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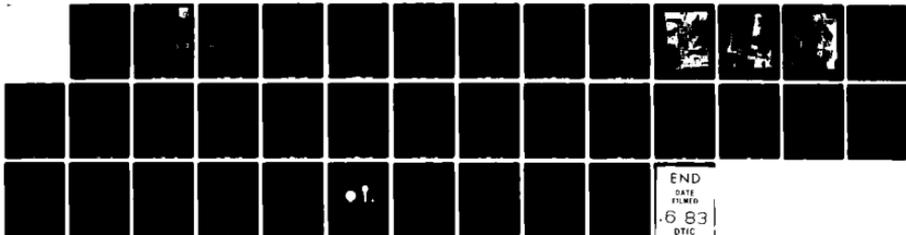
EPOXY ADHESIVE SURFACE ENERGIES VIA THE PENDANT DROP  
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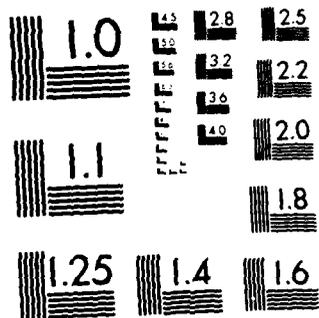
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EPOXY ADHESIVE SURFACE ENERGIES VIA THE PENDANT DROP METHOD

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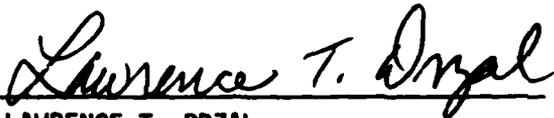
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) and its higher molecular weight homologue, Epon 1007F, yield surface energies which gradually decrease with time. Surface energies of amine-Epon 828 adhesives are reported as a function of temperature, cure time, and resin concentration.		

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## INTRODUCTION

Polymer surface properties are of both practical and theoretical importance in adhesion and adhesive formulation, stabilization of colloidal systems and the use of polymers as surface active additives. Theoretically, the description of polymer surface properties is progressing along with improved methods of measuring polymer surface energies<sup>1,2</sup>. Therefore we set out to accurately measure the surface energy of epoxy resins and resin adhesive mixtures as part of an effort to optimize epoxy resin adhesive formulations.

Of the numerous methods used to measure polymer surface energies, the pendant drop method has several advantages. It is an absolute method, it is static, it can be used with viscous liquids and, with viscous liquids, it provides a way to monitor the attainment of hydrodynamic equilibrium in the pendant drop. Enlarged photographs of the pendant drop provide a permanent record of the drop dimensions from which surface energy, drop profile area, drop surface area and drop volume can be determined<sup>3</sup>. Roe<sup>4</sup> has refined the technique of Andreas, Hauser and Tucker<sup>5</sup> in the pendant drop determination of polymer surface energies. From the measurement of two drop dimensions, the maximum drop diameter ( $d_e$ ), and the drop diameter at a distance  $d_e$  above the drop apex ( $d_g$ ), the surface energy was calculated from Equation 1 (see also Figure 1). Roe's refinement allows the calculation of  $1/H$  from the measurement of several, independent drop dimensions. Constant  $1/H$  values obtained from the same drop profile confirms that this profile obeys the

EQUATION 1

$$\gamma = \Delta \rho g d_e^2 (1/H)$$

$\gamma$  = SURFACE TENSION ( DYNES/CM )

$\Delta \rho$  = DENSITY DIFFERENCE BETWEEN TWO PHASES ( G/CM<sup>3</sup> )

$g$  = ACCELERATION OF GRAVITY ( CM/SEC<sup>2</sup> )

1/H = A CORRECTION FACTOR FROM TABLES OF  $S_N$  VERSUS 1/H

$$S_N = \frac{D_{S_N}}{D_E} \quad ( N = 8-12 )$$

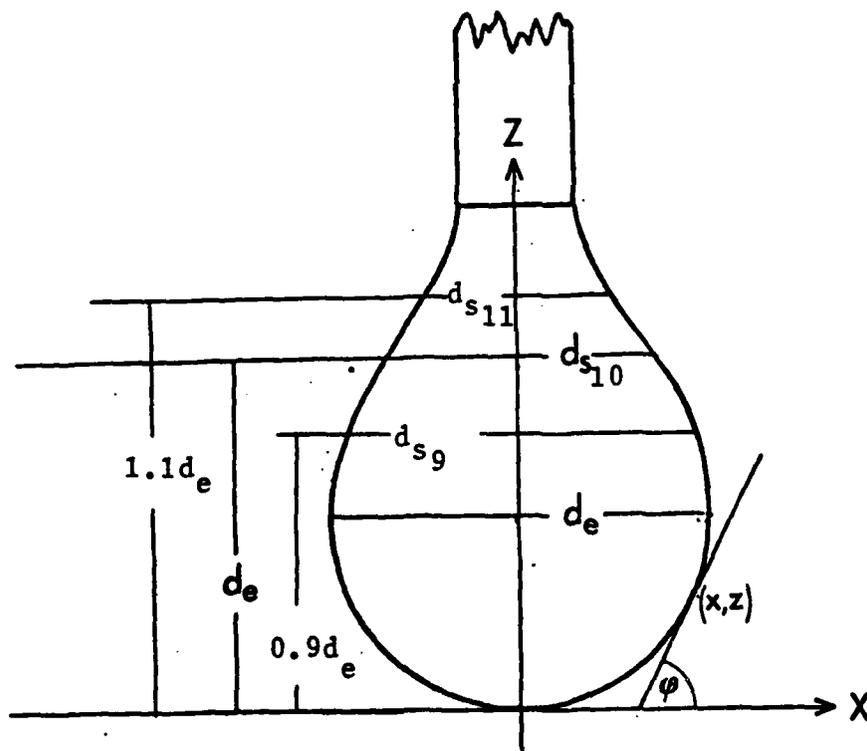


FIGURE 1. PENDANT DROP PROFILE AND DROP DIMENSIONS.

known differential equation describing a hanging pendant drop, and is not being influenced by viscous flow effects (see Figure 1 and Appendix 1). For most polymer fluids, Roe<sup>6</sup> reports hydrodynamic equilibrium is reached within ten minutes and surface energies (reproducible to  $\pm 0.3$  dynes/cm) are constant over periods of 80 minutes.

Dearlove<sup>7</sup> has reported the surface energy, viscosity and density of epoxy resins and epoxy resin adhesive mixtures. When a bisphenol A-epichlorohydrin resin of 360 average molecular weight and 47.2 dynes/cm surface energy ( $30^{\circ}$ ) is cured with diethylaminopropylamine of surface energy 27.0 dynes/cm, the surface tension of the mixture increases from 33.3 to 46.5 dynes/cm over a seven hour period. No correlation was found between the initial properties of the epoxy adhesives and their steel-to-steel lap shear strengths.

Patterson<sup>8</sup> has increased the accuracy of surface energies obtained by the pendant drop method, freeing the method from the measurement of arbitrary drop dimensions. From a photograph, the x, z coordinates (see Figure 1) of the drop profile at eight to fifteen points on the profile are used as input to a Fortran program which calculates the surface energy by an iterative method. Using this program and careful optical alignment techniques, Patterson reports accurate surface energies precise to  $\pm 0.2\%$ .

#### EXPERIMENTAL

Following Patterson's procedures we have assembled the apparatus shown in Figure 2 and Photographs 1-3. Vibration, a major impediment to obtaining sharp drop images, was eliminated

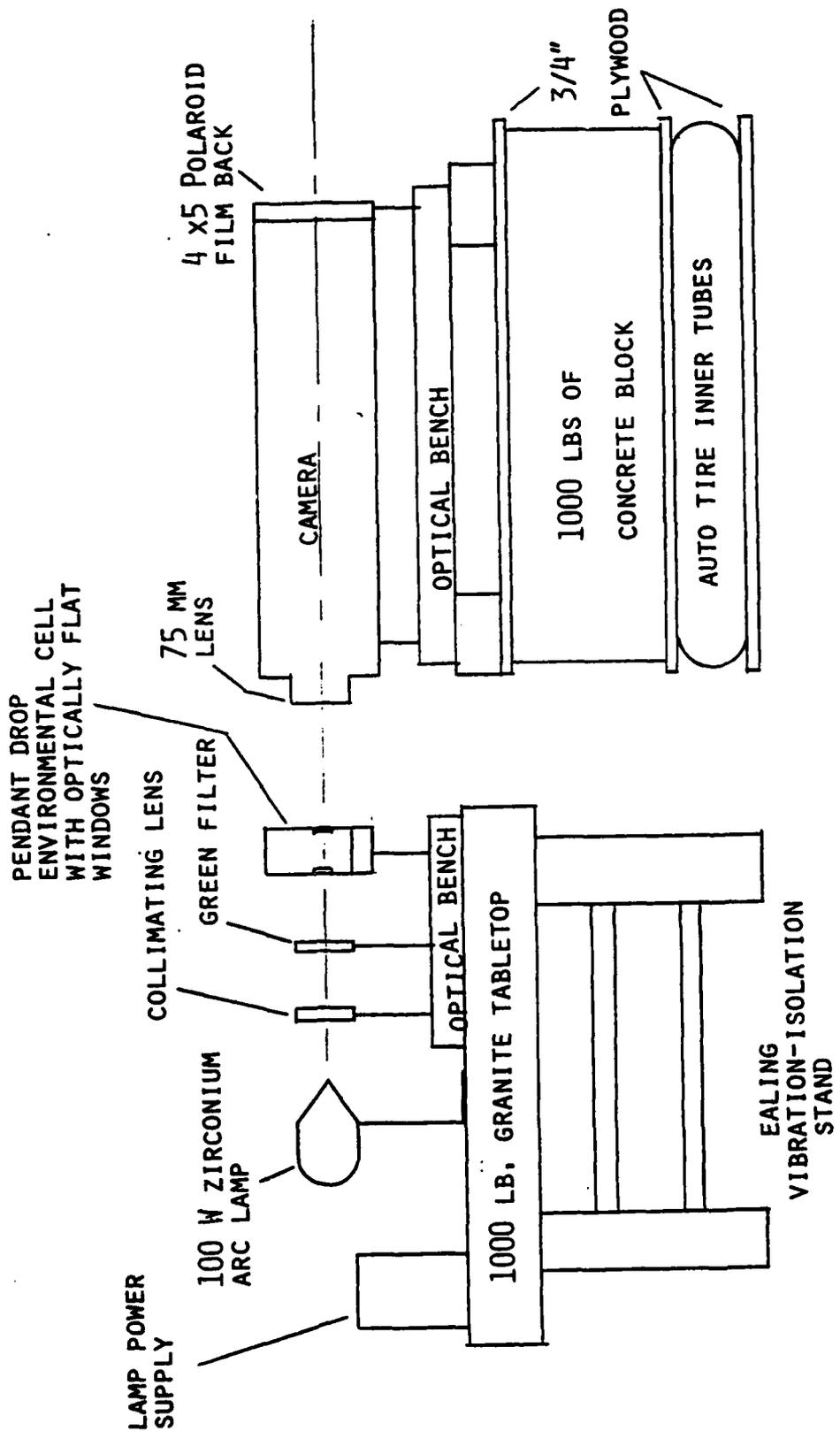


FIGURE 2. SCHEMATIC OF EXPERIMENTAL APPARATUS FOR PENDANT DROP PHOTOGRAPHY.

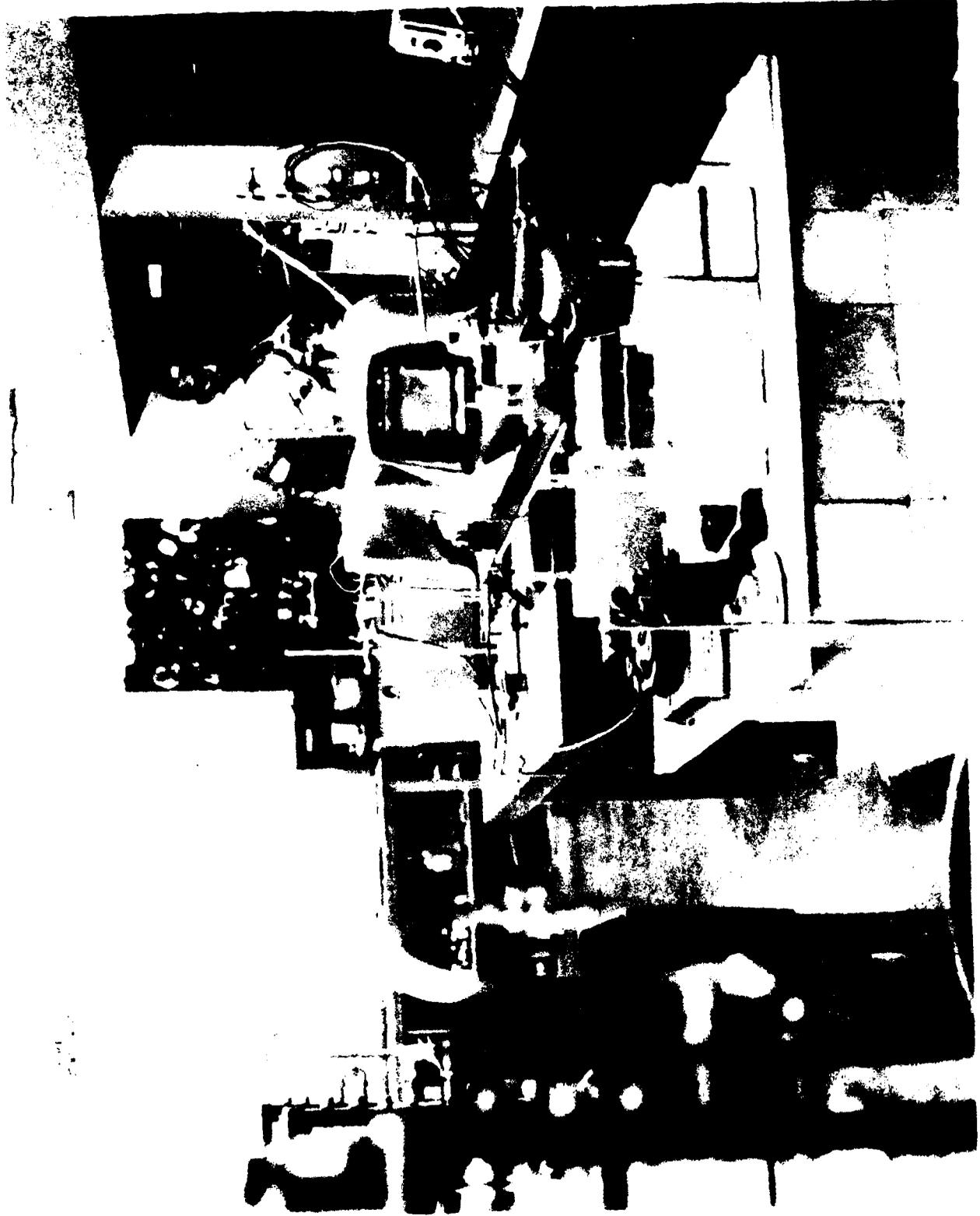


PHOTO 1. CAMERA AND  
ISOLATION TABLES.

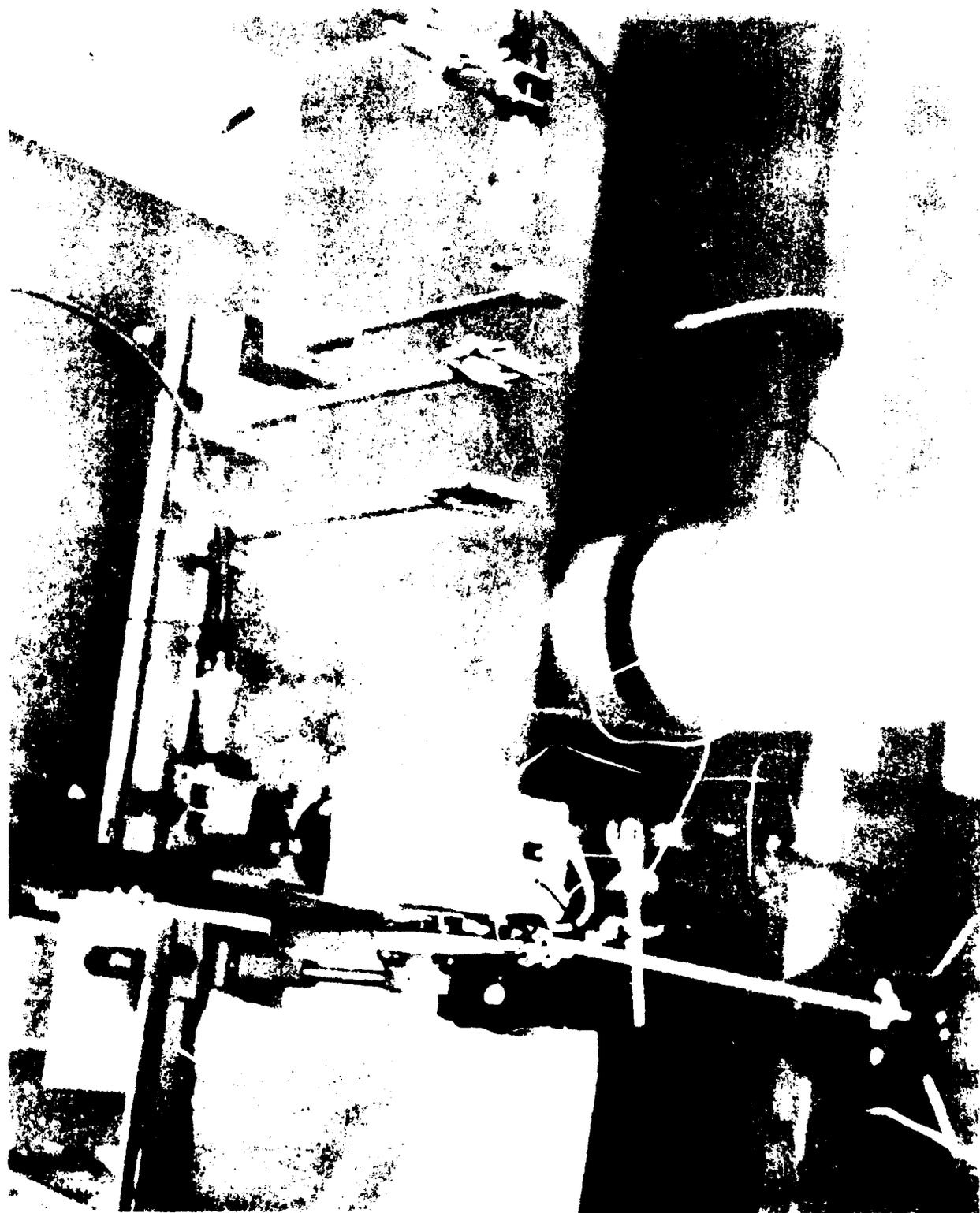


PHOTO 2. OPTICAL BENCH AND  
PENDANT DROP CELL.



FIGURE 3. MANN COMPARATOR  
AND ALPHACOMPUTER

by placing all equipment on two vibration-isolation systems. A Bruel-Kjaer vibration meter measured a vibrational acceleration of less than  $0.1 \text{ meter/sec}^2$  from 10 Hz to 10 kHz on the vibration-isolation systems compared with a vibrational acceleration of  $0.3 \text{ meter/sec}^2$  on an unisolated benchtop in the same room. Alignment procedures<sup>8</sup> insured that the light source, pendant drop, camera lens and camera film plane were all linear. Subsequent adjustment of the camera lens and film plane placed them precisely perpendicular to the optical path. A plumb line, placed in the rear of the camera near the film plane, provided a vertical reference line which appears on each photo negative. In addition to the pendant drop image and plumb line, each negative also displayed the image of a precision steel ball ( $0.1250 \pm 0.00005$  inch diameter) as a calibration standard. Focusing was done by movement of the pendant drop, which could be displaced precisely in any one of three mutually perpendicular directions. To facilitate vertical alignment, the syringe needle, from which the drop was suspended, could be precisely moved through a  $5^\circ$  arc perpendicular to the light path. With the 75 mm camera lens and the drop in focus, the combination of object and image distances afforded a magnification of about twelve diameters. A mechanical shutter, mounted independently of either vibration-isolation system, yielded exposures of sharp contrast with shutter speeds of  $1/4$  to  $1/2$  second. The pendant drop was formed within a closed container (see Photo 2 and Figure 9) to eliminate vibration due to air currents and to provide a controlled drop environment. The temperature of the

container was precisely ( $\pm 0.1^{\circ}\text{C}$ ) controlled by six 50 watt cartridge heaters powered by a proportional controller and utilizing a platinum resistance thermometer. The container could be readily evacuated and/or filled with an inert gas.

The densities of epoxy resins were determined at various temperatures using a pycnometer and a dilatometer. Densities of low viscosity liquids were determined by a bouyancy method.

Low viscosity liquids were generally purified by fractional distillation, then stored in a dry box. These liquids were further characterized by boiling point, refractive index and density determinations. Water, of  $1 \times 10^{-7}$  mhos conductivity, was obtained from a Continental Deionized Water System. We received epoxy resin samples from Miller-Stephenson Chemical Company and determined their surface energies under a nitrogen atmosphere after degassing the resins at  $50-75^{\circ}$  at pressures of 1-3 torr for 20-30 minutes.

A Mann linear comparator equipped with a Bausch & Lomb Accu-Rite digital readout was used to measure pendant drop dimensions from photographic negatives. Data reduction was done on a VAX 780 or Apple II computer.

#### RESULTS & DISCUSSION

Several tests were made of the accuracy of the photographic pendant drop technique. The average diameter of a precision steel ball, calculated from a photo negative displaying the ball and a millimeter scale, was accurate to  $\pm 0.1\%$ , while the magnification factor (  $\text{MAG} = \text{measured ball diameter from}$

the negative divided by 0.1250 inches, the actual ball diameter) over six negatives remained precise to 0.08% (MAG = 12.124 ± 0.009). Butler and Blo<sup>9</sup> recommend that a magnification factor precise to 0.02% be maintained in order to achieve surface energies as accurate as those afforded by measuring pendant drop dimensions directly (no photograph). Table 1 summarizes the results of surface tension measurements on several liquids calculated by Andreas' two parameter method, while Appendix 2 contains physical properties of the purified liquids. Table II contrasts surface energies of water calculated by the two-parameter method and the iterative computer program of Patterson. In addition to a calculated  $\gamma$ , Patterson's program calculates SDEV, a parameter which measures the "goodness of fit" of the experimentally determined coordinates of the drop profile to the theoretically determined profile coordinates. From our data, large differences in  $\gamma$  calculated by the two methods generally parallel large SDEV values. We believe that having a sharp profile edge around the entire drop perimeter and finding the coordinates of it is more critical for the determination of  $\gamma$  by the computer program than by the two-parameter method. The surface energies which follow were all calculated by the two-parameter method.

Surface energy measurements of Epon 828(see Figure 3) prior to degassing are presented in Figure 4. Prior to the calculation of surface energies, the density of Epon 828 at several temperatures was determined. These values are tabulated in Table III. For mixtures of Epon 828 with Epon 1007F, mixture

TABLE I. EXPERIMENTAL SURFACE ENERGIES AT 20° COMPARED WITH LITERATURE VALUES.

FLUID	γ THIS STUDY		PRECISION- STANDARD DEV. AS % OF AVG.	γ LITERATURE <sup>8</sup> mNm <sup>-1</sup>
	mNm <sup>-1</sup>	p.ft. <sup>-1</sup> x 10 <sup>-3</sup> <sup>a</sup>		
WATER	72.58 ± 0.82	(4.973 ± 0.0056)	1.1	72.88
CYCLOHEXANE	24.52 ± 0.37	(1.680 ± 0.0025)	1.2	24.98
BENZENE(30°)	27.25 ± 0.18	(1.867 ± 0.0012)	0.6	27.47
HEXANE	18.22 ± 0.15	(1.248 ± 0.0010)	0.8	18.41

a - English units for γ, 1 mNm<sup>-1</sup> = 6.852 x 10<sup>-5</sup> p.ft.<sup>-1</sup> .

TABLE II. CALCULATED SURFACE ENERGIES FOR WATER AT 25° C.

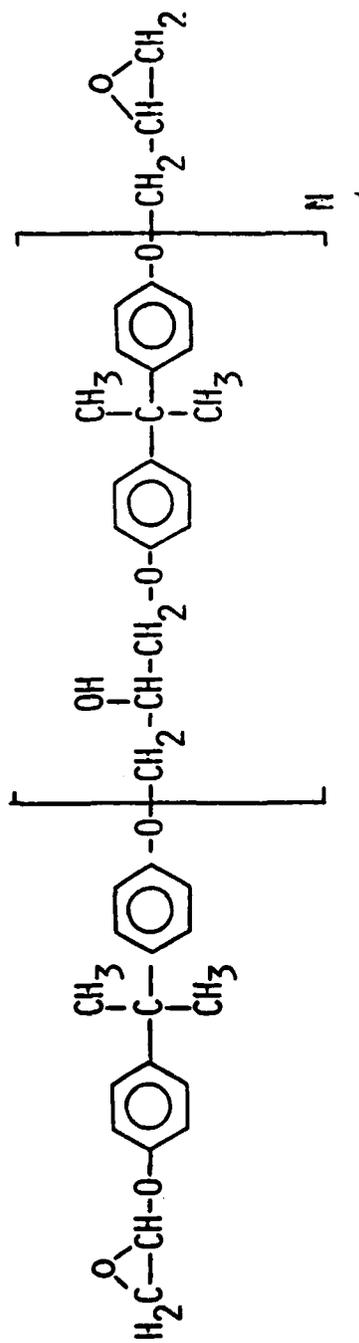
<u>EXPOSURE</u>	<u>Y-TWO PARAMETER</u>		<u>Y-COMPUTER</u>	<u>SDEV ( x 10<sup>-4</sup> )</u>
EW1-32-13	70.93 mNm <sup>-1</sup>	(4.860 x 10 <sup>-3</sup> p.ft <sup>-1</sup> )	69.03 mNm <sup>-1</sup>	1.32
EW1-32-11	70.00	(4.796 x 10 <sup>-3</sup> )	63.41	1.20
FW2-12-82	73.26	(5.019 x 10 <sup>-3</sup> )	77.83	0.89
EW1-32-14	71.31	(4.886 x 10 <sup>-3</sup> )	70.76	0.57
Patterson's results <sup>8</sup>	72.93	(4.997 x 10 <sup>-3</sup> )	72.68	0.14-0.65

TABLE III. DENSITIES OF RESINS & RESIN MIXTURES AT VARIOUS TEMPERATURES.

SAMPLE	DENSITY(kg/ m <sup>3</sup> )					TEMPERATURE
	20°	30°	40°	75°	90°	
EPON 828	1168 (72.90)	1159 (72.35)	1152 (71.91)	1126 (70.29)	1117 (69.72) <sup>a</sup>	150°
MPDA	1135 (70.88)	1129 (70.45)		1099 (68.61)	1087 (67.85)	
EPON 1007F	1160 (72.4)					1080 (estimated) (67.4)
1% EPON 1007F IN EPON 828	1168 (72.90)	1159 (72.35)	1152 (71.91)	1126 (70.29)	1117 (69.72)	
5% EPON 1007F IN EPON 828	1168 (72.90)	1159 (72.35)	1152 (71.91)	1126 (70.29)	1117 (69.72)	
14.6 g MPDA & <sup>b</sup> 100 g EPON 828	1160 (72.9)	1150 (71.8)		1120 (69.9)	1110 (69.3)	

a - Densities in units of lb.ft<sup>-3</sup>

b - These values calculated from equation 2.



X-22, N= 0

EPON 328, N= 0 (75%), N= 1 (25%)

EPON 1001, N=0 (11%), N= 1 (16%), N= 2-8 (72%)

EPON 1007F, N= 14.0 (AVERAGE).

FIGURE 3. STRUCTURES OF EPON BISPHENOL A-EPICHLOROHYDRIN RESINS.

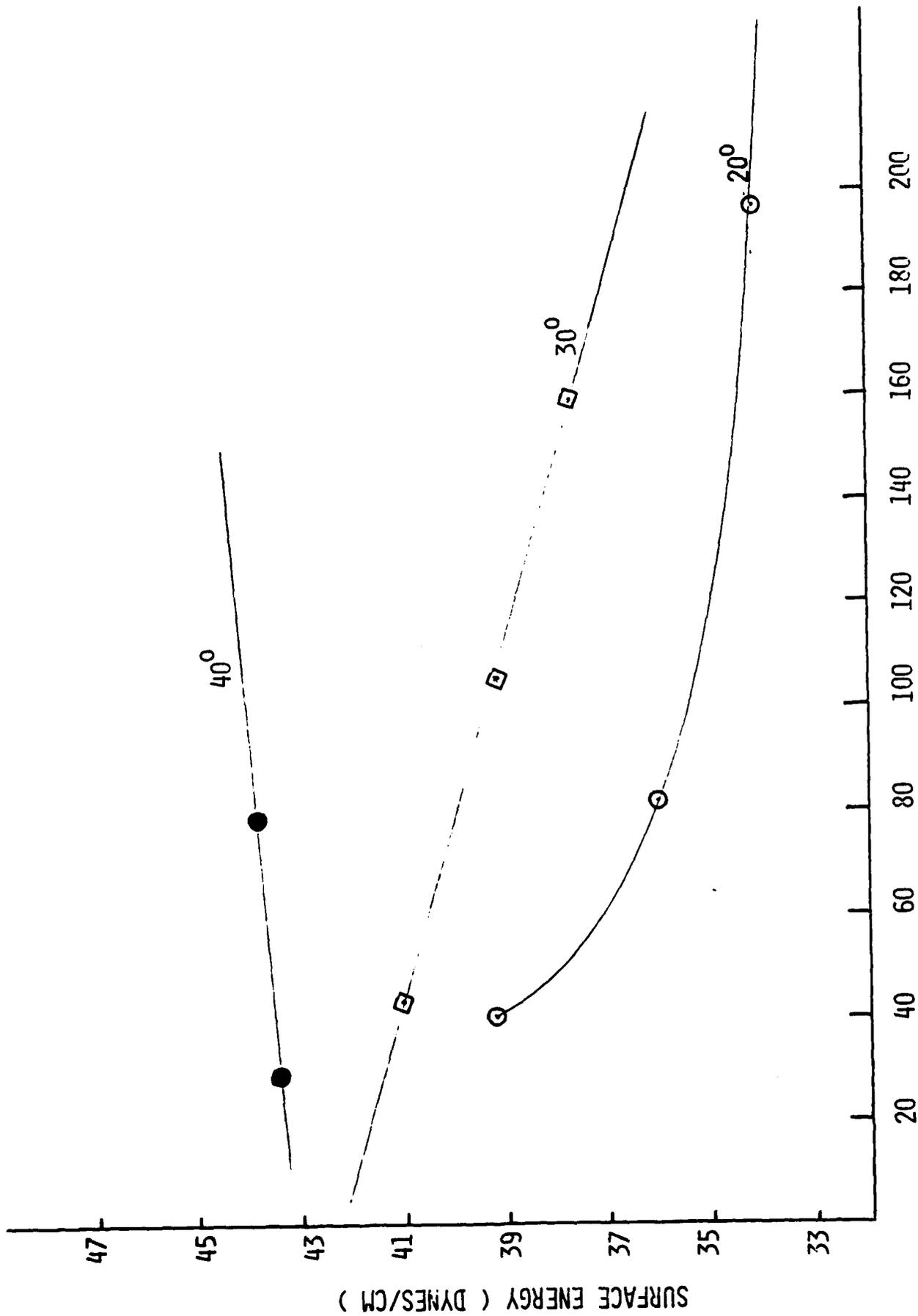


FIGURE 4. SURFACE ENERGY OF UNDEGASSED EPON 828 AT SEVERAL TEMPERATURES.

densities were assumed to be the same as the density of Epon 828 at that temperature. Equation 2<sup>7</sup> was used to calculate the densities of meta-phenylenediamine/Epon 828 mixtures.

$$\text{EQUATION 2} \quad \delta_{\text{mixture}} = (\delta_{828}) V_{828} + (\delta_Z) V_Z$$

where  $\delta_{\text{mixture}}$  = density of mixture

$\delta_{828}$  = density of Epon 828

$V_{828}$  and  $V_Z$  = volume fractions of Epon 828 and other mixture component, respectively.

From Figure 4, we attribute the gradual decrease in surface tension over time to the presence of a surface active impurity in commercial Epon 828 which slowly diffuses to the interface lowering the surface tension. At 40°C surface energies increase, probably due to evaporation of the impurity from the resin surface. When Epon 828 was degassed before pendant drop formation surface tension values remained constant over one to two hour intervals. Attempts to trap the volatile component from commercial Epon 828 and identify it by spectroscopy are so far unsuccessful. According to Paul Jones of Shell Oil in Texas, Epon 828 contains epichlorohydrin (less than 1 ppm), water (0.04-0.05%), and acetone (at the ppm level). The latter two polar compounds have surface tensions greater than that of degassed Epon 828 suggesting that they could not be responsible for the decreasing surface energies we observed.

To follow the rate of approach to hydrodynamic equilibrium

we tested the conformity of the drop profile, at various times, to the known differential equation which describes pendant drop profiles( Roe's procedure ). Table IV contains our results. Constant  $1/H$  values within 10 minutes of pendant drop formation confirm that hydrodynamic equilibrium has been reached.

Figure 5 displays Epon 828 surface energies from 20-140°C and yield a calculated temperature coefficient of  $-0.111 \text{ mNm}^{-1} \text{ } ^\circ\text{C}^{-1}$ . The apparent "break" in the line at 40° we believe to be an experimental artifact. The 20,30 and 40° surface energies were determined in a water-thermostatted Helma glass cell, while the remaining surface energies were determined in the electrically heated aluminium block cell. Our temperature coefficient of  $-0.111$  compares favorably with a value of  $-0.1 \text{ mNm}^{-1} \text{ } ^\circ\text{C}^{-1}$  reported by Dearlove<sup>7</sup> for a comparable bisphenol A-epichlorohydrin epoxy resin.

Mixing longer chain epoxy resins with Epon 828 yields amine-cured adhesives with better flexibility. Accordingly we have prepared 1% and 5% solutions of Epon 1007F in Epon 828 by heating the mixtures in dry air at 130° for 1.5 and 63 hours respectively. Surface tension values at various times and temperatures are presented in Figures 6 and 7. We believe the initial slight rise in surface tension in going from Epon 828 to the 1% Epon 1007F in Epon 828 ( from 47.47 to 48.63 at 20° and from 41.28 to 42.29 at 90°) reflects a surface composition which is the same as the bulk resin mixture, 99% Epon 828 and 1% Epon 1007F. A slightly higher surface tension for this surface composition implies that Epon 1007F has a higher surface tension than Epon 828. At 150°C this is indeed the case(see Table V).

TABLE IV.  $1/H$  VALUES OBTAINED FROM SEVERAL PENDANT DROP DIAMETERS (SEE FIGURE 1).

<u>EXPOSURE NO.</u>	<u><math>S_8</math></u>	<u><math>S_9</math></u>	<u><math>S_{10}</math></u>	<u><math>S_{11}</math></u>
FW-1-504	0.9007	0.8107	0.6939	0.5598
$1/H$	0.8211	0.8207	0.8224	0.8216

(Exposure 504 taken 124 minutes after drop formation).

TABLE V. SURFACE ENERGIES OF EPOXY RESINS OF DIFFERING AVERAGE MOLECULAR WEIGHT.

<u>RESIN</u>	<u>AVG. M.W.</u>	<u><math>\gamma</math> AT 150°</u>
X-22	340	29.0 mNm <sup>-1</sup> (1.98 x 10 <sup>-3</sup> p.ft <sup>-1</sup> )
EPON 828	380	35.1 (2.40 x 10 <sup>-3</sup> )
EPON 1007F	4316	38.2 (2.62 x 10 <sup>-3</sup> )

(See Figure 3 for resin structures).

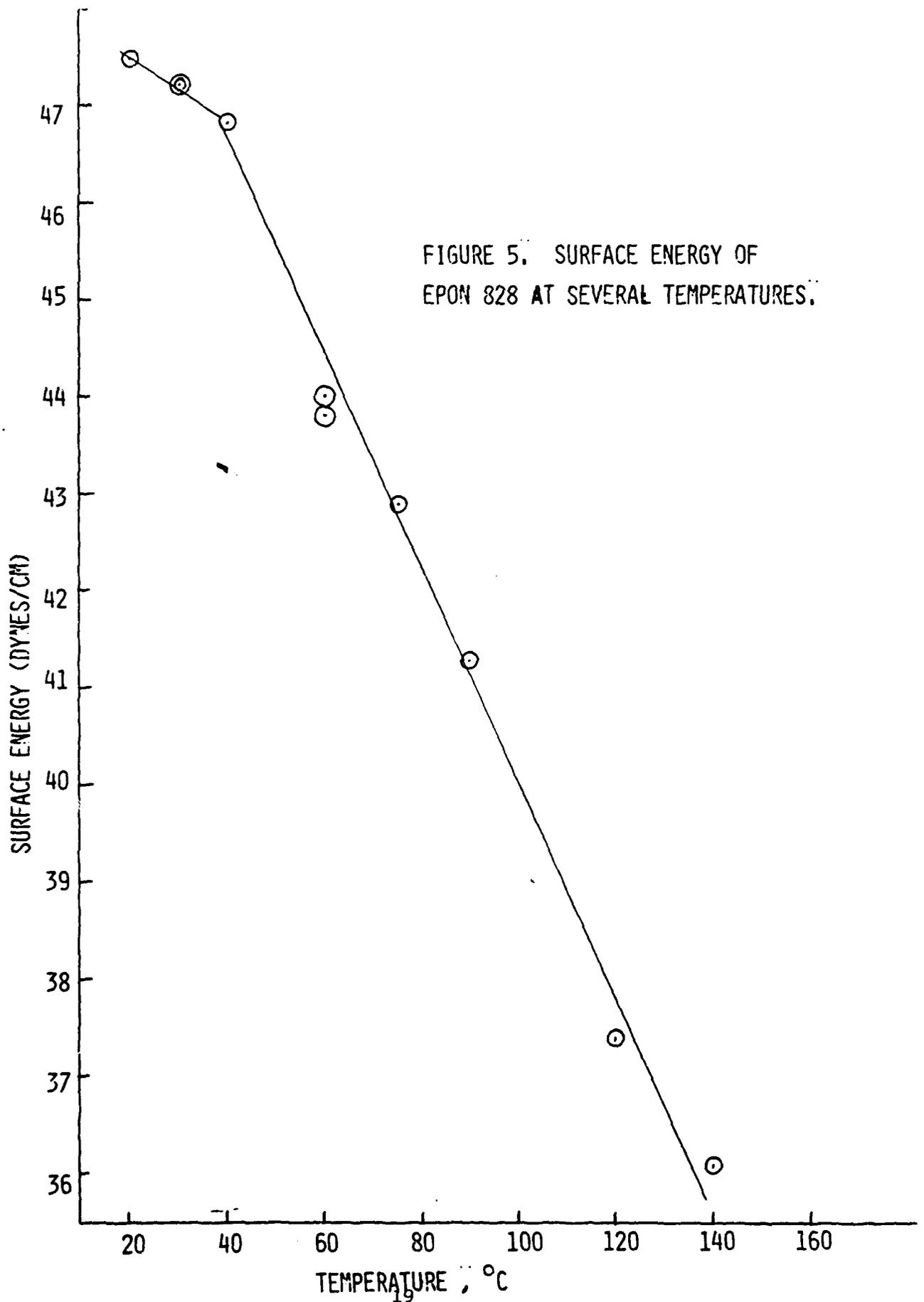


FIGURE 5. SURFACE ENERGY OF EPON 828 AT SEVERAL TEMPERATURES.

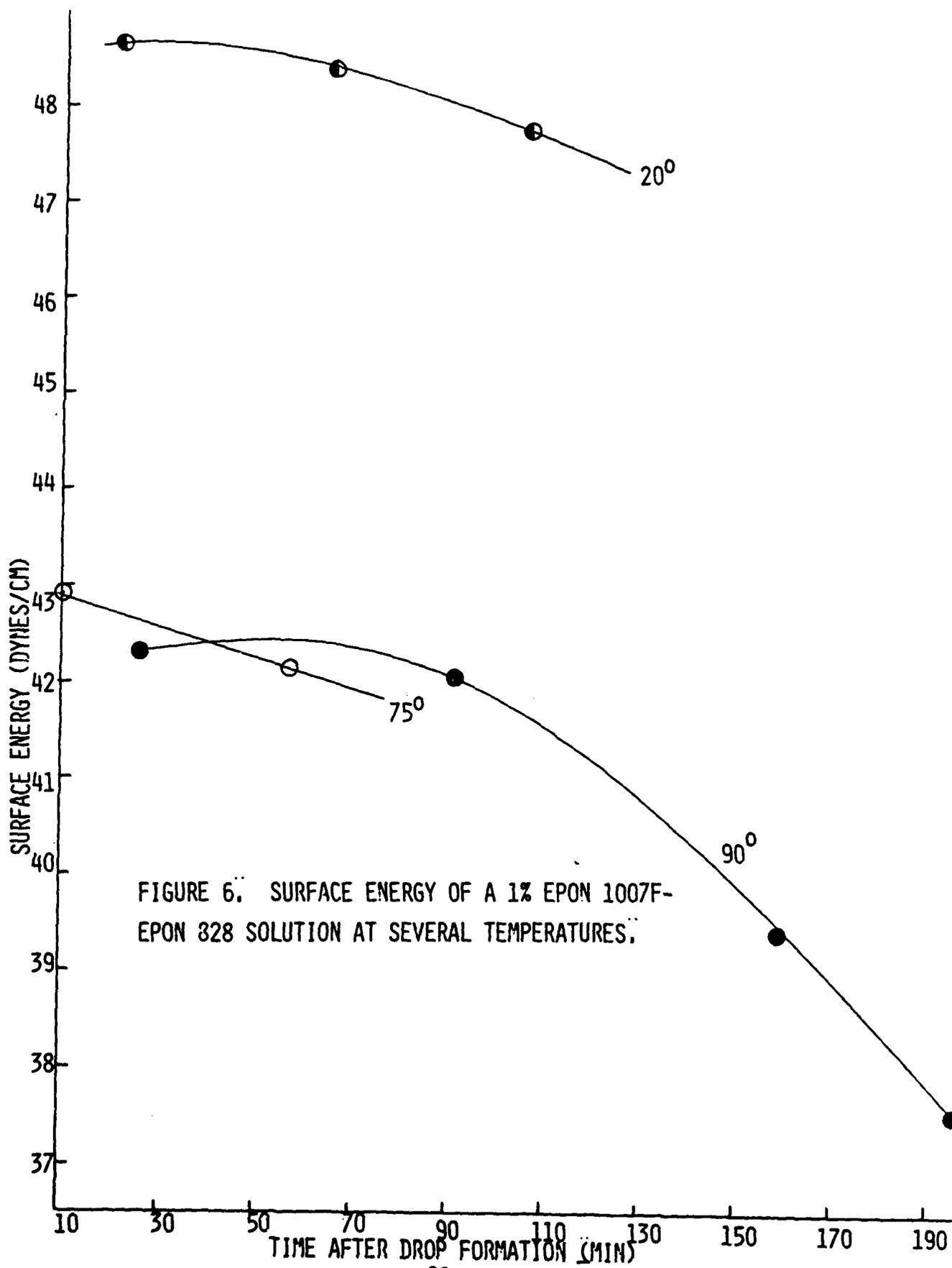


FIGURE 6. SURFACE ENERGY OF A 1% EPON 1007F-EPON 328 SOLUTION AT SEVERAL TEMPERATURES.

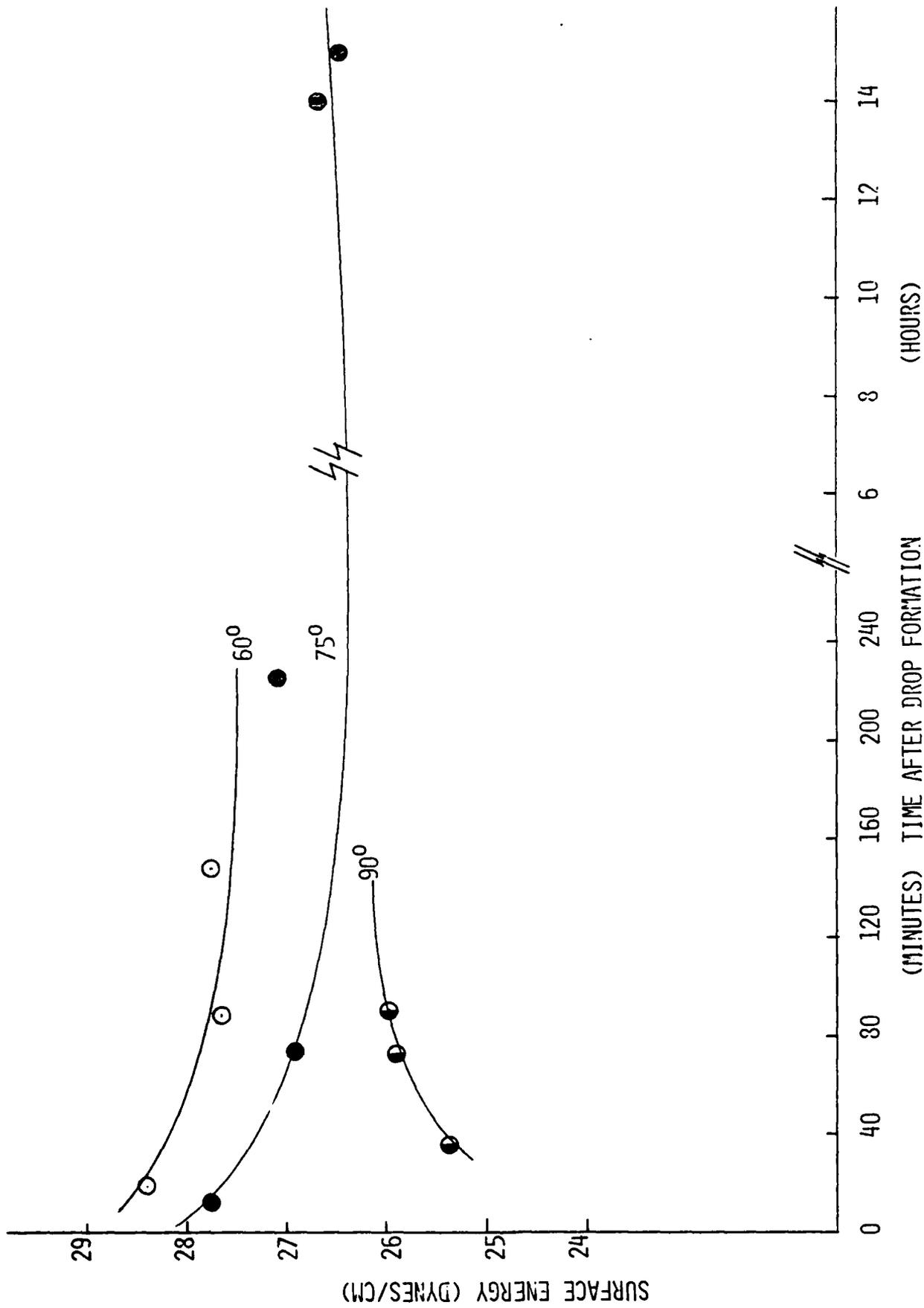


FIGURE 7. SURFACE ENERGY OF 5% EPON 1007F IN EPON 828 AT SEVERAL TEMPERATURES.

The decrease of mixture surface energy with time is baffling, however it may be due to the presence of a surface active impurity in the mixture. There is some indication<sup>9</sup> that the Epon resins designated "F" are prepared by catalytically reacting Epon 828 with bisphenol A. Epon 1007F therefore could contain a surface active catalyst which diffuses to the mixture surface lowering the surface energy. After a long period of heating, the 5% mixture displays rather constant surface energies with time suggesting a surface "equilibrium" has been reached. This equilibrium surface, with a  $\gamma$  of 26-28 mNm<sup>-1</sup> should be entirely composed of the catalyst impurity which, due to the relatively low  $\gamma$ , must be considerably less polar than the epoxy resins.

The surface energy of meta-phenylenediamine (MPDA), an aromatic amine curing agent, was measured. Although the solid amine oxidizes upon heating above the melting point (64<sup>o</sup>), at elevated temperatures we found surface energies constant over intervals of 100 minutes. Table VI lists MPDA results.

The MPDA-Epon 828 mixture was prepared by mixing stoichiometric amounts of the melted tetrafunctional amine (heated under N<sub>2</sub>) and the difunctional Epon 828 (14.6g of MPDA per 100g of Epon 828) at 60<sup>o</sup>. Surface energies of the mixture are listed in Table VII. At 30<sup>o</sup> the resin-amine pendant drop was clearly fluid, although viscous, after three hours. Between four and five hours the drop became rigid. At 75<sup>o</sup> the first pendant drop photo was taken one hour after mixing the epoxy resin and the amine, and the mixture cured to a hard solid 30 minutes later. Within the first ten minutes of drop formation the drop profile conformed to the theoretical drop profile as indicated by constant 1/H values at S<sub>8</sub>, S<sub>11</sub>, and S<sub>12</sub>, however as the viscosity increased the shape of the hanging mass of fluid became increasingly unsymmetrical generally resulting in a completely cured irregularly-shaped mass of polymer adhering to the needle tip. In summary, the surface energy

TABLE VI. SURFACE ENERGY OF MPDA AT SEVERAL TEMPERATURES.

EXPOSURE NO.	TEMP. (°C)	TIME AFTER DROP FORMATION	$\gamma$		DENSITY	
			$\frac{\text{mNm}^{-1}}{(\text{p}\cdot\text{ft}^{-1} \times 10^{-3})}$	$(\text{p}\cdot\text{ft}^{-1} \times 10^{-3})$	$\frac{\text{kg}\cdot\text{m}^{-3}}{(\text{lb}\cdot\text{ft}^{-3})}$	$(\text{lb}\cdot\text{ft}^{-3})$
622	75	36 minutes	49.26	(3.375)	1099.2	(68.61)
623	75	136	49.64	(3.401)		
624	90	20	47.77	(3.273)	1087	(67.85)
625	90	49	47.56	(3.258)		

MPDA PURIFIED BY FRACTIONAL DISTILLATION, MP.- 63-64°C.

TABLE VII. SURFACE ENERGIES OF A STOICHOOMETRIC EPON 828-MPDA MIXTURE.

<u>MIXTURE</u>	<u>TIME AFTER DROP FORMATION</u>	<u><math>\gamma^{30^\circ}</math> (mNm<sup>-1</sup>)</u>	<u>TOTAL CURING TIME @ 30<sup>o</sup></u>
MPDA-EPON 828	34 minutes	46.2 (3.16 x 10 <sup>-3</sup> )	100 minutes
	84	46.2 (3.16 x 10 <sup>-3</sup> )	150
	144	45.9 (3.14 x 10 <sup>-3</sup> )	244
MPDA-EPON 828 & AMINE OXIDATION PRODUCTS	33	47.1 (3.22 x 10 <sup>-3</sup> )	123
	61	47.1	151
	102	47.1	192
	150	46.8 (3.21 x 10 <sup>-3</sup> )	240
	203(75 <sup>o</sup> )	39.5	293
MPDA-EPON 828		<u><math>\gamma^{75^\circ}</math></u>	<u>CURE TIME @ 75<sup>o</sup></u>
	5	42.3 <sup>a</sup> (2.89 x 10 <sup>-3</sup> )	110
	19	42.8 (2.93 x 10 <sup>-3</sup> )	58
	7	42.6 (2.92 x 10 <sup>-3</sup> )	62

<sup>a</sup>- these values obtained with different pendant drops.

FOR COMPARISON;	<u><math>\gamma^{30^\circ}</math></u>	<u><math>\gamma^{75^\circ}</math></u>
EPON 828	47.26	42.89
MPDA	-	49.45

TABLE VIII. SURFACE ENERGIES OF MPDA-EPON 828 MIXTURES  
AT 75°C.

<u>PARTS MPDA PER 100 PARTS EPON 828</u>	<u><math>\gamma^{75^\circ}</math> (<math>\text{mNm}^{-1}</math>)</u>
5	$42.3 \pm 0.2$ ( $2.89 \times 10^{-3}$ p.ft <sup>-1</sup> )
14.6	$42.6 \pm 0.3$ ( $2.92 \times 10^{-3}$ )
20	$44.2 \pm 0.5$ ( $3.03 \times 10^{-3}$ )

TABLE IX. SURFACE ENTROPIES OF RESINS AND RESIN MIXTURES.

<u>SUBSTANCE</u>	<u><math>-\delta\gamma/\delta T</math></u>
EPON 828	0.111 $\text{mNm}^{-1}/^\circ\text{C}$
MPDA	0.119
14.6 PARTS PER HUNDRED MPDA-EPON 828	0.077
EPON 1001F <sup>a</sup> (AVG. M.W. OF 1000)	0.063

<sup>a</sup> -  $\gamma = 41.62 \pm 0.39$  at 90° and  $40.99 \pm 0.13$  at 100°.

of Epon 828-MPDA mixtures at 30 and 75°C is slightly lower than  $\gamma$  for Epon 828 alone and these mixture surface energies do not change with time. Secondly, it is not possible to follow surface energy changes during the latter stages of resin-amine curing because symmetrical pendant drops can not be maintained.

To evaluate the effect of molecular weight on the surface energy of a series of bisphenol A-epichlorohydrin resins we have formed pendant drops of X-22, a pure sample of the diglycidyl ether of bisphenol A (melting point 46°C) and Epon 1007F. The results are summarized in Table V. As with other organic homologous series, the surface energy increases with increasing molecular weight.

#### CONCLUSIONS

We have used the pendant drop method to accurately measure the surface energy of liquid epoxy resins and resin mixtures. Typically, over a temperature range of 20-100°C,  $\gamma$  values are reproducible to  $\pm 0.2 \text{ mNm}^{-1}$ . Even viscous liquid surface energies were measured by working at higher temperatures (160°C) under an inert atmosphere and following the attainment of hydrodynamic equilibrium in the pendant drop. With resin-amine mixtures our accurate measurement of surface energies was limited somewhat by the accuracy of measurement of resin mixture densities.

Surface energy measurements are one method of inspecting surface compositions. The Epon resins we examined all contained impurities produced in the manufacturing process. We have found that such impurities do significantly change epoxy resin

surface composition, however they can be removed generally by high temperature-reduced pressure degassing. Once impurities are removed, changes at the surface of epoxy resins and resin-resin mixtures are negligible as indicated by constant  $\gamma$  values over time.

Curing mixtures of Epon 828 and MPDA display little change in surface energy with changing bulk composition(see Table VIII). Nor does the surface energy change as curing proceeds to form high molecular weight polymers( Table VII). That curing is indeed occurring is reflected in the decrease in surface entropy(Table IX) as we proceed from resin and amine starting materials to highly polymerized products. The  $\delta\gamma/\delta T$  value of  $-0.077 \text{ mNm}^{-1}/^{\circ}\text{C}$  for a stoichiometric Epon 828-MPDA mixture cured for 100 minutes is comparable to values for other polymeric liquids( $-0.05$ - $0.075 \text{ mNm}^{-1}/^{\circ}\text{C}$ ).

Additional work with the Epon resin systems which would further clarify resin surface properties include: complete molecular and ionic characterization of the Epon resins; more extensive investigation of resin molecular weight and its influence on surface properties; measurement of Epon resin interfacial tensions with model metals and/or model hydrocarbons.

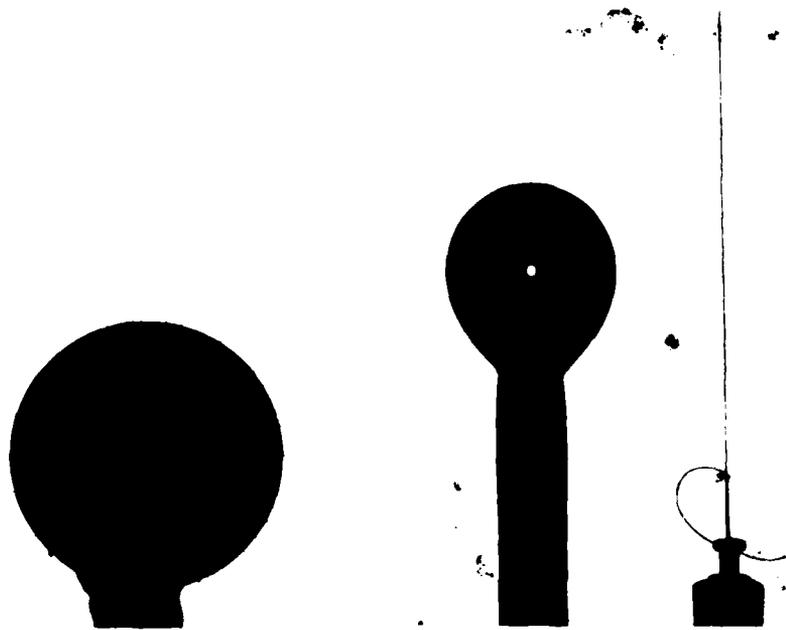
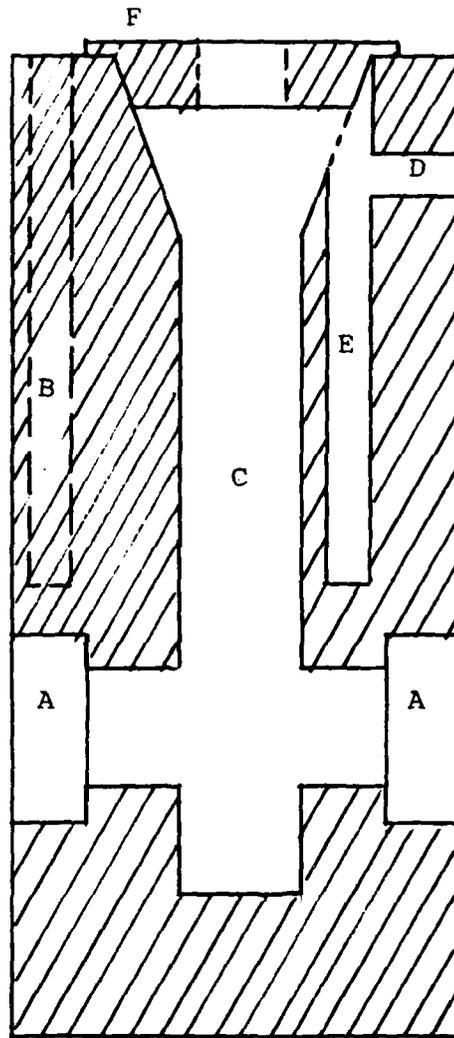


FIGURE 8. TYPICAL PENDANT DROP PHOTO NEGATIVE SHOWING CALIBRATION BALL AND VERTICAL REFERENCE LINE.



- A- optically flat windows
- B- Cavities for cartridge heaters
- C- Cavity for syringe and needle
- D- Gas inlet
- E- Thermocouple well
- F- Cell cover with opening

FIGURE 9. Schematic of High Temperature Aluminum Block Cell.

APPENDIX 1.

The profile of a pendant drop is given by

$$\frac{1}{(\delta/b)} + \frac{\sin\phi}{(x/b)} = 2 + \lambda \left( \frac{z}{b} \right)$$

where  $x$ ,  $z$ , and  $\phi$  are shown in Figure 1,  $\delta$  is the radius of curvature at  $(x,z)$  and  $b$  equals  $\delta$  at  $(0,0)$ . The size of the drop is determined by  $b$ , and its shape is determined by  $\lambda$ , where

$$\lambda = \frac{\Delta\rho g b^2}{\gamma}$$

and  $\Delta\rho$  is the density difference between the two phases.

APPENDIX 2. Physical Properties of Purified Liquids

<u>Liquid</u>	<u>Boiling Pt. (°C)</u>	<u>Index of Refraction (20°)</u>	<u>Density (20°)</u>
cyclohexane	81 (80.7)	1.4264 (1.4266) <sup>a</sup>	0.7769 (0.7785g/ml)
ethylene glycol	88/7torr	1.4324 (1.4318)	1.1139 (1.1088)
water	-	1.3330 (1.3329)	0.9981 (0.9982)
benzene	(80.1)	(1.5011)	(0.8786)
hexane	(68.9)	(1.3750)	(0.6603)

<sup>a</sup> - Literature values (Handbook of Chemistry & Physics, 54th Ed.) listed in parentheses.

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9. Conversation with Justin Bolger

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