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COMPATIBILITY OF MATERIALS WITH 7500 PSI OXYGEN

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FOREWORD

This investigation was initiated by the Biomedical Laboratory of the Aerospace Medical Research Laboratories, Aerospace Medical Division, Wright-Patterson Air Force Base, Ohio. The research was conducted by Union Carbide Corporation, Linde Division, Cryogenic Development Laboratory, Tonawanda, New York under Contract No. AF33(657)-11686. Mr. C. P. Smith, Section Engineer, and Mr. G. J. Nihart, Staff Chemist, were the principal investigators for Union Carbide Corporation. Contract monitor for the Aerospace Medical Research Laboratories was Mr. Irving H. Lantz. The work was performed in support of Project No. 6373, "Equipment for Life Support in Aerospace," and Task No. 637302, "Respiratory Support Equipment." The work sponsored by this contract was started in June 1963 and was completed in June 1964.

The authors acknowledge the invaluable assistance of Mr. R. M. Thayer, Mr. C. M. Heath, Mr. C. F. Gottzmann, Mr. E. S. Kordyban, and Mr. J. Notaro of Union Carbide Corporation, Linde Division.

This technical report has been reviewed and is approved.

WAYNE H. McCANDLESS
Technical Director
Biomedical Laboratory

ABSTRACT

A research program was conducted to develop ignition data on thread lubricants, thread sealants, fluorocarbon plastics, and metals. Spontaneous ignition temperatures were determined in both 2000 psi and 7500 psi oxygen for all the above materials except metals. The spontaneous ignition temperatures for these materials were found to be essentially the same in 7500 psi oxygen and in 2000 psi oxygen. Only three of the tested lubricants are recommended for possible use in 7500 psi systems. None of the thread sealants are recommended. Glass-filled polytetrafluoroethylene is usable only if tightly confined. The relative ease of ignition of metals and alloys was determined by promoted ignition methods in oxygen at 7500 psi. Inconel alloy 600, brass, Monel alloy 400, and nickel were found to have the highest resistance to ignition and combustion among the common alloys and metals. Of the materials tested, stainless steel and aluminum are the least satisfactory for use at oxygen pressures of 7500 psi. A test system was constructed to evaluate the hazards in rapidly charging a 65 cubic inch nickel-lined vessel with high pressure oxygen. A series of rapid charging tests up to as high as 8000 psi proceeded without incident. Electrostatic charges measured during the charging were negligible.

TABLE OF CONTENTS

Page

TABLE OF CONTENTS
(Continued)

LIST OF FIGURES

LIST OF FIGURES
(Continued)

LIST OF FIGURES
(Continued)

LIST OF TABLES

COMPATIBILITY OF MATERIALS WITH 7500 PSI OXYGEN

INTRODUCTION

The ease with which materials of construction ignite and combust in a gaseous oxygen environment has been a problem of long standing to those concerned with the safe design of oxygen handling equipment. Use of oxygen containing equipment has generally been limited to pressures below 3000 psi due to lack of knowledge of behavior of materials at higher pressures.

The use of gaseous oxygen at higher pressures in aerospace breathing systems (4), (17)* has increased the hazards inherent in an oxygen system (13) and additionally introduced several unknown entities. Some of these unknowns were investigated and evaluated by Baum, Goobich, and Trainer (1).

The present investigation was initiated to obtain further data on the behavior of materials in high pressure oxygen. Specifically, the purpose of the study was threefold:

1. Develop ignition data on thread lubricants, thread sealants, fluorocarbon plastics, and metals in 7500 psi oxygen.
2. Determine the hazards in charging a simulated system to 7500 psi with oxygen.
3. Measure the electrostatic charge developed during the charging of the simulated system.

PROGRAM DEVELOPMENT

The program was divided into three major areas of investigation and information was developed in each of these areas:

1. Selection of Test Methods.
2. Selection of Materials for Test.
3. Design of Equipment

Selection of Test Methods

Evaluation of various test methods was made to determine the amount of useful information which might be derived from a particular type of test in order to select the tests which would yield the most information in the time interval of the contract. It was recognized that each test would require development of parameters at 7500 psi which would give reliable and consistent data for comparison of materials.

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* Numbers in parentheses refer to references on page 57.

The tests considered for use in testing relative compatibility of materials in this program were:

1. Oxygen Bomb Test
2. Impact Sensitivity Test
3. Velocity Impact Test
4. Hot Wire Test
5. Promoted Ignition Test
6. Adiabatic Compression Test

Oxygen Bomb Test

The oxygen bomb test is a standard test used to determine the spontaneous ignition temperature of organic materials in 2000 psi oxygen. A specimen of the material to be tested is placed in an oxygen bomb and subjected to 2000 psi oxygen pressure; the bomb is then heated until spontaneous ignition of the sample material occurs or until the temperature reaches 500° C, whichever occurs first.

This test is considered to be an excellent test for thread lubricants, thread sealants, and fluorocarbon plastics. By comparing tests at 2000 psi and at 7500 psi, the effect of oxygen pressure on the spontaneous ignition temperature of the material could be determined.

Impact Sensitivity Test

This test is made by impregnating a specimen of the material with liquid oxygen and dropping a known weight through a given distance so that a hammer with a specified area impacts the sample. This test yields values denoting the amount of kinetic energy required to produce a reaction. This subject has been well covered by Reynales (11), (12), and it was not felt that enough additional information would be derived from the test to justify its use.

Velocity Impact Test

The velocity impact test can be used to test either non-metallic material or metallic material. The test is made in the following manner: A standard powdered material is injected into a high velocity oxygen stream and, after being accelerated to the velocity of the stream, is impacted against a specimen of the material to be tested. The heat generated during the impact ignites the powdered material and subjects the specimen to a known quantity of energy at an oxygen pressure variable from 50 to 700 psig.

This test has been used at Linde as a sorting tool to provide information on metals and alloys and the results concur with Linde experience. However, the specimen itself is in comparatively low-pressure oxygen and further tests by this method were not expected to yield as much additional information as other tests.

Hot Wire Test

This test was first used in 1923 by the Bureau of Mines (7), (8) to determine the ignition temperature of metals in oxygen at various pressures. It is based on the measurement of the electrical resistance of a wire specimen of the metal or alloy at its ignition point. Prior calibration of the resistance of wire specimens versus temperature must be made.

The test suffers from a number of deficiencies and has yielded anomalous results in Linde tests which were made in 2000 psi oxygen. It might be possible to overcome the deficiencies of this test if enough time were available but even then there would be no guarantee that the results would be satisfactory.

Promoted Ignition Test

The promoted ignition test is designed to determine the resistance to ignition and the amount of burning after ignition of the more resistant metals. It has been used previously at Linde to develop information on the combustion of metals and the ability of a given system to resist conflagration. Its use in this program would be an extension of the test to higher pressures to supplement the information which has been developed to date.

The test is made by subjecting the metal specimen to the energy released when a promoter material is heated to its spontaneous ignition temperature in oxygen. This test has considerable built-in flexibility by varying the weight of the promoter material, the weight of the metal, or the configuration of the metal specimen.

Adiabatic Compression Test

This test could be used to test either metal specimens or nonmetallic specimens. The sample contained in a test vessel at 15 psia of oxygen is subjected to the temperature developed when the vessel is rapidly pressurized to oxygen pressures ranging from 2000 psi to 8000 psi. Theoretically, extremely high temperatures may be reached by adiabatic compression (Appendix I) and it was expected that this test would yield some good data.

This type of test was also expected to be applicable in defining the hazards in charging a simulated system.

The test methods selected for use in this program were:

1. Oxygen Bomb Test
2. Promoted Ignition Test
3. Adiabatic Compression Test

Selection of Materials for Test

The number of materials available for testing in a program of this type is almost unlimited. Therefore, it was necessary to select those materials which it was thought would yield the most information. Selection was made by correlation of information found in the literature and that derived from unpublished Linde test programs.

Thread Lubricants and Thread Sealants

The subject of available thread lubricants and thread sealants has been covered quite well by Reynales (12). Using this information and that available from Linde files, a number of materials were chosen to be tested.

It was known beforehand that most of the materials selected for test would not be suitable for use in 7500 psi oxygen. In fact, a few are not suitable for 2000 psi oxygen. However, the most useful information expected from the oxygen bomb test was whether the spontaneous ignition temperature of a material in 7500 psi oxygen would differ from its spontaneous ignition temperature in 2000 psi oxygen. By testing some materials which would ignite at low temperatures as well as some which would ignite at high temperatures, the effect of pressure on the spontaneous ignition temperature of typical materials could be determined.

The materials selected for test are as follows:

1. Thread Lubricants

a. Aroclor 1254 - A chlorinated phenyl compound produced by Monsanto Chemical Company

b. Dixon's Flake Graphite No. 1 - Dixon Corporation, Bristol, Rhode Island

c. Oxweld Anti-Friction Compound No. 54 - A proprietary compound of Union Carbide Corporation, Linde Division. It is not recommended for service above 150° F or 300 psi of oxygen.

d. Kel-F 90 Grease - A formulation of poly chlorotrifluoroethylene oils and waxes with an inert gelling agent manufactured by Minnesota Mining and Manufacturing Company.

e. Halocarbon Grease Series 25-10 - A polychlorotrifluoroethylene formulation manufactured by Halocarbon Products Corporation, Hackensack, New Jersey.

f. Halocarbon Oil Series 13-21 - A polychlorotrifluoroethylene oil marketed by Halocarbon Products Corporation.

g. Molykote Z - Molybdenum disulfide in dry powder form produced by The Alpha Corporation, Greenwich, Connecticut.

h. Burnil Brand Microplates - Thin platelets of synthetic mica available as a powder, as a suspension of the powder in water, or as a thin paper from Minnesota Mining and Manufacturing Company.

i. Oxweld Anti-Friction Compound No. 64 - A proprietary product of Union Carbide Corporation, Linde Division. It is not recommended for service above 250° F or 500 psi of oxygen.

j. Almasol Powder (325 mesh) - This powder is composed of aluminum-magnesium-silicate of laminar lattice structure. It is used as an ingredient in solid film lubricants marketed by The Almasol Corporation, Fort Worth, Texas.

k. High Purity Goop - A proprietary lubricant made by Crawford Fitting Company, Cleveland, Ohio.

l. Everlube Solid Film Lubricant No. 811 - A molybdenum disulfide dispersion available from Eve lube Corporation of America, North Hollywood, California.

m. Oxylube No. 703 - A dry film lubricant based on molybdenum disulfide which is marketed by Drilube Company, Glendale, California.

2. Thread Sealants

a. Oxyseal - Parker-Hannifin Corporation, Cleveland, Ohio.

b. Rectorseal No. 15 - Rector Well Equipment Company, Fort Worth. Texas

c. Key Abso-lute - W. K. M. Division of ACF Industries, Houston, Texas

d. Mano Pipe and Joint Compound - Mano Industries, Woodside, Long Island, New York

e. Linde Green Pipe Joint Compound - Union Carbide Corporation, Linde Division.

f. 50/50 Soft Solder

Fluorocarbon Plastics and Elastomers

1. There were three types of fluorocarbon plastics which were of interest:

a. Polytetrafluoroethylene(TFE) - Marketed under the trade name of "Teflon" by E. I. du Pont de Nemours and Company, Inc. and as "Halon" TFE by Allied Chemical Corporation.

b. Polychlorotrifluoroethylene(CTFE) - Marketed as "Kel-F" by Minnesota Mining and Manufacturing Company,as "Polyfluoron" by Acme Resin Corporation, and as "Halon" VK and TVS by Allied Chemical Corporation.

c. Polyfluoroethylenepropene (FEP) - A copolymer of tetrafluoroethylene and hexafluoropropylene marketed under the trade name "Teflon" 100X by E. I. du Pont de Nemours and Company, Inc.

2. There were two types of fluoroelastomers of interest:

a. Copolymers of vinylidene fluoride and hexafluoropropylene - Available as "Fluorel" from Minnesota Mining and Manufacturing Company, and as "Viton" A, "Viton" A-HV, and "Viton" B from E. I. du Pont de Nemours and Company, Inc.

b. Copolymers of vinylidene fluoride and chlorotrifluoroethylene - Marketed by Minnesota Mining and Manufacturing Company, in two types, "KEL-F" Elastomer 3700 and "KEL-F" Elastomer 5500.

3. Additionally there are numerous companies which supply filled, formulated, or reinforced TFE.

4. The following materials were selected for test: *

a. Teflon (Virgin TFE) - E. I. du Pont de Nemours and Company, Inc.
b. Teflon 100X (FEP) - E. I. du Pont de Nemours and Company, Inc.
c. Viton A (Virgin) - E. I. du Pont de Nemours and Company, Inc.
d. Viton B (Virgin) - E. I. du Pont de Nemours and Company, Inc.
e. Rulon A (Reinforced TFE) - Dixon Corporation
f. Rulon B (Reinforced TFE) - Dixon Corporation
g. Rulon C (Reinforced TFE) - Dixon Corporation
h. Duroid 5600 (60% Teflon, 40% aluminum silicate ceramic fibers) - Rogers Corporation
i. Duroid 5650 (75% Teflon, 25% aluminum silicate ceramic fibers) - Rogers Corporation
j. Duroid 5870 (85% Teflon, 15% glass fibers) - Rogers Corporation
k. Duroid 5813 (60% Teflon, 40% glass fibers with MoS_2 filler) - Rogers Corporation
m. Kel-F 81 (CTFE) - Minnesota Mining and Manufacturing Company
n. Kel-F Elastomer 3700 - Minnesota Mining and Manufacturing Company
o. Kel-F Elastomer 5500 - Minnesota Mining and Manufacturing Company

Metals and Alloys

The oxidation, ignition, and combustion of metals in air and oxygen have received considerable attention in the past decade, but the ignition and burning of metals is still poorly understood. Knowledge of reaction kinetics and mechanics is sadly lacking. There are many investigators who have made significant contributions to this field. Their work is reviewed by Smeltzer and Perrow (16) and by Markstein (10). Because it is not the purpose of this investigation to become involved in the complicated mechanisms of ignition and combustion, only those articles which were considered to be of direct value to the present work have been referenced (1, 3, 6, 9, 14, 15).

The investigation of Dean and Thompson (3) yielded data which was directly applicable in helping to select the metals to be tested in the present investigation. Metal tubes were electrically heated to destruction in 50, 300, and 800 psia of oxygen, in a 50-50 mixture of oxygen and carbon dioxide, and in 100% carbon dioxide. Color motion picture photography recorded the manner in which the tubes heated and failed. The results of their tests showed that:

1. Stainless steels ignited within their melting range.

2. Steel alloys with no nickel content ignited at temperatures below their melting points.

3. Most of the nickel-based alloys tested did not ignite until the melting point was reached. At 800 psia both Inconel X and Monel failed at temperatures 250° to 500° F below their respective melting points.

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* Appreciation is expressed to the following companies for samples furnished for these tests: E. I. du Pont de Nemours and Company, Inc., Rogers Corporation, Dixon Corporation, The Fluorocarbon Company, Minnesota Mining and Manufacturing Company.

4. Nickel A did not ignite, only melted.

5. Copper ignited slightly below its melting point At a pressure of 300 psia approximately 70% of the test specimen was destroyed.

6. No ignition or burning of the aluminum alloy occurred, only melting.

7. For all the ferrous alloys tested, the rate of burning increased with the oxygen pressure.

8. Cobalt-based alloys ignited within their melting point range.

9. Titanium was the only metal which ignited in an atmosphere of carbon dioxide.

Reynolds (14) found that 18-8 stainless steel, copper, nickel, Inconel, and Inconel X melted before igniting in oxygen at pressures up to eight atmospheres.

Hill, Adamson, Foland, and Brissette (9) report that Inconel, copper, Monel, and aluminum did not have a spontaneous ignition temperature in the solid phase, and that iron, carbon steel, and common iron alloy had spontaneous ignition temperatures in the solid phase (below their melting points) and melted very rapidly while burning.

Baum, Goobich, and Trainer (1) found Monel and Type 316 stainless steel acceptable alloys of construction for 7500 psi oxygen systems but copper and brass possessed some undesirable characteristics.

Riehl, Key, and Gayle (15) report aluminum in gaseous oxygen to be sensitive to explosive shock while stainless steel is not.

Investigations previously made at Linde using the "Velocity Impact Test" (e.g. A known weight of powdered material was accelerated in a high velocity oxygen stream and impacted against the metal specimen) yielded the following results:

1. Carbon steel, cast iron, stainless steel, and aluminum could be ignited and completely consumed with the evolution of large quantities of energy.

2. Stainless steel and aluminum burned with explosive violence.

3. Copper, copper alloys, and Monels had a decided quenching effect on combustion.

The "Promoted Ignition Test" has been used in earlier work at Linde to investigate the resistance to ignition and the amount of burning after ignition of various metals in 2000 psi oxygen. In this test the metal specimen was subjected to the energy released when a promoter material was spontaneously ignited. Although this test does not provide absolute values it does effectively sort the metals in regard to their relative resistance to combustion. The results of this work are:

1. In general, metal alloys with a high percentage of nickel such as the Monels and Inconels have the highest resistance to combustion.

2. The resistance to combustion appeared to be related to the percentage of iron in an alloy.

3. The size of the metal sample will affect its resistance to combustion (e.g. the more subdivided a metal specimen, the easier it will ignite).

The results of Dean and Thompson (3) referred to earlier were evaluated in regard to the relative resistance to ignition and combustion of each metal tested and the metals are listed in Table 1 in the order of their decreasing resistance, with the most resistant metal at the top of the list.

The relative resistance of metals as determined by the "Velocity Impact Test" and the "Promoted Ignition Test" at Linde are also listed in Table 1 for comparison. It should be pointed out that there might be some shifting in the position of the metals in the "Promoted Ignition Test" column because in those cases where two or more metals showed similar resistance to ignition, the violence of the combustion determined the position. Also if a large enough number of tests were to be performed on each metal, statistically the position of a metal might be changed.

Of particular interest is the position occupied by aluminum. Dean and Thompson's work would place it at the top of the list while it is at the bottom of the list in the Linde tests. This is no doubt due to the fact that Dean and Thompson only heated the tube to its melting point. Grosse and Conway (6) have shown the ignition temperature of aluminum to be > 1000° C which is considerably above its melting point of 660° C. Aluminum is at the bottom of the ladder on the Linde tests because of its violent reaction once it becomes ignited.

It will be noted that nickel and copper alloys are at the top of the list in all three columns.

For the present investigation, metals and alloys were selected which would be representative of materials already tested in oxygen pressures up to 2000 psi. Results at 7500 psi could thus be compared to those obtained at the lower pressures. Additionally some other metals were chosen because of their possible usefulness in high pressure systems.

The metals and alloys selected for test, the melting point of each, and the reported ignition temperature in oxygen are presented in Table 2.

Design of Equipment

The materials of construction and the design of the equipment to be used in the test program was an area that received considerable attention. Because of the inherent hazards with gaseous oxygen and the magnification of these hazards with 7500 psi oxygen, it was necessary to select those materials which would be considered relatively safe by present available knowledge. Consequently nickel alloys were used in all critical pieces of apparatus. This resulted in considerable delay in initial construction of equipment.

TABLE 1.

RELATIVE RESISTANCE OF METALS AND ALLOYS TO IGNITION AND COMBUSTION IN OXYGEN IN DECREASING ORDER

Dean and Thompson (3)	Velocity Impact	Promoted Ignition
50-800 psi	50-100 psi	2000 psi
Aluminum	**Monel	**Monel
Nickel A	**K-Monel	**Inconel 600
*Hastelloy C	***Tobin bronze	**Monel S
**Monel	Copper	***Tobin bronze
*Hastelloy X	Steel	**Duranickel
**Inconel X	18-8 stainless steel	*****Ampco alloy No. 15
*Hastelloy R	Aluminum	**Permanickel
Copper		**K-monel
*Haynes 25		*Hastelloy R-235
*Multimet		Maraging Steel
18-8 Stainless Steel		Beryllium Copper
Other Stainless Steel		******Elgiloy
Carbon Steel		****Rene' 41
Titanium		**Inconel X-750
		*Multimet
		*Hastelloy X
		*Haynes 25
		***Everdur
		18-8 Stainless Steel
		Aluminum

TRADEMARKS

*Union Carbide Corporation, Stellite Division
**The International Nickel Company, Inc.
***Anaconda American Brass Company
****General Electric Company
*****Ampco Metal, Inc.
******Elgin National Watch Company

TABLE 2.

METALS AND ALLOYS SELECTED FOR TEST IN 7500 PSI OXYGEN

METAL OR ALLOY	MELTING POINT °C	IGNITION TEMPERATURE IN OXYGEN, °C
1. **Stainless Steel**		
Type 316	1375-1400	----
Type 304	1400-1475	M.P. (a)
Type 301	1400-1425	----
2. **Age-Hardening Stainless Steel**		
*Type 17-7 PH	1415-1450	150-275 below M.P. (a)
3. **Nickel-Chromium Alloys**		
Inconel alloy 600 (formerly Inconel)	1400-1425	M.P. (b)
Inconel alloy X-750 (formerly Inconel X)	1400-1425	150-275 below M.P. (a)
4. **Nickel - Copper Alloy**		
Monel alloy 400 (formerly Monel)	1300-1350	150-275 below M.P. (a)
5. **Other Metals and Alloys**		
Aluminum	660	>1000 (c)
Nickel	1455	M.P. (a)
Copper	1082	<M.P. (a) (d)
Brass	932	<850 (d)
Silver	960	----
Gold	1063	----
Lead	327	870 (c)

(a) Reference 3
(b) Reference 14
(c) Reference 6
(d) Reference 7

* Trademark of Armco Steel Corporation

High Pressure Oxygen Warm Converter

High pressure oxygen was generated in a warm converter. Required quantities of liquid oxygen were introduced into the warm converter and vaporized into the gaseous state to provide the necessary pressure. The warm converter was contained in a water bath with an inlet and outlet through which tap water flowed.

The converter was patterned after a previous Linde design which was modified to provide a larger capacity and a higher working pressure. The internal geometric volume of the converter is 425 cubic inches. Pressures up to 15,000 psi oxygen have been generated. Materials of construction were Inconel alloy X-750 and Monel alloy 400 which provided the strength for the pressures to be developed and also furnished a definite measure of safety because of their relative resistance to ignition. Figure 1 shows the top of the converter extending out of the water bath. All high pressure oxygen for the entire test program was generated in this warm converter.

Compressibility factors were calculated for oxygen at various pressures (Appendix II) for use during the test program.

High Pressure Oxygen Bomb

The original consideration given to a high pressure oxygen bomb was a design patterned after the one presently used at Linde for tests at 2000 psi. This bomb has a high mass with built-in cooling passages and integral heating unit. However, the use of a smaller bomb had several advantages relative to the present program and a small high pressure bomb was designed after substantial testing of a prototype bomb.

Figure 2 shows the high pressure bomb assembled and disassembled. Inconel X-750 was the material of construction because of the strength needed at the high temperatures to which the bomb would be heated.

Valves, Fittings, and Tubing

Initially all valves were constructed of Monel alloy 400 with stems of Monel alloy K-500. Packing was glass filled TFE. All fittings and tubing were made of Monel 400. Fabrication was by a commercial vendor of high pressure equipment. At a later date, some stainless steel parts were substituted to expedite test work and to evaluate the use of stainless steel.

All valves were rated at 30,000 psi with the exception of an air-operated valve which was rated at 10,000 psi. All valves were of standard construction as shown in Figure 3.

All fittings and tubing were rated at 30,000 psi with the exception of some 9/16" O.D. x 5/16" I.D. tubing which was only rated for 10,000 psi. The standard threaded and coned tubing connection was used as shown in Figure 4.

Simulated System

Construction of the simulated system will be discussed later under the experimental program.

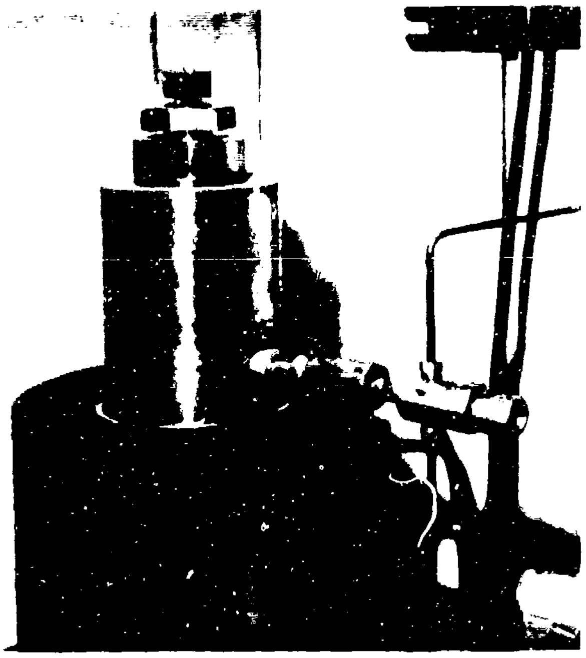
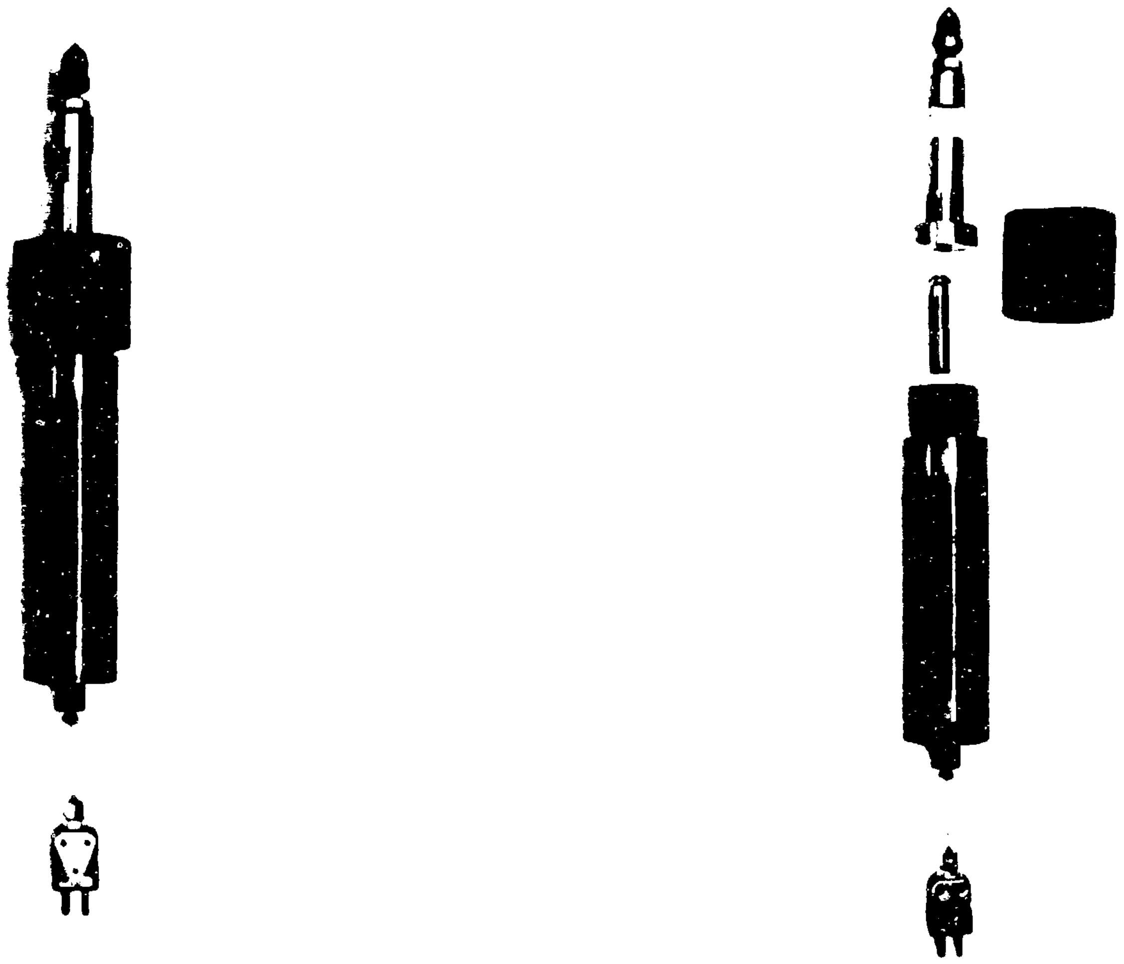


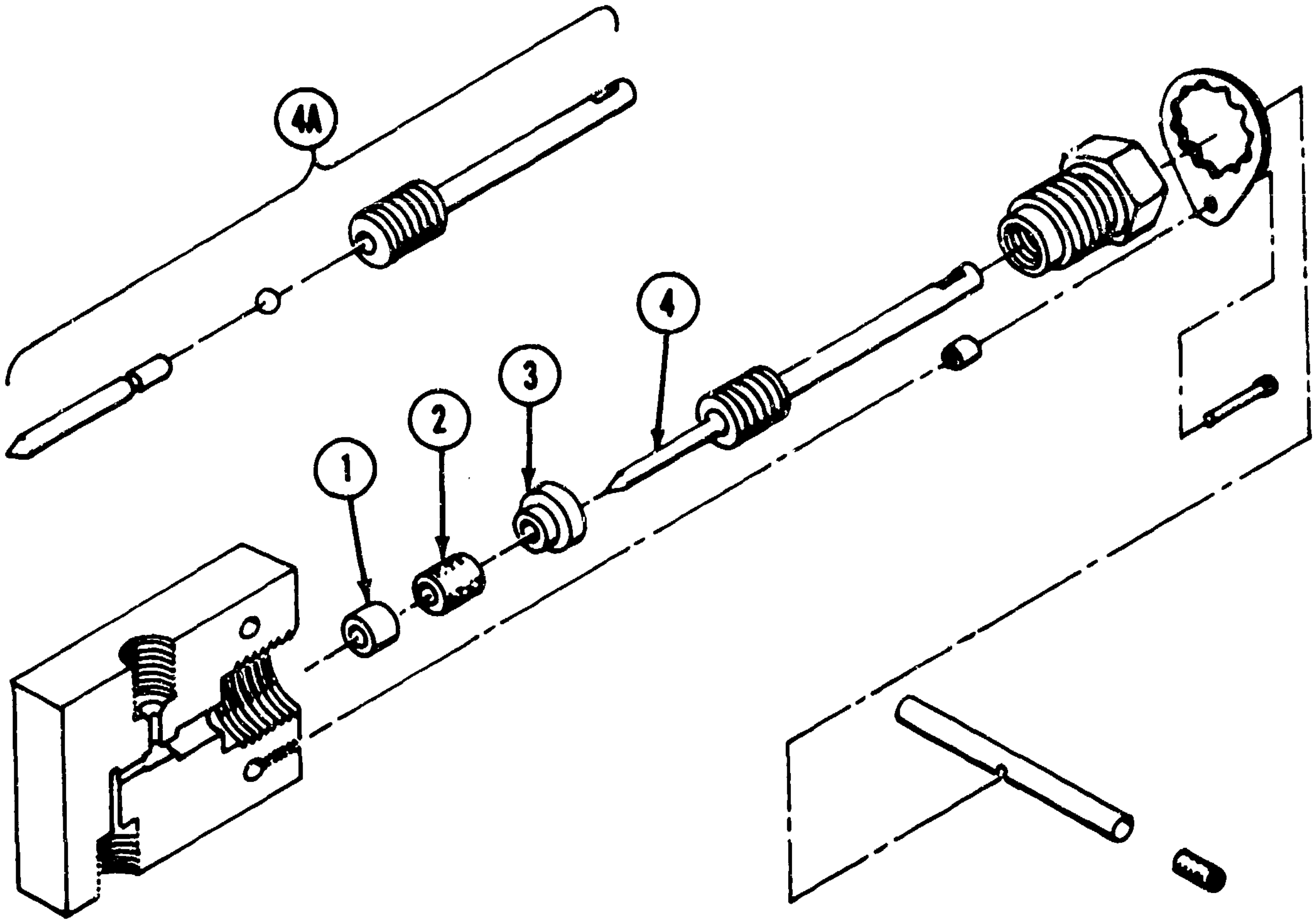
FIGURE 1. WARM CONVERTER FOR HIGH PRESSURE OXYGEN



A. ASSEMBLED

B. DISASSEMBLED

FIGURE 2. HIGH PRESSURE OXYGEN BOMB



KEY

1. Bottom Packing Washer
2. Glass Filled Teflon Packing
3. Top Packing Washer
4. Rotating Stem

4A. Non-Rotating Stem

FIGURE 3. HIGH PRESSURE VALVE

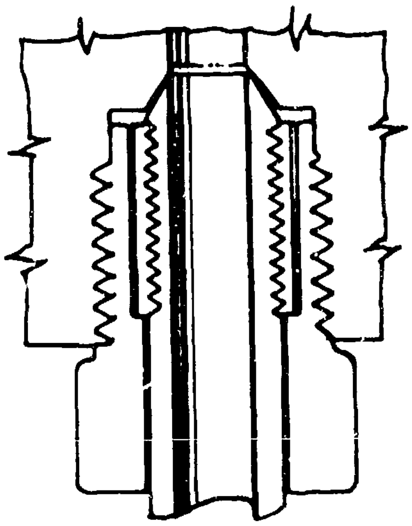


FIGURE 4. TUBING CONNECTION

EXPERIMENTAL PROGRAM

High Pressure Laboratory

Construction of Laboratory

All experimental tests were performed in two high pressure bays of a high pressure laboratory shown in Figure 5.

The bays of the laboratory are constructed of reinforced concrete walls with one blow-out wall. The blow-out wall faces a dirt hill at the rear of the laboratory. Heavy blast mats extend upward from the top of the hill to stop flying projectiles. The same type of blast mat is used to cover the ceiling and other critical points in each bay.

All valves and controls are operated from outside the test area by extensions through the heavy concrete wall (Figure 6). All visual observations are made from the operating area with the use of mirrors.

Physical Arrangement of Test Equipment

A common entrance door from the operating area serves both high pressure bays with the entry way separated from each bay by heavy steel plate. The warm converter was placed in one bay and the test equipment was placed in the other bay so as to isolate the high pressure oxygen generating equipment from the test equipment in the event of a failure in either section. Tubing connection was carried via a small tunnel between the two bays. Additionally the warm converter was sandbagged. This physical arrangement can be seen in Figure 7 which shows test equipment set up for the high pressure oxygen bomb test. Figure 8 shows the sandbagged warm converter.

Cleaning and Assembly of Test Equipment

All valves, fittings, tubing, and apparatus were disassembled and cleaned for oxygen service by standard vapor degreasing and washing methods. Threads of valve stems, tubing, and gland nuts were given a very thin film of fluorocarbon lubricant immediately prior to assembly. These threads are normally not exposed to high pressure oxygen because of weep holes in the fittings.

All liquid oxygen entering the warm converter was passed through a filter which is rated to remove 99.5% of all particles greater than one micron.

High Pressure Oxygen Bomb Tests

Purpose

The oxygen bomb test determines the spontaneous ignition or explosion temperature of a material in contact with oxygen at some given pressure. The test provides a means for determining the allowable temperature to which a material may be exposed in oxygen at the given pressure without the possibility

FIGURE 5. HIGH PRESSURE LABORATORY

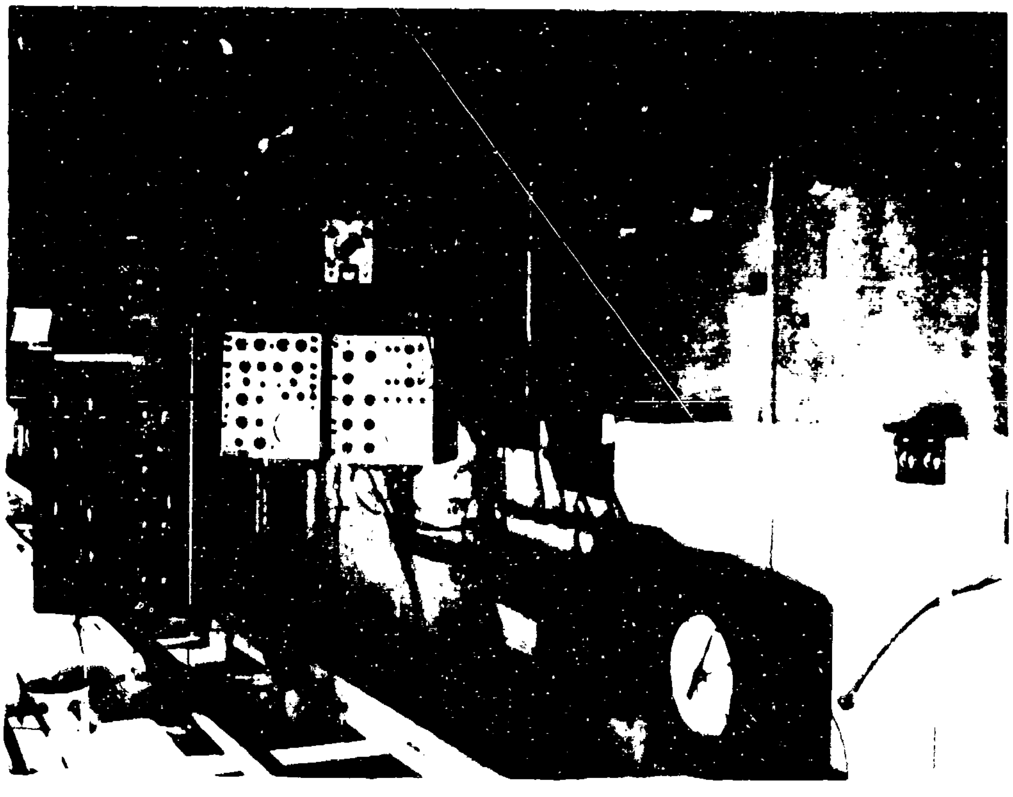


FIGURE 6. CONTROL AREA OF HIGH PRESSURE TEST EQUIPMENT

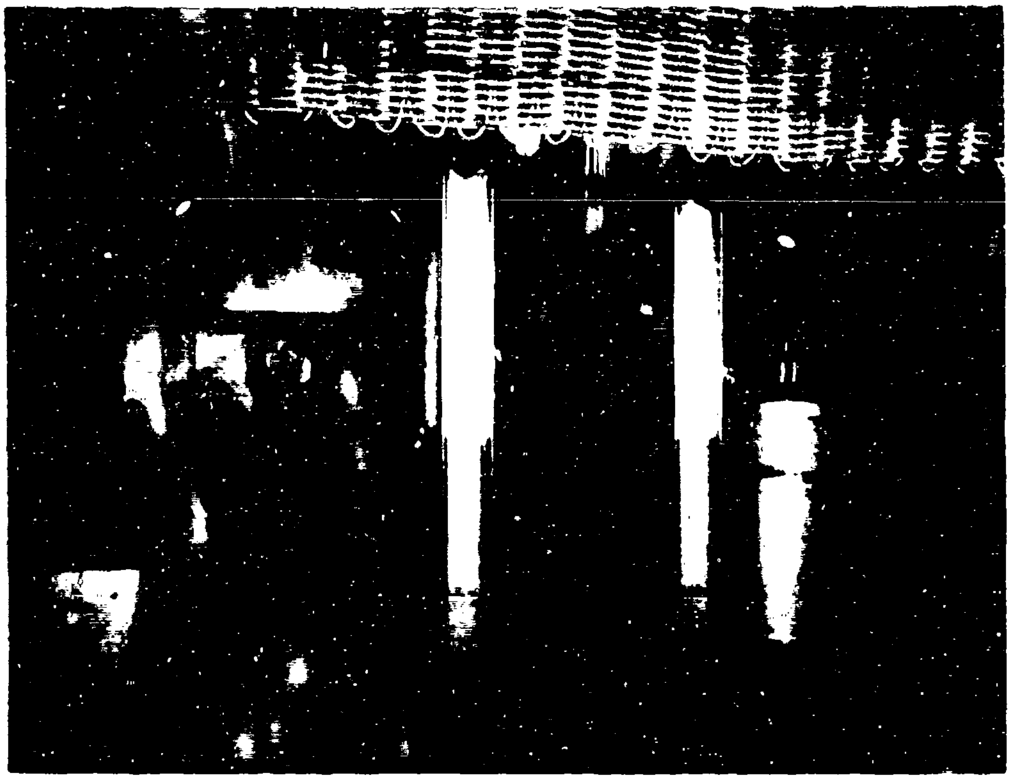


FIGURE 7. PHYSICAL ARRANGEMENT OF TEST EQUIPMENT IN HIGH PRESSURE BAYS

FIGURE 8. SANDBAGGED WARM CONVERTER

of spontaneous ignition. A material should not be used in oxygen service where its spontaneous ignition temperature might be approached; a substantial safety factor is normally provided.

Procedure

The test is made by placing a sample of the material in the oxygen bomb, pressurizing with oxygen, and heating the bomb until the spontaneous ignition occurs. The temperature and pressure are recorded at which the ignition takes place.

Preliminary Tests of Prototype Bomb and Final Bomb

1. Prototype Bomb

As mentioned earlier the use of an oxygen bomb with a small mass would have some advantages in the 7500 psi program over the design used for the standard Linde 2000 psi bomb which has a large mass. Therefore, a prototype bomb (Figure 9) was purchased for making preliminary tests at 2000 psi. It was rated for 10,000 psi at a temperature of 1000°F.

This bomb was essentially a piece of high pressure stainless steel tubing having a one-half inch inside diameter. Each end of the tubing was coned and threaded into a hexagonal cap with a coned seating surface. The opposite end of each cap had a 1/4" high pressure connection. A pencil iron-constantan thermocouple with an Inconel shield was introduced through one end and oxygen gas through the other end.

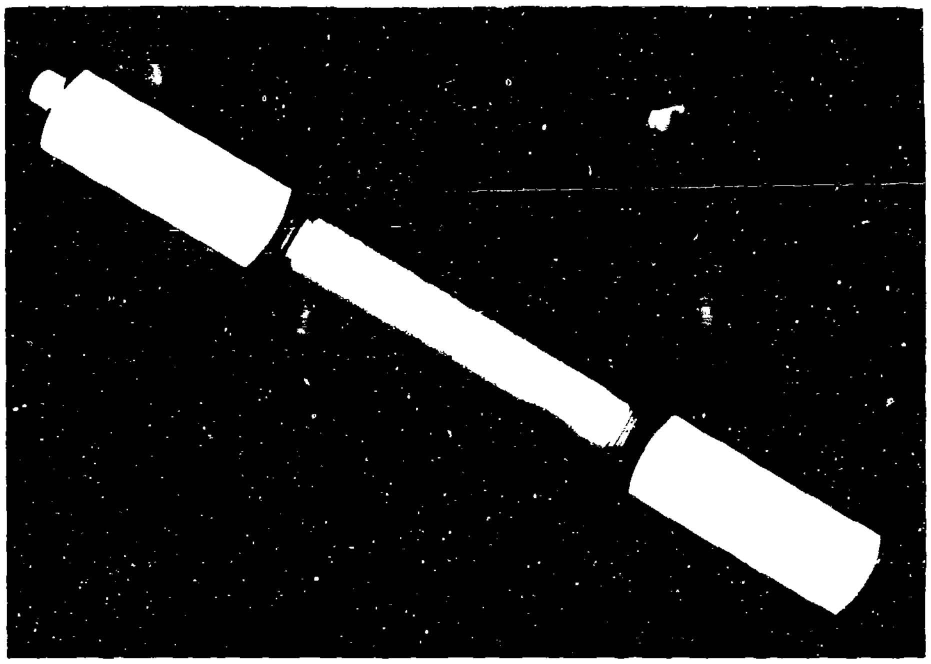


FIGURE 9. PROTOTYPE HIGH PRESSURE OXYGEN BOMB

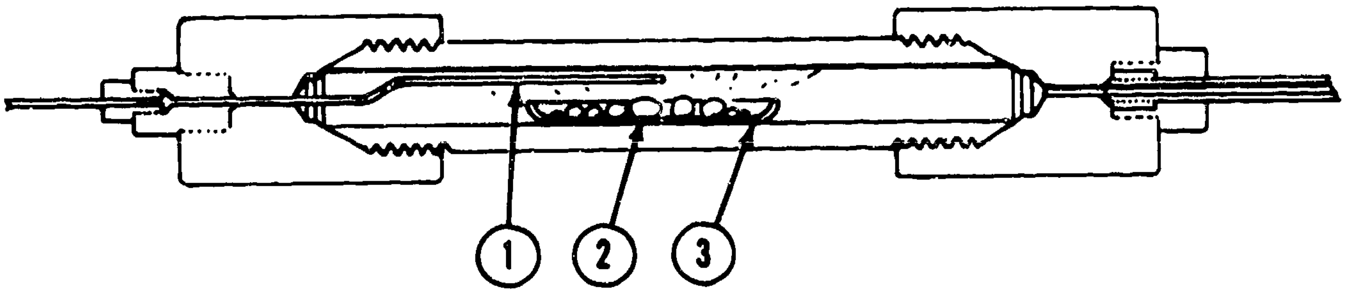


FIGURE 10. SCHEMATIC OF PROTOTYPE OXYGEN BOMB IN HORIZONTAL POSITION

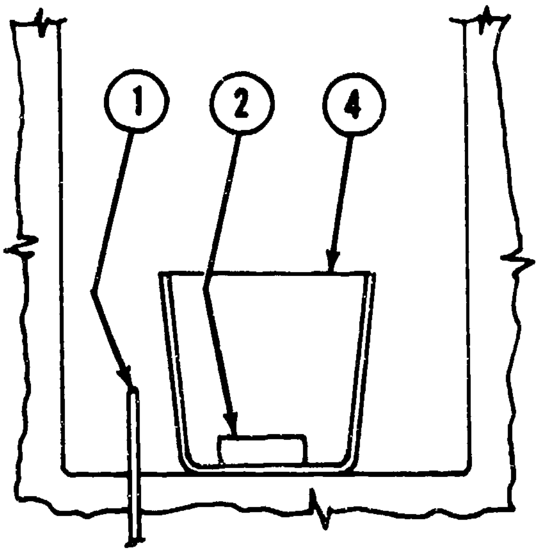


FIGURE 11. SCHEMATIC OF LINDE STANDARD OXYGEN BOMB

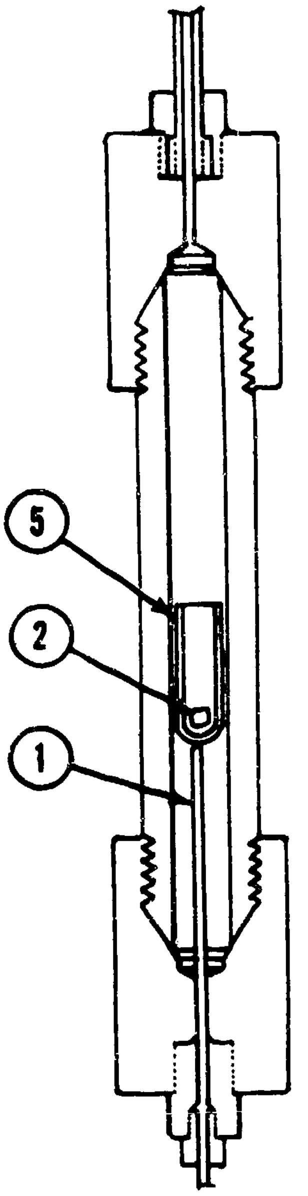


FIGURE 12. SCHEMATIC OF PROTOTYPE OXYGEN BOMB IN VERTICAL POSITION

KEY

1. Thermocouple
2. Specimen
3. Specimen Boat
4. Specimen Crucible
5. Specimen Test Tube

By testing materials which had previously been tested in the Linde standard bomb, it was possible to determine if comparable ignition temperatures were obtained. Initially the prototype bomb was tried in a horizontal position as shown in the schematic of Figure 10. The sample was placed in a quartz boat and slid in one end of the tube which was then placed in a tube furnace and heated. Results were unpredictable and one low-ignition material decomposed without the occurrence of spontaneous ignition.

Because spontaneous ignition is thought to occur in the gas phase by the decomposition products of the test material reaching an explosive concentration in the oxygen, it was postulated that the decomposition products were diffusing through the bomb as it lay in the horizontal position and not reaching an explosive concentration. In the Linde standard bomb (shown schematically in Figure 11), the sample is contained in a crucible and the gases collecting above the sample are confined to the crucible where they reach an explosive concentration.

By placing the prototype bomb in a vertical position (schematically shown in Figure 12) and using a 10 mm x 35 mm Pyrex glass test tube (without lip) as a sample holder, it was found that the spontaneous ignition temperature obtained on typical materials was the same as that found in the standard Linde bomb.

The prototype bomb, as pictured in Figure 13, was used to investigate many parameters of the bomb test to determine the effect on the spontaneous

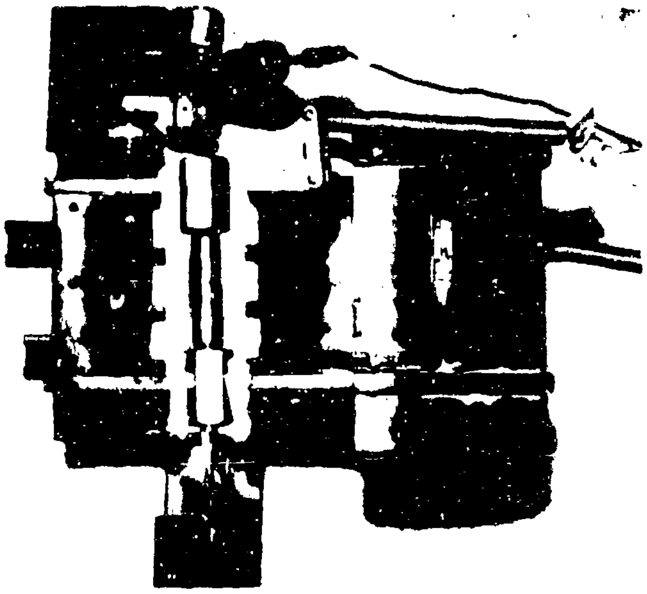


FIGURE 13. PROTOTYPE BOMB AS USED FOR PRELIMINARY TESTS

ignition temperature caused by variations in sample weight, heating rates, etc. The results of these tests are presented in Table 7 of Appendix IV. Heating rate and sample size of the materials tested were found to have little effect on the spontaneous ignition temperature.

During these tests the prototype bomb began to leak because scoring occurred at the seating interfaces of the tube and cap. The leakage could be stopped by resurfacing the seating surfaces or by the use of thin copper gaskets, but the leakage was severe enough at 2000 psi to make a new bomb design desirable for the 7500 psi tests.

2. Final Bomb

The geometric configuration of the prototype bomb had been proven out in preliminary tests, so the final bomb was designed using a 1/2" I.D. opening. Inconel X-750 was used as the material of construction because of its strength at high temperatures. A lens ring was used to provide a leak-tight seal as shown in a schematic of the bomb in Figure 14B.

A few tests with standard materials in the new bomb yielded high spontaneous ignition temperatures. This was demonstrated to be due to convection currents set up in the bomb by part of the bomb cavity at the top being outside the heating zone of the furnace. (The entire cavity of the prototype bomb was physically in the furnace.) A convection from the cold zone at the top to the warm zone at the bottom caused the decomposition products to diffuse. Thus an explosive concentration did not materialize until a higher temperature was reached.

This situation was corrected by making a lens ring plug as shown schematically in Figure 14A. This is the design finally used for all the high pressure tests. In preliminary tests made with this design at 2000 psi, it was found that the spontaneous ignition temperature of standard materials compared favorably with those determined in the standard Linde bomb and in the prototype bomb. Preliminary tests made on these same standard materials at oxygen pressures of 5500 to 9000 psi indicated that their spontaneous ignition temperatures at these higher pressures were going to be the same as that at 2000 psi.

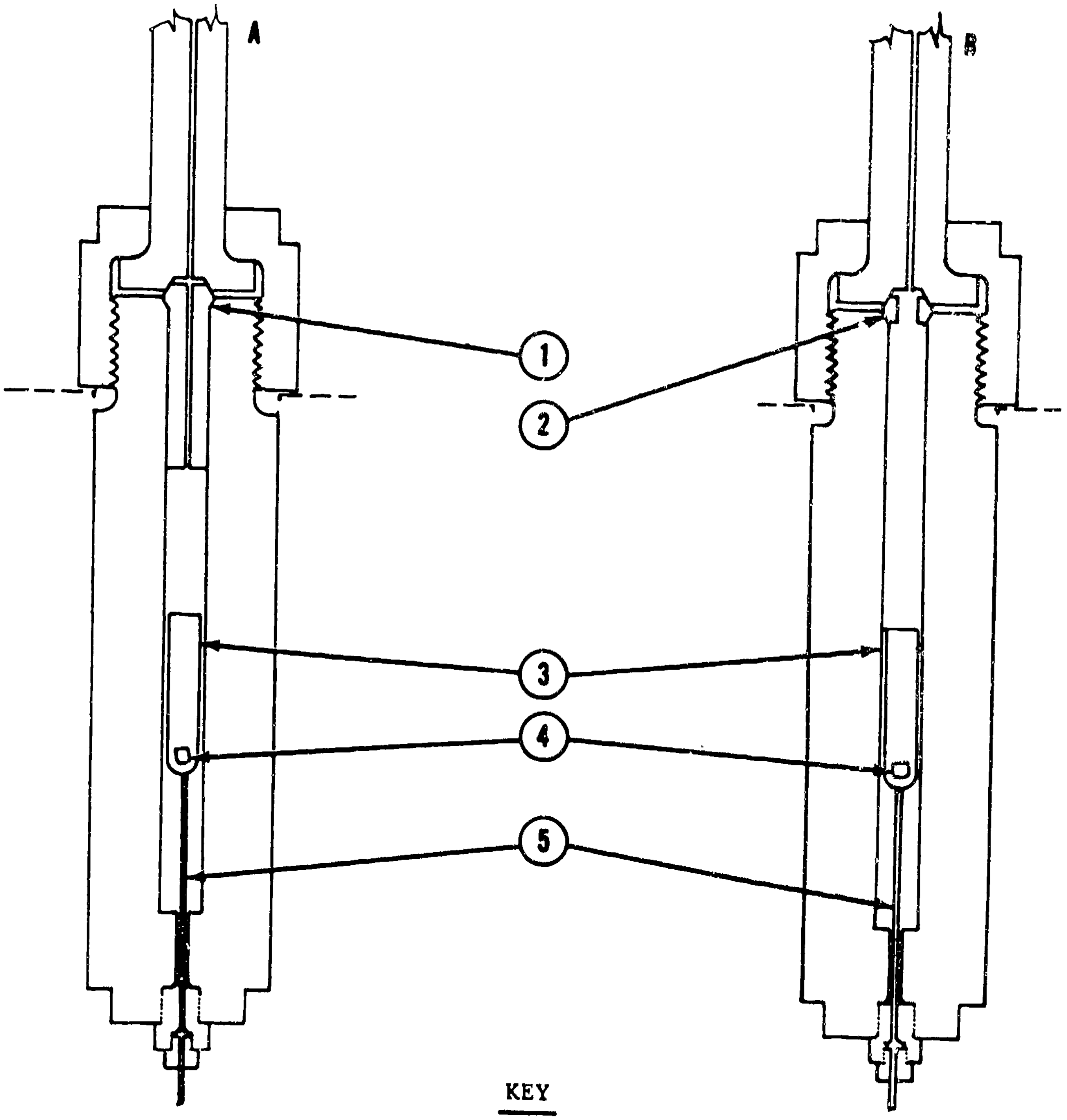
This preliminary data is also presented in Table 7 of Appendix IV.

7500 psi Tests

1. Test Set-up and Procedure

A schematic of the test set-up for the high pressure oxygen bomb is shown in Figure 15 and a pictorial view of the set-up is shown in Figure 16.

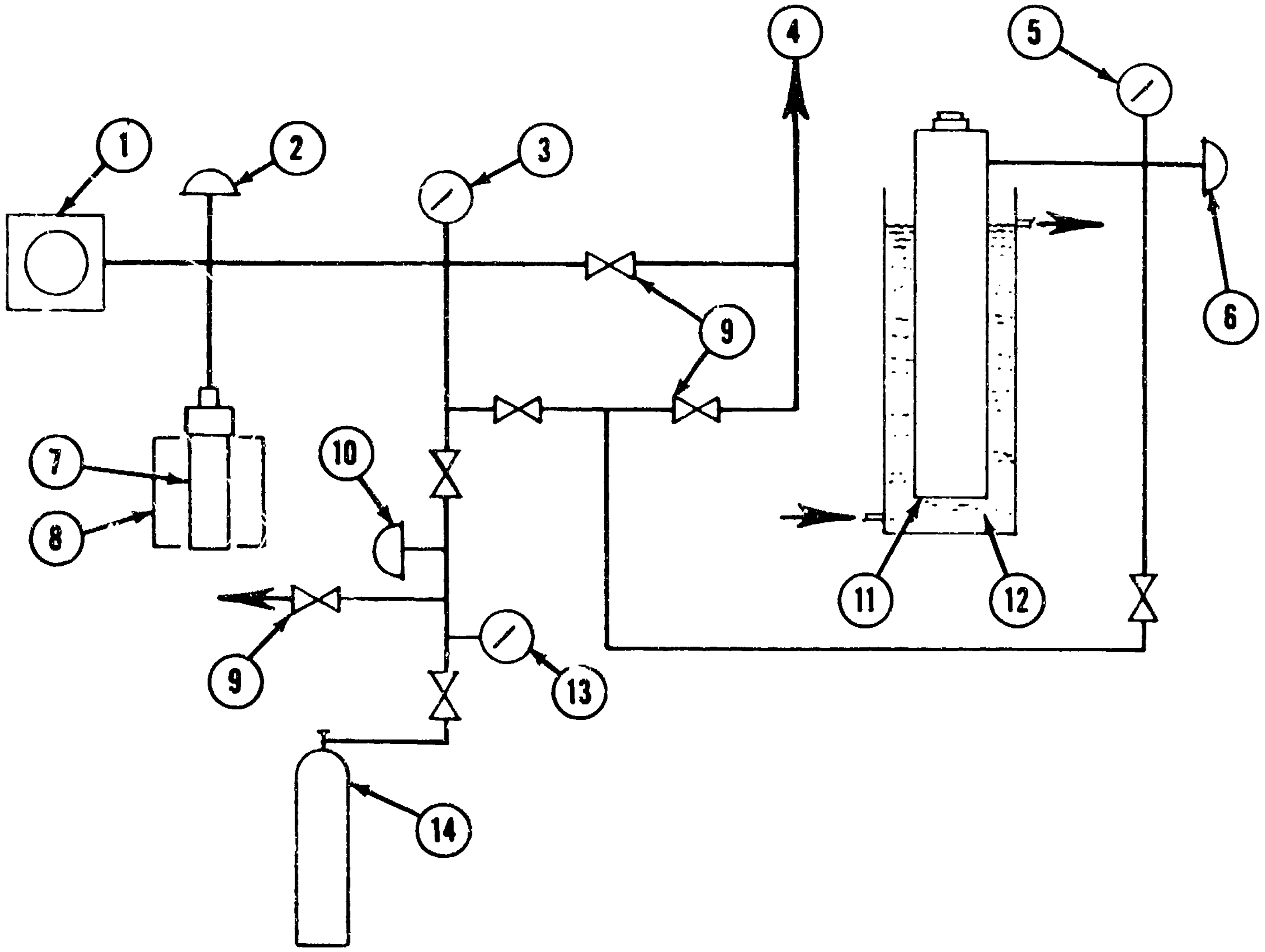
With the sample in the bomb, the bomb is successively pressurized with cylinder oxygen and blown down to atmospheric pressure four times. This is done slowly. Finally the bomb is pressurized with high pressure oxygen from the converter. The fill lines from the converter and the 2000 psi cylinder oxygen are blown down to isolate the bomb from oxygen sources. The bomb is then heated in the furnace until spontaneous ignition occurs.



KEY

1. Lens Ring Plug ("A" Only)
2. Lens Ring ("B" Only)
3. Pyrex Test Tube, 10 mm X 35 mm
4. Specimen
5. Thermocouple

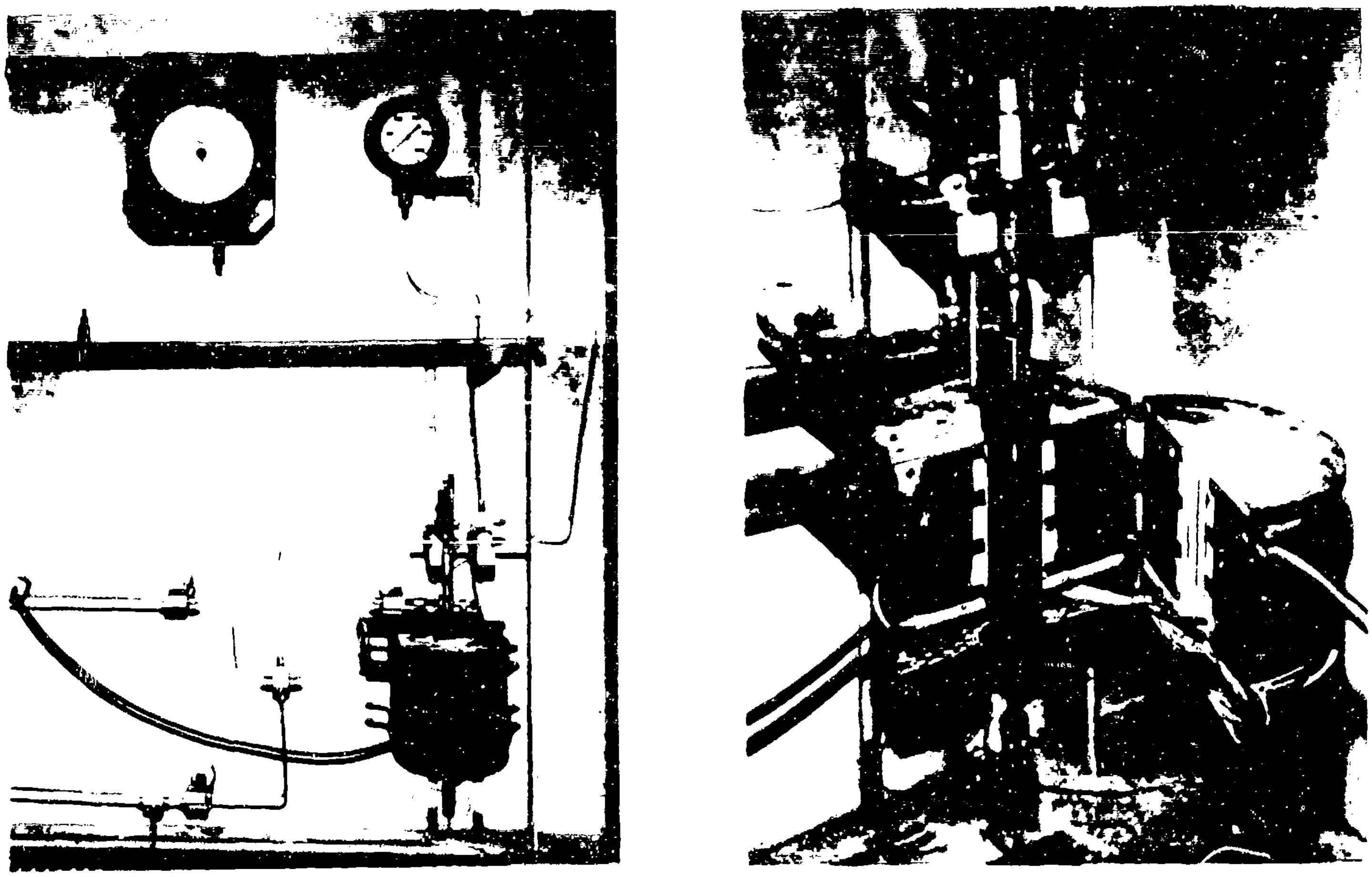
FIGURE 14. 10,000 PSI OXYGEN BOMB WITH TEST TUBE ENCLOSED



KEY

1.	10,000 psi Pressure Recorder	8.	Furnace
2.	10,000 psi Bursting Disc	9.	Blow Down Valve
3.	15,000 psi Pressure Gage	10.	3,000 psi Safety Disc
4.	Blowdown to Outside	11.	Warm Converter
5.	50,000 psi Pressure Gage	12.	Water Bath
6.	16,500 psi Bursting Disc	13.	3,000 psi Oxygen Gage
7.	Oxygen Bomb	14.	Cylinder Oxygen- 2,000 psi

FIGURE 15. SCHEMATIC FOR 7500 PSI OXYGEN BOMB TEST



A. COMPLETE SET-UP B. CLOSE-UP OF BOMB IN FURNACE

FIGURE 16. HIGH PRESSURE OXYGEN BOMB TEST SET-UP

Because the pressure in the bomb increases with temperature, it is necessary to select a starting pressure which will yield a final pressure of 7500 psi at the spontaneous ignition temperature of the material under test.

A variac was used to control the rate of heating of the furnace. For expected low ignition temperatures, the variac was set at a lower value than for expected high ignition temperatures. This produced comparable heating rates over the last twenty minutes prior to ignition.

2. Spontaneous Ignition Temperatures

Ignition temperatures were determined for each material in 7500 psi oxygen and in 2000 psi oxygen using the high pressure bomb and the same test parameters. The lowest ignition temperature found for each material in 7500 psi oxygen and in 2000 psi oxygen are presented in Table 3. Complete information on all materials tested may be found in Table 8 of Appendix IV. In general, the ignition temperatures at 7500 psi were found to be the same as those at 2000 psi.

Results with Oxylube No. 703 demonstrate the fact that with some materials the weight of the sample is significant in determining its spontaneous ignition temperature in the bomb. Results with Oxweld Anti-Friction Compound

No. 64 also demonstrate the effect of weight with some materials and results with Everlube No. 811 demonstrate the effect that the physical geometry of the sample can have on the ignition point.

After a conversation with personnel of the Swagelok Company, the results with High Purity Goop were attributed to the possible non-homogeneity of the product. High Purity Goop is a mixture of fluorocarbon materials and one test sample may have contained more of one component than that contained in another sample.

3. Evaluation of Results

a. Thread Lubricants

A study of ignition temperatures presented in Table 3 shows the fluorocarbon lubricants to have high spontaneous ignition temperatures.

Dixon's Flake Graphite, Burnil Brand Microplates, and Almasol Powder did not ignite at 500°C and therefore may be applicable to lubrication problems in high pressure oxygen. However, experience with these materials is lacking and other problems that might be associated with their use would need to be ascertained.

Molykote Z and products formulated with molybdenum disulfide, Everlube No. 811 and Oxylube No. 703, have low ignition temperatures and are not recommended for use in either 7500 psi or 2000 psi oxygen service.

b. Thread Sealants

Ignition temperatures of those thread sealants tested are all approximately the same except for Mano Pipe and Joint Compound and 50-50 soft solder. If the temperature rise and pressure rise when ignition occurs (Table 8 of Appendix IV) are evaluated in reference to the sample weight, it appears that ignition of Key Abso-Lute or Linde Green Pipe Joint Compound results in the liberation of less energy than the ignition of Rectorseal No. 15 or Parker Oxyseal.

Mano Pipe and Joint Compound has a relatively high ignition temperature and a comparatively small amount of energy release but Linde experience with the sealing characteristics of the product show that it does not meet the specifications required for high pressure oxygen service. However, further development of this product might make its sealing characteristics acceptable.

Soft solder did not ignite at 500°C, but its melting point is 225°C. It would not present any ignition hazard but evaluation of its use as a sealant by tinning of threads would need to be made.

c. Fluorocarbon Plastics and Elastomers

Teflon (TFE) and filled TFE products have the highest spontaneous ignition temperatures with Kel-F 81 being next highest. The ignition temperatures of the fluorocarbon elastomers are substantially lower than those of the fluorocarbon plastics.

TABLE 3

LOWEST SPONTANEOUS IGNITION TEMPERATURE FOUND IN 7500 PSI OXYGEN AND IN 2000 PSI OXYGEN, °C

	7500 psi	2000 psi
Thread Lubricants		
Aroclor 1254	355	376
Dixon's Flake Graphite No. 1	No ignition 500°C	No ignition 500°C
Kel-F 90 Grease	435	435
Oxweld Anti-Friction Compound No. 54	230	237
Burnil Brand Microplates	No ignition 500°C	No ignition 500°C
Molykote Z	267	277
Halocarbon Oil Series 13-21	435	427
Halocarbon Grease Series 25-10	438	431
Oxweld Anti-Friction Compound No. 64	410	410
Everlube No. 811	216	250
Oxylube No. 703	190	238
High Purity Goop	411	398
Almasol Powder	No ignition 500°C	No ignition 500°C
Thread Sealants		
Oxyseal	347	360
Mano Pipe and Joint Compound	422	430
Key Abso-lute	342	355
Rectorseal No. 15	355	374
Linde Green Pipe Joint Compound	362	356
50-50 Soft Solder	No ignition 500°C	No ignition 500°C
Fluorocarbon Plastics and Elastomers		
Viton A (Virgin)	300	310
Viton B (Virgin)	316	325
Teflon (Virgin)	465	469
Teflon 100 X	410	413
Rulon A	465	463
Rulon B	460	466
Rulon C	465	458
Duroid 5600	470	468
Duroid 5650	444	461
Duroid 5870	463	452
Duroid 5813	463	463
Kel-F 81	425	431
Kel-F Elastomer 3700	332	341
Kel-F Elastomer 5500	340	352
Rubber Compound		
Neoprene	190	200

TFE products and Kel-F 81 both liberate only a small amount of energy on ignition, indicated by the low temperature and pressure rise on ignition when compared to that of other materials.

4. Conclusions

a. Because the spontaneous ignition temperatures of the majority of the materials tested can be attained by adiabatic compression, only those that did not ignite at 500°C can be recommended as not presenting a hazard. Further investigation would be required to determine other problems that might be associated with their use.

b. It is recognized that some type of material is needed for dynamic and static seals. The use of TFE or glass-filled TFE in these instances would be recommended because of the low energy release of TFE. However, these would be recommended only if tightly confined. Because TFE, per se, cold flows, the glass-filled TFE would receive a higher recommendation.

Adiabatic Compression Tests

Purpose and Discussion

Originally it was thought that this test could be used to test specimens of various metallic and non-metallic materials to show that the material could or could not be ignited by adiabatic compression. Calculation of theoretical temperatures attainable by adiabatic compression (Appendix I) indicated that the melting points of most metals to be tested would be exceeded. Because these temperatures would not be reached in actual practice, it was thought that experimental tests would be in order to determine the peak temperatures attainable.

Reynales [(13)] reports that temperature rises of 560°F were measured by sudden opening of a shut-off valve and pressurizing with 2200 psi nitrogen. In shock tube experiments with oxygen, peak temperatures of over 2000°F were recorded and all materials tested including Teflon, Kel-F, Buna-n, and butyl rubber were burned. Greenspan[(5)] reported ignition of Teflon and Kel-F by adiabatic compression.

A shock tube could have been used in the present program for this type of experiment but this would require the use of a bursting disc and the likely probability that a piece of the bursting disc might impact the specimen and invalidate results. It was also thought that a shock tube would not duplicate any condition that might exist during charging of a receiver at 7500 psi. Therefore, a quick-opening air-operated valve was purchased for use in adiabatic tests.

Preliminary Tests

Before the air-operated valve was received, a few crude tests were made at 2000 psi to determine possible test parameters. The tests were made using the apparatus shown in Figure 17. Both nitrogen and oxygen at 2000 psi were used for these tests and a small test volume varying from 10 cc to 50 cc was suddenly charged to 2000 psi by opening a ball-cock valve by hand as quickly

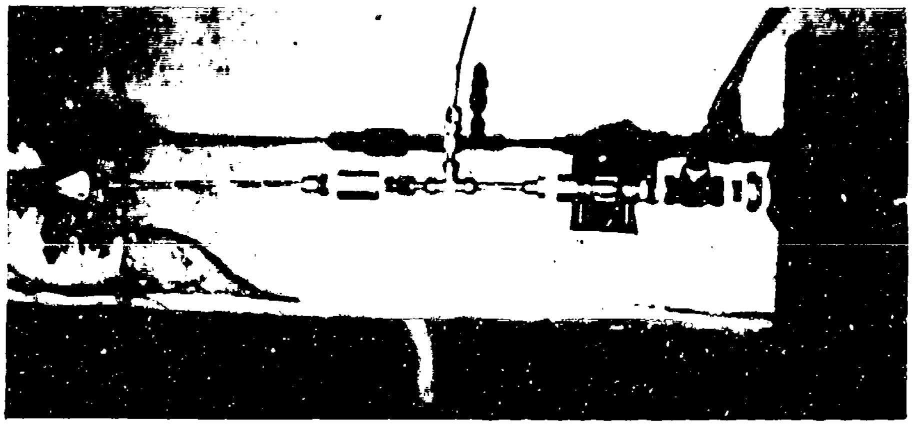


FIGURE 17. PRELIMINARY ADIABATIC COMPRESSION TEST SET-UP

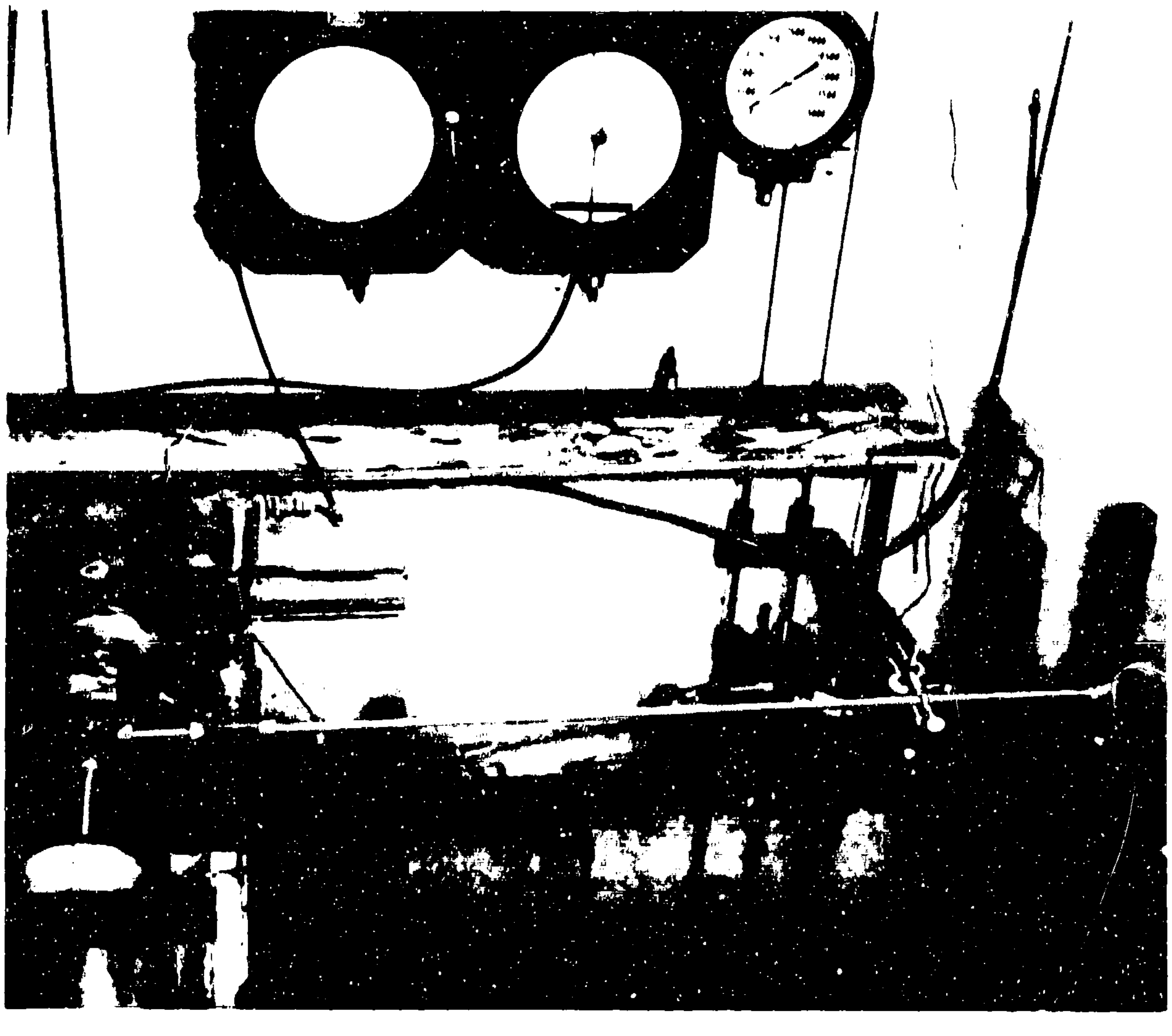


FIGURE 18. 7500 PSI ADIABATIC COMPRESSION TEST SET-UP

as possible. Results of these tests are not tabulated because of information which was developed later. It will suffice here to say that the maximum rise in temperature measured was only 93°C but that two-milligram slivers of lead were melted in this apparatus which indicated that temperatures in the order of 327°C were momentarily reached.

7500 psi Tests

When the quick-opening air-operated valve was received, experimental tests at 2000 psi showed that the opening time of the valve was in the order of 0.5 second and the pressurization time of a 20-inch section of 5/16" I.D. tubing required 0.05 second. Calculations based on these measurements indicated that the temperatures developed would not begin to approach theoretical. (See Appendix III). Consequently the use of this test became unattractive in the testing of metals, and because it was already known that organic materials could be raised to their spontaneous ignition temperatures by 2000 psi oxygen,(5) there was no advantage in further use of the test.

The air-operated valve (with 5/16" I.D. ports) was purchased for a dual purpose. It was to be used in the charging of a simulated system. While setting up for the simulated system the opportunity to make some quick temperature measurements presented itself. The tip of a 1/16" inconel shielded pencil thermocouple was removed to expose the bare iron-constantan junction. This was placed at the end of various lengths of 5/16" I.D. tubing. The tubing was then suddenly pressurized from an intermediate storage vessel by opening the air-operated valve. During these quick tests a maximum temperature of 378°C was recorded when a 51-inch section of tubing was pressurized to 6650 psi. Temperature measurements beyond this pressure were not made. The arrangement for the test is shown in Figure 18 and a schematic of the set-up in Figure 19.

At a later date, samples of Kel-F 81, virgin Viton A, and virgin Teflon were quickly tested in the same set-up by placing the specimens at the dead end of the tube and suddenly pressurizing them to pressures ranging from 6300 to 6900 psi with oxygen. All specimens spontaneously ignited. Results are reported in Table 12 of Appendix IV. During these tests a pressure transducer and recorder in the system indicated that pressurization occurred in 0.05 sec. (Figure 20). Results of these tests only proved that the ignition temperatures of these materials could be reached in this set-up which was expected.

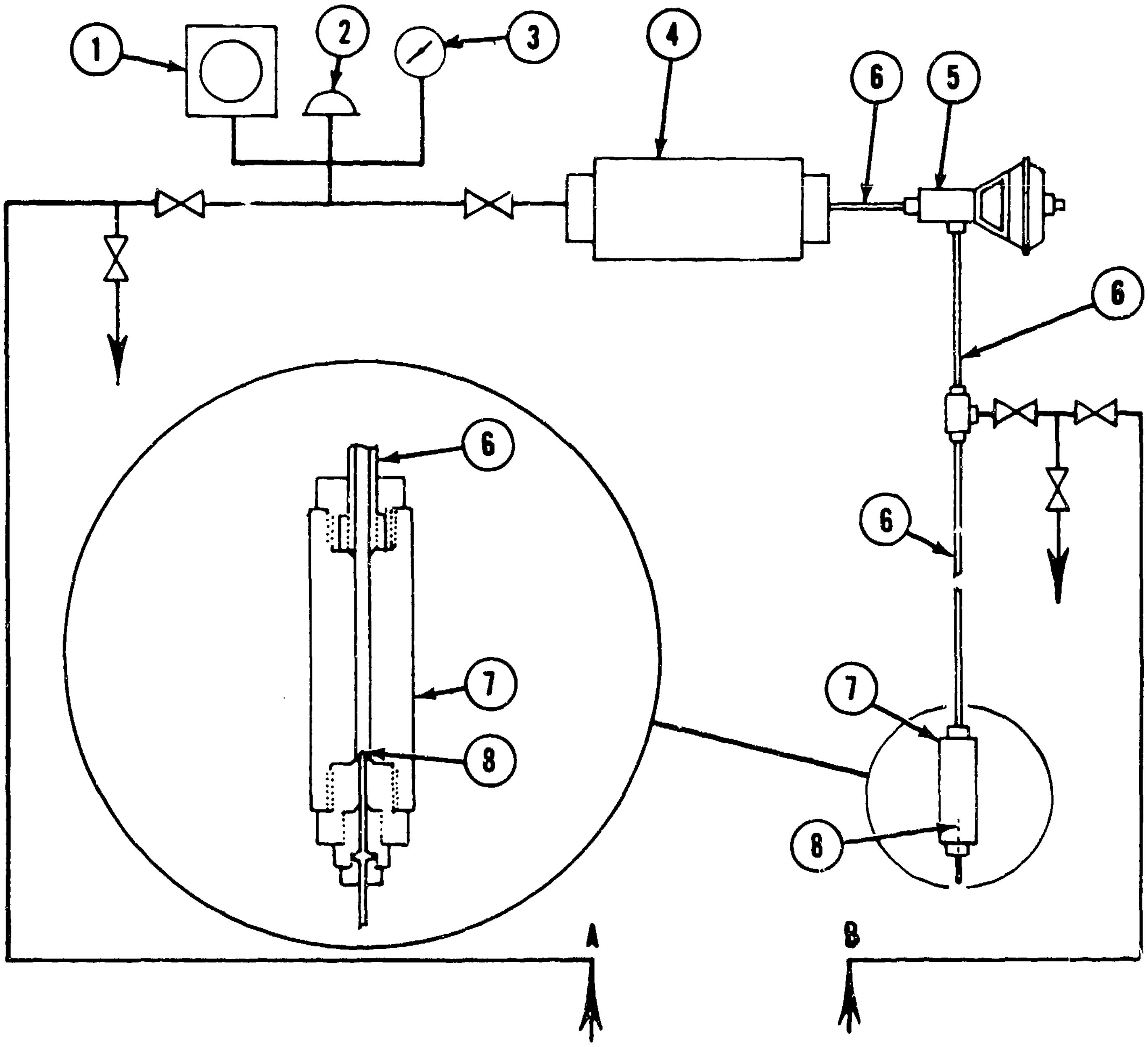
Promoted Ignition Tests

Purpose

The promoted ignition test is a means of evaluating the relative resistance to ignition and combustion of various metals by subjecting the metal specimen to the energy released when a promoter material is heated to its spontaneous ignition temperature in oxygen at a given pressure. The test from a practical standpoint shows what can happen when an elastomeric material, such as a gasket or O-ring, ignites in an oxygen system.

Procedure

The procedure for this test is the same as the procedure for the oxygen bomb test but a metal specimen as well as the promoter material is placed in the Pyrex test tube. Refer to Figure 15 for a schematic of the test set-up.



KEY

1. 0-10,000 psi Recorder
2. 10,000 psi Bursting Disc
3. 10,000 psi Gage
4. Storage Vessel
5. Air-Operated Valve
6. 5/16" I.D. Tubing
7. 5/16" I.D. Coupling
8. 1/16" Thermocouple or Specimen of Material

A. High Pressure Oxygen from Converter
B. 2000 psi Cylinder Oxygen

FIGURE 19. SCHEMATIC FOR ADIABATIC COMPRESSION TEST

Test No. 11040-41

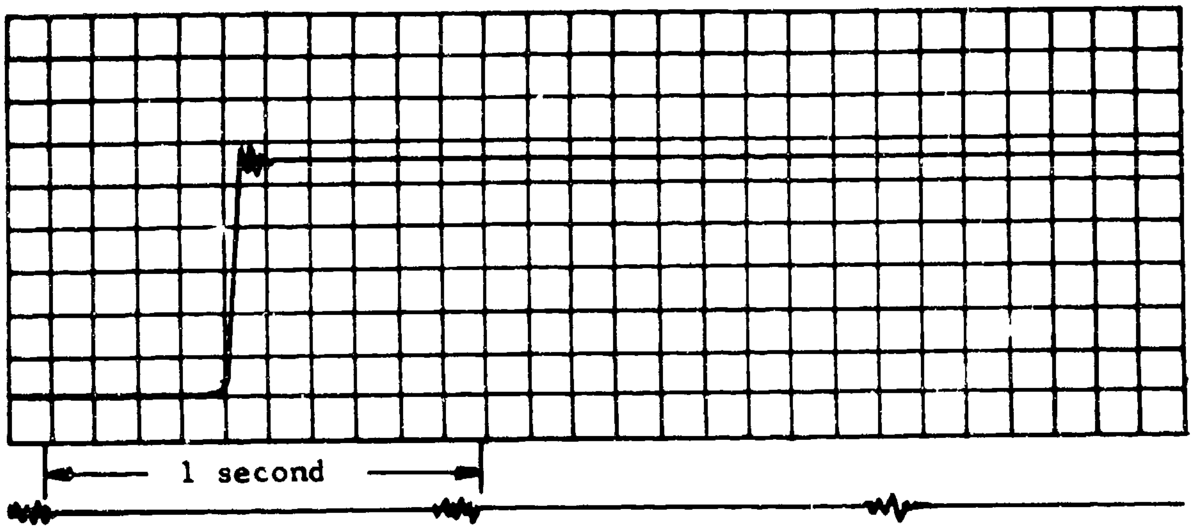


FIGURE 20. PRESSURE PROFILE OF ADIABATIC COMPRESSION TEST

Preliminary Tests

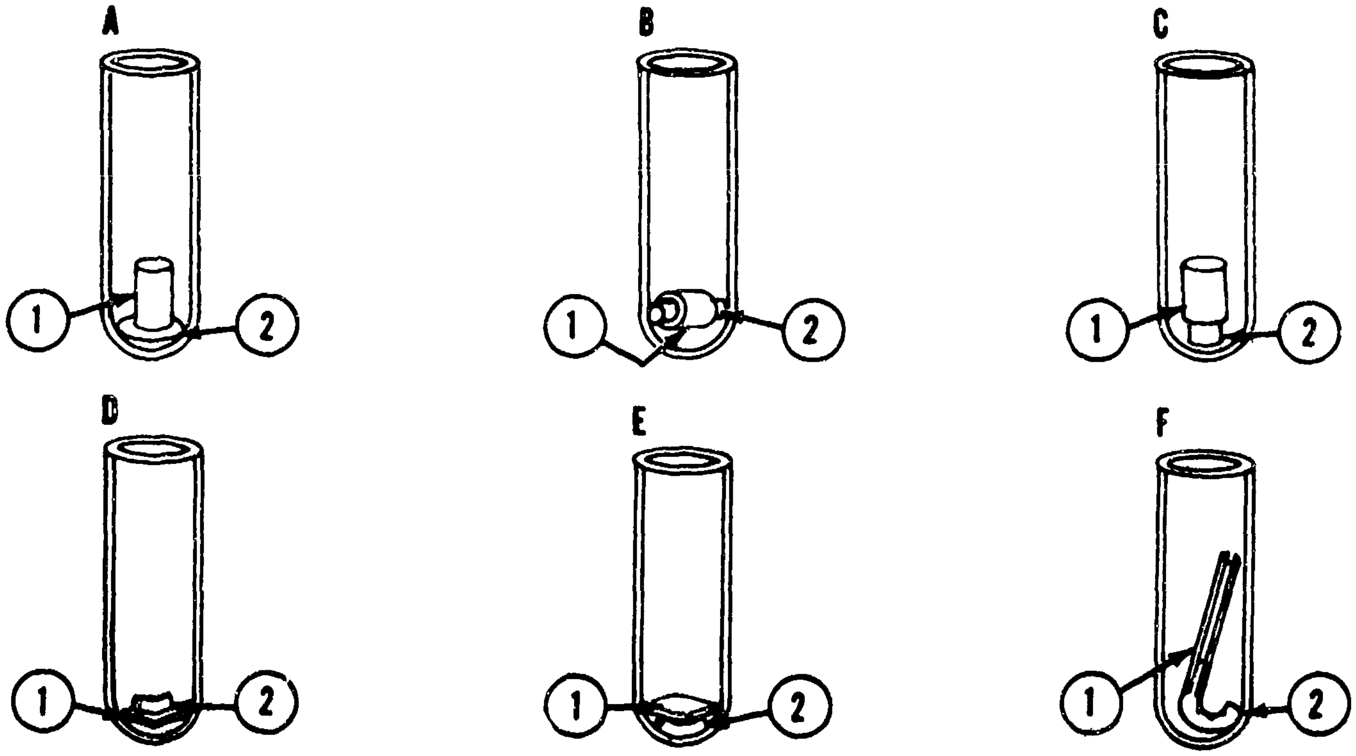
It was necessary to determine the parameters which would give reproducible and comparable results on the various metals to be tested. Preliminary tests were carried out at 2000 psi. A review of promoter materials had narrowed the choice to neoprene or Viton A. Two or three tests were made with Viton A but neoprene was selected because of its lower ignition temperature and the possibility that decomposition products from the Viton A might effect the outcome of the test.

Neoprene O-rings were ordered for these tests and purchased with the understanding that all O-rings would be from the same batch of neoprene in order to obtain uniform spontaneous ignition temperatures. However, difficulty was experienced in that the ignition temperature of the neoprene varied substantially which is typical with this type of compound. This did not seem to materially affect the test results but it did cause the pressure at the spontaneous ignition temperature to vary because of the pressure dependency on the temperature of the bomb.

Various weight and size combinations as well as physical relationships were tried until one was found which would yield comparable results. These various configurations are schematically shown in Figure 21.

Metal specimens of 0.005-inch thick foil were used. Certified analyses of the metal foils were obtained. Original plans were either to use a constant weight of promoter and vary the physical dimensions of the metal specimen or to use fixed physical dimensions for the metal specimen and vary the weight of the promoter. The latter condition appeared to be more attractive after completion of all the 2000 psi tests and some of the 7500 psi tests. The final physical configuration chosen for the 7500 psi tests is that shown in Figure 21F.

Twenty-nine preliminary tests were performed at 2000 psi (Table 9 of Appendix IV). However, when tests were started at 7500 psi it was discovered



KEY

A. Formed a cylinder of the metal specimen to fit inside the neoprene O-ring. Specimen in a vertical position.

B. Rolled the neoprene O-ring within a cylinder of the metal specimen. Ends were open and specimen in a horizontal position.

C. Formed a cylinder of the metal specimen and lightly crimped the promoter into the bottom of the specimen which was in a vertical position.

D. Metal specimen in a flat horizontal position and the neoprene on top of the specimen.

E. Metal specimen in a flat horizontal position and the neoprene underneath the specimen.

F. Formed a half-cylinder from a long metal specimen with the neoprene crimped into the bottom of the specimen which was always in a semi-vertical position.

1. METAL 2. NEOPRENE

FIGURE 21. PHYSICAL CONFIGURATIONS USED WITH PROMOTER MATERIAL AND METAL SPECIMENS IN PYREX TEST TUBE DURING PROMOTED IGNITION TESTS

that results were significantly different from those obtained in the 2000 psi tests in that the weight of neoprene required to bring about combustion of the metal was less. This was attributed to the geometric configuration of the bomb, the physical position of the metal specimen in the test tube during preliminary tests, and the difference in oxygen pressures. All of these factors would favor the blanketing of the metal specimen by the ignition products of CO_2 and H_2O formed during combustion of the neoprene. At the lower pressure of 2000 psi the combustion products would occupy a much greater volume than at 7500 psi.

Although the tests at 2000 psi were informative it was necessary to develop new parameters at 7500 psi. Original intentions had been to perform identical tests at 2000 psi and at 7500 psi for comparison purposes, but tests at 2000 psi now had to be eliminated and only the 7500 psi tests were made.

7500 psi Tests

1. Discussion of Test

After completion of a number of tests at 7500 psi, a geometrical configuration of 5 millimeters by 30 millimeters was selected as the standard size for the 5-mil metal foil samples. By determining the weight of neoprene necessary to combust, partially combust, melt, or partially melt a specimen, the relative resistance to ignition and combustion was obtained for the metals and alloys.

The metals and alloys have been arranged in Table 4 in order of their decreasing resistance to ignition as determined by these tests in 7500 psi oxygen. Complete data on the tests is presented in Table 10 of Appendix IV.

It had been hopefully expected that we might be able to extract some temperature data from the pressure profiles in the bomb during the ignitions and a pressure transducer was connected into the bomb system. The pressure was recorded on a Sanborn 150 recorder which permitted suppression of several thousand pounds of pressure and, by means of an attenuator, the recorder could be spanned over a narrow pressure range if it were so desired. After a large number of tests and a study of the pressure profiles, the conclusion was reached that prediction of ignition temperatures could not be determined from the profiles. A typical pressure profile is shown in Figure 22.

2. Discussion of Results

a. From Table 4 it can be seen that nickel is by far the most resistant to ignition (gold and silver excepted) but if enough energy is present nickel will burn.

b. A standard specimen of Monel alloy 400 was never completely combusted so the amount of neoprene shown in Table 4 to completely combust the standard size specimen was estimated. The required weight of neoprene might actually be higher than the estimate which would improve the relative position of Monel 400.

TABLE 4

METALS AND ALLOYS ARRANGED IN ORDER OF DECREASING RESISTANCE TO IGNITION AND COMBUSTION IN 7500 PSI OXYGEN

Metal or Alloy	Weight of Neoprene to Completely Combust Standard Specimen 5 mm x 30 mm x 0.005"
Gold	Only melts
Silver	Only melts
Nickel	48 to 56 mg*
Monel alloy 400	18 to 19 mg*
Yellow Brass (partial combustion only)	11.8 to 15.2 mg
Inconel alloy 600	13.2 mg
Aluminum	11.0 to 16.4 mg
Copper	10.5 mg*
Inconel alloy X-750	9.0 mg
Stainless steels	7.1 to 8.5 mg

*Estimated from results of a number of tests which were either standard with only part of the specimen consumed or were not standard and either complete or partial combustion occurred.

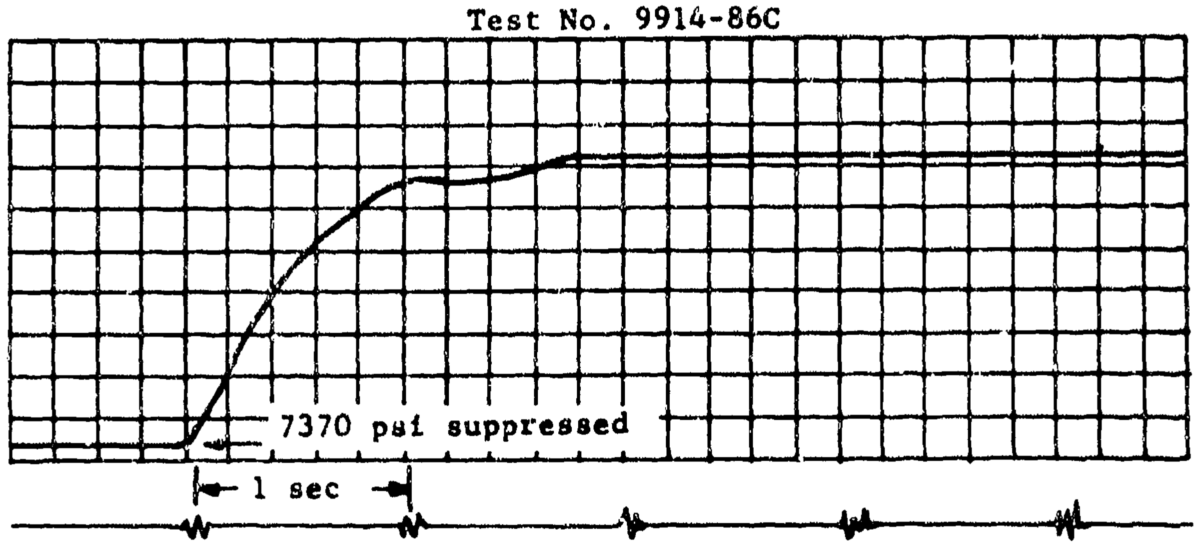


FIGURE 22. PRESSURE PROFILE OF PROMOTED IGNITION TEST

c. It has been possible to demonstrate with some of the metals (e.g. copper, brass, Monel 400, and nickel) that ignition can be started and part of the specimen combusted without consuming all of the specimen. By varying the weight of neoprene only slightly, it has been possible to combust a small part of the specimen, a large part of the specimen, or all of the specimen. This is characteristic of these metals and demonstrates their quenching effect once ignition has started. This is no doubt due to the low heats of combustion of these materials. Evidence of this is shown in Figures 26, 27, 29, and 30. Figures 23, 24, 25, 28, 31 and 32 show other ignition results.

d. Lead was not listed in Table 4 because 12.6 mg of neoprene melted the specimen into a ball with very little oxidation and no further tests were run. Therefore it was difficult to position it in the table.

e. Aluminum was tested with two different anodized surface thicknesses and also with what was thought to be normal surface condition if just exposed to the atmosphere. The amount of neoprene necessary to ignite the aluminum was not as clear cut as with other metals. However a range was established between 11.0 and 16.4 mg. Once ignited, the aluminum burned violently.

f. Gold and silver required more neoprene to melt the specimens than was required to ignite most of the other metals.

g. Brass had high resistance to ignition but it was always heavily oxidized.

h. Stainless steels were least resistant to ignition.

i. Samples of stainless steel enclosed in 5-mil Kel-F and 5-mil Teflon FEP were tested. This was intended to simulate a coating of these materials on the metal. Complete ignition occurred in all cases.

j. Two samples of aluminum coated on one side with Kel-F were tested. The Kel-F disappeared in one case without ignition and ignited in the other case. The aluminum was not affected in either case.

3. Evaluation of Test Results

a. With the exception of aluminum, results of these tests generally agree with those previously made at Linde in 2000 psi oxygen and with those of Dean and Thompson (3). (See Table 1.)

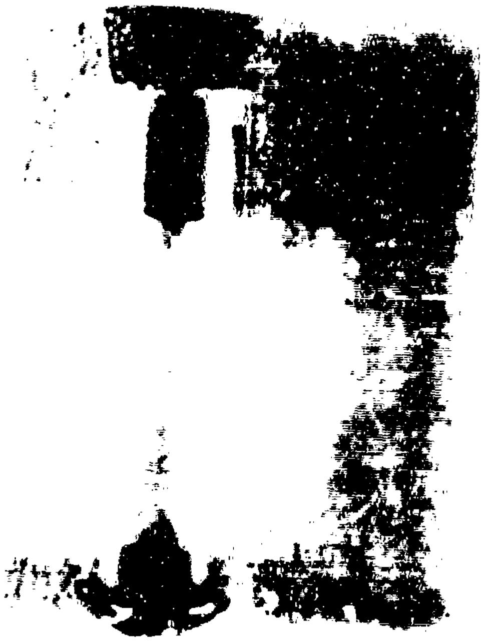
b. Nickel has the highest resistance to ignition and combustion. Therefore it would be the first choice for construction of a high pressure oxygen receiver from the standpoint of compatibility with oxygen.

c. Monel alloy 400, Inconel alloy 600, and brass, would be rated as the next best materials.

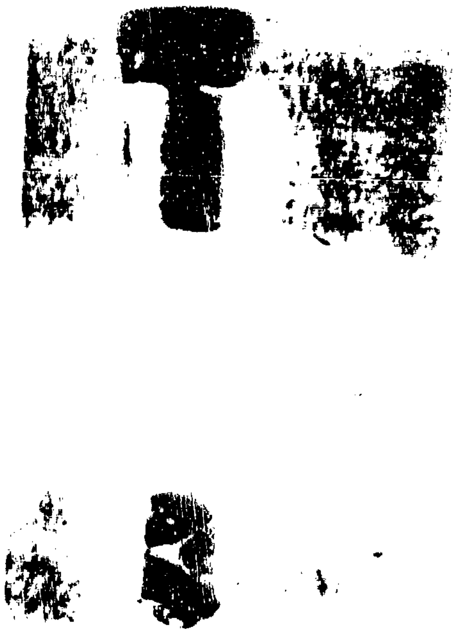
92 D
7.1 mg NEOPRENE
153 mg 301 S.S.
5 mm X 30 mm
7600 PSIG

90 A
7.2 NEOPRENE
147 mg 301 S.S.
5 mm X 30 mm
8220 PSIG

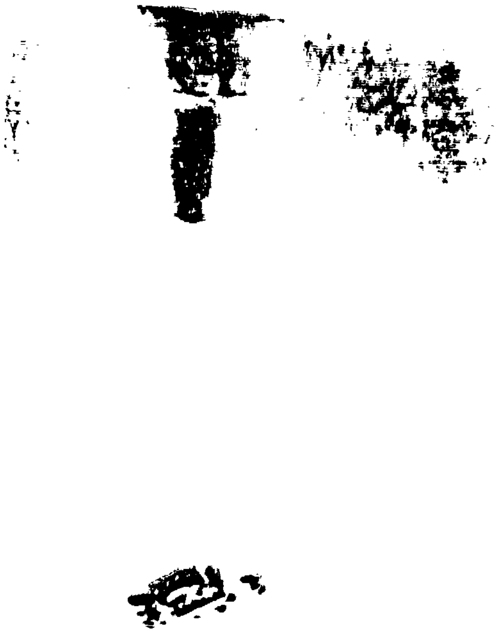
90 B
9.5 mg NEOPRENE
161 mg 301 S.S.
5 mm X 30 mm
8090 PSIG



73 C
8.5 mg NEOPRENE
161 mg. 304 S.S.
5 mm X 30 mm
7000 PSIG



42 B
8.8 mg NEOPRENE
168 mg 17-7 PH, S.S.
5 mm X 30 mm
8700 PSIG



94 A
9.0 mg NEOPRENE
171 mg 316 S.S.
5 mm X 30 mm
8700 PSIG

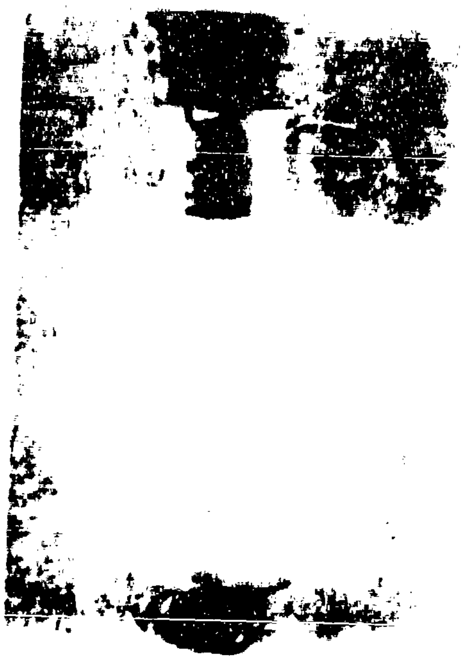
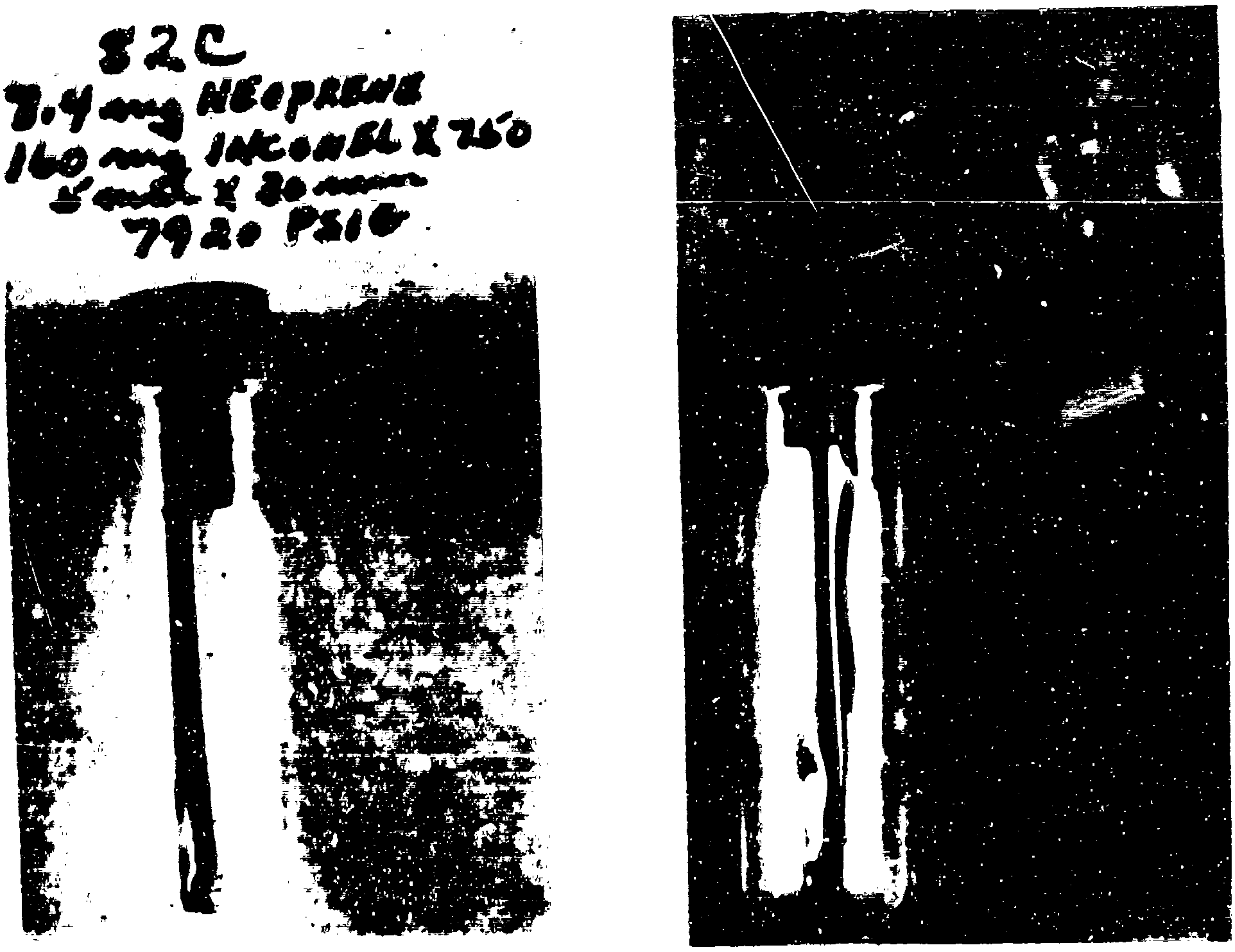


FIGURE 23 PROMOTED IGNITION TEST, STAINLESS STEEL (ENLARGED 2X)



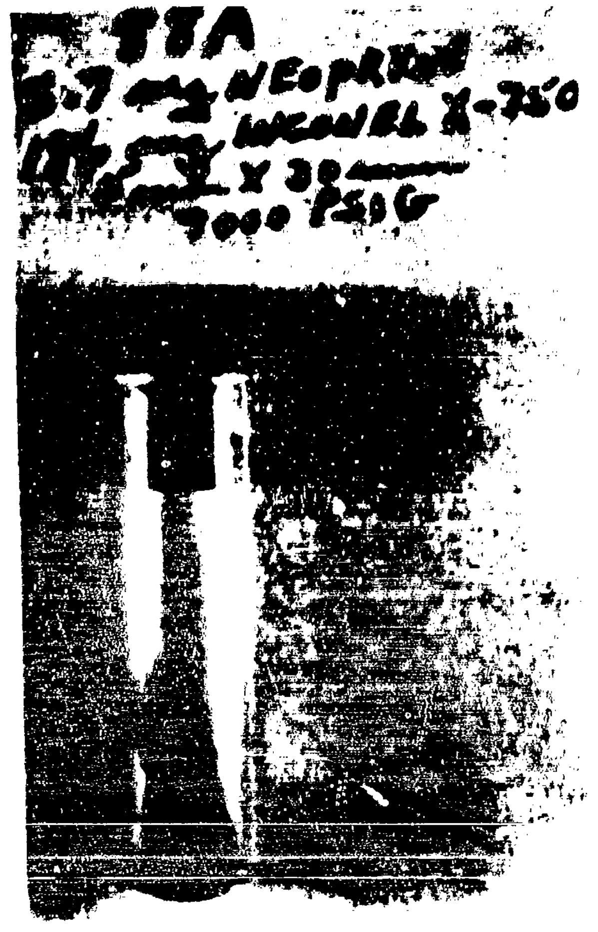


FIGURE 24.

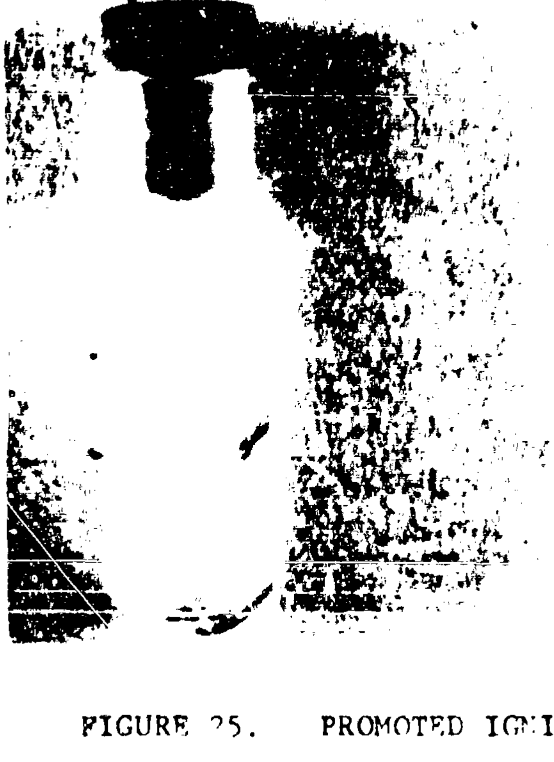
PROMOTED IGNITION TEST

INCONEL X-750 (ENLARGED 2X)

97A
11.3 mg NEOPRENE
47 mg ANODIZED Al
[illegible] mil COAT
5 mm X 30 mm
8100 PSIG

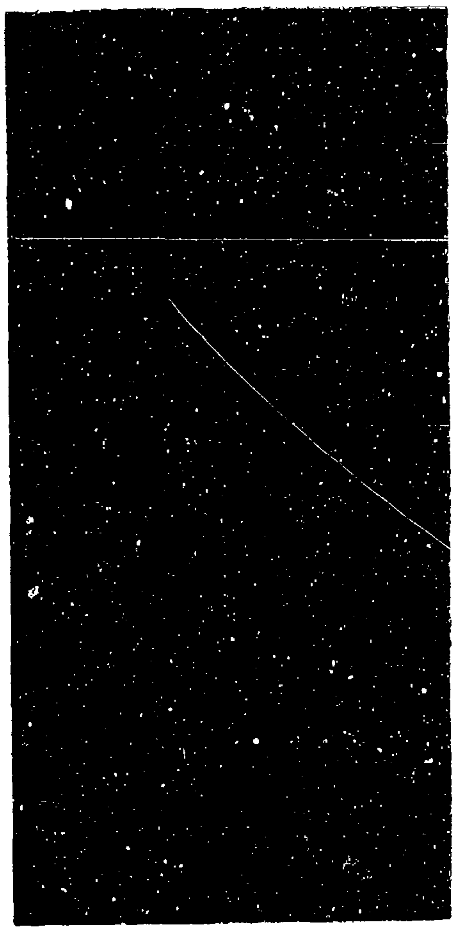
81C
14.1 mg NEOPRENE
20.3 mg ALUMINUM
5 mm X 30 mm
7850 PSIG

97D
14.6 mg NEOPRENE
50 mg ANODIZED Al
0.29 mil COAT
5 mm X 30 mm
7700 PSIG



15.5 mg NEOPRENE
54 mg ANODIZED Al
0.29 mil COAT
5 mm X 30 mm
7300 PSIG

FIGURE 25. PROMOTED IGNITION TEST, ALUMINUM (ENLARGED 2X)



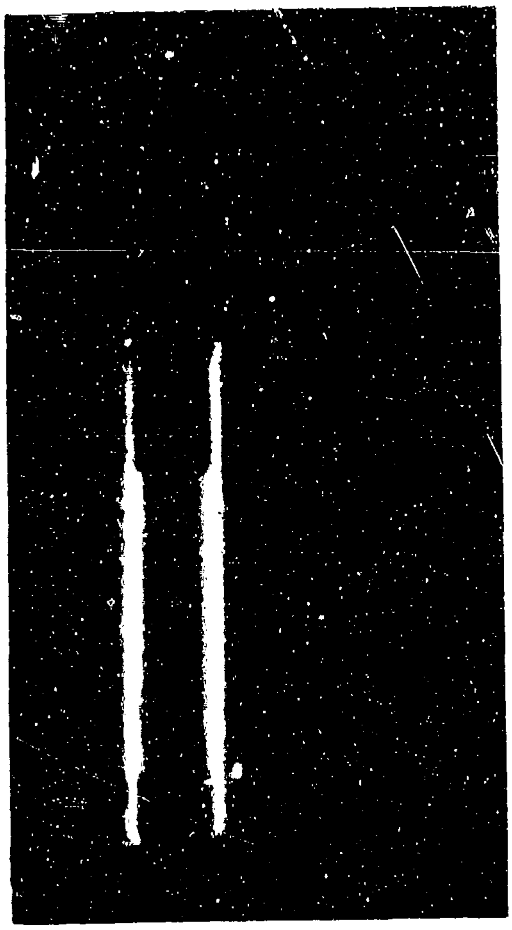


FIGURE 26.

PROMOTED IGNITION TEST

COPPER AND LEAD (ENLARGED 2X)

90 C
11.8 mg NEOPRENE
177 mg YELLOW BRASS
5 mm X 30 mm
7325 PSIG

90 D
13.2 mg NEOPRENE
166 mg YELLOW BRASS
5 mm X 30 mm
7350 PSIG

92 C
15.2 mg NEOPRENE
160 mg YELLOW BRASS
5 mm X 30 mm
7700 PSIG

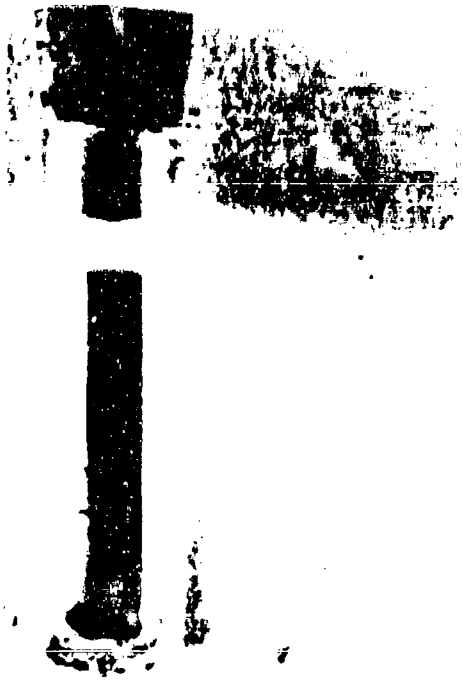


FIGURE 27.

PROMOTED IGNITION TEST

BRASS (ENLARGED 2X)

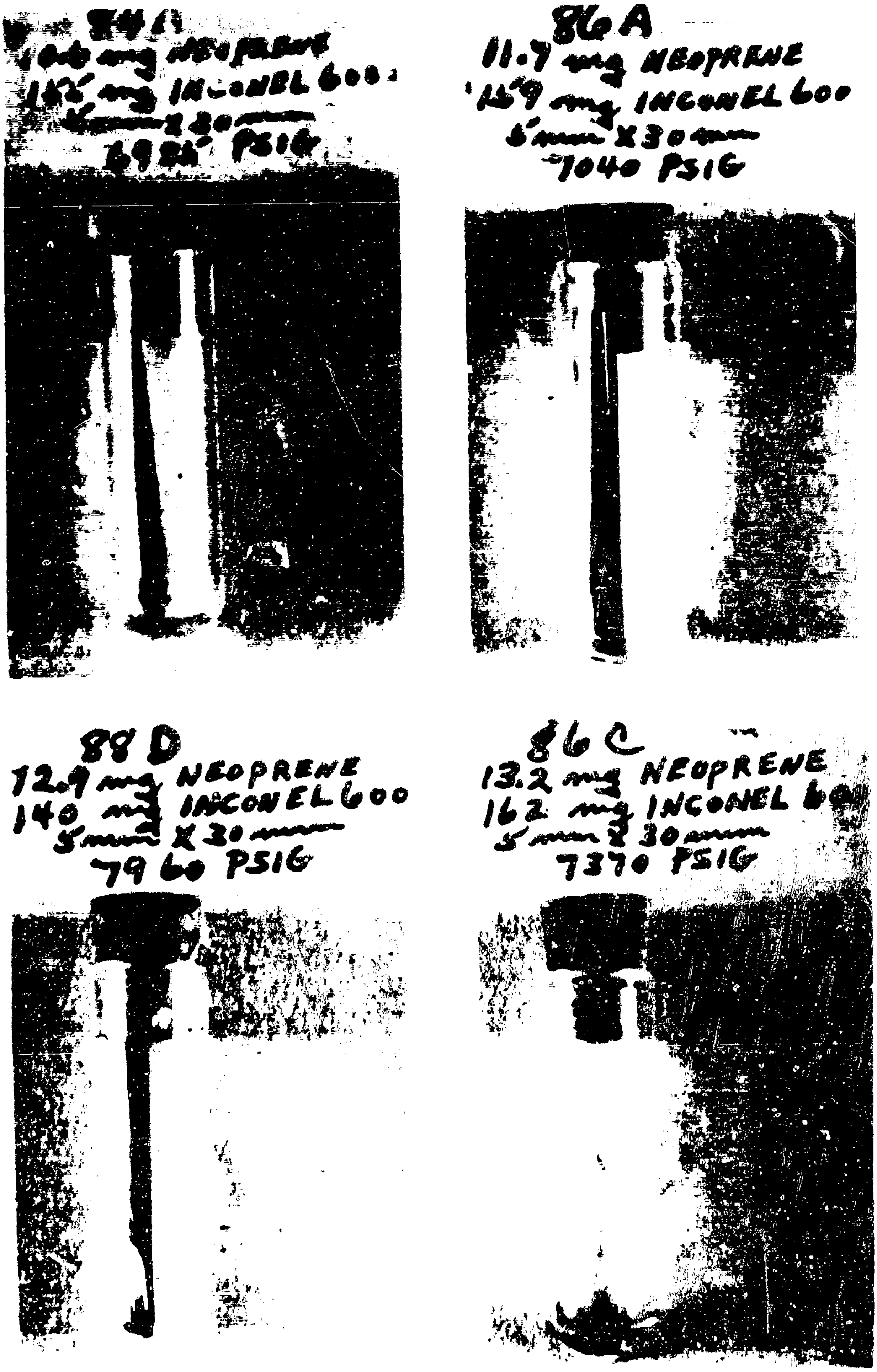


FIGURE 28. PROMOTED IGNITION TEST, INCONEL 600 (ENLARGED 2X)

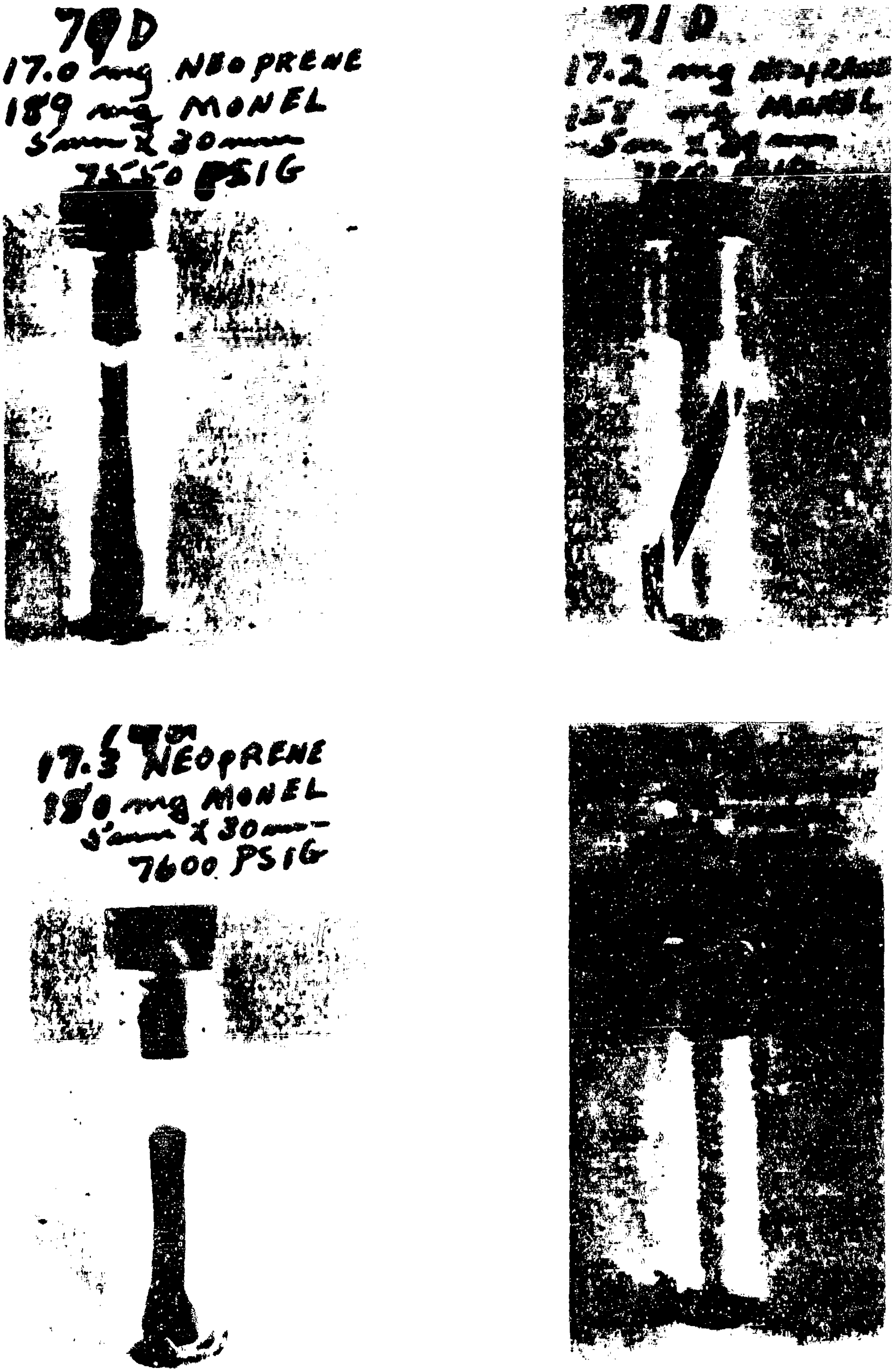


FIGURE 29. PROMOTED IGNITION TEST, MONEL 400 (ENLARGED 2X)

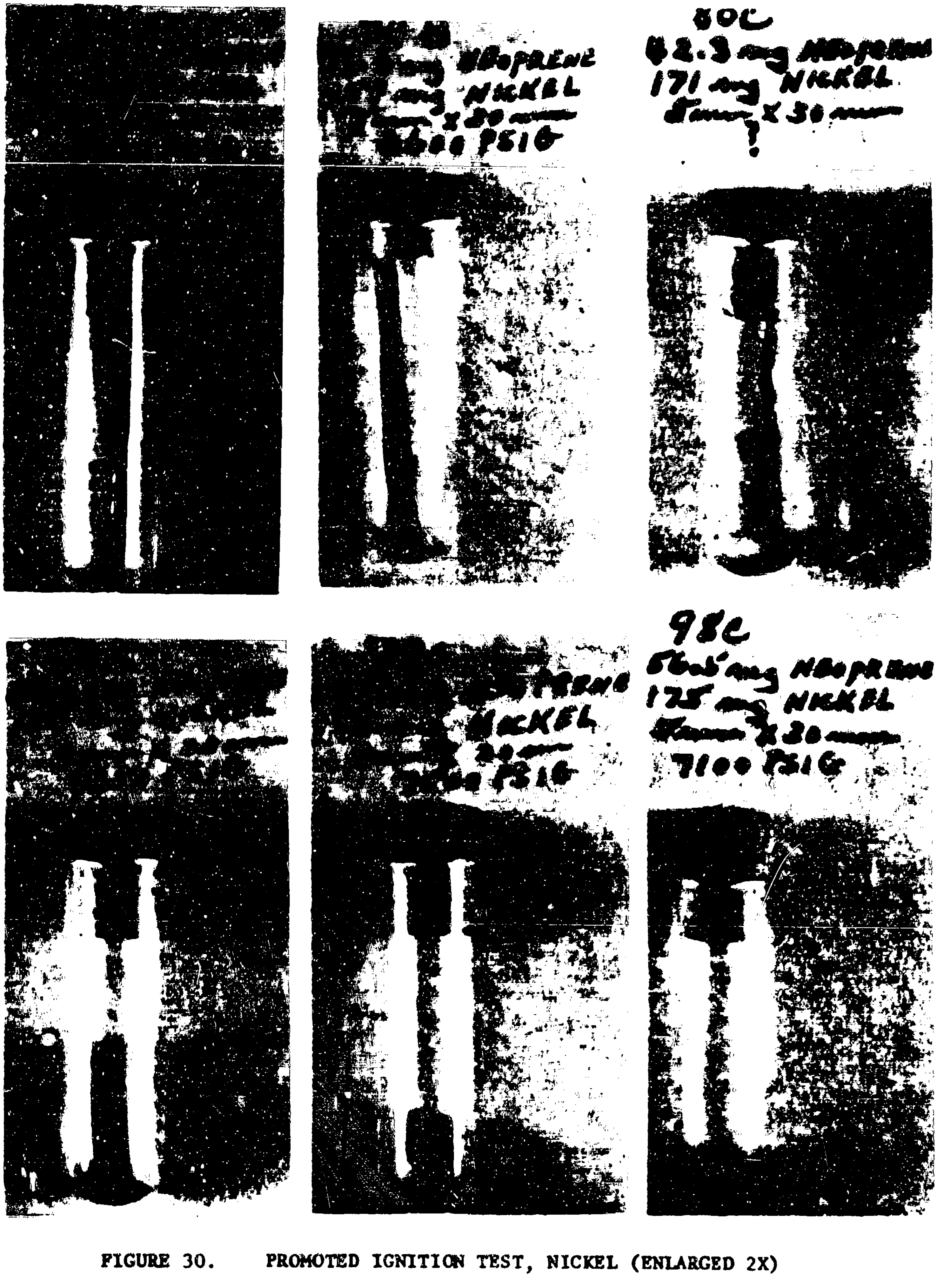
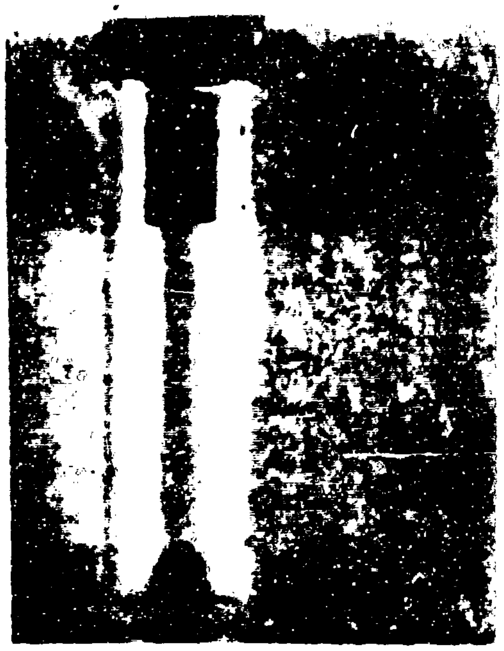


FIGURE 30. PROMOTED IGNITION TEST, NICKEL (ENLARGED 2X)

94D
15.3 mg NEOPRENE
372 mg GOLD
5 mm X 30 mm
7400 PSIG

96D
59 mg NEOPRENE
396 mg GOLD
5 mm X 30 mm
7350 PSIG



94B
14.3 mg NEOPRENE
229 mg SILVE
5 mm X 3
8300 PSIG

78B
54.6 mg NEOPRENE
205 mg SILVER
5 mm X 30 mm
7000 PSIG

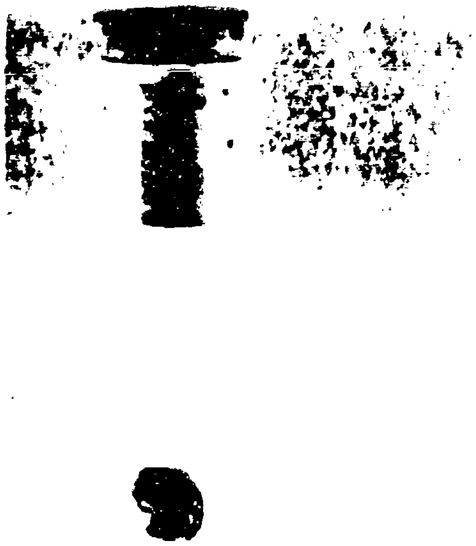
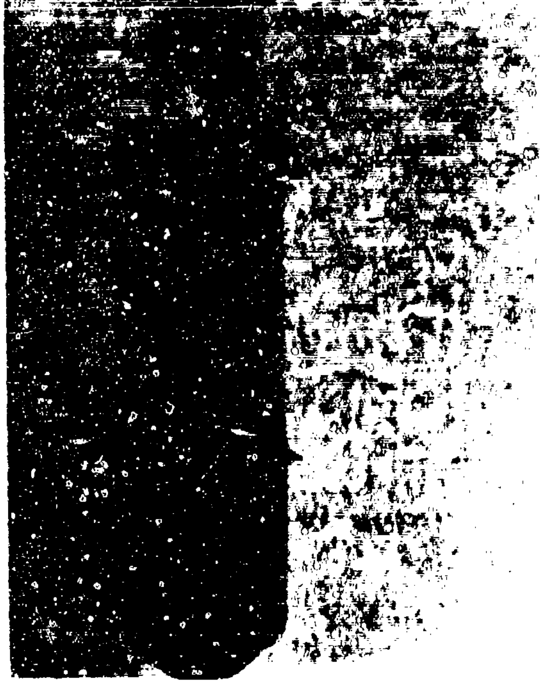


FIGURE 31. PROMOTED IGNITION TEST, GOLD AND SILVER (ENLARGED 2X)

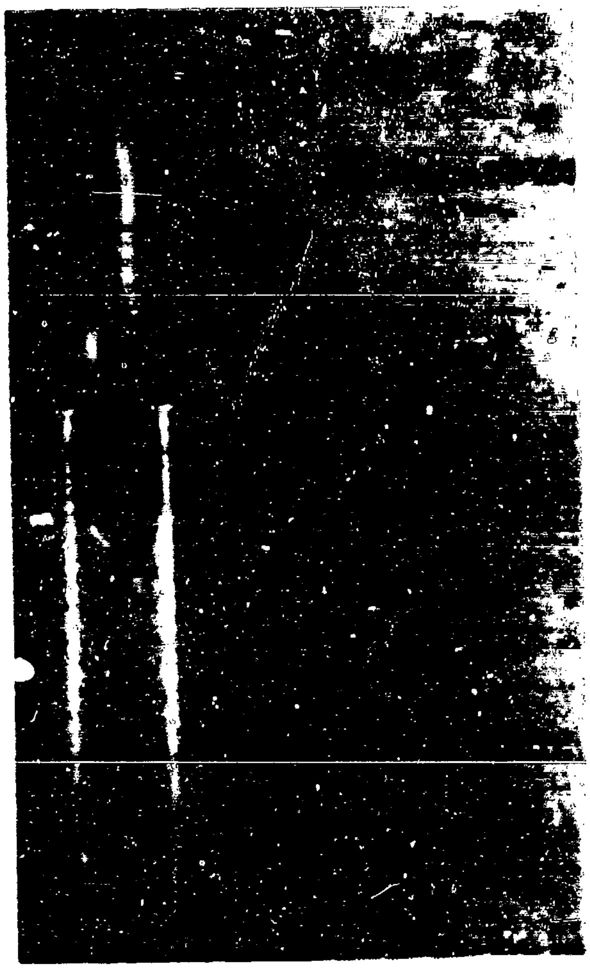
95C
176 mg 304 S.S.
Sealed in 275 mg KEL-F
5mm x 30 mm
8400 PSIG

A.

96A
176 mg 304 S.S.
Sealed in 218 mg Teflon
5mm x 30mm
8300 PSIG



B.



C.

FIGURE 32.

PROMOTED IGNITION TEST

A. STAINLESS STEEL SEALED IN KEL-F
B. STAINLESS STEEL SEALED IN TEFLON FEP
C. ALUMINUM COATED WITH KEL-F

(ENLARGED 2X)

d. Gold and silver might have applications in plating other metals. Their use would not present an ignition hazard.

e. Copper is not recommended because of other shortcomings.(1)

f. Of the materials tested, stainless steel and aluminum are the least satisfactory for use at oxygen pressures of 7500 psi.

Simulated System Tests

Purpose

The simulated system tests had three objectives:

1. To experimentally determine the hazards involved in charging a simulated system with 7500 psi oxygen.

2. To determine any electrostatic charge developed during charging of the simulated system.

3. To determine the flow rate at which a hazardous condition is approached.

Philosophy

The philosophy used in constructing and testing the system was as follows:

1. Use the best possible materials as determined by previous test data.

2. Use equipment and arrange it in a manner which would best simulate an actual system.

3. Pressurize the system rapidly with oxygen to various pressure levels and measure the pressure rise by means of a pressure transducer and recorder. This information will permit calculation of flow rates.

4. By means of a probe and oscilloscope, measure the electrostatic charge of the high pressure oxygen gas in the receiver as it is charged.

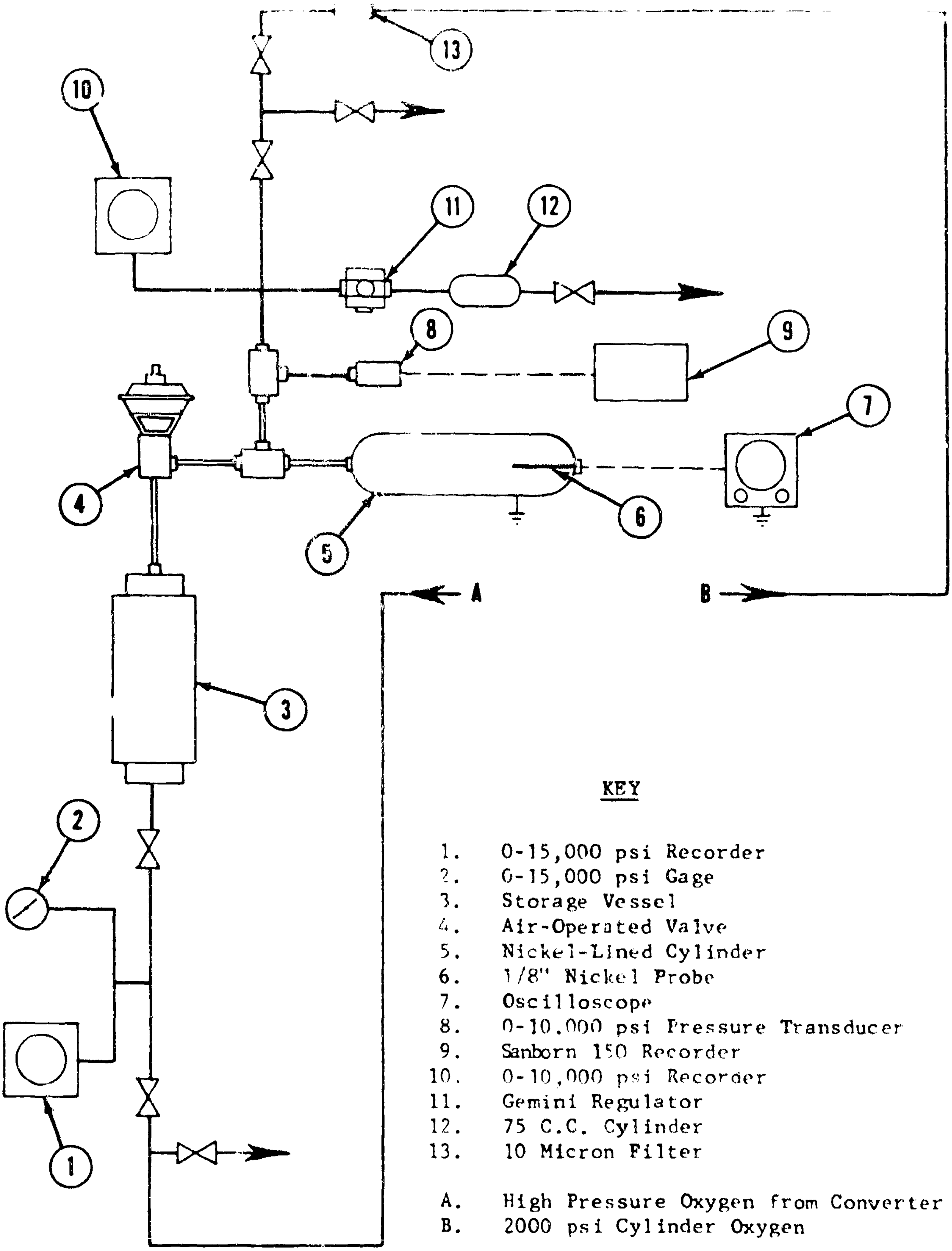
Materials of Construction and Arrangement of Equipment

The schematic of the test set up is shown in Figure 33. Figures 34 and 35 are pictorial presentations. The equipment, its material of construction and the reason for its position are:

1. Storage Vessel

The storage vessel was used as a source of high pressure oxygen after it had been charged by the converter. It was used for two reasons:

a. As an intermediate vessel between the converter and the simulated system to isolate the converter in case of a mishap.



KEY

1. 0-15,000 psi Recorder
2. 0-15,000 psi Gage
3. Storage Vessel
4. Air-Operated Valve
5. Nickel-Lined Cylinder
6. 1/8" Nickel Probe
7. Oscilloscope
8. 0-10,000 psi Pressure Transducer
9. Sanborn 150 Recorder
10. 0-10,000 psi Recorder
11. Gemini Regulator
12. 75 C.C. Cylinder
13. 10 Micron Filter

A. High Pressure Oxygen from Converter
B. 2000 psi Cylinder Oxygen

FIGURE 33. SCHEMATIC FOR SIMULATED SYSTEM TESTS

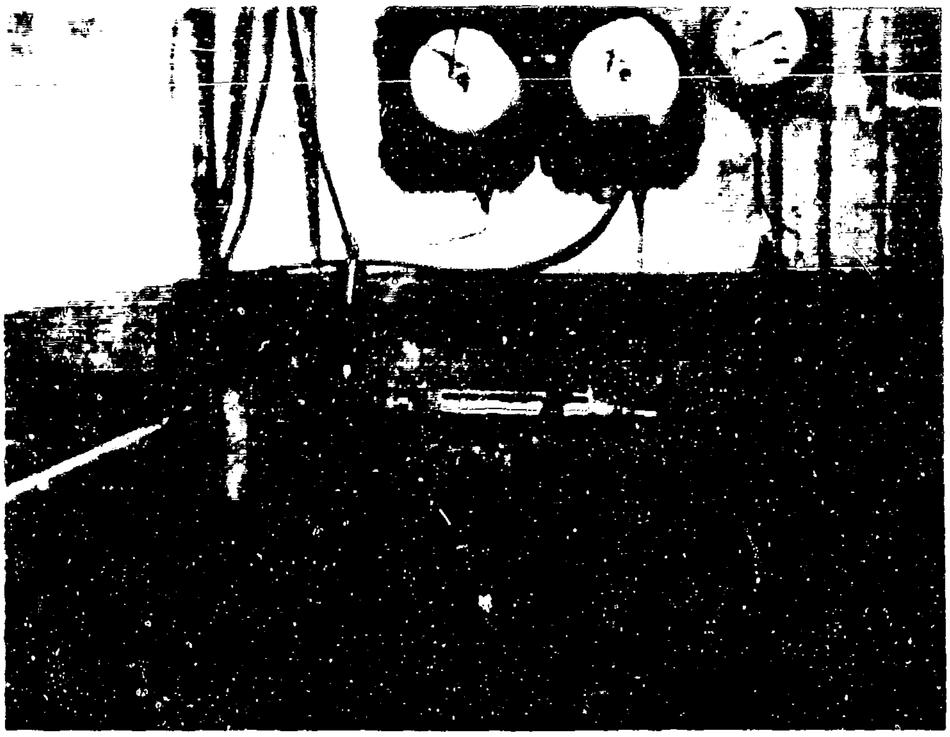


FIGURE 34. PICTURE OF SIMULATED SYSTEM TEST

FIGURE 35. VIEW OF GEMINI REGULATOR LOCATION IN SIMULATED SYSTEM

b. To warm up the oxygen by pressurization and provide gas at essentially ambient temperature.

The storage vessel had an internal geometric volume of 341 cubic inches, was constructed of 316 stainless steel and was rated at 15,000 psi. It was considered expendable in the event of a mishap. A 5/16-inch diameter hole through the head of the vessel was given a funnel-shaped, curved radius on the inside face of the head to facilitate gaseous flow.

2. Air-operated valve

The valve body, removeable seat, stem, and packing washers were made from Monel 400 and Monel K-500. The packing was glass-filled TFE. The valve contained 5/16-inch I.D. ports and was rated at 10,000 psi. It was actually necessary to use the valve at higher pressures for these tests but because the tests were conducted in a high pressure area and the valve had a built in safety factor we were able to do so. Valves rated for higher pressures contained smaller port openings which would not have been as satisfactory for the tests.

The upstream port of the valve was placed in a direct line with the storage vessel and the downstream port was placed in a direct line with the receiver to provide the minimum resistance to gas flow.

3. Receiver

Because nickel was found to be the best material of construction, a nickel-lined stainless steel cylinder was purchased. The cylinder wall consisted of 0.20 inch of nickel and 0.30 inch of stainless steel. The cylinder was rated for 7500 psi. Internal geometric volume was 65 cubic inches. The receiver was placed in direct line with the air-operated valve to minimize pressurization time.

4. Probe

A 1/8-inch nickel rod was selected for the electrostatic probe because of its resistance to ignition. It entered the receiver through an insulated fitting and was sealed with TFE packing. A nickel washer fastened to the rod on the inside prevented its being blown out by the combination of high pressure and the lubricity of the TFE.

5. 10,000 psi Pressure Transducer

The transducer was used to measure the pressurization time of the simulated system. Because it was made of stainless steel it was placed in a physical position to minimize its being impacted by any particles that might be in the system.

6. Regulator

Because we were trying to simulate a system, it was felt that a pressure regulator which is currently used in high pressure oxygen should be included.

The regulator used was identical to that which will be used in the Gemini capsule. The regulator is only used at 5000 psi in the Gemini capsule but is similar in construction to the regulator used in the Mercury capsule at 7500 psi. Aluminum was used for the body of the Mercury regulator while stainless steel is used for the body of the Gemini regulator.

The regulator contains a relief device on the downstream side to handle all the flow in the event of catastrophic failure in the regulator itself. The relief device limits pressure downstream to a maximum of 200 psi.

Elastomeric materials are used in the regulator for static and dynamic seals.

The regulator was placed in a position which was thought to be comparable to one that would be used in a system: not too far from the receiver but not in a direct line with the charging line.

7. Valves, Fittings, and Tubing

All valve bodies were Monel 400 but some of the valve stems were Monel K-500 and some were 17-4 PH stainless steel. Likewise packing washers were either Monel 400 or stainless steel. Packing for the valve stems was glass-filled TFE.

All fittings were Monel 400 except for two stainless steel tees to connect the 9/16" O.D. x 5/16" I.D. tubing. All tubing was also Monel 400.

8. Lubricants and Thread Sealants

TFE tape was used both as a lubricant and a sealant for pipe threads on both ends of the receiver and at the transducer because other materials were either considered to be unsuitable or sealing characteristics were unknown.

A thin film of fluorocarbon oil was used to lubricate the threads of the tubing, gland nuts and valve stems. However, these points are not exposed to high pressure oxygen either because of weep holes or isolation by the valve stem packing.

9. 10,000 psi Pressure Recorder

This recorder contained a stainless steel bourdon tube but shock effects on it were minimized by using 1/8-inch O.D. tubing to connect it to the system.

Procedure

The following steps were followed in making a simulated test:

1. High pressure oxygen from the warm converter was used to pressurize the storage vessel to the desired pressure.

2. Initially the receiver was successively pressurized and blown down several times with 2000 psi oxygen to be certain an oxygen atmosphere existed. This was also done with the Gemini regulator after the first series of tests. (For immediate succeeding tests, step 2 was eliminated.)

3. The blowdown valves were closed and the air-operated valve was opened.

4. The rate of pressure build-up was recorded on a Sanborn 150 recorder and any electrostatic charge developed was recorded photographically with an oscilloscope. A typical pressure profile is shown in Figure 36, and a typical photographic record of the electrostatic charge is shown in Figure 37.

5. The air-operated valve was closed and the simulated system and the downstream side of the Gemini regulator were blown down.

6. The electrostatic probe was grounded for several minutes between tests to be certain there was no charge on it at the beginning of the next test.

7. Steps, 1, 3, 4, 5, and 6 were repeated for the next test.

8. Temperature of the gas in the storage vessel just prior to making a test was always between 10°C and 20°C.

7500 psi Tests

1. Results

a. The simulated system was pressurized with oxygen to instantaneous pressures ranging from 665 psi to 7100 psi without any mishaps. Pressurization time varied from 0.5 second to 0.21 second.

b. Ten pressurizations were made to instantaneous pressures of 7000 psi. Final pressures of 8000 psi were developed when the air-operated valve was left open and the receiver and storage vessel were permitted to equalize pressures over a period of a few minute The gas in the storage vessel (initially at 12,000 psi) was cooled tremendously during pressurization of the simulated system. The temperature of the gas in the storage vessel was not measured but Byrnes, Reid and Ruccia (2) have shown that the temperature of a gas in a cylinder during depressurization may be lowered substantially.

c. Electrostatic charges measured varied from 0.0 volt to -0.32 volt.

d. Substituting thirty inches of 1/4-inch O.D. x 0.083-inch I.D. tubing for the 9/16-inch O.D. by 5/16-inch I.D. charging line between the air-operated valve and the receiver did not increase the electrostatic charge developed. The pressurization time recorded for the receiver when 1/4-inch tubing was used for the charging line is in error because of the position occupied by the transducer during these tests.

e. Temperature measurement of gas in the receiver during pressurization showed a maximum temperature rise of 80°C.

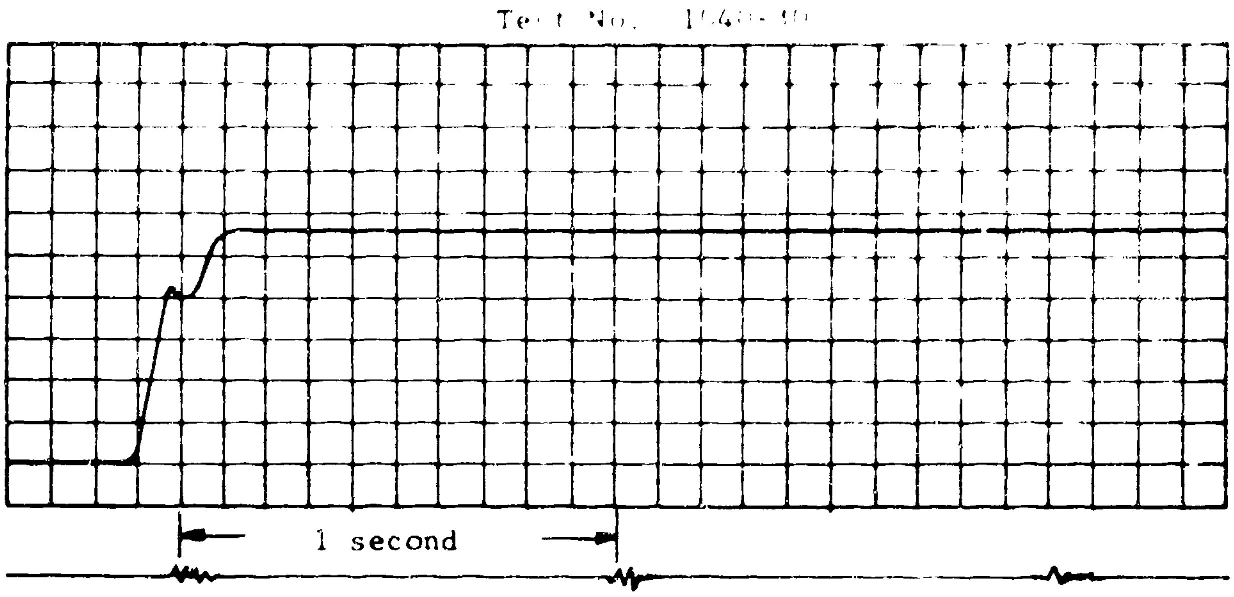


FIGURE 36. PRESSURE PROFILE OF SIMULATED SYSTEM TEST

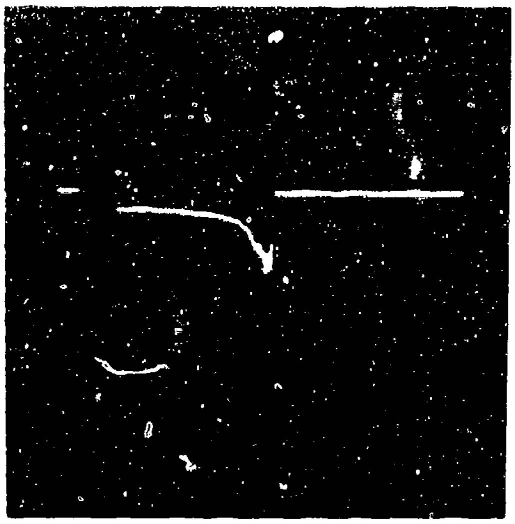


FIGURE 37.

TYPICAL PHOTOGRAPHIC RECORD OF ELECTROSTATIC CHARGE DEVELOPED IN SIMULATED TEST

f. Complete data may be found in Table 11 of Appendix IV.

2. Evaluation of Results

a. The fact that the simulated system was tested thoroughly without any malfunctions does not alter the fact that charging of a system of this type always carries with it the inherent hazards associated with high pressure gaseous oxygen nor does it imply that oxygen can be handled in the system with absolute safety.

b. Because there were no malfunctions, the flow approaching a hazardous condition cannot be defined.

c. There were no significant electrostatic charges developed during these tests. Therefore, it must be concluded that this does not represent a hazard in the system as it was tested. If the gas were to travel through lengthy lines before reaching the receiver, more of a charge might be developed.

d. A dip always occurred in the pressure profile (Figure 36) and correlated with a hissing sound from the test area. This was found to be due to the relief valve opening on the downstream side of the Gemini regulator. Apparently because of the rapid pressurization, the downstream pressure exceeded 200 psi before the regulator could shut off the flow. This momentarily caused a dip in the pressure curve until the regulator started to control, then the relief valve would close and the pressure in the system would rise. Of course this all happened in a small fraction of a second.

e. The Gemini regulator performed perfectly during all the tests. However, in view of the results of oxygen bomb tests and promoted ignition tests, some of its materials of construction are not recommended for 7500 psi oxygen service.

f. After one of the tests, the receiver started to leak around the threads where the fitting for the probe threaded into the receiver. It was thought that the TFE tape might have partially ignited but when the fitting was removed it was not possible to determine that any ignition had taken place.

Evaluation of Hardware Used in the Experimental Program

Gemini Regulator

As previously mentioned this regulator performed perfectly but some of the materials of construction are not recommended for 7500 psi oxygen service.

High Pressure Valves

1. Serious galling occurred between the packing rings and the stem of the original Monel valves (Figure 38 A, B, C, D, and F). These valves were standard construction but custom-made. The difficulty was attributed to improper machining and surface finish of the parts, as well as tolerances which were too close. Reworking of the pieces provided better operation, but

the problem was never completely eliminated. No doubt use of similar metals together contributed to the over-all galling problem.

2. The seating surfaces of the Monel K-500 stem and the Monel 400 body tended to gall so that it was necessary to tighten the valve more and more to obtain a leak-tight seal. Finally it would be necessary to reface the seating surfaces of both the stem and the body. Figures 38 A, C, and D show galling as it occurred on the seating surface of the stem.

3. During the latter part of the program, 17-4PH stainless steel valve stems were substituted for the Monel K-500 valve stems in the Monel 400 bodies and stainless steel packing rings were substituted for Monel packing rings. This was done both to expedite test work and to permit some experience to be gained. The 17-4 PH valve stems performed better mechanically than the Monel K-500 stems because the seating surface did not gall as severely. Both rotating stems and non-rotating stems were used. (See Figure 3). The non-rotating stem gives good leak-tight seals with no scoring of the seating surface but it gives very poor control over gas flow because of backlash.

4. Complete stainless steel valves (body and stem) were used at times to keep the test work going and to gain some experience with their use in high pressure oxygen.

5. On occasion, when a valve was taken apart, there would be a slick dark film on the valve stem below the bottom packing washer (Figure 38 E). This appeared to be TFE which had coated the stem as it was opened and closed. This represents a possible source of ignition and, because it is not confined, would be more likely to be exposed to ignition conditions.

6. In spite of the fact that no mishaps occurred, stainless steel valves are the least satisfactory for use in 7500 psi oxygen service because of the low resistance of stainless steel to ignition.

7. Considerable effort needs to be expended to design a high pressure valve from materials that are more compatible with high pressure oxygen but still will not gall.

Tubing Connection and Fittings

The mechanical connection shown in Figure 4 was used throughout the tests except in those places that required pipe connections. This is a standard high pressure connection and, although there are some problems associated with its use, in general it performed satisfactorily and provided leak-tight seals.

It is recommended that some consideration be given to a means of locking the gland nut in position after the fitting has been tightened. This would prevent loosening due to vibration.

For maximum safety, all welded connections are recommended.

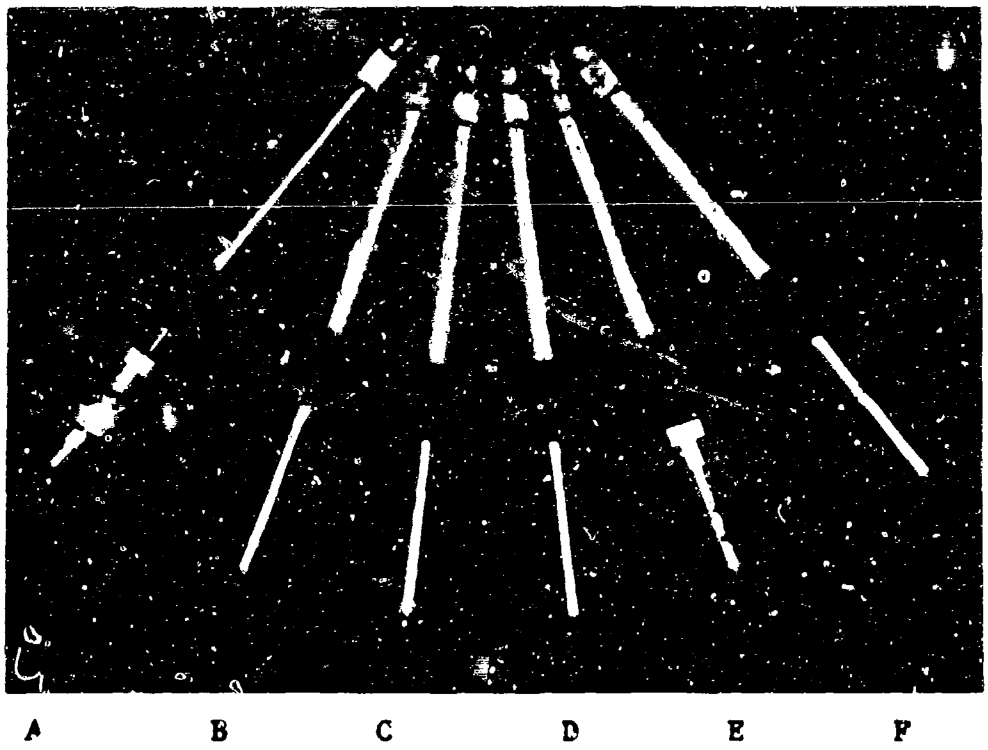


FIGURE 38. HIGH PRESSURE VALVE STEMS

FIGURE 39. RUPTURED 10,000 PSI BURSTING DISCS

Miscellaneous

Four 10,000 psi safety discs were ruptured during the experimental test program. These are shown in Figure 39. These discs were fabricated from Monel alloy 400. The torn edges of these discs showed no evidence of ignition or combustion being caused by the rupture.

CONCLUSIONS

The following conclusions are based upon the results of the present experimental test program and other data referenced in this report.

1. The spontaneous ignition temperatures of the materials tested are essentially the same in 7500 psi oxygen as in 2000 psi oxygen.

2. The relative resistance of metals and alloys to ignition and combustion in 7500 psi oxygen agrees in general with results obtained in earlier Linde test work at 2000 psi and with the results of Dean and Thompson (3) except for aluminum.

3. Only three thread lubricants might have possible safe application in 7500 psi oxygen. These lubricants are Dixon's Flake Graphite No. 1, Burnil Brand Microplates, and Almasol Powder. Further information on their lubricating properties and thermal stability would need to be known before a complete recommendation could be made. One drawback to this type of lubricant is that there is always a possibility of particulate matter inadvertently getting into the high pressure system.

4. The only thread sealant tested in this program which might be recommended for 7500 psi oxygen service would be 50-50 soft solder (which could be used in tinning threads). TFE tape is not recommended because of the possibility of pieces getting into the gas stream.

5. Recognizing that some type of packing is needed for static and dynamic seals, glass-filled TFE would be the only material presently recommended for use Even its use presents a hazard and it should only be used when tightly confined.

6. Inconel alloy 600, brass, Monel alloy 400, and nickel are recommended for use in 7500 psi oxygen. Other metals might be acceptable if plated with nickel, silver, or gold. Monel alloy K-500 might also be acceptable but it was not given the promoted ignition test in 7500 psi oxygen.

7. Of the materials tested, stainless steel and aluminum are the least satisfactory for use at oxygen pressures of 7500 psi.

8. Copper is not recommended because of the results of Baum, Goobich, and Trainer (1). Other copper alloys might be acceptable material but would need to be tested.

9. Electrostatic charges developed during the charging of a small high pressure receiver were found to be negligible.

10. The simulated system was pressurized with oxygen to instantaneous pressures ranging from 665 psi to 7100 psi without incident. Pressurization time varied from 0.5 to 0.21 seconds.

The hazards considered to be most responsible for ignitions in oxygen systems are:

a. Adiabatic Compression - High temperatures may be produced when low pressure oxygen is suddenly brought to a high pressure, such as when a valve is suddenly opened between a high pressure and a low pressure portion of the system. These temperatures can be high enough to ignite organic materials or small particles of metal.

b. Particulate Matter - Particles may be accelerated in a high velocity stream and impacted against organic materials or metallic burrs, projections, etc. Their energy is converted to high local temperatures by this impact with subsequent ignition of associated materials. Even the particles themselves might be ignited if they were either organic or metallic in nature.

These hazards may be substantially reduced by proper cleaning of the equipment initially, use of filters (especially ahead of regulators) minimum use of organic material, and judicious operation of valves and regulators to prevent rapid increases in pressure.

There are other mechanisms by which ignition may occur but these have been well covered by Reynales (13).

RECOMMENDATIONS

1. Further study should be made on the copper alloys to define more clearly their compatibility and applicability in 7500 psi systems.

2. More tests should be conducted on metals and alloys to determine the effect of geometric configuration on the relative ignition temperatures.

3. There is need for further investigation into thread lubricants and thread sealants. This study would need to cover their physical properties such as lubrication and sealing qualities as well as their compatibility with oxygen. Development of special lubricants and sealants might be required.

4. A program is needed to develop and build better hardware constructed of materials more compatible with high pressure oxygen. The program should cover any and all equipment which would be used in a 7500 psi oxygen system.

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(15) Riehl, W. A., Key, C. F., and Gayle, J. B., *Reactivity of Titanium with Oxygen*, NASA Technical Report R-180, National Aeronautics and Space Administration, Washington, D. C., 1963.

(16) Smeltzer, W. W., and Perrow, J. M., "Oxidation of Metals," *Industrial and Engineering Chemistry*, *53* (4), 319-324, April 1961.

(17) Watkins, H. D., "Basic Gemini ECS Keyed to 14-day Flight," *Aviation Week and Space Technology*, *79*, 52-69, August 19, 1963.

APPENDIX I

CALCULATION OF THEORETICAL TEMPERATURE ATTAINED BY ADIABATIC COMPRESSION

The theoretical temperature attained by adiabatic compression was calculated using the expression:

$$\frac{T_2}{T_1} = \left(\frac{P_2}{P_1}\right)^{\frac{n-1}{n}} \qquad \text{where } n = \frac{C_p}{C_v}$$

substituting for n, we obtain:

$$\frac{T_2}{T_1} = \left(\frac{P_2}{P_1}\right)^{\frac{1.4-1}{1.4}} = \left(\frac{P_2}{P_1}\right)^{0.286}$$

Using an initial pressure of 14.7 psia and an initial temperature of 293°K:

$$T_2 = 293\left(\frac{P_2}{14.7}\right)^{0.286}$$

This equation was solved for various pressures to obtain the theoretical temperature rise. These are presented in Table 5 and Figure 40.

TABLE 5

THEORETICAL TEMPERATURE ATTAINED BY ADIABATIC COMPRESSION

CALCULATED FROM THE EXPRESSION $\frac{T_2}{T_1} = \left(\frac{P_2}{P_1}\right)^{\frac{n-1}{n}}$

P_2, psia	Temperature	
	T_2, °K	T_2, °C
100	506	233
200	622	349
400	761	488
800	922	649
1,200	1036	763
1,600	1122	849
2,000	1199	926
2,400	1266	993
2,800	1313	1040
3,200	1363	1090
4,000	1455	1182
5,000	1561	1288
6,000	1639	1366
7,000	1711	1438
7,500	1750	1477
10,000	1900	1627
15,000	2123	1850

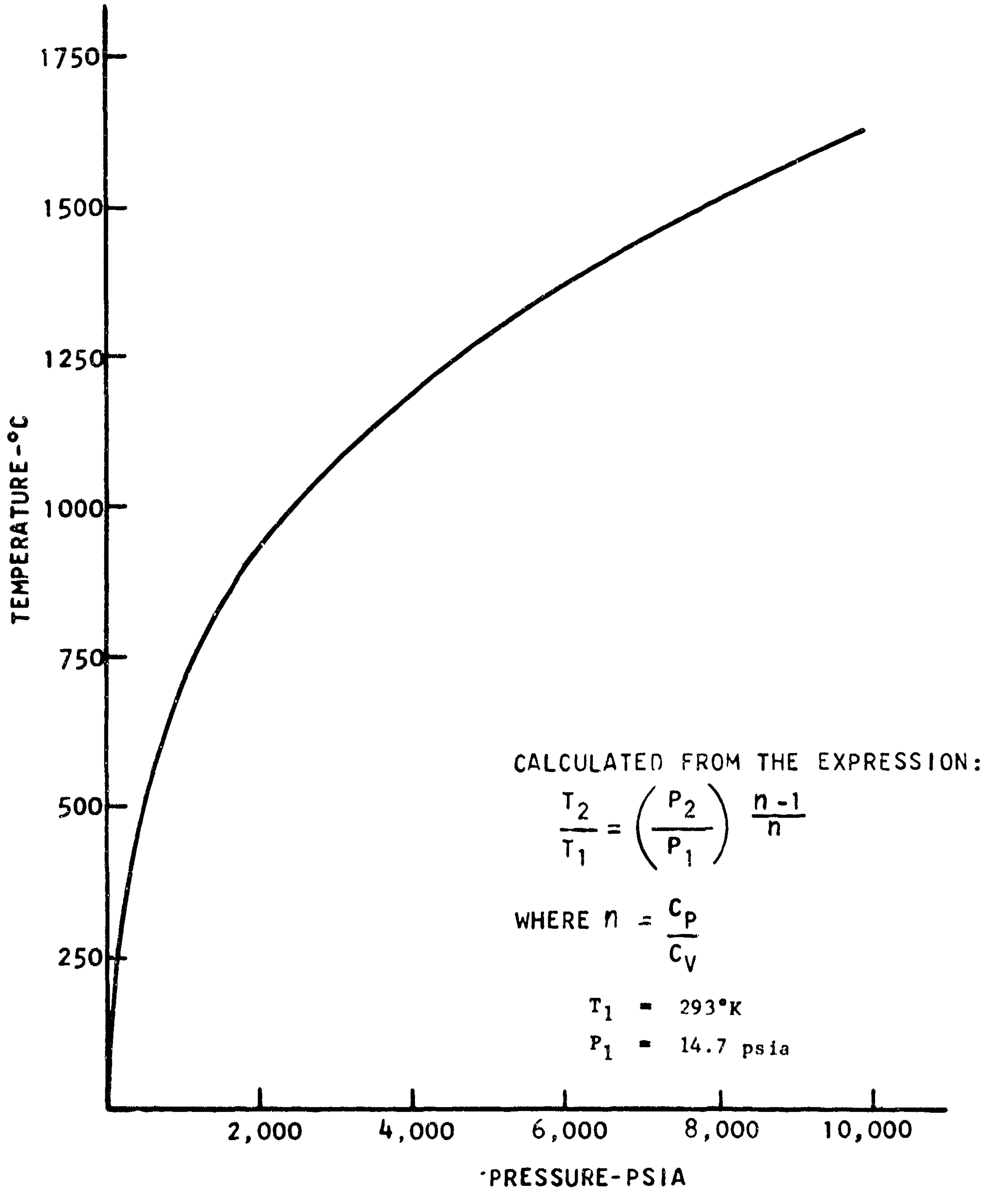

FIGURE 40. THEORETICAL TEMPERATURE ATTAINED BY ADIABATIC COMPRESSION

APPENDIX II

CALCULATION OF COMPRESSIBILITY FACTORS FOR OXYGEN

The compressibility factor (Z) for oxygen was calculated for various pressures using charts(1) which comprised a series of lines representing compressibility factor, $Z = \left[\frac{PV}{P_oV_o}\right]_{T\,=\,\text{const.}}$, plotted against reduced pressure, $P_r = \frac{P}{P_c}$, for different values of reduced temperature, $T_r = \frac{T}{T_c}$

The pressures and compressibility factor for each are presented in Table 6 and Figure 41 for $T_r = 1.9$.

TABLE 6

COMPRESSIBILITY FACTOR FOR OXYGEN AT VARIOUS PRESSURES

P, psia	P_R	Z
200	0.271	0.995
400	0.543	0.99
600	0.815	0.985
800	1.08	0.97
1,000	1.36	0.965
1,200	1.63	0.955
1,400	1.90	0.95
1,600	2.17	0.942
2,000	2.71	0.938
2,400	3.26	0.935
2,800	3.80	0.938
3,200	4.35	0.945
4,000	5.42	0.97
5,000	6.78	1.02
6,000	8.15	1.07
7,000	9.50	1.125
8,000	10.85	1.193
9,000	12.20	1.272
10,000	13.58	1.35
11,000	14.92	1.42
12,000	16.30	1.49
13,000	17.65	1.56
14,000	19.0	1.63
15,000	20.4	1.71
16,000	21.7	1.78
17,000	23.1	1.85
18,000	24.4	1.92
19,000	25.8	1.99
20,000	27.2	2.06

(1) "Compressibility Charts and Their Application to Problems Involving Pressure-Volume-Energy Relation for Real Gases" - Research Bulletin P-7637, Worthington Corporation, Harrison, New Jersey (1952)

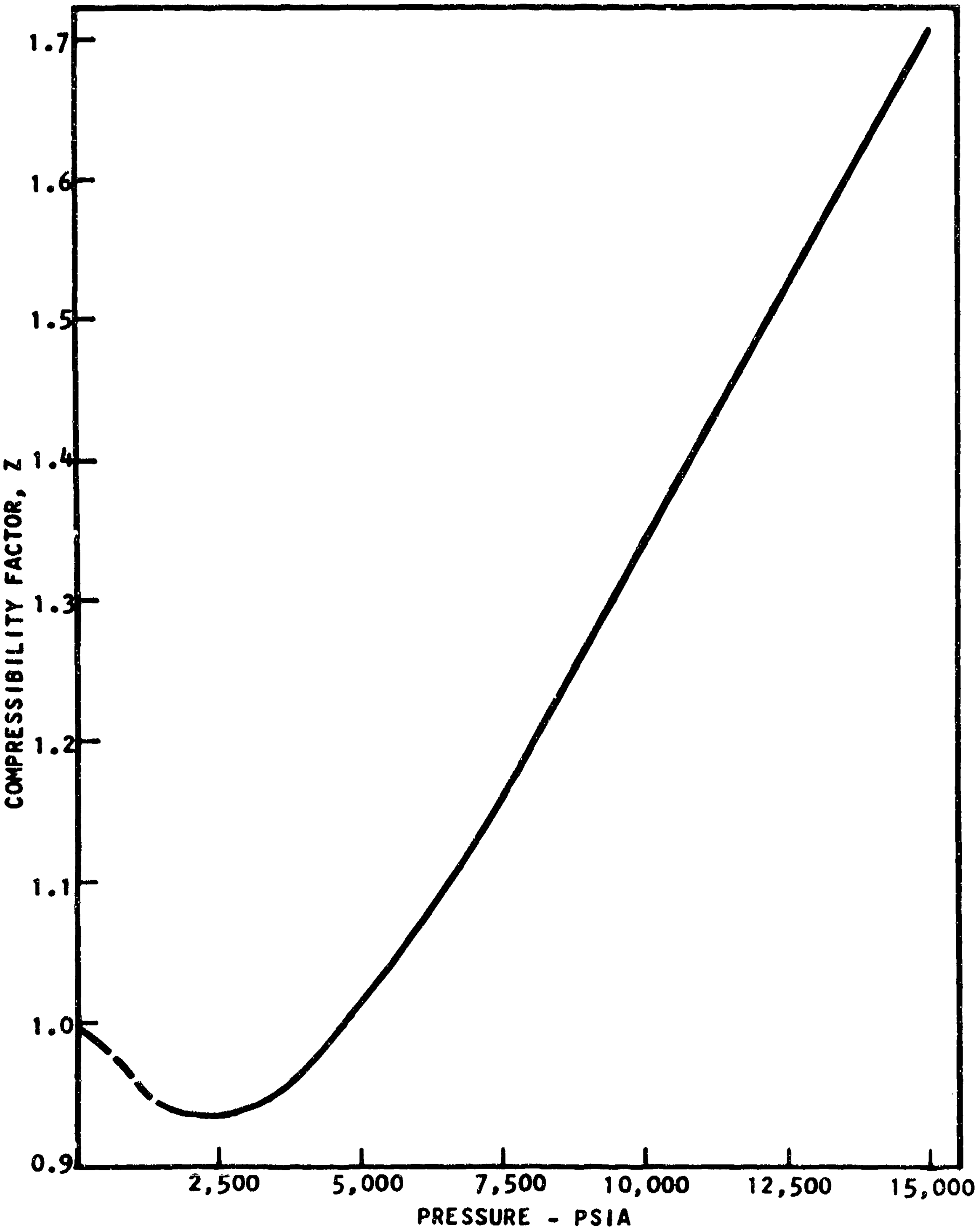

FIGURE 41. COMPRESSIBILITY FACTOR FOR OXYGEN AT 293°K

APPENDIX III

ADIABATIC COMPRESSION - A CONTROLLED IGNITION MECHANISM

Compression Apparatus

The envisioned apparatus as shown in Fig. 42 was considered equivalent to the model of Fig. 43 where the advancing high pressure gas front is equivalent to a piston moving at the velocity of sound (C_o). In this case a shock wave advances into the gas at rest at a velocity (V_W) greater than the velocity of the piston (V_p). The values for these velocities and resulting pressure and temperature rise behind the shock wave can be calculated.

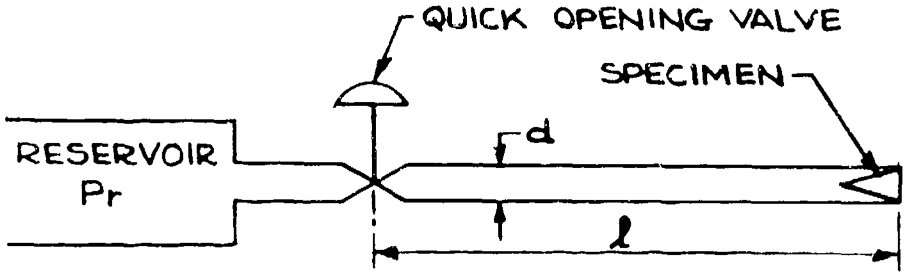

FIGURE 42

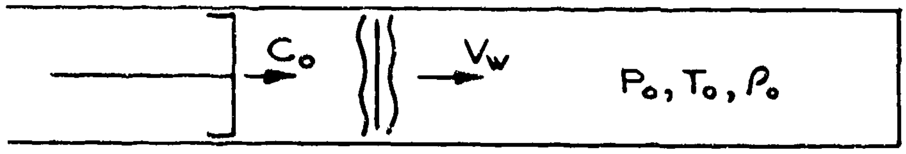

FIGURE 43

When the shock wave preceeding the piston reaches the wall at the end of the tube it is reflected giving rise to further compression.

1st Shock

$$V_W = \frac{1}{2}\frac{V_p}{1-\mu^2} + C_o^2 + \frac{1}{4}\left(\frac{V_p}{1-\mu^2}\right)^2 \qquad (1)\ (\text{Ref. } 1)$$

where:

$$\mu^2 = \frac{k-1}{k+1}$$

for $V_p = C_o$ and representative values of $K = 1.4$ and C_o - 1130 ft./sec.

$$V_W = 1990 \text{ ft./sec.}$$

$$M_o = \frac{V_W}{C_o} = 1.76 \qquad (2)$$

Ref. 1: R. Courant, K. O. Friedricks - Supersonic Flow and Shock Waves Interscience Publishers Inc., 1948.

$$\frac{P_1}{P_o} = (1 + \mu^2) M_o^{\,2} - \mu^2 = 3.494 \tag{3}$$

$$\frac{\rho_1}{\rho_o} = \frac{P_1 + \mu^2 P_o}{P_o + \mu^2 P_1} = 2.3 \tag{4}$$

$$\frac{T_1}{T_o} = \frac{P_1}{P_o} \frac{\rho_o}{\rho_1} = 1.52 \tag{5}$$

from (5) $T_1 = 1.52\ (530°R) = 805°R$

2nd Shock

$$\frac{P_2}{P_1} = \frac{(2\mu^2 + 1) \frac{P_1}{P_o} - \mu^2}{\mu^2 \frac{P_1}{P_o} + 1} = 5.06 \tag{6}$$

and from expressions similar to (4) and (5)

$T_2 = 1430\ °R$

The conclusion is that in the assumed model a series of shock reflections will rapidly tend to build up pressures and corresponding high temperatures in the tube. Actually friction effects would tend to dampen the shock waves fairly soon.

To determine how clearly the above model can be simulated tests were run on the quick-opening valve. The observed results are indicated below.

Preliminary Tests with "Quick Opening" Valve

Measurements made of the rate of pressure build-up in the actual tube indicates flow velocities substantially below sonic so that the actual mechanism of compression cannot utilize the idealized piston described above.

Valve opening time - 0.5 sec.

Pressure build-up to 2000 psi in 20 in. long 5/16 in diameter tube takes - 0.05 sec.

The rate of pressure build-up is nearly linear and approximately proportional to the length of test section.

In this instance, the mechanism of pressure build-up involves mixing of the gas at rest in the tube and the inrushing gas.

In view of the fact that a high degree of mixing takes place the anticipated temperature rise becomes uncertain and of questionable reproducibility.

Comparison of Gas Heat Capacity to Heat Loss to Surroundings

In this case a control volume of 1.07×10^{-3} ft.^{3} was taken representing a tube 5/16 in. I. D. and 24 in. long.

then $Q = V \rho C_p$

$$= 1.07 \times 10^{-3} (.089) .3 = .29 \times 10^{-4} \text{ Btu/°F} \tag{7}$$

The heat transfer from the tube can be expressed as:

$$dQ = h A \Delta T \, dt \tag{8}$$

where both the heat transfer area (A) and the temperature gradient are time dependent.

then: $$Q = \int_0^t h \; A(t) \; \Delta T(t) \, dt \tag{9}$$

From thermodynamic considerations an expression for ΔT was derived as follows:

$$\frac{T_2}{T_1} = \left(\frac{V_1}{V_2}\right)^{k-1} \tag{10}$$

$$\Delta T = T_2 - T_1 = T_i \left[\left(\frac{V_1}{V_2}\right)^{k-1} - 1 \right] \tag{11}$$

since:

$$V_2 = V_1 - AX \tag{12}$$

where $X = \frac{dX}{dt} t$ and letting $\frac{dX}{dt} = K$

$$V_2 = V_1 - AKt \tag{13}$$

Substituting (13) in (11) and simplifying:

$$\Delta T = T_1 \left[\left(\frac{\rho}{\rho - kt} \right)^{k-1} - 1 \right] \tag{14}$$

The heat transfer area made up of the tube ends and the varying surface area is expressed as:

$$\begin{aligned} A &= 2 \frac{\pi}{4} d^2 + \pi d \ (\rho - x) \\ &= 2 \frac{\pi}{4} d^2 + \pi d \ (\rho - kt) \end{aligned} \tag{15}$$

Substituting (14) and (15) in (9) and simplifying the heat transfer to the surroundings is expressed as:

$$\begin{aligned} Q = h T_1 & \frac{\pi d^2 l^{(k-1)}}{2k \ (2-k)} \left[l^{2-k} - (l-kt)^{2-k} \right] + \frac{\pi d \ l^{k-1}}{(3-k) \ k} \left[l^{3-k} - (l-kt)^{3-k} \right] \\ & - \frac{\pi}{2} d^2 t - \pi d \left(l \ t - \frac{k}{2} t^2 \right) \end{aligned} \tag{16}$$

for the representative values of:

$d = \frac{.31}{12} = .026$ ft.

$l = 2$

$t = .04$ sec.

$k = 25$ ft./sec.

$T_1 = 530$ °R

$Q = .06$ Btu

This heat loss to the surroundings is too large compared to the heat capacity of the gas to maintain adiabatic conditions during compression.

APPENDIX IV.

TABLES OF EXPERIMENTAL DATA

TABLE 7

SUMMARY OF PRELIMINARY HIGH PRESSURE OXYGEN BOMB TESTS
AND CORRELATION WITH 2000 PSI PROTOTYPE BOMB TESTS AND LINDE STANDARD 2000 PSI OXYGEN BOMB TESTS

HIGH PRESSURE BOMB									PREVIOUS DATA						
									PROTOTYPE BOMB					LINDE STANDARD BOMB	
Test date 1963	Sample material and test No.	Notes	Sample weight in grams	Ignition temp. °C	Ignition pressure psi	Temp. rise on ignition °C	Pressure rise on ignition psi	Average ht'g rate °C/min during last 20 min.	Test date 1963	Sample weight in grams	Ignition temp. °C	Ignition pressure psi	Average ht'g rate °C/min.	Ignition temp. °C	Ignition pressure psi
	Durable Gasket														
10-7	9914-14D		0.1	156	2300	20	50	8.0	8-6	0.1	147	2275	3	165	1900
10-13	9914-16A		0.1	153	2500	20	200	4.8	8-7	0.1	152	2200	4		
10-18	9914-16B		0.1	149	8100	12	500	4.4	8-14	0.1	160	2230	10		
									8-14	0.1	146	2230	5		
									8-14	0.3	155	2200	6		
									8-14	0.3	165	2230	12		
									8-15	0.1	146	2250	6		
	Filled Teflon														
10-2	9914-14A		0.3	461	2200	30	50	5.0	8-13	0.3	446	2280	4	No explosion at 410°C and 2000 psi	
10-7	9914-14B		0.3	460	2300	30	50	5.5	8-13	0.3	451	2140	9		
10-7	9914-14C		0.3	460	2170	30	70	1.8	8-13	0.3	455	2160	10		
10-18	9914-16C		0.3	456	5550	17	1000	4.3	8-13	0.15	451	2200	10		
10-21	9914-17A	(1)	0.3	455	7200	--	---	4.0	8-14	0.3	446	2300	10		
10-22	9914-18A	(2) (3)	0.15	N.E.340	---	--	---	--	8-14	0.5	459	2280	10		
10-22	9914-18B	(4)	0.15	469	9000	--	---	5.2	8-15	0.3	446	2300	10		
10-22	9914-18C		0.15	456	6450	23	600	4.1							
10-23	9914-19A	(5)	0.15	218	5500	40	400	7.8							
10-23	9914-19B		0.15	460	8000	15	1000	4.7							
10-24	9914-21A	(6)	0.15	471	8050	16	900	5.8							
	Viton A Compound														
10-8	9914-14E		0.1	330	2220	60	40	7.2	8-12	0.1	310	2300	5	330	2250
10-23	9914-19C		0.1	330	8600	50	600	8.8	8-14	0.1	325	2250	6		
									8-15	0.3	310	2100	10		
	Tricresyl Phosphate														
10-18	9914-16D		0.1	254	6900	32	1200	8.2	8-7	0.1	240	2400	4	260	2150
	Leather		---	---	----	---	---	---	8-12	0.1	163	2300		170	2100
									8-12	0.1	160	2280			

(1) High pressure oxygen leaked through valve into bomb and pressure in bomb was reduced by blowing to atmosphere three times during test.
(2) N.E. - means no explosion at the indicated temperature
(3) Test stopped at 340°C because of leakage. Sample had started to decompose.
(4) High pressure oxygen leaked through valve into bomb.
(5) It is thought that this sample may have been contaminated.
(6) This sample was composed entirely of shavings.

TABLE 8

SUMMARY OF HIGH PRESSURE OXYGEN BOMB TESTS

Test date 1963	Sample material and test No.	Notes	Sample weight in grams	Ignition temp °C	Ignition pressure psi	Temp. rise on ignition °C	Pressure rise on ignition psi	Initial oxygen press. in bomb before ht'g	Voltage setting on furnace variac	Average ht'g rate °C/min. during last 20 min.
	Omweld Anti-Friction Compound No. 54									
10/23	9914-20A		0.1	235	7400	40	900	5550	95	8.5
10/25	9914-23B		0.12	230	7600	31	950	6000	80	6.0
10/25	9914-23C		0.11	237	2300	50	200	1900	80	5.9
	Aroclor 1254									
10/23	9914-20B		0.15	355	7550	35	1400	5150	95	8.3
10/23	9914-20C		0.11	355	8100	17	1000	5750	95	7.8
10/24	9914-21B		0.14	376	2300	31	300	1750	95	6.7
10/24	9914-21C		0.13	385	2400	90	400	1800	95	8.3
	Virgin Teflon									
10/25	9914-22A		0.1	474	7870	16	600	4850	95	5.6
10/25	9914-22B	(leak)	0.1	471	7150	12	600	4850	95	5.6
10/25	9914-22C		0.2	465	7750	16	1200	5150	95	5.3
10/25	9914-23A		0.1	469	2650	20	100	1950	95	6.3
	Teflon 100X									
10/30	9914-24A		0.1	414	7800	9	800	4600	95	5.2
10/30	9914-24B	(leak)	0.1	412	6600	10	600	4600	95	5.1
10/30	9914-24C		0.1	410	8150	10	800	5250	95	4.8
10/30	9914-25A		0.1	413	1850	32	50	1400	95	6.8
	Kel-F 5500									
10/31	9914-25B		0.1	345	7650	11	600	5050	90	6.5
10/31	9914-25C	(leak)	0.1	340	7200	16	600	5400	85	5.7
10/31	9914-25D		0.1	352	2200	38	100	1900	85	5.9
	Kel-F 3700									
10/31	9914-26A		0.1	332	7600	15	800	5300	85	5.9
10/31	9914-26B		0.1	331	6900	15	700	5050	85	5.8
10/31	9914-26C		0.1	341	2350	30	100	1900	85	5.8
	Kel-F 81									
11/1	9914-26D	(1)	0.1	433	8300	10	500	5150	85	3.8
11/1	9914-27A	(1)	0.1	433	7300	8	400	4750	95	6.2
11/1	9914-27B	(1)	0.1	431	2500	15	200	1850	87	4.2
11/4	9914-27C	(1)	0.1	432	7800	10	500	4900	95	5.9
11/11	9914-32B	(2)	0.1	425	8100	10	400	5050	95	5.9

(1) 1/8" rod from Adam Spence Corp.
(2) 1/16" sheet from Fluorocarbon Company.

Test date 1963	Sample material and test No.	Notes	Sample weight in grams	Ignition temp °C	Ignition pressure psi	Temp. rise on ignition °C	Pressure rise on ignition psi	Initial oxygen press. in bomb before ht'g	Voltage setting on furnace variac	Average ht'g rate °C/min. during last 20 min.
	Kel-F 90 Grease									
11/4	9914-27D		0.14	435	7200	10	500	4800	95	6.6
11/4	9914-30A	(leak)	0.12	448	7200	5	300	5000	95	6.7
11/5	9914-30B		0.13	445	8500	7	500	5150	95	5.8
11/5	9914-30C		0.11	435	2500	16	300	1900	95	8.0

TABLE 8 (Cont'd)

SUMMARY OF HIGH PRESSURE OXYGEN BOMB TESTS

Test date 1963	Sample material and test No.	Notes	Sample weight in grams	Ignition temp °C	Ignition pressure psi	Temp. rise on ignition °C	Pressure rise on ignition psi	Initial oxygen press. in bomb before ht'g	Voltage setting on furnace variac	Average Ht'g rate °C/min during last 20 min.
	High Purity Goop									
11/5	9914-28A	(1) (2)	0.1	N.E.500	8300	---	---	5100	95	5.5
11/5	9914-28B		0.22	411	6100	21	800	4400	95	7.0
11/5	9914-28C		0.1	419	2550	7	300	1950	95	6.8
11/6	9914-28D	(1) (2) (leak)	0.15	N.E.500	6000	--	---	5000	95	5.0
11/7	9914-30D	(1) (2)	0.18	N.E.500	8200	--	---	5050	90	3.8
11/8	9914-31A	(1) (2)	0.1	N.E.450	2900	--	---	1900	95	5.8
11/8	9914-31B	(2) (3)	0.3	N.E.440	7500	--	---	4850	86	2.8
	(1) When bomb disassembled, sample had disappeared and evidence of ignition, but no indication of ignition on charts- slight fumes were also noticed when bomb disassembled. (2) N. E. - means no explosion at indicated temperature (3) A residue of .03 gram of sample remained									
	Halocarbon Grease 25-10									
11/6	9914-29A	(leak)	0.12	438	7000	12	500	4800	90	5.0
11/6	9914-29B		0.13	444	7650	7	600	5000	90	5.0
11/6	9914-29C		0.12	431	2750	14	200	1850	95	6.6
11/6	9914-29D		0.11	439	7700	5	500	4900	95	6.2
	Molykote Z									
11/8	9914-31C		0.13	270	7000	140	350	5000	95	9.6
11/8	9914-31D		0.12	267	7300	120	400	5300	85	4.1
11/8	9914-32A		0.11	277	2700	103	50	1900	85	6.9
	Duroid 5600									
11/11	9914-32C		0.1	480	7300	10	300	4900	95	5.5
11/11	9914-32D		0.2	470	8100	12	600	5200	95	5.5
11/12	9914-33A	(1)	0.2	468	2450	12	400	1900	95	6.2
	(1) Residue same shape as initial sample, but it collapsed to dust when touched									
	Duroid 5650									
11/12	9914-33B		0.2	458	8000	17	1000	4900	95	5.7
11/12	9914-33C	(leak)	0.2	444	7450	17	900	4850	95	5.7
11/12	9914-33D	(1)	0.2	461	2450	20	150	1750	95	5.7
	(1) White residue same shape as initial sample									
	Duroid 5813									
11/13	9914-34A		0.2	464	8050	16	1000	4850	95	6.5
11/13	9914-34B	(leak)	0.2	463	7000	17	800	4900	95	6.3
11/13	9914-34C		0.2	463	2300	16	200	1650	95	6.0
	Duroid 5870									
11/13	9914-34D	(leak)	0.2	463	7550	18	1200	5000	95	6.3
11/14	9914-35A		0.2	469	7900	16	1200	4850	95	6.0
11/14	9914-35B		0.2	452	2650	30	250	1800	95	5.9

N.E. - means no explosion at the indicated temperature.

TABLE 8 (Cont'd)

SUMMARY OF HIGH PRESSURE OXYGEN BOMB TESTS

Test date 1963	Sample material and Test No.	Notes	Sample weight in grams	Ignition temp. °C	Ignition pressure psi	Temp. rise on ignition °C	Pressure rise on ignition psi	Initial oxygen press. in bomb before ht'g	Voltage setting on furnace variac	Average ht'g rate °C/min during last 20 min.
	Rulon A									
11/15	9914-35C	(leak)	0.2	465	7200	22	800	4900	95	6.1
11/15	9914-35D		0.2	466	7700	20	900	4800	95	6.8
11/18	9914-36A		0.2	463	2600	22	150	1900	90	4.9
	Rulon B									
11/18	9914-36B		0.2	460	7900	25	600	4850	95	6.0
11/18	9914-36C		0.2	465	7400	25	500	4700	95	7.0
11/18	9914-36D		0.2	466	2600	20	200	1900	85	3.8
	Rulon C									
11/19	9914-37A		0.2	465	7300	15	700	4600	95	5.9
11/19	9914-37B		0.2	471	7750	16	750	4750	95	6.3
11/19	9914-37C		0.2	458	2600	25	300	1900	95	7.0
	Everlube Solid Film Lubricant No. 811 (Dried)									
11/19	9914-37D	(Powder)	0.21	216	6200	220	600	4800	92	7.3
11/19	9914-38A	(1/16" chunks)	0.20	250	2350	240	100	1850	88	7.5
11/20	9914-38B	(1/16" chunks)	0.20	254	8700	175	900	6000	88	7.0
	Halocarbon Oil Series 13-21									
11/20	9914-38C		0.17	442	7700	10	600	4800	95	7.6
11/20	9914-38D	(1)	0.11	N.E.500	7800	---	---	4800	92	5.0
11/20	9914-39A		0.18	435	7800	15	800	4800	92	6.3
11/21	9914-39B		0.18	427	2400	23	400	1700	88	5.0
		(1) Sample ignited without any indication being evident on the recorder charts								
	Burnil Brand Microplates									
11/21	9914-39C	(1) (2)(Powder)	0.08	N.E.500	9200	---	---	5600	90	4.0
11/21	9914-39D	(1) (2)(Powder)	0.09	N.E.500	2400	---	---	1700	95	5.6
11/21	9914-40A	(2)(Paper)	0.2	N.E.500	8000	---	---	4700	95	5.4
		(1) Unable to get any more weight of powder into test tube (2) All samples were still intact when the bomb was disassembled								
	Viton A (Virgin)									
11/22	9914-40B		0.11	310	7200	16	500	4900	90	7.6
11/22	9914-40C		0.12	310	2400	80	100	1900	85	6.4
11/22	9914-40D		0.13	300	7000	60	600	5000	85	6.8
	Viton B (Virgin)									
11/22	9914-41A		0.11	318	7000	47	600	4800	85	6.2
11/22	9914-41B		0.11	316	7700	19	800	5300	85	7.0
11/23	9914-41C		0.11	325	2350	30	100	1700	85	5.0

N.E. - means no explosion at the indicated temperature.

TABLE 8 (Cont'd)

SUMMARY OF HIGH PRESSURE OXYGEN BOMB TESTS

Test date 1963	Sample Material and Test No.	Notes	Sample weight in grams	Ignition temp. °C	Ignition pressure psi	Temp. rise on ignition °C	Pressure rise on ignition psi	Initial oxygen press. in bomb before ht'g	Voltage setting on furnace variac	Average ht'g rate °C/min during last 20 min
	Nano Pipe and Joint Compound									
11/26	9914-41D	(1)	0.17	N.E.500	8150	---	---	4950	95	6.0
11/26	9914-42A	(2)	0.21	422	7700	12	100	4900	95	6.5
11/26	9914-42B	(2)	0.19	427	7900	10	100	5050	95	6.7
11/27	9914-42C	(2)	0.20	430	2600	16	25	1850	95	5.4
		(1) Sample may be partly decomposed. Color changed from dark to light gray.								
		(2) Sample sintered								
	Dixon's Flake Graphite No.1									
11/27	9914-43A		0.23	N.E.500	7500	---	---	4650	95	3.5
11/27	9914-43B		0.24	N.E.500	7500	---	---	4650	95	5.0
11/27	9914-43C		0.23	N.E.500	2700	---	---	2000	95	6.0
	Oxylube No. 703 (Dried)									
12/2	9914-43D	(1)	0.1[illegible]	N.E.500	8600	---	---	4900	95	5.0
12/2	9914-44A	(1)	0.23	N.E.500	2400	---	---	1700	90	4.0
12/2	9914-44B	(1)	0.20	N.E.500	7550	---	---	4650	92	3.0
12/4	9914-45B	(6)(2)	estimated wt.0.5+	190	6200	45	300	4700	95	7.7
12/4	9914-45C	(3)(4)	0.22	N.E.500	8800	---	---	5150	95	4.0
12/4	9914-45D	(5)(2)	0.21	N.E.310	7300	---	---	4900	95	7.2
12/4	9914-46A	(6)(2)	0.53	200	6500	40	500	4900	95	8.0
12/13	9914-51D	(6)(2)	0.40	238	2200	42	25	1800	95	8.0
		(1) Sample tested as chunk; some oxidation on outside of chunk evidenced by yellow color.								
		(2) Sample powdered in mortar and tested in powdered state.								
		(3) Sample composed of small pieces and powder as present in original dried sample.								
		(4) Sample intact when bomb disassembled; slight oxidation on bottom of sample evidenced by yellow color.								
		(5) Sample intact, no discoloration. Powder slightly sintered.								
		(6) Sample completely disappeared. Bomb thoroughly gumked with combustion products and corrosion.								
	Oxyseal (dried)									
12/3	9914-44C	(1)	0.15	347	7900	153	500	4950	95	7.4
12/3	9914-44D	(1) (leak)	0.20	348	6800	160	500	4900	90	6.4
12/3	9914-45A	(1)	0.20	360	2700	160	500	2000	90	4.2
		(1) Enough heat was liberated to melt the bottom of the pyrex test tube.								
	Omrald Anti-Friction Compound No. 64									
12/6	9914-47A	(1)	0.22	N.E.500	8500	---	---	5000	95	5.0
12/8	9914-47B	(2)	0.21	N.E.400	8000	---	---	5000	95	5.0
12/8	9914-47C		0.80	414	6700	46	1900	4400	95	5.0
12/8	9914-47D		0.74	410	7600	Bursting Disc	Ruptured	4800	95	5.0
12/8	9914-47E		0.52	410	2500	65	1000	1900	95	4.1
		(1) Sample completely disappeared when bomb disassembled.								
		(2) Sample partly decomposed, but mostly intact.								

N.E. - means no explosion at the indicated temperature.

TABLE 8 (Cont'd)

SUMMARY OF HIGH PRESSURE OXYGEN BOMB TESTS

Test date 1963	Sample material and test No.	Notes	Sample weight in grams	Ignition temp. °C	Ignition pressure psi	Temp. rise on ignition °C	Pressure rise on ignition psi	Initial oxygen press. in bomb before ht'g	Voltage setting on furnace variac	Average ht'g rate °C/min during last 20min.
	<u>Rectorseal No. 15</u> (Dried) MIL-T-5542B (ASG)									
12/10	9914-48A		0.28	374	7700	104	2500	4900	98	6.3
12/10	9914-48B		0.18	355	6800	80	1000	4600	96	6.9
12/10	9914-48C		0.16	374	2500	70	400	2000	96	6.9
	<u>50/50 Soft Solder</u>									
12/10	9914-48D	(1)	0.6	N.E.500	7500	---	---	4700	96	3.4
12/10	9914-49A	(1)	0.5	N.E.500	7600	---	---	4800	100	5.0
12/10	9914-49B	(1)	0.5	N.E.500	2600	---	---	1900	100	6.3
		(1) Solder melted and a considerable amount of oxide formed.								
	<u>Almasol Powder (325 Mesh)</u>									
12/11	9914-49C	(1)	0.10	N.E.500	8000	---	---	4600	100	7.8
12/11	9914-49D	(1) (leak)	0.14	N.E.500	7000	---	---	4600	100	6.0
12/11	9914-49E	(1)	0.15	N.E.500	2900	---	---	2000	100	5.5
12/11										
		(1) Sample intact. No evidence of oxidation.								
	<u>Key Absolute</u> (Dried) MIL-T-5542-B (ASG)									
12/11	9914-49F	(1)	0.12	N.E.500	7300	—	---	4550	95	3.0
12/12	9914-50A		0.56	342	6600	40	1200	4300	95	6.1
12/12	9914-50B	(2)	0.75	355	2500	40	500	1900	92	5.8
		(1) Sample decomposed, white ash left. (2) Bursting disc ruptured.								
	<u>High Purity Goop</u>									
12/12	9914-50C	(1)	0.51	N.E.420	8500	---	---	5100	95	4.0
12/13	9914-51A		0.50	398	2800	55	900	1900	95	5.0
		(1) Decomposition occurred, clear oily liquid condensed on bomb head, part of sample remaining. Fumes issued from bomb when it was disassembled.								
	<u>Neoprene O-Ring</u> (MIL-R-1149, Type II, Class I)									
12/13	9914-51B		0.11	200	2500	175	200	2000	95	7.4
12/13	9914-51C		0.05	190	7100	90	900	5500	89	6.0
	<u>Linde Green Pipe Compound</u>									
12/5	9914-46B		0.26	373	7450	14	250	4650	95	7.4
12/5	9914-46C		0.52	362	6900	30	600	4500	92	6.9
12/5	9914-46D		0.31	356	2350	54	200	1750	90	7.1

N.E. - means no explosion at the indicated temperature.

TABLE 9

PRELIMINARY DATA - PROMOTED IGNITION TEST

1964 date	Test No.	Promoter	Promoter wt. mg.	Specimen	Specimen wt. mg.	Physical configuration	Ignition temp. °C	Ignition pressure psig.	Approximate specimen size width x length mm.	Remarks
1-3	9914-59A	Neoprene	61	Stainless Steel Type 304	54	A	N.R.340	2400	6 x 8	O-ring charred and hard but original shape
1-3	9914-59B	"	55	Stainless Steel Type 304	52	A	200	2100	6 x 8	Complete combustion
1-3	9914-59C	"	59	Stainless Steel Type 304	53	A	325	2300	6 x 8	Complete combustion
1-3	9914-59D	"	57	Monel	50	A	N.R.220	2200	6 x 8	O-ring hard but original shape
1-6	9914-60A	"	57	Monel	51	A	N.R.350	2250	6 x 8	Ignition occurred as oxygen pressure reduced from bomb. Monel melted and oxidized.
1-6	9914-60B	"	59	Monel	52	A	198	2000	6 x 8	Monel melted into ball and oxidized on outer surface.
1-6	9914-60C	"	57	Monel	49	A	350	2300	6 x 8	Monel melted into ball and oxidized on outer surface.
1-6	9914-60D	"	55	Monel	50	A	358	2250	6 x 8	Monel melted into ball and oxidized on outer surface.
1-7	9914-61A	"	59	----	--	--	360	2250	-----	-------------------------
1-7	9914-61B	"	55	----	--	--	360	2300	-----	-------------------------
1-7	9914-61C	"	55	Monel	150	A	360	2300	8 x 20	Monel melted and surface oxidation
1-7	9914-61D	"	55	Monel	150	A	200	2050	8 x 20	Monel melted and surface oxidation
1-8	9914-62A	"	55	Silver	140	A	349	2200	6 x 15	Silver melted, slight oxidation
1-8	9914-62B	"	56	Gold	170	A	200	2000	7 x 9	Gold melted into ball, no oxidation.
1-8	9914-62C	"	29	----	--	--	365	2000	-----	-------------------------
1-8	9914-62D	"	15	----	--	--	374	1950	-----	-------------------------
1-9	9914-63A	"	6	----	--	--	378	2150	-----	-------------------------
1-9	9914-63B	"	6	Stainless Steel Type 304	60	B	365	2300	7 x 8	Slight oxidation no ignition of stainless steel
1-9	9914-63C	"	7	Stainless Steel Type 304	44	B	375	1950	5 x 8	Slight oxidation no ignition of stainless steel
1-9	9914-63D	"	15	Stainless Steel Type 304	60	B	366	2000	7 x 8	Slight oxidation no ignition of stainless steel
1-10	9914-64A	"	29	Stainless Steel Type 304	49	B	343	2100	7 x 7	Oxidation of metal in contact with Neoprene.
1-10	9914-64B	"	29	Stainless Steel Type 304	55	B	365	2200	7 x 8	Complete combustion.
1-10	9914-64C	"	13	Stainless Steel Type 304	81	B	318	2200	7 x 7	Slight oxidation of stainless steel.
1-10	9914-64D	"	28	Stainless Steel Type 304	60	B	378	2000	7 x 8	Complete combustion.
1-13	9914-65A	"	19	Stainless Steel Type 304	59	B	358	1650	7 x 8	Stainless steel oxidized but did not burn.
1-13	9914-65B	"	21	Stainless Steel Type 304	56	B	327	1900	7 x 8	Stainless steel oxidized but did not burn.
1-13	9914-65C	"	24	Stainless Steel Type 304	57	B	316	2100	7 x 8	Stainless steel oxidized but did not burn.
1-13	9914-65D	"	26	Stainless Steel Type 304	55	B	378	1800	7 x 8	Stainless steel oxidized but did not burn.
1-15	9914-66A	"	26	Stainless Steel Type 304	55	B	345	2500	6 x 8	Complete combustion
1-15	9914-66B	Viton A	51	Stainless Steel Type 304	51	C	316	2200	6 x 8	Complete combustion
1-15	9914-66C	Viton A	50	Monel	50	C	310	2200	6 x 8	Monel melted, slight oxidation.
1-15	9914-66D	Viton A	73	Monel	52	C	307	2000	6 x 8	Monel melted, partly oxidized.
1-15	9914-67A	Viton A	50	Monel	50	C	312	7500	5 x 10	Monel melted some and surface oxidized.
1-13	9914-67B	Viton A	34	Stainless Steel Type 304	50	C	332	2200	5 x 10	Complete combustion.

TABLE 10

PROMOTED IGNITION TEST

1964 date	Test No.	Neoprene wt. mg.	Specimen	Specimen wt. mg.	Physical configuration	Ignition temp. °C	Ignition pressure psig.	Specimen size width x length mm.	Remarks
1-15	9914-67C	35	Monel	49	C	345	1900	5 x 10	Monel melted into ball.
1-16	9914-67D	31	Stainless Steel Type 304	51	C	194	6000	5 x 10	Complete combustion
1-16	9914-68A	21	Stainless Steel Type 304	51	C	310	8000	5 x 10	Complete combustion
1-16	9914-68B	30	Monel	50	C	315	7500	5 x 10	Monel melted and oxidized.
1-16	9914-68C	18	Stainless Steel Type 304	52	C	189	6750	5 x 10	Complete combustion
1-16	9914-68D	15	Monel	50	C	190	6700	5 x 10	Monel melted and oxidized.
1-16	9914-69A	13	Stainless Steel Type 304	50	C	189	7400	5 x 10	Complete combustion
1-16	9914-69B	12	Monel	[illegible]	C	190	7450	5 x 10	Monel partly oxidized, not melted.
1-17	9914-69C	7.6	Stainless Steel Type 304	49	C	190	7400	5 x 10	Complete combustion
1-17	9914-69D	7.0	Gold	128	C	189	7400	5 x 10	Gold melted at bottom where promoter contacted it.
1-17	9914-70A	4.4	Stainless Steel Type 304	50	C	192	7200	5 x 10	Complete combustion
1-17	9914-70B	1.8	Stainless Steel Type 304	50	C	N.E.280	7700	5 x 10	O-ring oxidized but still intact. No oxidation of stainless steel.
1-17	9914-70C	14.5	Monel	98	C	190	7000	5 x 18	Monel melted at bottom and oxidized 1/2 way up.
1-17	9914-70D	14.4	Monel	60	C	191	7200	5 x 10	Monel melted at bottom and side. Heavy oxidation.
1-17	9914-71A	2.5	Stainless Steel Type 304	49	C	316	7450	5 x 10	Stainless oxidized but did not burn.
1-17	9914-71B	14.0	Monel	41	C	191	5650	5 x 7	Oxidized, started to melt one corner.
1-17	9914-71C	4.3	Stainless Steel Type 304	49	C	306	7100	5 x 10	Stainless oxidized, but did not burn.
1-20	9914-71D	17.2	Monel	158	F	184	7850	5 x 29	13 mm burned 16 mm remained
1-20	9914-72A	17.6	Monel	50	C	186	7450	5 x 10	Complete combustion.
1-20	9914-72B	17.9	Monel	102	C	185	7400	5 x 18	Complete combustion.
1-20	9914-72C	7.8	Stainless Steel Type 304	47	C	194	7250	5 x 10	Oxidized 5 mm from bottom but did not burn.
1-20	9914-72D	11.0	Stainless Steel Type 304	150	F	186	7500	5 x 30	Complete combustion.
1-20	9914-73A	8.5	Stainless Steel Type 304	50	C	190	7400	5 x 10	Complete combustion.
1-20	9914-73B	6.5	Gold	86	C	195	6700	5 x 7	Gold partly melted.
1-20	9914-73C	8.5	Stainless Steel Type 304	161	F	190	7000	5 x 30	Complete combustion.
1-21	9914-73D	10.0	Nickel	159	F	288	7350	5 x 30	Oxidation inside and out where neoprene touched. Hole burned through nickel.
1-21	9914-74A	13.4	Nickel	159	F	190	6800	5 x 30	Bottom 3 or 4 mm melted with surface oxidation.
1-21	9914-74B	17.6	Nickel	159	F	189	6600	5 x 30	Bottom 2 or 3 mm melted with light surface oxidation.
1-21	9914-74C	24.0	Nickel	158	F	185	6600	5 x 30	Light oxidation of bottom
1-21	9914-74D	31.7	Nickel	160	F	188	6500	5 x 30	Light yellow oxidation lower 1/3 of specimen.
1-22	9914-75A	6.5	------	---	--	190	7150	------	--------------------------------
1-22	9914-75B	7.0	------	---	--	192	6100	------	--------------------------------
1-23	9914-75C	17.3	Nickel	55	C	185	7400	5 x 10	Slight oxidation. Bottom corner melted.
1-23	9914-75D	17.0	Monel	58	C	185	7300	5 x 10	Complete combustion.
1-23	9914-79A	17.3	Monel	180	F	178	7600	5 x 30	Partial combustion 16 mm length left.
1-23	9914-79B	8.3	Stainless Steel Type 304	109	F	185	7400	5 x 20	Heavy oxidation at bottom of specimen but did not ignite.

N.E. - means no explosion at the indicated temperature.

TABLE 10 (Cont'd)

PROMOTED IGNITION TEST

1964 date	Test No.	Neoprene wt. mg.	Specimen	Specimen wt. mg.	Physical configuration	Ignition temp. °C	Ignition pressure psig.	Specimen size width x length mm.	Remarks
1-23	9914-79C	56.0	Nickel	179	A	178	7500	8 x 20	Complete combustion.
1-23	9914-79D	17.0	Monel	189	F	280	7550	5 x 30	Partial combustion, 18 mm length left.
1-24	9914-80A	44.4	Nickel	189	A	180	7250	9 x 20	Partial combustion, 14 mm length left.
1-24	9914-80B	41.6	Nickel	113	C	178	8450	5 x 20	Nickel did <u>not</u> ignite. (Neoprene completely wrapped in metal)
1-24	9914-80C	42.3	Nickel	171	F	Recorder not operating.		5 x 30	Nickel partly ignited. 24 mm left. (Only part of neoprene wrapped in metal.)
1-27	9914-80D	40.9	Nickel	42	C	367	8375	5 x 10	Nickel 1/2 melted, some oxidation. 750 psi blow down when pressure too high.
1-27	9914-81A	47.7	Nickel	45	C	174	6810	5 x 10	Nickel 1/2 melted, some oxidation.
1-27	9914-81B	19.2	Aluminum	12.1	C	177	7200	5 x 10	Complete combustion. oxide fused.
1-27	9914-81C	14.1	Aluminum	20.3	C	174	7850	5 x 20	Aluminum just started to melt. Little oxidation.
1-27	9914-81D	16.4	Aluminum	13.0	C	179	7600	5 x 10	Complete combustion.
1-28	9914-82A	10.9	Stainless Steel Type 304	110	C	Test stopped at 226	Pressure to high	5 x 20	Neoprene and specimen intact. Neoprene charred.
1-28	9914-82B	10.8	Inconel X-750	171	F	259	8315	5 x 30	Complete combustion, magnetic oxide.
1-28	9914-82C	7.4	Inconel X-750	160	F	276	7920	5 x 30	Heavy oxidation and small corner missing at bottom but did not ignite.
1-28	9914-82D	13.0	Stainless Steel Type 304	111	F	177	7460	5 x 20	Complete combustion.
1-28	9914-84A	10.6	Inconel 600	155	F	179	6985	5 x 30	Melting and some oxidation at bottom. 25 mm left.
1-29	9914-84B	9.4	Inconel X-750	171	F	185	7810	5 x 30	Complete combustion.
1-29	9914-84C	13.7	Inconel 600	153	F	185	6310	5 x 30	Complete combustion magnetic oxide.
1-29	9914-84D	8.5	Inconel X-750	17.	F	185	7440	5 x 30	Started to melt and heavy oxidation at bottom.
1-29	9914-86A	11.7	Inconel 600	159	F	170	7040	5 x 30	Started to melt and oxidize at bottom.
1-29	9914-86B	9.0	Inconel X-750	183	F	285	8360	5 x 30	Complete combustion.
1-30	9914-86C	13.2	Inconel 600	162	F	190	7370	5 x 30	Complete combustion.
1-30	9914-86D	10.3	Copper	190	F	277	7540	5 x 30	Almost complete combustion. 5 mm left. Surface oxidation.
1-31	9914-88A	8.7	Inconel X-750	186	F	183	7000	5 x 30	Complete combustion.
1-31	9914-88B	8.2	Copper	183	F	170	8440	5 x 30	Partial combustion. 22 mm left - surface oxidation. Black and red crystals in slag.
1-31	9914-88C	12.5	Inconel 600	164	F	183	6380	5 x 30	Bottom 2 or 3 mm melted out of one side with surface oxidation only at bottom.
2-3	9914-88D	12.9	Inconel 600	140	F	183	7960	5 x 30	Bottom two or three mm melted with heavy surface oxidation on melted portion.

TABLE 10 (Cont'd)

PROMOTED IGNITION TEST

1964 date	Test No.	Neoprene wt. mg.	Specimen	Specimen wt. mg.	Physical configuration	Ignition temp. °C	Ignition pressure psig.	Specimen size width x length mm.	Remarks
2-3	9914-90A	7.2	Stainless Steel Type 301	147	F	303	8220	5 x 30	No ignition. Only slight discoloration of upper part. Heavy oxidation inside at bottom.
2-4	9914-90B	9.5	Stainless Steel Type 301	161	F	188	8090	5 x 30	complete combustion.
2-3	9914-90C	11.8	Yellow brass	177	F	186	7325	5 x 30	Bottom 7 or 8 mm melted with heavy oxidation. Heavy black oxidation over entire length of specimen.
2-4	9914-90D	13.2	Yellow brass	166	F	180	7350	5 x 30	Melted bottom 10 mm into ball with heavy oxidation. Black oxidation over entire length of specimen.
2-4	9914-92A	8.0	Stainless Steel Type 301	157	F	190	7860	5 x 30	Complete combustion.
2-18	9914-92B	8.8	Stainless Steel Type 17-7PH	168	F	320	8700	5 x 30	Complete combustion. Bottom of test tube melted.
2-16	9914-92C	15.2	Yellow brass	160	F	185	7700	5 x 30	Bottom 8mm completely combusted. Heavy oxidation over entire length of specimen.
2-18	9914-92D	7.1	Stainless Steel Type 301	153	F	188	7600	5 x 30	Complete combustion. Bottom of test tube melted.
2-18	9914-93A	16.4	Anodized aluminum (<0.1 mil coat)	50	F	181	7700	5 x 30	Complete combustion. Side of test tube melted.
2-18	9914-93B	11.0	Anodized aluminum (<0.1 mil coat)	47	F	185	7450	5 x 30	Complete combustion. Side of test tube melted.
2-18	9914-93C	8.2	Anodized aluminum (<0.1 mil coat)	50	F	185	7500	5 x 30	No melting or significant oxidation .
2-18	9914-93D	9.8	Anodized aluminum (<0.1 mil coat)	52	F	185	7300	5 x 30	No melting or significant oxidation.
2-18	9914-94A	9.0	Stainless Steel Type 316	171	F	324	8700	5 x 30	Complete combustion.
2-18	9914-94B	14.7	Silver	229	F	376	8300	5 x 30	Bottom 6 mm melted.
2-18	9914-94C	13.2	Nickel	168	F	361	8600	5 x 30	2 mm melted out of each of lower corners.
2-19	9914-94D	15.3	Gold	372	F	183	7400	5 x 30	Bottom 3 or 4 mm melted.
2-19	9914-95A	12.6	Lead	209	F	186	7700	5 x 30	Melted into ball. Very little oxidation.
2-19	9914-95B	15.2	Anodized 25 mil aluminum (0.2mil coat)	163	F	185	7200	5 x 20	No ignition.
2-19	9914-95C		176 mg stainless steel Type 304 sealed in 275 mg of Kel-F		--	380	8400	5 x 30	Complete combustion.
2-19	9914-95D		176 mg stainless steel Type 304 sealed in 300 mg of Kel-F		--	415	7500	5 x 30	Complete combustion.
2-19	9914-96A		176 mg stainless steel Type 304 sealed in 218 mg of Teflon FEP		--	419	8300	5 x 30	Complete combustion.
2-19	9914-96C		1 x 4 x 4 mm Kel-F coated on 4 x 4 x 4 mm aluminum	---	--	N.E.500	9000	------	Aluminum intact. No Significant oxidation. Kel-F gone.
2-25	9914-96D	59	Gold	396	A	180	7350	5 x 30	Melted into ball.
2-25	9914-97A	11.3	Anodized aluminum (0.3 mil coat)	57	F	184	8100	5 x 30	Lower 2 or 3 mm melted. No significant oxidation.
2-25	9914-97B	13.8	Anodized aluminum (0.3 mil coat)	53	F	180	8000	5 x 30	Bottom few mm started to melt.
2-27	9914-97C	15.5	Anodized aluminum (0.3 mil coat)	54	F	186	7800	5 x 30	Complete combustion.
2-27	9914-97D	14.6	Anodized aluminum (0.3 mil coat)	50	F	180	7700	5 x 30	Complete combustion.
2-28	9914-98A		1 x 4 x 4 mm Kel-F coated on 1 x 4 x 4 mm aluminum	--	--	385	8000	------	Kel-F ignited, aluminum intact
2-28	9914-98B	54.0	Silver	205	A	182	7000	5 x 30	Entire specimen melted
2-28	9914-98C	56.5	Nickel	175	A	186	7100	5 x 30	Complete combustion.

N.E. - means no explosion at the indicated temperature.

TABLE 11

PRESSURIZATION OF SIMULATED SYSTEM WITH OXYGEN

Date 1964	Test No.	Pressure in Storage Vessel			Pressure in Simulated System			Initial Pressurization time of simulated system, sec.	Electrostatic charge measured volts
		Before Test psi	Immediately after test psi	On Warming after test psi	Immediately after test psi	On Cooling after test psi	When Completely equalized with storage vessel psi		
9/16" O.D. x 5/16" I.D. tubing. Gemini Regulator blanked on low pressure side.									
3-4	11040-3-1	approx. 1100	----	----	900	----	----	0.52	*N.D.
3-4	11040-3-2	approx. 900	----	----	775	----	----	0.51	*N.D.
3-4	11040-3-3	approx. 775	----	----	665	----	----	0.55	*N.D.
3-4	11040-3-4	approx. 1100	----	----	875	----	----	0.52	*N.D.
3-5	11040-3-5	approx. 1200	----	----	975	----	----	0.5	*N.D.
3-5	11040-3-6	approx. 975	----	----	850	----	----	0.5	*N.D.
3-5	11040-3-7	approx. 850	----	----	750	----	----	0.5	*N.D.
3-5	11040-6	2200	----	----	1700	----	----	0.3	< -0.05
3-5	11040-7	1800	----	----	1500	----	----	0.4	< -0.05
3-5	11040-8	3000	----	----	2400	----	----	0.30	< -0.05
3-5	11040-9	2500	2100	2200	2100	1800	----	0.30	< -0.05
3-5	11040-10	3800	2950	3200	2950	----	----	0.30	< -0.05
3-5	11040-11	5100	3850	4200	3850	3250	----	0.26	< -0.05
3-5	11040-12	4700	3600	3800	3600	3100	----	0.28	-0.05
3-5	11040-13	5900	4200	4700	4200	3900	----	0.26	-0.25
3-5	11040-14	7400	5050	5800	5050	4200	----	0.24	----
3-5	11040-15	8500	5600	6500	5600	4800	----	0.24	-0.20
3-5	11040-16	6500	4600	5200	4600	3850	----	0.26	-0.15
3-5	11040-17	5200	3850	4300	3850	3300	----	0.28	-0.14
3-5	11040-18	4300	3300	----	3300	2850	----	0.30	-0.10
Geometric volume of 80 cc added to low pressure side of Gemini Regulator and valve installed to blow down low pressure side.									
3-6	11040-19	3200	2600	2800	2700	2300	----	0.28	-0.10
3-6	11040-20	6300	4400	5100	4400	3500	----	0.26	-0.10
3-6	110-0-21	9000	5750	6600	5750	----	----	0.24	-0.14
3-6	11040-22	10700	6600	7500	6600	----	----	0.22	-0.15
3-6	11040-23	11800	6500	7350	6500	----	7350	0.21	-0.13
30 inches of 1/4" O.D. x 0.083" I.D. tubing substituted for 9/16" O.D. x 5/16" I.D. tubing.									
3-6	11040-24	9200	6200	6800	5700	----	6800	0.14	< -0.05
3-6	11040-25	6800	----	5000	4600	----	5000	0.16	< -0.05
3-6	11040-26	3800	----	3100	2600	----	3100	0.20	< -0.05
Reinstalled 9/16" O.D. x 5/16" I.D. tubing.									
3-9	11040-27	12000	7000	8000	7000	----	8000	0.22	-0.08
3-9	11040-28	12000	7000	8000	7000	----	8000	0.22	-0.08
3-9	11040-29	12000	7000	8000	7000	----	8000	0.22	-0.09
3-9	11040-30	12000	7000	8000	7000	----	8000	0.23	-0.11
3-9	11040-31	12100	7100	8100	7100	----	8100	0.22	-0.09
3-9	11040-32	10500	6300	7250	6300	----	7250	Not recorded	-0.09

*N.D. - None detected

TABLE 11 (Cont'd)

PRESSURIZATION OF SIMULATED SYSTEM WITH OXYGEN

Date 1964	Test No.	Pressure in Storage Vessel			Pressure in Simulated System			Initial Pressurization time of simulated system, sec.	Electrostatic charge measured volts
		Before Test psi	Immediately after test psi	On Warming after test psi	Immediately after test psi	On Cooling after test psi	When Completely equalized with storage vessel psi		
3-10	11040-33	12000	7000	8000	7000	----	8000	0.22	-0.14
3-10	11040-34	12000	7000	8000	7000	----	8000	0.22	-0.16
3-10	11040-35	12200	7100	----	7100	----	----	0.22	-0.12
3-10	11040-36	12000	7000	----	7000	----	----	0.22	-0.12
3-10	11040-37	12000	7000	----	7000	----	----	0.22	-0.32
1/16" Dia. inconel shielded iron-constantan thermocouple substituted for nickel probe. Thermocouple extended approximately two inches into gas space of receiver.									**Increase in Temperature, °C**
3-10	11040-38	10200	6200	----	6200	----	----	0.23	Not recorded
3-10	11040-39	9000	5600	----	5600	----	----	0.23	68°C
1/16" Dia. unshielded iron-constantan thermocouple substituted for inconel shielded thermocouple.									
3-10	11040-40	8100	5200	----	5200	----	----	0.24	80°C

TABLE 12

ADIABATIC COMPRESSION TESTS

Date 1964	Pressure in Storage Vessel		Sample	Weight of sample in grams	Pressurization time of 51" length of 5/16" I.D. tubing and sample, sec	Remarks
	Before test psi	Immediately after test psi				
3-10	7200	6900	Viton A (Virgin)	0.217	0.05	Sample ignited
3-10	7100	6700	Viton A (Virgin)	0.223	0.05	Sample ignited
3-10	7000	6500	Kel-F 81	0.240	0.05	Sample ignited
3-10	6900	6300	Teflon (Virgin)	0.161	0.05	Sample ignited

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13. ABSTRACT

A research program was conducted to develop ignition data on thread lubricants, thread sealants, fluorocarbon plastics, and metals. Spontaneous ignition temperatures were determined in both 2000 psi and 7500 psi oxygen for all the above materials except metals. The spontaneous ignition temperatures for these materials were found to be essentially the same in 7500 psi oxygen and in 2000 psi oxygen. Only three of the tested lubricants are recommended for possible use in 7500 psi systems. None of the thread sealants are recommended. Glass-filled polytetrafluoroethylene is usable only if tightly confined. The relative ease of ignition of metals and alloys was determined by promoted ignition methods in oxygen at 7500 psi. Inconel alloy 600, brass, Monel alloy 400, and nickel were found to have the highest resistance to ignition and combustion among the common alloys and metals. Of the materials tested, stainless steel and aluminum are the least satisfactory for use at oxygen pressures of 7500 psi. A test system was constructed to evaluate the hazards in rapidly charging a 65 cubic inch nickel-lined vessel with high pressure oxygen. A series of rapid charging tests up to as high as 8000 psi proceeded without incident. Electrostatic charges measured during the charging were negligible.

14. KEY WORDS	LINK A		LINK B		LINK C	
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