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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

CAMBRIDGE 39, MASSACHUSETTS

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The work was conducted at Parametrics, Inc., Waltham, Massachusetts, under P/O No. IL-B 79285.

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 pyrolisis products of these samples were done by Dr. Paul Vouros, under the direction of Professor Klaus Biemann, Chemistry Department, MIT.

The assistance of Mr. George Reickert and of Mr. Stephen Dismar is gratefully acknowledged for their part in this work during the summer of 1964.

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° 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

	Reference Table		ound ound ound ound ound ound ound ound	11 18
	Sleeve		Wire Wound Wire Wound Wire Wound Wire Wound Unfreated	
ı	Dynamic Oxygen Liter/min		⊣ବ ଉମ ଉବଦେଶଶମନେମ	on on
Environment	Static Oxygen		*** * ** * * * * * * * * * * * * * * *	
7	Ambient		мммм мм	•
	Internal Current		ик ининимининининини	ĸи
Heating	External Tape		ими	
	Open Flame	,	××	
Pressure	(atm)		Ambient Ambient Ambient Ambient Ambient Ambient 1/3 1/3 1/3 4/3 1/3 1/3 1/3 1/3 1/3 1/3 1/3 1/3 1/3 1	1/3
Treatment	L-1483	,	1 coats 3 coat	
Trea	None		* * * * * * * * * * * * * * * * * * * *	MM
	Run No.		44 0222	1-2
	Number of Wires	A. : Polyolefin insulation, #24 AWG		B. Teflon-polyamide insulation Thirteen #22 AWG, 7" Thirteen #24 AWG, 7"

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°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 on 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 repellant 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 trichorethylene 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 Trichlorethylene.

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.

Vapo	r 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			
D	Ignition	Time to	D	Ignition	Time to	
Run	Temp (^O F)	Ignite (sec)	Run	Temp (^O F)	Ignite (sec)	
1	1050	10	1	1100	18	
2	1150	8.	2	1050	. 7	
3	1100	10	3	1100	15	
4	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

		Untreated	Trea	ted
Current (amps)	Temperature (^O F)	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 Dis- coloration	Yellow Dis- coloration	Yellow Dis- coloration

2.2.2 Tests in One-Third Atmosphere of 100% O₂

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_2 at 1/3 Atmosphere.

	Run 1			Run 2	
Temp. (^o F)	Pressure (in of Hg)	Observation	Temp. (^O F)	Pressure (in. of Hg)	Observation
200	10	No change	75	10	No change
300	11	No change	167	10.5	No change
350	4	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	<u>.</u>	Tarry (fusing of insulation)	700	13.5	Black
		0. 1115.01.011,	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
		·			·

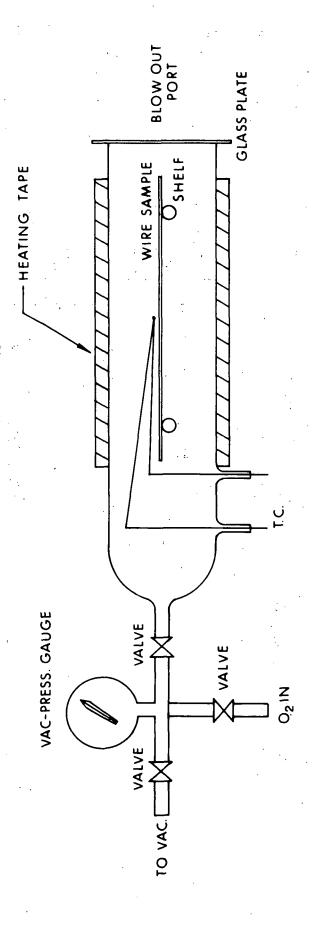


Figure 2.1 Externally Heated Glass Apparatus.

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 ${\rm O_2}$.

Temp. (°F)	Pressure (in. of Hg)	Observations	Temp.	Pressure (in. of Hg)	Observations
Run 1 -	(1 coating o	f L-1483)	Run 3 -	(3 coatings	of L-1483)
75 200 300 400 500 600 700 800 900 950 1000 1050	10 10.5 11.2 11.7 12.0 12.6 13.0 13.7 13.5 13.0	No change No change No change Discoloration Brown Dark Brown Black Black Tarry Tarry-Bubbling Tarry-Bubbling	800	10 11 11,7 12 12,2 12,5 13,0 13,5 13,8 14, 14,2 14,7 15,0 15,0	No change No change No change No change No change Discoloration Brown Dark Brown Dark Brown Brown-Black Black Tarry Tarry Tarry-Bubbling Tarry-Bubbling
Run 2 -	(1 coating o	f L-1483)			
400 450 500 550 600 700 725 800	11, 0 13, 0 14, 0	No change Discoloration Brown Brown Dark Brown Dark Brown Black Tarry			•
850		Tarry-Bubbling	•		

2.3 Final Experiments

2.3.1 Ambient Conditions

Thriteen 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 termperature). 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. (^O F)	External Temp. (^O F)	Current (amps)	Time (min)	Observations
Untreated	1			,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
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
Treated				
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
	And the second second		ř	

2.3.2 Tests in One-Third Atmosphere of 100% O2.

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 forepump. Valve 1 is then closed and 100% O_2 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 guage 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

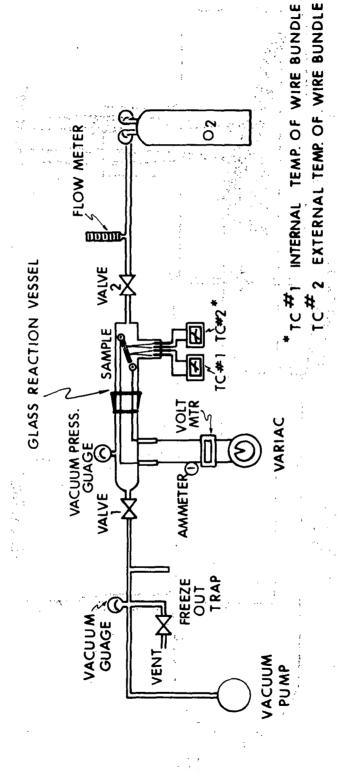


Figure 2.2 Glass Test System.

* 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 ${\rm O}_2$.

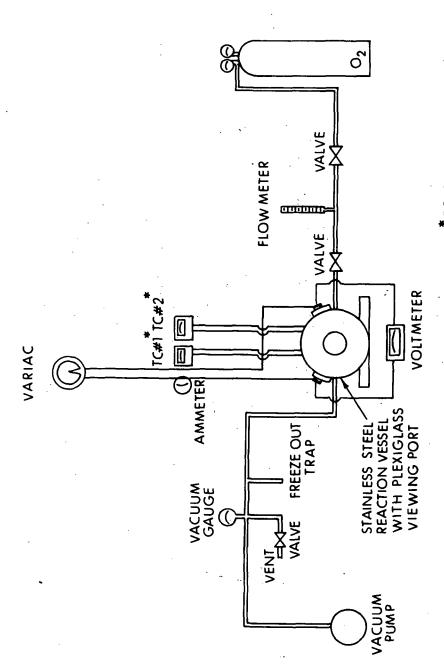
	. <u> </u>		1			
Internal Temp. (^O F)	External Temp.	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
(F)				<u></u>	<u></u>	
Run 1 - S	tatic Test			•	÷	
ı	·	•			,	•
Ambient 140 155 260 500 800 1050	Ambient 140 150 220 375 550 620	10.0 10.2 10.4 10.8 11.7	0 10 14 21 26.5	0 0.2 0.3 0.4 0.7	0 1.5 3.5 5.5 8.0 10.0 10.2	No change No change No change External wire brown Dense Smoke Insulation Charred off-
Run 2 - S	tatic Test					Central wire Glowing, then Melted
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 con- tinuing
650	400	13	0		2.0	Current Shut off, Wire Continued Burning
Run 3 - D	ynamic Te	st, Oxygen at	IL/min.			
Ambient 93 205 550	Ambient 100 150 200	10 10 10.5	0 34 34 	0 1.2 1.2	0 0.2 0.5	Discoloration Heavy Smoke Rapid Ignition and Explos- ion

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-guage polyolefin heated wires, bound with sleeving, were tested under a pressure of 1/3 atm of 100% $\rm O_2$. 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.

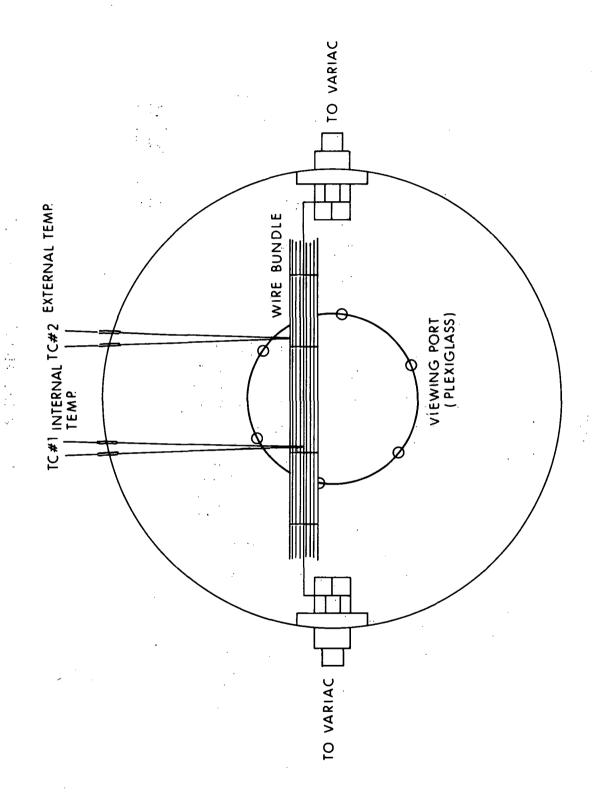


Figure 2. 5 Stainless Steel Reaction Vessel (rear view).

TABLE 2.9
Bundles of Three Treated Wires with Untreated Sleeve.

Internal Temp. (^O F)	External Temp. (^O F)	Pressure (in. of Hg)	Current (amps)	Volts	Time (min)	Observations				
Run 1 - Dynamic System, Oxygen at 6 L/min at 1/3 atm.										
	Ambient 100 125 130 200 250	10 11 11.5 17.0 17.5	0 34 34 34 40 40	-	0 0.5 1.0 3.5 4.0 5.0	No change Discoloration Smoking Heavy Smoking Ignition-Burning				
5 0 0	Ů-		10		0.0	ignition Burning				
Run 2 - S	tatic Syster	<u>n</u>								
	Ambient 145 175 250	10 10 10.2	0 35 34		0 0.5 1.0	Discoloration Brown Discolor- ation Ignition-Explos-				
:						ion				
Run 3 - S	tatic Syster	<u>n</u>				•				
Ambient 150 250 375 600	Ambient 105 150 200 275	10 10 10 10 10 10.3	0 5 15.5 20.0 25.0		0 4.5 10.5 14.5 19.0	No change No change No change No change Brown Discolor- ation Ignition-Explos- ion				
Run 4 - S	tatic Syster	<u>n</u>								
Ambient 200 405 500	Ambient 150 250 300	10 10 10 10.2	0 25 25 25	0 0.5 0.6 0.65	0 1.0 2.5 4.5	No change No change Brown Discol- oration				
550	310	10.3	25	0.66	8.0	Brown Discol-				
675 900 1100	390 450 	10.3 10.8 11.3	30 30 30		8.5 9.0 9.3	oration Smoking Heavy Smoking Ignition-Explos- ion				

TABLE 2.9 (continued)

Internal Temp. (^O F)	External Temp. (^O F)	Pressure (in. of Hg)	Current (amps)	Volts	Time (min)	Observations
Run 5 - D	ynamic Sys	stem Oxygen	at 3 L/min	1,		
				_		
	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 Discol-
, :	340	10.3	30	0.90	2.0	oration Dark Brown Discoloration
	350	10.3	30	1.10	2.5	Dark Brown Discoloration
ji v	400	10.8	30	1.20	3.0	Ignition-Explos-

TABLE 2.10

Bundle of Three Treated Wires with Treated Sleeves.

Internal Temp. (°F)	External Temp.	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
Run 1 - D	ynamic Sys	tem Oxygen	at 3 L/mi	n at 1/3	atm.	
	Ambient 150 160	11 11.5 11.8 11.9 12.2 12.5 13.0 17.0 19.0	0 20 20 20 20 30 30 30 30 30	0	0 1.5 2.5 3.0 4.5 5.0 5.5 6.5 7.0 7.5	No change No change Thermocouple moved No change No change Discoloration Smoking Heavy Smoke Ignition- Explosion
Run 2 - S	tatic Syster	n.				2. probron
Ambient 240 575	Ambient 100	10 10 10.2	27.5 27.5	_	0 0.5 2.5	No change Thermocouple moved, Dis-
640		10.2	27.5	0.88	4.0	coloration Dark Brown -
705 740 875		10.6 10.9 11.0	27.5 27.5 30		7.0 11.0 13.0	Light Smoke Heavy Smoke Heavy Smoke Voluminous Smoke 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. (^O F)	External Temp.	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
Run 1 - 1	Dynamic Sy	stem Oxyger	at 3 L/m	in at 1/3 a	atm.	
Ambient 325 700 800	Ambient 200 300 	10 10.5 15.0 17.0	0 34 34 34	0 0.8	0 0.5 1.0 1.5	Smoking Heavy Smoke Heavy Smoke Ignition - Burning
Run 2 -	Dynamic Sy	stem Oxygen	at 6 L/mir	n at 1/3 a	tm.	
	Ambient 110 140 170 200 200	9.1 9.1 9.2 10.0 11.0 13.2	0 30 30 30 30 30 30	0	0 0.5 1.0 1.5 2.0 2.5	No change No change No change No change No change Discoloration Smoking Ignition - Explosion

TABLE 2.12

Bundle of Thirteen Treated Wires

External Temp. (^O F)	Pressure (in. of Hg)	Current (amps)	Voltage (volts)	Time (min)	Observations
Run 1 - Dy	namic System	n Oxygen at	6 L/min a	t 1/3 atm.	
					
Ambient	9.8	₂ 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
400	15.0	30		3.0	Brown Discol- oration Ignition - Exploration
Run 2 - Dy	namic System	Oxygen at	6 L/min a	$t_1/3$ atm.	ť
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-
	10.0	9.0	1 00	9.0	oration
	10.2	30	1.20	2.0	Smoking
	10.5 11.0	30 30	1.35 1.50	2.5 3.0	Heavy Smoking Ignition - Explosion

TABLE 2.13

Bundle of Thirteen Untreated Wires with Sleeve

External (^O F	_	Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
Thermo couple	Balco*	(1 . 0. 1.g,	(amps)	(111211)	•
Run 1 -	Dynamic :	System, Oxyg	gen at 3 L/n	nin at $1/3$	atm.
Ambient		9,2	0 .	0	
125		9.2	30	0.5	No change
150	222	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
Run 2 - Ambient	Dynamic	System Oxyge	en at 3 L/m	in at 1/3 a	atm.
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
	<u>_</u>	11.0	30	2.5	Heavy Smoking
300		12.0	30	3.0	Ignition - Explosion
					Explosion

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

TABLE 2.14

Bundle of Thirteen Treated Wires with Sleeve

The	ernal Temp. (^O F) rmo Balco* uple	Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
Run	1 - Dynamic	Oxygen at 3 L	/min at $1/3$	atm.	\$) -
Amk 125 160 200 225 275 300 340		10.2 10.2 10.2 10.2 10.2 10.3 10.4 12.4	0 30 30 30 30 30 30 30	0 0.5 1.0 1.5 2.0 2.5 3.0 3.5	No change No change No change No change Discoloration Smoking Heavy Smoke Ignition-Fire
Run	2 - Dynamic	Oxygen at 3 L	/min at 1/3	atm.	
Amb 110 150 160	pient	10 10 10 10.4	0 30 30 30	0 0.5 1.0 1.5	No change Discoloration Sleeving Split Ignition-Spark- ing, burning
Run	3 - Dynamic Three co	Oxygen at 3L/ atings of L-14	min at $1/3$	atm.	
Amb 100 180 300	oient	10 10 10.5 11.0	0 30 30 30 30	0 0.5 1.0 1.5 2.0	No change No change Smoking Ignition - Burning

^{*}See comment at Table 2.13

TABLE 2.15

Bundle of 43 Untreated Wires with Sleeve

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

TABLE 2.16

Bundle of Untreated Wires With Copper Mesh Sleeves

External Temp.	Pressure	Current	Time	Observations
(°F)	(in. of Hg)	(amps)	(min)	•
Run 1 - Bundle o				
Dynamic	System Oxyge	n at 3 L/m	in at $1/3$	atm.
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	1.0	30	2.0	Heavy Smoking
375	11.5	30	2.5	Heavy Smoking
450	15.0	30	3.0	Ignition-Explosion
Run 2 - Bundle o	of 3 Wires with	Copper Br	aid As Sle	eeve
	System Oxyge			
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. (^O F)	Pressure (in. of Hg)	Current (amps)	Time (min)	Observations
Dynamic System	Oxygen at 3 L	/min at 1/3	3 atm.	
Ambient 150 250 300 350 360	10 10.3 10.6 11.0 11.0	0 0 30 30 30 30 30	0 0.5 2.0 3.0 5.5 6.5 8.0	No change No change No change No change No change No change eno visible effect on wire.

TABLE 2.18

Bundle of 13 Teflon-Polyamide Insulated Wire,

24 Guage, Untreated.

Internal Temp.	Pressure	Current	Time	Observations
(⁰ F)	(in, of Hg)	(amps)	(min)	
Run 1 - Dynami	c System 3 L/	min at 1/3	atm.	
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 wir
				No burning or
	·			explosion
Run 2 - Dynami			•	
Ambient	10	0	0	explosion
Ambient 210	10 11	0 30	0 0.5	explosion No change
Ambient	10 11 11	0 30 30	0 0.5 1.0	explosion No change No change
Ambient 210 300	10 11	0 30	0 0.5	explosion No change

APPENDIX A

PRYOLISIS 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 pyrolisis.

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, (^O 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 pryolisis showing. A more accurate Mass Spectrometric analysis should be made. (See Note 1)
1" TFE	1,000	Same products of pyrolisis showing in 2 to 3 time the abundance at 900 degree F. (See Note 1)
1" TFE	1,100	Same products of pyrolisis showing in about 2 times the abundance at 1,000°F. (See Note 1)
1" TFE	1,200	No analysis was possible, as the prod- ducts of pryolisis were in such abund- ance that the ampoule ruptured in the baking oven.

- Note 1. Some of the more abundant products of this pyrolitic degradation of the TFE insulation are given in the order of the mass spectrometric peak amplitudes shown:
- 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 SiF $_4$ molecule, which, in turn is formed by reaction between either F_2 or HF and the Silicon Dioxide of the pyrexampoules. The F_2 originates from the pryolitic decomposition of the TFE, or the HF originates from reaction between te F_2 from the TFE and F_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_2 , 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.

- C₃F₅, 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 Fluoracetic Acid. It has been found that Fluorinated Hydrocarbons containing an even number of Carbon Atoms may upon ingestion-form Fluoracetic acid, the toxicity of which is very high. Of this Fluoracetic Acid, the Handbook on Industrial Hygiene and Toxicology, Volume II, Interscience, 1962, states on page 1774: "Fluoracetic 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 Fluoroactic Acid, oningestion, with the added lethal biological effects noted above.
- C₂F₄, 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 $450^{\circ}F$ ". In this Table, the maximum temperature given is $800^{\circ}F$, whereas in our sampling and baking of Teflon wires we indicated (Table A-1) that at $900^{\circ}F$ and above, the Mass spectrometer showed significant products of pyrolisis.

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 pyrolisis 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

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

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

or

The possibility that the even-numbered products of Teflon pyrolisis 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 of 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 $400^{\circ} F$ (204°C); at this temperature the weight loss is only 0.0002% per hour and does not increase significantly until the temperature reaches $680^{\circ} F$ (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 dissappear 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 hazadous 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 $400^{\circ}F$ ($204^{\circ}C$) 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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