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# Technical Report No. 1

EVALUATION OF THIN WALL SPACECRAFT WARING Volume I: Test Methods and Facilities

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### TECHNICAL REPORT NO. 1

### EVALUATION OF THIN WALL SPACECRAFT ELECTRICAL WIRING

### VOLUME 1: TEST METHODS AND FACILITIES

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Report Prepared for:

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# Volume I

# TABLE OF CONTENTS

Page No.

I.	OBJECTI	VE 1	
II.	EVALUAT	ION PROGRAM	
	A. Gen	eral	
	B. Tes	t Methods	
	1.	Insulation Resistance - Total Sample	
	2.	Voltage Withstand	
	3.	Insulation Resistance	
	4.	Corona	
	5.	Voltage Breakdown	
	6.	Voltage Flashover	
	7.	Outside Diameter	
	8.	Concentricity	
	9.	Conductor Dimension	
	10.	Weight Per 1000 Feet	
	11.	Stripability	
	12.	Solderability	
	13.	Color Durability	
-	14.	Marking Legibility	
	15.	Compatibility with Potting Compounds	
	16.	Flexibility13	
.*	17	Scrape Abrasion	
	18.	Blocking	
	19.	Cut-Through	
	20.	Thermal Creep	
	21.	Wicking	
	22.	Thermal Aging	
	23.	Exposure to Ultra-Violet Radiation	
- · · · ·	24.	Exposure to X-Ray Radiation	
	25.	Flammability	
	26.	Chemical Compatibility	
	27.	Offgassirg in 5 psia Oxygen	
	28.	Vacuum Volatility	
	-	•	

#### LIST OF FIGURES

PAGE NO. 1 Cabled Specimen for Insulation Resistance and Corona Measurement 40 Chamber for Aging Cabled Specimens at 50°C and 100% 2 RH plus Dew in Pure Oxygen at 15 psia . . 41 3 Cabled Specimen Mounted in High Voltage Cell . . . 42 4 Jig for Forming Twisted Pair Specimens 43 5 High Voltage Cell for Voltage Breakdown Measurments in Oxygen and in Vacuum. . . . . . . . . . . . . . 44 Cross Sectional View of Sec-Up for Electric Strength 6 45 7 Cell for Flashover Measurement in Wet Oxygen at 5 psia 46 8 Typical X-ray of Wire Specimens (5X Magnification) . . 47 9 Wire No. 3 After Thermal Stripping . . . . . 48 10 Electrical and Mechanical Specimens for Potting Compound Compatibility Tests . . . . . . . 49 Potting Compound Compatibility Specimens being Cured 11 50 12 Repeated Reversed Mandrel Flexibility Test Apparatus for use with 0.030" to 0.125" Diameter Mandrels. . . . 51 13 Repeated Reversed Mandrel Flexibility Test Apparatus for use with 1/4" to 1 3/4" Diameter Mandrels. . . . 52 14 MIT Fold Endurance Flex Tester ..... 53 Loading Nose for MIT Fold Endurance Flex Tester. . 15 .54 16 Dimensions of Modified Loading Nose for MIT Flex 55 56 17 NEMA Scrape Abrasion Tester. . . . . . . 18 Close-up of Scrape Abrasion Head . . . . . . . . 57 19 Compressions Cage Equipped for Thermal Creep Test. . 58 20 Instron Testing Machine with Conditioning Chamber for 59 Thermal Creep and Cut-Through Tests. . . . . . . Wicking Specimens in Fluorescent Dye Solution. . . 60 21 ٠ 22 Specimens for Thermal Aging Tests at 150°C in Vacuum . 61 23 Chamber for Ultraviolet Exposure in Vacuum . . . . . 62 63 24 Test Assembly for Flammability Test. . . . • • • 25 Flammability Test with External Heater . . . . 64 . 65 26 Flammability Test, Voltage Drop Technique. . . . 27 Measured Temperature vs. Resistance (1 inch length) 66 Stranded #20 Nickel Plated Copper Wire • • • • 28 Measured Temperature vs. Resistance (1 inch length) Stranded #20 Silver Plated Wire (wire #6). . . 67 29 Chemical Compatibility Specimens in a Test Fluid . . 68 30 Quartz Spring Balance Assemblies for Measurement of 69 70 31 Quartz Spring Balance Specimen Chamber • • • • • • Schematic Diagram of Quartz Spring Balance • • • • 71 32 33 Flow Diagram Atmospheric Control System for Quartz 72 Spring Balance Apparatus . . . . . . . . . . 34 Sample Holding Furnace Tube for Gas Analysis • • • 73 Balance System Showing Vacuum Station and Associated 35 Recording and Power Supply Equipment 74 Recording Microbalance and Furnace Used in Vacuum 36 75 Vulatility Study

FIGURE NO.

#### EVALUATION OF THIN WALL SPACECRAFT ELECTRICAL WIRING

#### I. OBJECTIVE

The objective of this program is to determine the performance characteristics of various thin wall, spacecraft, electrical wiring under simulated spacecraft environments. The data will permit wire selection for manned spacecraft to be made on the basis of comparative performance. Further, recommendations will be made regarding the development of specifications for comparative evaluation and qualification testing of manned spacecraft electrical wire insulation.

### 11. EVALUATION PROGRAM

#### A. General

Approximately 1000 feet of each wire type is required to fabricate the various types of specimens that are used in the program. The samples were purchased directly from each wire manufacturer. It was specified that the wire be spark tested and that each reel contain only one piece, with both ends accessible.

Upon receipt of each sample, a sufficient quantity of wire was set aside for the specimens that are used in the experiments on off gassing in oxygen, volatility in vacuum, gas analysis and markability. The remainder of each sample was immersed in distilled water for the three day insulation resistance and voltage withstand measurements.

The evaluation program consists of the following tests:

#### Electrical Tests

Insulation Resistance	-	Total sample immersed in water at 23 <sup>°</sup> C
Voltage Withstand - 1600 volts for 1 min.	-	Total sample immersed in water
Insulation Resistance*	-	As a function of exposure time at 100% RH + dew in 15 psia pure oxygen at 50°C
Corona Start Voltage	-	In 5 psia pure dry oxygen at $93^{\circ}$ C and in 15 psia 0 <sub>2</sub> at 100% RH + dew
Voltage Breakdown	•	In wet oxygen at 5 psia and 23 <sup>°</sup> C, and at 150 <sup>°</sup> C in vacuum, 10 <sup>-6</sup> torr
Voltage Flashover	<b>,</b>	In 5 psia pure oxygen at 23 <sup>0</sup> C and 100% RH + dew.

\*Note: Insulation Resistance and voltage breakdown are used as end point criteria of certain other tests.

### Mechanical Tests

Outside Diameter	-	at 23C and 50% RH
Concentricity of Insulation	-	fi 12 12 12 12 12
Conductor Dimensions	-	17 11 11 11 11
Weight per 1000 ft.	-	H 4 H H A S
Stripability	-	11 11 11 11 12
Solderability	-	Solder pot at 320 <sup>0</sup>
Color Durability	-	See Test Method
Marking Legibility	. <b>-</b>	See Test Method
Compatibility with Potting Compounds	•	See Test Method
Flexibility*	. •	At 23°C and -196°C
Abrasion	· •	At 230
Blocking	· -	150°C and 10 <sup>-6</sup> torr
Cut-through	-	23°C and 150°C
Thermal Creep ("Cold" Flow)	۰ ۱ <b>۰</b>	23°C and 150°C
Wicking	-	In water at 23 <sup>0</sup> C

\*Note: Flexibility is used as an end point criterion of certain other tests.

## Physical - Chemical Tests

Thermal Aging	-	At 150 <sup>0</sup> C in oxygen at 15 psia and in vacuum.
Exposure to Ultra-Violet	-	Approx. 1.4 x $10^6$ ergs/cm <sup>2</sup> /sec/ $\mathcal{M}$ equiv. at 4000 A for 1 month At 85C in wet oxygen at 15 psia and at 150C in vacuum.
Exposure to Radiation	-	10 hrs. at 6000 rads/hr at $150^{\circ}$ C and $10^{-6}$ torr and 100 rads/hr at 93C in 5 psia pure $0_2$
Flammability Smoke, flash and fire points	-	In wet flowing oxygen at 5 psia.
Chemical Compatibility	-	(See Test Method)

### Analytical Tests

Offgassing	in	Oxygen	-	TGA	and	Analysis	of	Gases	6
Volatility	in	Vacuum	-	TGA	and	Analysis	at	10 <sup>-7</sup>	torr

### B. Test Methods

### 1. Insulation Resistance - Total Sample

This test is one of the two screening tests that are performed on the total sample. The full length of the test sample (except for the ends, which are suitably protected) are immersed in water. The DC resistance between the conductor and the water is measured at 500 volts after 1 hour, 1 day and 3 days. The measurements are made with a Kiethly electrometer after a one-minute electrification time. If a specimen fails to meet the acceptance criterion  $(3 \times 10^{10} \text{ ohms per 1000 feet})$ , the electrification time is increased to 5 minutes. The 5 minute value will be significantly greater than the one minute value if the conduction is not associated with the absorption of water into the insulation structure, either in a fault or throughout the volume of the insulating wall.

#### 2. Voltage Withstand

At the conclusion of the insulation resistance test, a voltage of 1600 volts RMS at 60 cps is applied for one minute. If failure occurs, the failure point (or points) are removed from the water and the test is repeated for one minute.

Only those portions of the wire sample which pass the voltage withstand test and have a resistance greater than 30,000 megohms per 1000 feet after 3 days immersion are used for further evaluation.

#### 3. Insulation Resistance

A cabled test specimen is used to determine the effect of exposure at  $50^{\circ}$ C and 100% RH + dew in pure oxygen at 15 psia on insulation resistance. The cabled specimen consists of six wires that are wrapped around a seventh central wire, as shown in Figure 1. Fabrication is accomplished by passing the bundled wires through a circular guide, which will allow the wires to pass through only if they are in the proper configuration. The cabled wires are held in place with short lengths of heat-shrinkable tubing. The cabled specimens are mounted in chambers by inserting them in holes in the chamber walls, as shown in Figure 2.

-3-

RTV silicone rubber is used to seal the holes after the wires are in place. This scheme permits the ends of the specimens to remain outside of the chamber and eliminates the need for feed-through bushings that would introduce undesirable leakage currents when wet.

A heated water bath is located in the bottom of each chamber. The whole arrangement is placed in an oven and maintained at  $50^{\circ}$ C. The additional heat in the water bath provides the required 100% RH  $\pm$  dew. The chamber is purged and filled with oxygen at 15 psia and twice each day fresh oxygen is allowed to flow through the chamber.

Insulation resistance is measured between the six outer wires, which are connected together, and the central wire. Again, the Kiethly electrometer is used with a one minute electrification time.

### 4. Corona

At the conclusion of the insulation resistance measurement, corona measurements are made on the cabled specimens before they are removed from the conditioning chamber -  $(50^{\circ}C, 100\% \text{ RH}, 15 \text{ psia } \Omega_2)$ . The specimens are then removed and tested in a high-voltage cell, which is described below, in dry oxygen at 5 psia. Figure 3 shows the method of connecting the cabled specimens.

Corona tests are made according to ASTM D1868-61T. The high frequency components of the corona are amplified and detected using a 100 kc resonant circuit and a wide band amplifier with a 300 kc high frequency and a 15 kc low frequency cut off. The signals are displayed on an oscilloscope and the variation of the amplitude of the detected signa' with applied 60 cycle voltage is noted. The voltage is increased slowly and the corona initiation voltage (C.I.V.) is noted. Then the voltage is increased about 20% above the C.I.V. and then decreased slowly and the corona extinction voltage (C.E.V.) is noted. The C.E.V. is considered to be more significant than the C.1.V. for it is the highest voltage at which corona will persist if it is ever initiated by a momentary high voltage surge.

Whenever the C.E.V. is lower than expected, a resistivity check is made after the completion of the corona test to determine if a failure has occurred.

-4-

### 5. Voltage Breakdown

Voitage breakdown tests are made on NEMA twisted pur specimens, which provide positive pressure at the cross-overs. The specimens are fabricated with a special jig as shown in Figure 4.

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The test cell consists of a vacuum tank, 10.5 inches in diameter which is fitted with a cover containing ten feed-throughs. Figure 5 shows the chuber and the associated vacuum system, Figure 3 shows details of the feed-throughs, which are made with Veeco 1/4 inch diameter, 0-ring type C-25 quick vacuum couplings. Eighteen inch lengths of silicone insulated solid copper wire are passed through each coupling, with a six inch extension in the tank. This arrangement provides a flashover strength of at least 25 kv, even at the reduced pressure of 5 psia. These feed-throughs are corona-free at voltages that are higher than the corona start voltages of the cabled specimens. For tests in vacuum, flashover is not a problem and the six inch extension can be reduced to one inch to minimize the amount of organic material in the chamber.

For tests of twister pair specimens in 5 psia  $\theta_2$  is necessary to minimize the chance for flashower between leads of the twisted pairs and also between the high voltage connection to the twisted part and the tank.

The twisted pairs were made with long leaf  $s \in t$  sions so that the opposite ends of each wire could be brought cogether have a good separation between the ends of the two wires could be obtained.

Figure 6 is a sketch of the twisted pair in the tank. It will be noted that each specimen is surrounded with a pyrex tube. The primary function of the pyrex tube is to prevent breakdown to the tank. It also shields the specimens from the breakdown products of other specimens and diminishes fire hazards.

For the tests at 5 psia  $0_2$ , a vacuum of less than 50 microns was first established in the tank. Then  $0_2$  was bubbled through water at  $30^{\circ}$ C into the tank and pressure adjusted.

For the tests in vacuum, a diffusion pump was used to establish a vacuum of  $10^{-5}$  torr or less at a temperature of 150C.

-5-

The tank was grounded and one wire of twisted pair was connected to it. Sidey cycle voltage was increased at a 500 volts/sec. rate and at a 100 volts/sec. rate. Nine specimens were tested at each rate. In order to conserve time, on the 100 volt/sec. tests, the voltage was raised quickly to 0.5 times the minimum breakdown strength noted on the 500 volt/sec. test and 100 volt/sec. rate was started at this voltage. This should have a negligible effect on the breakdown voltage at the slow rate of rise.

#### 6. Voltage Flashover

Voltage failure may occur along the surface of hook-up insulation (voltage "flashover") as well as through its volume (voltage "breakdown"). For this program a .010 inch diameter nichrome wire was wrapped around the hook-up wire (approximately 5 turns) to serve as an electrode and was accurately spaced 3/16" from the cut end of the insulation at the end of the wire. Conventional mechanical wire strippers could not be used to strip the insulation from the wire since they damaged some of the insulations used. Instead the insulation was carefully removed from the end of the wire with a razor blade.

Voltage flashover tests (see Figure 7) were made with the test specimen in flowing, wet oxygen at 5 psia absolute. The 60 cps voltage was raised so that flashover occurred in about 1 minute. The voltage at flashover was noted. A 50,000 ohms resistance was used in series with the specimen so that flashover current continued to flew until voltage was removed. In this way the tendency of the surface to track or to flame could be evaluated and visual results reported.

### Significance and Use

If a crack or other type of discontinuity is present in hook-up wire or develops during installation or use, dielectric failure can occur along the surface of the wire from the conductor at the point of the discontinuity to ground or to another component. Since the flashover voltage is reduced at low pressures it is important to determine its value under the worst conditions of use. In this program, 5 psia wet oxygen is considered to be the worst service condition, although it should be noted that the somewhat lower pressures which might occur on depressurization of a capsule in space could result in considerably lower flashover voltages.

Even though normal voltages in spacecraft wiring may be quite low, it should be recognized that considerable overvoltage can develop in switching, particularly with inductive circuits. Once an arc has been established it may persist even at very low voltages.

Unfortunately, surface arcs tends to carbonize or "track" many insulation surfaces so that permanent damage occurs. Moreovor, the high temperature arc may readily ignite flammable insulating materials.

-7-

#### 7. Outside Diameter

Ten pieces of wire, 26.46 inches long, are removed at random intervals during the preparation of specimens These pieces are first used for weight determination (see Section 10 below) and then for dimension measurements. One piece, six inches long, is cut from each of the randomly selected weight specimens. The outside diameter is measured with a hand micrometer at three locations on the specimen. Two measurements, at right angles to each other, are made at each of the three locations, giving r total of six measurements for each specimen, or 60 measurements for the entire sample.

The average, maximum and minimum outside diameter is reported for each specimen. These values are then averaged for the ten specimens.

The same samples are then examined by industrial x-ray techniques. The specimens are mounted so as to provide an undistorted image on x-ray film. An enlarged portion of a typical x-ray photograph is shown in Figure 8. Additional measurements of outside diameter, as well as other dimensions, are made by examining the x-ray with a measuring microscope.

The same specimens are also viewed with an optical comparator to give additional measurements of maximum and minimum outside diameter.

### 8. Concentricity

The x-ray examination yields data on insulation wall thickness. Six measurements are made for each specimen. Concentricity is defined as the ratio of the minimum wall thickness to the maximum wall thickness, expressed as a percentage. The percent concentricity for each specimen is reported, and the average value of the ten specimens is computed. The acceptance criterion is 80%.

#### 9. Conductor Dimension

The overall conductor dimensions are determined from the x-ray examination, as described above. Such measurements permit a determination of the degree of bunching in the stranding. In addition, the wire insulation is stripped and the overall stranded diameter, as well as the diameter of the individual wires, is measured with a hand micrometer.

### 10. Weight Per 1000 Feet

Ten randomly selected specimens are cut to a length of 26.46 inches.

-8-

The length is determined by stretching the wire over a precisely machined bar of the proper length Uniform tension among specimens is achieved by hanging a 3 lb. weight from one end of the wire.

Each specimen is wiped with a lint-free cloth and rolled into a small coil. A Mettler balance is used to weigh the specimens to the nearest 0.1 mg. The weight in grams of the 26.46 inch specimen is equivalent to the weight in pounds of a 1000 foot specimen.

### 11. Stripability

Ten test specimens approximately six inches long are evaluated. A properly adjusted precision cutting type wire stripper is used to strip one inch of insulation from each end of the specimen. Where appropriate, a thermal type, hot wire stripper is used.

The ease of stripping is reported and the exposed conductor is examined under a stereomicroscope with at least 5X magnification for evidence of mechanical damage such as nicks and scrapes. After thermal stripping, change in color or other evidence of thermal damage is locked for. After examining the bunched stranding it may be useful to separate the stranding to permit additional observation. Lighting of the test specimen is critical and the microscope lamp is moved into various positions during the visual study.

If nicks or scrapes are observed on the conductor after the use of a properly adjusted mechanical stripper, photomicrographs of the defect will be made with adequate magnification (at least 5X).

The cut ends of the insulation are also examined for damage caused by crushing in the jaws of the mechanical stripper or by heating in the case of the thermal stripper.

Permanent records of damage to conductor or insulation may be made with photomicrographs (at least 5X), such as that shown in Figure 9.

### 12. Solderability

A six-inch wire test specimen with 1/2 inch of insulation removed from one end is bent around a mandrel of its own diameter at a point 1/2 inch back from the cut edge of the insulation so that an approximate  $90^{\circ}$  bend in the wire is maintained. The stripped conductor is immersed in molten solder to a

**-9**-9

depth of 3/8 inches, or within 1/8 inch of the insulation, and held for 5 seconds. Upon removal, the specimen is examined for evidence of insulation damage at the cut edge and the bent section. Observation of discoloration, melting, shrinking, cracking, splitting, flaring or charring are reported.

The 60/40 (tin/lead) solder is heated to  $320^{\circ}$ C in a solder pot and the temperature is controlled to  $\pm 5^{\circ}$ C. In those cases where the solder does not wet the conductor, a suitable flux is applied before dipping. This insures adequate thermal contact and increases the heat ransfer to the conductor and, hence, to the insulation.

13. Color Durability

Evidence and extent of visual color change is determined after exposure to the various environmental conditions of the program. Comparisons are made with unemposed specimens. If a color change is noted, the specimen is retained.

Special attention will be paid to color characteristics after thermal ageing, ultra-violet and radiation exposure.

14 Marking Legibility

Marked specimens are to be provided by the NASA Manned Spacecraft Center. Specimens are prepared by the Kingsley Machine Company, Hollywood, California.

Approximately 100 feet of each sample is shipped to Kingsley for marking. Twenty feet is for experimenting by Kingsley; the remainder is required for use as test specimens.

Marked test specimens are subjected to a 1600 volt withstand test after immorsion in water for 24 hours. After passing the voltage withstand test, the marked area of the test specimen will be exposed to human perspiration, preferably by wiping several times over the outside of the nose. The marked area of the wire will then be pulled between the thumb and forefinger 10 times, while held tightly together in an effort to remove the marking. The legibility of the marking to the naked eye after test will be reported.

Marked test specimens will also be subjected to the thermal ageing ultra-vilot and chemical compatibility tests. The legibility of the markings to the naked eye after such exposure will be reported.

-10-

### 15. Compatibility with Potting Compounds

Two types of specimens, a mechanical and an electrical, are used to test. (a) the mechanical bond between wire and potting compounds, and (b) the resistance to moisture penetration along the interface between the wire insulation and the resin.

The mechanical specimen consists of a straight piece of wire 36 inches in length with one end potted. Four wires are potted in a mol- that consists of a one inch diameter pill-box with masking tape applied to increase the depth to about 3/4 inches. One end of each wire is inserted in a hole in the bottom of the mold. This holds the wires in position during the potting operation. The mold is removed before the specimen is aged for testing. Figure 10 shors both the mechanical and electrical specimens, with and without molds.

The mechanical specimens are aged for 15 days at  $150^{\circ}$ C in pure oxygen at 15 psia before testing. In the mechanical bond test, the wire is attached to the upper crosshead of an Instron testing machine by making three turns around a capstan. The potted end of the specimen is restrained by a fork that is attached to the lower crosshead. With a crosshead speed of one inch per minute, the wire is loaded until: (a) the wire is pulled completely free of the potting compounds, (b) the compound fails in shear, or (c) the wire fails in tension. The maximum load carried by each wire joint is reported.

The electrical specimen consists of a twisted pair (see page 5) for description) that is potted in a cylindrical mold 3/4 inches in diameter and  $4\frac{1}{2}$  inches long. The mold is formed by wrapping aluminum foil around a glass test tube and then removing the test tube. Two electrical specimens, with and without the aluminum mold, are shown in Figure 10. For the purpose of this test, the twisted pairs are cut in the center of the lwisted portion and the ends are bent back to provide adequate spacing between the bare conductor ends.

A duplicate set of electrical specimens is prepared using twisted pairs that are nicked with a razor blade. The blade is drawn across both wires so that the nicks are adjacent to each other.

The electrical specimens are aged for 15 days at 150°C in pure oxygen at 15 psia. They are then immersed in water at room temperature for three days. The free ends of the wires are held out of the water to avoid wicking along the conductors. At the end of the three day immersion period, insulation

-11-

resistance is measured between the wires and the water bath. The specimens are then removed from the water bath and tested to voltage breakdown. These tests will detect the penetration of moisture along the interface between the wire insulation and the potting compound.

Standard potting techniques are used in fabricating the specimen. Figure 11 shows a group of specimens being cured. Manufacture recommendations are followed in preparing the various wire insulation surface for potting. Detailed descriptions of the potting compounds, etchants and primers are included in the discussion of results.

#### 16. Flexibility

#### a. Mandrel Flexibility Test

The mandrel flexibility test provides a visual indication of the ability of a wire to be flexed around a mandrel of specified size. The test may be performed in several ways. In the simplest way the wire is carefully straightened without elongation and wrapped around itself (1X mandrel). If cracking or delamination of the insulation is noted, the wire may be wrapped around a metal mandrel which is approximately twice the diameter of the wire (2X mandrel). The test is continued with larger and larger mandrels until a mandrel diameter is found with which damage to the insulation is not observed. It is customary to report the mandrel diameter with which no insulation failure is obtained as well as the next smaller diameter with which failure of the insulation is observed. The extent and character of the visual damage to the insulation is reported also.

The uniformity and reproducibility of the mandrel flexibility test may be improved by winding the wire specimen back and forth between two mandrels of the same diameter. A "reverse" bend will take place if the wire is wrapped in opposite rotational direction between two mandrels. A simple mandrel holder for making repeated "reverse" mandrel flexibility tests is shown in Figure 12. The end of the test specimen is first inserted through the hole in one mandrel to hold it in place. The mandrel is then twisted by hand while slight tension is applied to the wire so that a uniform and reasonably tight winding can be formed on the mandrel. If cracking or other insulation damage takes place, the result is reported and a larger mandrel diameter is used. When four to six turns of vire have been applied to one mandrel, the other end of the wire is fastened through the hold of the adjoining mandrel. The wire is then wound from the first to the second mandrel so that the reverse bend takes place. A restraining torque is applied to the first mandrel so that sufficient tension is maintained to produce a uniform winding on the second mandrel. The wire is wound back and forth between the two mandrels five times. If after examination some slight evidence of insulation damage is noted, it may be desirable to continue winding the wire specimen back and forth five times more.

The mendrel assembly shown in Figure 1? is designed to be used at room temperature and also while immersed in liquid nitrogen. The liquid

-13-

nitrogen can be held in a polystyrene foam container as shown in the photograph. In a NASA (Huntsville) program, NAS 8-2442, mandrels for the repeated flexibility test were designed to operate in liquid helium. To reduce the heat leak the mandrels were supported at the end of long stainless steel tubes as shown in Figure 13. Since these mandrels were available in several diameters, chey were used in the subject program even though heat loss is not so important with liquid nitrogen.

At low temperatures some wire insulations may become quite brittle so that large mandrel diameters must be used. For the #20 hook-up wire used in this program, the following mandrel sizes were standardized: The wire itself (1X) and for the repeated mandrel flexing test - .060, 0.125, 0.25, 0.50, 0.75, 1.0, 1.25, 1.50, 1.75, 2.0 inch diameters.

It is thecretically possible to calculate the elongation in the insulating coating as the wire is bent around a mandrel of a given size. However, a stranded wire may tend to change shape when bent so that such calculations are apt to be in error. When wire of a single size is to be compared (as in this program), it is better to compare flexibility results as a function of mandrel diameter without attempting the calculation of elongation. When wires of different size are to be compared, it is desirable to make an arbitrary comparison by describing the vandrel diameter in terms of a multiple of the overall wire diameter - 2X, 3X, 5X, etc., (i.e., a 0.125 in. dia. (2X) mandrel or a 0.50 in. dia. (8X) mandrel for a .062 in. dia. wire).

### Significance and Use

At room temperature hook-up wire usually can be wrapped on its own diameter (IX mandrel) without visible damage. The mandrel flexibility test is used primarily to determine the influence of ambient or aging conditions.

Exposure at elevated temperatures may embrittle hook-up wire insulation by loss of voltage plasticizer or low molecular weight polymer fragments, by polymer scission or increased cross-linking or by oxidative degradation. Exposure to radiation, such as ultra-violet light or gamma and neutron flux, may cause molecular scission or cross-linking and may also promote oxidative degradation. A mandrel flexibility test made at a room temperature provides a simple means of showing the effect of ageing.

-14-

As the test temperature is decreased, the wire insulation may become stronger but decreases in elongation to break - becomes more brittle. For most elastomeric materials such as silicone rubber, brittleness increases relatively slowly as the temperature is decreased until, finally, a sudden increase occurs at what is conventionally called the "glass transition" temperature. For non-elastomeric macerials such as H-film, much smaller changes in elongation occur as the temperature is decreased. While information is quite incomplete, available data for both elastomeric and non-elastomeric materials indicate that, below the temperature of liquid nitrogen (-196°C), brittieness increases very little. Particularly interesting and useful comparisons of flexibility can be made, therefore, at the liquid nitrogen temperature.

Flexibility measurements made at liquid nitrogen temperatures provide data of functional significance for space applications where very cold ambient temperatures may be encountered. In NASA contract NAS8-2442 it was discovered that mandrel flexibility tests on wire at liquid nitrogen temperatures provided a very sensitive means for determining the effect of thermal aging and hydrolytic molecular scission with great sensitivity. The effects of such aging degradation could be detected with measurements made in liquid nitrogen or liquid helium long before the aging effects became apparent with tests at room temperature. To capitalize on this sensitivity, mandrel flexibility tests in liquid nitrogen are being used in this program to detect aging in wire after exposure to high temperature in oxygen, to ultra-violet light, to x-ray radiation and to various chemicals.

#### Analysis from Visual Observation

Mandrel flexibility tests can provide considerable information when carried out by a skilled observer. For example, lack of adhesion between layers in a taped construction or between overcoats and base insulation may be observed and point to weakness in particular wire insulation constructions. A tendency for dispersion overcoats to "mud flat cracking" becomes more visible when the coating is flexed. A weak "knit line" in an extrusion tends to develop longitudinal splits when the wire is flexed in liquid nitrogen.

It must be recognized that such visual observations are subjective and not quantitative. In this program only one individual has made the flexibility test so as to provide a uniform basis for comparison between wires.

-15-

### 17. Scrape Abrasion

Α

The NEMA (GE) repeated scrape abrasion tester subjects the wive to the back and forth scraping action of a No. 11 (.016 inch diameter) steel sewing needle mounted at a right angle to the wire which is clamped in an anvil, as shown in Figure 17. The side of the needle rests on the wire. The needle is held in the abrading head, Figure 18, and loaded with weights as shown by eccentine action. The needle under the compression load is moved back and forth along the wire for a distance of 3/8 inches, 60 times per minute. When the needle ubrades through the insulation coating on the wire, electrical contact (limited to .063 amperes) is established which actuates a relay to stop the driving motor.

In the operation of the test, the wire is straightened without elongation and clamped in the anvil. The counter is set to zero. The desired load is added to the abrading head and the needle is set carefully on the wire. The motor is started and the repeated scraping action progresses until contact with the wire turns off the driving motor. The number of strokes to failure is recorded. For the next test the wire is rotated  $120^{\circ}$  and the test repeated at another position along the wire. For the third test the wire is again rotated  $120^{\circ}$ . In this fashion three points about the circumference are evaluated so that effects of eccentricity will be included. It is customary to make at least three measurements on each wire specimen.

The scrape abrasion tester was originally developed for use with film-coated, magnet wire. With magnet wire insulation the effects of load, insulation thickness, and wire diameter have been determined as represented by the following equation:

$$= \frac{S p^{3}}{t^{2} d}$$
 where A = Abrasion factor  
S = Average number of scrapes to failure  
p = Load in grams  
t = Insulation thickness  
d = Solid conductor diameter in mils

With stranded hook-up wire this relationship may not hold, but does indicate the need to obtain test results at several loads. For the #20 stranded wire used in this program, load of 500, 800 and 1000 grams have generally been used. This range of loads will permit adequate comparison of the different wires in this program.

### Significence and Use

The repeated scrape abrasion test gives completely arbitrary results but permits comparison between wires with different insulation coatings. It does have the advantage that the effect of load can be evaluated. However, many other factors contribute to the abrasion of hook-up wire:

- i. Nature and uniformity of the abrading surface (sharp edge, wire on wire, etc.)
- ii. The tension in the wire.
- iii. Test temperature.
- iv. Thermal or other aging.

In service many kinds of abrasion may be encountered. The sawin<sub>6</sub> action of wire against a sharp metal edge (i.e., when a protective grommet is missing) is unfortunately a common and very destructive type of abrasion. Abrasion of wire against wire when cabled together can also occur and may be most serious under a clamp. Experience indicates that in such cases failure usually occurs by cutthrough. However, the tendency for cut-through may be increased by vibrational forces.

Many types of abrasion test have been proposed for hook-up wire. A commonly used test makes use of a sandpaper abrading surface. This surface is hardly representative of normal service conditions. The sandpaper can be constantly renewed so that it presents a new abrading surface. Even so, sandpaper is sufficiently variable that uniform test results are difficult to achieve. A wire-on-wire test is occasionally suggested since the uniformity of the abrading surface is thereby eliminated as a test factor. In practice, such tests are difficult to run and require unrealistically large compressive loads to achieve failure in a reasonable time.

Considerable attention has been directed to the type of abrading surface used. A sewing needle has been found to be very uniform, hard and inexpensive. The diameter of the needle can, of course, be varied to achieve different types of abrasion. Sharp cutting edges generally have proved to be impractical as abrading surfaces, although a sapphire cutting edge has shown some promise. Small balls have also proven useful as abrading surfaces on flat wires, but are not useful with small, round wires. The repeated scrape abrasion tester is now being replaced for magnet wire evaluation by a single scrape abrasion test in which the loal is gradually increased until failure occurs. While reasons exist for substituting the single scrape abrasion test with film coated magnet wire, it is not a suitable test for hook-up wire insulation. With the single scrape test, the relatively soft insulation of hook-up wire "piles up" ahead of the abrading edge and very non-uniform tearing results.

Until some other test is developed which meets functional need better, it is suggested that the repeated scrape abrasion test can provide a comparative, even though arbitrary, evaluation of abrasion resistance for hook-up wire insulation.

### 18. Blocking

Blocking is difficult to determine in a quantitative fashion. However, qualitative evidence of blocking is watched for throughout the program in the cabled specimens and twisted pairs under the various exposure conditions. Since blocking will be evaluated in this way, no special test specimens or procedures are considered to be necessary

### 19. Cut-Through

This test evaluates the ability of the insulation to withstand highly localized pressures without cutting through to the conductor. The test specimen consists of a straight piece of wire about six inches long that is mounted on a flat base plate(200 RMS minimum surface finish). The base plate is placed in the compression cage of an Instron Testing Machine, as shown in Figure 19 Pressure is applied to the wire insulation by a steel paddle 1/16 inch thick with a 1/32 inch radius. (The paddle shown in Figure 19 is used for thermal creep tests and is thicker than the cut-through paddle). A 115 volt alarm circuit is connected between the conductor and the paddle so that a signal is given when the insulation is ruptured.

A crosshead speed of 0.005 inches per minute is used to obtain load versus deflection curves for three specimens at room temperature and three specimens at  $149^{\circ}$ C. The curves for the test insulations are compared to that for MIL-W-16878 Type E Teflon insulated wire, and the load to cut-through is reported.

Figure 20 is an overall view of the test equipment that is required for the cut-through and thermal creep tests.

### 20. Thermal Creep

The specimen and test apparatus for thermal creep measurements is the same as that for the cut-through tests except for the shape of the steel paddle, which is 1/4 inch thick with a flat bottom surface (200 RMS) and a 1/32 inch radius at each longitudinal edge. The shape of the paddle is shown in Figure 19.

The purpose of the creep test is to evaluate the ability of the wire insulation to withstand constant compressive loads such as those encountered in tight bundle tier. The acceptance criterion is that the time to breakthrough must exceed one hour for a specified load that is defined as the load required to fail Type E Teflon in one hour. In addition to determining if the failure time of an insulation exceeds that of Teflon, it is desirable to rank the various insulation in the order of their performance. For insulation with creep characteristics greatly different from those of Teflon, it is not feasible to obtain both kinds of information with a single test employing only one test load. The H-film constructions, for instance, will run for thousands of hours at the load (116 pounds) that fails Teflon in one hour. Since it is a foregone conclusion that certain wire constructions will pass the acceptance criterion, the test was modified to provide data on the comparative creep characteristics of all the samples in the program.

Fxperience has shown that with many materials the creep rupture load is approximately 50% to 75% of the ultimate short-time tensile load. This principle was applied to the wire specimens to obtain an estimate of the load that would yield a creep failure in a reasonable length of time. The specimen is subjected to a short-time test, which consists of applying a load, with the creep fixture, at a steady rate of .002 inches per minute until failure occurs. Approximately 75% of this short-time failure load is then used as the constant load in the first creep test. The load is applied for one hour and, if failure does not occur, it is increased by about 10% for the next 15 minutes. This procedure is repeated until failure occurs. Two additional specimens are then tested at a load determined by the results of the first test.

This test procedure permits the wires with failure times much greater than that of Teflon to be ranked in order of performance. With wires that fail in shorter time than Teflon, there is no need to modify the procedure.

-20-

The tests are also conducted at  $149^{\circ}C$  to determine the effect of temperature on creep characteristics. At the elevated temperature the one hour failure load for the Teflon wire was only 33 pounds. Again, this load is too small to cause failure of most of the other wires in a reasonable time. Therefore, the procedure used at room temperature, as described above, is also applied at  $149^{\circ}C$ .

### 21. Wicking

Specimens for the wicking test consist of straight pieces of wire six inches in length. Each specimen is wiped with a clean, dry, lint-free cloth and then weighed to the nearest 0.1 mg. The specimen is then vertically immersed for two inches of its length in a fluorescent dye solution of the following composition:

0.1 gm	Rhodamine B dye
2.0 gm	Urea
15 c.c.	ethyl alcohol
l c.c.	10% water solution of Triton X-100
1000 c.c.	Water

Figure 21 shows the manner in which the specimens are held in the dye solution.

After an immersion period of 24 hours, at room temperature, the specimens are removed from the solution, wiped with a clean, dry, lint-free cloth, ond weighed. After weighing, the specimens are taken to a dark-room and examined under an ultra-violet light for evidence of fluorescent dye on the stripped conductor. With transparent insulation the dye can be detected without stripping.

The weight gain and the wicking distance are reported for each specimen.

### 22. Thermal Aging

Thermal aging is carried out both in vacuum and in oxygen. Flexibility specimens and twisted pairs are aged at  $150^{\circ}$ C for 15 days in vacuum  $(10^{-6}$  torr range) or in oxygen (15 psia). Figure 22 shows a group of specimens in a glass bottle that is located in an oven and attached to the manifold of a high vacuum pumping system.

After the aging period, insulation resistance and short-time voltage breakdown tests are made at room condition  $(23^{\circ}C/50\% \text{ RH})$ . Mandrel flexibility tests (see page 13) are made at  $23^{\circ}C$  and at  $-196^{\circ}C$  in liquid nitrogen. The degree of degradation is determined by comparison with unaged specimens.

### 23. Exposure to Ultra-Violet Radiation

Flexibility specimens and twisted pairs are exposed to ultra-violet radiation in vacuum and in wet oxygen at 15 psia for 30 days. In each case the wires are held vertically on a cylindrical rack that is coaxially positioned with respect to a cylindrical UV lamp Figure 23 shows this arrangement for the vacuum radiation. A silvered reflector, which is mounted inside the metal bell-jar, is not visible in the photograph.

The lamp is a General Electric Company UA-3 Fhotochemical Lamp. It has a lighted length of six inches, producing approximately 39 watts in the ultra-violet range, between 2200 and 4000A. To provide the required radiation of 0.0056 watts cm<sup>-2</sup> (56,000 ergs cm<sup>-2</sup> sec<sup>-1</sup>) the radial distance from the lamp to the specimen holder is 18.5 cm.

The temperature of the wires is measured with a thermocouple. In vacuum the specimens are maintained at about  $145^{\circ}$ C. In oxygen the temperature is approximately  $90^{\circ}$ C. These temperatures are reached as the result of radiated energy, from the 360 watt U.V. lamp, no additional heat is required.

### 24. Exposure to X-Ray Radiation

Five twisted pairs and five flexibility specimens of each wire sample are exposed to 50 KVP x-rays in vacuum at  $150^{\circ}C$  and in oxygen at 5 psia and  $93^{\circ}C$ .

-22-

The x-ray source is a Machlett AEG50S tube (tungsten target, beryllium window) rated at 50 KVP, 50 ma. In vacuum the specimens are exposed for ten hours at a dose rate of 6000 rads per hour. In oxygen the dose rate is reduced to 100 rads per hour. After irradiation, insulation resistance, voltage breakdown and mandrel flexibility are measured at room condition and mandrel flexibility is also measured in liquid nitrogen at  $-196^{\circ}C$ .

#### 25. Flammability

In this program flammability tests have been performed in wet flowing oxygen at 5 psi (254 mm. of Hg) absolute pressure. The oxygen was saturated with moisture by passing it through water held at about  $10^{\circ}$ C above room temperature. The flammability test chamber was evacuated, flushed once with pure oxygen and then evacuated to the test pressure. Provision was made for connecting a vacuum bottle so that gases evolved could be analyzed as described in the section under analysis.

Three approaches were taken to produce the high temperatures needed to cause ignition (if possible) in wires mounted vertically.

Test I - The wire was subjected to external heat from an incandescent, heater coil so that the wire temperature increased rapidly to about 480 to  $500^{\circ}$ C. If ignition did not occur after a five minute exposure, the wire was heated by passing current chrough it sufficient to achieve a temperature of  $600^{\circ}$ C or somewhat higher.

Test II A - An external heat source was not used but the conductor was heated by passing a large steady current through it so that a very high temperature was achieved quickly. In various phases of this test a nominal initial current of 40, 45, or 50 ammeres was used for the #20 wire. If ignition or considerable degradation was not obtained in 5 minutes, the current was increased by 5 amperes (i.e. from 40 to 45 or from 45 to 50).

Test II B - An external heat source was not used and the wire was brought up to high temperatures somewhat more slowly than in II A above by passing smaller currents at first through the wire and then increasing the current in steps at 2.5 minutes intervals. The following nominal current steps were adopted: 20, 30, 32.5, 35, 37.5, 40, 42.5, 45 and 47.5 amperes. Throughout the flammability test the test specimen was watched carefully for evidence of physical change and the formation of smoke or visible vapor. Periodically during the test at intervals of about 15 seconds a conventional spark plug with platinum plated electrodes set at a gap distance of 0.125 inches was activated for about a second by applying the output of a 15 KV sign-lighting transformer with output current limited to .020 amperes by resistance in the primary circuit of the transformer. The spark gap was located within 1/32 inch of the wire and about 1½ inches above the hottest portion of the wire so that inflammable degradation gases would be readily ignited. The spark gap was not operated continuously when it was discovered that periodic operation of the spark gap was more likely to cause ignition than continuous operation.

Various types of ignition were encountered as will be reported under test results. Efforts to use photoelectric indication of ignition were not very successful. It was possible to obtain reliable photoelectric indication of the faint flashes of non-continuing combustion only in the absence of background light which made other visual observations impossible. It was indicated that photoelectric techniques could detect a maintained flame even in the presence of some background lighting but such detection was inadequate for the present program. Visual observation appears to be more sensitive and discriminatory than photoelectric techniques for the subject purpose.

### Description of Apparatus

The test assembly except for the DC power supply is shown in Figure 24. In this photograph the bell jar, which contained the oxygen at 5 psi, is shown in the lifted position. The bell jar was supported by a rope passing over a pulley and was counterweighted so that even the development of a small pressure within the jar would automatically lift the bell jar and vent it to the atmosphere in an explosion resistant hood. Provisions were made also to flood the chamber with nitrogen in case of uncontrolled fire. Thes precautions so far have not been needed.

-25-

The various points of the test assembly are listed and described in more detail below.

**a.** An 8 inch diameter glass bell jar with a brass base plate containing suitable gas ports and electrical bushings and a circular silicone rubber gasket to provide a seal.

b. A DC power supply capable of supplying a controlled current up to 250 amperes. A G.F. DC arc welder modified with an external electronic field supply for better current control was used.

c. A means for supporting and clamping the 6 inch long wire specimen in a vertical position (see Figure 25). The clamps and leads were designed so that 50 amperes flowing through them would produce negligible heating.

d. A heater coil (see Figure 25) consisting of 9 turns of #26 nichrome wire wound in a diameter of  $\frac{1}{2}$  inch and a length of  $\frac{1}{2}$  inch. A variable, low voltage power supply to permit bringing the heater coil to a yellow heat - 7 volts from a Variac was used.

e. A spark plug with platinum plated electrodes set to a gap of 0.125 inches. The power supply for the spark plug consists of a 15 KV, 50 ma sign-lighting transformer with a resistance in the 115 volt primary circuit to limit the current through the gap to .020 amperes. The primary circuit of the transformer passes through an electrical interlock on the cage to prevent accidental contact with high voltage. The spark is activated by an external switch in the 115 volt supply.

The spark gap is located within 1/32 inch of the wire and  $1\frac{1}{2}$  inches above the center of the test specimen.

f. A .003 inch chromel-alumel thermocouple is used to measure the temperature of the wire when the heater coil is used. A hole sufficiently large to accept the thermocouple junction (about .014 inch diameter) is drilled through the center of the wire. The uninsulated thermocouple leads are brought down through the heater coil and along

the test wire but separated from both as can be seen in Figure 25. The uninsulated thermocouple leads are connected to suitable and larger insulated leads and led through the bottom place of the bell jar assembly to a thermocouple chart recorder. A thermocouple potentiometer is available also for auxiliary and calibration purposes.

When the external heater coil was not used, the wire temperature g. was determined by measuring the potential drop along a one inch length at the center of the wire while current was flowing in the wire. In this way it was unnecessary to drill a hole for the thermocouple and thereby disrupt the continuity of the wire. Moreover, initial tests showed that the temperature along a one inch length at the center of the wire test specimen was substantially uniform. It is necessary to cut the wire insulation at each end of the one inch length in the center so as to apply the .003 inch diameter platinum drop leads as shown in Figure 26. These fine drop leads are then soldered to leads which pass out through the base plate. These leads are connected to a recorder with a full scale of 0.150 volt. Actual voltage drops along the one inch section of the test wire may be as high as 0.150 inch at the highest currents. (The internal resistance of the recording voltmeter is high compared to the lead resistance so that the voltage drop along the wire can be accurately measured without error from the leads).

The current is recorded simultaneously with the voltage on a twopen strip recorder. A suitable current shunt is used to provide the input to the recorder.

From the current and voltage measurement the resistance of the hot wire can be easily calculated.

$$R = \frac{E}{I}$$
 Where R = resistance, ohms.  
E = voltage, volts.  
I = current, amperes.

Wire resistance changes with temperature and it is theoretically possible to calculate the temperature from the charge in resistance. Unfortunately such calculations depend upon the temperature coefficient of resistance for the conductor and this coefficient appeared to depend to a larger extent than expected on the wire plating and perhaps some unknown factors such as the diffusivity of silver plating into the copper. Oxidation may also be a factor. In consequence in proved necessary to run calibration curves of resistance against the temperature measured with a thermocouple. In this case, to avoid disrupting the wire by drilling a hole, the insulation was removed from a short section at the center of the wire and the strands untwisted sufficiently to permit the entrance of the .003 inch wire thermocouple. The wire was then retwisted. The experimentally determined calibration curves for the nickel placed and silver plated, #20 stranded wires used in this program are drawn in Figures 27 and 28.

If current alone would serve to indicate the temperature of the wire, the measurement problem would be solved. At the lower wire temperatures, current could be related directly to temperature. Unfortunately at the higher temperatures, differences in thermal emissivity and exothermic (or endothermic) reactions at the wire surface seemed to produce temperature changes not directly related to the amount of current flowing. From the functional point of view, the current may be a more important parameter than the resulting temperature. However, a knowledge of the temperature is helpful in making comparisons between wires and in considering the mechanism of deterioration and ignition.

At the conclusion of the first phase of this program it is reluctantly concluded that neither the thermocouple nor the voltage-drop technique for measuring wire temperature is adequate in every respect. While the voltage-drop technique has provided temperature measurements, which are believed to be reasonably accurate, it is concluded that the thermocouple technique is more direct and less tedious. In the remainder of the program a thermocouple will be inserted within a small section of wire which has been untwisted at the center of the test specimen and then retwisted before test.

-28-

### Significance and Use

The importance of resistance to ignition and combustion in spacecraft wiring is obvious. The psychological as well as physical hazard of smoke which may be toxic or coat optical elements is also well recognized. The danger of toxic products, which may not form visible smoke, should be recognized also. Finally, so far as possible, hook-up wire should continue to perform its function even though exposed to very high temperatures. As an ideal, a hook-up wire should maintain adequate insulation until it melts and fuses itself. Perhaps more realistically, the wires surrounding such a melting wire in a cabled bund. should continue to function and remain electrically isolated. The flammability uest should permit analysis of the suitabilicy of a specific insulated, hook-up wire in the foregoing respects.

The three types of flammability test are designed to represent three types of field conditions:

Test I. The test with an external heat source represents the situation in which a hook-up wire accidently may be adjacent to or come in contact with another very hot wire or a very hot element or device. It is the condition most likely to produce combustion since the heater coil operating at about  $900^{\circ}$ C itself serves as an ignition. source and the heat is applied very rapdily to the outside of the wire insulation.

Test II A. The test with a high current applied at the start of the test represents performance under a short-circuit condition. Because heat is applied so suddenly to the conductor, it is most likely to cause maximum physical damage to the wire insulation.

Test II B. The test in which current is slowly increased represents the characteristic of an increasingly overloaded (racher than short circuited) wire. Smoke is more likely to evolve with this procedure. The test also permits somewhat more quantitative comparison of the physical degradation of different types of wire. Additional comment concerning the test specimen appears important. The specification in the request for proposal called for the flammability test to be made on a single wire mounted so that the 5 psi oxygen would have free access to it. The test results to date indicate that the vertical mounting chosen does tend to promote the progress of combustion along the wire. It is likely also that the free access to the atmosphere may promote ignition. For future evaluation programs, it is recommended that, in addition to the freely mounted specimen, a cabled specimen should be considered. A suitable 7 wire cabled test specimen has been used in this program for measuring insulation resistance and corona intensity (see Figure 1). With this sample the high current could be applied to the central conductor of the bundle and a test voltage simultaneously applied between the bot central conductor and the surrounding 6 wires in the bundle.

The 5 psi wet oxygen atmo phere represents the environment of veral spacecraft and is, therefore, important for this evaluation program. nowever, a very few limited tests indicate that more serious ignition and combustion problems may occur in oxygen at atmospheric pressure. For example, the wire itself will burn in oxygen at 15 psi. A polyolefin insulated wire even ignited and burned in 15 psi oxygen under the influence of just the high voltage spark <u>at</u> <u>room temperature</u>. In future evaluation programs it may be important to consider the effect of 15 psi as well as 5 psi cxygen on flammability since the higher o xygen pressures may be encountered in some operational phases.

-30-

#### 26. Chemical Compatibility

To determine the resistance of the wire insulation to various chemicals used in or about manned spacecraft, three twisted pair specimens and six flexibility specimens are exposed to the chemicals for specified periods and then tested for insulation resistance, voltage breakdown and mandrel flexibility at room temperature and mandrel flexibility in liquid nitrogen at -196°C. The period of exposure is 14 days for all of the chemicals except the fuels and oxidizers. For these compounds the period is reduced to 20 hours.

The twisted pairs are made from extra long (35 inches) pieces of wire so that the flexibility samples can be cut from the long tails after the exposure. Figure 29 shows the method of immersing the specimens and bringing the ends out through a two-hole rubber stopper. The ends are sealed with an RTV silicone rubber to protect the conductors from direct contact with the vapor from the test liquid The test tubes are 38 mm. diameter and 300 mm. long. Specimens of only one wire sample are placed in each test tube. This eliminates the possibility of having the reaction products from one sample influencing the reaction with another sample.

Special techniques and apparatus are required for handling fluorine and  $N_{\gamma}O_{L}^{0}$ .

In the case of Freon 114, which boils at  $3.6^{\circ}$ C, the gas was liquified and then held at  $-7^{\circ}$ C during the exposure period.

The chemicals that are used as test fluids are:

a. Hypergolic Fuels (M-1) UDMH MMH Fydrazine (MIL-P-26536) A-50 (MIL-P-27402)

b. Oxidizers

N<sub>2</sub>0<sub>4</sub> Fluorine

-31-

c. 0ils (M-2)

Lube cil (MTL-1-7808) Hydraulic oil (NIL-H-5606)

d. Solvents

Ethyl alcohol JP-4 (MIL-J-5624) Freon TF Freon 114 Trichloroehtylene Acetone

e. Miscellaneous Liquids (M-2)

5% salt solution

Ethylene glycol (67.5%) and water (32.5%) with inhibitors per A. Research Specification RS-89.

In addition, a salt fog test is conducted in accordance with MIL STD 810.

Any evidence of discoloration, delamination, cracking, swelling, flaking, corrosion or other unusual condition is reported along with any change in insulation resistance, voltage breakdown strength and flexibility.

27. Offgassing in 5 psia Oxygen

#### Apparatus

The spring balance apparatus system constructed for determining the weight loss in oxygen is shown in the photographs of Figures 30 and 31. The apparatus basically comprised duplicate spring balances of conventional decign and a common gas handling system for atmosphere control.

The spring balance utilized quartz springs (Worden Quartz Products) specified for a 500 mg. maximum load and a sensitivity of 1 mm./mg. Spring displacement was measured optically using a cathetometer (Gaertner Scientific Corp.) which provided measurements to 0.05 mm. The nominal detection limit for these balances was, therefore, 0.05 mgs. The balance reading was based on double location measurements: the position of the spring index relative to the position of a reference mark on the body of the spring housing tube.

-32-
The design of the spring balance unit is shown schematically in Figure 32. The balance chamber assembly was fabricated from Pyrex glass utilizing "standard taper" joints between sections. A load support link of 6 mil platinum wire was used to suspend the test specimen within the hot zono. The weight of these links was about 80 mgs. thus limiting the sample capacity of the balance to 420 mgs.

The sample chamber was heated by a tube furnace, the temperature of which was automatically controlled by means of a thermocouple placed in the open hot zone. Specimen temperature was monitored by a second thermocouple located in a well within the sample chamber. This temperature was recorded and is the temperature listed in the data. The furnaces were mounted on jack units for ease in positioning.

The two balance units were mounted in a rack which was supported on a vibration dampening pad. The gas delivery lines were manifolded into the atmosphere control system, the flow diagram for which is shown in Figure 33.

## Calibration Tests

The sensitivity of the springs was determined experimentally in the 300-400 mg, load region. This was accomplished by applying a base load to the balance and measuring the spring extension resulting from the addition of known load increments. The results in this test are tabulated below:

Load Range (mgs.)	Sensitivity Balance "A"	(mm./mg.) Balance "B"
331 to 381	0.973	0.971
331 to 406	0.973	0.970

The system was also checked for temperature effects. Loads of 400 mgs. (nichrome wire) were placed on the balances and an atmosphere of 5 psia argon was established in the balance chambers. Balance readings were then made at the several temperature levels of the test schedule: room temperature,  $150^{\circ}$ C,  $200^{\circ}$ C,  $250^{\circ}$ C, and  $300^{\circ}$ C. No significant shift in balance reading resulted from the change in temperature.

## Operating Procedure

The procedure in this test comprised the following operations:

- 1. Wire specimens are cut to a weight of 400-410 mgs. The test piece is cleaned by wiping with lintless tissues dampened with ethanol. The specimen is then formed for hanging on the spring balance, after which its weight is measured on an anlytical 'alance. All specimen handling is by means of gloves or forceps.
- 2. After suspension of the specimen from the balance, the specimen and also the glass surfaces of the chamber system are freed of static surface charges by means of a Static Master Ionizing Unit. Neutralization of these charges is required in order to prevent the sample and load support link from being pulled against the walls.
- 3. After complete assembly of the system and positioning of the furnaces, an initial balance reading is obtained. The atmosphere of the balance chamber is then changed to 5 psia oxygen. This is accomplished by evacuation of the system (ca 50 microns) and back-filling with argon twice, after which the system was re-evacuated (50 microns or less) and charged with oxygen\* to 5 psia pressure as measured on a diaphram type vacuum gauge. The stopcocks on the balance chambers are then closed after which balance readings are obtained. This balance reading is used as the base for the weight loss data.
- 4. Both balance systems are then heated to 150°C at relatively rapid rates (room temperature to 150°C in about 15 minutes). The temperature is maintained at 150°C overnight. Surveillance of weight changes is maintained during the first 1½ hours of this 16 hour exposure.

\*Linde Co. Aviator Breathing Grade: Minimum Purity Specification 99.5%.

The balance of the heat schedule comprises increasing the temperature sequentially to  $200^{\circ}$ C,  $250^{\circ}$ C, and  $300^{\circ}$ C with 30 minute hold; g periods at each temperature. A contining surveillance of weight change is maintained throughout this exposure. The balances are run singly in this heat schedule in order to permit the close following of any reactions. As a consequence of this scheduling, additional data are obtained on the inactive system. While the first balance is operated through the high temperature program, the second balance is held at  $150^{\circ}C$  (ca  $2\frac{1}{4}$  hours) and confirmatory rate data re obtained relative to the overnight heating at this temperature. Similarly, while the  $200-250-300^{\circ}$  program is being conducted on the second balance, the first unit is maintained at  $300^{\circ}$ C and data covering an additional  $2\frac{1}{2}$  hours at the terminal temperature are obtained.

- The systems are cooled rapidly by withdrawal of the furnaces. Terminal balance readings are made after cooling to room temperature.
- 6. Prior to disassembly a check is made to insure the absence of leakage into the balance chambers. This is accomplished by readjusting the pressure in the gas manifold to the initial level and then noting any change in pressure on reopening the balance chambers into the manifold.
- The terminal weight of the specimen is determined on an analytical balance.

## Data Tabulation

The results in these tests are tabulated in terms of the cumulative weight loss of the specimen versus the various thermal exposure events (time and temperature defining the extent of exposure). Thus, the indicated weight loss represents the full weight loss accumulated as a result of the referenced exposure event plus the summation of all prior events. The weight loss values are based on the initial spring balance reading at 5 psia oxygen (room temperature).

It should perhaps be noted that the balance chamber temperature has a cyclic characteristic based on the operating differential of the controller and the thermal lag in the system. The cycle was generally uniform and amounted to  $\pm 3^{\circ}$ C for Unit A and  $\pm 1\frac{1}{2}^{\circ}$ C for Unit B. The temperatures listed for the constant temperature periods are the mean values of recorded temperature for that period.

# Gas Analysis

In addition to determining the weight loss in vacuum, separate analytical experiments are conducted to identify the off-gassing products, both in oxygen and in vacuum. These analyses are done with an Analytical Mass Spectrometer (General Electric Cat. No. 8665934 G-1) and a Recording Infrared Spectrometer (Perkin-Fimer Model 21).

In making the studies, 18 inch lengths of the wire sample are first cleaned with ethyl alcohol, then cut into short  $(1\frac{1}{4}")$  lengths, and weighed. This sample of wire is then placed in the side arm of the quartz furnace tube shown in Figure 34. The quartz tube is then placed in a Hoskins Furnace so that the lower portion of the tube (up to the side arm) can be heated. The furnace is held at  $600^{\circ}$ C for one hour under vacuum. The side arm and the upper portion of the quartz sampling tube are kept at ambient temperature during the heating.

After one hour of heating the sampling tube is sealed off with the stopcock and removed from the furnace. It is allowed to cool to below  $100^{\circ}C$  and then tipped to move the wire to the bottom of the tube. The tube is then placed in a furnace held at  $150^{\circ}C$  with an Amplitral Controller for one hour.

After one hour, the tube, still in the oven, is attached to the mass spectrometer sample system and a portion of the outgassing products that have been given off are admitted to the mass spectrometer and analyzed. Following this, the sample tube is placed in liquid nitrogen to freeze out all condensably compounds especially  $CO_2$ . Since the mass spectrometer cannot easily separate CO from N<sub>2</sub>, those gases not condensed at the liquid nitrogen to freeze out all temperature are admitted to the mass spectrometer through a small tube

-36-

containing a catalyst to convert CO to  $CO_2$ . In this way the  $CO-N_2$  ratio can be determined using mass 28 (N<sub>2</sub>) and mass 44 (CO<sub>2</sub>).

When the analyses are completed at  $150^{\circ}$ C the sample tube is again evacuated - this time only for five minutes to pump off the residual material. The tube is again sealed off and the same procedure followed using an oven controlled by an Amplitrol at  $300^{\circ}$ C.

The foregoing discussion gives the details when studying outgassing and degradation under vacuum. When the studies are made in the presence of oxygen some of the procedures are different. The initial evacuation of the sample tube is carried out for 15 minutes, then oxygen at 5 lbs. absolute is admitted to the sample tube, sealed off, and heated for one hour with oxygen. After one hour the oxygen and outgassing products are pumped away and the sample tube cooled. The wire is dropped into the loter part of the furnace and brought up to  $150^{\circ}$ C and then filled to 5 psis with the oxygen present the degassing products are considerably diluted, the method of analysis consists of first taking a sample of the total gas, with the oxygen, then freezing out the remainder, and allowing this to pass through the catalyst to check for CO. After this is completed, the non condensible gases are pumped away and the sample again brought up to temperature. In this way a more sensitive analyses of the trace degradation products in the sample can be achieved.

## 28. Vacuum Volatility

#### Apparatus

The weight change measurements for the vacuum exposure are made in the apparatus shown in Figures 35 and 36. This apparatus consists of a microbalance and associated vacuum system furnace and recording equipment.

The electronic recording microbalance (Sartorius-Werke, Gottingen, model Electrona) was designed for vacuum operation. The maximum load and accuracy are specified as 1000 mg. and 0.001 mg. respectively. The balance amplifier scales can be adjusted to detect weight changes in the following ranges: 0-20, 0-10, 0-5, 0-2, 0-1, 0-0.5, and 0-0.2 mg. full scale. The balance sensitivity is about 0.2% of the full scale magnitude. The balance is mounted on a table designed to minimize transmittal of shocks and vibration from the environment to the balance. The output signal is continuously recorded (Leeds and Northrup, Philadelphia, Pa., Mcdel Speedomax H). Because of the weight of the wire used to suspend the specimen, the specimen weight was held at 800 to 815 mg.

The balance is connected through metallic bellows to a vacuum pumping station (Veeco Vacuum Corp., N.Y. Model VS-9) designed to produce vacuums of  $1 \times 10^{-7}$  torr. The system as used readily pumped down to  $10^{-6}$  to  $10^{-7}$  torr. during test.

The specimens were suspended from the right arm of the balance in a vertical, closed-end quartz tube. Tare weights were suspended from the left balance arm. The specimens were heated by raising a tube furnace mounted on sliding rods over the furnace tube. The furnace was continuously held at the operating temperature which buld maintain the specimen at  $150^{\circ}$ C.

#### **Operating Procedure**

The procedure in this test consists of the following steps:

1. Wire specimens are cut to a weight of 800 to 815 mg. to the nearest 0.1 mg. on an analytical balance. The length of wire is recorded. The wire specimens are cleaned by wiping with a lintless tissue dampened with ethanol. The specimen is formed into a coil of

-38-

suitable size for insertion into the furnace tube. The specimen is re-weighed and differences from the original weight noted. The specimens are handled by means of gloves or forceps during this operation.

2. The specimen is hung on the balance arm and the position on the chart noted. If repositioning is required, other than that which could be accomplished by readjusting the torsion of the beam wire, tare weights are removed or added at this time. The quartz furnace tube is then positioned. No further operations are conducted until a stable signal is obtained.

3. The roughing pump is then used to evacuate the balance chamber. The range switch is turned to the 20 mg. setting during this initial evacuation, which was manually controlled at a rate slow enough to prevent excessive force on the balance arm (as indicated by "apparent" weight changes observed during this operation). Evacuation is continued with the diffusion pump, and the weight change during this operation is recorded.

4. When the weight becomes constant, the heated furnace is raised around the specimen. Continuous weight loss rate measurements are recorded until the slope of the curve obtained is less than 0.0025% per hour (based on insulation weight which was assumed to be 10% of the wire weight). The furnace is cooled by lowering the furnace, and weight changes, if any, are noted.

5. The vacuum pump is isolated and air introduced slowly, following the precautions taken during initial evacuation. When the specimen is subjected to air at atmospheric pressure, the weight change is recorded for one half hour.

6. The specimen is removed from the microbalance and weighed on the analytical balance.

### Gas Analysis

The procedures used in analyzing the gases collected during heating in vacuum are described in the previous section.

-39-



Figure 1: Cabled Specimen for Insulation Resistance and Corona Measurements



Figure 2: Chamber for Aging Cabled Specimens at 50°C and 100% RH plus Dew in Pure Oxygen at 15 psia



Figure 3: Cabled Specimen Mounted in High Voltage Cell



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Figure 6: Cross Sectional View of Set-Up for Electric Strength Measurements at 5 psia  $0_2$ 

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Figure 7: Cell for Flashover Measurement in Wet Oxygen at 5 psia



Figure 8: Typical X-ray of Wire Specimens (5X Magnification)



Figure 9: Wire No. 3 After Thermal Stripping

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Figure 10: Electrical and Mechanical Specimens for Potting Compound Compatibility Tests



Figure 11: Potting Compound Compacibility Specimens being Cured in Aluminum Foil Molds



Figure 12: Repeated Reversed Mandrel Flexibility Test Apparatus for use with 0.030" to 0.125" Diameter Mandrels



Figure 13: Repeated Reversed Mandrel Flexibility Test Apparatus for use with 1/4" to 1 3/4" Diameter Mandrels



Figure 14: MIT Fold Endurance Flex Tester



Figure 15: Loading Nose for MIT Fold Endurance Flex Tester



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Figure 16: Dimensions of Modified Loading Nose for MIT Flex Tester



Figure 17: NEMA Scrape Abrasion Tescer



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Figure 19: Compression Cage Equipped for Thermal Creep Test



Figure 20: Instron Testing Machine with Conditioning Chamber for Thermal Creep and Cut-Through Tests



Figure 21: Wicking Specimens in Fluorescent Dye Solution



Figure 22: Specimens for Thermal Aging Tests at 150°C in Vacuum



Figure 23: Chamber for Ultraviolet Exposure in Vacuum



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Figure 24: Test Assembly for Flammability Test



Figure 25: Flammability Test with External Heater



Figure 26: Flammability Test, Voltage Drop Technique








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Figure 30: Quartz Spring Balance Assemblies for Measurement of Offgassing in Oxvgen



Figure 31: Quartz Spring Balance Specimen Chamber



Figure 32: Schematic Diagram of Quartz Spring Balance



Figure 33: Flow Diagram Atmospheric Control System for Quartz Spring Balance Apparatus





Figure 35: Balance System Showing Vacuum Station and Assoclated Recording and Power Supply Equipment



Figure 36: Recording Microbalance and Furnace Used in Vacuum Volatility Scudy