AD-A145 860 INFRARED SPECTROSCOPY STUDY OF THE SP-250 EPOXY RESIN SYSTEM(U) ARMY MATERIALS AND MECHANICS RESEARCH CENTER WATERTOWN MA R E SACHER ET AL. JUL 84 AMMRC-TR-84-29 1/1

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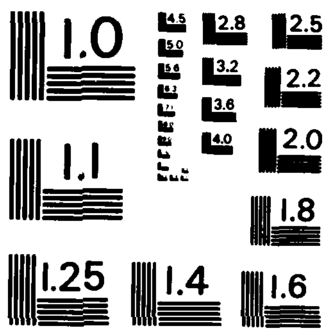

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INFRARED SPECTROSCOPY STUDY OF THE SP-250 EPOXY RESIN SYSTEM

ARMY MATERIALS AND MECHANICS RESEARCH CENTER

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July 1984

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: AMMRC TR 84-29	2. GOVT ACCESSION NO.: AD-A145860
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle): INFRARED SPECTROSCOPY STUDY OF THE SP-250 EPOXY RESIN SYSTEM	5. TYPE OF REPORT & PERIOD COVERED: Final Report
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Robert E. Sacher and Bernard R. LaLiberte	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Army Materials and Mechanics Research Center, Watertown, Massachusetts 02172, DRXMR-OP	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: D/A Project: 1L162105AH84; Agency Accession: DA OGH815
11. CONTROLLING OFFICE NAME AND ADDRESS: U.S. Army Materiel Command, Alexandria, Virginia 22333	12. REPORT DATE: July 1984
	13. NUMBER OF PAGES: 27
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS. (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Epoxy resins
Curing agents
Monuron
Dicyandiamide
Infrared spectroscopy

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

(SEE REVERSE SIDE)

Block No. 20

ABSTRACT

The various epoxides which are part of the SP-250 formulation (3M Company, St. Paul, Minnesota) were treated to three different curing procedures. In this study, the epoxides and the epoxy blends were individually cured with dicyandiamide (Dicy) and Monuron, respectively, and in combination (Dicy/Monuron). The acceleration of the Dicy-containing epoxy resin cure is linked to the formation of cyclic 2-oxazolidone. The characteristic nature of the carbonyl band of these cyclic derivatives prompted us to monitor the hardening process by infrared spectroscopy. The cure conversion was evaluated to be the highest for the EPON 828 + Dicy/Monuron and ECN 1273/ EPON 828 + Dicy/Monuron systems. The epoxide ± accelerator reaction affording 2-oxazolidone was found to be the greatest in the active diluent + Monuron system. Regarding neat EPON 828, the presence of Dicy significantly enhanced the formation of 2-oxazolidone.

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INTRODUCTION

The various epoxy combinations in the SP-250 system were recently examined by differential scanning calorimetry (DSC) under dynamic conditions. The results of this study[1] and the military aspects of the epoxy system such as the usage in helicopter blades necessitated further investigation of 3M's formulation. Consequently, the same mixtures prepared for the previous study were reexamined by infrared spectroscopy (IR).

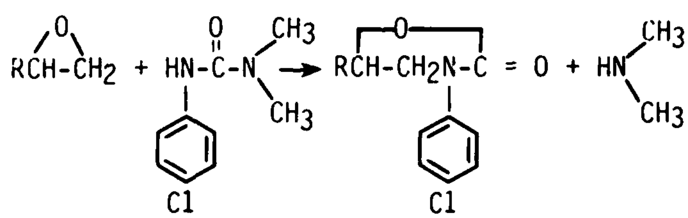
EXPERIMENTAL

The preparation of mixtures used in this study has been described.[1] For this IR investigation, the mixtures containing Monuron were cured at 130°C and those consisting of dicyandiamide and having "no Monuron" were hardened at 200°C. The curing time for all systems was two hours.

The preparation of samples for IR examination involved two different techniques. Solid materials were measured as potassium bromide (KBr) wafers and slurries were analyzed as thin films on KBr salt plates. The IR spectra were recorded on a Digilab FTS-10M Fourier transform infrared spectrometer. Each spectrum consisted of 120 co-added interferometric scans at 4-cm^{-1} resolution. All uncured specimens were measured from 4000 cm^{-1} to 400 cm^{-1}.

DISCUSSION AND RESULTS

The accelerated cure of dicyandiamide (Dicy)-containing epoxy resins is a complicated process and the cure mechanism was elucidated in this laboratory.[2-4] The low temperature Dicy cure is dependent on the fact that the trisubstituted urea accelerator (Monuron) first reacts with the oxirane ring to form a matrix which is partially composed of cyclic 2-oxazolidones as indicated below:



Although the by-product, dimethylamine, is able to readily react with the epoxy functionally, the amine plays a more important role in the hardening process. This amine

1. LaLIBERTE, B. R., and SACHER, R. E. *The Dicyandiamide Cure of SP-250 Epoxy Resin Accelerated by Monuron.* Army Materials and Mechanics Research Center, AMMRC TR 83-17, April 1983.
2. LaLIBERTE, B. R., SACHER, R. E., and BORNSTEIN, J. *Acceleration of the Epoxy Resin-Dicyandiamide Cure Cycle by Trisubstituted Ureas.* Army Materials and Mechanics Research Center, AMMRC TR 81-30, June 1981.
3. LaLIBERTE, B. R. *The Reaction of Diuron With Epoxy Groups.* Army Materials and Mechanics Research Center, AMMRC TN 79-1, January 1979.
4. BORNSTEIN, J., LaLIBERTE, B. R., and SACHER, R. E. *The Formation of Cyclic 2-Oxazolidones With Respect to the Acceleration of the Dicyandiamide Cure of Epoxy Resins.* Army Materials and Mechanics Research Center, AMMRC TN 82-2, June 1982.

does, in fact, increase the reactivity of Dicy. It has been proposed[5] that dimethylamine has a strong proclivity to remove a proton from Dicy, thus, enhancing the nucleophilic nature of the hardening agent (Dicy). This would account for the increased reactivity of Dicy.

The SP-250 resin is a mixture of three very different types of epoxy materials. The SP-250 formulation is summarized as to reflect a distribution of Monuron and Dicy on the basis of weight percent for each epoxide as shown in Table 1 below:

Table 1.

Epoxide	Epoxide (wt. %)	Dicy (wt. %)	Monuron (wt. %)
ECN 1273 (Ciba Geigy)	45.8	3.88	1.96
EPON 828 (Shell)	38.1	3.23	1.63
Active Diluent	4.7	0.40	0.20
	88.6	7.51	3.79

ECN 1273 is a low melting polymeric material having an epoxy novalac structure. EPON 828 comes in a liquid form and this mixture has a considerable amount of diglycidyl ether of bisphenol-A. The active diluent is described as a liquid flexibilizer having one epoxy group per molecule.

Infrared analysis of the curing behavior of the blends depicted in Tables 2 and 3 was performed using three different IR frequencies. These bands include the 1510 cm^{-1} (phenyl C=C semicircle stretch), the 915 cm^{-1} (the oxirane ring deformation), and the 1745 cm^{-1} frequency (carbonyl stretch which is characteristic of 2-oxazolidone derivatives). The results of the analysis are listed in Tables 2 and 3.

The maximum cure conversion which yielded the lowest 915 cm^{-1} to 1510 cm^{-1} ratio was found to be the EPON 828/Monuron/Dicy system which is also associated with the highest ratio of oxazolidone regarding the epoxy band. These results were somewhat anticipated based on predictions contained in our recent study.[1] The speculations resulted from the introduction of scanning rates having the dimensions of temperature per time. These values are believed to be a measure of the rapidity in which a mixture will achieve about 50% of its hardening cycle. Also, the ECN 1273/EPON 828/Monuron/Dicy system, 0.032 cm^{-1}, and the ECN 1273/EPON 828/Monuron system, 0.043 cm^{-1}, yielded high epoxy conversion. The active diluent system itself with Dicy and/or Monuron does not cure that well. The lack of cure is illustrated with the values of 0.182 cm^{-1} to 0.195 cm^{-1} and with the ratio of 910 cm^{-1} to 1510 cm^{-1}. These results are understandable because of the monofunctional characteristic of the active diluent which would not favor the formation of a high molecular weight product. However, the active diluent/Monuron mixture afforded the greatest 2-oxazolidone formation. Based on the previous predications,[1] this observation was not much of a surprise.

The EPON 828 system cures better than the ECN 1273 mixture and this is illustrated in Table 2. Copies of the infrared scans used in compiling the data in Tables 2 and 3 are incorporated in the Appendix.

5. LaLIBERTE, B. R., and BORNSTEIN, J. *Mechanism of Monuron-Accelerated Dicyandiamide Cure of Epoxy Resins.* Army Materials and Mechanics Research Center, AMMRC TR 81-34, July 1981.

Table 2. INFRARED ANALYSES OF CURING BEHAVIOR

Sample Identification	Ratio of 910 cm^{-1} to 1510 cm^{-1}
EPON 828 + DICY (uncured)	0.246
EPON 828 + DICY (cured)	0.178
EPON 828 + MON (uncured)	0.416
EPON 828 + MON (cured)	0.212
EPON 828 + MON + DICY (uncured)	0.313
EPON 828 + MON + DICY (cured)	0.018
ECN 1273 + DICY (uncured)	0.686
ECN 1273 + DICY (cured)	0.186
ECN 1273 + MON (uncured)	0.810
ECN 1273 + MON (cured)	0.588
ECN 1273 + MON + DICY (uncured)	0.777
ECN 1273 + MON + DICY (cured)	0.195
DILUENT + DICY (uncured)	0.315
DILUENT + DICY (cured)	0.245
DILUENT + MON (uncured)	0.307
DILUENT + MON (cured)	0.195
DILUENT + MON + DICY (uncured)	0.505
DILUENT + MON + DICY (cured)	0.182
EPON 828 + ECN 1273 + DICY (uncured)	0.350
EPON 828 + ECN 1273 + DICY (cured)	0.236
EPON 828 + ECN 1273 + MON (uncured)	0.398
EPON 828 + ECN 1273 + MON (cured)	0.043
EPON 828 + ECN 1273 + MON + DICY (uncured)	0.400
EPON 828 + ECN 1273 + MON + DICY (cured)	0.032
EPON 828 + DILUENT + DICY (uncured)	0.313
EPON 828 + DILUENT + DICY (cured)	0.114
EPON 828 + DILUENT + MON (uncured)	0.225
EPON 828 + DILUENT + MON (cured)	0.198
EPON 828 + DILUENT + MON + DICY (uncured)	0.862
EPON 828 + DILUENT + MON + DICY (cured)	0.570
ECN 1273 + DILUENT + DICY (uncured)	0.581
ECN 1273 + DILUENT + DICY (cured)	0.158
ECN 1273 + DILUENT + MON (uncured)	0.548
ECN 1273 + DILUENT + MON (cured)	0.375
ECN 1273 + DILUENT + MON + DICY (uncured)	0.771
ECN 1273 + DILUENT + MON + DICY (cured)	0.129

Table 3. INFRARED ANALYSIS OF 2-OXAZOLIDONE FORMATION

Sample Identification	Ratio of 1745 cm^{-1} to 910 cm^{-1}
EPON 828 + MON (uncured)	0
EPON 828 + MON (cured)	0.666
EPON 828 + MON + DICY (uncured)	0
EPON 828 + MON + DICY (cured)	6.0
DILUENT + MON (uncured)	0
DILUENT + MON (cured)	0.888
DILUENT + MON + DICY (uncured)	0
DILUENT + MON + DICY (cured)	1.1
EPON 828 + DILUENT + MON (uncured)	0
EPON 828 + DILUENT + MON (cured)	0.807
EPON 828 + DILUENT + MON + DICY (uncured)	0
EPON 828 + DILUENT + MON + DICY (cured)	0.200

CONCLUSION

This manuscript is an initial study on the application of infrared spectroscopy as an analytical tool for the study of the curing process of exoxy-based systems. In this study, two specific vibrational frequencies were utilized to follow the cure. The epoxy frequency was normalized to the phenyl C=C semicircle stretch at 1510 cm^{-1} which is considered to be inert in the course of the hardening process. The carbonyl frequency was found to increase in the presence of Dicy and this band was normalized to the epoxy absorbance.

For a better understanding of the cure mechanism, the various SP-250 mixtures should be cured in the IR spectrometer with respect to temperature and time.

Further work is ongoing in the evaluation of the extent of cure with respect to chemistry. This work includes the following considerations:

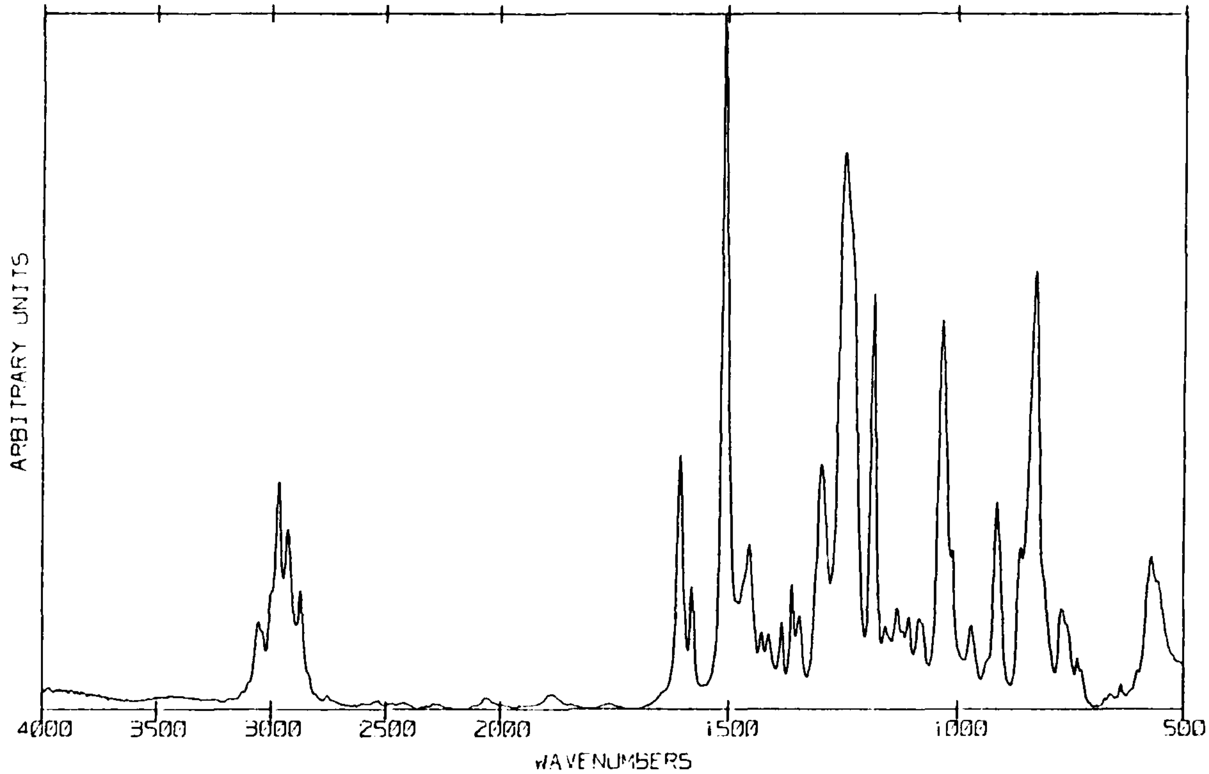
A. The evaluation of other inert frequencies for normalization such as the 1600 cm^{-1}, phenyl C=C quadrant stretch, and the 830 cm^{-1}, p-substitution frequency. The 1240 cm^{-1} bond phenyl-O-C stretch is also being studied as a normalizing frequency.

B. The use of the region from 3600 cm^{-1} to 3200 cm^{-1} (formation of alcohols during the amide reaction), 2400 cm^{-1} to 2000 cm^{-1} (C-N stretch characteristic of dicyandiamide), and the 1200 cm^{-1} to 900 cm^{-1} may be employed as a measure of cross-linking.

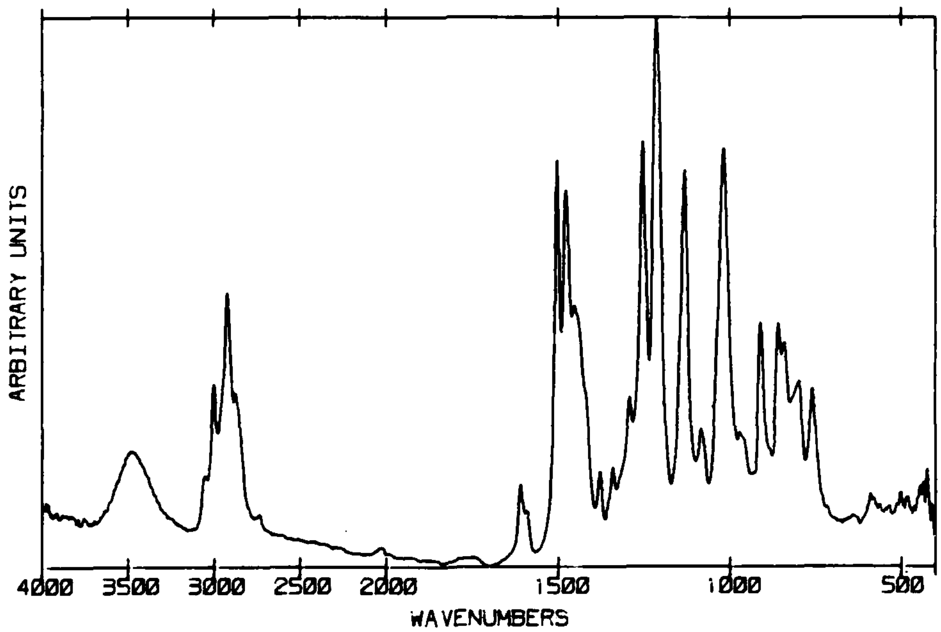
C. The formation of C-N linkages between the epoxy system and the Dicy hardener are part of the cure process. These linkages are difficult to detect by IR analysis but are pertinent to the cure cycle. Investigations of this type have commenced and will continue in the future.

A DSC paper is being prepared which involves the isothermal analysis of the SP-250 mixtures. The results of this study added technical substance to our scanning rates.[1] Likewise, this IR investigation indicates that the rates are worthy of a theoretical consideration.

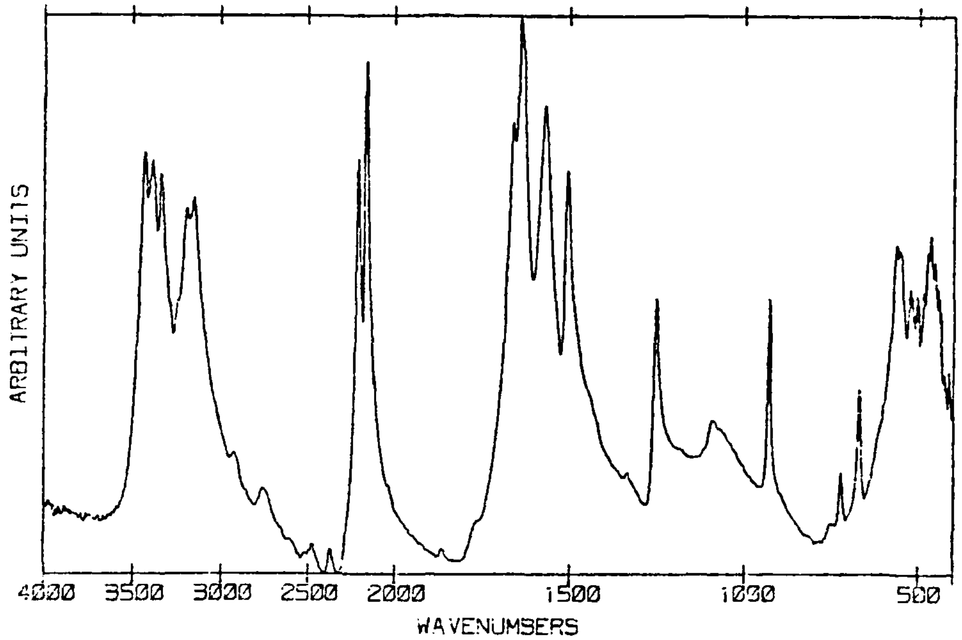
APPENDIX



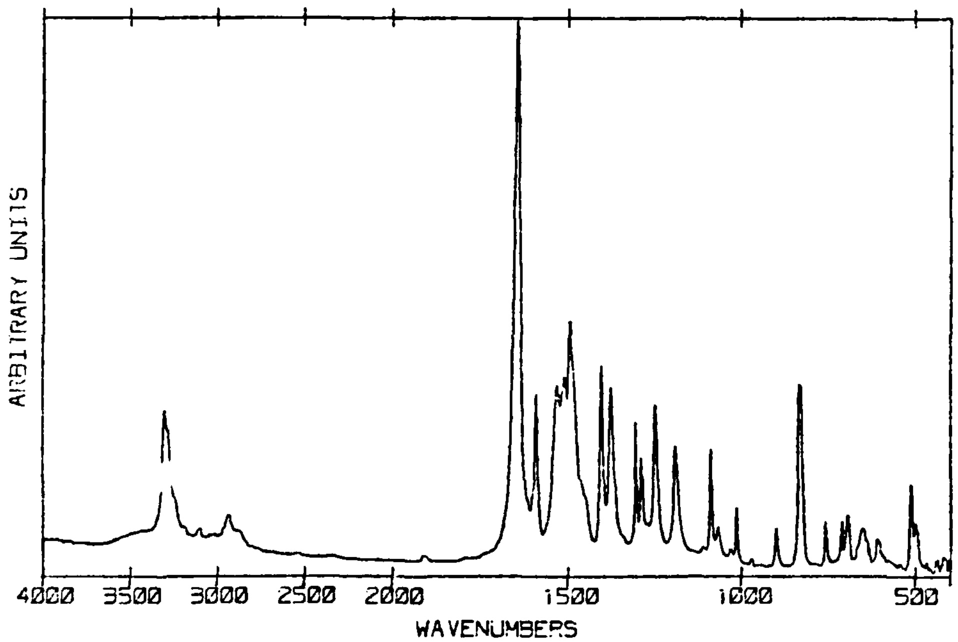
DIGLYCIDYLETHER OF BISPHENOL A (DGEBA) MONOMER



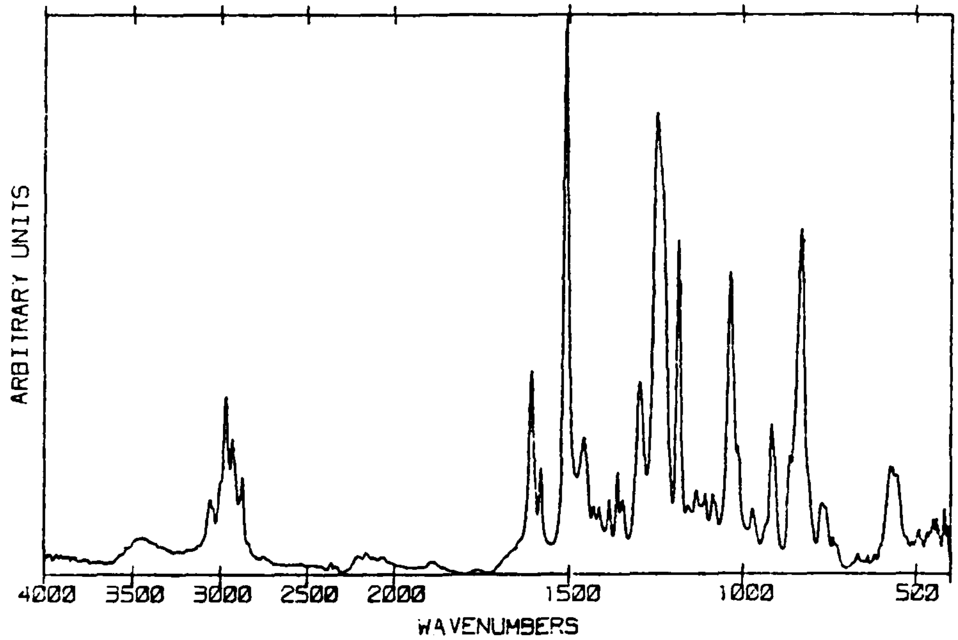
ECN 1273 RESIN KBR PELLET



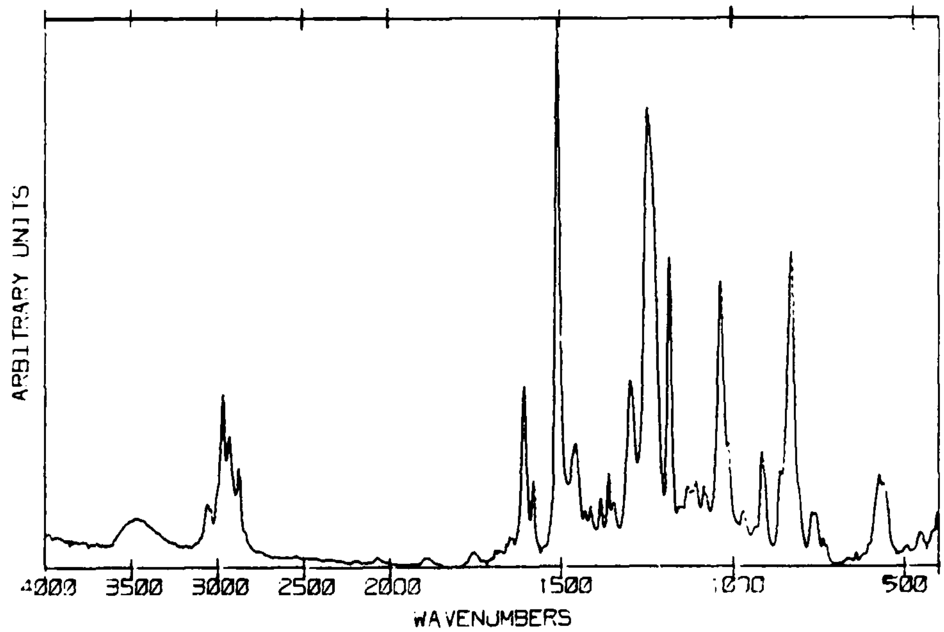
DICYANDIAMIDE 97% KBR PELLET



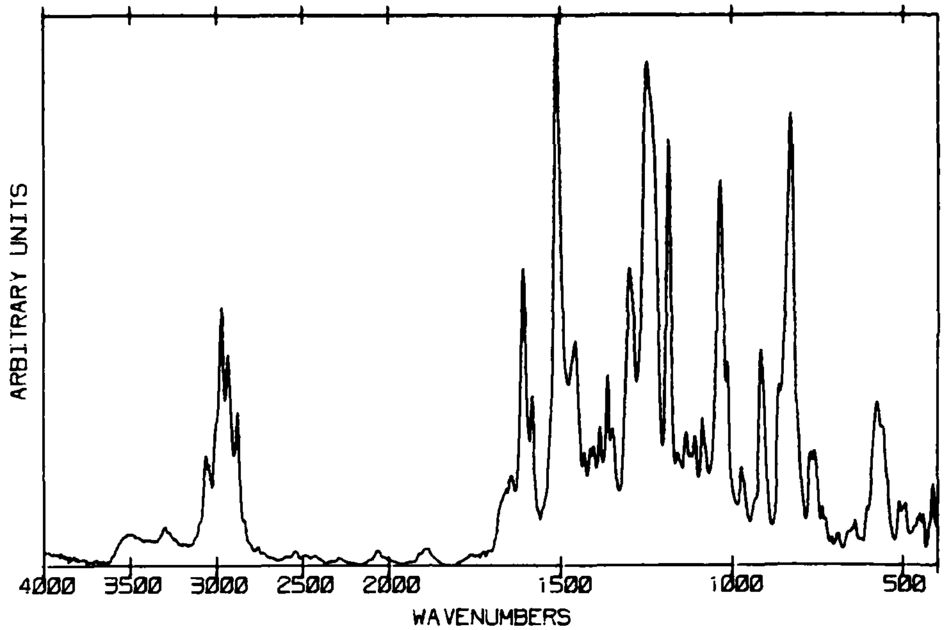
MONURON KBR PELLET



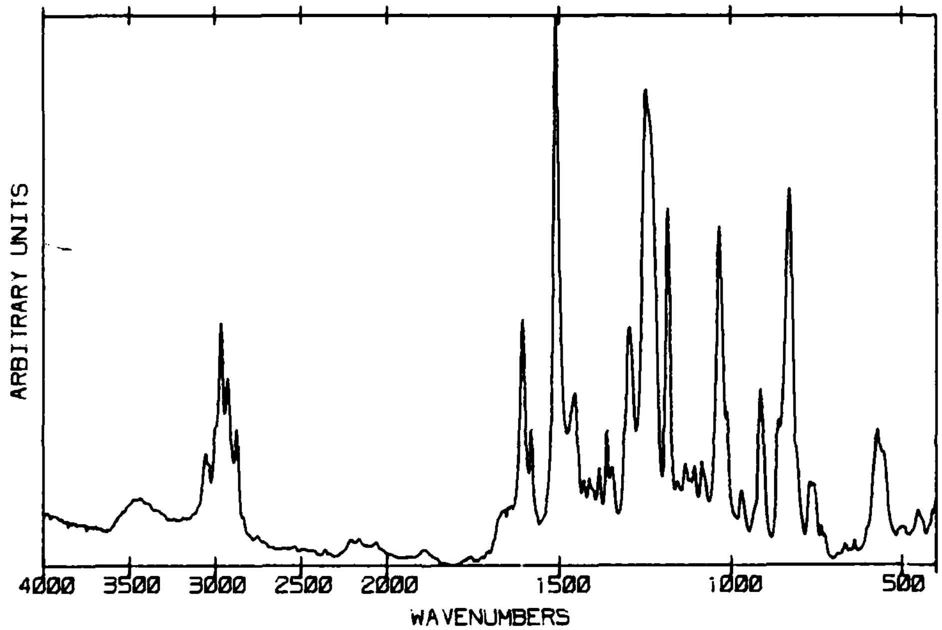
33.1% EPON 828, 3.23% DICY UNCURED THIN FILM



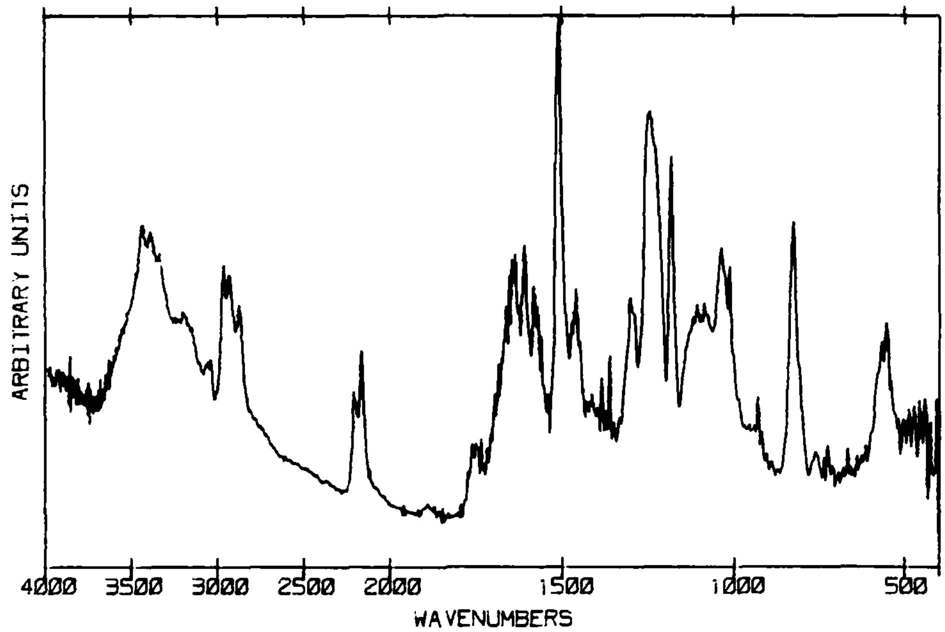
33.1% EPON 828, 3.23% DICY CURED @ 180 C FOR 2 HRS. THIN FILM



EPON 828, 1.63% MONURON UNCURED THIN FILM



38.1% EPON 828, 3.23% DICY , 1.63% MONURON UNCURED



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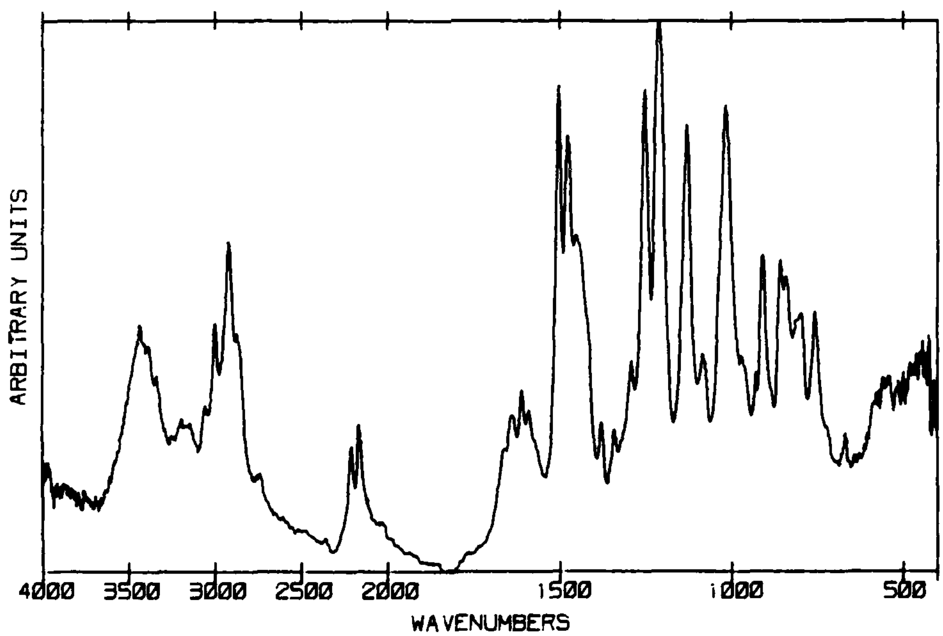
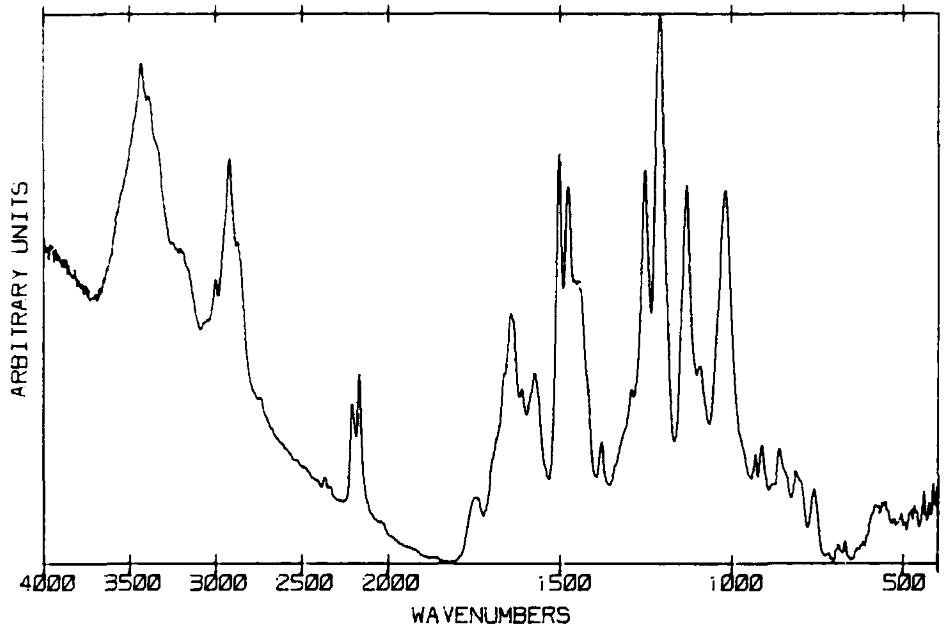
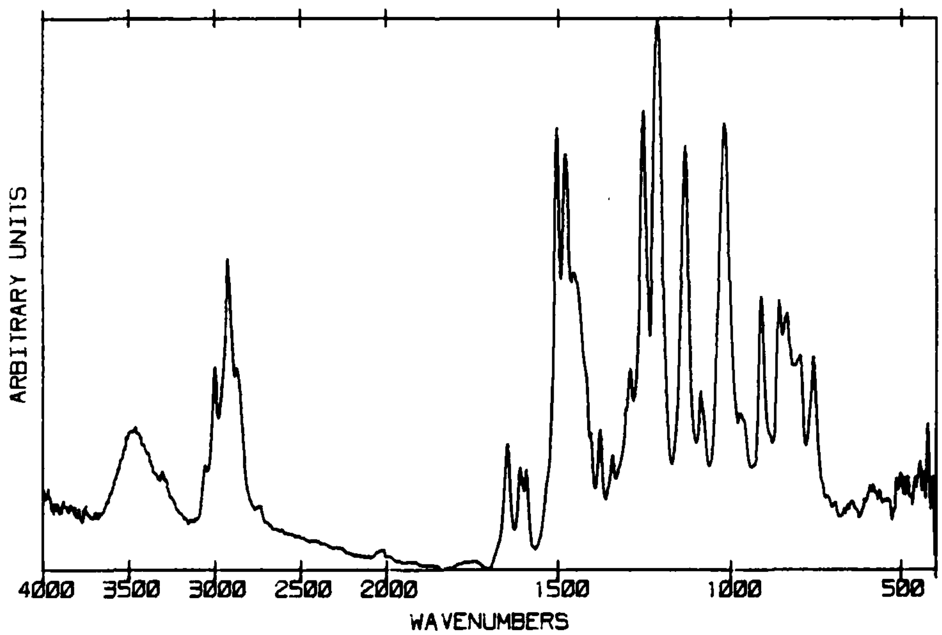


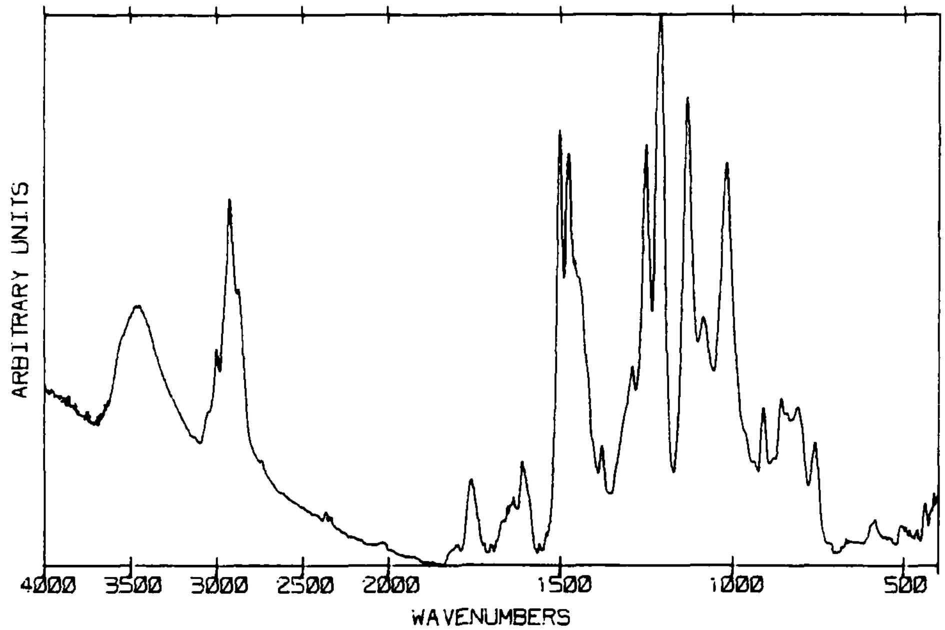
ECN1273 3.88% DICY UNCURED KBR PELLET



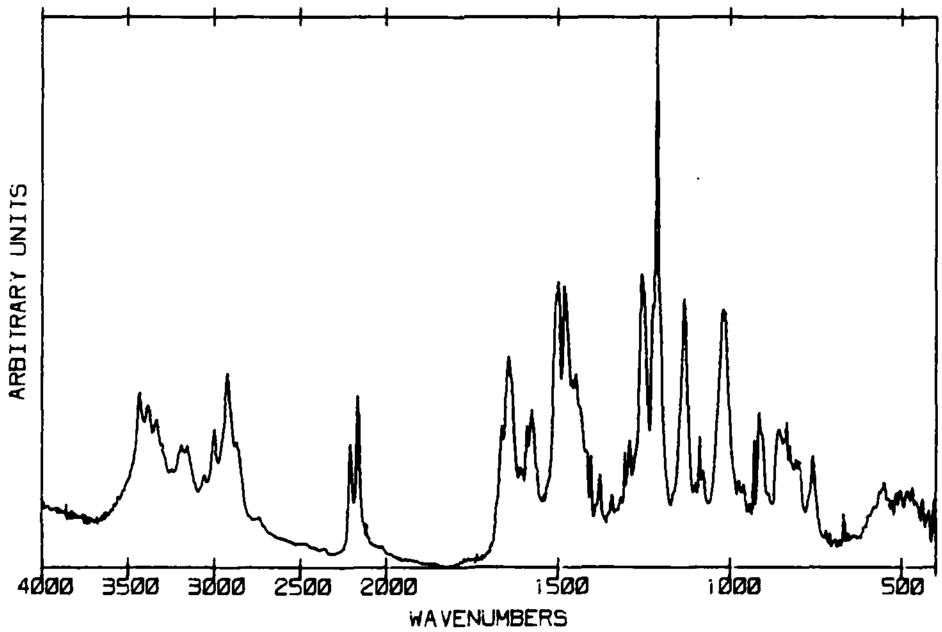
ECN 1273 3.88% DICY (CURED)



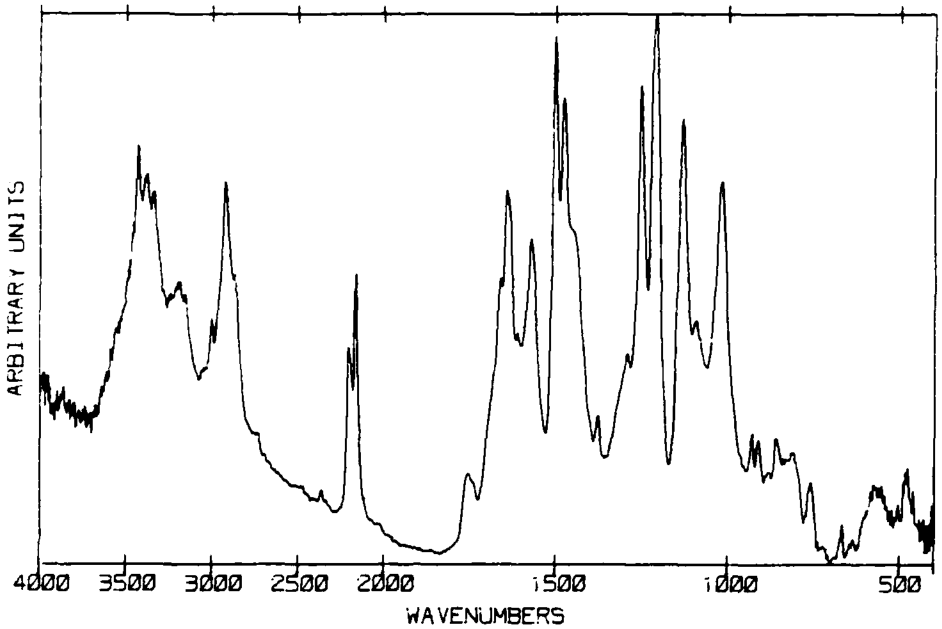
ECN1273 1.96% MONURON UNCURED KBR PELLET



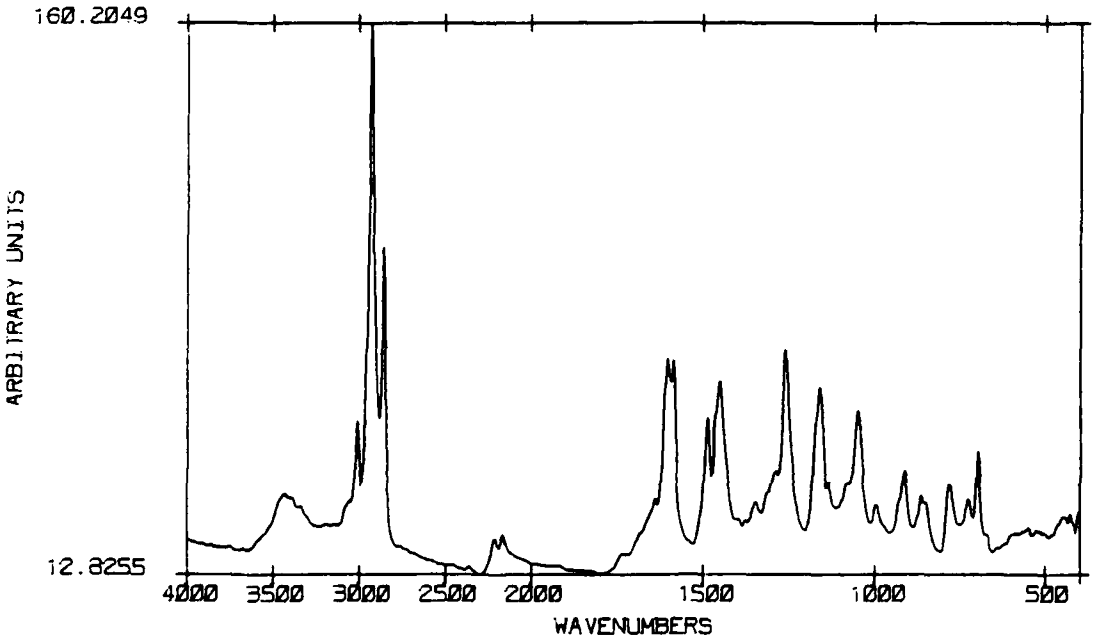
ECN1273 1.96% MONURON (CURED)



ECN 3.88% DICY, 1.96% MONURON UNCURED KBR PELLET



ECN 1273 3.88% DICY 1.96% MONURON (CURED)



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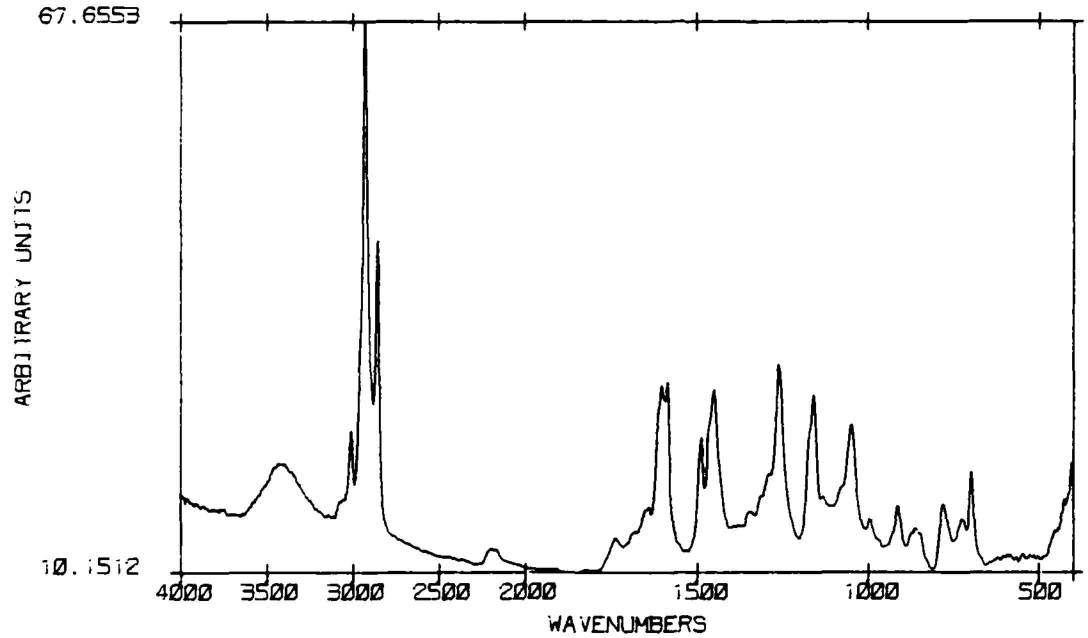

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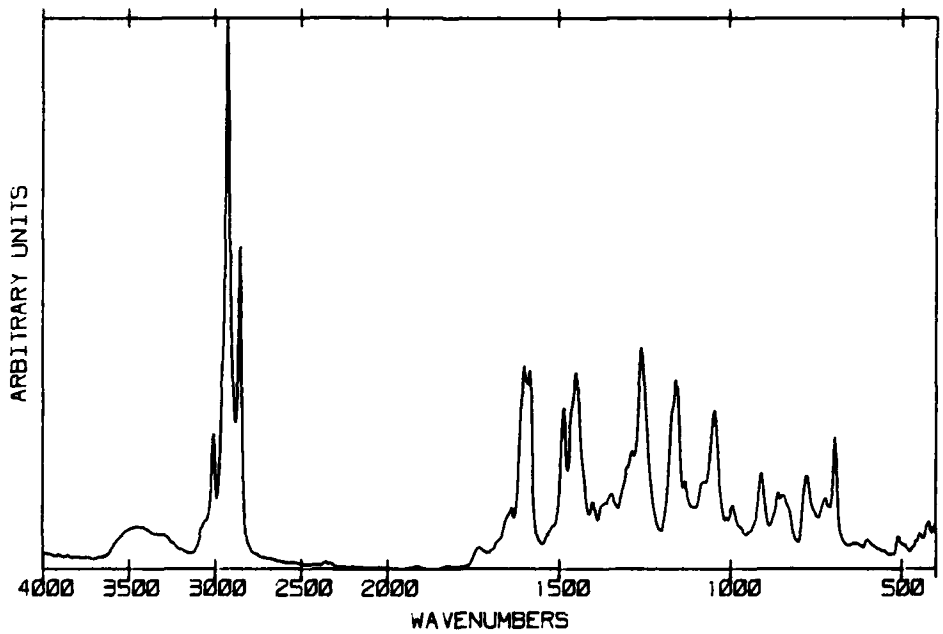

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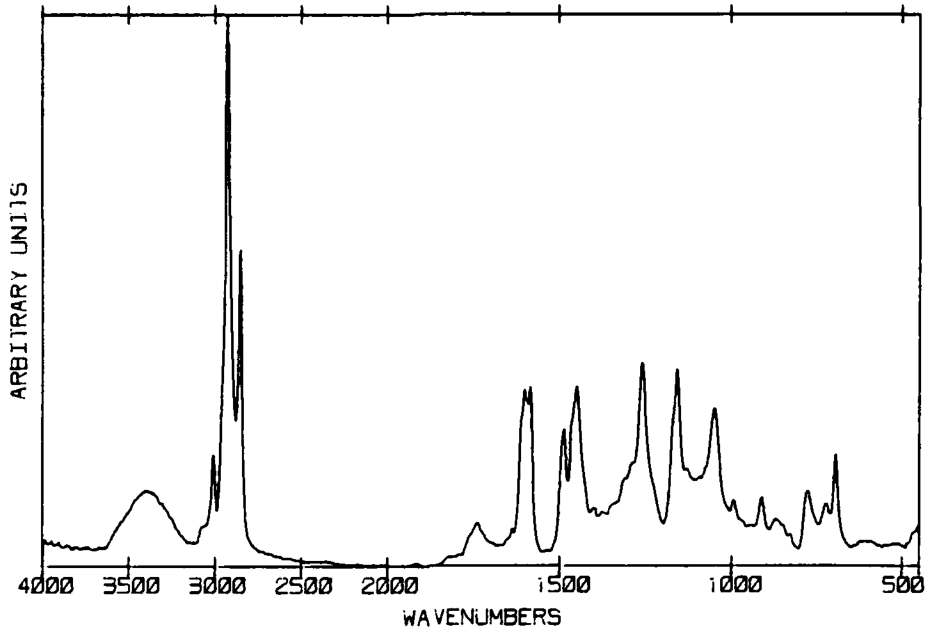

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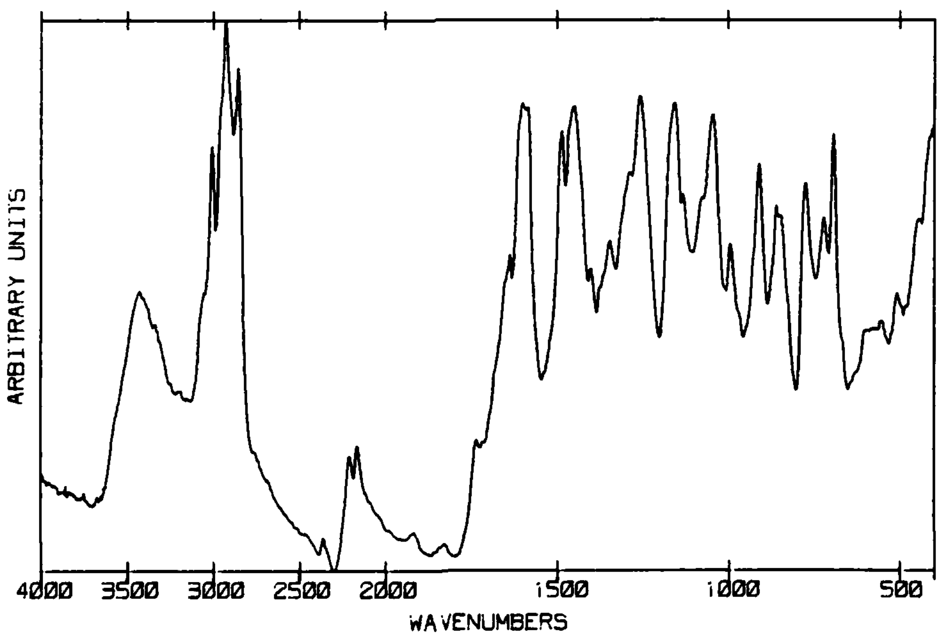

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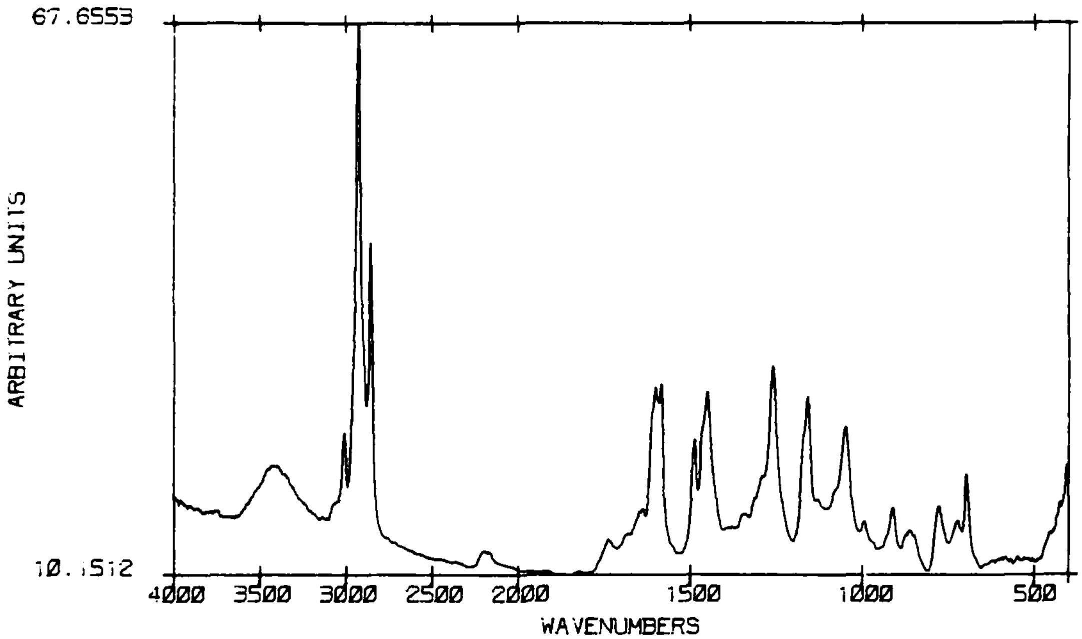

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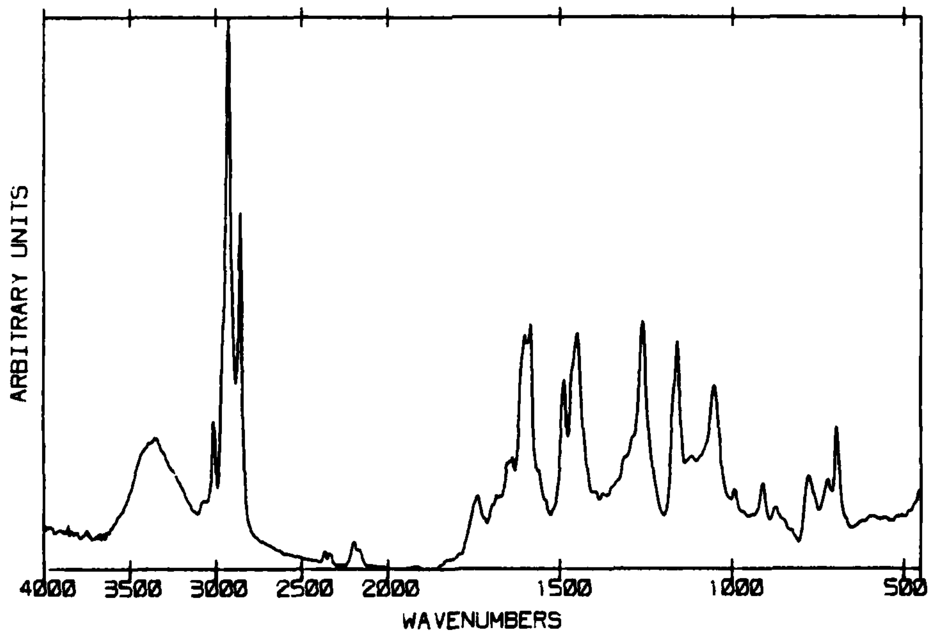
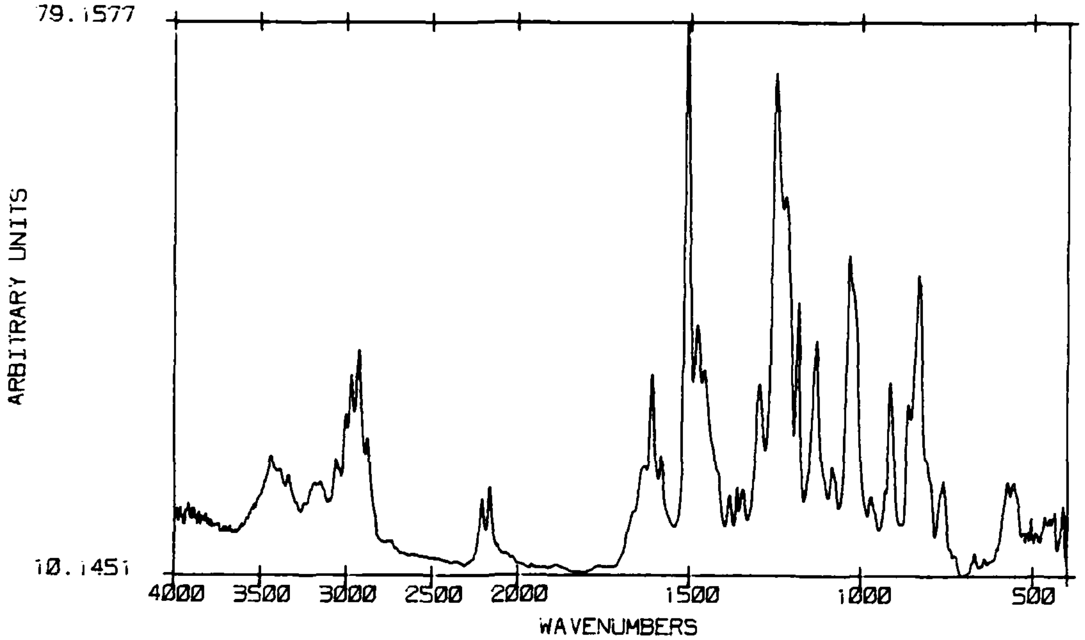
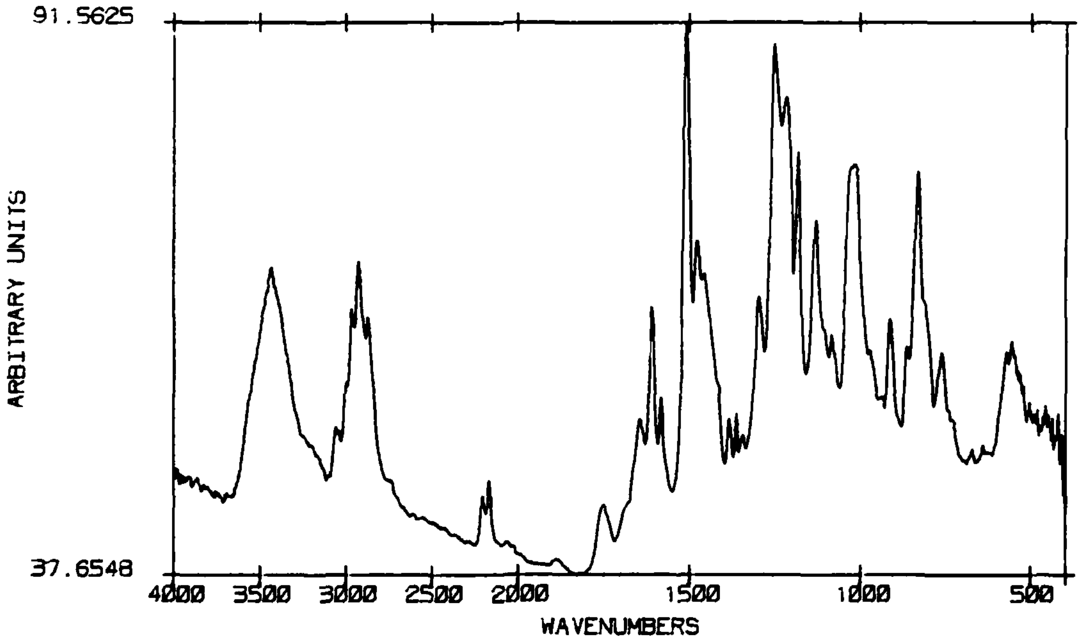


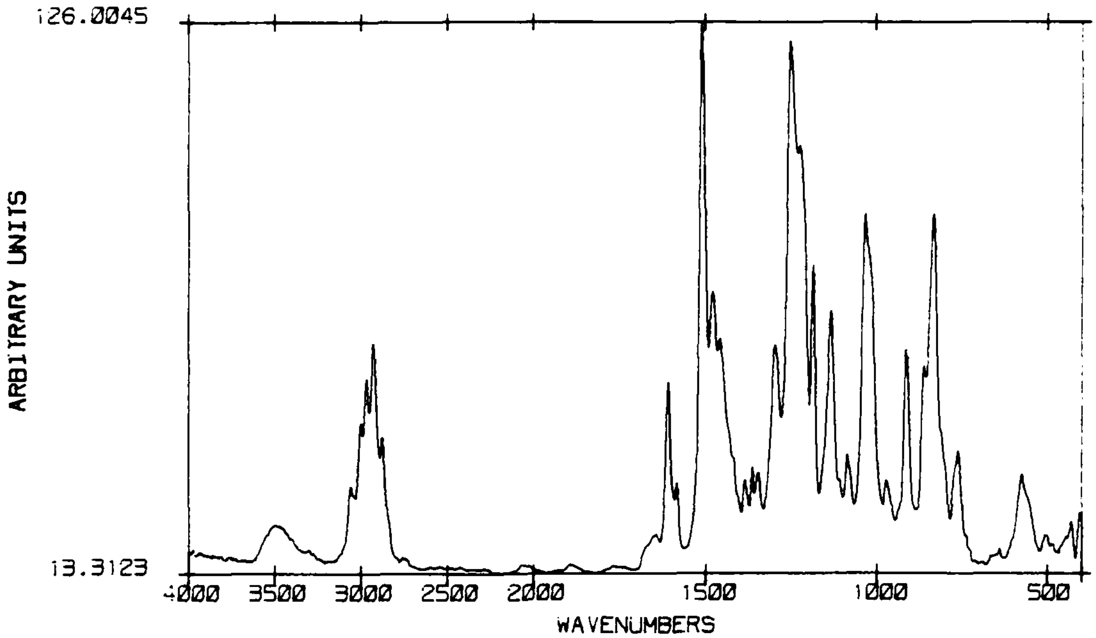
CARDOLITE 513 (4.7%), MONURON (0.2%), DICY (0.4%) CURED @ 130C 2 HRS.



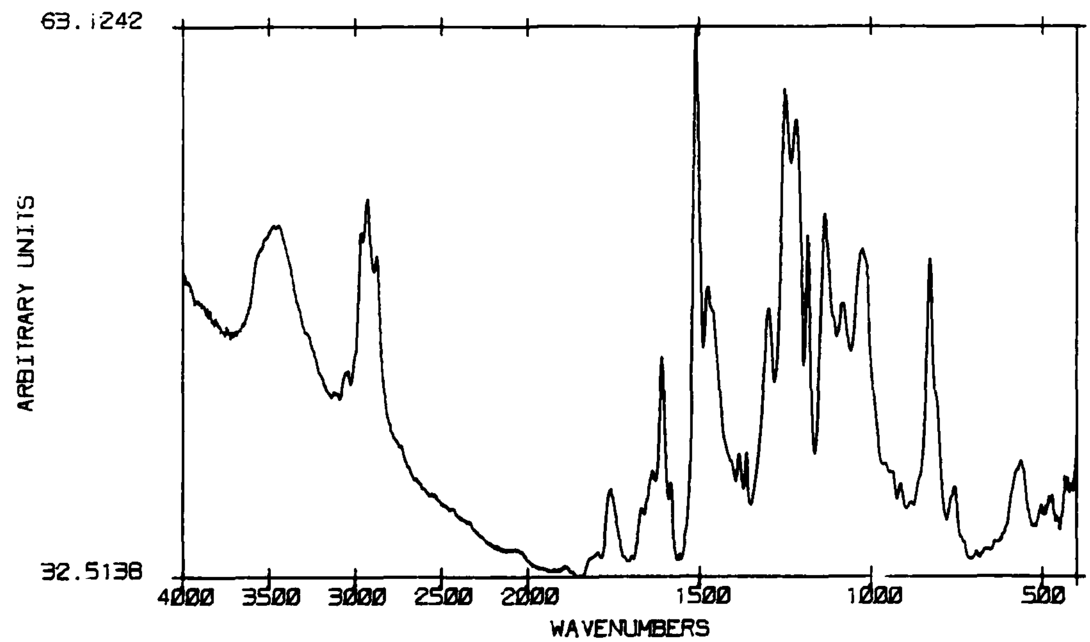
FCN 1273 EPON 828 DICY (7.11) UNCURED



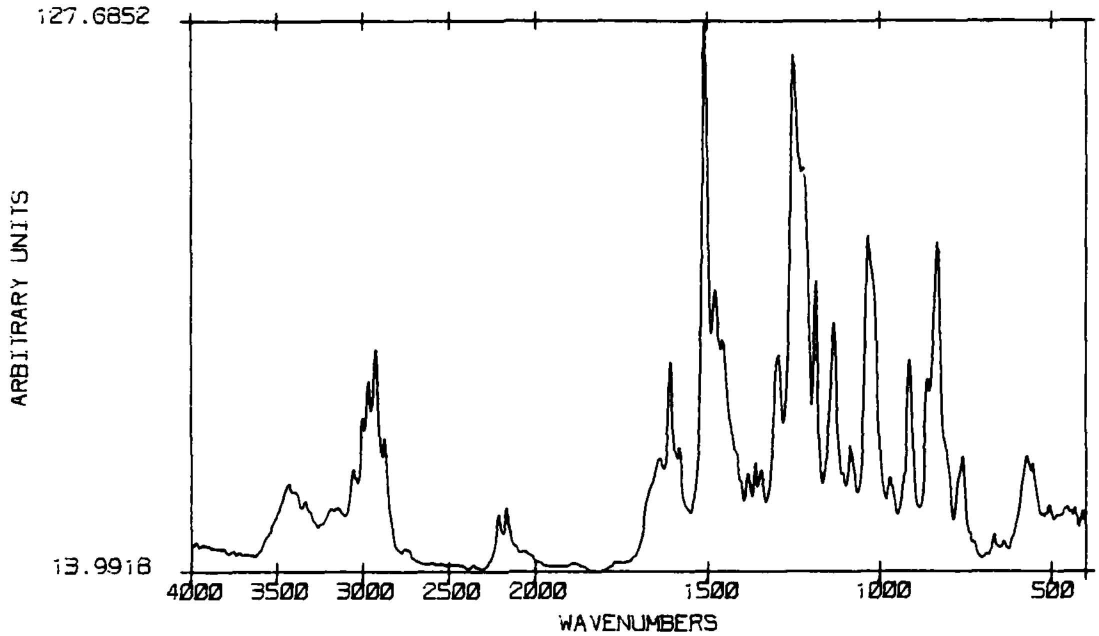
ECN 1273 EPON828 DICY 7.11 CURED 180 C 2 HRS.



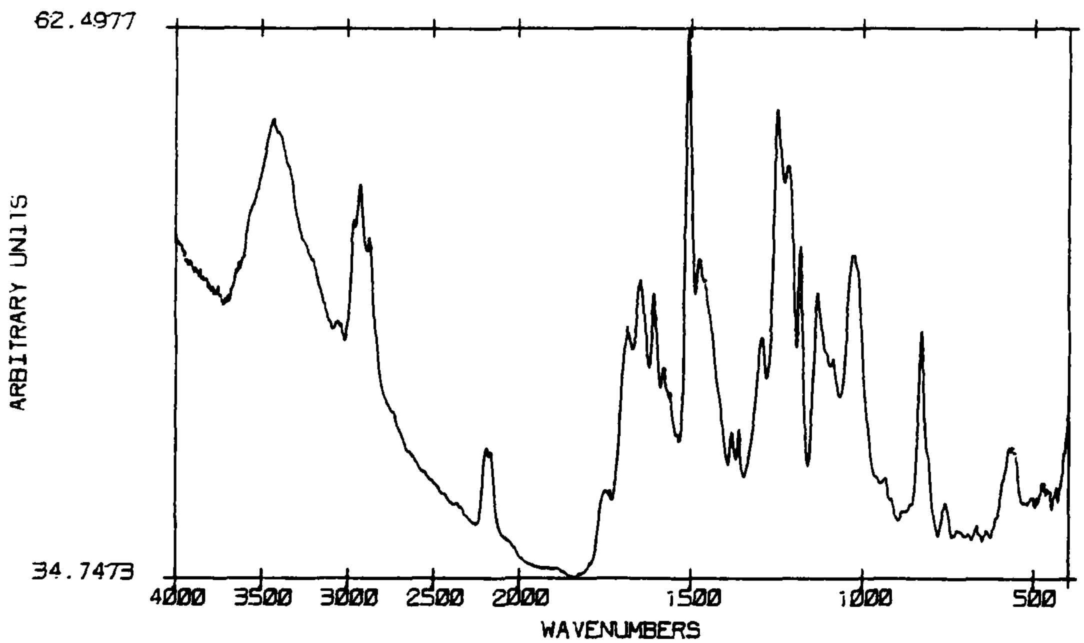
ECN & EPON & MONURON UNCURED THIN FILM



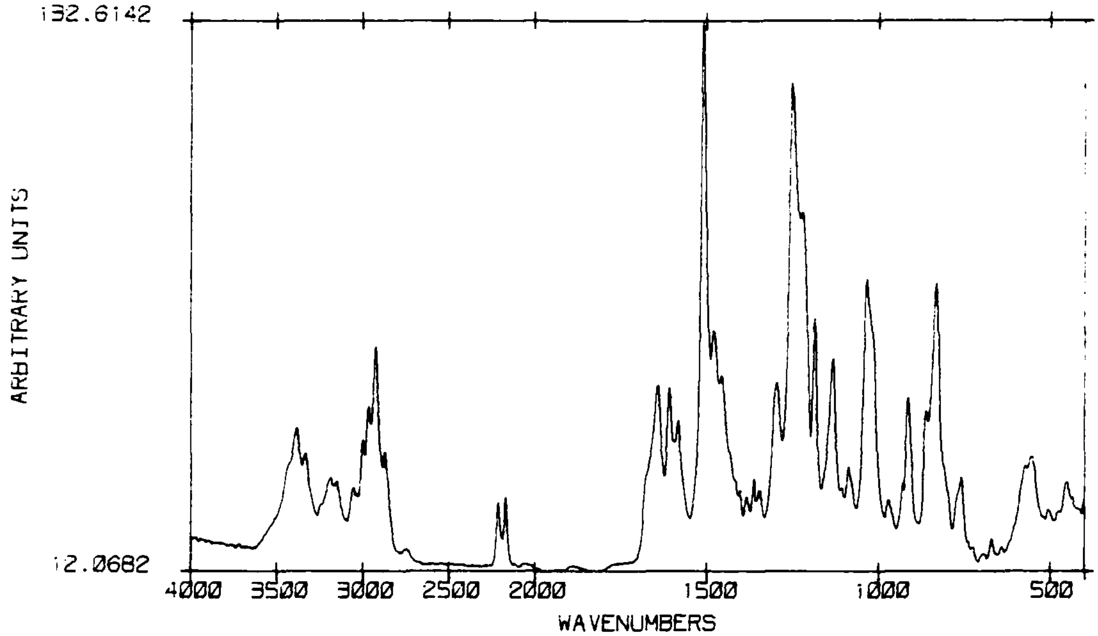
ECN,EPON,MONURON(3.59%) CURED 2 HRS. @ 130 C KBR



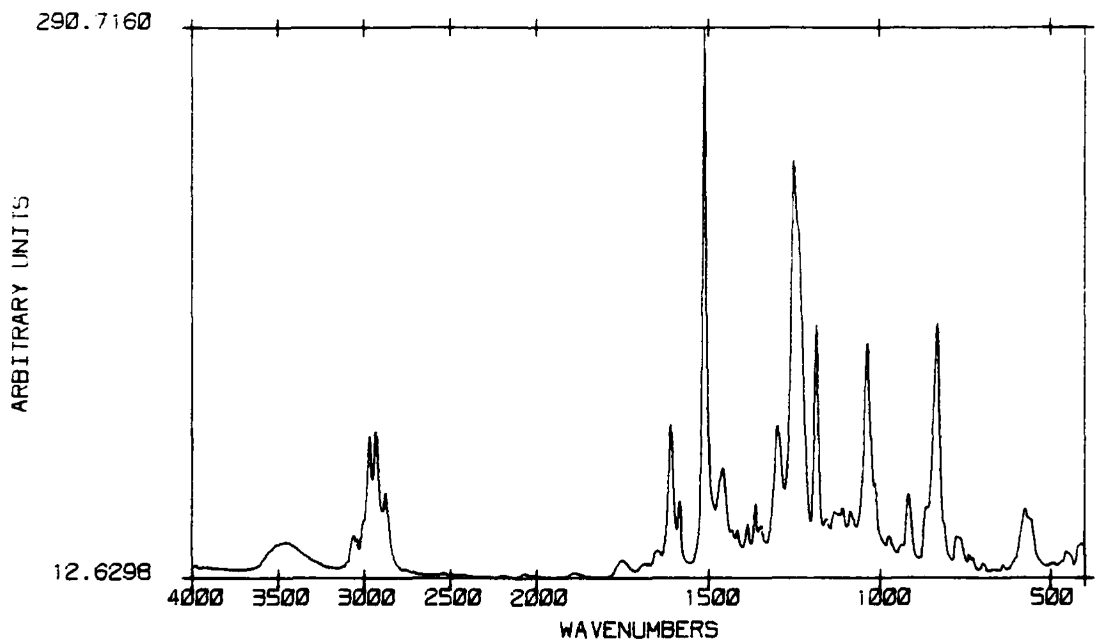
EPON 828,ECN 1273,MONURON(3.59%),DICY(7.11%) UNCURED TF



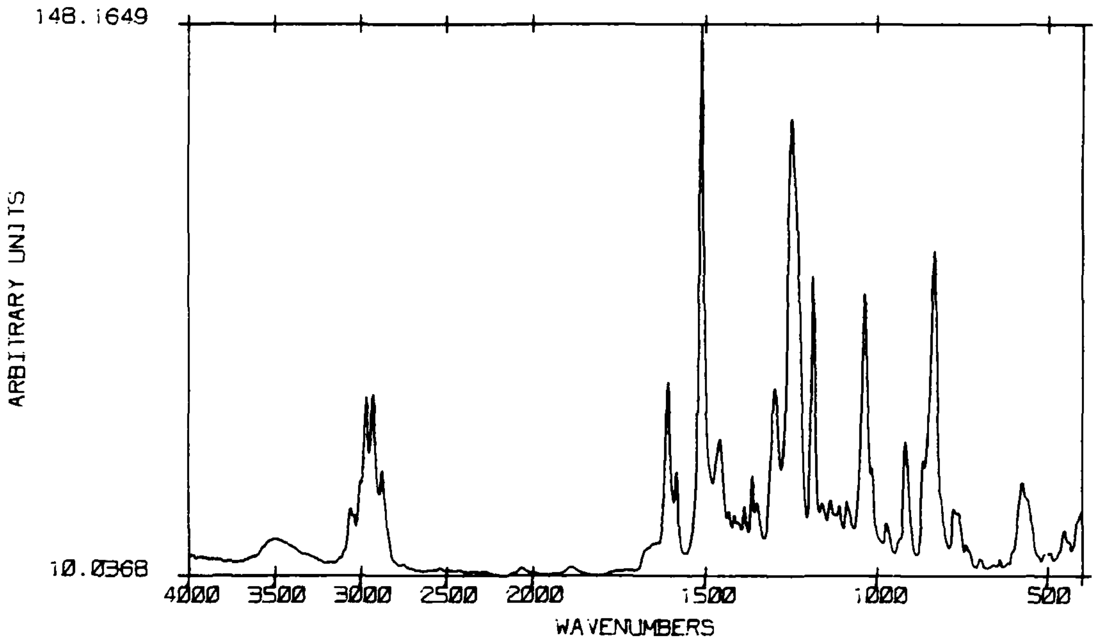
EPON 828,ECN 1273,MONURON(3.59%),DICY(7.11%) CURED 2 HRS. @ 130 C KE



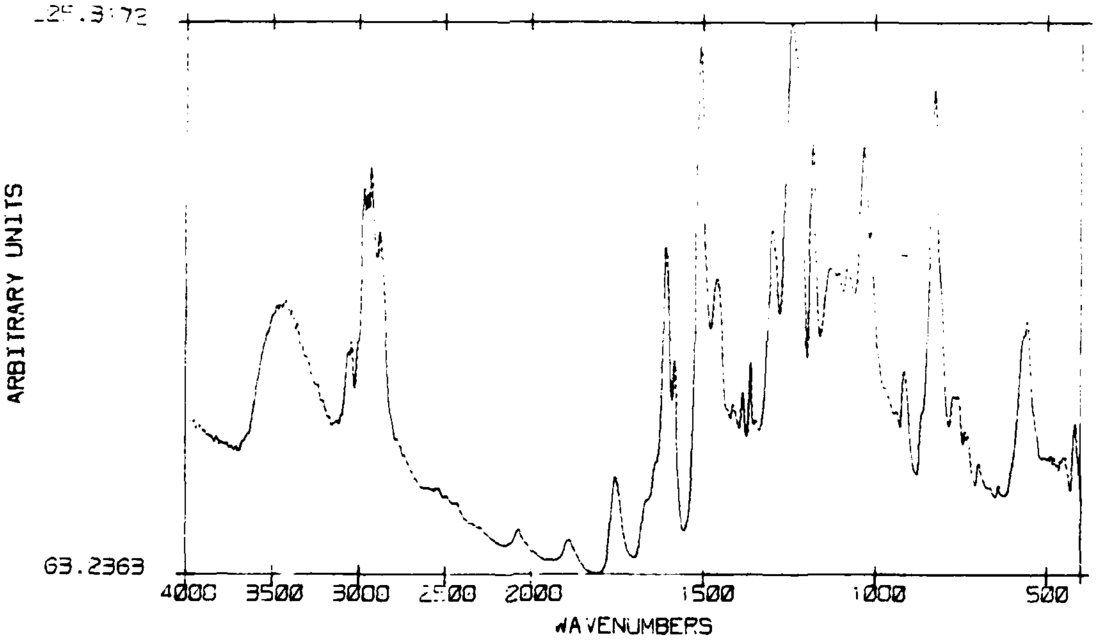
EPON, CORDOLITE & DICY (3.63%) UNCURED THIN FILM



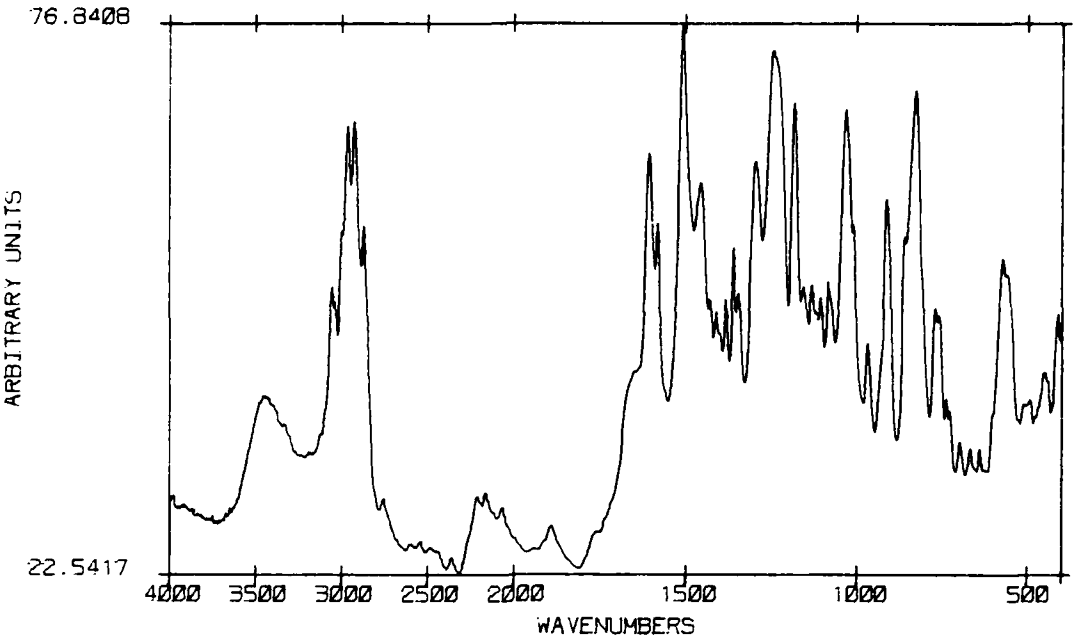
EPON, CARDOLITE & DICY (3.63%) CURED FOR 2 HRS. @ 180 C



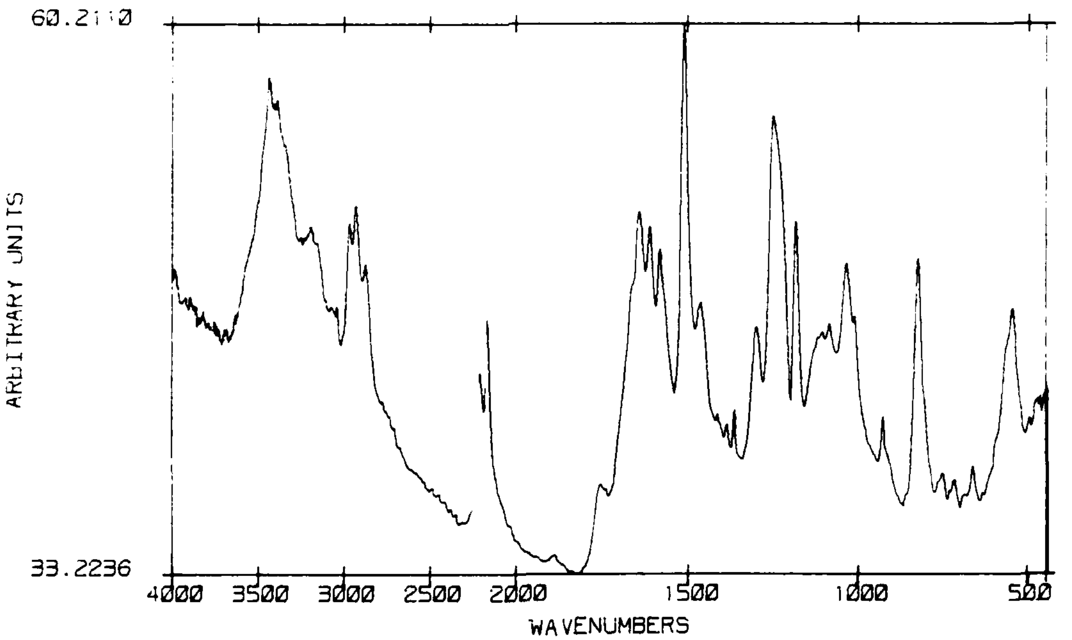
EPON↓PON 828(38.1%)CARDOLITE(4.7%),MONURON(9.83%) UNCURED THIN FILM



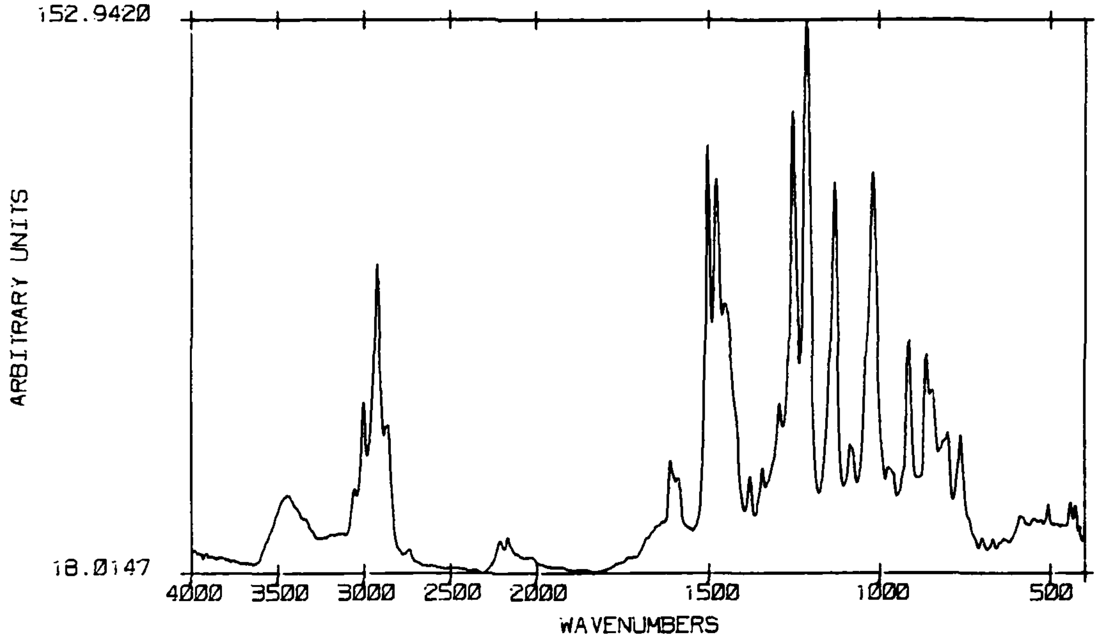
EPON 828(38.1%),CARDOLITE 513(4.7%),MONURON(1.83%) CURED 2 HRS. @ 130



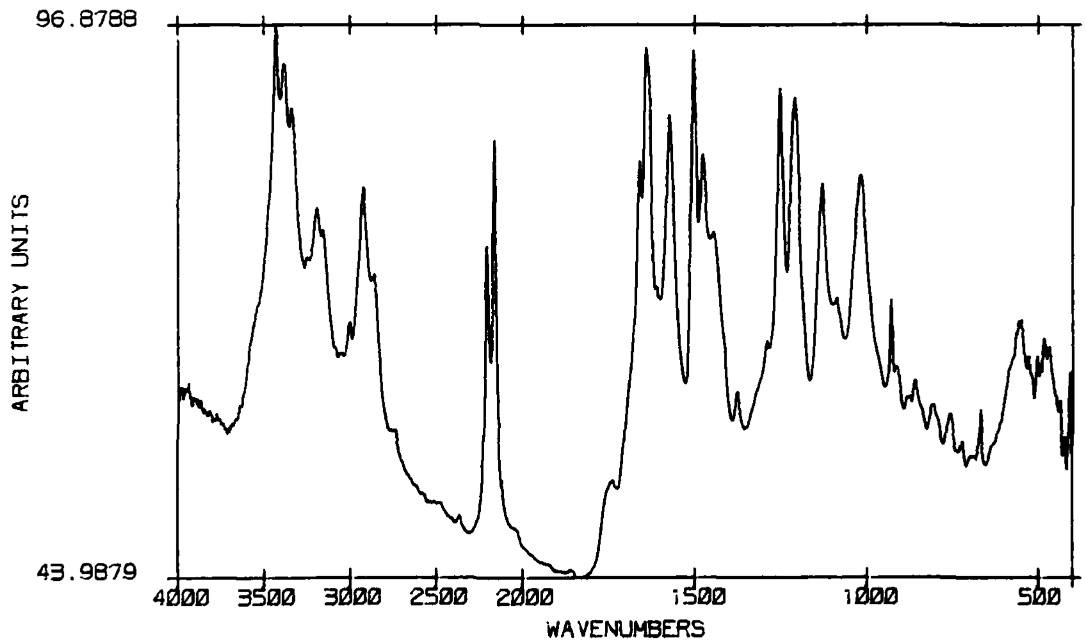
EPON 828, CARDOLITE, MONURON, DICY UNCURED



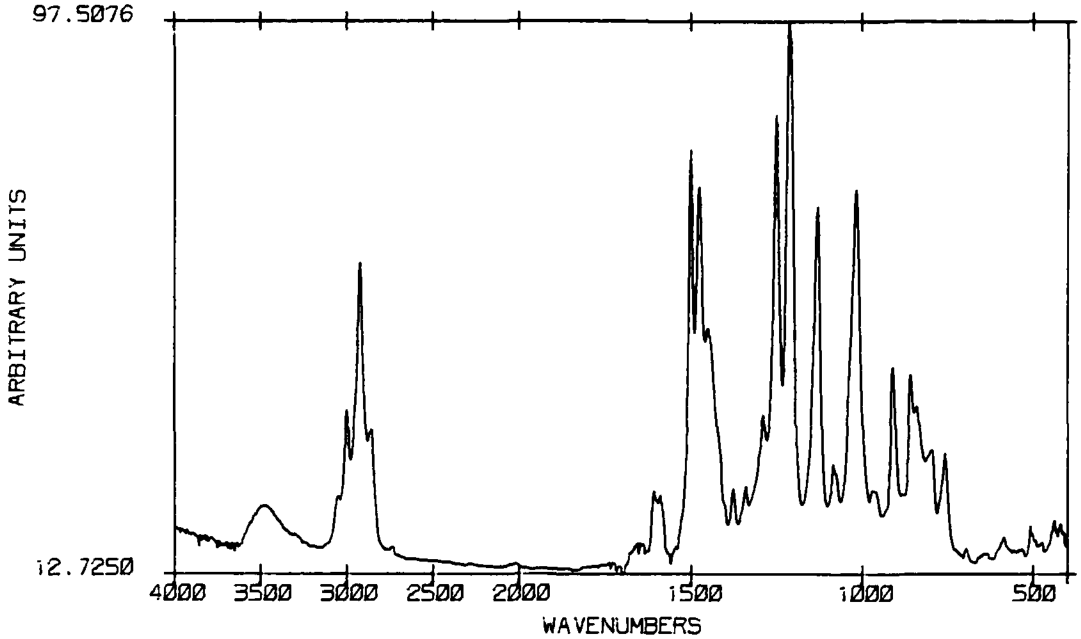
EPON 828, CARDOLITE 1273, MONURON, DICY CURED FOR 2 HRS. @ 130 C KBR



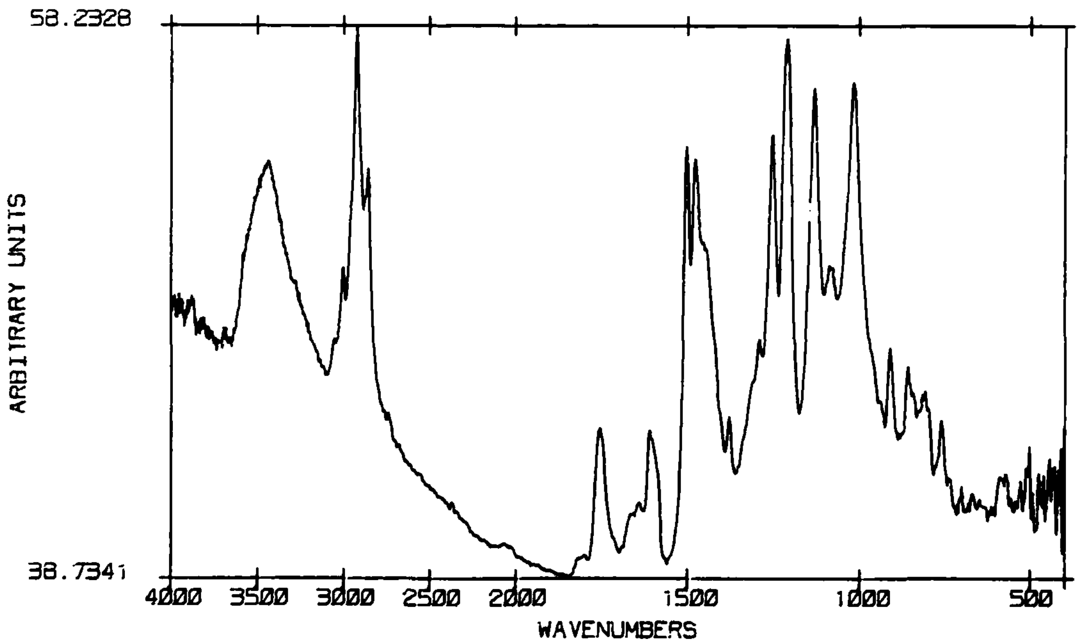
ECN1273 DICY CARDOLITE 513 UNCURED



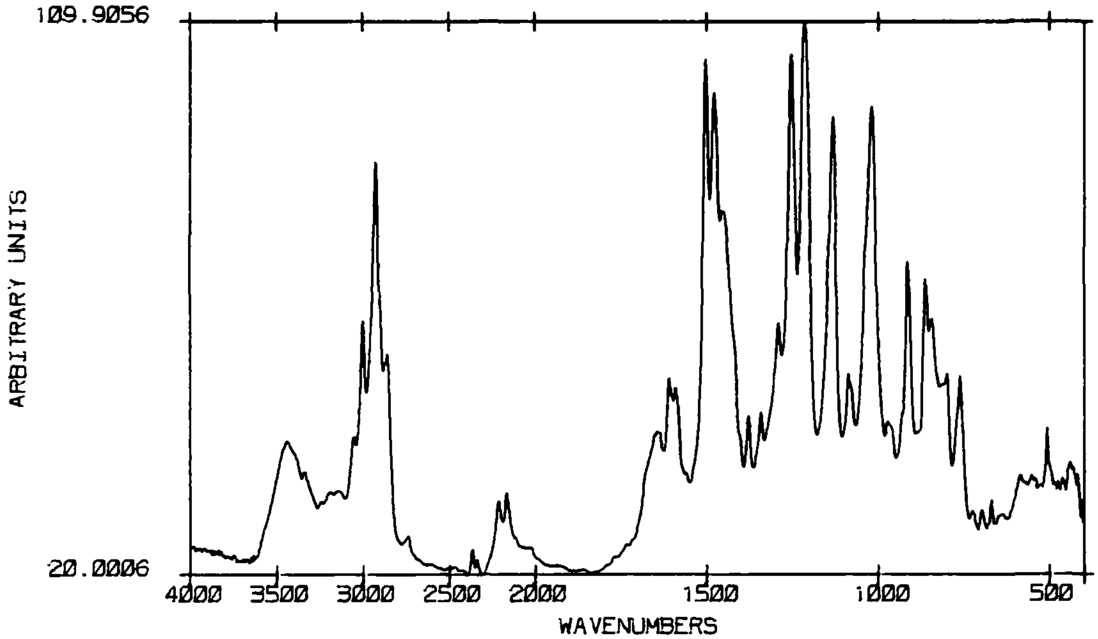
ECN 1273 CARDOLITE 513 DICY CURED 180 C 2 HRS.



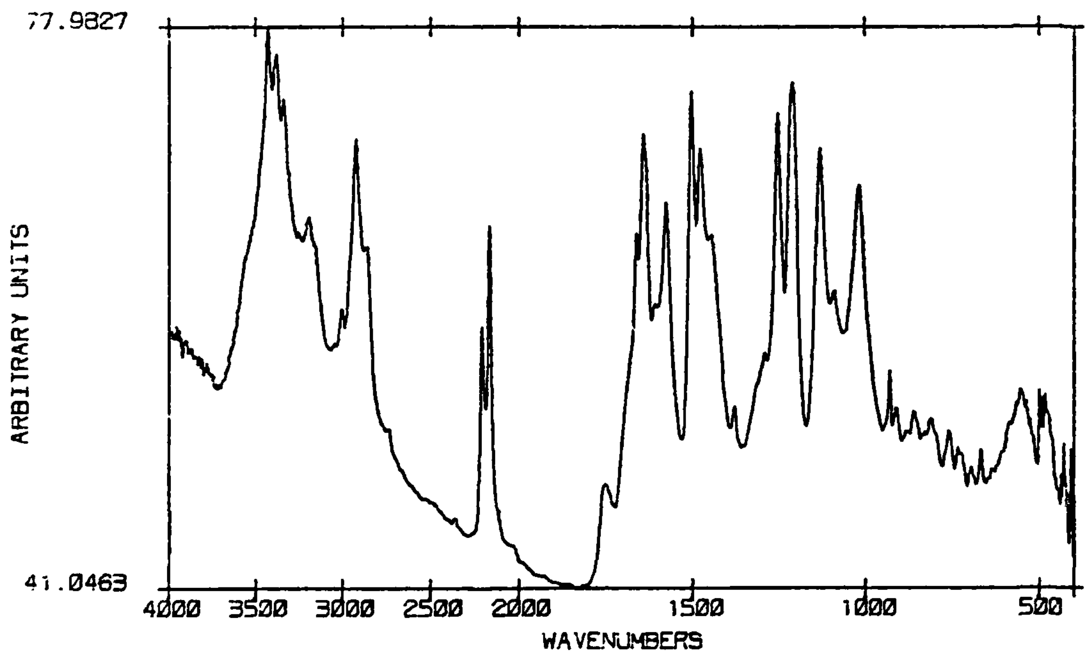
ECN 1273, CARDOLITE 513 MONURON UNCURED



ECN 1273, CARDOLITE 513 MONURON: CURED 130 2 HRS.



ECN 1273, CARDOLITE 513 MONURON, DICY UNCURED



ECN 1273 CARDOLITE 513 DICY MONURON CURED 130 C 2 HRS.

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INFRARED SPECTROSCOPY STUDY OF THE SP-250
EPOXY RESIN SYSTEM -
Robert E. Sacher and Bernard R. LaLiberte

Technical Report AMMRC TR 84-29, July 1984, 26 pp -
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Monuron

The various epoxides which are part of the SP-250 formulation (3M Company, St. Paul, Minnesota) were treated to three different curing procedures. In this study, the epoxides and the epoxy blends were individually cured with dicyandiamide (Dicy) and Monuron, respectively, and in combination (Dicy/Monuron). The acceleration of the Dicy-containing epoxy resin cure is linked to the formation of cyclic 2-oxazolidone. The characteristic nature of the carbonyl band of these cyclic derivatives prompted us to monitor the hardening process by infrared spectroscopy. The cure conversion was evaluated to be the highest for the EPON 828 + Dicy/Monuron and ECN 1273/EPON 828 + Dicy/Monuron systems. The epoxide ± accelerator reaction affording 2-oxazolidone was found to be the greatest in the active diluent + Monuron system. Regarding neat EPON 828, the presence of Dicy significantly enhanced the formation of 2-oxazolidone.

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