Flammability in Unusual Atmospheres

Part 1 - Preliminary Studies of Materials in Hyperbaric Atmospheres Containing Oxygen, Nitrogen, and/or Helium

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CONTENTS

Abstract Problem Status Authorization	ii ii ii
INTRODUCTION	1
LITERATURE SUMMARY	2
Effect of Oxygen Enrichment Effect of Increased Air Pressure Effect of Helium Substitution for Nitrogen	2 3 3
DEVELOPMENT OF APPARATUS AND EXPERIMENTAL PROCEDURES	3
Exposure Chambers Apparatus for Ignition and Flame Speed Studies Gas Mixtures Materials Used Preparation and Treatment of Sample Position and Support of the Sample	4 5 6 7 9
GENERAL EXPERIMENTAL PROCEDURE	9
EFFECTS OF ATMOSPHERIC VARIABLES ON FLAMMABILITY	9
Effect of Oxygen Enrichment Effect of Increased Pressure Effect of Substitution of Helium for Nitrogen	10 12 12
EFFECT OF ATMOSPHERIC COMPOSITION ON IGNITER TEMPERATURE	12
FLAMMABILITY OF OIL-SOAKED PAPER	14
FLAME RESISTANCE OF SELECTED MATERIALS	15
SUMMARY	15
CONCLUSIONS	16
REFERENCES	17
APPENDIX A - Description of Materials Tested	18

ABSTRACT

A study of the flammability of fabrics and other solids under unusual atmospheric conditions has been initiated. The most profound effect on both ease of ignition and linear burning rate was caused by oxygen enrichment. For example, many materials which did not ignite in 21% oxygen ignited and burned readily at 31% or 41% oxygen. With a given atmosphere, increase in pressure was often effective in causing ignition where no ignition occurred at lower pressures. Substitution of helium for nitrogen in mixtures with oxygen had two generally significant effects. Helium decreased the tendency of a material to ignite. This effect was shown to be due largely to the high thermal conductivity of helium. Once ignited, burning rates were often much faster in helium than nitrogen.

PROBLEM STATUS

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

AUTHORIZATION

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FLAMMABILITY IN UNUSUAL ATMOSPHERES

PART 1 - PRELIMINARY STUDIES OF MATERIALS IN HYBERBARIC ATMOSPHERES CONTAINING OXYGEN, NITROGEN, AND/OR HELIUM

INTRODUCTION

In February 1965, a disastrous fire occurred in a decompression chamber at the Navy Experimental Diving Unit (NEDU) killing the two occupants (1). To help in determining the cause of the fire, special analyses and other assistance were contributed by the Chemistry Division of NRL (2). Since then, the Fuels Branch of NRL has cooperated with NEDU in certain aspects of planning chamber safety, including the selection of materials for clothing, mattresses, and other items. In this regard, it was soon found that there was a decided lack of the quantitative information needed for understanding ignition and combustion of materials in unusual (nonterrestrial) atmospheres, especially at superatmospheric (hyperbaric) pressures. This lack of knowledge is important because of the increasing number of activities of the Navy and other interests, including hyperbaric medicine, which are exposing men and materials to atmospheres at higher pressures, often enriched with oxygen. Consequently, a study was initiated at this Laboratory to examine experimentally the ignition and combustion of materials in these unusual environments.

The source of ignition in the decompression chamber fire was believed to be an overheated motor (1). This motor was in a carbon dioxide scrubber which had a resinimpregnated fluted paper filter on the outlet side. The paper filter (Spec. MIL-F-5504B) which was adopted for use with this scrubber is used ordinarily for fuel and hydraulic fluid filtration in jet aircraft. Each element is tested in such a way that residual hydrocarbon fluid remains on the paper when packaged. Although it had seen many hours of previous use, this paper filter in this instance is believed to have been ignited by proximity to the hot electric motor.

In the NEDU decompression chamber fire, witnesses reported that fierce, extremely rapid burning of materials in the chamber caused a very fast pressure rise; nearly 100 psi in perhaps 15-30 seconds. To reach the maximum pressure observed, calculations show that a temperature increase of about 400°C must have occurred. This heat could have been furnished by the complete burning of as little as 500 grams of fuel, calculated as cellulose.

The composition of the atmosphere in the chamber at the onset of the fire was stated to be about 28% oxygen, 36% nitrogen, and 36% helium, at a chamber pressure of 55 psia. Although such an environment might not seem to be unduly hazardous, the disastrous results proved that atmospheric conditions in the chamber were conducive to extremely fast burning rates. The major ways in which this system differs from ordinary air at one atmosphere pressure in regard to flammability effects are: (a) enriched oxygen percentage, (b) increased pressure, and (c) partial substitution of helium for nitrogen. Therefore, emphasis was placed on learning the influence of these three atmospheric variables on the ignition and combustion of materials such as fabrics, films, and other solids. Experimentally, conditions were chosen to permit the separation of these three factors in terms of effectiveness. In addition, a survey was made of a number of different materials under conditions selected to show their relative flame resistance and, from this, their suitability for use in pressure chambers.

LITERATURE SUMMARY

A search of published literature yielded only limited information on the effects of the pertinent atmospheric factors on flammability of materials. Although a number of recent papers and reports discuss the effect of enrichment of oxygen on combustion of solids, most of them are related to the manned spacecraft effort, and since Mercury, Gemini, and Apollo craft have all been committed to 100% oxygen at 258 mm Hg, almost all of these references treat the subject at total pressures of less than one atmosphere.

Only one reference (3) was found of work done in enriched oxygen at pressures greater than one atmosphere. Only one reference (4) was found involving air at higher pressures. In only one instance (5) was helium used as a diluent gas, and this only at reduced pressures.

It should be emphasized that the present discussion does not apply to the homogeneous or heterogeneous combustion of gases and vapors. Nor does it include heterogeneous combustion of liquids, such as hydraulic fluids as films or sprays. A considerable literature has been built up concerning combustion of such vapors and liquids. Although this information has not been ignored, the following discussion is limited to the ignition and combustion of solids, particularly in the form of fabrics and thin sheets.

Effect of Oxygen Enrichment

The first quantitative work on the effects of increased oxygen concentration on flammability of fabrics appears to be that published in 1959 by Coleman (4). All work was done at one atmosphere pressure. The types of fabric materials included in this study were wool, wool-Terylene mixture, cotton, P.V.C.-coated cotton, and cotton fabrics which had been treated with various inorganic flame retardants. The principal findings were the following:

1. Although wool fabrics were difficult to ignite in air and only partially burned, slight increases in oxygen content (e.g., to 26%) caused complete combustion.

2. Likewise, P.V.C.-coated cotton did not burn in air but was consumed at 26% oxygen.

3. There were limits of oxygen concentration beyond which flame retardants were ineffective. For example, the best cotton flame-retardant tried (30% boric acid/70% borax) was not effective above 32% oxygen, regardless of loading.

Klein (5) studied the effect of oxygen concentration on the burning rate of a standard cotton fabric in oxygen-nitrogen mixtures at pressures of one atmosphere and lower. The burning rate was increased greatly as the oxygen/nitrogen ratio was increased at any given pressure. At a given oxygen/nitrogen ratio, an increase in total pressure increased the burning rate. This effect of pressure was enhanced at higher oxygen percentages.

Hall and Fang (6) found that paper ignited much more readily and burned 6 times as fast in 5-psia oxygen as in air at one atmosphere pressure. Many materials that did not burn in air burned readily in oxygen at 5 psia. Chianta and Stoll (7,8) showed that oxygen enrichment at reduced pressures had a profound effect on the ease of ignition and rate of burning of cotton and polyamide (Nomex) fabric. Roth (9) has summarized other available information on oxygen enrichment at reduced pressures. Huggett, et al. (10) recently published a comprehensive report on the ignition and combustion of a large number of materials after prolonged exposure in 100% oxygen at 258 mm Hg pressure. The enhanced burning rate compared to air was reaffirmed. No major differences were found in the

minimum ignition energy required in air, in oxygen, or after "soaking" in oxygen for as long as 30 days. The ignition delay time and rate of burning were profoundly affected, however.

Denison, Ernsting, and Cresswell (11) recently published a dramatic report on the possible fire risks to man in oxygen-rich atmospheres. They concluded that, in oxygen, the hazards are increased by a roughly "one thousandfold rise" in ignitability and a five-fold increase in burning rate. Most conventional fire-proofing agents were ineffective as was smothering as a means of extinction. Only an instantaneous water spray was able to control such a fire successfully. In simulated clothed-man experiments in oxygen atmospheres using pigs and mannikins, they observed the following:

1. A flashover flame instantly covered the pig and clothing, singeing the pig and causing the clothing to ignite on all edges.

2. The flashover could propagate on the pig's skin beneath one or two layers of flame-proofed material.

3. The flashover did not occur on flame-proofed denim (boric acid/borax).

4. Dropping the partial pressure of oxygen to 0.5 atm just prevented flashover in most materials and markedly slowed the spread of the main fire.

5. Igniting a clothed man in an oxygen atmosphere may lead to fatal damage within 5 to 20 seconds of onset of the fire.

Effect of Increased Air Pressure

Very limited data were found which concerned the effect of enriched oxygen atmospheres at greater than one atmosphere pressure on the flammability of materials. The flammability of cosmetic and hair preparations in the presence of 100% oxygen at one and 2 atmospheres pressure was studied to assay the hazard of using oxygen breathing masks in aviation (3). It was concluded that such preparations are inadvisable for use in oxygen. Another reference (4) discusses the results of experimentation with air pressures up to 75 psig. These experiments showed that "doubling the oxygen concentration from 21 to 42% by enrichment has produced a much greater effect (on burning rate) than increasing the oxygen by doubling the air pressure to 2 atmospheres." Increasing air pressure from one to 6 atmospheres lowered the fabric burning time from about 14 sec to 8 sec.

Effect of Helium Substitution for Nitrogen

Only one instance of the effect of helium versus nitrogen as the inert gas in flammability of fabrics was found (5). The author concluded that the burning rate of cotton cloth in atmospheres of oxygen and helium differs very little from that in atmospheres of oxygen and nitrogen. These data were taken at pressures of about 0.5 atmosphere or less.

DEVELOPMENT OF APPARATUS AND EXPERIMENTAL PROCEDURES

In designing the methods of evaluation to be used to determine the fire resistance and burning rates of fabrics and other materials in the present work, many experimental factors were considered. A large number of methods are used in commerce, and many are accepted as standard methods by such technical societies as the American Society for



Fig. 1 - Small oven used in flammability studies

Testing Materials and the American Association of Textile Chemists and Colorists and by government agencies for materials specifications. Practically all of these methods are based on exposures in a confined space to an ignition source in ordinary air at ambient pressure. Consequently, none of these standard methods was acceptable without modification. The general apparatus and procedures developed for the present work are described below.

Exposure Chambers

The nature of the data required from these studies made it necessary to perform the experiments in closed chambers which could be evacuated and/or pressurized. In experiments of this type, it is important that the burning of the sample not significantly change the composition of the confined atmosphere either by depletion of oxygen or dilution by the gaseous combustion products. Although this factor suggests that the vessel must not be too small, excessive size was also not desirable because of the need to use various gas mixtures. The amounts of these gases used will be greatly increased at high pressures. In the smaller of two chambers used in the present work (12.6 liters), consecutive 1-inch burning rates on the same strips showed no evidence of change. Since these tests were made at one atmosphere in the smaller volume, they were the most liable to any effect due to change in the composition of the atmosphere. These results confirmed the calculated expectations.

<u>Atmospheric Pressure Chamber</u> – For convenience, most of the experiments at one 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 laboratory fume hood.

<u>Hyperbaric Pressure Chamber</u> – For experiments at increased pressure, a chamber obtained from NEDU was used. This chamber, shown in Fig. 2, had a volume of 142 liters and a maximum working pressure of 75 psia. The chamber was equipped with suitable valves and pressure tubing to permit flushing and pressurizing the chamber with the



Fig. 2 - Chamber used for pressure experiments (from NEDU)

desired gas mixture in preparation for each experiment, and subsequent release of the gas pressure. This chamber was not equipped for evacuation, so that flushing was needed to set up the desired atmospheres. Gas analyses were made on the chamber contents before each run to ensure that the atmospheres were correct. All experiments with this chamber were performed in a safety cell, with the operator protected by a suitable barricade.

Apparatus for Ignition and Flame Speed Studies

Because of the need to perform the experiments in sealed chambers, an electrical method of ignition using 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 is pictured in front of the oven in Fig. 1. Another view is shown in Fig. 3. A clamp holder for the specimen and a Nichrome wire heater were mounted on a block of transite. The Nichrome heater was made of 25-gauge wire wound on a $1/4 \times 7/8$ inch form, and was operated at 18 volts and 4.0 amperes for most experiments. Three thermocouples were spaced above the specimen, which was held between the clamp and the igniter. The iron-constantan thermocouples,



Fig. 3 - View of igniter apparatus

spaced 1 inch apart, provided a convenient means of timing flame speeds. A similar holder was used for vertically mounted specimens. Figure 4 is a schematic diagram of the combustion system of the chamber shown in Fig. 1.

Gas Mixtures

Five principal gas mixture compositions were used in this study:

(a)	O_2/N_2	:	21/79
(b)	O ₂ /He	:	21/79
(c)	O_2/N_2	:	31/69
(d)	O ₂ /He	:	31/69
(e)	$O_2/N_2/He$:	31/34.5/34.5

The numerical ratios refer to percents by volume. All the mixtures except (a) (ordinary air) were prepared by NEDU and delivered to NRL in large gas cylinders. 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. This paper



Fig. 4 - Schematic diagram of the combustion apparatus

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 (see Table 1 and Table 2). These were principally a cotton terry cloth, FM-3, and a similar cloth treated with a commercial fire retardant, FM-4. This cloth was impregnated with a chemical, tetrakis (hydroxymethyl) phosphonium chloride (THPC), which has the structural formula (HOCH₂)₄PC1.

Preparation and Treatment of Sample

It was anticipated that the previous treatment of the sample might influence the results. However, several experiments showed no measurable difference in flame speeds for filter paper strips which had been exposed for 24 hours to relative humidities (R.H.) of 50% and 100%. However, it is likely that materials such as cotton fabrics would be affected. In most instances specimens were used after exposure to approximately 50% R.H., the more severe condition. Several experiments were designed to detect any differences in ignition or burning rate which might be caused by the length of time of exposure of the test specimen to the test atmosphere. No differences were detected. In this connection, it was comforting to note that Huggett et al. (10) observed no significant

		Horizonta	al Position	Vertical Position	
Atmosphere	Pressure (psia)	Ign. Delay (sec)	Burn Rate (cm/sec)	Ign. Delay (sec)	Burn Rate (cm/sec)
0 /1: .01/70	15	NITE	NI	NT	
O ₂ /N ₂ :21/79	15	NI*	NI	NI	NI
	30	NI	NI	9.6	0.101
	45	NI	NI	7.2	0.13
	60	NI	NI	6.6	0.31‡
	75	5.0	0.031	-	-
O ₂ /He:21/79	75	NI	NI	t	t
O ₂ /N ₂ : 31/69	15	18.0	S	t	†
•	30	7.2	0.23	6.3	2.61
O2/He:31/69	15	NI	NI	- I	-
-	30	NI	NI	-	-
	60	NI	NI	-	-
	75	ş	0.05	5	ŝ
O ₂ /N ₂ /He:					
31/34.5/34.5	15	NI	NI	- 1	-
	30	NI	NI	S	S
	45	9.0	0.38	7,2	2,54

 Table 1

 Combustion of THPC-Treated Terry Cloth

 When Held Horizontally or Vertically

*NI denotes no ignition under the test conditions. †Burned or charred about one-half length of test strip. II lamed momentarily, then smoldered for entire length of cloth strip. §Did not flame, but smoldered for entire length of cloth strip.

	- Station - St	Paj	per	Terry	Cloth	Treated Te	erry Cloth*
Atmosphere	Pressure (psia)	Ign. Delay (sec)	Burn Rate (cm/sec)	Ign. Delay (sec)	Burn Rate (cm/sec)	Ign. Delay (sec)	Burn Rate (cm/sec)
O ₂ /N ₂ 21/79	15	6.5	0.23	8.4	0.20	NIţ	NI
	30	6.0	0.25	6.6	0.61	NI	NI
	45	7.2	0.30		•	NI	NI
	60	6.0	0.30	4.8	1.17	NI	NI
	75	4.8	0.28	4.9	0.89	5.0	0.03 t
O,/He:31/79	15	NI	NI	-	-	_	-
	30	NI	NI	1	-	-	-
	45	NI	NI	-		-	-
	60	NI	NI	-	-	-	-
	75	18.0	0.48	18.0	0.40	NI	NI
O, /N,:31/69	15	6.0	0.43				
0, 11,01/00	30	5.7	0.47				
	45	6.0	0.45				
	60	4.8	0.53				
	75	5.4	0.53				1
O ₂ He:31/69	15	17.0	0.76	22.5	1.27	NI	NI
-1	30	14.3	0.69	27.0	1.02	NI	NI
	45	8.1	0.70	_	-	NI	NI
	60	7.5	0.76	-	-	NI	NI
	75	7.5	0.86	-	-	\$	0.05
O, /N, /He:	1.						
O ₂ /N ₂ /He: 31/34.5/34.5	15	10.2	0.61	10.8	0.94	NI	NI
	30	11.1	0.67	10.2	1.70	NI	NI
	45	8.4	0.56		-	9.0	0.38
	60	6.6	0.56	-	-	-	-
	75	5.4	0.68	-	-	-	-

Table 2
Combustion of the Standard Paper, a Terry Cloth, and a
Terry Cloth Treated With a Fire Retardant

*See the first four columns of Table 1. †NI denotes no ignition under the test conditions, tFlamed momentarily, then sinoldered for entire length of cloth strip. \$Did not flame, but smoldered for entire length of cloth strip.

enhancement in ignition or flame speed in oxygen after soaking for 30 days. These data indicate that equilibrium is established very quickly between the atmosphere and the surface of the specimen. Consequently, no extended soaking period was used in most of our experiments.

It was established that, with the present method of ignition, the width of the test strip had little effect on ignition or on burning rate. Table 3 contains data of this sort. Most subsequent runs were made using 1/4-inch strips for convenience. The strips used were 4 inches long to simplify the measurement of burning rates. It was established by weighing test strips that the variation in density of the standard filter paper was not significant in its influence on burning rate.

Table 3
Burning Rates of Filter Paper Strips
(FM-1) in Air at One Atmosphere
Pressure

Strip Size	Burning Rate (cm/sec)			
(in.)	Horizontal	Vertical		
$1/2 \times 4$	0.20	-		
	0.22	-		
	0.22	-		
1/4 × 4	0.22	1.7		
	C.23	1.8		
	0.23	1.7		

Position and Support of the Sample

It is known from practical experience that a vertical sample position with the ignition source applied at the bottom is more conducive to ignition and flame propagation. A rather comprehensive study of the flammability of materials at one atmosphere of air showed that the vertical burning rate was on the order of 10 times faster than the horizontal rate (12). The data in Table 3 also bear this out. In addition, the data in Table 1 on the flammability of cotton cloth which has been treated for fire resistance show that both ignitability and burning rate tend to increase substantially in a vertical position. For this reason the determinations of fire resistance (ignitability) for a variety of materials were made in the vertical position (see Table 4). However, most comparative measurements were made in the horizontal position because it was often very difficult to measure the speed of propagation of the flame in the vertical position. The more moderate burning rates obtained horizontally (Table 3) permitted more meaningful study of the effects of other factors.

GENERAL EXPERIMENTAL PROCEDURE

The procedure for a typical experiment was as follows: 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. This apparatus was placed in the chamber, the latter was evacuated to about 30 inches Hg, and the desired atmosphere admitted. This evacuation and fill procedure was repeated several times to ensure that no dilution of the gas mixture had occurred. 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. Ignition delay was measured visually using a stopwatch, and flame speed was measured either visually with a stopwatch or by examining a strip chart record of the response of the parallel thermocouples.

EFFECTS OF ATMOSPHERIC VARIABLES ON FLAMMABILITY

Experiments were designed to permit the determination of the effect of the following factors on ease of ignition and burning rates: oxygen enrichment, increased pressure,

NRL Sample Material		Combustion in O_2/N_2 Mixtures		
Number			314 O2	41% Oz
FM - 1	Resin-impregnated paper			
FM-3	Cotton terry cloth	Burned		
FM-28	Cotton cloth, white duck	Burned		
FM-4	Cotton terry cloth, Roxel-treated	No	No	Burned
FM-5	Fleece-backed cotton cloth, Roxel-treated	Surface only	Burned	Burned
FM-14	Cotton, O.D. Sateen, Roxel-treated	No	Burned	-
FM-15	Cotton, green whipcord, Roxel-treated	No	Burned	-
FM-16	Cotton white duck, Roxel-treated	No	Burned	-
FM-17	Cotton, King Kord, Roxel-treated	No	Burned	-
FM-29	Cotton white duck, treated with 30% boric acid- 70% borax	No	Burned	Burned
FM-30	Cotton terry cloth, treated with 30% boric acid-70% borax	No	Burned	Burned
FM-6	Fire-resistant cotton ticking	No	Burned	-
FM-7	Fire-resistant foam rubber		No	Burned
FM-9	Nomex temperature-resistant Nylon	No	Burned	-
FM-10	Teflon fabric		No	No
FM-11	Teflon fabric	No	No	No
FM-12	Teflon fabric	No	No	No
FM-13	Teflon fabric	No	No	No
FM-19	Verel fabric	No	Burned	Burned
FM-22	Vinyl-backed fabric	No	Burned	Burned
FM-23	Omnicoated Dupont high-temperature fabric	No	Burned*	Burned
FM-24	Omnicoated glass fabric	No	No	Burned*
FM-20	Glass fabric, fine weave	No	No	No
FM-21	Glass fabric, knit weave	No	No	No
FM-25	Glass fabric (coarse weave)	No	No	No
FM-26	Glass fabric (coarse weave)	No	No	No
FM-27	Aluminized asbestos fabric	No	No	Burned
FM-32	Rubber from aviator oxygen mask	Burned	Burned	Burned
FM-33	Fluorolube grade 362	No	No	Not
FM-34	Belco no-flame grease	No	No	Not

Table 4	
Flame Resistance of Materials Held Vertically at One Atmosphere Pressure in O, /N, Mixtu	res

Burned ... ly over igniter. † White soke only.

substitution of helium for oxygen, and igniter temperature. The data which are tabulated often represent the average of soveral experimental values. Detailed data are also given in Table 2. All data were obtained at the same igniter energy input unless otherwise stated.

Effect of Oxygen Enrichment

The effect of increasing the percentage of oxygen on the ease of ignition can be summarized as follows: (a) when the material ignited readily, increasing the oxygen content from 21% to 31% did not affect the ignition delay time appreciably, as illustrated by the data in Table 5. (b) When ignition was difficult, increase in oxygen content tended to have a decided effect. For example, as shown in Table 5, filter paper did not ignite in He/O₂ mixtures at 21% O₂ until a pressure of 75 psia was reached. In 31% oxygen, ignition occurred readily at 15 psia. (c) In many other instances, increasing the percentage of oxygen allowed ignition of materials which did not ignite at lower percentages of oxygen (see Table 4).

	Eace of 18th		iter raper		
Total		Ignition Delay (sec)			
Pressure	In N ₂ /O ₂				
(psia)	21% O ₂	31% O2	21% O ₂	31% O ₂	
15	6.5	6.0	NI	17.0	
30	6.0	5.7	NI	14.3	
60	6.0	4.8	NI	7.5	
75	4.8	5.4	18.0	7.5	

Table 5
Effect of Oxygen Enrichment on the
Ease of Ignition of Filter Paper

In general, the burning rate of materials was markedly increased by increasing the oxygen content of the atmosphere. An example of such data is given in Table 6. Filter paper burned approximately twice as fast when the oxygen content in N_2/O_2 mixtures was increased from 21% to 31%. A graphical presentation of burning data for the filter paper in O_2/N_2 mixtures is given in Fig. 5. These data confirm that the burning rate of filter paper is approximately doubled as the oxygen is increased from 21 to 31% with nitrogen as the diluent.

Table 6Effect of Oxygen Enrichment on Burning
Rates with Nitrogen as the Diluent

Total	Burning Rate (cm/sec)					
Pressure	Filter Paper		Terry Cloth			
(psia)	21% O ₂	31% O ₂	21% O ₂	31% O ₂ _		
15	0.23	0.43	0.20	1.67		
30	0.28	0.45	0.61	1.57		



Fig. 5 - Effect of pressure and oxygen enrichment on burning rate

Whereas an increase of oxygen from 21% to 31% in nitrogen caused an approximate doubling of the burning rate of the filter paper, the increase in burning rate for cotton terry cloth was much more pronounced (Table 6). At one atmosphere (15 psia), the burning rate in O_2/N_2 was 0.20 cm/sec at 21% O_2 and 1.67 cm/sec at 31% O_2 .

The effect of increasing the oxygen percentage in admixture with helium is similar, although the data are limited because in many instances at 21% oxygen in helium no ignition occurred. However, it was observed (Table 2) that filter paper ignited in O_2 /He:21/79 at 75 psia and burned at 0.48 cm/sec. In O_2 /He:31/69, at 15 psia, the burning rate was 0.76 cm/sec, a pronounced increase in rate.

Effect of Increased Pressure

In general, it was observed that an increase in pressure did not have as marked an effect on ignition and combustion of materials as did an increase in the oxygen percentage. However, it was observed that increased pressure often showed decided leverage in enhancing combustibility. For example, in many instances an increase in pressure caused ignition which did not occur at lower pressures. This fact is illustrated in Table 5 in that filter paper did not ignite in 21% oxygen in helium until a pressure of 75 psia was reached. Other examples are evident in Tables 1 and 2.

It was particularly true of the treated terry cloth that an increase of pressure caused ignition and combustion where none was evident at lower pressures (see Table 1). For instance, treated terry cloth, in a vertical position in an atmosphere of 31% oxygen in nitrogen, charred only half the length of the test strip at 15 psia, but ignited readily at 30 psia.

The effect of increased pressure on burning rate was also most noticeable in causing very rapid burning in materials that would not even ignite at lower pressures (see Table 1). As shown in Fig. 5, the burning rate of the standard filter paper was increased moderately by increase in pressure.

Effect of Substitution of Helium for Nitrogen

The data given in Table 7 very clearly show two effects of the substitution of helium for nitrogen on the flammability of filter paper. These data are representative of a greater number of experiments, some of which are collected in Tables 1 and 2. It is evident that ignition is more difficult to obtain with helium present. The mixed helium/ nitrogen diluent gives results for ignition delay that are intermediate between nitrogen and helium. With helium, ignition delays are invariably considerably longer, often resulting in no ignition when easy ignition occurs with nitrogen as diluent.

Although ignition is more difficult in helium/oxygen, Table 7 suggests that the burning rate is often greater than in nitrogen/oxygen. However, the results in Table 2 with terry cloth suggest that the effect of helium substitution for nitrogen upon burning rates is complex. In several instances the terry cloth burning rate was greater in nitrogen mix-tures than in helium mixtures.

EFFECT OF ATMOSPHERIC COMPOSITION ON IGNITER TEMPERATURE

One of the effects observed when equal energies were applied to the igniter was that in He/O₂ mixtures the ignition delays were much longer than in N₂/O₂. In many cases no ignition occurred in He/O₂, whereas the material did ignite and burn in N₂/O₂.

12

 Table 7

 Effect of Substitution of Helium for Nitrogen on the Flammability

of Filter	Paper Held	Horizontall	y at 15 psia			
Dilucut	Ignition De	elay (sec)	Burn Rate (cm/sec)			
Diluent	21% O2	31% O2	21 ⁰⁷ O ₂	3.1% O2		
Nitrogen	6.5	6.0	0.23	0.43		
Helium	NI	17.0	NI	0.76		
Equal Volumes of Helium and Nitrogen	-	10.2	-	0.61		



Fig. 6 - Effect of different atmospheres on igniter temperature

Since we can assume that N_2 and He are inert and do not affect ignition chemically, we may look to the physical properties of these gases for explanation. A property in which He excels is thermal conductivity. Heat would be conducted away from the igniter wire much faster in He/O₂ than in N_2/O_2 , so that in He/O₂ the material does not reach as high a temperature. Because of this likely explanation, experiments were designed to test it.

First, the temperature of the igniter wire as a function of applied current was measured by optical pyrometry in $21\% O_2/79\%$ He; $31\% O_2/69\%$ He; and $21\% O_2/79\% N_2$. The results are plotted in Fig. 6. It is seen that at a given current the temperature of the igniter is, indeed, considerably lower in the helium mixture than in the nitrogen mixture. When oxygen in He/O₂ was increased from 21% to 31%, a significant increase in temperature was observed. No significant effect was found in N_2/O_2 mixtures in going from 21% to 31% oxygen, ns would be expected due to the similar thermal conductivities for oxygen and nitrogen.

The igniter was set at 4.0 amperes for most of the experiments recorded earlier. It was found here that at 4.0 amperes applied current, the temperature of the igniter in 21% O₂/79% N₂ was 970°C, and in 21% O₂/79% He, it was 850°C, a difference of 120°C. By increasing the current to 4.7 amperes, the temperature in O₂ /He was brought up to 970°C.

		Juc Aunos	phere Pres	Suie			
Filament Current (amp)	Igniter Temp. (°C)	Ignition Delay (sec)	Burn Rate (cm/sec)	Igniter Temp. (°C)	Ignition Delay (sec)	Burn Rate (cm/sec)	
	31% O ₂ /69% He			$31\% O_2/69\% N_2$			
4.0	860	15.0	0.79	960	6.4	0.52	
4.6	970	9.2	0.70	-	-	-	
4.7	980	7.8	0.93	-	-	-	
4.8	990	7.5	0.85	-	-		
	21% O ₂ /79% He			21	% O₂/79%	N ₂	
4.0	850	NI	NI	960	10.6	0.24	
4.6	93 5	NI	NI	-	-	-	
4.7	970	NI	NI	-	· _	-	

Table 8Effect of Igniter Temperature on the Flammability of Filter Paper
at One Atmosphere Pressure

Ignition data are given in Table 8 which show the effect of increasing the igniter temperatures in He/O₂ mixtures to match those in N₂/O₂ mixtures. The ignition delay in 31% O₂/69% He was in fact shortened considerably as the igniter temperature was increased. However, there was still a significantly longer lag than in N₂/O₂ at equivalent igniter temperatures. This effect is even more pronounced in the 21% O₂ mixtures. No ignition was induced in the He/O₂ mixture even at 970°C.

The fact that equal igniter temperatures did not cause equal ignition effects on filter paper in He and N_2 mixtures may be due to yet another effect of the much greater thermal conductivity of the He. That is, ignition of a material results when the material on heating releases vapors in sufficient quantity and at high enough temperatures to ignite. Although the igniter wire may be equally hot in mixtures of N_2 and He, the material is also losing heat at a greater rate in He and the vapors are diffusing away at a greater rate, which would make ignition of a material more difficult even though equal energies are applied.

Although the greater difficulty of igniting materials with a hot wire in helium atmospheres was shown to be largely due to the much higher thermal conductivity in helium mixtures, the greater ease of ignition as pressure was increased cannot be explained in this way. In general, thermal conductivity tends to increase as pressure is increased; but this effect is relatively small, especially with helium (13). It is likely that the better ignitability with increased pressure is due to the increased availability of oxygen atoms in contact with the heated solid.

FLAMMABILITY OF OIL-SOAKED PAPER

Besides the atmospheric factors which contributed to increased ignitability and faster burning rates in the NEDU fire, an additional factor was present. The paper filter in the CO_2 absorber had been contaminated by hydraulic oil in testing for compliance with the specifications, as described in the Introduction. Although the amount of residual oil contamination on the NEDU filter is not known because of its previous use, it seemed useful to impregnate filter paper with hydraulic oil and determine its flammability. The data, taken in N_2/O_2 and He/O_2 atmospheres, are given in Table 9.

No pronounced effect on ignition delay time was observed. This fact may imply that it is necessary to ignite the paper itself before flame propagation will occur, because it is

14

Composition of Atmosphere (wt-%)		Burn Rate (cm/sec)		Ignition Delay (sec)		
O ₂	N ₂	He	Oil-Free	Oiled	Oil-Free	Oiled
21	79	-	0.23	0.77	6.5	6.8
21	-	79	NI	NI	NI	NI
31	69	-	0.43	2.2	6.0	5.2
31	-	69	0.76	3.2	17.0	20.2

Table 9Effect of Oil Impregnation (30% MIL-H-5606A HydraulicFluid) on Flammability of Filter Paper Held Horizontally
at Atmospheric Pressure

probable that the oil is evaporated and carried away from the igniter by convection during the heating process before a flame is generated. Once inflamed, however, the burning rate of the oil-impregnated paper was much faster: from 3 to 5 times as fast as the oilfree paper. This is probably due to a large extent to the fact that the hydrocarbon vapors from the hydraulic fluid are available to feed the flame as soon as they are volatilized. In contrast, the oil-free paper and its polymer coating must be pyrolyzed to provide vapors for combustion, a process which should tend to modulate the burning rate.

FLAME RESISTANCE OF SELECTED MATERIALS

A survey was made of a number of candidate materials such as clothing for possible use in hyperbaric chambers and in other applications. In order to save time, certain experimental conditions were selected based on previous experimental findings. Since the factor with the greatest impact was found to be oxygen enrichment, it was decided to use nitrogen/oxygen mixtures at atmospheric pressure for this survey. The data are summarized in Table 4. As can be seen, although a number of materials are fire-resistant at 21% oxygen, most of them will burn when oxygen is increased to 31%. Only two types of materials did not ignite in an atmosphere containing 41% oxygen. These, Teflon and glass, are known to resist ignition even in 190% oxygen atmospheres (10).

These results do not include allowance for increased pressure or helium as a diluent. Both of these factors will in some instances certainly cause ignition and combustion of some of the materials that did not burn at 21% and 31% oxygen in nitrogen at atmospheric pressure (15 psia). For example, Item FM-4, THPC-treated cotton terry cloth, did not burn in 31% oxygen/nitrogen at 15 psia, but burned readily when the pressure was increased to 30 psia (Table 1). It is important that all of the factors of interest be invoked whenever a decision as to suitability of a material is in question.

SUMMARY

The effect of oxygen enrichment, increased pressure, and substitution of helium for oxygen on the flammability of fabrics and other solid materials was studied. Oxygen enrichment had the most pronounced effect. Linear burning rates were increased by a factor of about 2 or 3 when oxygen content was increased from 21% to 31%. Oxygen enrichment was especially effective in inducing ignition. Many materials which did not ignite at 21% oxygen, ignited and burned readily when oxygen content was increased to 31% or 41%.

Increase in pressure of a given atmosphere often had a pronounced effect on the ignition of materials, especially in borderline cases. In atmospheres of a given composition, ignition frequently occurred at higher pressures where no ignition occurred at lower pressures.

Substitution of helium for nitrogen as the diluent gas was found to decrease the tendency of a material to ignite. This effect was shown to be caused largely by the relatively high thermal conductivity of helium, which dissipated the heat from the igniter. Once ignited, however, most of the materials tested had a decidedly greater burning rate in the helium mixtures than in nitrogen mixtures.

Although a number of the potentially combustible materials studied did not ignite in ordinary air at one atmosphere pressure, upon increase of oxygen content to 41%almost all these materials ignited and burned.

CONCLUSIONS

Although it is recognized that the data given in this report are limited and much more needs to be done, they are being reported because of the present interest in, and the importance of, the results. Up to now, very few experimental data have been available concerning the effect on the flammability of materials of atmospheric variables such as pressures greater than one atmosphere, increased percentages of oxygen, and diluents such as helium. This lack of information is brought out in the literature summary in this report.

On the basis of this exploratory study, it was found that there were very few materials which could not be ignited at 41% oxygen in nitrogen at one atmosphere. Although this study was not exhaustive, only Teflon and glass fabrics were judged to be nonflammable under these conditions. Of course, many metals and other inorganic materials such as asbestos should be suitably nonflammable also. The NEDU is presently designing the interior furnishings of their experimental pressure chambers to use such materials. Based on these results, NEDU is also assessing the suitability of glass (Beta fibers) and Teflon in fabrics used for garments to be worn by chamber occupants. Of particular interest is the potential irritation to the skin by these materials.

During this study, it became apparent that factors such as increased oxygen content and increased pressure could significantly increase the flammability of materials. However, at this stage it must be concluded that no simple multiplication factors can be used in predicting the flammability of materials in such artificial atmospheres. Therefore, before any material can be judged safe for a given application, all the pertinent conditions of use should be examined experimentally.

The present work is being extended to include conditions where no data exist at present. In particular, it is planned to study flammability at pressures ranging from subatmospheric up to 300 psi, and to study the effects of a wider range of concentrations of oxygen, especially in helium atmospheres.

16

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Appendix A

DESCRIPTION OF MATERIALS TESTED

- FM-1 <u>Resin-impregnated paper</u>. 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."
- FM-3 Cotton terry cloth. This cotton terry cloth robe was supplied by the Navy Experimental Diving Unit (NEDU), Navy Yard, Washington, D.C.
- FM-4 <u>Cotton terry cloth, Roxel-treated.</u> This Roxel-treated cotton terry cloth robe was supplied by NEDU.
- FM-5 <u>Fleece-backed cotton cloth, Roxel-treated</u>. These fleece-backed cotton sweat pants were supplied by NEDU.
- FM-6 <u>Fire-resistant cotton ticking</u>. This cotton mattress ticking was supplied by NEDU.
- FM-? <u>Fire-resistant foam rubber.</u> This fire-resistant foam rubber mattress padding was supplied by NEDU.
- FM-9 <u>Nomex temperature-resistant Nylon</u>. This high-temperature resistant Nylon (manufactured by the DuPont Company) was supplied by NEDU
- FM-10 <u>Teflon fabric</u>. This fabric, manufactured by Stern & Stern, identification number T-3-42, was obtained from Code 6120 at NRL.
- FM-11 <u>Teflon fabric.</u> This fabric, manufactured by Stern & Stern, identification number T-9-21, was obtained from Code 6120 at NRL.
- FM-12 <u>Teflon fabric</u>. This fabric, manufactured by Stern & Stern, identification number T-2-42, was obtained from Code 6120 at NRL.
- FM-13 <u>Teflon fabric.</u> This fabric, manufactured by Stern & Stern, identification number T-93-42, was obtained from Code 6120 at NRL.
- FM-14 <u>Cotton O.D. Sateen, Roxel-treated</u>. This fire-resistant cloth was supplied by the Hooker Chemical Corporation, Niagara Falls, New York.
- FM-15 Cotton green whipcord, Roxel-treated. This fire-resistant cloth was supplied by the Hooker Chemical Corporation.
- FM-16 <u>Cotton white duck, Roxel-treated.</u> This fire-resistant cloth was supplied by the Hooker Chemical Corporation.
- FM-17 <u>Cotton King Kord, Roxel-treated.</u> This fire-resistant cloth was supplied by the Hooker Chemical Corporation.

- FM-19 <u>Verel fabric.</u> This fire-resistant fabric, made of Eastman modified acrylic staple fibers, was obtained from the Southern Regional Research Laboratory, USDA, New Orleans, Louisiana.
- FM-20 <u>Glass fabric</u>. This fine-weave fabric, made of Beta glass fibers, was obtained from the NASA Manned Space Center, Houston, Texas.
- FM-21 <u>Glass fabric</u>. This knit weave fabric of Beta glass fibers was obtained from the NASA Manned Space Center, Houston, Texas.
- FM-22 <u>Vinyl-backed fabric.</u> This fire-resistant mattress covering material was supplied by NEDU.
- FM-23 Omn coated DuPont high-temperature fabric. This fabric (manufactured by the DuPont Company) was supplied by NEDU.
- FM-24 <u>Omnicoated glass fabric</u>. This fabric (manufactured by the DuPont Company) was supplied by NEDU.
- FM-25 <u>Glass fabric.</u> This glass fabric, designated Style 539, was manufactured by Hess, Goldsmith & Company, Inc., and was supplied by NEDU.
- FM-26 <u>Glass fabric</u>. This glass fabric, designated Style 217, was manufactured by Hess, Goldsmith & Company, Inc., and was supplied by NEDU.
- FM-27 <u>Aluminized asbestos fabric</u>. This material is similar to that used in NASA space suits and was supplied by NEDU.
- FM-28 <u>Cotton cloth, white duck</u>. This laboratory coat was manufactured by the Fisher Scientific Company and obtained at NRL.
- FM-29 <u>Cotton cloth</u>. This is the same material as FM-28, but treated for fire resistancy with 13.8 wt-% of 70% borax and 30% boric acid.
- FM-30 <u>Cotton terry cloth</u>. This is the same material as FM-3, but treated for fire resistancy with 23.3 wt-% of 70% borax and 30% boric acid.
- FM-32 <u>Natural rubber</u>. This rubber from aviators mask, oxygen, MIL-M-6482B(4), was supplied by NEDU.
- FM-33 <u>Fluorolube grade 362</u>. This material was manufactured by the Hooker Chemical Corporation and supplied by NEDU.
- FM-34 <u>Belco no-flame grease</u>. This grease, manufactured by the Bel-Ray Company, Inc., was supplied by NEDU.

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