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THE CHEMICAL, THERMAL, AND DYNAMIC
MECHANICAL PROPERTIES OF FIVE
ADHESIVES

By G. K. Baker

Published August 1978

Prepared for the United States Department of Energy
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**Kansas City
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THE CHEMICAL, THERMAL, AND DYNAMIC MECHANICAL PROPERTIES OF FIVE ADHESIVES

BDX-613-1853 (Rev.), UNCLASSIFIED Final Report, Published August 1978

Prepared by G. K. Baker

The chemical, thermal, and dynamic mechanical properties of five adhesives have been determined. The analyses performed include the determination of the amount and identity of the unbound chemicals extracted from the cured adhesives, the swell ratio, the amount of residual amino, isocyanate, and epoxy groups, dynamic thermogravimetric analysis, isothermal gravimetric analysis, thermal mechanical analysis, storage shear modulus, loss shear modulus, and loss tangent. Two adhesives, Halthane 73-18 and Halthane 87-1, exhibit the best overall properties. They have a high degree of cure, good thermal stability, and elastomeric behavior in the general temperature range required.

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CONTENTS

Section	Page
SUMMARY.	7
DISCUSSION	8
SCOPE AND PURPOSE.	8
ACTIVITY	8
<u>Description of Adhesives</u>	8
<u>Chemical Analyses</u>	9
<u>Thermal Analyses</u>	21
<u>Dynamic Mechanical Analyses</u>	24
ACCOMPLISHMENTS.	35
APPENDIX. SHEAR MODULUS DATA.	38
DISTRIBUTION	49

ILLUSTRATIONS

Figure		Page
1	Infrared Spectra of Methylene Chloride Extracts of Halthane 73-18	13
2	Infrared Spectra of Methylene Chloride Extracts of Halthane 87-1.	13
3	Infrared Spectra of Methylene Chloride Extracts of LW520/MDIPA.	14
4	Infrared Spectra of Methylene Chloride Extracts of LW520/ER508/MDA.	14
5	Infrared Spectra of Methylene Chloride Extracts of ER508/ER505/TEPA	15
6	Gel Permeation Chromatographs of Methylene Chloride Extracts of Halthane 73-18.	16
7	Gel Permeation Chromatographs of Methylene Chloride Extracts of Halthane 87-1	17
8	Gel Permeation Chromatographs of Methylene Chloride Extracts of LW520/MDIPA	18
9	Gel Permeation Chromatographs of Methylene Chloride Extracts of LW520/ER508/MDA	19
10	Gel Permeation Chromatographs of Methylene Chloride Extracts of ER508/ER505/TEPA.	20
11	Thermal Mechanical Thermograms of Halthane 73-18	23
12	Thermal Mechanical Thermograms of Halthane 87-1.	24
13	Thermal Mechanical Thermograms of LW520/MDIPA.	25
14	Thermal Mechanical Thermograms of LW520/ER508/MDA.	26
15	Thermal Mechanical Thermograms of ER508/ER505/TEPA	27
16	Dynamic Thermogravimetric Analyses of Five Adhesives	29

17	Isothermogravimetric Analyses of Five Adhesives.	30
18	Storage Shear Modulus of Halthane 73-18 as a Function of Temperature	32
19	Storage Shear Modulus of Halthane 87-1 as a Function of Temperature	33
20	Storage Shear Modulus of LW520/MDIPA as a Function of Temperature.	34
21	Storage Shear Modulus of LW520/ER508/MDA as a Function of Temperature	36
22	Storage Shear Modulus of ER508/ER505/TEPA as a Function of Temperature	37

TABLES

Number		Page
1	Swell Ratio and Extractables Content of Five Adhesives in Methylene Chloride	11
2	Residual Amine, Epoxy, and Isocyanate Groups in Five Adhesives	12
3	Transistion Temperatures of Five Adhesives Determined by Thermal Mechanical Analysis. .	28
4	Weight Loss of Five Adhesives After Thermo- gravimetric Analysis	31
A-1	Shear Modulus Data of Five Adhesives	38

SUMMARY

The chemical, thermal, and dynamic mechanical properties of five adhesives, cured at room temperature, 50°, and 70°C, have been determined. The adhesives were developed at Lawrence Livermore Laboratory (LLL) and the Union Carbide Corporation's Y-12 Plant.

Two adhesives, Halthane 73-18 and Halthane 87-1, have the best overall chemical, thermal, and dynamic mechanical properties for all three cure conditions. They have a high degree of cure, good thermal stability, and show elastomeric behavior in the general temperature range required.

Halthane 73-18, Halthane 87-1, and LW520/ER508/MDA have a good degree of cure for all three cure conditions. The adhesives, LW520/MDIPA and ER508/ER505/TEPA, require a cure temperature of 70°C to develop a high degree of crosslinking.

All the adhesives exhibit good thermal stability up to 150°C as determined by dynamic thermogravimetric analysis. Only one adhesive, ER508/ER505/TEPA, exhibited continued weight loss when held at 150°C for 15 minutes. The thermal mechanical analyses show three adhesives, Halthane 73-18, Halthane 87-1, and LW520/MDIPA, have subambient glass transition temperatures and reasonably high softening temperatures indicating elastomeric behavior over the potential operating range.

The storage and loss shear moduli and loss tangent of each adhesive were determined by the forced torsional pendulum geometry method over a temperature range of -120° to 120°C at a frequency of 0.2 Hz. The data correlate well with that of the chemical and thermal analyses. The moduli of Halthane 73-18 having various cure temperatures are virtually identical, and the data for Halthane 87-1 indicate the room temperature cure material crosslinks in a different manner than the elevated cure adhesives. The two adhesives containing epoxy resin, LW520/ER508/MDA and ER508/ER505/TEPA, have high shear moduli at room temperature and below. They also show significant changes in modulus with the different cure conditions.

DISCUSSION

SCOPE AND PURPOSE

New adhesives are required for use in some of the newer programs. Several potential adhesive formulations have recently been developed at Lawrence Livermore Laboratory and the Union Carbide Corporation's Y-12 Plant. Because the adhesives are new, certain material property evaluations are required to ascertain their usefulness in a given application. As a result, Bendix Kansas City was requested to determine the chemical, thermal and dynamic mechanical properties of five adhesives systems.

ACTIVITY

Description of Adhesives

Five adhesives were analyzed. They were prepared at UCC/Y-12 on January 5, 6, and 7, 1976 and cured using three conditions: room temperature, 48 hours at room temperature plus 7 days at 50°C, and 48 hours at room temperature plus 7 days at 70°C. The following formulations were used.

Halthane 73-18 is a polyol cured urethane adhesive. It consists of 64.4 parts by weight of LPA-19-73 and 35.6 parts by weight of HGH-18. LPA-19-73 is an isocyanate prepolymer of 4,4'-methylenebis(phenylisocyanate) and a mixture of Polymeg 1000 and Polymeg 2000 which are polytetramethylene ether glycols made by the Quaker Oats Company. HGH-18 is a mixture of 85 percent Polymeg 1000, 10 percent 1,4-butanediol, and 5 percent Quadrol.

Halthane 87-1 is an amine cured urethane adhesive and has 93 parts by weight LPA-19-87 (an isocyanate prepolymer of Hylene W and Polymeg 2000) and 7 parts by weight of Tonox 60/40 which is a mixture of aromatic amines.

LW520/MDIPA is an amine cured urethane adhesive consisting of 64.3 parts by weight of Adiprene LW520 (an isocyanate prepolymer of Hylene W and polytetramethylene ether glycols made by Du Pont Corporation) and 10.7 parts by weight 4,4'-methylenebis-2,6-diisopropylaniline (MDIPA).

LW520/ER508/MDA is a combination urethane/epoxy adhesive containing 50 parts by weight each of Adiprene LW520 and Epi-Rez 508 (an epoxy resin made from the diglycidyl ether of bisphenol-A by Jones-Dabney Company) and 19.5 parts by weight of 4,4'-methylenebisaniline (MDA).

ER508/ER505/TEPA is an epoxy adhesive consisting of 50 parts by weight of Epi-Rez 508, 50 parts by weight of Epi-Rez 505, (a modified resin made from the diglycidyl ether of bisphenol A), and 8 parts by weight tetraethylenepentamine (TEPA).

Chemical Analyses

The five adhesives, cured at each of the three conditions, were analyzed for the following characteristics:

The amount of unbound chemicals (extractables);

The swell ratio;

The identification of the extracts by infrared spectrophotometry, gel permeation chromatography, and gas chromatography/mass spectrometry; and

Residual amino, isocyanate, and epoxy groups.

The extractables or amount of unbound chemicals were determined by Soxhlet extraction of 3 to 4 grams of adhesive in methylene chloride for 24 hours. The swell ratio was determined by calculating the ratio of the length of a sample swollen in methylene chloride to the length before swelling occurred.

Infrared analyses of thin films of the extracts from the Soxhlet extractions were made using a Model 221 Perkin-Elmer infrared spectrophotometer. The extracts were also analyzed with the Waters Associates Gel Permeation Chromatograph, Model 200 using tetrahydrofuran as the solvent and the low columns set. Identification of the extracts was further obtained by gas chromatography/mass spectrometry analyses by the following method. Two microliters of the extract dissolved in methylene chloride were injected into the gas chromatograph. The inlet temperature was 300°C and the temperature of the column was programmed from 55° to 275°C at a rate of 16°C/minute. The identification of the peaks separated in the column was made by a Hewlett-Packard Model 5982A mass spectrometer.

Residual amino groups in the adhesives were determined by swelling chopped portions of the adhesive in toluene and titrating the amino groups with perchloric acid in acetic acid. The residual isocyanate groups were determined by back titration with hydrochloric acid of an aliquot of 2N dibutylamine which had been allowed to react with the free isocyanate groups. Residual epoxy groups were determined by titration with perchloric acid in acetic acid in the presence of tetraethylammonium bromide.

The data for the extractables content and swell ratio of the five adhesives cured at the three different conditions are summarized in Table 1. The results of the analyses for residual amine, epoxy, and isocyanate groups are given in Table 2.

The infrared spectra of thin films of the methylene chloride extracts from the samples cured at room temperature and 70°C are shown in Figures 1 through 5. The spectra of the extracts of samples cured at 50°C are not given since they are similar to those of the 70°C cure specimens. The spectrum of the extract of LW520/MDIPA cured at 50°C (Figure 3) is given rather than the one cured at room temperature since the latter completely dissolved during the extraction procedure.

The results given in Table 1 primarily reflect the degree of cure or crosslinking of the adhesives. The extractables content represents the amount of unbound material in the adhesive, and the swell ratio is a qualitative method of comparing the extent of crosslinking. The lower the swell ratio, the greater the crosslinking.

The data show the two Halthane adhesives have very low extractables content and the least change in swell ratio for all cure conditions compared to the other three adhesives. Such data indicate virtually complete cure is achieved by the Halthane adhesives at room temperature. The LW520/ER508/MDA adhesive appears to be similar to the Halthane adhesives in this respect; however, the difference in extractables between room temperature cure (2.64 percent extractables) and 50°C (1.13 percent extractables) is greater compared to the Halthanes (0.71 percent at room temperature and 0.68 percent at 50°C for 73-18 and 1.33 percent at room temperature and 1.01 percent at 50°C for 87-1).

The other two adhesives, ER508/ER505/TEPA and LW520/MDIPA, require an elevated cure of 70°C to achieve good crosslinking. The adhesive, ER508/ER505/TEPA, cured at room temperature and 50°C has very low crosslinking as shown by the high amount of extractables, 17.2 and 11.3 percent respectively, and the fact both disintegrated during the swell test. The LW520/MDIPA adhesive, cured at room temperature, dissolved during the tests which means it was not crosslinked at all.

The gel permeation chromatograph (GPC) curves of the five adhesives cured at three different temperatures are given in Figures 6 through 10. Such data provide information about the number of species in the extracts, their molecular weight, and a semiquantitative estimate of their concentration.

Table 1. Swell Ratio and Extractables
Content of Five Adhesives in
Methylene Chloride

Cure Temperature (°C)	Extractables Content (Percent)	Swell Ratio
Halthane 73-18		
*	0.71	2.08
50	0.68	2.13
70	0.50	2.17
Halthane 87-1		
*	1.33	4.34
50	1.01	4.21
70	0.81	3.98
LW520/MDIPA		
*	Dissolved	Dissolved
50	8.76	6.0
70	0.86	2.81
LW520/ER508/MDA		
*	2.64	1.48
50	1.13	1.87
70	0.31	1.53
ER508/ER505/TEPA		
*	17.16	Disintegrated
50	11.34	Disintegrated
70	4.54	1.32
*Room temperature		

Table 2. Residual Amine, Epoxy, and Isocyanate Groups in Five Adhesives

Cure Temperature (°C)	Residual		
	Amine as NH ₂ (Percent)	Epoxy as H ₃ C ₂ O (Percent)	Isocyanate as NCO (Percent)
Halthane 73-18			
*	**	**	0.075 ±0.018
50	**	**	0.125 ±0.010
70	**	**	0.113 ±0.039
Halthane 87-1			
*	0.204 ±0.004	**	0.061 ±0.020
50	0.194 ±0.001	**	0.054 ±0.024
70	0.183 ±0.001	**	0.055 ±0.019
LW520/MDIPA			
*	0.304 ±0.004	**	0.700 ±0.032
50	0.292 ±0.001	**	1.117 ±0.045
70	0.314 ±0.014	**	0.900 ±0.207
LW520/ER508/MDA			
*	1.761 ±0.029	0.038 ±0.001	***
50	1.678 ±0.012	0.022 ±0.005	***
70	1.674 ±0.004	0.016 ±0.003	***
ER508/ER505/TEPA			
*	2.670 ±0.034	0.010 ±0.001	**
50	2.217 ±0.060	0.034 ±0.002	**
70	2.155 ±0.054	0.001 ±0.001	**

*Room temperature

**None present

***Could not be determined because of interference by epoxy groups

Figure 1. Infrared Spectra of Methylene Chloride Extracts of Halthane 73-18

Figure 2. Infrared Spectra of Methylene Chloride Extracts of Halthane 87-1

Figure 3. Infrared Spectra of Methylene Chloride Extracts of LW520/MDIPA

Figure 4. Infrared Spectra of Methylene Chloride Extracts of LW520/ER508/MDA

Figure 5. Infrared Spectra of Methylene Chloride Extracts of ER508/ER505/TEPA

Extracts of all the adhesives except LW520/ER508/MDA are shown to consist mainly of high molecular weight polymeric material as indicated by the height of the peaks between elution counts 16 and 20. In GPC, a lower elution count indicates a higher molecular weight.

The extracts of Halthane 73-18, Halthane 87-1, and LW520/MDIPA are qualitatively similar (Figures 6, 7, and 8). The three peaks between elution counts 23 and 27 are believed to be the low molecular weight oligomers of polybutylene oxide observed in the gas chromatography/mass spectrometry analyses (GC/MS), discussed later. The peak between 26 and 27 counts may be due to 1,4-butanediol, although this has not been confirmed. In the case of the Halthanes, the type and quantity of a given species in the extract is approximately the same regardless of the cure condition. The extracts of LW520/MDIPA show a significant decrease in both high and low molecular weight material with 70°C cure adhesive compared to 50°C cure adhesive. All of the specimen of LW520/MDIPA cured at room temperature dissolved in methylene chloride and, therefore, was not analyzed by GPC. Most of the extract of LW520/ER508/MDA is low molecular weight, eluting between counts 22 and 28 and is a mixture of polyoxybutylene oxide, diglycidyl ether of bisphenol-A (DGEBA), and low molecular weight epoxy oligomers, according to the GC/MS data presented in this report. The extract from the 70° cure sample shows virtually no high molecular weight material. The GPC of the extracts of the epoxy adhesive, ER508/ER505/TEPA, indicate they are largely of higher molecular weight material, eluting between counts 17 and 21. The cure temperature of 70°C

Figure 6. Gel Permeation Chromatographs of Methylene Chloride Extracts of Halthane 73-18

Figure 7. Gel Permeation Chromatographs of Methylene Chloride Extracts of Halthane 87-1

Figure 8. Gel Permeation Chromatographs of Methylene Chloride Extracts of LW520/MDIPA

Figure 9. Gel Permeation Chromatograms of Methylene Chloride Extracts of LW520/ER508/MDA

Figure 10. Gel Permeation Chromatograms of Methylene Chloride Extracts of ER508/ER505/TEPA

essentially eliminates the low molecular weight components. The results of the gas chromatography/mass spectrometry analyses of the extracts of each adhesive are given.

Halthane 73-18

The extracts from the samples having all three cure conditions were the same. The main components were low molecular weight oligomers of polyoxybutylene (from the Polymeg products). A trace amount of BHT (2,6-di-ter-butyl-p-cresol), an antioxidant added to many commercial products, was found as well as a small amount of bisphenol A.

Halthane 87-1

The results for the 87-1 extracts were identical to that of the 73-18 extracts.

LW520/MDIPA

Similar results to that of the Halthanes were obtained. The main outgassing components were polyoxbutylene. Again, bisphenol A and a trace amount of an adipate ester were observed.

LW520/ER508/MDA

The same grouping of polyoxybutylene oligomers was observed in this extract. Also, DGEBA and fragments of DGEBA from the epoxy resin were found. The amount of DGEBA was less in the extract having the 70°C cure temperature.

ER508/ER505/TEPA

The main components were low molecular weight adducts of polyglycol and DGEBA, presumably from Epi-Rez 505. Some polyglycol, DGEBA, and an adipate ester were found, and an amine identified as N,N'-di-secbutyl-p-phenylenediamine was seen. The presence of this amine is not understood. Tetraethylenepentamine (TEPA) was not observed in the mass spectrum.

Thermal Analyses

Each material was analyzed by dynamic thermogravimetric analysis in dry nitrogen from ambient to 150°C at a heating rate of 10°C per minute, and then by isothermal gravimetric analysis by holding the temperature at 150°C for 15 minutes. Thermal mechanical analyses were made on each adhesive from -150° to 250°C using the penetration probe and a heating rate of 10°C per minute to determine the glass transition temperature and softening temperature.

The thermograms of the thermal mechanical analyses (TMA) are given in Figures 11 through 15. For convenience, a summary of the glass transition and softening temperatures determined by TMA for each adhesive is given in Table 3. As seen in Figures 12 and 13, the glass transition temperatures of Halthane 87-1 and LW520/MDIPA are not readily apparent. The scatter in the data given in Table 3 for these adhesives reflect this problem. Also, the glass transition temperature of ER508/ER505/TEPA cured at 70°C is not easily determined (Figure 15).

Three adhesives, Halthane 73-18, Halthane 87-1, and LW520/MDIPA, have subambient glass transition temperatures. The adhesives, LW520/ER508/MDA and ER508/ER505/TEPA, have glass transition temperatures ranging from 45° to 100°C. The higher glass transition temperatures for these two adhesives is due to the epoxy resin in the formulations.

As would be expected, adhesives containing epoxy resin have the highest softening temperatures of above 250°C. The adhesive containing both urethane and epoxy resins, LW520/ER508/MDA, shows several transitions after the glass transition. The transition occurring at about 160°C may be due to softening of the urethane portion.

The temperature range between the glass transition and softening temperatures for polymeric materials is the useful range for elastomeric behavior. As seen from the data in Table 3, Halthane 73-18 has the largest temperature range for elastomeric behavior of the five adhesives. Also it should be noted that Halthanes 73-18 and 87-1 and LW520/MDIPA exhibit elastomeric behavior in the potential range of temperatures of interest.

The thermograms of the thermogravimetric analyses are given in Figures 16 and 17. Both the dynamic thermogravimetric analyses and the isothermal analyses are presented. A summary of the weight loss for each adhesive from ambient temperature to 150°C after dynamic thermogravimetric analyses and the weight loss after holding the temperature at 150°C for 15 minutes is given in Table 4.

All the adhesives exhibited about the same weight loss of 0.22 to 0.42 percent during the dynamic thermogravimetric analysis from 25° to 150°C. Much of this is probably due to the loss of moisture. Isothermal heating at 150°C for 15 minutes more caused very little additional weight loss except in the case of the epoxy adhesive ER508/ER505/TEPA. The epoxy adhesive is either degrading at 150°C or a low molecular weight, unbounded material is volatilizing. The GPC analysis of the extract of ER508/ER505/TEPA shows very little low molecular weight material in the 70°C cure

Figure 11. Thermal Mechanical Thermograms of Halthane 73-18

specimen, and yet the isothermal TGA data show it has the greatest weight loss of all the adhesives. This would tend to support the suggestion of degradation.

It is noted the adhesives which had been cured at 70°C generally lost more weight compared to the specimens cured at 50°C and room temperature. The reason for this is not understood at this time.

Figure 12. Thermal Mechanical Thermograms of Halthane 87-1

Dynamic Mechanical Analyses

The storage shear modulus, loss shear modulus, and loss tangent of each adhesive was determined with the Rheometric Mechanical Spectrometer using the forced torsional pendulum geometry. Measurements were made at a frequency of 0.2 Hz and over a temperature range of -120° to 120°C . The test samples were rectangles

Figure 13. Thermal Mechanical Thermograms of LW520/MDIPA

approximately 38.1 mm long by 12.7 mm wide by the thickness of the slab received from UCC/Y-12 (usually 3.2 mm).

Plots of the storage shear modulus G' as a function of temperature for each adhesive and cure condition are given in Figures 18 through 22. The tabulated data of storage shear modulus, loss shear modulus (G''), and loss tangent ($\tan \delta$) for each adhesive and cure temperature are provided in the Appendix.

Figure 14. Thermal Mechanical Thermograms of LW520/ER508/MDA

The storage shear moduli of the Halthane 73-18 specimens are all very similar regardless of the cure condition. They have the sharp drop in modulus from about 900 to 4 MPa after passing through the viscoelastic transition region (-60° to -20°C) which is characteristic of polyol cured isocyanate prepolymers.

Figure 15. Thermal Mechanical Thermograms of ER508/ER505/TEPA

The storage shear modulus of the Halthane 87-1 adhesive changes less rapidly than the 73-18 adhesive, and it also has a higher modulus in the rubbery region. This is characteristic of amine cured isocyanate prepolymers. It is reasoned the urea crosslinks of amine cured isocyanates provide stronger secondary (hydrogen)

Table 3. Transition Temperatures of Five Adhesives
Determined by Thermal Mechanical Analysis

Cure Temperature (°C)	Glass Transition Temperature, Tg (°C)	Softening Temperature, Ts (°C)	Ts-Tg (°C)
Halthane 73-18			
*	-42	185	227
50	-35	190	225
70	-35	205	240
Halthane 87-1			
*	-50	85	135
50	-85	105	190
70	-75	110	185
LW520/MDIPA			
*	-60	80	140
50	-40	85	125
70	-65	125	190
LW520/ER508/MDA			
*	45	>250	>205
50	80	>250	>130
70	100	>250	>150
ER508/ER505/TEPA			
*	50	230	180
50	60	>250	>190
70	100	275	175
*Room temperature

Figure 16. Dynamic Thermogravimetric Analyses of Five Adhesives

bonding than the urethane crosslinks of polyol cured isocyanates. Hydrogen bonding would interfere with conformational changes in the polymer chains as segmental rotation between crosslinks. The sample cured at room temperature had a higher modulus at nearly all of the temperatures tested compared to the samples cured at 50° and 70°C. This has been previously observed and indicates the cure mechanism at room temperature is different than that at the elevated temperatures.

The storage shear modulus of the LW520/MDIPA adhesive changes even more slowly with temperature than that of Halthane 87-1. As seen in Figure 20, the storage shear modulus of the 70°C cure

Figure 17. Isothermogravimetric Analyses of Five Adhesives

Table 4. Weight Loss of Five Adhesives After Thermogravimetric Analysis

Cure Temperature (°C)	Weight Loss From 25° to 150°C (Percent)	Additional Weight Loss After Holding at 150°C for 18 Minutes	Total Weight Loss (Percent)
Halthane 73-18			
*	0.22	0.02	0.24
50	0.22	0.03	0.25
70	0.22	0.01	0.23
Halthane 87-1			
*	0.18	0.02	0.20
50	0.32	0.02	0.34
70	0.40	0.01	0.41
LW520/MDIPA			
*	0.20	0.02	0.22
50	0.18	0.02	0.20
70	0.40	0.01	0.41
LW520/ER508/MDA			
*	0.20	0.02	0.22
50	0.22	0.02	0.24
70	0.20	0.02	0.22
ER508/ER505/TEPA			
*	0.22	0.30	0.52
50	0.22	0.30	0.52
70	0.42	0.30	0.72
*Room temperature

Figure 18. Storage Shear Modulus of Halthane 73-18 as a Function of Temperature

Figure 19. Storage Shear Modulus of Halthane 87-1 as a Function of Temperature

Figure 20. Storage Shear Modulus of LW520/MDIPA as a Function of Temperature

specimen is significantly higher than the others from 60° to 140°C. This result correlates with the extraction and swell test data.

The modulus curves in Figure 21 for LW520/ER508/MDA show this adhesive has a high modulus up to 40° to 60°C and then, in the case of the room temperature and 50°C cure specimens, drops sharply to around 3 MPa. The effect of cure temperature on the storage shear modulus of this adhesive is definitely observable. The higher the cure temperature, the higher is the temperature of the viscoelastic transition. These data are in good agreement with the results of the TMA analyses. As seen in the data in the Appendix, the maximum value for the loss tangent occurs at the same temperature as the glass transition temperature. This was generally observed for all the adhesives.

The epoxy adhesive, ER508/ER505/TEPA, given in Figure 22 also shows the effect of an elevated cure. The room temperature cure specimen could not be tested above 70°C because of severe distortion (melting) of the test sample.

ACCOMPLISHMENTS

The chemical, thermal, and dynamic mechanical properties of five adhesives have been determined. The two Halthane adhesives, 73-18 and 87-1, have the best overall chemical, thermal, and dynamic mechanical properties for all three cure conditions. They have a high degree of cure, good thermal stability and show elastomeric behavior in the general temperature range needed. The adhesives, LW520/MDIPA and ER508/ER505/TEPA, require an elevated cure temperature of 70°C to develop a high degree of crosslinking. The adhesives containing epoxy resin, LW520/ER508/MDA and ER508/ER505/TEPA, have high shear moduli at room temperature.

Figure 21. Storage Shear Modulus of LW520/ER508/MDA as a Function of Temperature

Figure 22. Storage Shear Modulus of ER508/ER505/TEPA as a Function of Temperature

APPENDIX

SHEAR MODULUS DATA

Table A-1. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Modulus (MPa)	Shear Modulus (MPa)
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Halthane 73-18, Room Temperature Cure Only

-100	0.0320	840.8	26.9
- 90	0.0349	780.7	27.2
- 80	0.0349	741.4	25.9
- 70	0.0349	723.6	25.3
- 60	0.0757	579.0	43.2
- 50	0.1461	394.0	90.3
- 40	0.3640	101.8	52.2
- 30	0.6329	32.6	20.6
- 20	0.4521	8.6	3.9
- 10	0.1975	4.9	1.0
0	0.1109	4.1	0.5
10	0.0554	3.7	0.2
20	0.0466	3.5	0.2
30	0.0349	3.4	0.1
40	0.0145	4.8	0.07
60	0.0145	4.7	0.07
80	0.0145	4.6	0.07

Halthane 73-18, 48 Hours Room Temperature Cure
Plus 7 Days at 50°C

-120	0.0420	1283	53.9
-110	0.0522	1136	59.2
-100	0.0473	1091	51.6
- 80	0.0424	987	41.8
- 70	0.0691	834	57.6
- 60	0.0936	750	70.2
- 50	0.2964	315	93.4
-	0.5212	88	46.0
- 30	0.5542	13.6	7.5
- 20	0.3282	7.7	2.5
- 10	0.2153	6.5	1.4
0	0.1098	4.7	0.52
10	0.0508	4.3	0.21
20	0.0404	4.1	0.17
30	0.0337	4.1	0.14
40	0.0274	4.0	0.11
50	0.0244	4.0	0.09

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Modulus (MPa)	Shear Modulus (MPa)
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Halthane 73-18, 48 Hours Room Temperature Cure
Plus 7 Days at 50°C

60	0.0176	3.8	0.06
70	0.0214	3.9	0.08
90	0.0238	3.7	0.09
100	0.0346	3.6	0.1
120	0.0296	2.7	0.07

Halthane 73-18, 48 Hours Room Temperature Cure
Plus 7 Days at 70°C

-100	0.0391	857	33.5
- 80	0.0313	780	24.4
- 70	0.0378	712	26.9
- 60	0.0790	596	47.1
- 50	0.2086	391	81.5
- 40	0.5208	102	53.2
- 30	0.6849	21.1	14.4
- 20	0.4569	8.2	3.7
- 10	0.2102	5.1	1.0
0	0.0879	3.7	0.3
10	0.0600	3.5	0.2
20	0.0440	3.3	0.1
40	0.0366	3.2	0.1
60	0.0157	3.0	0.04
80	0.0228	3.4	0.07

Halthane 87-1, Room Temperature Cure Only

-120	0.0407	1552	63.2
-100	0.0292	1378	40.2
- 90	0.1051	1317	138.4
- 80	0.2277	646	147.1
- 70	0.2711	214	58.0
- 60	0.2217	122	27.0

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)
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Halthane 87-1, Room Temperature Cure Only

- 50	0.1733	76.6	13.3
- 40	0.1436	58.0	8.3
- 20	0.1109	39.8	4.4
0	0.0993	28.0	2.8
20	0.0787	23.0	1.8
40	0.0729	19.0	1.4
60	0.0817	16.0	1.3
80	0.1139	11.7	1.3
100	0.1673	7.0	1.2
120	0.2126	4.2	0.9

Halthane 87-1, 48 Hours Room Temperature Cure
Plus 7 Days at 50°C

-120	0.0449	1143	51.5
-100	0.0506	1070	54.1
- 90	0.1313	697	91.5
- 80	0.2725	213	58.0
- 70	0.2878	162	46.6
- 60	0.2139	83	17.8
- 50	0.1933	53.7	10.4
- 40	0.1539	39.4	6.1
- 30	0.1450	33.3	4.8
- 20	0.1249	25.5	3.2
0	0.1197	15.4	1.8
20	0.1114	10.8	1.2
40	0.1164	9.5	1.1
60	0.1079	10.6	1.3
80	0.1106	10.3	1.1
100	0.1524	8.3	1.3
120	0.2122	4.7	1.0

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Modulus (MPa)	Shear Modulus (MPa)
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Halthane 87-1, 48 Hours Room Temperature Cure
Plus 7 Days at 70°C

-120	0.0390	1105	43.0
-100	0.0622	870	55.3
- 90	0.1186	695	82.4
- 80	0.2394	361.7	86.6
- 70	0.2673	103.4	27.6
- 60	0.2410	53.2	12.8
- 50	0.2070	36.3	7.5
- 40	0.1618	26.6	4.3
- 30	0.1294	19.8	2.6
- 20	0.1235	17.0	2.0
- 10	0.1640	15.2	2.4
0	0.1271	12.2	1.5
10	0.1131	10.1	1.1
20	0.1100	9.2	1.0
40	0.1397	7.9	1.0
60	0.1110	7.8	0.9
80	0.1140	8.0	0.9
100	0.1340	7.1	0.9
120	0.1890	4.5	0.8

LW520/MDIPA, Room Temperature Only

-140	0.0626	1473.6	92.25
-140	0.0607	1475.3	89.55
-120	0.0417	1269.8	52.95
-100	0.0460	1102.6	50.72
- 80	0.0698	924.8	64.27
- 70	0.1313	646.9	84.94
- 60	0.1585	390.5	61.89
- 50	0.1481	223.5	33.10
- 40	0.1347	178.6	24.06
- 30	0.1332	126.4	17.03
- 20	0.1350	103.7	14.00
- 10	0.1348	79.6	10.73
+ 0	0.1310	63.9	8.30
+ 10	0.1278	52.0	6.65
+ 20	0.1219	43.4	5.29

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Modulus (MPa)	Shear Modulus (MPa)	Loss Shear Modulus (MPa)
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LW520/MDIPA, Room Temperature Only

30	0.1196	36.7		4.39
40	0.1193	31.7		3.78
50	0.1221	26.2		3.19
60	0.1400	21.5		3.01
80	0.2333	10.5		2.4
100	0.3337	4.4		1.47
120	0.2460	2.8		0.69
140	0.1793	1.7		0.31

LW520/MDIPA, 48 Hours Room Temperature Cure Plus 7 Days at 50°C

-120	0.0356	1248		44.4
-100	0.0458	1020		46.7
- 80	0.0742	810		60.0
- 70	0.1334	564		75.2
- 60	0.1467	373		54.7
- 50	0.1552	214		33.2
- 40	0.1543	172		26.6
- 30	0.1492	129.5		19.3
- 20	0.1501	89.1		14.9
- 10	0.1507	80.9		12.8
0	0.1491	63.7		9.4
10	0.1229	48.1		5.9
20	0.1059	42.7		4.5
40	0.1026	31.6		3.2
60	0.1219	21.8		2.7
80	0.1517	7.7		1.3
100	0.2517	5.3		1.3
120	0.2103	2.8		0.6

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Modulus (MPa)	Shear Modulus (MPa)	Loss Modulus (MPa)
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LW520/MDIPA, 48 Hours Room Temperature Cure Plus
7 Days at 70°C

-140	0.0593	1387.6	82.3
-120	0.0400	1295.3	51.8
-100	0.0416	1116.5	46.4
- 80	0.0642	920.9	59.1
- 70	0.1256	653.1	82.0
- 60	0.1610	427.1	68.8
- 50	0.1735	258.0	44.8
- 40	0.1740	160.9	28.0
- 30	0.1519	119.4	18.1
- 20	0.1475	91.1	13.4
- 10	0.1451	70.4	10.2
- 0	0.1415	53.9	7.6
10	0.1303	44.0	5.7
20	0.1201	36.8	4.4
30	0.1061	31.3	3.3
40	0.0950	28.9	2.7
50	0.0863	25.2	2.2
60	0.0812	23.3	1.9
80	0.0748	19.4	1.4
100	0.0731	15.7	1.1
120	0.0943	11.8	1.1
140	0.1023	5.3	0.5

LW520/ER508/MDA, Room Temperature Cure Only

- 80	0.0533	1207	64.4
- 40	0.0400	998	39.9
- 20	0.0452	866	39.2
0	0.0565	821	46.4
10	0.0655	727	47.6
20	0.0743	654	48.7
30	0.0878	602	52.9
40	0.1257	400	50.3
50	0.3731	138	51.6
60	0.8427	20	16.9

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)
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LW520/ER508/MDA, Room Temperature Cure Only

70	0.7316	13.3	9.7
80	0.3854	2.9	1.1
90	0.2174	2.3	0.5
100	0.1387	2.6	0.4
120	0.0411	2.6	0.1

LW520/ER508/MDA, 48 Hours Room Temperature Cure Plus 7 Days at 50°C

-120	0.0342	1709	58.4
-100	0.0508	1522	77.3
-80	0.0556	1321	73.4
-60	0.0495	1142	56.5
-50	0.0473	1102	52.1
-40	0.0442	1043	46.1
-30	0.0451	947	42.7
-20	0.0478	904	43.2
-10	0.0513	800	41.0
0	0.0541	750	40.6
10	0.0584	698	40.8
20	0.0553	682	37.7
30	0.0612	615	37.6
40	0.0699	500	37.7
50	0.0905	410	37.1
60	0.1285	300	38.6
70	0.3739	96	35.9
80	0.7265	15	10.9
90	0.4914	5.0	2.5
100	0.2309	3.4	0.8
120	0.0379	4.3	0.2
140	0.0058	3.4	0.2

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)
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LW520/ER508/MDA, 48 Hours Room Temperature Cure
Plus 7 Days at 70°C

-120	0.0274	1732	47.5
- 80	0.0609	1361	82.9
- 40	0.0518	978	50.7
- 20	0.0529	838	44.4
- 10	0.0550	728	40.2
0	0.0574	712	40.9
10	0.0509	663	33.8
20	0.0538	606	32.6
30	0.0594	535	31.8
40	0.0630	484	30.6
50	0.0769	409	31.5
60	0.0849	358	30.5
70	0.0937	300	28.1
80	0.1361	224	30.6
100	0.5743	17	9.9
120	0.1330	12	1.6
140	0.0153	11	0.18
160	0.0023	13	0.03

ER508/ER505/TEPA, Room Temperature Cure Only

-140	0.0224	2247	50.3
-120	0.0210	2131	44.8
-100	0.0156	2081	32.5
- 80	0.0253	1780	45.1
- 60	0.0636	1424	90.6
- 55	0.0662	1295	85.8
- 50	0.0713	1191	85.0
- 45	0.0750	1095	82.2
- 40	0.0823	988	81.4
- 35	0.0855	874	74.7
- 30	0.0905	827	74.9
- 20	0.1039	619	64.4
- 10	0.1274	413	52.6
0	0.1388	345	48.1
10	0.1476	289	42.7

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Modulus (MPa)	Shear Modulus (MPa)
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ER508/ER505/TEPA, Room Temperature Cure Only

20	0.1743	206	35.9
30	0.1978	130	25.9
40	0.2179	114	24.0
45	0.2348	115	27.1
50	0.2982	56	16.7
60	0.5615	13	7.3
70	0.5200	5	2.6

ER508/ER505/TEPA, 48 Hours Room Temperature Cure Plus 7 Days at 50°C

-120	0.0185	2456	45.4
-100	0.0254	2370	60.2
- 80	0.0321	2047	65.7
- 60	0.0536	1727	92.6
- 40	0.0720	1228	88.4
- 30	0.0842	1020	86.0
- 20	0.0886	866	76.8
- 10	0.1143	717	82.0
0	0.1148	521	59.8
10	0.1246	401	50.1
20	0.1393	299	41.7
30	0.1598	231	37.0
40	0.1942	143	27.9
50	0.2074	160	33.3
60	0.2413	116	28.2
80	0.3022	31	9.6
100	0.1039	21	2.2

ER508/ER505/TEPA, 48 Hours Room Temperature Cure Plus 7 Days at 70°C

-140	0.0285	2355	67.1
-120	0.0283	2050	58.0
-100	0.0300	1885	56.6

Table A-1 Continued. Shear Modulus Data of Five Adhesives

Temperature (°C)	Loss Tangent	Storage Modulus (MPa)	Shear Modulus (MPa)	Loss Modulus (MPa)
ER508/ER505/TEPA, 48 Hours Room Temperature Cure Plus 7 Days at 70°C				
- 80	0.0356	1729		61.6
- 70	0.0385	1682		64.8
- 60	0.0534	1667		89.0
- 50	0.0663	1463		97.0
- 40	0.0711	1311		93.2
- 30	0.0714	1078		77.0
- 20	0.0801	905		72.5
- 10	0.0898	746		67.0
0	0.0994	621		61.7
10	0.1102	489		53.9
20	0.1177	361		42.5
30	-----	285		----
40	-----	193		----
50	-----	141		----
60	-----	104		----
70	0.2152	58		12.6
80	0.2287	37		8.6
100	0.1785	21		3.8
120	0.0531	15		0.81
140	0.0223	16		0.4
160	0.0181	18		0.3

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BDX-613-1853 (Rev.)

THE CHEMICAL, THERMAL, AND DYNAMIC MECHANICAL
PROPERTIES OF FIVE ADHESIVES, G. K. Baker,
UNCLAS Final August 1978.

The chemical, thermal, and dynamic mechanical
properties of five adhesives have been deter-
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determination of the amount and identity of
the unbound chemicals extracted from the cured
adhesives, the swell ratio, the amount of
residual amino, isocyanate, and epoxy groups,
dynamic thermogravimetric analysis, isothermal
gravimetric analysis, thermal mechanical
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PLASTICS: Adhesives

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Halthane 73-18 and Halthane 87-1, exhibit the best overall properties. They have a high degree of cure, good thermal stability, and elastomeric behavior in the general temperature range required.

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