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CHARACTERIZATION OF
FILM ADHESIVES

EP 46385, Final Report

K. C. Atkinson, Project Leader

Published October, 1971

Prepared for the
United States Atomic Energy Commission
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**Kansas City
Division**

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BDX-613-528

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Project Leader:
K. C. Atkinson
Department 421

EP 46385
Final Report

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KANSAS CITY DIVISION
KANSAS CITY, MISSOURI

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ABSTRACT

Chemical characterization of four film adhesives (Ablefilm 501-T, Plymaster ACG 2080, CMC 11-2, and FM 123-5 currently in use at Bendix Kansas City) was performed by Bendix Test Laboratory for Sandia Laboratories Albuquerque to help select the best adhesive for microcircuits. Test results eliminated none of these candidate adhesives, and the outgassing products proved non-hazardous to the circuitry.

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SUMMARY

The purpose of this analysis is to assist in selecting an adhesive for micro-circuit application and to assist in choosing suitable acceptance tests. Specific goals are to

- Identify the resin system, cure agent, filler, and reinforcement
- Determine the amount of each in the system
- Determine the volatiles, ash content, and molecular weight distributions
- Subject the samples to thermomechanical analysis and to differential scanning calorimetry; and
- Determine epoxy equivalent weights on both B-staged and cured epoxy adhesives.

Other areas of interest are ease of processing and the possibility of circuit damage resulting from volatiles in the adhesive.

All analytical goals were met, except the identification of agents in the epoxy adhesives. Specific identities and amounts could not be determined. The adhesives tested were Ablefilm 501-T, Plymaster ACG 2080, CMC 11-2, and FM 123-5. In the absence of acceptance requirements, none of the data obtained eliminated any of these candidate materials and it is concluded that the outgassing products offer no hazard to the circuitry.

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DISCUSSION

SCOPE AND PURPOSE

Chemical characterization of four film adhesives was requested by George Volda of Sandia Laboratories Albuquerque. The adhesives are candidates for the manufacture of a ceramic microcircuit assembly. The selected adhesive is to be cut to an appropriate shape, laid up in the assembly, and cured with heat and pressure.

Data obtained will be used in selecting the adhesive and in choosing acceptance tests. Sandia has requested that Bendix test laboratory:

- Identify and determine the amount of each component in the system;
- Determine the volatiles;
- Determine the ash content;
- Determine the molecular weight distribution;
- Make a thermal analysis; and
- Determine the epoxy equivalents on both B-staged and cured epoxies.

Other major considerations are ease of processing and possible hazards to the circuits from volatiles in the adhesives. This work was performed under Purchase Order 11-2454, EP 46385.

ACTIVITY

Adhesives

CMC 11-2 is an unsupported film of butadiene-acrylonitrile mixed (and partly copolymerized) with a phenolic resole. It is nominally 4 mils thick, non-tacky, flexible, and easily stretched. Volatiles (run at 100°C for 1 hour) were 3.58 percent, mostly low molecular weight phenolics. The ash, 0.73 percent by weight, was calcium sulfate and zinc oxide. Volatility, stretchiness, and the lack of thickness were judged to be disadvantages.

Plymaster is a vinyl butyral phenolic copolymer with some free phenolic material. It is supported on a mat of random glass fibers, 14.9 percent by weight. It is 26 mils thick, non-tacky, and firm to the touch. Volatiles were 3.52 percent by weight, about the same as that of CMC 11-2. Only the volatiles were considered disadvantageous.

FM 123-5 is an epoxy resin supported on hosiery knit nylon 6-6 fabric (8.2 percent by weight). It is 9 mils thick but quite flexible and must be refrigerated when not in use. At room temperature, FM 123-5 is so tacky that it is difficult to remove the backing. Volatiles are 0.78 percent, and chlorine is 0.52 percent. The refrigeration requirement and the tackiness are disadvantages.

Ablefilm 501-T is an epoxy adhesive supported on glass cloth, 30.1 percent by weight. It is 4 mils thick and is brittle even at room temperature. Evidence indicates that it is a mixture of a standard diglycidyl ether of Bisphenol A (DGEBA) and an epoxy novolac cured with an aromatic amine. This system must be refrigerated at -40°F when not in use. Volatiles are 0.79 percent, and residual chlorine is 0.65 percent. The refrigeration requirement and the frangible character of the film are considered serious drawbacks.

Infrared Analysis

For identification purposes, infrared spectra of all the samples were run (Appendix B). A Perkin-Elmer Model 221 was used to record the spectra from samples solvent-cast on NaCl plates. Identification was based on comparison with reference spectra in the Sadtler collection and other sources.

The spectrum of the Plymaster adhesive is shown on page 27. The adhesive is a copolymer and mixture of vinyl butyral with a phenolic. The butyral is prepared by hydrolyzing vinyl acetate to vinyl alcohol. The vinyl alcohol polymer, which is essentially a 1,3 glycol, forms cyclic acetals by condensation with aldehydes. The alcohol and ester functionality are not completely converted; typically, the finished material will be 79 to 89 percent butyral, 9 to 21 percent alcohol, and 0 to 2.5 percent acetate. The phenolic is added as an additional cross-linking agent and perhaps to improve its temperature capabilities.

The spectrum of CMC 11-2, a butadiene-acrylonitrile phenolic resole adhesive formulation, is shown on page 28. Some of the phenolic component was extracted with alcohol (page 29). The unextractable portion seems to be reacted with the butadiene-acrylonitrile.

Ablefilm 501-T (page 30) is an epoxy material. Indications from the spectrum and from gel permeation chromatography are that it is a blend of an epoxidized novolac with a standard DGEBA resin. FM 123-5 (page 31) is an epoxy resin. Its support is nylon 6-6 (page 32).

Thermal Analysis

Differential scanning calorimetry (DSC) of CMC 11-2 shows a broad but low profile exotherm beginning at 124°C and peaking at 155°C. This profile

corresponds to the cure reaction (page 33). Thermogravimetric analysis (TGA) of CMC 11-2 shows a loss of volatiles from about 100 to 275°C, amounting to about 10 percent by weight. At roughly 300°C (after a pause) the rate of weight loss accelerates to 450°C. The rate of loss levels off between 450 and 500°C where the run terminates. It is probable that the initial loss (below 275°C) consists of low molecular weight phenolic species. Above 300°C, the cured polymer is being thermally degraded (page 34). Thermo-mechanical analysis (TMA) was not run on CMC 11-2.

The DSC of the uncured Plymaster material shows a small endotherm between 65 and 70°C not present in the scan of the cured material. The uncured samples also show what resembles a second order enthalpic transition about 200°C. The transition is weak in the uncured and much weaker in the cured material. Both cured and uncured materials show a large endotherm just below 400°C (page 35). TGA curves of Plymaster also show minor rate-of-loss changes at approximately 200°, and the steepest portion of the curve occurs just below 400°C (pages 36 and 37).

Thermomechanical analysis consists of recording the position of a weighted probe versus temperature. The Plymaster samples were run in the expansion mode, that is, with a broad foot on the probe. However, the probe sank into the Plymaster material, beginning at 58°C, pausing at 70°C, then sinking more slowly to the limit of travel. The cured material softened just above 60°C, and the probe stopped sinking between 80 and 100°C (pages 38 and 39).

The DSC of Ablefilm shows a large cure exotherm peaking at 140 to 144°C (pages 40 and 41). The TGA curves are very similar for both cured and uncured samples of Ablefilm. Both are stable to about 200°C, lose less than 2 percent by weight before 300°C, and then slide into major degradation, the steepest portion of which is between 350° and 450° (pages 42 and 43). A TMA was run in the expansion mode on uncured Ablefilm, anticipating softness. The probe sank immediately and rapidly, finally stopping when the temperature reached 125°C. The cured material, on the other hand, expanded through 65°C, and then allowed the probe to sink through 130°C (pages 44 and 45).

The DSC and TGA curves of the FM 123-5 were essentially identical to those of the Ablefilm. It is interesting to note that the cure exotherms are the same, although the recommended cure temperatures differ. Probably the optimum cure would be obtained at 135 to 140°C below the peak of the exotherm (pages 46 and 49). TMA penetration started at once and continued to 130°C. No expansion was observed (page 50).

Molecular Weight Distribution

Gel permeation chromatography is an analytical technique which effectively sorts polymer molecules according to size. A dissolved sample is washed

through a series of columns packed with porous beads. Pore sizes vary widely so that the smallest molecules may diffuse into many more pores than do the large molecules. Consequently, the largest molecules elute first, followed by decreasing sizes down to dissolved gases. Detection is by differential refractometer. If suitable standards are available, the elution times can be translated into molecular weights.

In this study no standards were available, although the DGEBA monomer could be identified. The distribution of sizes, however, is of interest. Both CMC 11-2 and Plymaster have bimodal distribution. The larger molecules of CMC 11-2, copolymers of butadiene-acrylonitrile and phenolic, elute first in a broad band from 20 to 30 counts. A set of phenolics of much lower weight comes out between 31 and 38 counts (page 51). Plymaster exhibits a similar distribution. Evidently the molecular weights correspond closely to those of CMC 11-2 (page 52).

The epoxy chromatograms indicate that FM 123-5 has a considerably higher percentage of monomer (the peak is just below 36) than does Ablefilm, which seems to have all the molecular weight species fairly evenly distributed. The two higher resolution runs (eight columns, small pore gel) emphasize the difference between FM 123-5 and Ablefilm (pages 53 and 56).

Mass Spectrometry

Each sample was inserted directly into the source of a CEC 21-110C mass spectrometer and analyzed at various temperatures. The resulting mass spectra were normalized, assigning the most intense peak the value of 100. Structural assignments were made by comparison with standard mass spectrum patterns of materials which might be present. Since the unknown spectra represent mixtures and the interpretation was made without computer assistance, the assignments are not complete, nor are they entirely certain.

No volatiles exist below 70°C in uncured FM 123-5. At this temperature the pattern could fit either an isobutyl ketone or a furfuryl alcohol. The pattern is largely replaced at 150°C by diethyl amine. At 215°C and above, polymer fragments make up the pattern. Outgassing was very much reduced above 215°C. The cured material did not outgas much at any temperature (pages 57 through 60).

At 80°C, the major effluent of Ablefilm appears to be carbon dioxide accompanied by acetone. With increasing temperature, phenolic fragments replace the carbon dioxide and acetone. Above 250°C the pattern stabilizes, showing adhesive fragments. Above 300°C and above mass 77, the mass spectra of Ablefilm and FM 123-5 are nearly identical (pages 61 through 66).

From 60° to 100°C evidence of water and aromatics are found in Plymaster. Water decreases after 100°C and is replaced by butyral fragments. At 150°C, water increases again, perhaps because of the onset of cure (pages 67 through 73).

Volatiles in CMC 11-2 were observed during the temperature interval of 50 to 130°C. No further outgassing took place until the temperature reached 330°C. The initial outgassing contained an abundance of short-chain, unsaturated hydrocarbons such as propene, and butene. Some water and phenolic material may be present. The high end of this interval (100 to 120°C) produces a spectrum similar to that of Bisphenol-F. When the temperature reached 330°C, further outgassing began. This pattern is probably made up of fragments from the thermal degradation of the polymer (pages 74 and 75).

Ash

To determine ash content various adhesive samples were ignited in a muffle at 600°C for 4 hours. The residues of Plymaster (14.9 percent) and of Ablefilm (30.2 percent) were taken as glass reinforcement; no non-glass ash was observed. CMC 11-2 was not reinforced. Its ash was 0.73 percent by weight and consisted of zinc oxide and calcium sulfate. Zinc oxide is a cure agent for butadiene rubber but is ordinarily used in concentrations of about 5 percent. FM 123-5 has an ash content of 0.24 percent. The residue was not identified. The nylon mesh support was, of course, burned off. Photomicrographs (110X) of the support layers are shown in Figures A-1 through A-5.

Epoxide Equivalent

The epoxide equivalent (equivalents of epoxy per 100 grams of resin) was determined by reacting the epoxy groups with HCl in dioxane. The unused HCl was back-titrated potentiometrically with silver nitrate. Ablefilm had an epoxide equivalent of 0.164, as received. No residual epoxy was detected after cure. The epoxide equivalent of FM 123-5 was 0.262, as received. Curing reduced the value to 0.02.

Elemental Analysis

Elemental analysis using a Perkin-Elmer Model 240 Elemental Analyzer was run on the four materials. The results are presented with other single-valued data in Table 1.

Table 1. Results of Elemental Analysis

Sample	C* (Percent)	H* (Percent)	N* (Percent)	Cl (Percent)	Ash** (Percent)	Volatile*** (Percent)	EE****
CMC 11-2	77.82	8.05	4.95	0.04	0.73	3.58	--
Plymaster	65.00	9.22	--	0.01*	14.9	3.52	--
Ablefilm	65.80	6.73	1.42	0.65*	30.2	0.79	0.164
FM 123-5	65.96	7.11	3.59	0.52	0.24	0.78	0.262

*Figures adjusted to exclude reinforcement.

**Residue after ignition at 600°C for 4 hours.

***Weight loss during 1 hour at 100°C.

****Epoxy equivalents per 100 grams of resin.

Appendix A
PHOTOMICROGRAPHS

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Figure A-1. Ablefilm 501-T Before Ignition:
Uncured (110X)

Figure A-2. Ablefilm 501-T After Ignition:
600°C (110X)

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Figure A-3. Plymaster FP 97-H Before Ignition:
Uncured, Master Roll 1-165 (110X)

Figure A-4. Plymaster FP 97-H After Ignition:
600°C, Master Roll 1-165 (110X)

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Figure A-5. Nylon Mesh FM 123-5 (110X)

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Appendix B
CHARTS

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Polyvinyl butyral

SPECTRUM NO. _____	ORIGIN _____	LEGEND _____	REMARKS <i>thin film</i>
SAMPLE <i>Plymaster</i>		1. _____	<i>from HCCl₃ (uncured)</i>
<i>FP-97-H</i>	PURITY _____	2. _____	<i>on NaCl disk</i>
<i>Master roll 1-165</i>	PHASE _____	DATE <i>1-11-71</i>	
	THICKNESS _____	OPERATOR <i>L. J. J.</i>	

SPECTRUM NO. _____
SAMPLE _____

SPECTRUM NO. _____	ORIGIN _____	LEGEND _____	REMARKS _____
SAMPLE <u>CMC</u>		1. _____	<u>Cast from MeCl₂</u>
<u>11-2 Adhesive</u>	PURITY _____	2. _____	
	PHASE <u>Film</u>	DATE _____	
	THICKNESS _____	OPERATOR <u>KCA</u>	

SPECTRUM NO. _____
SAMPLE

SPECTRUM NO. _____	ORIGIN _____	LEGEND _____	REMARKS _____
SAMPLE _____	<i>isopropanol</i>	1. _____	<i>phenolic</i>
<i>CMC 11-2</i>	PURITY <i>extract</i>	2. _____	_____
_____	PHASE _____	DATE _____	_____
_____	THICKNESS _____	OPERATOR <i>KCA</i>	_____

SPECTRUM NO. _____
SAMPLE _____

SPECTRUM NO. _____	ORIGIN _____	LEGEND _____	REMARKS <i>film from</i>
SAMPLE <i>Able film</i>		1. _____	<i>HCCl₃</i>
<i>501</i>	PURITY _____	2. _____	
	PHASE _____	DATE <i>1-6-71</i>	
	THICKNESS _____	OPERATOR <i>Dubiel</i>	

SPECTRUM NO. _____
SAMPLE

SPECTRUM NO. _____	ORIGIN _____	LEGEND _____	REMARKS _____
SAMPLE _____	_____	1. _____	<i>cast from MeCl₂</i>
<i>FM 123-5</i>	PURITY _____	2. _____	_____
_____	PHASE <i>film</i>	DATE _____	_____
_____	THICKNESS _____	OPERATOR <i>KCA</i>	_____

SPECTRUM NO. _____
SAMPLE _____

SPECTRUM NO. _____	ORIGIN _____	LEGEND _____	REMARKS _____
SAMPLE <u>Support</u>		1. <u>0.3% in KBr</u>	
<u>from FM 123-5</u>	PURITY _____	2. _____	
	PHASE _____	DATE _____	
	THICKNESS _____	OPERATOR <u>KCA</u>	

SPECTRUM NO. _____
SAMPLE

PART NO. 900325

DU PONT INSTRUMENTS

SAMPLE: <i>CMC - 11-2</i> <i>Adhesive</i> <i>uncured</i>	SIZE <i>Large</i>	ATM. <i>N₂</i>		RUN NO. _____
	REF. <i>Empty Pan</i>	T _____	ΔT _____	DATE <i>2-3-71</i>
ORIGIN:	PROGRAM MODE <i>Heat</i>	SCALE SETTING <i>20</i> $\frac{\%}{\text{in}}$ <i>0.5</i> $\frac{\%}{\text{in}}$		OPERATOR <i>Long</i>
	RATE <i>10</i> $\frac{\%}{\text{min}}$ START _____ °C

PART NO. 950251

 INSTRUMENT PRODUCTS DIVISION
 DuPont Instrument Products Division

SAMPLE: <u>Plymaster</u> <u>OSC</u> <u>Adhesive</u> <u>Cured & Uncured</u> ORIGIN:	SIZE <u>Large Par</u>	ATM. <u>N₂</u>	RUN NO. _____
	REF. <u>Empty Par</u>	T _____	DATE <u>1-11-71</u>
	PROGRAM MODE <u>Heat</u>	SCALE SETTING <u>50</u> °C <u>0.5</u> °C	OPERATOR <u>L. Jones</u>
	RATE <u>10</u> °C/MIN, START _____ °C

PART NO. 950251

 DU PONT
 INSTRUMENT PRODUCTS DIVISION
 WILMINGTON, DELAWARE

SAMPLE: <i>Plymaster Adhesive Coated film PPO Ind. Uncoated</i> SIZE <i>144</i> mg.	X-AXIS		Y-AXIS		RUN NO.	DATE <i>1-5-51</i>
	TEMP. SCALE <i>50</i> °C inch		SCALE <i>2</i> mg. inch (SCALE SETTING X 2)		OPERATOR <i>Long</i>	
	SHIFT <i>0</i> inch		SUPPRESSION		HEATING RATE <i>10</i> °C min.	
	TIME SCALE (ALT.)		mg.		ATM. <i>N₂</i>	
				TIME CONSTANT <i>1</i> sec.

PART NO. 900325

INSTRUMENTS

SAMPLE: <i>Plymaster</i> TMA Expansion Cured	SIZE <i>0.51 mm</i>	ATM. <i>Static</i>		RUN NO. _____
	REF. <i>1 gm load expansion</i>	T	ΔT	DATE <i>1-15-71</i>
	PROGRAM MODE <i>Heat</i>	SCALE SETTING	20 $\frac{^{\circ}\text{C}}{\text{IN.}}$	1.0 $\frac{^{\circ}\text{C}}{\text{IN.}}$
ORIGIN:	RATE <i>10 $\frac{^{\circ}\text{C}}{\text{MIN}}$</i>	START $^{\circ}\text{C}$

PART NO. 900325

INSTRUMENTS

SAMPLE: <i>Plymaster</i> TMA Expansion	SIZE <i>0.65 mm</i>	ATM. <i>Static</i>	RUN NO. _____
	REF. <i>1 gm load Expansion</i>	T _____ ΔT _____	DATE <i>1-15-71</i>
ORIGIN:	PROGRAM MODE <i>Heat</i>	SCALE SETTING <i>20</i> $\frac{1}{100}$ <i>1.0</i> $\frac{1}{100}$	OPERATOR <i>Long</i>
	RATE <i>10</i> $\frac{1}{min}$ START _____ $^{\circ}C$

PART NO. 900325

INSTRUMENTS

SAMPLE: <i>Able film</i> <i>OSC</i> <i>Uncured</i>	SIZE <i>20 mg</i>	ATM. <i>N₂</i>		RUN NO.
	REF. <i>Empty Pan</i>	T	ΔT	DATE <i>1-21-71</i>
ORIGIN:	PROGRAM MODE <i>Heat</i>	SCALE	20 $\frac{^{\circ}\text{C}}{\text{IN}}$	0.5 $\frac{^{\circ}\text{C}}{\text{IN}}$
	RATE <i>15</i> $\frac{^{\circ}\text{C}}{\text{MIN}}$ START $^{\circ}\text{C}$	SETTING	OPERATOR <i>Long</i>

PART NO. 900325

DUPONT INSTRUMENTS

SAMPLE: <i>Abk film Adhesive</i> <i>DSC</i> <i>Cured 30 min at</i> <i>300°F</i>	SIZE <i>20 mg</i>	ATM. <i>N₂</i>		RUN NO. _____
	REF. <i>Empty Pan</i>	T	ΔT	DATE _____
ORIGIN:	PROGRAM MODE <i>Heat</i>	SCALE SETTING	<i>20</i> $\frac{\%}{\text{in}}$	<i>0.5</i> $\frac{\%}{\text{in}}$
	RATE <i>10</i> $\frac{\%}{\text{min}}$ START $^{\circ}\text{C}$			OPERATOR <i>L. J. J.</i>

PART NO. 950251

 INSTRUMENT PRODUCTS DIVISION
 A DIVISION OF DUPONT

FORM 10, 450251

DUPO INSTRUMENT PRODUCTS DIVISION
A DIVISION OF BELL & HOWELL

SAMPLE: <i>Ablefilm 501</i> <i>Cured</i>	X-AXIS		Y-AXIS		RUN NO.	DATE <i>1-9-71</i>
	TEMP. SCALE $\frac{^{\circ}\text{C}}{\text{inch}}$	SHIFT $\frac{\text{inch}}{\text{inch}}$	SCALE <i>2</i> $\frac{\text{mg.}}{\text{inch}}$ (SCALE SETTING X 2)		OPERATOR <i>Long</i>	
SIZE <i>17.0 mg. 100% Span</i>	TIME SCALE (ALT.)		SUPPRESSION $\frac{\text{mg.}}{\text{mg.}}$		HEATING RATE <i>10</i> $\frac{^{\circ}\text{C}}{\text{min.}}$	
					ATM. <i>N₂</i>	
					TIME CONSTANT <i>1</i> sec.

PART NO. 900325

INSTRUMENTS

SAMPLE: Able Film TMA Penetration film Adhesive cured	SIZE .11 mm	ATM. Static		RUN NO.
	REF. 25m load	T	ΔT	DATE 7-19-71
ORIGIN:	PROGRAM MODE Heat	SCALE SETTING 20 $\frac{1}{10}$	0.2 $\frac{1}{10}$	OPERATOR Long
	RATE 10 $\frac{1}{10}$ MIN, START °C

PART NO. 900325

INSTRUMENTS

PART NO. 900325

SAMPLE: <i>FM-123 Adhesive</i> DSC Cured 30 min at 250°F	SIZE	ATM. <i>N₂</i>	RUN NO.
	REF. <i>Empty Pan</i>	T <i>1</i> Δ T	DATE <i>1-21-71</i>
ORIGIN:	PROGRAM MODE <i>Heat</i>	SCALE SETTING <i>20</i> $\frac{\%}{\text{min}}$ <i>0.5</i> $\frac{\%}{\text{min}}$	OPERATOR <i>Long</i>
	RATE <i>10</i> $\frac{\%}{\text{min}}$.START. <i>20</i> °C

PART NO. 950251

 DU PONT DE NEMOURS & CO.
 INSTRUMENT PRODUCTS DIVISION
 WILMINGTON, DELAWARE

SAMPLE: FM 123 Bendix adhesive with organic fiber Cured 30 min at 250°F SIZE 12.8 mg.	X-AXIS TEMP. SCALE 50 °C inch SHIFT 0 inch TIME SCALE (ALT.)		Y-AXIS SCALE 2 mg. inch (SCALE SETTING X 2) SUPPRESSION 0 mg.		RUN NO. DATE 1-21-71 OPERATOR Long HEATING RATE 10 °C/min. ATM. N ₂ TIME CONSTANT 1 sec.

PART NO. 900325

INSTRUMENTS

SAMPLE: FM-123 TMA Penetration at 250°F	SIZE 20 mm	ATM. Static		RUN NO.
	REF. 2 gm load	T	ΔT	DATE 1-19-71
ORIGIN:	PROGRAM MODE Heat	SCALE SETTING 20 $\frac{^{\circ}\text{C}}{\text{IN}}$	0.5 $\frac{^{\circ}\text{C}}{\text{IN}}$	OPERATOR Long
	RATE 10 $\frac{^{\circ}\text{C}}{\text{MIN}}$ START $^{\circ}\text{C}$

PERMEATION CHROMATOGRAPHY ANALYSIS

10⁶, 10⁵, 10⁴, 10³ Å

DATE 1-5-71

SENSITIVITY 2X

SAMPLE B4-2859

CALIBRATION BC-9B

SOLVENT THF

TEMP. 26°C

CONCENTRATION 0.5%

TIMING 170 Sec

TYPE OF MATERIAL CMC 11-2

FILM ADHESIVE

Batch 11-2459 (17741)

GEL PERMEATION CHROMATOGRAPHY ANALYSIS	
10 ⁶ , 10 ⁵ , 10 ⁴ & 10 ³ Å	
DATE 1-5-71	SENSITIVITY 2X
SAMPLE Bx-2860	CALIBRATION BC-3B
SOLVENT THF	TEMP 26°C
CONCENTRATION 1.0%	TIMING 120 Sec
TYPE OF MATERIAL PLYMASTER FP 97-H	

ACG 2080
 Film ADHESIVE
 Batch 1-165

GEL PERMEATION CHROMATOGRAPHY ANALYSIS

10⁶, 10⁵, 10⁴ & 10³ Å

DATE 1-2-71 SENSITIVITY 2x

SAMPLE Bx-2866 CALIBRATION BC-3B

SOLVENT THF TEMP. 26°C

CONCENTRATION 0.5% TIMING 120 Sec

TYPE OF MATERIAL Fm-123

Film ADHESIVE

PERMEATION CHROMATOGRAPHY ANALYSIS	
DATE	1-5-71
SAMPLE	Bx-2861
SOLVENT	THF
CONCENTRATION	1.09%
TYPE OF MATERIAL	ABLE Film 5017
SENSITIVITY	2x
CALIBRATION	Bc-3B
TEMP.	26°C
TIMING	120 Sec

Film Adhesive

Batch 9-29-30-1

GEL PERMEATION CHROMATOGRAPHY ANALYSIS	
9 Column Line	
DATE 1-20-78	CHROMAT 2X
SAMPLE Bx-2882	
SOLVENT THF	27°C
CONCENTRATION 1.0%	120 Sec
TYPE OF MATERIAL ABLEFILM 5017

Mass Spectra of Volatile from Unknown FM-123

70°C

1.

Unknown FM-123

120°C

2.

Mass Spectra of Volatiles from Uncond FM-123

150°C

3.

Mass Spectra of Volatiles from uncond FM-123

160°C

4.

Mass Spectra of Volatile from Cured
FM-1123

run at 210°C for 20 min

215°C

MASS SPECTRUM OF VOLATILES FROM FM-123

270°C

6.

CURED 30 MIN AT 275°F

CONT

MASS SPECTRUM OF VOLATILES FROM ABLE FILM

80°C UNCURED

8

ABLE FILM

100°C UNCURED

9

MASS SPECTRUM OF VOLATILES FROM ABLF FILM150° UNMURED

10

MASS SPECTRA OF VOLATILES FROM ABLE FILM

270°C

11

CON'T

MASS SPECTRUM OF OUTGASSING VOLATILES OF ABLE FILM

200°C

ABLE FILM

250°C

13

MASS SPECTRUM OF VOLATILES OF ABLE FILM

ABLE 300°C 14

cont.

DOW-DGEB

MW 340

150°C

1.

MASS SPECTRA OF POLYVINYL BUTYRAL

60°* Uncond

* m/e 18 most intense peak (H₂O)
(PEAKS NORMALIZED TO m/e 94)
(EXCEPT m/e 16, 17, 18)

Poly vinyl butyral

100° C Uncond

MASS SPECTRUM OF POLYVINYL BUTYRAL PUB

UNPURED 110°C

MASS SPECTRA OF POLYVINYL BUTYRAL

UNPURED 130°C

Mass Spectra of Polyvinylbutyral

Uncured 150°C*

MASS SPECTRUM OF POLYVINYL BUTYRAL PVB

Cured 150°C

m/e 18 highest peak... (100%)
NORMALIZED TO m/e 28 ...

MASS SPECTRUM OF POLYVINYL BUTYRAL

CURED 170°C

MASS SPECTRUM OF POLYVINYL BUTYRAL

PVB CURED

140°C

m/e

MASS SPECTRUM OF Polyvinylbutyral PVB (PURE)
B-76

80°C

1,1-DIETHOXYBUTANE

TOT: 8095 MAP:103 101 55 47 57 INLET:150 ION:

EV:

SIR-2291 MW: 146.1307
C8 H18 O2

M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.
27	262	28	201	29	425	30	24	31	290	41	180	42	42	43	428
44	130	45	188	46	59	47	794	55	808	56	54	57	629	58	25
59	70	60	4	61	9	69	17	70	9	71	81	72	97	73	512
74	24	75	556	85	146	89	30	100	146	101	810	102	44	103	1000

1,1-DIETHOXYBUTANE

TOT: 7004 MAP:103 47 55 101 75 INLET

ION:100 EV:

CUR-0013 MW: 146.1307
C8 H18 O2

M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.	M/E	R.A.
15	25	19	54	26	23	27	245	28	78	29	450	30	22	31	201
32	12	39	47	40	12	41	125	42	20	43	337	44	102	45	139
46	26	47	838	48	11	53	15	54	8	55	745	56	38	57	207
58	11	59	65	60	6	61	9	69	7	71	43	72	45	73	514
74	24	75	523	76	21	85	58	89	26	100	54	101	70	102	48
103	1000	104	52	117	6	145	6

MASS SPECTRUM OF POLYVINYL BUTYRAL PVB (PURE)

B-76

170°C

CMC 11-2

Phenol Formaldehyde Butadiene Acrylonitrile
Adhesive

50-60°C

70V

100µA

Phenol Formaldehyde Butadiene Acrylonitrile
Adhesive

80°C

CMC 11-2

70V 100µA

CMC 11-2

PHENOL FORMALDEHYDE BUTADIENE ACRYLONITRILE
OUTGAS PRODUCTS

100°C - 120°C

2-2- METHYLENE DI PHENOL

2-2- METHYLENE DI PHENOL

ALL PEAKS ENTERED ABOVE 7%

$C_{12}H_{10}O_2$ M.W. 200

250 EV. DOW-5240