NRL Report 6606

Flammability in Unusual Atmospheres
Part 2 - Selected Materials in Oxygen-Nitrogen and Oxygen Helium Mixtures at Pressures up to 315 PSIA

September 22, 1967

DEPARTMENT OF THE NAVY
OFFICE OF NAVAL RESEARCH

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PREVIOUS REPORT IN THIS SERIES

"Flammability in Unusual Atmospheres, Part 1—Preliminary Studies of Materials in Hyperbaric Atmospheres Containing Oxygen, Nitrogen, and/or Helium," J. E. Johnson and F. J. Woods, NRL Report 6470, Oct. 31, 1966

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Flammability in Unusual Atmospheres
Part 2 - Selected Materials in Oxygen-Nitrogen and Oxygen Helium Mixtures at Pressures up to 315 PSIA

F. J. Woods and J. E. Johnson

Fuels Branch
Chemistry Division

September 22, 1967

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ABSTRACT

The Naval Research Laboratory has been investigating the flammability of materials under unusual atmospheric conditions. Ignition and flame spread of several fabrics and paper were measured at pressures from 315 psia down to the limiting pressures for ignition. The minimum total pressure limit for the standard filter paper used was much lower at 41% O_2 than at 21% O_2 in O_2/N_2 mixtures. Also, the partial pressure of O_2 required for ignition at 41% was much lower than at 21% O_2. Although, in general, materials have a faster burning rate in helium mixtures than in nitrogen mixtures, there are some significant exceptions to this finding. The nature of the material has been shown to have a marked influence on the effect that variables, such as pressure, oxygen content, and diluent, have on the rate of burning.

PROBLEM STATUS

This is an interim report; work is continuing on the problem.

AUTHORIZATION

NRL Problem C01-03
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FLAMMABILITY IN UNUSUAL ATMOSPHERES

PART 2 - SELECTED MATERIALS IN OXYGEN-NITROGEN AND OXYGEN-HELIUM MIXTURES AT PRESSURES UP TO 315 PSIA

INTRODUCTION

In recent years men have become increasingly accustomed to living in unusual atmospheric environments for various reasons. All manned space flights by the U.S. to date have used essentially 100% oxygen at the reduced pressure of 5.5 psia. Hyperbaric medicine has provided therapeutic environments based on atmospheres at pressures approaching 100 psia, in which patients breathe pure oxygen (1). The average oxygen concentration of such a system is understandably variable. Aquanauts have been living deep in the sea in gaseous environments composed almost entirely of helium, at the ambient pressure of many atmospheres, purposely "contaminated" with sufficient oxygen necessary to maintain a life-supporting supply. It is expected that men will soon be living and working at the approximate depth of 200 meters and ambient pressure of over 20 atmospheres (300 psia), with oxygen at about 1 percent.

In these various situations the deviations from the normal earth atmosphere at sea level include reduced or increased pressure, various percentages of oxygen, and substitution or removal of the diluent nitrogen. These strange environments have been approached somewhat gingerly with regard to possible deleterious physiological effects, such as oxygen toxicity, nitrogen narcosis, and "the bends." However, it appears that, until very recently, not nearly enough attention had been given to the flammability hazards in these situations, which are especially serious because it is difficult or impossible to escape the fire or its combustion products in the confined spaces involved. Of course, it was generally known that pure oxygen constituted an unusually hazardous atmosphere because of the extreme ease of ignition and astonishingly high burning rates which could occur (2-4). Although several serious incidents had occurred in spacecraft simulators on the ground (2), it was not until early 1967 that two closely spaced fatal fires in oxygen atmospheres emphasized the extreme flammability hazard inherent in undiluted oxygen, and the scarcity of information on this subject. These two incidents were the Apollo I capsule fire of January 27 and the fire in an experimental chamber at USAF Aeromedical Laboratory at Brooks Air Force Base on January 30.

Two years earlier, in February 1965, a fatal fire occurred in a chamber at the Navy Experimental Diving Unit, Washington, D.C. This fire demonstrated the disaster which can occur in a confined chamber even at a much lower percentage of oxygen (5,6). At the onset of the fire the composition of the atmosphere in the chamber was 28% oxygen, 36% nitrogen, and 36% helium, at a chamber pressure of 55 psia. Preliminary studies at NRL (6) showed that burning rates of materials under these atmospheric conditions were substantially higher than in air at sea level because of three major factors: increased oxygen percentage, increased pressure, and substitution of helium for nitrogen. The most important factor in this situation was the oxygen enrichment.

These preliminary studies revealed a decided lack of experimental data exists concerning the effect of several atmospheric variables, such as oxygen content, pressure, and diluents, on flammability. The present work was planned to extend the study of the flammability of materials to pressures ranging from subatmospheric to 300 psia. Operations with the Sea Lab chambers in the near future are not expected to exceed depths corresponding to this pressure. To get a better understanding of the ignition and combustion of materials in unusual gas mixtures, it was also an aim to extend the present studies to the lower pressure limits for ignition, i.e., those pressures below which the material does not ignite in the particular gas mixture being studied.

The gases studied were limited to mixtures of oxygen, nitrogen, and helium. Some mention has been made of the use of neon and hydrogen as diluents (7), but apparently no serious practical use of these gases is contemplated soon.

LITERATURE SUMMARY

In the previous report (6) on this problem a brief literature summary was included, which showed the limited amount of information that had been published about the effect of atmospheric variables, such as gas composition and pressure, on the flammability of materials.

A recent progress report from the U.S. Bureau of Mines (8) discussed the initiation of a project undertaken at the request of the Compressed Gas Association with support by the U.S. Public Health Service. This work is intended to determine the ignition and combustion characteristics of a variety of combustible materials in hyperbaric atmospheres. Minimum ignition temperatures, minimum ignition energies, and rates of burning or flame spread are to be determined in air and other oxygen-nitrogen atmospheres at pressures to 75 psig. Both spontaneous ignition and induced ignition (by hot wire and electrostatic spark) will be studied. Some preliminary data confirmed the much greater flame spread, by a factor of about 10:1, of fabric specimens held vertically rather than horizontally (9 .

Huggett, von Elbe, and Haggerty (10) recently published data on the combustibility of materials in oxygen-helium and oxygen-nitrogen atmospheres at pressures of 258 to 760 mm Hg. This work was an extension of that published earlier (4) on oxygen alone at 258 mm Hg. Their study showed that flames spread faster over combustible solids as the percentage of oxygen is increased. Flames spread faster in helium-oxygen mixtures than in nitrogen-oxygen mixtures. They further concluded that the flame spread rate in an atmosphere of constant O_2/He or O_2/N_2 ratio is independent of the total pressure in the range studied. The "apparent" ignition energy was strongly dependent on the atmospheric composition and the mechanism by which the ignition energy was supplied. However, the "true" ignition energy was approximately independent of the atmosphere in the flammable range. A roughly linear correlation was shown between rate of flame spread for a given material and the logarithm of the heat capacity of the gas mixtures per mole of oxygen.

In previous work at NRL Johnson and Woods (6) studied the effect of oxygen enrichment, increased pressure, and substitution of helium for nitrogen on the flammability of fabrics and other solids. Oxygen enrichment was very important, in that many materials which would not ignite at all in 21% oxygen, ignited and burned readily in 31% or 41% oxygen. System pressure often had a pronounced effect, especially in borderline cases, in that ignition frequently occurred at several atmospheres pressure, whereas no ignition occurred at lower pressures. Substitution of helium for nitrogen as the diluent gas decreased the tendency of materials to ignite using a hot wire igniter, due largely to the high thermal conductivity of helium dissipating the heat from the igniter. However, once ignited most materials tested burned faster in the atmospheres containing helium. Finally, it was concluded that no simple multiplication factors can be used at present to predict the flammability of materials in the unusual atmospheres. Before any material can be judged safe, it should be examined experimentally under all the important atmospheric conditions of use.

Turner and Segal (1) studied the burning behavior of various combustible materials in air at pressures of 1 to 4 atmospheres. The materials included fabrics, methenamine (hexamethylene tetramine) tablets, and flammable liquids. They found that all these materials burned more rapidly as pressure was increased. In air at 4 atmospheres, for example, the mass burning rate increased by about 80% for cotton sateen and 120% for methyl ethyl ketone as compared to 1 atmosphere pressure.

APPARATUS AND PROCEDURES

The experimental apparatus, procedures, and significance of the experimental conditions used in this investigation were similar to those described in the first report of this series (6). Consequently, only a brief description of the important items will be given here.

Exposure Chambers

The nature of the data required from these studies made it necessary to perform the experiments in closed chambers. Factors that influenced design and selection of the apparatus for these investigations were the following:

1. Closed chambers were needed, capable of evacuation to reasonably low pressures and able to withstand pressures to 315 psia.

2. A remotely controlled method for ignition of material was required.

3. Sufficient volume was needed to avoid significant depletion of oxygen or dilution by the gaseous combustion products.

4. A window was needed for observation of the nature of the reaction and for measuring ignition delay and flame speed visually.

Factor 3 suggests that the vessel must not be too small. However, excessive size also was not desirable because of difficulties, cost of construction, and the need to use various gas mixtures. The amounts of these gases used would be greatly increased at high pressure. The capability of the new hyperbaric chamber for evacuation permitted conservation of gases, because it removed the necessity for flushing the chamber. However, approximately 110 cubic feet (STP) of gas are required to fill this chamber to a pressure of 315 psia.

Atmospheric Pressure Chamber - For convenience, many of the experiments at 1 atmosphere pressure were made in a cubical vacuum oven, shown in Fig. 1, which was commercially available. This chamber had a volume of 12.6 liters and was provided with two valves and a vacuum gauge so that the chamber could be exhausted and the desired gas mixture admitted. The small size of this chamber permitted it to be used in an ordinary fume hood.

Hyperbaric Pressure Chamber - For experiments at both reduced and increased pressures, a larger chamber was constructed by NRL. The NRL chamber, shown in Fig. 2, was constructed so that it could be exhausted and the gas mixture under study admitted at the desired pressure. The chamber was equipped with suitable electric solenoid valves and pressure tubing to permit the gas transfer operations to be done from behind a protective barricade, as shown in Fig. 3. This chamber had a volume of 142 liters and a maximum working pressure of 315 psia.

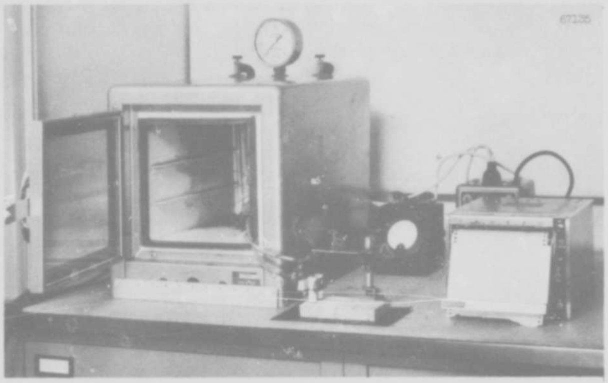

Fig. 1 - Small oven used in flammability studies

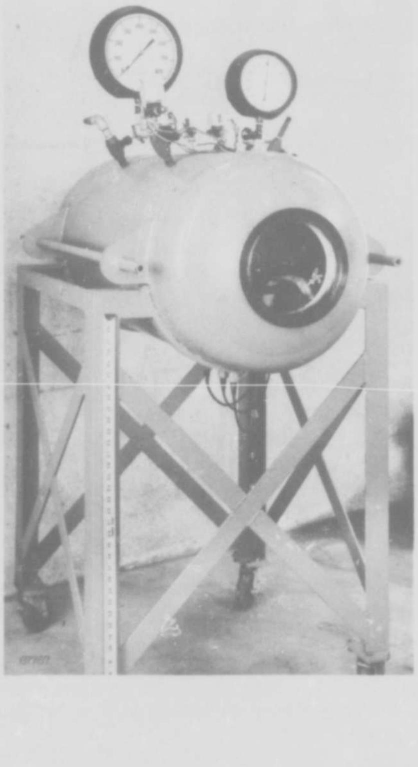

Fig. 2 - Hyperbaric pressure chamber used for flammability studies

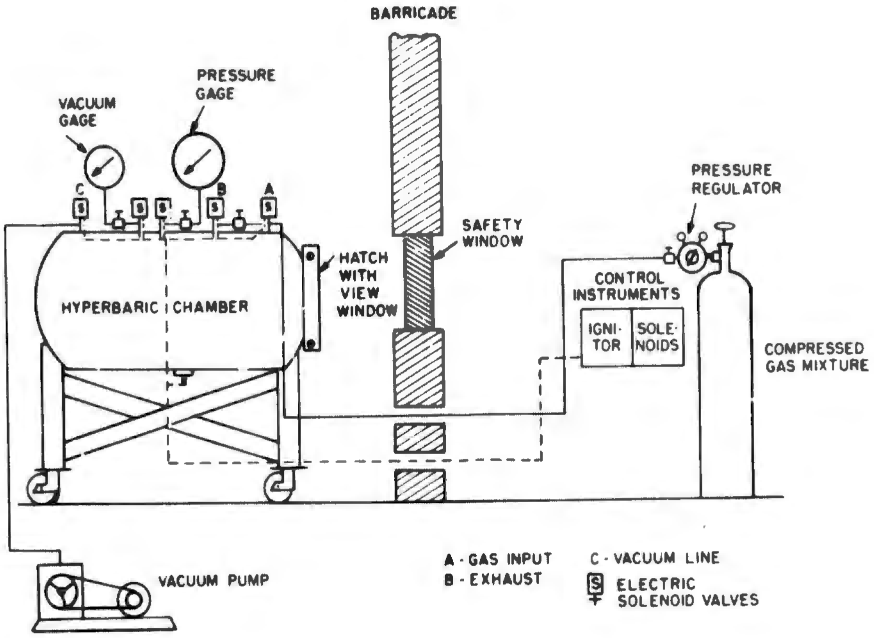

Fig. 3 - Hyperbaric chamber and auxiliary equipment

Ignition and Burning - Because of the need to perform the experiments in sealed chambers, a remotely controlled electrical method of ignition with a heated wire was used throughout. The amount of energy used to heat the igniter wire influenced the ignition delay time but had no effect on the burning rate. The latter was based on the average time (seconds) required to burn along 2 inches of the specimen, not including the first 1-inch segment which touched the igniter.

The ignition apparatus used in the flammability measurements, in both the atmospheric pressure chamber and the hyperbaric pressure chamber, is pictured alongside the oven in Fig. 1 and shown schematically in Fig. 4. A clamp holder for the specimen and a Nichrome wire heater were mounted on a block of transite. The heater was made of 25-gauge Nichrome wire wound on a 1/4 by 7/8 inch form and was operated at 20 volts and 4.3 amperes for most experiments. This ignition apparatus could be operated in either horizontal or vertical position.

Gas Mixtures

Seven principal gas mixture compositions were used in this study:

Table 1
Gas Mixtures Studied

Mixture	Composition (vol %)		
	O_2	N_2	He
A	21	79	—
B	21	—	79
C	31	69	—
D	31	34.5	34.5
E	25.6	10.4	64
F	41	59	—
G	41	—	59

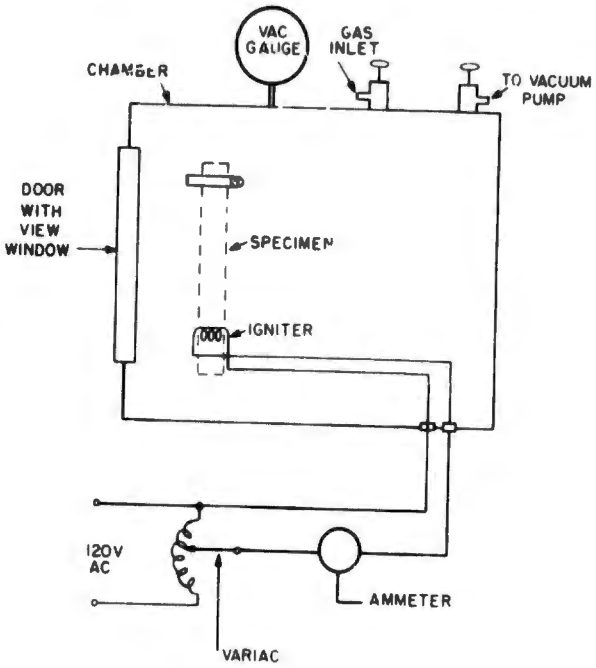

Fig. 4 - Ignition apparatus

Mixture D was prepared by NEDU and delivered to NRL in large gas cylinders. The remaining gas mixtures were obtained from Air Products and Chemicals, Inc., Southern Oxygen Division. Oxygen percentages were determined by means of a Model E-2 Beckman Oxygen Analyzer. In addition, the compositions of the gas mixtures were determined by a gas chromatographic method.

Materials Used

A summary description of each material, identified by a code number, is given in Appendix A. A resin-impregnated filter paper, FM-1, was used as a standard. The paper was of the same material used in the filter involved in the NEDU chamber fire, except that the paper used in these present studies was free of hydrocarbons. The resin impregnation provided a paper with sufficient stiffness so that it could be supported horizontally by clamping only one end.

In addition to the standard paper, several other materials were chosen for more extensive tests. These were principally a cotton terry cloth, FM-3, white duck cotton cloth, FM-28, and a high-temperature-resistant nylon, FM-9.

The cotton terry cloth was made flame-retardant by soaking strips of the material in a solution of 70% borax and 30% boric acid solution. The degree of impregnation with the 70% borax/30% boric acid mixture is expressed as a percentage increase above the original weight of the cloth.

Preparation and Treatment of Sample

Earlier experiments (6) showed no measurable differences in flame speeds after filter paper strips (FM-1) had been exposed for 24 hours to relative humidities (R.H.) of

50% and 100%. Similarly, the experiments with cotton fabrics that were exposed to 50% R.H. showed no difference in ignition or burning rate after exposure to higher relative humidities. In the previous report reference was made to investigations by Huggett, et al. (4), who observed no significant enhancement in ignition or flame speed in oxygen after soaking the samples for 30 days. These data indicated that equilibrium was established very quickly between the atmosphere and the surface of the specimen. Consequently, no extended soaking periods were used in the experiments reported here.

Position and Support of the Sample

Investigations at this Laboratory (6) and elsewhere (8,9) have shown that a vertical sample position with the ignition source applied at the bottom is very conducive to easier ignition and faster flame propagation. The vertical burning rate is as much as 10 times faster than horizontal. For this reason determinations of the fire resistance (ignitability) of materials have been made in the vertical position (6). However, comparative measurements are usually made in the horizontal position because it is difficult to measure the speed of propagation of the flame in the vertical position. The more moderate burning rates obtained horizontally permit the study of the effects of other factors.

GENERAL EXPERIMENTAL PROCEDURE

The procedure for a typical experiment generally followed the one used previously (6); i.e., the specimen of material (4 inches by 1/4 inch for the filter paper and 4 inches by 3/8 inch for the cloth samples) was mounted with one end in the clamp and the other end resting on the heater coil. The cloth specimens were supported by a length of Nichrome wire threaded through the fabric. The apparatus was placed in the chamber, the latter evacuated to about 30 inches Hg, and the desired atmosphere admitted. This evacuation and fill procedure was repeated several times during experimentation at below atmospheric pressure to insure that no dilution of the gas mixture had occurred. A single evacuation and fill was used for all experiments at higher pressures.

When the desired atmospheric conditions had been established, the heater was energized and the progress of the experiment was viewed through the window in the chamber door or hatch in the hyperbaric pressure chamber. Ignition delay and flame speed were measured visually and timed with a stopwatch.

EFFECT OF PRESSURE ON FLAMMABILITY

One of the principal extensions of this work over the previous one (6) was to increase the gas pressure limits in this study to 315 psia.

Table 2 contains data on the flammability of the resin-impregnated filter paper (FM-1) in O_2/N_2 mixtures showing the effect of pressure as the O_2 content was varied from 21% to 41%. Figure 5 illustrates the effect of hyperbaric pressures on flame speeds. At 21% O_2 there is about a 50% increase in burning rate at 315 psia as compared to 15 psia. This moderate increase appears to be essentially linear. At 41% O_2, however, the increase in burning rate is more than 100% at 315 psia over 15 psia. No significant effect of increased pressure on ignition delay time is seen in the data of Table 2. This insensitivity of ignition delay time to pressure was observed previously with materials which are readily flammable under the conditions of study.

Table 2
Effect of Pressure on the Flammability of Filter Paper
in Oxygen-Nitrogen Atmospheres

Total Pressure	Ignition Delay (sec)			Burning Rate (cm/sec)		
	21% O_2	31% O_2	41% O_2	21% O_2	31% O_2	41% O_2
High-Pressure Data						
15 psia	6.5	-	6.9	0.23	-	0.64
30 psia	7.0	-	-	0.26	-	-
65 psia	4.9	-	4.4	0.24	-	0.82
115 psia	4.1	-	5.1	0.27	-	1.10
165 psia	4.7	-	3.1	0.27	-	1.20
215 psia	3.9	-	3.4	0.31	-	1.27
265 psia	4.0	-	4.4	0.31	-	1.34
315 psia	3.5	-	2.9	0.33	-	1.47
Low-Pressure Data						
89 mm Hg	-	-	NI*	-	-	NI
102 mm Hg	-	-	38.7	-	-	0.64
152 mm Hg	-	NI	17.0	-	NI	0.54
178 mm Hg	-	13.0	-	-	0.43	-
203 mm Hg	-	-	15.5	-	-	0.64
229 mm Hg	-	12.0	-	-	0.43	-
279 mm Hg	-	13.4	-	-	0.39	-
381 mm Hg	NI	-	-	NI	-	-
406 mm Hg	16.0	-	10.2	0.18	-	0.59
432 mm Hg	15.0	-	-	0.19	-	-
508 mm Hg	14.1	-	-	0.21	-	-
632 mm Hg	12.4	-	9.2	0.21	-	0.60

*NI denotes no ignition under test conditions.

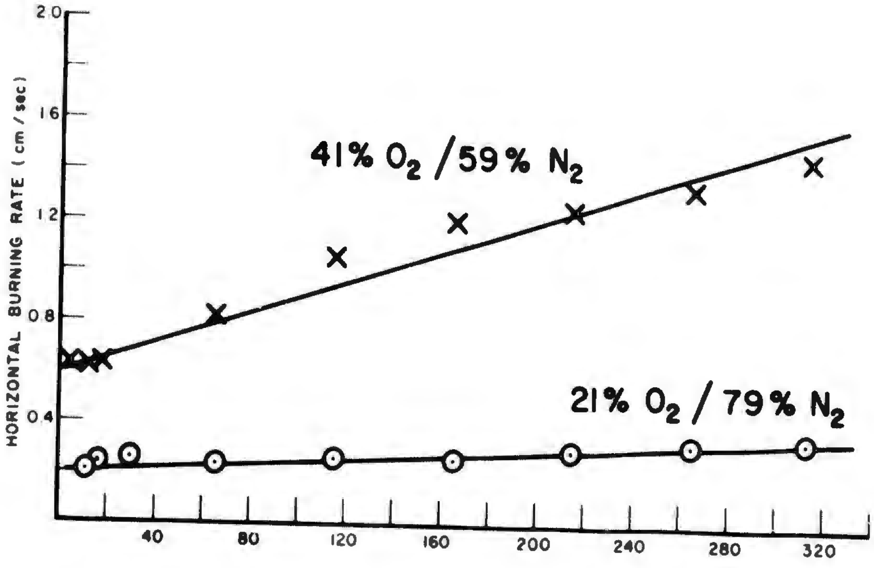

Fig. 5 - Effect of pressure and oxygen enrichment on burning rates of filter paper, FM-1, in oxygen-nitrogen mixtures

Figure 6 focuses on the burning rates given in Table 2 for the filter paper in oxygen-nitrogen mixtures at pressures less than 1 atmosphere. In all minimum pressure limit experiments the igniter energy was increased in an effort to extend the limit to the lowest pressure. Several effects are apparent from the curves in Fig. 6. A curved line, drawn to the left of the horizontal burning rate curves, denotes the minimum pressure limit for ignition of the impregnated filter paper under these conditions. As the O_2 was increased from 21% to 41%, the minimum pressure limit decreased markedly from 400 mm Hg to 100 mm Hg. At the same time, the partial pressure of O_2 at the minimum pressure limits decreased from 85 mm Hg at 21% O_2 to 42 mm Hg at 41% O_2. It is also of interest to note that the burning rate at a given percent of O_2 did not significantly decrease as it approached the minimum pressure limit until ignition failed altogether.

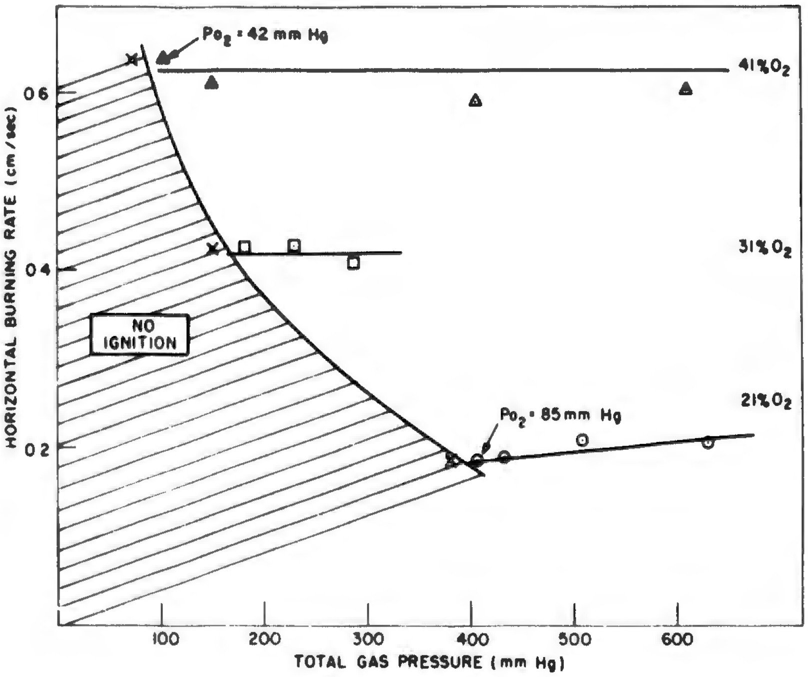

Fig. 6 - The effect of oxygen enrichment on minimum pressure limits for combustion of filter paper, FM-1, in oxygen-nitrogen mixtures

Table 3 and Figure 7 contain data in regard to the effect of total gas pressure on the flammability of filter paper (FM-1) in oxygen-helium mixtures. These data resemble, somewhat, the data for O_2/N_2 mixtures in Fig. 5. However, no significant change in the burning rate with pressure is apparent at 21% O_2. A pronounced increase in burning rate at 41% O_2 is noted, however, as the pressure is increased up to 300 psia.

Table 4 contains data which corroborate the previous report (6) that ignition occurs in air at higher pressures in materials which would not ignite at 1 atmosphere pressure. No literature reference was found in which other investigators report this effect.

The effects of total pressure on flame speed are somewhat variable and are related to the system under study. It had been reported previously (1,6,11) that increasing air pressures from about 15 to 75 psia caused a moderate increase in the flame speeds for flammable materials. The fact that gas mixtures with higher percentages of oxygen showed a more rapid increase in flame speed with pressure was observed in previous

Table 3
Effect of Pressure on the Flammability of Filter Paper in Oxygen/Helium Atmospheres

Total Pressure	Ignition Delay (sec)		Horizontal Burning Rate (cm/sec)	
	21% O_2	41% O_2	21% O_2	41% O_2
High-Pressure Data				
15 psia	NI*	12.6	NI	1.06
30 psia	NI	-	NI	-
45 psia	NI	-	-	-
55 psia	18.1	-	0.43	-
65 psia	-	9.8	-	1.2
75 psia	20.0	-	0.48	-
115 psia	21.0	5.4	0.48	1.41
165 psia	-	-	-	-
215 psia	15.5	8.5	0.45	1.36
265 psia	-	-	-	-
315 psia	14.2	6.9	0.41	1.70
Low-Pressure Data				
89 mm Hg	-	NI	-	NI
102 mm Hg	-	26.4	-	1.00
114 mm Hg	-	33.2	-	0.94
152 mm Hg	-	16.4	-	1.06

*NI denotes no ignition under test conditions.

Table 4
Flammability Resistance of Materials in Air at Pressures to 315 psia

Total Pressure (psia)	Flammability Observation			
	FM-4, Terry Cloth Treated With THPC	FM-14, O.D. Sateen Treated With THPC	FM-19, Verel	FM-9, Nomex
15	NI*	NI	NI	NI
65	NI	†	-	-
75	‡	-	-	-
115	‡	†	†	†
315	§	‡	†	†

*NI denotes no ignition under test conditions.
†Flamed over ignitor only.
‡Flamed momentarily; then smoldered length of cloth strip.
§Burned or charred one-half length test strip.
-Not tested.

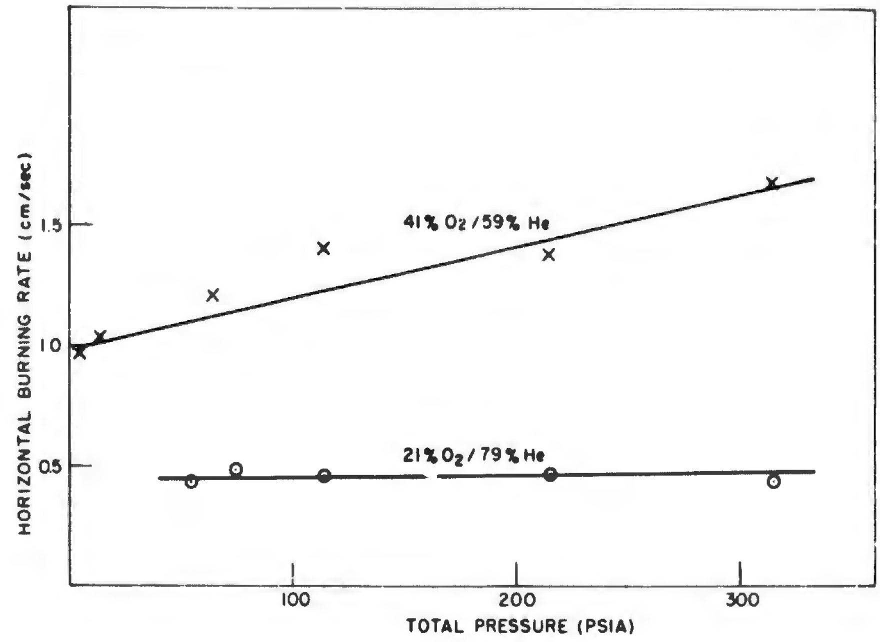

Fig. 7 - Effect of pressure and oxygen enrichment on burning rates of filter paper, FM-1, in oxygen-helium mixtures

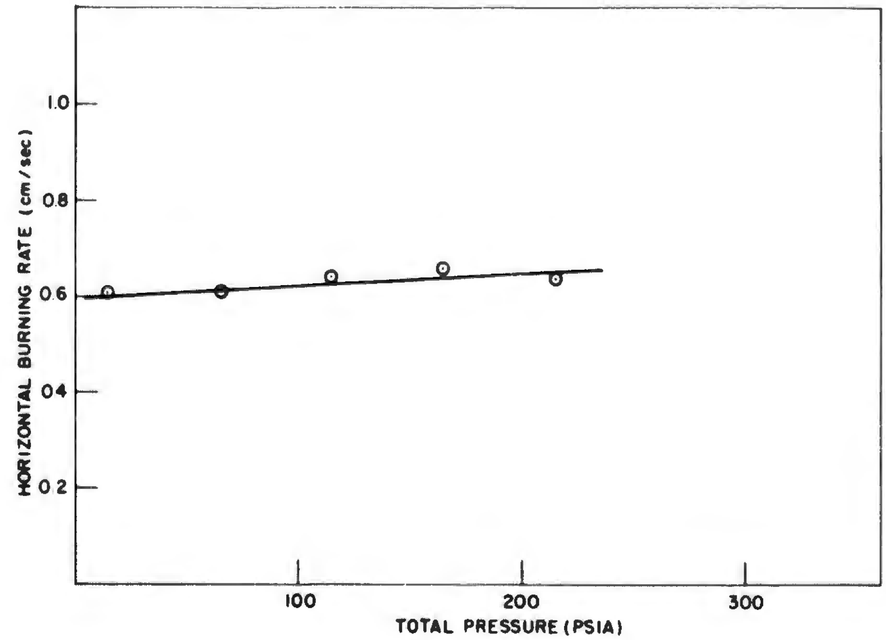

Fig. 8 - Effect of pressure on burning rates of filter paper, FM-1, in a mixture of 25.6% O_2, 10.4% N_2, and 64% He

work (6), but seems definitely established by the present work (Figs. 5 and 7). Huggett, et al. (10), concluded that "the rate of flame spread at constant atmospheric composition is approximately independent of pressure over the range studied (258 to 760 mm Hg)." This statement agrees very well with the results shown in Fig. 6 for data at subatmospheric pressures. It is also noteworthy that under some conditions, in mixtures containing helium and lower percentages of oxygen, flame speeds were essentially independent of pressures up to 200 to 315 psia (Figs. 7 and 8).

EFFECT OF HELIUM SUBSTITUTION FOR NITROGEN

It was observed in Fig. 7 that with 21% O_2 in helium the burning rate of filter paper was essentially constant from 15 to 315 psia, in contrast to 21% O_2 in nitrogen, where the burning rate increased by 50% as pressure was increased to 315 psia as shown in Fig. 5. Another example of this constancy of burning rate is shown in Fig. 8, where filter paper had a horizontal burning rate of 0.63 ± 0.03 cm/sec over the pressure range of 15 to 200 psia. These data were taken from Table 5. This phenomenon has been observed, thus far, only in mixtures predominant in helium with low (21–25%) oxygen. Nitrogen mixtures under the same conditions have shown a moderate, but significant, increase in flame speed with pressure increases of 75 to 315 psia. At 41% O_2, burning rates increased significantly with pressure in both helium and nitrogen as shown in Fig. 9.

Table 5
Effect of Pressure on the Flammability of Filter Paper,
FM-1, in Oxygen/Nitrogen/Helium Atmospheres

Total Pressure	25.6% O_2, 10.4% N_2, 64% He		31% O_2, 34.5% N_2, 34.5% He	
	Ignition Delay (sec)	Horizontal Burn Rate (cm/sec)	Ignition Delay (sec)	Horizontal Burn Rate (cm/sec)
High-Pressure Data				
15 psia	13.8	0.61	-	-
65 psia	9.0	0.61	7.3	0.66
115 psia	6.8	0.64	4.3	0.77
165 psia	6.6	0.66	-	-
215 psia	9.9	0.63	-	-
Low-Pressure Data				
305 mm Hg	-	-	15.6	0.57
381 mm Hg	-	-	14.3	0.57

It had been observed earlier (6) that most of the materials had a decidedly greater burning rate in oxygen-helium mixtures than in oxygen-nitrogen mixtures. However, some exceptions to this generalization were seen in the present data. In Fig. 9 the burning rate of filter paper is significantly higher in 41% O_2/He than in 41% O_2/N_2. However, in Fig. 10 the data show that in 41% O_2 mixtures a temperature-resistant nylon (Nomex, FM-9) burns much faster at higher pressures with nitrogen as the diluent as compared to helium. The data in Fig. 10 were taken from Table 6. In Table 6 it is observed that the ignition delays with a standard igniter energy are much longer in helium than in nitrogen mixtures, which is to be expected from earlier work (6), in which this result was shown to be due largely to the high heat conductivity of helium.

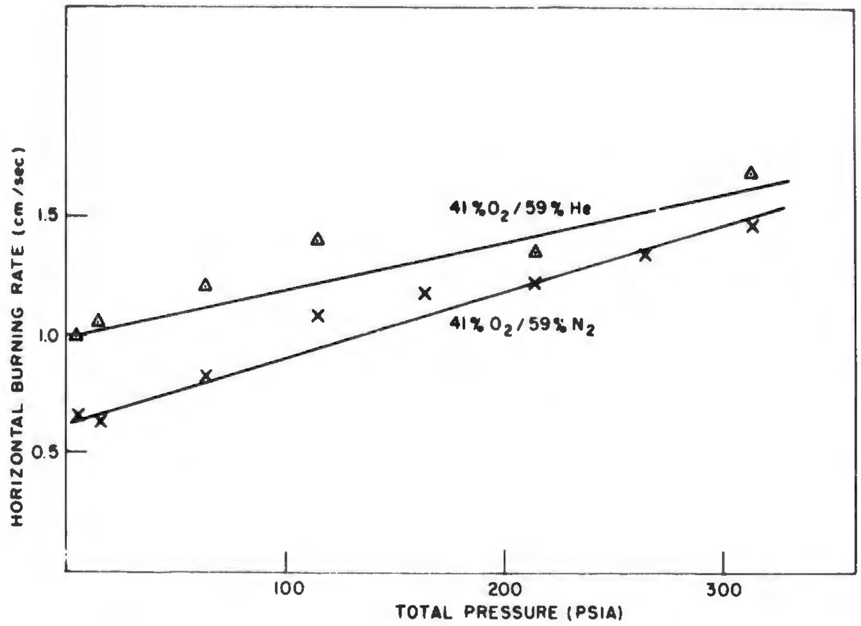

Fig. 9 - The effect of pressure and oxygen enrichment on burning rates of filter paper, FM-1, in oxygen-nitrogen and oxygen-helium mixtures

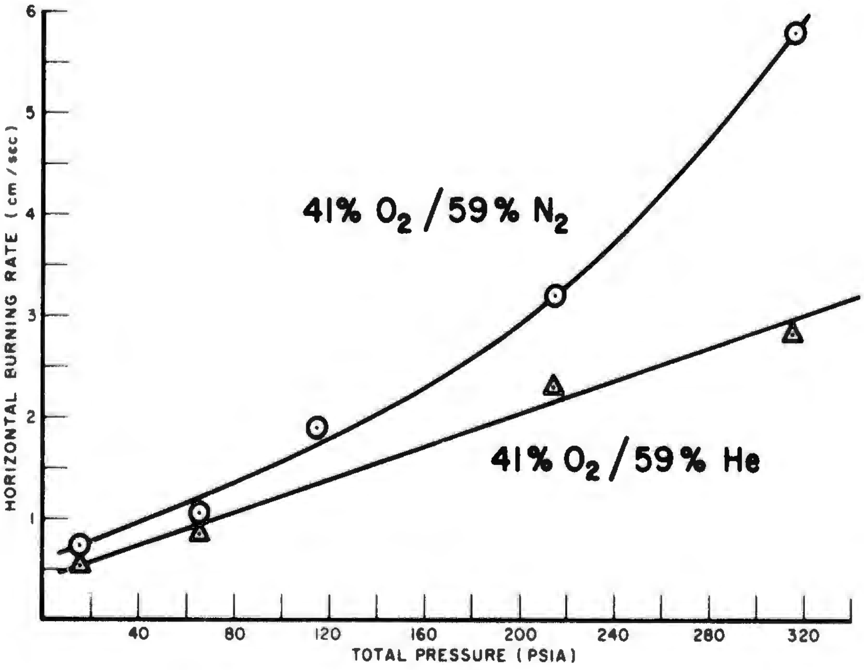

Fig. 10 - The effect of pressure on the burning rates of high-temperature-resistant nylon (Nomex), FM-9, in nitrogen-oxygen and helium-oxygen atmospheres

Table 6
Combustion of High Temperature Nylon (Nomex) in Mixtures Containing 41% Oxygen

Total Pressure (psia)	Warp Direction				Fill Direction			
	41% O_2/N_2		41% O_2/He		41% O_2/N_2		41% O_2/He	
	Ignition Delay (sec)	Burn Rate (cm/sec)	Ignition Delay (sec)	Burn Rate (cm/sec)	Ignition Delay (sec)	Burn Rate (cm/sec)	Ignition Delay (sec)	Burn Rate (cm/sec)
15	10.0	0.64	22.4	0.54	12.0	0.52	24.6	0.48
65	5.1	1.02	24.0	0.89	-	-	22.2	0.79
115	5.8	1.87	18.0	1.76	6.8	1.43	15.8	1.04
215	5.0	3.2	15.0	2.31	-	-	-	-
315	4.2	5.77	14.9	2.82	-	-	8.9	1.58

Only two instances of the effect of helium versus nitrogen as the inert gas in the flammability of solid materials have been found (10,12) other than that from this Laboratory (6). Klein (12) concluded that the burning rate of cotton cloth in atmospheres of oxygen and helium differs very little from atmospheres of oxygen and nitrogen at pressures of 0.5 atm. or less. Huggett, et al. (10), concluded that nitrogen is more effective than helium in reducing flame spread rate (at 760 mm Hg and less). In our earlier work (6) it was observed that "most of the materials tested had a decidedly greater burning rate in the helium mixtures than in nitrogen mixtures." In the present work this statement can still be made (Fig. 9), but in one important case shown in Fig. 10, high temperature nylon (FM-9) burned much faster in 41% O_2/N_2 than in 41% O_2/He. This case is discussed in more detail in the next section of this report.

EFFECT OF THE NATURE OF THE MATERIAL

The first report of this series (6) clearly indicated that materials vary in their degree of flammability — from highly flammable (easily ignitable and fast burning) to nonflammable under the test conditions. However, the effect of variables, such as pressure and diluent, on flammability is influenced by the nature of the material being studied. The following studies will confirm these facts.

A temperature-resistant nylon (FM-9) was studied in 41% O_2 mixtures in both nitrogen and helium up to total pressures of 315 psia. The data are given in Table 6 and a comparative plot in Fig. 10 shows increased burning rates for Nomex nylon in 41% O_2/N_2 over helium. In contrast with this data Fig. 6 shows that filter paper burned faster in 41% O_2/He than in 41% O_2/N_2.

In Fig. 11 a plot is made comparing rates for Nomex and the filter paper (FM-1) in 41% O_2/N_2. This graph shows that Nomex burned much faster than filter paper in this atmosphere at higher pressures. For example, at 315 psia Nomex burned about four times as fast as the filter paper. The significance of this difference is underscored by the fact that Nomex would not burn in ordinary air (21% O_2) even at 315 psia, whereas the filter paper ignited readily in air at pressures as low as 8 psia. The differences in pyrolysis rates between Nomex nylon and paper under various conditions probably account for their different flammability behavior. Plans have been made to study this phase of the work in more detail.

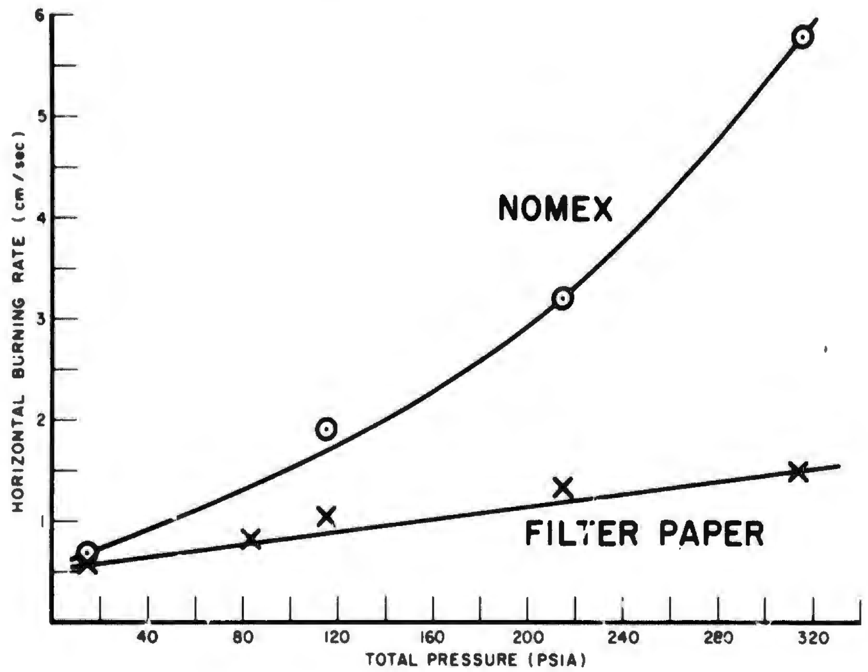

Fig. 11 - Effect of pressure on burning rates of high-temperature-resistant nylon (Nomex), FM-9, and filter paper, FM-1, in 41% O_2 - 59% N_2 atmosphere

Data on the minimum pressure limits for ignition of filter paper and Nomex nylon are given in Table 7. The filter paper ignited at total pressures of the gas mixture considerably lower than required for ignition of Nomex. It is to be noted, also, that the minimum ignition pressures were lower for nitrogen-oxygen than helium-oxygen.

Table 7
Minimum Pressure Limits for Ignition

NRL Sample Number	Material	Minimum Pressure for Ignition (mmHg)	
		41% O_2/59% N_2	41% O_2/59% He
FM-1	Filter Paper	100	200
FM-9	Nomex Nylon	230	460

It should also be pointed out that, for certain materials, the preparation of the sample in terms of direction of flame spread was important. This is illustrated in Table 6, in that the Nomex strips in the "fill direction" cut were significantly slower burning than the strips in the "warp direction," cut in both O_2/N_2 and O_2/He mixtures. Therefore, it was important, for comparative results, that the materials being examined be cut and mounted the same way.

In the early quantitative work on the effects of increased oxygen concentration by Coleman (11) in 1959, the best cotton flame retardant he used was a mixture of 30% boric acid and 70% borax. He found that even this treatment was ineffective in preventing inflammation above 32% oxygen at 1 atmosphere. However, it was of interest to compare this inorganic treatment and the treatment with THPC (tetrakis hydroxymethyl phosphonium chloride) reported previously (6). The data obtained with cotton terry cloth impregnated with the boric acid/borax (B/B) mixture is given in Table 8. It was found that even the unrealistic high loading of 28.2% retardant was insufficient to protect the cotton cloth in the vertical position at 41% oxygen. At 31% oxygen 25.4% B/B could not prevent the cloth strip from charring for its entire length, although it did not burst into flame. It was concluded that these results essentially corroborated the work of Coleman (11). Also, the data in Table 8 emphasize, once again, the much more severe stress imposed by the vertical over the horizontal position of the cloth strip.

Table 8
Flammability of Boric Acid/Borax-Treated Cotton Terry Cloth
and THPC-Treated Terry Cloth at 1 Atmosphere Pressure

Atmosphere	Treatment[*]	Horizontal Position		Vertical Position	
		Ignition Delay (sec)	Burn Rate (cm/sec)	Ignition Delay (sec)	Burn Rate (cm/sec)
O_2/N_2:21/79	THPC	NI[†]	NI	NI	NI
	B/B, 10.6%	NI	NI	NI	NI
O_2/N_2:31/69	THPC	18	§	9.6[‡]	1.37[‡]
	B/B, 10.6%	-	-	13.8	0.56
	B/B, 15.2%	-	-	11.4	0.30
	B/B, 25.4%	NI	NI	§	§
O_2/N_2:41/59	THPC	5.4	1.31	11.7	1.7
	B/B, 10.1%	-	-	10.4	0.69
	B/B, 15.3%	-	-	10.2	0.57
	B/B, 22.0%	§	§	-	-
	B/B, 23.3%	-	-	13.7	0.46
	B/B, 28.2%	-	-	14.0	0.38

[*] B/B % denotes percent by weight of 70% borax/30% boric acid added to the cloth.
[†] NI denotes no ignition under the test conditions.
[‡] Variable results, some strips charred about one-half length of test strip.
[§] Did not flame, but smoldered for entire length of test strip.

SUMMARY

In reporting the experimental results of the present work on the flammability of materials, it must be realized that some of the data are limited and should be applied to the evaluation of flammable hazards with caution, pending further verification and study. It is believed, however, that the lack of information in the literature on this subject, which is of great current interest, warrants the present report.

The increase in pressure of air to 315 psia caused a moderate increase in the flame speed of filter paper. Gas mixtures containing higher percentages of oxygen exhibited greater percentage increases in flame speed with increased pressure. In some mixtures containing helium and lower percentages of O_2, flame speeds were essentially independent of pressure up to as much as 315 psia. Minimum pressure limits were obtained for the flammability of filter paper in O_2/N_2 mixtures containing 21% to 41% O_2. Not only was the minimum total pressure limit much lower at 41% O_2, but the partial pressure of O_2 at these limits decreased from 85 mm Hg to 42 mm Hg at 41% O_2.

As reported earlier (6) it appears that materials usually have a faster burning rate in helium mixtures than in nitrogen mixtures. However, in this present work an important exception to this general statement was observed. High-temperature-resistant nylon burned much faster in 41% O_2/N_2 than in 41% O_2/He at higher pressures. At 315 psia the burning rate in O_2/N_2 was 5.8 cm/sec versus 2.8 cm/sec in O_2/He. It is apparent that the nature of the material can be an overriding factor in determining rate of burning.

It is important to note, also, that the high-temperature-resistant nylon burned four times as fast as the standard filter paper in 41% O_2/N_2 at 315 psia. This faster burning of the nylon was in spite of the fact that the nylon did not burn at 21% O_2 even at 315 psia, while the filter paper burned readily in air at pressures below 1 atmosphere.

Some studies were made of the flammability of cotton terry cloth impregnated with a mixture of borax and boric acid. Even high loading of this flame retardant mixture was ineffective at high concentrations of oxygen (31% to 41%).

CONCLUDING REMARKS

This extension of work on the flammability of materials at this Laboratory to include previously unexplored atmospheric conditions shows, even more strongly, the dangers of making broad generalizations from limited data. Although it is expected that the information in this report will be useful, it must be stated again that, at this stage, no simple multiplication factors can be used in predicting the flammability of materials in these artificial atmospheres. No extrapolation to other atmospheric conditions seems justified without experimental verification. Before any materials can be judged safe for a given application or the degree of hazard estimated, all the pertinent conditions of use must be examined experimentally.

The present work is being extended to provide minimum ignition pressure data for other materials at different percentages of oxygen and diluents. Further studies into the effect of the nature of materials on combustion in unusual atmospheres are planned.

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Appendix A

DESCRIPTION OF MATERIALS TESTED

FM-1	*Resin-impregnated paper*. This nominal 5-micron-pore-size filter paper was obtained from the Skinner Purifier Division of the Bendix Aviation Corporation. It meets MIL-F-5504B specifications, "Filters and Filter Elements, Fluid Pressure, Hydraulic, Micronic Type." The paper contained no hydrocarbons which are normally used in specification tests.
FM-3	*Cotton terry cloth*. This cotton terry cloth was cut from a robe supplied by the Navy Experimental Diving Unit (NEDU), Navy Yard, Washington, D.C.
FM-4	*Cotton terry cloth, fire-resistant*. This cloth was treated for fire resistance by impregnating with a chemical, tetrakis (hydroxymethyl) phosphonium chloride (THPC) and was supplied by NEDU.
FM-9	*High-temperature-resistant nylon (Nomex)*. This high-temperature-resistant nylon, 3.5 oz/yd^2 (manufactured by the Dupont Company), was supplied by NEDU.
FM-14	*Cotton O.D. Sateen, fire-resistant*. This cloth was treated for fire resistance by impregnating with a chemical, tetrakis (hydroxymethyl) phosphonium chloride (THPC), and was supplied by the Hooker Chemical Corporation, Niagara Falls, New York.
FM-19	*Verel fabric*. This fire-resistant fabric, 5.0 oz/yd^2, made of Eastman modified acrylic staple fibers, was obtained from the Southern Regional Research Laboratory, USDA, New Orleans, Louisiana.
FM-28	*Cotton cloth, white duck*. This 7.0 oz/yd^2 duck was cut from a laboratory coat manufactured by the Fisher Scientific Company and obtained at NRL.

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13. ABSTRACT

The Naval Research Laboratory has been investigating the flammability of materials under unusual atmospheric conditions. Ignition and flame spread of several fabrics and paper were measured at pressures from 315 psia down to the limiting pressures for ignition. The minimum total pressure limit for the standard filter paper used was much lower at 41% O_2 than at 21% O_2 in O_2/N_2 mixtures. Also, the partial pressure of O_2 required for ignition at 41% was much lower than at 21% O_2. Although, in general, materials have a faster burning rate in helium mixtures than in nitrogen mixtures, there are some significant exceptions to this finding. The nature of the material has been shown to have a marked influence on the effect that variables, such as pressure, oxygen content, and diluent, have on the rate of burning.

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