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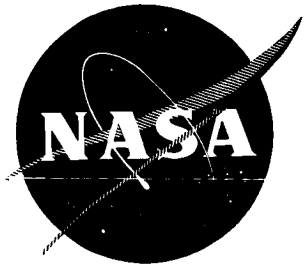

OXYGEN DIFLUORIDE RESEARCH STUDY

by

Ralph B. Jackson
ALLIED CHEMICAL CORPORATION

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
CONTRACT NAS 3-6298
Theodore Male, Project Manager

Requests for copies of this report should be referred to

National Aeronautics and Space Administration
Scientific and Technical Information Facility
P. O. Box 33
College Park, Maryland 20740

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FINAL REPORT

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Ralph B. Jackson

ALLIED CHEMICAL CORPORATION
Morristown, New Jersey 07960

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CONTRACT NAS 3-6298

NASA Lewis Research Center

Cleveland, Ohio

Theodore Male, Project Manager

Chemical Rocket Division

FOREWORD

This report was prepared by Allied Chemical Corporation under NASA Contract No. NAS 3-6298. The program was initiated and administered by Lewis Research Center, Liquid Rocket Technology Branch, Chemical Rocket Division. The project manager for the contract was Mr. Theodore Male.

The work was performed by the Industrial Chemicals Division of Allied Chemical Corporation at Morristown, New Jersey.

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ABSTRACT

The compatibility of several materials to oxygen difluoride under dynamic conditions was investigated. Metal test orifices were successfully exposed to liquid OF_2 flows at pressures to 1500 psig and to gaseous OF_2 at sonic velocity. Plastic orifices were tested and found to be compatible to liquid OF_2 flows at pressures to 500 psig.

A metal ignition study was conducted. Wires were heated electrically in an OF_2 atmosphere and the ignition temperatures were calculated from resistivity-temperature data.

The attempt to modify a Tracerlab Fluorine Monitor so that it would be suitable for OF_2 service was unsuccessful.

1. INTRODUCTION

Oxygen difluoride is a powerful oxidizing agent. Because of its relatively high boiling point and excellent specific impulse, with certain fuels, it is being considered seriously for upper stage rocket applications.

From the standpoint of equipment design, it is imperative that a knowledge of material compatibility be available. Similarly, a knowledge of the ignition temperature of construction metals in OF_2 is important so that proper material selections can be made to help eliminate failures from this source. Further, when working with high energy toxic propellants, it is necessary to monitor surrounding areas to maintain concentrations within tolerable limits.

This study is directed toward furnishing this required knowledge. It consists of three separate tasks: an evaluation of the compatibility of various metals in liquid and gaseous OF_2 under dynamic conditions, the investigation of metal ignition in OF_2, and the development of an OF_2 detector.

2. DYNAMIC TESTING OF METALS IN LIQUID OF_2

The compatibility of several metals with liquid OF_2 under dynamic conditions was investigated. A test loop was constructed in which were installed test orifices fabricated from candidate materials. These orifices, 0.0135 inch diameter, were exposed to liquid flows of OF_2 at several pressures ranging from 120 to 1500 psig. The dynamic exposure time at each pressure increment for each specimen was a minimum of approximately ten minutes. Photomicrographs taken before and after exposure were used to measure any changes in the orifices.

2.1. Apparatus

The apparatus for the liquid OF_2 dynamic tests consisted of three basic assemblies: a valve manifold, a test loop and an insulated liquid-nitrogen container. The complete setup is represented schematically in Figure 1.

Valves 1 to 7 as shown in this drawing were high pressure manually operated needle valves. These valves and the associated manifold hardware were located within a high pressure cubicle. The valves were safely operated by extension handles which passed through the cubicle wall. The manifold was virtually rebuilt twice during this program because of unsatisfactory valve performance. Originally, Valves 1-7 were high pressure needle valves manufactured by

Pressure Products Industries, Inc. After modest service, several valves developed leaks across the valve seats and were replaced. Of greater significance, in three instances the valve stems sheared with the plug in a closed position making it impossible to open the valve. This happened to Valves 2 and 3 when the test loop contained almost three pounds of liquid OF_2. In order to empty the system, it was necessary to break into the service lines and by-pass the manifold. As a result of this hazardous occurrence, it was decided to rebuild the manifold using Hoke M343 needle valves. These valves too developed leaks in this service and were eventually replaced with Hoke Y344H blunt needle valves. Although some valve replacements were needed from time to time, these blunt needle valves were used for the remainder of the dynamic program.

In addition to the aforementioned needle valves, the manifold included Valves 1B and 2B which were Hoke solenoid valves. These also leaked after short service and were therefore backed up by manual needle valves (1BB and 2BB) operated with extension handles. These valves were used to protect the compound gauge from the high operating pressures.

The test loop and liquid nitrogen tank were located outside the cubicle but within the wooden barricade wall since the large size of this equipment precluded installation inside the cubicle. The setup was protected from the weather by a roof and sliding plexiglass

panels. This permitted visual observation of the setup and offered additional protection in the event of an explosion or other mishap.

The liquid nitrogen tank was welded from stainless steel sheets. Six inches of rigid polyurethane foam insulation was placed between the inner and outer walls of the tank. To further minimize nitrogen evaporation, the tank was covered by 2-inch thick panels of rigid polyurethane through which extended the Annin valves and service lines of the test loop.

The test loop included two 1000 cc. capacity cylinders. The dip tubes in these cylinders as well as the test loop lines were 1/2" Monel tubing. The specimen holders were fabricated from heavy wall 1/2" Monel unions (Figure 2). Soft aluminum gaskets, approximately 0.95" O.D. x .75" I.D. and 0.020" thick, were placed on either side of the test specimens. These gaskets provided a leak tight seal at the highest test pressure. The liquid OF_2 flow through the loop was controlled by two 1/2" Annin valves, Model 3620. All the fittings and associated hardware were likewise Monel. Wherever possible, Monel Swagelok fittings were used for closures. Threaded connections when used were generally back brazed to prevent leakage. After an initial adjustment, the Annin valves performed satisfactorily throughout the entire program and can therefore be highly recommended for this service. In order to operate safely at the maximum required pressure of 1500 psig, it was

necessary to back pressurize the bellows of the Annin valves. This was accomplished by opening Hoke solenoid Valves 1C and 2C (Figure 1). As can be seen from this drawing, pressure on both sides of the bellows was thereby readily equalized.

The Annin valves were pneumatically operated by nitrogen pressure applied to a domotor via solenoid Valves 1A or 2A. Nitrogen at 100 psig assured rapid valve operation and leak tight closure.

In addition, the manifold was tied into a vacuum system (not shown), the pump of which was protected by a hot charcoal scrubber and a soda lime trap. Pressure in the loop was released by bleeding the gases through the charcoal scrubber which effectively decontaminated OF_2 exits. This scrubber, designed with a water cooled inlet, operated satisfactorily with ordinary charcoal briquettes.

The electric circuits and solenoid valves were controlled from a panel board. The circuitry was designed so that all switches could be operated individually or in any desired combination. Circuits were provided so that runs could be started manually and terminated automatically when the runs were completed. These run circuits were tied into a timer which recorded to 0.1 seconds the duration of the dynamic flow of OF_2. The automatic circuits were controlled by the compound gauge or pressure switch. The circuit was also designed

to prevent over-pressurization of the compound gauge. A second electrode in this gauge was tied into an alarm system. The timer and pressure alarm were likewise located on the panel board. The operation of the automatic run circuits will be more completely described under "Operating Procedure".

2.2. Materials

Twelve materials were exposed to liquid OF_2 under dynamic conditions. Test specimens included nine alloys and samples of welded, brazed, and silver soldered Monel. The test specimens, discs of approximately one inch in diameter, had been machined from sheet stock of the parent metal. An orifice was drilled through the center of the disc with a No. 80 drill, 0.0135 inch diameter. The inlet side of the orifice was slightly enlarged using a counter-sink but the outlet edge remained untapered.

In the case of welded or brazed materials, a disc of Monel was used as the parent metal. A 1/4 inch hole was drilled through the center of the disc and the hole was filled with brazing rod, silver solder, or Monel weld rod. In all cases, the molten alloy was applied using the approved technique for the specific material. The hole was overfilled with the filler metal which was then machined flush with the parent Monel disc. The orifice was drilled through the filler metal. Test materials together with their suppliers and chemical analyses are shown in Table 1.

2.3. Cleaning and Passivation

All components of the test equipment as well as the test specimens were subjected to a very rigid cleaning procedure. The material was sonically cleaned in a hot water solution of detergent followed by repeated hot water rinses to remove any trace of detergent. This was followed by several rinses with distilled water. The water was then removed by several rinses with Genesolv DI, a mixture of 35% isopropanol and 65% Genesolv D. The pieces were then given a final sonic wash and rinse with Genesolv D. All materials were then dried in a vacuum oven.

The orifice specimens were examined under a microscope and metallic burrs and other contaminants were removed from the orifices before being cleaned as described above.

The valves, lines, and gauges in the manifold and the test loop were passivated with fluorine upon assembly and after each re-installation of test specimens. The system was evacuated and then slowly pressurized with fluorine to 100 psig for approximately one hour. The fluorine was then vented, the system purged with nitrogen, and evacuated.

2.4. Operating Procedure

The test loop had been designed so that two specimens could be evaluated with one charge of OF_2. The OF_2 was charged to one service cylinder and pressurized to the desired pressure. The other side of the loop had

been evacuated. When the appropriate Annin valve was opened, the OF_2 flowed through one orifice and was collected in the evacuated cylinder. The completion of the run was noted by a pressure rise in the receiving cylinder as helium entered. The process was then reversed with flow directed from the second cylinder and through the second orifice.

In describing the operation of this dynamic test equipment reference has been made to the valves as numbered in Figure 1. It should be noted that all the solenoid valves shown in this drawing (Nos. 1A, B, C and 2A, B, C) are normally closed. Valves 1A and 2A actuate the respective Annin valves since they control the nitrogen flow to the Annin Domotor. The stepwise procedure which is described below was performed after the leak testing and passivation had been completed.

a. Entire system evacuated.
b. Liquid nitrogen tank filled until test loop submerged.
c. Gaseous OF_2 condensed into one of the service cylinders. (Cylinder 1 through Valves 7 and 2, for example.) The amount of OF_2 charged to the cylinder was measured from the pressure drop in the main OF_2 supply cylinder. The calibrated gauge used for this purpose and the OF_2 cylinder are not shown.

d. When the desired amount of OF_2 had been condensed, Valve No. 7 was closed and the helium used to pressurize the OF_2 was then fed from a pre-set pressure regulator through Valves 4, 5, 2 and 2C. Valve 2C was used to pressurize the Annin valve bellows when run pressures were 600 psig or greater, thus maintaining equal pressure on both sides of the bellows. Approximately three pounds were condensed. Since the normal holdup in lines and cylinder heels was 442 gms. of OF_2, approximately two pounds of OF_2 was available for transport through the orifices.

e. Valve 2B which remained open to measure the vacuum on the unfilled side of the loop (Cylinder No. 2) was at this time switched over to automatic control. On automatic it opened and closed in series with Valve 2A, the Annin valve control solenoid. The compound gauge shown in the drawing was equipped with two electrodes and also served as a pressure switch. The electrode set at the lower pressure permitted the automatic circuit to operate when a lesser pressure was in Cylinder No. 2. When pressure rises, the electrode makes contact and the automatic circuit is broken, shutting off the timer, closing the Annin valve and Valve 2B.

f. To start a test run, a switch was thrown which simultaneously opened Valves 1A and 1B and started the timer. When all the OF_2 had been forced through the orifice in sample holder No. 1, the helium entered the OF_2 receiver causing the pressure in this vessel to increase. This pressure change opened the pressure switch circuit thus causing Annin Valve No. 1 (through 1A) and Valve 1B to close and shut off the timer. The timer recorded the OF_2 flow duration to a tenth of a second. If pressure continued to increase in the OF_2 receiver through valve leakage or electrical malfunction, the second electrode in the pressure switch was actuated. This automatically closed all valves and sounded an alarm. The pressure switch was sensitive to pressure changes of less than 1 psig.

g. The next run was through the second test specimen. To prepare for this run the residual pressure in the left side of the loop was vented and this section was then evacuated with Valve 1B opened to the compound gauge. When evacuation had been completed, Cylinder No. 2, which now contained the OF_2, was pressurized with helium using the corresponding valves for this side of the loop. In this manner, runs could be repeated in either direction until the desired dynamic flow time had accrued.

h. When shutting down the system for weekends, or to replace test specimens, the helium was vented and the system evacuated to remove any residual helium. The system was then closed off, the LN_2 drained from the tank and the OF_2 allowed to vaporize. The OF_2 gas was vented back to the OF_2 supply cylinder until a pressure equilibrium was reached. The remaining OF_2 gas in the loop was then vented through the charcoal scrubber and the system purged and evacuated.

2.5. Experimental Data

In the evaluation of the twelve materials, a total of 610 runs were recorded and an equivalent of almost 1200 pounds of liquid OF_2 was forced through the test orifices. The total dynamic test time for all specimens was 150 hours. (Table 2) These totals do not include many runs that were manually terminated when the orifices appeared to have become plugged. Since it was impossible to determine at what time during the run blockage occurred, these runs were totally discarded. Runs where a partial plug occurred as indicated by a significant increase in the run duration were included in the total OF_2 throughput and the cumulative dynamic time. However, such runs were not used in calculating average mass flows or measured velocities. Each run has been reported and the mass flows and measured velocities calculated. In addition, the average data for each material at each pressure increment has also been listed. The tables

for each material are identified in the section describing the particular specimen.

2.5.1. Monel and Nickel

The initial runs were made with the nickel and Monel orifices. These specimens were tested at pressures of 120, 300, 500, 700, 900, 1200, and 1500 psig. It appeared that the difference in mass flows at 200 psi increments was rather small and it was decided to go to 300 psi increments after the 900 psig run. It should, of course, be noted that it took considerable time to prepare for each run and it was therefore not feasible to continue to run at 200 psi increments. The complete experimental data for Monel and nickel are shown in Tables 3 and 4, respectively. Summaries for each metal are shown in Tables 5 and 6.

During this first series of runs, considerable orifice plugging occurred and an investigation was made as to the cause of this plugging. The test loop was dismantled and inspected for any particulate matter that could be the cause of this blockage. A small amount of contaminants was recovered from the lines and cylinders and examined under a microscope. Identified contaminants included fine metallic slivers, copper flecks, and Teflon particles. The slivers had abraded from the several pipe thread connections and the Teflon from the pipe thread tape. The copper apparently had abraded from service lines when the Swagelok connections were tightened.

In addition, there was a small amount of metallic fluorides which probably formed during passivation. The particulate matter was believed to have contaminated the system after the cleaned components had been assembled. We therefore re-assembled the loop in several sub-assemblies which could be flushed completely. The cleaned assemblies were then connected with a minimum of Swagelok closures which we believed reduced the contaminants in the system. In addition, before making the final connection, the loop was completely blown out with high pressure nitrogen. It should be noted that the dip tubes in the cylinders had been shortened to permit a larger heel of OF_2 to remain behind. This reduced the possibility of carrying over any sediment or heavy particles that might be present.

The OF_2 was checked to see if the contamination that caused the plugging came from this source. CO_2 and HF are normal condensible impurities in OF_2. A sample of OF_2 from our supply cylinder was condensed and the liquid visually examined for solid particles (CO_2 and HF). No particles were observed. This procedure was repeated with a second OF_2 sample and again no solids could be seen. An infrared analysis of a sample of OF_2 disclosed: CO_2 not detected, HF $\simeq$ 0.02%, CF_4 trace. This checked with the analysis supplied with the OF_2 cylinder. Therefore, it was felt conclusively that the OF_2 was not the source of contamination.

When the loop was re-assembled the tests were conducted with fewer orifice plugs. Upon completion of the tests, the system was purged and evacuated before the specimens were removed. The specimens were re-weighed and photomicrographs taken for comparison with the orifice before exposure. Both specimens showed a slight weight gain which is assumed to be a fluoride film. The diameter of the Monel orifice remained virtually unchanged. The increase of the orifice diameter was 0.0002 inches. The nickel orifice, however, shows an enlargement of approximately 1 mil., changing from 0.0139 to 0.0150 inches. This indicates an enlargement of almost 8%. The photomicrographs of these specimens are shown as Exhibits 1 and 2 in the Appendix.

2.5.2. S.S. 304 and Aluminum 2024

Before starting the runs with these materials the valve manifold was completely rebuilt with Hoke M343 valves in place of the Pressure Products valves. The system was then leak tested and passivated. Runs were made at 120, 300, 600, 900, 1200, and 1500 psig. The run data for the S.S. 304 and aluminum 2024 are shown respectively in Tables 7, 9 and 8, 10. Although some plugging was still encountered during this series of runs, the performance was definitely an improvement over the previous series. Some slight leaks did occur during this work but none was deemed serious enough to curtail the test program. The specimens were weighed

and photographed before and after exposure to liquid OF_2. The stainless steel 304 specimen showed a slight gain in weight (+0.0013 gms.) while the aluminum 2024 showed a very slight loss (-0.0003 gms.). Neither specimen showed any change in the orifice diameter. The S.S. 304 specimen had a slightly dulled appearance while the aluminum 2024 remained relatively unchanged. Photomicrographs of these materials are shown in Exhibits 3 and 4.

2.5.3. Aluminum 6061 and Titanium

To further reduce the possibility of orifice plugging the system was completely purged with nitrogen gas at high pressure before replacing the specimen holders. It was felt that this procedure would blow out any extraneous particulate matter which we believed had caused the orifice blockage. The specimen holders were then installed and the test loop pressure tested and repassivated before the runs were started. Runs were made at six pressure increments ranging from 120 to 1500 psig. The results of the aluminum 6061 and the titanium runs are shown respectively in Tables 11, 13 and 12, 14. Photomicrographs are shown as Exhibits 5 and 6.

This series of runs had far fewer plugs than the previous series. It was felt that the added precaution of blowing out the loop with high pressure nitrogen was instrumental in the performance improvement. The specimens were

weighed and photographed both before and after exposure. The aluminum 6061 showed no weight change while the titanium showed a gain of 0.0013 gms. Comparison of the photomicrographs taken before and after showed an orifice enlargement of 1 mil. for the aluminum 6061. This specimen appeared to be very slightly tarnished. The titanium specimen showed no enlargement. However, several rust colored areas were noted on the surface of this specimen. Under a microscope, these discolored areas appeared to be either tiny blisters or pits. It appeared that the initial corrosion effect was the formation of a tiny blister. The blisters then apparently broke leaving a pit. Some blisters were seen with cracks or partially broken open. It should be noted that these corroded areas represented only a very small percentage of the total specimen surface. Peculiarly this surface discoloration was noted only near the specimen edge which was in contact with the aluminum gasket.

2.5.4. Stainless Steel 301 and Inconel

As in the previous series of tests, the loop was blown out before installing the specimen holders. When the loop was closed it was leak tested and passivated. Runs were made at six pressures ranging from 120 to 1500 psig. Some severe plugging problems were encountered at the very onset of this program. In fact, the first ten runs were marred by plugging. A severe snowstorm prevented any work on this setup for two days. During this period, the OF_2 remained

in the loop and the LN_2 supply was replenished as needed. Peculiarly when work was resumed not a single plug was encountered until halfway through the 900 psig runs. At the conclusion of the series of runs at 900 psig, the system was shut down for a weekend. The weekend shutdown involved the evacuation of helium from the system and draining the liquid nitrogen from the tank. The liquid OF_2 was then slowly vaporized and permitted to return to the original supply cylinder. Normally by Monday morning the entire system had warmed to ambient temperature and the loop pressure was approximately equal to the cylinder pressure which was recorded before charging OF_2 to the loop. On this occasion, the pressure was quite low and a strong odor of OF_2 was noted. The leak was located at one of the specimen holders and it was necessary to remove it to make repairs. After repairs, it was replaced, and the system again leak tested and re-passivated. It should be noted that, except for the first series of runs, this was the only case where a specimen was removed from the loop before the completion of all the required runs. During the time the specimen (S.S. 301) was out, it was kept dry and clean in a vacuum oven.

The specimens had been weighed and photographed both before and after the runs were completed (Exhibits 7 and 8). The S.S. 301 showed a loss of 0.0009 gms. while the Inconel gained 0.0006 gms. The S.S. 301 micrographs indicated an orifice enlargement of

.001 inches but the Inconel orifice remained unchanged. Both specimens had a slightly tarnished appearance after exposure but otherwise showed no other signs of corrosion. The experimental data for the S.S. 301 are shown in Tables 15 and 17. The data for the Inconel are reported in Tables 16 and 18.

2.5.5. Brazed Monel and Welded Monel

Before installing these specimens, the loop was again blown out to remove any particulate matter. After installation of the specimen holders, the system was again leak tested and passivated before OF_2 was charged to the loop. Runs were made at 120, 300, 600, 900, 1200 and 1500 psig. The results of these runs are shown in Tables 19, 21 and 20, 22, respectively, for the brazed and welded specimens. Some plugging was encountered in these runs. Of greater concern was an increased incidence of valve problems. As a result of the frequency of valve manipulations, leakage occurred through several valve packings including a packing seal in an Annin valve. This packing seal permitted the back pressurization of the bellows. Tightening the packing nuts on the Hoke valves became a twice daily routine during the runs at 1200 and 1500 psig. Both specimens were weighed and photographed, Exhibits 9 and 10, before and after exposure. Both specimens showed very small weight gains. The brazed specimen picked up 0.0005 gms. and the welded specimen gained 0.0003 gms. The welded orifice showed no enlargement but the brazed unit showed an increase in

diameter of .0003 inches. The welded specimen had a somewhat tarnished appearance with no significant difference between the weld and the parent metal. The brazed specimen was discolored. The brazed metal per se was a dark brown in sharp contrast to the parent metal. Microscopic examination of the brazed area disclosed a very slight surface etching. No significant corrosion was noted at the braze-Monel interface.

2.5.6. Silver Soldered Monel and Copper-Chromium

Before the specimens were installed, the loop was blown out as previously described. As a result of the leakage that occurred towards the end of the last series of runs, the entire system was given an extensive leak test. During passivation, fluorine odors were noted which were eventually traced to the packing glands of Valves 2 and 3 in the manifold. These valves were removed and we found that the Teflon seals had been extruded from the packing gland and were no longer functioning properly. The valves were replaced with new Hoke Y344 valves and the system again passivated. Runs were completed at 120 and 300 psig but during the 600 psig runs another manifold valve started to leak and the system was shut down to replace this valve. A leak in the water cooled inlet of the charcoal burner was discovered at this point as the burner filled with water. This in turn necessitated the replacement of the burner with a spare before we could vent the system. After the

valve was replaced, the system was again passivated before continuing the runs. The 900 and 1200 psig runs were completed despite some orifice plugging problems and assorted valve leaks and malfunctions. During the 1500 psig runs, Valve No. 3 in the manifold developed a leak across the seat. It therefore became impossible to maintain a vacuum on the No. 2 cylinder side of the loop and tests through the silver soldered orifice were curtailed. The required dynamic time, however, was achieved through the copper orifice.

The specimens were weighed and photographed both before and after exposure, as shown in Exhibits 11 and 12. We were quite surprised to discover that the copper specimen had become dish shaped as a result of these tests. (The distorted specimen is compared to an unused specimen in Exhibit 12D.) Before starting this work calculations had been made to determine whether the test materials would become distorted at 1500 psig and all appeared to have more than sufficient strength. It appears that the distortion was a result of the hammer effect of the liquid slugging against the orifice when the Annin valve was opened. As a result of the distortion and the consequent stretching of the metal, the orifice outlet showed an appreciable enlargement, approximately 5 mils. However, the outlet edge was still sharp and showed no signs of corrosion or erosion. This specimen showed a gain in weight of .0012 gms. It is believed

that if the aperture enlargement was due to erosion or corrosion the specimen would have lost weight. The appearance of the specimen was good, being only slightly tarnished by the OF_2. The silver soldered specimen gained .0009 gms. and showed no change in orifice diameter. It did, however, appear to be more tarnished. The silver solder area was moderately darkened and this surface showed etching. No significent corrosion was noted at the interface of the solder and parent metal.

The experimental data for the silver soldered specimen are reported in Tables 23 and 25. The data for the copper-chromium alloy is shown in Tables 24 and 26.

2.6. Conclusions

The physical changes in the test specimens after their dynamic exposure to liquid OF_2 have been shown in Table 27. No specimen was considered to be unsuitable for subject service on the basis of weight change. The slight changes in weight are not unusual considering the length of time and the varied conditions of exposure to OF_2. It should be noted that many specimens were subjected to OF_2 exposure for as long as two weeks. This exposure included both gas and liquid phase OF_2 contact. In addition, the specimens were exposed to OF_2 at ambient as well as cryogenic temperatures. During the weekends, for example, specimens were exposed to gaseous OF_2 at cylinder pressure as the gas was vented to the cylinders. In addition, all specimens were initially exposed

to fluorine gas at elevated pressures during the passivation period. In view of this background, little significance can be attached to a slight change in weight.

The appearance of the orifices is a better criterion to rate the compatibility of the specimens to liquid OF_2. The orifice diameters were measured from photomicrographs taken before and after exposure. The untapered exit sides of the orifices were used for these measurements. The micrographs were 150X enlargements. Hence, a diameter change of 0.05 mm., which was easily measured represented an actual diameter change of approximately 0.00015 inches. As shown in Table 27, half the specimens showed some measurable enlargement. Of these only nickel and aluminum 6061 showed a significant enlargement, one mil or more. These materials would therefore be of questionable utility in dynamic service requiring a high degree of dimensional stability.

The copper-chromium alloy showed an apparent severe orifice enlargement. However, we strongly believe that this was merely the result of the metal stretching when it deformed as a result of the hammer effect of the OF_2. We have attempted to verify this by photographing the inlet side of the specimen as seen in Exhibit 12C. Despite the taper on this side the microscope was focused at the point where the normal diameter starts. Measurements of these photographs indicate no change from the original orifice

diameter taken from the exit side of the specimen. No photographs were taken of the inlet side before exposure. Therefore, a more exact comparison could not be made. Copies of the several photomicrographs referred to in this report may be found in the Appendix. Unless otherwise indicated all photomicrographs show the outlet side of the orifice.

No attempts have been made to rationalize the accumulated data. We realize there are some overlaps in the data from consecutive pressure increments in a few instances. However, the main objective of this phase of the contract had been to establish the compatibility of the various test specimens to liquid OF_2 under dynamic conditions. This we have achieved and we have firmly established that with suitably designed equipment which has been properly cleaned and passivated, liquid OF_2 can be safely handled at high pressures and velocities.

3. DYNAMIC TESTING OF PLASTICS IN LIQUID OF_2

Eight plastic materials were exposed to liquid OF_2 under dynamic conditions in the same test equipment used for the metal specimens. This work, authorized under NASA Contract No. NAS 3-2564, was held in abeyance pending completion of the dynamic compatibility tests being conducted under Contract NAS 3-6298. The test requirements for these plastic materials were less demanding than for the metals. The minimum required duration of dynamic exposure at each pressure increment was five seconds and the maximum test pressure was 500 pounds.

3.1. Apparatus

The apparatus was the same as used for the metal specimens shown in Figure 1. This equipment has been fully described in Section 2.1. of this report. Since the plastic specimens require greater support, new specimen holders were designed and fabricated as shown in Figure 3. These holders, fabricated from heavy wall 1/2" Monel unions, provided the necessary backup for the plastic materials. The mating faces of the holders were serrated and the test specimen itself therefore served as a seal obviating the need for gaskets.

3.2. Materials

The eight materials selected were those that appeared to be most suitable for dynamic exposure on the basis of static liquid OF_2 exposure and cryogenic tensile testing. The test materials together with their chemical composition, the manufacturer, and fabricator, are listed in Table 28. The test specimens were discs of approximately 1/2" diameter punched out of sheet stock approximately 1/8" thick. The orifices were drilled through the centers of the test discs using a No. 80 drill, 0.0135 inch diameter. One end of each orifice was slightly countersunk and was used as the inlet side. The outlet edge of the orifice remained untapered.

3.3. Cleaning and Passivation

The specimens were subjected to a microscopic examination during which plastic shavings or "burrs" from the drilling operation were removed. Considerable difficulty was encountered in removing the shreds of plastic that formed at the edges of the orifices as the drill broke through. Pulling off such shreds generally raised others. The small size of the particles precluded removal by cutting. Attempts to remove same by polishing were unsuccessful and in fact worsened the appearance of the orifices. The fuzzy edges are quite obvious in some of the photomicrographs* included in the Appendix of this report.

* Exhibit 13, Almac CTFE is an example.

When the specimens were reasonably free of these particles, they were cleaned by boiling in concentrated nitric acid for two hours. After a thorough water wash to remove the acid, they were rinsed with distilled water. The specimens were then dried with acetone and thoroughly rinsed in Genesolv D before overnight drying in a vacuum oven at 80°C. The cooled specimens were weighed and photographed before installation in the test loop.

It should be noted that unlike the procedure for the metal orifice specimens, the test loop was not passivated with the plastic orifices in place. It was felt that the fluorine might have a deleterious effect upon the plastic specimens. The test loop, however, was passivated with fluorine at 110 psig for two hours without the specimen holders in place by plugging off the connections to the holders. The holders, since they were not passivated, were subjected to a very rigorous cleaning procedure as described in Section 2.3.

3.4. Operating Procedure

When the specimen holders were in place, the test loop was pressure tested before the OF_2 was charged to the system. The charge of OF_2 was sufficient to provide a liquid transport through the orifices of approximately 100 gms. It was estimated that this would provide OF_2 for 20 second runs at 120 psig and also meet the minimal run duration requirement of five seconds at 500 psig.

All sets of specimens were exposed at three pressure increments, 120, 300 and 500 psig. At least two runs were made through each specimen at each pressure increment. The actual operating procedure was identical to that used for the metal specimens as described in Section 2.4.

3.5. Experimental Data

A total of 58 runs were made through the plastic orifices with no failures or burnouts occurring. This is equivalent to almost 13 pounds of liquid OF_2 in transport. The experimental data for these runs have been reported in Tables 29 to 36. In most cases, good correlation was found in the duplicate runs. When two runs showed poor correlation, a third or fourth run was made on that particular specimen.

Specimens were weighed both before and after exposure. the specimens generally had a strong odor of OF_2 when removed from the holder despite the fact the test loop had been evacuated overnight before being opened. It was felt that this reflected absorbed or adsorbed OF_2. Therefore the specimens, in addition to an immediate weighing after removal, were placed in a vacuum oven for three hours at 75°C and then re-weighed. These weights, together with the weight changes in grams between the initial and the final weighings, are shown in Table 37. Photomicrographs were made of the orifices before and after exposure to determine if enlargement had occurred. To our surprise, six orifices appeared to have diminished after exposure.

These materials were the relatively soft tetrafluoroethylene (TFE)* and FEP specimens. The more rigid materials, trifluoromonochloroethylene (CTFE), remained unchanged.** We therefore believed that the apparent decrease in orifice diameter resulted from the specimens deforming or cold flowing as a result of the high pressure applied by the specimen holder. Some selected micrographs illustrating this phenomenon have been included in the Appendix of this report.

To prove that the reduction in orifice was, as suspected, a result of cold flow rather than an effect of the OF_2, an additional specimen was prepared for use as a blank. The specimen, Halon TFE G-50, was prepared and cleaned in the same manner as the test specimens. The blank, after being photographed, was secured in a specimen holder and then immersed in liquid nitrogen to simulate the actual test temperature. After three days immersion, it was removed from the holder and placed in a heated vacuum oven as were the test specimens before their final re-weighing. Photomicrographs taken of the orifice after this treatment were compared to those previously taken and clearly showed the orifice diameter had changed from 0.0122" to 0.0096". This is a decrease of approximately 21% and firmly established that cold flow and permanent set could occur as a result of the

* Exhibits 14 and 15

** Exhibit 13

physical environment. The dimensional changes were therefore considered not indicative of chemical reactions with OF_2. The micrographs of this blank specimen may also be found in the Appendix as Exhibit 16.

3.6. Conclusions

On the basis of this study it appears that the several plastic materials are chemically compatible with liquid OF_2 under conditions of high pressure and velocity. The meticulous attention given to the preparation and cleaning of the specimens was undoubtedly a significant factor in the successful completion of this particular investigation.

No specimen showed any erosion or corrosion effect after OF_2 exposure. The weight changes in the several specimens are quite small and are not considered to be a significant indication of degradation or chemical reaction. Therefore on the basis of weight change the materials likewise appear to be satisfactorily resistant to the OF_2 under the subject test conditions.

An inconsistency was noted in the Reynolds numbers (Re.No.) achieved with the plastic materials as compared to the numbers calculated for the metal specimens for the same pressure increments.

REYNOLDS NUMBERS

Material	120 psig	300 psig	500 psig
Monel	14,985	24,515	28,722
Nickel	15,768	24,370	32,639
Teflon 5	13,171	28,287	49,320
Halon TFE G-50	16,537	26,546	41,052

You will note that at 120 and 300 psig, the calculated Re. Nos. are in excellent correlation. Yet at 500 psig the numbers for the plastics are much higher. We believe that this is evidence of the temporary deformation of the 1/8" thick discs at 500 pounds pressure. Such deformation would result in orifice enlargement. When the pressure was relieved the disc recovered its original shape. Since the Reynolds numbers were calculated on the basis of the initial orifice area, the listed results are obviously not indicative of the true measured velocities and Reynolds numbers.

It would therefore appear that despite the compatibility of these materials with liquid OF_2, their tendency to cold flow and deform under pressure would render them of questionable utility under dynamic conditions. However, where dimensional stability is not a critical factor, these materials could find useful application in liquid OF_2 service.

4. DYNAMIC TESTING OF METALS IN OF_2 GAS

Twelve metal orifices were exposed to OF_2 gas at approximately sonic velocity. Exposure time for each specimen was ten minutes. All tested materials appeared to be completely unaffected by this exposure.

4.1. Apparatus

The test setup, shown in Figure 4, provided a relatively unsophisticated method of transporting a controlled flow of OF_2 gas through a test orifice. The OF_2 was fed directly from a supply cylinder into the setup. Two Hoke 344 needle valves (Nos. 1 & 2) in conjunction with a pressure gauge (A) were used to measure the supply cylinder pressure before and after each run, thus providing a convenient means of estimating the gas flow through the orifices. Valve No. 3, a Hoke 343, was used for controlling the upstream pressure on the orifice which was indicated on compound gauge B. The orifice was mounted in a specimen holder (Figure 2) located between the two compound gauges (B & C). Gauge C was used to measure the pressure downstream from the orifice. Valve No. 4, which opened during the runs, was closed when the setup was pressure tested or evacuated. The spent OF_2 was vented through a charcoal burner where it was effectively decomposed. The setup was provided with connections for various services. Fluorine was available for passivation, nitrogen for purging and pressure testing, and a

vacuum line to remove residual traces of OF_2 before opening the system to change specimens. The entire system functioned satisfactorily throughout the test program.

4.2. Materials

The materials tested consisted of a set of orifice test specimens identical to those used in the liquid OF_2 dynamic investigations. The materials are completely identified in Table 1. The specimens consisted of discs of metal through which orifices of 0.0135" diameter were drilled using a No. 80 drill. One end of the orifice was slightly countersunk and this was used as the upstream side of the orifice. The downstream opening remained untapered.

4.3. Cleaning and Passivation

The orifices were examined under a microscope and all burrs and drill turnings removed. Where necessary, the faces were polished to provide a sharp outlet edge to the orifice. When the specimens were satisfactorily free of particulate contamination, they were subjected to a multi-step cleaning procedure as described in Section 2.3.

The lines, gauges, and valves used in the test setup were removed from the liquid OF_2 dynamic setup and therefore did not require disassembly and re-cleaning. As a routine precaution, however, the assembled setup was passivated with fluorine at 75 psig for one hour. The system was then vented, flushed and evacuated

before runs were made. It was not deemed necessary to re-passivate the system before each subsequent run. However, extreme care was taken during the removal and re-installation of the specimen holders. When the holder was not in place, the lines were capped or plugged to prevent the entrance of atmospheric moisture into the system.

4.4. Operating Procedure

The setup was leak tested and passivated before the initial run. The fluorine was then vented to the charcoal burner, the system flushed with nitrogen and then evacuated to assure the removal of all traces of fluorine. In preparation for the initial run, the main cylinder valve and Valve No. 1 were opened and the cylinder pressure shown on Gauge A was recorded. With Valves 5, 6, and 7 closed, Valve No. 2 was partially opened and was used as a throttling valve during the run. To start the run, Valve No. 4 was opened fully. Valve No. 3, the control valve, was used to regulate the upstream pressure on the orifice at 60 psig for the ten minute run. The two compound gauges (B & C) were monitored continually to assure a constant pressure differential across the orifice of 60 psia. At no time was a pressure buildup on Gauge C noted.

At the completion of the run, Valve No. 2 was closed and the system flushed out with nitrogen. The pressure reading on Gauge A was again taken, and the pressure drop in the supply cylinder recorded. When the system

was deemed to be relatively free of OF_2, it was closed off and evacuated to remove the last traces of OF_2. Before removing the specimen holder, the system was padded with nitrogen to a slight positive pressure. Thus, when the connections were broken the nitrogen leaked out, preventing the atmospheric air from entering the system. When the holder had been removed for specimen replacement, the open connections were sealed to prevent possible contamination.

When the next specimen was installed, the system was pressure tested with nitrogen before preparing for the run. In these succeeding runs, all steps previously described were followed except that the initial fluorine passivation was omitted. No difficulties were encountered at any time with this equipment.

4.5. Experimental Data

Each test specimen was exposed to gaseous OF_2 at approximately sonic velocity for ten minutes. This velocity was achieved by maintaining a pressure differential of 60 psia across the orifice. Our preliminary calculations had indicated that this pressure would be more than adequate to achieve sonic velocity through the 0.0135" diameter orifice. To ascertain our actual run velocities, the OF_2 in transport was calculated based on the pressure drop in our OF_2 supply cylinder (3016 $in.^3$ capacity). This data is shown in Table 38. It should be noted that the observed pressure differentials varied slightly from run to run, ranging from a low of

8 psia to a high of 12 psia. The majority of runs appeared to have a pressure drop of 10 psia. It must be emphasized that the gauge used for this purpose was a high pressure gauge capable of taking full-cylinder pressures. The smallest gradations on this gauge were therefore in five-pound increments. Readings were estimated to the nearest pound, but the accuracy of each reading was possibly ±1/2 lb. Therefore, the precision of the measurements is somewhat less than we would have preferred. However, calculations based on this estimated OF_2 transport data confirm that we did achieve approximate sonic velocity in these runs. This data calculated for two runs with different pressure drops in our supply cylinder are shown below:

Pressure Drop	Sonic Velocity (ft./sec.)		Mass Flow (lbs./sec.)	
Psia	Calc.	Meas.	Calc.	Meas.
12	740	732	3.27×10^{-4}	3.22×10^{-4}
10	740	615	3.27×10^{-4}	3.00×10^{-4}

The sonic velocity, gas density, and mass flow were computed assuming isentropic flow of an ideal gas through the orifice. The critical pressure ratio for OF_2 was calculated to be 0.538 based on a specific heat ratio (cp/cv) of 1.33.

The specimens were weighed both before and after exposure. All specimens except the Monel showed either a negligible weight change or none at all. The Monel specimen, which was in the setup during the initial

fluorine passivation procedure, gained 0.0008 gms. This, we must assume, was a result of its exposure to fluorine rather than the OF_2 contact. These weights are also included in Table 38.

Photomicrographs were taken of the specimens, both before and after their exposures. In no case could any change in appearance or orifice dimension be seen.

4.6. Conclusions

All test orifices are completely compatible to gaseous OF_2 at sonic velocity at ambient temperatures. A ten minute exposure to OF_2 did not produce any discernible changes in either the appearances or the orifice dimensions of the specimens. Weight changes were considered to be negligible, and were probably more a reflection on the sensitivity of the balance used for these weighings, than an indication of chemical reaction.

5. IGNITION OF METALS IN OXYGEN DIFLUORIDE

The ignition temperatures of metal wires in OF_2 gas at atmospheric pressure were determined. Preliminary runs were made to establish suitable techniques and adequate equipment for this investigation and to verify the accuracy of the resistivity-temperature data obtained from the literature. In addition, approximately 100 runs were made using a programmed constant power supply. The resultant wire burnout curves were plotted on a recorder. About one-third of these runs involved wire ignition in a helium atmosphere and were used to determine the optimum wire length and to examine the effect of various wire coil geometries. The remaining runs involved wire ignitions in OF_2 under carefully controlled conditions.

5.1. Preliminary Study

While awaiting the delivery and assembly of a constant power source and accessory equipment, exploratory wire ignition tests were conducted. These preliminary investigations were made primarily to check the accuracy of the resistivity-temperature data we had obtained for the several test materials.

5.1.1. Preliminary Apparatus and Equipment

The initial ignition chamber used for the exploratory tests was a glass tube sealed at each end with rubber stoppers. Inserted through these stoppers were copper rods for electrodes and 1/4" copper tubing which served as gas inlet and outlet. This unit was quickly seen to

be inadequate and was replaced by the more efficient setup shown in Figure 5. This unit utilized a one liter resin flask, the head of which provided four openings through which standard tapered joints could be inserted. Two electrodes were machined from copper rods to mate snugly with ground glass standard tapered adaptors. The remaining two openings were used to accommodate a glass gas inlet tube which extended to the bottom of the flask, and a vent outlet, respectively. A rotameter located in the exit line was used to measure gas flow.

For both setups, the voltage input was regulated by a 20 amp. capacity powerstat. The amperage readings were taken from a G.E. Amprobe and a Simpson voltmeter. The wire temperatures were determined from the resistivity calculated from the voltage-amperage readings and checked with a Leeds & Northrup optical pyrometer. No attempts were made to finely calibrate these instruments since one of the purposes of this exploratory work was to visually observe the phenomenon of wire ignition rather than to provide finite measurements.

5.1.2. Test Materials

Seven different materials were included in this investigation; in addition to these materials, a second sample of nickel wire of a different size was also tested. Monel 400 wire obtained from two different sources was used. The test materials, initial wire diameters, sources of supply, and the

reported nominal melting points of the materials are listed in Table 39.

5.1.3. Preliminary Experimental Procedure and Data

The glass tube reactor was chosen for the initial studies because of its simplicity. However, the inadequacy of this reactor became readily apparent. When the tube was mounted in a horizontal position, the wires expanded and sagged as they were heated (by increasing the voltage output of the powerstat), thereby contacting the reactor wall. The tube was then tested in a vertical position. In three runs the burnout was always initiated at the upper end of the test wire as a result of heat buildup at this point. These tests also demonstrated the need for better closures since the stoppers were ignited by the burning wires.

Tests verified the feasibility of the resin flask setup (Figure 5) and this unit was used subsequently. The initial test on each wire in this equipment was conducted in a nitrogen atmosphere to establish the approximate amperage-voltage limits. The system was then flushed with OF_2 and the wires re-heated in an OF_2 atmosphere. The several preliminary runs conducted in this manner are described in detail and summarized in Table 40.

The wire ignitions were carefully observed and a detailed description of this phenomenon has been included in the data for these preliminary runs. It should be noted that these visual descriptions

are equally applicable for the later runs in which better instrumentation was available.

Run No. 1. 32 ga. nickel wire (.0088"dia.), wound into a coil containing approximately 30 inches of wire, was checked in nitrogen before testing in OF_2. In the OF_2 atmosphere, the voltage was slowly increased until, at 43 volts, the coil started to glow at one point. The wire immediately ignited and an amperage reading could not be obtained. The wire broke into fragments, each of which ignited vigorously with sparks flying about the flask. The chamber fogged up and the exit lines and rotameter were fouled with a white deposit believed to be NiF_2. White fumes were also seen exiting from the vent line. It was noted that the wire glowed prior to ignition at one end near an electrode. The remainder of the coil did not glow. It was felt that this glow was actually the inception of ignition.

Run No. 2. A 3-7/16" length of .0088" dia. nickel wire was heated in a nitrogen atmosphere. Initial glow occurred at a setting of 3 volts and 2.4 amps. Power was increased in 1 volt increments. At 8 volts, it drew 4.2 amps. At this point, wire temperature was estimated to be approximately 1050°C. An optical pyrometer was used and the temperature was just below the instrument's minimum scale calibration at 1075°C. When the voltage was increased to 9, the wire burned out. The wire was very brittle and discolored, possibly owing to nitride formation.

Run No. 3. As in Run No. 2, a 3-7/16" length of .0088" dia. nickel wire was checked in nitrogen up to 7 volts (3.6 amps.). The nitrogen was then replaced with OF_2. Voltage was increased in 1 volt increments. Between 2 and 3 volts (2.0-2.5 amps.) wire began to glow. Wire remained intact at 7 volts and 3.8 amps., at which point current was shut off. Several minutes later current was re-applied as before. At 6 volts, the amperage started to flicker and the wire burned out. Wire was not totally consumed. Remaining wire had a gray-white coating.

Run No. 4. Copper wire (3-7/15" x .0126" dia.) was tested in nitrogen. The low resistance of this material caused a high current flow which blew the fuse in a small powerstat. A 20 amp. powerstat was then substituted and used in all subsequent preliminary runs. At 2 volts and 10 amps., the wire barely glowed. At 3.5 volts and 12.5 amps., it burned out.

Run No. 5. A second piece of copper wire (3-7/16" x .0126" dia.) was installed in the flask and checked for circuit continuity before charging OF_2 to the flask. In the OF_2 atmosphere, the wire barely glowed at 2 volts and 10 amps. Immediately upon increasing the voltage, the wire ignited at one point with considerable sparking. No increase in the wire glow intensity other than that at the ignition point was noted prior to ignition. The chamber clouded up as ignition began, and the wire was consumed back to the electrodes. The rotameter was clogged and the exit lines fouled with copper fluoride and/or oxide.

Run No. 6. A Monel wire (3-7/16" x .0100" dia.) was heated in a nitrogen atmosphere. A slight glow was observed at 5 volts which produced 1.9 amps. At 7 volts and 3.1 amps., the wire glowed with a moderate brightness and the current was shut off. The system was flushed with OF_2 and the wire retested in an OF_2 atmosphere. Again at 5 volts and 1.9 amps., a very faint glow was observed. Voltage was increased in one-volt increments and at 8 volts (3.5 amps.) a slight fogging was noted in the flask. While still at this voltage setting, the wire glowed brightly at one point, and the brightness traveled along the wire in both directions to the electrodes as though a surface film were burning off. The wire then continued to glow with the same reduced intensity noted before this brightness developed. (This strange phenomenon was explored more thoroughly in later work.) While the brightness persisted, the amperage fluctuated between 2.5 and 3.1 amps. and finally stabilized at 3.1 amps. when the wire color intensity again became normal. The voltage was increased to 9 and amperage rose to 3.5, at which point the flask began to get fogged. An increase to 10 volts showed no change in amperage. At 11 volts, a sudden loss of continuity was noted (amperage zero), but no wire break was observed. When the system was flushed out and the wire examined, it was found to be intact. After moving the wire about, continuity was re-established. It was not known if the current interruption was caused by the formation

of an insulating film of nickel or copper fluoride at the electrode connection or resulted from a poor electrical contact. Since it had been established that volatile materials were formed before ignition of this material, no further tests were made with this wire.

Run No. 7. A tungsten wire (3-7/16" x .0120" dia.) was checked in a nitrogen atmosphere. The wire glowed slightly when the current was 3 volts, at which point a reading of 5.0 amps. was obtained. Voltage was increased in one-volt increments until 7 volts (7.5 amps.) were reached, at which setting the wire was almost white hot. After cooling, the nitrogen was replaced with OF_2. In OF_2, a setting of 2 volts indicated 3.2 amps. As the voltage was being increased to 3 volts, the wire ignited with the light intensity of a flash bulb and was completely consumed. After the burning subsided, the resin flask walls showed a film deposit. Upon flushing the system with N_2, copious white fumes came out of the vent line.

Run No. 8. A S.S.302 wire (3-7/16" x .0200" dia.) was first tested in a nitrogen atmosphere. At 4 volts and 4.1 amps., a slight glow was seen. At 5 volts (4.9 amps.) the glow was brighter and the current was shut off. After cooling and flushing the system with OF_2, the wire was re-tested in an OF_2 atmosphere. At 4 volts (3.8 amps.) no glow was seen. At 4.5 volts (4.5 amps.) a yellow tint was forming on the flask and the amperage started to drop. At 5 volts (4.2 amps.)

the coating on the flask increased and a very faint glow appeared. At 6 volts, the amperage read 5.2, but slowly fell off to 4.8 at which point a bright spot appeared on the wire and the wire then slowly burned back to the electrodes. The ignition was accompanied by numerous burning particles which bounced around inside the flask. The flask was completely coated with a yellow-orange deposit and filled with a similarly colored smoke. The exit lines and rotameter were severely fouled with this same deposit.

Run No. 9. A molybdenum wire (3-7/16" x .0151" dia.) was heated in a nitrogen atmosphere. At 3 volts the current flow was 7.0 amps. and the wire glowed slightly. At 4 volts (8.7 amps.) the glow was brighter and the current was then shut off. After cooling and flushing the system with OF_2, the wire was re-heated in an OF_2 atmosphere. At 1 volt, 1 amp. was noted. As the voltage was being increased to 2 volts, the wire ignited with a very bright white light. Wire particles richoceted from the flask walls like tiny fireballs. The pressure generated by the ignition in the flask blew one of the copper electrodes out of its fitting. Some white residue was seen in the flask, together with white smoke in the vent exit after the ignition was completed. The wire was totally consumed during this pyrotechnic display.

5.1.3.1. Preliminary Measurements of Ignition Temperatures vs. Melting Points

A series of experiments were run with the previously described equipment to determine the burnout temperatures of the test materials in an inert atmosphere as calculated from the voltage-amperage data. These calculated temperatures were then compared to the melting point data for these materials which had been obtained from the literature. It was assumed that the two temperatures should be in fairly close agreement and generally they were. When possible the calculated temperatures were also checked by optical pyrometer.

The tungsten wire, however, gave very poor checks between the calculated temperatures and those observed with the optical pyrometer. As the wire temperature increased the difference between the pyrometer reading and the calculated temperature became greater. For example, at an observed pyrometer temperature of 1272°C, the calculations indicated a temperature in excess of 2000°C. The cause of this disparity was investigated. The purge gas was found to have no influence since the same large differences were noted when the wire was heated in a vacuum. It was finally concluded that the pyrometer reading was low because readings were taken through a fairly thick, non-optical glass flask. Some evidence to confirm this was obtained by changing the sighting position. When the position was shifted so that the light path through the glass was longer, the observed temperature decreased.

We made no attempt to determine if this was a result of light diffraction or diffusion. In addition, metal vaporization and vapor deposition on the flask walls tended to mask the light intensity and produced low readings. As a result of this work, it was concluded that a pyrometer was not sufficiently reliable for final temperature evaluations in subsequent runs.

Since the greatest error appeared with the tungsten wire, the accuracy of the resistivity data was checked. The resistance of the tungsten wire was measured from 24° to 700°C with a Wheatstone bridge. Excellent agreement with the published data (Ref. 1) was obtained up to 500°C. However, runs at higher temperature were less successful, since the wire oxidized despite attempts to shield it with helium. It was concluded that air in the system oxidized the wire. When the wire had been cooled to room temperature, the oxidation was shown as an increase in the wire resistance. This was again demonstrated by heating a tungsten wire in the resin flask setup to approximately 1250°C (observed with the pyrometer) while leaving the power setting unchanged. The initial reading of 6.8 volts and 7.2 amps. decreased over 40 minutes to 6.4 volts and 5.5 amps., indicating increased wire resistance. This change, the result of oxidation, produced a great increase in calculated temperature, although pyrometer readings remained virtually constant. As a result of this, the system was re-designed to prevent air contamination.

With the improved setup, which included a liquid seal in the vent system to maintain a very slight positive pressure of inert gas (nitrogen or helium) in the system, measurements were made on the other test materials. Despite our relatively crude instrumentation, calculated burnout temperatures for nickel, iron, copper, and Monel 400 were obtained which checked closely with their reported melting points. Other calculated burnout temperatures obtained in this series of runs are also included in Table 41, together with the nominal melting points for the materials.

5.1.4. Preliminary Study Conclusions

One of the additional purposes of this preliminary work was to establish the feasibility of an optical method of determining the ignition temperatures of wires in OF_2. On the basis of this work, an optical device such as a pyrometer or a photomultiplier tube did not appear to be suitable. Of the six materials tested, tungsten and molybdenum ignited in OF_2 before any glow was noted. Copper and S.S. 302 ignited when a very faint glow occurred which was below the limits of the optical devices. In addition, all four of these materials formed volatile matter before ignition which masked the intensity of the light emitted from the wire. Only the nickel and Monel wires glowed significantly before ignition. The Monel wire, however, also gave off volatile matter which coated the flask prior to ignition. Since the coating cut down light emission, optical measurement

would produce a low reading. Therefore, only the nickel wire could be considered to be within the range of an optical device, but it was still below the range of our pyrometer.

We therefore decided that we would use the temperatures obtained from our resistivity-temperature data. These resistivities were based on our recorded amperage-voltage information. This method has been used by Godwin and Lorenzo (Ref. 2) who conducted wire ignition studies in fluorine. However, unlike Godwin and Lorenzo who did their ignitions in a stainless steel bomb, we believed that conducting our tests in glass vessels was a significant improvement since we were able to visually observe the phenomenon of ignition. The resin flask showed slight etching and many pit marks where it was struck by glowing particles. However, we believed that this equipment would be used successfully.

Our preliminary work in glass with both coils and straight wires showed that a straight wire was to be preferred. The upper section of a tight coil always glowed first, indicative of a higher temperature than the lower part of the coil loops, but the straight wire appeared to be uniform in temperature.

This preliminary study also indicated that our resistivity-temperature data was generally satisfactory for the investigation. Better resistivity data was subsequently obtained for those materials which did

not show good temperature correlation. Sources of this data are included in our references.

5.2. Experimental Study

5.2.1. Apparatus and Equipment

The resin flask setup as shown in Figure 5 was used in the following series of tests with some modifications made to the accessory equipment. The vent outlet was connected to a vacuum system so that the entire setup, back to the OF_2 and helium gas cylinders, could be evacuated. The test gas was then introduced to the system until a pressure slightly above atmospheric was reached. At this point, while the test gas continued to flow into the system at a low rate, the vent line was opened. This prevented the entrance of air through the vent line. Each electrode was drilled and tapped at the bottom end to accommodate a 1/8" brass machine screw the end of which was rounded and polished to achieve good point contact. These screws secured the wire which passed through the hole in each electrode. These holes had been countersunk to prevent the wire from contacting the electrode at any point other than the screw contact. The wire length was then measured from these two contact points.

The equipment used in this work included a Kepco Inc., Power Supply Model KS 36-30M, with a maximum output rating of 36 volts, 30 amperes, and regulation of 0.01%. This power source was programmed to furnish power linearly from zero to maximum output at any one of

of six pre-determined rates. These rates ranged from 3.33 volts per minute to 0.0667 volts per minute. The amperage and voltage across the wire were continuously plotted with a Mosely X-Y recorder.

5.2.2. Test Materials and Cleaning Procedure

The test materials consisted of wires of nickel "A", Monel 400, molybdenum, tungsten, stainless steel 302, copper, and iron. The materials are further identified in Table 39. The cut wires were washed in acetone and rinsed with Genesolv "D" before each test. The air dried wire was then handled with tweezers to prevent surface contamination.

5.2.3. Experimental Procedure

Two series of experiments were performed. The first series served to determine wire burnout temperatures in a helium atmosphere; the second series was conducted to determine wire ignition temperatures in an atmosphere of oxygen difluoride. The same setup and equipment were used for both series of tests.

In all runs the cleaned wire was fastened into the electrodes, the system was then sealed and evacuated to less than 1 mm. of Hg, and the test gas was admitted to the system until a pressure slightly in excess of atmospheric was obtained. The vacuum line vent was then opened and a reduced gas flow was maintained throughout the run. The vacuum pump and the mercury manometer were isolated from the system

after the system was evacuated but before the gas was admitted. A compound gauge was used to monitor the gas pressure in the system during the filling operation.

The run was started when the voltage programmer was turned on. The voltage-amperage for the test wire was plotted by the recorder until ignition occurred and the resistivity was calculated from the amperage-voltage shown at the wire ignition. The ignition temperature was then taken from the resistivity-temperature curves for the particular material.

5.2.4. Experimental Data

5.2.4.1. Wire Ignition in a Helium Atmosphere

It was originally felt that wire ignition in a helium atmosphere would give an insight into what could be expected in an OF_2 atmosphere. It was also hoped that the plotted resistance curves from the helium burnout study could be used as a background reference for the OF_2 curves. The differences in the slopes of the curves obtained in the different atmospheres would perhaps give some indication of the effect of OF_2 corrosion. This series of runs was also made to demonstrate the effects of various lengths of test material, the effect of coil geometry, and the effect of different voltage increase rates. The data obtained from the ignitions in helium are shown in Table 42. Coils were formed on mandrels ranging from 1/8" to 1" diameter. Our visual observations indicated that

each size coil showed different heating characteristics. For a given length of wire, the 1/8" and 1/4" coils showed the greatest temperature difference between the top and the bottom of the coil since these coils were more tightly wound. The top of the loop always glowed long before the bottom. Radiant heating also caused the more closely spaced loops to glow first when unevenly spaced coils were used. Burnout always initiated at the top of the coil loops. Larger coils (1/2" and 1") showed a tendency to sag appreciably, bringing the center loops of the coil into close proximity and causing the formation of hot spots. In one case, the loops actually sagged until they touched and therefore shorted.

The calculated temperatures obtained in this series of tests were generally lower than the listed melting points for the several materials. This was largely because the burnouts were initiated at the local coil hot spots. The burnout temperatures calculated for Monel were quite consistent regardless of the wire length or coil geometry. However, all runs showed burnout temperatures approximately 400 to 500° below the nominal melting point of this alloy. Our analysis confirmed that the alloy was well within the specifications for Monel 400.* Resistance measurements for our wire made at room temperature also checked with the resistance reported in the literature. (Ref. 3)

* Actual analysis: 64.6% Ni, 33.1% Cu, 1.16% Fe

Samples of both an ignited and an untested wire were examined by International Nickel Co., Inc., Huntington Alloy Products Division, who also identified this material as Monel alloy 400 (Ref.4). Their analysis of the ignition-tested wire confirmed that the dendritic structure of this specimen proved it had melted. Chemically, this wire showed a higher silicon content than the unexposed sample. We believe this was caused by the hot wire contacting the Pyrex resin flask when it broke. International Nickel Co. also performed a melting point determination on the unexposed wire and reported it had a normal response to temperature. However, their communication did state that chemistry variation in the wire or generation of a contact potential could result in an actual temperature at our indicated resistivity of 68 microhm-cm. of possibly 250°C higher than indicated by the resistivity-temperature curve. They concluded, therefore, that the actual ignition temperature of the Monel wire was probably much higher than our measurements indicated.

Several additional runs were made in an attempt to explain this paradox. The ignition curves for the Monel in a helium atmosphere were all similar although runs were made using different voltage increase rates. All the curves showed a very large, almost instantaneous increase in amperage at a calculated temperature of approximately 900°C. This rapid amperage increase, with no measurable increase in voltage, terminated

in ignition. The curve for Run #80 shown in Figure 6 is typical of this phenomenon. In order to ascertain whether this peculiarity was related to helium, ignition runs were made in argon (Run #82) and in a vacuum (Run #83). As was the case in a helium atmosphere, the peculiar extremely rapid increase in amperage occurred virtually simultaneously with ignition, and initiation of this phenomenon occurred at approximately the same temperature as calculated for the helium runs. Two runs were made in helium (81 & 85A) in which the runs were terminated just prior to this point. In both runs, wire resistance measurements were made at room temperature both before and after the wires were heated, but no significant change in wire resistance was measured.

From these curves it would appear that this phenomenon signified a sudden phase change in the structure of the wire at this temperature. Such a phase change could cause a sudden variation in the wire resistance with a resultant significant error in our temperature data. The fact that the amperage increased rapidly indicated a sudden decrease in wire resistance which in turn is consistent with our temperature error on the low side. The data for these Monel runs have been included in Table 42.

5.2.4.2. Wire Ignition in an OF_2 Atmosphere

Based on the heating characteristic of the helium tests, the wire lengths chosen for the OF_2 series of runs were 3.4" and 5.75". The short length of wire was fastened tautly between the electrodes and then accurately measured with a vernier caliper. The exposed length of the longer wire was always 5.75" of wire between the centers of the two electrode contact points. This wire was in the form of a loosely wound spiral which had been shaped on a 1/8" mandrel. The spiral form avoided the formation of the hot spots that occurred with tight wire coils. This chosen length produced a spiral which showed a uniform temperature when heated. Longer lengths produced closer wound spirals or coils which showed the effect of radiant heating.

At least two runs were made at each wire length for each material. When good duplication was not achieved, additional runs were made. The results of these runs are shown in Table 43.

5.2.4.2.1. Nickel Ignition in OF_2

Nickel was the only material which was tested in two wire diameter sizes. Tests were run with two lengths of wire (3.4 and 5.75") for each diameter. The ignition temperatures for both wires were approximately 1200°C. The wire diameter did not seem to affect the ignition temperature when the shorter length was tested. However, the thinner wire showed ignition temperature

approximately 100° lower when the 5.75" length was tested. The data from the nickel runs are summarized in Table 44.

Temperatures were calculated for twelve runs which were run to completion. Other runs were made to determine the effects of corrosion. The resistances of the nickel wires were measured at 25°C using a Wheatstone bridge when the wires were first fastened between the electrodes. The wires were then heated to just below the ignition point. The current was then turned off and the test wire allowed to cool rapidly. The wire resistance was then again measured at 25°C. The effects of OF_2 corrosion resulted in a higher resistance for the second measurement. Run #88 was made for the purpose of evaluating the change in resistance. A wire 5.75" long x .0150" diameter showed an increase in resistance from 0.1563 Ohms to 0.1626 Ohms. The increase in resistance, due to corrosion,can be equated to a change in the effective conductive diameter of the wire. The corrected cross sectional area of the wire was then used to calculate the resistivity and a corrected temperature was obtained.

It can be seen from Table 43 that factors such as rate of power increase, wire diameter, and wire length all produce different run times until ignition is achieved. Since the exact time of exposure could be determined, proportionate corrections could be made for each set of conditions. The uncorrected as

well as corrosion-corrected ignition temperatures are shown in Table 43. This table indicates that the corrected temperatures as a group show a smaller deviation than do the uncorrected temperatures. The curves obtained from identical wires are virtually identical. This consistency enabled us to calculate the resistance change for one wire and apply to the other wires. Each material tested produced its own type of curve. An example of a curve produced by the ignition of a nickel wire in OF_2 is shown in Figure 7 (data from Run #69).

Several sources were consulted (Ref. 5,6,7) for resistivity-temperature data. Since all three references were in very close agreement, a composite curve was used to determine the ignition temperatures of these runs.

5.2.4.2.2. Monel Ignition in OF_2

Particular attention was given to the Monel wire because this material showed a rather peculiar behavior. It appeared to have two reaction points (Table 45). The initial reaction occurred at approximately 700°C. It was evidenced by a bright glow which initiated at one point and traveled the length of the wire in both directions before subsiding. This phenomenon had been noted earlier in our preliminary work. All Monel wires which were ignited in OF_2 produced a very unusual but consistent plot on the recorder chart as a result of this phenomenon. Figure 8 (Run #41) is a typical example. Apparently,

this primary reaction, shown in Figure 8, caused an increase in resistance as indicated by the reduced amperage. It is surmised that the reaction is a rapid but non-catastropic surface corrosion which in turn results in an apparent decrease in the conductive wire diameter. The initiating mechanism could not be definitely proven but our hypothesis is included in our conclusions, Section 5.2.6.

Several runs were made in an attempt to explain this phenomenon. The resistances of the Monel wires were measured when the wires were first fastened in the electrodes. The wires were then heated until the initial reaction point was just passed, at which point the voltage was shut off and the OF_2 flushed from the system. When ambient temperature was reached, the resistance was again measured. Based on the increase in resistance, a new apparent wire area was computed and a corrected temperature for this first reaction point was then calculated. It should be noted this break in the curve at approximately 700°C occurs only in the presence of OF_2. Wires heated in helium, argon, or vacuum showed no break at this point. It should also be noted that wires were heated to just past this reaction point and then brought back to room temperature. Upon being re-heated to final ignition the curve does not show another reaction at the initial reaction temperature (Fig. 9).

Referring to Figures 8 & 9, you will note the second reaction point or ignition point is not based on readings at the extreme upper limit of the curve. The ignition temperature as herein reported is assumed to be that point at which the wire resistance (diameter) was changing so rapidly as a result of corrosion that no increase in current resulted from a further increase in voltage. This, in effect, assumes that above this temperature corrosion becomes virtually catastropic. This phenomenon was noted with the metals having high ignition temperatures such as nickel and Monel.

The apparent ignition temperatures from Monel as calculated from our measurements and uncorrected for the effects of corrosion were extremely high. In fact, it was often calculated as being above the melting point.

Resistance measurements were made on fresh Monel wires using a Wheatstone bridge. The wires were then heated in OF_2 to just below the ignition point. The current was then shut off and the wires allowed to cool rapidly. The resistances of the wires were again measured at 25°C and compared to the resistances measured before their exposure to OF_2. A Monel wire, 5.75" long, showed an increase in resistance from 1.5022 to 1.9235 Ohms after exposure. The change in the resistance was caused by the corrosion of the metal in the OF_2 at high temperature. This in

turn also produced a decrease in the conductive diameter of the wire. The apparent conductive wire diameter and area could then be calculated from the resistance measurements. Using this new area, corrected temperatures for the wires at this point were determined. With this corrected area, the calculated temperatures were much lower. Run #79 (Figure 10) illustrates the calculated temperatures using the wire cross sectional areas as determined by the resistance measurements. In this particular run, the test was terminated just before ignition. Run #41 (Figure 8) was also a Monel wire 5.75" long but was run to ignition. The two curves match quite closely. Since this is true it can be assumed that the corrosion rate, resistance, and diameter changes measured for Run #79 are equally applicable to Run #41. Calculations for the 5.75" length of Monel wire used in Runs #40 and 41 now indicate ignition temperatures of 895 and 890°C respectively, whereas the uncorrected temperatures were considerably above the melting point for this material. International Nickel's resistivity-temperature data was used in this study (Ref. 3).

A further attempt was made to determine the mechanism that produced the initial but non-catastropic reaction between the Monel and OF_2. Additional runs were made that were terminated at various points along the curves. Samples of these wires were sent out for metallographic examination. Photomicrographs

of cross-sections of eight specimens of Monel wire, prepared by W. B. Coleman Co., Philadelphia, failed to reveal any explanation for the corrosion mechanism or the initiating factor for this observed reaction in OF_2. The micrographs clearly revealed the grain sizes and crystal growths, all of which were in line with what could be expected at the calculated specimen temperature.

We also obtained another sample of Monel 400 wire with a different "heat" number from a second supplier (Newark Wire Cloth Co.) together with a certificate of analysis. We made sufficient runs with the new wire to establish that the phenomenon displayed by the original wire is characteristic of Monel 400 and not merely an isolated occurrence.

5.2.4.2.3. Stainless Steel 302 Ignition in OF_2

Six runs were made with S.S. 302 wire in an OF_2 atmosphere using two different lengths of wire. The corrected average ignition temperature for wires 5.75" long was approximately 1000°C. The shorter wires (3.4") had ignition temperatures of approximately 900°C. The curves obtained from the six runs showed a very sharp break at the ignition point indicating sudden and complete ignition. The slopes of the curves showed only a nominal change as the ignition point was approached which, unlike the Monel curves, is interpreted as indicative of relatively slight corrosion. The duplicate wires produced virtually identical curves.

Resistance measurements were made (Run #75) on a wire both before and after exposure to OF_2 to slightly below the ignition point. The resistance increased from 0.592 Ohms to 0.610 Ohms after exposure. The resistances were then used to calculate a new wire area. This reduced wire size was then used to recalculate the ignition temperatures. The uncorrected or apparent ignition temperatures together with the corrected readings are shown in Table 43. As a further check on the effect of corrosion, one wire (Run #74) was run at a voltage increase rate of 1.67 volts/min., whereas the other S.S. 302 wires were run at the usual rate of 3.33 volts/min. This wire was therefore exposed to OF_2 for twice as long and the apparent ignition temperature for this run as a result of the lengthy exposure was very high. When the proper corrosion correction was applied to the data from this run, the corrected temperature fell into line. This factor was also applied to Run #75 which having been heated in OF_2 twice, likewise initially showed an erroneously high ignition temperature. Again, the corrected temperature matched that for the other wires of identical length. The curve from this run (#75) has been shown as Figure 11. This figure shows the initial track plotted when the wire was used for resistance measurements (#75A) as well as the subsequent run to ignition (#75B). The resistivity-temperature curve used for the S.S. 302 was based on data published by International Nickel (Ref. 8).

5.2.4.2.4. Copper Ignition in OF_2

Five runs were made with copper wire in an OF_2 atmosphere. Duplicate runs were made using a wire length of 3.43". The remaining three runs involved wires 5.75" long. The two short wires ignited at 620°C while the long wires ignited at 700°C. Because of the extremely low resistance of copper, a small voltage produced a high amperage. As a result, ignition occurred in 13 seconds for the 3.43" wire and 23 seconds for the 5.75" wires. Since the exposure times were extremely brief it was felt that corrosion measurements would be impractical. No corrections were therefore made for corrosion, although it obviously had some effect as shown by the higher apparent ignition temperature for the long wires which were subjected to greater corrosion owing to their longer exposure. This increased corrosion would have reduced the conductive area of the wire. Our failure to correct for this resulted in the higher ignition temperature. The curve developed for Run #46, which is typical of all the copper ignition curves, is shown in Figure 12. The data from these ignition runs with copper wire are included in Table 43. The resistivity-temperature data used in this study were taken from Reference 5.

5.2.4.2.5. Iron Ignition in OF_2

Four runs were made using high purity iron wire in an OF_2 atmosphere. Wire lengths of 3.2" and 5.75" were run to ignition at temperatures of 623 and 665°C, respectively. Run #60 produced a typical OF_2 ignition curve for this material and is shown in Figure 13. It is apparent that the curve is quite smooth until a power input of 2 volts was reached. At this point, the curve trace became slightly irregular, possibly indicating corrosion. Both the helium and OF_2 ignition curves showed a somewhat similar change in slope between 400 and 500°C. However, the helium ignition curve remained quite smooth up to the ignition point, in sharp contrast to the erratic tracing of the OF_2 curve. This erratic pattern in OF_2 is apparent only during the final 30 seconds of exposure, indicating that little corrosion occurred prior to this time. Although no corrections for corrosion were made for this material, the curves suggested that the corrected ignition temperature would be between 500-600°C. The uncorrected ignition temperatures, however, are sufficiently low to preclude consideration of pure iron as a compatible material for OF_2 service.

It should be noted that with iron wire, as with the uncorrected ignition temperatures for the other materials, the longer wire was found to have a higher ignition temperature. Again, this was a result of the extended exposure which consequently created a greater error in the wire area. The data for these iron runs were also included in Table 43.

The resistivity-temperature data for iron were obtained from three sources (Ref. 5, 6, 7). Since the data were in very close agreement, our data curve was a composite of the data of all three references.

5.2.4.2.6. Molybdenum Ignition in OF_2

Four runs were made with molybdenum wires in an OF_2 atmosphere using wire lengths of 3.41 and 5.75". As with the copper wires, ignition occurred after a very brief exposure. The short wires ignited in 10 seconds at a calculated temperature of 290°C. The long wires ignited in 16 seconds at a calculated temperature of 320°C. Since the wires ignited rapidly and the calculated temperatures were so low, no corrections were made for the effect of corrosion. All four ignition curves for molybdenum were quite similar. The curve for Run #53, which was typical, is shown in Figure 14. The smoothness of this curve as compared to that for iron (Figure 13) indicated that corrosion was not significant until the last few seconds preceding ignition. The ignition temperatures were obtained from the resistivity-temperature data of Agte and Vacek (Ref. 1). The data obtained from this series of runs were included in Table 43.

5.2.4.2.7. Tungsten Ignition in OF_2

Four specimens of tungsten wires were ignited in an OF_2 atmosphere. Wires of 3.41" and 5.75" were tested in duplicate. All four wires showed approximately the same ignition temperatures which ranged from 255°C

to 280°C. As was observed with the copper and molybdenum wires, the tungsten wires also ignited after a very brief exposure ranging from 12 to 18 seconds. The rapid ignition made it impractical to evaluate corrosion effects, and the reported temperatures were not corrected for corrosion. Again, all curves were quite similar and a sample curve from Run #58 is shown in Figure 15. The data for these four runs have likewise been included in Table 43. The reported ignition temperatures were obtained from the resistivity-temperature data of Agte and Vacek (Ref. 1).

5.2.5. Calculations

Basically, two equations were used for the calculations involved in this ignition study. The resistivities were calculated from the following equation, using the voltages and amperages obtained from the plotted ignition curves:

$$\text{Resistivity} = \frac{\text{Volts} \times A}{\text{Amps} \times L}$$

Resistivity is expressed in microhms-cm.

A = the cross sectional area of the test wire in cm^2

L = the wire length in centimeters

The temperature was then obtained from the resistivity-temperature curve for the particular material.

The wire resistances were measured for the wires both before and after exposure to OF_2. The change in resistance was used to calculate a new wire area corrected for corrosion as follows:

$$\frac{R_1 \times A_1}{R_2} = A_2$$

5.2.6. Conclusions

The average ignition temperatures as shown in Table 46 generally conformed to the accepted order of material compatibility with OF_2. The one significant exception appeared to be the stainless steel 302 which, in this listing, was equivalent or perhaps slightly superior to Monel. However, we believe that the very short exposure periods used in this investigation mitigated the effects of corrosion. Our own experience has demonstrated that Monel is far more resistant than S.S. 302 to both OF_2 and F_2 at elevated temperatures contrary to these ignition results. This paradox was also noted by Godwin and Lorenzo (Ref. 2) who reported that Monel ignited in fluorine at 400°C compared to S.S. 302 ignition at almost 700°C.

Our own studies in fluorine (Ref. 9) indicated that the corrosion rates with 18-8 stainless steels tended to accelerate as exposure time increased. Monel on the other hand became passivated and the corrosion rate slightly decreased with longer exposure. It would therefore appear that these wire ignition temperatures cannot be used as an absolute indication of the relative compatibilities of the several materials.

The techniques developed in this investigation have provided a new look at the phenomenon of ignition. For the first time, the actual ignition has been both visually observed and constantly monitored by instruments. It is felt that these techniques could be adapted to corrosion studies by the comparison of material resistances both before and after exposure.

The Monel wires were explored more thoroughly than the other materials because of two unusual occurrences: The low ignition temperature in helium, and the two reaction points in OF_2. The helium ignition curve is perhaps indicative of a phase change if we accept the resistivity-temperature data (Ref. 3) as being correct. Verification of this data was beyond the scope of this program. The two reaction points in OF_2 had likewise been well studied. It was noted that the temperature at which the first reaction occurred was very close to the ignition temperature of copper. Copper is also a major constituent of Monel (33%). We therefore suspect that this first reaction is the ignition of the exposed surface copper, and the reaction is subsequently quenched because of the presence of the more corrosion-resistant nickel. It is possible that the quenching mechanism was the formation of a passivating fluoride film on the nickel particles which retarded further attack, or the nickel may have served merely as a heat sink to prevent the initial copper ignition from becoming catastrophic. However, we were unable to ascertain if either hypothesis was correct.

It should be noted that, despite our attempts to find the best resistivity-temperature data, these curves may not fit our materials exactly. Chemical composition may vary by several percent within the specifications of a particular alloy. The material, although meeting specifications, could conceivably have a range of resistances within these specification limits. The

degree of accuracy to which these curves can be applied to a particular sample was not known. It is suspected that this may be an inherent, albeit minor, source of error in our work.

Our work has considered the effects of corrosion for three of the materials tested. In determining corrosion, resistance measurements were taken after exposure to OF_2 but prior to burnout. This resistance measurement by necessity was taken on a wire which had approached but had not reached ignition. Hence, the corrosion for the last few seconds of exposure, which undoubtedly was significant, could not be measured. All corrections therefore must be considered to be conservative. The actual ignition temperatures for all the tested materials may therefore be considered to be actually slightly lower than indicated in our results.

6. OXYGEN DIFLUORIDE DETECTION

Oxygen difluoride, in addition to being a highly energetic propellant, is quite toxic. It is therefore desirable that a suitable detection device be available to monitor locations where OF_2 is used and adjacent areas to maintain the atmospheric concentration of OF_2 within acceptable limits. A task of this program was to attempt to modify an existing fluorine detector so that it could be utilized for OF_2 service.

6.1. OF_2 Detection Equipment

The instrument which we had used in this investigation was a "Fluorine Monitor", manufactured by Tracerlab Division, Laboratory for Electronics, Inc., Waltham, Mass. This instrument was designed to detect small concentrations of fluorine gas in air. The sensing element is a krypton-85 quinol clathrate which releases the radioactive gas Kr-85 in proportion to the amount of fluorine present. The quinol is oxidized by fluorine to quinone and the cage-like structure of the clathrate is destroyed releasing the krypton-85. The released gas is then swept along in the air stream to a counter. The counting rate is proportional to the fluorine input, and the instrument can therefore be calibrated in terms of fluorine concentration.

Although the instrument is quite sensitive to fluorine, OF_2 apparently does not have sufficient oxidizing power to quantitatively destroy the clathrate cage and therefore does not produce an equivalent release of

krypton. The detector as initially designed is therefore totally inadequate for service as an OF_2 detector.

We had proposed to modify the instrument by interjecting a pyrolyzer in the gas stream before it entered the detector. Ths purpose of this heater was to thermally decompose the OF_2 to fluorine and oxygen according to the equation

$$2OF_2 \longrightarrow 2F_2 + O_2$$

The fluorine thus produced should be capable of reacting with the clathrate and could then be used as a measure of the OF_2 concentration in the test gas. The initial pyrolyzer designed for this purpose consisted of a 1" diameter Monel pipe packed with sodium fluoride pellets. A thermocouple well was installed through the central axis of the pipe so that temperature measurements could be made at any point of the heater. The pipe was mounted in a Hoskins electric furnace. As an added precaution the outside pyrolyzer wall temperature was monitored with a second thermocouple to maintain a minimal temperature drop across the heater packing.

The Tracerlab detector was reported to be humidity sensitive and was equipped with an adjustment dial which was used to compensate for the relative humidity at time of use. Our initial setup therefore included a chamber which was used to humidify our nitrogen feed in order to simulate atmospheric conditions. This initial test setup has been shown in Figure 16.

6.1.1. Instrument Calibration

Our initial work was to calibrate the instrument for fluorine and to establish its sensitivity limits. The setup (Figure 16) used a mixture of 0.6% F_2 in N_2 as a feed gas for calibration purposes in place of the OF_2-N_2 mixture shown in the schematic drawing. The F_2-N_2 mixture was carefully metered through a flowmeter and further diluted with nitrogen fed through a large capacity flowmeter. The feed method was extremely accurate and the fluorine concentration could be controlled within 1 ppm. The equipment could be used to feed either dry or humidified nitrogen by manipulating the appropriate valves. The humidity chamber was equipped with both a wet and dry bulb thermometer to determine the relative humidity of the diluent nitrogen.

The instrument was calibrated using the nitrogen diluted fluorine. The two highest sensitivity scales of the detector were calibrated over the ranges of 0-0.5 and 0.5-10 ppm fluorine respectively using dry gases. We were unable to obtain good calibration data using a humidified test gas. We suspected that some of the fluorine and moisture reacted to produce HF since the same fluorine concentration in dry diluent nitrogen produced a stronger signal from the detector. We verified the formation of HF analytically. A sample of the humidified feed mix was introduced into a 10 cm IR cell equipped with calcium fluoride windows. The cell contents were evaluated with a Carey recording

spectrophotometer and HF was positively identified as being present but the concentration could not be determined. The feed gas initially contained 8 ppm of fluorine. The HF was therefore much less than this concentration. A sample of the dry gas mixture containing 8 ppm of fluorine showed no HF by the same analytical technique. Calibration curves supplied by Tracerlab were based on humidified gas mixtures and therefore contained HF which had no effect on the clathrate. As a result we had established that the detector had a greater sensitivity to fluorine than was claimed by the manufacturer.

6.1.2. Analytical Procedure for Fluorine

The fluorine content of the test gas mixture was determined analytically. The gas was fed through a calibrated flowmeter for an accurately timed period and scrubbed through a solution of potassium iodide. The fluorine quantitatively oxidized the iodide to iodine as follows:

$$F_2 + 2KI \longrightarrow 2KF + I_2$$

The iodine was then titrated with 0.01N sodium thiosulfate using Thyodene as an indicator. The fluorine could then be calculated as ppm in the feed gas.

6.2. Experimental Procedure

Since F_2-N_2 mixtures in ppm concentrations gave reproducible results with the detector we next established that the passage of feed gases through the furnace posed no problem. The furnace was removed from the setup and completely passivated at elevated temperatures using straight fluorine. After flushing out any absorbed F_2, ppm quantities were passed through the heated furnace and the gas exits were chemically analyzed. No loss of fluorine was noted when the furnace temperature was below 350°C. Losses increased with higher temperatures. We therefore selected 300°C as the pyrolyzer temperature for the OF_2 decomposition to mitigate the corrosion losses.

As previously stated, it was our intention to thermally decompose the OF_2 to fluorine and oxygen and use the detector's response to the fluorine to measure the OF_2. In principle this appeared to be a simple and accurate method of utilizing the instrument as an OF_2 detector. However, in practice it proved to be far from simple. The primary problem appeared to be that at the temperatures at which quantitative decomposition of OF_2 should occur, corrosion was also encountered. The second problem was the difficulty in analytically determining the composition of the pyrolyzer exit gases which could contain both OF_2 and F_2. The reactions of these oxidizers in KI solutions

are not identical:

$$OF_2 + 4KI + H_2O \longrightarrow 2KF + 2KOH + 2I_2$$
$$F_2 + 2KI \longrightarrow 2KF + I_2$$

In addition to the problem of the OF_2 and F_2 reacting differently in KI, corrosion losses were unknown and wet analyses of the exit gases were impractical. In the ppm concentrations desired for the detector range no other analytical methods proved suitable.

We were therefore faced with monitoring our pyrolyzer gas exits with the detector. The initial setup (Fig. 16) was modified slightly since the humidifier had been found to be undesirable. However, this setup produced a negligible response on the detector when the feed was directed through the pyrolyzer. It was then learned that the detector pump is pre-set at a constant rate. The pump which pulls the gases across the clathrate would also be required to pull the gas through the pyrolyzer at a constant rate. However, the pump was built with a bypass. As soon as back pressure was sensed on the inlet line because of the packed pyrolyzer, the pump compensated by obtaining an increased flow through the bypass line. Therefore, little if any pyrolyzer exit gas ever reached the counting chamber in the detector. We proved this by inserting a flowmeter between the pyrolyzer exit and the detector. Detaching the heater produced a normal flow to the instrument. In place, the flow became negligible.

The next change was to locate the pyrolyzer before the mixing chamber. Again, problems arose and the results were completely unsatisfactory. The diluent gas passed through the pyrolyzer at rates up to 6 liters/minute which decreased the OF_2 residence time in the heater to where efficient decomposition did not occur. In addition, N_2 feed rate changes caused variations in the pyrolyzer temperature. The detector showed a very slow response to changes in OF_2 feed rates and no reproducibility of results could be obtained.

A third arrangement of the components as shown in Figure 17 was hoped to be more satisfactory. The OF_2/N_2 mixture was fed to the pyrolyzer padded with additional N_2 so that a constant gas rate into the pyrolyzer was maintained regardless of the OF_2 concentration. Thus, we had eliminated gas residence time variations and temperature shifts in the pyrolyzer. The bulk of the N_2 diluent was added downstream of the pyrolyzer. To prevent large flow rates of diluent causing back pressure at the pyrolyzer, these lines were changed from 1/4 to 3/8" copper tubing. Despite an intensive investigation we were never able to obtain detector signals that were proportional to the OF_2 feed concentration or even achieve reproducibility of data when duplicate runs were made.

Several fresh approaches were taken in an effort to obtain some significant data. One series of investigations consisted of the systematic variation of the OF_2 feed rates to cover the entire range of the detector's scales while maintaining a constant pyrolyzer temperature. This was repeated several times using different pyrolyzer temperature settings. It was hoped that this would provide a family of curves from whence could be derived optimum pyrolyzer temperatures. This method failed to give anything near a quantitative response to the OF_2 feed. In fact, significant signal responses were not noticed until feed rates approached 1000 ppm, an intolerably high concentration. We were unable to determine if this was due to poor OF_2 decomposition or a result of the liberated F_2 reacting with the heater wall.

A final program was started to see if the pyrolyzer design could be changed to improve the instrument response. The Monel pyrolyzer tube was evaluated without packing. A lengthy coil of Monel tubing to provide increased gas contact time was also tested. Despite intensive passivation before being placed in service, neither unit produced any improvement. A third pyrolyzer tested was a lengthy coil of aluminum tubing. It was completely passivated but it likewise failed to provide acceptable data.

We realized that our setup involved an OF_2 feed in the pyrolyzer that was more concentrated than the feed gas to the detector. A last revision of the setup

placed the furnace after the mix chamber with a flowmeter installed between the furnace and the detector. By controlling the exit from the mix chamber a flow was maintained through the furnace that was just sufficient to allow the pump to pull its normal feed rate as verified by the flowmeter. Thus, the back pressure problem was overcome. However, again results were unfavorable.

It was reasoned that despite the inability of cold OF_2 to react with the clathrate in the detector, warm OF_2 might produce some quantitative results. The feed line directly before the instrument was therefore heated while the ppm feed of OF_2 was introduced. This change was likewise unproductive.

6.3. Conclusions

Our investigations have indicated that the Tracerlab Fluorine Detector could not be readily modified for use in OF_2 service. This investigation was based on a theory that OF_2 could be thermally decomposed and the by-product fluorine could then be detected by the instrument. All of our attempts to produce this effect quantitatively or reproducibly were unsuccessful. We were unable to achieve quantitative decomposition without significant corrosion.

This approach was taken despite some inherent disadvantages. The pyrolyzer would require a power supply limiting the portability of the instrument. Secondly, and perhaps more significantly, in practice OF_2 would react with moisture in the air at the pyrolyzer temperature

to form HF which does not elicit a response from the instrument. We had considered the investigation of drying agents to remove atmospheric moisture before the pyrolyzer. However, in view of the poor pyrolyzer performance, this problem remained academic.

The instrument despite its known sensitivity to fluorine showed several serious drawbacks. It should be noted that our device was an early production model. Tracerlab had reported that some of its deficiencies had been eliminated in later models. The shortcomings of our instrument are listed below:

1. Clathrate service life was rather short and required factory replacement.

2. Instrument was battery-powered. In continuous service the battery required recharging after three or four hours.

3. Battery charger must be connected and disconnected manually and the battery cannot be charged overnight. A timer could not be used in series with the charger.

4. Clathrate unit was secured with a Teflon fitting which also served as a gas feed inlet. When this Teflon support broke from fatigue the glass clathrate container broke. The clathrate then contaminated the entire unit. A complete decontamination was required before the instrument could be repaired.

5. The device required frequent rezeroing as the battery charge changed.

6. No provision was provided to run the unit by standard electric current.

There were other shortcomings to this instrument which were only realized through the several months of usage. It is therefore questionable whether the detector in its present form could be considered a suitable device even if our modification efforts had been fruitful. Since the completion of our investigation we have been advised that a similar detector has been developed by Panametrics, Waltham, Mass. This detector uses a sensitized clathrate which can detect OF_2 directly. If further OF_2 detection studies are being considered, the evaluation of this instrument should be included.

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TABLE 1

OF_2 DYNAMIC TESTS
METAL TEST MATERIALS

MATERIAL	SUPPLIER	ANALYSIS	% CHEMICAL COMPOSITION
Monel 400	International Nickel Co.	Actual	66.05Ni, 31.50Cu, .14C, 1.11Fe, 1.00Mn, .17Si, .007S.
Nickel 200	Whitehead Metals Inc.	Actual	99.44Ni, .01Cu, .05Fe, .26Mn, .13Si, .005S.
Stainless Steel 301	Peter A. Frasse & Co., Inc.	Actual	.092C, 18.23Cr, 1.19Mn, 7.30Ni, .54Si, .016P, .012S, Bal. Fe.
Stainless Steel 304	U.S. Steel Co., Inc.	Actual	.05C, 18.36Cr, 1.31Mn, 8.95Ni, .44Si, .023P, .024S, Bal. Fe.
Inconel 600	International Nickel Co.	Actual	.05C, 15.59Cr, .04Cu, 6.91Fe, .16Mn, 76.97Ni, .25Si, .007S.
Aluminum 6061-T6	Whitehead Metal Inc.	Nominal	0.15-0.35Cr, 0.15-0.4Cu, 0.7 Max Fe, 0.8-1.2Mg, 0.4-0.8Si, 0.15 Max Ti, 0.2 Max Zn; Others .05 Max each, .15 total; Bal. Al.
Aluminum 2024-T3	Aluminum Co. of America	Actual	4.15Cu, .28Fe, 1.45Mg, .60Mn, .13Si, .01Ti, .08Zn; .00 each Ni, Pb, Sn, Bi; Bal. Al.
Copper-Chromium 182	Anaconda American Brass Co.	Actual	.68Cr, 99.21Cu, .07Fe, .01Si.
Silver Solder Rod #1801	Eutectic Welding Alloys Corp.	Actual	3.Cu, 0.3Sn, 2.Zn, 2.Cd, Bal. Ag.
Brazing Rod, Oxweld #25	Linde Corporation	Nominal	88Cu, 2Sn, 10Zn.
Monel Filler Metal 40	International Nickel		
Titanium A-110AT	Crucible Steel Co. of America	Actual	.08C, .02N, .17Fe, 4.7Al, 2.5 Sn, .0166 H_2

TABLE 2

LIQUID OF_2 DYNAMIC TESTS

SUMMARY

Material	No. Runs	Lbs. OF_2*	Time Sec.
Monel	56	116.35	4597.7
Nickel	63	130.73	5193.6
S. S. 304	50	107.16	4407.3
Al 2024	66	141.36	6256.5
Al 6061	40	91.34	4115.0
Titanium	42	96.05	4490.2
S. S. 301	46	93.45	4021.6
Inconel	47	97.41	3953.9
Brazed Monel	51	109.99	4547.2
Welded Monel	47	101.13	4203.1
Silver Soldered Monel	46	92.22	3730.6
Copper Alloy	56	112.72	4467.2
TOTAL	610	1,172.91	53,983.9
			149.96 hrs.

*Total lbs. OF_2 through orifice.

TABLE 3

LIQUID OF_2 DYNAMIC TESTS
MONEL ORIFICE

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
120	2.41	231.0	.0104	93.4	
"	1.98	171.7	.0115	103.3	
"	2.45	203.0	.0121	108.7	
"	2.01	167.0	.0120	107.8	
Totals:	8.85	772.7			
Avg:	2.21	193.2	.0115	103.3	14,985
300	1.98	106	.0187	167	
"	1.98	107	.0185	166	
"	1.98	100	.0198	177	
"	1.98	100	.0198	177	
"	1.98	112	.0177	158	
Totals:	9.90	525			
Avg:	1.98	105	.0189	169	24,515
500	1.98	81	.0245	218	
"	1.98	87	.0228	203	
"	1.98	90	.0220	197	
"	1.98	95	.0208	187	
"	1.98	90	.0220	197	
"	1.98	97	.0204	183	
"	1.98	89	.0223	200	
Totals:	13.86	629			
Avg:	1.98	89.9	.0221	198	28,722
700	1.98	73	.0272	242	
"	1.98	78	.0254	227	
"	1.98	61	.0325	290	
"	1.98	88	.0225	202	
"	1.98	73	.0272	243	
"	1.98	74	.0268	240	
"	1.98	77	.0257	230	
"	1.98	71	.0280	250	
"	1.98	65	.0305	272	
Totals:	17.82	660			
Avg:	1.98	73.3	.0273	244	35,395
900	1.98	63.0	.0315	282	
"	1.98	58.0	.0342	306	
"	1.98	76.9	.0257	229	
"	1.98	59.8	.0332	296	
"	1.98	62.1	.0318	285	
"	1.98	64.8	.0308	276	

TABLE 3 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
900	1.98	65.4	.0303	272	
"	1.98	58.4	.0339	303	
"	2.17	73.8	.0294	264	
"	2.17	72.5	.0299	269	
"	2.17	72.8	.0298	268	
"	2.17	69.7	.0311	279	
"	2.17	72.6	.0299	269	
Totals:	26.69	869.8			
Avg:	2.05	66.9	.0306	276	40,037
1200	2.17	62.5	.0347	312	
"	2.18	66.0	.0330	297	
"	2.18	69.7	.0313	281	
"	2.18	70.6	.0309	278	
"	2.18	71.0	.0307	276	
"	2.18	66.8	.0326	293	
"	2.18	67.7	.0322	289	
"	2.18	66.9	.0326	293	
Totals:	17.43	541.2			
Avg:	2.18	67.7	.0322	289	41,922
1500	2.18	57.9	.0377	339	
"	2.18	59.5	.0366	329	
"	2.18	60.0	.0363	326	
"	2.18	59.4	.0367	330	
"	2.18	65.5*	-----	---	
"	2.18	59.0	.0370	332	
"	2.18	59.8	.0365	328	
"	2.18	60.0	.0363	326	
"	2.18	59.7	.0365	328	
"	2.18	59.2	.0368	331	
Totals:	21.80	600.0			
Avg:	2.18	59.39	.0367	330	47,870

*Partial plug, not included in average.

TABLE 4

LIQUID OF_2 DYNAMIC TESTS

NICKEL ORIFICE

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
120	1.98	166	.0119	106.9	
"	1.98	183	.0108	97.0	
"	2.01	181	.0111	99.7	
"	2.01	138	.0146	131.0	
Totals:	7.98	668			
Avg:	1.995	167	.0121	108.7	15,768
300	1.98	110	.0180	160	
"	1.98	95	.0208	187	
"	1.98	111	.0178	159	
"	1.98	106	.0187	167	
"	1.98	110	.0180	160	
Totals:	9.90	532			
Avg:	1.98	106	.0187	168	24,370
500	1.98	76	.0260	233	
"	1.98	83	.0238	213	
"	1.98	79	.0251	224	
"	1.98	119*	-----	---	
"	1.98	76	.0260	232	
"	1.98	78	.0254	227	
"	1.98	84	.0237	212	
Totals:	13.86	595			
Avg:	1.98	79	.0251	225	32,639
700	1.98	77	.0257	228	
"	1.98	100*	-----	---	
"	1.98	63	.0315	281	
"	1.98	65	.0305	272	
"	1.98	71	.0280	250	
"	1.98	69	.0288	257	
"	1.98	70	.0283	253	
"	1.98	72	.0275	245	
"	1.98	70	.0283	253	
Totals:	17.82	657			
Avg:	1.98	70.0	.0283	254	36,845
900	1.98	58.0	.0342	305	
"	1.98	57.1	.0347	310	
"	1.98	53.9	.0368	329	
"	1.98	55.5	.0357	319	
"	1.98	55.6	.0356	318	
"	1.98	67.3	.0295	263	

TABLE 4 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
900	1.98*	109.0*	.0182*	163*	
"	1.98*	101.6*	.0195*	174*	
"	2.17	76.0	.0286	257	
"	2.17	71.6	.0303	272	
"	2.17	71.8	.0302	271	
"	2.17	72.1	.0301	270	
"	2.17	71.9	.0302	271	
Totals:	26.69	921.4			
Avg:	2.07	64.6	.0320	287	41,632
1200	2.17*	83.7*	-----	---	
"	2.17*	93.7*	-----	---	
"	2.18*	144.8*	-----	---	
"	2.18	72.6	.0300	270	
"	2.18	74.2	.0294	264	
"	2.18*	126.6*	-----	---	
"	2.18	72.0	.0303	272	
"	2.18	66.0	.0330	297	
"	2.18	67.1	.0325	292	
"	2.18	72.7	.0300	270	
"	2.18	66.6	.0327	294	
"	2.18	65.2	.0334	300	
"	2.18	65.7	.0332	298	
Totals:	28.32	1070.9			
Avg:	2.18	69.1	.0316	284	41,197
1500	2.18	58.6	.0372	334	
"	2.18	58.6	.0372	334	
"	2.18	58.4	.0373	335	
"	2.18	59.2	.0368	331	
"	2.18	58.6	.0372	334	
"	2.18	58.0	.0376	338	
"	2.18	58.4	.0373	335	
"	2.18	59.4	.0367	330	
"	2.18	59.6	.0366	329	
"	2.18	67.7*	-----	---	
"	2.18	82.4*	-----	---	
"	2.18	70.4*	-----	---	
Totals:	26.16	749.3			
Avg:	2.18	58.76	.0371	333	48,305

*Not included in averages. Partial plug suspected.

TABLE 5

LIQUID OF_2 DYNAMIC TESTS
MONEL SUMMARY

	TOTAL			AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	4	8.85	772.7	4	2.21	193.2	.0115	103.3	14,985
300	5	9.90	525.0	5	1.98	105.	.0189	169.0	24,515
500	7	13.86	629.0	7	1.98	89.9	.0221	198	28,722
700	9	17.82	660.0	9	1.98	73.3	.0273	244	35,395
900	13	26.69	869.8	13	2.05	66.9	.0306	276	40,037
1200	8	17.43	541.2	8	2.18	67.7	.0322	289	41,922
1500	10	21.80	600.0	9	2.18	59.4	.0367	330	47,870
TOTAL	56	116.35	4597.7						

TABLE 6

LIQUID OF_2 DYNAMIC TESTS
NICKEL SUMMARY

	TOTAL			AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	4	7.98	668	4	1.995	167	.0121	108.7	15,768
300	5	9.90	532	5	1.98	106	.0187	168	24,370
500	7	13.86	595	6	1.98	79	.0251	225	32,639
700	9	17.82	657	8	1.98	70	.0283	254	36,845
900	13	26.69	921.4	11	2.07	64.6	.0320	287	41,632
1200	13	28.32	1070.9	9	2.18	69.1	.0316	284	41,197
1500	12	26.16	749.3	9	2.18	58.8	.0371	333	48,305
TOTAL	63	130.73	5193.6						

TABLE 7

LIQUID OF_2 DYNAMIC TESTS
S.S. 304 ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	2.60	260.0	.0100	89.8	
	"	2.16	205.3	.0105	94.3	
	"	2.16	195.2	.0111	99.7	
	"	2.16	203.1	.0106	95.2	
Totals:		9.08	863.6			
Avg:		2.27	215.9	.0106	94.8	13,752
	300	2.16	127.9	.0169	152	
	"	2.16	138.5	.0156	140	
	"	2.16	135.6	.0159	143	
	"	2.16	147.8	.0146	131	
	"	2.16	124.5	.0174	156	
Totals:		10.80	674.3			
Avg:		2.16	134.9	.0161	144	20,889
	600	2.16	89.0	.0243	218	
	"	2.16	89.7	.0241	217	
	"	2.16	96.0	.0225	202	
	"	2.16	90.4	.0239	215	
	"	2.16	92.2	.0234	210	
	"	2.16	88.4	.0244	219	
	"	2.16	89.5	.0241	217	
Totals:		15.12	635.2			
Avg:		2.16	90.7	.0238	214	31,043
	900	2.16	73.6	.0293	263	
	"	2.16	74.3	.0291	261	
	"	2.16	78.3	.0276	248	
	"	2.16	74.1	.0291	261	
	"	2.16	73.9	.0292	262	
	"	2.16	77.9	.0277	249	
	"	2.16	75.7	.0285	256	
	"	2.16	73.1	.0295	265	
	"	2.16	95.9*	.0225*	202*	
	"	2.16	74.9	.0288	259	
Totals:		21.60	771.7			
Avg:		2.16	75.1	.0288	258	37,425
	1200	2.16	64.1	.0337	303	
	"	2.16	64.5	.0335	301	
	"	2.16	64.3	.0336	302	
	"	2.16	64.6	.0334	300	

TABLE 7 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
1200	2.16	63.6	.0340	305	
"	2.16	64.6	.0334	300	
"	2.16	64.6	.0334	300	
"	2.16	62.6	.0345	310	
"	2.08	64.1	.0324	291	
"	2.08	60.9	.0342	307	
Totals:	21.44	637.9			
Avg:	2.14	63.8	.0336	302	43,808
1500	2.08	51.1	.0407	366	
"	2.08	55.0	.0378	340	
"	2.08	89.1*	-----	---	
"	2.08	57.6	.0361	324	
"	2.08	65.6*	-----	---	
"	2.08	62.3*	-----	---	
"	2.08	57.6	.0361	324	
"	2.08	60.9*	-----	---	
"	2.08	54.2	.0384	345	
"	2.08	56.5	.0368	331	
"	2.08	54.6	.0381	342	
"	2.08	53.4	.0390	350	
"	2.08	53.6	.0388	349	
"	2.08	53.1	.0392	352	
Totals:	29.12	824.6			
Avg:	2.08	54.7	.0381	342	49,610

*Partial plug, not included in averages.

TABLE 8

LIQUID OF_2 DYNAMIC TESTS
ALUMINUM 2024 ORIFICE

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
120	2.16	211.8	.0102	91.6	
"	2.16	189.7	.0114	102.4	
"	2.16	194.9	.0111	99.7	
Totals:	6.48	596.4			
Avg:	2.16	198.8	.0109	97.9	14,201
300	2.16	133.4	.0162	146	
"	2.16	149.9	.0144	129	
"	2.16	158.9	.0136	122	
"	2.16	216.4*	-----	---	
"	2.16	174.0*	-----	---	
"	2.16	202.0*	-----	---	
"	2.16	155.8	.0139	125	
"	2.16	120.2	.0180	162	
"	2.16	119.6	.0181	163	
Totals:	19.44	1430.2			
Avg:	2.16	139.6	.0155	141	20,453
600	2.16	84.3	.0256	230	
"	2.16	129.5*	-----	---	
"	2.16	113.0*	-----	---	
"	2.16	82.6	.0262	235	
"	2.16	84.1	.0257	231	
"	2.16	115.1*	-----	---	
"	2.16	87.2	.0248	223	
"	2.16	116.4*	-----	---	
"	2.16	104.5*	-----	---	
"	2.16	90.4	.0239	215	
"	2.16	85.2	.0254	228	
"	2.16	84.6	.0255	229	
Totals:	25.92	1176.9			
Avg:	2.16	85.5	.0253	227	32,929
900	2.16	69.5	.0311	279	
"	2.16	87.9*	-----	---	
"	2.16	130.2*	-----	---	
"	2.16	90.3*	-----	---	
"	2.16	80.3*	-----	---	
"	2.16	69.9	.0309	278	
"	2.16	71.1	.0304	273	
"	2.16	142.6*	-----	---	

TABLE 8 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
900	2.16	88.2*	-----	---	
"	2.16	77.0	.0281	252	
"	2.16	113.3*	-----	---	
"	2.16	92.7*	-----	---	
"	2.16	75.1	.0288	259	
"	2.16	68.7	.0314	282	
"	2.16	95.8*	-----	---	
"	2.16	83.0*	-----	---	
"	2.16	65.9	.0328	295	
"	2.16	70.0	.0309	278	
Totals:	38.88	1571.5			
Avg:	2.16	70.9	.0305	274	39,746
1200	2.16	60.8	.0355	319	
"	2.16	61.4	.0352	316	
"	2.16	60.5	.0357	321	
"	2.16	70.4*	.0307	276	
"	2.16	57.0	.0379	340	
"	2.16	60.6	.0356	320	
"	2.16	80.8*	-----	---	
"	2.16	50.0**	-----	---	
"	2.16	60.5	.0357	321	
"	2.08	65.7	.0317	285	
"	2.08	57.8	.0360	323	
Totals:	23.60	685.5			
Avg:	2.15	61.6	.0349	314	45,549
1500	2.08	54.3	.0383	344	
"	2.08	51.2	.0406	365	
"	2.08	50.5	.0412	370	
"	2.08	83.2*	-----	---	
"	2.08	83.4*	-----	---	
"	2.08	50.3	.0414	372	
"	2.08	70.5*	-----	---	
"	2.08	50.4	.0413	371	
"	2.08	54.5	.0382	343	
"	2.08	58.4	.0356	320	
"	2.08	60.8*	-----	---	
"	2.08	50.4	.0413	371	
"	2.0	78.1*	-----	---	
Totals:	27.04	796.0			
Avg:	2.08	52.5	.0397	357	51,786

*Partial plug, not included in averages.
**Incomplete run, not included in averages.

TABLE 9

LIQUID OF_2 DYNAMIC TESTS
S.S. 304 SUMMARY

	TOTAL			AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	4	9.08	863.6	4	2.27	215.9	.0106	94.8	13,752
300	5	10.80	674.3	5	2.16	134.9	.0161	144	20,889
600	7	15.12	635.2	7	2.16	90.7	.0238	214	31,043
900	10	21.60	771.7	9	2.16	75.1	.0288	258	37,425
1200	10	21.44	637.9	10	2.14	63.8	.0336	302	43,808
1500	14	29.12	824.6	10	2.08	54.7	.0381	342	49,610
TOTAL	50	107.16	4407.3						

TABLE 10

LIQUID OF_2 DYNAMIC TESTS
ALUMINUM 2024 SUMMARY

	TOTAL			AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	3	6.48	596.4	3	2.16	198.8	.0109	97.9	14,201
300	9	19.44	1430.2	6	2.16	139.6	.0155	141	20,453
600	12	25.92	1176.9	7	2.16	85.5	.0253	227	32,929
900	18	38.88	1571.5	8	2.16	70.9	.0305	274	39,746
1200	11	23.60	685.5	8	2.15	61.6	.0349	314	45,549
1500	13	27.04	796.0	8	2.08	52.5	.0397	357	51,786
TOTAL	66	141.36	6256.5						

TABLE 11

LIQUID OF_2 DYNAMIC TESTS
ALUMINUM 6061 ORIFICE

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
120	2.19	221.0	.0099	88.9	
"	2.19	204.7	.0107	96.1	
"	2.19	205.1	.0107	96.1	
Totals:	6.57	630.8			
Avg:	2.19	210.3	.0104	93.7	13,592
300	2.19	131.1	.0167	150	
"	2.19	127.8	.0171	154	
"	2.19	127.3	.0172	155	
"	2.30	142.9	.0161	145	
"	2.30	140.1	.0164	147	
Totals:	11.17	669.2			
Avg:	2.23	133.8	.0167	150	21,759
600	2.30	97.7	.0235	211	
"	2.30	102.8	.0224	201	
"	2.30	128.9	.0178	160	
"	2.30	121.0	.0190	171	
"	2.30	107.8	.0213	191	
"	2.30	122.0	.0189	170	
"	2.30	106.8	.0215	193	
Totals:	16.10	787.0			
Avg:	2.30	112.4	.0205	184	26,691
900	2.30	87.2	.0264	237	
"	2.30	87.2	.0264	237	
"	2.30	92.9	.0248	223	
"	2.30	86.2	.0267	240	
"	2.30	87.4	.0263	236	
"	2.30	88.0	.0261	234	
"	2.30	86.3	.0267	240	
Totals:	16.10	615.2			
Avg:	2.30	87.9	.0262	235	34,089
1200	2.30	75.9	.0303	272	
"	2.30	74.5	.0309	278	
"	2.30	74.8	.0307	276	
"	2.30	77.2	.0298	268	
"	2.30	74.6	.0308	277	
"	2.30	74.7	.0307	276	
"	2.30	73.2	.0314	282	
"	2.30	84.9	.0271	243	
Totals:	18.40	609.8			
Avg:	2.30	76.2	.0302	271	39,311

TABLE 11 (Continued)

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	1500	2.30	104.0*	-----	---	
	"	2.30	69.2	.0332	298	
	"	2.30	68.0	.0338	304	
	"	2.30	66.9	.0344	309	
	"	2.30	75.8	.0303	272	
	"	2.30	72.6	.0317	285	
	"	2.30	86.0*	-----	---	
	"	2.30	115.0*	-----	---	
	"	2.30	71.7	.0321	288	
	"	2.30	73.8	.0312	280	
Totals:		23.00	803.0			
Avg:		2.30	71.1	.0323	290	42,067

*Partial plug, not included in averages.

TABLE 12

LIQUID OF_2 DYNAMIC TESTS
TITANIUM ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	2.19	210.5	.0104	93.4	
	"	2.19	203.2	.0108	97.0	
	"	2.19	201.5	.0109	97.9	
Totals:		6.57	615.2			
Avg:		2.19	205.1	.0107	96.1	13,940
	300	2.19	141.8	.0154	138	
	"	2.19	128.8	.0170	153	
	"	2.30	153.6	.0150	135	
	"	2.30	142.4	.0162	146	
	"	2.30	141.8	.0162	146	
Totals:		11.28	708.4			
Avg:		2.26	141.7	.0160	144	20,889
	600	2.30	168.9*	-----	---	
	"	2.30	136.9*	-----	---	
	"	2.30	111.6	.0206	185	
	"	2.30	102.0	.0225	202	
	"	2.30	108.9	.0211	190	
	"	2.30	106.4	.0216	194	
	"	2.30	111.7	.0206	185	
	"	2.30	111.6	.0206	185	
Totals:		18.40	958.0			
Avg:		2.30	108.7	.0212	190	27,561
	900	2.30	92.9	.0248	223	
	"	2.30	119.0*	-----	---	
	"	2.30	92.0	.0250	225	
	"	2.30	94.1	.0244	219	
	"	2.30	92.6	.0248	223	
	"	2.30	93.6	.0246	221	
	"	2.30	138.5*	-----	---	
	"	2.30	95.3	.0241	217	
Totals:		18.40	818.0			
Avg:		2.30	93.4	.0246	221	32,058

TABLE 12 (Continued)

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	1200	2.30	77.0	.0299	269	
	"	2.30	78.9	.0292	262	
	"	2.30	80.3	.0286	257	
	"	2.30	81.8	.0281	252	
	"	2.30	79.0	.0291	261	
	"	2.30	80.1	.0287	258	
	"	2.30	77.9	.0295	265	
	"	2.30	80.4	.0286	257	
Totals:		18.40	635.4			
Avg:		2.30	79.5	.0289	260	37,716
	1500	2.30	68.8	.0334	300	
	"	2.30	72.0	.0319	287	
	"	2.30	72.3	.0318	286	
	"	2.30	70.6	.0326	293	
	"	2.30	68.8	.0334	300	
	"	2.30	70.2	.0328	295	
	"	2.30	70.6	.0326	293	
	"	2.30	72.7	.0316	284	
	"	2.30	71.8	.0320	287	
	"	2.30	117.4*	-----	---	
Totals:		23.00	755.2			
Avg:		2.30	70.9	.0324	291	42,212

*Partial plug, not included in averages.

TABLE 13

LIQUID OF_2 DYNAMIC TESTS
ALUMINUM 6061 SUMMARY

TOTAL				AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	3	6.57	630.8	3	2.19	210.3	.0104	93.7	13,592
300	5	11.17	669.2	5	2.23	133.8	.0167	150	21,759
600	7	16.10	787.0	7	2.30	112.4	.0205	184	26,691
900	7	16.10	615.2	7	2.30	87.9	.0262	235	34,089
1200	8	18.40	609.8	8	2.30	76.2	.0302	271	39,311
1500	10	23.00	803.0	7	2.30	71.1	.0323	290	42,067
TOTAL	40	91.34	4115.0						

TABLE 14

LIQUID OF_2 DYNAMIC TESTS
TITANIUM SUMMARY

TOTAL				AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	3	6.57	615.7	3	2.19	205.1	.0107	96.1	13,940
300	5	11.28	708.4	5	2.26	141.7	.0160	144	20,889
600	8	18.40	958.0	6	2.30	108.7	.0212	190	27,561
900	8	18.40	818.0	6	2.30	93.4	.0246	221	32,058
1200	8	18.40	635.4	8	2.30	79.5	.0289	260	37,716
1500	10	23.00	755.2	9	2.30	70.9	.0324	291	42,212
TOTAL	42	96.05	4490.2						

TABLE 15

LIQUID OF_2 DYNAMIC TESTS
STAINLESS STEEL 301 ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	2.19	219.9	.0100	89.8	
	"	2.19	222.7	.0098	88.0	
	"	2.19	221.0	.0099	88.9	
Totals:		6.57	663.6			
Avg:		2.19	221.2	.0099	88.9	12,895
	300	2.19	141.9	.0154	138	
	"	2.19	142.3	.0154	138	
	"	2.19	142.0	.0154	138	
	"	2.19	142.6	.0154	138	
	"	2.19	141.6	.0155	139	
Totals:		10.95	710.4			
Avg:		2.19	142.1	.0154	138	20,018
	600	2.19	100.8	.0217	195	
	"	2.19	99.8	.0219	197	
	"	2.19	99.8	.0219	197	
	"	2.19	99.1	.0221	199	
	"	2.19	99.3	.0221	199	
	"	2.19	99.0	.0221	199	
Totals:		13.14	597.8			
Avg:		2.19	99.6	.0220	198	28,722
	900	2.19	83.6	.0262	235	
	"	2.19	81.8	.0268	241	
	"	2.19	81.6	.0268	241	
	"	2.19	81.6	.0268	241	
	"	2.19	83.0	.0264	237	
	"	2.19	87.2	.0251	225	
	"	2.19	85.7	.0256	230	
	"	2.19	84.7	.0259	233	
	"	2.19	80.4	.0272	244	
Totals:		19.71	749.6			
Avg:		2.19	83.3	.0263	236	34,234
	1200	1.96	56.4	.0348	313	
	"	1.96	56.4	.0348	313	
	"	1.96	60.6	.0323	290	
	"	1.96	58.6	.0334	300	
	"	1.96	58.2	.0337	303	

TABLE 15 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
1200	1.96	57.0	.0344	309	
"	1.96	57.0	.0344	309	
"	1.96	57.4	.0341	306	
"	1.96	57.0	.0344	309	
"	1.96	56.7	.0346	311	
"	1.96	67.5*	-----	---	
Totals:	21.56	642.8			
Avg:	1.96	57.5	.0341	306	44,388
1500	1.96	69.9*	-----	---	
"	1.96	60.5*	-----	---	
"	1.96	60.6*	-----	---	
"	1.96	45.7	.0429	385	
"	1.96	50.8	.0386	347	
"	1.96	55.3	.0354	318	
"	1.96	49.9	.0393	353	
"	1.96	51.4	.0381	342	
"	1.96	51.7	.0379	340	
"	1.96	54.7	.0358	322	
"	1.96	52.4	.0374	336	
"	1.96	53.9	.0364	327	
Totals:	23.52	657.8			
Avg:	1.96	51.9	.0378	340	49,320

*Partial plug, not included in averages.

TABLE 16

LIQUID OF_2 DYNAMIC TESTS
INCONEL ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	2.19	229.4	.0095	85.3	
	"	2.19	218.5	.0100	89.8	
	"	2.19	217.2	.0101	90.7	
Totals:		6.57	665.1			
Avg:		2.19	221.7	.0099	88.9	12,895
	300	2.19	139.4	.0157	141	
	"	2.19	135.5	.0162	146	
	"	2.19	135.5	.0162	146	
	"	2.19	141.3	.0155	139	
	"	2.19	135.4	.0162	146	
Totals:		10.95	687.1			
Avg:		2.19	137.4	.0159	143	20,744
	600	2.19	94.3	.0232	208	
	"	2.19	93.7	.0234	210	
	"	2.19	93.4	.0234	210	
	"	2.19	93.7	.0234	210	
	"	2.19	93.7	.0234	210	
	"	2.19	93.1	.0235	211	
Totals:		13.14	561.9			
Avg:		2.19	93.7	.0234	210	30,463
	900	2.19	76.2	.0287	258	
	"	2.19	76.6	.0286	257	
	"	2.19	75.6	.0290	261	
	"	2.19	77.7	.0282	253	
	"	2.19	89.2	.0246	221	
	"	2.19	86.6	.0253	227	
	"	2.19	84.8	.0258	232	
	"	2.19	74.6	.0294	264	
	"	2.19	74.3	.0295	265	
Totals:		19.71	715.6			
Avg:		2.19	79.5	.0277	249	36,120
	1200	1.96	55.3	.0354	318	
	"	1.96	65.6	.0299	269	
	"	1.96	49.6	.0395	355	
	"	1.96	74.4*	-----	---	
	"	1.96	60.7	.0323	290	
	"	1.96	58.0	.0338	303	

TABLE 16 (Continued)

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	1200	1.96	57.6	.0340	305	
	"	1.96	52.8	.0371	333	
	"	1.96	52.5	.0373	335	
	"	1.96	52.4	.0374	336	
	"	1.96	52.6	.0373	335	
	"	1.96	52.4	.0374	336	
	"	1.96	52.0	.0377	339	
Totals:		25.48	735.9			
Avg:		1.96	55.1	.0356	320	46,419
	1500	1.96	46.4	.0422	379	
	"	1.96	46.4	.0422	379	
	"	1.96	45.0	.0436	392	
	"	1.96	62.6*	-----	---	
	"	1.96	52.6	.0373	335	
	"	1.96	67.3*	-----	---	
	"	1.96	56.2	.0349	314	
	"	1.96	46.2	.0424	381	
	"	1.96	56.0	.0350	314	
	"	1.96	66.0*	-----	---	
	"	1.96	43.6	.0450	404	
Totals:		21.56	588.3			
Avg:		1.96	49.1	.0399	358	51,061

*Partial plug, not included in averages.

TABLE 17

LIQUID OF_2 DYNAMIC TESTS
S.S. 301, SUMMARY

TOTAL				AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	3	6.57	663.6	3	2.19	221.2	.0099	88.9	12,895
300	5	10.95	710.4	5	2.19	142.1	.0154	138	20,018
600	6	13.14	597.4	6	2.19	99.6	.0220	198	28,722
900	9	19.71	749.6	9	2.19	83.3	.0263	236	34,234
1200	11	21.56	642.8	10	1.96	57.5	.0341	306	44,388
1500	12	23.52	657.8	9	1.96	51.9	.0378	340	49,320
TOTAL	46	95.45	4021.6						

TABLE 18

LIQUID OF_2 DYNAMIC TESTS
INCONEL SUMMARY

TOTAL				AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	3	6.57	665.1	3	2.19	221.7	.0099	88.7	12,895
300	5	10.95	687.1	5	2.19	137.4	.0159	143	20,744
600	6	13.14	561.9	6	2.19	93.7	.0234	210	30,463
900	9	19.71	715.6	9	2.19	79.5	.0277	249	36,120
1200	13	25.48	735.9	12	1.96	55.1	.0356	320	46,419
1500	11	21.56	588.3	8	1.96	49.1	.0399	358	51,061
TOTAL	47	97.41	3953.9						

TABLE 19

LIQUID OF_2 DYNAMIC TESTS
BRAZED MONEL ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	2.19	211.5	.0104	93.4	
	"	2.19	197.3	.0111	99.7	
	"	2.19	196.6	.0111	99.7	
Totals:		6.57	605.4			
Avg:		2.19	201.8	.0109	97.6	14,158
	300	2.19	129.1	.0170	153	
	"	2.19	126.1	.0174	156	
	"	2.19	127.1	.0172	155	
	"	2.19	126.0	.0174	156	
	"	2.19	126.0	.0174	156	
Totals:		10.95	634.3			
Avg:		2.19	126.9	.0173	155	22,484
	600	2.19	88.6	.0247	222	
	"	2.19	88.7	.0247	222	
	"	2.19	88.5	.0247	222	
	"	2.19	94.4	.0232	208	
	"	2.19	116.0*	-----	---	
	"	2.19	97.2	.0225	202	
	"	2.19	87.8	.0249	224	
Totals:		15.33	661.2			
Avg:		2.19	90.9	.0241	217	31,478
	900	2.19	88.6	.0247	222	
	"	2.19	73.4	.0298	268	
	"	2.19	89.3	.0245	220	
	"	2.19	90.2	.0243	218	
	"	2.19	112.1*	-----	---	
	"	2.19	89.1	.0246	221	
	"	2.19	68.3	.0321	288	
	"	2.19	72.5	.0302	271	
	"	2.19	72.2	.0303	272	
	"	2.19	103.5*	-----	---	
	"	2.19	83.0	.0264	237	
Totals:		24.09	942.2			
Avg:		2.19	80.7	.0271	243	35,250

TABLE 19 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
1200	2.19	63.1	.0347	312	
"	2.19	63.3	.0346	311	
"	2.19	79.9	.0274	246	
"	2.19	85.7	.0256	230	
"	2.19	80.6	.0272	244	
"	2.19	77.2	.0284	255	
"	2.19	71.8	.0305	274	
"	2.19	64.0	.0342	307	
"	2.09	104.0*	-----	---	
"	2.09	70.6	.0296	266	
"	2.09	88.9	.0235	211	
"	2.09	80.0	.0261	234	
"	2.09	77.4	.0270	243	
"	2.09	68.4	.0306	275	
Totals:	30.06	1074.9			
Avg:	2.15	74.7	.0288	259	37,570
1500	2.09	56.7	.0369	332	
"	2.09	65.1	.0321	288	
"	2.09	56.3	.0371	333	
"	2.09	59.7	.0350	314	
"	2.09	59.3	.0352	316	
"	2.09	55.8	.0375	337	
"	2.09	55.6	.0376	338	
"	2.09	55.6	.0376	338	
"	2.09	55.4	.0377	339	
"	2.09	55.1	.0379	340	
"	2.09	54.6	.0383	344	
Totals:	22.99	629.2			
Avg:	2.09	57.2	.0365	328	47,580

*Partial plug, not included in averages.

TABLE 20

LIQUID OF_2 DYNAMIC TESTS
WELDED MONEL ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	2.19	209.6	.0104	93.4	
	"	2.19	207.7	.0105	94.3	
	"	2.19	205.8	.0106	95.2	
Totals:		6.57	623.1			
Avg:		2.19	207.7	.0105	94.3	13,679
	300	2.19	133.0	.0165	148	
	"	2.19	133.7	.0164	147	
	"	2.19	133.2	.0164	147	
	"	2.19	133.4	.0164	147	
	"	2.19	133.6	.0164	147	
Totals:		10.95	666.9			
Avg:		2.19	133.4	.0164	147	21,324
	600	2.19	92.7	.0236	212	
	"	2.19	93.3	.0235	211	
	"	2.19	93.5	.0234	210	
	"	2.19	90.5	.0234	217	
	"	2.19	94.7	.0231	208	
	"	2.19	100.4*	-----	---	
	"	2.19	92.3	.0237	213	
Totals:		15.33	657.4			
Avg:		2.19	92.8	.0236	212	30,753
	900	2.19	84.8	.0258	232	
	"	2.19	75.7	.0289	260	
	"	2.19	75.8	.0289	260	
	"	2.19	77.1	.0284	255	
	"	2.19	76.0	.0288	259	
	"	2.19	76.8	.0285	256	
	"	2.19	76.6	.0286	257	
	"	2.19	75.8	.0289	260	
	"	2.19	82.2	.0266	239	
Totals:		19.71	700.8			
Avg:		2.19	77.9	.0281	252	36,555

TABLE 20 (Continued)

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	1200	2.19	66.3	.0330	296	
	"	2.19	111.7*	-----	---	
	"	2.19	69.6	.0314	282	
	"	2.19	67.1	.0326	293	
	"	2.19	65.0	.0337	303	
	"	2.09	64.9	.0322	289	
	"	2.09	65.3	.0320	287	
	"	2.09	62.7	.0333	299	
	"	2.09	65.0	.0322	289	
	"	2.09	70.4	.0297	267	
	"	2.09	71.5	.0292	262	
Totals:		23.49	779.5			
Avg:		2.14	66.8	.0320	287	41,632
	1500	2.09	54.8	.0381	342	
	"	2.09	58.1	.0360	323	
	"	2.09	59.7	.0350	314	
	"	2.09	119.6*	-----	---	
	"	2.09	73.3*	-----	---	
	"	2.09	65.7*	-----	---	
	"	2.09	57.6	.0363	326	
	"	2.09	57.5	.0363	326	
	"	2.09	57.5	.0363	326	
	"	2.09	57.4	.0364	327	
	"	2.09	57.3	.0365	328	
	"	2.09	56.9	.0367	330	
Totals:		25.08	775.4			
Avg:		2.09	57.4	.0364	327	47,435

*Partial plug, not included in averages.

TABLE 21

LIQUID OF_2 DYNAMIC TESTS
BRAZED MONEL SUMMARY

TOTAL				AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	3	6.57	605.4	3	2.19	201.8	.0109	97.6	14,158
300	5	10.95	634.3	5	2.19	126.9	.0173	155	22,484
600	7	15.33	661.2	6	2.19	90.9	.0241	217	31,478
900	11	24.09	942.2	9	2.19	80.7	.0271	243	35,250
1200	14	30.06	1074.9	13	2.15	74.7	.0288	259	37,570
1500	11	22.99	629.2	11	2.09	57.2	.0365	328	47,580
TOTAL	51	109.99	4547.2						

TABLE 22

LIQUID OF_2 DYNAMIC TESTS
WELDED MONEL SUMMARY

TOTAL				AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	3	6.57	623.1	3	2.19	207.7	.0105	94.3	13,679
300	5	10.95	666.9	5	2.19	133.4	.0164	147	21,324
600	7	15.33	657.4	6	2.19	92.8	.0236	212	30,753
900	9	19.71	700.8	9	2.19	77.9	.0281	252	36,555
1200	11	23.49	779.5	10	2.14	66.8	.0320	287	41,632
1500	12	25.08	775.4	9	2.09	57.4	.0364	327	47,435
TOTAL	47	101.13	4203.1						

TABLE 23

LIQUID OF_2 DYNAMIC TESTS
SILVER SOLDERED MONEL ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	2.09	181.2	.0115	103	
	"	2.09	181.5	.0115	103	
	"	2.09	182.4	.0115	103	
	"	2.09	179.5	.0116	104	
Totals:		8.36	724.6			
Avg:		2.09	181.2	.0115	103	14,941
	300	2.09	170.3*	-----	---	
	"	2.09	123.8	.0169	152	
	"	2.09	121.4	.0172	155	
	"	2.09	115.7	.0181	163	
	"	2.09	113.8	.0184	165	
	"	2.09	115.4	.0181	163	
Totals:		12.54	760.4			
Avg:		2.09	118.0	.0177	159	23,065
	600	2.09	80.7	.0259	233	
	"	2.09	82.6	.0253	227	
	"	2.09	80.3	.0260	234	
	"	2.09	80.0	.0261	234	
	"	2.09	81.6	.0256	230	
	"	2.09	79.5	.0263	236	
	"	1.88	71.1	.0264	237	
	"	1.88	70.8	.0265	238	
Totals:		16.30	626.6			
Avg:		2.04	78.3	.0261	234	33,944
	900	1.88	58.2	.0323	290	
	"	1.88	55.5	.0339	305	
	"	1.88	55.2	.0341	306	
	"	1.88	54.3	.0346	311	
	"	1.88	62.4	.0301	270	
	"	1.88	60.3	.0312	280	
	"	1.88	54.8	.0343	308	
	"	1.88	61.0	.0308	277	
	"	1.88	55.1	.0341	306	
	"	1.88	55.5	.0339	305	
	"	1.88	56.5	.0333	299	
Totals:		20.68	628.8			
Avg:		1.88	57.2	.0329	296	42,938

TABLE 23 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
1200	1.88	63.4*	-----	---	
"	1.88	49.9	.0377	339	
"	1.88	48.2	.0390	350	
"	1.88	48.7	.0386	347	
"	1.88	47.9	.0392	352	
"	1.88	47.0	.0400	359	
"	1.88	47.8	.0393	353	
"	1.88	48.0	.0392	352	
"	1.88	74.1*	-----	---	
"	1.88	60.5*	-----	---	
"	2.22	73.9*	-----	---	
"	2.22	69.3*	-----	---	
Totals:	23.24	678.7			
Avg:	1.88	48.2	.0390	350	50,771
1500	2.22	62.2	.0357	321	
"	2.22	62.1	.0357	321	
"	2.22	62.5	.0355	319	
"	2.22	61.7	.0360	323	
"	2.22	63.0	.0352	316	
Totals:	11.10	311.5			
Avg:	2.22	62.3	.0356	320	46,419

*Partial plug, not included in averages.

TABLE 24

LIQUID OF_2 DYNAMIC TESTS
COPPER ALLOY ORIFICE

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
120	2.09	180.9	.0116	104	
"	2.09	166.6	.0125	112	
"	2.09	166.1	.0126	113	
"	2.09	165.5	.0126	113	
Totals:	8.36	679.1			
Avg:	2.09	169.8	.0123	111	16,102
300	2.09	115.1	.0182	164	
"	2.09	115.7	.0181	163	
"	2.09	116.5	.0179	161	
"	2.09	120.8	.0173	155	
"	2.09	119.6	.0175	157	
"	2.09	120.5	.0173	155	
Totals:	12.54	708.2			
Avg:	2.09	118.0	.0177	159	23,065
600	2.09	87.2	.0240	216	
"	2.09	88.9	.0235	211	
"	2.09	86.4	.0242	217	
"	2.09	86.4	.0242	217	
"	2.09	84.6	.0247	222	
"	2.09	86.4	.0242	217	
"	1.88	80.7	.0233	209	
"	1.88	76.7	.0245	220	
Totals:	16.30	677.3			
Avg:	2.04	84.7	.0241	217	31,478
900	1.88	84.1*	-----	---	
"	1.88	101.1*	-----	---	
"	1.88	43.4**	-----	---	
"	1.88	60.1	.0313	281	
"	1.88	92.9*	-----	---	
"	1.88	81.6*	-----	---	
"	1.88	45.5**	-----	---	
"	1.88	40.3**	-----	---	
"	1.88	56.9	.0330	296	
"	1.88	60.0	.0313	281	
"	1.88	60.0	.0313	281	
"	1.88	62.0	.0303	272	
"	1.88	61.7	.0305	274	

TABLE 24 (Continued)

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
900	1.88	60.0	.0313	281	
"	1.88	60.0	.0313	281	
"	1.88	60.0	.0313	281	
Totals:	30.08	1029.6			
Avg:	1.88	60.1	.0313	281	40,762
1200	1.88	50.0	.0376	338	
"	1.88	55.8	.0337	303	
"	1.88	51.5	.0365	328	
"	1.88	51.7	.0364	327	
"	1.88	51.6	.0364	327	
"	1.88	51.0	.0369	332	
"	1.88	50.0	.0376	338	
"	1.88	69.8*	-----	---	
"	1.88	76.4*	-----	---	
"	1.88	58.6	.0321	288	
"	2.22	73.4*	-----	---	
"	2.22	72.5*	-----	---	
Totals:	23.24	712.3			
Avg:	1.88	52.5	.0358	322	46,709
1500	2.22	64.9	.0342	307	
"	2.22	65.1	.0341	306	
"	2.22	66.5	.0334	300	
"	2.22	64.9	.0342	307	
"	2.22	65.1	.0341	306	
"	2.22	64.8	.0343	308	
"	2.22	68.9	.0322	289	
"	2.22	67.5	.0329	296	
"	2.22	66.4	.0334	300	
"	2.22	66.6	.0333	299	
Totals:	22.20	660.7			
Avg:	2.22	66.1	.0336	302	43,808

*Partial plug, not included in averages.

**Flow in reverse direction in attempt to dislodge plug. These runs are not included in averages.

TABLE 25

LIQUID OF_2 DYNAMIC TESTS
SILVER SOLDERED MONEL SUMMARY

	TOTAL			AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	4	8.36	724.6	4	2.09	181.2	.0115	103	14,941
300	6	12.54	760.4	5	2.09	118.0	.0177	159	23,065
600	8	16.30	626.6	8	2.04	78.3	.0261	234	33,944
900	11	20.68	628.8	11	1.88	57.2	.0329	296	42,938
1200	12	23.24	678.7	7	1.88	48.2	.0390	350	50,771
1500	5	11.10	311.5	5	2.22	62.3	.0356	320	46,419
TOTAL	46	92.22	3730.6						

TABLE 26

LIQUID OF_2 DYNAMIC TESTS
COPPER-CHROMIUM SUMMARY

	TOTAL			AVERAGE					
PSIG	No. Runs	Lbs. OF_2	Time Sec.	No. Runs	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Meas. Vel.	Re. No.
120	4	8.36	679.1	4	2.09	169.8	.0123	111	16,102
300	6	12.54	708.2	6	2.09	118.0	.0177	159	23,065
600	8	16.30	677.3	8	2.04	84.7	.0241	217	31,478
900	16	30.08	1029.6	9	1.88	60.1	.0313	281	40,762
1200	12	23.24	712.3	8	1.88	52.5	.0358	322	46,709
1500	10	22.20	660.7	10	2.22	66.1	.0336	302	43,808
TOTAL	56	112.72	4467.2						

TABLE 27

LIQUID OF_2 DYNAMIC TESTS
PHYSICAL CHANGES OF TEST SPECIMENS

	Weight - grams			Orifice Diameter (inches)		
	Before	After	Δ	Before	After	Δ
Monel	12.0856	12.0869	+.0013	.0136	.0138	+.0002
Nickel	12.6882	12.6894	+.0012	.0139	.0150	+.0011
S. S. 304	10.5671	10.5685	+.0014	.0133	.0133	.0000
Al 2024	4.0820	4.0817	-.0003	.0135	.0135	.0000
Al 6061	3.8860	3.8860	.0000	.0130	.0140	+.0010
Titanium	5.2949	5.2962	+.0013	.0132	.0132	.0000
S. S. 301	5.5740	5.5731	-.0009	.0133	.0134	+.0001
Inconel	12.1580	12.1586	+.0006	.0130	.0130	.0000
Brazed Monel	10.2288	10.2293	+.0005	.0139	.0142	+.0003
Welded Monel	10.4740	10.4743	+.0003	.0139	.0139	.0000
Silver Soldered Monel	11.0175	11.0184	+.0009	.0143	.0143	.0000
Copper-Chromium	5.1274	5.1286	+.0012	.0145	.0194*	+.0049*

*Specimen distorted and orifice enlargement due to stretching of metal. Measurements taken on the inlet side of the orifice after exposure indicate no enlargement.

TABLE 28

LIQUID OF_2 DYNAMIC TESTS
PLASTIC IDENTIFICATION

No.	Name	A	B	C	D
1	Teflon 5	(1)	(4)	(5)	
2	Teflon 7	(1)	(4)	(5)	
3	Halon TFE G-80(H)	(1)	(5)	(5)	(8)
4	Halon TFE G-80(L)	(1)	(5)	(5)	(9)
5	Almac CTFE	(2)	(6)	(7)	
6	Plaskon 2200	(2)	(5)	(5)	
7	FEP	(3)	(4)	(7)	
8	Halon TFE G-50	(1)	(5)	(5)	

A. Composition

B. Resin Manufacturer

C. Fabricator

D. Remarks

(1) Tetrafluoroethylene polymer.
(2) Trifluoromonochloroethylene polymer.
(3) Copolymer of tetrafluoroethylene and perfluoropropylene.
(4) DuPont Chemical Company.
(5) Allied Chemical Corporation.
(6) 3 M Company.
(7) Almac Plastics.
(8) High crystallinity.
(9) Low crystallinity.

TABLE 29

LIQUID OF_2 DYNAMIC TESTS
TEFLON 5 ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	.22	26.3	.0084	75.5	
	"	.22	18.6	.0118	106	
Totals:		.44	44.9			
Avg:		.22	22.5	.0101	90.8	13,171
	300	.22	10.0	.0220	198	
	"	.22	10.3	.0214	192	
Totals:		.44	20.3			
Avg:		.22	10.2	.0217	195	28,287
	500	.22	5.8	.0379	340	
	"	.22	5.8	.0379	340	
Totals:		.44	11.6			
Avg:		.22	5.8	.0379	340	49,320

TABLE 30

LIQUID OF_2 DYNAMIC TESTS
TEFLON 7 ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	.22	26.5	.0083	74.6	
	"	.22	26.2	.0084	75.5	
Totals:		.44	52.7			
Avg:		.22	26.4	.0084	75.1	10,894
	300	.22	12.9	.0171	154	
	"	.22	13.5	.0163	146	
Totals:		.44	26.4			
Avg:		.22	13.2	.0167	150	21,759
	500	.22	11.9	.0185	166	
	"	.22	9.8	.0224	201	
Totals:		.44	21.7			
Avg:		.22	10.9	.0205	184	26,691

TABLE 31

LIQUID OF_2 DYNAMIC TESTS
HALON TFE G-80(L) ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	.22	11.4	.0193	173	
	"	.22	15.1	.0145	130	
Totals:		.44	26.5			
Avg:		.22	13.3	.0169	152	22,049
	300	.22	8.2	.0268	241	
	"	.22	8.0	.0275	247	
Totals:		.44	16.2			
Avg:		.22	8.1	.0272	244	35,395
	500	.22	5.0	.0440	395	
	"	.22	6.4	.0344	309	
Totals:		.44	11.4			
Avg:		.22	5.7	.0392	352	51,061

TABLE 32

LIQUID OF_2 DYNAMIC TESTS
HALON TFE C-80(H) ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	.22	21.5	.0102	92	
	"	.22	21.4	.0103	93	
Totals:		.44	42.9			
Avg:		.22	21.5	.0103	93	13,490
	300	.22	11.3	.0195	175	
	"	.22	10.8	.0204	183	
Totals:		.44	22.1			
Avg:		.22	11.0	.0200	179	25,966
	500	.22	7.4	.0297	267	
	"	.22	7.9	.0278	250	
Totals:		.44	15.3			
Avg:		.22	7.7	.0288	259	37,570

TABLE 33

LIQUID OF_2 DYNAMIC TESTS
ALMAC CTFE ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	.22	22.3	.0099	89	
	"	.22	16.7	.0132	119	
	"	.22	18.0	.0122	110	
Totals:		.66	57.0			
Avg:		.22	19.0	.0118	106	15,376
	300	.22	10.0	.0220	198	
	"	.22	8.1	.0272	244	
Totals:		.44	18.1			
Avg:		.22	9.1	.0246	221	32,058
	500	.22	6.5	.0338	304	
	"	.22	7.4	.0297	267	
Totals:		.44	13.9			
Avg:		.22	7.0	.0318	286	41,487

TABLE 34

LIQUID OF_2 DYNAMIC TESTS
PLASKON 2200 ORIFICE

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
120	.22	13.4	.0164	147	
"	.22	16.0	.0138	124	
"	.22	15.3	.0144	129	
Totals:	.66	44.7			
Avg:	.22	14.9	.0149	134	19,438
300	.22	8.0	.0275	247	
"	.22	9.2	.0239	215	
Totals:	.44	17.2			
Avg:	.22	8.6	.0257	231	33,509
500	.22	6.6	.0333	299	
"	.22	6.6	.0333	299	
Totals:	.44	13.2			
Avg:	.22	6.6	.0333	299	43,373

TABLE 35

LIQUID OF_2 DYNAMIC TESTS
FEP ORIFICE

PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
120	.22	24.6	.0089	80	
"	.22	39.7	.0055	49	
"	.22	30.4	.0072	65	
"	.22	31.8	.0069	62	
Totals:	.88	126.5			
Avg:	.22	31.7	.0072	64	9,284
300	.22	9.6	.0229	206	
"	.22	18.7	.0118	106	
"	.22	11.8	.0186	167	
Totals:	.66	40.1			
Avg:	.22	13.4	.0178	160	23,210
500	.22	6.5	.0338	304	
"	.22	9.5	.0232	208	
"	.22	7.2	.0306	275	
Totals:	.66	23.2			
Avg:	.22	7.8	.0292	263	38,151

TABLE 36

LIQUID OF_2 DYNAMIC TESTS
HALON TFE G-50 ORIFICE

	PSIG	Lbs. OF_2	Time Sec.	Mass Flow Lbs/Sec.	Measured Vel. Ft/Sec.	Re. No.
	120	.22	19.6	.0112	101	
	"	.22	17.2	.0128	115	
	"	.22	15.9	.0138	124	
Totals:		.66	52.7			
Avg:		.22	17.6	.0126	114	16,537
	300	.22	16.7	.0132	119	
	"	.22	9.4	.0234	210	
	"	.22	9.2	.0239	215	
	"	.22	10.6	.0208	187	
Totals:		.88	45.9			
Avg:		.22	11.5	.0203	183	26,546
	500	.22	7.1	.0310	279	
	"	.22	6.9	.0319	287	
Totals:		.44	14.0			
Avg:		.22	7.0	.0315	283	41,052

TABLE 37

LIQUID OF_2 DYNAMIC TESTS
PLASTIC MATERIAL WEIGHT CHANGES

Material	Wt. 1	Wt. 2	Wt. 3	Wt. 1 - Wt. 3 **
Teflon 5	1.1305	1.1309	1.1309	+0.0004
Teflon 7	1.1910	1.1920	1.1917	+0.0007
G-80 Low *	0.9896	0.9906	0.9897	+0.0001
G-80 High *	0.9968	0.9982	0.9974	+0.0006
Almac CTFE	1.0414	1.0408	1.0408	-0.0006
Plaskon 2200	1.1286	1.1276	1.1278	-0.0008
Almac FEP	1.1505	1.1515	1.1514	+0.0009
Halon G-50	1.0520	1.0532	1.0532	+0.0012

*Halon TFE G-80 low and high crystallinity as indicated.

**+ indicates gain in weight for exposed specimen
- indicates loss in weight for exposed specimen

TABLE 38

OF_2 GAS DYNAMIC TESTS
METAL ORIFICES

Material	OF_2 Cylinder PSIG Before	OF_2 Cylinder PSIG After	°C	Initial Wt. gms.	Final Wt. gms.	△Wt.
Monel 400	216	207	29	12.4755	12.4763	+.0008
Welded Monel	207	195	29	10.6506	10.6508	+.0002
Brazed Monel	195	185	29	11.6294	11.6296	+.0002
Silver Soldered Monel	185	175	29	11.4191	11.4193	+.0002
Nickel 200	170	160	25	12.2603	12.2603	.0000
S. S. 301	160	150	25	5.6245	5.6248	+.0003
S. S. 304	150	140	25	10.6775	10.6775	.0000
Inconel	140	130	26	12.2984	12.2986	+.0002
Aluminum 6061	131	120	28	3.9146	3.9146	.0000
Aluminum 2024	120	110	28	4.0870	4.0870	.0000
Copper-Chromium	111	100	29	5.1872	5.1875	+.0003
Titanium	100	88	29	5.4097	5.4097	.0000

Note: OF_2 cylinder volume is 3016 $in.^3$.

TABLE 39

WIRE IGNITION STUDY
TEST MATERIALS

	Material	Source	Diameter	M.P. °C
1.	Nickel "A"	(a)	.0150"	1450
2.	Nickel "A"	(a)	.0088"	1450
3.	Molybdenum	(a)	.0151"	2620
4.	Tungsten	(a)	.0120"	3370
5.	Monel 400	(a)	.0100"	1330
6.	S. S. 302	(a)	.0200"	1400
7.	Copper	(a)	.0126"	1083
8.	Iron	(b)	.0090"	1535
9.	Monel 400	(c)	.0101"	1330

(a) Magnetic Wire Corporation, N.Y., N.Y.

(b) B&A Code 1805 High Purity Iron (99.90%) wire, Allied Chemical Corporation.

(c) Newark Wire Cloth Co., Newark, New Jersey.

TABLE 40

WIRE IGNITION IN OF_2
PRELIMINARY TESTS

Run	Material	Length	Diam.	Initial Glow Volts	Initial Glow Amps	Ignition Volts	Ignition Amps	Remarks
1	Nickel	30"	.0088"	43	--	43	--	Chamber was fogged. Wire ignited and consumed.
3	Nickel	3-7/16"	.0088"	3-4	2.0-2.5	6	3.0	Wire broke - not consumed.
5	Copper	3-7/16"	.0126"	2	10	2	12.5	Wire consumed. Chamber clouded. Exit lines clogged.
6	Monel	3-7/16"	.0100"	5	1.9	11	3.5	Loss of continuity occurred, no ignition. Chamber was quite cloudy. Rotameter tube coated.
7	Tungsten	3-7/16"	.0120"	--	--	3	--	Ignition occurred with very bright white light. Flask walls coated.
8	S.S. 302	3-7/16"	.0200"	4-1/2	4.5	6	4.8	At initial glow, a pale yellow deposit appeared on glass. At ignition, voltage amps fell off from 5.2 to 4.8 before ignition. Wire slowly consumed and flask filled with yellow-orange smoke.
9	Molybdenum	3-7/16"	.0151"	--	--	2	--	Bright white light as wire was consumed. White residue and smoke in flask and exit.

TABLE 41

PRELIMINARY WIRE STUDY
IGNITIONS IN HELIUM ATMOSPHERE

	Material	Diam. (inches)	Length (inches)	Burnout Volts	Burnout Amps	Calc. Temp. °C	M.P. °C
1.	Nickel	.0088	5-1/2	16.6	7.2	1450	1450
2.	Nickel	.0150	11-3/4	19.0	11.5	1465	1450
3.	Molybdenum	.0151	11-3/4	37	15	3108*	2620
4.	Tungsten	.0120	11-3/4	85	17	3537	3370
5.	Monel 400	.0100	11-3/4	28	5.9	1270	1330
6.	S.S. 302	.0200	11-3/4	24	11.5	1090*	1400
7.	Copper	.0126	11-1/2	6-1/2-7	17	1110-1205**	1083
8.	Iron	.0090	11-3/4	43.5	4.7	1535	1535
9.	Nickel	.0150	11-3/4	17	10.5	1420	1450
10.	S.S. 302	.0200	11-3/4	26	12.3	1140*	1400
11.	Iron	.0090	11-3/4	46.8	4.75	1530	1535
12.	Tungsten	.0120	11-3/4	85	17	3537*	3370
13.	Copper	.0126	11-3/4	6-1/2-7	17	1125-1205**	1083

*The error could be a combination of poor resistivity-temperature data or lack of precision in our measurements. However, more accurate data was obtained and used in our precise experimental work performed later.

**A precise amperage reading could not be obtained. Reading estimated between 6-1/2 and 7 Amps and temperatures calculated for both limits.

TABLE 42

WIRE STUDY
IGNITION IN HELIUM

Run	Material	Form[a]	Wire Length (in.)	Diam. (in.)	Volt. Rate	Burnout Volts	Burnout Amps	Calc. Temp. °C
1	Nickel	1/8 coil	11-3/4	.0150	1.667	Not burned out		
2A[b]	Nickel	1/8 coil	11-3/4	.0150	3.33	Not burned out		
2B[b]	Nickel	1/8 coil	11-3/4	.0150	.667	Not burned out		
3	Nickel	1/8 coil	11-3/4	.0150	.667	Not burned out		
4	Nickel	1/8 coil	11-3/4	.0150	1.667	16.7	10.95	1305
5	S.S. 302	1/4 coil	11-3/4	.0200	1.667	18.6	10.1[c]	1220
6	S.S. 302	1/4 coil	11-3/4	.0200	1.667	20.2	10.8	1310
7	Nickel	1/4 coil	11-3/4	.0150	1.667	14.9	9.8	1315
8	Nickel	1/2 coil	17-3/4	.0150	1.667	24.4	10.65	1290
9	S.S. 302	1/2 coil	17-3/4	.0200	1.667	29.9	10.35	1480
10	Copper	1/4 coil	11-3/4	.0126	1.667	5.75	17.5	1020
11	Copper	1/2 coil	17-3/4	.0126	1.667	6.60	15.7	875
12	Copper	1/2 coil	17-3/4	.0126	1.667	8.30	17.4	1000
13	Copper	1 coil	17-3/4	.0126	1.667	Coil shorted		----
14	Copper	1/4 coil	17-3/4	.0126	1.667	7.33	16.7	850
15	Copper	1/4 coil	7-3/4	.0126	1.667	4.33	19.4	1055
16	Monel	1/4 coil	11-3/4	.0100	1.667	23.4	5.98	745
17	Monel	1/4 coil	7-3/4	.0100	1.667	18.7	7.03	855
18	Monel	1/8 coil	11-3/4	.0100	1.667	23.25	5.90	790
19	Monel	1/8 coil	11-3/4	.0100	3.33	22.5	5.66	820
20	Monel	wire	3.45	.0100	1.667	8.4	7.30	870
21	Monel	wire	3.45	.0100	1.667	9.1	7.85	880
22	Monel	wire	4.0	.0100	1.667	9.85	7.45	840
23	Monel	wire	4.0	.0100	1.667	9.80	7.35	870
24	Iron	wire	4.0	.0090	1.667	19.05	4.13	>MP
25	Iron	wire	3.43	.0090	1.667	15.1	5.83	1190
26	Tungsten	wire	3.42	.0120	1.667	23.4	18.9	2987
27	Iron	wire	3.48	.0090	1.667	15.65	5.81	1260
28A[b]	Molybdenum	wire	3.46	.0151	1.667	10.22	>20.0[d]	>MP
28B[b]	Molybdenum	wire	3.46	.0151	1.667	10.80	>20.0[d]	>MP
32	Nickel	wire	3.45	.0150	1.667	7.0	15.3	1360
80	Monel	1/8 coil	5.75	.0100	3.33	13.2	6.93	840
81	Monel	1/8 coil	5.75	.0100	3.33	12.3	6.50	830[e]
82	Monel[f]	1/8 coil	5.75	.0100	3.33	9.40	4.85	910
83	Monel[g]	1/8 coil	5.75	.0100	3.33	5.38	2.74	960

TABLE 42

(Continued)

Run	Material	Form[a]	Wire Length (in.)	Diam. (in.)	Volt. Rate	Burnout Volts	Burnout Amps	Calc. Temp. °C
84	Monel	1/8 coil	5.75	.0100	(h)	13.5	7.02	890
85A	Monel	1/8 coil	5.75	.0100	3.33	12.90	6.76	850[e]
85B	Monel	1/8 coil	5.75	.0100	3.33	13.6	7.10	870
86	Monel	1/8 coil	5.75	.0100	1.67	13.4	6.95	890

(a) Number indicates diameter in inches of the mandrel on which the coil was formed.

(b) A & B indicates the same wire was run twice, the first run, "A", did not ignite.

(c) Estimated.

(d) Reached upper limit of amperage capacity without igniting.

(e) Run terminated just before burnout for resistance measurements.

(f) Run made in Argon.

(g) Run made in Vacuum.

(h) Rate to 6 amps = 3.33 volts/min.; from 6-6-1/2 amps, 1.67 volts/min.; from 6-1/2 amps to burnout, 0.167 volts/min.

TABLE 43

WIRE STUDY
IGNITION IN OF_2

Run	Material	Wire (Inches) Length	Wire (Inches) Diam.	Power Volts/min.	Ignition Volts	Ignition Amps	Calc. Temp. °C	(a) Corrected Temp. °C
30B	Nickel	3.43	.0150	1.667	4.65	10.55	1280	----
31	Nickel	3.45	.0150	1.667	4.75	10.65	1290	1220
33	Nickel	3.44	.0150	1.667	4.75	10.66	1305	1220
34	Nickel	3.44	.0150	3.33	4.75	10.9	1270	1225
35	Nickel	3.44	.0150	3.33	4.80	10.9	1285	1240
36	Nickel	5.75	.0150	3.33	8.00	10.6	1330	1265
37	Nickel	5.75	.0150	3.33	7.6	10.35	1270	1210
38	Monel	3.43	.0100	3.33	7.4	5.30	1650	825
39	Monel	3.43	.0100	3.33	7.4	5.35	1620	795
40	Monel	5.75	.0100	3.33	12.0	4.95	>1680	820
41	Monel	5.75	.0100	3.33	12.1	5.0	>1680	810
42	Copper	3.43	.0126	3.33	0.76	12.9	615	----
43	Copper	3.43	.0126	3.33	0.77	12.83	630	----
44	Copper	5.75	.0126	3.33	1.29	11.72	705	----
45	Copper	5.75	.0126	3.33	1.29	11.82	700	----
46	Copper	5.75	.0126	1.667	1.249	11.64	680	----
47	S.S. 302	3.41	.0200	3.33	3.34	6.58	930	880
48	S.S. 302	3.41	.0200	3.33	3.30	6.51	920	880
49	S.S. 302	5.75	.0200	3.33	5.77	6.54	1060	960
50	S.S. 302	5.75	.0200	3.33	5.80	6.53	1090	980
51	Molybdenum	3.42	.0151	3.33	.551	6.27	290	----
52	Molybdenum	3.42	.0151	3.33	.557	6.28	295	----
53	Molybdenum	5.75	.0151	3.33	.90	5.55	327	----
54	Molybdenum	5.75	.0151	3.33	.875	5.57	315	----
55	Tungsten	3.41	.0120	3.33	.670	4.66	275	----
56	Tungsten	3.41	.0120	3.33	.625	4.56	255	----
57	Tungsten	5.75	.0120	3.33	.975	4.15	273	----
58	Tungsten	5.75	.0120	3.33	1.00	4.20	280	----
59	Iron	3.42	.0090	3.33	3.50	2.30	623	----
60	Iron	3.41	.0090	3.33	3.50	2.30	623	----
61	Iron	5.75	.0090	3.33	6.50	2.30	665	----
63	Iron	5.75	.0090	3.33	6.50	2.32	665	----
64B	Nickel	3.41	.0088	3.33	7.70	4.72	>MP	----
65	Nickel	3.41	.0088	3.33	6.8	5.23	1365	1270
66	Monel	3.43	.0100	3.33	7.5	5.31	>MP	885

TABLE 43

(Continued)

Run	Material	Wire (Inches) Length	Wire (Inches) Diam.	Power Volts/min.	Ignition Volts	Ignition Amps	Calc. Temp. °C	Corrected Temp. °C
67	Nickel	3.41	.0088	3.33	6.70	5.27	1285	1230
68	Nickel	5.75	.0088	3.33	10.5	5.12	1200	1110
69	Nickel	5.75	.0088	3.33	10.7	5.1	1250	1145
70	Nickel	5.75	.0088	3.33	11.0	5.13	1300	1190
73B	Monel	5.75	.0100	3.33	11.5	4.82	>MP	760
74	S.S. 302	5.75	.0200	1.667	6.0	6.45	1450	1050
75B	S.S. 302	5.75	.0200	3.33	6.0	6.60	1320	960
76	Monel	5.75	.0100	3.33	8.3	4.45	----	740[b]
77	Monel	5.75	.0100	3.33	8.3	4.50	----	705[b]
78	Monel	5.75	.0100	3.33	7.5	4.12	----	665[c]
79	Monel	5.75	.0100	3.33	12.0	4.95	----	740[d]
87	Nickel	5.75	.0150	3.33	6.9	10.0	----	1100[d]
88	Nickel	5.75	.0150	3.33	7.5	10.4	----	1175[d]
89	Nickel	5.75	.0088	3.33	11.35	5.13	----	1245[d]

(a) Corrections made for corrosion affect on wire diameter.

(b) Temperature at first reaction point not run to ignition.

(c) Run terminated before first reaction point.

(d) Run terminated before final ignition.

Note: Runs marked (b), (c), (d) were used for wire resistance measurements after OF_2 exposure.

TABLE 44

NICKEL "A" WIRE
IGNITION IN OF_2

Run	Length	Diam.	Rate Volts/min.	Corrected Ignit. Temp. °C
31	3.45	.0150	1.667	1220
33	3.44	.0150	1.667	1220
34	3.44	.0150	3.33	1225
35	3.44	.0150	3.33	1240
36	5.75	.0150	3.33	1265
37	5.75	.0150	3.33	1210
65	3.41	.0088	3.33	1270
67	3.41	.0088	3.33	1230
68	5.75	.0088	3.33	1110
69	5.75	.0088	3.33	1145
70	5.75	.0088	3.33	1190
87	5.75	.0150	3.33	1100*
88	5.75	.0150	3.33	1175*
89	5.75	.0088	3.33	1245

*Not run to burnout.

TABLE 45

MONEL 400 WIRE

IGNITION IN OF_2

Run	Wire Length	1st Reaction Point				2nd Reaction Point			
		Volts	Amps	Resist.*	Temp.	Volts	Amps	Resist.*	Temp.
38	3.43	4.90	4.45	64.3	720	7.4	5.30	69.7	825
39	3.43	4.90	4.48	63.9	695	7.4	5.35	68.5	795
40	5.75	7.92	4.32	64.5	740	12.0	4.95	72.0	820
41	5.75	8.40	4.47	65.1	770	12.1	5.0	71.6	810
66	3.43	4.95	4.50	64.5	735	7.5	5.31	70.5	885
71	3.41	4.80	4.45	63.5	680	Not run to burnout.			
72	3.41	4.87	4.50	63.5	680	Not run to burnout.			
73	5.75	8.20	4.42	64.5	740	11.5	4.82	70.4	760
76	5.75	8.30	4.45	65.2	770	Not run to burnout.			
77	5.75	8.3	4.5	63.9	705	Not run to burnout.			
78	5.75	7.5	4.12	63.2	665**	Not run to burnout.			
79	5.75	8.2	4.5	63.3	670	12.0	4.95	64.6	740

*Resistivity in microhm-cm.

**Run terminated just below 1st reaction point.

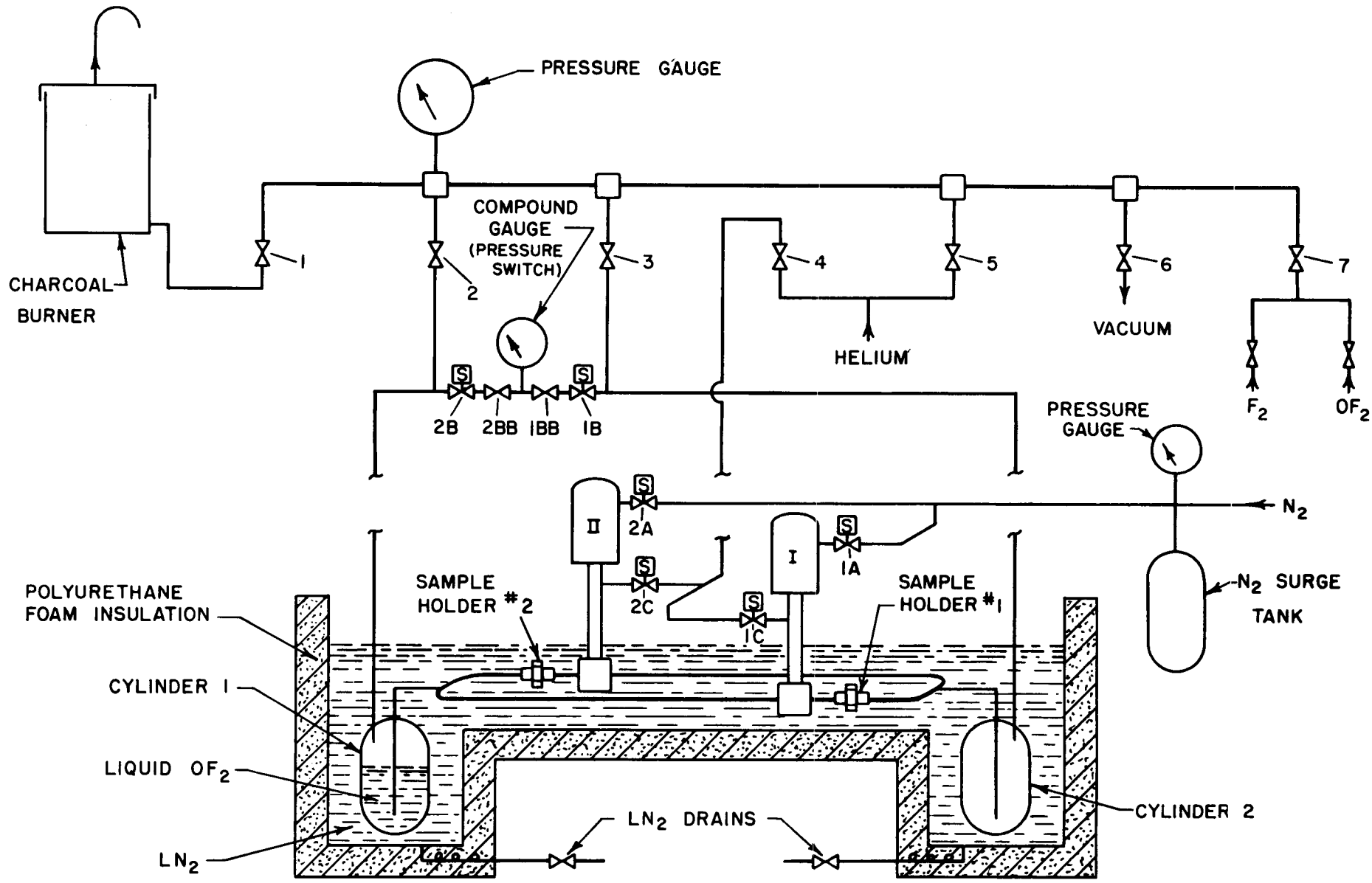
Note: All temperatures have been corrected for corrosion.

TABLE 46

WIRE STUDY IN OF_2

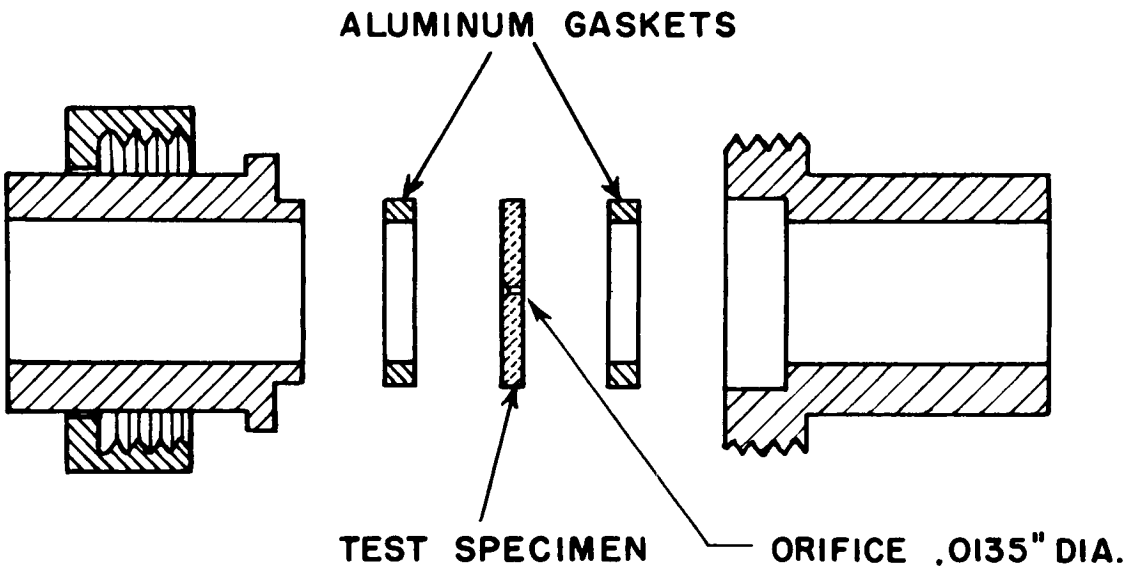
AVERAGE IGNITION TEMPERATURE

Material	Initial Diam.	Initial Length	Avg. Ignition Temp. °C
Nickel	.0150	3.44	1226
Nickel	.0150	5.75	1238
Nickel	.0088	3.41	1250
Nickel	.0088	5.75	1172
Monel	.0100	3.43	835
Monel	.0100	5.75	783
S.S. 302	.0200	3.41	880
S.S. 302	.0200	5.75	988
Copper	.0126	3.43	623
Copper	.0126	5.75	695
Iron	.0090	3.41	623
Iron	.0090	5.75	665
Molybdenum	.0151	3.42	292
Molybdenum	.0151	5.75	321
Tungsten	.0120	3.41	265
Tungsten	.0120	5.75	276



OF_2 DYNAMIC SETUP

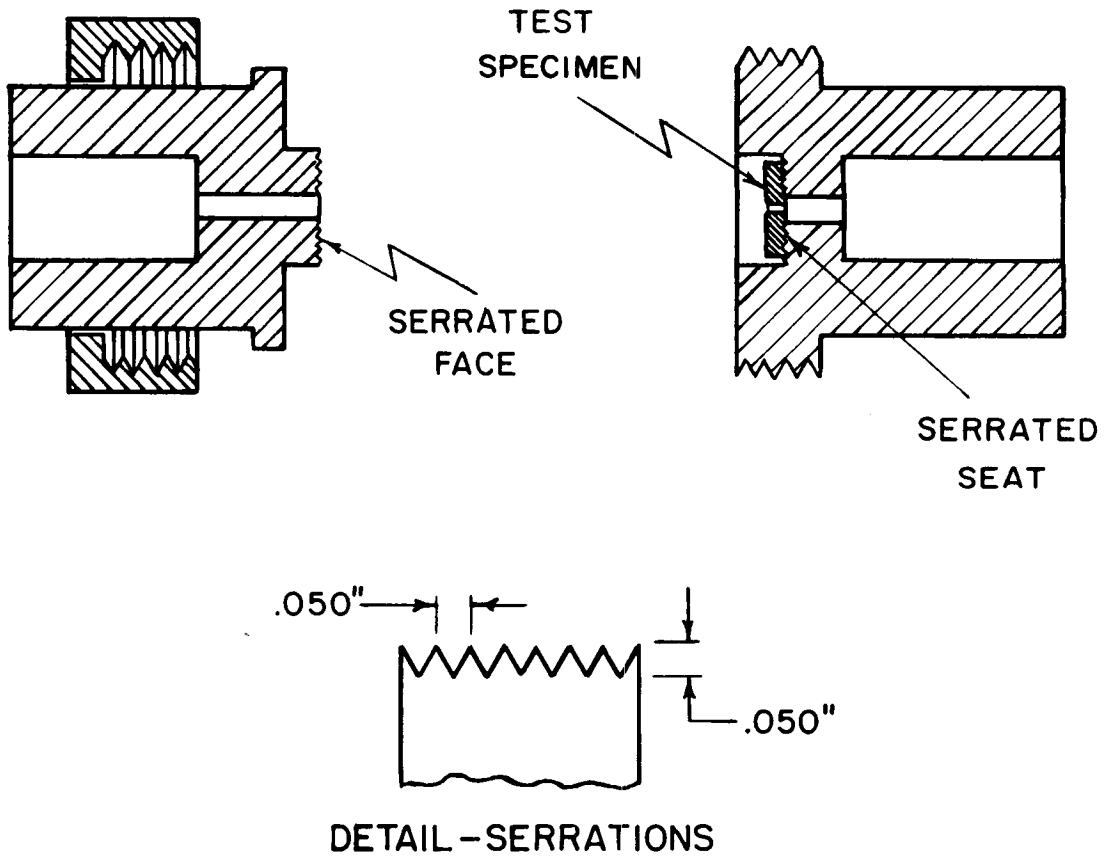
FIGURE 1.



SPECIMEN HOLDER – OF_2 DYNAMIC STUDY.

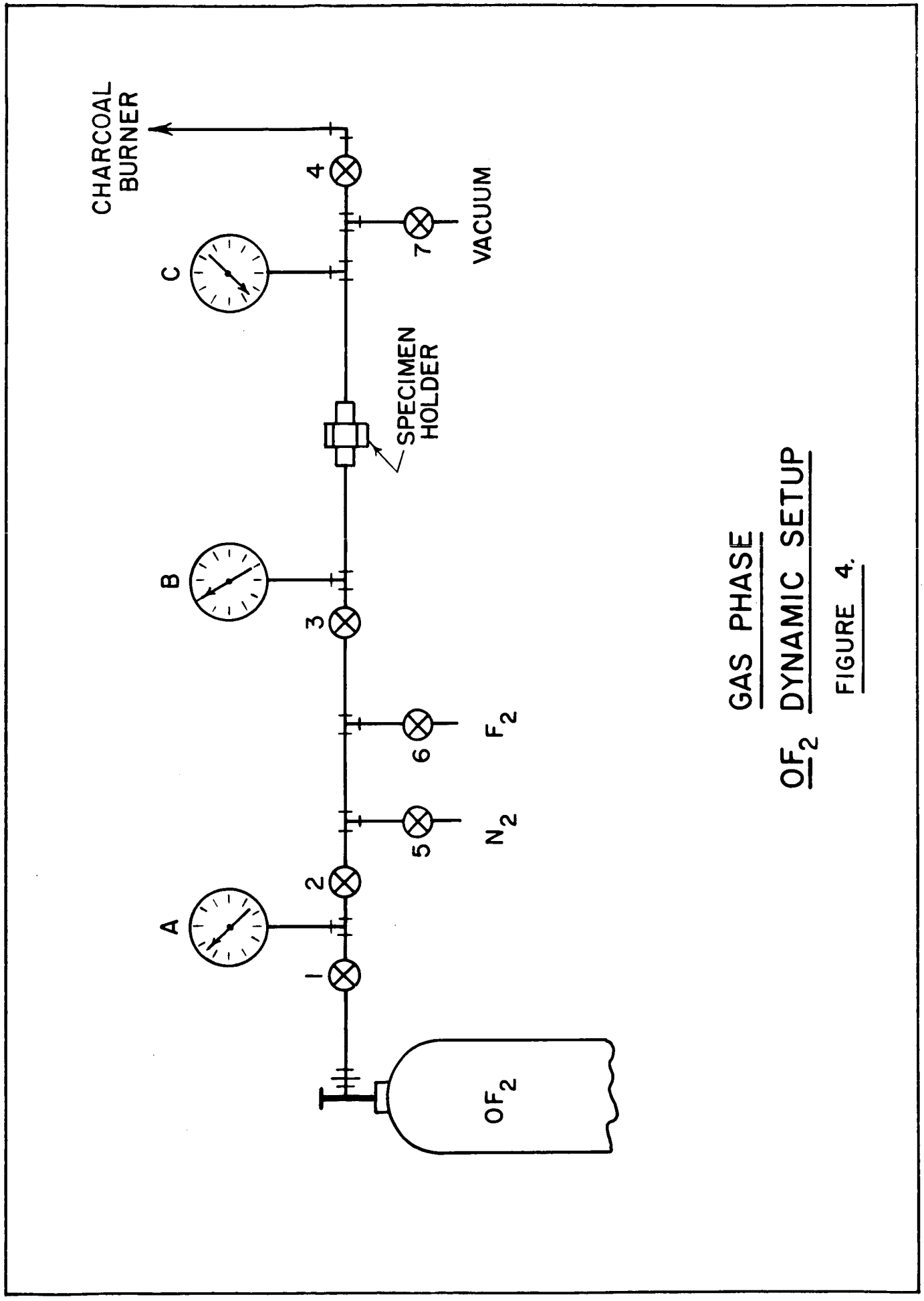
FIGURE 2.

S.H. G.C.R.L. 4-21-66



SPECIMEN HOLDER – OF_2 DYNAMIC STUDY

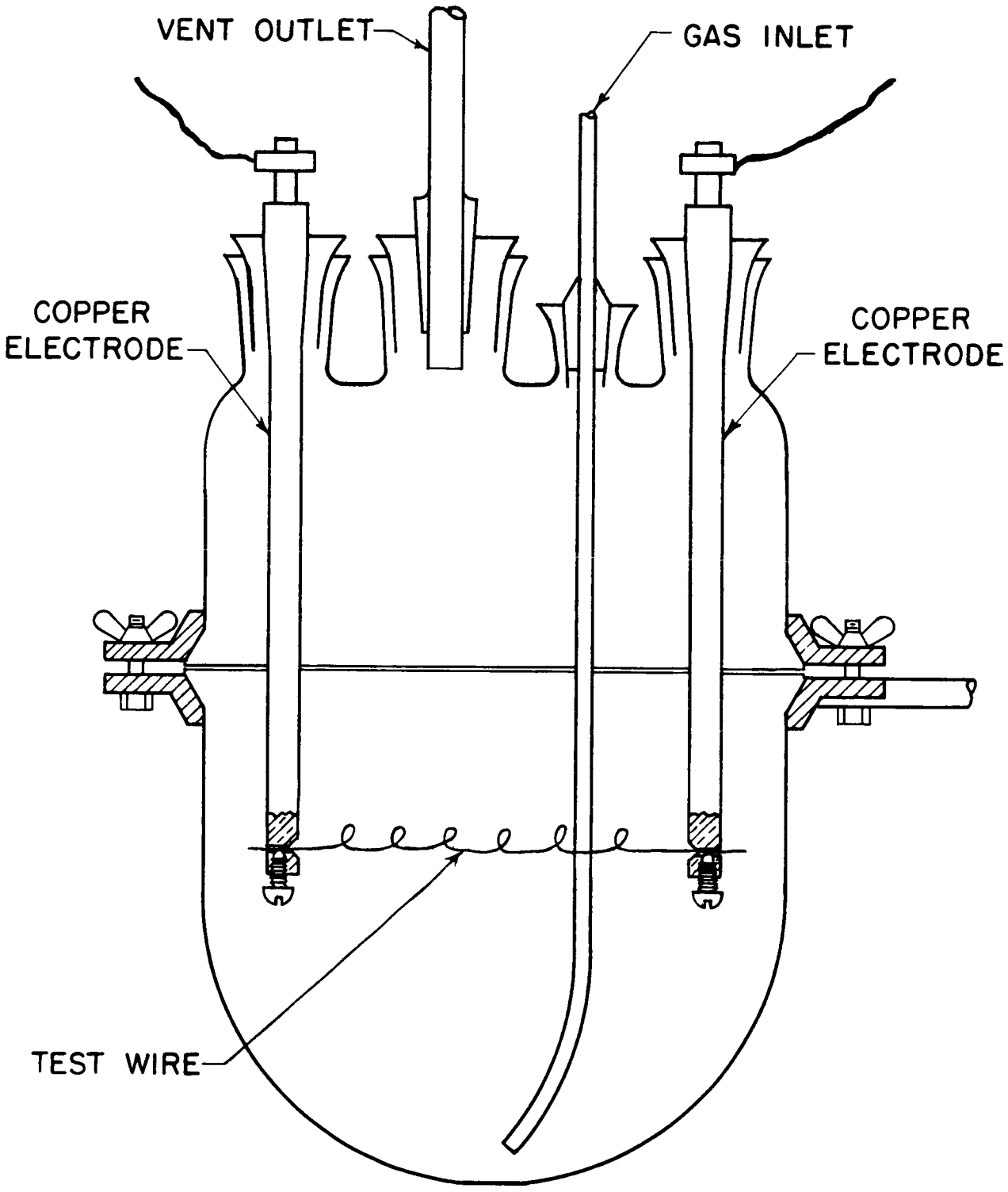
FIGURE 3.



GAS PHASE

OF_2 DYNAMIC SETUP

FIGURE 4.



OF_2 WIRE IGNITION TEST SETUP

FIGURE 5.

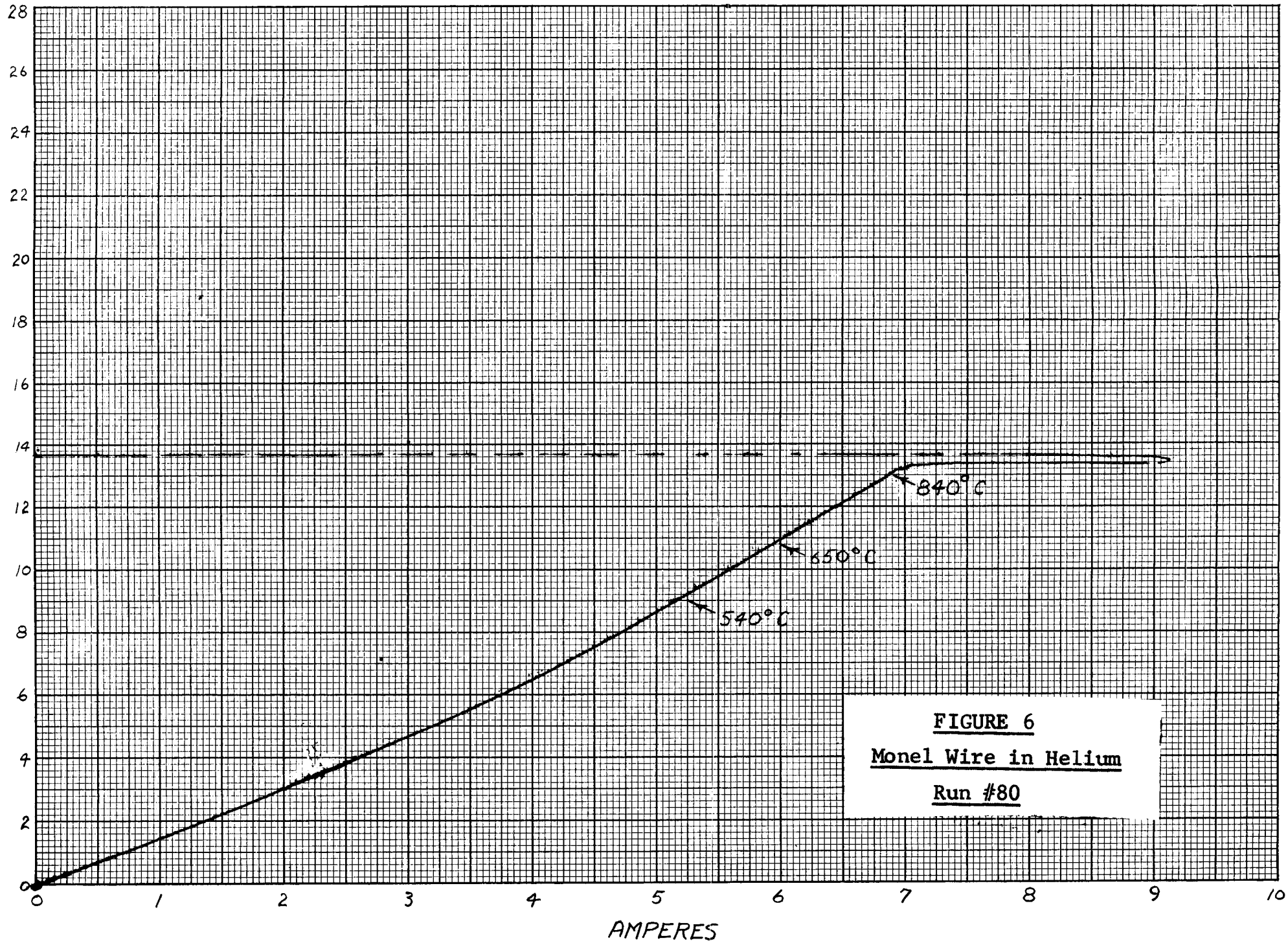


FIGURE 6

Monel Wire in Helium

Run #80

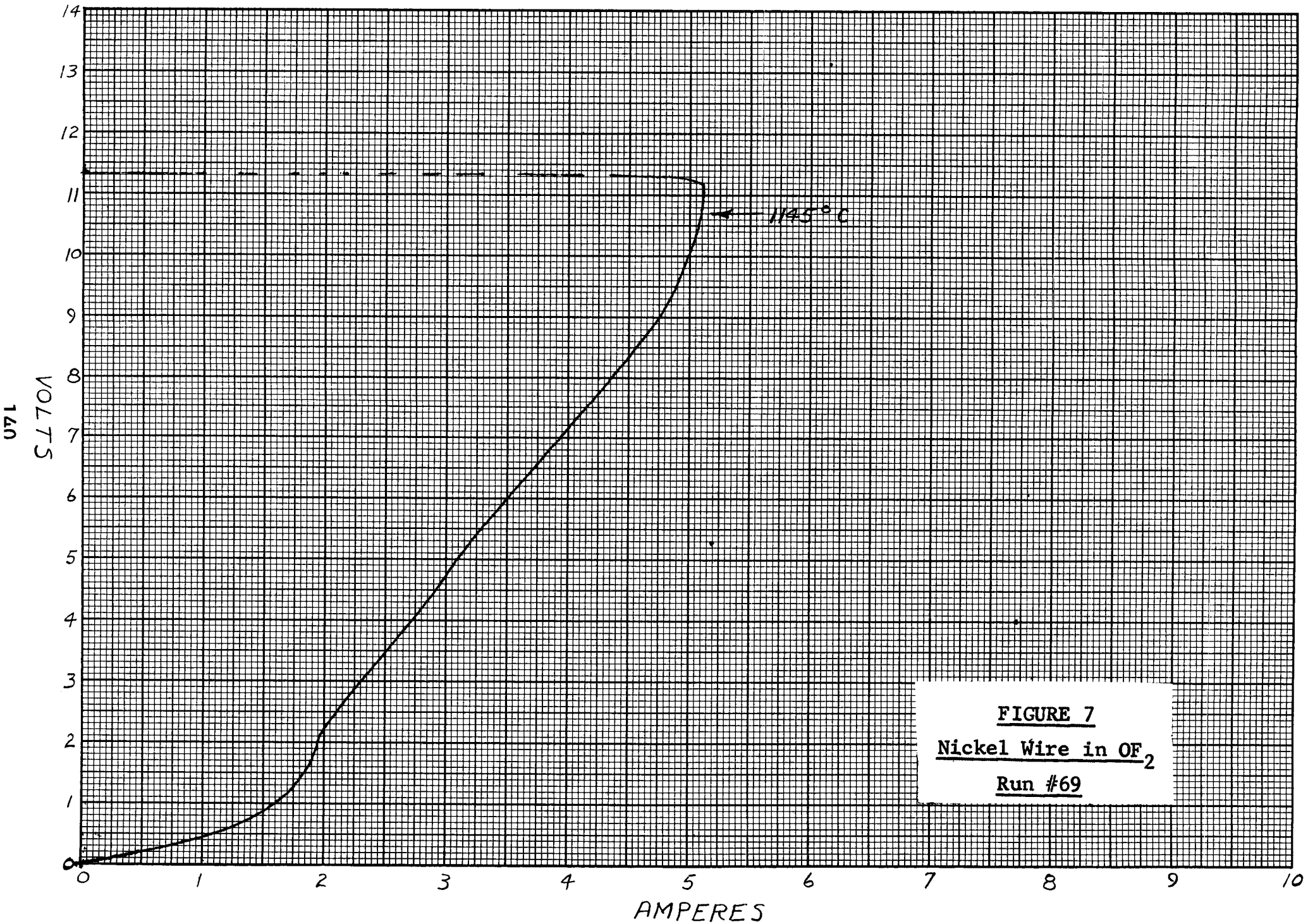


FIGURE 7

Nickel Wire in OF_2

Run #69

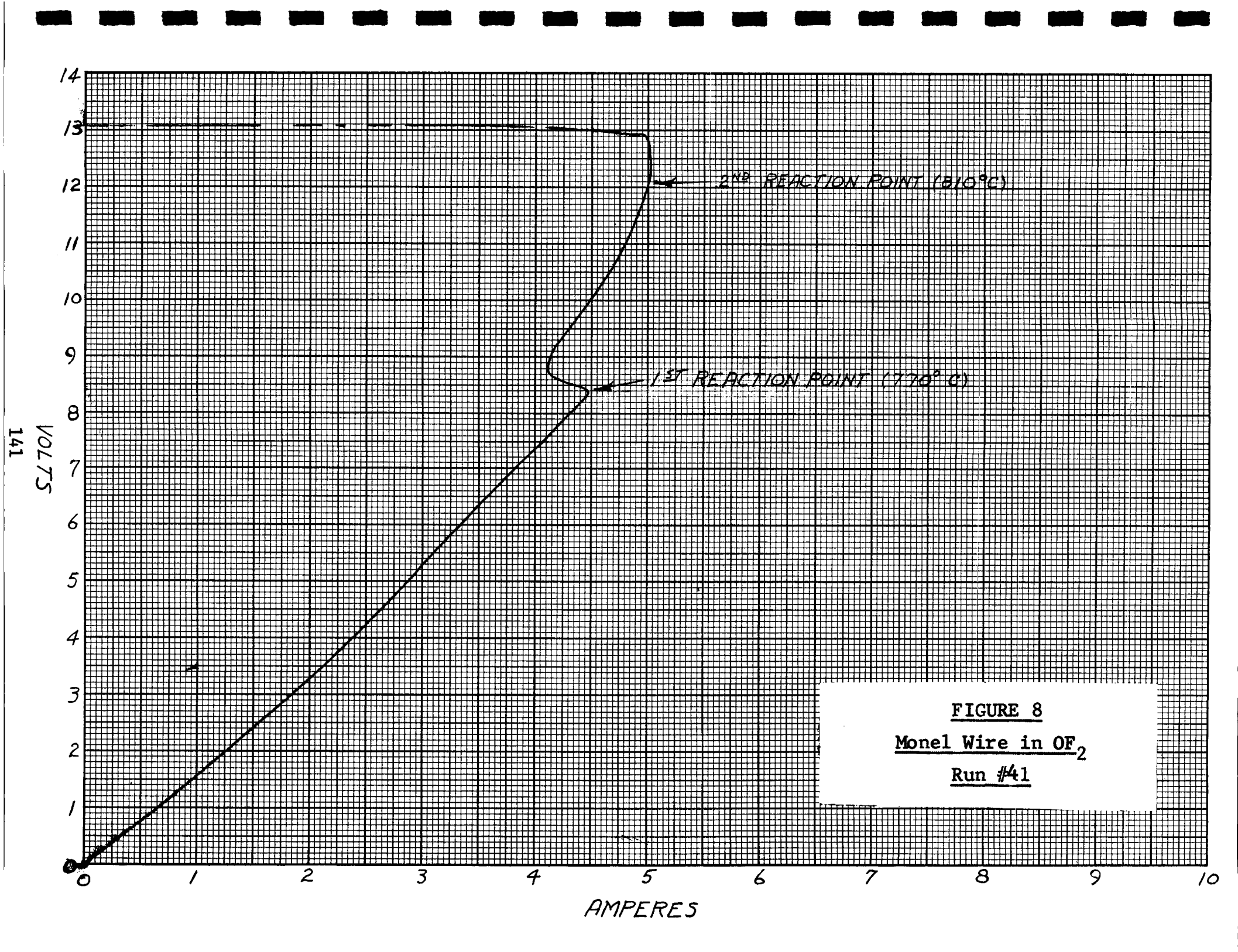


FIGURE 8

Monel Wire in OF_2

Run #41

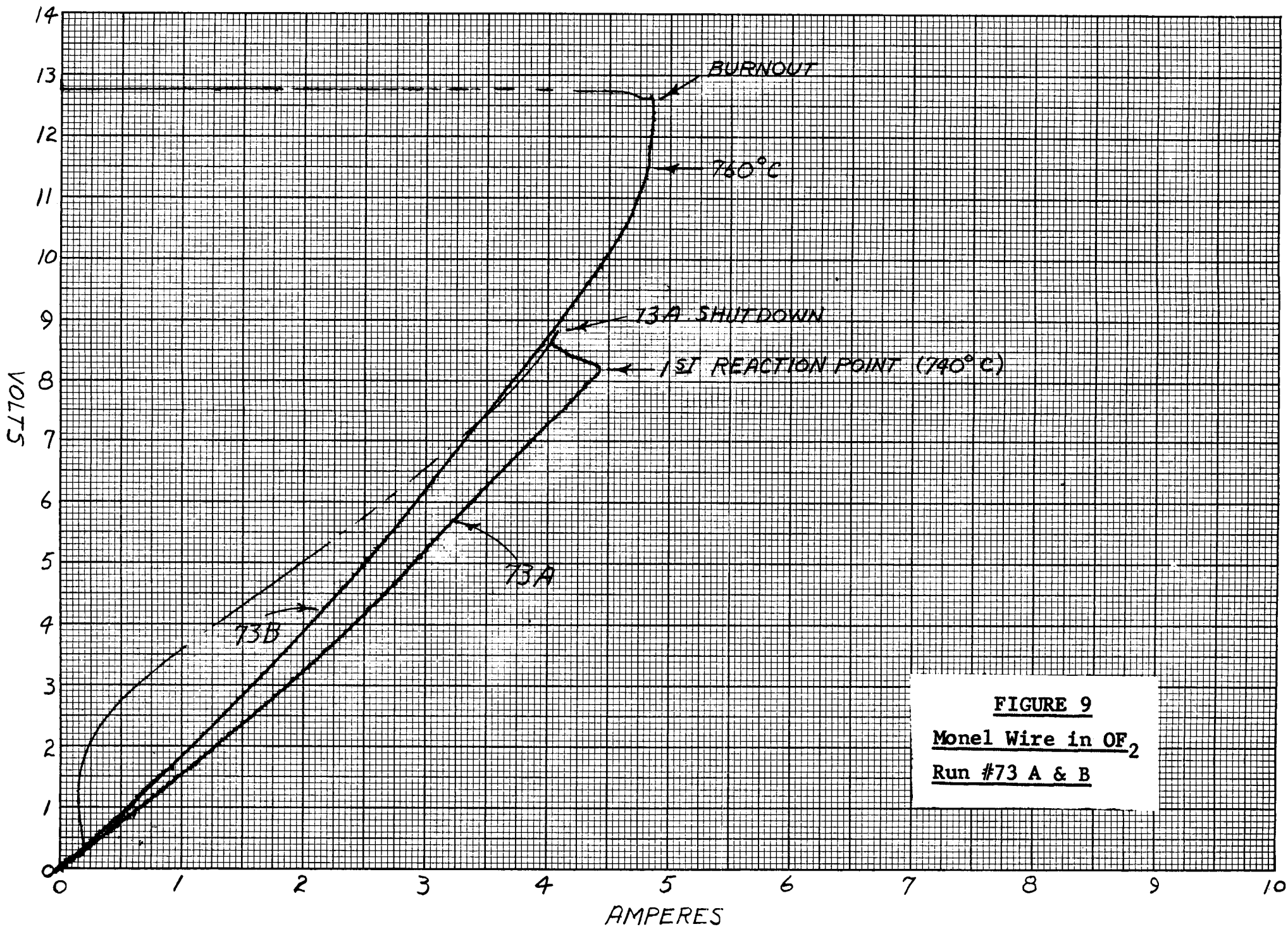


FIGURE 9

Monel Wire in OF_2

Run #73 A & B

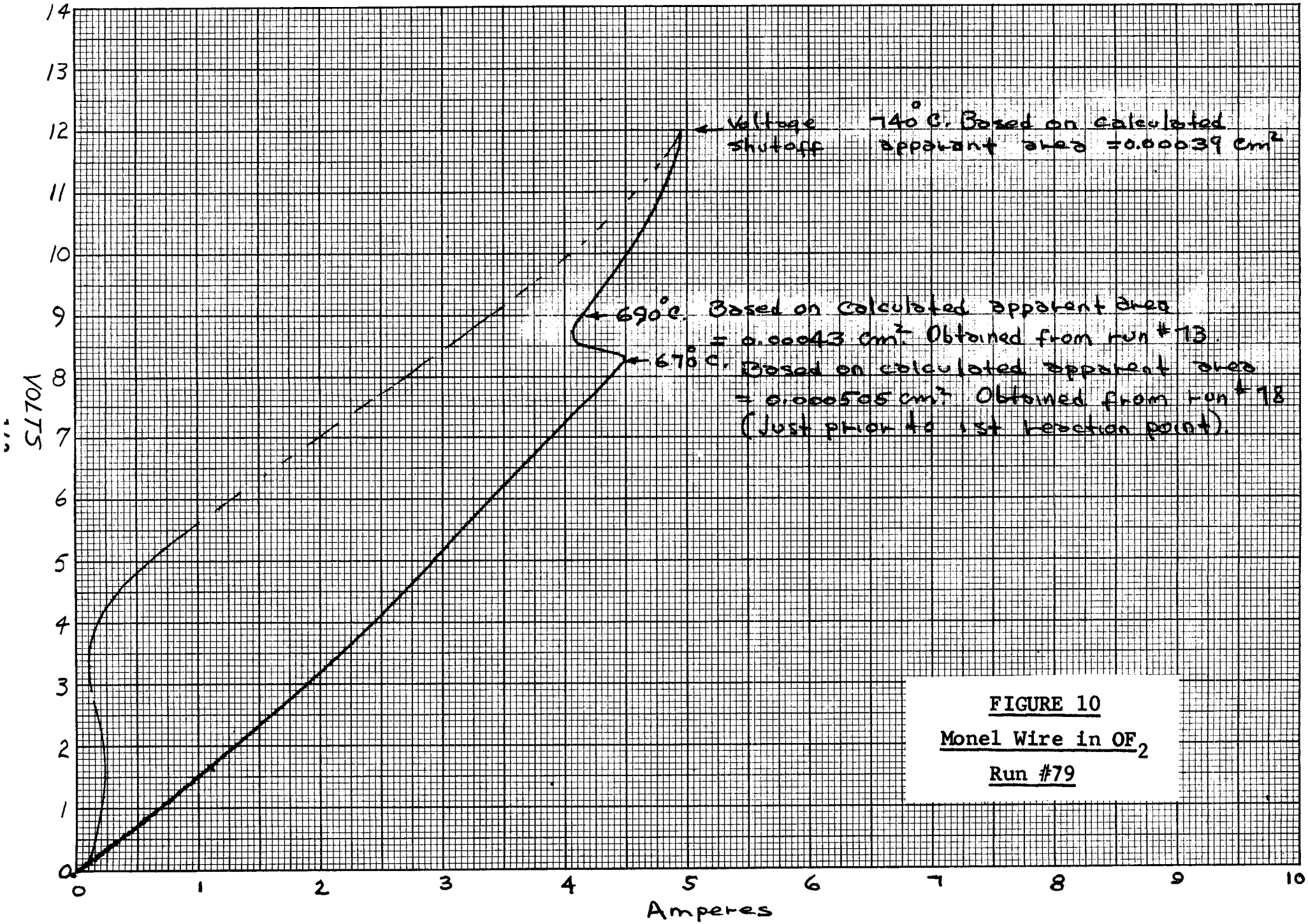


FIGURE 10

Monel Wire in OF_2

Run #79

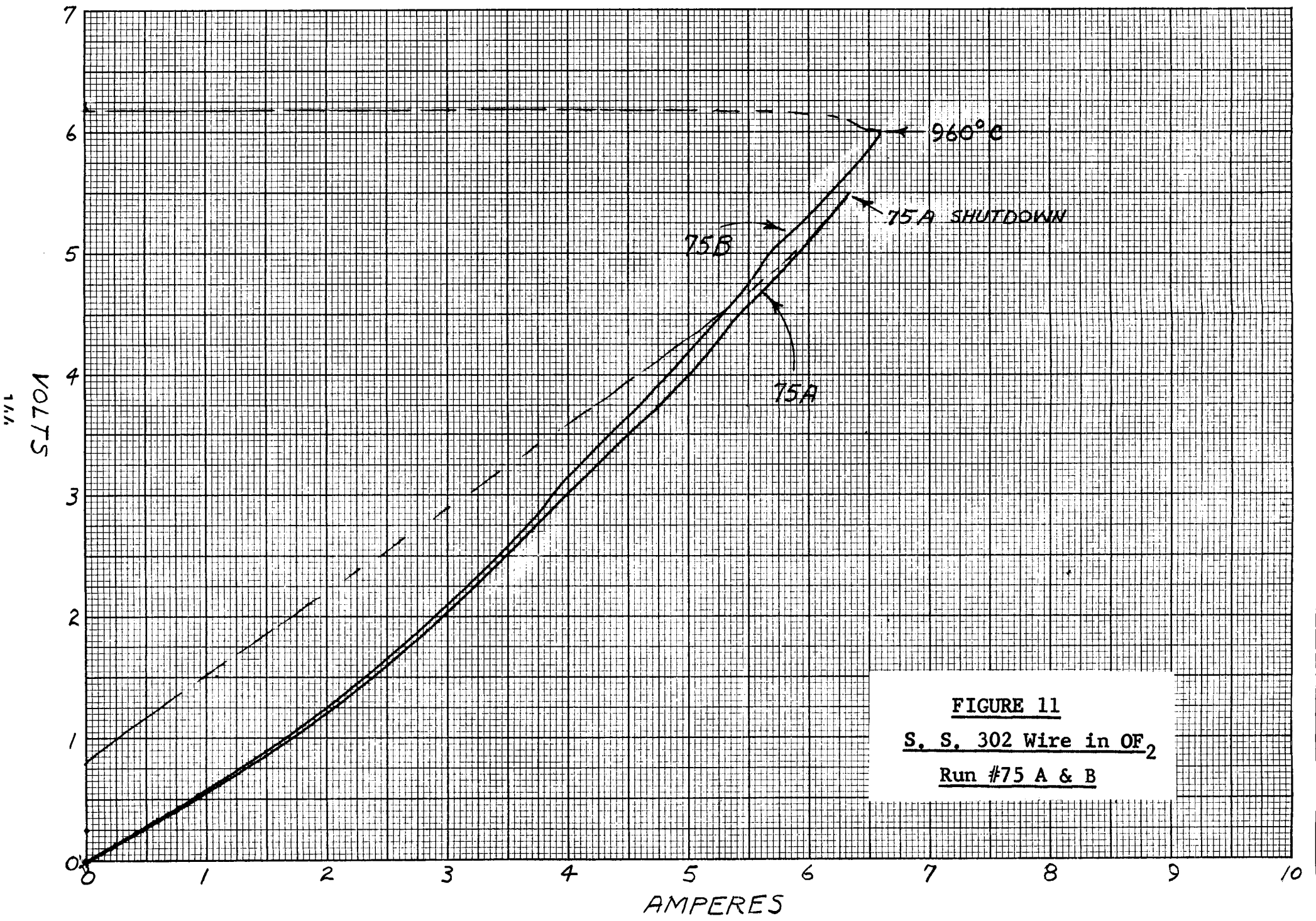


FIGURE 11

S. S. 302 Wire in OF_2

Run #75 A & B

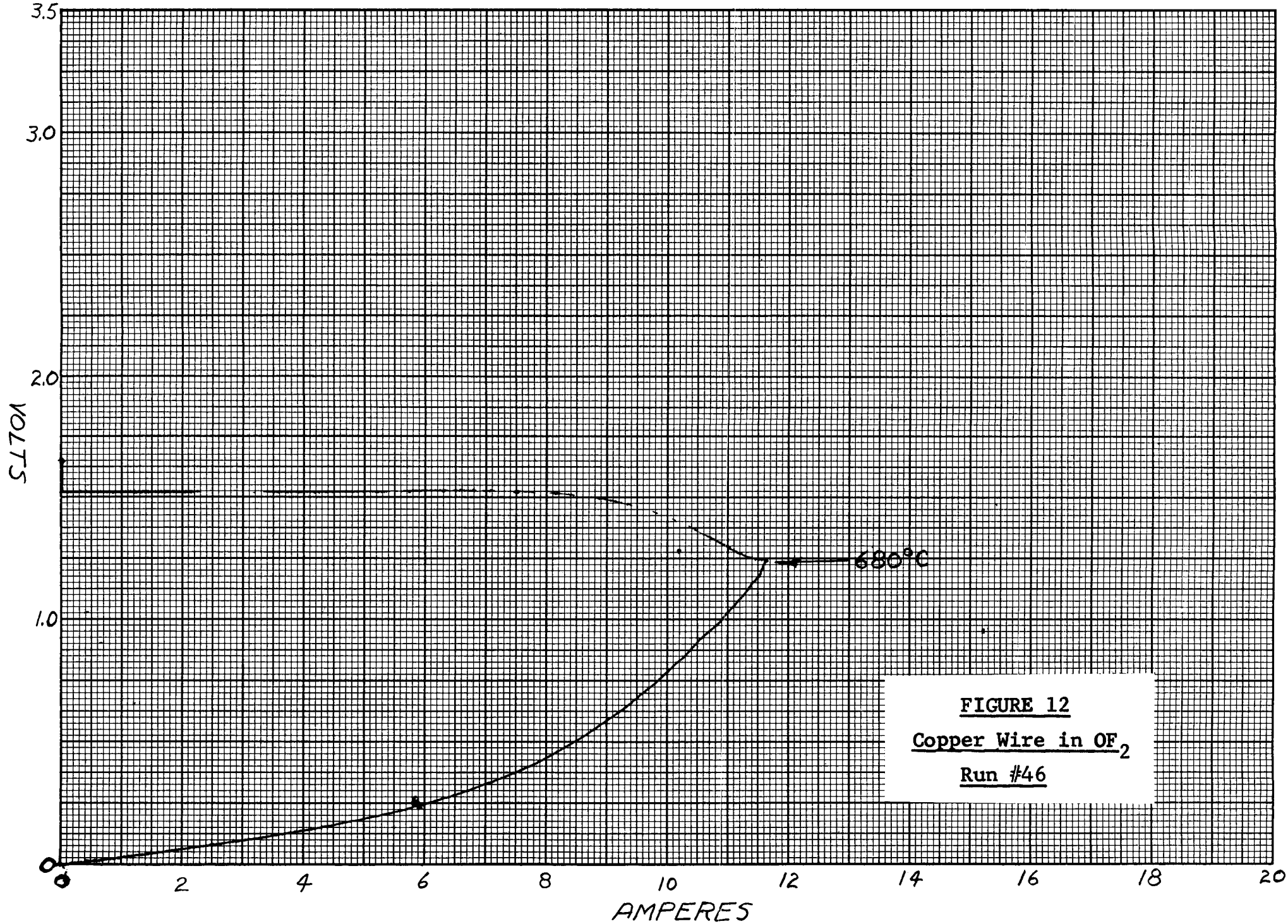


FIGURE 12

Copper Wire in OF_2

Run #46

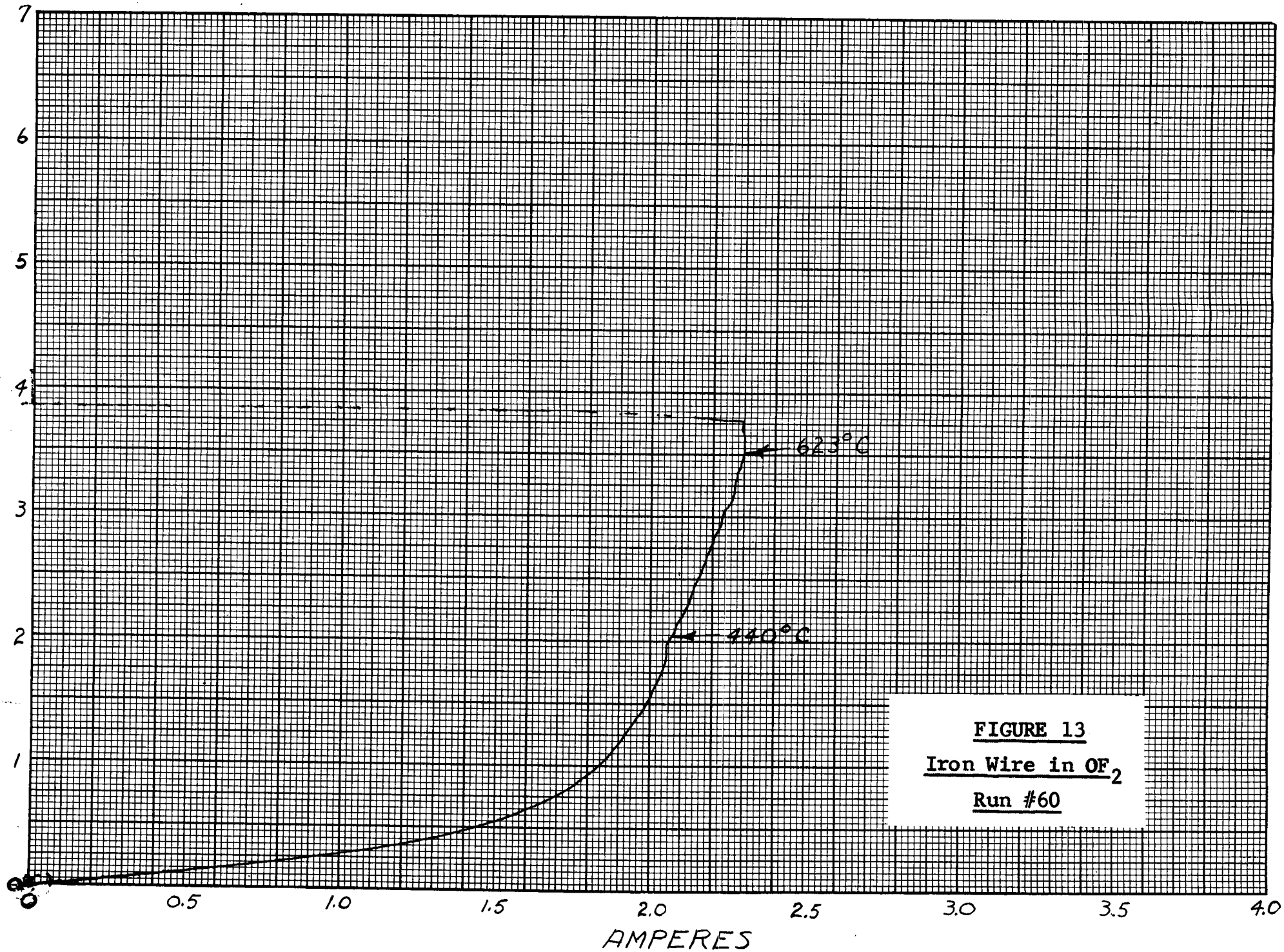


FIGURE 13

Iron Wire in OF_2

Run #60

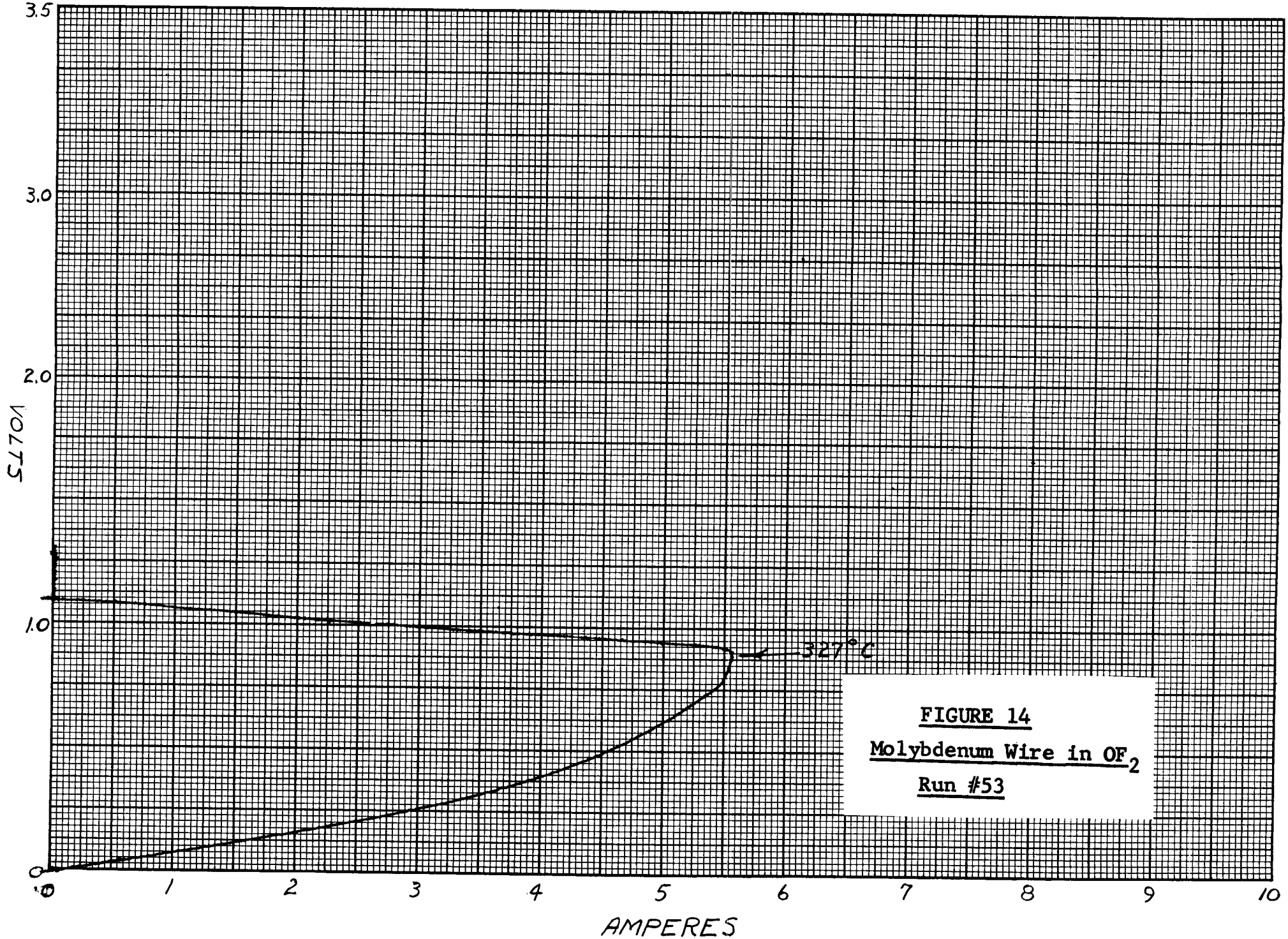


FIGURE 14

Molybdenum Wire in OF_2

Run #53

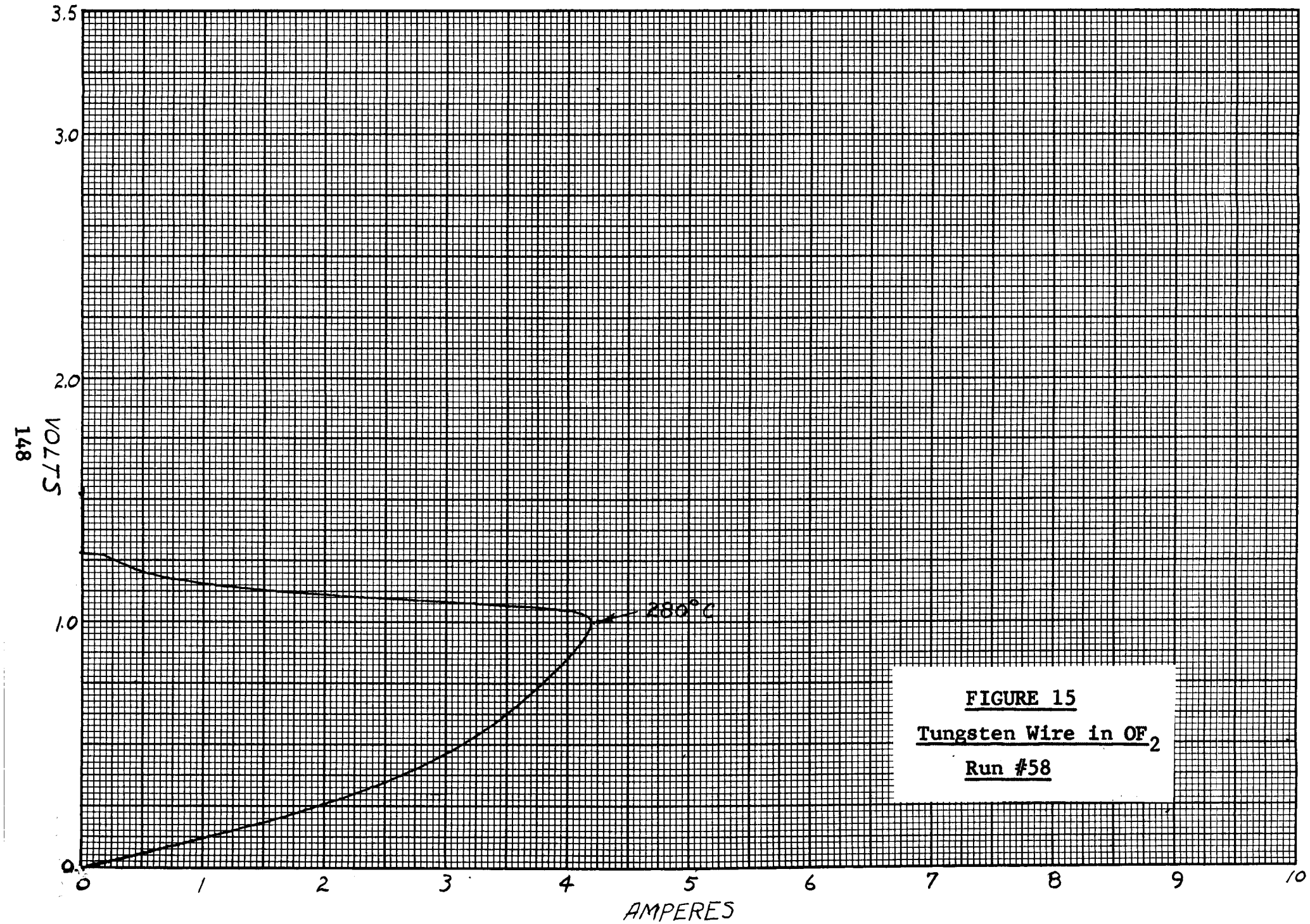
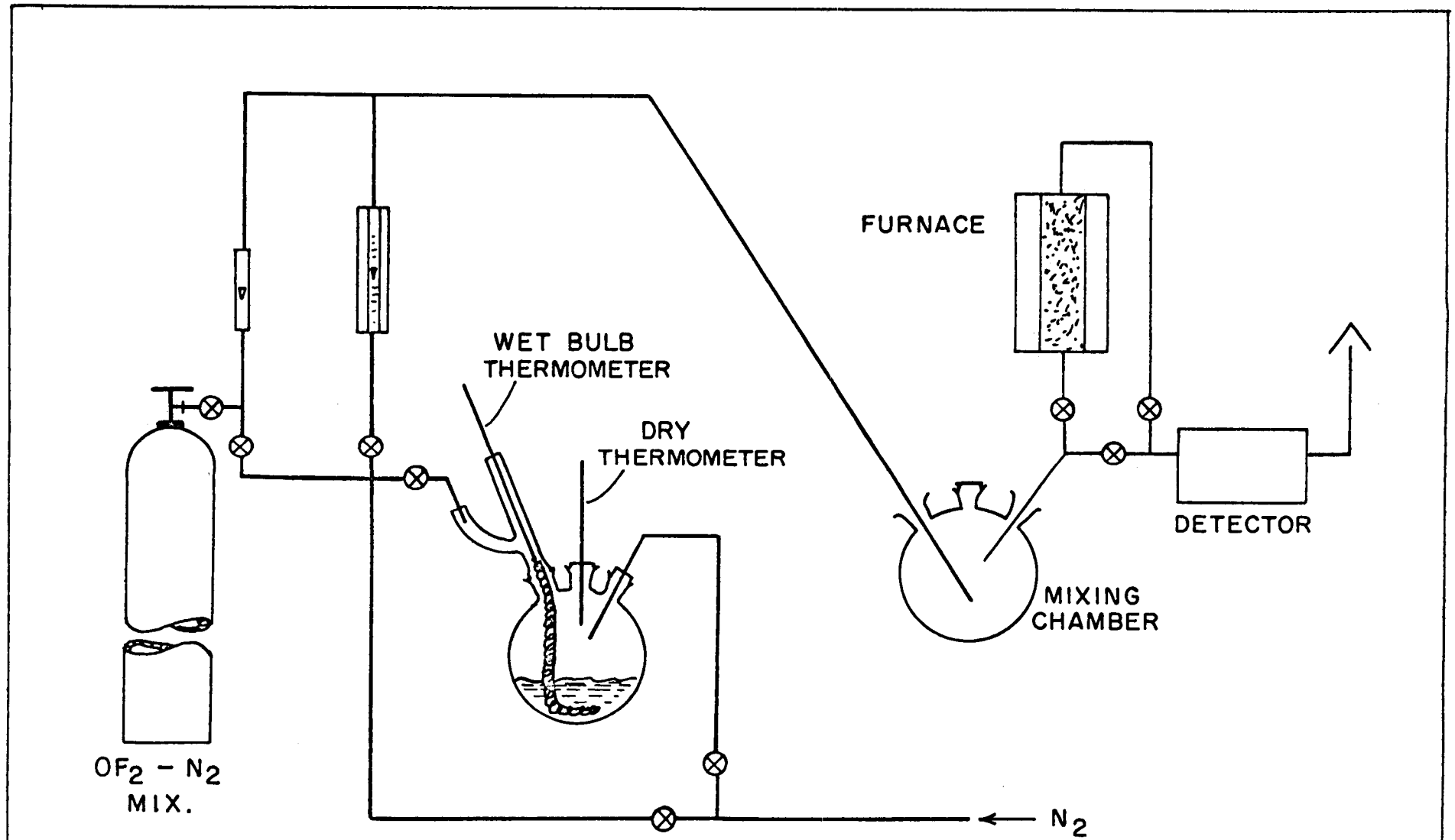


FIGURE 15

Tungsten Wire in OF_2

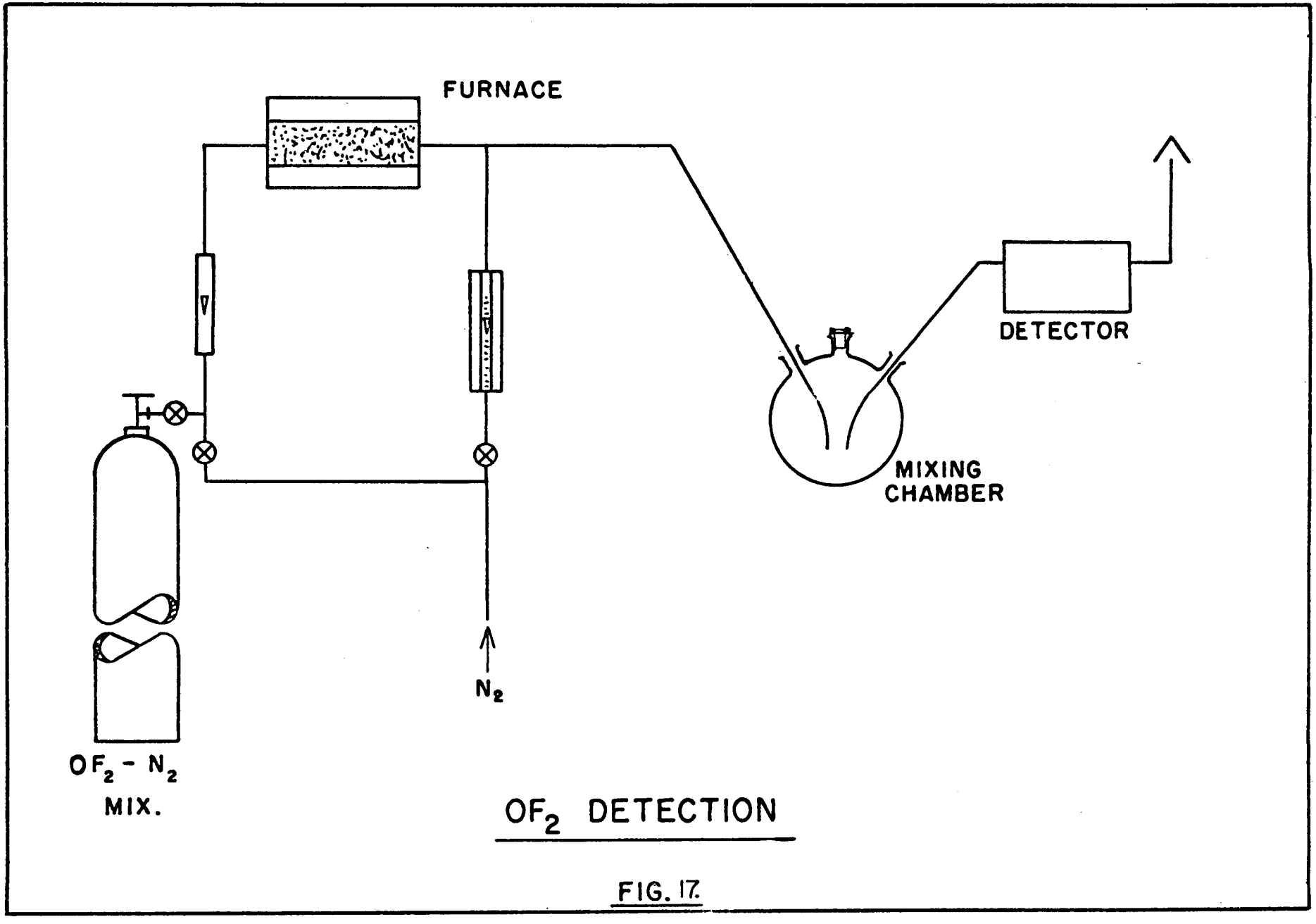
Run #58



OF_2 DETECTION

FIG. 16.

S.H. G.C.R.L. 9-26-66



OF_2 DETECTION

FIG. 17.

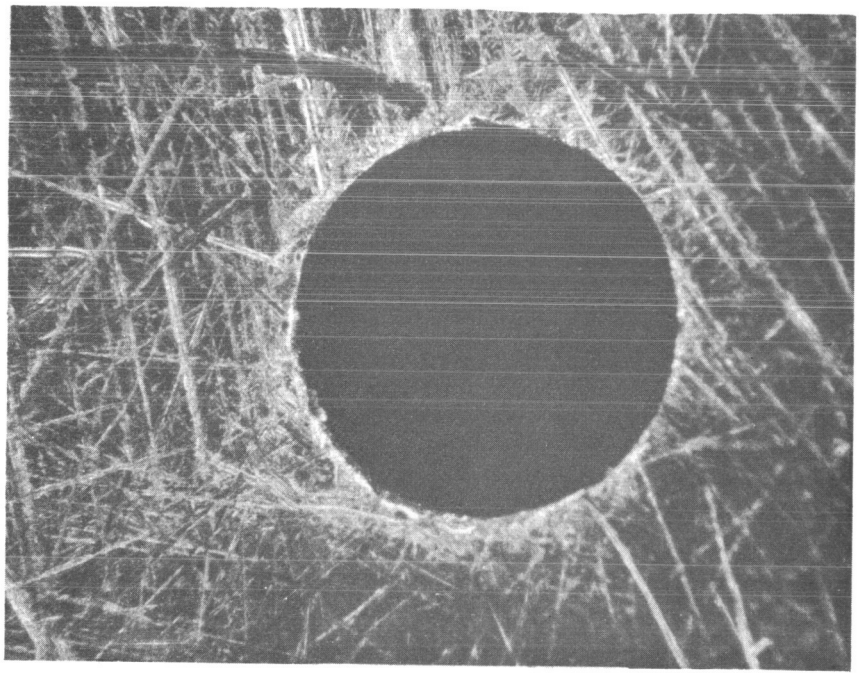


Exhibit 1A - Monel orifice before exposure to liquid OF_2. 150X

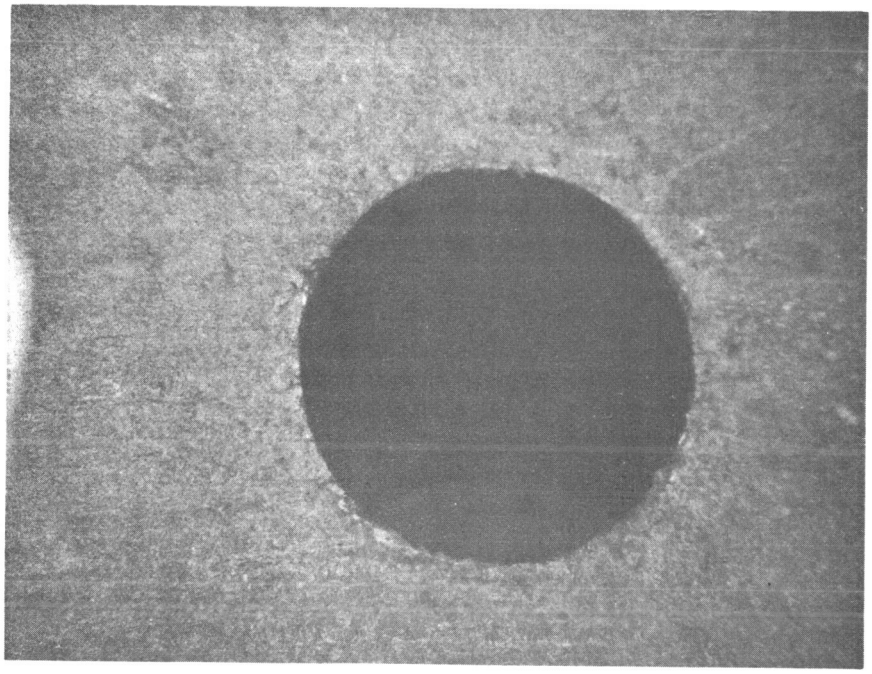


Exhibit 1B - Monel orifice after exposure to liquid OF_2. 150X

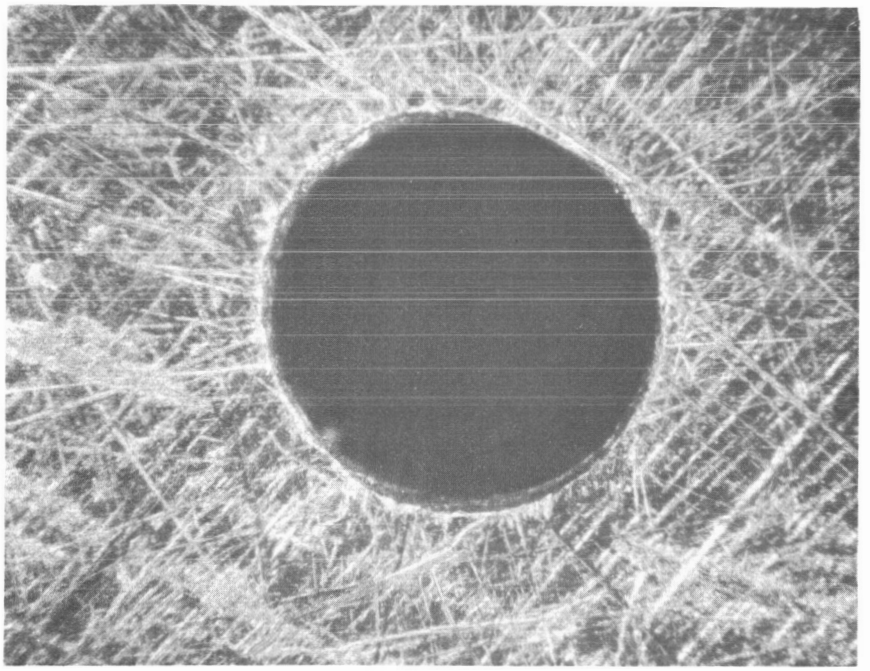


Exhibit 2A - Nickel orifice before exposure to liquid OF_2. 150X

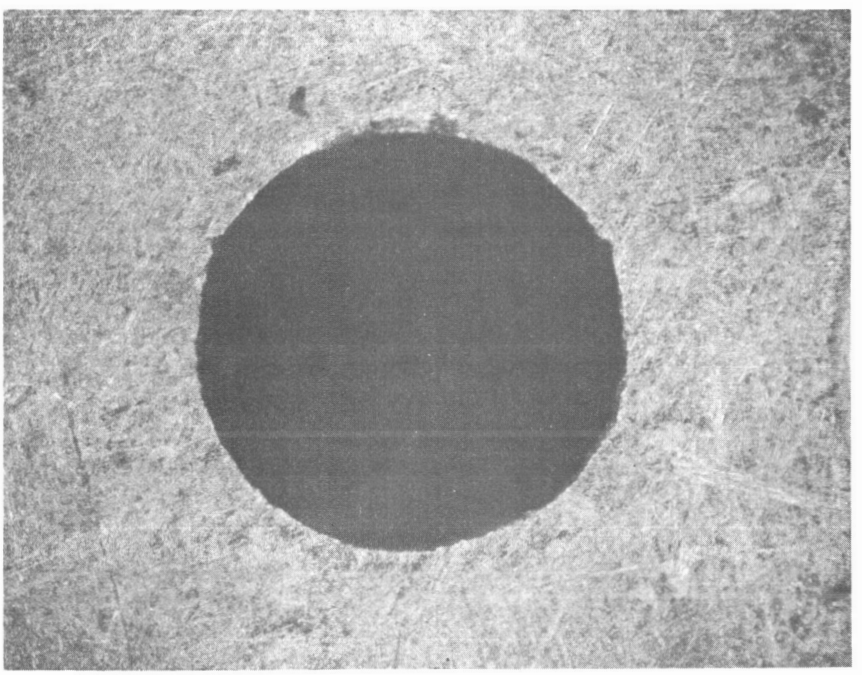


Exhibit 2B - Nickel orifice after exposure to liquid OF_2. 150X

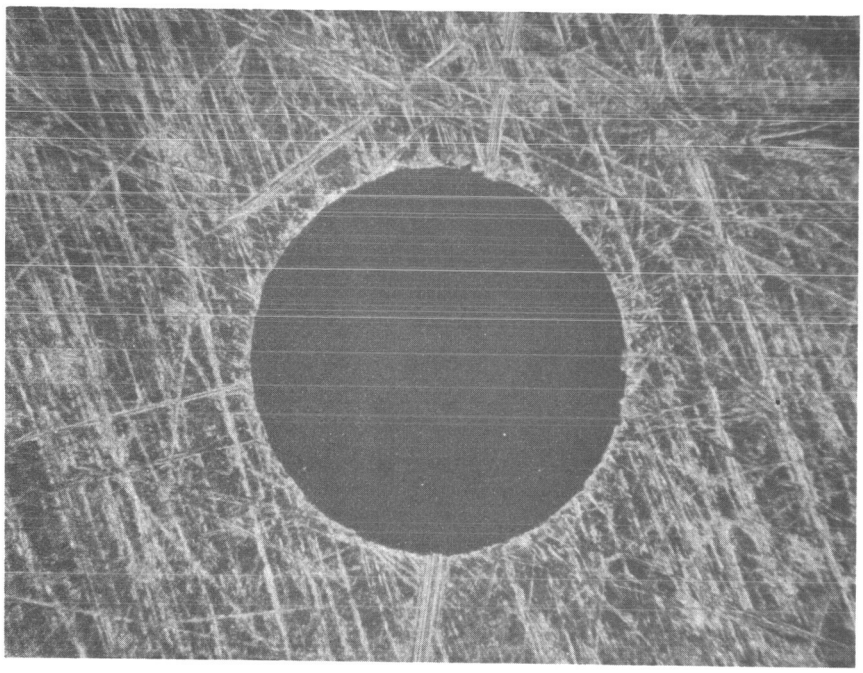


Exhibit 3A - Stainless Steel 304 orifice before exposure to liquid OF_2. 150X

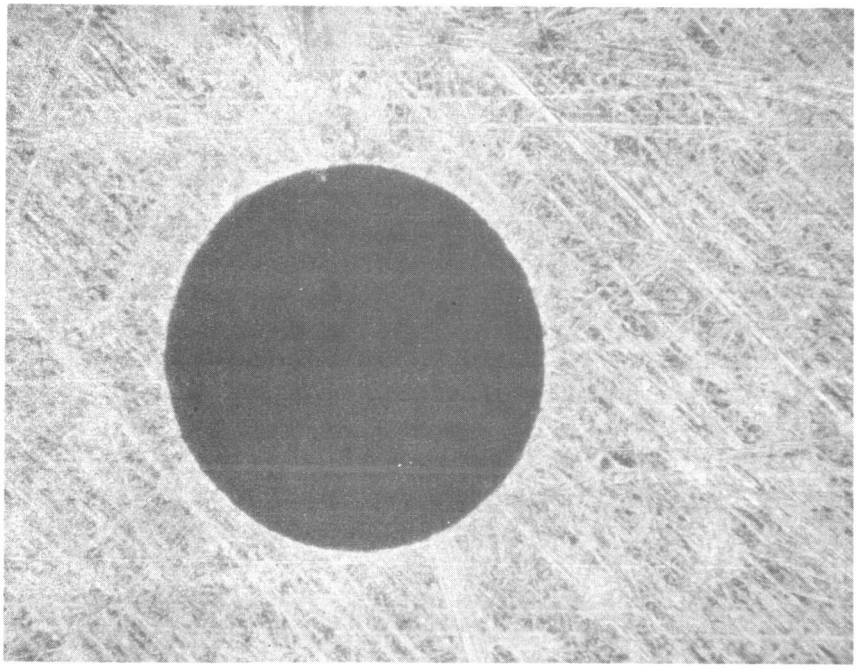


Exhibit 3B - Stainless Steel 304 orifice after exposure to liquid OF_2. 150X

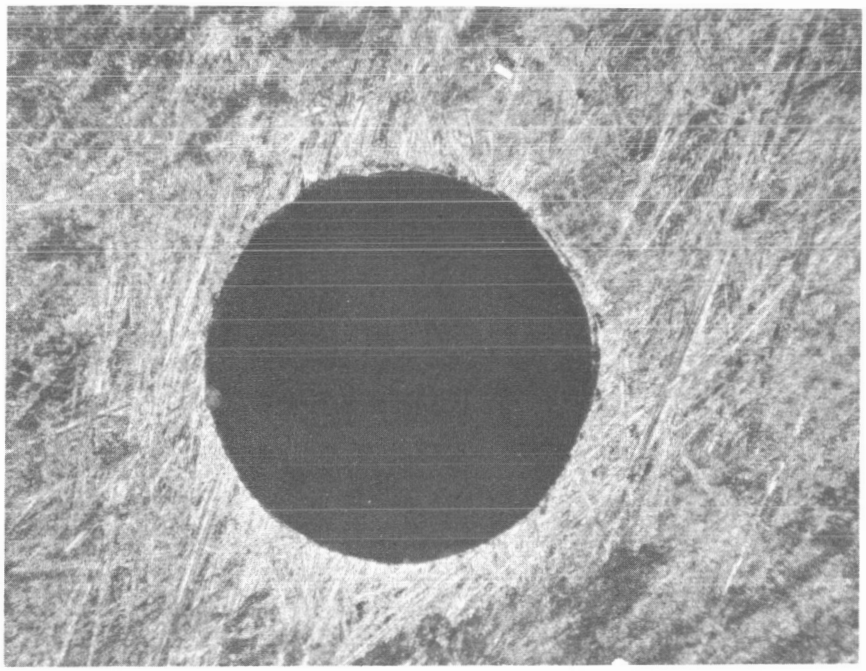


Exhibit 4A - Aluminum 2024 orifice before exposure to liquid OF_2. 150X

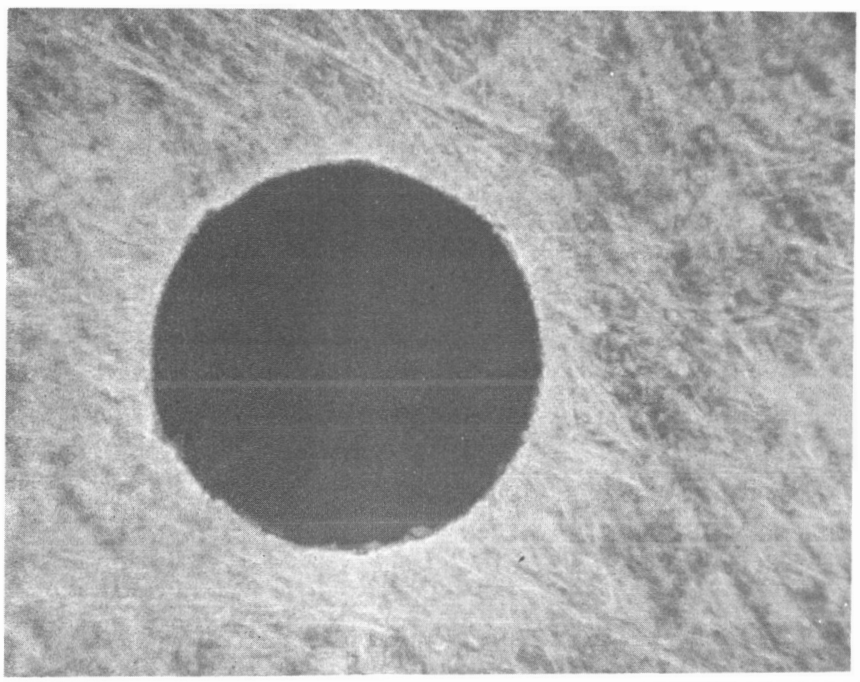


Exhibit 4B - Aluminum 2024 orifice after exposure to liquid OF_2. 150X

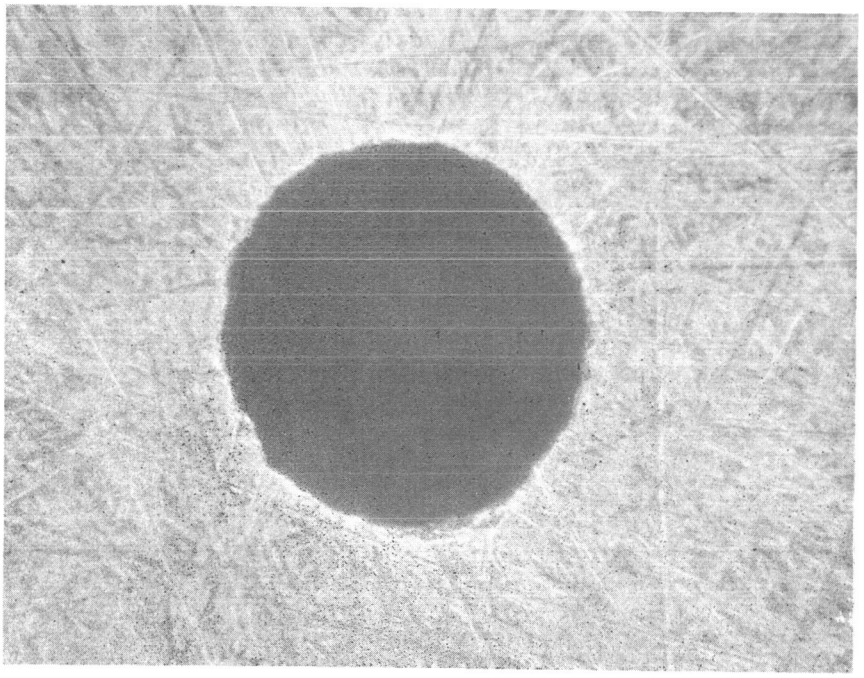


Exhibit 5A - Aluminum 6061 orifice before exposure to liquid OF_2. 150X

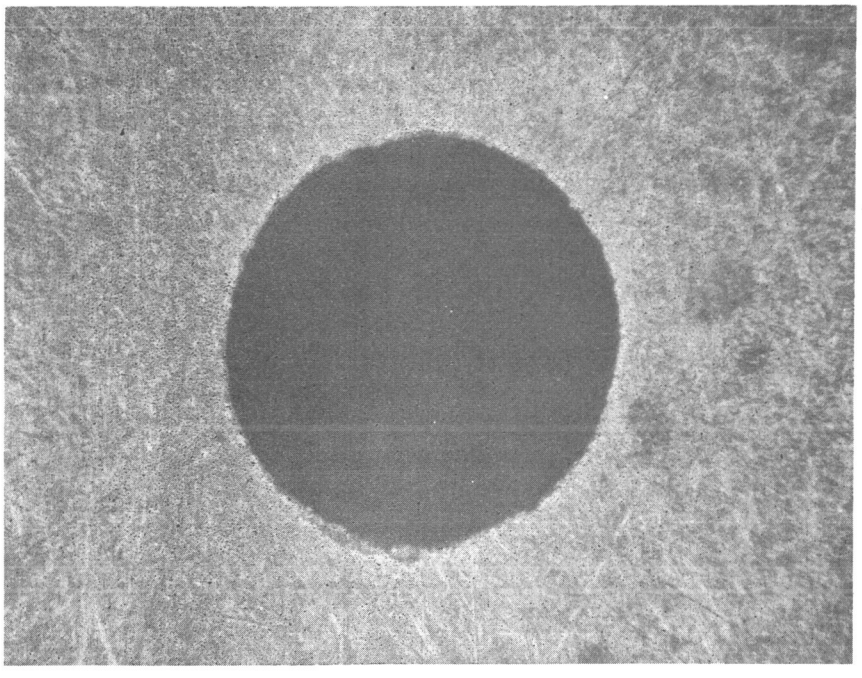


Exhibit 5B - Aluminum 6061 orifice after exposure to liquid OF_2. 150X

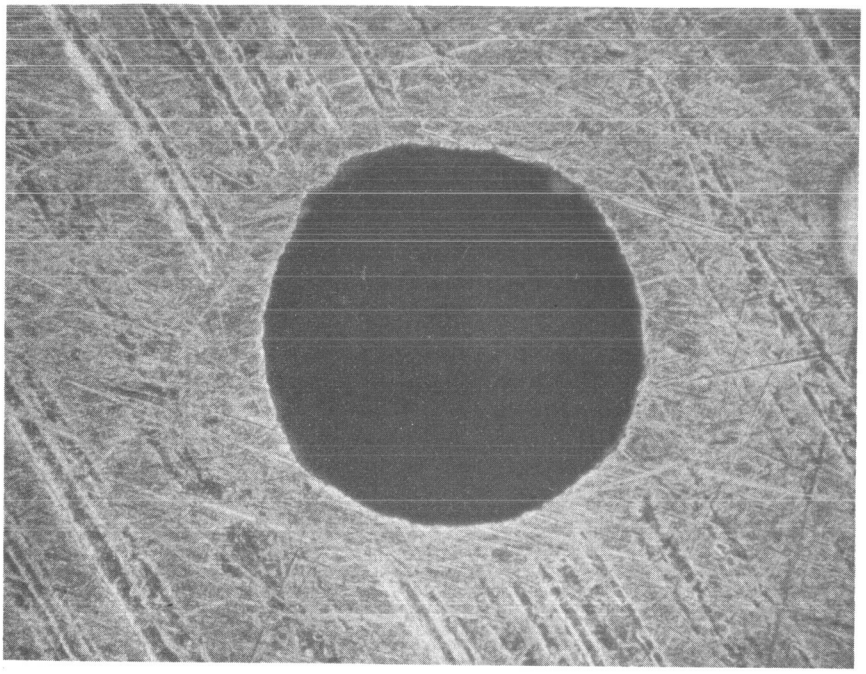


Exhibit 6A - Titanium orifice before exposure to liquid OF_2. 150X

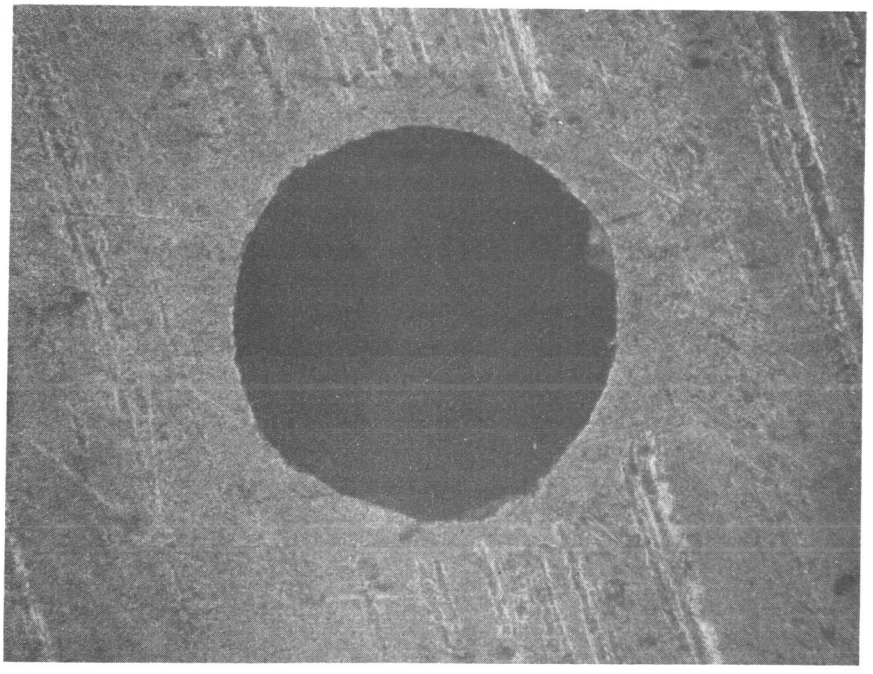


Exhibit 6B - Titanium orifice after exposure to liquid OF_2. 150X

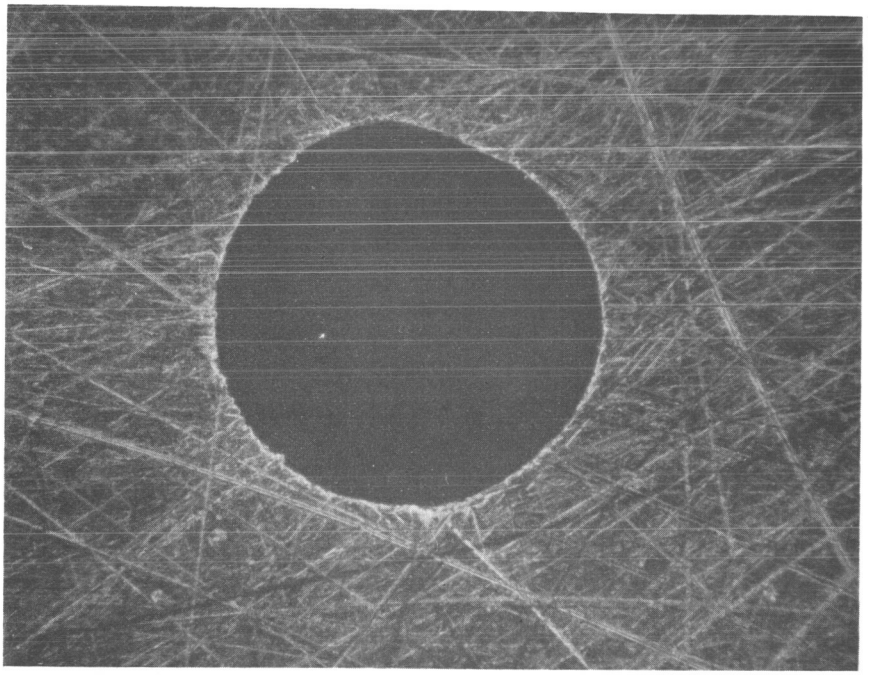


Exhibit 7A - Stainless Steel 301 orifice before exposure to liquid OF_2. 150X

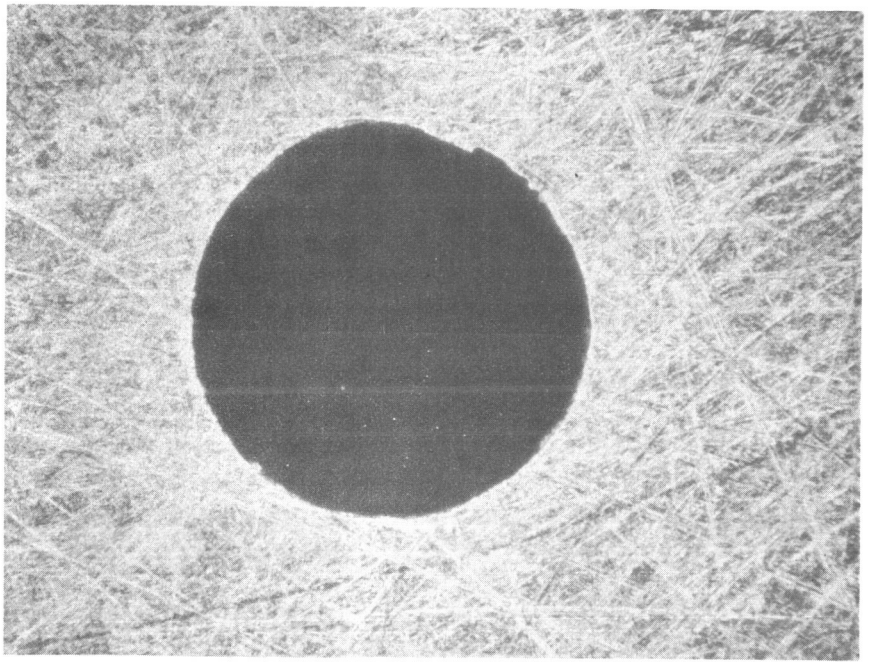


Exhibit 7B - Stainless Steel 301 orifice after exposure to liquid OF_2. 150X

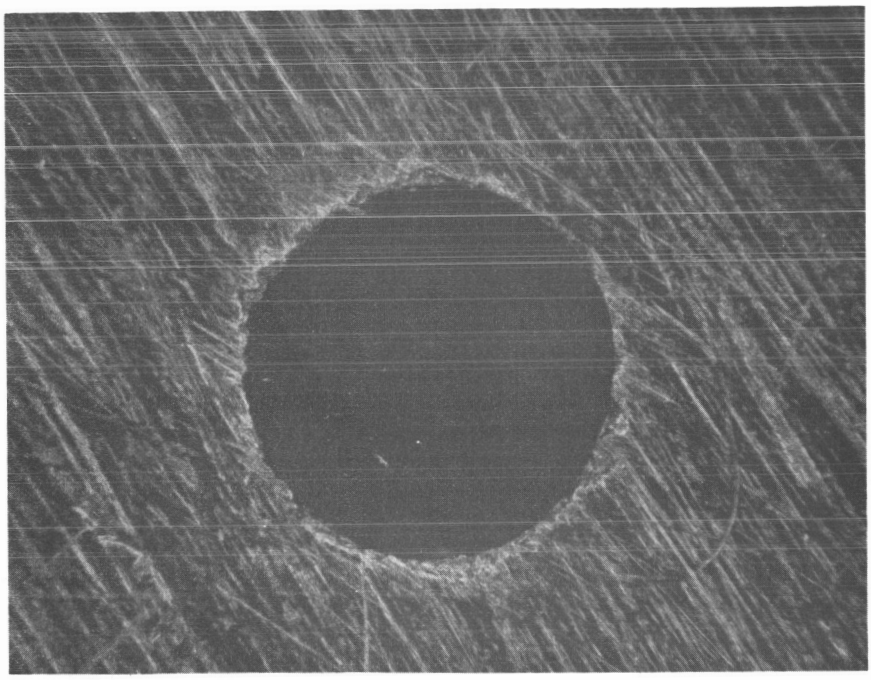


Exhibit 8A - Inconel orifice before exposure to liquid OF_2. 150X

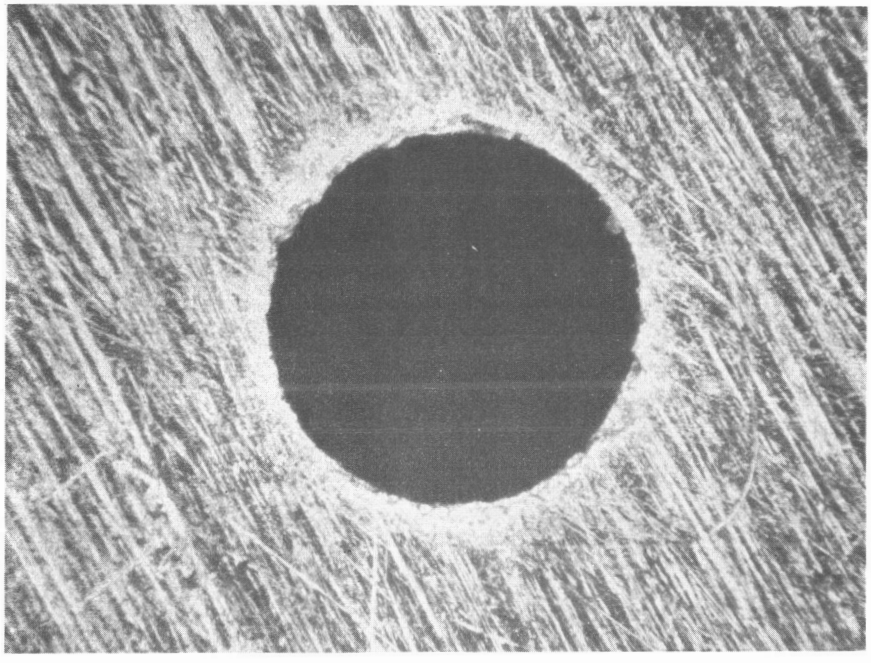


Exhibit 8B - Inconel orifice after exposure to liquid OF_2. 150X

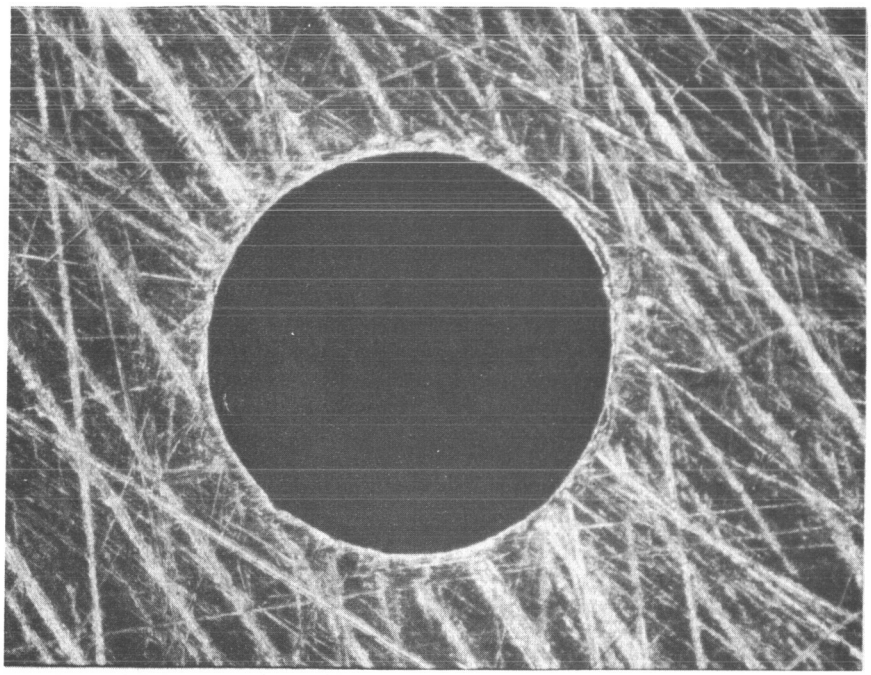


Exhibit 9A - Brazed Monel orifice before exposure to liquid OF_2. 150X

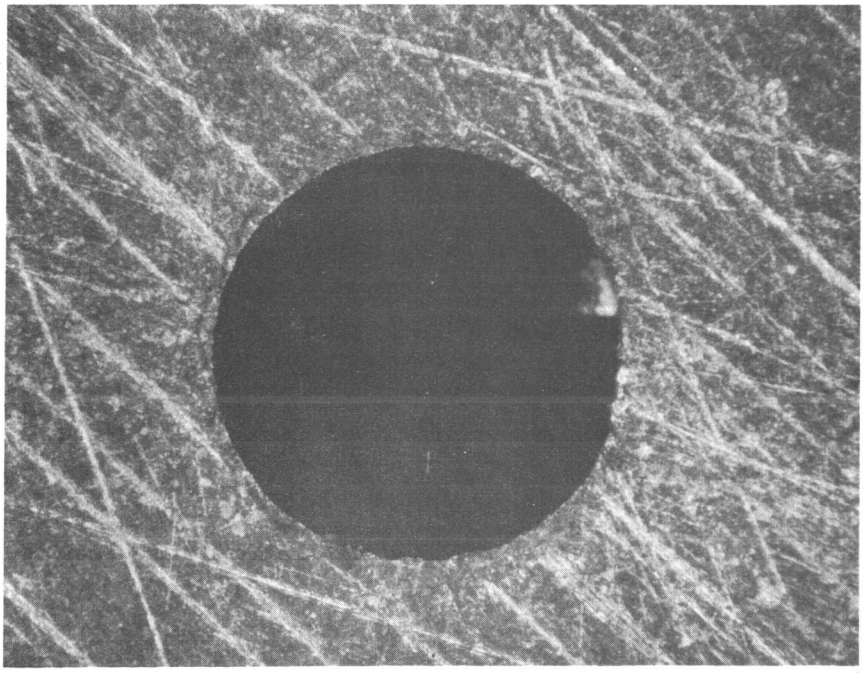


Exhibit 9B - Brazed Monel orifice after exposure to liquid OF_2. 150X

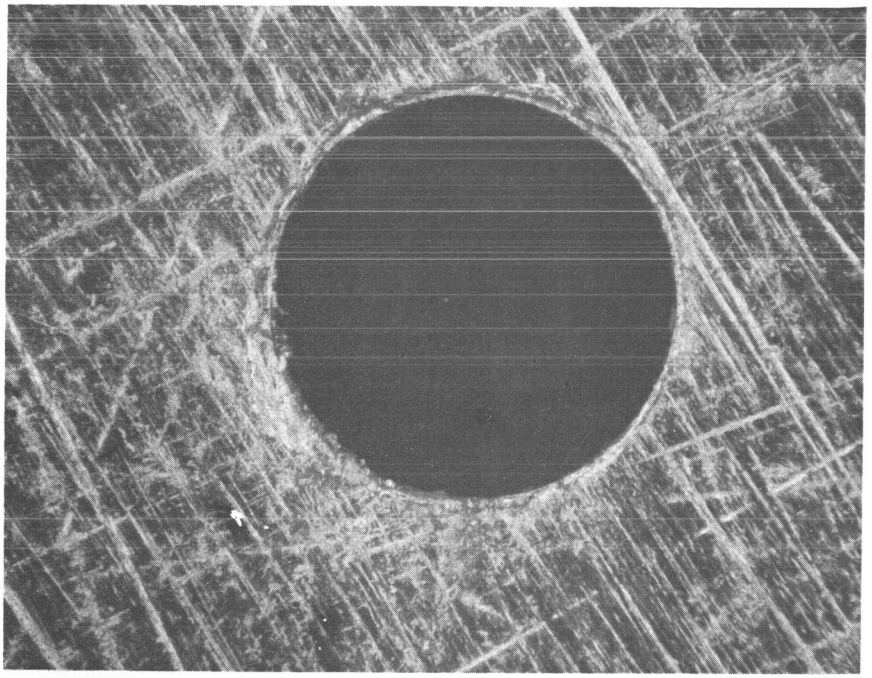


Exhibit 10A - Welded Monel orifice before exposure to liquid OF_2. 150X

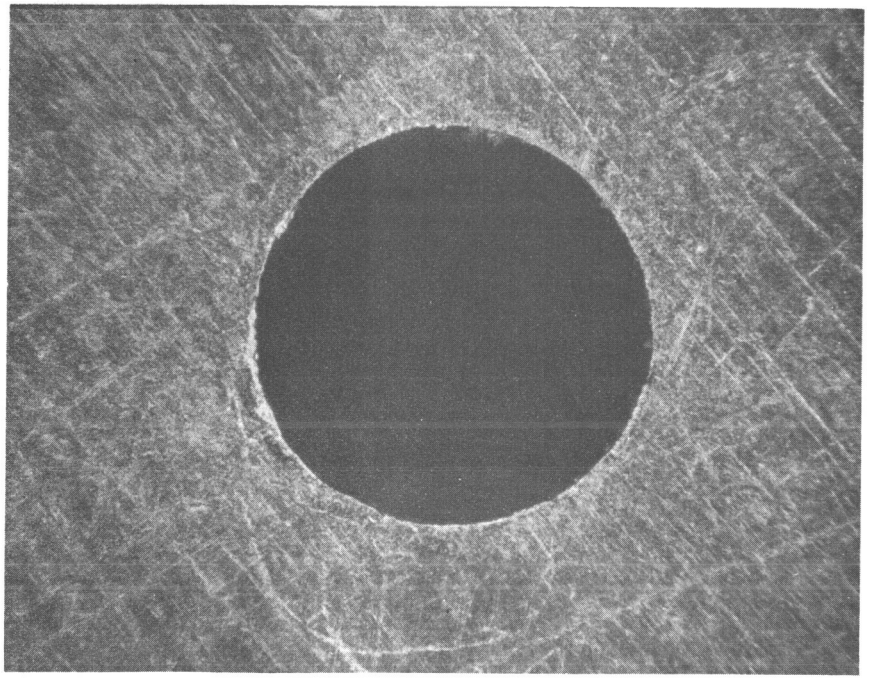


Exhibit 10B - Welded Monel orifice after exposure to liquid OF_2. 150X

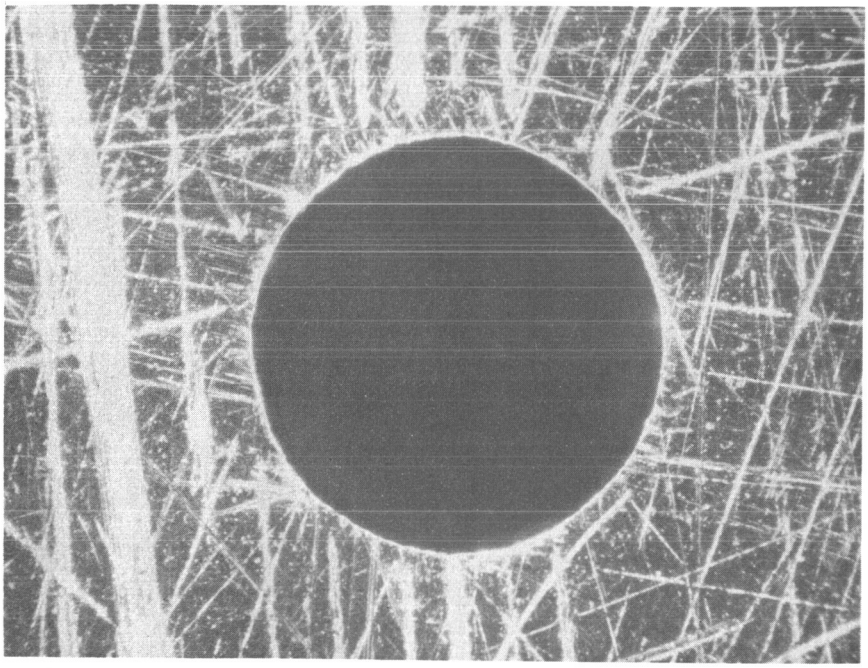


Exhibit 11A - Silver soldered Monel orifice before exposure to liquid OF_2. 150X

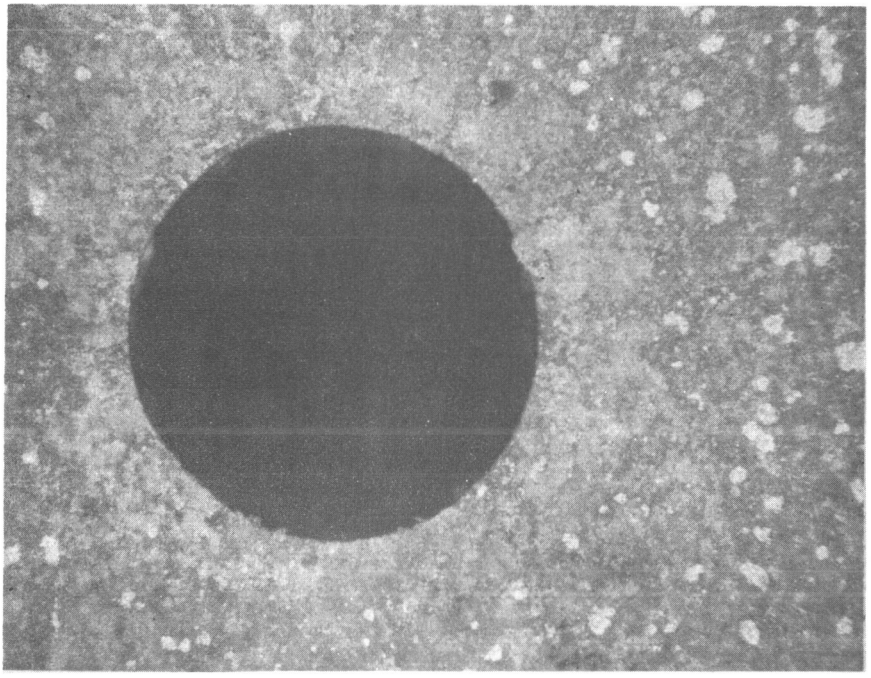


Exhibit 11B - Silver soldered Monel orifice after exposure to liquid OF_2. 150X

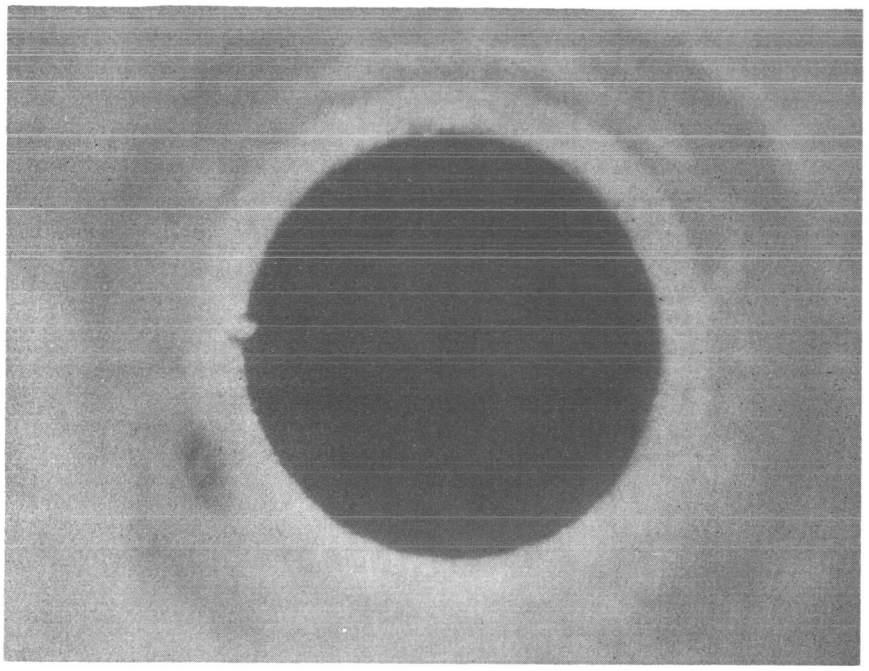


Exhibit 12C - Copper-chromium orifice after exposure to liquid OF_2. Inlet side. 150X

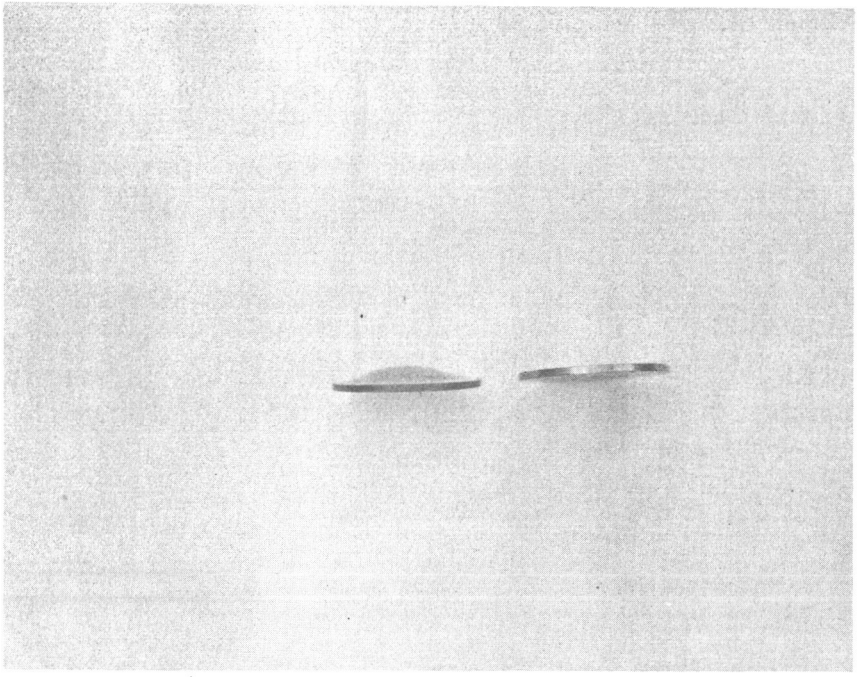


Exhibit 12D - Distorted copper-chromium specimen after liquid OF_2 exposure compared with unexposed specimen.

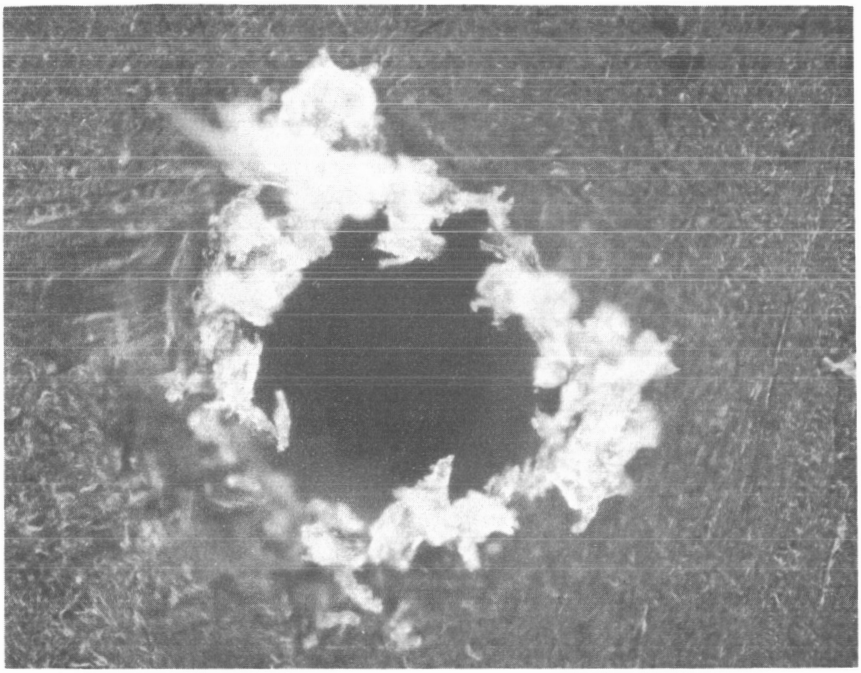


Exhibit 13A - Almac CTFE orifice before exposure to liquid OF_2. 150X

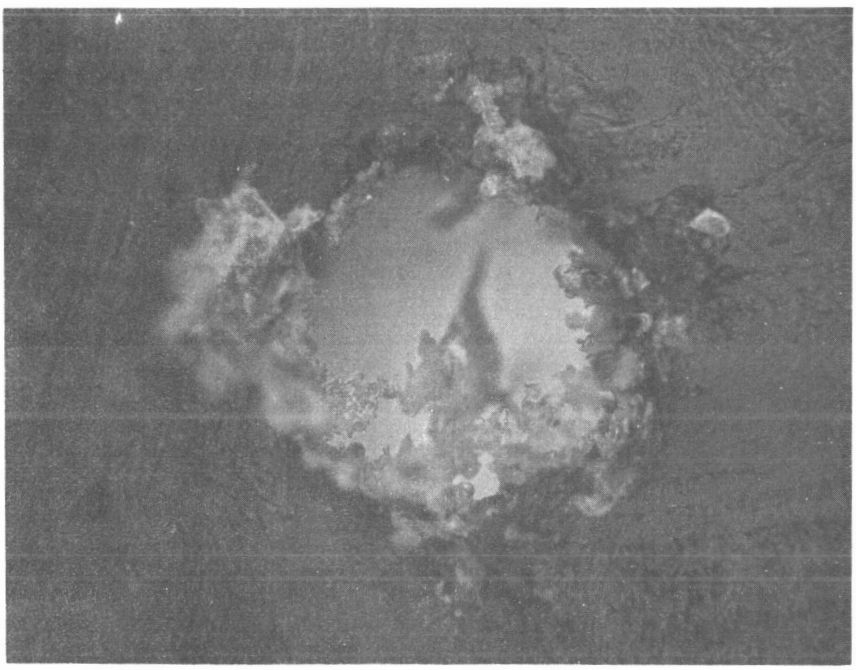


Exhibit 13B - Almac CTFE orifice after exposure to liquid OF_2. 150X

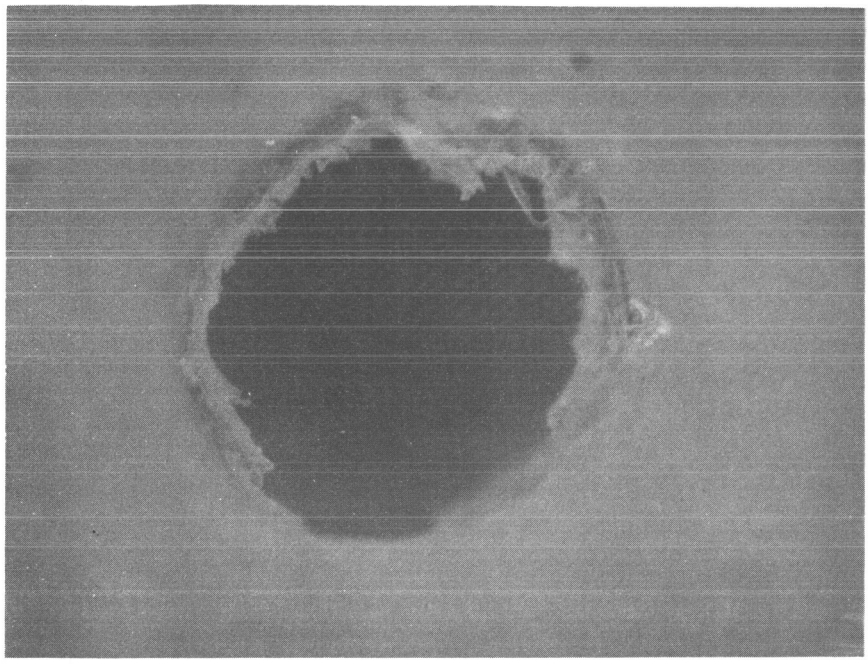


Exhibit 14A - Teflon 5 orifice before exposure to liquid OF_2. 150X

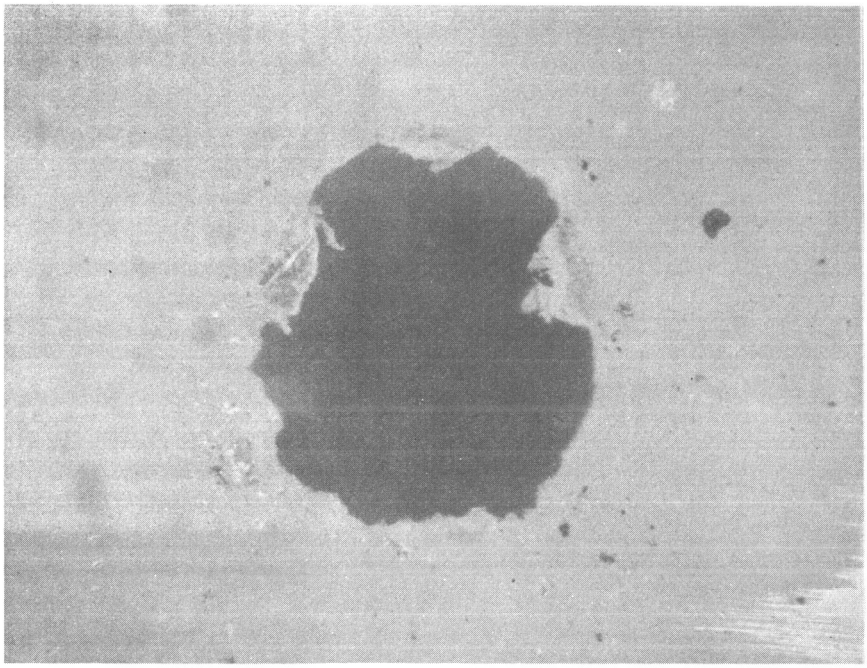


Exhibit 14B - Teflon 5 orifice after exposure to liquid OF_2. 150X

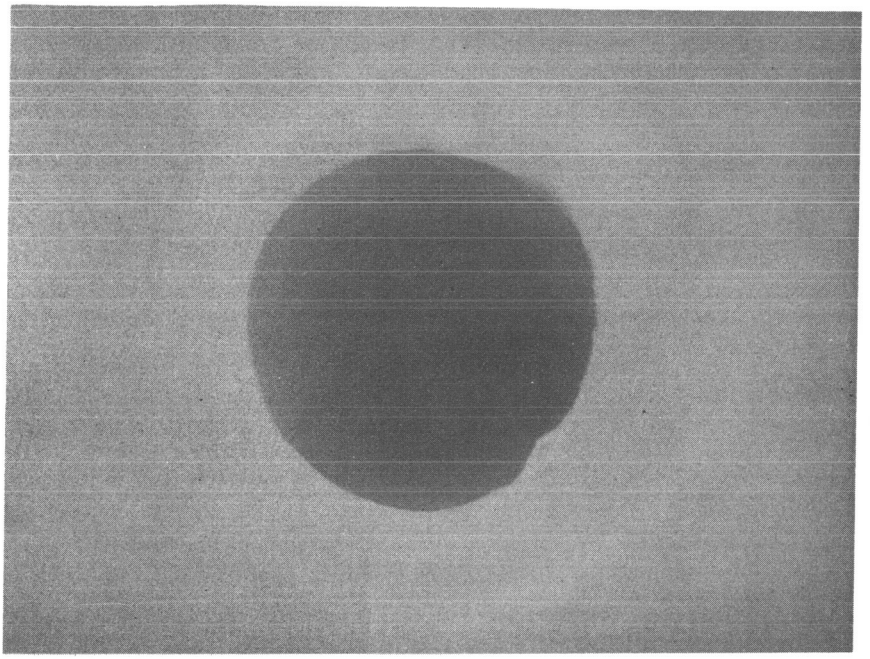


Exhibit 15A - Halon TFE G-80 High Crystallinity orifice before exposure to liquid OF_2. 150X

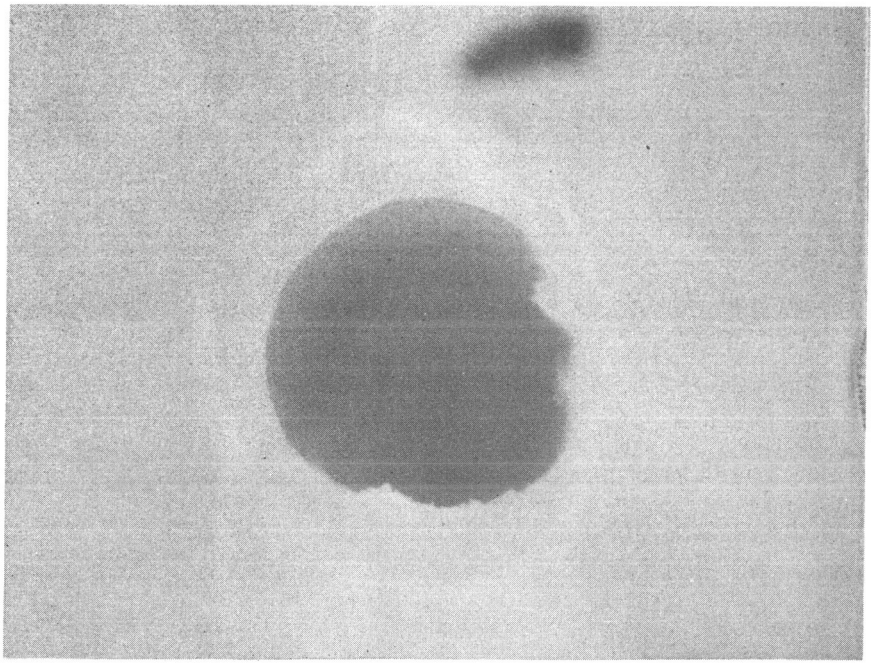


Exhibit 15B - Halon TFE G-80 High Crystallinity orifice after exposure to liquid OF_2. 150X

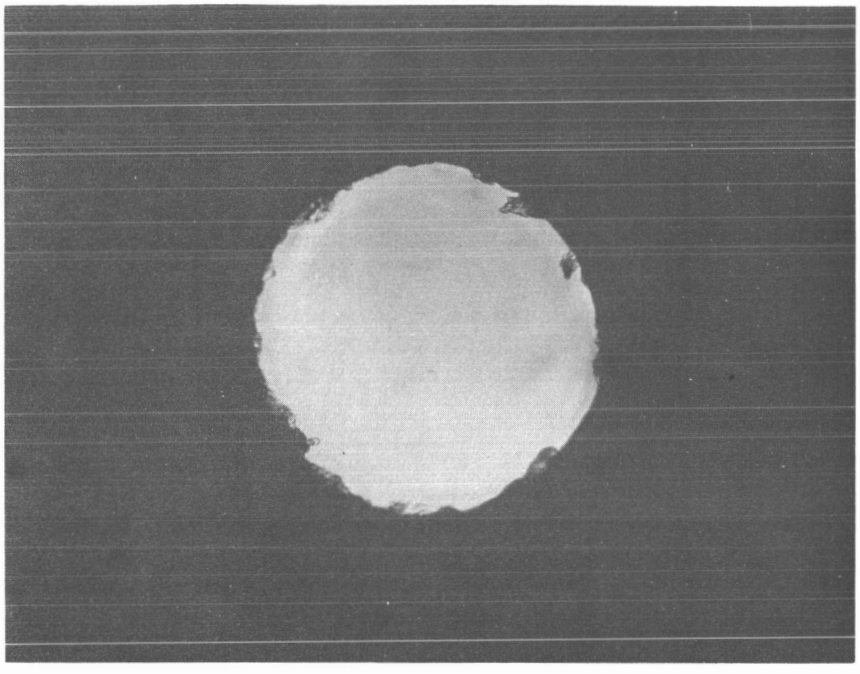


Exhibit 16A - Halon TFE G-50 orifice before exposure to simulated test conditions. 150X

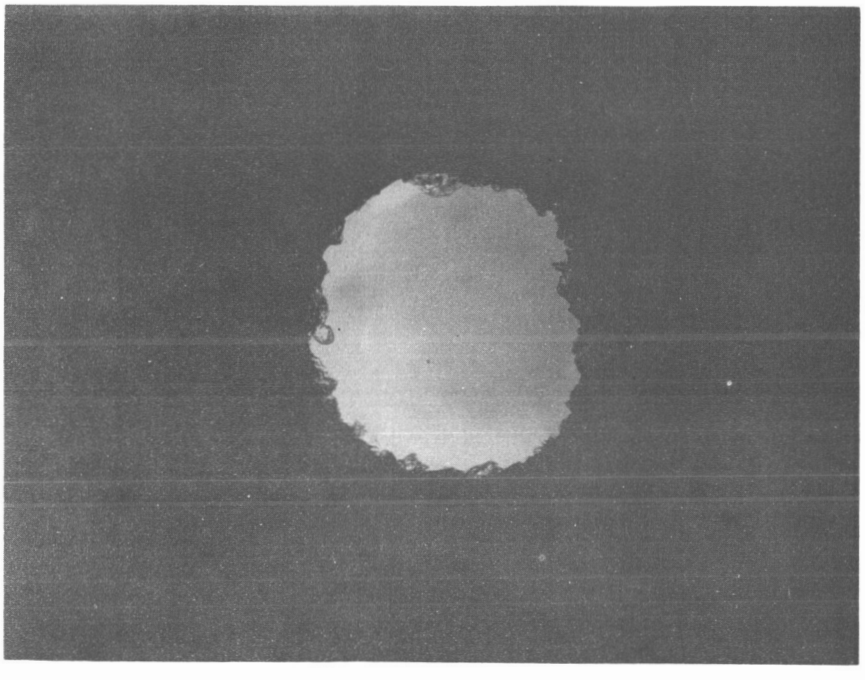


Exhibit 16B - Halon TFE G-50 orifice after exposure to simulated test conditions. 150X