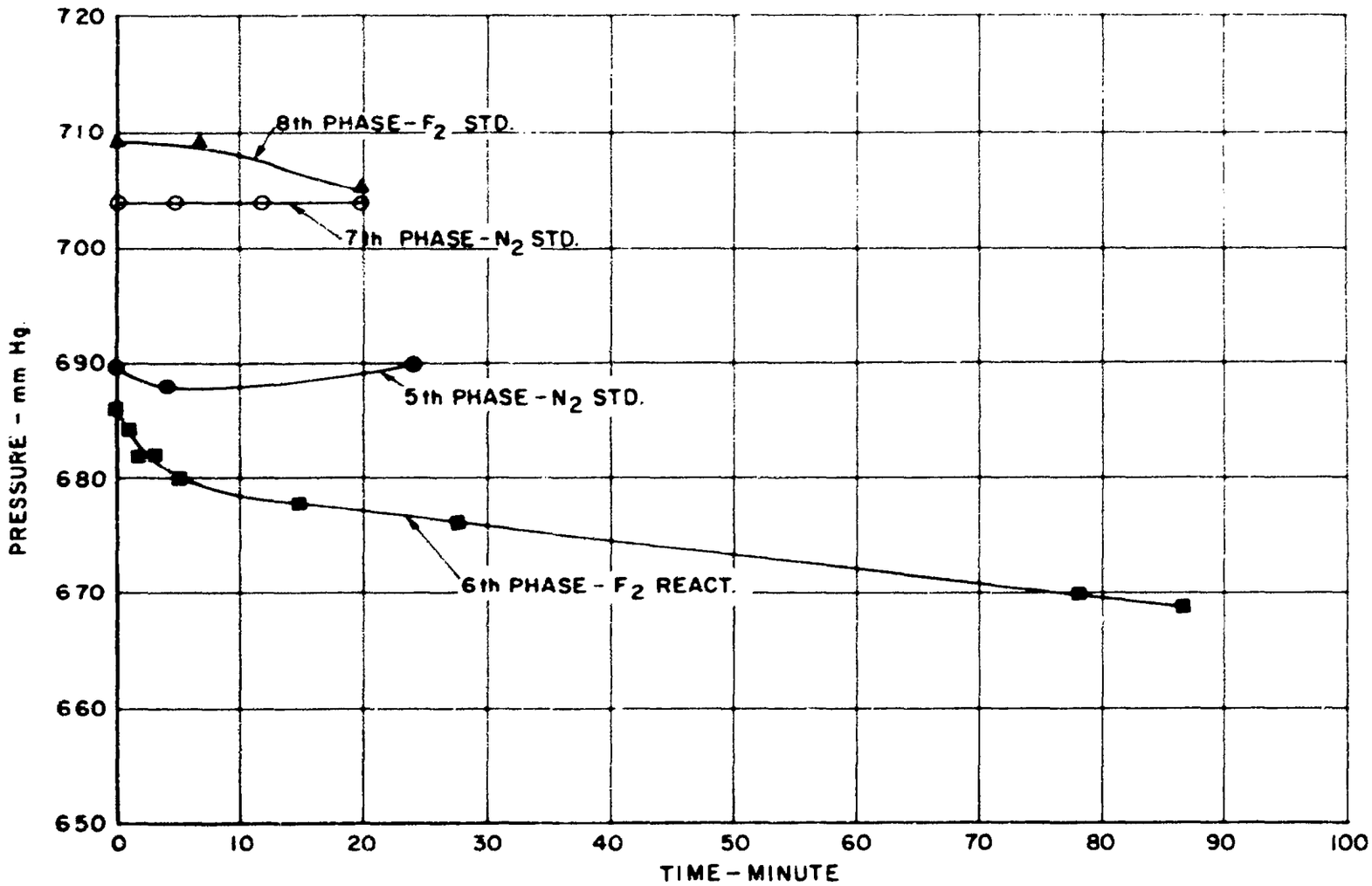


FIGURE 18c

RUN 54, EXPOSURE OF GASEOUS FLUORINE TO MONEL POWDER AT -297°F

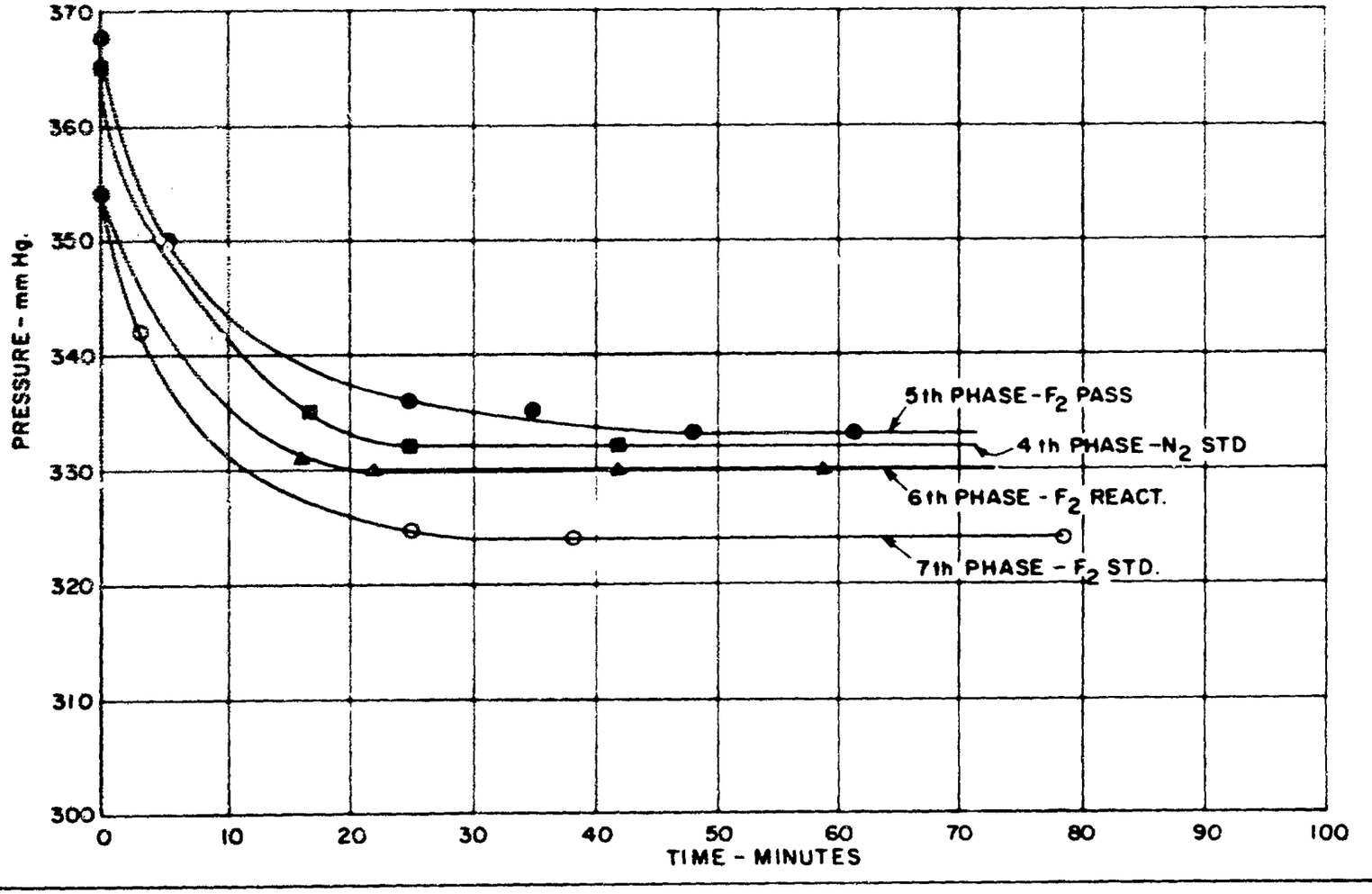
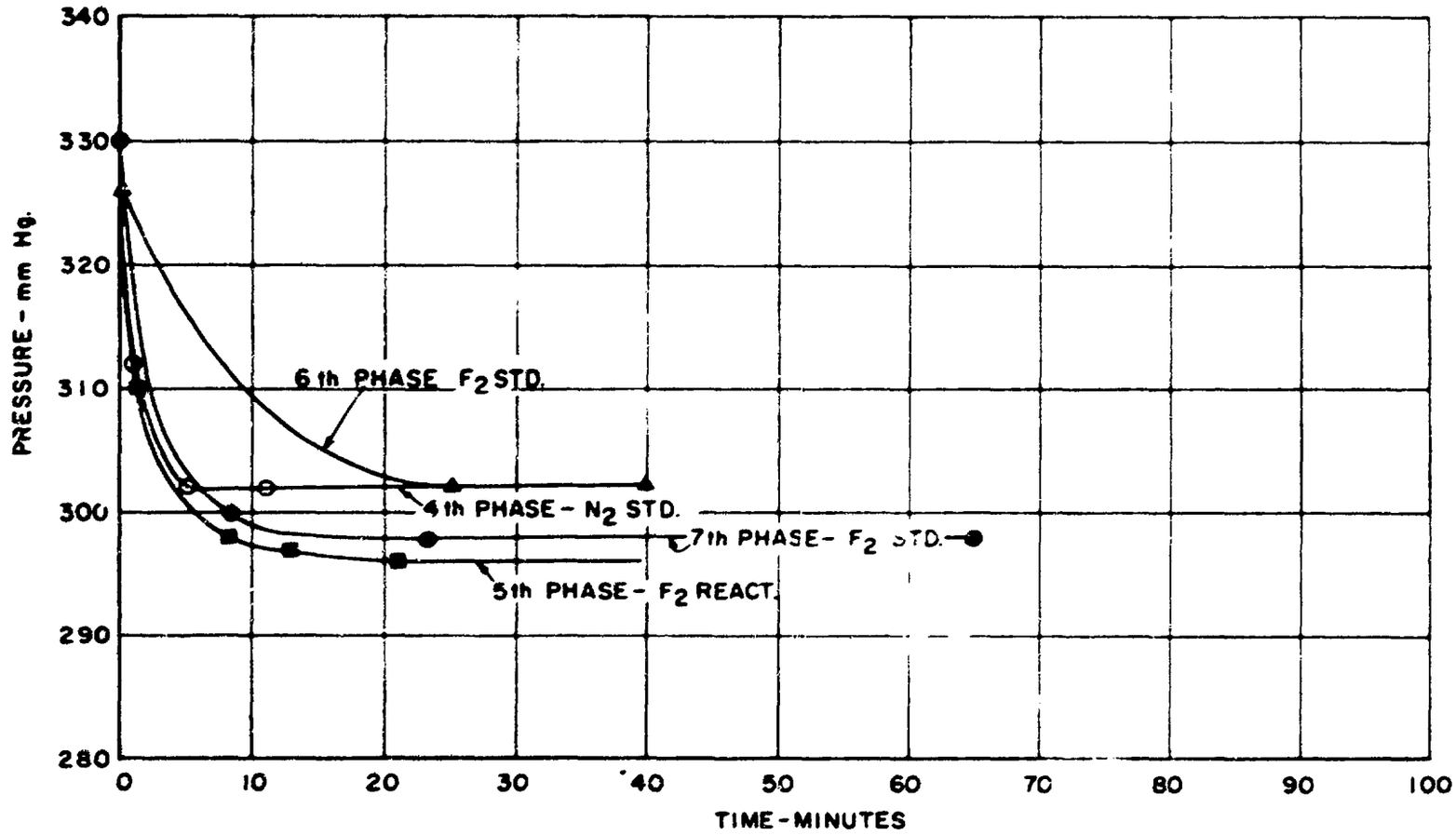


FIGURE 18d

RUN 65, EXPOSURE OF GASEOUS FLUORINE TO TITANIUM POWDER AT -297°



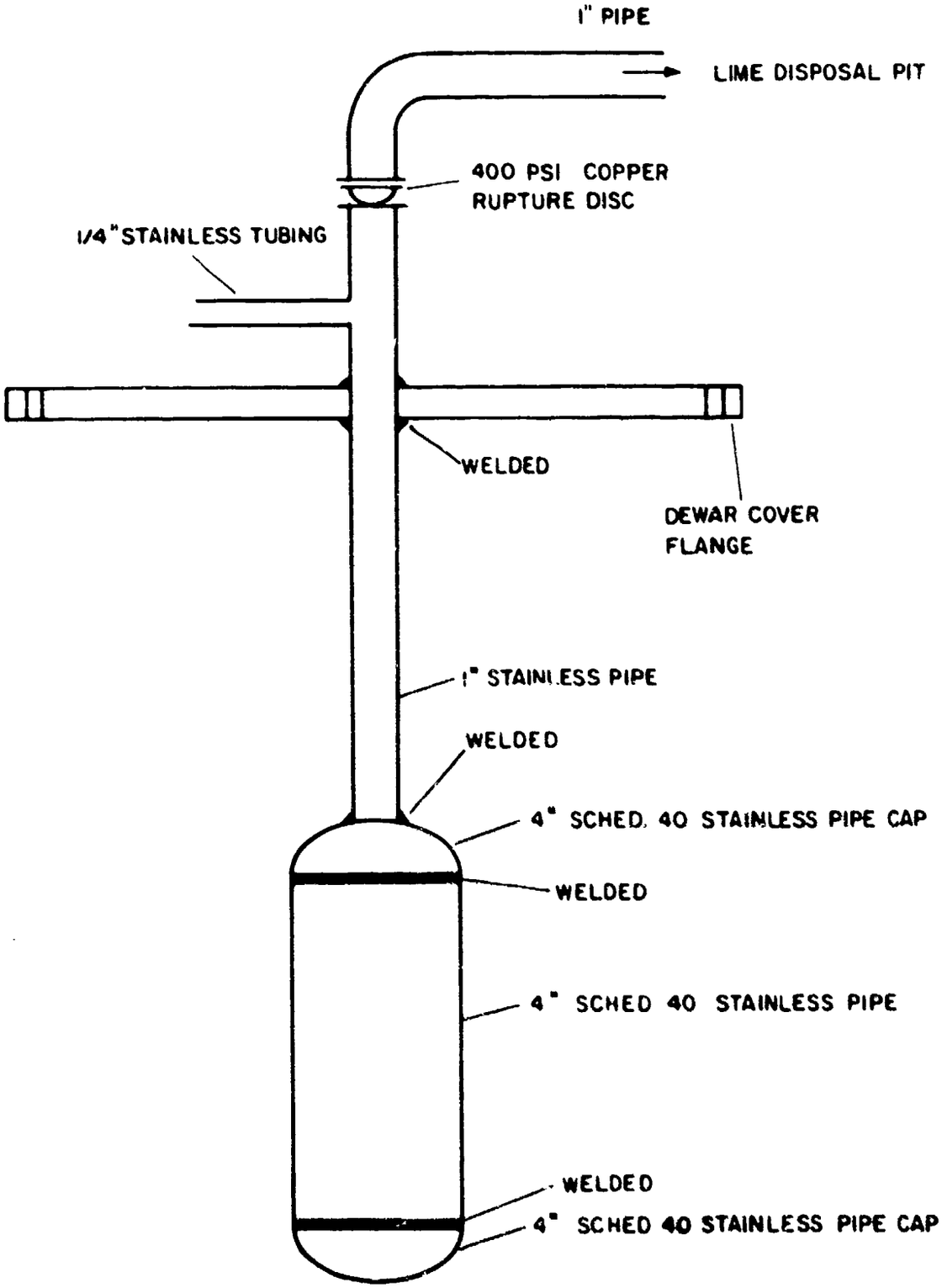


Figure 19 Liquid Fluorine Container for Tensile Specimens

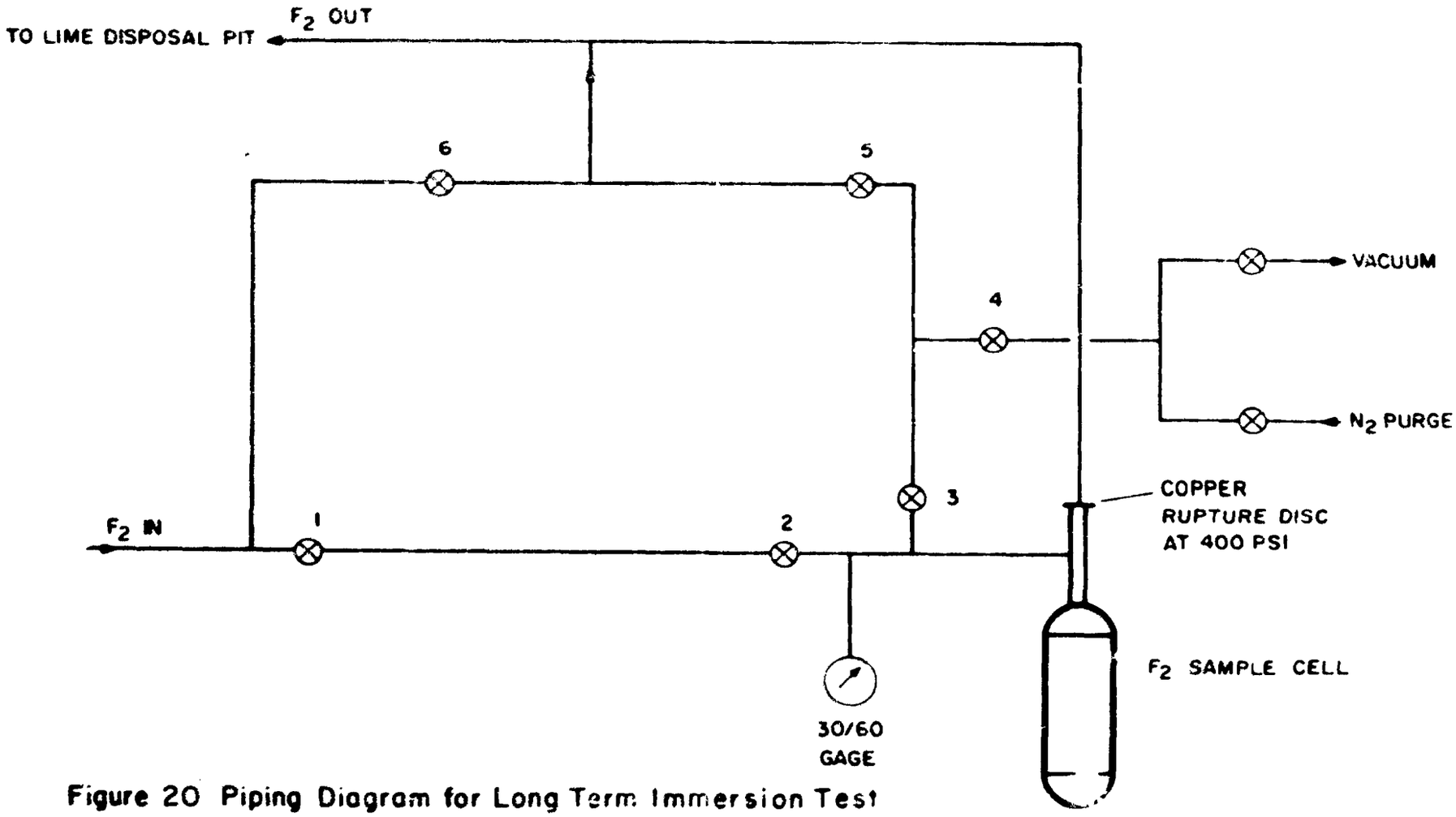


Figure 20 Piping Diagram for Long Term Immersion Test

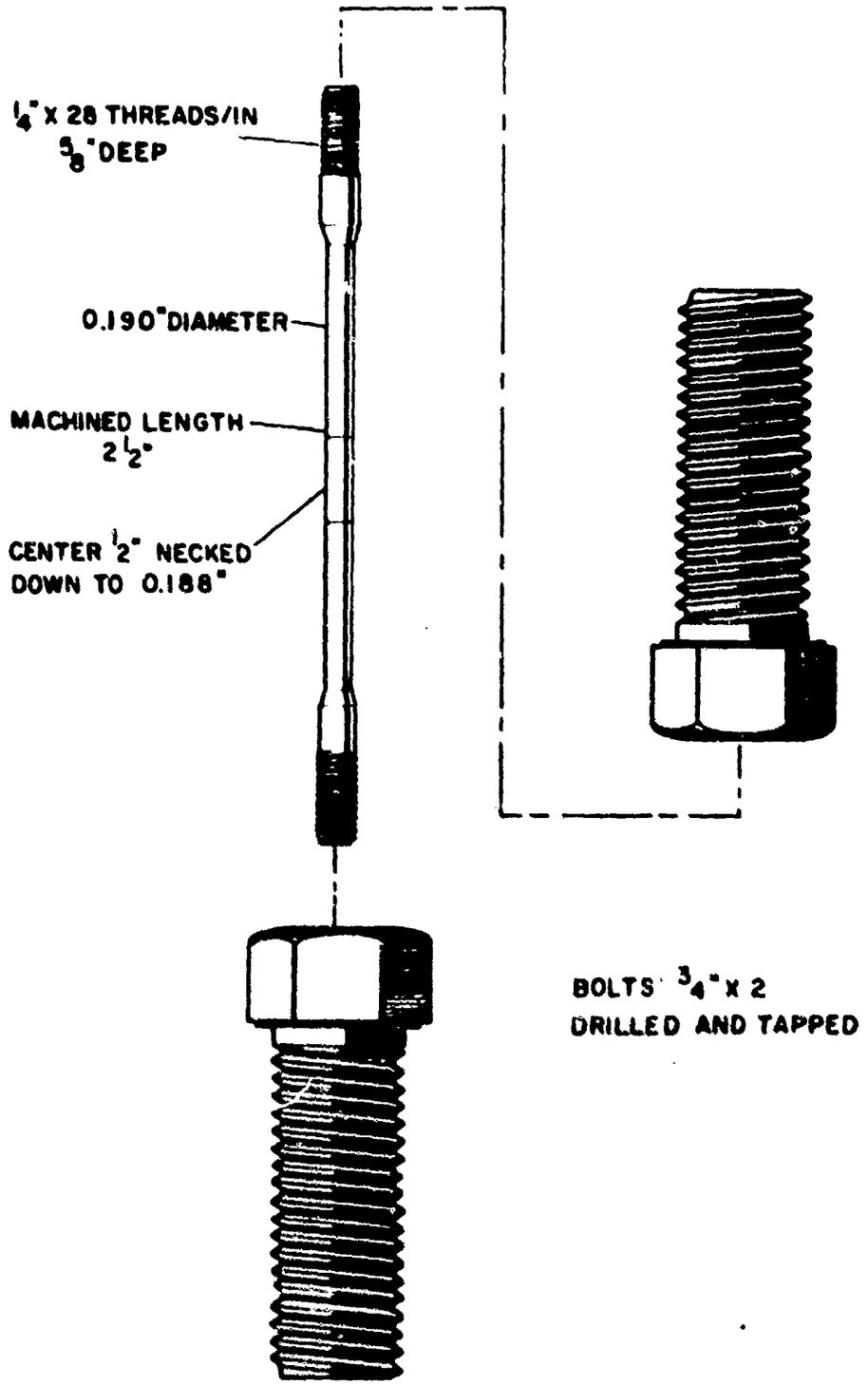


Figure 21 Diagram of Typical Machined Tensile Specimen used for One-Year Immersion Tests

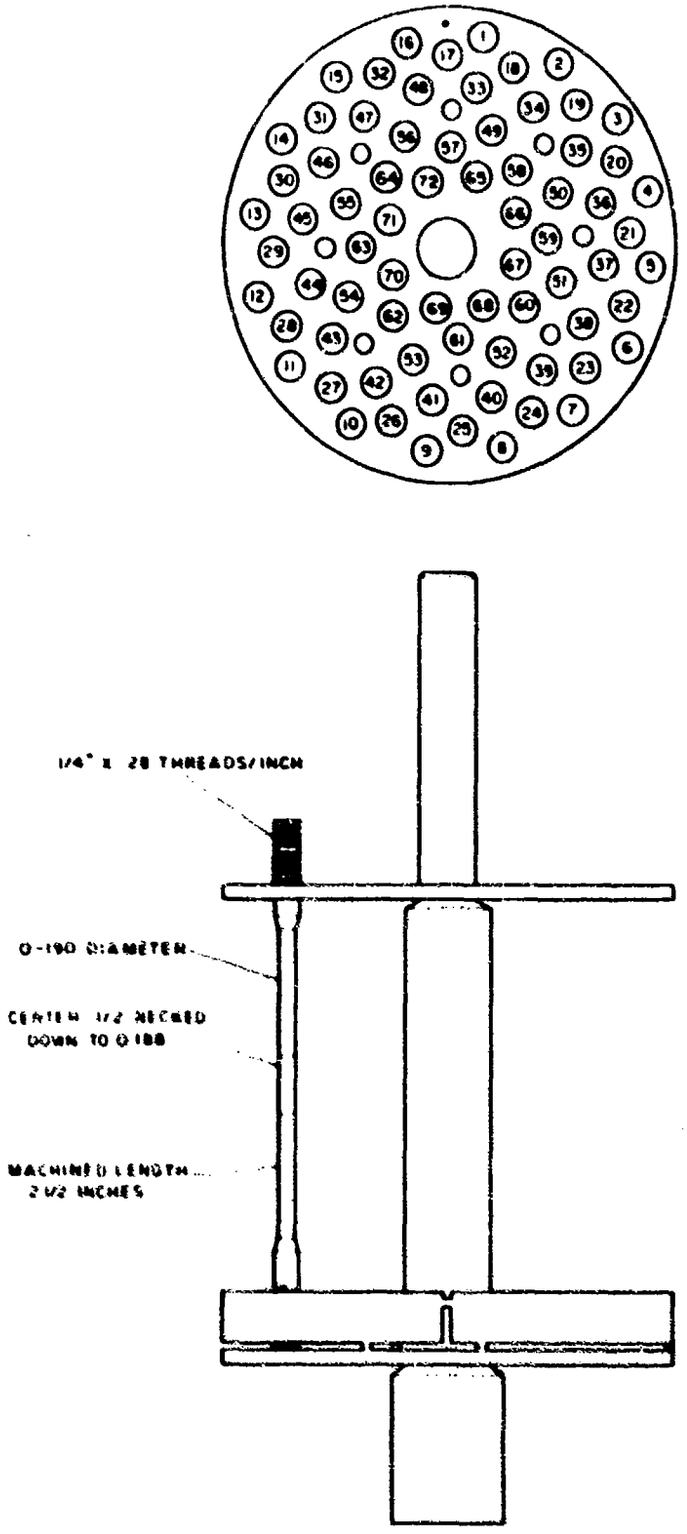


Figure 22. Rack for Holding Tensile Specimens

APPENDIX II

TABLES

TABLE I

CONTAMINANT IDENTIFICATION IN GASEOUS FLUORINE AND PREPARATION OF PURE FLUORINE

Sample No.	Remarks	
21	Fluorine gas taken directly from cylinder. Fluorine boiled and vacuum was pulled on the system at liquid oxygen temperature. Residue analyzed.	SO_2F_2, SiF_4, SF_6, CF_4, and CO_2
22	Cylinder fluorine passed through NaF trap and boiled; vacuum pulled on the system at liquid oxygen temperature. Residue analyzed.	SO_2F_2, SiF_4, SF_6, CF_4, and CO_2
23	Fluorine gas taken from cylinder. Fluorine boiled at liquid oxygen temperature. Contaminants were recondensed at -330° F (vacuum liquid nitrogen bath). Further vacuum was pulled and temperature reduced to -336° F. Residue analyzed.	Less SO_2F_2 than above. More SiF_4, SF_6, CF_4, and CO_2
24	Duplicate of previous run.	SO_2F_2, SiF_4, SF_6, CF_4, CO_2, and OF_2
25	Recovered fluorine from which contaminants were removed. Fluorine stored in stainless steel cylinder.	0.7% HF
26	Passivated system with fluorine from sample 25. Evacuated passivated receiver before distillation. Collected pure fluorine.	HF - nil
27	Stored sample in stainless steel cylinder over weekend.	0.1% HF
28	Filtered fluorine sample.	0.1% HF
29	Sample 28 boiled away at liquid oxygen temperature. Residue collected.	Trace of HF
30	Collected cylinder fluorine in a Monel cell and boiled the fluorine at liquid oxygen temperature. Residue analyzed.	SO_2F_2, SF_6, CF_4, trace of SiF_4 and HF

TABLE II

DETERMINATION OF SOLUBILITY OF CONTAMINANTS IN LIQUID FLUORINE

Run Date	Composition of Gas Mix	Temp. °F	Appearance	Remarks
1/25, 26, 27	. . . . .	. . . .		Set up system used fo
1/30	F_2 = 95% CF_4 = 5%	-320	Liquid clear with a few small particles floating in the liquid. Liquid was yellowish orange in color.	Explosion at filter ga until after the run. L must have been conta liquid N_2, explaining orange color.
1/31	F_2 = 94.3% CF_4 = 5.7%	-320	Liquid was clear for approximately 15 minutes. Fine particles were then observed in the liquid. Liquid was orange in color.	. . . . .
2/2	F_2 = 90% CF_4 = 10%	-320	Liquid cloudy, full of fine particles.	. . . . .
2/6	F_2 = 93% CF_4 = 7%	-320	Liquid cloudy.	After condensation, n appeared until approx
2/7	F_2 = 95% CF_4 = 5%	-320	Liquid was clear and then turned cloudy.	After condensation the contained fine particle
2/8	F_2 = 97% CF_4 = 3%	-320	Liquid remained clear, no solids noticeable.	. . . . .
2/9	F_2 = 95% CF_4 = 5%	-320	Liquid cloudy, full of fine particles.	. . . . .
2/10	F_2 = 96% CF_4 = 4%	-320	Liquid clear.	No solids appeared un mately 1/4 of the liqui away.
2/13	F_2 = 95.5% CF_4 = 4.5%	-320	Liquid clear.	No solids appeared un mately 1/2 of the liqui away.
6/8	SiF_4 = 3% F_2 = 97%	-320	Liquid; orange in color and cloudy. White solids along sides of test cell, the depth of the liquid. Also a deposit on the bottom of the cell. The vapor space above the liquid was yellowish in color.	. . . . .
6/9	SiF_4 = 1% F_2 = 99%	-320	The appearance of test cell was similar to the previous test (6/8) but less solids were observed.	. . . . .
6/13	SiF_4 = .2% F_2 = 99.8%	-320	The appearance of test cell was similar to the two previous runs. Less solids than in test of 6/9.	Discovered a leak. T peated.

TABLE II

TERMINATION OF SOLUBILITY OF CONTAMINANTS IN LIQUID FLUORINE

ion Mix	Temp. °F	Appearance	Remarks
	. . . .		Set up system used for solubility runs.
%	-320	Liquid clear with a few small particles floating in the liquid. Liquid was yellowish orange in color.	Explosion at filter gasket not detected until after the run. Liquid mixture must have been contaminated with liquid N_2, explaining the yellowish orange color.
3% 7%	-320	Liquid was clear for approximately 15 minutes. Fine particles were then observed in the liquid. Liquid was orange in color.	. . . . .
%	-320	Liquid cloudy, full of fine particles.	. . . . .
%	-320	Liquid cloudy.	After condensation, no particles appeared until approximately 15 minutes.
%	-320	Liquid was clear and then turned cloudy.	After condensation the mixture contained fine particles
	-320	Liquid remained clear, no solids noticeable.	. . . . .
	-320	Liquid cloudy, full of fine particles.	. . . . .
	-320	Liquid clear.	No solids appeared until approximately 1/4 of the liquid had boiled away.
5% 5%	-320	Liquid clear.	No solids appeared until approximately 1/2 of the liquid had boiled away.
	-320	Liquid; orange in color and cloudy. White solids along sides of test cell, the depth of the liquid. Also a deposit on the bottom of the cell. The vapor space above the liquid was yellowish in color.	. . . . .
	-320	The appearance of test cell was similar to the previous test (6/8) but less solids were observed.	. . . . .
% %	-320	The appearance of test cell was similar to the two previous runs. Less solids	Discovered a leak. Test will be repeated.

			orange in color.	
2/2	F_2 = 90% CF_4 = 10%	-320	Liquid cloudy, full of fine particles.	. . . . .
2/6	F_2 = 93% CF_4 = 7%	-320	Liquid cloudy.	After condensatio appeared until app
2/7	F_2 = 95% CF_4 = 5%	-320	Liquid was clear and then turned cloudy.	After condensation contained fine parti
2/8	F_2 = 97% CF_4 = 3%	-320	Liquid remained clear, no solids noticeable.	. . . . .
2/9	F_2 = 95% CF_4 = 5%	-320	Liquid cloudy, full of fine particles.	. . . . .
2/10	F_2 = 96% CF_4 = 4%	-320	Liquid clear.	No solids appeared mately 1/4 of the liq away.
2/13	F_2 = 95.5% CF_4 = 4.5%	-320	Liquid clear.	No solids appeared mately 1/2 of the liq away.
3/8	SiF_4 = 3% F_2 = 97%	-320	Liquid; orange in color and cloudy. White solids along sides of test cell, the depth of the liquid. Also a deposit on the bottom of the cell. The vapor space above the liquid was yellowish in color.	. . . . .
6/9	SiF_4 = 1% F_2 = 99%	-320	The appearance of test cell was similar to the previous test (6/8) but less solids were observed.	. . . . .
6/13	SiF_4 = .2% F_2 = 99.8%	-320	The appearance of test cell was similar to the two previous runs. Less solids than in test of 6/9.	Discovered a leak. peated.
6/15	SiF_4 = .2%	-320	No change in appearance from previous test (6/13).	. . . . .
6/17	SF_6 = 3% F_2 = 97%	-320	Liquid appeared cloudy. White solids on sides and bottom of cell. Vapor above cell appears yellow.	. . . . .
7/11	SF_6 = 1.3% F_2 = 98.7%	-320	Liquid appeared cloudy as it condensed. White solids on bottom of the cell.	. . . . .
7/17	SF_6 = 0.2% F_2 = 99.8%	-320	No solids floating in liquid, but liquid is is not clear. Solids on bottom of cell.	. . . . .
7/17	SF_6 = 0.1% F_2 = 99.9%	-320	Liquid full of fine particles. Solids on bottom of cell.	. . . . .

-320	Liquid cloudy, full of fine particles.	. . . . .
-320	Liquid cloudy.	After condensation, no particles appeared until approximately 15 minutes.
-320	Liquid was clear and then turned cloudy.	After condensation the mixture contained fine particles
-320	Liquid remained clear, no solids noticeable.	. . . . .
-320	Liquid cloudy, full of fine particles.	. . . . .
-320	Liquid clear.	No solids appeared until approximately 1/4 of the liquid had boiled away.
-320	Liquid clear.	No solids appeared until approximately 1/2 of the liquid had boiled away.
-320	Liquid; orange in color and cloudy. White solids along sides of test cell, the depth of the liquid. Also a deposit on the bottom of the cell. The vapor space above the liquid was yellowish in color.	. . . . .
-320	The appearance of test cell was similar to the previous test (6/8) but less solids were observed.	. . . . .
-320	The appearance of test cell was similar to the two previous runs. Less solids than in test of 6/9.	Discovered a leak. Test will be repeated.
-320	No change in appearance from previous test (6/13).	. . . . .
-320	Liquid appeared cloudy. White solids on sides and bottom of cell. Vapor above cell appears yellow.	. . . . .
-320	Liquid appeared cloudy as it condensed. White solids on bottom of the cell.	. . . . .
-320	No solids floating in liquid, but liquid is is not clear. Solids on bottom of cell.	. . . . .
-320	Liquid full of fine particles. Solids on bottom of cell.	. . . . .

TABLE III

PASSIVATION OF CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F_2-

Run No.	Alloy	Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Rem
262	Nickel	F_2	19.8" Hg.	1.1 mg.	1.6 mg.	None	Thick white film
263	Nickel	F_2	18 " Hg.	1.2 mg.	.05 mg.	None	Thick white film
264	Nickel	F_2	18" Hg.	0.6 mg.	.7 mg.	None	Thick white film
265	Nickel	F_2	18" Hg.	1.5 mg.	2.6 mg.	None	Heavy oil preser thick white film.
266	Nickel	F_2	18" Hg.	1.9 mg.	2.7 mg.	Moderate	Film oil, thick
267	Nickel	F_2	18" Hg.	3.1 mg.	5.3 mg.	Moderate	Thick layers of
268	Al 6061	F_2	10" Hg.	0.2 mg.	.2 mg.	None	No change visib
269	Al 1100	F_2	10" Hg.	0.2 mg.	.3 mg.	None	No change visib
270	Al 1100	F_2	10" Hg.	8.7 mg.	2.5 mg.	Moderate	Oil film like vas
271	Al 6061	F_2	10" Hg.	5.6 mg.	9.6 mg.	Moderate	Oil film like vas
272	Al 1100	F_2	10 psig	5.1 mg.	10.1 mg.	Moderate	White film.
273	Al 6061	F_2	10 psig	15.4 mg.	18.5 mg.	Moderate	White film, spo
274	A-110-AT	F_2	10 psig	5.7 mg.	16.4 mg.	Moderate	Thick oil film,
275	A-110-AT	F_2	10 psig	4.4 mg.	14.0 mg.	Moderate	Thick oil film,
276	A-110-AT	F_2	10 psig	13.0 mg.	15.9 mg.	Moderate	Dish black, wh
277	A-1100	F_2	1 atms.	16.2 mg.	16.4 mg.	Moderate	Dish completely
278	Nickel	F_2	10 psi	19.5 mg.	36.2 mg.	Moderate	Heavy oil film.
279	A-110-AT	F_2	10 psig	14.8 mg.	10.2 mg.	Moderate	Circled area of
280	AL-1100	F_2	10 psig	12.1 mg.	13.5 mg.	Moderate	Black scars.
281	Nickel	F_2	10 psig	24.7 mg.	20.1 mg.	Heavy	Black scars on
282	Al 6061	F_2	10 psi	28.2 mg.	27.1 mg.	Moderate	Black heavy gr
283	Nickel	F_2	10 psi	29.6 mg.	26.1 mg.	Moderate	Black spots on
284	Nickel	F_2	10 psi	38.2 mg.	34.6 mg.	Heavy	Heavy black gr
285	Nickel	F_2	10 psi	19.1 mg.	30.4 mg.	Moderate	Gold film prese

TABLE III

N OF CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F_2-ClF_3 MIXTURES

Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Remarks
F_2	19.8" Hg.	1.1 mg.	1.6 mg.	None	Thick white film on dish.
F_2	18 " Hg.	1.2 mg.	.05 mg.	None	Thick white film on dish.
F_2	18" Hg.	0.6 mg.	.7 mg.	None	Thick white film.
F_2	18" Hg.	1.5 mg.	2.6 mg.	None	Heavy oil present on both corners, thick white film.
F_2	18" Hg.	1.9 mg.	2.7 mg.	Moderate	Film oil, thick white film.
F_2	18" Hg.	3.1 mg.	5.3 mg.	Moderate	Thick layers of oil, white film.
F_2	10" Hg.	0.2 mg.	.2 mg.	None	No change visible.
F_2	10" Hg.	0.2 mg.	.3 mg.	None	No change visible.
F_2	10" Hg.	8.7 mg.	2.5 mg.	Moderate	Oil film like vasoline, white film.
F_2	10" Hg.	5.6 mg.	9.6 mg.	Moderate	Oil film like vasoline, white film.
F_2	10 psig	5.1 mg.	10.1 mg.	Moderate	White film.
F_2	10 psig	15.4 mg.	18.5 mg.	Moderate	White film, spotted.
F_2	10 psig	5.7 mg.	16.4 mg.	Moderate	Thick oil film, white film.
F_2	10 psig	4.4 mg.	14.0 mg.	Moderate	Thick oil film, white film.
F_2	10 psig	13.0 mg.	15.9 mg.	Moderate	Dish black, white film.
F_2	1 atms.	16.2 mg.	16.4 mg.	Moderate	Dish completely black.
F_2	10 psi	19.5 mg.	36.2 mg.	Moderate	Heavy oil film, white film.
F_2	10 psig	14.8 mg.	10.2 mg.	Moderate	Circled area of black coke on coupon.
F_2	10 psig	12.1 mg.	13.5 mg.	Moderate	Black scars.
F_2	10 psig	24.7 mg.	20.1 mg.	Heavy	Black scars on face.
F_2	10 psi	28.2 mg.	27.1 mg.	Moderate	Black heavy grit on face.
F_2	10 psi	29.6 mg.	26.1 mg.	Moderate	Black spots on white film.
F_2	10 psi	38.2 mg.	34.6 mg.	Heavy	Heavy black grit on face.
F_2	10 psi	19.1 mg.	30.4 mg.	Moderate	Gold film present on surface.

267	Nickel	F_2	18" Hg.	3.1 mg.	5.3 mg.	Moderate	Thick layers of oil,
268	Al 6061	F_2	10" Hg.	0.2 mg.	.2 mg.	None	No change visible.
269	Al 1100	F_2	10" Hg.	0.2 mg.	.3 mg.	None	No change visible
270	Al 1100	F_2	10" Hg.	8.7 mg.	2.5 mg.	Moderate	Oil film like vasoli
271	Al 6061	F_2	10" Hg.	5.6 mg.	9.6 mg.	Moderate	Oil film like vasoli
272	Al 1100	F_2	10 psig	5.1 mg.	10.1 mg.	Moderate	White film.
273	Al 6061	F_2	10 psig	15.4 mg.	18.5 mg.	Moderate	White film, spotted
274	A-110-AT	F_2	10 psig	5.7 mg.	16.4 mg.	Moderate	Thick oil film, whi
275	A-110-AT	F_2	10 psig	4.4 mg.	14.0 mg.	Moderate	Thick oil film, whi
276	A-110-AT	F_2	10 psig	13.0 mg.	15.9 mg.	Moderate	Dish black, white f
277	A-1100	F_2	1 atms.	16.2 mg.	16.4 mg.	Moderate	Dish completely bl
278	Nickel	F_2	10 psi	19.5 mg.	36.2 mg.	Moderate	Heavy oil film, whi
279	A-110-AT	F_2	10 psig	14.8 mg.	10.2 mg.	Moderate	Circled area of bla
280	AL-1100	F_2	10 psig	12.1 mg.	13.5 mg.	Moderate	Black scars.
281	Nickel	F_2	10 psig	24.7 mg.	20.1 mg.	Heavy	Black scars on face
282	Al 6061	F_2	10 psi	28.2 mg.	27.1 mg.	Moderate	Black heavy grit on
283	Nickel	F_2	10 psi	29.6 mg.	26.1 mg.	Moderate	Black spots on whit
284	Nickel	F_2	10 psi	38.2 mg.	34.6 mg.	Heavy	Heavy black grit on
285	Nickel	F_2	10 psi	19.1 mg.	30.4 mg.	Moderate	Gold film present o
286	Monel	F_2	10 psi	18.8 mg.	8.5 mg.	Moderate	Black film on surfa
287	Nickel	F_2	16 psi	18.7 mg.	9.9 mg.	Heavy	. . . . .
288	Monel	F_2	10 psi	16.3 mg.	10.5 mg.	Heavy	Black surface
289	.001 Monel	F_2	10 psi	32.3 mg.	. . . . .	Heavy	Heavy corrosion on and yellow fluorides
290	.001 Monel	F_2	10 psi	59.9 mg.	. . . . .	Heavy	Complete black film
291	.001 Monel	F_2	10 psi	21.1 mg.	. . . . .	Heavy	Black grit with yell
292	.001 Monel	F_2	10 psi	. . . . .	. . . . .	Heavy	Yellowish green fluo
293	.001 Monel	F_2	10 psig	1.7 mg.	. . . . .	Heavy	Completely full of f
294	.001 Monel	F_2	10 psig	1.7 mg.	. . . . .	Heavy	Completely full of f

(Continued)

F_2	10" Hg.	0.2 mg.	.2 mg.	None	No change visible.
F_2	10" Hg.	0.2 mg.	.3 mg.	None	No change visible.
F_2	10" Hg.	8.7 mg.	2.5 mg.	Moderate	Oil film like vasoline, white film.
F_2	10" Hg.	5.6 mg.	9.6 mg.	Moderate	Oil film like vasoline, white film.
F_2	10 psig	5.1 mg.	10.1 mg.	Moderate	White film.
F_2	10 psig	15.4 mg.	18.5 mg.	Moderate	White film, spotted.
F_2	10 psig	5.7 mg.	16.4 mg.	Moderate	Thick oil film, white film.
F_2	10 psig	4.4 mg.	14.0 mg.	Moderate	Thick oil film, white film.
F_2	10 psig	13.0 mg.	15.9 mg.	Moderate	Dish black, white film.
F_2	1 atms.	16.2 mg.	16.4 mg.	Moderate	Dish completely black.
F_2	10 psi	19.5 mg.	36.2 mg.	Moderate	Heavy oil film, white film.
F_2	10 psig	14.8 mg.	10.2 mg.	Moderate	Circled area of black coke on coupon.
F_2	10 psig	12.1 mg.	13.5 mg.	Moderate	Black scars.
F_2	10 psig	24.7 mg.	20.1 mg.	Heavy	Black scars on face.
F_2	10 psi	28.2 mg.	27.1 mg.	Moderate	Black heavy grit on face.
F_2	10 psi	29.6 mg.	26.1 mg.	Moderate	Black spots on white film.
F_2	10 psi	38.2 mg.	34.6 mg.	Heavy	Heavy black grit on face.
F_2	10 psi	19.1 mg.	30.4 mg.	Moderate	Gold film present on surface.
F_2	10 psi	18.8 mg.	8.5 mg.	Moderate	Black film on surface.
F_2	16 psi	18.7 mg.	9.9 mg.	Heavy	. . . . .
F_2	10 psi	16.3 mg.	10.5 mg.	Heavy	Black surface
F_2	10 psi	32.3 mg.	. . . . .	Heavy	Heavy corrosion on sides. Purple and yellow fluorides.
F_2	10 psi	59.9 mg.	. . . . .	Heavy	Complete black film.
F_2	10 psi	21.1 mg.	. . . . .	Heavy	Black grit with yellow fluoride.
F_2	10 psi	. . . . .	. . . . .	Heavy	Yellowish green fluoride.
F_2	10 psig	1.7 mg.	. . . . .	Heavy	Completely full of fluorides.
F_2	10 psig	1.7 mg.	. . . . .	Heavy	Completely full of fluorides.

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TABLE III (Continued)

CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F_2-ClF_3 MIXTURES

Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Remarks
F_2	10 psi	.4 mg	.....	.....	Black spots.
F_2	10 psi	.2 mg.	.1 mg.	.....	Black spots.
F_2	10 psig	.2 mg.	.....	None	Clean.
F_2	10 psig	.....	.....	None	Clean.
F_2	10 psig	2.6 mg.	.....	Moderate	Heavy black area.
F_2	10 psig	2.0 mg.	.....	Moderate	Heavy black area.
F_2	10 psig	20.9 mg.	.....	Heavy	Heavy black film.
F_2	10 psig	43.3 mg.	.....	Heavy	Scattered black films.
F_2	10 psi	1.5 mg.	.....	Moderate	Shiny.
F_2	10 psi	1.5 mg.	.....	Moderate	20% black.
F_2	10 psi	3.4 mg.	.....	Moderate	Oil turned to a gold color - 85% of area.
F_2	10 psi	3.3 mg.	.....	Moderate	Oil turned to black about 75% of area.
F_2	10 psi	29.5 mg.	.....	Moderate	100% black.
F_2	10 psig	44.6 mg.	.....	Heavy	Sample black, spotted.
F_2	10 psig	28.5 mg.	.....	Moderate	Sample 100% black.
F_2	10 psig	40.2 mg.	.....	Moderate	Sample 100% black.
F_2	10 psig	5.2 mg.	.....	Moderate	Sample 60% black.
F_2	10 psi	4.6 mg.	.....	Moderate	Sample 40% black.
F_2	10 psi	93.6 mg.	.....	Heavy	Sample 100% black.
F_2	10 psi	62.4 mg.	.....	Heavy	Sample 100% black.
F_2	10 psi	70.9 mg.	8.1 mg.	Heavy	Sample 100% black.
F_2	10 psi	57.4 mg.	4.9 mg.	Heavy	Sample 100% black.
F_2	25 psi	67.7 mg.	.....	Heavy	Sample 90% black.
F_2	25 psi	61.6 mg.	.....	Heavy	Sample 90% black.

300	.005 Cu.	F_2	10 psig	2.0 mg.	. . . . .	Moderate	Heavy black area.
301	.005 Cu.	F_2	10 psig	20.9 mg.	. . . . .	Heavy	Heavy black film.
302	.005 Cu.	F_2	10 psig	43.3 mg.	. . . . .	Heavy	Scattered black film
303	.005 Cu.	F_2	10 psi	1.5 mg.	. . . . .	Moderate	Shiny.
304	.005 Cu.	F_2	10 psi	1.5 mg.	. . . . .	Moderate	20% black.
305	.005 Cu.	F_2	10 psi	3.4 mg.	. . . . .	Moderate	Oil turned to a gold co
306	.005 Cu.	F_2	10 psi	3.3 mg.	. . . . .	Moderate	Oil turned to black ab
307	.010 Cu.	F_2	10 psi	29.5 mg.	. . . . .	Moderate	100% black.
308	.010 Cu.	F_2	10 psig	44.6 mg.	. . . . .	Heavy	Sample black, spotted
309	.010 Cu.	F_2	10 psig	28.5 mg.	. . . . .	Moderate	Sample 100% black.
310	.010 Cu.	F_2	10 psig	40.2 mg.	. . . . .	Moderate	Sample 100% black.
311	.010 Cu.	F_2	10 psig	5.2 mg.	. . . . .	Moderate	Sample 60% black.
312	.010 Cu.	F_2	10 psi	4.6 mg.	. . . . .	Moderate	Sample 40% black.
313	.010 Cu.	F_2	10 psi	93.6 mg.	. . . . .	Heavy	Sample 100% black.
314	.010 Cu.	F_2	10 psi	62.4 mg.	. . . . .	Heavy	Sample 100% black.
315	.005 Cu.	F_2	10 psi	70.9 mg.	8.1 mg.	Heavy	Sample 100% black.
316	.005 Cu.	F_2	10 psi	57.4 mg.	4.9 mg.	Heavy	Sample 100% black.
317	.005 Cu.	F_2	25 psi	67.7 mg.	. . . . .	Heavy	Sample 90% black.
318	.005 Cu.	F_2	25 psi	61.6 mg.	. . . . .	Heavy	Sample 90% black.
319	.005 Brass	F_2	50 psi	54.9 mg.	. . . . .	. . . . .	Sample 50% black.
320	.005 Brass	F_2	50 psi	72.6 mg.	. . . . .	. . . . .	Sample 75% black.
321	.005 Cu.	F_2	60 psi	64.7 mg.	4.2 mg.	Heavy	Sample 30% black.
322	.005 Cu.	F_2	60 psi	116.6 mg.	4.7 mg.	Heavy	Sample 90% black.
323	.001 Monel	ClF_3	?	14.02 mg.	. . . . .	Heavy	Some of the carbon on di
324	.001 Monel	ClF_3	?	14.02 mg.	. . . . .	Heavy	Some of the oily materia
325	.001 Monel	ClF_3	1 atms.	14.02 mg.	. . . . .	Heavy	. . . . .
326	.001 Monel	ClF_3	1 atms.	14.02 mg.	. . . . .	Heavy	Small pool of liquid on di
327	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Bottom side of dish show yellow fluorides.

(Continued)

$_2$	10 psig	2.0 mg.	. . . . .	Moderate	Heavy black area.
$_2$	10 psig	20.9 mg.	. . . . .	Heavy	Heavy black film.
$_2$	10 psig	43.3 mg.	. . . . .	Heavy	Scattered black films.
$_2$	10 psi	1.5 mg.	. . . . .	Moderate	Shiny,
$_2$	10 psi	1.5 mg.	. . . . .	Moderate	20% black.
$_2$	10 psi	3.4 mg.	. . . . .	Moderate	Oil turned to a gold color - 85% of area.
$_2$	10 psi	3.3 mg.	. . . . .	Moderate	Oil turned to black about 75% of area.
$_2$	10 psi	29.5 mg.	. . . . .	Moderate	100% black.
$_2$	10 psig	44.6 mg.	. . . . .	Heavy	Sample black, spotted.
$_2$	10 psig	28.5 mg.	. . . . .	Moderate	Sample 100% black.
$_2$	10 psig	40.2 mg.	. . . . .	Moderate	Sample 100% black.
$_2$	10 psig	5.2 mg.	. . . . .	Moderate	Sample 60% black.
$_2$	10 psi	4.6 mg.	. . . . .	Moderate	Sample 40% black.
$_2$	10 psi	93.6 mg.	. . . . .	Heavy	Sample 100% black.
$_2$	10 psi	62.4 mg.	. . . . .	Heavy	Sample 100% black.
$_2$	10 psi	70.9 mg.	8.1 mg.	Heavy	Sample 100% black.
$_2$	10 psi	57.4 mg.	4.9 mg.	Heavy	Sample 100% black.
$_2$	25 psi	67.7 mg.	. . . . .	Heavy	Sample 90% black.
$_2$	25 psi	61.6 mg.	. . . . .	Heavy	Sample 90% black.
$_2$	50 psi	54.9 mg.	. . . . .	. . . . .	Sample 50% black.
$_2$	50 psi	72.6 mg.	. . . . .	. . . . .	Sample 75% black.
$_2$	60 psi	64.7 mg.	4.2 mg.	Heavy	Sample 30% black.
$_2$	60 psi	116.6 mg.	4.7 mg.	Heavy	Sample 90% black.
F_3	?	14.02 mg.	. . . . .	Heavy	Some of the carbon on dish was flaky.
F_3	?	14.02 mg.	. . . . .	Heavy	Some of the oily material still on dish.
F_3	1 atms.	14.02 mg.	. . . . .	Heavy	. . . . .
F_3	1 atms.	14.02 mg.	. . . . .	Heavy	Small pool of liquid on dish.
F_3	5 psig	14.02 mg.	. . . . .	Moderate	Bottom side of dish showed some yellow fluorides.

1 89 1

TABLE III (Continued)

PASSIVATION OF CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F_2-ClF_3

Run No.	Alloy	Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Remarks
328	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	None	. . . . .
329	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Some of the carbon wa and crumbled up.
330	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Light	. . . . .
331	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Yellow and mostly whi over top and bottom of side of test cell were
332	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Heavy string-shaped c on top of dish, with ye fluorides on bottom si
333	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Inside of test cell was
334	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	None	No carbon or fluoride
335	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped c on top of dish.
336	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped c on top of dish.
337	.010 Cu.	ClF_3	9 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped c on top of dish.
338	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	None	Heavy string-shaped c on top of dish.
339	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped c on top of dish.
340	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	None	Heavy string-shaped c on top of dish.
341	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped c on top of dish.
342	.010 Cu.	ClF_3	5 psig	1.8 mg.	. . . . .	None	No carbon or fluoride
343	.010 Cu.	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluoride
344	.010 Cu.	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluoride
345	.010 Cu.	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluoride
[illegible]	.001 Monel	ClF_3	[illegible] psig	1.8 mg.	[illegible]	None	No carbon or fluoride

1 89 1 2

TABLE III (Continue.)

ONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F_2-ClF_3 MIXTURES

as sed	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Remarks
ClF_3	5 psig	14.02 mg.	. . . . .	None	. . . . .
ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Some of the carbon was string-shaped and crumbled up.
ClF_3	5 psig	14.02 mg.	. . . . .	Light	. . . . .
ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Yellow and mostly white fluorides over top and bottom of dish, also inside of test cell were pools of liquid.
ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Heavy string-shaped carbon deposits on top of dish, with yellow and green fluorides on bottom side of dish.
ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Inside of test cell was completely white.
ClF_3	5 psig	14.02 mg.	. . . . .	None	No carbon or fluorides on dish.
ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped carbon deposits on top of dish.
F_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped carbon deposits on top of dish.
F_3	9 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped carbon deposits on top of dish.
F_3	5 psig	14.02 mg.	. . . . .	None	Heavy string-shaped carbon deposits on top of dish.
F_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped carbon deposits on top of dish.
F_3	5 psig	14.02 mg.	. . . . .	None	Heavy string-shaped carbon deposits on top of dish.
F_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped carbon deposits on top of dish.
F_3	5 psig	1.8 mg.	. . . . .	None	No carbon or fluoride on dish.
F_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluoride on dish.
F_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluoride on dish.
F_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluoride on dish.
F_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluoride on dish.

							over top and bottom side of test cell aer-
332	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Heavy string-shaped on top of dish, with fluorides on bottom
333	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	Moderate	Inside of test cell
334	.001 Monel	ClF_3	5 psig	14.02 mg.	. . . . .	None	No carbon or fluori
335	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped on top of dish.
336	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped on top of dish.
337	.010 Cu.	ClF_3	9 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped on top of dish.
338	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	None	Heavy string-shaped on top of dish.
339	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped on top of dish.
340	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	None	Heavy string-shaped on top of dish.
341	.010 Cu.	ClF_3	5 psig	14.02 mg.	. . . . .	Heavy	Heavy string-shaped on top of dish.
342	.010 Cu.	ClF_3	5 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
343	.010 Cu.	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
344	.010 Cu.	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
345	.010 Cu.	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
346	.001 Monel	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
347	.001 Monel	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
348	.001 Monel	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
349	.001 Monel	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
350	.001 Monel	ClF_3	4 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
351	.001 Monel	ClF_3	5 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
352	.001 Monel	ClF_3	5 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
353	.001 Monel	ClF_3	7 psig	1.8 mg.	. . . . .	None	No carbon or fluorid
354	.001 Monel	25% ClF_3 75% F_2	20 psi	1.8 mg.	. . . . .	None	No carbon or fluorid

(Continued)

to the two previous runs. Less solids [illegible] Discovered a leak. Test will be repeated.

ClF_3	5 psig	14.02 mg.	Moderate	Heavy string-shaped carbon deposits on top of dish, with yellow and green fluorides on bottom side of dish.
ClF_3	5 psig	14.02 mg.	Moderate	Inside of test cell was completely white.
ClF_3	5 psig	14.02 mg.	None	No carbon or fluorides on dish.
ClF_3	5 psig	14.02 mg.	Heavy	Heavy string-shaped carbon deposits on top of dish.
ClF_3	5 psig	14.02 mg.	Heavy	Heavy string-shaped carbon deposits on top of dish.
ClF_3	9 psig	14.02 mg.	Heavy	Heavy string-shaped carbon deposits on top of dish.
ClF_3	5 psig	14.02 mg.	None	Heavy string-shaped carbon deposits on top of dish.
ClF_3	5 psig	14.02 mg.	Heavy	Heavy string-shaped carbon deposits on top of dish.
ClF_3	5 psig	14.02 mg.	None	Heavy string-shaped carbon deposits on top of dish.
ClF_3	5 psig	14.02 mg.	Heavy	Heavy string-shaped carbon deposits on top of dish.
ClF_3	5 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	4 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	5 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	5 psig	1.8 mg.	None	No carbon or fluoride on dish.
ClF_3	7 psig	1.8 mg.	None	No carbon or fluoride on dish.
% ClF_3 % F_2	20 psi	1.8 mg.	None	No carbon or fluoride on dish.

1 p 40 1

TABLE III (Continued)

PASSIVATION OF CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, an

Run No.	Alloy	Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	
355	.001 Monel	25% ClF_3 75% F_2	20 psi	1.8 mg.	. . . . .	None	Two carbon
356	.001 Monel	25% ClF_3 75% F_2	20 psi	1.8 mg.	. . . . .	None	Spotted carb
357	.001 Monel	25% ClF_3 75% F_2	20 psi	1.8 mg.	. . . . .	None	No carbon o
358	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Dish disinte
359	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	No carbon o
360	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Dish disinte
361	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Some white
362	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
363	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
364	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
365	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
366	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
367	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
368	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
369	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon o
370	.062 Monel	25% ClF_3 75% F_2	60 psi	1.3 mg.	2.9 mg.	None	No carbon o waxy film.
371	.062 Monel	25% ClF_3	60 psi	1.3 mg.	4.2 mg.	None	No carbon

TABLE III (Continued)

F CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F_2-ClF_3 MIXTURES

Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Remarks
25% ClF_3 75% F_2	20 psi	1.8 mg.	. . . . .	None	Two carbon spots on dish.
25% ClF_3 75% F_2	20 psi	1.8 mg.	. . . . .	None	Spotted carbon spots on dish.
25% ClF_3 75% F_2	20 psi	1.8 mg.	. . . . .	None	No carbon or fluorides on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Dish disintegrated to a white powder.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	No carbon or fluorides on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Dish disintegrated to a white powder.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Some white spotted fluorides on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
25% ClF_3 75% F_2	60 psi	1.3 mg.	2.9 mg.	None	No carbon or fluoride deposits on dish, waxy film.
25% ClF_3 75% F_2	60 psi	1.2 mg.	4.2 mg.	None	No carbon or fluoride deposits on dish, waxy film.

[illegible]	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Dish disintegra
359	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	No carbon or fl
360	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Dish disintegr
361	.001 Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Some white spo
362	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fl
363	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fl
364	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fl
365	.001 Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fl
366	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fl
367	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . .	None	No carbon or fl
368	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fl
369	.005 Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fl
370	.062 Monel	25% ClF_3 75% F_2	60 psi	1.3 mg.	2.9 mg.	None	No carbon or fl waxy film.
371	.062 Monel	25% ClF_3 75% F_2	60 psi	1.2 mg.	4.2 mg.	None	No carbon or fl waxy film.
372	.062 Monel	25% ClF_3 75% F_2	60 psi	1.3 mg.	2.6 mg.	None	No carbon or fl waxy film.
373	.062 Monel	25% ClF_3 75% F_2	60 psi	1.5 mg.	3.8 mg.	None	No carbon or fl waxy film.
375	.062 Monel	F_2	60 psi	1.7 mg.	0.5 mg.	None	No carbon or fl waxy film.
376	.062 Monel	F_2	60 psi	1.2 mg.	1.0 mg.	None	No carbon or fl waxy film.
377	.062 Monel	F_2	60 psi	1.2 mg.	0.8 mg.	None	No carbon or fl waxy film.

(Continued)

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Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	No carbon or fluorides on dish.
Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Dish disintegrated to a white powder.
Monel	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	Moderate	Some white spotted fluorides on dish.
Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Brass	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Cu.	25% ClF_3 75% F_2	60 psi	1.8 mg.	. . . . .	None	No carbon or fluoride deposits on dish.
Monel	25% ClF_3 75% F_2	60 psi	1.3 mg.	2.9 mg.	None	No carbon or fluoride deposits on dish, waxy film.
Monel	25% ClF_3 75% F_2	60 psi	1.2 mg.	4.2 mg.	None	No carbon or fluoride deposits on dish, waxy film.
Monel	25% ClF_3 75% F_2	60 psi	1.3 mg.	2.6 mg.	None	No carbon or fluoride deposits on dish, waxy film.
Monel	25% ClF_3 75% F_2	60 psi	1.5 mg.	3.8 mg.	None	No carbon or fluoride deposits on dish, waxy film.
Monel	F_2	60 psi	1.7 mg.	0.5 mg.	None	No carbon or fluoride deposits on dish, waxy film.
Monel	F_2	60 psi	1.2 mg.	1.0 mg.	None	No carbon or fluoride deposits on dish, waxy film.
Monel	F_2	60 psi	1.2 mg.	0.8 mg.	None	No carbon or fluoride deposits on dish, waxy film.

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TABLE III (Continued)

PASSIVATION OF CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F

Run No.	Alloy	Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Re
378	.062 Monel	F_2	60 psi	1.3 mg.	1.4 mg.	None	No carbon or fl waxy film.
379	.062 Cu.	F_2	60 psi	1.6 mg.	3.9 mg.	None	No carbon or fl waxy film.
380	.062 Cu.	F_2	60 psi	1.8 mg.	2.4 mg.	None	No carbon or fl waxy film.
381	.062 Cu.	F_2	60 psi	1.8 mg.	2.8 mg.	None	No carbon or fl waxy film.
382	.062 Cu.	F_2	60 psi	1.3 mg.	2.9 mg.	None	No carbon or fl waxy film.
384	.062 Cu.	25% ClF_3 75% F_2	60 psi	1.0 mg.	2.0 mg.	None	No carbon or fl waxy film.
385	062 Cu.	25% ClF_3 75% F_2	60 psi	0.5 mg.	2.6 mg.	None	No carbon or f waxy film.
386	.062 Cu.	25% ClF_3 75% F_2	60 psi	0.2 mg.	3.1 mg.	None	No carbon or f waxy film.
387	.062 Cu.	25% ClF_3 75% F_2	60 psi	1.1 mg.	4.2 mg.	None	No carbon or f waxy film.
**392	.062 Cu.	25% ClF_3 +F_2	60 psi	0.8 mg.	3.6 mg.	None	No carbon or f oily liquid.
**393	.062 Cu.	25% ClF_3 +F_2	60 psi	1.7 mg.	2.5 mg.	Moderate	Carbon deposi liquid.
**394	.062 Cu.	25% ClF_3 +F_2	60 psi	1.6 mg.	2.4 mg.	Moderate	Carbon deposi liquid.
**395	.062 Cu.	25% ClF_3 +F_2	60 psi	1.1 mg.	3.5 mg.	Moderate	Carbon deposi liquid.
**396	100% Blank	Cl_4	.....	.....	.....	.....	.....
397	.062 Cu.	100% F_2	60 psi	1.6 mg.	3.4 mg.	None	No carbon or waxy solid.
398	.062 Cu.	100% F_2	60 psi	1.6 mg.	4.4 mg.	None	No carbon or waxy solid.
399	.062 Cu.	100% F_2	60 psi	1.6 mg.	3.7 mg.	None	No carbon or waxy solid.
400	.062 Cu.	100% F_2	60 psi	1.2 mg.	3.6 mg.	None	No carbon or waxy solid.

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TABLE III (Continued)

OF CONTAMINATED METAL SURFACES WITH FLUORINE, ClF_3, and F_2-ClF_3 MIXTURES

Gas Used	Pressure	Wt. of Hexadecane Before Exposure	Wt. of Residue After Exposure	Reaction* Violence	Remarks
F_2	60 psi	1.3 mg.	1.4 mg.	None	No carbon or fluoride deposits on dish, waxy film.
F_2	60 psi	1.6 mg.	3.9 mg.	None	No carbon or fluoride deposits on dish, waxy film.
F_2	60 psi	1.8 mg.	2.4 mg.	None	No carbon or fluoride deposits on dish, waxy film.
F_2	60 psi	1.8 mg.	2.8 mg.	None	No carbon or fluoride deposits on dish, waxy film.
F_2	60 psi	1.3 mg.	2.9 mg.	None	No carbon or fluoride deposits on dish, waxy film.
25% ClF_3 75% F_2	60 psi	1.0 mg.	2.0 mg.	None	No carbon or fluoride deposits on dish, waxy film.
25% ClF_3 75% F_2	60 psi	0.5 mg.	2.6 mg.	None	No carbon or fluoride deposits on dish, waxy film.
25% ClF_3 75% F_2	60 psi	0.2 mg.	3.1 mg.	None	No carbon or fluoride deposits on dish, waxy film.
25% ClF_3 75% F_2	60 psi	1.1 mg.	4.2 mg.	None	No carbon or fluoride deposits on dish, waxy film.
25% ClF_3 +F_2	60 psi	0.8 mg.	3.6 mg.	None	No carbon or fluoride deposits on dish, oily liquid.
25% ClF_3 +F_2	60 psi	1.7 mg.	2.5 mg.	Moderate	Carbon deposit on dish; spotted, oily liquid.
25% ClF_3 +F_2	60 psi	1.6 mg.	2.4 mg.	Moderate	Carbon deposit on dish; spotted, oily liquid.
25% ClF_3 +F_2	60 psi	1.1 mg.	3.5 mg.	Moderate	Carbon deposit on dish; spotted, oily liquid.
Cl_4	.....	.....	.....	.....	.....
100% F_2	60 psi	1.6 mg.	3.4 mg.	None	No carbon or fluoride deposit on dish, waxy solid.
100% F_2	60 psi	1.6 mg.	4.4 mg.	None	No carbon or fluoride deposit on dish, waxy solid.
100% F_2	60 psi	1.6 mg.	3.7 mg.	None	No carbon or fluoride deposit on dish, waxy solid.
100% F_2	60 psi	1.2 mg.	3.6 mg.	None	No carbon or fluoride deposit on dish, waxy solid.

F_2	25 psi	61.6 mg.	. . . . .	Heavy	Sample 90% black.
ClF_3 F_2	60 psi	1.0 mg.	2.0 mg.	None	No carbon or fluoride deposits on dish, waxy film.
ClF_3 F_2	60 psi	0.5 mg.	2.6 mg.	None	No carbon or fluoride deposits on dish, waxy film.
ClF_3 F_2	60 psi	0.2 mg.	3.1 mg.	None	No carbon or fluoride deposits on dish, waxy film.
ClF_3 F_2	60 psi	1.1 mg.	4.2 mg.	None	No carbon or fluoride deposits on dish, waxy film.
ClF_3 F_2	60 psi	0.8 mg.	3.6 mg.	None	No carbon or fluoride deposits on dish, oily liquid.
ClF_3 F_2	60 psi	1.7 mg.	2.5 mg.	Moderate	Carbon deposit on dish; spotted, oily liquid.
ClF_3 F_2	60 psi	1.6 mg.	2.4 mg.	Moderate	Carbon deposit on dish; spotted, oily liquid.
ClF_3 F_2	60 psi	1.1 mg.	3.5 mg.	Moderate	Carbon deposit on dish; spotted, oily liquid.
Cl_4	. . . . .	. . . . .	. . . . .	. . . . .	. . . . .
F_2	60 psi	1.6 mg.	3.4 mg.	None	No carbon or fluoride deposit on dish, waxy solid.
F_2	60 psi	1.6 mg.	4.4 mg.	None	No carbon or fluoride deposit on dish, waxy solid.
F_2	60 psi	1.6 mg.	3.7 mg.	None	No carbon or fluoride deposit on dish, waxy solid.
F_2	60 psi	1.2 mg.	3.6 mg.	None	No carbon or fluoride deposit on dish, waxy solid.
ClF_3	8 psi	1.3 mg.	3.2 mg.	None	No carbon or fluoride deposit on dish, oily liquid.
ClF_3	8 psi	1.5 mg.	3.7 mg.	None	No carbon or fluoride deposit on dish, oily liquid.
ClF_3	8 psi	2.0 mg.	3.5 mg.	None	No carbon or fluoride deposit on dish, oily liquid.
ClF_3	8 psi	1.9 mg.	3.8 mg.	None	No carbon or fluoride deposit on dish, oily liquid

**Hexadecane in Analysis

ressure surge while adding gas to cell.
psi pressure surge
pressure surge

Run #392	0.3 mg.
Run #393	0.2 mg.
Run #394	0.2 mg.
Run #395	0.2 mg.
Run #396	0.2 mg.

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TABLE IV

SHORT TERM IMMERSION TESTS OF METAL SPECIMENS IN LIQUID CYLINDER

Sample	Alloy	Exposure Time (mins.)	Original Weights (gms.)
655	Al 1100	15	2.3074
656	Al 1100	15	2.4113
690	Al 1100	15	2.3162
691	Al 1100	15	2.3034
712	Al 1100	15	2.0689
713	Al 1100	15	2.4202
653	Al 1100	75	2.2731
654	Al 1100	75	2.3424
710	Al 1100	75	2.3657
711	Al 1100	75	2.3299
666	Al 2017	15	2.6916
667	Al 2017	15	2.7966
664	Al 2017	75	2.7624
665	Al 2017	75	2.8273
714	Al 2017	75	2.8224
715	Al 2017	75	2.7909
662	Al 5052	15	2.3719
663	Al 5052	15	2.3221
692	Al 5052	15	2.4936
693	Al 5052	15	2.4781
720	Al 5052	15	2.5003
721	Al 5052	15	2.4972
660	Al 5052	75	2.4649
661	Al 5052	75	2.5082
718	Al 5052	75	2.4194
719	Al 5052	75	2.4257
652	Al 6061	15	2.3051
657	Al 6061	15	2.1508
658	Al 6061	75	2.3384
659	Al 6061	75	2.1948
670	Al 7079	15	2.4807
671	Al 7079	15	2.5671
688	Al 7079	15	2.5911
689	Al 7079	15	2.6286
668	Al 7079	75	2.4598
669	Al 7079	75	2.6967
716	Al 7079	75	2.5888
717	Al 7079	75	2.5320
684	SS 304	15	15.5228
685	SS 304	15	14.5951
672	SS 304	75	15.2750
673	SS 304	75	14.3820
702	SS 304	75	14.8949
703	SS 304	75	15.1732

TABLE IV

DER RM IMMERSION TESTS OF METAL SPECIMENS IN LIQUID CYLINDER FLUORINE

ple	Alloy	Exposure Time (mins.)	Original Weights (gms.)	Weight Change (mg.)
5	Al 1100	15	2.3074	-.2
6	Al 1100	15	2.4113	0
0	Al 1100	15	2.3162	-.1
1	Al 1100	15	2.3034	-.1
2	Al 1100	15	2.0689	0
3	Al 1100	15	2.4202	0
3	Al 1100	75	2.2731	+.2
4	Al 1100	75	2.3424	+.6
0	Al 1100	75	2.3657	-.2
1	Al 1100	75	2.3299	0
6	Al 2017	15	2.6916	+.1
7	Al 2017	15	2.7966	+.4
4	Al 2017	75	2.7624	-.1
5	Al 2017	75	2.8273	+.1
4	Al 2017	75	2.8224	+.2
5	Al 2017	75	2.7909	+.1
2	Al 5052	15	2.3719	-.4
3	Al 5052	15	2.3221	-.1
2	Al 5052	15	2.4936	-.1
3	Al 5052	15	2.4781	-.1
0	Al 5052	15	2.5003	+.1
1	Al 5052	15	2.4972	+.1
0	Al 5052	75	2.4649	+.3
1	Al 5052	75	2.5082	+.3
8	Al 5052	75	2.4194	0
9	Al 5052	75	2.4257	+.1
2	Al 6061	15	2.3051	-.1
7	Al 6061	15	2.1508	0
8	Al 6061	75	2.3384	0
9	Al 6061	75	2.1948	+.1
0	Al 7079	15	2.4807	0
1	Al 7079	15	2.5671	+.1
8	Al 7079	15	2.5911	-.2
9	Al 7079	15	2.6286	0
8	Al 7079	75	2.4598	-.1
9	Al 7079	75	2.6967	+.2
6	Al 7079	75	2.5888	-.6
7	Al 7079	75	2.5320	0
4	SS 304	15	15.5228	-.1
5	SS 304	15	14.5951	0
2	SS 304	75	15.2750	-.3
3	SS 304	75	14.3820	+.2
2	SS 304	75	14.8949	+.1
3	SS 304	75	15.1732	+.2
0	SS 316	15	16.0333	0

[illegible]	901 Magnesium	GLF	4 psia	1.8 mg	None	No carbon or fluoride

666	Al 2017	15	[illegible]	
667	Al 2017	15	2.6916	
664	Al 2017	75	2.7966	
665	Al 2017	75	2.7624	
714	Al 2017	75	2.8273	
715	Al 2017	75	2.8224	
662	Al 5052	15	2.7909	
663	Al 5052	15	2.3719	
692	Al 5052	15	2.3221	
693	Al 5052	15	2.4936	
720	Al 5052	15	2.4781	
721	Al 5052	15	2.5003	
660	Al 5052	75	2.4972	
661	Al 5052	75	2.4649	
718	Al 5052	75	2.5082	
719	Al 5052	75	2.4194	
652	Al 6061	15	2.4257	+
657	Al 6061	15	2.3051	-
658	Al 6061	75	2.1508	
659	Al 6061	75	2.3384	
670	Al 7079	15	2.1948	+
671	Al 7079	15	2.4807	
688	Al 7079	15	2.5671	+
689	Al 7079	15	2.5911	-
668	Al 7079	75	2.6286	
669	Al 7079	75	2.4598	-
716	Al 7079	75	2.6967	+
717	Al 7079	75	2.5888	-
684	SS 304	15	2.5320	
685	SS 304	15	15.5228	-
672	SS 304	75	14.5951	
673	SS 304	75	15.2750	-
702	SS 304	75	14.3820	+
703	SS 304	75	14.8949	+
680	SS 316	15	15.1732	+
681	SS 316	15	16.0333	
678	SS 316	75	16.7154	
679	SS 316	75	15.6023	+
704	SS 316	75	17.7163	-
705	SS 316	75	19.0055	
682	SS 347	15	18.1909	
683	SS 347	15	13.4750	-
674	SS 347	75	13.3431	-
675	SS 347	75	14.2175	-
686	SS 420	15	14.6835	-
687	SS 420	15	14.0845	+
706	SS 420	15	13.5779	-
707	SS 420	15	14.3415	-
676	SS 420	75	13.9555	-
677	SS 420	75	13.9185	
700	Copper	15	13.7174	
701	Copper	15	8.9397	-
698	Copper	75	8.8082	
699	Copper	75	9.0681	+
694	Cartridge Brass	75	8.5638	
695	Cartridge Brass	75	7.4784	-
696	Cartridge Brass	15	7.4907	-
697	Cartridge Brass	15	7.2524	-
			7.3131	

fluoride	ClF3	4 psig	1.8 mg	None	No carbon or fluoride on dish.
	Al 2017	15	2.6916	+.1	
	Al 2017	15	2.7966	+.4	
	Al 2017	75	2.7624	-.1	
	Al 2017	75	2.8273	+.1	
	Al 2017	75	2.8224	+.2	
	Al 2017	75	2.7909	+.1	
	Al 5052	15	2.3719	-.4	
	Al 5052	15	2.3221	-.1	
	Al 5052	15	2.4936	-.1	
	Al 5052	15	2.4781	-.1	
	Al 5052	15	2.5003	+.1	
	Al 5052	15	2.4972	+.1	
	Al 5052	75	2.4649	+.3	
	Al 5052	75	2.5082	+.3	
	Al 5052	75	2.4194	0	
	Al 5052	75	2.4257	+.1	
	Al 6061	15	2.3051	-.1	
	Al 6061	15	2.1508	0	
	Al 6061	75	2.3384	0	
	Al 6061	75	2.1948	+.1	
	Al 7079	15	2.4807	0	
	Al 7079	15	2.5671	+.1	
	Al 7079	15	2.5911	-.2	
	Al 7079	15	2.6286	0	
	Al 7079	75	2.4598	-.1	
	Al 7079	75	2.6967	+.2	
	Al 7079	75	2.5888	-.6	
	Al 7079	75	2.5320	0	
	SS 304	15	15.5228	-.1	
	SS 304	15	14.5951	0	
	SS 304	75	15.2750	-.3	
	SS 304	75	14.3820	+.2	
	SS 304	75	14.8949	+.1	
	SS 304	75	15.1732	+.2	
	SS 316	15	16.0333	0	
	SS 316	15	16.7154	0	
	SS 316	75	15.6023	+.5	
	SS 316	75	17.7163	-.8	
	SS 316	75	19.0055	0	
	SS 316	75	18.1909	0	
	SS 347	15	13.4750	-.8	
	SS 347	15	13.3431	-.8	
	SS 347	75	14.2175	-.1	
	SS 347	75	11.6835	-.2	
	SS 420	15	14.0845	+.5	
	SS 420	15	13.5779	-.1	
	SS 420	15	14.3415	-.3	
	SS 420	15	13.9555	-.1	
	SS 420	75	13.9185	0	
	SS 420	75	13.7174	0	
	Copper	15	8.9397	-.1	
	Copper	15	8.8082	0	
	Copper	75	9.0681	+.3	
	Copper	75	8.5638	0	
	Cartridge Brass	75	7.4784	-.1	
	Cartridge Brass	75	7.4907	-.1	
	Cartridge Brass	15	7.2524	-.1	
	Cartridge Brass	15	7.3131	0	

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TABLE V

CORROSION OF METAL SAMPLES IN LIQUID FLUORINE CONTAMINATED WITH

Sample Number	Exposure Time (mins.)	Alloy	Original Weights (gms.)	Final Weights (gms.)	We Cha (m
722	75	Al 1100	2.0456	2.0453	-0
723	75	Al 1100	2.0187	2.0186	-0
724	75	Al 1100	2.2056	2.2056	0
725	75	Al 1100	2.0331	2.0330	-0
726	15	Al 1100	1.9811	1.9810	-0
727	15	Al 1100	2.1019	2.1016	-0
728	15	Al 1100	2.1322	2.1322	0
729	15	Al 1100	2.0516	2.0515	-0
730	75	Al 6061	2.2730	2.2729	-0
731	75	Al 6061	2.2058	2.2059	+0
732	75	Al 6061	2.1143	2.1141	-0
733	75	Al 6061	2.1978	2.1977	-0
734	15	Al 6061	2.3081	2.3083	+0
735	15	Al 6061	2.1238	2.1239	+0
736	15	Al 6061	2.2031	2.2032	+0
737	15	Al 6061	2.0605	2.0605	0
738	75	Al 5052	1.9011	1.9010	-0
739	75	Al 5052	2.0357	2.0363	+0
740	75	Al 5052	1.8868	1.8865	-0
741	75	Al 5052	1.9023	1.9017	-0
742	15	Al 5052	2.0676	2.0674	-0
743	15	Al 5052	2.0010	2.0008	-0
744	15	Al 5052	1.9500	1.9499	-0
745	15	Al 5052	2.0425	2.0423	-0
746	75	Al 2017	2.6273	2.6272	-0
747	75	Al 2017	2.7278	2.7277	-0
748	75	Al 2017	2.7966	2.7963	-0
749	75	Al 2017	2.8717	2.8716	-0
750*	15	Al 2017	2.7598	2.7585	-1
751*	15	Al 2017	2.7936	2.7926	-1
752*	15	Al 2017	2.8255	2.8244	-1
753*	15	Al 2017	2.6892	2.6886	-0
754*	See Note**	Al 7079	2.7593	2.7584	-0
755*	See Note**	Al 7079	2.6860	2.6851	-0
756*	See Note**	Al 7079	2.6921	2.6916	-0
757*	See Note**	Al 7079	2.4778	2.4773	-0

Notes: * The last eight samples were weighed 1 1/2 weeks after the expo test.

** Explosion occurred in sample cell after 30 minutes. Sample 75 showed a slight reaction.

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TABLE V

WITH

ON OF METAL SAMPLES IN LIQUID FLUORINE CONTAMINATED WITH 1.0% OF_2

We Cha (m	nple nber	Exposure Time (mins.)	Alloy	Original Weights (gms.)	Final Weights (gms.)	Weight Change (mg.)
-0						
-0	22	75	Al 1100	2.0456	2.0453	-0.3
0	23	75	Al 1100	2.0187	2.0186	-0.11
-0	24	75	Al 1100	2.2056	2.2056	0
-0	25	75	Al 1100	2.0331	2.0330	-0.1
-0	26	15	Al 1100	1.9811	1.9810	-0.1
0	27	15	Al 1100	2.1019	2.1016	-0.3
-0	28	15	Al 1100	2.1322	2.1322	0
-0	29	15	Al 1100	2.0516	2.0515	-0.1
+0	30	75	Al 6061	2.2730	2.2729	-0.1
-0	31	75	Al 6061	2.2058	2.2059	+0.1
-0	32	75	Al 6061	2.1143	2.1141	-0.2
+0	33	75	Al 6061	2.1978	2.1977	-0.1
+0	34	15	Al 6061	2.3081	2.3083	+0.2
+0	35	15	Al 6061	2.1238	2.1239	+0.1
0	36	15	Al 6061	2.2031	2.2032	+0.1
-0	37	15	Al 6061	2.0605	2.0605	0
+0	38	75	Al 5052	1.9011	1.9010	-0.1
-0	39	75	Al 5052	2.0357	2.0363	+0.6
-0	40	75	Al 5052	1.8868	1.8865	-0.3
-0	41	75	Al 5052	1.9023	1.9017	-0.6
-0	42	15	Al 5052	2.0676	2.0674	-0.2
-0	43	15	Al 5052	2.0010	2.0008	-0.2
-0	44	15	Al 5052	1.9500	1.9499	-0.1
-0	45	15	Al 5052	2.0425	2.0423	-0.2
-0	46	75	Al 2017	2.6273	2.6272	-0.1
-0	47	75	Al 2017	2.7278	2.7277	-0.1
-0	48	75	Al 2017	2.7966	2.7963	-0.3
-1	49	75	Al 2017	2.8717	2.8716	-0.1
-1	750*	15	Al 2017	2.7598	2.7585	-1.3
-1	751*	15	Al 2017	2.7936	2.7926	-1.0
-0	752*	15	Al 2017	2.8255	2.8244	-1.1
-0	753*	15	Al 2017	2.6892	2.6886	-0.6
-0	754*	See Note**	Al 7079	2.7593	2.7584	-0.9
-0	755*	See Note**	Al 7079	2.6860	2.6851	-0.9
-0	756*	See Note**	Al 7079	2.6921	2.6916	-0.5
	757*	See Note**	Al 7079	2.4778	2.4773	-0.5

expo

Notes: • The last eight samples were weighed 1 1/2 weeks after the exposure test.

ple 75

•• Explosion occurred in sample cell after 30 minutes. Sample 756 showed a slight reaction.

	[illegible]	Al 1100	2.1322	2.1322
729	15	Al 1100	2.0516	2.0515
730	75	Al 6061	2.2730	2.2729
731	75	Al 6061	2.2058	2.2059
732	75	Al 6061	2.1143	2.1141
733	75	Al 6061	2.1978	2.1977
734	15	Al 6061	2.3081	2.3083
735	15	Al 6061	2.1238	2.1239
736	15	Al 6061	2.2031	2.2032
737	15	Al 6061	2.0605	2.0605
738	75	Al 5052	1.9011	1.9010
739	75	Al 5052	2.0357	2.0363
740	75	Al 5052	1.8868	1.8865
741	75	Al 5052	1.9023	1.9017
742	15	Al 5052	2.0676	2.0674
743	15	Al 5052	2.0010	2.0008
744	15	Al 5052	1.9500	1.9499
745	15	Al 5052	2.0425	2.0423
746	75	Al 2017	2.6273	2.6272
747	75	Al 2017	2.7278	2.7277
748	75	Al 2017	2.7966	2.7963
749	75	Al 2017	2.8717	2.8716
750*	15	Al 2017	2.7598	2.7585
751*	15	Al 2017	2.7936	2.7926
752*	15	Al 2017	2.8255	2.8244
753*	15	Al 2017	2.6892	2.6886
754*	See Note**	Al 7079	2.7593	2.7584
755*	See Note**	Al 7079	2.6860	2.6851
756*	See Note**	Al 7079	2.6921	2.6916
757*	See Note**	Al 7079	2.4778	2.4773

Notes: * The last eight samples were weighed 1 1/2 weeks after the test.

** Explosion occurred in sample cell after 30 minutes. Samp showed a slight reaction.

758	75	Al 7079	3.0240	3.0245
759	75	Al 7079	2.9980	2.9982
760	75	Al 7079	2.9847	2.9848
761	75	Al 7079	2.9957	2.9956
762	15	Al 7079	2.4843	2.4842
763	15	Al 7079	2.5828	2.5823
764	15	Al 7079	2.4579	2.4573
765	15	Al 7079	2.5659	2.5655
766	75	SS 304	13.4790	13.4789
767	75	SS 304	13.8023	13.8022
768	75	SS 304	14.2583	14.2581
769	75	SS 304	14.8753	14.8753
770	15	SS 304	14.7416	14.7418
771	15	SS 304	14.9058	14.9059
772	15	SS 304	13.8328	13.8329
773	15	SS 304	14.1542	14.1543
774	75	SS 316	13.5268	13.5264
775	75	SS 316	13.0103	13.0099
776	75	SS 316	13.8580	13.8581
777	75	SS 316	14.9162	14.9168

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	75	Al 6061	2.2730	2.2729	-0.1
	75	Al 6061	2.2058	2.2059	+0.1
	75	Al 6061	2.1143	2.1141	-0.2
	75	Al 6061	2.1978	2.1977	-0.1
	15	Al 6061	2.3081	2.3083	+0.2
	15	Al 6061	2.1238	2.1239	+0.1
	15	Al 6061	2.2031	2.2032	+0.1
	15	Al 6061	2.0605	2.0605	0
	75	Al 5052	1.9011	1.9010	-0.1
	75	Al 5052	2.0357	2.0363	+0.6
	75	Al 5052	1.8868	1.8865	-0.3
	75	Al 5052	1.9023	1.9017	-0.6
	15	Al 5052	2.0676	2.0674	-0.2
	15	Al 5052	2.0010	2.0008	-0.2
	15	Al 5052	1.9500	1.9499	-0.1
	15	Al 5052	2.0425	2.0423	-0.2
	75	Al 2017	2.6273	2.6272	-0.1
	75	Al 2017	2.7278	2.7277	-0.1
	75	Al 2017	2.7966	2.7963	-0.3
	75	Al 2017	2.8717	2.8716	-0.1
50*	15	Al 2017	2.7598	2.7585	-1.3
51*	15	Al 2017	2.7936	2.7926	-1.0
52*	15	Al 2017	2.8255	2.8244	-1.1
53*	15	Al 2017	2.6892	2.6886	-0.6
54*	See Note**	Al 7079	2.7593	2.7584	-0.9
55*	See Note**	Al 7079	2.6860	2.6851	-0.9
56*	See Note**	Al 7079	2.6921	2.6916	-0.5
57*	See Note**	Al 7079	2.4778	2.4773	-0.5

otes: * The last eight samples were weighed 1 1/2 weeks after the exposure test.

** Explosion occurred in sample cell after 30 minutes. Sample 756 showed a slight reaction.

58	75	Al 7079	3.0240	3.0245	+0.5
59	75	Al 7079	2.9980	2.9982	+0.2
60	75	Al 7079	2.9847	2.9848	+0.1
61	75	Al 7079	2.9957	2.9956	-0.1
62	15	Al 7079	2.4843	2.4842	-0.1
63	15	Al 7079	2.5828	2.5823	-0.5
64	15	Al 7079	2.4579	2.4573	-0.6
65	15	Al 7079	2.5659	2.5655	-0.4
66	75	SS 304	13.4790	13.4789	-0.1
767	75	SS 304	13.8023	13.8022	-0.1
768	75	SS 304	14.2583	14.2581	-0.2
769	75	SS 304	14.8753	14.8753	0
770	15	SS 304	14.7416	14.7418	+0.2
771	15	SS 304	14.9058	14.9059	+0.1
772	15	SS 304	13.8328	13.8329	+0.1
773	15	SS 304	14.1542	14.1543	+0.1
774	75	SS 316	13.5268	13.5264	+0.4
775	75	SS 316	13.0103	13.0099	-0.4
776	75	SS 316	13.8580	13.8581	+0.1
777	75	SS 316	14.9162	14.9168	+0.6

TABLE V (Continued)

CORROSION OF METAL SAMPLES IN LIQUID FLUORINE CONTAMINATED WITH 1.0% OF_2

Sample Number	Exposure Time (mins.)	Alloy	Original Weights (gms.)	Final Weights (gms.)	Weight Change (mg.)
778	15	SS 316	14.2930	14.2932	+0.2
779	15	SS 316	14.4870	14.4871	+0.1
780	15	SS 316	13.7539	13.7536	-0.3
781	15	SS 316	14.2764	14.2765	+0.1
782	75	SS 347	13.4460	13.4459	-0.1
783	75	SS 347	13.1328	13.1325	-0.3
784	75	SS 347	13.1461	13.1458	-0.3
785	75	SS 347	12.5850	12.5848	-0.2
786	15	SS 347	14.0841	14.0838	-0.3
787	15	SS 347	13.1410	13.1419	+0.9
788	15	SS 347	13.0368	13.0368	0
789	15	SS 347	13.5973	13.5965	-0.8
790	75	SS 420	12.4321	12.4328	+0.7
791	75	SS 420	13.7836	13.7842	+0.6
792	75	SS 420	13.0705	13.0713	+0.8
793	75	SS 420	13.2577	13.2561	-1.6
794	15	SS420	13.1820	13.1828	+0.8
795	15	SS 420	13.0607	13.0610	+0.3
796	15	SS 420	13.4896	13.4902	+0.6
798	75	Cartridge Brass	7.4749	7.4751	+0.2
799	75	Cartridge Brass	7.4866	7.4868	+0.2
800	75	Cartridge Brass	7.2449	7.2447	-0.2
801	75	Cartridge Brass	7.3088	7.3088	0
802	15	Cartridge Brass	7.3150	7.3158	+0.8
803	15	Cartridge Brass	7.3383	7.3392	+0.9
804	15	Cartridge Brass	7.3731	7.3738	+0.7
805	15	Cartridge Brass	7.1715	7.1721	+0.6
806	75	Copper	5.8939	5.8935	-0.4
807	75	Copper	5.9902	5.9908	+0.6
808	75	Copper	6.1970	6.1971	+0.1
809	75	Copper	5.6426	5.6431	+0.5
810	15	Copper	6.2655	6.2663	+0.8
811	15	Copper	5.8575	5.8583	+0.8
812	15	Copper	5.4876	5.4882	+0.6
813	15	Copper	5.6777	5.6786	+0.9
854	75	Nickel	16.0574	16.0573	-0.1
855	75	Nickel	19.1638	19.1640	+0.2
856	75	Nickel	19.8291	16.8294	+0.3
857	75	Nickel	19.5873	19.5873	0
858	15	Nickel	19.5418	19.5417	-0.1
859	15	Nickel	15.4427	15.4429	+0.2
860	15	Nickel	18.8230	18.8233	+0.3
861	15	Nickel	17.9968	17.9967	-0.1

TABLE VI

CORROSION OF METAL SPECIMENS IN LIQUID FLUORINE CONTAMINATED WITH 10% OF_2

Sample Number	Exposure Time (mins.)	Alloy	Original Weights (gms.)	Final Weights (gms.)	Weight Change (mg.)
814	75	Al 6061	2.1593	2.1593	0
815	75	Al 6061	1.9998	1.9997	-0.1
816	75	Al 6061	2.1955	2.1954	-0.1
817	75	Al 6061	2.1750	2.1749	-0.1
818	15	Al 6061	2.0487	2.0485	-0.2
819	15	Al 6061	2.0200	2.0199	-0.1
820	15	Al 6061	2.0242	2.0241	-0.1
821	15	Al 6061	2.0646	2.0644	-0.2
822	75	Copper	6.5514	6.5525	+1.1
823	75	Copper	6.7172	6.7182	+1.0
824	75	Copper	5.8251	5.8261	+1.0
825	75	Copper	6.3102	6.3108	+0.6
826	15	Copper	6.2154	6.2161	+0.7
827	15	Copper	6.1841	6.1849	+0.8
828	15	Copper	6.7667	6.7685	+1.8
829	15	Copper	6.3422	6.3426	+0.4
830*	75	Al 6061	2.1584	2.1594	+1.0
831*	75	Al 6061	1.9977	1.9994	+1.7
832*	75	Al 6061	2.1942	2.1975	+3.3
833*	75	Al 6061	2.1739	2.1759	+2.0
834*	15	Al 6061	2.0477	2.0479	+0.2
835*	15	Al 6061	2.0188	2.0189	+0.1
836*	15	Al 6061	2.0234	2.0236	+0.2
837*	15	Al 6061	2.0636	2.0635	-0.1
838	15	SS 304	13.4808	13.4810	+0.2
839	15	SS 304	13.8048	13.8050	+0.2
840	15	SS 304	14.2599	14.2605	+0.6
841	15	SS 304	14.8782	14.8777	-0.5
842	75	SS 304	14.7346	14.7349	+0.3
843	75	SS 304	14.9080	14.9081	+0.1
844	75	SS 304	13.8347	13.8348	+0.1
845	75	SS 304	14.1560	14.1560	0
846	15	Cartridge Brass	7.2713	7.2717	+0.4
847	15	Cartridge Brass	7.4773	7.4777	+0.4
848	15	Cartridge Brass	7.2389	7.2392	+0.3
849	15	Cartridge Brass	7.3021	7.3026	+0.5
850	75	Cartridge Brass	7.2990	7.2991	+0.1
851	75	Cartridge Brass	7.3180	7.3181	+0.1
852	75	Cartridge Brass	7.3573	7.3576	+0.3
853	75	Cartridge Brass	7.1541	7.1542	+0.1

*A recheck was run on the Aluminum 6061 samples.

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TABLE VII

COMPARISON OF AVERAGE WEIGHT CHANGE OF METAL SAMPLES EXPOSED TO LIQUID CYLINDER FLUORINE AND LIQUID FLUORINE CONTAMINATED WITH OF_2

	Average Weight Change - Mg					
	15 Minute Exposure			75 Minute Exposure		
Alloy	In Cylinder Fluorine	In Fluorine and 1% OF_2	In Fluorine and 10% OF_2	In Cylinder Fluorine	In Fluorine and 1% OF_2	In Fluorine and 10% OF_2
Aluminum 1100	-0.067	-0.125	- - - -	+0.150	-0.125	- - - -
Aluminum 2017	+0.250	-1.000	- - - -	+0.075	-0.150	- - - -
Aluminum 5052	-0.083	-0.175	- - - -	+0.175	-0.100	- - - -
Aluminum 6061	-0.050	+0.100	-0.150 +0.100*	+0.050	-0.075	-0.075 +2.000*
Aluminum 7079	-0.025	-0.400	- - - -	-0.125	+0.175	- - - -
304 S.S.	-0.050	+0.125	+0.125	+0.050	-0.100	+0.125
316 S.S.	0.000	+0.025	- - - -	-0.075	+0.175	- - - -
347 S.S.	-0.800	-0.050	- - - -	-0.150	-0.225	- - - -
420 S.S.	0.000	-0.567	- - - -	0.000	+0.125	- - - -
Copper	-0.050	0.775	+0.925	+0.150	+0.200	+0.925
Cartridge Brass	-0.050	0.750	+0.400	-0.100	+0.050	+0.150

*Duplicate Run

TABLE

SUMMARY OF FILM THICKNESS MEAS

Run No.	Metal Powder	Weight Sample (gm.)	Temp. °F	Initial F_2 Pressure in Contact with Sample (mmHg abs)	Thickness After 1 hr.
16	Nickel	119.63	86	496	9.1
17	Copper	61.42	86	485	7.7
18	Nickel	133.39	86	512	10.3
19	Monel	126.79	86	516	19.3
20	Brass	130.10	86	517	3.27
21	Copper	68.18	86	494	4.56
22	Aluminum	36.53	86	533	4.2
23	Monel	137.03	86	541	9.0
24	Brass	83.99	86	505	5.75
25	Titanium	50.46	86	538	- - -
26	316 S.S.	111.7	86	535	5.0
27	347 S.S.	103.4	86	533	5.4
28	304 S.S.	100.3	86	500	8.0
29	Monel	111.8	-113	690	6.7
30	Brass	102.8	-113	698	3.1
31	Monel	98.31	+183	498	9.9

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TABLE VIII

KNESS MEASUREMENTS ON METAL POWDERS

Th After	kness 1 hr.	Thickness After 2.5 hr.	End of Run	Length of Run (hrs.)	Sensitivity A	Residual Gas Analysis	Remarks
9	1	9.7	12.42	24.2	0.2		
9	7	9.4	23.0	95.0	0.2		
11	3	11.1	14.8	23.4	0.1		
10	3	10.2	29.8	71.6	0.3		
3	27	3.37	9.98	46.7	0.1		
5	56	5.5	19.62	94.0	0.1		
5	2	5.0	13.9	91.1	0.3		
9	0	9.9	10.87	24.5	0.4		
6	75	6.24	10.62	67.3	0.2		
1	- -	- - -	- - -	- - -	- - -		
5	0	5.8	16.7	22.0	0.2		Powder caked on sides of cell. Powder found in coil used to increase system volume. Reaction increased after overnight exposure.*
6	4	6.6	11.4	18.8	0.4		Olive and orange fluoride in cell nozzle. Powder removed and recharged. Reaction occurred.
8	0	8.8	14.4	22.1	0.3		Powder removed and recharged. Reaction occurred.
8	7	8.8	167.0	27.9	0.6		Note large film after overnight exposure.
3	1	3.4	6.8	26.7	0.2		Powder removed and recharged. Reaction occurred.
9	9	9.1	44.6	74.8	0.5	7.7% HF, also CO_2, CF_4, SiF_4 and unknown.	Nickel and copper fluorides in passivated powder which was darker than fresh powder. Reaction increased after 68 hrs.* Reacted during F_2 standardization due to possible leak. Analysis of gas after F_2 standardization HF, CO_2, SiF_4.

TABLE VIII (Conti

SUMMARY OF FILM THICKNESS MEASURI

Run No.	Metal Powder	Weight Sample (gm.)	Temp. °F	Initial F_2 Pressure in Contact with Sample (mmHg abs)	Thickness After 1 hr.	Thic After
40	Aluminum	28.26	-113	687	6.5	- -
41	410 S.S.	100.06	+ 86	503	8.5	10.
42	316 S.S.	84.22	-113	679	5.0	5.
43	304 S.S.	91.93	-113	680	5.1	6.
44	Nickel	106.30	+183	479	5.8	6.
45	347 S.S.	97.83	-113	686	13.0	14.
46	316 S.S.	89.34	183	471	30.8	32.9
47	410 S.S.	93.3	-113	686	22.1	24.3
48	304 S.S.	92.43	+183	474	12.0	16.8
49	Titanium	39.93	-113	656	76.6	77.0
50	Titanium	10.50	-113	624	5.6	5.9

TABLE VIII (Continued)

KNESS MEASUREMENTS ON METAL POWDERS

Thickness After 1 hr.	Thickness After 2.5 hr.	End of Run	Length of Run (hrs.)	Sensitivity A	Residual Gas Analysis	Remarks
.5	- - -	163	118.0	0.6	No HF	
.5	10.5	63.8	71.0	0.7	8.5% HF	
.0	5.2	8.6	21.3	0.4	2.3% HF	
.1	6.1	44.0	25.6	0.5	1.1% HF	
.8	6.1	8.1	21.0	0.1	21.9% HF	
.0	14.1	40.6	28.2	0.6	0.6% HF	After F_2 standardization, the test cell was purged and evacuated for 1 1/2 hrs. When opened the cell contained a golden fluorescent vapor which illuminated the interior of the cell when a light was shined in. This vapor disappeared as the atmosphere contacted it. The vapor had no odor. The powder had a yellow-green film when removed but this disappeared as the atmosphere contacted it.
.8	32.9	35.4	97.8	0.2	HF-Nil, 1.1% CO_2, 10 ppm CF_4	Run 4 days to check effect of prolonged exposure.
[illegible]	24.3	25.9	25.2	1.2	0.2% HF, 20 ppm CF_4	
0	16.8	46.8	24.4	0.2		
[illegible]	77.0	77.1	5.4	0.2		One minute file of 74.1 A indicated violent initial reaction.
[illegible]	5.9	23.5	24.0	1.0		Repeat of previous run with smaller sample.

TABLE VIII (Continued)

SUMMARY OF FILM THICKNESS MEASUREMENT

Run No.	Metal Powder	Weight Sample (gm.)	Temp °F	Initial F_2 Pressure in Contact with Sample (mmHg abs)	Thickness After 1 hr.	Thickness After 2.5 hr
51	347 S.S.	94.78	+183	470	9.3	11.9
52	Nickel	93.65	-297	322	4.4	3.9
53	410 S.S.	88.48	+183	470	29.3	43.0
54	Monel	102.31	-297	323	0	0
55	Brass	150.27	+183	487	1.9	2.6
57	Copper	56.47	+183	458	7.1	9.6
59	Aluminum	33.02	+183	468	1.6	- - -
64	Titanium	39.95	-297	302	System pressure dropped mmHg abs in 7 minutes c 0.28 gm F_2 (equivalent t film)	

*Leaks possibly caused by tapping sample bomb during run to see if agitation of the powder woul tapping. The effect of a tiny leak is to reduce system pressure as water seeps into the system is consumed. Pressure increases in the system when water enters faster than HF reacts with found in the cell and IR analysis showing HF present in residual gas.

LE VIII (Continued)

ESS MEASUREMENTS ON METAL POWDERS

ss hr.	Thickness After 2.5 hr.	End of Run	Length of Run (hrs.)	Sensitivity A	Residual Gas Analysis	Remarks
	11.9	27.8	30.4	0.3	15.2% HF	Leaks in IR Cell discovered for this run and several others where residual gas composition is not reported.
	3.9	3.25	21.4	0.6	0.7% HF	First run at -297° F. Technique for runs at this temperature was being developed. Pressure began to increase after first hour exposure.
	43.0	90.7	24.5	0.6	12.9% HF	
	0	0	6.5	2.0	6.6% HF, 10 ppm CF_4	No reaction detected.
	2.6	3.46	23.1	0.1	29.2% HF, traces CO_2 and CF_4	
	9.6	23.0	24.7	0.14	19.2% HF, traces CO_2 and CF_4	Passivated powder was purple and lumpy.
	- - -	- - -	21.6	0.3		Pressure dropped to a minimum in 101 minutes then increased 15 mm in 15 hours.
em pressure dropped to 2 Hg abs in 7 minutes consuming gm F_2 (equivalent to 78.7 A)			- - -	0.5		

on of the powder would increase reaction. In run 31, system pressure increased after such seeps into the system and reacts with the fluorine to produce HF which attacks the powder and than HF reacts with the powder. The leaks were confirmed by corrosion products being

TABLE IX

SUMMARY OF FILM THICKNESSES ON METAL POWDEI TEMPERATURES AND TIMES OF EXPOSUF

Metal	+183° F Film Thickness - Å 1 hr	+183° F Film Thickness - Å 2.5 hrs	+183° F Film Thickness - Å End of Run	Total Time (hrs)	+86° F Film Thickness - Å 1 hr	+86° F Film Thickness - Å 2.5 hrs	+86° F Film Thickness - Å End of Run
Aluminum	1.6	1.7	--	21.6	4	5	13.9
Brass	1.9	2.1	3.46	23.1	3.28 (5.64)*	3.4	10.0
Copper	7.1	10.5	23.0	24.7	4.26 (7.70)*	5.8	19.7
Monel							
Nickel	5.8	6.1	8.11	21	9.02 (10.3)*	11.1	15.8
304 Stainless Steel	12.0	16.8	46.8	24.4	8.0	8.8	14.35
316 Stainless Steel	30.8	32.9	35.4	97.8	5.0	5.8	16.7
347 Stainless Steel	9.3	11.9	27.8	30.4	5.4	6.6	11.35
410 Stainless Steel	29.3	43.0	90.7	24.5	8.5	10.5	63.8
Titanium	----------	----------	----------	**	----------	----------	----------

* Duplicate Run.

** Sample exploded on exposure to fluorine.

TABLE IX

OF FILM THICKNESSES ON METAL POWDERS AT VARIOUS TEMPERATURES AND TIMES OF EXPOSURE

3° F			+86° F				-113° F			
s - Å	Total	Film Thickness - Å			Total	Film Thickness - Å			Total
End of Run	Time (hrs)	1 hr	2.5 hrs	End of Run	Time (hrs)	1 hr	2.5 hrs	End of Run	Time (hrs)
--	21.6	4	5	13.9	91	6.5	10.5	16.3	118
3.46	23.1	3.28 (5.64)*	3.4	10.0	47	3.1	3.4	6.81	26.7
23.0	24.7	4.26 (7.70)*	5.8	19.7	98.5	1.8	1.9	3.18	22.5
8.11	21	9.02 (10.3)*	11.1	15.8	25	1.0	2.3	11.8	25.0
46.8	24.4	8.0	8.8	14.35	22.1	5.1	6.1	44.0	26.5
35.4	97.8	5.0	5.8	16.7	22.0	5.0	5.2	8.6	21.3
27.8	30.4	5.4	6.6	11.35	18.8	13.0	14.1	40.6	28.2
90.7	24.5	8.5	10.5	63.8	71	22.1	24.3	25.9	25.2
----------		**	----------			5.6	5.9	23.5	24.0

orine.

TABLE X

SUMMARY OF FILM THICKNESSES ON MONEL POWDER AT VARIOUS TEMPERATURES AND TIMES OF EXPOSURE

Run No.	Temp. °F	Film Thickness, (A) 1 hr	2 hr	End Run	Run Time (hours)	Average Formation Rate, (A/hr)
19	86	9.0	9.9	29.8	71.3	0.417
23	86	9.0	9.9	11.2	24.1	0.469
29	-113	6.7	8.4	167	27.9	6.0
31	183	8.9	9.2	31.0 44.6*	68.25 74.75	0.452 0.600*
34	86	8.3	8.6	13.9	19.1	0.728
35	86	11.45	---	35.3	22.0	1.61
37	86	3.1	3.8	8.3	70	0.119
39	86	7.2	8.8	30.3	115	0.263

*Reaction during fluorine standardization due to possible leak.

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TABLE XI

APPARENT REACTIONS OF METAL POWDERS AND GASEOUS FLUORINE A

Run No. and Alloy	Pressure Drop mmHg			Apparent Film Th	
	15 Min.	30 Min.	60 Min.	15 Min.	30 Mi
52 - Nickel	+20.5	+27.8	+27.8	+ 6.67	+9.04
54 - Monel	-12.0	-11.0	- 9.0	- 1.00	-0.92
56 - 316 S.S.	+ 7.5	+ 9.5	+ 7.0	+ 2.94	+3.73
58 - 304 S.S.*	+10.0	+13.7	+14.0	+ 5.34	+7.32
60 - 347 S.S.	+ 1.0	+ 2.6	- 2.5	+ 0.78	+2.03
61 - 410 S.S.	+ 9.5	+ 4.0	+ 2.0	+12.91	+5.44
62 - Brass	- 4.6	- 0.6	- 1.8	- 7.21	-0.09
63 - Copper	- 0.5	0	0	- 0.14	0
65 - Titanium	+ 1.5	+ 2.0	+ 2.0	+ 1.25	+1.67
66 - Aluminum	+ 4.5	+ 4.0	+ 4.0	+ 2.64	+2.35

Method of Calculation: (1) Plot nitrogen standards, fluorine standard, and reaction curves - system pressure vs. time of exposure.

(2) Read pressure difference from curves: fluorine standar fluorine reaction pressure at 15, 30, and 60 minute tim

(3) Calculate film thickness: $d = \frac{F\ V_T\ \Delta P}{T\ W_s}$ (see text).

*No fluorine standard was taken. Nitrogen standard was used to get Δ P.

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TABLE XI

PARENT REACTIONS OF METAL POWDERS AND GASEOUS FLUORINE AT -297° F

Alloy	Pressure Drop mmHg			Apparent Film Thickness - A		
	15 Min.	30 Min.	60 Min.	15 Min.	30 Min.	60 Min.
l	+20.5	+27.8	+27.8	+ 6.67	+9.04	+9.04
	-12.0	-11.0	- 9.0	- 1.00	-0.92	-0.75
S.	+ 7.5	+ 9.5	+ 7.0	+ 2.94	+3.73	+2.75
S.*	+10.0	+13.7	+14.0	+ 5.34	+7.32	+7.46
S.	+ 1.0	+ 2.6	- 2.5	+ 0.78	+2.03	-1.95
S.	+ 9.5	+ 4.0	+ 2.0	+12.91	+5.44	+2.72
	- 4.6	- 0.6	- 1.8	- 7.21	-0.09	-0.28
r	- 0.5	0	0	- 0.14	0	0
um	+ 1.5	+ 2.0	+ 2.0	+ 1.25	+1.67	+1.67
num	+ 4.5	+ 4.0	+ 4.0	+ 2.64	+2.35	+2.35

Calculation: (1) Plot nitrogen standards, fluorine standard, and reaction of fluorine curves - system pressure vs. time of exposure.

(2) Read pressure difference from curves: fluorine standard pressure - fluorine reaction pressure at 15, 30, and 60 minute times.

(3) Calculate film thickness: $d = \frac{F\,V_T\,\Delta P}{T\,W_s}$ (see text).

e standard was taken. Nitrogen standard was used to get Δ P.

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TABLE XII

CORROSION OF METAL SPECIMENS IMMERSED IN LIQUID FLUORINE FOR ONE Y

Sample Number	Metal	A Sample Weight Before Exposure (gm)	B Sample Weight After Exposure (gm)	C Weight Difference Before Cleaning (gm, B-A)	D Sample Weight After Cleaning (gm)	E Weigh Differe After Cleani (gm, D-
1	S.S. 304	18.6849	18.6873	+0.0024	18.6858	+0.0009
2		18.5512	18.5540	+0.0028	18.5486	-0.0026
3		18.7305	18.7298	-0.0007	18.7303	-0.0002
4		18.6154	18.6210	+0.0056	18.6163	+0.0009
5		18.5245	18.5325	+0.0080	18.5267	+0.0022
6	S.S. 410	17.8507	17.8298	-0.0209	17.8024	-0.0483
7		18.1199	18.0981	-0.0218	18.0772	-0.0427
8		17.8918	17.8766	-0.0152	17.8676	-0.0342
9		17.9080	17.8905	-0.0175	17.8857	-0.0223
10		18.0191	18.0123	-0.0068	17.9984	-0.0207
11	15-7 Monel	17.9713	17.9760	+0.0047	17.9721	+0.0008
12		17.9272	17.9371	+0.0099	17.9266	-0.0006
13		18.1022	18.1104	+0.0082	17.1044	+0.0022
14		17.9481	17.9553	+0.0072	17.9510	+0.0029
15		17.9088	17.9144	+0.0056	17.9089	+0.0001
16	Copper	19.5659	19.5715	+0.0056	19.5632	-0.0027
17		19.7531	19.7639	+0.0108	19.7536	+0.0005
18		19.6306	19.6379	+0.0073	19.6298	-0.0008
19		19.7691	19.7752	+0.0061	19.7697	+0.0006
20		19.6955	19.7001	+0.0046	19.6938	-0.0017
21	Al 1100	6.3930	6.3616	-0.0314	6.3442	-0.0488
22		6.4349	6.4106	-0.0243	6.3927	-0.0422
23		6.3277	6.3046	-0.0233	6.2878	-0.0399
24		6.3876	6.3688	-0.0188	6.3561	-0.0315
25		6.3828	6.3675	-0.0153	6.3514	-0.0314
26	Al 6061	6.0088	5.9928	-0.0160	5.9773	-0.0315
27		5.9818	5.9697	-0.0121	5.9523	-0.0295
28		6.0216	6.0163	-0.0053	5.9784	-0.0437
29		5.9744	5.9578	-0.0166	5.9411	-0.0333
30		5.9193	5.8984	-0.0209	5.8860	-0.0333
31	Nickel	20.4919	20.4952	+0.0033	20.4884	-0.0035
32		20.4087	20.4149	+0.0062	20.4082	-0.0006
33		20.6207	20.6281	+0.0074	20.6221	+0.0014
34		20.5791	20.5999	+0.0208	20.5823	+0.0032
35		20.6101	20.6206	+0.0105	20.6139	+0.0038
36	Monel	20.5294	20.5366	+0.0072	20.5284	-0.0010
37		20.4504	20.4604	+0.0100	20.4500	-0.0004
38		20.4651	20.4715	+0.0064	20.4616	-0.0035
39		20.6064	20.6157	+0.0093	20.6043	-0.0021
40		20.6896	20.6916	+0.0020	20.6862	-0.0034

TABLE XII

ORROSION OF METAL SPECIMENS IMMERSED IN LIQUID FLUORINE FOR ONE YEAR

Metal	A Sample Weight Before Exposure (gm)	B Sample Weight After Exposure (gm)	C Weight Difference Before Cleaning (gm, B-A)	D Sample Weight After Cleaning (gm)	E Weight Difference After Cleaning (gm, D-A)	F Metal Corrosion (mils/year)
S. 304	18.6849	18.6873	+0.0024	18.6858	+0.0009	---
	18.5512	18.5540	+0.0028	18.5486	-0.0026	0.0048
	18.7305	18.7298	-0.0007	18.7303	-0.0002	0.0003
	18.6154	18.6210	+0.0056	18.6163	+0.0009	** ---
	18.5245	18.5325	+0.0080	18.5267	+0.0022	* ---
S. 410	17.8507	17.8298	-0.0209	17.8024	-0.0483	0.0905
	18.1199	18.0981	-0.0218	18.0772	-0.0427	0.0802
	17.8918	17.8766	-0.0152	17.8676	-0.0342	0.0640
	17.9080	17.8905	-0.0175	17.8857	-0.0223	0.0418
	18.0191	18.0123	-0.0068	17.9984	-0.0207	0.0387
-7 Monel	17.9713	17.9760	+0.0047	17.9721	+0.0008	---
	17.9272	17.9371	+0.0099	17.9266	-0.0006	0.0010
	18.1022	18.1104	+0.0082	17.1044	+0.0022	* ---
	17.9481	17.9553	+0.0072	17.9510	+0.0029	* ---
	17.9088	17.9144	+0.0056	17.9089	+0.0001	* ---
pper	19.5659	19.5715	+0.0056	19.5632	-0.0027	0.0044
	19.7531	19.7639	+0.0108	19.7536	+0.0005	*** ---
	19.6306	19.6379	+0.0073	19.6298	-0.0008	0.0013
	19.7691	19.7752	+0.0061	19.7697	+0.0006	*** ---
	19.6955	19.7001	+0.0046	19.6938	-0.0017	0.0028
1100	6.3930	6.3616	-0.0314	6.3442	-0.0488	0.2638
	6.4349	6.4106	-0.0243	6.3927	-0.0422	0.2272
	6.3277	6.3046	-0.0233	6.2878	-0.0399	0.2155
	6.3876	6.3688	-0.0188	6.3561	-0.0315	0.1700
	6.3628	6.3675	-0.0153	6.3514	-0.0314	0.1698
6061	6.0088	5.9928	-0.0160	5.9773	-0.0315	0.1700
	5.9818	5.9697	-0.0121	5.9523	-0.0295	0.1594
	6.0216	6.0163	-0.0053	5.9784	-0.0432	0.2321
	5.9744	5.9578	-0.0166	5.9411	-0.0333	0.1800
	5.9193	5.8984	-0.0209	5.8860	-0.0333	0.1800
kel	20.4919	20.4952	+0.0033	20.4884	-0.0035	0.0053
	20.4087	20.4149	+0.0062	20.4082	-0.0006	0.0093
	20.6207	20.6281	+0.0074	20.6221	+0.0014	* ---
	20.5791	20.5999	+0.0208	20.5823	+0.0032	* ---
	20.6101	20.6206	+0.0105	20.6139	+0.0038	* ---
nel	20.5294	20.5366	+0.0072	20.5284	-0.0010	0.0017
	20.4504	20.4604	+0.0100	20.4500	-0.0004	0.0007
	20.4651	20.4715	+0.0064	20.4616	-0.0035	0.0058
	20.6064	20.6157	+0.0093	20.6043	-0.0021	0.0035
	20.6886	20.6916	+0.0020	20.6862	-0.0036	0.0060

18		19.6306	19.6379	+0.0073	19.6298	-0.00
19		19.7691	19.7752	+0.0061	19.7697	+0.00
20		19.6955	19.7001	+0.0046	19.6938	-0.00
21	Al 1100	6.3930	6.3616	-0.0314	6.3442	-0.04
22		6.4349	6.4106	-0.0243	6.3927	-0.04
23		6.3277	6.3046	-0.0233	6.2878	-0.03
24		6.3876	6.3688	-0.0188	6.3561	-0.03
25		6.3828	6.3675	-0.0153	6.3514	-0.03
26	Al 6061	6.0088	5.9928	-0.0160	5.9773	-0.03
27		5.9818	5.9697	-0.0121	5.9523	-0.02
28		6.0216	6.0163	-0.0053	5.9784	-0.04
29		5.9744	5.9578	-0.0166	5.9411	-0.03
30		5.9193	5.8984	-0.0209	5.8860	-0.03
31	Nickel	20.4919	20.4952	+0.0033	20.4884	-0.00
32		20.4087	20.4149	+0.0062	20.4082	-0.00
33		20.6207	20.6281	+0.0074	20.6221	+0.00
34		20.5791	20.5999	+0.0208	20.5823	+0.00
35		20.6101	20.6206	+0.0105	20.6139	+0.00
36	Monel	20.5294	20.5366	+0.0072	20.5284	-0.00
37		20.4504	20.4604	+0.0100	20.4500	-0.00
38		20.4651	20.4715	+0.0064	20.4616	-0.00
39		20.6064	20.6157	+0.0093	20.6043	-0.00
40		20.6896	20.6916	+0.0020	20.6862	-0.00
41	Ti A-110 AT	10.4057	10.3207	-0.0850	10.3157	-0.09
42		10.3669	10.2730	-0.0939	10.2661	-0.10
43		10.4500	10.3724	-0.0776	10.3653	-0.08
44		10.3437	10.2718	-0.0719	10.2642	-0.07
45		10.2572	10.1847	-0.0725	10.1777	-0.07
46	Ti C-120 AV	10.3558	10.2710	-0.0848	10.2661	-0.08
47		10.3333	10.2516	-0.0817	10.2456	-0.08
48		10.3732	10.3085	-0.0647	10.3002	-0.07
49		10.4072	10.3659	-0.0413	10.3418	-0.06
50		10.3463	10.3091	-0.0372	10.2947	-0.05
51	Mg AZ-31	3.9513	3.9416	-0.0097	3.9053	-0.04
52		3.9398	3.9696	+0.0298	3.9223	-0.01
53		3.9748	3.9706	-0.0042	3.9263	-0.04
54		3.9345	3.9097	-0.0248	3.8844	-0.05
55		3.9334	3.9261	-0.0073	3.8903	-0.04
56	Mg HM-31	3.9925	3.9491	-0.0434	3.9618	-0.03
57		4.0097	3.9558	-0.0539	3.9205	-0.08
58		4.0078	3.9671	-0.0407	3.9235	-0.08
59		4.0017	3.9215	-0.0802	3.8995	-0.10
60		3.9625	3.9942	+0.0317	3.8652	-0.09

* Copper film on threads could not be removed.
** Fluoride in specimen's center drill holes could not be removed.
*** Black and yellow film on threads could not be removed.

AREA OF SPECIMEN - 4.20 in^2

NOTE: Corrosion based upon weight difference after cleaning.

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0.0008		19.6955	19.7001	-0.0046	19.6938	-0.0017	0.0028
0.0006							
0.0017	100	6.3930	6.3616	-0.0314	6.3442	-0.0488	0.2638
		6.4349	6.4106	-0.0243	6.3927	-0.0422	0.2272
0.04		6.3277	6.3046	-0.0233	6.2878	-0.0399	0.2155
0.042		6.3876	6.3688	-0.0188	6.3561	-0.0315	0.1700
0.0399		6.3828	6.3675	-0.0153	6.3514	-0.0314	0.1698
0.0317							
0.0314	061	6.0088	5.9928	-0.0160	5.9773	-0.0315	0.1700
		5.9818	5.9697	-0.0121	5.9523	-0.0295	0.1594
0.0317		6.0216	6.0163	-0.0053	5.9784	-0.0432	0.2321
0.0299		5.9744	5.9578	-0.0166	5.9411	-0.0333	0.1800
0.0432		5.9193	5.8984	-0.0209	5.8860	-0.0333	0.1800
0.0333							
0.0333	1	20.4919	20.4952	.0.0033	20.4884	-0.0035	0.0053
		20.4087	20.4149	.0.0062	20.4082	-0.0006	0.0093
0.0037		20.6207	20.6281	.0.0074	20.6221	.0.0014	• ---
0.0006		20.5791	20.5999	.0.0208	20.5823	.0.0032	• ---
0.0013		20.6101	20.6206	.0.0105	20.6139	.0.0038	• ---
0.0032							
0.0038		20.5294	20.5366	.0.0072	20.5284	-0.0010	0.0017
		20.4504	20.4604	.0.0100	20.4500	-0.0004	0.0007
0.0010		20.4651	20.4715	.0.0064	20.4616	-0.0035	0.0058
0.0004		20.6064	20.6157	.0.0093	20.6043	-0.0021	0.0035
0.0035		20.6896	20.6916	.0.0020	20.6862	-0.0036	0.0060
0.0021							
0.0036	110 AT	10.4057	10.3207	-0.0850	10.3157	-0.0900	0.2910
		10.3669	10.2730	-0.0939	10.2661	-0.1008	0.3239
0.0900		10.4500	10.3724	-0.0776	10.3653	-0.0847	0.2740
0.1008		10.3437	10.2718	-0.0719	10.2642	-0.0795	0.2572
0.0847		10.2572	10.1847	-0.0725	10.1777	-0.0795	0.2572
0.0795							
0.0795	20 AV	10.3558	10.2710	-0.0848	10.2661	-0.0897	0.2164
		10.3333	10.2516	-0.0817	10.2456	-0.0877	0.2706
0.0897		10.3732	10.3085	-0.0647	10.3002	-0.0730	0.2252
0.0877		10.4072	10.3659	-0.0413	10.3418	-0.0654	0.2020
0.0730		10.3463	10.3091	-0.0372	10.2947	-0.0516	0.1591
0.0654							
0.0516	-31	3.9513	3.9416	-0.0097	3.9053	-0.0460	0.3350
		3.9398	3.9696	.0.0298	3.9223	-0.0175	0.1465
0.0460		3.9748	3.9706	-0.0042	3.9263	-0.0485	0.4060
0.0175		3.9345	3.9097	-0.0248	3.8844	-0.0501	0.4200
0.0485		3.9334	3.9261	-0.0073	3.8903	-0.0431	0.3608
0.0501							
0.0431	-31	3.9925	3.9491	-0.0434	3.9618	-0.0307	0.2564
		4.0097	3.9558	-0.0539	3.9205	-0.0892	0.7470
0.0307		4.0078	3.9671	-0.0407	3.9235	-0.0843	0.7060
0.0892		4.0017	3.9215	-0.0802	3.8995	-0.1022	0.3460
0.0843		3.9625	3.9942	.0.0317	3.8652	-0.0973	0.3150
1022							
0973							

threads could not be removed.
ecimen's center drill holes could not be removed.
low film on threads could not be removed.

MEN - 4.20 in^2

based upon weight difference after cleaning.

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TABLE XIII

AVERAGE CORROSION OF METAL SPECIMENS IMMERSED IN LIQUID FLUORINE FOR ONE YEAR

Sample Number	Metal	Metal Corrosion (mils/year)
1-5	304 S.S.	0.0026
6-10	410 S.S.	0.0630
11-15	15-7 Monel	0.0010
16-20	Copper	0.0028
21-25	Al 1100	0.2093
26-30	Al 6061	0.1843
31-35	Nickel	0.0034
36-40	Monel	0.0035
41-45	Ti A-110	0.2807
46-50	Ti C-120	0.2267
51-55	Mg AZ-31	0.3437
56-60	Mg HM-31	0.6741

TABLE XIV

MECHANICAL PROPERTIES OF TENSILE TEST SI

IN LIQUID NITROGEN AND LIQUID FLUORINE

Sample Number	Metal	Initial Diameter (inches)	Final Diameter (inches)	Initial Area (sq in)	Final Area (sq in)	Yield Stress (psi)	Ultimate Load (lbs)
N-1	304 S.S.	0.1886	0.075	0.0281	0.0044	60,100	2620
N-2		0.1885	0.0903	0.0283	0.00641	60,100	2640
N-3		0.1888	0.0865	0.02795	0.00588	59,400	2620
N-4		0.1886	0.0850	0.0279	0.00568	61,600	2640
N-5		0.1886	0.0835	0.0278	0.00548	61,900	2640
F-1	304 S.S.	0.1891	0.073	0.0281	0.0042	60,100	2615
F-2		0.1885	0.0830	0.0279	0.00541	62,700	2570
F-3		0.1880	0.0845	0.02778	0.00566	62,300	2650
F-4		0.1884	0.0965	0.0279	0.00731	62,000	2660
F-5		0.1883	0.0885	0.0278	0.00615	70,100	2630
N-6	410 S.S.	0.1885	0.085	0.0278	0.0057	75,500	2500
N-7		0.1887	0.0990	0.0278	0.00764	73,700	2450
N-8		0.1881	0.00970	0.0278	0.00740	72,700	2510
N-9		0.1881	0.0992	0.0278	0.00773	71,600	2390
N-10		0.1887	0.0957	0.0279	0.00720	79,900	2520
F-6	410 S.S.	0.1884	0.097	0.0278	0.0074	74,500	2495
F-7		0.1884	0.0990	0.0278	0.00769	73,400	2470
F-8		0.1887	0.0965	0.0280	0.00731	72,900	2520
F-9		0.1886	0.0955	0.0280	0.00718	74,300	2510
F-10		0.1883	0.1005	0.0278	0.00865	73,400	2470
N-11	15-7 Monel	0.1836	0.123	0.0264	0.0119	75,400	3965
N-12		0.1884	0.1260	0.0278	0.01250	71,000	3920
N-13		0.1879	0.1230	0.0277	0.01195	70,400	3930
N-14		0.1886	0.1285	0.0279	0.0129	71,000	2980
N-15		0.1884	0.1275	0.0278	0.0128	70,500	3930
F-11	15-7 Monel	0.1880	0.125	0.0278	0.0123	71,200	3970
F-12		0.1884	0.124	0.0279	0.0121	71,300	3960
F-13		0.1877	0.1262	0.0278	0.0125	70,500	3950
F-14		0.1882	0.1225	0.0279	0.0118	70,600	3950
F-15		0.1888	0.1214	0.0279	0.0116	69,900	3940

TABLE XIV

SP S OF TENSILE TEST SPECIMENS IMMERSED

INE AND LIQUID FLUORINE FOR ONE YEAR

nate ad s)	l a n)	Yield Stress (psi)	Ultimate Load (lbs)	Ultimate Stress (psi)	Reduction in Area (%)	Percent Elongation	Modulus of Elasticity (psi)	Yield Load (lbs)
0	44	60,100	2620	93,200	84.3	40.6	30.1×10^6	1690
0	541	60,100	2640	93,300	77.3	39.7	24.5×10^6	1700
0	588	59,400	2620	93,700	79.0	39.3	27.0×10^6	1660
0	568	61,600	2640	94,600	79.6	39.0	23.7×10^6	1720
0	548	61,900	2640	95,000	80.3	38.0	26.1×10^6	1720
5	42	60,100	2615	93,100	85.1	39.2	25.9×10^6	1690
0	541	62,700	2570	92,100	80.6	39.2	28.5×10^6	1750
0	566	62,300	2650	95,400	79.6	37.5	27.1×10^6	1730
0	731	62,000	2660	95,300	73.8	39.5	26.5×10^6	1730
0	615	70,100	2630	94,600	77.9	38.6	22.6×10^6	1950
0	57	75,500	2500	89,900	79.5	10.4	27.0×10^6	2100
0	764	73,700	2450	88,100	72.5	8.14	22.3×10^6	2050
0	740	72,700	2510	90,300	[illegible] 4	7.90	28.0×10^6	2020
0	773	71,600	2390	86,000	72.2	10.4	29.6×10^6	1990
0	720	79,900	2520	90,300	74.2	8.16	26.5×10^6	2230
5	74	74,500	2495	89,700	73.4	8.62	28.2×10^6	2070
0	769	73,400	2470	88,800	72.3	8.80	25.3×10^6	2040
0	731	72,900	2520	90,000	73.9	8.47	27.6×10^6	2040
1	718	74,300	2510	89,600	74.4	7.14	27.7×10^6	2080
0	865	73,400	2470	88,800	68.9	7.64	25.9×10^6	2040
5	19	75,400	3965	150,000	54.9	29.0	28.6×10^6	1990
0	250	71,000	3920	141,000	55.0	24.9	25.7×10^6	1975
0	195	70,400	3930	141,000	56.9	26.3	24.8×10^6	1950
0	29	71,000	2980	142,700	53.8	25.6	26.0×10^6	1980
0	28	70,500	3930	141,400	54.0	26.2	24.9×10^6	1960
0	23	71,200	3970	142,800	55.8	25.9	25.0×10^6	1980
	21	71,300	3960	141,900	56.6	26.6	21.6×10^6	1990
	25	70,500	3950	142,000	55.0	24.5	25.5×10^6	1960
	18	70,600	3950	141,600	57.7	25.1	25.4×10^6	1970
	16	69,900	3940	141,200	58.4	24.3	26.0×10^6	1950

TABLE XIV (Contin

Sample Number	Metal	Initial Diameter (inches)	Final Diameter (inches)	Initial Area (sq in)	Final Area (sq in)	Yield Stress (psi)
N-16	Copper	0.1778	0.1046	0.0248	0.0086	52,800
N-17		0.1797	0.1085	0.0256	0.00925	54,300
N-18		0.1790	0.1090	0.0252	0.00934	53,200
N-19		0.1782	0.1100	0.0250	0.00950	54,400
N-20		0.1785	0.120	0.0250	0.01065	53,200
F-16	Copper	0.1784	0.109	0.0250	0.0093	54,200
F-17		0.1783	0.1348	0.0250	0.01327	54,800
F-18		0.1784	0.1125	0.0250	0.00991	54,200
F-19		0.1792	0.1219	0.0252	0.01082	52,800
F-20		0.1784	0.1252	0.0250	0.01238	54,400
N-21	Al 1100	0.1885	0.083	0.0278	0.0054	21,600
N-22		0.1876	0.0856	0.0276	0.00590	21,600
N-23		0.1875	0.0934	0.0276	0.00684	21,500
N-24		0.1893	0.0978	0.0281	0.00750	21,300
N-25		0.1882	0.0875	0.0278	0.00600	21,400
F-21	Al 1100	0.1886	0.079	0.0278	0.0049	22,300
F-22		0.1890	0.0926	0.0281	0.00675	21,700
F-23		0.1885	0.0855	0.0279	0.00575	21,600
F-24		0.1892	0.0843	0.0281	0.00560	21,400
F-25		0.1884	0.0851	0.0278	0.00562	21,500
N-26	Al 6061	0.1785	0.125	0.0250	0.0123	41,200
N-27		0.1782	0.1180	0.0250	0.01095	42,100
N-28		0.1780	0.1185	0.0249	0.01120	41,900
N-29		0.1780	0.1318	0.0249	0.01360	41,800
N-30		0.1780	0.1246	0.0249	0.01220	42,200
F-26	Al 6061	0.1781	0.122	0.0249	0.0117	41,900
F-27		0.1782	0.1234	0.0250	0.01200	41,300
F-28		0.1775	0.1159	0.0247	0.01055	41,000
F-29		0.1780	0.1142	0.0249	0.01024	42,000
F-30		0.1778	0.1179	0.0249	0.01097	41,200

TABLE XIV (Continued)

Final Area (sq in)	Yield Stress (psi)	Ultimate Load (lbs)	Ultimate Stress (psi)	Reduction in Area (%)	Percent Elongation	Modulus of Elasticity (psi)	Yield Load (lbs)
0.0086	52,800	1410	56,900	65.3	3.73	18.0×10^6	1310
0.00925	54,300	1455	56,800	63.9	3.69	19.0×10^6	1390
0.00934	53,200	1445	57,300	62.9	4.04	19.0×10^6	1340
0.00950	54,400	1460	58,400	62.0	4.08	18.1×10^6	1360
0.01065	53,200	1470	58,800	57.4	4.03	17.7×10^6	1330
0.0093	54,200	1450	58,000	62.8	4.31	16.9×10^6	1355
0.01327	54,800	1470	58,800	46.9	5.32	18.0×10^6	1370
0.00991	54,200	1475	59,000	60.4	4.71	19.2×10^6	1355
0.01082	52,800	1490	59,100	57.1	3.68	18.2×10^6	1330
0.01238	54,400	1460	58,400	50.5	4.03	18.0×10^6	1360
0.0054	21,600	644	23,200	80.6	10.7	9.73×10^6	600
0.00590	21,600	644	23,300	78.6	5.96	10.1×10^6	596
0.00684	21,500	640	23,200	75.2	5.31	10.6×10^6	594
0.00750	21,300	642	22,800	73.3	5.67	9.51×10^6	600
0.00600	21,400	641	23,100	78.4	6.10	10.4×10^6	594
0.0049	22,300	641	23,100	82.4	4.65	9.87×10^6	619
0.00675	21,700	634	22,600	76.0	5.00	10.1×10^6	610
0.00575	21,600	638	22,900	79.4	5.72	10.1×10^6	604
0.00560	21,400	648	23,100	80.1	5.32	10.5×10^6	602
0.00562	21,500	640	23,000	79.8	5.65	11.0×10^6	599
0.0123	41,200	1124	45,000	50.8	8.11	10.2×10^6	1029
0.01095	42,100	1140	45,600	56.2	7.62	10.6×10^6	1053
0.01120	41,900	1135	45,600	55.0	7.33	10.1×10^6	1043
0.01360	41,800	1158	46,500	45.4	6.62	10.5×10^6	1040
0.01220	42,200	1155	46,400	51.0	6.38	9.75×10^6	1050
0.0117	41,900	1135	45,600	53.0	8.61	10.4×10^6	1044
0.01200	41,300	1136	45,400	52.0	7.36	10.0×10^6	1033
0.01055	41,000	1132	45,800	57.3	7.26	9.90×10^6	1012
0.01024	42,000	1237	49,700	58.9	7.02	9.72×10^6	1045
0.01097	41,200	1128	45,300	55.9	7.05	10.2×10^6	1027

TABLE XIV (Continued)

Sample Number	Metal	Initial Diameter (inches)	Final Diameter (inches)	Initial Area (sq in)	Final Area (sq in)	Yield Stress (psi)	Ult L (l
N-46	Ti C-120-AV	0.1882	0.140	0.0279	0.0154	123,000	4
N-47		0.1869	0.151	0.0275	0.0180	125,000	4
N-48		0.1875	0.149	0.0276	0.0175	124,000	4
N-49		0.1886	0.153	0.0280	0.0184	124,000	4
N-50		0.1870	0.161	0.0275	0.0204	124,000	4
F-46	Ti C-120-AV	0.1881	0.149	0.0278	0.0175	122,000	4
F-47		0.1882	0.123	0.0279	0.0161	120,000	4
F-48		0.1883	0.150	0.0279	0.0177	121,000	4
F-49		0.1881	0.148	0.0278	0.0172	121,000	4
F-50		0.1882	0.149	0.0279	0.0175	123,000	4
N-51	Mg AZ-31	0.1784	0.1371	0.0250	0.0148	35,200	1
N-52		0.1786	0.1384	0.0251	0.0151	35,800	1
N-53		0.1788	0.1383	0.0251	0.0150	35,300	1
N-54		0.1793	0.1386	0.0253	0.0151	35,600	1
N-55		0.1785	0.1375	0.0250	0.0149	35,600	1
F-51	Mg AZ-31	0.1778	0.1511	0.0249	0.0180	34,000	1
F-52		0.1786	0.1423	0.0251	0.0157	34,500	1
F-53		0.1787	0.1600	0.0251	0.0201	33,900	1
F-54		0.1796	0.1505	0.0254	0.0178	34,400	1
F-55		0.1791	0.1554	0.0252	0.0190	33,800	1
N-56	Mg HK-31	0.1785	0.1422	0.0250	0.0176	49,600	1
N-57		0.1781	0.1506	0.0249	0.0178	48,700	1
N-58		0.1783	0.1487	0.0250	0.0174	47,700	1
N-59		0.1785	0.1519	0.0250	0.0181	49,600	1
N-60		0.1780	0.1484	0.0249	0.0174	49,400	1
F-56	Mg HK-31	0.1797	0.1688	0.0254	0.0225	46,100	1
F-57		0.1786	0.1570	0.0251	0.0194	47,700	1
F-58		0.1787	0.1563	0.0251	0.0192	46,700	1
F-59		0.1786	0.1622	0.0251	0.0205	46,500	1
F-60		0.1794	0.1650	0.0253	0.0215	43,000	1

ued)

TABLE XIV (Continued)

Ultim Loa (lbs	Final Area (sq in)	Yield Stress (psi)	Ultimate Load (lbs)	Ultimate Stress (psi)	Reduction in Area (%)	Percent Elongation	Modulus of Elasticity (psi)	Yield Load (lbs)
4240								
4225	.0154	123,000	4240	152,000	44.8	6.02	14.8×10^6	3420
4300	.0180	125,000	4225	154,000	34.5	5.54	15.1×10^6	3425
4220	.0175	124,000	4300	156,000	36.6	5.70	15.2×10^6	3435
4200	.0184	124,000	4220	151,000	34.3	6.53	14.9×10^6	3465
	.0204	124,000	4200	153,000	25.8	4.67	15.2×10^6	3410
4230								
4250	.0175	122,000	4230	152,000	37.1	7.07	15.0×10^6	3385
4245	.0161	120,000	4250	152,000	42.3	6.19	15.0×10^6	3350
4200	.0177	121,000	4245	152,000	36.6	6.33	15.5×10^6	3375
4310	.0172	121,000	4200	151,000	38.1	6.40	15.1×10^6	3355
	.0175	123,000	4310	154,000	73.3	5.46	15.1×10^6	3430
1038								
1047	.0148	35,200	1038	41,500	40.8	6.29	6.40×10^6	880
1044	.0151	35,800	1047	41,700	39.8	6.29	6.51×10^6	898
1050	.0150	35,300	1044	41,600	40.2	5.67	6.04×10^6	886
1042	.0151	35,600	1050	41,500	40.3	5.65	5.78×10^6	900
	.0149	35,600	1042	41,700	40.4	5.33	6.41×10^6	889
1119								
1040	.0180	34,000	1119	44,900	27.7	6.00	6.32×10^6	851
1035	.0157	34,500	1040	41,400	31.5	7.05	6.30×10^6	865
1039	.0201	33,900	1035	41,200	19.9	5.33	6.23×10^6	850
1013	.0178	34,400	1039	40,900	29.9	5.67	6.24×10^6	873
	.0190	33,800	1013	40,200	24.6	4.01	6.35×10^6	852
1260								
1250	.0176	49,600	1260	50,400	29.6	7.49	6.31×10^6	1240
1240	.0178	48,700	1250	50,200	28.5	8.20	6.03×10^6	1212
1255	.0174	47,700	1240	49,600	30.4	8.94	6.28×10^6	1193
1230	.0181	49,600	1255	50,200	27.6	6.33	6.05×10^6	1240
	.0174	49,400	1230	49,400	30.1	6.67	6.59×10^6	1230
1220								
1230	.0225	46,100	1220	48,600	11.4	4.10	6.29×10^6	1170
1220	.0194	47,700	1230	48,900	22.7	3.67	6.28×10^6	1197
1172	.0192	46,700	1220	48,600	23.5	5.32	6.53×10^6	1173
1240	.0205	46,500	1172	48,700	18.3	4.67	6.29×10^6	1158
	.0215	45,000	1240	49,000	15.0	2.01	6.31×10^6	1087

TABLE XV

AVERAGE RESULTS OF MECHANICAL PROPERTIES OF TENSILE SPECIMEN

Sample Number	Metal	Yield Stress (psi)	Ultimate Stress (psi)	Modulus of Elasticity (psi)	Reduction in Area (%)
N-1-5	304 S.S.	60,620	93,960	62.28×10^6	80.10
F-1-5	304 S.S.	63,440	94,100	26.12×10^6	79.40
Nominal		30,000		29×10^6	
N-6-10	410 S.S.	74,680	88,920	26.68×10^6	74.36
F-6-10	410 S.S.	73,700	89,380	26.94×10^6	72.58
Nominal		60,000		29×10^6	
N-11-15	15-7 Monel	71,660	143,420	26.00×10^6	54.92
F-11-15	15-7 Monel	70,700	141,900	24.70×10^6	56.70
Nominal		55,000		29×10^6	
N-16-20	Copper	53,580	57,640	18.36×10^6	62.30
F-16-20	Copper	54,080	58,660	18.02×10^6	55.54
Nominal		48,000		16×10^6	
N-21-25	Al 1100	21,480	23,120	10.07×10^6	77.22
F-21-25	Al 1100	21,700	22,940	10.31×10^6	79.54
Nominal		17,000		10.0×10^6	
N-26-30	Al 6061	41,840	45,820	10.23×10^6	51.68
F-26-30	Al 6061	41,480	46,360	10.04×10^6	55.42
Nominal		40,000		10.0×10^6	
N-31-35	Nickel	89,960	96,900	29.60×10^6	77.80
F-31-35	Nickel	89,680	96,420	30.48×10^6	78.12
Nominal		25,000		30×10^6	
N-36-40	Monel	58,020	93,740	26.12×10^6	72.60
F-36-40	Monel	58,120	93,040	25.52×10^6	71.16
Nominal		50,000		26×10^6	
N-41-45	Ti A-110-AT	125,400	151,600	15.66×10^6	19 25
F-41-45	Ti A-110-AT	126,200	152,000	15.84×10^6	16.96
Nominal		112,200		16×10^6	
N-46-50	C-120-AV	124,000	153,200	15.04×10^6	35.20
F-46-50	C-120-AV	121,400	152,200	15.14×10^6	38.28
Nominal		130,000		16×10^6	
N-51-55	Mg AZ-31	35,500	41,600	6.23×10^6	40.30
F-51-55	Mg AZ-31	34,160	41,720	6.29×10^6	27.92
Nominal		30,000		6.5×10^6	
N-56-60	Mg HK-31	49,000	49,960	6.25×10^6	29.24
F-56-60	Mg HK-31	46,000	48,240	6.34×10^6	18.18
Nominal		30,000		6.5×10^6	

TABLE XV

AVERAGE RESULTS OF MECHANICAL PROPERTIES OF TENSILE SPECIMENS

Metal	Yield Stress (psi)	Ultimate Stress (psi)	Modulus of Elasticity (psi)	Reduction in Area (%)	Elongation (%)
304 S.S.	60,620	93,960	62.28×10^6	80.10	39.32
304 S.S.	63,440	94,100	26.12×10^6	79.40	38.80
	30,000		29×10^6		
410 S.S.	74,680	88,920	26.68×10^6	74.36	9.00
410 S.S.	73,700	89,380	26.94×10^6	72.58	8.13
	60,000		29×10^6		
15-7 Monel	71,660	143,420	26.00×10^6	54.92	26.40
15-7 Monel	70,700	141,900	24.70×10^6	56.70	25.28
	55,000		29×10^6		
Copper	53,580	57,640	18.36×10^6	62.30	3.91
Copper	54,080	58,660	18.02×10^6	55.54	4.41
	48,000		16×10^6		
Al 1100	21,480	23,120	10.07×10^6	77.22	6.75
Al 1100	21,700	22,940	10.31×10^6	79.54	5.27
	17,000		10.0×10^6		
Al 6061	41,840	45,820	10.23×10^6	51.68	7.21
Al 6061	41,480	46,360	10.04×10^6	55.42	7.46
	40,000		10.0×10^6		
Nickel	89,960	96,900	29.60×10^6	77.80	6.10
Nickel	89,680	96,420	30.48×10^6	78.12	6.24
	25,000		30×10^6		
Monel	58,020	93,740	26.12×10^6	72.60	25.56
Monel	58,120	93,040	25.52×10^6	71.16	24.70
	50,000		26×10^6		
Ti A-110-AT	125,400	151,600	15.66×10^6	19.25	5.38
Ti A-110-AT	126,200	152,000	15.84×10^6	16.96	5.53
	112,200		16×10^6		
C-120-AV	124,000	153,200	15.04×10^6	35.20	5.69
C-120-AV	121,400	152,200	15.14×10^6	38.28	6.29
	130,000		16×10^6		
Mg AZ-31	35,500	41,600	6.23×10^6	40.30	5.85
Mg AZ-31	34,160	41,720	6.29×10^6	27.92	5.61
	30,000		6.5×10^6		
Mg HK-31	49,000	49,960	6.25×10^6	29.24	7.53
Mg HK-31	46,000	48,240	6.34×10^6	18.18	3.95
	30,000		6.5×10^6		

TABLE XVI

STANDARD AREAS OF METAL POWDERS USED IN CALCULATIONS

Aluminum	0.3591
Brass	0.2465
Copper	0.36
Monel	0.0615
Monel (rerun)	0.0416
Nickel	0.17
316 Stainless Steel	0.2161
347 Stainless Steel	0.1075
304 Stainless Steel	0.1519
410 Stainless Steel	0.0650
Titanium	1.0374

Note: A computer was used to arrive at the above numbers and the results reported are read from the computer sheet. The limitations of the method are discussed in the text.

TABLE XVII

AFTER-PASSIVATION AREAS OF METAL POWDERS

Run No.	Metal	Area m^2/gm	Difference from Standard Area (%)
16	Nickel	0.0934	- 45
18	Nickel	0.2128	+ 25
20	Brass	0.177	- 28
24	Brass	0.1711	- 30
30	Brass	0.1683	- 32
27	347 S.S.	0.0868	- 19
28	304 S.S.	0.1475	- 3
19	Monel	0.0587	- 5
23	Monel	0.0765	+ 24
29	Monel	0.1005	+ 63
34	Monel	0.0352	+ 42
31	Monel	0.0874	+ 63
35	Monel	0.1352	+120
17	Copper	0.238	- 34
21	Copper	0.7288	+102
32	Copper	0.2222	- 36
22	Aluminum	0.1951	- 46

NOTE: A computer was used to arrive at the above numbers and the results reported are read from the computer sheet. The limitations of the method are discussed in the text (page 16).

TABLE XVIII

COMPARISON OF FLUORINE REACTED WITH FRESH AND PASSIVATED POWDERS

Run No.	Metal	Temp. °F	Fluorine Compound - gm: 1st passivation	Fluorine Compound - gm: 2nd passivation
27	347 S.S.	86	1.52×10^{-2}	0.17×10^{-2}
28	304 S.S.	86	2.66×10^{-2}	1.83×10^{-2}
30	Brass	-113	2.84×10^{-2}	2.45×10^{-2}
32	Copper	-113	2.09×10^{-2}	1.54×10^{-2}
33	MgF_2*	86	77×10^{-2} (after 2 hours)	
34	Monel	86	1.44×10^{-2}	0.466×10^{-2}
35	Monel	86	1.99×10^{-2}	0.592×10^{-2}
37	Monel	86	0.924×10^{-2}	0.873×10^{-2}
38	Nickel	-113	3.45×10^{-2}	0.691×10^{-2}
39	Monel	86	2.65×10^{-2}	1.156×10^{-2}
42	316 S.S.	-113	1.942×10^{-2}	2.118×10^{-2}
43	304 S.S.	-113	7.47×10^{-2}	0.774×10^{-2}
44	Nickel	183	2.60×10^{-2}	0.1092×10^{-2}
45	347 S.S.	-113	5.16×10^{-2}	0.64×10^{-2}
47	410 S.S.	-113	1.753×10^{-2}	1.84×10^{-2}
48	304 S.S.	183	7.72×10^{-2}	6.56×10^{-2}
50	Titanium	-113	2.195×10^{-2}	0.2745×10^{-2}
51	347 S.S.	183	3.42×10^{-2}	0.831×10^{-2}

*Reacted with fluorine until no further reaction occurred. Removed sample and exposed to atmosphere. Powder returned to the cell and re-exposed to fluorine. Reaction occurred but not run to completion.

TABLE XIX

CHEMICAL ANALYSIS OF STAINLESS STEELS

Composition %	Type 304	316	347	410
Iron	69.28	65.74	67.73	86.15
Nickel	10.57	13.16	13.18	- - - -
Chromium	18.40	17.45	16.62	11.80
Carbon	0.06	0.06	0.03	0.04
Manganese	0.66	0.41	1.74	0.80
Silicon	1.00	1.06	1.23	1.18
Sulfur	0.017	0.022	0.012	0.014
Phosphorous	0.014	0.018	0.011	0.017
Columbium	- - - -	- - - -	0.45	- - - -
Molybdynum	- - - -	2.08	- - - -	- - - -