Industrial Summit Technology • Parlin, New Jersey 08859

PYRE-M.L. Wire Enamel

Introduction

Pyre-M.L. products are part of a family of materials based on aromatic polyimides which are one of the most thermally stable organic materials. These products include Pyre-M.L. Wire Enamels, Pyre-M.L. Liquid "H" Enamels, Pyre-M.L. Insulating Varnish, and Pyre-M.L. Thinner. Table 1 describes these products and their associated codes/ grades.

During decades of commercial and military use, Pyre-M.L. Wire Enamels have been recognized as having the finest thermal stability, radiation resistance, solvent resistance, cryogenic resistance, and electrical properties of any enamel available. Precisely because of these properties, Pyre-M.L. has been used in motors, generators, transformers, and other apparatus which operate continuously at temperatures up to 240°C. Pyre-M.L. is also useful in heavy duty motors which, due to temporary extreme overloads, acceleration torques, or stalls, are subjected to even higher temperatures (up to 400°C) for limited periods (see Graph No. 1) . Its thermal stability and very low tendency to give off volatile matters has led to its use in sealed relays designed for use in vacuum. The almost absolute insolubility of Pyre-M.L. has led to their use in hermetic refrigeration systems and certain liquid filled transformers. Pyre-M.L. products are U.L.® approved.

Description of Product

Pyre-M.L. wire enamels are solutions of polyamic acids formed by reacting aromatic diamines with aromatic dianhydrides. Sometimes aromatic hydrocarbon or xylene solvent is added as a diluent. The monomers used in Pyre-M.L. formulation and the accompanying reaction are shown in figure 1. Various grades of Pyre-M.L. enamels with their properties/specifications are described in Table 2.

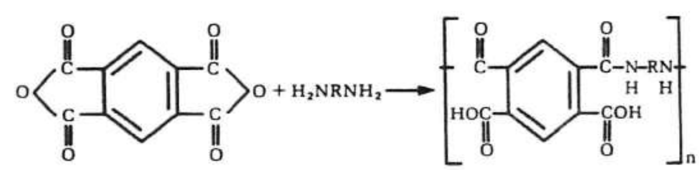
Pyre-M.L. wire enamels can be applied to all sizes of round, square, and rectangular wires by conventional die coating techniques. They have also been applied to irregular surfaces using other techniques such as spraying, drop dispense, and brushing. Copper, aluminum, brass, nickel, and chrome alloy have all been coated successfully. Pyre-M.L. has also been used for coating glass served wire and special hook up wires insulated with surface treated Teflon® fluorocarbon resin.

Table 1

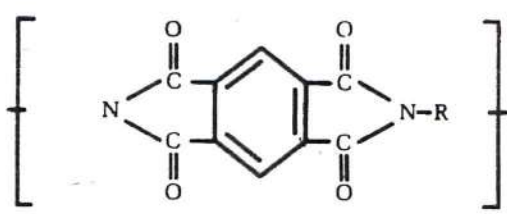
LIST OF PYRE-M.L. PRODUCT OFFERINGS

Category	IST Code	Tech. Bull.
Wire Enamel	RC-5019 RC-5057 RC-5069 RC-5083	ML-1-95
Insulating Varnish	RK-692	ML-2-95
Liquid "H" Enamel	RC-5097	ML-3-95
Thinner	T-8585	ML-4-95

Figure: - 1



When the enamel is baked it is converted to an inert polyimide.



Viscosity Stability

The viscosity, as manufactured, is not stable but gradually drops by 35%, and then increases to gel. Gelation occurs in approximately 14 days at 120°F (49°C), 30 days at 100°F (38°C), and 90 days at 77°F (25°C). At 40°F (4°C), it will not gel in one year. Hence, it is recommended that Pyre-M.L. be stored under refrigeration conditions prior to use. Pyre-M.L. is stored at 40°F in the Pyre-M.L. manufacturing plant warehouse in Parlin, New Jersey, USA. Storage in an unheated building may be acceptable during winter. Viscosity measurements are strongly affected by temperature. See Graph No.2 for temperature-viscosity conversion.

Moisture contamination has a detrimental effect on viscosity stability, and hence, must be avoided. Allow all chilled containers to reach room temperatures before opening them to avoid moisture from condensing.

Properties of Magnet Wire Coated with Pyre-M.L. Enamel:

Thermal Properties

Tests made according to IEEE 57 or ASTM D2307 indicated that wire coated with Pyre-M.L. has a thermal life of 20,000 hours at 243°C. This value was obtained with no extrapolation. Samples aged at 200°C and 220°C show no failures after 40,000 hours at which time the test was discontinued. Again, refer to Graph No.1 which shows the thermal life of wire coated with Pyre-M.L. based on data from four different laboratories. Temperature Vs. Weight Loss is shown in Table 3. Wire coated with Pyre-M.L. is suitable for use in Class H or 220°C insulating systems.

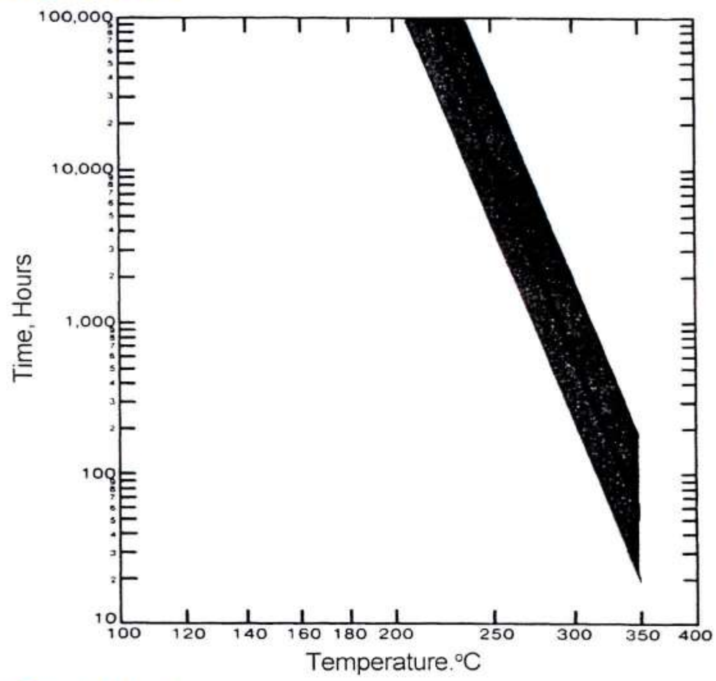
Table 2

PYRE-M.L. PRODUCTS SPECIFICATION
I.S.T. (USA) CORPORATION OFFERINGS

Code	RC-5019	RC-5057	RC-5069	RC-5083
Solids(%)(1)	15.0-16.0	14.5-15.5	12.3-13.3	18.0-19.0
Viscosity (Poise)(2)	50-70	50-70	5-12	20-40
Solvent System				
NMP(%)	100	75-85	75-85	15
Aromatic Hydrocarbon(%)	-	15-25	15-25	0
DMAC	0	0	0	85
Density				
LBS/Gal (+/-0.05)	9.11	8.80	8.72	8.57-8.67
KGS/Gal (+/-0.05)	4.13	3.99	3.96	3.89-3.94

Note-(1) NEMA Test Method - 2gm/2hr/200°C Bake
(2) Measured at 25°C with Brookfield Viscometer #3 Spindle @ 12 RPM

Graph No. 1
Thermal Life - Unvarnished PYRE-M.L.®
Coated Wire



Graph No. 2
Effect of Temperature on Viscosity of
Pyre-M.L. Wire Enamel

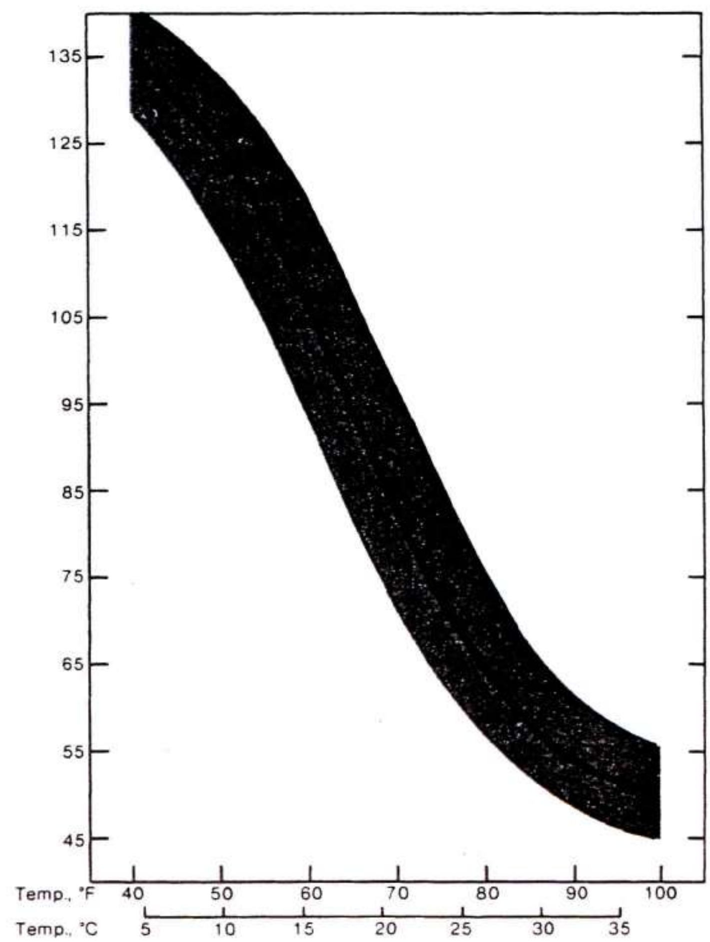


Table 3

Weight Loss at Elevated Temperatures

Hours At 220°C	%Loss
2300	3.3
6900	4.8
13000	5.6
Hours At 240°C	**%Loss**
2300	5.0
6200	9.0
12500	13.4
Hours At 260°C	**%Loss**
2200	10.5
6500	23.2
11500	27.1
Hours At 300°C	**%Loss**
144	4.1
195	5.1
404	7.0
500	8.3
645	8.3
808	8.3

Electrical Properties

The electrical properties are remarkably stable over a wide range of temperatures as illustrated in Table 4.

Refrigerant Resistance

Sixty day sealed tube tests at elevated temperatures in the presence of copper, iron, aluminum, and either FREON® 12 or FREON® 22 refrigerants indicate that Pyre-M.L. is unaffected. Pyre-M.L. was found to have only 0.01% extractables in FREON 12 and 0.02% extractables in FREON 22 based on the weight of the wire enamel. These favorable conditions have been confirmed by reports received from hermetic system manufacturers.

Dielectric Gas Resistance

Laboratory tests indicate that the electrical properties of Pyre-M.L. are not significantly affected by exposure for 150 days to FREON 116 (hexafluoroethane) FREON C318 (octaflourocyclobutane), or nitrogen at 300°F. Class H dry type transformers utilizing magnet wire coated with Pyre-M.L. are in commercial use.

Dielectric Liquid Resistance

Laboratory tests indicate that moderately or thoroughly cured Pyre-M.L. has excellent resistance to oil. However, Pyre-M.L. can be degraded by water at elevated temperatures and pressures. It should not be used at elevated temperatures in sealed systems containing any significant amounts of water or materials such as paper which decompose to water without preliminary tests to show that service will be satisfactory. Thoroughly cured wire performs best under these circumstances.

Heat and Cold Resistance

a. Weight loss data from coated aluminum strip (in air) at 5 minutes at 300°C shows:

Polyester	7.5%
Epoxy	16.5% based on original film weight
PYRE-M.L.	0.75%

b. Retention of elongation (hours to 1% elongation) on heat aging. (initial %E=25%)

These are described in Table 5

Table 5

Hours	Temperature
11	752°F (400°C)
85	662°F (350°C)
700	572°F (300°C)
4500	482°F (250°C)

The mechanical properties of these polymer films are excellent and are retained over a wide temperature range. The zero strength temperature is 1472°F (800°C); this is 270°F (150°C) above the melting point of aluminum. The flex modulus, approximately 400,000 psi at ambient temperature, increases by only 25% when measured at

Table 4

Dissipation Factor

Frequency cps.	25°C	150°C	175°C	200°C	225°C	250°C
10^2	0.0025	.0062	.0180	>0.1000	>0.1000	>0.1000
10^3	0.0025	.0056	.0072	>0.0177	0.1152	0.5810
10^4	0.0030	.0048	.0061	>0.0101	0.0207	0.163
Dielectric Constant						
10^2	3.66	3.11	3.43	3.63	5.29	11.88
10^3	3.68	3.09	3.40	3.60	3.62	5.58
10^4	3.65	3.07	3.41	3.60	3.55	3.56

-310°F (-190°C) and decreases by about 50% when measured at 482°F (250°C).

The retention of physical properties at low temperatures, as indicated, is exemplified by the results obtained with magnet wire coated with Pyre-M.L. enamel in liquid helium at -269°C (4°K). Samples of 25 - mil diameter wire were cooled to 4°K and subjected to a mandrel bend. The radius at which failure occurred on forward and reverse bends is shown in Table 6. The superiority over the materials tested is clearly shown.

These properties are described in details in Tables 7,8, and 9.

Table 6

	Failure Diameter In Inches	
	Forward Bend	Reverse Bend
"Formvar"	3/4	1
Polyester	1/2	3/4
PYRE-M.L.	OK at 1/8	OK at 1/8
Filled TEFLON®		
TFE-flourocarbon	1/2	1/2
Asbestos	1/2	1/2
Polyvinyl Chloride	1 3/4	1 3/4
11	752°F (400°C)	
85	662°F (350°C)	
700	572°F (300°C)	
4500	482°F (250°C)	

Table 7

Coefficient of Kinetic Friction

PYRE-M.L.	0.17
Graphite	0.18
TEFLON TFE-flourocarbon	0.04-0.09

Table 8

Linear Coefficient of Thermal Expansion	
PYRE-M.L	$4.0 \times 10^{-5/0}$C
TEFLON TFE-flourocarbon	$5.5 \times 10^{-5/0}$C
Nylon	$8\text{-}10 \times 10^{-5/0}$C
DELRIN® acetal resin	$8.1 \times 10^{-5/0}$C
Epoxy	$5\text{-}9 \times 10^{-5/0}$C

Table 9

Coefficient of Thermal Conductivity	Cal/sec/cm $^{2/0}$ C/CM
PYRE-M.L	35×10^{-5}
Silicon	35×10^{-5}
Epoxy	5×10^{-5}
Nylon	55×10^{-5}
DELRIN	55×10^{-5}
TEFLON	60×10^{-5}

Dependence of Properties on Degree of Cure

Many of the properties of wire coated with Pyre-M.L. Wire Enamel depend on the degree to which it is cured. Magnet wire manufacturers and users should select the proper degree of cure to give the balance of properties which best meets their requirements.

Determining Degree of Cure

The dissipation factor test is recommended for controlling the degree of cure of wire in production, NEMA Publication MW-1000 lists a limit of no greater than 0.006 for wire coated with Pyre-M.L. Other useful control tests can be developed using weight loss at elevated temperatures and crazing tendencies. These are shown in Table 10.

Table 10

Properties of Heavy Build No. 18 (1.024mm) Wire Coated with PYRE-M.L.®

Degree of Cure	**Incomplete**	**Moderate**	**Thorough**	**Very Thorough**
Dissipation Factor[1]	0.020	0.004	0.0025	0.0015
Dielectric Constant[1]	4.5	3.9	3.7	3.2
Weight Loss[2]	16%	3%	1.6%	1.3%
Crazing[3]	Very Severe	Moderate	Slight	Very Slight
Crazing after 1/2 hr. x 150⁰ C[4]	Severe	None	None	None
Crazing after 1 hr. x 200⁰ C[5]	None	None	None	None
Flexibility[6]	Borderline 2x	Passes 2x	Borderline 1x	Passes 1x
Inter-coat Adhesion	Good	Good	Fair	Fair
Unidirectional Scrape Resistance (KG) (0.009")	-	1.02-1.17	1.06-1.27	1.19-1.50
G.E. Scrape	40-70	15-35	20-40	25-45
Dielectric Strength	8.5kV	12kV	11kV	11kV
Oil & Water Res.[7]				
Flexibility	Poor	Good	Good	Good
Dielectric Strength	2kV	7kV	8.5kV	5.5kV

1. Clean wires with soft cloth and bend into U shape. Dip wire in mercury. Connect one end of wire to bridge; place the other lead from the bridge in mercury. Make measurements at 25°C at 1000 cycles/sec
2. Weigh 70 grams of wire de-greased with acetone wet cloth. Bake 5 minutes at 300°C or 2 hours at 200°C. Cool for 2 minutes and weigh.
3. Wrap wires on 1x through 6x mandrels and dip N-methylpyrrolidone. Examine under 10x microscope. Many other solvents will give similar results.
4. As above, but bake coil for 30 minutes at 150°C before dipping in solvent.
5. Same as #4, but bake 1 hour at 200°C
6. Quick snap by NEMA Snap test and wrap on 1x and 2x mandrels.
7. Place NEMA twists in 7" (177.88mm) length of 3/4" (19.05mm) iron pipe. Add 50cc of transformer oil and three drops of water. Seal pipe ends and age 88 hours at 200°C. Cool. Remove wires and measure dielectric strength and note flexibility.

Comparison of Pyre-M.L. with other Enamels

The data in table 11 compare Pyre-M.L. and "Formvar" Wire Enamels applied on #18 Copper wire. The conditions under which the tests were conducted are defined in the footnotes. The results represent average values for a number of determinations and may vary somewhat from values determined by other methods.

Application of PYRE-M.L.:

As mentioned before, Pyre-M.L. is a solution of aromatic polyamic acid. The polyamic acid is then converted to an insoluble polyimide upon curing i.e. imidization. Water is formed during the cure.

Material of Construction

Pyre-M.L. Wire Enamels are weak acids, and hence, will react with iron or mild steel. Therefore, all equipment which come in contact with the enamel should be made of stainless steel, polyethylene, polypropylene or other appropriate material. The equipment include dies used for removing excess enamel from the wire, coating bath, rolls, reservoir for the enamel, pumps, filtration equipment, etc. If iron is leached from the contact surface into the enamel, the resulting dissolved iron has a detrimental effect on the enamel, and hence, the coated wire.

Table 11

Comparison of the Initial properties of Wire Coated with PYRE-M.L.® Wire Enamel and Another Commercial Enamel (applied on #18 copper wire) - 1.024mm

Test	PYRE-M.L.	"Typical Formvar"
Thermal Classification	220°c	105°c
Build (diam. increase)	0.0030 inch (0.076 mm)	0.0030 inch (0.076 mm)
Dry Dielectric Strength[1]	3400 volts/mil	2000 volts/mil.
Wet Dielectric Strength[2]	1900 volts/mil.	600 volts/mil.
Insulation Resistance	Infinite	Infinite
Dielectric Constant		
(100 cycles)[1]	3.9	3.71
(1000 cycles)	3.9	3.64
Dissipation Factor		
(100 cycles)[1]	0.0040	0.0275
(1000 cycles)	0.0040	0.0220

Table 11 (Continued)

Test	PYRE-M.L.	"Typical Formvar"
G.E. Scrape Abrasion[3]	15-35 scrapes	60-80 scrapes
Unidirection Scrape[4]	1.3-1.5kg	1.02-1.17kg
Cut Through Temperature[5]	Above 500°C	240°C.
Extractables[6]	Less than 0.1%	3.0-6.0%
Heat Shock[7]	Passes to 425°C	Passes to 124°C
Solderability	Poor	Fair
Chemical Resistance[8]		
5% Sulfuric Acid	V. Good	Good
5% Hydrochloric Acid	V. Good	Good
1% Potassium Hydroxide	Poor	V. Good
Quick Snap Flexibility	Passes	Passes
Quick Snap Wind on own diam.	Passes 2x	Passes 1x
...Solvent Resistance[8]		
Xylene	V. Good	V. Good
Cresylic Acid	V. Good	Poor
Carbon Tetrachloride	V. Good	V. Good
Naphtha	V. Good	V. Good
Acetone	V. Good	V. Good
Alcohol	V. Good	Good
Ethyl Acetate	V. Good	Fair
Hexane	V. Good	Good
Transformer Oil	V. Good	Good
Crazing (solvent Stress)	Passes if annealed	Crazes, cracks heal at 150°C
FREON*22 Resistance[9]	Passes	Fails

The Pyre-M.L. described above is baked to a moderate degree of cure.

Footnotes for Table of Initial Properties

1. At room temperature and humidity
2. Standard NEMA twist. Soak in distilled water 24 hours, remove, clip loop, shake off excess water and test.
3. Standard test, 700 grams load. At prevailing atmospheric conditions.
4. Indiana Technical College Unidirectional Scraper (0.009" needle).
5. ASTM D 1676
6. Standard Soxhlet type equipment. Extraction with toluene for 12 cycles each lasting for 15 minutes followed by extraction with methanol for six cycles each lasting 20-25 minutes.
7. Wind on own diameter, then heat for one hour and examine for cracks.
8. Immerse in various reagents for 24 hours at room temperature. Examine for blistering, swell and softening.
9. A. four days immersion in liquid FREON 22 at 60°C. Place in 120°C oven (immediately after opening bomb) for 10 minutes. Observations made at 20x magnification. Mandrels of 1x, 3x, 5x and strength all unaffected.
   B. Stressed PYRE-M.L. placed in tube containing FREON and refrigerant oil. Held under pressure at 120°C. No evidence of film failure at six weeks.
   + If crazed, crazes will not heal. Annealing for 30 minutes to one hour at 175-200°C recommended.

Application Techniques

Except for the use of stainless steel or polyethylene equipment, Pyre-M.L. is applied in much the same manner as "Formvar" polyvinyl acetal enamels. Techniques used for coating different wire sizes are as follows:

Large Size Wires

Round wires larger than about No. 12 AWG (2.05mm or .0808 inches diameter) and square and rectangular wire are usually coated on vertical ovens about 24ft. (7.3 meters) high. The best results have been obtained on ovens which have forced convection heat (up draft in the bottom and down draft at the top) supplemented by radiant heating panels near the bottom of the oven. Acceptable wire can be produced with other types of ovens.

The optimum oven operating temperatures will have to be determined on each oven, but in a two zone oven a bottom zone temperature of about 650°F (343°C) and a top zone temperature of about 900°F (482°C) will probably allow the most economical coating speeds. If a radiant panel is used, a panel temperature of 1000° F (538°C) has been found to be best. Excellent adhesion has been obtained by curing the enamel under these conditions to a dissipation factor of 0.002 at 1000 cycles.

The dies used for square and rectangular wire should be made of stainless steel. Dies of any type of design which have given satisfactory results with polyvinyl acetal type enamels will probably be satisfactory for Pyre-M.L., if they are made of stainless steel.

The two common problems which have been encountered in coating rectangular wire are lack of adhesion and smoothness. Adhesion is controlled by the temperature at which the enamel is cured and the degree of cure. Adhesion is usually improved by increasing wire metal temperature near the bottom of the oven. Radiant heating panels are particularly useful for improving adhesion. Smoothness can be improved by adjusting the die sizes or by slight reductions in the viscosity of the enamel with N-methyl-2--pyrrolidone. The optimum viscosity will usually be the minimum viscosity which gives adequate corner coverage.

Medium Size Wires

Wires from about No.13 (0.072 inch or 1.83mm in diameter) to about No. 28 (0.0126 inch or 0.32mm in diameter) are coated on either horizontal or vertical machines. Phosphorbronze and stainless steel dies are being used commercially.

A typical oven pattern for an 18ft. (5.5 meter) laboratory vertical oven is describes in Table 12.

Table 12

Feet from Bottom	Temp., °F	Meters from Bottom	Temp., °C
1	225	.3	107
3	260	.9	127
5	310	1.7	154
7	460	2.3	238
9	570	3.0	299
11	585	3.7	307
13	600	4.3	316
15	700	5.0	371
17	725	5.7	385

The optimum heat pattern for any particular oven must be found experimentally. In general, the best results are obtained by adjusting the temperature in the bottom zone of the oven cool enough to prevent immediate blistering, but not enough to evaporate sufficient solvent to prevent blistering in the top zone. The top zone temperature is adjusted to give adequate cure.

Fine Wires

Pyre-M.L. is usually applied to wires smaller then No. 28 (0.0126 inch or 0.32mm in diameter) on horizontal machines using stainless steel roller dies.

To obtain smooth wire it is usually desirable to dry pass fine wire once through the oven before applying any enamel. This helps remove any oil or dirt. If necessary, Pyre-M.L. can be reduced about 10% with either NMP or Aromatic Hydrocarbon for application to fine wire. The solvent should be added slowly with good agitation.

Degree of Cure

Three round wire samples were coated under different conditions with an 18ft. L.M. machine. The first sample is badly under baked, the dissipation factor is .023 and the loss in weight of the coated wire is 19.1% of the weight of the coating when heated for five minutes at 300° C. The second sample is cured to the degree typical of much of the commercial polymide coated wire. The third sample is well cured. The results are described in Table 13.

Table 13

Curing PYRE-M.L.

Temp. °C	Speed Ft/Min	D.F. at 10^3c.p.s.	Weight Loss 5min at 300°C
55/315	35	.023	19.1%
55/370	25	.004	2.9%
135/515	25	.0015	1.4%

The dissipation factor is related to the energy necessary to realign the polar groups on the molecules each time the field changes. Changes in dissipation factor therefore reflect the loss of carboxyl groups, amide groups, water and N-methyl pyrrolidone during cure. Dissipation factor is the most precise way we know for measuring degree of cure.

Mandrel wrap test after quick snap illustrate the dependence of flexibility on degree cure. In contrast to most enamels, Pyre-M.L. has better flexibility when thoroughly cured than when under cured. They are shown in Table 14.

Table 14

Flexibility No. 18 AWG Wire

D.F. at 1000 c.p.s.	Snap & Wrap
.02	2X - 3X
.004	2X
.0015	1X - 2X

Panels were prepared by casting films (0.8-1.0 mils dry) on a 22mil thick copper panel. All panels initially baked at 10 minutes at 150°C to remove solvents before being given the bake above. Adhesion was tested with a sharp knife.

The adhesion to copper is a function of the temperature of cure. Even under cured Pyre-M.L. can have good adhesion if it is baked at a high temperature. All of these wires have thoroughly cured coatings, but the adhesion is influenced by both the degree and the temperature of cure.

Subsequent experiments have shown that radiant panel temperatures in the range of 538°C - 593°C are even better than the 482°C temperature used above.

Adhesion was determined after elongation to break, elongation of 30%, and bending on a 1" mandrel. All three tests gave identical results. Various adhesion results are shown in Tables 15 and 16.

Table 15

Adhesion to Panels

Minutes	Temperature	Adhesion	D.F.
0.5	400°C	Excellent	.017
1.0	"	"	.007
2.0	"	"	.004
5.0	"	"	.003
2.0	200°C	Poor	.06
8.0	"	"	.02
20.0	"	"	.011

Table 16

Adhesion to No. 3 Square Wire

Temperature °C Bottom	Top	Panel	Radiant Speed Ft./Min	D.F. at 10^3 cps	Adhesion
310	465	Off	9	.0022	poor
315	465	427	9	.0018	poor
315	465	427	7	.0019	fair
315	465	482	7	.0015	fair
315	488	482	7	.0017	good

Recirculating Gas Ovens

The most common types of ovens used for rectangular Pyre-M.L. coated wire have forced air convection heat which is updraft in the bottom section of the oven and down draft in the top section. The convection heat is supplemented by electrically heated radiant panels near the bottom of the oven. The solvents are catalytically burned in the air stream after it has passed through the bottom section of the oven.

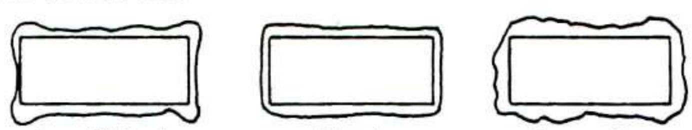
Data from a number of trial runs and demonstrations are shown in Tables 17 & 18. In several cases these do not represent the optimum production conditions, but rather the first set of conditions we could find to produce acceptable wire.

All of the conditions shown in this table produced wire with good inter-coat adhesion and adhesion to copper. For wire between 0.114 and 0.500 inch wide, between .096 and .229 inch thick and having a cross-sectional area between 1.3×10^4 square mils, the approximate speed is given by the following expression:

$$\text{Speed (ft./min.)} = \frac{4 \times 10^5}{\text{cross section area (sq. mils)}}$$

Corner Coverage

Conventional universal wire enamel dies deposit excessive enamel at the corners. The ideal combination enamel and coating conditions allow just the right amount of flow to leave the same thickness of enamel on the corners as there is on the flat surface, excessive flow leaves the corners thin.



Insufficient Flow — Ideal Flow — Excessive Flow

Insufficient flow ("dog bone") can be corrected by lowering the viscosity, by decreasing the volatility of the solvents and thinners, by decreasing the temperature in the bottom oven, or by decreasing speed.

Table 17

Radiantiy Heated Ovens

The data below show conditions used on three radiantly heated ovens.

	Test 1	Test 2	Test 3
Oven Type	electric	indirect gas	electric
Oven Hight	20 ft.	20 ft.	10 ft.
Temperature			
3 ft	-	-	310°C
5 ft.	-	313°C	-
6 ft.	288°C	-	400°C
10 ft.	343°C	432°C	377°C
15 ft.	400°C	466°C	x
17 ft.	-	493°C	x
Wire Size	.040 x. .200	098 x .300	.070 x .190
Speed (ft./min.)	10.5	10	4.5
No. of Coats	8	6	10
Build-width (mils)	3.2	2.5	4.5
Build-thickness (mils.)	5.7	4.0	4.1
Dielectric Strength (kv)	8.0	5.5	8.0
Type of Dies	cone	universal	universal
Enamel Code	RC-5057	RC-5019	RC-5019
Viscosity (poises)	52	63	28

Table 19

Overload Resistance

(NEMA Method)

	$\Sigma I^2t/10^5$
Polyimide	13.70 - 17.70
Polyamide-imide	8.06 - 10.01
Polyester	3.34 - 5.87
Formvar	2.27 - 6.31
Nylon/Formvar	2.85 - 5.48

Selected data from:
"Overload Test" by W. W.Pendleton of Anaconda Wire & Cable given at NEMA Magnet Wire Committee Presentation, Insulation Conference, Chicago, October 17,1967.

Table 20

Overload Resistance

(Essex Method)

	Minutes	
	30 amp.	35 amp
Polyimide	>5	1.32
Polyvinyl Formal	1.57	.83
Nylon/Formal	1.70	.90
Polyester	1.75	.90
Nylon/Polyester	1.06	-
Polyester-imide	1.70	.92
Amide-imide/ Polyester	2.50	1.08

Selected data from:
"Magnet Wire Temperature at Overload Conditions" by E.W. Daszewski of Essex Wire Corperation, Proceeding of Seventh EI Conference, p. 137.

Table 18

Production Trials Recirculating Gas Ovens Rectangular Wire

Wire Sizes (inches)	Area Sq. Mils. x10^4	Speed Ft./ Min.	Coats	Build Thickness	Build Width	Bottom Air °C	Top Air °C	Radiant Panel °C	Viscosity at 25°C	Dielectric Strength (KV)
.229 x .229	5.23	7	6	3.8	4	316	488	482	60	6.5
.100 x .500	5.00	8-1/2	6	4.4	2.6	316	488	482	60	5.0
.114 x .228	2.60	10	6	-	-	316	488	455	30	-
.229 x .229	5.23	7	6	4.5	5.0	316	482	455	30	7.5
.114 x .114	1.30	11-½	6	4.5	5.0	316	482	455	30	5.2
.096 x .229	2.87	13-1/2	8	5.0	4.0	343	493	565	12	3.5
.170 x .270	4.59	8	8	5.0	-	280	480	470	60	7.3
.060 x .210	1.26	16	4	2.0	2.0	232	538	577	32	-
.170 x .250	4.24	8	8	5.0	6/5	277	450	550	14	3.5
.140 x .280	3.92	11	8	-	-	277	450	550	14	4.0

Flow is also controlled by the cross section of the bare copper. The greater the radius of the corner, the less the tendency to flow away from it. Sharp corners, burrs, and abrupt changes in curvature are hard to coat.

Enamel Circulation and Application

Pyre-M.L. wire enamel should be pumped, filtered and recirculated with an all stainless steel system. Contamination with iron can lead to gelation, rough wire and decreased thermal stability of the coated wire.

The most common dies are stainless steel models of the universal grooved pin dies. The disc type of die covered by G. E.'s patent #2,875,725 is also used in production.

Our experience has been that the preferred groove size is 15 mils. If the width of the wire is more than three times the thickness it may be necessary to limit the build on the edges by using groove-less die pins on the edges on some of the coats. This should not be don on the first coat. An increase in both width and thickness of at least 0.5 mils is preferred on the first coat to promote good adhesion and prevent oxidation of the copper.

Smoothness

Occasionally one encounters a ripple or "orange peel" in the surface of the enamel where one or more of the dies is applying a thick film. This sometimes results in periodic "globs" or even blisters at or near the corners. This can be corrected by reducing the film thickness with smaller die grooves, reducing the surface tension with "flow agent" or hydrocarbon thinners, or by lowering the viscosity.

Streaks

Conventional dies apply enamel in streaks. If the enamel does not flow to a uniform film on the first coat, there will be streaks caused by the oxidation of the copper where the first coat is thin. With relatively transparent enamels, this streak appearance is not hidden by the subsequent coats. It is not clear whether the objection to these streaks is based on engineering or aesthetic reasons, but they are difficult to correct without abandoning the convenience of universal dies.

With conventional dies, the streaks can be minimized, but usually not eliminated, by reducing the viscosity and applying a full wet coat on the first pass.

Blistering

Blistering is generally due to insufficient heat or too much heat in the bottom of the oven. It is, of course, a function of film thickness, any condition such as excessive corner coverage, rippling, irregular jerking of the wire, excessive variation, etc. which causes localized areas of excessive film thickness will exaggerate blistering.

Ovens which lack heat capacity in their bottom section can usually be run faster with RC-5069, which contains a fairly volatile hydrocarbon diluent than with the pure N-methyl pyrrolidone type enamel such as RC-5019. In contrast, short ovens with plenty of bottom zone heat capacity will run fastest with RC-5019.

Blistering is caused much more readily by convection heat, which skins over the wet film, than by radiant heat, which tends to heat from the metal.

Curing blistering is usually easy if one is willing to accept slower coating speeds. Otherwise it involves the careful selection of the solvents and oven pattern to optimize speed. On modern, high-heat capacity equipment, blistering will ultimately limit the speed.

Precipitation

If the polyamic acid polymer in Pyre-M.L. is cured to the polyimide before sufficient solvent is evaporated to lead to film formation, the polyimide will precipitate from solution as a yellow powder. This is frequently encountered when one attempts to cure thick films rapidly. When encountered, it is usually caused by excessive heat at the bottom of the oven. As with blistering, this phenomenon is a function of thickness, and it is exaggerated by poor flow, jerky wire movement, and vibrations which lead to localized thick films.

Pyre-M.L. tends to craze or crack when coated wire is first bent and then dipped in solvent or varnish. This tendency is dependent on the degree of cure. Crazing may be prevented by stress relieving the enamel with heat after the wire is bent or wound into a coil. We recommend stress relief for all coils before varnishing or de-greasing. See Table 21.

Table 21

Solvent Crazing

	Stress Relief		
D.F. at 10^3c.p.s.	None	30 min./ 150° C	60 min./ 220° C
.01	Severe	Slight	None
.004	Moderate	None	None
.002	Slight	None	None

TEST

Twisted pairs of 18HML sealed in iron pipe with 50g of 10C oil and 3 drops of water for 88 hours at 200ºC. Twists tested immediately for removal.

Oil filled transformers normally operate below 150ºC. This is an accelerated screening test to select materials or further evaluation in transformer insulation systems. Thorough cure improves oil and water resistance. The results are shown in Tables 22 and 23.

Table 22

Cure vs. Oil & Water Resistance

D.F. at 10^3c.p.s.	**Dielectric Strength (KV) Initial**	**Final**
.01	9.3	2.4
.004	11.5	7.0
.002	10.8	8.5

Table 23

Transformer Fluid Resistance
Varnished Twists
18 HML

	PYRE-M.L.	**VARNISH "X"**
Oil	10.0 KV	12.2 KV
Oil & Water	9.2 KV	8.3 KV
Askeral	11.2 KV	10.2 KV

This wire had a dissipation factor of about .004 before varnishing. Test conducted as above.

Major Advantages of PYRE-M.L.:

Thermal Stability
Overload Resistance
Chemical Inertness except alkali
Radiation Resistance

Uses for PYRE-M.L.:

Transformers
Hermetic Motors
Traction Motors
Appliances
Power Tools
Mine Motors
Aircraft Wire
Aerospace Wire
Gas Chromatograph Capillaries
Semi Conductor Coating
Alignment Coating
Instrument/Sensor Wire Coating

Varnishing Magnet Wire Coated with PYRE-M.L.

Pyre-M.L. Insulating Varnishes RK-692 is recommended for use with magnet wire coated with Pyre-M.L. (See Bulletin ML-2 95) This varnish has outstanding thermal stability and mechanical and electrical properties. They maintain their strength and electrical properties at temperatures above 250ºC.

Insulating systems employing wire coated with Pyre-M.L. with certain silicone, epoxy, and phenolic varnishes are in commercial use and are giving satisfactory results. Certain commercially available varnishes degrade the thermal stability of wire coated with Pyre-M.L. according to laboratory results. All systems should be thoroughly tested before commercial use.

Wire coated with Pyre-M.L. should be heated for 30 minutes to one hour at 175ºC to 200ºC after winding and before varnishing to relieve stresses in the film and to prevent crazing of the wire enamel by the varnishing solvents.

Safety and Handling Procedures

While we are not aware of any health problems attributed to the use of this and related products during the past 20 years, good industrial hygiene should be practiced. Pyre-M.L. wire enamels and varnishes should be handled in well-ventilated areas, eye contact must be prevented, and skin contact should be minimized.

The TLV for Xylene is 100ppm (ACGIH). If ventilation is not adequate to maintain work place air concentrations below this level, NIOSH/MSHA approved respirators should be worn. Eye protection and Neoprene gloves should be used.

Animal tests have shown that these materials are only slightly toxic when ingested. They do cause skin irritation after prolonged or repeated contact, and any inadvertent skin contamination should be promptly washed off with water. In the event of eye contact, flush immediately with water for 15 minutes and see a physician.

The solvents typically used with Pyre-M.L. are extremely flammable and must be kept away from sparks, open flame, or other sources of ignition. Although we have not run tests, we would predict the by-products of combustion would be toxic by inhalation.

Waste disposal of Pyre-M.L. should be carried out in an EPA approved incinerator. Under these conditions, there is enough oxygen present that toxic by-products are minimized. The disposal of Pyre-M.L. must comply with Local, State and Federal regulations.

FDA Status

Pyre-M.L. products are not FDA approved. I.S.T. neither warranties these products nor sanctions their usage in medical applications unless explicitly approved.

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PYRE-M.L. INSULATING VARNISH - RK-692

Introduction

Pyre-M.L. products are part of a family of materials based on aromatic polyimides which are one of the most thermally stable organic materials. These products include Pyre-M.L. Wire Enamels, Pyre-M.L. Liquid "H" Enamels, Pyre-M.L. Insulating Varnish, and Pyre-M.L. Thinner. Table 1 (see page 1) describes these products, associated code/grade, and ML Technical Bulletin number.

RK-692 is a high temperature varnish developed for use in conjunction with Pyre-M.L. enameled wire. It is a solution of aromatic polyamic acid in N-methyl pyrrolidone. The polyamic acid is produced by reacting the aromatic diamine with the aromatic dianhydride. The application process involves drying of the solvent from the solution followed by curing and baking of the polyamic acid left behind. During the latter process, water is removed, and the polyamic acid turns into polymide film. The film so formed is mechanically tough, chemically inert, thermally stable, and radiation and cryogenic resistant. It also has excellent electrical insulating properties which remain essentially unchanged over a wide range of temperatures for extended time periods. Refer to Tech. Bulletin ML-1-95 for a detailed description of these properties. Pyre-M.L. products are U.L.® approved.

Product Properties

A. Liquid Varnish:

Pyre-M.L. Varnish is a coating solution which is converted into polyimide film by the applicators. These product properties/specifications are described in Table 24. These meet the commercial and military specifications required by the industry.

Table 24

PYRE-M.L. PRODUCTS SPECIFICATION

Code	RK-692
Solids (%)(1)	11.5 - 12.5
Viscosity (Poise) (2)	6 - 10
Solvent System	
NMP (%)	75 - 85
Xylene (%)	15 - 25
Density	
LBS/Gal (+/-0.05)	8.59
KGS/Gal (+/-0.023)	3.90

Note - (1) NEMA Test Method - 2gm/2hr. /200°C Bake
(2) Measured at 25°C with Brookfield Viscometer #3 Spindle @ 12 RPM.

B. Dried Film

The major properties of the varnished film are described below:

(1) Thermal Stability - excellent
Expected life at 220°C over 100,000 hours
Expected life at 240°C over 25,000 hours
Expected life at 300°C over 1,000 hours

(2) Electrical properties - excellent from -250°C to +250°C temperature. Electric strength exceeds 3000 volts per mil dry, and 1500 volts per mil when wet. It remains essentially the same over this entire temperature range. Dissipation factor (about .003 at 100 cycles at room temperature) increases only slightly and dielectric constant (about 3.8 at 100 cycles at room temperature) actually drops as the temperature is raised to 300°C. Insulation resistance is infinite when dry and $5x10^5$ megahms after boiling 10 minutes in water.

(3) Radiation Resistance - very good.
(Exposure to 3,000 megarads of electrons caused only minor changes in electrical and physical properties.)

(4) Representative values for thermal conductivity are:

PYRE-M.L.	35×10^{-5} cal/(sec) (Cm^2) (°C/CM)
Silicone	35×10^{-5} cal/(sec) (Cm^2) (°C/CM)
Epoxy	5×10^{-5} cal/(sec) (Cm^2) (°C/CM)
TEFLON® fluorocarbon	60×10^{-5} cal/(sec) (Cm^2) (°C/CM)

(5) Linear coefficient of thermal expansion:

PYRE-M.L.	4×10^5/°C
TEFLON®	5.6×10^5/°C
Epoxy	5 to 9×10^5/°C

(6) Coefficient of Kinetic friction:

PYRE-M.L.	0.17
Graphite	0.18
TEFLON	0.04 to 0.09

(7) Solvent and Oil Resistance - excellent

(8) Flame Resistance - excellent
(The dried film will not propagate flame although the varnish solvents are flammable.)

(9) Bond Strength by helical coil method on PYRE-M.L.:

25°C		6 to 8 lbs.
125°C		6 to 8 lbs.
150°C		6 to 8 lbs.
200°C		6 to 8 lbs.
300°C	after 4 weeks	3 to 5 lbs.

(10) Weight Loss:

300°C	after 3 weeks	1%

C. Properties of RK-692 Varnish are as indicated in Table 25.

Viscosity Stability

Viscosity, as manufactured, is not stable but gradually drops by 35%, and then increases to gel. Gelation occurs in approximately 14 days at 120°F (49°C), 30 days at 100°F (38°C), and 90 days at 77°F (25°C). At 40°F (4°C), it will not gel in one year. Pyre-M.L. is stored at 40°F in the Pyre-M.L. manufacturing plant warehouse in Parlin, New Jersey, USA.

Storage in an unheated building may be acceptable where winters are cold. Viscosity measurements are strongly affected by temperature. See Graph No. 2 (page 2) for temperature-viscosity conversion. Refrigeration is highly recommended for longer shelf life.

Moisture contamination has a detrimental effect on viscosity stability and must be avoided. Allow all chilled containers to reach room temperature before opening to avoid water condensation.

Table 25

Insulating Varnish Properties

Physical Properties	**RK-692**
Elongation at 25°C	10 - 25%
Tensile Strength at 25°C (10^3psi)	15
Weight Loss	
50 hours at 300°C	1%
150 hours at 300°C	1%
3.3 hours at 400°C	11%
3.3 hours at 450°C	20%
Bond Strength (lbs.)*	
Initial at 25°C	6 - 9
Initial at 180°C	6 - 8
at 25°C after 2 wks. at 300°C	-
at 180°C after 2 wks. at 300°C	-
at 25°C after 4 wks. at 300°C	3 - 5
at 25°C after 3 wks. at 260°C	5 - 7
at 180°C after 3 wks. at 260°C	3 - 5
Temp. up to which dissipation factor is essentially constant	300°C
Temp. at start of decomposition in air by thermal gravimetiric analysis (12°C/min. rate of rise)	540-550°C
Film thickness applied per coat (panel dipped at 4 in/min at 25°C) (in mils)	1.0
Dielectric Constant (25°C 100 cps)	3.5
Dielectric Strength (kV/mil)	4.0

Process

Application Procedure

Preheat the wound unit 1/2 hour or longer at 175° to 200°C to relieve stresses that may have been developed in the Pyre-M.L. enamel on the wire during winding.

Pyre-M.L. Varnish may be successfully applied by brush, vacuum impregnation, total immersion, end turn dipping, and roll over methods depending on the size of equipment being varnished and customer preference.

Stainless steel equipment is recommended, but inert or polyethylene plastic type lining of containers will suffice.

The key to the successful use of Pyre-M.L. Varnish is to remove the solvent before polymer conversion. Insufficient solvent removal can be detected after the fact by the presence of opaque yellow film or yellow powder. This means that the polymer was converted to its final insoluble, infusible form before the formation of a film. Adequate film formation takes place only by the evaporation of the solvent from a solution of the unconverted polymer.

Solvent removal will be facilitated by the use of well ventilated, forced convection ovens.

Best results are obtained by preheating the units before varnishing and allowing the heat form the mass to drive off most of the solvent before subjecting the unit to further bakes in the 100°C - 150°C range and final bakes at 215°C - 230°C.

When Pyre-M.L. enameled conductors are present in the unit being varnished, the windings stresses should be relieved by annealing the conductors at temperatures of 175°C to 200°C for at least 1/2 hour.

If units are being varnished by means other than total immersion or end turn dipping, for example spraying, temperature of the varnished unit should be limited to below 100°C to prevent bubbling of the film. Units of sufficient mass to provide 90°C - 100°C heat removal from varnish will tend to become tack free in a relatively short time.

Typical cure procedures for units employing the Pyre-M.L. system - conductor, coated fabric and varnish are as follows: All temperatures are metal temperatures. The length of time required to get the unit to temperature must be considered.

Small Servo Units

Reduce one part RK-692 with 2-3 parts T-8585 Solvent.

1. Preheat unit to 175°C - 200°C to relieve stresses.
2. Cool to below 150°C.
3. Immerse while hot in varnish until bubbling stops (approximately one minute).
4. Drain and allow solvent to flash. Hold for 15 minutes after all evidence of solvent flashing is no longer visible.
5. Bake 1 hour at 100°C.
6. Bake 1 hour at 150°C - Re-dip for one minute if necessary after 150°C bake. Repeat steps 4, 5, and 6.
7. Bake 1 hour at 215°C or higher.

1. Preheat unit to 175°C - 200°C to relieve stresses, then cool to below 150°C.
2. Immerse while hot in varnish until bubbling stops (approximately 3-5 minutes).
3. Drain and allow solvent to flash. Hold for at least 1/2 hour after all evidence of solvent flashing is no longer visible.
4. Bake 2 hours at 100°C.
5. Bake 2 hours at 150°C - Redip for one minute if necessary after 150°C bake. Repeat steps 3, 4, and 5.
6. Bake 2 hours at 215°C or higher.

The 100°C bake may be eliminated if windings are not tight.

1. Preheat to 175°C - 200°C to relieve stresses.
2. Cool down to 100°C.
3. Roll over in varnish at approximately 5-10rpm for five minutes. Drop or remove pan from heat radiating from unit. Continue rotating unit until tack free.
4. Bake 3 hours at 150°C.
5. Bake 2 hours at 215°C or higher.

In a continuous operation, the Pyre-M.L. Varnish in the tanks should be maintained at a temperature which will insure stability with relationship to consumption or the addition of fresh material. In some cases cooling may be necessary.

With periodic varnish applications, arrangements should be made to seal up and store the varnish under refrigeration while not in use (see "Viscosity Stability").

Pyre-M.L. Varnish may be applied to surfaces by spray, dip, roller coat, or brush. One spray coat will give approximately 0.2 mil (0.0051 mm) dry film. If heavier films are desired, these may be applied by multiple coats with a 5 to 15 minute bake at 150°C between coats to remove solvent. When desired thickness is obtained, the whole structure can be given the final high temperature bake necessary for conversion to polyimide.

Film thickness of 0.5 mil (0.0127 mm) per coat may be applied by dip, roll coat, or brush with little or no thinning. Multiple coats may be applied with complete solvent removal between coats at 100°C to 150°C for 5 to 15 minutes. A final high temperature cure may then be given the complete film.

These suggested baking equivalents may be used as a guide for determining the cure bake on Pyre-M.L. coating:

60 minutes	at 200° C
3 minutes	at 300° C
1 minute	at 400° C

These bakes represent ***minimum*** cure. An adequate baking schedule for a particular application should be determined by experiment.

Packaging

Stainless steel tanks and other containers are recommended for application of this varnish. Polyethylene lined containers are also adequate. The varnish should be kept out of contact with mild steel, brass, copper, and aluminum alloys containing over 1/2% copper to avoid polymer degradation. The status of other aluminum alloys is still doubtful.

Safety and Handling Procedures

While we are not aware of any health problems attributed to the use of this and related products during the past 20 years, good industrial hygiene should be practiced. Pyre-M.L. wire enamels and varnishes should be handled in well-ventilated areas, eye contact must be prevented, and skin contact should be minimized.

The TLV for Xylene is 100ppm (ACGIH). And while the ACGIH has not been established a TLV for n-methyl-pyrrolidone, IST has established an internal exposure limit of 25ppm. If ventilation is not adequate to maintain work place air concentrations below these levels, NIOSH/MSHA approved respirators should be worn. Eye protection and Neoprene gloves should be used.

Animal tests have shown that these materials are only slightly toxic when ingested. They do cause skin irritation after prolonged or repeated contact, and any inadvertent skin contamination should be promptly washed off with water. In the event of eye contact, flush immediately with water for 15 minutes and see a physician.

The solvents typically used with Pyre-M.L. are extremely flammable and must be kept away from sparks, open flame, or other sources of ignition. Although we have not run tests, we would predict the by-products of combustion would be toxic by inhalation.

Waste disposal of Pyre-M.L. should be carried out in an EPA approved incinerator. Under these conditions, there is enough oxygen present that toxic by-products are minimized. The disposal of Pyre-M.L. must comply with Local, State and Federal regulations.

FDA Status

Pyre-M.L. products are not FDA approved. I.S.T. neither warranties these products nor sanctions their usage in medical applications unless explicitly approved.

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PYRE-M.L. - LIQUID "H" ENAMEL - RC-5097

Introduction

Pyre-M.L. products are part of a family of materials based on aromatic polyimides which are one of the most thermally stable organic materials. These products include Pyre-M.L. Wire Enamels, Pyre-M.L. Liquid "H" Enamels, Pyre-M.L. Insulating Varnish, and Pyre-M.L. Thinner. Table 1 (see page 1) describes these products and their associated codes/grades.

Liquid "H" Enamel (also known as RC-5097) was developed for top coating insulated airframe and aerospace wires. RC-5097 is a solution of aromatic polyamic acid in N-methyl pyrrolidone. The polyamic acid is produced by reacting an aromatic diamine with an aromatic dianhydride. The application process involves drying of the solvent from the solution followed by curing and baking of the polyamic acid left behind. During the latter process, water is removed and the polyamic acid turns into polyimide film. The film so formed is mechanically tough, chemically inert, thermally stable, radiation and cryogenic resistant. It also has excellent electrical insulating properties which remain essentially unchanged over a wide range of temperature for extended time periods. Refer to Tech. Bulletin ML-1-95 for a detailed description of these properties. Pyre-M.L. products are U.L.® approved.

Description of Product

Pyre-M.L. Liquid "H" is a coating solution which is converted into polyimide film by the applicator. The product properties are described in Table 27. The specifications stated herein apply to RC-5097 used for top coating airframe and aerospace wires either for commercial uses or in compliance with military uses such as called out in Military specs MIL W-81381. Frequently, customers will pigment RC-5097 for color coding or cosmetic reasons. The procedure for facilitating pigmentation is described in Appendix I.

Table 27

PYRE-M.L. PRODUCTS SPECIFICATION

Code	RC-5097
Solids(%)(1)	10.5-11.5
Viscosity (Poise)(2)	105-135
Solvent System NMP(%)	75 - 85
Aromatic Hydrocarbon(%)	15 - 25
Density LBS/Gal (+/-0.05)	8.64
KGS/Gal (+/-0.023)	3.92

Note: (1) NEMA Test Method - 2gm/2hr./200°C Bake
(2) Measured at 25°C with Brookfield Viscometer #3 Spindle @ 6 RPM.

Viscosity Stability

The viscosity as manufactured, is not stable but gradually drops by 35%, and then increases to gel. Gelation occurs in approximately 14 days at 120°F (49°C), 30 days at 100°F (38°C), and 90 days at 77°F (25°C). At 40°F (4°C), it will not gel in one year. Hence, it is recommended that Pyre-M.L. be stored under refrigeration conditions prior to use. Pyre-M.L. is stored at 40°F in the Pyre-M.L. manufacturing plant warehouse in Parlin, NJ, USA. Storage in an unheated building may be acceptable during winter. Viscosity measurements are strongly affected by temperature. See Graph No. 2 (page 2) for temperature-viscosity conversion.

Moisture contamination has a detrimental effect on viscosity stability, and hence, must be avoided. Allow all chilled containers to reach room temperatures before opening them to avoid moisture from condensing.

Safety and Handling Procedures

While we are not aware of any health problems attributed to the use of this and related products during the past 20 years, good industrial hygiene should be practiced. Pyre-M.L. wire enamels and varnishes should be handled in well-ventilated areas, eye contact must be prevented, and skin contact should be minimized.

The TLV for Xylene is 100ppm (ACGIH). And while the ACGIH has not established a TLV for n-methyl-pyrrolidone, IST has established an internal exposure limit of 25ppm. If ventilation is not adequate to maintain work place air concentrations below these levels, NIOSH/MSHA approved respirators should be worn. Eye protection and Neoprene gloves should be used.

Animal tests have shown that these materials are only slightly toxic when ingested. They do cause skin irritation after prolonged or repeated contact, and any inadvertent skin contamination should be promptly washed off with water. In the event of eye contact, flush with water for 15 minutes and see a physician.

The solvents typically used with Pyre-M.L. are extremely flammable and must be kept away from sparks, open flame, or other sources of ignition. Although we have not run tests, we would predict the by-products of combustion would be toxic by inhalation.

Waste disposal of Pyre-M.L. should be carried out in an EPA approved incinerator. Under these conditions, there is enough oxygen present that toxic by-products are minimized. The disposal of Pyre-M.L. must comply with Local, State and Federal regulations.

FDA Status

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Industrial Summit Technology • Parlin, New Jersey 08859

PYRE-M.L. THINNER - T-8585

Introduction

Pyre-M.L. products are part of a family of materials based on aromatic polyimides which are one of the most thermally stable organic materials. These products include Pyre-M.L. Wire Enamels, Pyre-M.L. Liquid "H" Enamels, Pyre-M.L. Insulating Varnish, and Pyre-M.L. Thinner. Table 1 (see page 1) describes these products, their associated codes/ grades, and ML Technical Bulletin numbers. Pyre-M.L. products are U.L.® approved.

Description of Product

Pyre-M.L. Thinner (T-8585) is a mixture of N-Methyl-Pyrrolidone and aromatic hydrocarbon solvents in an 80/20 ratio. This product was designed to meet the "thinning" needs of Pyre-M.L. customers.

Application of PYRE-M.L. Thinner:

T-8585 is added on an as needed basis, to Pyre-M.L. enamels or varnishes to lower their viscosities and/or solids. The amount of T-8585 required depends upon the extent of viscosity/solids reduction desired. For reducing viscosity, T-8585 amount is estimated using past experience or running a small pilot in the laboratory. For reducing % solids, T-8585 amount is calculated by using the following formula:

T-8585 to be added (in Lbs. per 100 lbs. of Pyre-M.L.)

= (old solids in %) x 100 / (new solids in %) - 100.

If both the solid and viscosities need to be lowered, then lower the solids using the above formula. Recheck the new viscosity. If it is still high, then lower it by running a pilot or estimating from the past experience.

The thinner must be missed thoroughly with Pyre-M.L. prior to use.

Shelf Life

Three year shelf life is recommended for T-8585 from unopened containers. Frequent opening and closing of the T-8585 containers will lower its shelf life.

Storage Conditions

Unlike Pyre-M.L. wire enamels or varnishes, refrigeration is not required for Pyre-M.L. Thinner T-8585. Room temperature storage is adequate.

Materials of Construction

For thinning, stainless steel 316, HDPE, polypropylene, or other suitable material of construction is required for the mixing equipment and the contact surfaces.

Safety and Handling Procedures

While we are not aware of any health problems attributed to the use of this and related products during the past 20 years, good industrial hygiene should be practiced. Pyre-M.L. wire enamels and varnishes should be handled in well-ventilated areas, eye contact must be prevented, and skin contact should be minimized.

The TLV for xylene is 100ppm (ACGIH). And while the ACGIH has not established a TLV for n-methyl-pyrrolidone, IST has established an internal exposure limit of 25ppm. If ventilation is not adequate to maintain work place air concentrations below these levels, NIOSH/MSHA approved respirators should be worn. Eye protection and Neoprene gloves should be used.

Animal tests have shown that these materials are only slightly toxic when ingested. They do cause skin irritation after prolonged or repeated contact, and any inadvertent skin contamination should be promptly washed off with water. In the event of eye contact, flush with water for 15 minutes and see a physician.

The solvents typically used with Pyre-M.L. are extremely flammable and must be kept away from sparks, open flame, or other sources of ignition. Although we have not run tests, we would predict the by-products of combustion would be toxic by inhalation.

Waste disposal of Pyre-M.L. should be carried out in an EPA approved incinerator. Under these conditions, there is enough oxygen present that toxic by-products are minimized. The disposal of Pyre-M.L. must comply with Local, State and Federal regulations.

FDA Status

Pyre-M.L. products are not FDA approved. I.S.T. neither warranties these products nor sanctions their usage in medical applications unless explicitly approved.

Appendix I

COLORING LIQUID H POLYIMIDE WIRE ENAMEL

Introduction

Polymide jacketed wire, insulated with Teflon®, can be colored by three methods - topcoats, show through, and pigmentation. The natural color of cured Liquid H is yellow depending on coat thickness and curing conditions. If the polyimide is no more than one or two mils thick, the color of the Teflon® will show through although it will be distorted toward amber by the polyimide. Blues and white are particularly affected. Pigmenting the Liquid H produces colors that are unaffected by coating thickness, are most durable and less expensive than topcoats and retain the printing, potting and thermal properties of the base polyimide. Blues and whites cannot be ready by this route.

Description of the Process

Most pigments suitable for coloring Teflon® will perform well in Liquid H. A color concentrate will first be made in N-methyl-2-pyrrolidone. This is done by blending the ingredients to make a 20% (weight) pigment mixture and grinding 24 hours in a ball mill for five minutes in a homogenizer. The resulting color concentration should then be screened through a 200 mesh sieve to remove any large particles.

Liquid H is pigmented by adding one or more color concentrates and stirring with a suitable propeller type mixer. Since the natural polyimide color is so intense, a high pigment loading is needed to get reasonable colors. The highest loading attempted was 30% (based on the final product). At high loadings the polyimide becomes brittle; we do not suggest loadings beyond 20% without extensive testing. Concentrate addition to Liquid H should be done while the polymer is being stirred to get the best mixing effect. Because Liquid H is so viscous, a one hour mixing time should be allowed to ensure homogenous blend and the mixing energy should be ten horsepower per hundred gallons. Sample color formulations are shown in Table 28. They are calculated to produce 28% pigment in the final product.

Coating with pigmented Liquid H is the same as the natural product except the viscosity is slightly lower. Adding color concentrates raises both solids level and NMP content. Liquid H is 12% solids and the concentration 20%. No change in coating build should be noticed. The stripping die diameter will remain at 6-10 mils larger than the wire for 1/4 mil build per pass. The oven profile should be 250°F at the bottom to 450°F at the top and the wire residence time will remain at one to one and one quarter minutes per pass.

Safe Handling Practices

The organic solvents used in Liquid H are combustible. Keep away from heat and oven flame. Use only with adequate ventilation and avoid breathing vapors. Skin contact should be avoided. Exposed areas should be flushed with water immediately. Wash hands thoroughly after handling. Keep container closed.

Rubber gloves resistant to the solvents used with Liquid H can be purchased from Richmond Rubber Company, 618 East Leigh Street, Richmond, Virginia. Specify "Will-Guard" No. 64.

Some of the pigments used to color Liquid H are highly toxic. The manufacturer's suggestions for handling them should be learned and followed.

Storage and Handling

Pigmented Liquid H should be stored below 70°F. It must be kept in closed containers, free of moisture. Some pigments may tend to settle after prolonged storage and will require reconstitution immediately before use.

Table 28
LIQUID H COLOR FORMULATIONS

	Parts by Weight
YELLOW	
Liquid H 301 (12% solids)	100
White Conc. (20% TiPure R-900 in NMP)*	15
BROWN	
Liquid H 301	100
White Conc. (20% TiPure R-900 in NMP)	5
Brown Conc. (20% Ferro Brown V5102 in NMP)**	10
ORANGE	
Liquid H 301	100
Orange Conc. (20% Ferro Orange in NMP)	10
White Conc. (20% TiPure R-900 in NMP)	5
RED	
Liquid H 301	100
Red Conc. (20% Ferro Red V8845 in NMP)	14
White Conc. (20% TiPure R-900 in NMP)	1
BLACK	
Liquid H 301	100
Orange Conc. (20% Ferro Black V302 in NMP)	10
PURPLE	
Liquid H 301	100
Red Conc. (20% Ferro Red V8845 in NMP)	14
Blue Conc. (20% Ferro Blue V3285 in NMP)	1

* "TiPure" is a Du Pont Registered Trademark
** "Ferro is a Ferro Corporation Trademark