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# APOLLO

## GUIDANCE AND NAVIGATION

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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E-1713

COMBUSTION OF POLYOLEFIN  
INSULATION AND TOXICITY  
OF TEFLON UNDER  
ELECTRICAL OVERLOADS

by

A. O. Mirarchi

December 1964

**MIT**

**INSTRUMENTATION  
LABORATORY**

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## GUIDANCE AND NAVIGATION

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# INSTRUMENTATION LABORATORY

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The preparation and baking of Teflon insulated wires was done at the Instrumentation Laboratory by Mr. M. Soikkeli, while the Mass Spectrometric Analyses of the pyrolysis products of these samples were done by Dr. Paul Vouros, under the direction of Professor Klaus Biemann, Chemistry Department, MIT.

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The publication of this report does not constitute approval by the National Aeronautics and Space Administration of the findings or the conclusions contained therein. It is published only for the exchange and stimulation of ideas.

## SUMMARY

Test were conducted on polyolefin insulated #24 gauge wires to determine conditions of insulation breakdown, ignition, and explosion. Some wires were treated with a water-repellant-type-coatings.

Test conditions in single strands of wire included

- (a) an open flame,
- (b) at pressure of 1/3 atmosphere in static oxygen with vessel externally heated up to 725<sup>o</sup>F, and
- (c) at ambient atmosphere with wire heated internally by current of 10, 15, 20, and 25 amps.

Bundles of three insulated wires inside a plastic sleeving material were tested at 1/3 atmosphere with internal current heating in one wire ranging to 34 amps.

- (a) in static oxygen
- (b) in oxygen flowing at 3 liters/min, and
- (c) in oxygen flowing at 6 liters/min:

A bundle of three wires in a copper braid sleeve was tested under similar conditions.

Bundles of thirteen wires were tested with internal current heating of about 30 amps (except as noted).

- (a) under ambient atmosphere (with 10 to 33 amps)
- (b) at 1/3 atmosphere in static oxygen
- (c) at 1/3 atmosphere in oxygen flowing at 1 liter/min,
- (d) at 1/3 atmosphere in oxygen flowing at 3 liter/min,
- (e) at 1/3 atmosphere in oxygen flowing at 6 liter/min,
- (f) inside plastic sleeving at 1/3 atmosphere in oxygen flowing at 3 liters/min,
- (g) in copper mesh at 1/3 atmosphere in oxygen flowing at 3 liter/min.

One bundle of forty-three wires in a plastic sleeve was tested at 1/3 atmosphere in oxygen flowing at 3 liters/min with 30 amp current in one central wire.

In addition, one bundle of thirteen Teflon-polyamide insulated #22 gauge wire was tested at 1/3 atmosphere in oxygen flowing at 3 liters/min with a 30 amp current in a central wire. Finally, these same conditions were applied to a similarly insulated bundle of thirteen #24 gauge wires.

The following Summary Table shows the various tests and test conditions, with reference to the particular detailed table in the text. All observations made are included in the respective tables.

SUMMARY TABLE

Number of Wires	Run No.	Treatment		Pressure (atm)	Heating			Environment		Reference Table No.	
		None	L-1483		Open Flame	External Tape	Internal Current	Ambient	Static Oxygen		Dynamic Oxygen Liter/min
<b>A. Polyolefin insulation, #24 AWG</b>											
Single, 3"	1-4	X		Ambient							3
Single, 3"	1-4		X	Ambient		X					3
Single, 3"	1	X		Ambient			X				4
Single, 3"	1-2	X		Ambient			X				4
Single, 3"	1-2		X	1/3			X				5
Single, 3"	1-2		X	1/3			X				6
Single, 3"	3		X	1/3			X				6
Single, 3"	1	X		Ambient			X				7
Thirteen, 3"	2	X		Ambient			X				7
Thirteen, 3"	1-2	X		1/3			X				8
Thirteen, 3"	3	X		1/3			X				8
Thirteen, 3"	3	X		1/3			X				8
Three, 7"	1		X	1/3			X				9
Three, 7"	4		X	1/3			X				9
Three, 7"	5		X	1/3			X				9
Three, 7"	1		X	1/3			X				9
Three, 7"	2		X	1/3			X				9
Thirteen, 7"	1	X		1/3			X				10
Thirteen, 7"	2	X		1/3			X				11
Thirteen, 7"	1-2	X		1/3			X				11
Thirteen, 7"	1-2	X		1/3			X				12
Thirteen, 7"	1	X		1/3			X				13
Thirteen, 7"	2		X	1/3			X				13
Thirteen, 7"	3		X	1/3			X				13
Thirteen, 7"	3		X	1/3			X				14
Thirteen, 7"	3		X	1/3			X				14
Thirteen, 7"	3		X	1/3			X				14
Forty-three, 7"	1	X		1/3			X				15
Forty-three, 7"	1	X		1/3			X				15
Forty-three, 7"	2	X		1/3			X				15
Three, 7"	3	X		1/3			X				16
<b>B. Teflon-polyamide insulation</b>											
Thirteen #22 AWG, 7"	1	X		1/3			X				17
Thirteen #24 AWG, 7"	1-2	X		1/3			X				18

## CONCLUSIONS AND RECOMMENDATIONS

The condition of electrical overloading of wires, having different insulation materials, leads to the formation of gaseous mixtures between the products formed by the pyrolytic degradation of the insulation and the oxygen (or other) atmosphere surrounding them. These gaseous mixtures can be combustible, as is the case when hydrocarbons are given off from polyolefin insulation. Or they may be noncombustible, as is the case when fluorocarbons are given off from the Teflon. Combustion will occur, in the case of hydrocarbon and oxygen mixture, only if the mixture falls within the limits ranging from fuel-rich to oxygen-rich-mixture and, at the same time, a hot spot of about  $1000^{\circ}\text{C}$  temperature is present which can trigger the ignition. While the hydrocarbons are not toxic, the fluorocarbons are toxic; hence, this additional hazard must be recognized in the confines of the space capsule.

Consequently, the need is indicated for a conservative approach in the electric and electronic design of all equipments on board to provide automatic fail-safe features. Wiring should be selected on the basis of anticipated overloads. Where this condition could happen, the insulation should be of a nontoxic, noncombustible nature, such as glass or asbestos insulation, and, additionally, these circuits should be kept physically separated from all others.

## SECTION 1.

### INTRODUCTION

The tests undertaken for this work were made for the purpose of obtaining confirmation on the expected behavior of wire insulation materials when heated, by electrical overloading, in an atmosphere of oxygen. The atmosphere of the Command Module for the Apollo mission is composed of oxygen, at a pressure of 5 psi. Superposed to this, an anticipated (temperature dependent) moisture component of 1 to 2 psi will be established soon after launch, due to fluids and humans on board. This moisture component has not been introduced in the tests herein reported because of the time requirements for obtaining the test data, and because it is believed that such an additional moisture component would have complicated considerably the test procedure and instrumentation, without changing the basic findings. The selection of electrical insulation materials to be tested was based essentially on the fact that, chronologically, the irradiated polyolefins (Thermorad) had been selected as suitable and acceptable on the basis of their other desirable properties as wire insulation materials (cut-through, thickness, density, insulation resistance, radiation resistance, etc). Teflon insulation was tested to obtain a comparison versus irradiated polyolefins with respect to combustibility and toxicity. The Teflon-polyamides (Kynar) were tested, as a few samples became available, again for the purpose of comparison, inasmuch as this combination of insulation materials showed desirable properties falling between those of Teflon alone, and those of irradiated polyolefins.

The reader will notice that electrical overloading was at first attained by increasing the amount of current flowing through the wires in some arbitrary increments of time and of current. This was done deliberately to get a feel for the most desired way in which to conduct the tests. In general, such step-wise procedure leads to a higher tolerance by the system for the overload, because the system has time in which to dissipate thermal energy through mechanisms of radiation, conduction, and convection. A proper technique for these studies would seem to be one in which overloading would be accomplished exactly as it might occur during operation of the electrical-electronic equipments on board: i. e. , the intensity, the frequency, and the

duration of the overloads should be duplicated in wires of the same make and gauge as those used in the actual circuits.

Another point of considerable importance must be made of the techniques referred to in this report as "static" or "dynamic" oxygen. The former means that the experimental vessel was given an initial charge of oxygen to the desired pressure, 5 psi. The latter means that inlet and outlet valves of the experimental vessel were manipulated to obtain the desired oxygen pressure in the vessel while oxygen was flowing. The former technique leads to the formation of combustible mixtures of oxygen and fuel (distillate from the wire insulations) ranging widely in composition, and showing some typical relationship between composition of the gaseous mixtures and wire temperatures. The latter technique leads to the formation of an entirely different set of corresponding values.

These two different techniques can be summarized and generalized by noting that the static tests showed no combustion, i. e., the attainment of combustible gaseous mixture between distillate and oxygen occurred before the wires were hot enough to trigger combustion; and, at a later time, when the wires became hot enough to trigger the combustion, this could not occur because the gaseous mixture was fuel-rich.

The dynamic tests showed violent combustion because even though some of the fuel was being continuously swept out of the experimental vessel, the rate of distillation was continuously increasing as the wire got hotter, and the oxygen was continuously being replenished until eventually the wire temperatures reached the ignition point of the oxygen-fuel mixtures.

Analogous conditions to these described can be expected to be generated on board, depending on circulation of the Command Module atmosphere, on the ventilation, or lack of it, in some recessed part of the equipment, and on the interplay between the mechanisms of radiation, conduction, and convection in dissipating the heat generated in one or more wires.

## SECTION 2

### PROCEDURE AND RESULTS

#### 2.1 Application of Retardant to Cables

Water repellent L-1483 is available from Minnesota Mining and Manufacturing Co. The suspension as received from the manufacturer was diluted to achieve a desired coating thickness. All coatings at the MIT/IL laboratory were applied from a mixture of 13.3 gms of L-1483 (as received from the manufacturer) and 86.7 gms of a 4:1 solution of 1, 1, 1 trichlorethane and methyl ethyl ketone. This mixture resulted in a 2% L-1483 solution.

The polyolefin wires were dipped in this solution and withdrawn at a time rate of approximately three seconds per inch. This procedure insured uniformity and reproducibility of the coating.

Before applying the retardant to the wires, the wires were vapor degreased with trichloroethylene. The insulated wires were exposed to trichloroethylene fumes at 80 to 90°C for approximately one hour. Three inch lengths of the 24-gauge wire were weighed before and after dipping in the L-1483 solution. The curing time at room temperature was approximately sixteen hours. A comparison of the amount of L-1483 coating adhered to wires degreased and not degreased is shown in Table 2.1.

TABLE 2.1

Effect of Amount of L-1483 Adhered When Degreased with Trichloroethylene.

Not Degreased		Degreased	
Wire No.	Wgt. of L-1483	Wire No.	Wgt. of L-1483
1	0.0 mg	4	0.5 mg
2	0.3 mg	5	0.6 mg
3	0.5 mg	6	0.6 mg

Although a more uniform and reproducible coating of the retardant is obtained when the wire is degreased with trichloroethylene, this solvent swells the polyolefin insulation to 50 percent of its original size. When

drying, a 16-percent reduction in the length of the original insulation was observed.

A similar procedure was followed using methyl ethyl ketone as the degreasing solvent. The insulation did not swell when exposed to these vapors, nor was any shrinkage observed upon drying. The wires were degreased with methyl ethyl ketone by vapor degreasing as well as by wiping with a paper tissue saturated with this solvent. The results are shown in Table 2.2.

TABLE 2.2

Amount of L-1483 Adhered When Degreased With Methyl Ethyl Ketone.

Vapor Degreased		Wiped with MEK	
Wire No.	Wgt. of L-1483	Wire No.	Wgt. of L-1483
1	0.5 mg	1	0.6 mg
2	0.6 mg	2	0.6 mg
3	0.5 mg	3	0.5 mg

In all subsequent coating applications, the wires were degreased by wiping the insulation with a paper tissue saturated with methyl ethyl ketone.

## 2.2 Preliminary Experiments to Determine Ignition of Temperature and Currents.

### 2.2.1 Ambient Conditions

The effect of an open flame on treated (in all subsequent discussions "treated" indicates a coating of L-1483 had been applied) and untreated polyolefin cables was determined. An open flame (propane torch) was maintained five inches from a single strand of wire. A thermocouple was placed in contact with the insulation of this wire. These open flame experiments were conducted under normal atmospheric conditions. The results of these experiments are shown in Table 2.3.

TABLE 2.3

## Open Flame Experiments, Ambient Atmosphere

Treated Wires			Untreated Wires		
Run	Ignition Temp ( $^{\circ}$ F)	Time to Ignite (sec)	Run	Ignition Temp ( $^{\circ}$ F)	Time to Ignite (sec)
1	1050	10	1	1100	18
2	1150	8	2	1050	7
3	1100	10	3	1100	15
4	1050	12	4	1050	8

Under the conditions of the above experiment the wires continued to burn while in proximity of the torch. Removal of the torch caused a cessation of the flame in both the treated and untreated samples.

The charring temperature and current were determined under ambient conditions. Wires three inches in length were successively connected in series to an ammeter and a 50-amp Variac. Currents of 10, 15, 20 and 25 amps were passed through each wire. The temperature at the surface of the wire was determined using temperature-sensitive paints. The wire was held for 120 seconds at each current setting. The results of treated and untreated wires subjected to identical conditions are shown in Tables 2.4.

TABLE 2.4

## Internally Heated Wires, Ambient Atmosphere

Current (amps)	Temperature ( $^{\circ}$ F)	Untreated	Treated	
		Observations	Run 1 Observations	Run 2 Observations
10	140	No change	No change	No change
15	160	No change	No change	No change
20	200	No change	No change	No change
25	400	Yellow Discoloration	Yellow Discoloration	Yellow Discoloration

## 2.2.2 Tests in One-Third Atmosphere of 100% O<sub>2</sub>

Individual strands of wire three inches in length were mounted in the test apparatus shown in Fig. 2.1. The reaction vessel was evacuated by means of a fore pump to approximately  $10^{-3}$  torr. One hundred percent oxygen was then introduced to a pressure of 1/3 of an atmosphere (10 in. of Hg). The vessel was isolated by means of a valve; hence, the increases in pressure noted are attributed to volatiles distilled from the wire insulation. The insulated wire was heated in the reaction vessel from the outside by a heating tape encompassing the vessel. The results of this external heating on untreated wires under static conditions are shown in Table 2.5.

TABLE 2.5

Untreated Wires Externally Heated in O<sub>2</sub> at 1/3 Atmosphere.

Run 1			Run 2		
Temp. (°F)	Pressure (in. of Hg)	Observation	Temp. (°F)	Pressure (in. of Hg)	Observation
200	10	No change	75	10	No change
300	11	No change	167	10.5	No change
350		No change	200	11	No change
400	12	Yellowing	300	11.2	No change
450		Browning	350	11.6	No change
500	12.4	Dark Brown	400	11.9	Browning
550		Very Dark Brown	450	12.1	Browning
600	13.0	Black	500	12.3	Dark Brown
625		Black	550	12.8	Dark Brown
650		Black	600	13.0	Very Dark Brown
700	13.5	Tarry (fusing of insulation)	650	13.2	Brown Black
725		Tarry (fusing of insulation)	700	13.5	Black
			800	14.1	Tarry (fusing of insulation)
			850	14.3	Tarry (fusing of insulation)
			900	15	Tarry (fusing of insulation)
			950	15	Tarry-Bubbling
			1000	15	Tarry-Bubbling

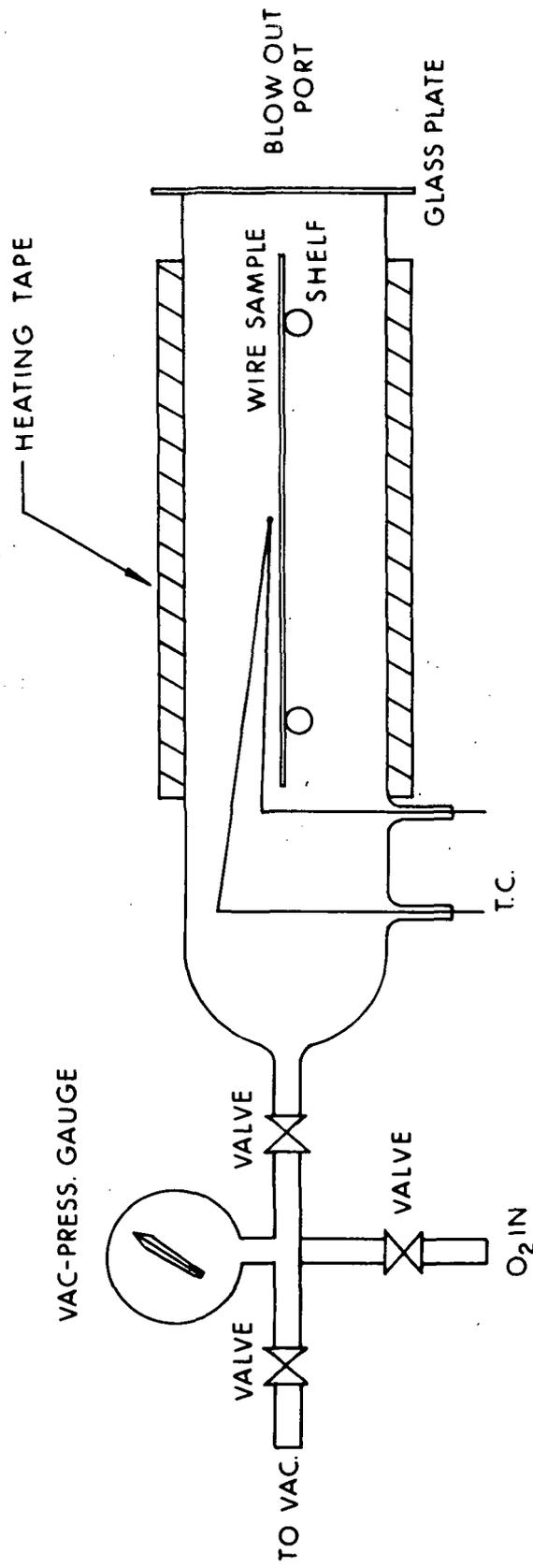


Figure 2.1 Externally Heated Glass Apparatus.

The identical test apparatus was used to determine the effect of wires treated with L-1483 by external heating under static conditions. These results are shown in Table 2.6. Runs #1 and #2 were conducted with wires which had been coated once with L-1483. Run #3 was conducted with a wire which had been coated three times with L-1483.

TABLE 2.6

Treated Wires Externally Heated in 1/3 atm of O<sub>2</sub>.

Temp. (°F)	Pressure (in. of Hg)	Observations	Temp. (°F)	Pressure (in. of Hg)	Observations
<u>Run 1 - (1 coating of L-1483)</u>			<u>Run 3 - (3 coatings of L-1483)</u>		
75	10	No change	75	10	No change
200	10.5	No change	200	11	No change
300	11.2	No change	300	11.7	No change
400	11.7	Discoloration	400	12	No change
500	12.0	Brown	450	12.2	Discoloration
600	12.6	Dark Brown	500	12.5	Brown
700	13.0	Black	600	13.0	Dark Brown
800	13.7	Black	650	13.5	Dark Brown
900	13.5	Tarry	700	13.8	Brown-Black
950	13.0	Tarry-Bubbling	750	14.	Black
1000	13.0	Tarry-Bubbling	800	14.2	Tarry
1050	13.0	Tarry-Bubbling	850	14.7	Tarry
			900	15.0	Tarry-Bubbling
			925	15.0	Tarry-Bubbling
<u>Run 2 - (1 coating of L-1483)</u>					
400	11.0	No change			
450		Discoloration			
500		Brown			
550		Brown			
600		Dark Brown			
700	13.0	Dark Brown			
725		Black			
800	14.0	Tarry			
850		Tarry-Bubbling			

## 2.3 Final Experiments

### 2.3.1 Ambient Conditions

Thirteen untreated wires of three-inch lengths were formed into a bundle of parallel wires. The polyolefin-insulated twenty-four gauge wires were bound with bus wire for rigidity. The central wire of the bundle was connected in series to an ammeter and a 60-amp Variac. Two thermocouples were placed in contact with the bundle, one in the center of the bundle in the proximity of the central wire (internal temperature) and one on the surface of the bundle (external temperature). Current was passed through the central wire in increasing steps of 5 amps, each step being maintained for a three-minute time period. A similar bundle of thirteen treated wires was constructed and tested under identical conditions. These experiments were conducted in room air under ambient conditions. The results are shown in Table 2.7.

TABLE 2.7

Bundles of Thirteen Wires Under Ambient Conditions.

Internal Temp. (°F)	External Temp. (°F)	Current (amps)	Time (min)	Observations
<u>Untreated</u>				
100	95	10	3	No change
140	100	15.5	6	No change
175	125	19.5	9	No change
250	140	24	12	Discoloration
380	155	29	15	Brown Discoloration
800	170	32.5	16	Charring-Heavy Smoke
<u>Treated</u>				
110	95	10	3	No change
150	100	15	6	No change
170	120	20	9	No change
260	150	25	12	Brown Discoloration
400	155	30	15	Black Discoloration
925	180	33	15.5	Charring-Heavy Smoke

### 2.3.2 Tests in One-Third Atmosphere of 100% O<sub>2</sub>

#### a) Glass Test System

The initial apparatus used to ignite by electrical overloading and to evaluate L-1483 as a fire retardant for bundles of irradiated polyolefin insulated wires is shown in Figure 2.2. This test system is constructed almost entirely of glass. The glass reaction vessel can be operated under either static or dynamic conditions. For a static test, the vessel is evacuated to approximately  $10^{-3}$  torr by means of the fore-pump. Valve 1 is then closed and 100% O<sub>2</sub> is introduced through Valve 2 until the vacuum gauge indicates a pressure of 1/3 atm, at which point valve 2 is closed. The vessel is then ready for the static test.

For a dynamic test, the vessel is first evacuated to a pressure of  $10^{-3}$  torr. Oxygen is then bled into the vessel until the vacuum gauge indicates 1/3 atm of oxygen. Valve 1 and Valve 2 are then manipulated to maintain the pressure constant while oxygen is being fed continuously into the vessel. The flow rate through the system is determined by the difference in flowmeter reading with valve 2 closed and opened at the desired operating conditions. (The excess flow is exhausted through the flowmeter which also serves as a vent). The vessel is then ready for the dynamic test.

An enlarged drawing of the reaction vessel itself is shown in Figure 2.3. Current is carried to the test bundle by 12 gauge copper wire. Thermocouples #1 and #2 measure the external and internal temperatures respectively. A blow-off window was provided to prevent destruction of the apparatus in the event of an explosion. (As seen below, this blow-off window proved inadequate).

Three runs were made with this system, all with bundles of thirteen untreated wires of three-inch lengths. In the first run, the current was gradually increased in steps of approximately 5-amp intervals. The vessel was maintained in a static condition. The second run was also a static test, only this time the current was raised to 34 amps immediately. The third run was a dynamic test with flow rate of

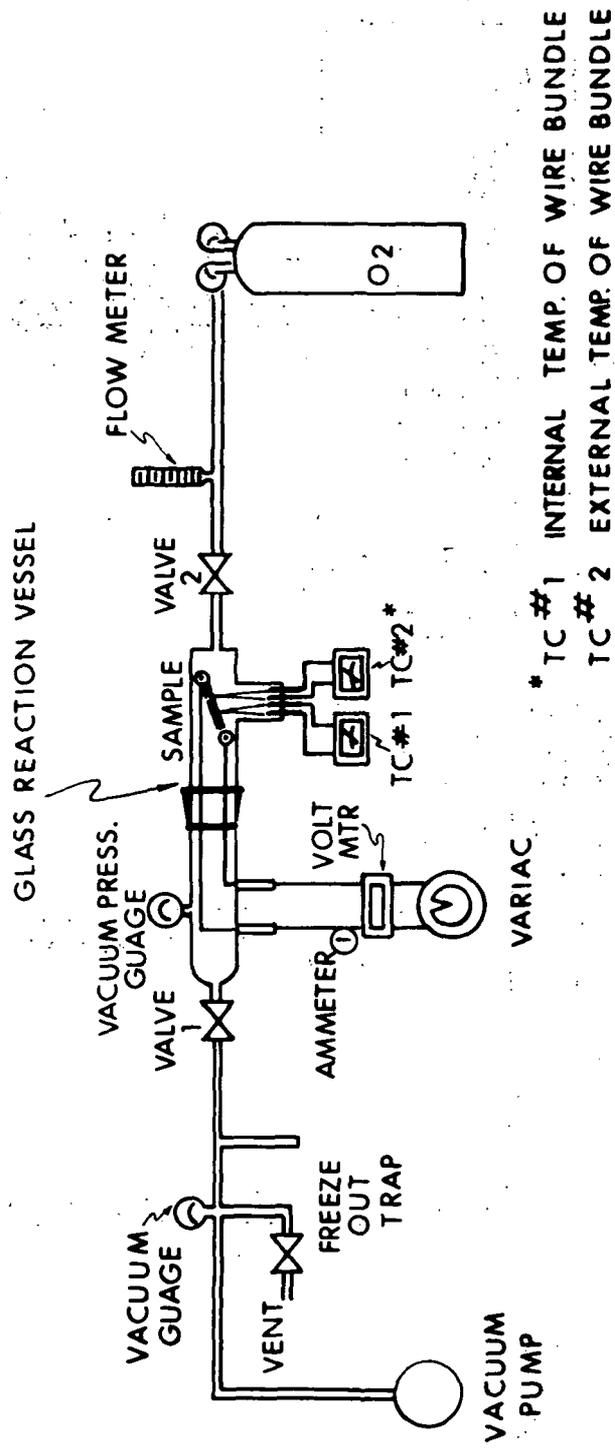
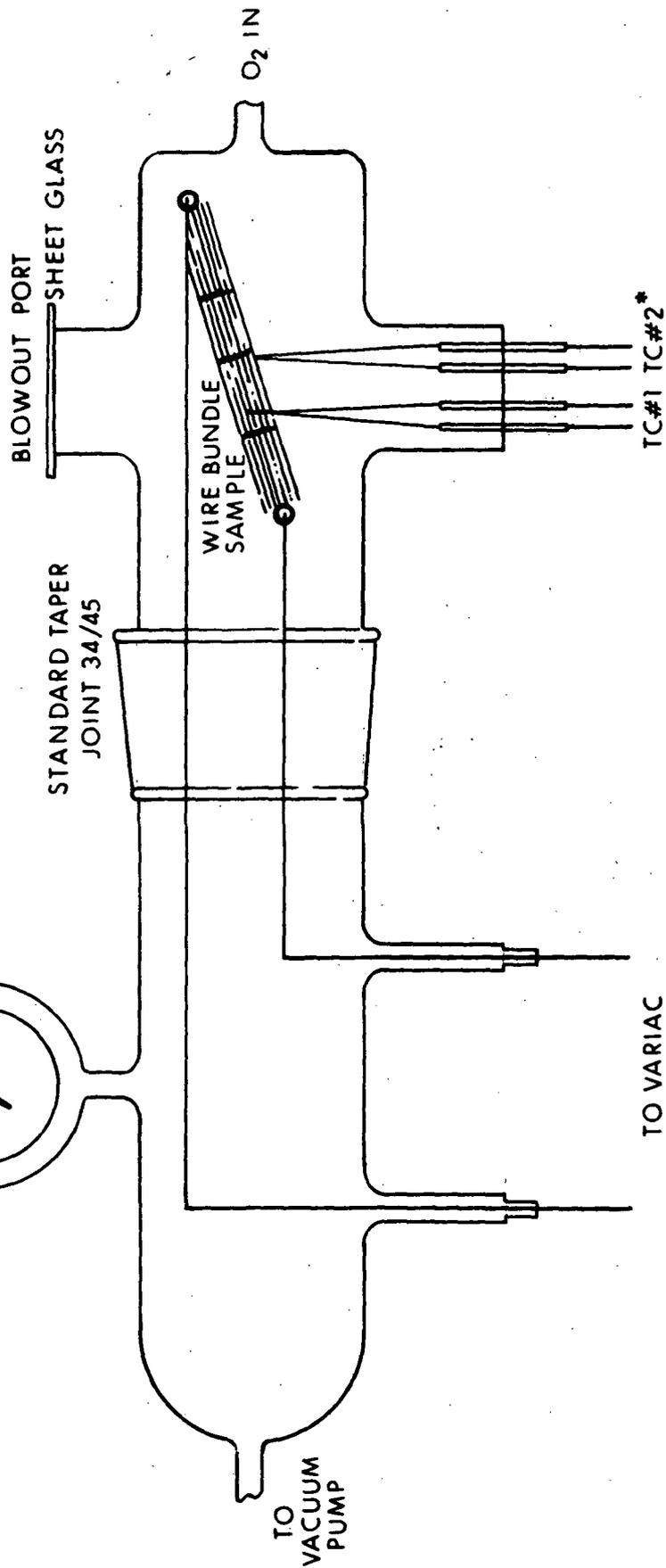
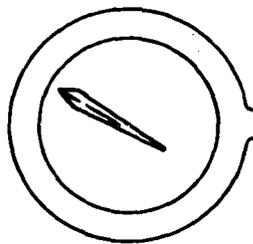


Figure 2.2 Glass Test System.

VACUUM-PRESSURE GAUGE



- \* TC#1 Internal Temp.
- \* TC#2 External Temp.

Figure 2.3 Glass Reaction Vessel.

1 liter/min at 1/3 atm oxygen. The current was raised immediately to 33 amps. In this last run an explosion occurred, shattering the apparatus. The results of these three runs are shown in Table 2.8.

TABLE 2.8

Bundle of Thirteen Untreated Wires in 1/3 Atmosphere O<sub>2</sub>.

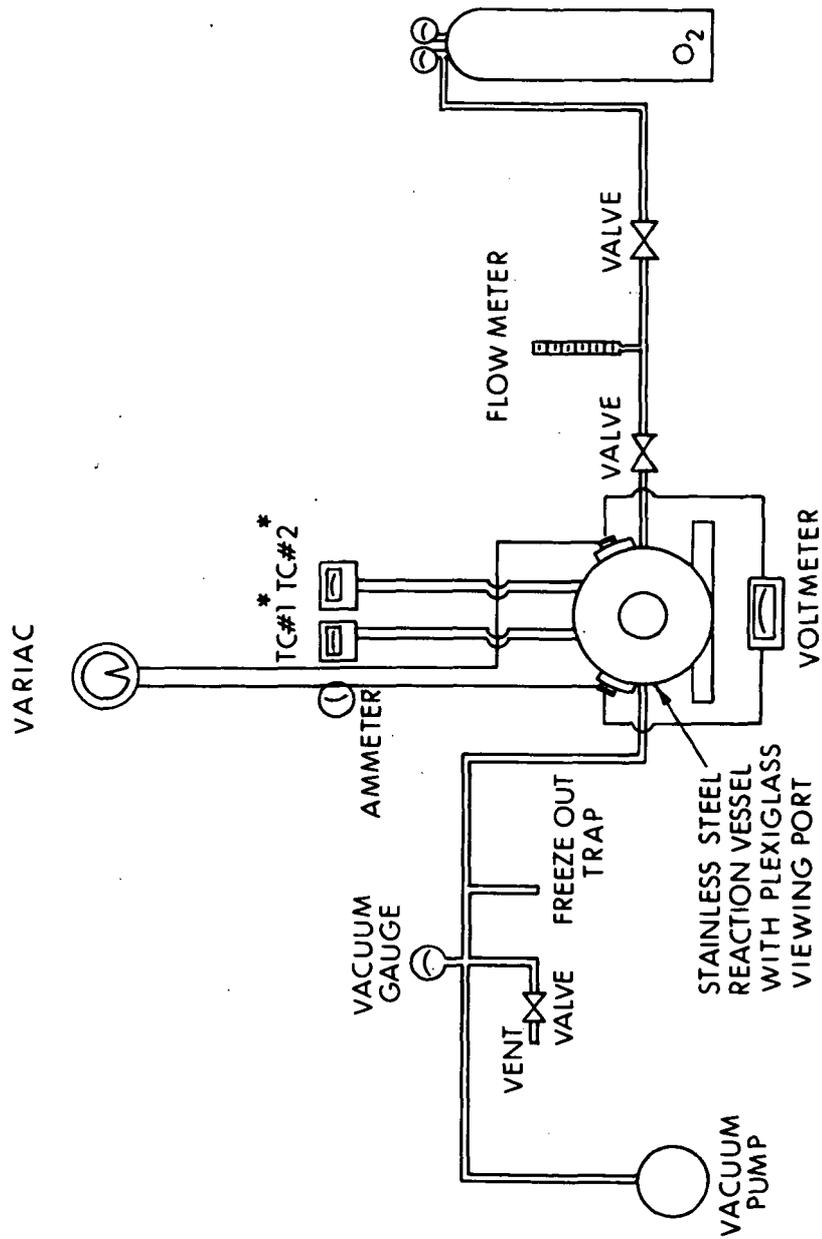
Internal Temp. (°F)	External Temp.	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
<u>Run 1 - Static Test</u>						
Ambient	Ambient	10.0	0	0	0	-----
140	140	10.2	10	0.2	1.5	No change
155	150	10.4	14	0.3	3.5	No change
260	220	10.8	21	0.4	5.5	No change
500	375	11.7	26.5	0.7	8.0	External wire brown
800	550	13.0	30	1.0	10.0	Dense Smoke
1050	620	19.3	34	---	10.2	Insulation Charred off-Central wire Glowing, then Melted
<u>Run 2 - Static Test</u>						
Ambient	Ambient	10	0	0	0	-----
193	158	11	34	1.1	0.5	Charring
288	350	11	34	1.35	1.0	Ignition of Insulation
330	360	13	34	1.48	1.5	Burning continuing
650	400	13	0	----	2.0	Current Shut off, Wire Continued Burning
<u>Run 3 - Dynamic Test, Oxygen at 1L/min.</u>						
Ambient	Ambient	10	0	0	0	-----
93	100	10	34	1.2	0.2	Discoloration
205	150	10.5	34	1.2	0.5	Heavy Smoke
550	200	----	--	---	---	Rapid Ignition and Explosion

### b) Metal Test System

A new test system was constructed. This system was based on the original concept but modified to an all-metal system, the main difference being in the reaction vessel. A stainless steel pressure cooker was modified and adapted to function as a reaction vessel. The cover of the reaction vessel served as a blow-off valve in the event of explosions. A Lucite window was mounted in the bottom of the vessel to observe the phenomena occurring during heating. The test system is shown in Figure 2.4, and a detailed drawing of the reaction vessel is shown in Figure 2.5. All the experiments described below were carried out in this apparatus.

### c) Three Wire Cables

Seven-inch lengths of cables consisting of three 24-gauge polyolefin heated wires, bound with sleeving, were tested under a pressure of  $1/3$  atm of 100%  $O_2$ . The tests were conducted under static and dynamic flow conditions for heated cables and for the same cables enmeshed in either treated or untreated sleeving. The results of these tests are shown in Tables 2.9 and 2.10.



- \* TC#1 Internal Temp. of Wire Bundle.
- \* TC#2 External Temp. of Wire Bundle.

Figure 2. 4 Metal Test System

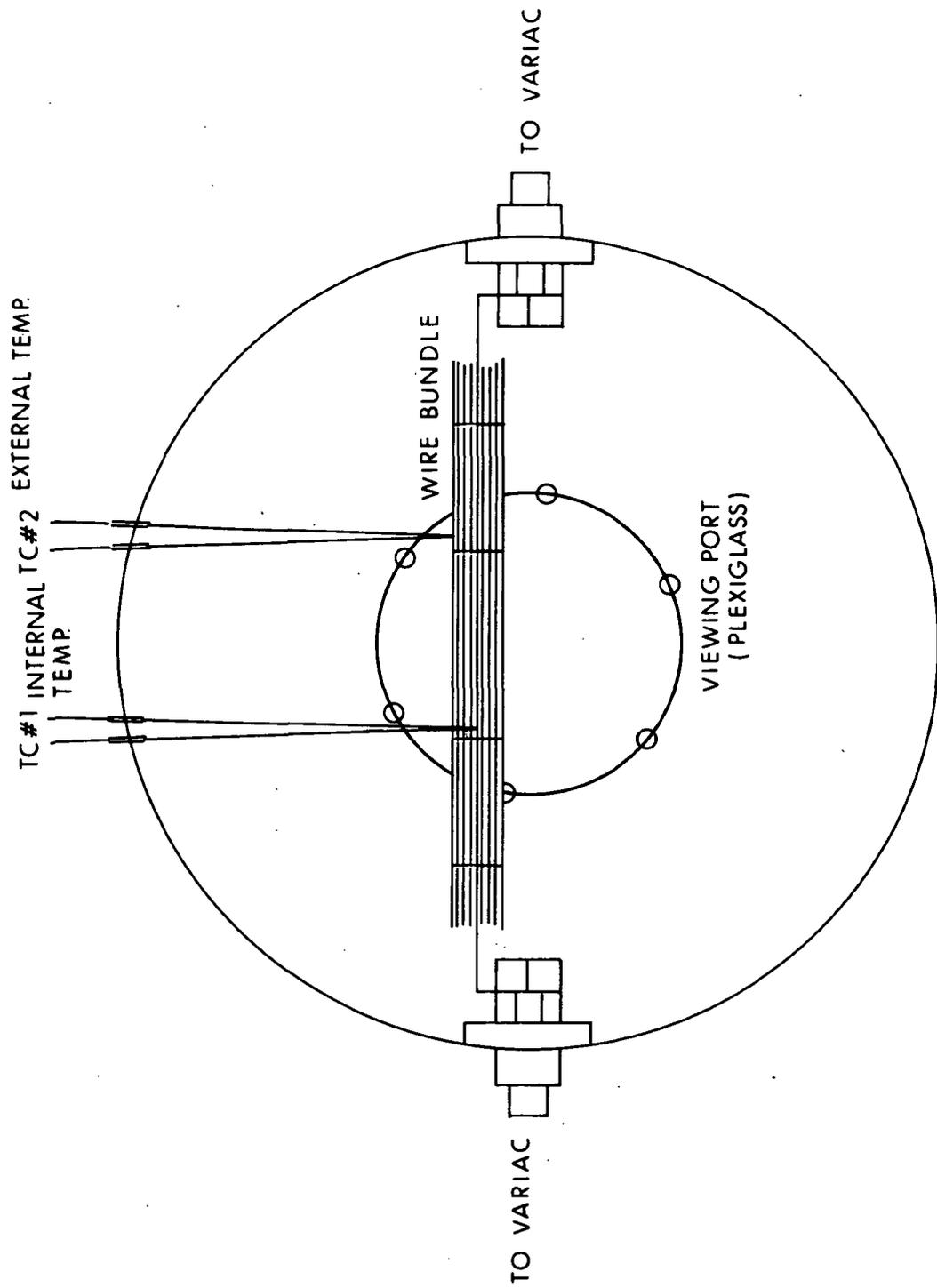


Figure 2. 5 Stainless Steel Reaction Vessel (rear view).

TABLE 2.9

## Bundles of Three Treated Wires with Untreated Sleeve.

Internal Temp. (°F)	External Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Volts	Time (min)	Observations
<u>Run 1 - Dynamic System, Oxygen at 6 L/min at 1/3 atm.</u>						
-----	Ambient	10	0		0	-----
	100	11	34		0.5	No change
	125	11.5	34		1.0	Discoloration
	130	17.0	34		3.5	Smoking
	200	17.5	40		4.0	Heavy Smoking
	250	18	40		5.0	Ignition-Burning
<u>Run 2 - Static System</u>						
-----	Ambient	10	0		0	-----
	145	10	35		0.5	Discoloration
	175	10.2	34		1.0	Brown Discoloration
	250	11.1	34		1.5	Ignition-Explosion
<u>Run 3 - Static System</u>						
Ambient	Ambient	10	0		0	-----
150	105	10	5		4.5	No change
250	150	10	15.5		10.5	No change
375	200	10	20.0		14.5	No change
600	275	10.3	25.0		19.0	Brown Discoloration
1000	325	11.2	30.0		20.5	Ignition-Explosion
<u>Run 4 - Static System</u>						
Ambient	Ambient	10	0	0	0	-----
200	150	10	25	0.5	1.0	No change
405	250	10	25	0.6	2.5	No change
500	300	10.2	25	0.65	4.5	Brown Discoloration
550	310	10.3	25	0.66	8.0	Brown Discoloration
675	390	10.3	30	0.90	8.5	Smoking
900	450	10.8	30	1.10	9.0	Heavy Smoking
1100	---	11.3	30	---	9.3	Ignition-Explosion

TABLE 2.9 (continued)

Internal Temp. (°F)	External Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Volts	Time (min)	Observations
<u>Run 5 - Dynamic System Oxygen at 3 L/min.</u>						
-----	Ambient	10.0	0	0	0	-----
	150	10.0	30	0.6	0.5	No change
	210	10.2	30	0.75	1.0	No change
	275	10.2	30	0.88	1.5	Brown Discoloration
	340	10.3	30	0.90	2.0	Dark Brown Discoloration
	350	10.3	30	1.10	2.5	Dark Brown Discoloration
	400	10.8	30	1.20	3.0	Ignition-Explosion

TABLE 2.10

## Bundle of Three Treated Wires with Treated Sleeves.

Internal Temp. (°F)	External Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
<u>Run 1 - Dynamic System Oxygen at 3 L/min at 1/3 atm.</u>						
-----	Ambient	11	0	0	0	-----
	150	11.5	20	----	1.5	No change
	160	11.8	20	----	2.5	No change
	---	11.9	20	----	3.0	Thermocouple moved
	---	12.2	20		4.5	No change
		12.5	30		5.0	No change
		13.0	30		5.5	Discoloration
		17.0	30		6.5	Smoking
		19.0	30		7.0	Heavy Smoke
		----	30		7.5	Ignition - Explosion
<u>Run 2 - Static System</u>						
Ambient	Ambient	10	0	0	0	-----
240	100	10	27.5	0.6	0.5	No change
575		10.2	27.5	0.8	2.5	Thermocouple moved, Discoloration
640		10.2	27.5	0.88	4.0	Dark Brown - Light Smoke
705		10.6	27.5	0.95	7.0	Heavy Smoke
740		10.9	27.5	1.0	11.0	Heavy Smoke
875		11.0	30	1.20	13.0	Voluminous Smoke
975		----	30	----	13.25	Ignition - Explosion

d) Bundle of Thirteen Wires

Bundles of 24-gauge polyolefin wires were constructed in a manner similar to that previously discussed. Both treated and untreated wires were tested. The treated wires had one coating of L-1483 retardant. All these runs were carried out under dynamic flow conditions. The results are shown in Tables 2.11 and 2.12.

e) Bundle of Thirteen Wires Sleeved

Bundles of 24-gauge polyolefin wires were constructed in a manner similar to that previously discussed; however, sleeving material was used to contain the wires. After the bundles had been prepared, Thermo-Fit tubing was shrunk on the bundle, forming a snug sleeve. Both treated and untreated wires were prepared in this manner. Wires were coated with one, two, and three coatings of L-1483 retardant. The results of untreated wires are shown in Tables 2.13 and 2.14.

f) Bundle of 43 Wires

A bundle of 43 polyolefin wires of 24-gauge was prepared and tested. These wires were not coated with L-1483. The results of one run of this bundle is shown in Table 2.15.

g) Bundle of 13 Wires with Copper Mesh Sleeves

Two bundles one with 13 and with 3, polyolefin wires of 24-gauge were prepared as before. A copper screening of 16 mesh was wrapped around each bundle forming a sleeve. The test results are shown in Table 2.16.

h) Teflon - Polyamide Insulated Wires

A bundle of 13 wires of 22-gauge Teflon-polyamide insulated wires was prepared and tested. The results are shown in Table 2.17.

Two bundles of 13 wires each of 24-gauge Teflon-polyamide insulated wires were prepared and tested. The results are shown in Table 2.18.

TABLE 2.11

## Bundle of Thirteen Untreated Wires

Internal Temp. (°F)	External Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
<u>Run 1 - Dynamic System Oxygen at 3 L/min at 1/3 atm.</u>						
Ambient	Ambient	10	0	0	0	-----
325	200	10.5	34	0.8	0.5	Smoking
700	300	15.0	34	---	1.0	Heavy Smoke
800	---	17.0	34	---	1.5	Heavy Smoke
						Ignition - Burning
<u>Run 2 - Dynamic System Oxygen at 6 L/min at 1/3 atm.</u>						
---	Ambient	9.1	0	0	0	-----
	110	9.1	30	---	0.5	No change
	140	9.2	30	---	1.0	No change
	170	10.0	30	---	1.5	No change
	200	11.0	30	---	2.0	No change
	200	13.2	30	---	2.5	Discoloration Smoking
	---	15.0	30	---	3.0	Ignition - Explosion

TABLE 2.12

## Bundle of Thirteen Treated Wires

External Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
<u>Run 1 - Dynamic System Oxygen at 6 L/min at 1/3 atm.</u>					
Ambient	9.8	0	0	0	-----
125	9.8	30	0.75	0.5	No change
160	9.8	30	0.80	1.0	No change
205	9.8	30	1.00	1.5	No change
290	11.2	30	1.25	2.0	Discoloration
350	13.2	30	1.50	2.5	Smoking-Dark Brown Discol- oration
400	15.0	30	----	3.0	Ignition - Exploration
<u>Run 2 - Dynamic System Oxygen at 6 L/min at 1/3 atm.</u>					
Ambient	10	0	0	0	-----
105	10	30	0.75	0.5	No change
125	10.2	30	0.90	1.0	No change
	10.2	30	1.10	1.5	Thermocouple moved, Discol- oration
	10.2	30	1.20	2.0	Smoking
	10.5	30	1.35	2.5	Heavy Smoking
	11.0	30	1.50	3.0	Ignition - Explosion

TABLE 2.13

## Bundle of Thirteen Untreated Wires with Sleeve

External Temp. (°F)		Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
Thermo couple	Balco*				
<u>Run 1 - Dynamic System, Oxygen at 3 L/min at 1/3 atm.</u>					
Ambient		9.2	0	0	-----
125	---	9.2	30	0.5	No change
150	---	10	30	1.0	No change
180	---	10.1	30	1.5	No change
200	180	10.2	30	2.0	Discoloration
225	260	10.5	30	2.5	Smoking
---	290	11.8	30	3.0	Charring - Smoking
---	300	12.5	30	3.5	Heavy Smoking
---	---	----	30	4.0	Ignition - Explosion
<u>Run 2 - Dynamic System Oxygen at 3 L/min at 1/3 atm.</u>					
Ambient		10	0	0	-----
130		10	30	0.5	No change
150		10.1	30	1.0	No change
180		10.3	30	1.5	Smoking
250		10.5	30	2.0	Brown Discol- oration
---		11.0	30	2.5	Heavy Smoking
300		12.0	30	3.0	Ignition - Explosion

\* Balco wire was incorporated along the length of wire bundle and gave an average temperature; whereas thermocouple were fixed at mid-length of wires, where thermal effects were more intense.

TABLE 2.14

## Bundle of Thirteen Treated Wires with Sleeve

External Temp. (°F)		Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
Thermo couple	Balco*				
<u>Run 1 - Dynamic Oxygen at 3 L/min at 1/3 atm.</u>					
Ambient	---	10.2	0	0	-----
125	---	10.2	30	0.5	No change
160	---	10.2	30	1.0	No change
200	---	10.2	30	1.5	No change
225	---	10.2	30	2.0	Discoloration
275	230	10.3	30	2.5	Smoking
300	260	10.4	30	3.0	Heavy Smoke
340	---	12.4	30	3.5	Ignition-Fire
<u>Run 2 - Dynamic Oxygen at 3 L/min at 1/3 atm.</u>					
Ambient		10	0	0	-----
110		10	30	0.5	No change
150		10	30	1.0	Discoloration
160		10.4	30	1.5	Sleeving Split Ignition-Spark- ing, burning
<u>Run 3 - Dynamic Oxygen at 3L/min at 1/3 atm.</u> <u>Three coatings of L-1483.</u>					
Ambient		10	0	0	-----
100		10	30	0.5	No change
---		---	30	1.0	No change
180		10.5	30	1.5	Smoking
300		11.0	30	2.0	Ignition - Burning

\*See comment at Table 2.13

TABLE 2.15

## Bundle of 43 Untreated Wires with Sleeve

External Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
Ambient	10	0	0	-----
125	10	30	0.5	No change
160	10.5	30	3.0	No change
200	10.7	30	4.5	Discoloration
200	12.0	30	5.0	Smoking
200	----	30	5.5	Ignition - Burning

TABLE 2.16

## Bundle of Untreated Wires With Copper Mesh Sleeves

External Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
<u>Run 1 - Bundle of 13 Wires With Copper Screening as Sleeves</u>				
<u>Dynamic System Oxygen at 3 L/min at 1/3 atm.</u>				
Ambient	10	0	0	-----
105	10	30	0.5	No change
150	10	30	1.0	No change
200	10	30	1.5	Smoking
275	10	30	2.0	Heavy Smoking
375	11.5	30	2.5	Heavy Smoking
450	15.0	30	3.0	Ignition-Explosion
<u>Run 2 - Bundle of 3 Wires with Copper Braid As Sleeve</u>				
<u>Dynamic System Oxygen at 3 L/min at 1/3 atm.</u>				
Ambient	9.2	0	0	-----
100	9.2	30	0.5	Smoking
130	9.8	30	1.0	Heavy Smoking
250	10.2	30	1.5	Ignition-Explosion

TABLE 2.17

Bundle of 13 Teflon-Polyamide Untreated Insulated Wire,  
22 Guage.

Internal Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
<u>Dynamic System Oxygen at 3 L/min at 1/3 atm.</u>				
Ambient	10	0	0	-----
150	10	0	0.5	No change
250	10.3	30	2.0	No change
300	10.6	30	3.0	No change
350	11.0	30	5.5	No change
360	11.0	30	6.5	No change
360	11.0	30	8.0	No visible effect on wire.

TABLE 2.18

Bundle of 13 Teflon-Polyamide Insulated Wire,  
24 Guage, Untreated.

Internal Temp. (°F)	Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
<u>Run 1 - Dynamic System 3 L/min at 1/3 atm.</u>				
Ambient	10.5	0	0	No change
200	10.5	30	0.5	No change
350	10.5	30	1.0	No change
425	10.5	30	1.5	No change
475	11	30	2.0	Discoloration
550	13	30	2.5	Insulation Melted and exposed wire No burning or explosion
<u>Run 2 - Dynamic System 3 L/min at 1/3 atm.</u>				
Ambient	10	0	0	-----
210	11	30	0.5	No change
300	11	30	1.0	No change
400	10.3	30	1.5	No change
450	11.5	30	2.0	No change
500	13	30	2.5	Wire exposed and shorted, No burn- ing or explosion.

## APPENDIX A

### PYROLYSIS AND TOXICITY OF TEFLON-INSULATED WIRES

#### Experimental Procedure

Electrical wire AWG 22 with Teflon insulation was cut in 1-inch lengths. Each sample was flame-sealed in a small pyrex ampoule. These were then baked for 10 minutes each at 600°F, 700°F, 800°F, 900°F, 1000°F, 1,100°F, and 1,200°F. The latter sample was lost through rupture of the ampoule due to the high pressure exerted by the products of pyrolysis.

Each ampoule was then introduced into a Bendix Time-of-Flight Mass Spectrometer and analyzed semiquantitatively. The results are shown in Table A. 1.

TABLE A.1

Sample	Pyrolized at T, (°F)	Mass Spectrometer Analyses
1" TFE	600	No significant findings
1" TFE	700	No significant findings
1" TFE	800	No significant findings
1" TFE	900	Products of pyrolysis showing. A more accurate Mass Spectrometric analysis should be made. (See Note 1)
1" TFE	1,000	Same products of pyrolysis showing in 2 to 3 times the abundance at 900 degree F. (See Note 1)
1" TFE	1,100	Same products of pyrolysis showing in about 2 times the abundance at 1,000° F. (See Note 1)
1" TFE	1,200	No analysis was possible, as the products of pyrolysis were in such abundance that the ampoule ruptured in the baking oven.

Note 1. Some of the more abundant products of this pyrolytic degradation of the TFE insulation are given in the order of the mass spectrometric peak amplitudes shown:

$\text{SiF}_3^+$ , showing an amplitude larger than that of Nitrogen in atmospheric air. If the partial pressure of this ion were known, and if it were equal to the partial pressure of Nitrogen in atmospheric air (it is believed to be higher), the percentage of this ion would be at least 89%, (Ref. 1). This ion forms from the  $\text{SiF}_4$  molecule, which, in turn is formed by reaction between either  $\text{F}_2$  or HF and the Silicon Dioxide of the pyrex ampoules. The  $\text{F}_2$  originates from the pyrolytic decomposition of the TFE, or the HF originates from reaction between the  $\text{F}_2$  from the TFE and  $\text{H}_2$  made available within the experimental ampoule. (Ref. 2) In either case the experimental evidence points to the availability of a large quantity of F, originating obviously from the TFE. The threshold set by the American Conference On Industrial Hygiene is 3 parts per million, or 2 milligrams of Hydrofluoric Acid per cubic meter of air, for one minute exposure.

- $C_3F_5^+$ , showing one of the many possible degradation products of TFE. This ion would react with moisture of organic living tissue (i. e. , mucous membrane of the human respiratory tract), to form noxious Fluorinated compounds. As it contains an un-even number of Carbon atoms, it would not produce Fluoroacetic Acid. It has been found that Fluorinated Hydrocarbons containing an even number of Carbon Atoms may -upon ingestion- form Fluoroacetic acid, the toxicity of which is very high. Of this Fluoroacetic Acid, the Handbook on Industrial Hygiene and Toxicology, Volume II, Interscience, 1962, states on page 1774: "Fluoroacetic Acid has extraordinary toxicity, having lethal effects in some species at 50 micrograms per 50 kilograms" (of body weight) ... "Symptoms are acute convulsive or cardiac effects, appearing after variable latent period".
- $C_4F_5^+$ , showing another of many possible degradation products of TFE. This ion appears in an abundance of approximately one half of that of the above radical,  $C_3F_5^+$ , and will lead to the formation of Fluoroacetic Acid, upon ingestion, as per the notation made immediately above.
- $C_4F_9^+$ , showing another of the many possible degradation products of TFE. This ion appears in an abundance of approximately one half of that of the ion immediately above,  $C_4F_5^+$ . In as much as it contains an even number of Carbon atoms, it will contribute to the formation of Fluoroacetic Acid, on ingestion, with the added lethal biological effects noted above.
- $C_2F_4$ , Perfluorethelene, showing an abundance similar to that of Oxygen in atmospheric air, i. e. 20% by volume, on the assumption, as stated above, of comparable partial pressures. This, again, being a fluorinated compound having an even number of Carbon atoms, would presumably lead to the formation of Fluoroacetic Acid on ingestion, and, hence, add to the lethality associated with this compound.

Similar information is given by the DuPont Company (Ref. 3) in their Bulletin A-28708, on page 5, Table I, entitled "Weight loss and ventilation recommended for Teflon fabricated resins above 450°F". In this Table, the maximum temperature given is 800°F, whereas in our sampling and baking of Teflon wires we indicated (Table A-1) that at 900°F and above, the Mass spectrometer showed significant products of pyrolysis.

The DuPont Table gives a weight loss of 0.15% at 800°F for Teflon and a recommended air ventilation of 325 cu ft/min/lb of resin for safety. A quick calculation of these data shows:

Weight of products of pyrolysis of Teflon (wire insulation) at 800°F:

$$\frac{0.15 \text{ gms}}{\text{gm hr}} = \frac{0.0025 \text{ gms}}{\text{gm min.}}$$

for which an air ventilation is recommended of

$$\frac{325 \text{ cu ft}}{\text{min lb (of resin)}} \quad , \quad \frac{\text{or } 0.7 \text{ cu ft}}{\text{min gm (of resin)}}$$

Taking an average molecular weight of the products of pyrolysis (see Mass Spectrometer Analysis, above) as 135, the potentially toxic concentration becomes:

$$\frac{2.5 \text{ mg}}{19.8 \text{ liters min gm}}, \quad \text{or} \quad \frac{0.126 \text{ mg}}{\text{L min gm}}$$

or

$$22.8 \text{ pp m (If average Mol Wt} = 135, \\ \text{then } 1 \text{ mg/L} = 181.1 \text{ pp m}).$$

The possibility that the even-numbered products of Teflon pyrolysis will react with moisture to form Fluoroacetic Acid has been indicated above. The Threshold Limit Value for this poison is given as 0.1 mg/cubic meter (see Reference 1, page 1801).

If we assume that the even-numbered radicals of Table A-1 constitute one half of the total radicals produced, the build up of the poison, in one minute, could reach a value of over ten times the Threshold Limit Value. This estimate is obtained on considering that the free volume of the Command Module is of the order of one cubic meter.

It is apparent that specific tests with live animals should be undertaken to confirm or deny the possibility of this toxic hazard stemming from the use of Teflon.

Finally, quoted below are the conclusions on this problem reached by the Occupational Medical Services of MIT in a bulletin revised December, 1961, entitled "Health Hazards Involved in the Use of Teflon".

"Teflon is a polymerized fluorocarbon which has many applications because of its physical, chemical, and electrical properties. For example, it is used in the form of tubing or gaskets for its chemical inertness, and as a coating for wire because of its excellent insulating properties; in the form of an aerosol spray it is used as a lubricant. MIT laboratories are using Teflon in significant amounts and many questions about its toxicity have come to the Occupational Medical Service.

"TOXICITY: Animal studies have shown that at ordinary temperatures, Teflon does not cause skin irritation, nor does it produce ill effects when taken by mouth in small quantities. Experience in industry confirms this observation.

Teflon, when heated, decomposes to products (gasses and fumes) which, when inhaled, may cause toxic symptoms. This decomposition starts about 400°F (204°C); at this temperature the weight loss is only 0.0002% per hour and does not increase significantly until the temperature reaches 680°F (360°C). The products evolved include Hydrogen Fluoride and fully Fluorinated Hydrocarbons, one of which, Octafluoroisobutylene, is reported to be more toxic than Phosgene.

"In serious cases, involving a large exposure to Teflon decomposition products, signs and symptoms, similar to the acute state of severe pneumonia, may occur suddenly or after a delay of several hours. In the event of such an exposure, followed by coughing, chest pain, and shortness of breath, prompt medical attention should be sought.

The most common effect of exposure to heated Teflon is "polyfume fever", which is similar to "metal fume fever", and is characterized by chills and fever occurring several hours after exposure. Symptoms disappear without special treatment within 36 to 48 hours. Inhalation of finely divided unheated Teflon has been reported to produce these same symptoms.

"SOURCE OF EXPOSURE: Since Teflon is potentially harmful at elevated temperatures, any heating process should be regarded as a source of exposure. Soldering of Teflon-coated wires is one possible source. In the Occupational Medical Service laboratory we have demonstrated that substantial amounts of fume may be produced by accidental burn-out of Teflon-coated wires in electrical systems. This could prove hazardous if such wires were used in improperly fused systems in confined spaces. Machining processes such as turning, cutting, grinding, or sawing may produce sufficient heat to cause thermal breakdown or considerable airborne dust and should be regarded as a source of exposure. If castings are to be made with Teflon a potential hazard undoubtedly exists.

"The manufacturers of Teflon state that contaminated cigarettes, cigars, and pipe tobacco may produce ill effects. Contamination of smoking materials by Teflon therefore must be prevented.

PRECAUTIONS: If it is found necessary to machine Teflon frequently or to heat it for a long period of time above 400°F (204°C) the operation should be performed under a suitable exhaust hood or local exhaust system. Use of coolants is advisable in high speed machining. If advice on ventilation is needed, call the Occupational Medical Service. Ext. 2596".

References:

- (1) J. H. Beynon, "Mass Spectrometry and its Applications to Organic Chemistry", Elsevier Publishing Company, 1960. Chapter 10.
- (2) Ibid. Section 9.17
- (3) E. I. DuPont De Nemours and Company, Inc., Plastics Department, "Handling and Use of "Teflon" Fluorocarbon Resins at High Temperatures", Bulletin A-28708.

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