ARMY RESEARCH LABORATORY

Ambient Temperature Storable Thermoset Resin

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This report studies chemical formulations that have demonstrated the capability of room temperature storability features having a minimum storage period demonstrated as six months. This report introduces resin systems that have met this requirement and have storage histories as long as five years without refrigeration or significant loss of mechanical properties.

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PREFACE

The work reported herein was conducted by Merlin Technologies, Incorporated, at their facilities located in San Carlos, California, under U.S. Army SBIR Contract Phase 1 DAAL04-94-C-4031 (SBIR Topic A93-116, "Ambient Temperature Storable Thermoset Resin"). The Principal Investigator was Clayton May. Invaluable support was provided by Arroyo Research and Consulting Corporation based on previous work in this area.

Dr. Seth Ghiorse of the U.S. Army Research Laboratory served as the Army Technical Monitor. The authors of this report would also like to acknowledge the assistance of Drs. Margaret Roylance and Walter Zukas of the U.S. Army Research Laboratory whose technical discussions and insights were a valuable contribution to the success of the program.

AMBIENT TEMPERATURE STORABLE THERMOSET RESIN

1.0 BACKGROUND

Current logic dictates that the best approach toward lower cost composite structures lies in simpler processing. Raw material prices, with the possible exceptions of some carbon fiber types and a few specialty matrix resins, will most probably remain constant or become only marginally less expensive. Market volumes are too low. On the other hand, changes in process technology offer two reasonable approaches to lower cost hardware. These are the focal points of this study.

The first approach is to eliminate the need for refrigerated storage of prepreg materials. Not only is the refrigerated storage itself an unwanted cost burden, but there are also cost problems associated with allowable inventory size and the clerical needs required to keep track of total out time for each specific lot or roll of material. This fact has been recognized and is the subject of a current SBIR contract.[1]

The second approach is to eliminate or substantially reduce autoclave processing times. There are numerous costs associated with autoclaving, the stand-by operator labor charges, the gas required (nitrogen) to provide inert internal pressurization, expensive vacuum bagging materials, the need (in most states) to have a standby boiler engineer, and the amortization of an expensive piece of equipment, to mention a few. A suggested solution to this latter cost was cited and demonstrated in a recent effort on Shell's HPT® matrix resins.[2] This effort also led to a concept for room temperature storable matrix resins and prepregs as discussed herein.

The solution to both of these problems can best be understood through comprehension of the 3T chart. This concept was first proposed by Professor John Gillham of Princeton University[3] and affords a graphic picture of a thermoset matrix throughout its heat history, during storage, during processing and in the final cured state.

A schematic representation is shown in Figure 1. This plot of temperature versus time shows that there are four major temperature dependent physical states in the life of a thermoset resin pertinent to this discussion; an ungelled glassy state, a liquid (processing) state prior to gellation, a rubbery gel region where the material has attained an infinite molecular weight, and a gelled glassy state wherein the resin becomes totally reacted chemically and attains its ultimate properties. There is a fifth material state, degradation, where a materials useful temperature range has been exceeded, but this is not of great importance for this discussion.

The 3T chart leads to a clear understanding of the various facets of thermoset resin chemistry vital to this program. Of prime importance is an understanding of how a thermoset resin behaves chemically in the glassy state. Thermoset resin systems consist of two types of materials, the resin itself and a curing agent or catalyst which converts the resin into an insoluble, three dimensional structure imparting elevated temperature performance, "toughness", chemical resistance, etc. Theoretically, when this mixture is an organic glass, its high modulus restricts the motion of the molecules therein, thereby stopping the curing reactions. However, it has been observed by a number of investigators that the cure chemistry actually continues in the glassy state 10-50 degrees Centigrade below the glass transition temperature (Tg). The magnitude of this difference and the degree of "inertness" is, of course, dependent on the chemical and physical nature of the materials involved.

Herein lies the uniqueness of this concept. As long as an ungelled composite matrix is substantially below its Tg (lower regions of the 3T chart), an uncured prepreg matrix should have an infinite shelf life. Currently available prepreg materials are normally supplied in a tacky, semi-liquid form and thus must be refrigerated during storage to artificially lower the Tg. By using prepregs wherein the matrix resin is substantially below the Tg, refrigeration is no longer prerequisite. Considering current fabrication technology, which includes heated layup tables, heated tape laying machines, etc., this should pose only minor problems to the hardware fabricator. It is more a matter of properly training the shop personnel. Thus, given admixtures of starting materials with sufficiently high Tg's, a variety of room temperature storable prepregs are available. The other approach is, of course, using a curing agent which forms an insoluble dispersion in the matrix resin. This is old technology widely used by the electrical prepreg industry, i.e. a dispersion of dicyanamide in a glassy matrix resin, a solid grade of a conventional epoxy resin. Since this involves a single, chemically specific curing agent, the range of affordable properties in the final lamination is obviously restricted.

However, there is more to the story. Further cost savings can be afforded the lamination processes by taking advantage of the ability of matrix resins to cure in the glassy state. Although processing rates undoubtedly depend upon the chemical nature of the materials involved (resins and current agents), our work has shown that the chemical reactions of the cure can occur quite rapidly in the glass state, providing the matrix is kept close to its ever increasing Tg.

Now let's see what this means in terms of the 3T chart (Figure 1) which is a modification of Gillham's original hypothesis. The lower part of the chart, ambient or below, is the stable storage region of the matrix material (a key goal in this study). As a resin system is heated from the glassy to the liquid state, it begins to react at an ever increasing rate. Concurrently, the viscosity of the matrix becomes increasingly fluid. This is the region where consolidation of the matrix and its reinforcement is normally accomplished. The duration of this part of a materials heat history should be minimized since this is the most expensive part of the fabrication process, i.e. autoclave and tooling usage, vacuum bagging, etc. It should also be noted that by keeping temperatures at a minimum during this period, less expensive bagging and tooling materials can be used. Once the partially cured material is recooled to the glassy state, bagging and tooling can be removed from the part. It should also be noted that at this point in the process, a properly designed matrix could still be an ungelled glass which would permit reworkability if needed. For optimal hardware properties, an understanding of the matrix rheology in this region is also prerequisite.

The remainder of the cure can be accomplished in a free standing glassy state by following a heating cycle such as that shown in Figure 1. Properly designed hardware should not warp under these conditions. In the glassy state, close to the Tg, the bulk modulus of the material is sufficiently high to resist warpage. The only stress on the part is the weight of the material itself. Stresses in composite hardware result only from differences in the thermal expansion coefficients of the matrix in the glassy state and the reinforcement as the final product is cooled down from the curing temperature. The magnitude of these forces is a factor of only about 10% of the modulus of the final hardware.

Summarily, evidence to date states that room temperature storable prepregs can be designed which can be formed into hardware using processing technology much simpler and less costly than the materials currently employed. Both the experimental and theoretical evidence as presented herein confirm this hypothesis.

2.0 EXPERIMENTAL RESULTS

2.1 INTRODUCTION

The original thrust of this investigation, as put forth in U.S. Army SBIR A93-116, was to develop a thermoset prepreg matrix resin for fiber reinforced composite structures which could be stored in normal manufacturing facility climates without the need for refrigeration. Our approach was to use commercially available epoxy resins and curing agents so that small businesses could readily avail themselves of these starting "formulations" and move quickly to the marketplace with a minimal effort. As our investigation progressed it not only became apparent that the storability objective could be readily achieved, but that with further investigation additional cost savings in the composite fabrication could be affected such as reduced autoclave usage, less expensive tooling, reduced labor charges, etc. A further outgrowth of the investigation dictates that the principles espoused herein could be fruitfully employed in other thermoset resin processes such as filament winding, RIM, RTM, adhesive bonding, repair resins, towpregs, tape winding and laying and the like.

It was thus readily apparent that all the goals set forth in the original Technology Proposal (Section 1.0) had been accomplished. At least five of the material systems investigated readily meet these objectives and several of the other formulations, with additional study, may also qualify.

2.2 MATERIALS SELECTION AND FORMULATION

All of the resins and curing agents used in this program are commercially available. They are listed by trade name and source in Table 1. Copies of the vendors' data sheets can be found in the Appendix. The chemical structures of the resins used in this investigation are shown in Figure 2 with the exception of EPON 1050® and ECN 1273® which are epoxy cresol novolacs which have the same general structures as DPS-

164®, but manufactured using cresol rather than phenol to form the precursor novolac resin. The chemical structures of the curing agents are shown in Figure 3.

The matrix candidates were formulated by combining each of the resins listed in Table 1 with each of the individual curing agents giving the 32 candidate matrices shown in Figure 4. The resin/curing agent mixtures were made by heating the resin indirectly (oil bath) to 100-125 degrees Centigrade and mixing in the curing agent while continuing the heating until a clear solution resulted. Once a clear solution was achieved, the mixture was cooled rapidly to room temperature.

Extreme care should be taken in any attempts to duplicate this process with these or any other experimental epoxy formulation. When heating the resin, curing agent combinations can become chemically quite reactive. Overheating can result in an uncontrollable exothermic reaction. This could cause loss of the material, fires, the generation of toxic fumes and possibly personal injury. In the laboratory, small lots of these materials can be readily handled in the aforementioned fashion. However, initial experimentation should be restricted to *small quantities* (10-25 grams). Indirect heating should always be used such as that supplied by an oil bath or other techniques which *avoid hot spots* in the mixing vessel.

On a larger production scale these problems can be avoided by the use of proper equipment. Since, by definition, all of the resins must be glassy and the curing agents are usually powdery solids, it is visualized that the initial mixing be accomplished by dry powder blending. Final resin, curing agent solution can then be accomplished by running the dry powder through a small extruder at a predetermined melt temperature. This should also permit a rapid process for manufacturing towpregs. The fibers could be introduced into the resin stream as it exits the extruder. The exit orifice size would be used to control the resin fiber ratio.

In our small scale preparations, as soon as each of the resin curing agent combinations was prepared, it was placed in a freezer at minus five degrees Centigrade to assure its stability prior to testing. Portions of the individual formulations were then exposed to ambient temperatures and tested for their residual reactivity at various time intervals.

Figure 4 also describes the physical appearance of the resin systems immediately after affecting solution of the curing agents. Some of the samples developed a hazy appearance on cooling to room temperature, indicating that the curing agent formed microcrystals within the resin. It should be noted, however, that most of these hazy mixtures continued to react, evidence that at least some of the curing agent stayed in solution. It was also found, not unexpectedly, that the materials described as viscous liquids or tacky solids polymerized further to non-tacky resins. This would be expected of any resin not in the glassy state. Whether or not these latter systems would be useful as room temperature stable matrices is a subject for further investigation. Samples of the non-gelled materials would have been retained after the six months' room temperature storage period for further testing.

2.3 ROOM TEMPERATURE STABILITY TESTS

The primary test used for room temperature storage stability was resin gel time at 350 degrees Fahrenheit. A small sample of the resin was placed on a smooth hot plate controlled at 350 +/- 5 degrees Fahrenheit and constantly tested for its flow characteristics by stroking the melted resin puddle until no flow (i.e. a gel) was observed. The time interval between initial placement of the sample on the hot plate and the observation of the gel point was measured with a stopwatch and reported in seconds. We found that small, round toothpicks were ideal for this purpose, disposable and inexpensive. Gel times were run after 0, 1, 3, 7, 14, 30, 60, 90, and 180 days' aging at room temperature as shown in Table 2.

It should be noted that geltime testing is a somewhat subjective procedure and will vary from operator to operator. We avoided this pitfall by having the same person run all of the gel times. Careful examination of Table 2 will also show that, even with the same operator, variances were observed. Nonetheless, the method provided a facile tool to measure room temperature storage stability. Since only six months were allowed for a program measuring six months' storage life and time was required to order materials and prepare the matrix resins, this technique was a valuable asset toward completion of the program with minimal tardiness.

As can be seen in Table 2, quite a number of the materials had at least six months' shelf life at room temperature. Four resin/curing agent combinations, EPON HPT 1079 with EPON Curing Agent HPT 1061, and EPON HPT 1071, 1079 and DPS 164 with DADS were among the more stable combinations based on these data. The data also shows that other resin/curing agent combinations have not gelled after six months at room temperature. The eventual utility of any of these "acceptable" systems will depend on whether or not the material has reached a stable state (gel time or % reacted) during the six month storage period. This is important to obtaining products with consistent processing characteristics on the manufacturing floor. Samples of all of these combinations have been retained and can be further evaluated after longer storage periods as the program continues.

It appears that a number of materials can meet the initial program criteria. If these prove stable, and further chemical reaction ceases during room temperature storage, this initial "screening" program will provide a broader than expected formulative base for further consideration. It is visualized that the various resin/curing agent combinations could be prereacted during the initial mixing to the point where a stable organic glass results. However, rheological testing and prepregging trials will also be required to see which of these prereacted matrices can be converted to useful laminated structures.

In addition to the gel time testing, differential scanning calorimetry was used to obtain a more quantitative view of the room temperature aging characteristics. The areas under the exotherm curves were measured before and after three and six months' aging for the ungelled combinations. The actual thermograms are shown in Figures 5-52. Analysis of the data in terms of residual heat of reaction (cals./g.) and percent reacted during the aging are summarized in Table 3. The HPT 1079/1061 resin system, which was the focal point of our earlier work, had reacted to the extent of 11.3 percent after three months. Interestingly, three other resin systems, 1079/DADS, 1071/DADS, and DPS 164/DADS, appear to be even more stable. Although more accurate, the DSC data also shows some inconsistencies, probably due to instrument to instrument variations and the accuracy of weighing very small samples.

2.4 CONCLUSIONS

There is little question that the primary objective of Phase I, "a room temperature storable thermoset resin", has been achieved. A number of the epoxy resin/curing agent combinations evaluated with this objective in mind appear to meet the program goal. As a result, the study now takes on added significance in that a formulary of resins and curing agents now appear to be available, not only providing the requisite room temperature storability, but also giving the prepreg supplier an opportunity to vary the properties of the resultant hardware.

Our initial hypothesis was that the resin in admixture with the curing agent should be deep in the glassy state at room temperature (well below the Tg). The chemical reactions of the cure could thus not transpire and the matrix would last indefinitely until heated. This is sound reasoning and the data bears out this conclusion. However, there is more to the story and a successful commercialization will require additional study and understanding.

The first question which comes to mind is the stability of a given system with regard to its processability after aging at room temperature. Will a fresh prepreg and one that has been stored at room temperature for six months give laminates with the same mechanical properties? Will the processing have to be varied to achieve this goal? If the latter proves true, can the prepreg be prereacted to the degree that processing will be identical regardless of the aging period? These questions can be answered only through a study more thorough than permitted under the current budget restraints. The importance of stable rheological properties as a matrix ages under ambient conditions thus also becomes important.

It was also noticed during our initial investigation that, in some of the DADS formulations, on cooling after dissolving the curing agent in the resin, the curing agent, at least in part, recrystallized from the resin, giving a hazy appearance to the matrix. For example, considering the DPS-164/DADS system, it suggests that a combination of a high matrix Tg with poor room temperature solubility could lead to even greater storage stability. Further knowledge of raw material solubility parameters would thus appear in order.

Based on a combination of the DSC data and the gel time tests, the resin systems which appear to be of the greatest interest for further study are 1071/DADS, 1079/DADS, DPS 164/DADS, 1071/1062 and 1079/1062.

Another possible area of exploration is the use of ETHACURE 100 type curing agents with a controlled prereaction of the components. As shown in the manufacturers' data sheet this is an 80:20 mixture of two diethyl m-phenylenediamine isomers. When used as a curing agent for HPT 1079, EPON 1031, DPS 164, ECN 1273, and possibly TACTIX 742, the extent of reaction after 6 months is 10-20%, indicating that one of the four amino groups is perhaps more reactive than the other three. The other amino groups may be sufficiently blocked by the ethyl groups to greatly retard further reaction in the glassy matrix resin.

Summarily, a successful small business venture at this stage of the investigation depends on the Tg of the matrix, the solubility parameters of the formulative components, and the rheological stability of the matrix.

3.0 FUTURE RECOMMENDATIONS

Our initial thinking on this program was that the Phase II effort would be to select two or three established small business prepreggers, provide data on two or three of the most promising resin systems, provide seed funding and work with them to develop room temperature storable prepregs. However, as noted in our conclusions, a successful development is probably a bit more complex. Other factors such as laminate properties, material solubility parameters and system rheology may all be prerequisite to a successful program. It is thus suggested that the Phase II effort entail working with one small business prepregger, become intimately involved in his developmental activities, and devote any remaining funding toward other new applications of the glassy state storage concept. In the latter area we feel that a room temperature stable adhesive tape, towpreg filament winding concepts and RIM/RTM applications are among the more important.

4.0 REFERENCES

1. U.S. Army SBIR Contract Phase I DAAL04-94-C-4031, SBIR Topic A93-116, "Ambient Temperature Storable Thermoset Resin".
2. W. B. Breightigam, R. S. Bauer, and C. A. May, CHEMTECH 23 (1), 38-42 (1993).
3. J. K. Gillham, Society of Plastics Engineers, Annual Technical Conference (1980), Proceedings, p. 268.

APPENDIX

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List of Figures.

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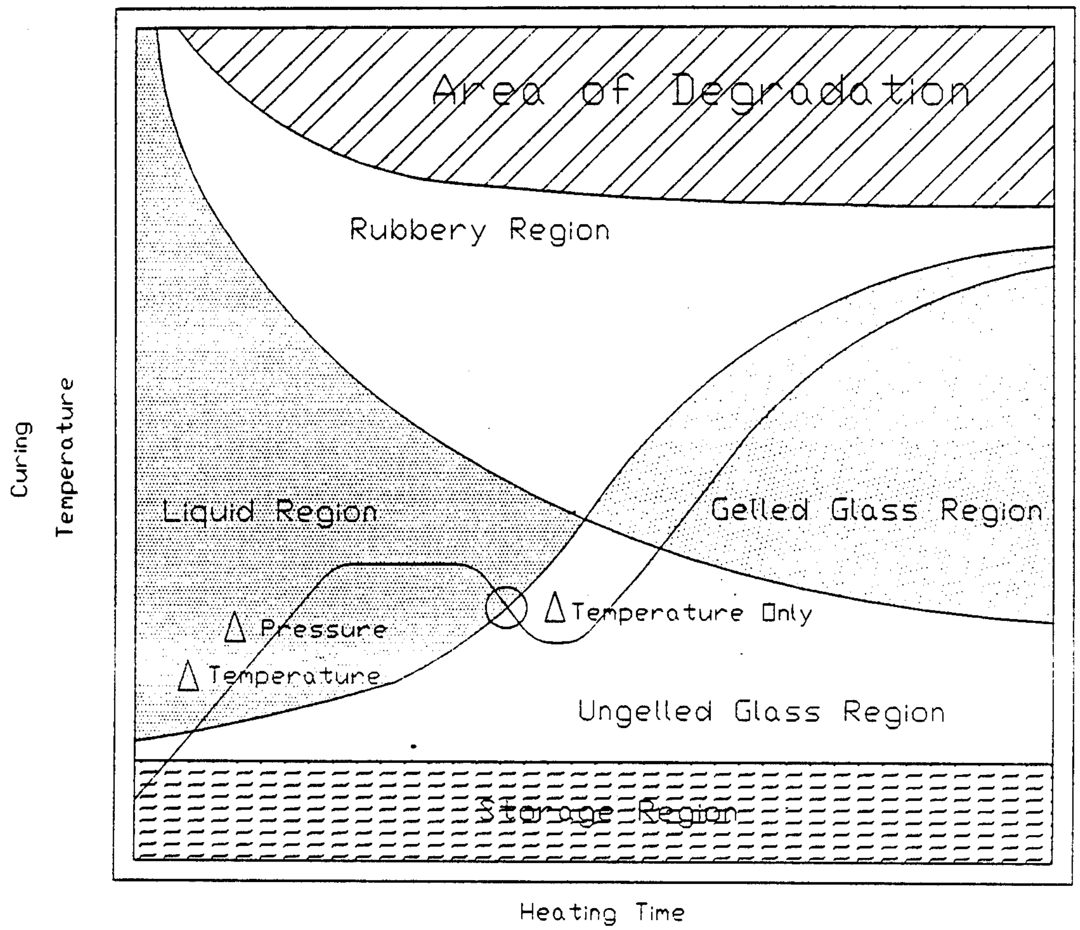
List of Tables.

Manufacturers' Data Sheets.

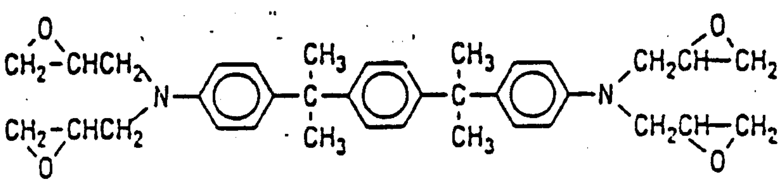


Time-Temperature-Transformation (TTT) Diagram

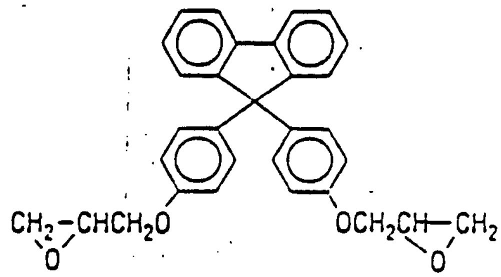
Figure 1.

Figure 2. Structural Formulae of Resins

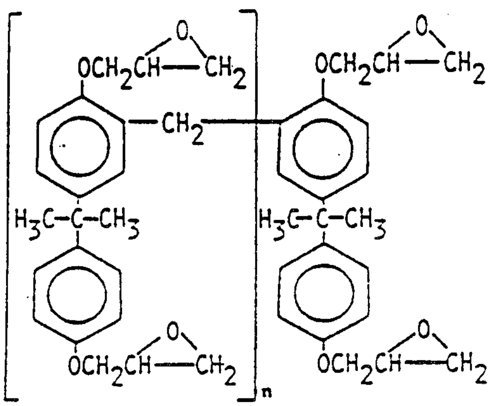
EPON HPT 1071



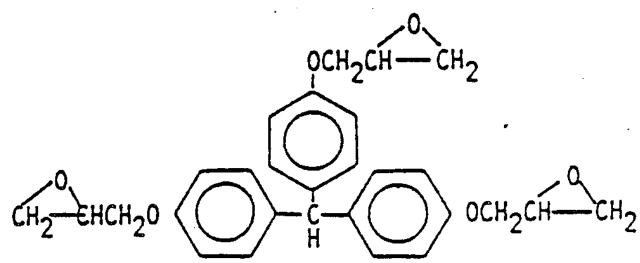
EPON HPT1079



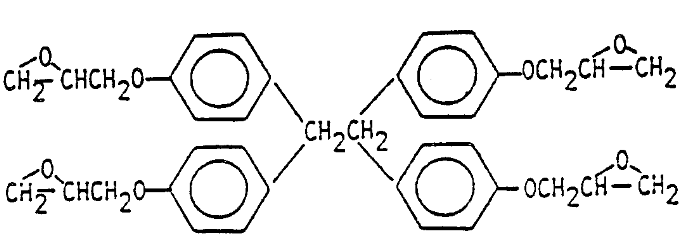
SHELL BPA NOVOLAC SU-8



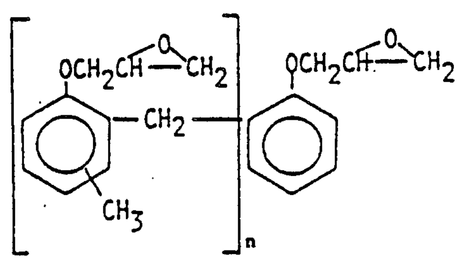
DOW TACTIX 742



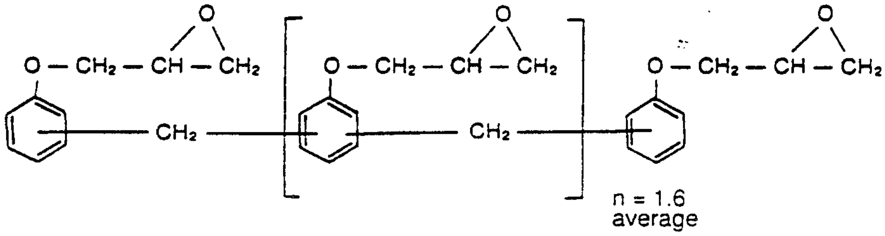
EPON 1031



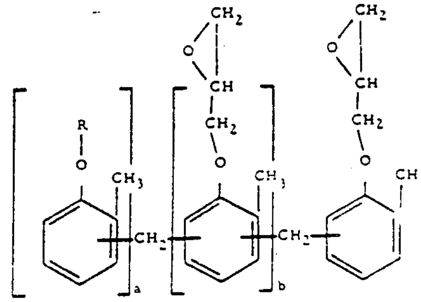
SHELL CRESOL NOVOLAC DPS-164



EPON HPT 1050



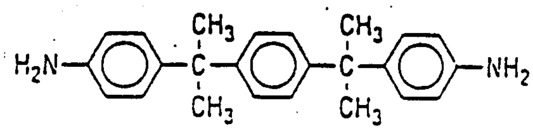
CIBA ECN 1273



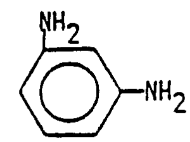
Note: R represents chlorohydrins, glycols, and/or polymeric ethers.

Figure 3. Structural Formulae of Curing Agents

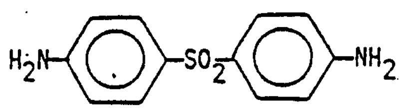
EPON HPT 1061



METAPHENYLENEDIAMINE (MPDA)



DIAMINODIPHHENYL SULFONE (DADS)



ETHACURE 100

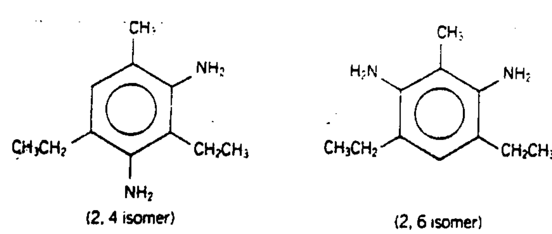


Figure 4. Candidate Epoxy Matrices and Their Physical Appearance on Mixing*.

Curing Agents	HPT 1061	MPDA	DADS	ETHACURE 100
Resins				
HPT 1071	C	CL	HT	CL
HPT 1079	C	C	C	CT
EPON SU-8	C	GEL	HT	CT
TACTIX 742	C	CL	H	CL
EPON 1031	C	C	C	T
DPS 16	C	CT	H	CT
EPON 1050	CT	CL	HT	CL
ECN 1273	C	CL	H	CT

* Physical appearance of matrix resins after mixing:

C - Clear resinous mixture
L - Clear viscous liquid
H - Hazy resin
T - Tacky solid.

Figure 5.

HPT RESIN 1071/HPT CURING AGENT 1061 BEFORE AGING.

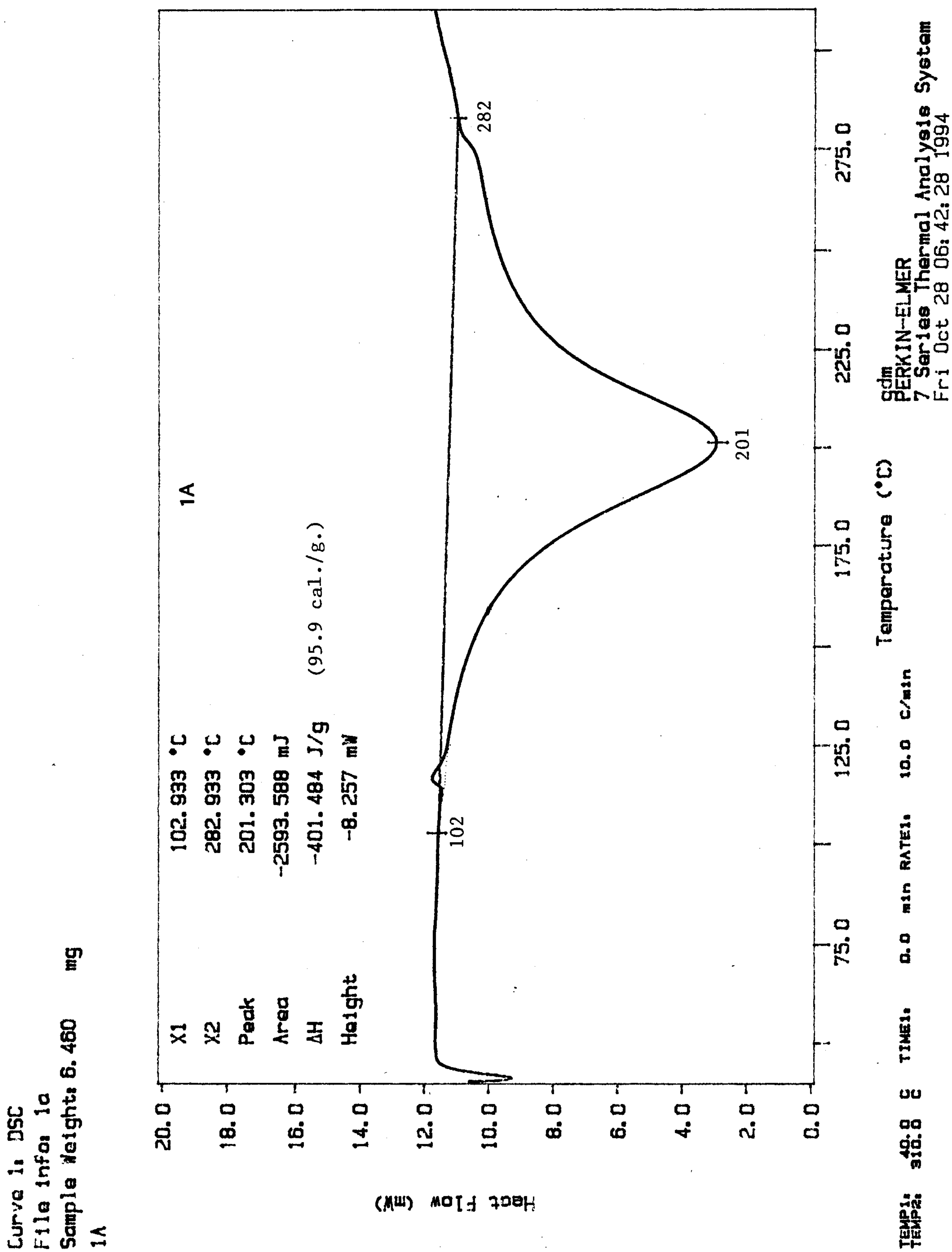


Figure 6.

SYSTEM FROM FIGURE 5 AFTER 3 MONTHS' ROOM TEMPERATURE STORAGE.

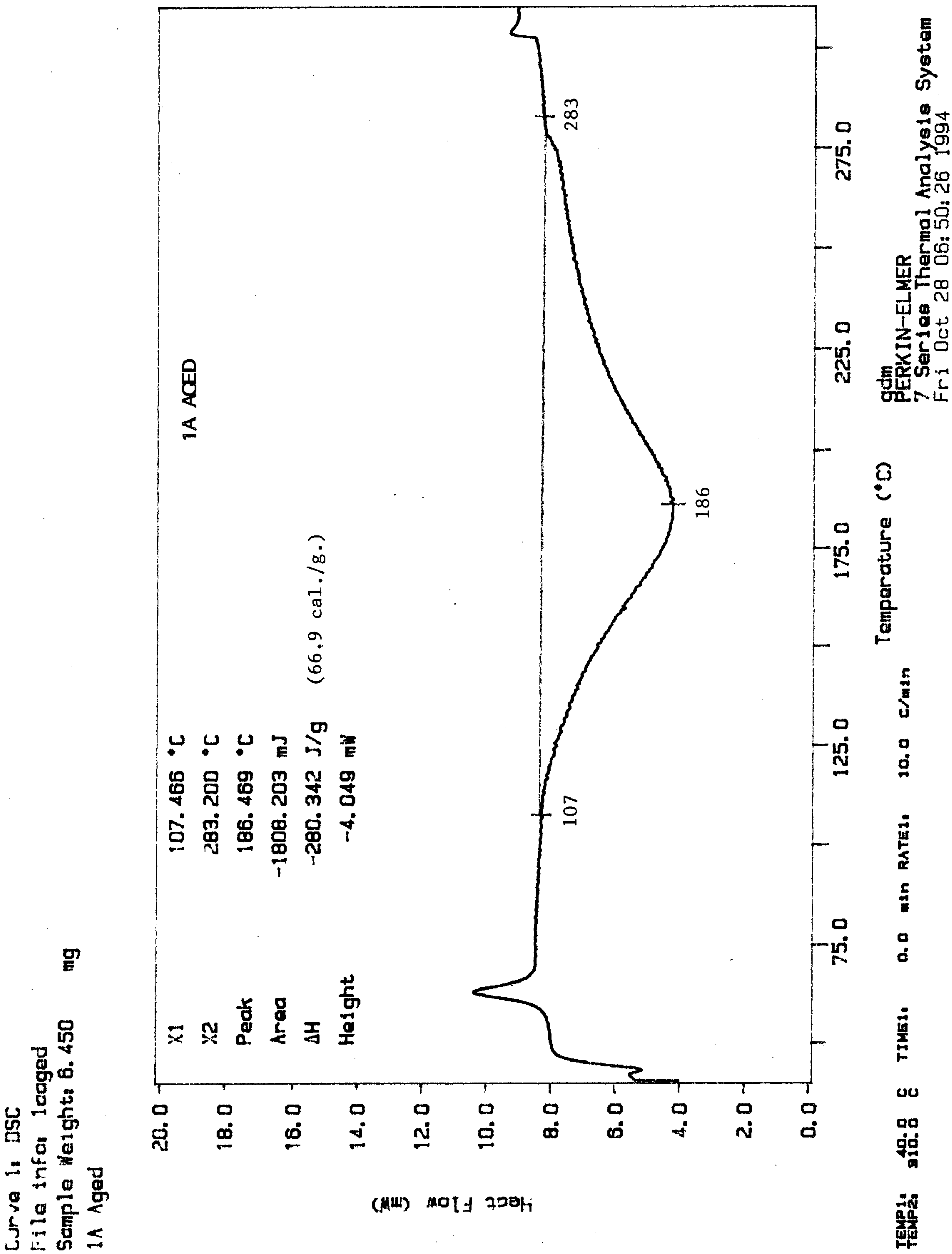


Figure 7.

SYSTEM FROM FIGURE 5 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

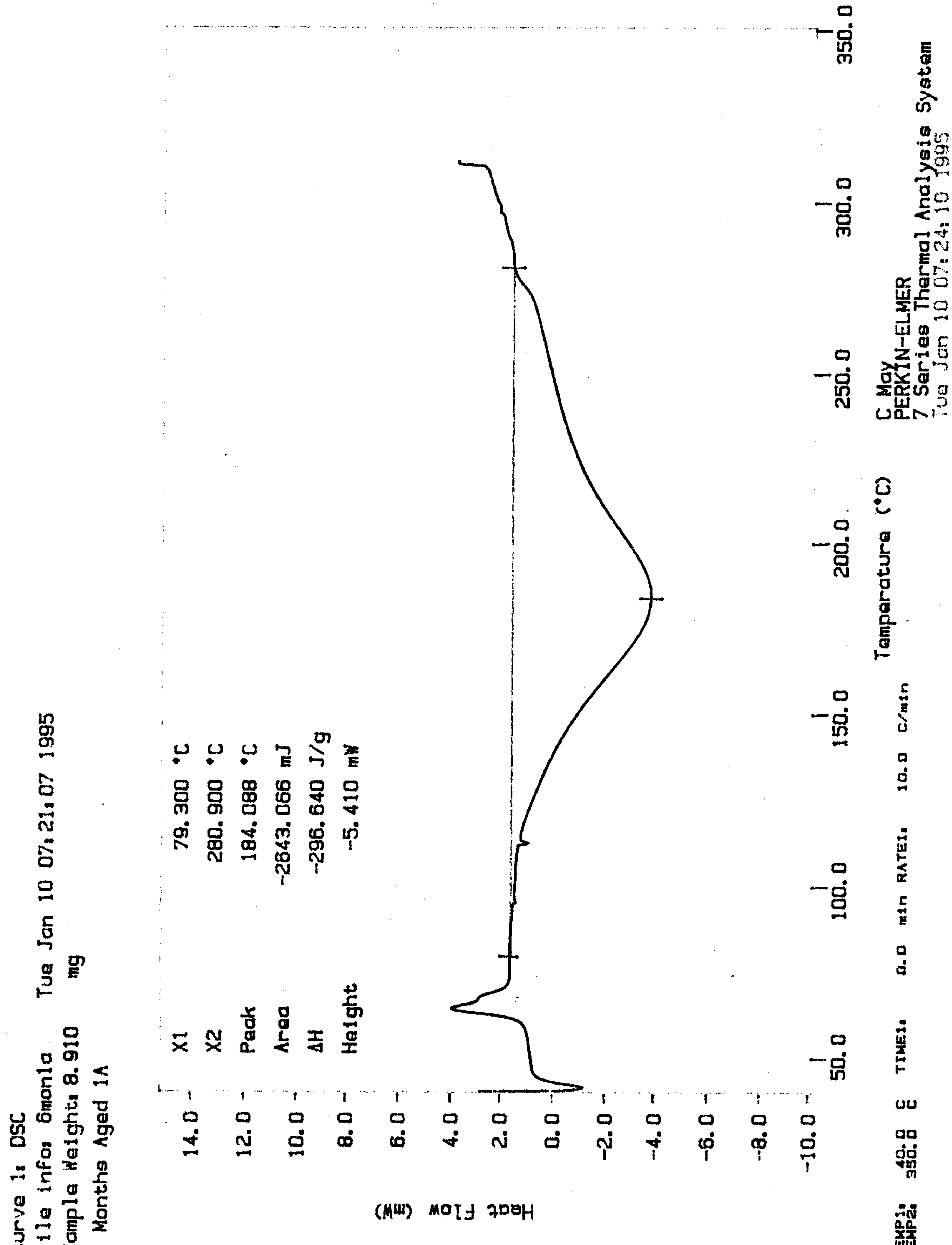


Figure 8.

HPT RESIN 1079/CURING AGENT 1061 BEFORE AGING.

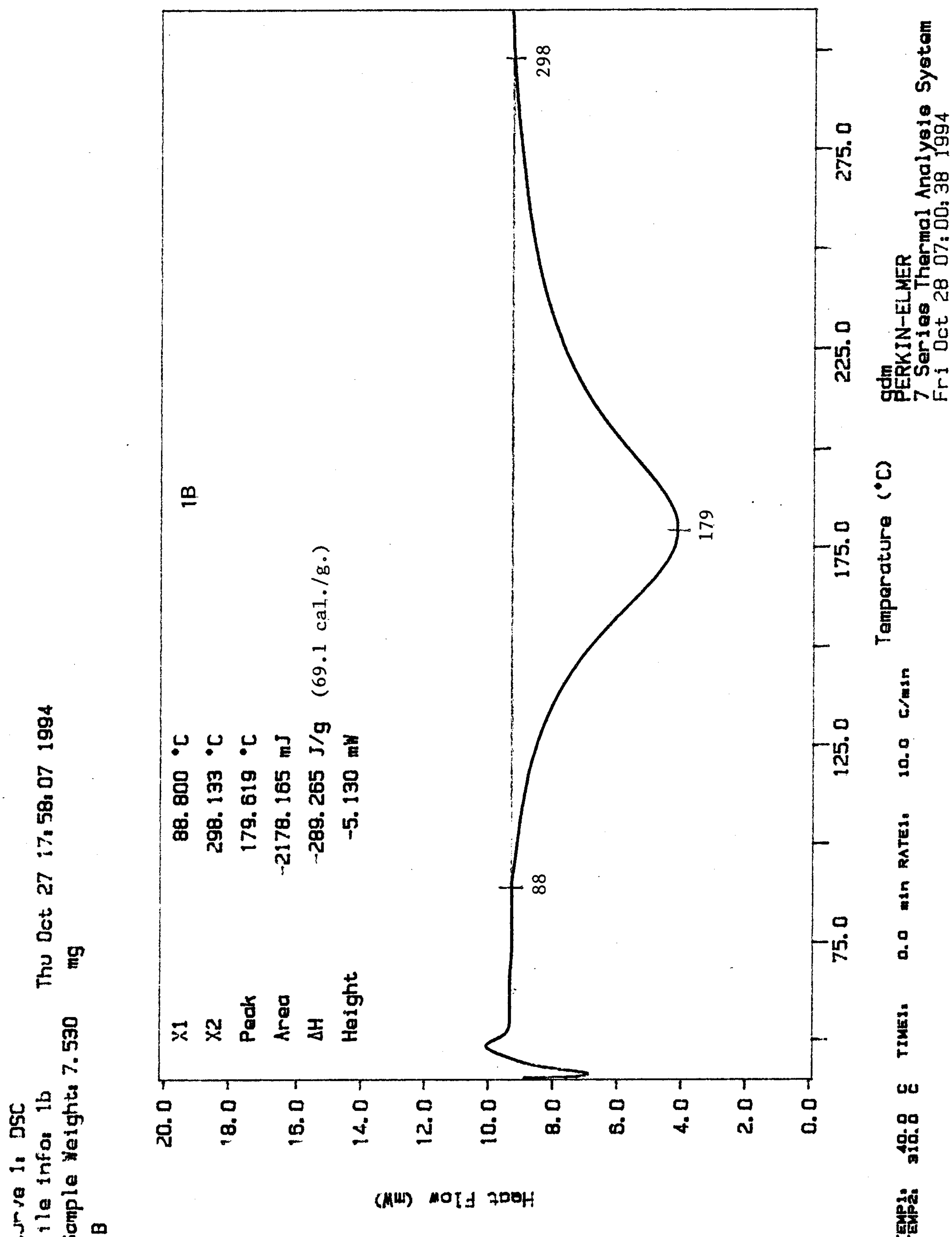


Figure 9

SYSTEM FROM FIGURE 8 AFTER 3 MONTHS' ROOM TEMPERATURE STORAGE.

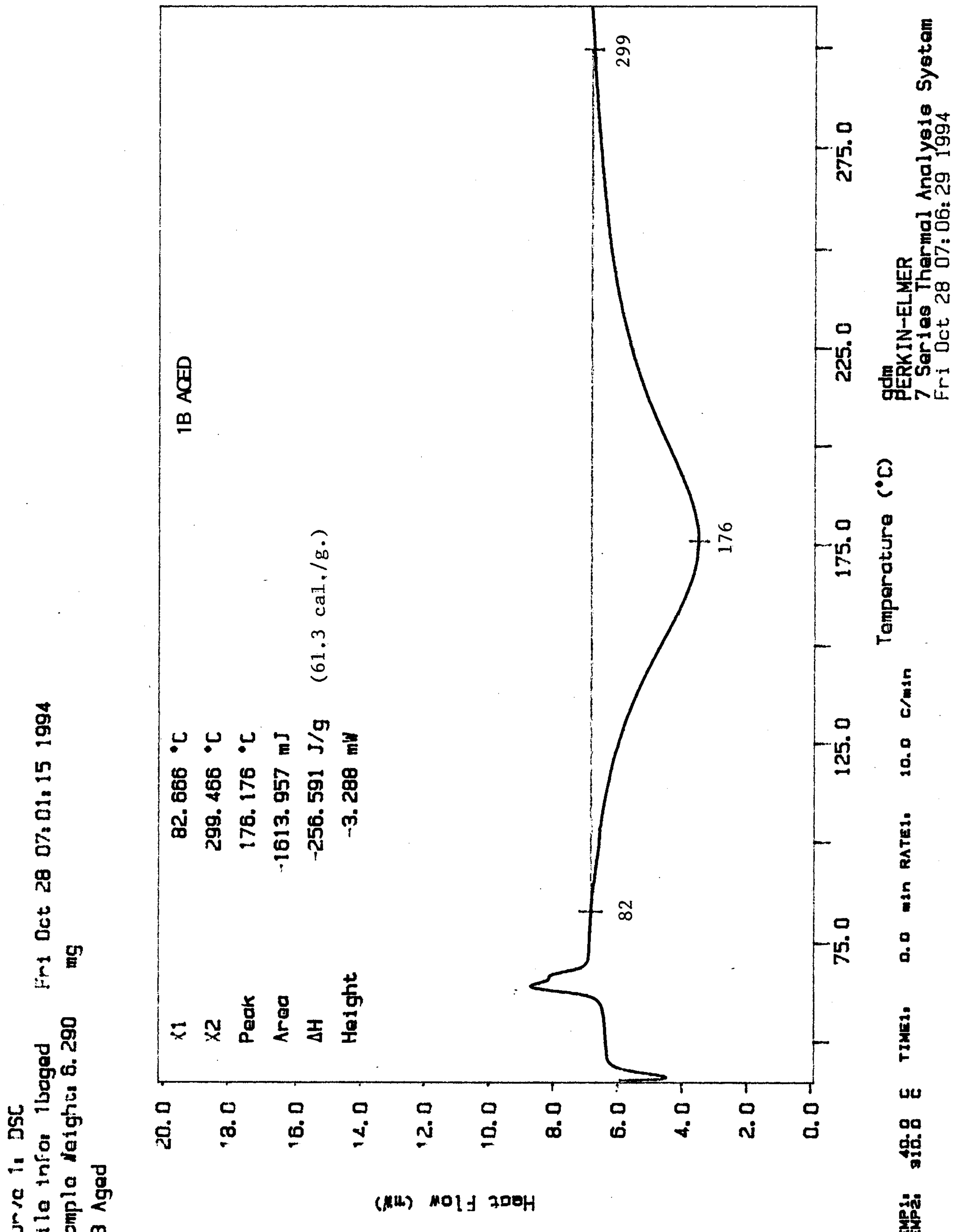


Figure 10.

SYSTEM FROM FIGURE 7 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE

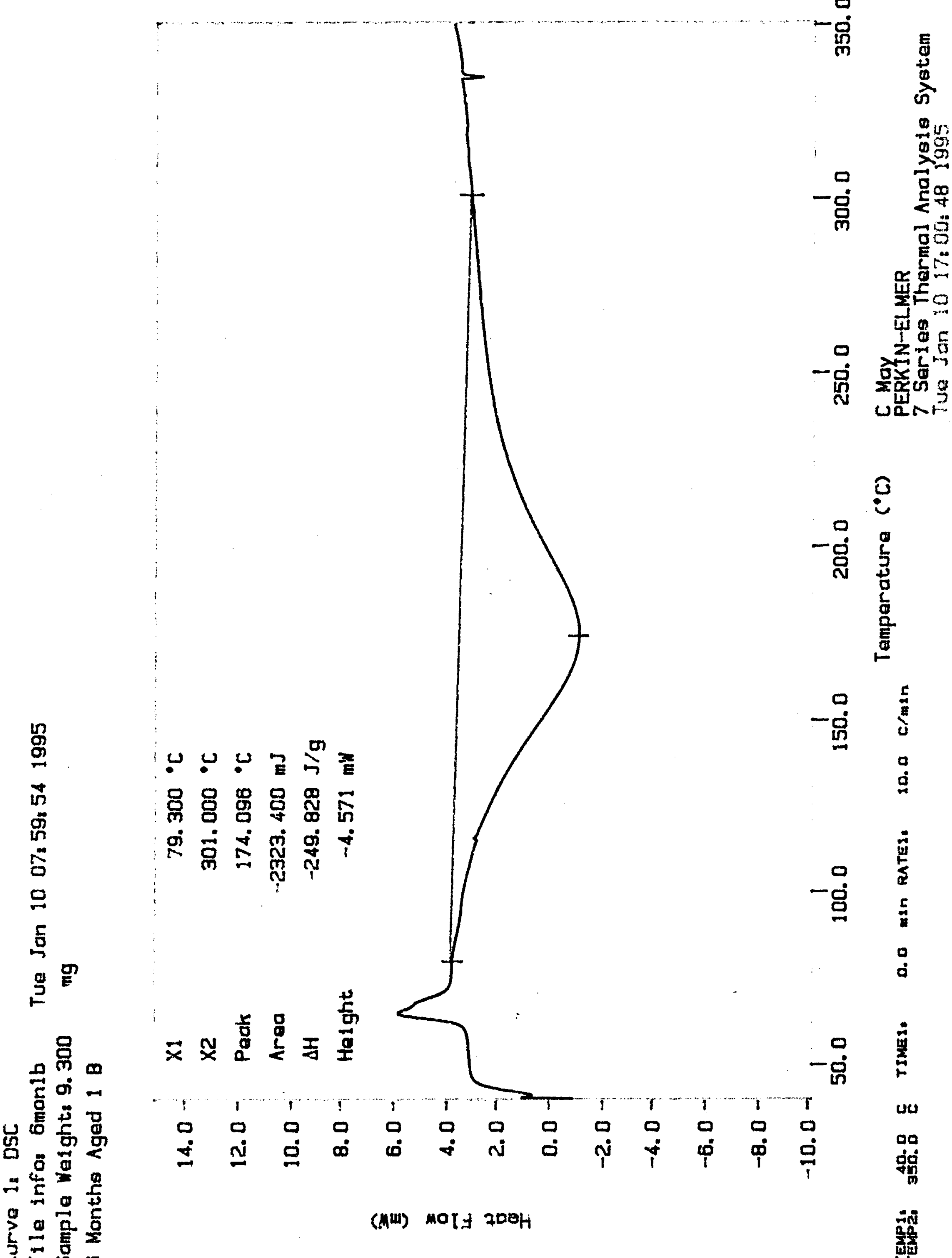


Figure 11.

TACTIX 742/HPT CURING AGENT 1061 BEFORE AGING.

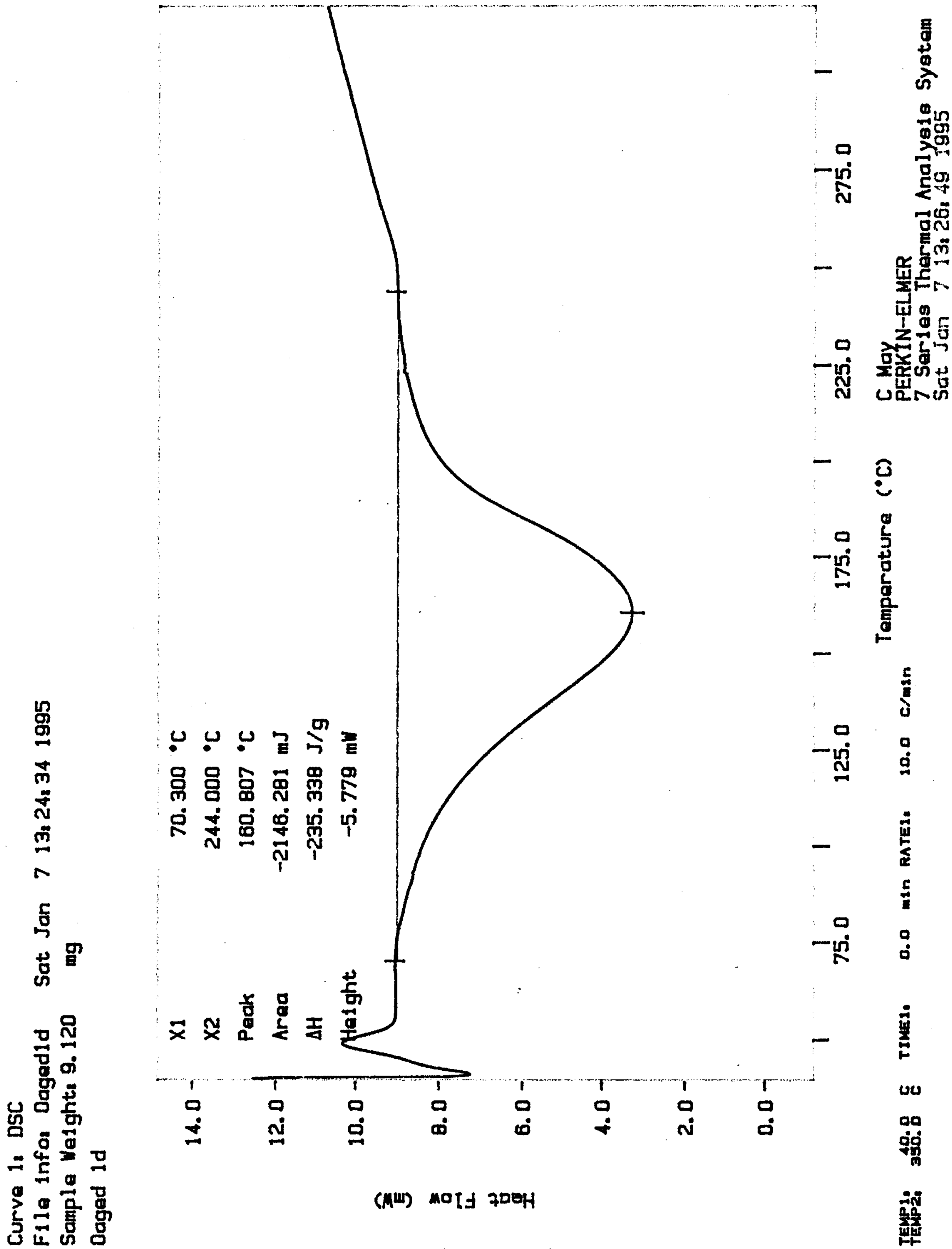


FIGURE 12.

SYSTEM FROM FIGURE 11 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

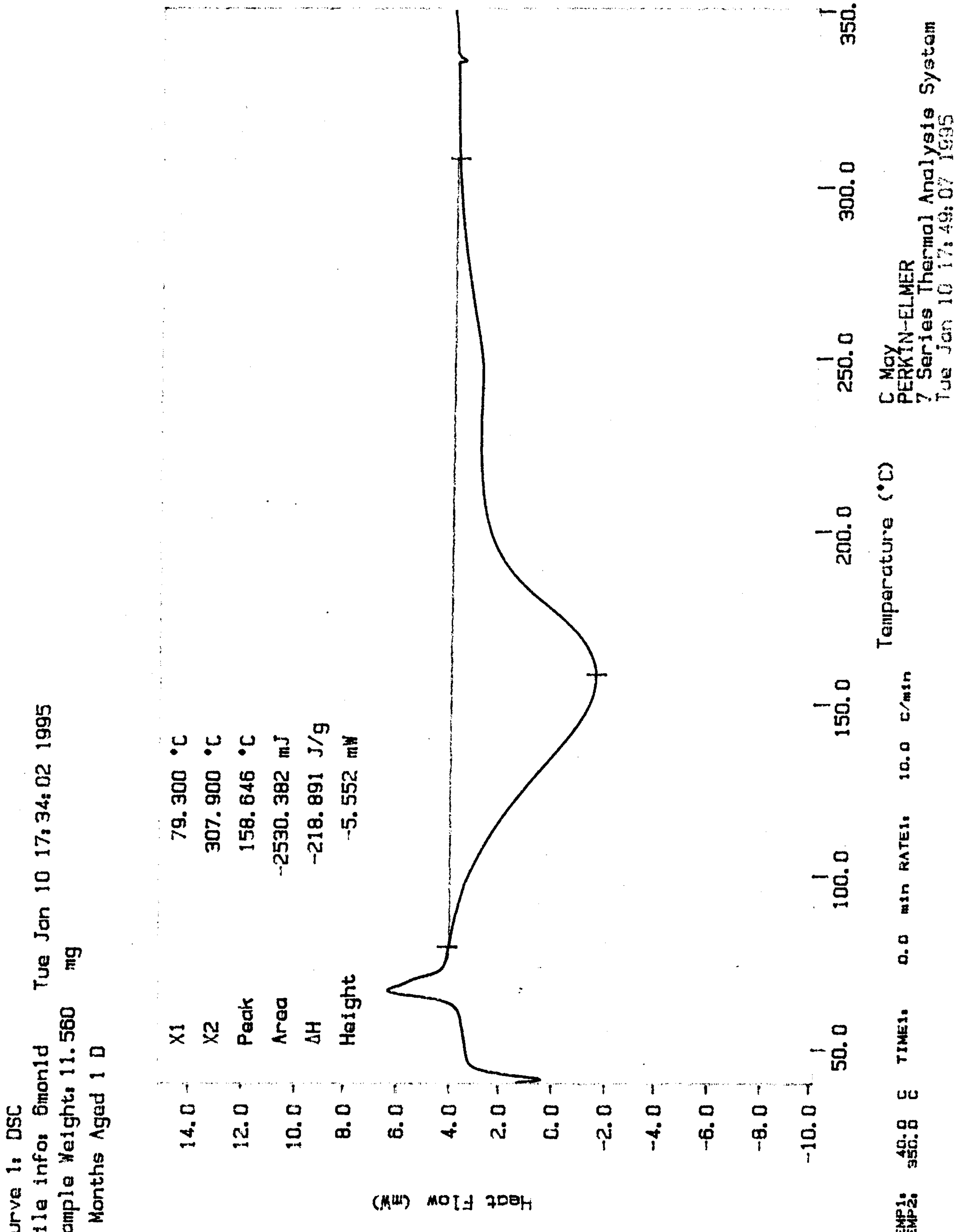


FIGURE 13.

EPON 1031/HPT CURING AGENT 1061 BEFORE AGING.

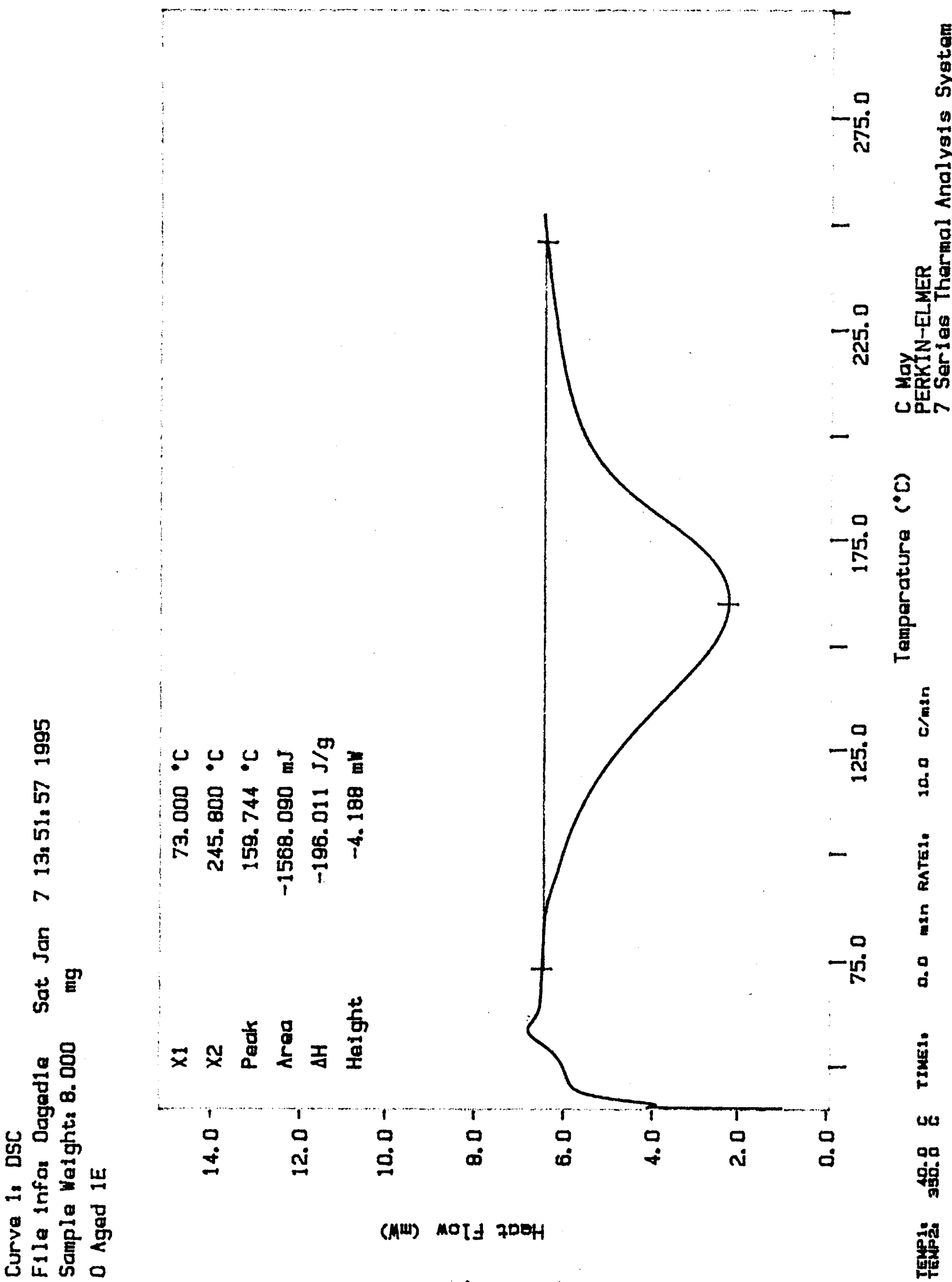


FIGURE 14.

SYSTEM FROM FIGURE 13 AFTER 6 MONTHS ROOM TEMPERATURE STORAGE.

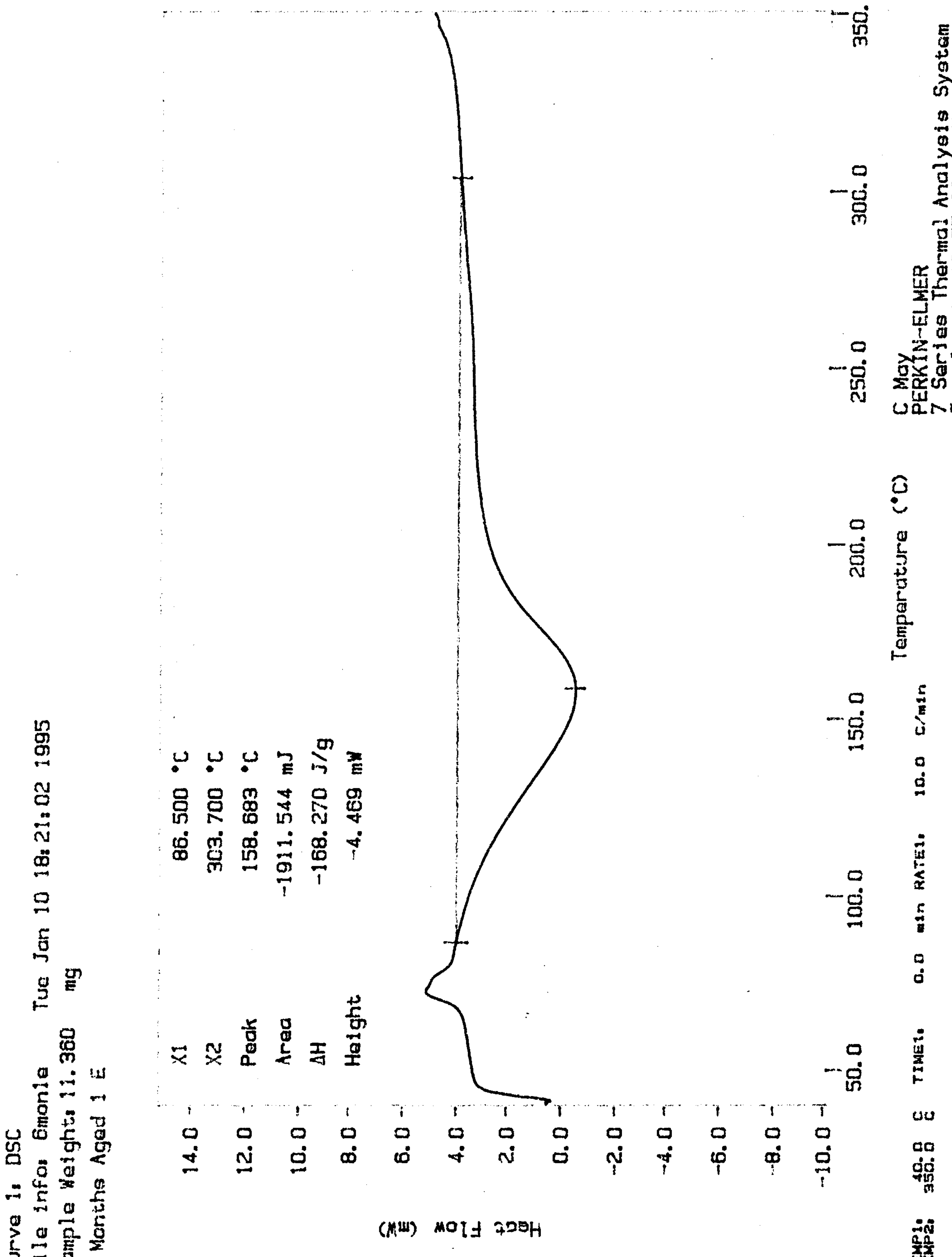


Figure 15.

DPS 164/HPT CURING AGENT 1061 BEFORE AGING.

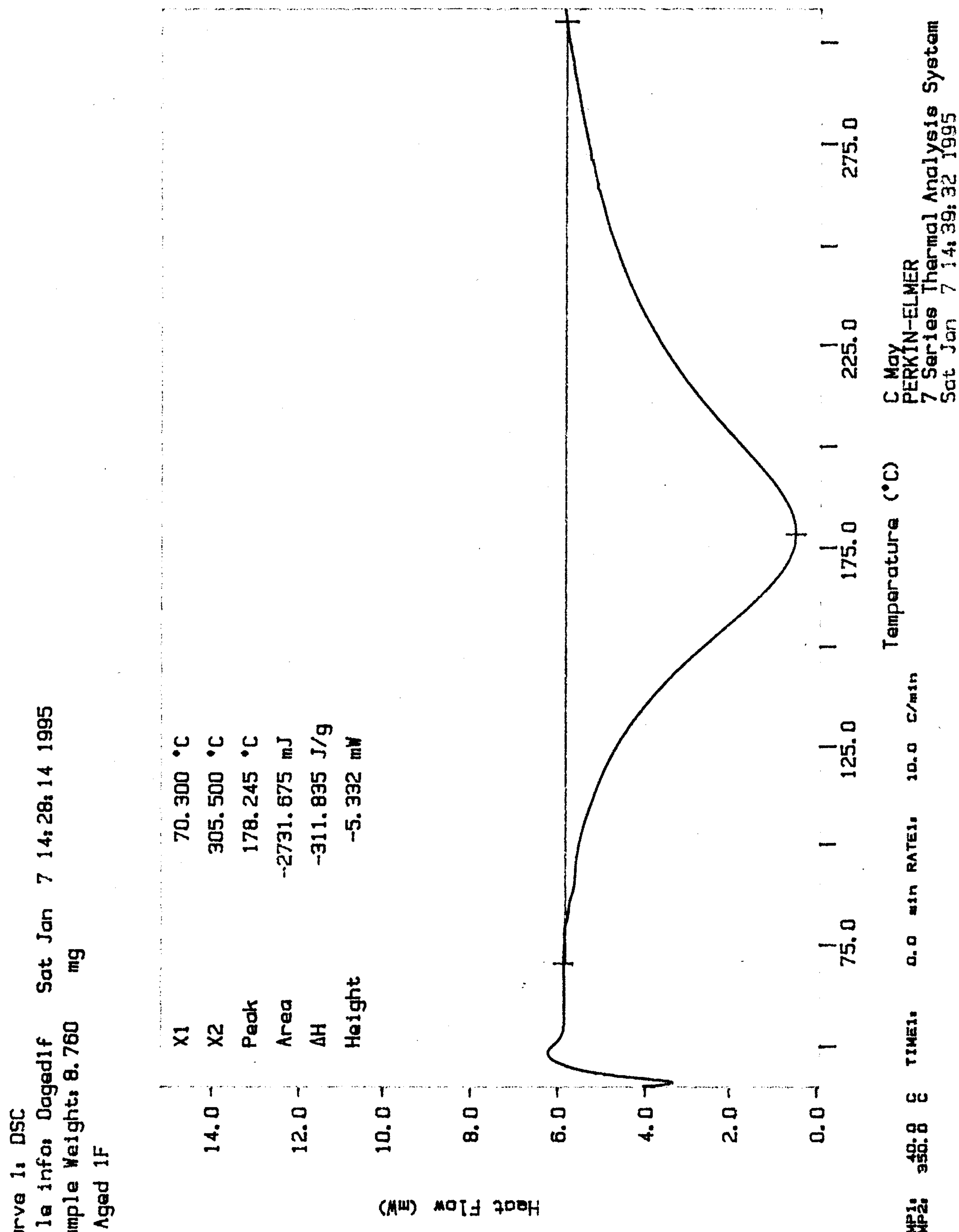


Figure 16.

SYSTEM FROM FIGURE 15 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

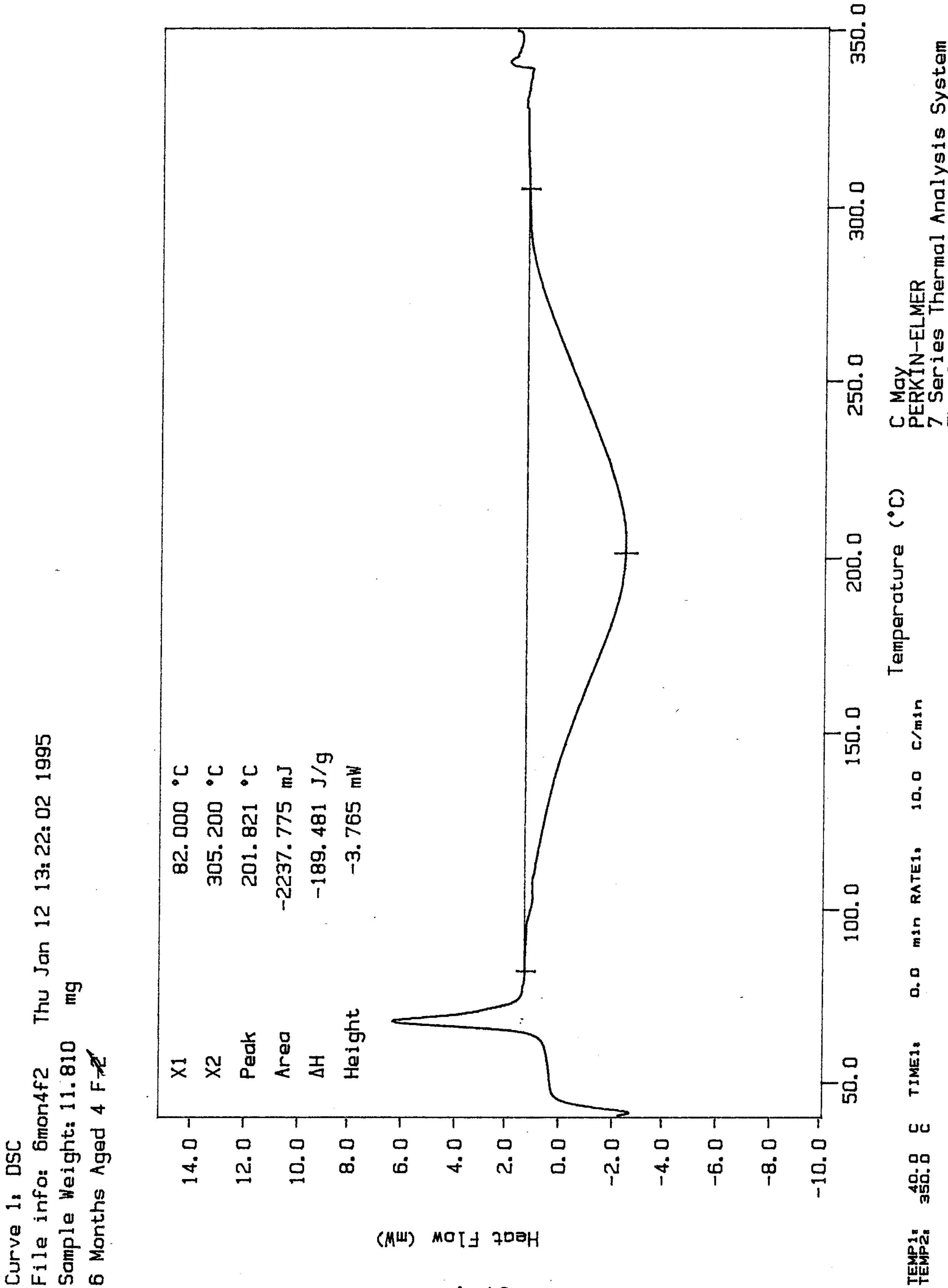


Figure 17.

ECN 1273/HPT CURING AGENT 1061 BEFORE AGING.

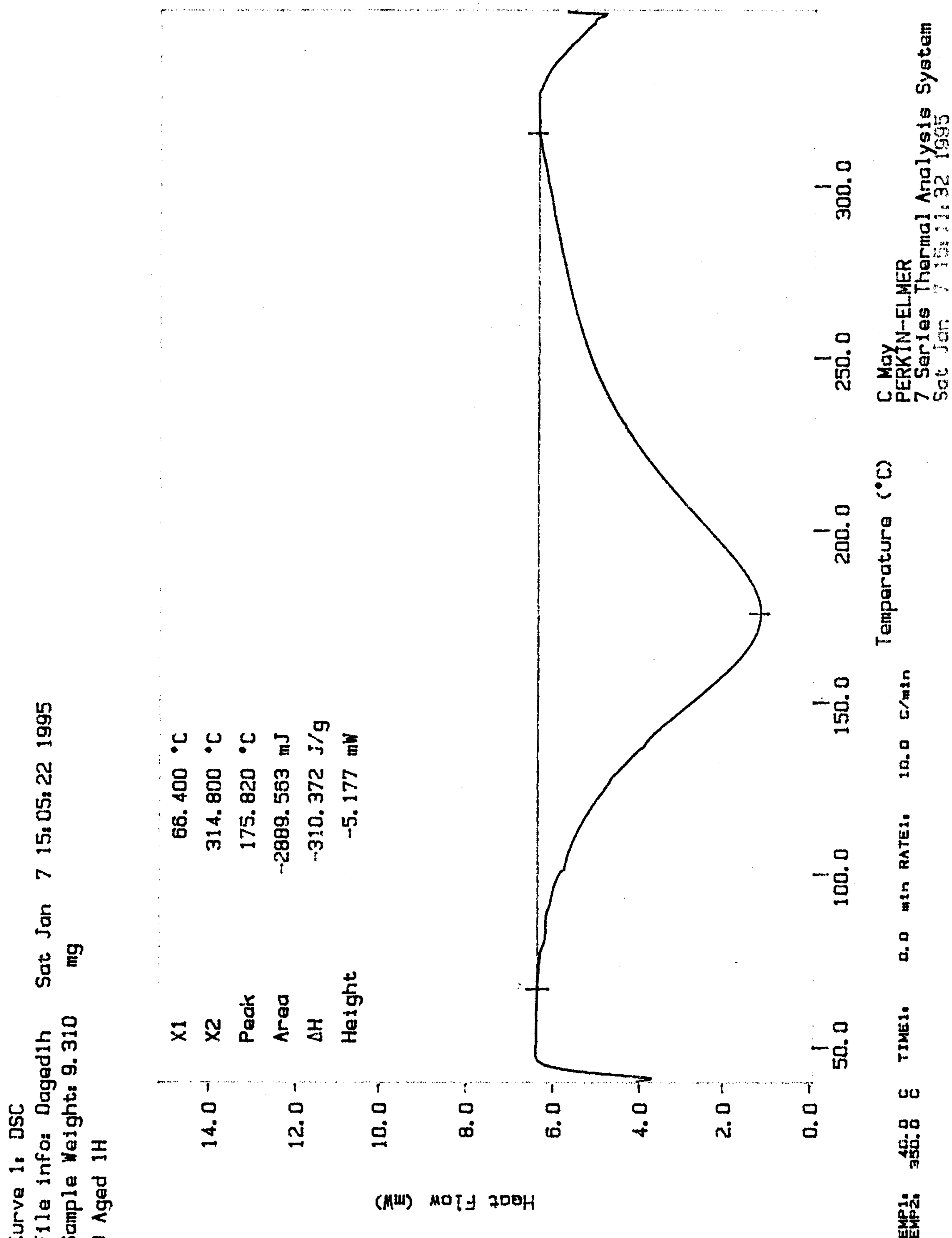


Figure 18.

SYSTEM FROM FIGURE 17 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

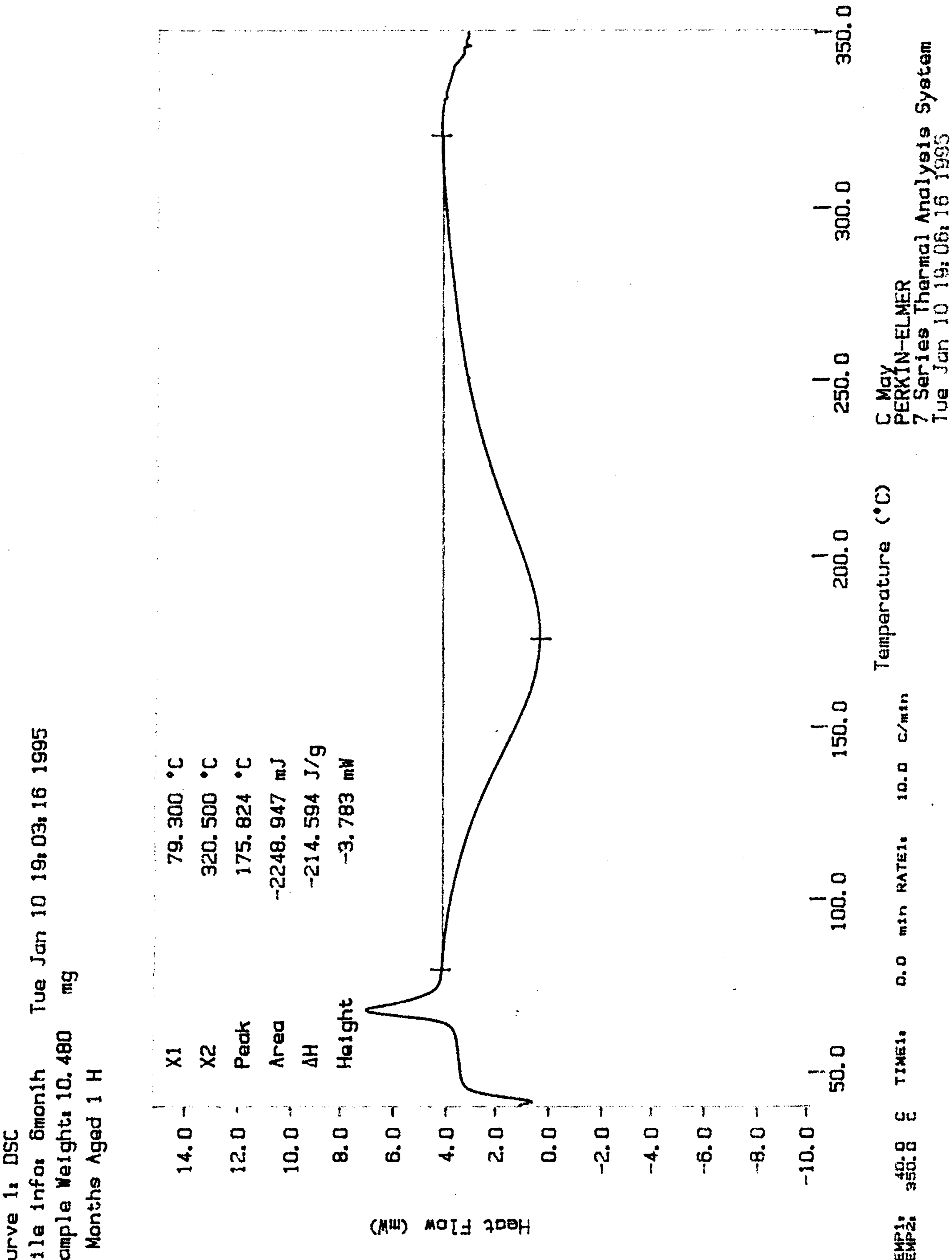


Figure 19.

HPT RESIN 1079/MPDA BEFORE AGING.

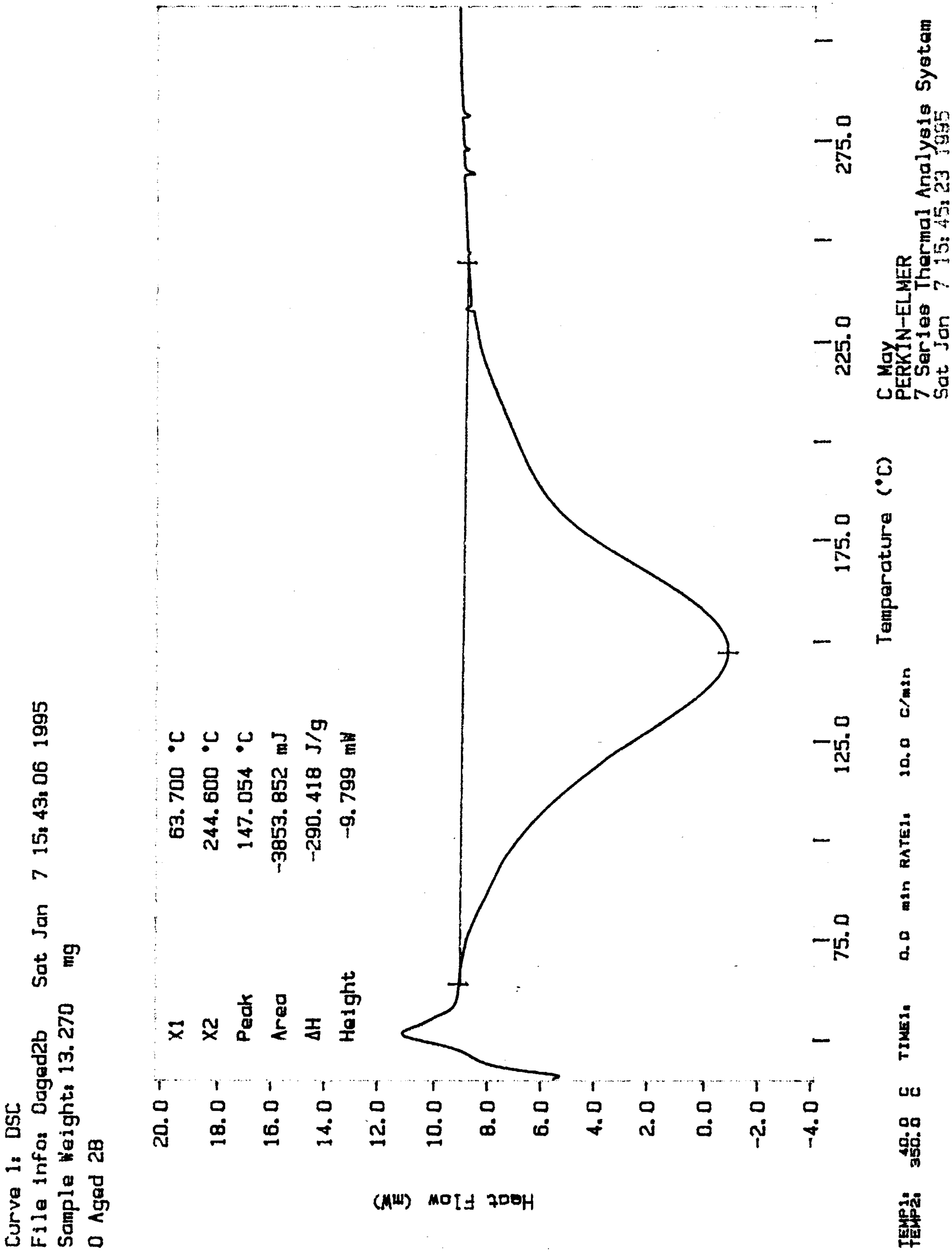


Figure 20.

SYSTEM FROM FIGURE 19 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

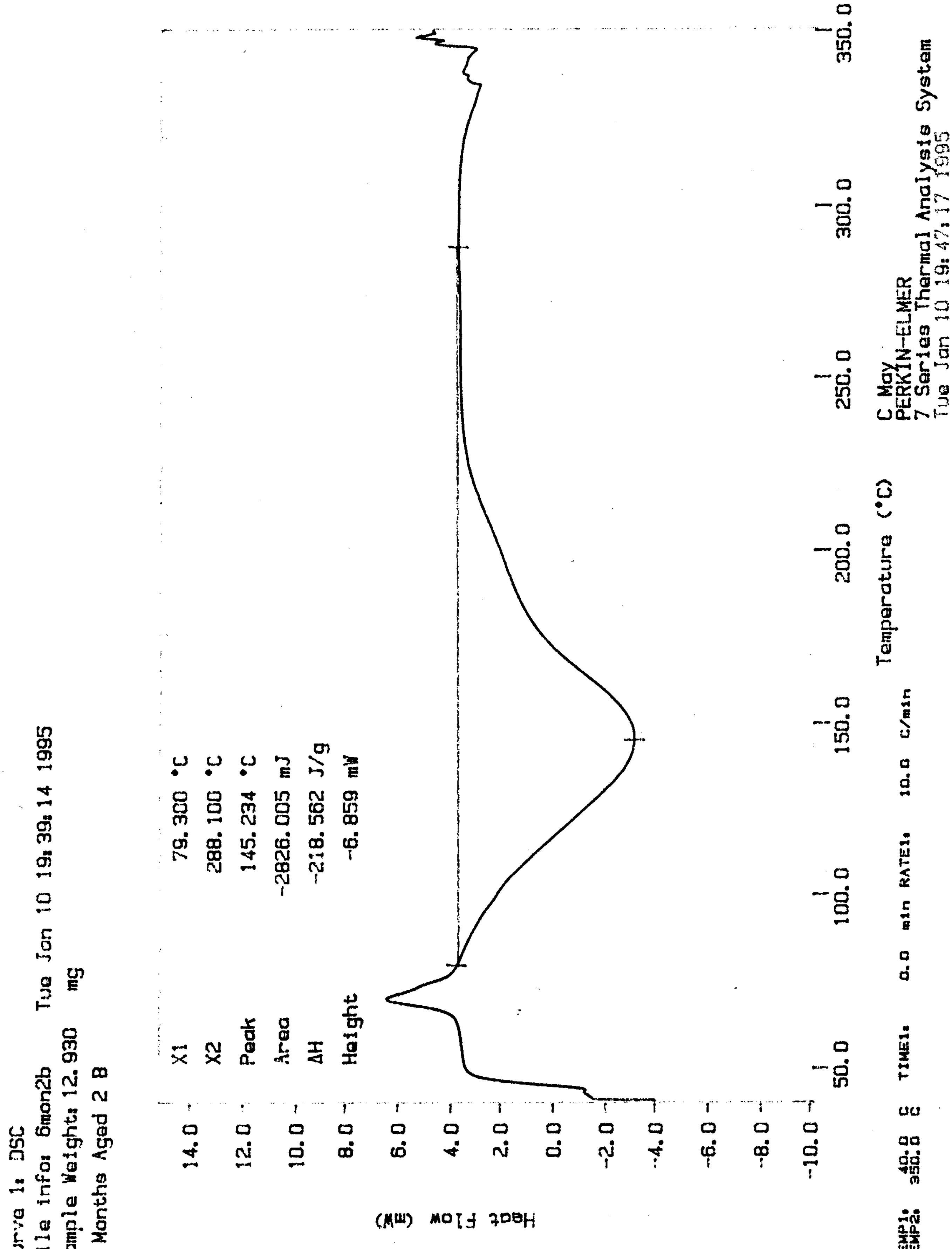


Figure 21.

HPT RESIN 1071/DADS BEFORE AGING

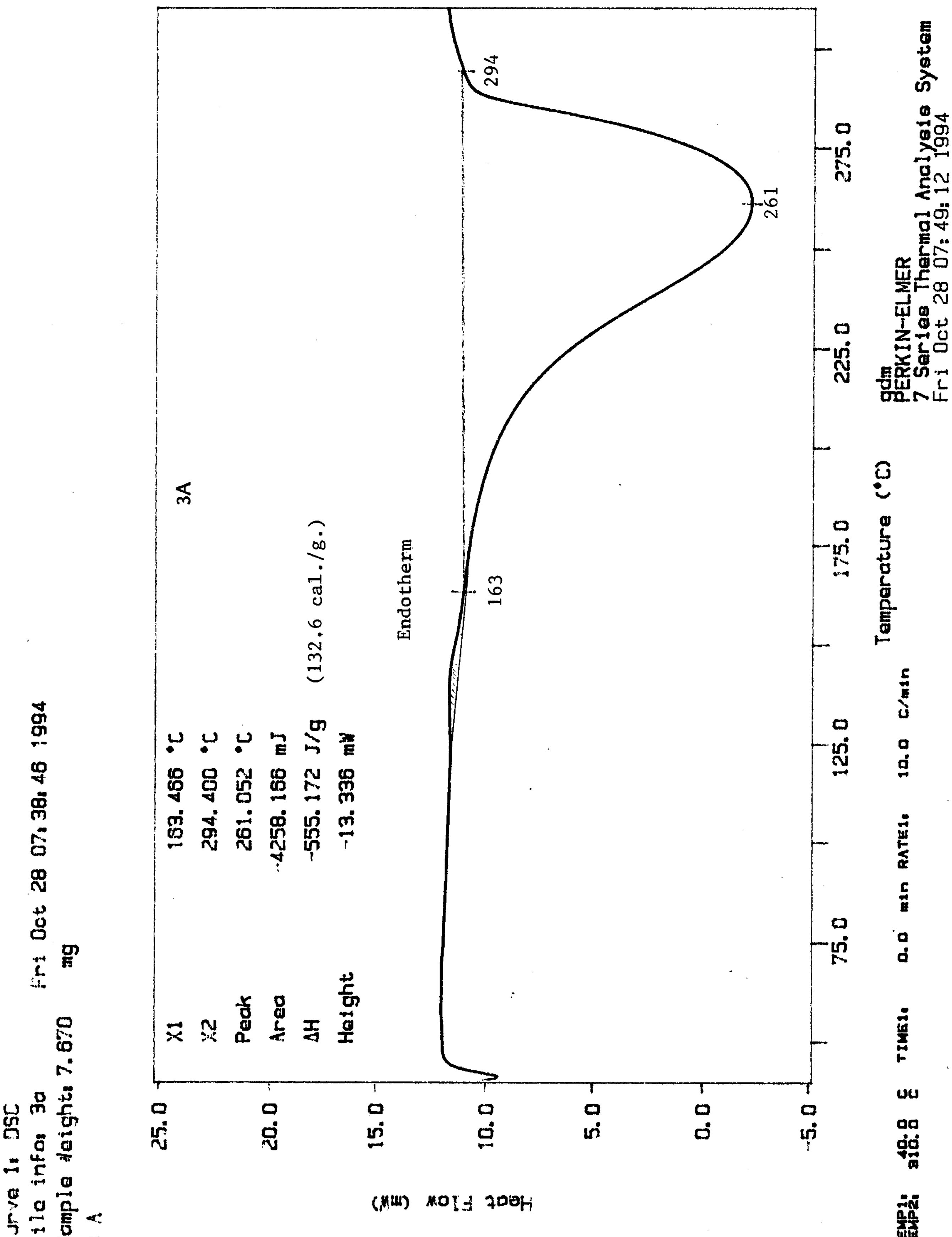


Figure 22.

SYSTEM FROM FIGURE 21AFTER 3 MONTHS' ROOM TEMPERATURE AGING.

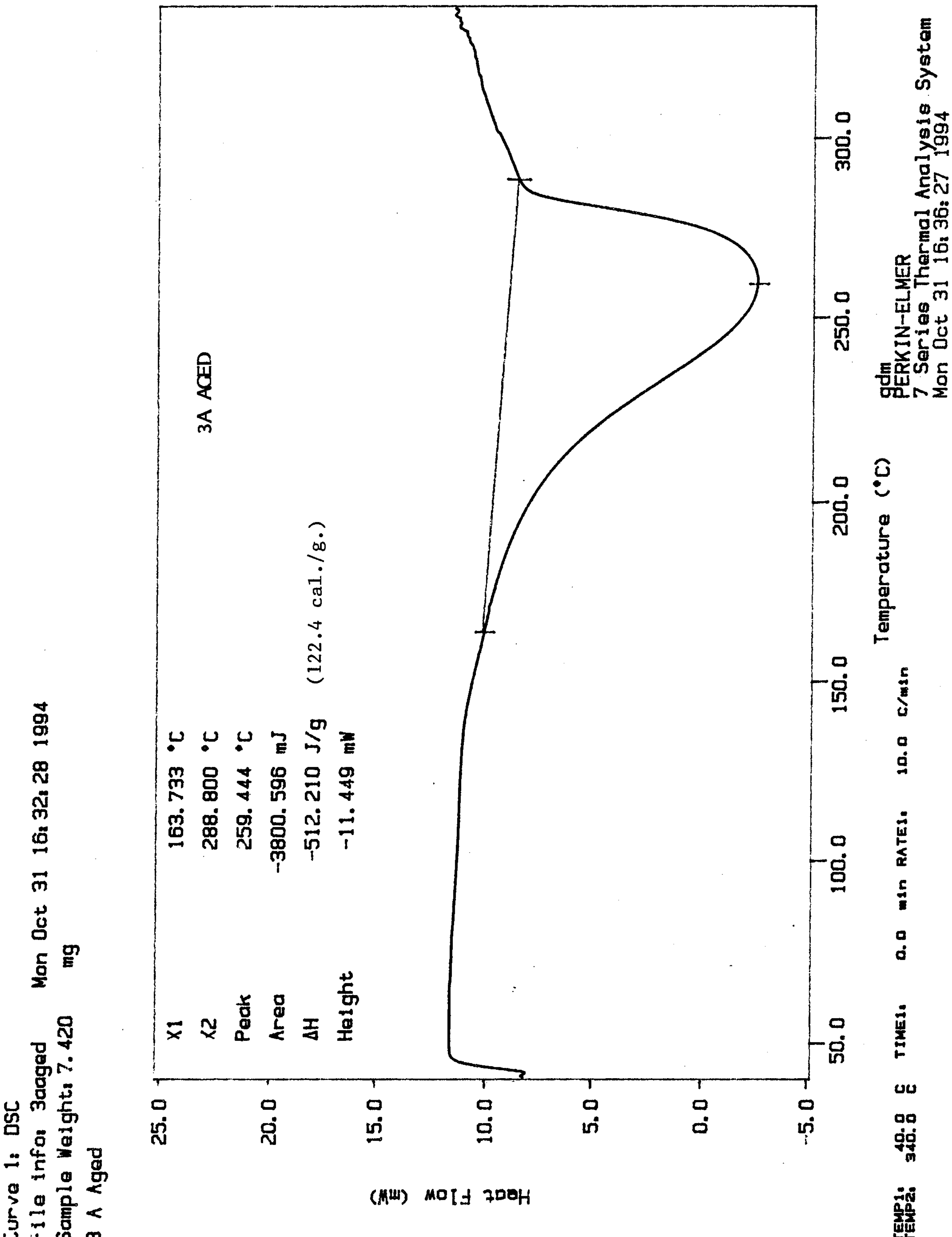


Figure 23.

SYSTEM FROM FIGURE 21 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

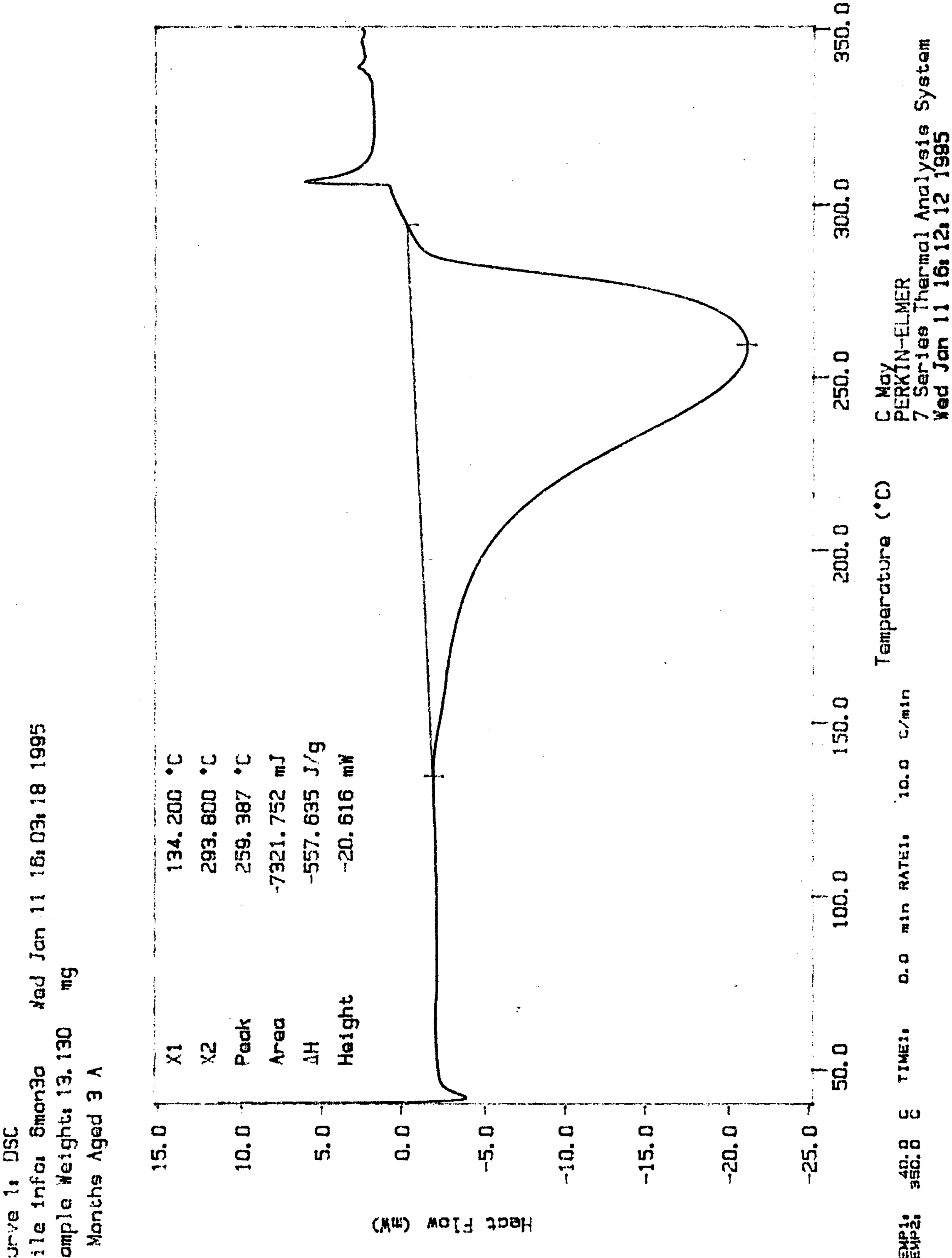


Figure 24.

HPT RESIN 1079/DADS BEFORE AGING

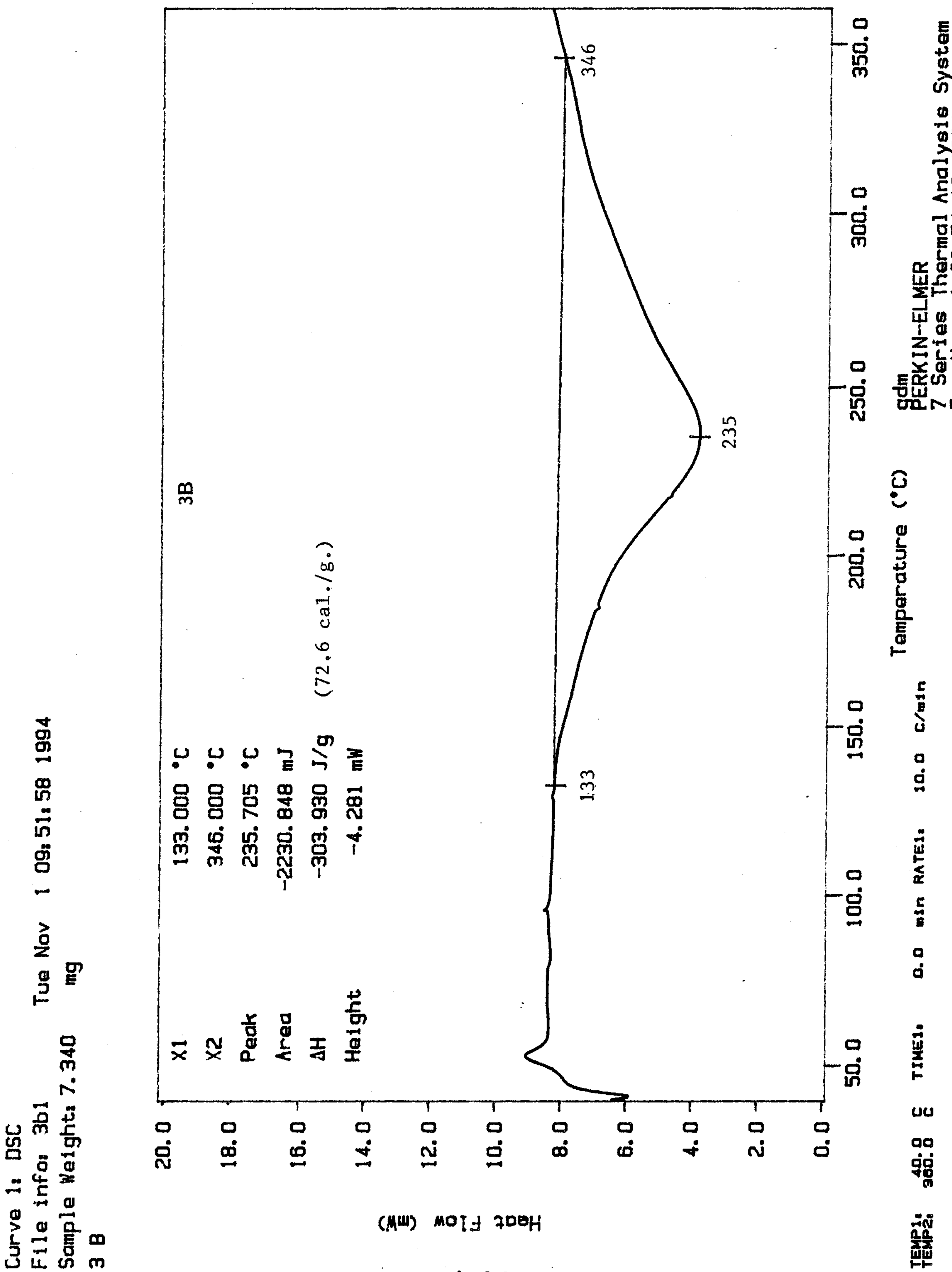


Figure 25.

SYSTEM FROM FIGURE 24 AFTER 3 MONTHS' ROOM TEMPERATURE AGING.

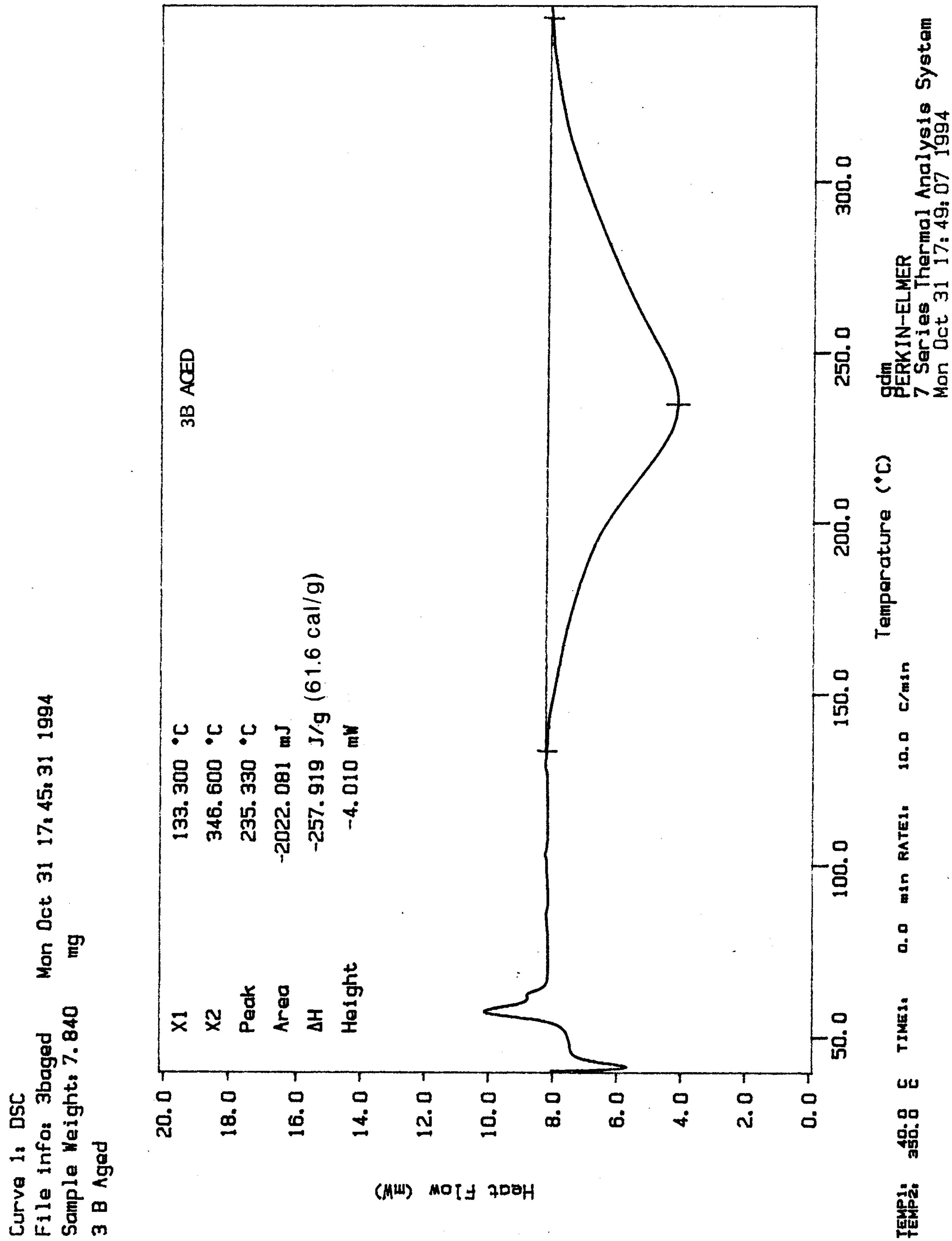


Figure 26.

SYSTEM FROM FIGURE 24 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

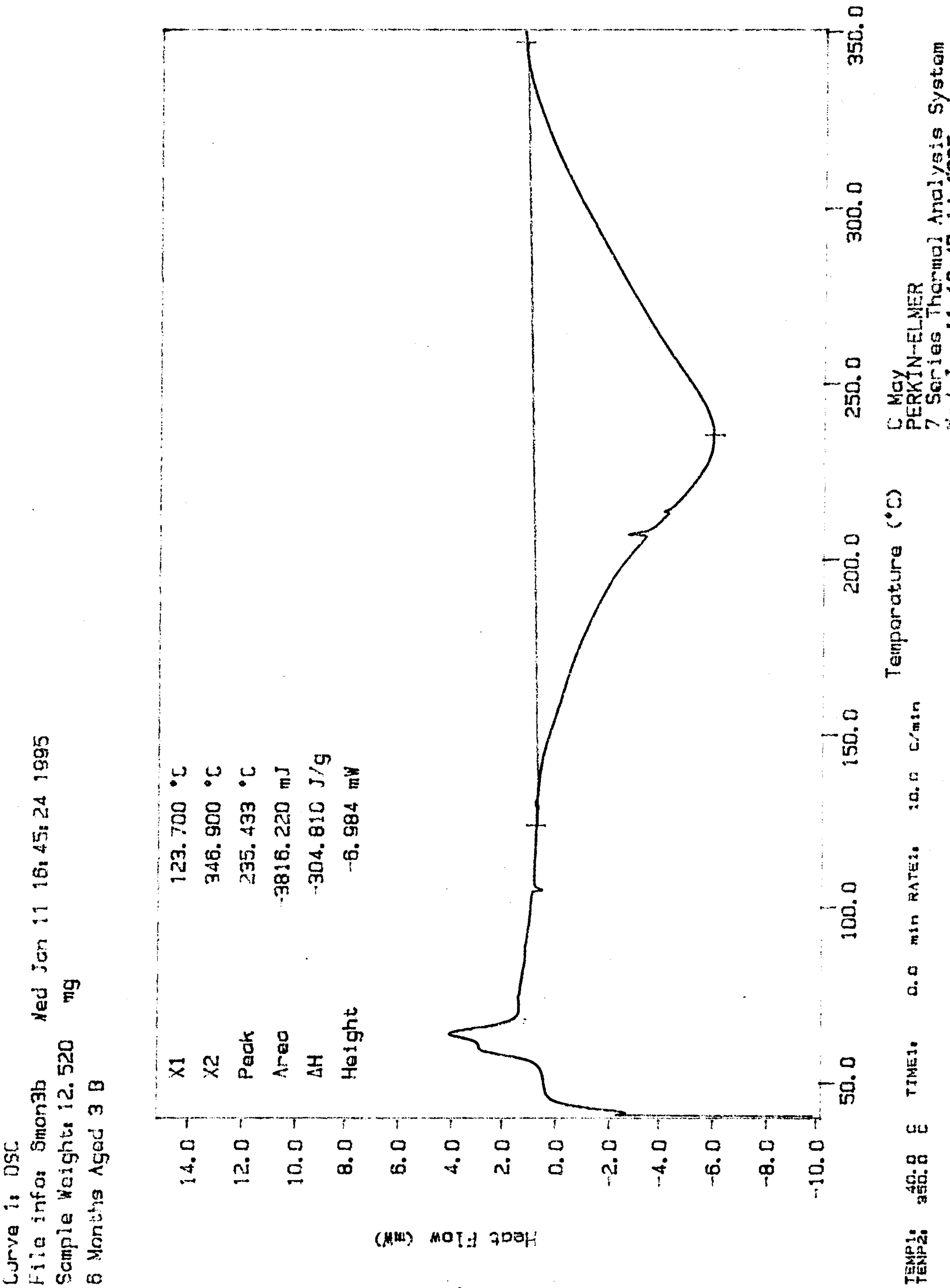


FIGURE 27.

SU-8/DADS BEFORE AGING.

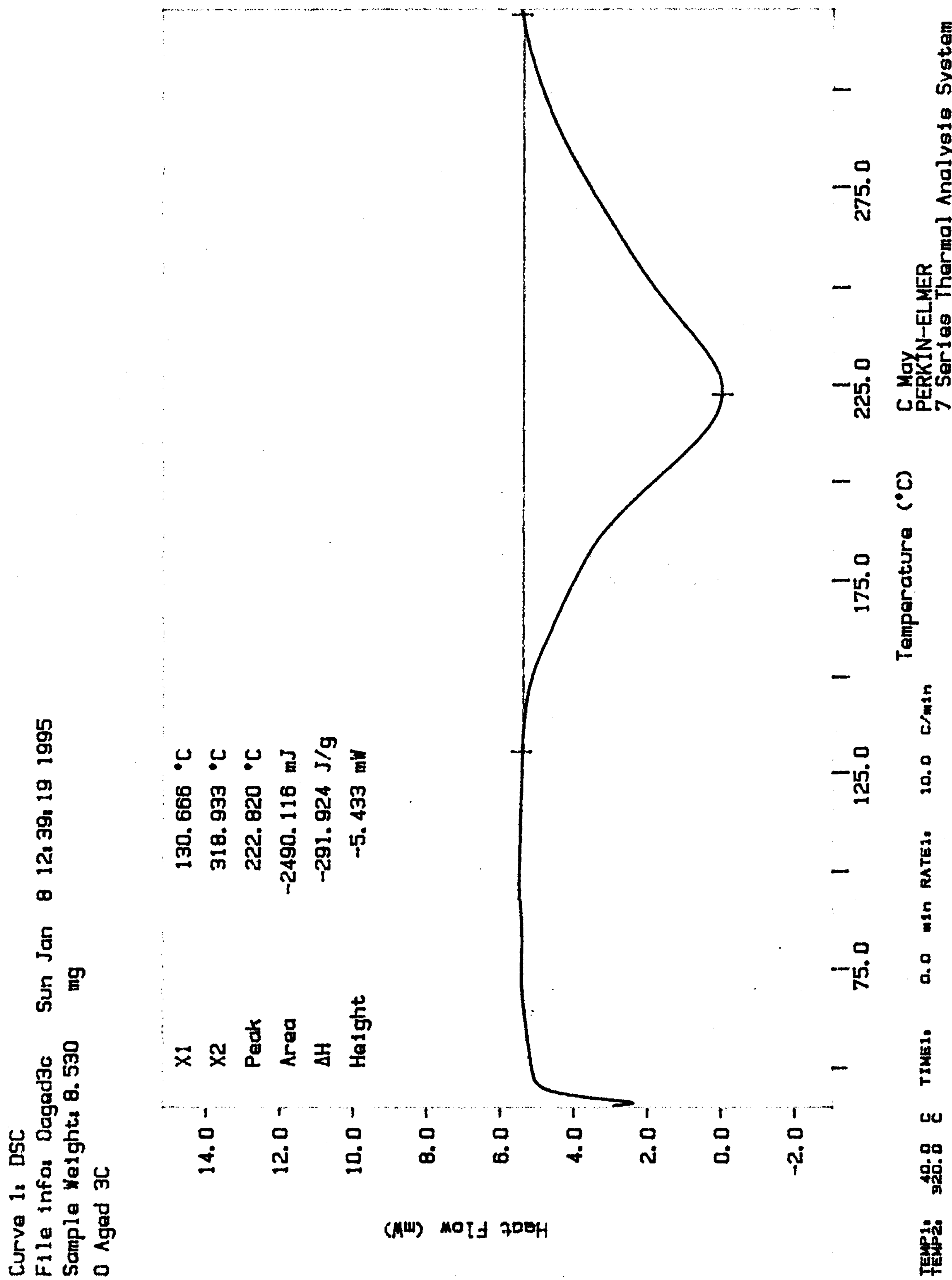


Figure 28.

SYSTEM FROM FIGURE 27 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

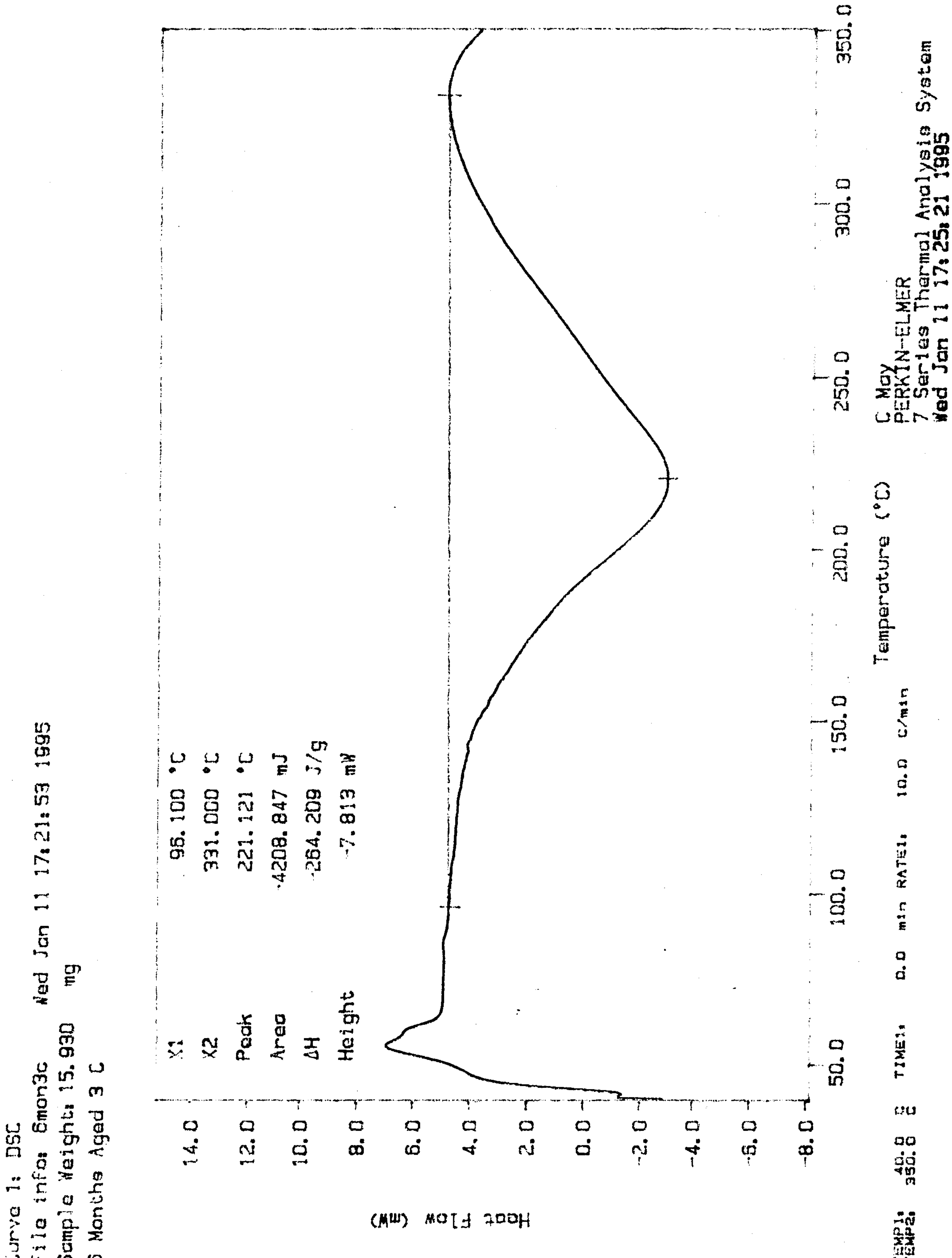


Figure 29.

TACTIX 742/DADS BEFORE AGING.

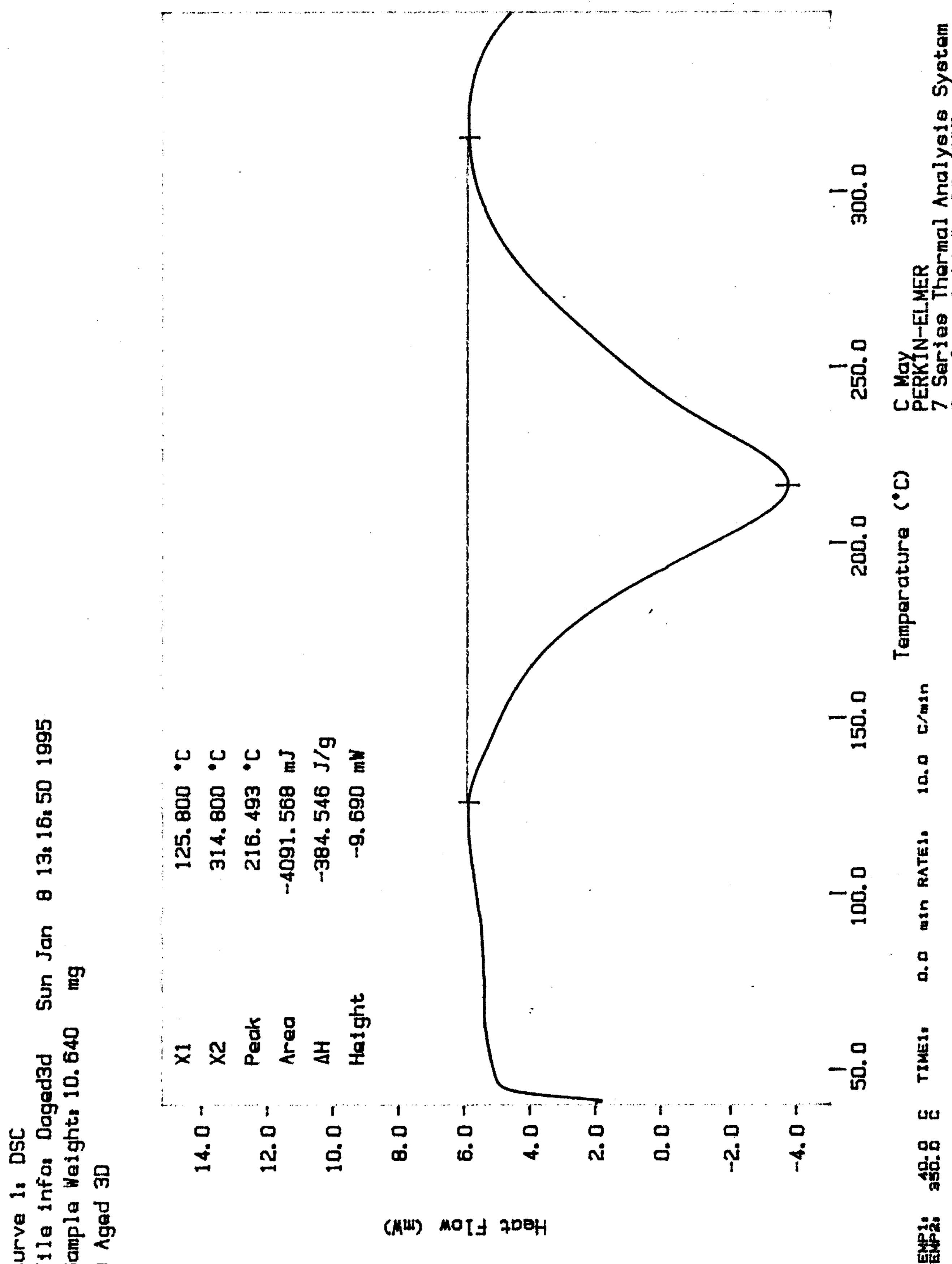


Figure 30.

SYSTEM FROM FIGURE 29 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

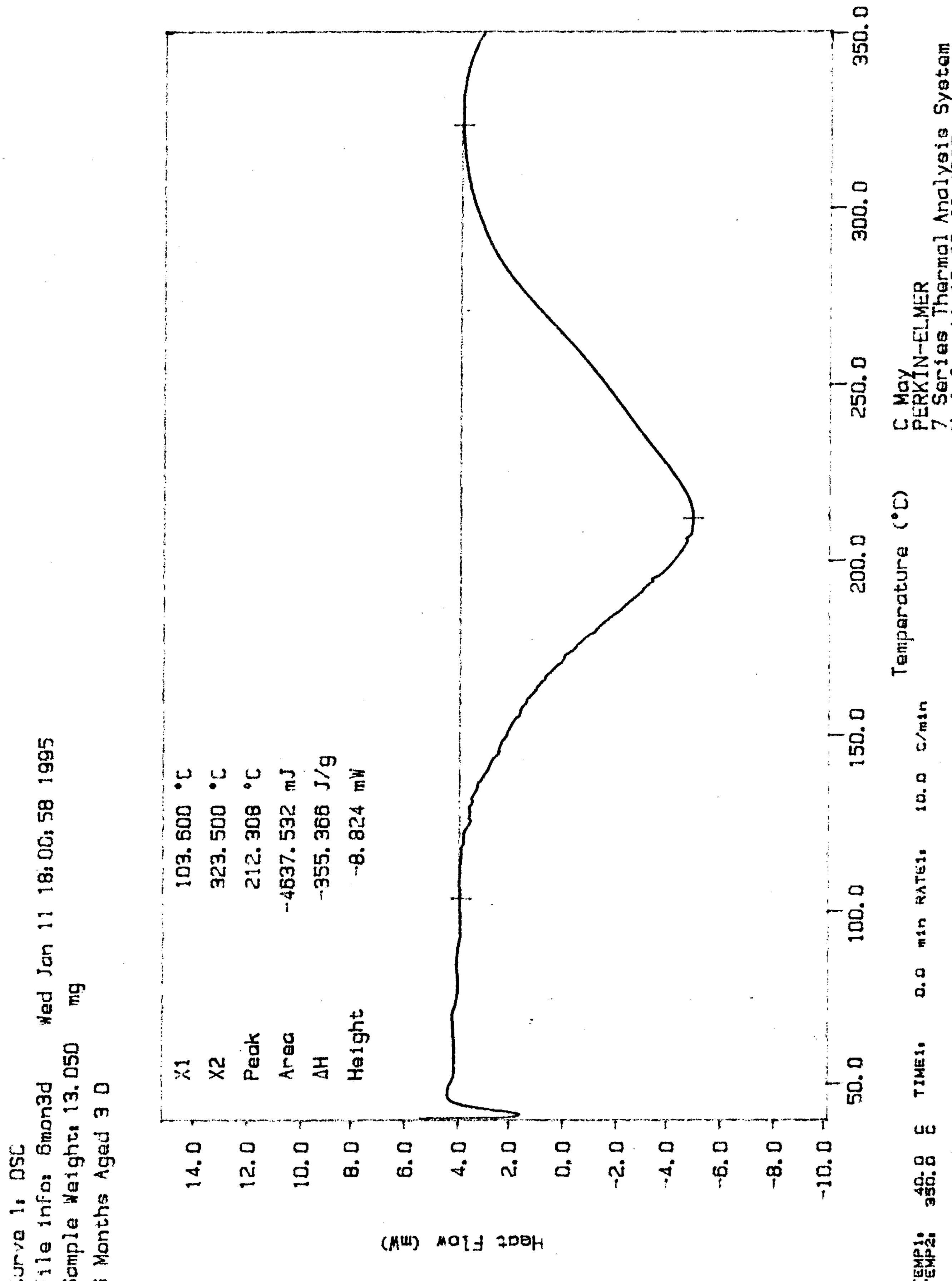


Figure 31.

EPON 1031/DADS BEFORE AGING.

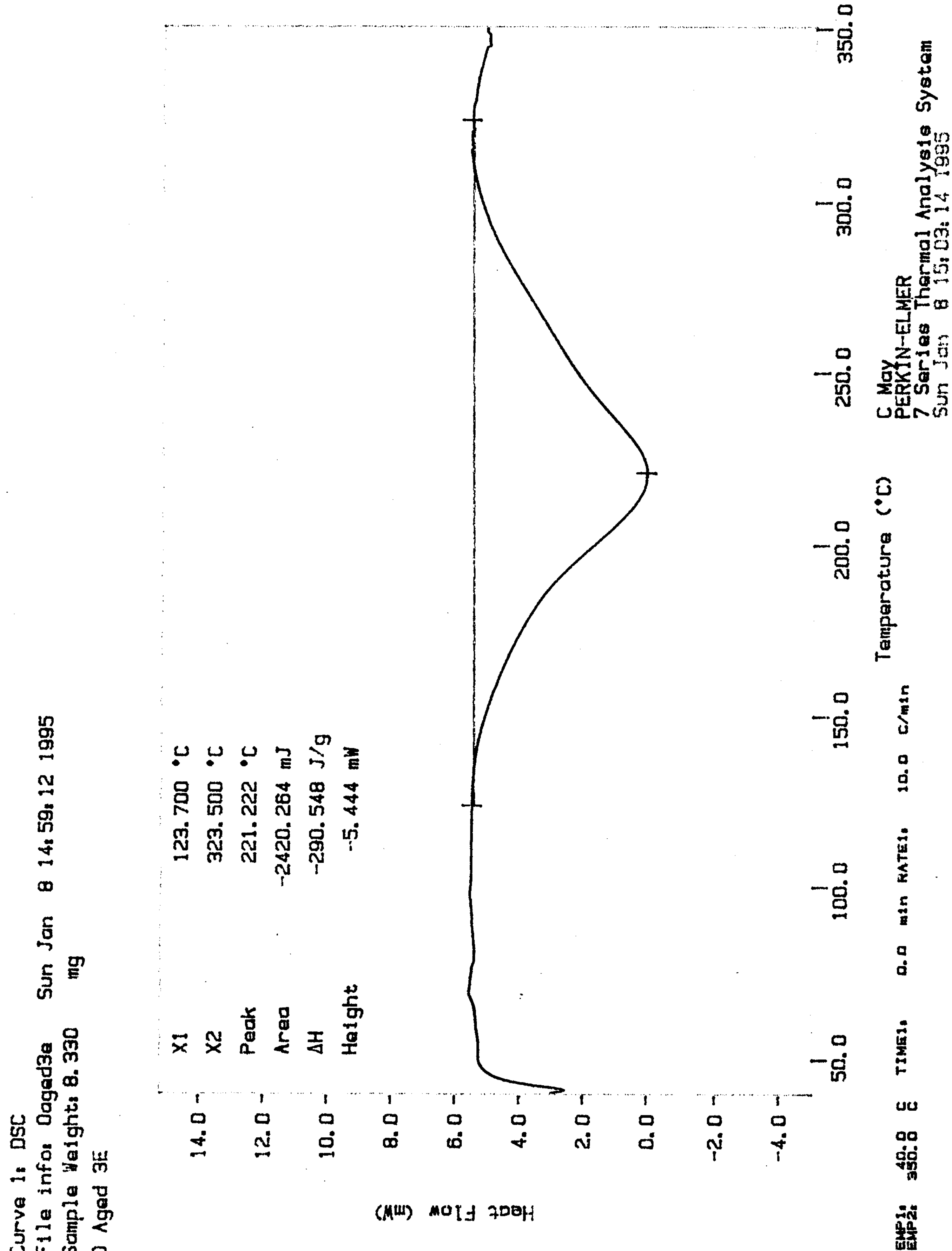


Figure 32.

SYSTEM FROM FIGURE 31 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

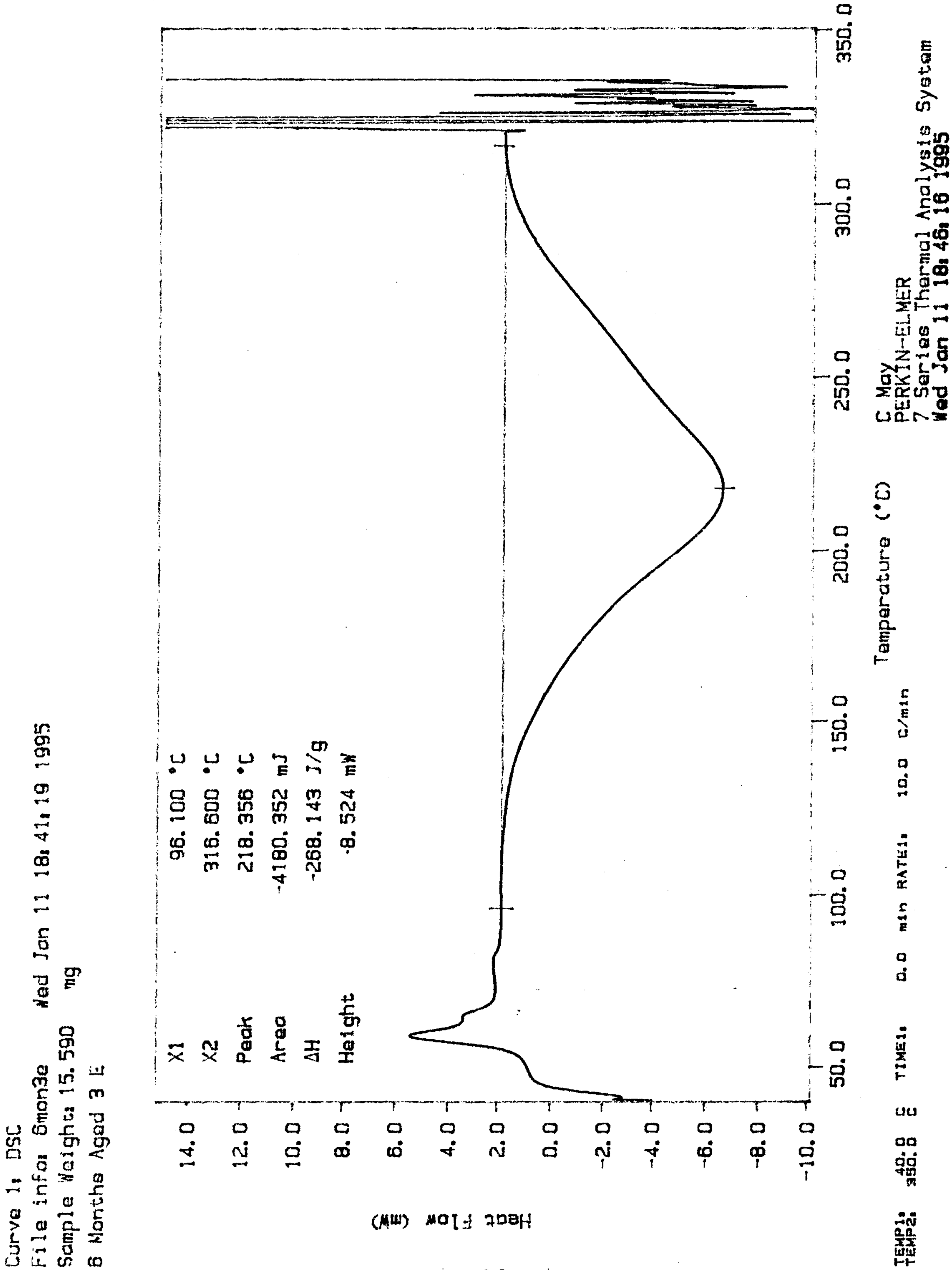


Figure 33.

DPS-164/DADS BEFORE AGING

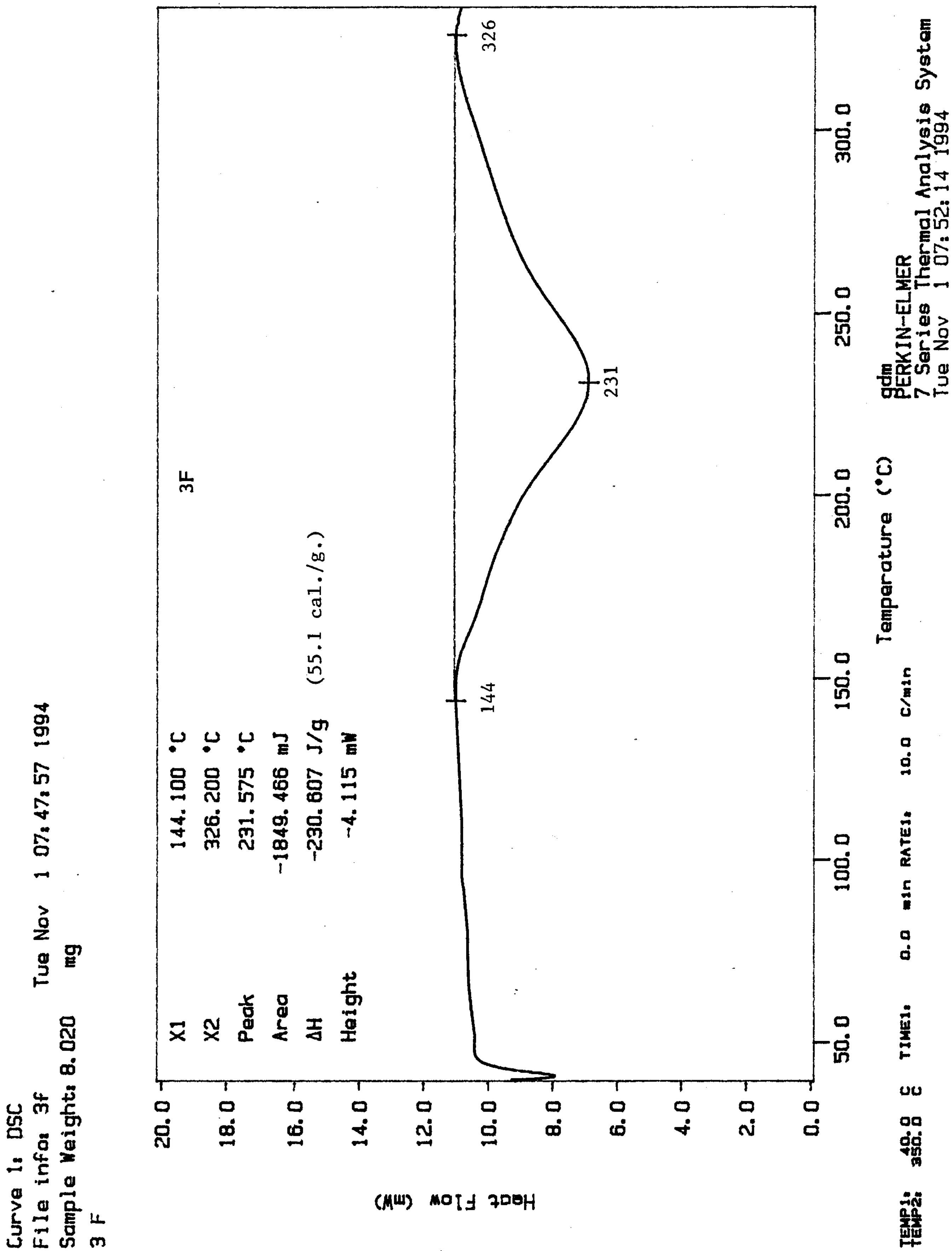


Figure 34.

SYSTEM FROM FIGURE 33 AFTER 3 MONTHS' ROOM TEMPERATURE AGING.

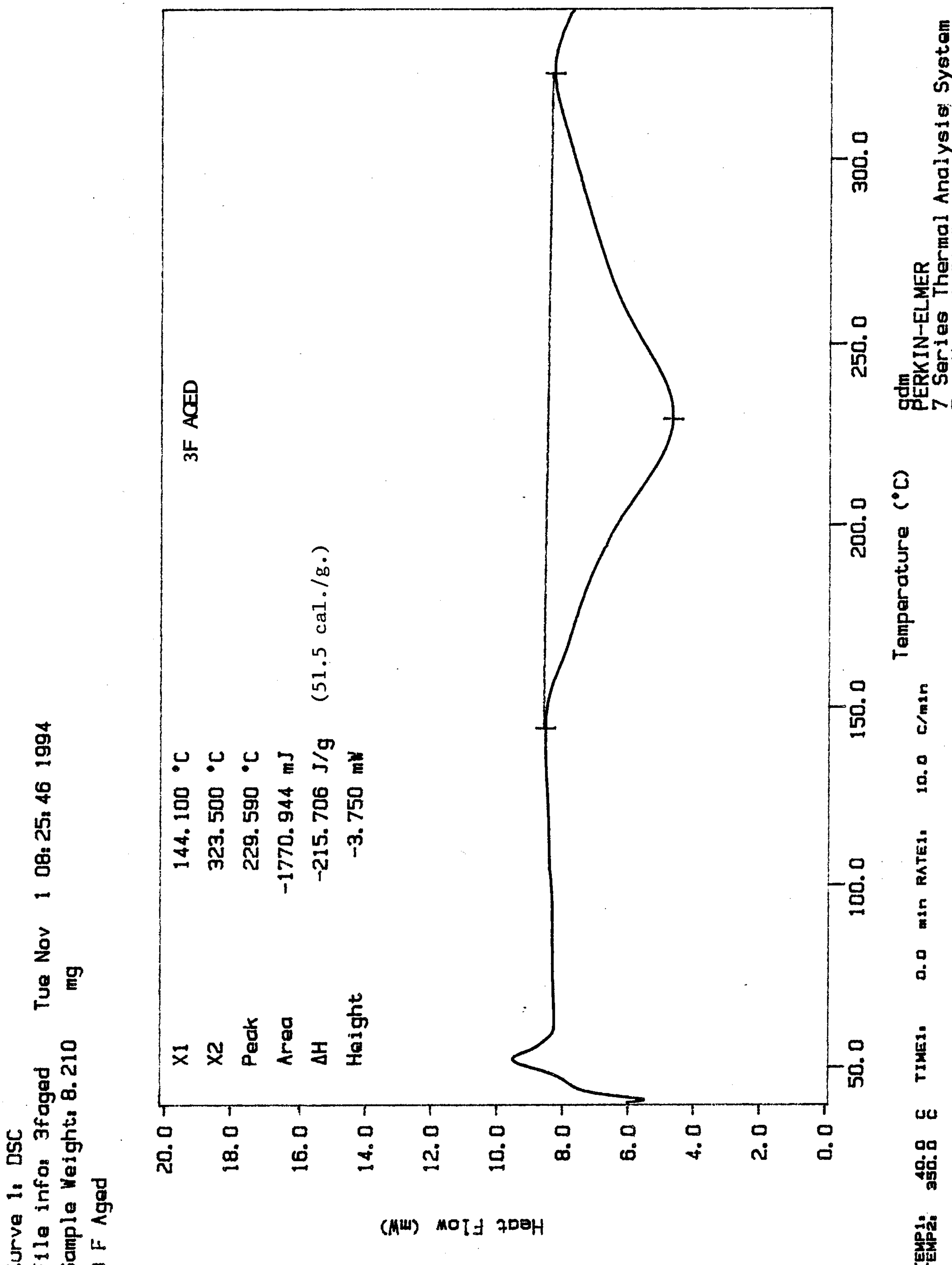


Figure 35.

SYSTEM FROM FIGURE 33 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

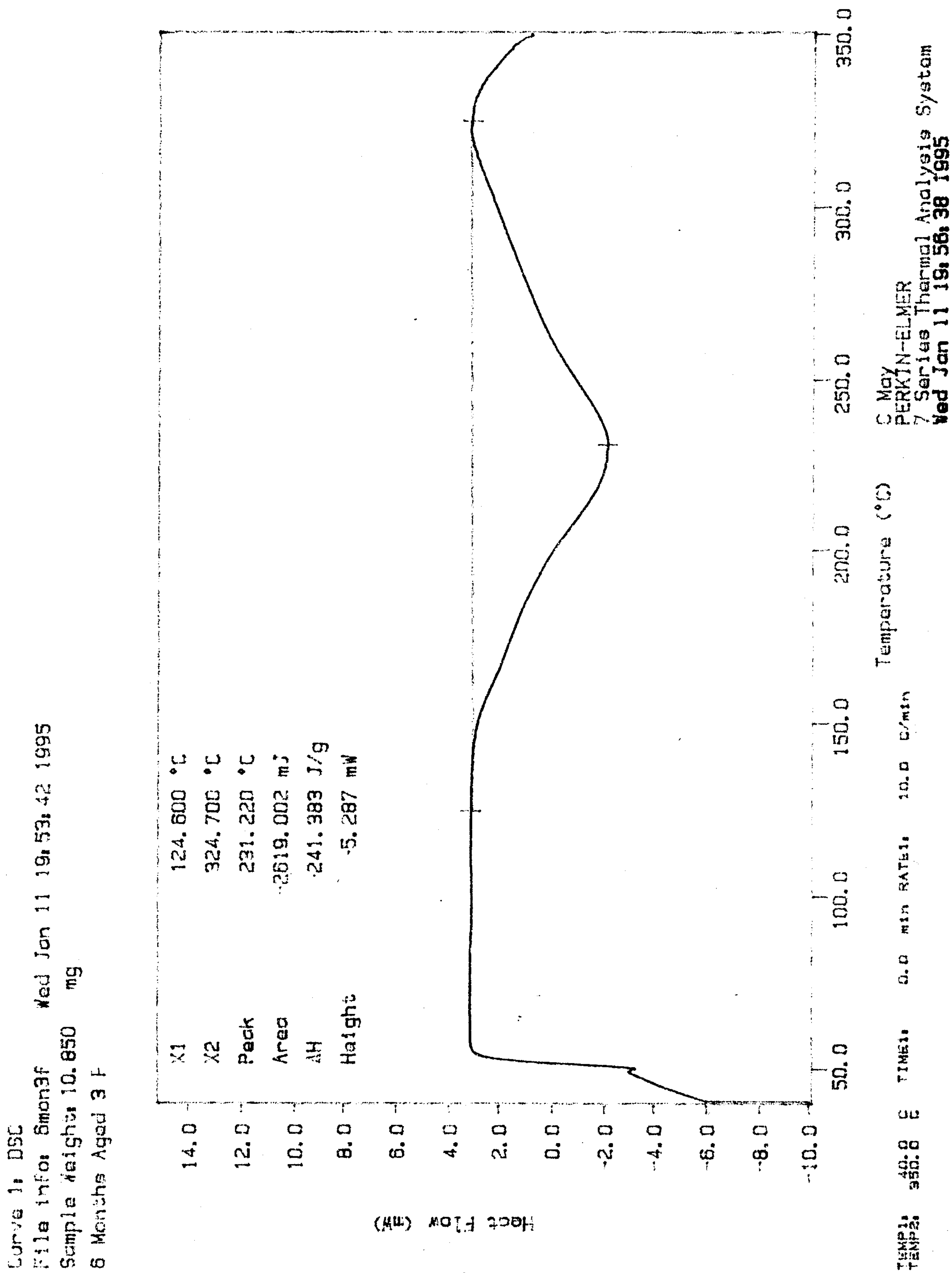


Figure 36.

EPON 1050/DADS BEFORE AGING.

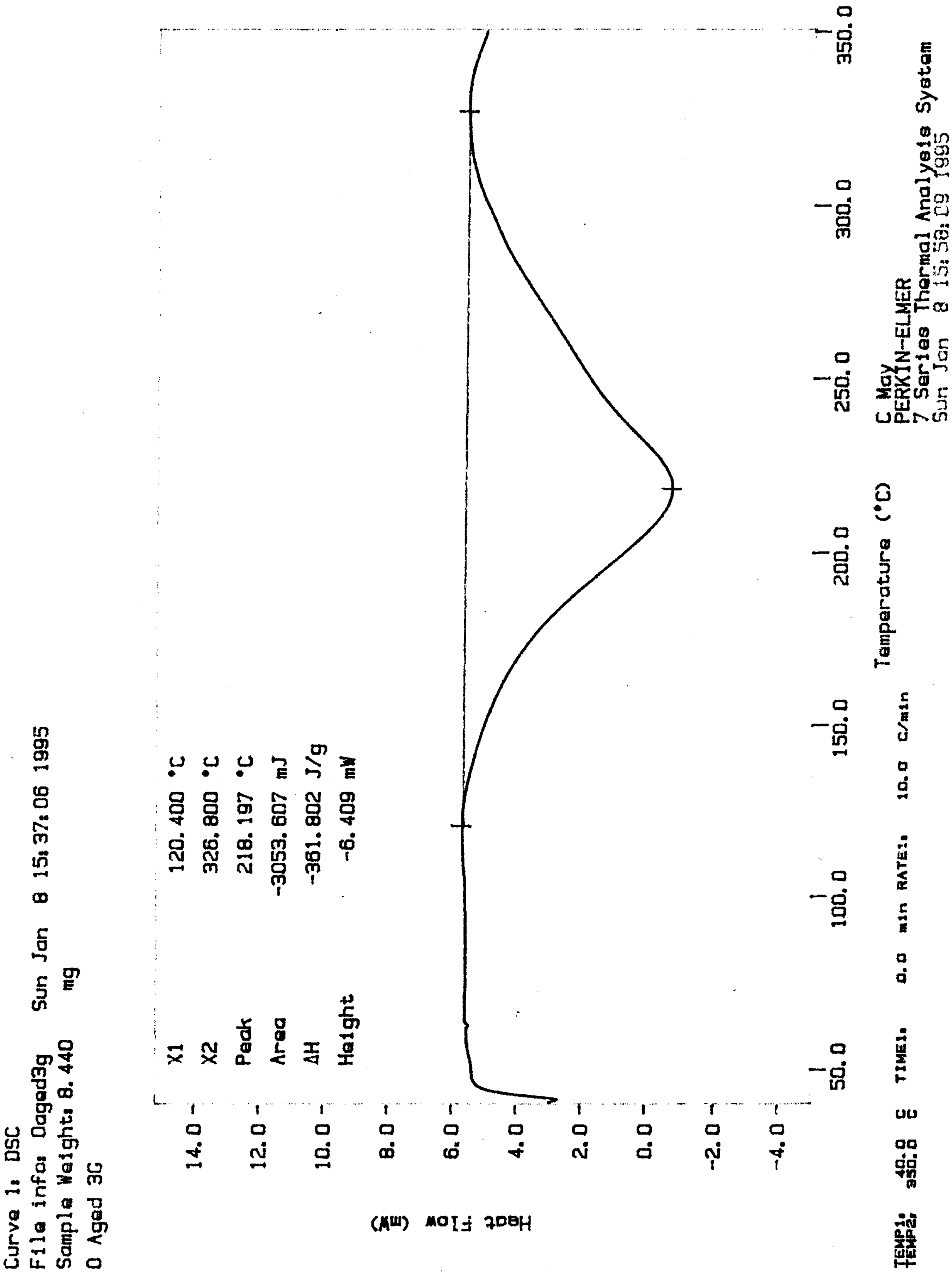


FIGURE 37.

SYSTEM FROM FIGURE 36 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

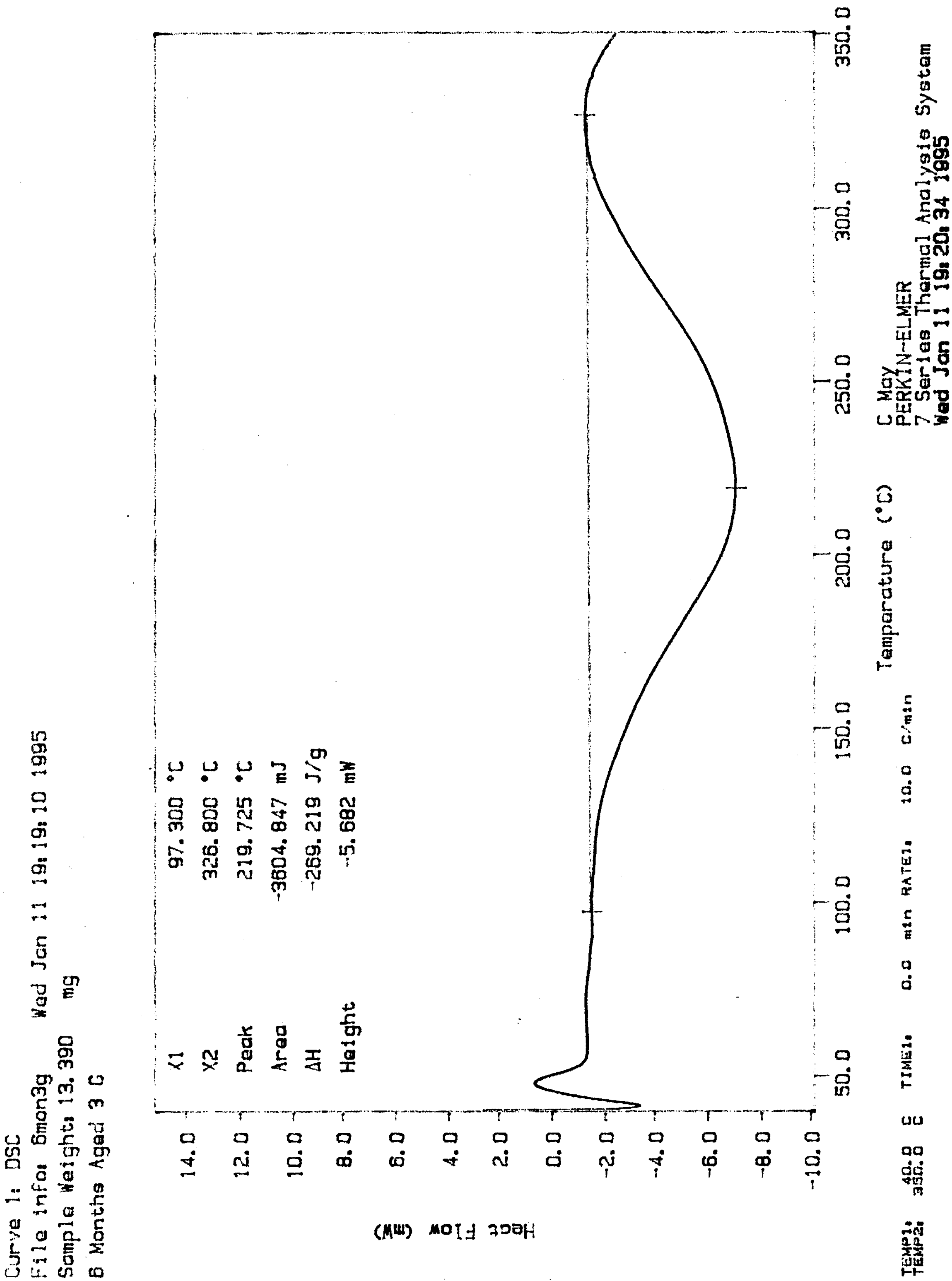


Figure 38.

ECN 1273/DADS BEFORE AGING.

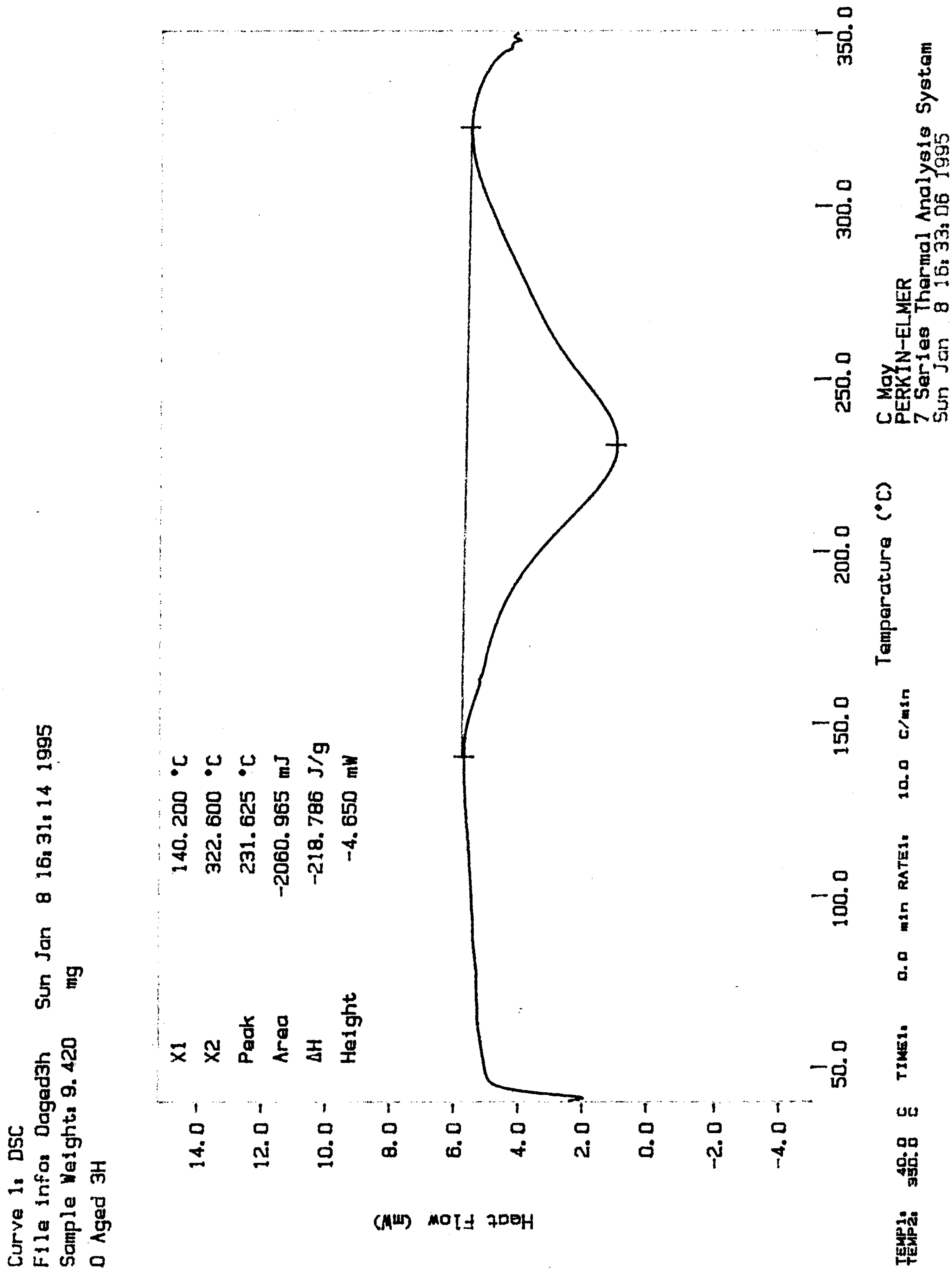


Figure 39.

SYSTEM FROM FIGURE 38 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

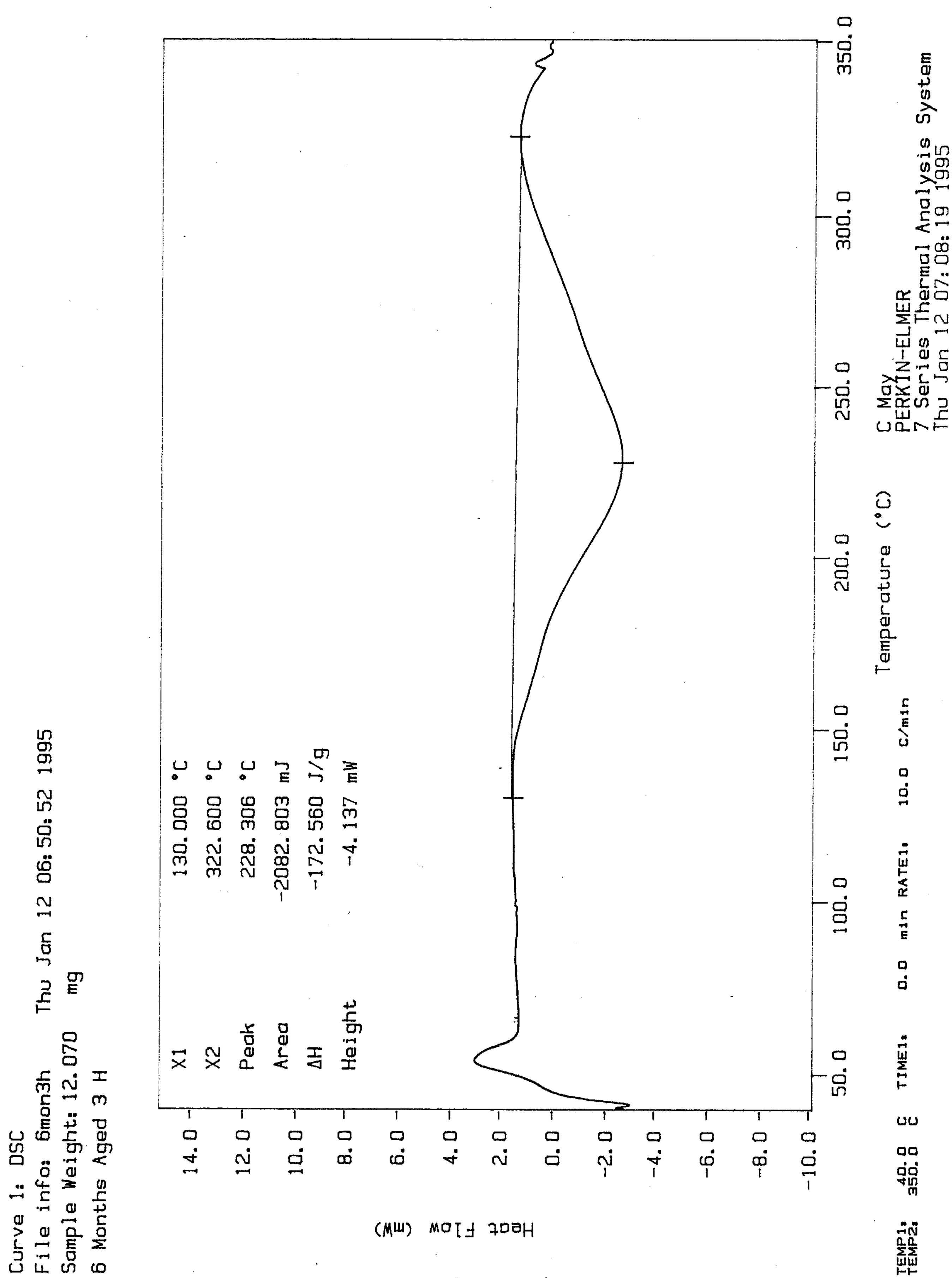


Figure 40.

HPT RESIN 1071/ETHACURE 100 BEFORE AGING.

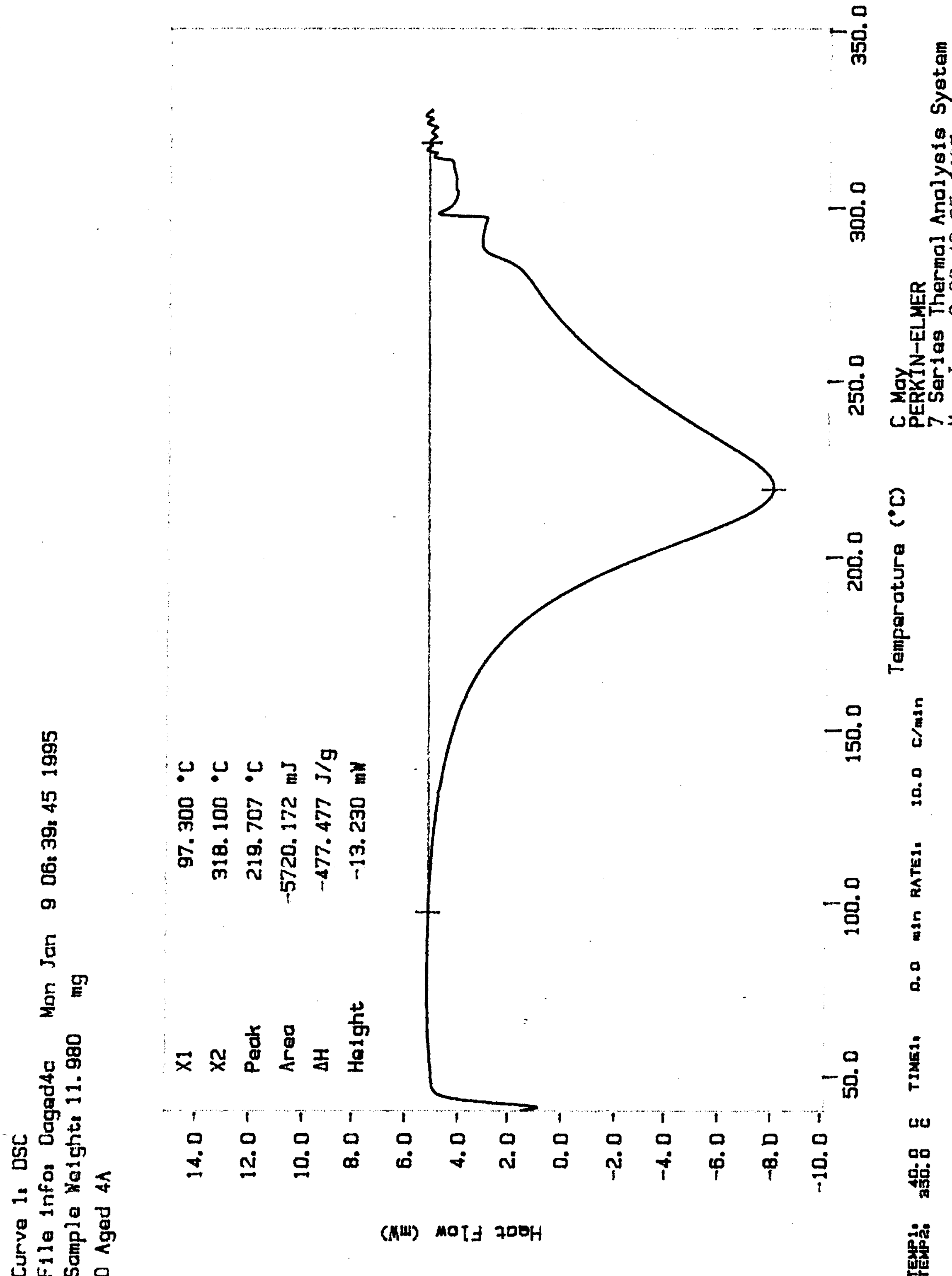


Figure 41.

SYSTEM FROM FIGURE 40 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

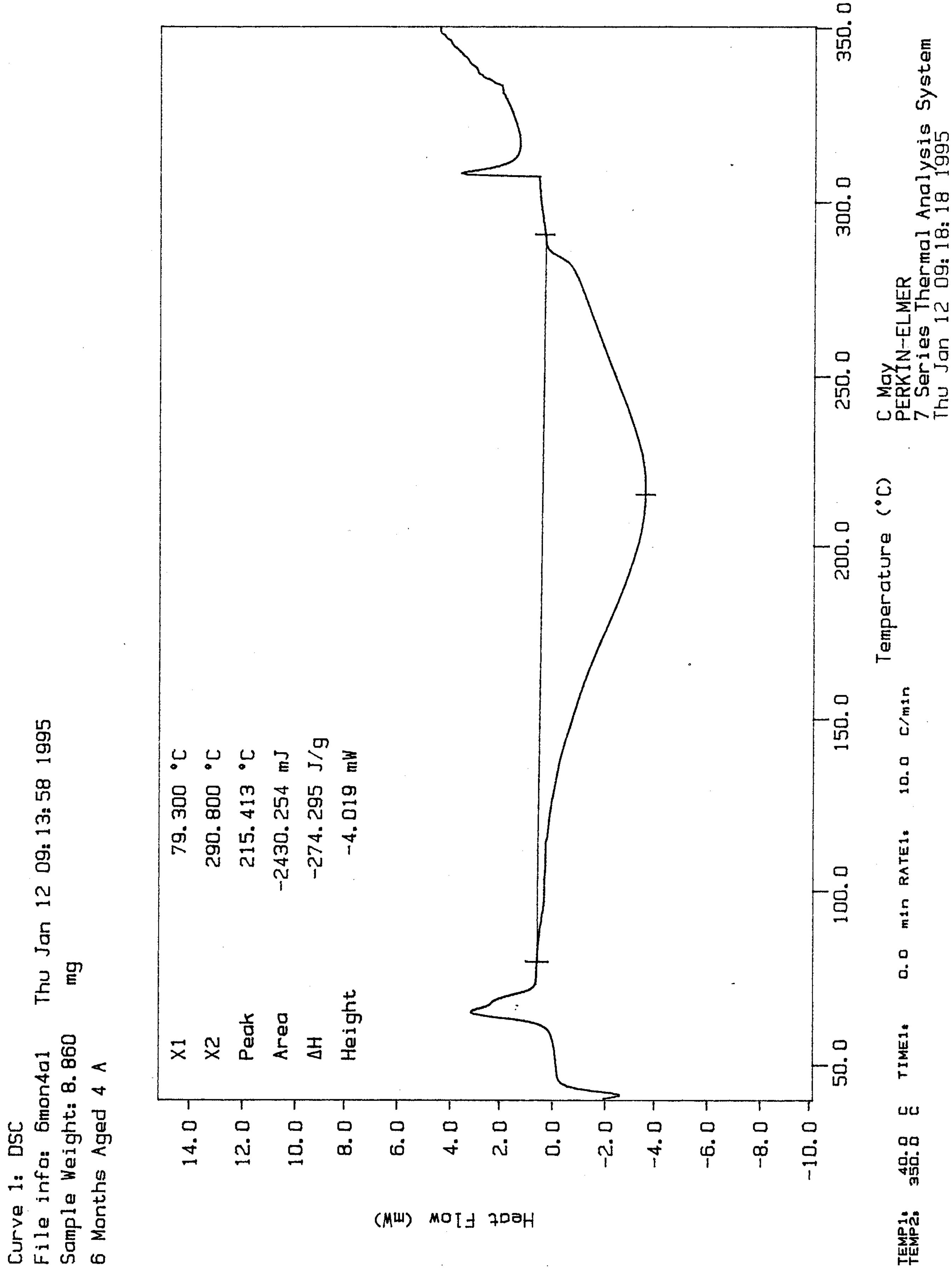


Figure 42.

HPT RESIN 1079/ETHACURE 100 BEFORE AGING

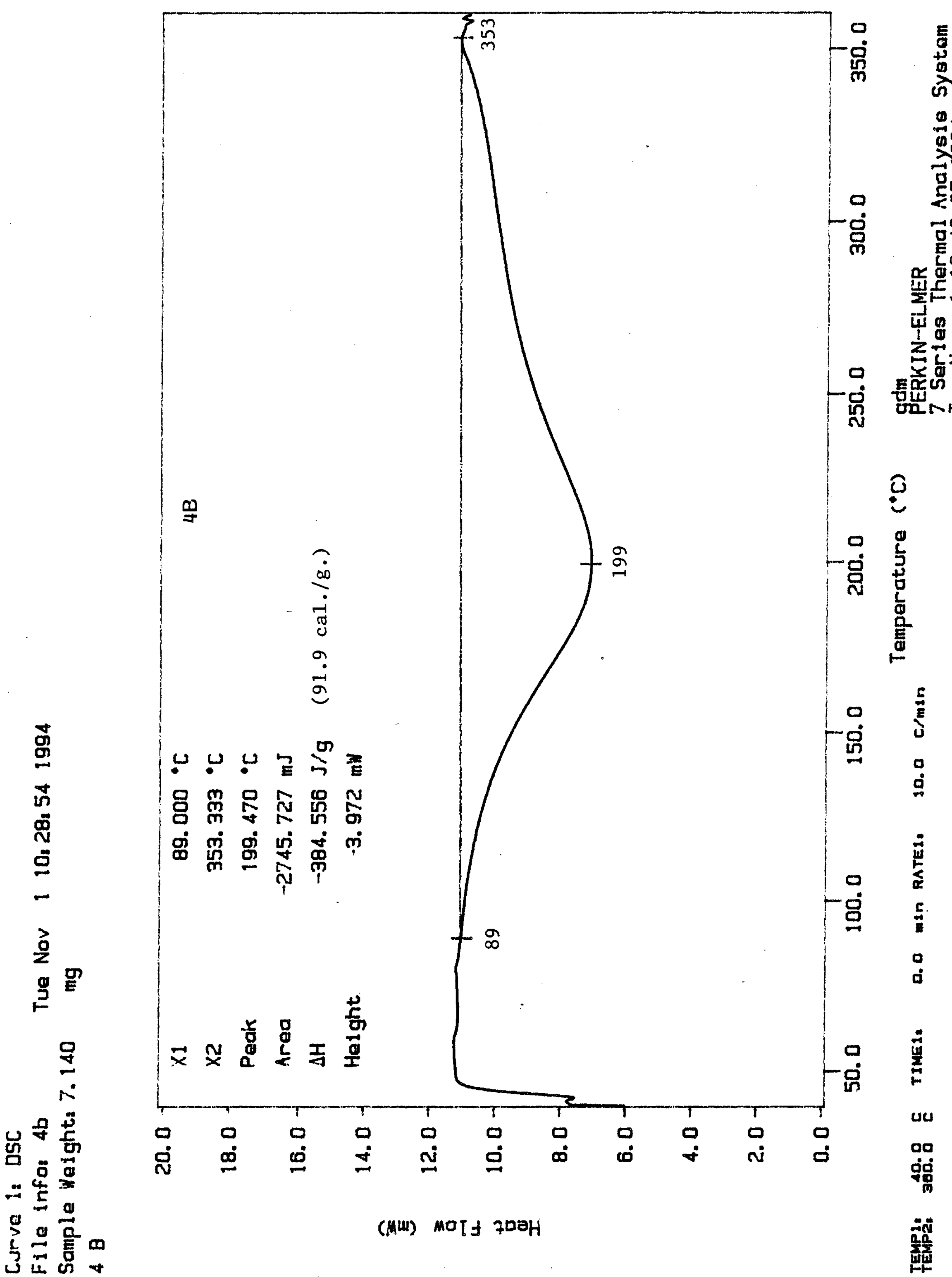


Figure 43.

SYSTEM FROM FIGURE 42 AFTER 3 MONTHS' ROOM TEMPERATURE AGING.

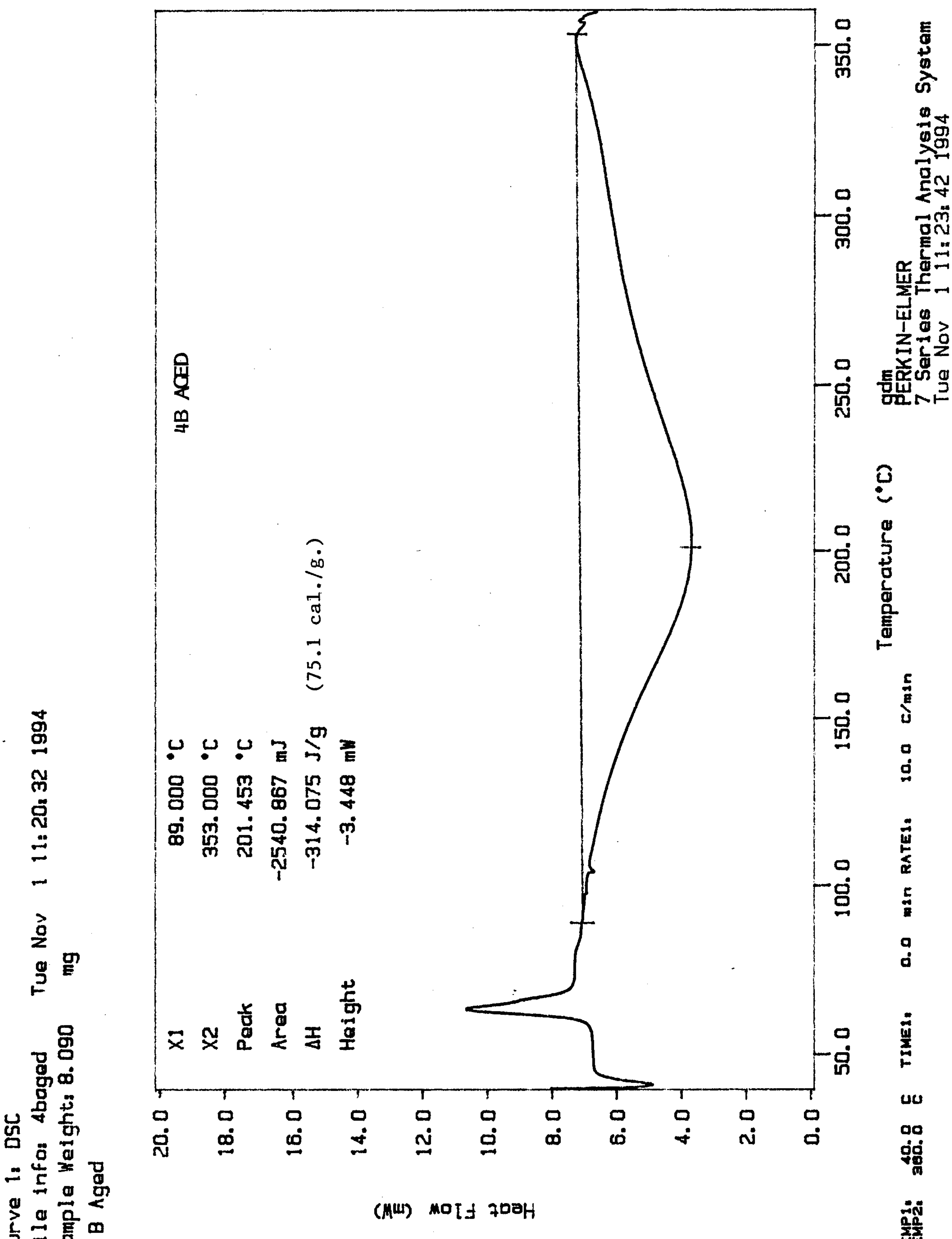


Figure 44.

SYSTEM FROM FIGURE 42 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

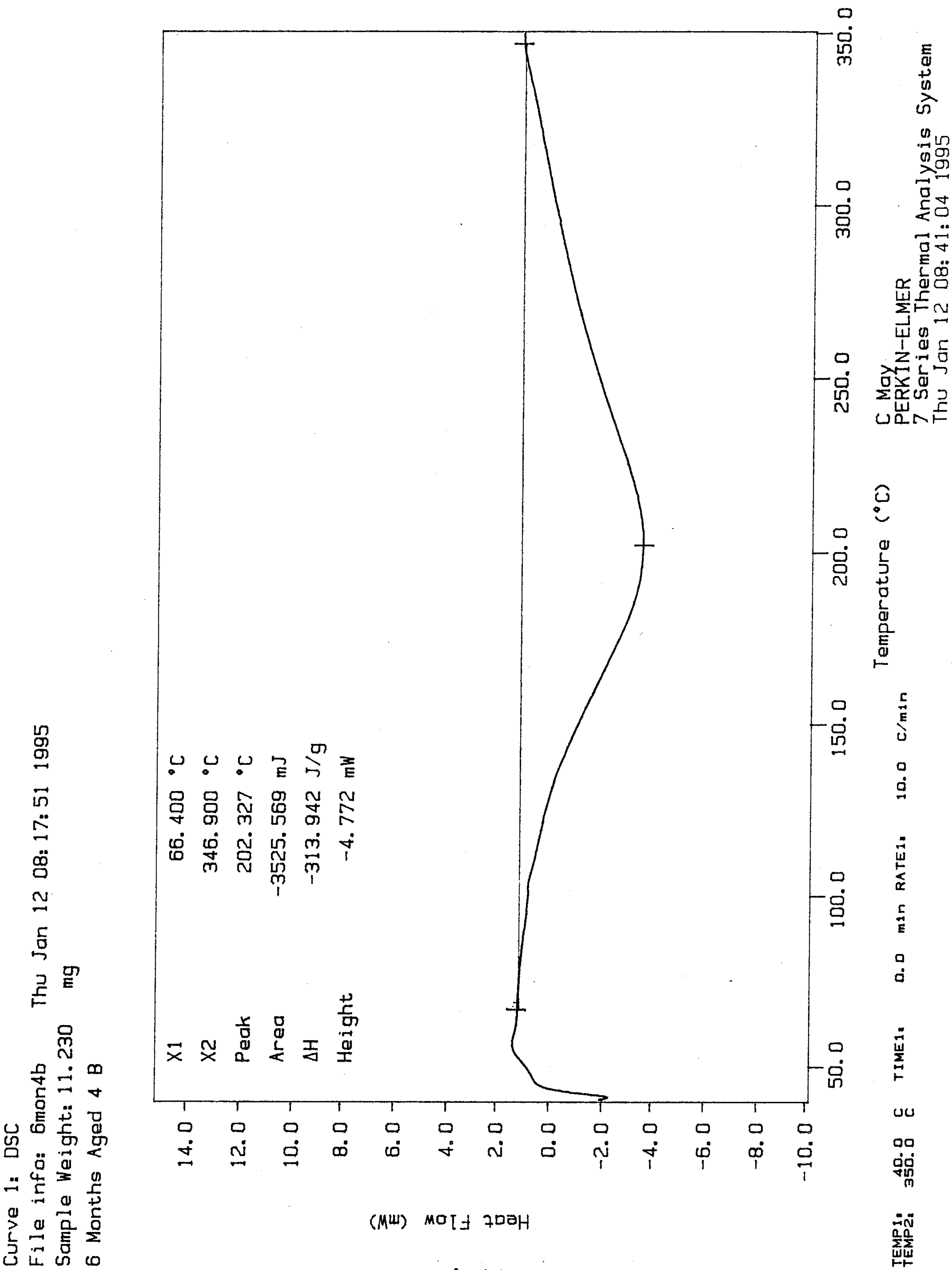


Figure 45.

TACTIX 742/ETHACURE 100 BEFORE AGING.

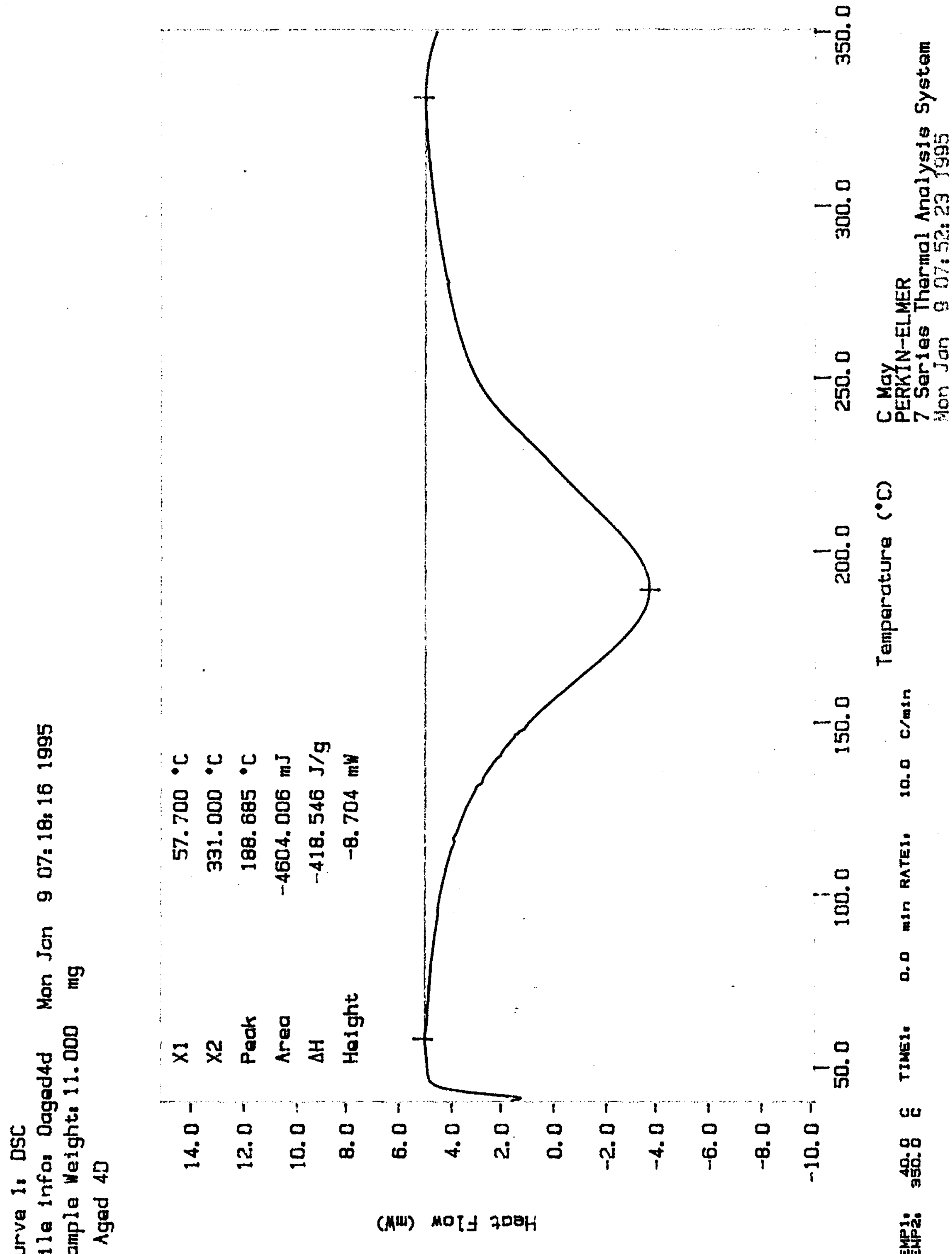


Figure 46.

System from Figure 45 After 6 Months' Room Temperature Storage.

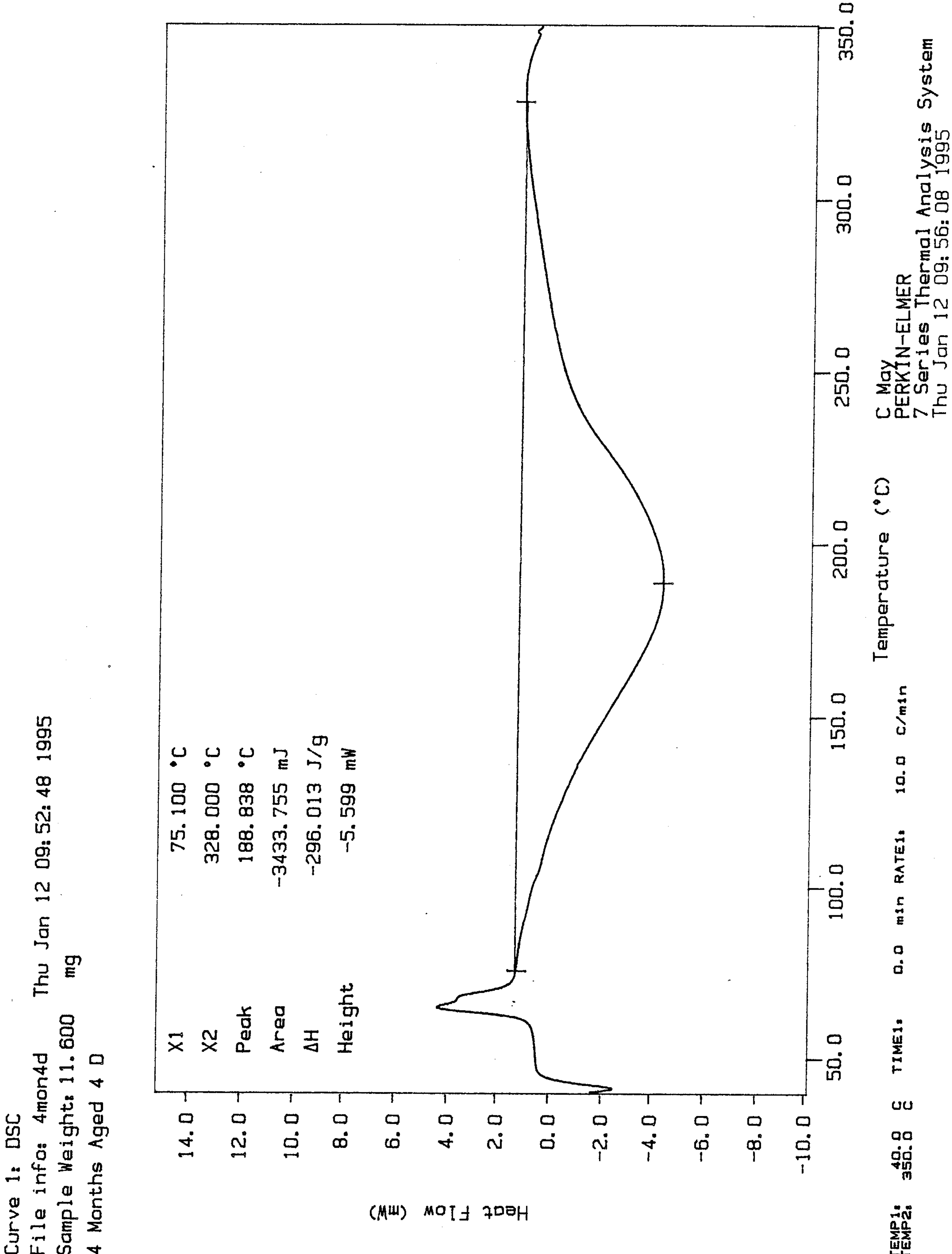


Figure 47.

EPON 1031/ETHACURE 100 BEFORE AGING.

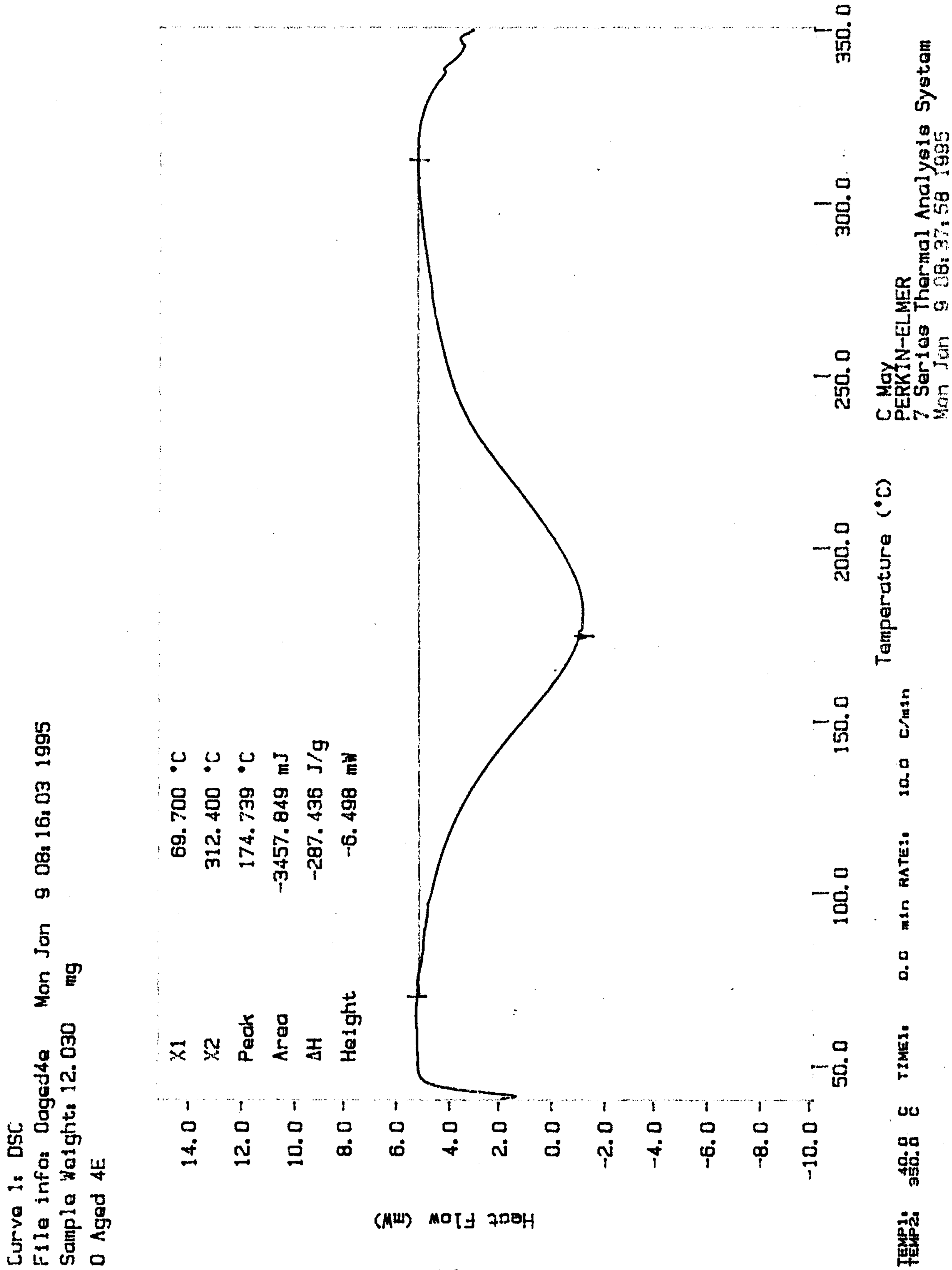


Figure 48.

SYSTEM FROM FIGURE 47 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

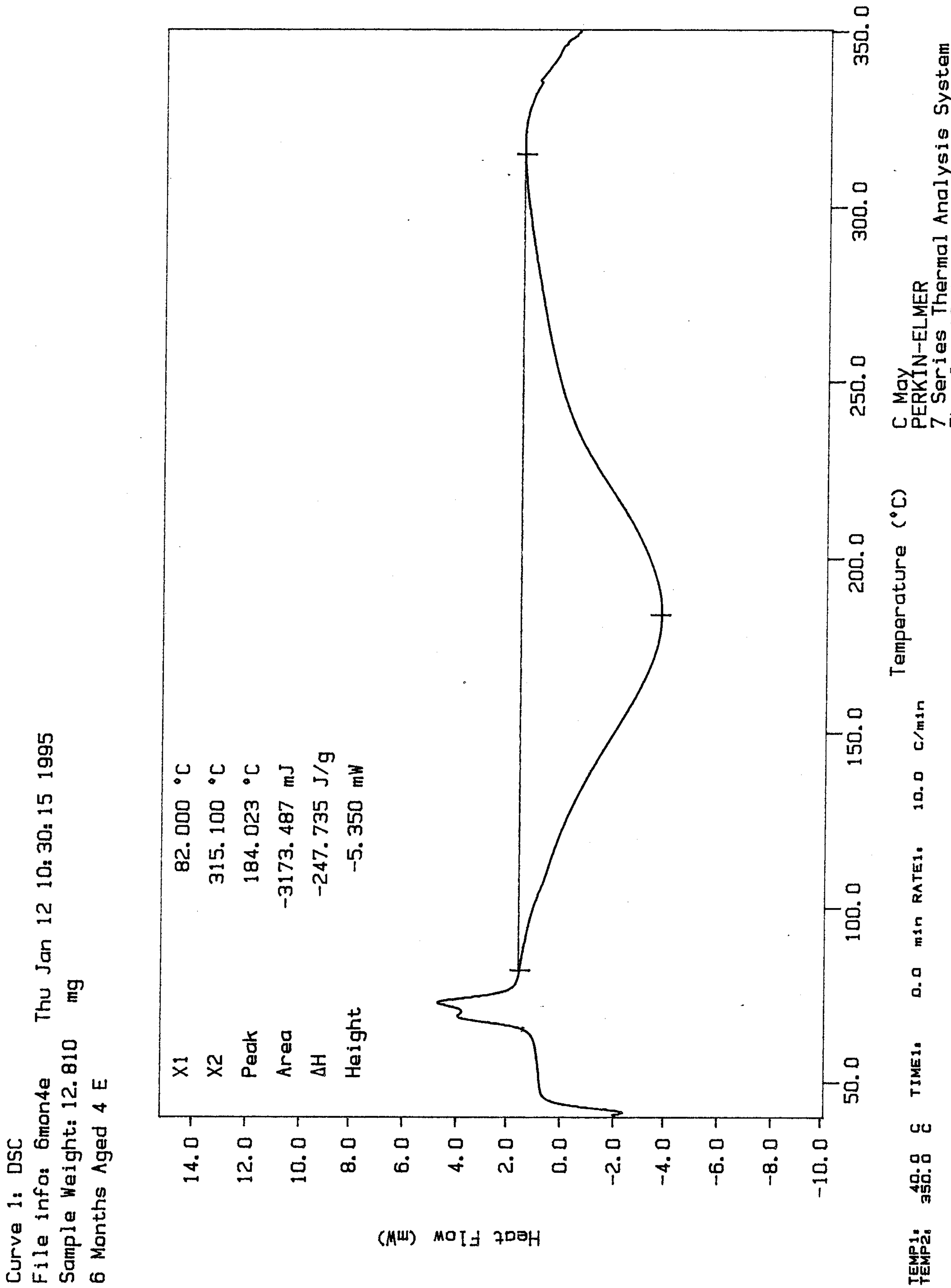


Figure 49.

DPS 164/ETHACURE 100 BEFORE AGING.

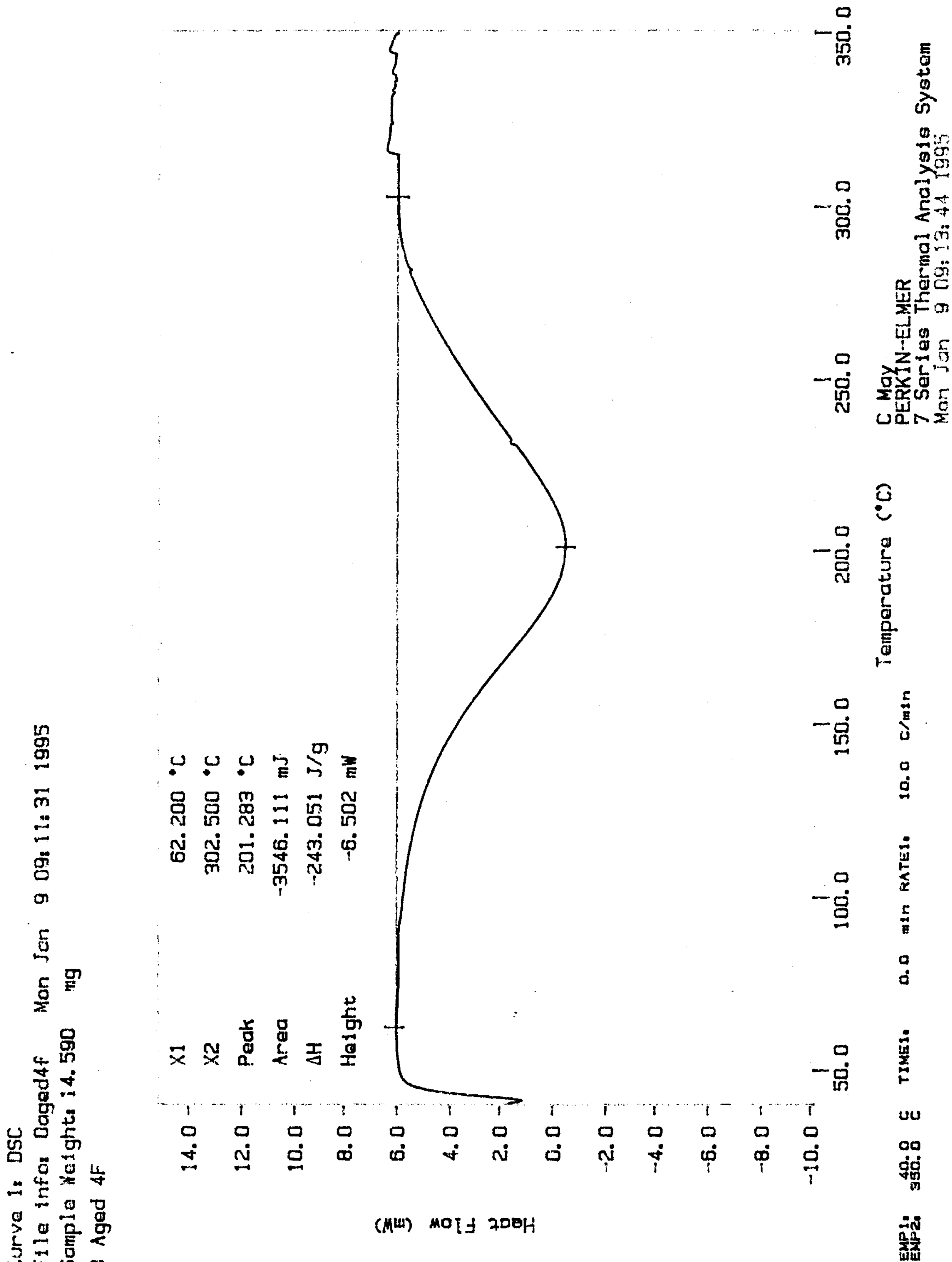


Figure 50.

SYSTEM FROM FIGURE 49 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

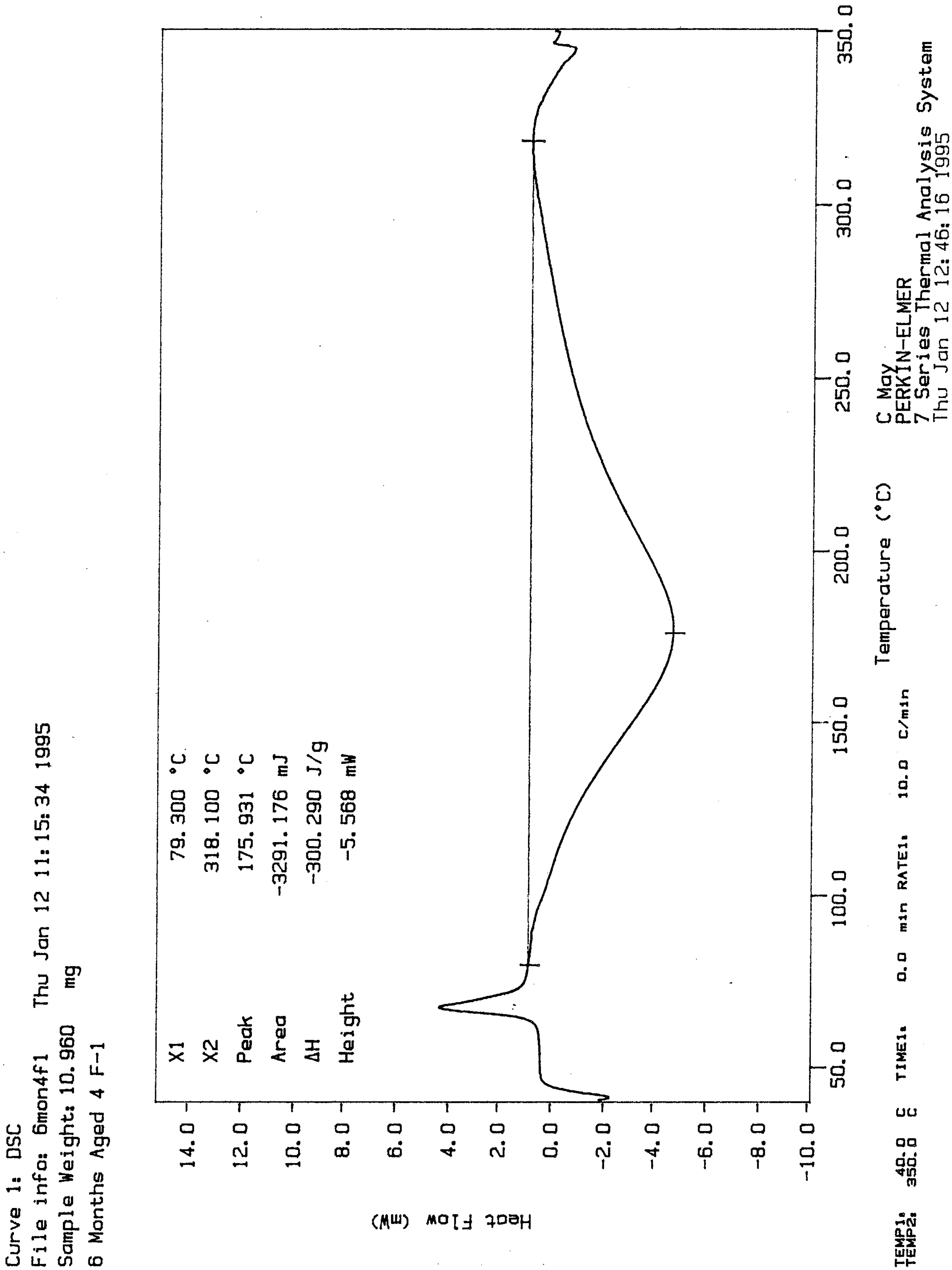


Figure 51.

ECN 1273/ETHACURE 100 BEFORE AGING.

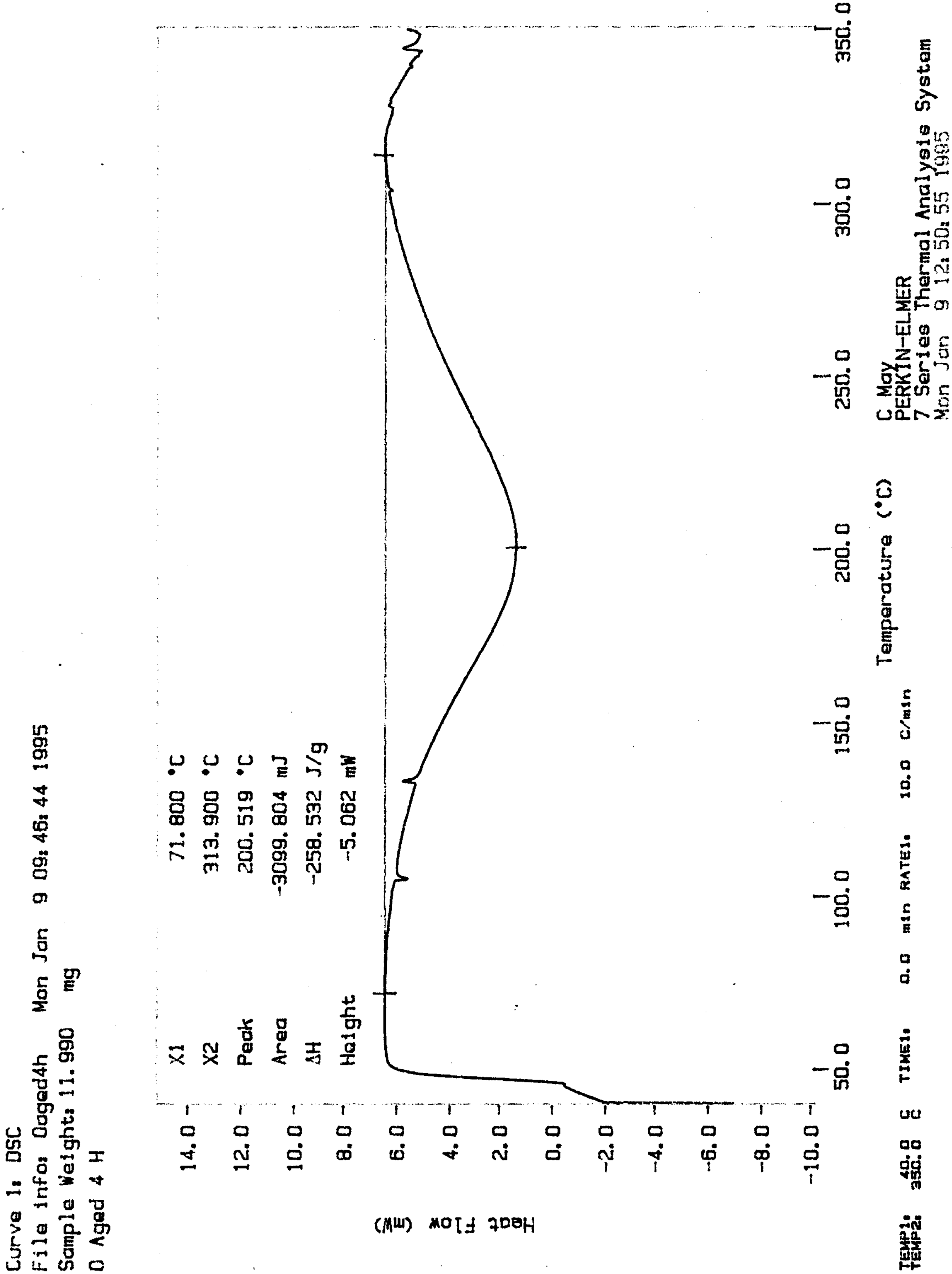


Figure 52.

SYSTEM FROM FIGURE 51 AFTER 6 MONTHS' ROOM TEMPERATURE STORAGE.

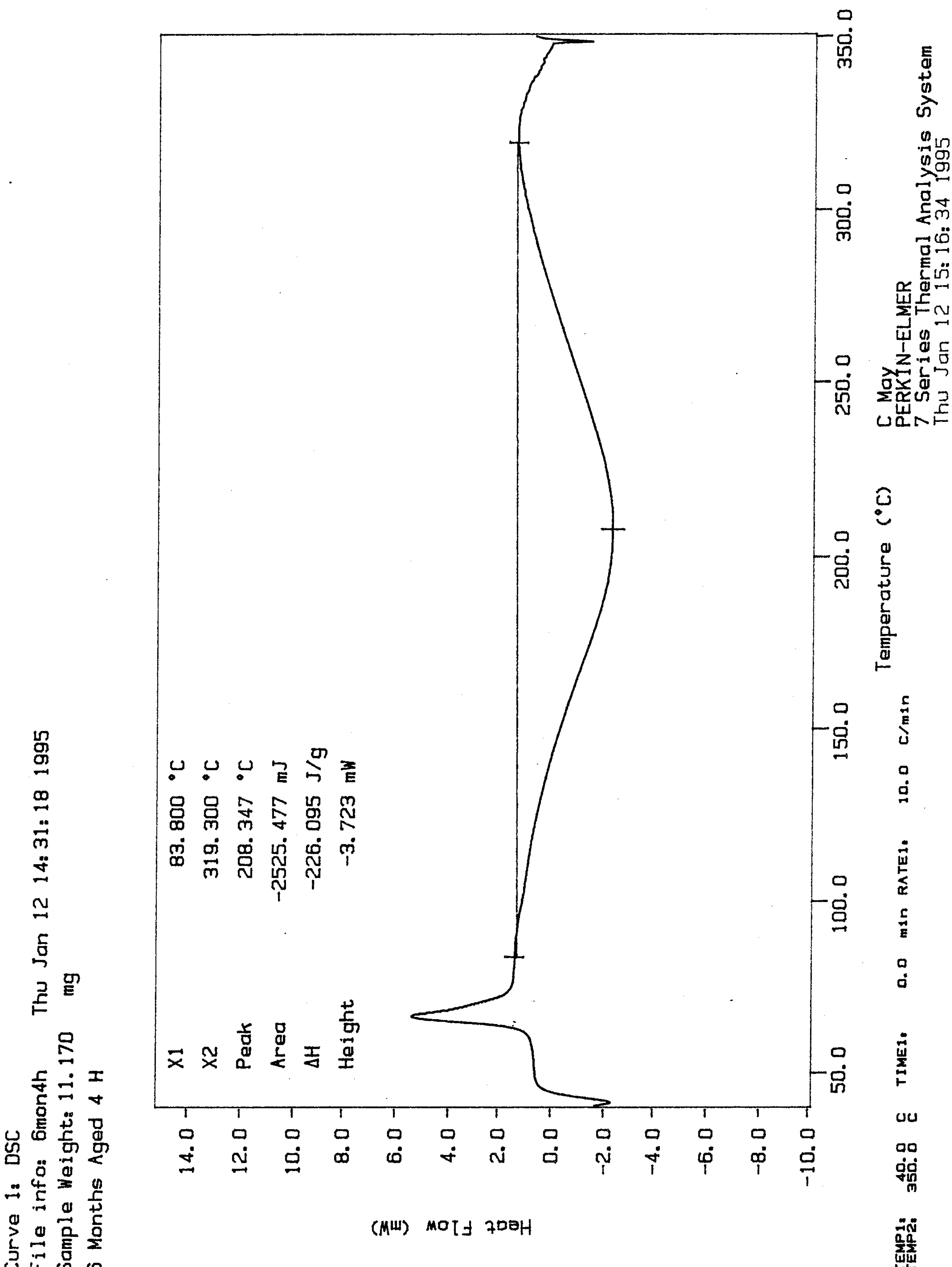


Table 1. Candidate Resins and Curing Agents

Resin System	Melting Point or Melt Viscosity	Commercial Source
EPON HPT Resin 1071	122° F[1]	Shell Chemical Co.
EPON HPT Resin 1079	176-180° F[1]	Shell Chemical Co.
EPON SU-8	82° C[2]	Shell Chemical Co.
TACTIX 742	Tacky Solid	Dow Chemical
EPON 1031	15 Poise @ 150°C[3]	Shell Chemical Co.
EPON DPS-164	82°C	Shell Chemical Co.
EPON 1050	2-500 Poise @ 52°C	Shell Chemical Co.
ECN 1273	73° C	CIBA

Curing Agent	Commercial Source
EPON HPT Curing Agent 1061	Shell Chemical Co.
m-Phenylenediamine (MPDA)	duPont
Diaminodiphenylsulfone (DADS)	CIBA
Ethacure 100	Ethyl Corporation

1 ASTM D-3461, Mettler, 1°C/minute
2 Durrans
3 Room Temperature Solid

Table 2. Gel Times as Days of Room Temperature Storage

(Times in seconds)

Resin (WPE)		HPT 1071 (160)	HPT 1079 (255)	SU-8 (215)	TCTX 742 (160)	EPON 1031 (213)	EPS 164 (220)	EPON 1050 (177)	ECN 1273 (225)
Curing Agent									
HPT 1061	0	438	260	43	80	55	101	55	85
(86)	1	385	247	41	70	53	101	42	75
(Amine Equivalent)	3	347	255	30	56	58	85	39	71
	7	194	241	24	53	52	83	26	49
	14	175	234	15	44	48	72	19	41
	30	171	250	GEL	44	45	67	11	32
	60	135	250	GEL	43	40	56	GEL	31
	90	123	233	GEL	39	34	46	GEL	20
	180	113	190	GEL	33	28	44	GEL	5
MPDA	0	142	112	GEL	40	23	28	17	26
(27)	1	51	85	GEL	21	20	22	12	18
	3	31	78	GEL	16	14	20	5	16
	7	27	73	GEL	14	17	14	GEL	1
	14	23	71	GEL	5	14	10	GEL	GEL
	30	20	75	GEL	1	12	GEL	GEL	GEL
	60	GEL	73	GEL	GEL	GEL	GEL	GEL	GEL
	90	GEL	51	GEL	GEL	GEL	GEL	GEL	GEL
	180	GEL	58	GEL	GEL	GEL	GEL	GEL	GEL
DADS	0	2355	1968	635	860	852	791	883	1168
(55)	1	2360	1959	546	866	716	890	846	1105
	3	2334	1914	534	819	707	1032	863	1123
	7	2334	1914	534	819	707	1032	863	1123
	7	2403	1890	535	806	682	1007	849	1060
	14	2433	1885	489	739	720	964	740	951
	30	2311	1864	480	563	673	925	584	916
	60	2313	1976	421	637	687	830	401	708
	90	2330	2004	474	633	661	888	294	735
	180	2232	1940	347	568	591	852	313	707
ETHACURE 100	0	1350	740	195	400	182	310	312	307
(44.5)	1	1606	732	108	330	153	218	218	441
	3	1550	667	48	272	129	305	123	180
	7	1400	612	36	118	99	147	52	101
	14	714	603	26	106	88	114	18	76
	30	404	607	20	102	91	110	15	69
	60	330	585	GEL	80	92	89	GEL	42
	90	299	540	GEL	77	89	78	GEL	34
	180	262	535	GEL	77	78	68	GEL	24

Table 3. Summary of Differential Scanning Calorimetry Studies.

Taken at Initial Mixing,
90 Days at Room Temperatures from 65-105 degrees F,
and 180 Days at Room Temperatures from 55-105 degrees F

Expressed in Calories/Gram (Joules/Gram)

RESIN/ CURING AGENT	Initial Reactivity	After 3 Mos.	% Reacted	After 6 Mos.	% Reacted
HPT 1071/ HPT 1061	95.9 (401.5)	66.9 (280.3)	30.2	70.8 (296.6)	26.2
HPT 1079/ HPT 1061	69.1 (289.3)	61.3 (256.6)	11.3	59.6 (249.8)	13.7
TACTIX 742/ HPT 1061	56.2 (235.3)	* *	*	52.3 (218.9)	6.9
EPON 1031/ HPT 1061	46.8 (196.0)	* *	*	40.2 (168.3)	14.5
DPS 164/ HPT 1061	74.4 (311.8)	* *	*	45.2 (189.5)	39.2
ECN 1273/ HPT 1061	74.1 (310.4)	* *	*	51.2 (214.6)	30.9
HPT 1079/ MPDA	69.3 (290.4)	* *	*	52.1 (218.6)	24.7
HPT 1071/ DADS	132.6 (555.2)	124.4 (512.2)	6.2	133.1 (557.6)	0.0
HPT 1079/ DADS	72.6 (303.0)	61.6 (257.9)	15.2	72.8 (304.8)	0.0
SU-8/ DADS	69.7 (291.9)	* *	*	63.1 (264.2)	9.5

(Continued)

Table 3. Summary of Differential Scanning Calorimetry Studies, Page 2.

RESIN/ CURING AGENT	Initial Reactivity	After 3 Mos.	% Reacted	After 6 Mos.	% Reacted
TACTIX 742/ DADS	91.8 (384.6)	* *	*	84.8 (355.4)	7.6
EPON 1031/ DADS	69.4 (290.6)	* *	*	64.0 (268.1)	7.7
DPS 164/ DADS	55.1 (230.6)	51.5 (215.7)	6.5	57.6 (241.4)	0.0
EPON 1050/ DADS	86.4 (361.8)	* *	*	64.3 (260.2)	25.8
ECN 1273/ DADS	52.2 (218.8)	* *	*	41.2 (172.6)	21.3
HPT 1071/ ETHACURE 100	114.0 (477.5)	* *	*	65.5 (274.3)	42.6
HPT 1079/ ETHACURE 100	91.9 (384.6)	75.1 (314.1)	18.2	74.9 (313.9)	18.5
TACTIX 742/ ETHACURE 100	99.9 (418.6)	* *	*	70.7 (296.0)	29.3
EPON 1031/ ETHACURE 100	68.6 (287.4)	* *	*	59.1 (247.7)	13.8
DPS 164/ ETHACURE 100	71.7 (300.3)	* *	*	58.0 (243.1)	19.1
ECN 1273/ ETHACURE 100	61.7 (258.5)	* *	*	54.0 (226.1)	12.5

* Not Tested

APPENDIX B

SC-522-88
(Supercedes SC:522-87)

Technical Bulletin
Shell Chemical Company

EPON HPT™ Curing Agent 1061-M

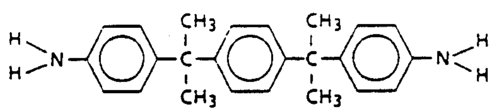
Epoxy curing agent for high performance matrix resin system in advanced composites and adhesives

General description

EPON HPT™ Curing Agent 1061-M is an aromatic diamine curing agent with a novel backbone for use in high performance composite applications and adhesives. The solid curing agent is supplied as a finely ground powder for ease of handling.

Chemical description

4,4'-[1,4-phenylene (1-methyl ethylidene)] bis (benzeneamine)



Advantages

- High performance aromatic amine curing agent
- Low moisture absorption
- Yields improved hot/wet performance
- Finely ground powder for ease of handling
- Rapid gel time

Applications

- Advanced composite structural formulations
- High performance structural adhesives

Typical properties

Physical form	Free flowing solid powder
Particle size	95% wt below 30 microns
Color	Tan to cream
Melting point, °F	322°-327°
Approximate equivalent weight, active hydrogen	86

Formulation/processing

Neat resin casting

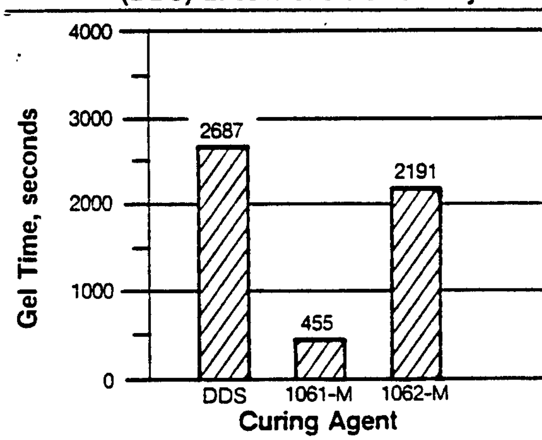
The following is a general procedure used in the laboratory for preparation of neat resin castings using Epon HPT curing agent 1061-M with multifunctional resins like EPON HPT™ Resin 1071 or TGMDA.

1. The Epon HPT resin, glycidyl ether of BPA Novolac, EPON® Resin 825, and/or triglycidyl aminophenol (TGAP) are heated in a circulating air oven to 300-325°F.
2. The Epon HPT curing agent 1061-M is melted in a second oven at 340°F.
3. Melted Epon HPT curing agent 1061-M dissolves rapidly in the resin with vigorous stirring resulting in a homogeneous resin/curing agent solution. (Caution: the gel time of Epon HPT curing agent 1061-M with these resins is rapid at 350°F, see Figure 1.)
4. The resin curing agent solution is then immediately degassed in a vacuum oven at 25 in. of Hg or less at 350°F. After degassing, the solution is ready to pour into a mold, which should be heated to about 300°F.

Gel and cure characteristics

Figure 1 shows the gel times of Epon HPT resin 1071 with Epon HPT curing agent 1061-M as compared with diaminodiphenylsulfone (DDS) and EPON HPT™ Curing Agent 1062-M. This shows the Epon HPT curing agent 1061-M gel time at 350°F is very fast compared to that of DDS and Epon HPT curing agent 1062-M.

Figure 1/**Gel times of EPON HPT™ Resin 1071 with EPON HPT™ Curing Agents 1061-M and 1062-M and diaminodiphenylsulfone (DDS) at 85% of stoichiometry**



Performance properties

Neat resin casting properties

Table 1 shows the neat resin casting properties of Epon HPT curing agent 1061-M with Epon HPT resin 1071. A comparison of performance obtained when cured with DDS is also provided. As is shown in the table, Epon HPT curing agent 1061-M gives improved retention of properties in hot/wet environments.

Table 1/**Neat resin casting properties of EPON HPT™ Resin 1071 cured with EPON HPT™ Curing Agent 1061-M and with diaminodiphenylsulfone (DDS)**

	Epon HPT resin 1071/ Epon HPT curing agent 1061-M	Epon HPT resin 1071/ DDS[1]
T_g (tan delta), °F	466	480
Moisture gain[2], %	2.1	3.6
Flexural properties (RT/dry)		
Strength, ksi	20	20
Modulus, ksi	494	563
Flexural properties (Hot/Wet)[3]		
Strength, ksi	14	13
Modulus, ksi	427	434
Retention of RT/dry properties at 200°F wet conditions,[3] %		
Strength, %	70	65
Modulus, %	86	70

[1]Cure schedule: 2 hours at 300°F; 4 hours at 392°F
[2]After 2 weeks immersion at 200°F
[3]Tested in water at 200 °F after two weeks immersion at 200°F

Packaging, storage and shipping

Epon HPT Curing Agent 1061-M is available for evaluation in 10-pound and 50-pound quantities. It is not defined as hazardous by criteria of DOT Regulations. Its shelf life is indefinite at room temperature (77°F).

For more information on EPON HPT™ Resin Systems contact:

Commercial Development Manager
Shell Chemical Company
One Shell Plaza
P. O. Box 2463
Houston, Texas 77252-2463
(713) 241-6227
(713) 241-0407

For Technical Assistance
Call toll free 1-800-TEC-EPON
In Texas call 1-800-222-EPON

Use and handling information for systems based on Epon HPT curing agent 1061-M

The recommendations for material selections made in this bulletin are based on Shell's experience and research and are believed to be sound technical approaches to the applications or end uses for which they are presented. However, these recommendations are directed solely toward technical performance and should not be taken as recommendations pertaining to health, safety or the environment.

Use of Epon HPT curing agent 1061-M is regulated by EPA under a TSCA Section 5(e) Consent Order. You will be notified by registered mail of the specific terms of the Order prior to the distribution of product to you. The requirements include use of personal protective equipment, use only at facilities under your control, use only as a component of prepregs, composites or adhesives for industrial applications, and no further distribution of the product before it has been formulated.

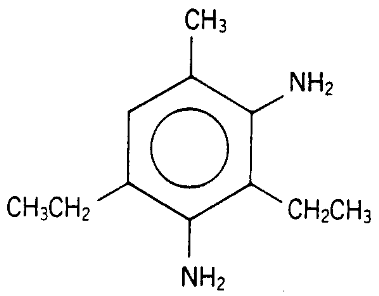
Epon HPT curing agent 1061-M and the auxiliary materials normally combined with this product are capable of producing adverse health effects ranging from minor skin irritation to serious systemic effects. Adverse effects can be minimized and most can be avoided through the observance of proper precautions, use of proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these depends on responsible action by adequately informed personnel.

A Material Safety Data Sheet (MSDS) is available for this product (MSDS # 690). **Transportation, storage, handling and use of this product should not occur until handling precautions and recommendations, as described in the MSDS, are understood by all persons who will work with it.** Questions and requests for MSDS sheets or other information should be directed to your Shell Chemical Company Sales Office. Information on non-Shell products should be obtained from the respective manufacturer or vendor.

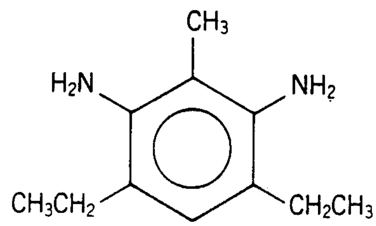
Printed in U.S.A.
February 1988
5M

Description

ETHACURE 100 curing agent is Diethyltoluenediamine (DETDA). It has the following composition and properties.



(2, 4 isomer)



(2, 6 isomer)

SPECIFICATIONS

1. Water, max.	0.08 wt %
2. Amine Number	628-635
3. 3, 5-Diethyltoluene-2, 4-diamine	74-80 wt %
4. 3, 5-Diethyltoluene-2, 6-diamine	18-24 wt %
5. 2, 4, 6-Triethylbenzene-1, 3-diamine, max.	1 wt %
6. Other polyalkylated-m-phenylenediamines	0-4 wt %
7. Appearance	Clear liquid

PHYSICAL PROPERTIES (TYPICAL)

Appearance	clear, amber liquid
Molecular weight	178.28
Boiling point, °C	308
Density at 20°C, g/ml	1.022
lbs/gal	8.50
Pour point, °C	−9
°F	15
Flash point, TCC, °C	>135
°F	>275
Viscosity, cs at 20°C	326
Solubility, (20°C), Ethanol	miscible
Toluene	miscible
Water, wt %	1.0

Uses

Polyurethane Chain Extender

ETHACURE 100 curing agent is a liquid aromatic diamine which is used as a chain extender for polyurethane resins, especially for reaction injection molded (RIM) elastomers (1-6). Additional polyurethane applications are for use in rigid foams (7), coatings (8), gaskets, sealants, encapsulation and other applications (9-12). Typical of aromatic diamines, physical properties of systems extended with ETHACURE 100 curing agent are very good. In general, compared with diol extended systems, ETHACURE 100 curative yields elastomers with:

- Higher modulus
- Increased hardness
- Greater compression set
- Good tensile strength
- High resistance to tear and abrasion

In some systems, ETHACURE 100 curing agent is less sensitive to stoichiometry, resulting in a larger processing window. Because it is more reactive than diols, a catalyst may not be required.

Epoxy Resin Curing Agent

ETHACURE 100 curing agent is a nonstaining high performance curing agent for epoxy resins (13-17). As a low viscosity liquid it is easy to mix with epoxy resins. Pot life is good and the exotherm during cure is low which allows the casting of large parts. The cured materials exhibit high deflection temperatures, excellent chemical resistance, and good retention of mechanical properties at elevated temperatures. Suggested applications are electrical encapsulation, filament winding, pre-pregs, tooling, potting and casting, and laminates.

Other Uses

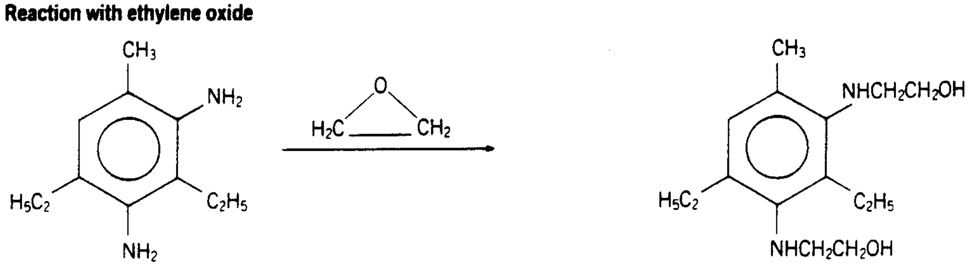
Other suggested uses are as a lube and fuel antioxidant (18), as a comonomer for polyimides, polyamides, or polyesterimides (for magnet wire enamels), and as a replacement for methylene dianiline (MDA), metaphenylenediamine (MPDA), and methylenebis-ortho-chloroaniline (MOCA).

Chemical Reactions

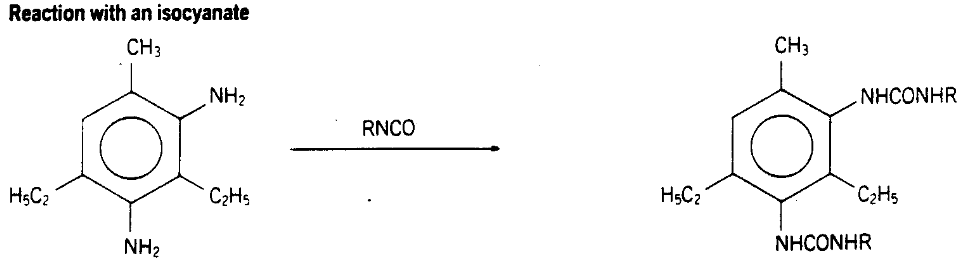
Because ETHACURE 100 curing agent is an aromatic diamine with reactive primary amine groups, it will undergo typical diamine reactions. For illustration purposes, only the 2, 4-isomer is depicted in each of the following examples; however, the 2, 6-isomer as well as other diamines will react similarly.

ETHACURE 100 Curing Agent	***Reactant***	***Product***

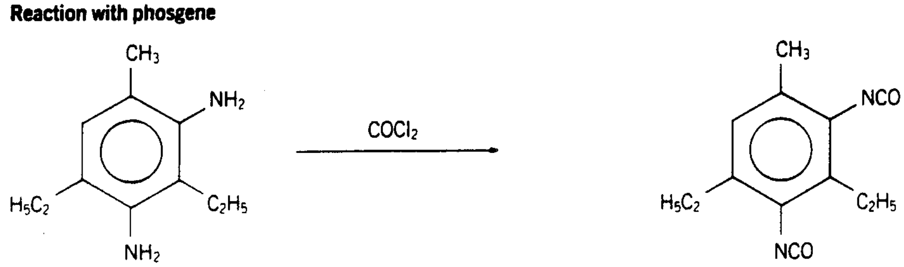
Reaction with ethylene oxide



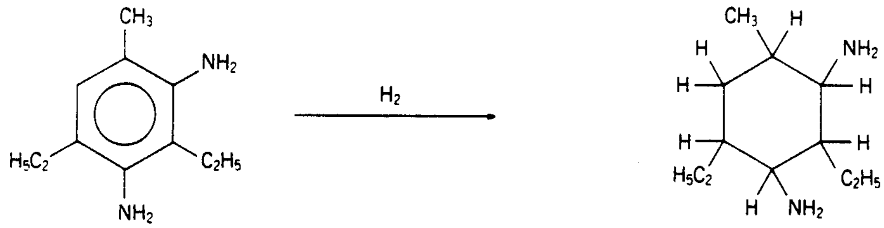
Reaction with an isocyanate



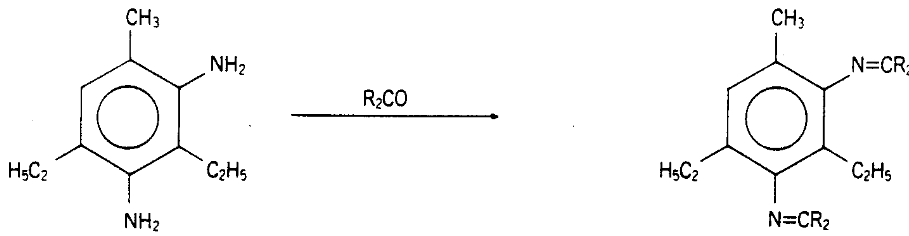
Reaction with phosgene



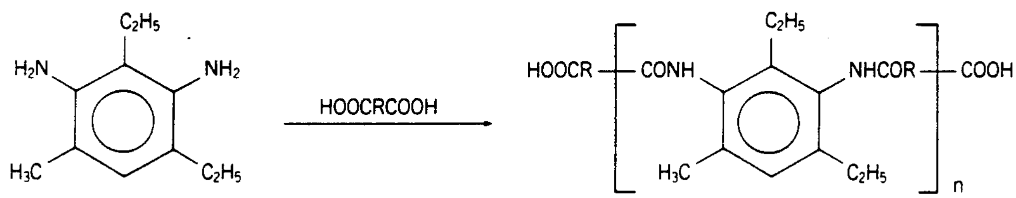
Hydrogenation



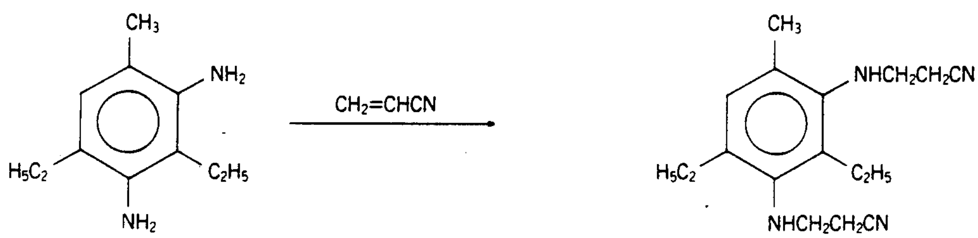
Reaction with ketones and aldehydes



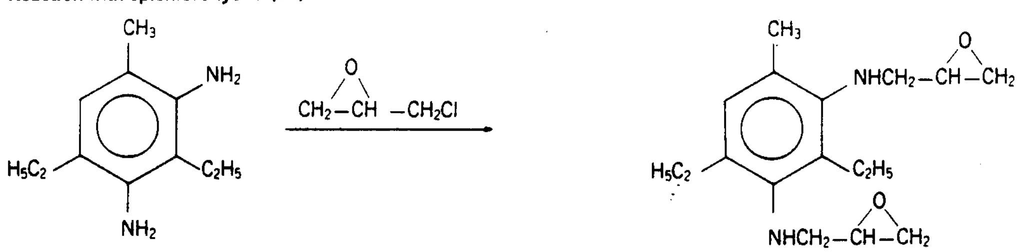
Reaction with dicarboxylic acid



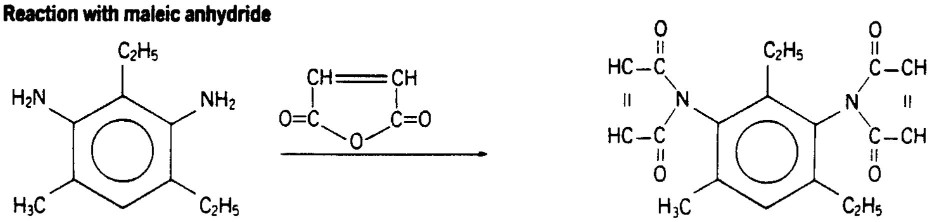
Reaction with acrylonitrile



Reaction with epichlorohydrin (19)



Reaction with maleic anhydride



References

1. Weber, C. and Schafer, H., U.S. Patent 4,218,543 (1980).
2. Ewen, J.H., McClellan, T.R., McMillin, M.H. and Murray, P.L., U.S. Patent 4,374,210 (1983).
3. Dominguez, R.J.G., Rice, D.M. and Lloyd, R.F., U.S. Patent 4,396,729 (1983).
4. Rice, D.M. and Dominguez, R.J.G., U.S. Patent 4,433,067 (1984).
5. Casey, J.P., Milligan, B. and Fasolka, M.J., "Chain Extender Structure-Activity Relationships," Proceedings SPI Annual Technical/Marketing Conf., 28th Polyurethane Meeting, 218-23, 1984.
6. Nissen, D. and Markovs, R.A., "Aromatic Diamines as Chain Extender in RIM Urethane Elastomers," SPI Proceedings, October 1982, p. 71.
7. Wiedermann, R., Blahak, J., and Prager, F.H., U.S. Patent 3,909,465 (1975).
8. Gruber, H. and Wellner, W., U.S. Patent 4,463,126 (1984).
9. Robinson, I.M., U.S. Patent 4,463,141 (1984).
10. Chung, D.A., U.S. Patent 4,487,913 (1984).
11. Schmidt, A.R., Strohmayer, H.F., Sibral, W. and Milligan, B., U.S. Patent 4,507,459 (1985).
12. Chang, E.Y. and Saxon, R., "Novel Cast Polyurethane Elastomers Based on p-TMXDI, An Aliphatic Diisocyanate," Elastomerics, June 1985, p. 18.
13. Andres, K.H. and Hahn, W., GB Patent 1,034,790 (1966).
14. Urech, K., Habermeier, J. and Moser, R., U.S. Patent 4,366,108 (1982).
15. Shimp, D.A., U.S. Patent 4,447,586 (1984).
16. Wiggins, P.L., "Diethyltoluenediamine, A Liquid Aromatic Diamine," SPI Meeting, Epoxy Resin Formulaters Division, Fall 1983, Atlanta, Georgia.
17. Henry, R.D., U.S. Patent 4,528,305.
18. Wright, W.E., U.S. Patent 4,456,541 (1984).
19. Shimp, D.A. and Graver, R.B., U.S. Patent 4,487,948.

Special Chemicals
Division, Ethyl Corporation
Ethyl Tower
451 Florida Street
Baton Rouge, Louisiana 70801
Telephone: 504/388-7899
Telex 586441

Ethyl Chemicals Group

SC-31R (188)

ETHYL FSC

Printed in U.S.A.

Curing Agent for Epoxy Resins

General Description

ETHACURE® 100 curing agent is a clear, light-colored, low-viscosity liquid aromatic diamine which is used as a high performance curing agent for epoxy resins. It does not contain MPDA (metaphenylenediamine) nor MDA (methylenedianiline) and does not have the staining characteristics of MPDA nor the toxicological concerns associated with MDA. As a low viscosity liquid it is easy to mix with epoxy resins. The resulting mixture will have a longer pot life than systems containing MPDA or MDA. Exotherm during the cure is low and allows the casting of large parts. The cured materials will exhibit high deflection temperatures, excellent chemical resistance, and good retention of mechanical properties at elevated temperatures.

Chemical Description

Diethyltoluenediamines (DETDA)

Suggested Uses

Filament winding
Electrical encapsulation
Pre-pregs
Tooling
Potting and casting
Laminating
Coating
Molding
Adhesive

Advantages

Low viscosity liquid
Toxicologically safer aromatic diamine
Low exotherm
Long pot life
Excellent chemical resistance
Good mechanical properties
Property retention at elevated temperatures
Good electrical properties

Ethyl Chemicals Group

Physical Properties

Boiling point, °C	308
Density at 20°C, g/mL	1.022
68°F, lb/gal	8.50
Pour point, °C	−9
°F	15
Flash point, TCC, °C	>135
°F	>275
Viscosity, cs at 20°C	326
Equivalent weight, g/eq	44.6

*Starting Formulation**

The stoichiometric amount of ETHACURE 100 to be used with an epoxy resin can be easily calculated by dividing the equivalent weight of ETHACURE 100 by the WPE (weight per epoxide) or the equivalent weight of the epoxy resin. The calculation of the amount of ETHACURE 100 to be used with the standard epoxy resin (i.e., the diglycidyl ether of bisphenol A) is shown below:

$$\frac{\text{equivalent weight of ETHACURE 100}}{\text{WPE}} \times 100 = \frac{44.6 \text{ g/eq}}{189 \text{ g/eq}} \times 100 = 23.6 \text{ phr}$$

*phr — parts by weight per one hundred parts by weight of resin.

The stoichiometric amount of ETHACURE 100 should be used as a starting formulation. A deviation from stoichiometry is not necessarily detrimental to the performance of the end product and can be beneficial in some cases. A specific property can be maximized by varying the concentration of ETHACURE 100 in the epoxy resin and measuring the desired property. An example of this is shown in the section entitled, "Optimum Concentration of ETHACURE 100 to Maximize Deflection Temperature".

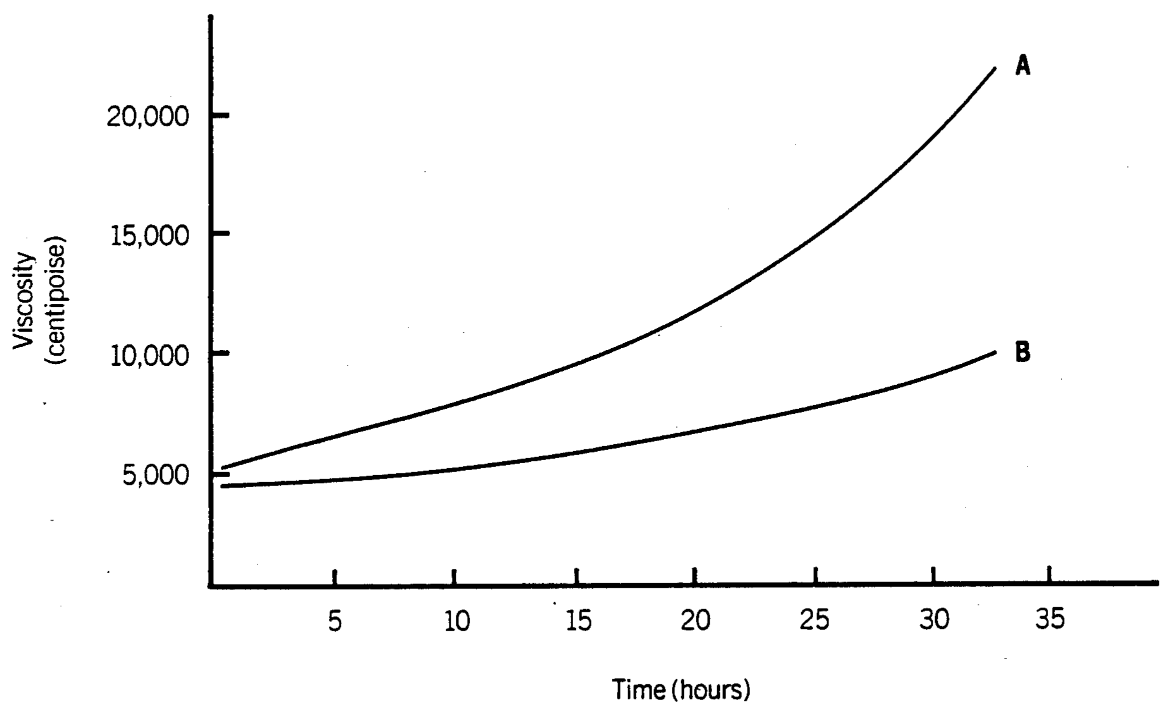
*Unless otherwise noted, all of the data in this bulletin is based on work with the diglycidyl ether of bisphenol A.

Pot Life

The pot life of ETHACURE 100/epoxy blends is greater than the pot life of typical aromatic amine/epoxy blends. ETHACURE 100 can be readily blended with liquid epoxy resins at room temperature to provide a low viscosity blend. The buildup of viscosity for an ETHACURE 100/epoxy system is very slow and provides a long working life. The viscosity of a 500 g mixture at 25.0°C doubled in 18 hours and 29 hours when the epoxy resin had a WPE of 189, and 183, respectively.

Viscosity Buildup at 25.0°C

FORMULATION:	**A**	Epoxy resin (WPE = 189), phr	100
		ETHACURE 100, phr	24
	B	Epoxy resin (WPE = 183), phr	100
		ETHACURE 100, phr	25.5



Mechanical Properties

When an epoxy resin is cured with ETHACURE 100, the resulting unfilled casting has outstanding mechanical property values of tensile strength, flexural strength, and deflection temperature. The values obtained were equivalent to those obtained with an epoxy resin cured with MDA and MDA-based curing agent.

Mechanical Properties of Unfilled Castings

FORMULATION: Epoxy resin (WPE = 189), phr	100	100
ETHACURE 100, phr	24.4	—
MDA, phr	—	26.5
CURE CYCLE: 2 hr at 100°C and 4 hr at 175°C		
MECHANICAL PROPERTIES:		
Specific gravity	1.166	1.193
Tensile strength, psi	11,000	10,400
Tensile modulus, psi	380,000	420,000
Elongation, %	4.9	4.5
Flexural strength, psi	18,000	17,000
Flexural modulus, psi	390,000	420,000
Izod impact, ft-lb/in	0.4	0.4
Deflection temperature, °C	169	160
Water boil, % wt gain	1.1	1.0

Mechanical Properties at Elevated Temperatures

An epoxy resin cured with ETHACURE 100 is suitable for applications at elevated temperatures because the system retains its strength when heated. Strength values and elongation were measured at 23°C and 100°C to get an indication of how an ETHACURE 100/epoxy resin system might be expected to perform at elevated temperatures. The values at 100°C showed only small losses compared to the values at 23°C, demonstrating that systems cured with ETHACURE 100 hardener have a high level of strength retention with increasing temperature in this range.

Effect of Temperature on Mechanical Properties

FORMULATION: Epoxy resin (WPE = 183), phr	100	
ETHACURE 100, phr	25.5	
CURE CYCLE: 2 hr at 100°C and 4 hr at 175°C		
Test temperature	23°C (73°F)	100°C (212°F)
Tensile strength, psi	10,000	8,000
Tensile modulus, psi	360,000	290,000
Elongation, %	4.4	5.4
Flexural strength, psi	17,000	13,000
Flexural modulus, psi	370,000	280,000

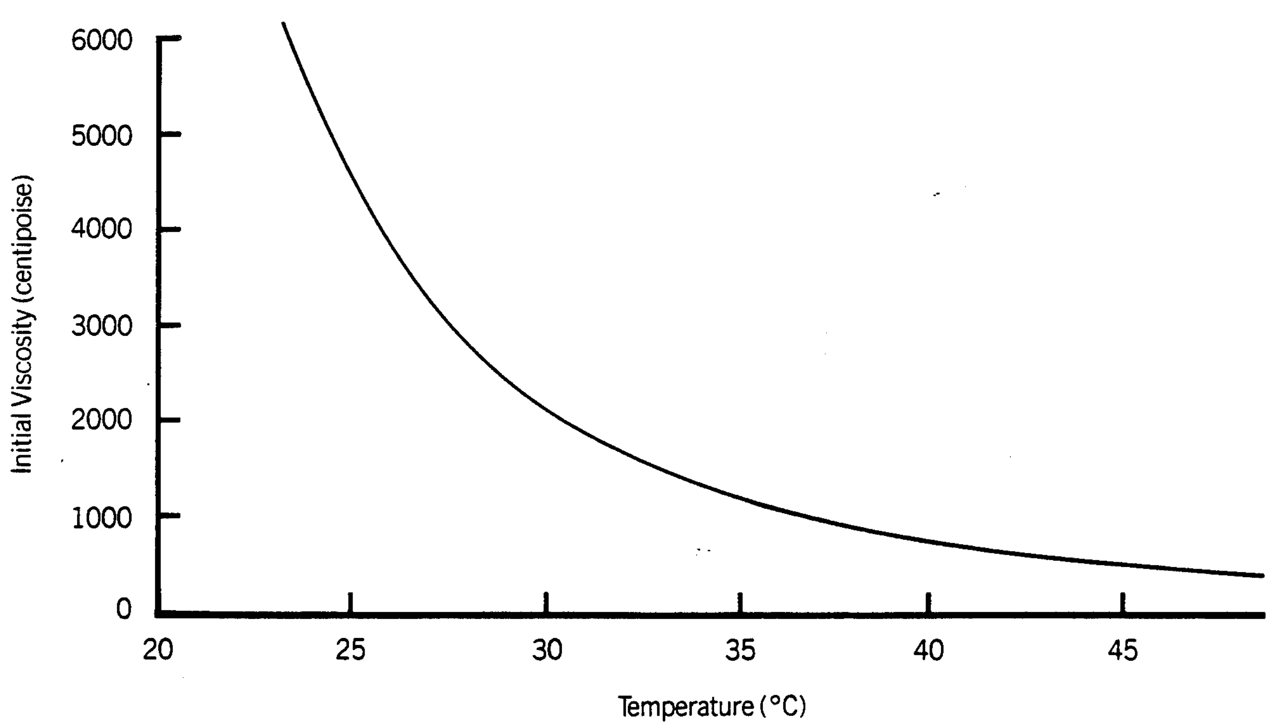
Initial Viscosity

ETHACURE 100 can be readily blended with epoxy resins even at room temperature since it is a low viscosity liquid at ambient temperature. The initial viscosity of the blend will decrease as the temperature is increased.

Initial Viscosity vs. Temperature

FORMULATION: Epoxy resin (WPE = 183), phr 100
ETHACURE 100, phr 25.5

Temperature °C	(°F)	Viscosity (centipoise)
22.9	(73)	5830
29.4	(85)	2280
33.8	(93)	1260
38.7	(102)	730
42.7	(109)	470
45.2	(113)	370
48.6	(119)	310

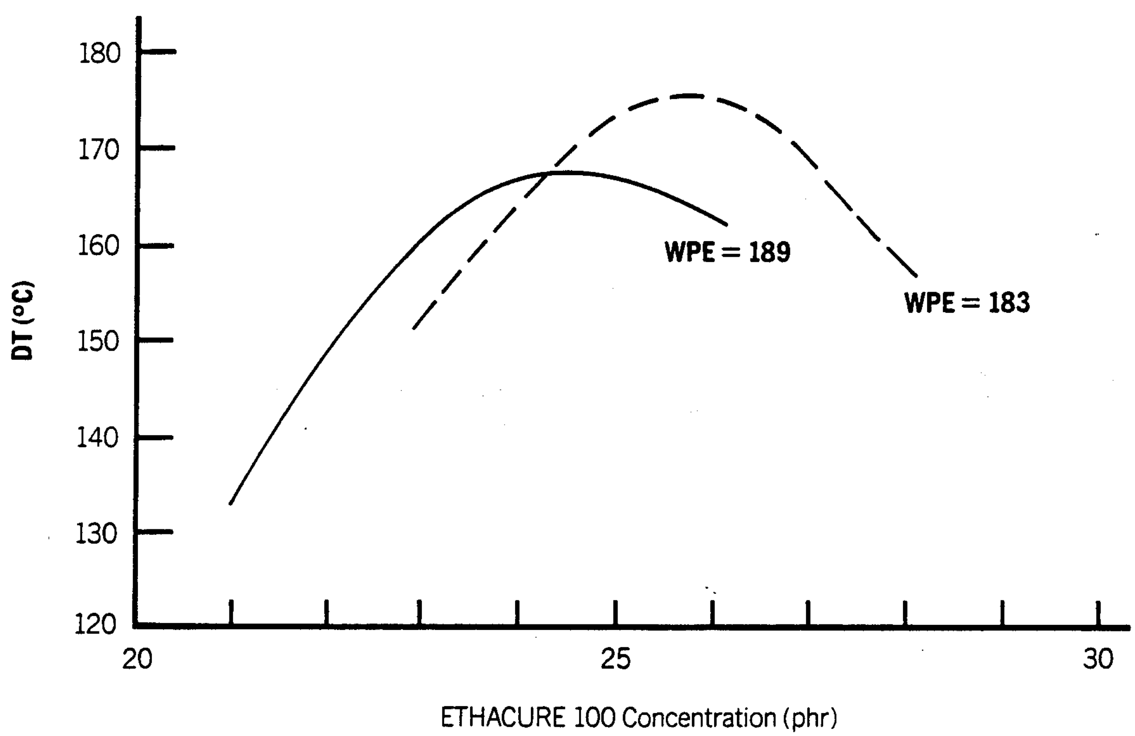


Optimum Concentration of ETHACURE 100 to Maximize Deflection Temperature

The optimum concentration of ETHACURE 100 is 25.5 phr for an epoxy resin with a W P E = 183, and 24.0 phr for an epoxy resin with a W P E = 189. The optimum DT (deflection temperature) will be obtained using the recommended concentrations and the cure cycle of 2 hr at 100°C and 4 hr at 175°C.

ETHACURE 100 Concentration Optimization

WPE = 183			WPE = 189		
phr	DT, °C	(°F)	phr	DT, °C	(°F)
28	157	(315)	26	163	(325)
27	165	(329)	25	167	(333)
26	175	(347)	24	168	(334)
25	173	(343)	23	160	(320)
24	162	(324)	22	146	(295)
23	151	(304)	21	133	(271)



Chemical Resistance

The 24 hr water boil test (ASTM 0543-67) has been performed on an epoxy resin-ETHACURE 100 casting to assess chemical resistance. Flexural strength after the water boil test was very good indicating a good retention of properties.

Chemical Resistance

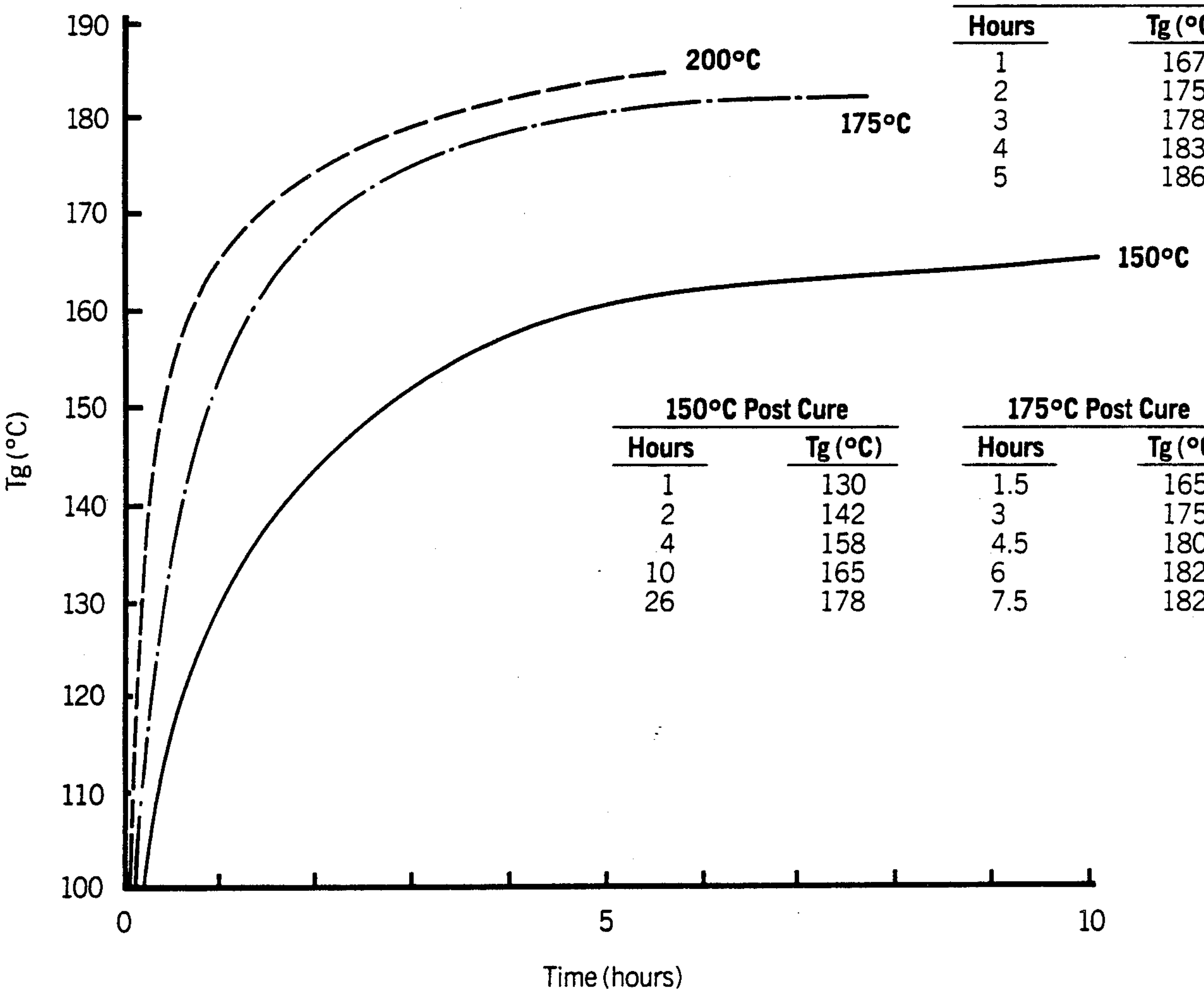
FORMULATION:	Epoxy resin (WPE = 189), phr	100	100
	ETHACURE 100, phr	24	—
	Curing agent Y, phr	—	25
CURE CYCLE:	2 hr at 100°C and 4 hr at 175°C		
CHEMICAL RESISTANCE:			
	3 hr acetone boil		
	% weight gain	1.6	0.5
	% flexural strength retention	90	—
	% flexural modulus retention	85	—
	24 hr water boil		
	% weight gain	1.4	1.1
	% flexural strength retention	92	—
	% flexural modulus retention	93	—

Effect of Post Cure Temperature and Time on Glass Transition Temperature

High Tg's (glass transition temperatures) and good retention of properties at elevated temperatures will be obtained after an adequate post cure at elevated temperatures. Samples were gelled at 100°C, and post-cured at 150°C, 175°C, and 200°C.

Effect of Post Cure on Tg

FORMULATION:	Epoxy resin (WPE = 189), phr	100
	ETHACURE 100, phr	24



200°C Post Cure

Hours	Tg (°C)
1	167
2	175
3	178
4	183
5	186

150°C Post Cure

Hours	Tg (°C)
1	130
2	142
4	158
10	165
26	178

175°C Post Cure

Hours	Tg (°C)
1.5	165
3	175
4.5	180
6	182
7.5	182

Cure Cycle

The cure cycle needed for satisfactory property development will vary considerably depending upon the application. In applications where maximum Tg's are less important than mechanical strengths, shorter cure cycles may be satisfactory.

Effect of Cure Cycle on Mechanical Properties

FORMULATION: Epoxy resin (WPE = 183), phr		100		
ETHACURE 100, phr		25.5		
CURE CYCLE:				
Temp, °C	**Time in Minutes**			
100	—	30	—	120
130	30	—	—	—
175	30	40	60	240
MECHANICAL PROPERTIES:				
Tensile strength, psi	12,600	12,200	11,900	9,970
Tensile modulus, psi	463,000	465,200	430,000	422,000
Elongation at break	7.7	6.6	6.2	4.5
Flexural strength, psi	21,500	21,700	20,100	17,000
Flexural modulus, psi	483,000	465,000	454,000	434,000
Izod impact, ft-lb/in	0.5	0.4	0.5	0.4
Tg, °C	134	136	152	174

Epoxy Novolac Resins

Epoxy novolac resins provide higher temperature performance. A higher deflection temperature is observed with the novolac resin cured with ETHACURE 100 hardener and indicates the suitability for a higher end use temperature.

Mechanical Properties

FORMULATION:	Epoxy novolac (WPE = 179), phr	100	100
	ETHACURE 100, phr	25	
	MDA phr	–	28
CURE CYCLE:	16 hr at 55°C (131°F), 2 hr at 125°C (257°F), 2 hr at 175°C (347°F), and 4 hr at 200°C (392°F)		
MECHANICAL PROPERTIES:			
Tensile strength, psi		8,200	9,100
Tensile modulus, psi		450,000	450,000
Elongation, %		2.2	2.9
Flexural strength, psi		17,000	15,400
Flexural modulus, psi		470,000	450,000
Izod impact, ft-lb/in		0.2	0.4
Deflection temperature, °C		228	182 (207)

MDA data was obtained from the literature. Cure cycle was 16 hours at 55°C, 2 hours at 125°C, and 2 hours at 175°C. The 207°C deflection temperature was obtained after an additional post cure of 4 hours at 200°C.

Toxicity

Acute Toxicity

ETHACURE 100 has a moderate to low degree of acute toxicity. The rat oral LD_{50} is 485 mg/kg. Rabbit dermal LD_{50} is approximately 700 mg/kg. Skin application on rabbits produced slight irritation. ETHACURE 100 was moderately to severely irritating to the eyes of rabbits. Rats were nominally exposed to 2.45 mg/L for 1 hour. This airborne exposure produced no deaths.

Sensitization

ETHACURE 100 was negative for skin sensitization when tested in guinea pigs. Repeated dermal applications resulted in irritation.

Genetic Toxicity

A battery of mutagenic assays was conducted on ETHACURE 100 showing no mutagenic activity in the Ames *Salmonella*/microsomal or *Saccharomyces* assays, no significant DNA damage in the *E.coli* DNA repair test, and no mutagenic activity in the dominant lethal test. No chromosome damage was detected by *in vivo* cytogenetics and micronucleus assays. A significant response was produced in the BALB/3T3 cell point mutation test without activation. However, this effect was not reproducible in a repeat test. Also, no significant response was produced in this assay after activation with the addition of a liver preparation. The results of these assays indicate ETHACURE 100 is not genetically active.

Subchronic Toxicity

A subchronic 21-day dermal toxicity study was conducted in rabbits. Repeated dermal applications of ETHACURE 100 at 1, 10 and 100 mg/kg for three weeks (five days/week) resulted in mild to moderate local irritation at the 10 and 100 mg/kg dosages. No significant local effects were observed at the 1 mg/kg dose. There were no significant systemic effects in any of the rabbits in this study.

Carcinogenicity

ETHACURE 100 showed no *in vitro* carcinogenic potential when its activity was evaluated in the *in vitro* BALB/3T3 cell transformation assay with and without metabolic activation. It did not induce morphological transformation in this assay. In addition, a screening test on a limited number of rats given ETHACURE 100 subcutaneously over a lifetime was negative for tumorigenicity.

Safety and Handling

ETHACURE 100 is a liquid aromatic diamine with a flash point (TCC) greater than 275°F. Special care should be exercised when storing or transferring it. This includes the following:

1. If possible, always blanket ETHACURE 100 with dry nitrogen.
2. If this is not possible, avoid outdoor storage and areas of high humidity.
3. Avoid prolonged storage at elevated temperatures (over 100°F).
4. To ensure homogeneity, warm the material to room temperature if stored below 15°F.

As a *normal* safety precaution, we recommend that personnel handling ETHACURE 100 always use chemical splash goggles or a face shield, neoprene or nitrile rubber gloves, resistent outer garments, and, if it is heated and exposed, a dual cartridge organic vapor respirator. Always handle ETHACURE 100 in a well ventilated area.

In case of contact, remove contaminated clothing, and wash the exposed skin or eyes for 15 minutes with plenty of water. If swallowed, drink two glasses of water and induce vomiting. Call a physician. Do not reuse contaminated clothing.

In case of a spill or leak, use the protective gear recommended above. Soak up the spilled material with sawdust, sand, vermiculite, or similar absorptive material, and shovel into a container for disposal in accordance with local regulations regarding organic chemical wastes.

In case of fire, use water spray, foam, dry chemical or CO_2.

Ordering and Shipping

For Order Placement:

U.S.A.
Ethyl Corporation, Chemicals Group
451 Florida Boulevard
Baton Rouge, Louisiana 70801 U.S.A.
Tel: (504) 388-7556 or
Toll Free (800) 535-3030 (except in Louisiana)

Shipping Point:
Ethyl's plant in Orangeburg, South Carolina, U.S.A.

Container Sizes:
TANK CAR
TANK TRUCK
DRUMS — 55 (425 lb net), 5 and 1 gallon nonreturnable, steel

Shipping Classifications:
U.S. DOT Description: Nonregulated
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Ethyl Chemicals Group

SC-522-88
(Supercedes SC:522-87)

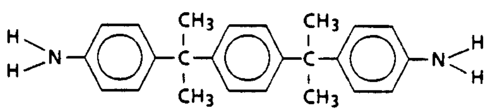
Technical Bulletin
Shell Chemical Company

EPON HPT™ Curing Agent 1061-M

Epoxy curing agent for high performance matrix resin system in advanced composites and adhesives

General description
EPON HPT™ Curing Agent 1061-M is an aromatic diamine curing agent with a novel backbone for use in high performance composite applications and adhesives. The solid curing agent is supplied as a finely ground powder for ease of handling.

Chemical description
4,4'-[1,4-phenylene (1-methyl ethylidene)] bis (benzeneamine)



Advantages
- High performance aromatic amine curing agent
- Low moisture absorption
- Yields improved hot/wet performance
- Finely ground powder for ease of handling
- Rapid gel time

Applications
- Advanced composite structural formulations
- High performance structural adhesives

Typical properties

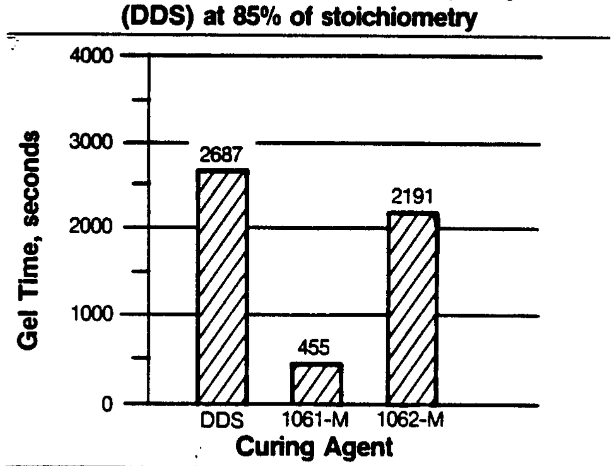
Physical form	Free flowing solid powder
Particle size	95% wt below 30 microns
Color	Tan to cream
Melting point, °F	322°-327°
Approximate equivalent weight, active hydrogen	86

Formulation/processing
Neat resin casting
The following is a general procedure used in the laboratory for preparation of neat resin castings using Epon HPT curing agent 1061-M with multifunctional resins like EPON HPT™ Resin 1071 or TGMDA.

1. The Epon HPT resin, glycidyl ether of BPA Novolac, EPON® Resin 825, and/or triglycidyl aminophenol (TGAP) are heated in a circulating air oven to 300-325°F.
2. The Epon HPT curing agent 1061-M is melted in a second oven at 340°F.
3. Melted Epon HPT curing agent 1061-M dissolves rapidly in the resin with vigorous stirring resulting in a homogeneous resin/curing agent solution. (Caution: the gel time of Epon HPT curing agent 1061-M with these resins is rapid at 350°F, see Figure 1.)
4. The resin curing agent solution is then immediately degassed in a vacuum oven at 25 in. of Hg or less at 350°F. After degassing, the solution is ready to pour into a mold, which should be heated to about 300°F.

Gel and cure characteristics
Figure 1 shows the gel times of Epon HPT resin 1071 with Epon HPT curing agent 1061-M as compared with diaminodiphenylsulfone (DDS) and EPON HPT™ Curing Agent 1062-M. This shows the Epon HPT curing agent 1061-M gel time at 350°F is very fast compared to that of DDS and Epon HPT curing agent 1062-M.

Figure 1/**Gel times of EPON HPT™ Resin 1071 with EPON HPT™ Curing Agents 1061-M and 1062-M and diaminodiphenylsulfone (DDS) at 85% of stoichiometry**



Performance properties

Neat resin casting properties

Table 1 shows the neat resin casting properties of Epon HPT curing agent 1061-M with Epon HPT resin 1071. A comparison of performance obtained when cured with DDS is also provided. As is shown in the table, Epon HPT curing agent 1061-M gives improved retention of properties in hot/wet environments.

Table 1/**Neat resin casting properties of EPON HPT™ Resin 1071 cured with EPON HPT™ Curing Agent 1061-M and with diaminodiphenylsulfone (DDS)**

	Epon HPT resin 1071/ Epon HPT curing agent 1061-M	Epon HPT resin 1071/ DDS[1]
T_g (tan delta), °F	466	480
Moisture gain[2], %	2.1	3.6
Flexural properties (RT/dry)		
Strength, ksi	20	20
Modulus, ksi	494	563
Flexural properties (Hot/Wet)[3]		
Strength, ksi	14	13
Modulus, ksi	427	434
Retention of RT/dry properties at 200°F wet conditions,[3] %		
Strength, %	70	65
Modulus, %	86	70

[1]Cure schedule: 2 hours at 300°F; 4 hours at 392°F
[2]After 2 weeks immersion at 200°F
[3]Tested in water at 200 °F after two weeks immersion at 200°F

Packaging, storage and shipping

Epon HPT Curing Agent 1061-M is available for evaluation in 10-pound and 50-pound quantities. It is not defined as hazardous by criteria of DOT Regulations. Its shelf life is indefinite at room temperature (77°F).

Use and handling information for systems based on Epon HPT curing agent 1061-M

The recommendations for material selections made in this bulletin are based on Shell's experience and research and are believed to be sound technical approaches to the applications or end uses for which they are presented. However, these recommendations are directed solely toward technical performance and should not be taken as recommendations pertaining to health, safety or the environment.

Use of Epon HPT curing agent 1061-M is regulated by EPA under a TSCA Section 5(e) Consent Order. You will be notified by registered mail of the specific terms of the Order prior to the distribution of product to you. The requirements include use of personal protective equipment, use only at facilities under your control, use only as a component of prepregs, composites or adhesives for industrial applications, and no further distribution of the product before it has been formulated.

Epon HPT curing agent 1061-M and the auxiliary materials normally combined with this product are capable of producing adverse health effects ranging from minor skin irritation to serious systemic effects. Adverse effects can be minimized and most can be avoided through the observance of proper precautions, use of proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these depends on responsible action by adequately informed personnel.

A Material Safety Data Sheet (MSDS) is available for this product (MSDS # 690). **Transportation, storage, handling and use of this product should not occur until handling precautions and recommendations, as described in the MSDS, are understood by all persons who will work with it.** Questions and requests for MSDS sheets or other information should be directed to your Shell Chemical Company Sales Office. Information on non-Shell products should be obtained from the respective manufacturer or vendor.

For more information on EPON HPT™ Resin Systems contact:

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For Technical Assistance
Call toll free 1-800-TEC-EPON
In Texas call 1-800-222-EPON

Warranty

All products purchased from or supplied by Shell are subject to terms and conditions set out in the contract, order acknowledgement and/or bill of lading. Shell warrants only that its product will meet those specifications designated as such herein or in other publications. All other information supplied by Shell is considered accurate but is furnished upon the express condition that the customer shall make its own assessment to determine the product's suitability for a particular purpose. **Shell makes no other warranty, either express or implied, including those regarding such other information, the data upon which the same is based, or the results to be obtained from the use thereof; that any product shall be merchantable or fit for any particular purpose; or that the use of such other information or product will not infringe any patent.**

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Technical Bulletin
Shell Chemical Company

EPON HPT® Resin 1071

New high performance epoxy matrix resin for advanced composites and adhesives.

- Prepreg
- Resin transfer molding
- Adhesives

General description

EPON HPT® Resin 1071 is a tetrafunctional epoxy resin suitable for high performance composite and adhesive applications. At elevated temperatures, Epon HPT resin 1071 provides outstanding performance characteristics. When cured at 400°F, the resin exhibits outstanding retention of properties at 300-350°F hot/wet.

Advantages

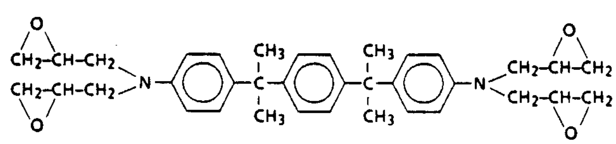
- Provides composites with outstanding hot/wet performance
- Outstanding heat resistance and deflection temperature
- Interchangeable with resins presently used in advanced composites

Applications

- Advanced composite structures with glass, carbon, aramid, or boron fibers
- High performance structural adhesives
- Processing methods
  - Prepreg
  - Resin transfer molding

Chemical description

- N,N,N,'N'-tetraglycidyl-α,α'-bis(4-aminophenyl)-p-diisopropylbenzene (CAS# is 103490-06-8).



Sales specifications

EPON HPT® Resin 1071	
Weight per epoxide (WPE)[4]	150-185

Typical properties

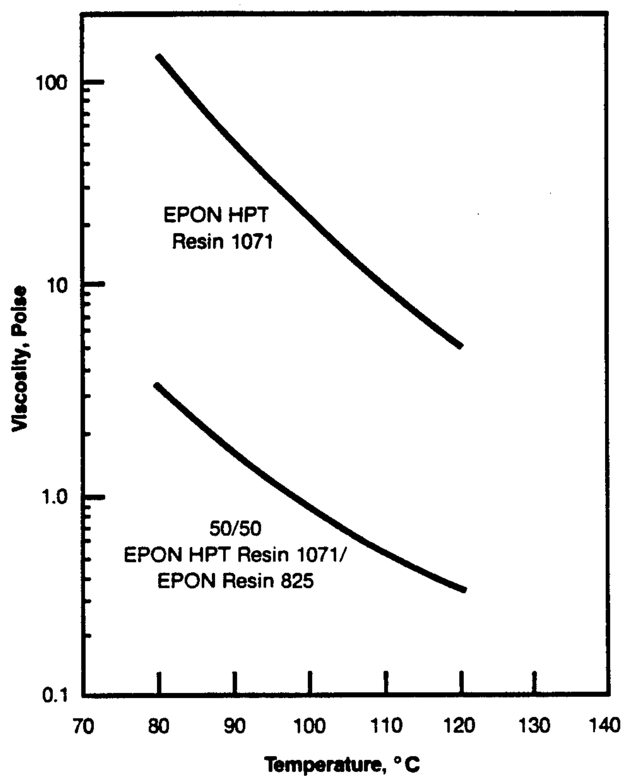
EPON HPT® Resin 1071	
Physical form	Dark colored solid
Melting point,[1] °F	122
T_g,[2] °F	73
Melt Viscosity,[3] at 230°F, Poise	18-22
Weight per epoxide (WPE)[4]	150-185

[1]ASTM D 3461, Mettler, 1°C/min
[2]DSC
[3]Brookfield
[4]Shell Method HC-427C-81, Perchloric Acid Method

Neat resin formulation

A number of specific formulations can be prepared using Epon HPT resin 1071. The general procedure used in preparation of neat resin castings using Epon HPT resin 1071 and curing agents such as diaminodiphenylsulfone (DDS) or EPON HPT® Curing Agent 1062-M is discussed below. In preparation of the formulations Epon HPT resin 1071 is blended with EPON® Resin 825, a glycidyl ether of bisphenol A. Melt viscosity vs. temperature is shown in Figure 1.

Figure 1/**Melt viscosity vs. temperature for EPON HPT® Resin 1071 and 50/50 blend with EPON® Resin 825**



Neat resin castings

Neat resin castings using Epon HPT resin 1071 are produced in the following manner.

1. The Epon HPT resin 1071, Epon resin 825, and/or triglycidylaminophenol (TGAP) are heated in a circulating air oven to 300-325° F.

2. When using DDS as a curing agent, it is also heated to 300-325° F in the same oven. If Epon HPT curing agent is used, it is melted in a second oven at 340° F.

3. The heated curing agent is then added to the resin mixture with vigorous stirring. The DDS system is returned to the 300-325° F oven and held there until the DDS dissolves. The mixture is periodically removed from the oven and stirred vigorously. Approximately 20-30 minutes are required for the DDS to dissolve into the resin mixture. When Epon HPT curing agents are used, their melts dissolve rapidly in the resin.

4. The resin/curing agent solution is then degassed in a vacuum oven at 125 mm of Hg or less at 350° F. After degassing, the solution is ready to pour into a mold preheated to 300° F.

Gel and cure characteristics

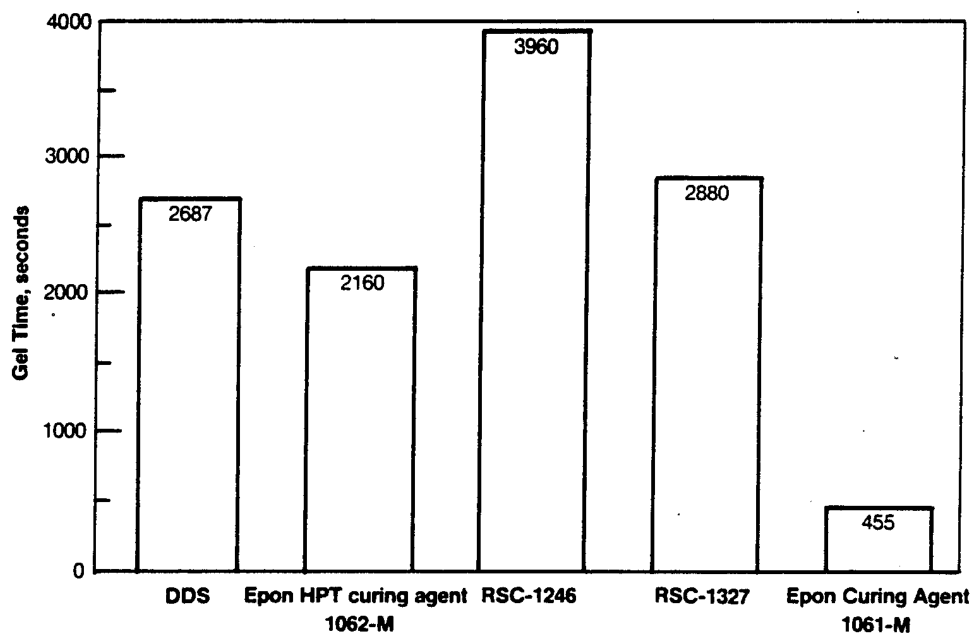
Epon HPT resin 1071 can be cured with a variety of curing agents. Gel times with Epon HPT curing agent 1062-M and DDS are shown in Table 1. Figure 2 compares DDS with Epon HPT curing agents and new Research Curing Agents RSC-1246 and RSC-1327.

Table 1/**Gel times versus temperature for EPON HPT® Resin 1071 cured with EPON HPT® Curing Agent 1062-M[1] and DDS[1]**

Temperature	Epon HPT curing agent 1062-M	DDS
248°F	> 2.5 hours[3]	> 2.5 hours
356°F	20-30 minutes	40 minutes
392°F	17 minutes	
446°F	7 minutes	

[1]100% stoichiometry
[2]Gel plate technique
[3]Resin mixture still fluid after 2.5 hours

Figure 2/**Gel times of EPON HPT® Resin 1071 with EPON HPT® Curing Agents and diaminodiphenylsulfone (DDS) at 85% of stoichiometry at 350°F**



The viscosity profiles of the Epon HPT resin 1071 system with Epon HPT curing agent 1062-M are shown in Figure 3 and Figure 4. The viscosity cure sweep and isothermal sweep show the system changes in viscosity at 100% stoichiometry. Variations of curing agent and cure schedule will impact the degree of cure (as measured by glass transition temperature, T_g) of Epon HPT resin 1071. This information is shown in Table 2.

Figure 3/**Viscosity cure sweep for EPON HPT® Resin 1071/EPON HPT® Curing Agent 1062-M system (61 phr) at 10°C/min using a Rheometrics Parallel Plate Viscometer**

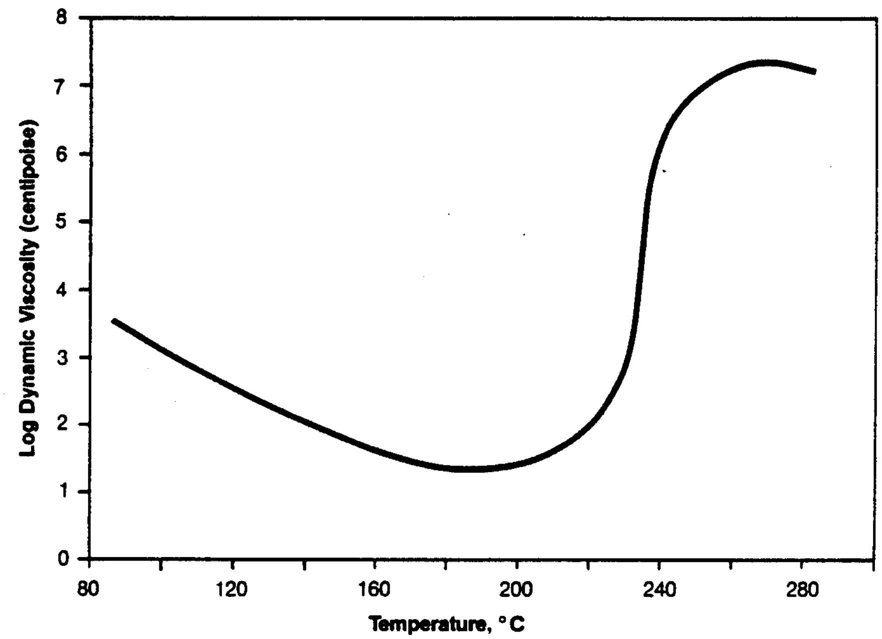
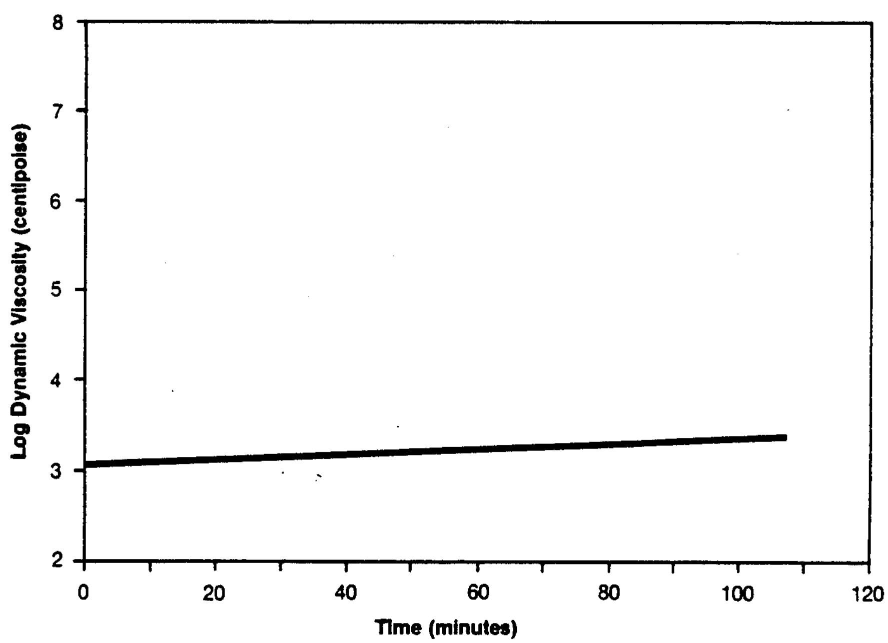


Figure 4/**Isothermal viscosity sweep for EPON HPT® Resin 1071/EPON HPT® Curing Agent 1062-M system (61 phr) at 220°F using a Rheometrics Parallel Plate Viscometer**



Gel and cure characteristics (Cont)

Table 2/**The effect of cure schedule on the glass transition temperature (T_g) of mixed resin/curing agent systems containing EPON HPT® Resin 1071, TGMDA, glycidyl ether of BPA Novolac, TGAP, DDS and EPON HPT® Curing Agent 1062-M**

Components	**Epon HPT resin 1071**	**TGMDA**	**Epon HPT resin 1071**	**Epon HPT resin 1071**
Resin component	100	100	100	100
EPI-REZ[1] SU-8, phr	8.2	8.2	—	—
TGAP,[2] phr	—	—	—	32
DDS, phr	22.8	28.0	—	—
Epon HPT 1062-M, phr	—	—	61	93
Stoichiometry, %	55	55	100	93
DMA[3] Glass transition temperature, °F at cure schedules				
2 hrs at 350°F	372	446	473	
2 hrs at 300°F 2 hrs at 392°F	455	518	471	
2 hrs at 300°F 4 hrs at 392°F	489	525	459	477

[1]EPI-REZ is a registered trademark of Interez, Inc.
[2]TGAP — Triglycidyl aminophenol
[3]DMA performed on DuPont 982 Dynamic Mechanical Analyzer at 9°F/min. Glass transition temperature determined from tan delta peak.

Composite fabrication

Prepreg formulation

The following is a general procedure used in the preparation of a prepreg formulation using Epon HPT resin 1071 with Epon HPT curing agent 1062-M and triglycidyl aminophenol (TGAP).

1. The Epon HPT resin 1071 and TGAP are blended together at 250°F in a high shear mixer.
2. The appropriate amount of Epon HPT curing agent 1062-M is then added while maintaining temperature.
3. The mixture is stirred until clear (20 minutes max.) and used immediately to cast a resin film.
4. The resin film is made using a doctor blade arrangement and controlling the following parameters:

Blade temperature, °F	175
Plate temperature, °F	175
Resin temperature, °F	175-200
Release paper film weight, g/m^2	97 (+5,-10)
thickness, in	0.005
width, in	12.5
Casting speed, ft/min	50

5. The cast film is then immediately put into 0°F (-18°C) storage. The above parameters result in a prepreg of 140-150 g/m^2) and a resin content of 35-42 wt%.
6. A 12-inch wide prepreg tape is produced with the cast resin film and fiber passing over a 200°F, two-foot long hot plate at speeds up to 10 ft/min with pressure applied by an oscillating sled.
7. The prepreg is then stored at 0°F.

The uncured Epon HPT resin 1071 system glass transition temperature (T_g) will vary with curing agent and diluents used. The uncured system T_g of two Research Curing Agents with Epon HPT resin 1071 is compared to that with Epon HPT curing agent 1062-M in Table 3.

Table 3/**Uncured T_g of EPON HPT® Resin 1071 with EPON HPT® Curing Agent 1062-M, RSC-1246, and RSC-1327**

	Uncured Tg (°F)
Epon HPT curing agent 1062-M	95
RSC-1246	68
RSC-1327	79

Figures 5a and 5b show the viscosity profiles of the Epon HPT resin 1071 with Epon HPT curing agent 1062-M and TGAP.

Prepreg formulation (Cont)

Figure 5a/**Cure viscosity sweeps (2° C/min and 10° C/min) for the EPON HPT® Resin 1071/TGAP/EPON HPT® Curing Agent 1062-M system (100/32/93 parts) at prepreg processing temperatures using a Rheometrics Parallel Plate Viscometer**

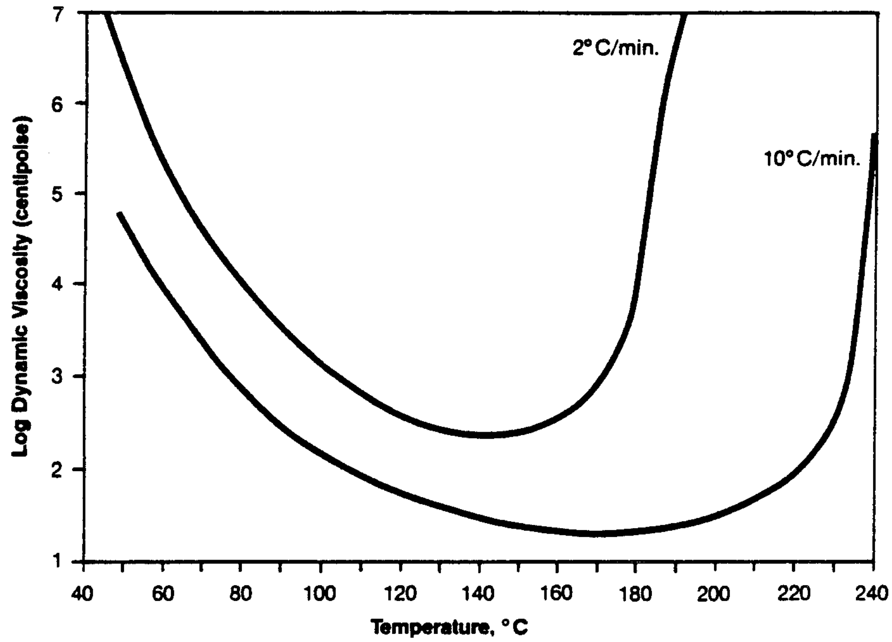
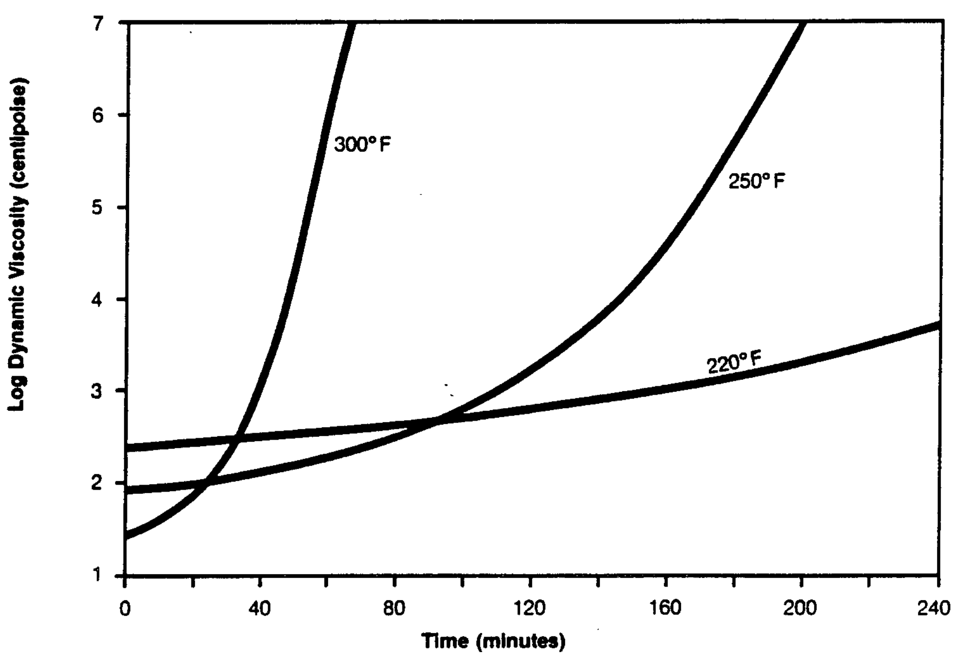


Figure 5b/**Isothermal viscosity sweeps, (220° F, 250° F and 300° F) for the EPON HPT® Resin 1071/TGAP/EPON HPT® Curing Agent 1062-M (100/32/93 parts) at prepreg processing temperatures using a Rheometrics Parallel Plate Viscometer**



Vacuum bag lay-up

The prepreg prepared using the above procedure should then be fabricated into panels or parts by following the general procedure outlined below.

1. The lay-up procedure for bag molding begins with an aluminum or steel base plate coated with mold release (Frekote 33).
2. An FEP release film, 2 mils thick, is then laid on the base plate with a porous release, Teflon-coated glass fabric on top of it.
3. The prepreg plies in the appropriate lay-up sequence are then placed on top of the glass fabric.
4. Two types of resin bleeders are typically used: a 120-style glass fabric/prepreg (1:3) and a 181-style glass fabric/prepreg (1:5).
5. A nonperforated release film is placed on the resin bleeders followed by a steel caul plate.
6. On top of the caul plate, 2 plies of 1518 style glass fabric are placed as a breather layer.
7. The vacuum bag, high temperature nylon, is then placed over the entire stack and sealed with vacuum bag sealant and blocked with edge dams.
8. The autoclave cure schedule for this system begins by applying full vacuum to the part (20 inches Hg minimum) and holding for 30 minutes.
9. Raise the temperature to 200° F at 2-5° F/min.
10. Apply 3 psi autoclave pressure and vent the vacuum bag.
11. Raise the temperature to 240° F at 1-3° F/min.
12. Hold there for 160 minutes.
13. Apply 85 psi autoclave pressure and heat to 350° F at 2-5° F/min.
14. Hold there for 120 minutes.
15. Cool under full pressure to below 140° F.
16. Post cure (free standing) for 120 minutes at 390° F.

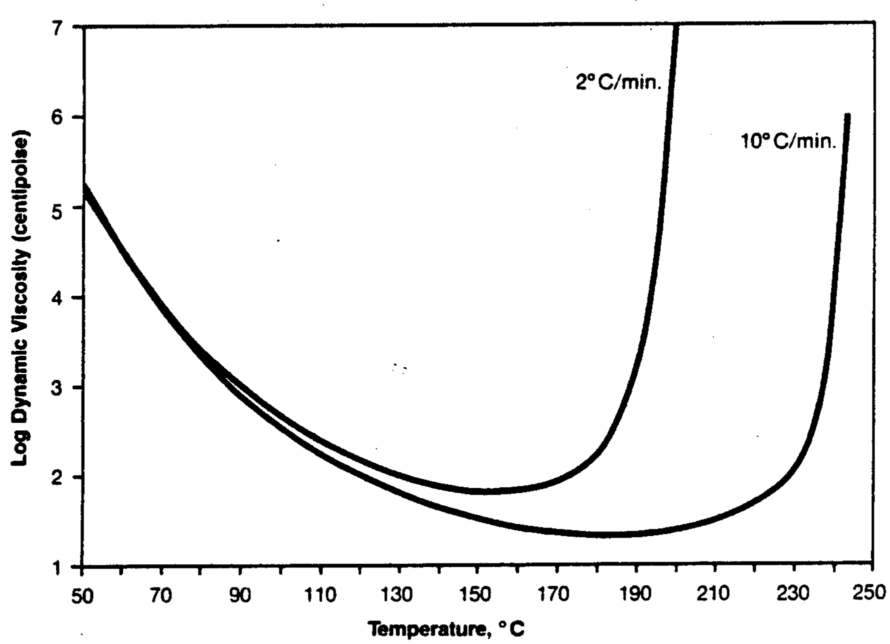
Resin transfer molding (RTM)

The following is a general procedure used for preparation of an advanced composite component using RTM with a blend of Epon HPT resin 1071 and Epon resin 825 cured with Epon HPT curing agent 1062-M.

1. The Epon HPT resin 1071 and Epon resin 825 are heated individually in a circulating air oven to 300° F.
2. The Epon HPT curing agent 1062-M is heated in a second oven at 325-340° F.
3. Once melted, the two resins are mixed together with vigorous stirring. After mixing, the resin mixture is returned to the oven to be reheated.
4. Once reheated, the resin mixture and molten curing agent are removed from the ovens and are mixed together with vigorous stirring. If increased pot life is critical, the temperature of the two components may be allowed to decrease somewhat, but care must be taken to prevent the solidification of the curing agent.
5. The resin-curing agent mixture is then placed in a vacuum oven at 250-300° F and degassed using 27 inches of Hg or less of vacuum for 5-20 minutes.
6. The resin-curing agent mixture should then be placed in the injection pot at 185-205° F or immediately cooled to inhibit further reaction.
7. The tool should be preheated to 230-250° F to decrease viscosity to an appropriate level.
8. Pressure (20-80 psi) or vacuum (27 inches Hg or more) may be used alone or in combination to inject the resin into the tool cavity. If vacuum is used, a slight positive pressure should be applied prior to the closing of the tool to reduce/eliminate voids and/or to inhibit air leaking into the system.
9. The cure schedule used may be adjusted to fit the requirements of the customer, but a typical cure schedules is 2 hours at 300° F and 2-4 hours at 392° F. Depending on the part size and geometry, the latter step can be performed outside of the tool.

The cure viscosity sweep of the Epon HPT resin 1071 system for RTM is shown in Figure 6. The heating rates are at 2° C/min. and 10° C/min.

Figure 6/**Cure viscosity sweeps (2° C/min and 10° C/min) for the EPON HPT® Resin 1071/EPON® Resin 825/EPON HPT® Curing Agent 1062-M system (50/50 wt%; 59.8 phr) at RTM processing temperature using a Rheometrics Parallel Plate Viscometer**



Performance properties

Neat resin castings

Epon HPT resin 1071 provides composites with outstanding hot/wet performance. When cured at 400°F, the resin exhibits outstanding retention of properties at 300-350°F hot/wet. The resin is also interchangeable with other resins presently used in advanced composites. The cured properties of unreinforced Epon HPT resin 1071 formulations are compared with TGMDA formulations in Table 4. The Epon HPT resin 1071 systems absorb less moisture and have improved hot/wet performance over TGMDA/DDS systems.

Table 4/**Neat resin casting properties for EPON HPT® Resin 1071, TGMDA and EPON HPT® Resin 1071 modified with EPON® Resin 825 and with triglycidyl aminophenol (TGAP) cured with EPON HPT® Curing Agent 1062-M and with diaminodiphenylsulfone (DDS)[1]**

	TGMDA	Epon HPT resin 1071	Epon HPT resin 1071	Epon HPT resin 1071/ Epon resin 825 (50/50 pbw)	Epon HPT resin 1071/ TGAP (100/32 pbw)
Components					
Resin component	100.0	100.0	100.0	100.0	100.0
Epon HPT curing agent 1062-M, phr	—	—	61.0	50.4	93.0
EPI-REZ[2] SU-8, phr	8.2	8.2	—	—	—
Diaminodiphenylsulfone (DDS), phr	28.0	22.8	—	—	—
Stoichiometry, %	55	55	100	100	93
T_g (tan delta),[3] °F	467	477	462	428	477
Moisture gain,[4] %	4.7	2.7	1.6	1.7	2.1
Cured density, g/cc			1.117		1.127
Flexural properties (RT/dry)					
Strength	17	17	19	19	18
Modulus, ksi	580	590	497	438	477
Flexural properties (Hot/wet)[5]					
Strength, ksi	12	11	14	13	15
Modulus, ksi	471	524	441	403	437
(Hot/wet vs. RT/dry) Retention, %	81	89	89	92	92

[1]Cure schedule: 2 hours at 300°F; 4 hours at 400°F.
[2]Glycidyl either of a bisphenol A Novolac (Interez, Inc.)
[3]Tg (tan delta) is obtained using a Rheometrics Viscoelastic Tester and the samples are heated from 86°F to a maximum of 572°F in 18°F increments with a 2-minute hold at temperature before obtaining the data.
[4]After 48 hour water boil.
[5]Tested in water at 200°F after two weeks immersion at 200°F.

Neat resin castings (Cont)

To provide materials to enhance the processing of Epon HPT resin 1071, new Research Curing Agents have been developed. These materials when combined with Epon HPT resin 1071 give lower uncured system T_g (Table 3) while providing high retention of properties at 200°F wet, shown in Table 5.

Table 5/**Neat resin casting properties for EPON HPT® Resin 1071 cured with EPON HPT® Curing Agent 1062-M, RSC-1246, and RSC-1327**[1]

	Epon HPT resin 1071		
	Epon HPT curing agent 1062-M	**RSC-1246**	**RSC-1327**
T_g (tan delta), °F	462	414	432
Flexural properties (RT/dry)			
Strength, ksi	19	16	16
Modulus, ksi	497	445	514
Flexural properties (Hot/wet)[2]			
Strength, ksi	14	13	14
Modulus, ksi	441	432	441

[1]Cured 2 hrs. at 300°F and 4 hours at 400°F.
[2]Tested in 200°F water after 2 weeks immersion in 200°F water.

Epon HPT resin 1071 maintains low moisture absorption when stoichiometry is varied. This results in high retention of properties in hot/wet environments as shown in Table 6. The resin when cured with Epon HPT curing agent 1062-M shows very high retention of properties at 300°F and 350°F. This retention of performance is compared to a TGMDA/DDS system in Table 7.

Table 6/**Effect of curing agent ratio on performance of the EPON HPT® Resin 1071/ EPON HPT® Curing Agent 1062-M system**

Stoichiometry, %	55	65	85	100
T_g (tan delta), °F	408	441	459	463
Moisture gain, %wt	1.8	1.7	1.7	1.6
Flexural properties (RT/dry)				
Strength, ksi	15	18	20	19
Modulus, ksi	544	531	506	497
Flexural properties (Hot/wet)[1]				
Strength, ksi	14	14	14	14
Modulus, ksi	459	428	427	441
Modulus (Hot/wet vs. RT/dry)				
Retention, %	84	81	84	89

[1]Tested under water at 200°F after two weeks immersion at 200°F.

Table 7/**High temperature performance of the EPON HPT ® Resin 1071/EPON HPT® Curing Agent 1062-M (61 phr) system compared to the TGMDA/SU-8/DDS system under dry and wet conditioning**[1]

	TGMDA/ SU-8/DDS[2]	Epon HPT resin 1071/ Epon HPT curing agent 1062-M[3]
Flexural modulus (RT/dry), ksi	555	500
300°F dry	370	400
300°F wet[4]	215	365
350°F dry	330	365
350°F wet[4]	155	320
Retention of RT/dry, %		
at 300°F wet	39	73
at 350°F wet	21	64

[1]Cure schedule: 2 hours at 300°F, 4 hours at 400°F
[2]TGMDA/SU-8/DDS = 90/10/29.7 parts
[3]1071/1062 = 100/61 parts
[4]After 48 hour water boil

Dynamic Mechanical Analysis profiles have been developed on these materials and can be obtained upon request.

Prepreg composite

Prepreg was prepared using Epon HPT Resin 1071, triglycidyl aminophenol (TGAP), and Epon HPT curing agent 1062-M. This resin system was applied to Hercules IM-6 fiber and made into a 12-inch wide prepreg tape. The physical, processing and performance properties are shown in Table 8 and Table 9.

Table 8/**Characteristics of EPON HPT® Resin 1071/EPON HPT® Curing Agent 1062-M/TGAP/Hercules IM-6 prepreg tape** [1]

	Epon HPT resin 1071/ Epon HPT curing agent 1062-M/ TGAP	Typical TGMDA/DDS system[2]
Property		
Resin content, %wt	35-40	32-42
Glass transition temperature,[3] °F	10	< 10
Gel time,[4] min		
200°F	> 480	
350°F	39	6-30
Volatile content, %wt	< 1	1 max
Tack,[5] RT, days	3-4	10 min
Drape, RT, days	8-10	10 min
Out time,[6] RT, days	> 70	> 70
Shelf life,[6] 0°F months, minimum	12	12
Fiber		
Density, g/cc	1.75	1.69-1.77
Epoxy sizing, %wt	0.8-0.9	0.5-1.1
Nominal aerial weight, g/m^2	144	150

[1]TGAP = triglycidyl aminophenol
IM-6 fiber is 12K, G-sized; 12 in. wide prepreg tape
[2]Range of data obtained from literature on several commercial systems, including accelerated systems
[3]Determined by Differential Scanning Calorimetry
[4]Gel plate technique
[5]Subjective evaluation
[6]System is meltable and flowable at 200°F.

Performance properties (Cont)

Prepreg composite (Cont)

Table 9/**Mechanical properties of EPON HPT® Resin 1071/EPON HPT® Curing Agent 1062-M/TGAP/Hercules IM-6 carbon fiber laminates**[1]

	Epon HPT resin 1071/ Epon HPT curing agent 1062-M/ TGAP	Typical TGMDA/DDS system[2]
Property		
Cured ply thickness, mils	5.3-5.9	5.5
Glass transition temperature,[3] °F	424	356
0° Flexural		
Strength, ksi	261	260
Modulus, msi	21	21
0° Tensile		
Strength, ksi	341	370
Modulus, msi	30	24
Elongation, %		1.4
Short beam shear, ksi	14	18
Edge delamination,[4] ksi	28	

[1]TGAP = triglycidyl aminophenol dry, room temperature properties IM-6 fiber is 12K, G-sized; 12 in. wide prepreg tape
[2]Range of data obtained from literature on several commercial systems, including accelerated systems.
[3]Determined by Differential Calorimetry.
[4]Layup $(\pm 25)_2$ 90 90 $(\pm 25)_2$

Packaging and storage

Epon HPT® resin 1071 is available in 2-pound, 8-pound, and 40-pound quantities. It is not defined as hazardous by criteria of DOT regulations. Product should be stored at standard ambient conditions less than 80° F.

Handling precautions

The recommendations for material selections made in this bulletin are based on Shell's experience and research and are believed to be sound technical approaches to the applications or end uses for which they are presented. However, these recommendations are directed solely toward technical performance and should not be taken as recommendations pertaining to health, safety or the environment.

Use of Epon HPT resin 1071 is regulated by EPA under a TSCA Section 5(e) Consent Order. You will be notified by registered mail of the specific terms of the Order prior to the distribution of product to you. The requirements include use of personal protective equipment, use only at facilities under your control, use only as a component of prepregs, composites or adhesives for industrial applications, and no further distribution of the product before it has been formulated.

Epon HPT resin 1071 and the auxiliary materials normally combined with this product are capable of producing adverse health effects ranging from minor skin irritation to serious systemic effects. Adverse effects can be avoided through the observance of proper precautions, use of proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these depends on responsible action by adequately informed personnel.

A Material Safety Data Sheet (MSDS) is available for this product (MSDS #693). **Transportation, storage, handling, and use of this product should not occur until handling precautions and recommendations, as described in the MSDS, are understood by all persons who will work with it.** Questions and requests for MSDS sheets or other information should be directed to your Shell Chemical Company Sales Office. Information on non-Shell products should be obtained from the respective manufacturer or vendor.

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Technical Bulletin
Shell Chemical Company

EPON HPT® Resin 1079

New high performance epoxy matrix resin for advanced composites and adhesives

- **Prepreg**
- **Adhesives**

General description

EPON HPT® Resin 1079 is a diglycidyl ether of a stiff backbone bisphenol. The resin was developed for applications in advanced composites and adhesives. When cured at 400° F, the resin exhibits outstanding retention of properties at 350° F hot/wet.

Advantages

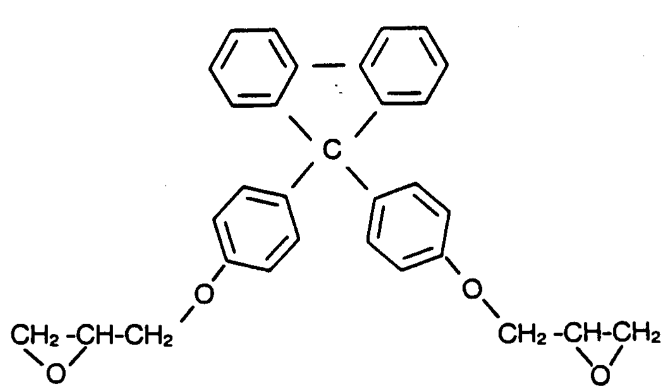
- Outstanding heat resistance and deflection temperature
- Low moisture absorption
- Outstanding hot/wet performance at 350° F
- Toughenable

Applications

- Advanced composite structures with glass, carbon, aramid, or boron fibers
- High performance structural laminates and adhesives
- Processing methods
  Prepreg

Chemical description

- Diglycidyl-9,9'-bis(4 hydroxyphenyl) fluorene
  (CAS# is 47758-37-2)



Sales specifications

EPON HPT® Resin 1079
Sales specifications not established

Typical properties

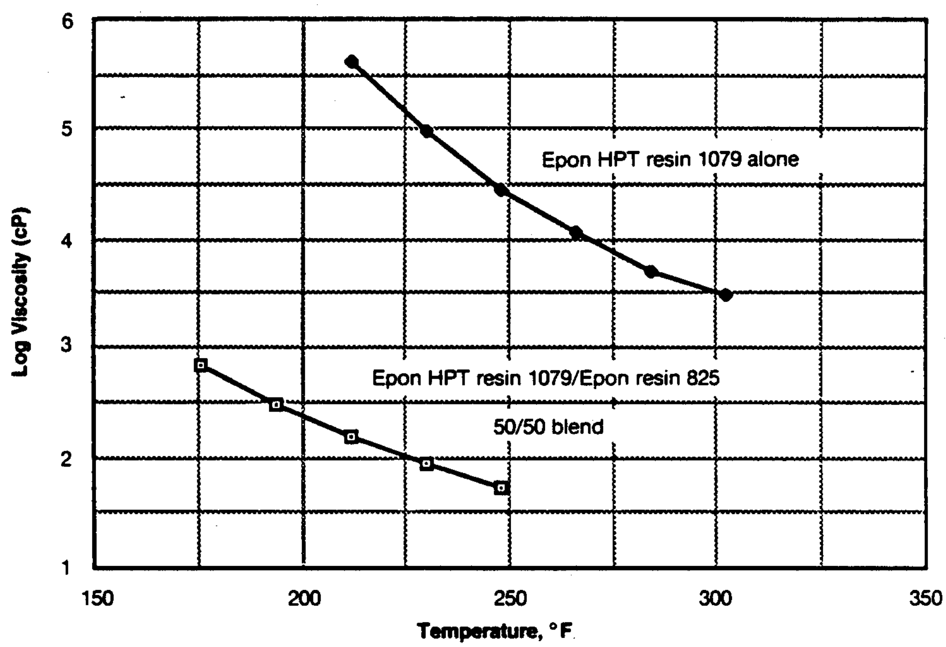
EPON HPT® Resin 1079	
Physical form	Glassy solid
Melting point,[1] °F	176-180
T_g,[2] °F	118
Melt viscosity,[3] at 350°F, cps	50-75
Weight per epoxide (WPE)[4]	240-270

[1]ASTM D 3461 Mettler, 1
[2]DSC
[3]Parallel Plate Viscosity
[4]Shell Method HC-427C-81, Perchloric Acid Method

Neat resin formulation

A number of specific formulations can be prepared using Epon HPT resin 1079. The general procedure used in preparation of neat resin castings using Epon HPT resin 1079 and curing agents such as diaminodiphenylsulfone (DDS) or EPON HPT® Curing Agent 1062-M is discussed below. In preparation of the formulations Epon HPT resin 1079 is blended with EPON® Resin 825, a glycidyl ether of bisphenol A. Melt viscosity versus temperature is shown in Figure 1.

Figure 1/**Melt viscosity versus temperature for EPON HPT® Resin 1079 and 50/50 blend with EPON® Resin 825**



Neat resin castings

Neat resin castings using Epon HPT resin 1079 are produced in the following manner.

1. The Epon HPT 1079, glycidyl ether of BPA Novolac, Epon resin 825, and/or triglycidyl aminophenol (TGAP) are heated in a circulating air oven to 300-325°F. (Blends with Epon resin 825 lower the melt viscosity of the base resin to aid in processing (Figure 1). In addition, any toughening agents can be mixed to the resin at this time.

2. When using diaminodiphenylsulfone (DDS) as a curing agent, it is also heated to 300-325°F in the same oven. If Epon HPT curing agent 1062-M is used, it is melted in a second oven at 340°F.

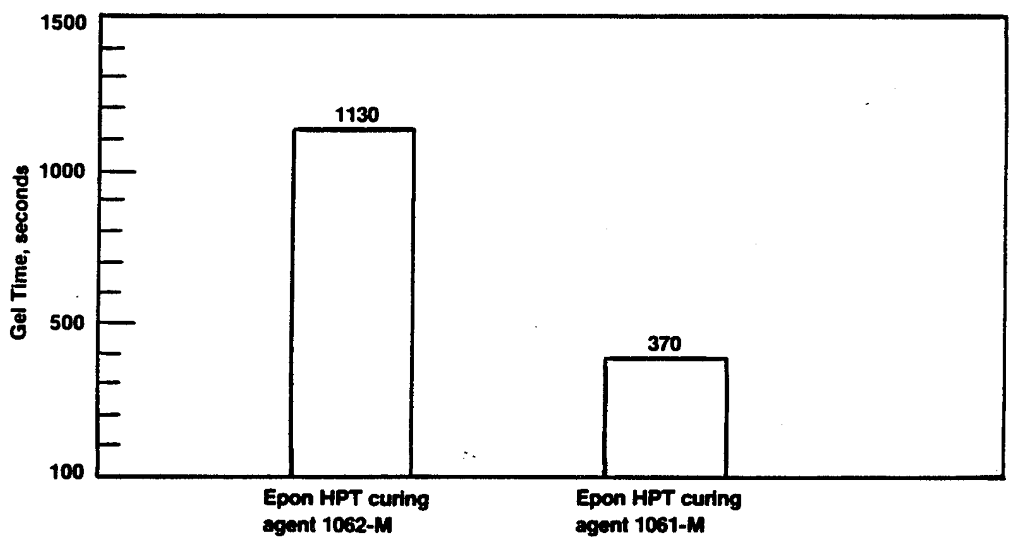
3. The heated curing agent is then added to the resin mixture with vigorous stirring. The DDS system is returned to the 300-325°F oven and held there until the DDS dissolves (during this time the mixture is periodically removed from the oven and stirred vigorously by hand). Approximately 20-30 minutes are required for the DDS to dissolve into the resin mixture. When Epon HPT curing agents are being used, their melts dissolve rapidly in the resin resulting in a resin/curing agent solution that can be degassed immediately as outlined in step 4, below.

4. The resin curing agent solution is then degassed in a vacuum oven at 25 inches of Hg or less at 350°F. After degassing, the solution is ready to pour into a mold, which should be heated to about 300°F.

Gel and cure characteristics

Epon HPT resin 1079 can be cured with a variety of curing agents. Gel times with DDS, and EPON HPT® Curing Agents 1061-M and 1062-M are shown in Figure 2.

Figure 2/**Gel times of EPON HPT® Resin 1079 with EPON HPT® Curing Agents and diaminodiphenylsulfone (DDS)**



The viscosity profile of the Epon HPT resin 1079 system with Epon HPT curing agent 1062-M is shown in Figure 3. Figures 4 and 5 show the viscosity cure sweep and the isothermal sweep for the Epon HPT resin 1079 system with Epon resin 825 and Epon HPT curing agent 1062-M. Variation of curing agent will have an impact on the measured glass transition temperature. The effect of various curing agents is shown in Table 1.

Figure 3/**Viscosity cure sweep for EPON HPT® Resin 1079/EPON HPT® Curing Agent 1062-M System at 10° C/min using a Rheometrics Parallel Plate Viscometer**

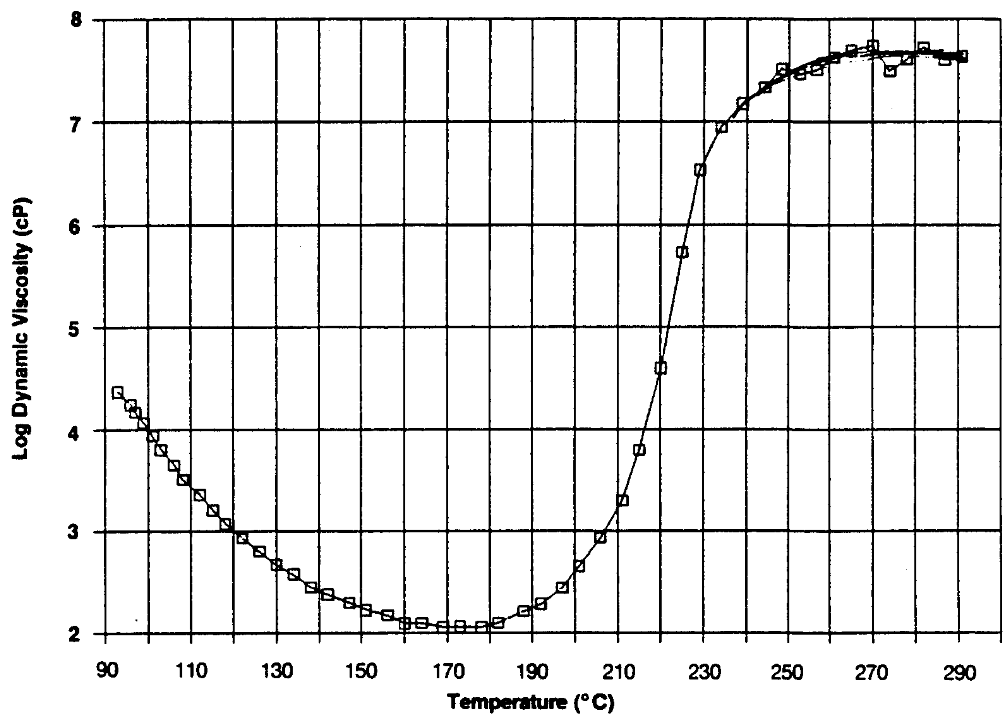


Figure 4/**Viscosity cure sweep for EPON HPT® Resin 1079/EPON Resin 825/EPON HPT® Curing Agent 1062-M at 10° C/min using Rheometrics Parallel Plate Viscometer**

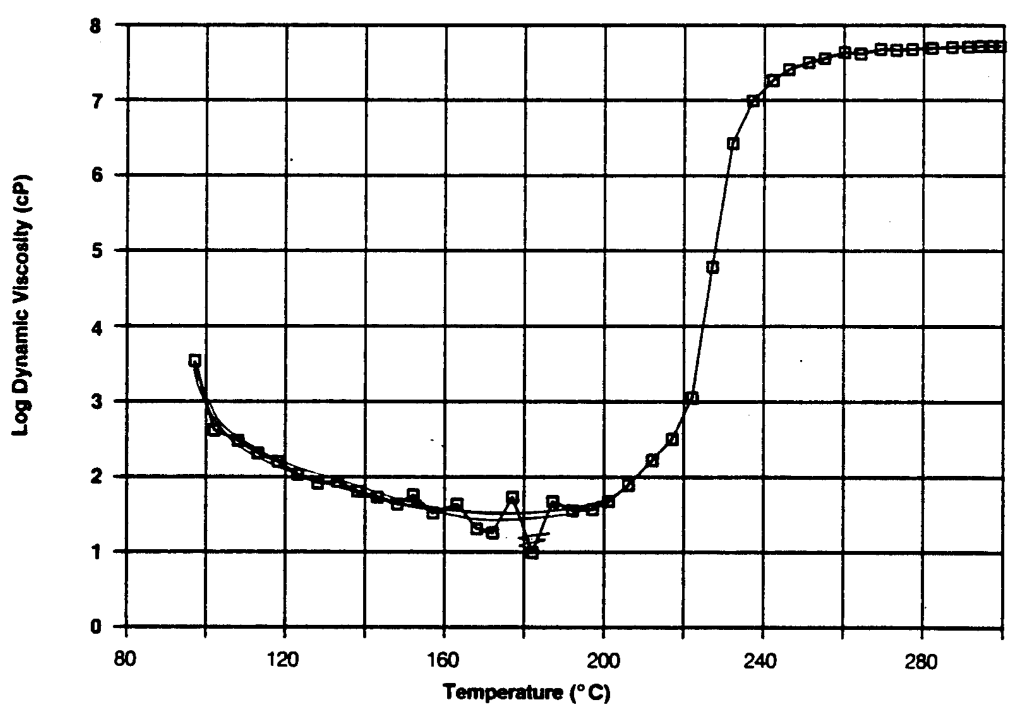


Figure 5/**Isothermal viscosity sweep for EPON HPT® Resin 1079/EPON 825/EPON HPT® Curing Agent 1062-M at 200°F using a Rheometrics Parallel Plate Viscometer**

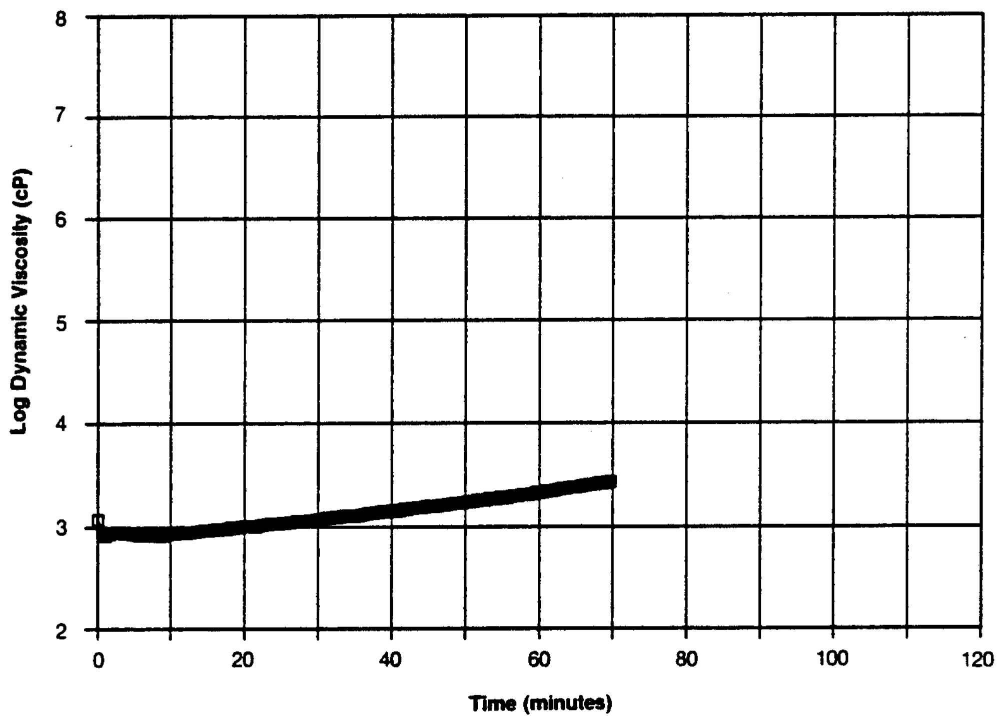


Table 1/**Glass transition temperature of EPON HPT® Resin 1079 with selected curing agents**[1]

	T_g (tan delta),[2] °F
Methylenedianiline	475
Diaminodiphenylsulfone	554
EPON HPT® Curing Agent 1061-M	482
EPON HPT® Curing Agent 1062-M	470

[1]Cure schedule 2 hrs. @ 300°F, 4 hrs. @ 400°F
[2]Rheometrics Viscoelastic Spectrometer

Performance properties

Neat resin castings

Epon HPT resin 1079 provides composites with outstanding hot/wet properties. When cured at 400°F, the resin exhibits outstanding retention of properties at 300°F and 350°F. The cured properties of unreinforced Epon HPT resin 1079 formulations are compared with TGMDA formulations in Table 2. The low water absorption and high T_g of the Epon HPT resin 1079 systems result in higher retention of properties than TGMDA/DDS systems when tested in hot/wet environments, Table 3.

Table 2/**Neat resin casting properties for EPON HPT® Resin 1079 and TGMDA cured with EPON HPT® Curing Agent 1062-M and with diaminodiphenylsulfone (DDS) including hot/wet exposure**[1]

Components			
Epon HPT resin 1079	—	100.0	100.0
TGMDA[2]	100.0	—	—
DDS[3]	49.4	24.3	—
Epon HPT curing agent 1062-M, phr	—	—	39.2
EPI-REZ* SU-8,[4] phr	8.2	—	—
% Stoichiometry	100	100	100
Properties			
T_g (tan delta),[5] °F	474	554	498
Moisture gain,[6] %	5.7	2.8	1.2
Flexural properties (RT/dry)			
Strength, ksi	20	18	22
Modulus, ksi	559	485	530
Flexural properties (Hot/wet)[7]			
Strength, ksi	11	13	14
Modulus, ksi	361	433	419
Modulus (Hot/wet vs. RT/dry)			
Retention, %	65	89	84

[1]Cure schedule: 2 hrs. @ 300°F; 4 hrs @ 400°F
[2]Tetraglycidylmethylenedianiline
[3]Diaminodiphenylsulfone, DDS (Registered trademark of Sumitomo Chemical Co., Ltd.)
[4]Glycidyl ether of a bisphenol A Novolac (Interez, Inc.)
[5]T_g (tan delta) is obtained using a Rheometrics Viscoelastic Tester
[6]After 48 hr. water boil
[7]Tested in water at 200°F after two weeks immersion at 200°F
*Reg. TM - EPI-REZ (Interez, Inc.)

Table 3/**High temperature performance of the EPON HPT® Resin 1079/EPON HPT® Curing Agent 1062-M (39 phr) system compared to the TGMDA/SU-8/DDS system under dry and wet conditioning[1].**

	TGMDA/ SU-8/DDS[2]	Epon HPT resin 1079/ Epon HPT curing agent 1062-M[3]
Flexural modulus, ksi		
Room temperature dry	555	460
300°F dry	370	380
300°F wet[4]	215	355
350°F dry	330	355
350°F wet[4]	155	330
% Retention of RT/dry		
@ 300°F wet	39	77
@ 350°F wet	21	72

[1]Cure schedule: 2 hrs. @ 300°F, 4 hrs. @ 400°F
[2]TGMDA/SU-8/DDS = 90/10/29.7 parts
[3]Epon HPT resin 1079/Epon HPT curing agent 1062-M = 100/39
[4]After 48 hr. water boil

Increasing the molecular weight between crosslinks does not significantly improve the toughness of Epon HPT resin 1079 formulations; i.e., increasing the concentration of curing agent does not improve the toughness of these materials. However, increasing the molecular weight between crosslinks improves the ability of Epon HPT resin 1079 formulations to be toughened using second phase toughening agents, such as CTBN rubbers or thermoplastics. Thus, by using excess curing agent second phase toughening of these materials becomes more effective, Table 4.

These concepts apply to other highly crosslinked thermosetting resins. However, due to the initially high temperature resistance of Epon HPT resin 1079 formulations, these concepts can be applied while maintaining a glass transition temperature above 400°F. Adhesive properties are shown in Table 5.

Table 4/**Neat resin casting properties for EPON HPT® Resin 1079 toughened with CTBN rubbers[1]**

Components		
Epon HPT resin 1079	100	100
Epon HPT curing agent 1062-M	39.2	57.1
CTBN rubber modifier	—	10.0
Stoichiometry, %	100	130
T_g (tan delta),[2] °F	498	324
Moisture gain,[3] %	1.2	1.2
Flexural properties (RT/dry)		
Strength, ksi	22	19
Modulus, ksi	530	501
Flexural properties (Hot/wet)[4]		
Strength, ksi	14	13
Modulus, ksi	419	429
Fracture toughness, K_q, psi-in$^{1/2}$	455	1,236

[1]Cure schedule: 2 hrs. @ 300°F, 4 hrs. @ 400°F
[2]T_g (tan delta) is obtained using a Rheometrics Viscoelastic Spectrometer
[3]After 48 hr. water boil
[4]Tested in water at 200°F after two weeks immersion at 200°F

Table 5/**Adhesives properties of EPON HPT® Resin 1079/EPON HPT® Curing Agent 1062-M modified with CTBN - 10%**

Temperature, °F	Lap shear, psi	Break type
77	2350	Adhesive
200	2740	Cohesive
250	3220	Cohesive
300	3290	Cohesive
350	2610	Cohesive
400	1670	Cohesive

Formulation: Epon HPT resin 1079/CTBN/Epon HPT curing agent 1062-M/talc = 90/10/51.7/45.3
Cure: 2 hours @ 300°F, 4 hours @ 400°F

Packaging and storage

Epon HPT resin 1079 is available in 2-pound, 8-pound, and 40-pound quantities. It is not defined as hazardous by criteria of DOT regulations. Product should be stored at standard ambient conditions, less than 80° F.

Handling precautions

The recommendations for material selections made in this bulletin are based on Shell's experience and research and are believed to be sound technical approaches to the applications or end uses for which they are presented. However, these recommendations are directed solely toward technical performance and should not be taken as recommendations pertaining to health, safety or the environment.

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Epon HPT resin 1079 and the auxiliary materials normally combined with this product are capable of producing adverse health effects ranging from minor skin irritation to serious systemic effects. Adverse effects can be minimized and most can be avoided through the observance of proper precautions, use of proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these depends on responsible action by adequately informed personnel.

A Material Safety Data Sheet (MSDS) is available for this product (MSDS #800633). **Transportation, storage, handling, and use of this product should not occur until handling precautions and recommendations, as described in the MSDS, are understood by all persons who will work with it.** Questions and requests for MSDS sheets or other information should be directed to your Shell Chemical Company Sales Office. Information on non-Shell products should be obtained from the respective manufacturer or vendor.

SHELL RESINS

EPON® Resin SU-8*

Introduction
EPON® Resin SU-8 is a polymeric solid epoxy resin possessing an average epoxide group functionality around eight. Epon SU-8 is compatible with bisphenol A-based epoxy resins, imparting improved high temperature strength, thermal stability, reactivity and chemical resistance. Prepreg laminates and graphite or boron reinforced composites with Epon SU-8 attain the maximum strength retention and thermal stability possible for an epoxy matrix system at elevated temperatures. Epoxy molding powders prepared with Epon SU-8 are characterized by an outstanding combination of flow stability and short press cycles.

Features
- Long shelf life
- Micropulverized at ambient temperatures
- Rapid development of hot hardness
- Maximum elevated temperature strength retention
- Improved tack qualities and lateral cohesiveness of unidirectional tapes

General Information
Epon Resin SU-8 can be used as a modifier for upgrading the elevated temperature properties and the reactivity of bisphenol A epoxy resin systems for molding powders or powder coatings. The high molecular weight of Epon SU-8 improves the tack qualities and lateral cohesiveness of unidirectional fiber reinforced tapes prepared with many epoxy matrix systems.

For prepreg applications, Epon SU-8 can be dissolved in numerous solvents such as most ketones, toluene, and diacetone alcohol.

*Formerly EPI-REZ® Resin SU-8.

Typical Properties

Melting Point, Durrans, °C	82
Reduced Viscosity[1], Gardner-Holdt	E
Pounds/Gallon	10.0
Color[1] Gardner	<5
Equivalent Weight	215
Flash Point, Pensky-Martens, °F	>200

1. Reduced to 40 percent nonvolatiles in 2-(2-butoxyethoxy) ethanol.

Suggested formulations are available illustrating the use of Epon SU-8 in transfer and compression molding powders, high temperature adhesives, NEMA G-11 and FR-5 circuit boards of both conventional thickness and MLC types, advanced engineering composites, and other high temperature reinforced plastics applications such as those requiring MIL-RS-9300A, Type II properties.

Epon SU-8 may be processed and cured with a variety of epoxy resin curing agents. Table 1 lists a number of curing agents for several different types of applications.

Table 2 and Figure 1 profile the excellent performance of Epon SU-8 relative to other commercially available high functionality resins.

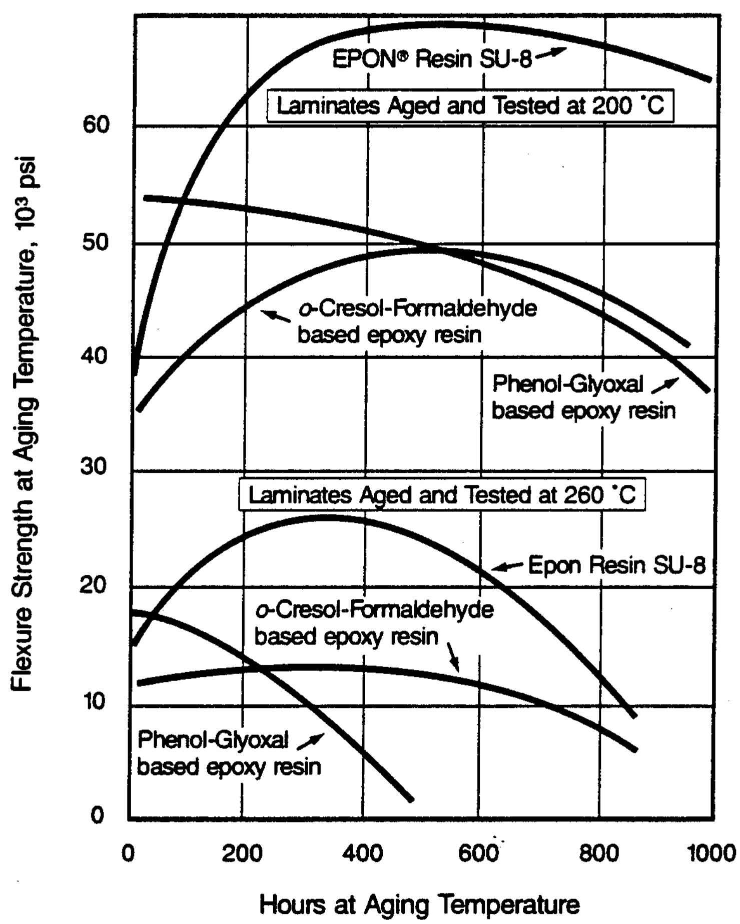
Storage

Epon Resin SU-8 should be stored in tightly sealed metal or polyolefin plastic containers at normal room temperature.

Handling Precautions

This product and the auxiliary materials normally combined with it are capable of producing adverse health effects ranging from minor skin irritation to serious systemic effects. Exposure to these materials should be minimized and avoided if feasible through the observance of proper precautions, use of appropriate engineering controls and proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these preventive measures depends upon responsible action by adequately informed persons. **None of these materials should be used, stored, or transported until the handling precautions and recommendations as stated in the Material Safety Data Sheets (MSDS) for this and all other products being used are understood by all persons who will work with them.** Questions and requests for information on Shell products should be directed to your Shell Chemical Company Sales Representative or the nearest Shell Chemical Sales Office. Information and MSDSs on non-Shell products should be obtained from the respective manufacturer or vendor.

Figure 1/**Thermal Stability of Glass Laminates[1] Prepared with High Functionality Solid Epoxy Resin/Anhydride/Catalyst/EMI-24 Binder Systems**



1. 12-ply laminates prepared with 181 Style glass cloth (I-550 finish). All binder systems consist of the designated epoxy resin cured with methylendomethylene tetrahydropthalic anhydride at 90 percent stoichiometric level and accelerated with 2-ethyl-4-methyl imidazole at 0.22 phr concentration. Laminates were press-cured 1 hour at 150 °C, then post-cured 16 hours at 200 °C prior to initiating aging tests in forced-air ovens.

Table 1/**Curing Agents for Several Applications**

Molding Powders	Reinforced Plastics	Powder Coatings
Phenolics	Phenolics	Phenolics
Dicyandiamide	Dicyandiamide	Dicyandiamide
Phthalic anhydride	Methyl endomethylene tetrahydrophthalic anhydride	Trimellitic anhydride
Tetrachloro-phthalic anhydride	BF_3MEA[1]	Melamine
4,4′-Methylene-dianiline	Imidazoles	
	4,4′-Diaminodiphenyl sulfone	

1. BF_3MEA is boron triflouride monoethylamine.

Table 2/**Contribution of Various High Functionality Solid Epoxy Resins to Properties of a Phenolic Cured Molding Powder Compound**

	EPON® Resin SU-8 82 °C Melting Point	***o*-Cresol/ Formaldehyde** 80 °C Melting Point
Composition (parts by weight)		
Epon Resin SU-8	100	—
o-Cresol-Formaldehyde Novolac Type, 80 °C Melting Point	—	100
Phenolic Curing Agent	52	48
Pigment	5	5
Candelilla Wax	3	3
325-Mesh Silica Filter	250	240
Spiral Flow[1], inches	31	42
Cure State Properties[2]		
Heat Deflection Temperature, °C	192	175
T_g °C	194	158
Tensile Strength, psi	7,400	7,200
Flexural Strength, psi	15,000	14,000
Compressive Yield Strength, psi	26,000	27,000
Hot Hardness[3], Shore D	92	84
Linear Shrinkage, inch/inch	0.0068	0.0070
Electrical Properties		
Dielectric Constant	5.07	5.13
Dissipation Factor	0.0070	0.0068

1. Determined at 1,000 psi and 150 °C per EMMI 1-66.
2. Determined on bars molded from dielectrically heated preforms in a transfer press at 1,000 psi and 150 °C for 3 minutes.
3. Determined immediately upon ejection from 150 °C mold.

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SC: 1580-93

TACTIX 742 RESIN

TACTIX 742 gives the highest glass transition temperature of any resin in the TACTIX line. When fully cured with DDS, this tri-functional, tris (hydroxyphenyl) methane-based epoxy creates a tightly cross-linked matrix with a T_g over 300°C.

Plus, TACTIX 742 has outstanding thermal oxidative stability, and good moisture resistance and modulus. When compared to Araldite MY-720, TACTIX 742 has both lower moisture pick-up and a higher dry T_g. And following a 200 hour water boil, TACTIX 742 retained 44% of its room temperature/dry modulus. These properties make TACTIX 742 an excellent matrix resin for both commercial and military structural composites and adhesives.

As with other solid resins, processing is easier with the addition of diluents. The reduction in viscosity for various blends of TACTIX 742 and TACTIX 123 can be seen in Figure 2 as a function of temperature. Using TACTIX 123 at

TABLE 2

Typical Resin Properties† of TACTIX* 742

Epoxide Equivalent Weight	150-170
Viscosity, cst @ 150°C	25-60
Resin Form	Semi-Solid
Volatiles, wt. %, max.	0.5
Color, Gardner, max.	13

†Typical properties; not to be construed as specifications.
*Trademark of The Dow Chemical Company

FIGURE 2

Viscosity Curves of TACTIX 742/TACTIX 123 Blends With Temperature

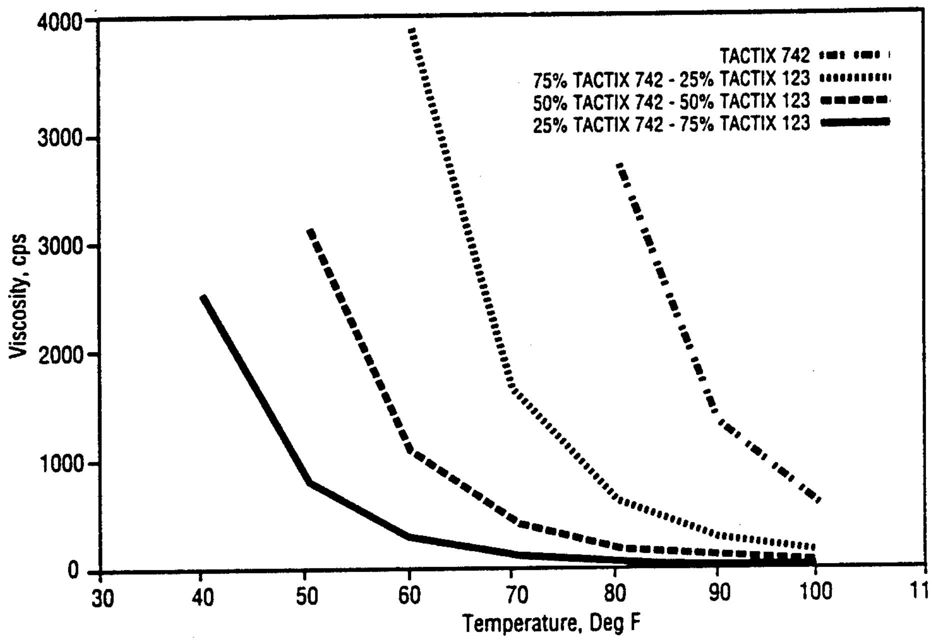


TABLE 3

Mechanical Properties of Unreinforced Resin Castings of 75% TACTIX* 742/25% TACTIX 123 Resins[1,2†]

Tensile (RT/dry[3])	
Strength, ksi	10.4
Modulus, ksi	455
Elongation, %	2.7
Flexural (RT/dry)	
Strength, ksi	18.0
Modulus, ksi	480
Moisture Absorbance, 200 hour water boil, wt. %	4.28
Glass Transition Temperature, °C by DSC	299
G_{IC}, in-lb/lb²	0.57

[1]% by weight
[2]Resin cured with 4,4-diaminodiphenylsulfone, 3 hours @ 177°C + 2 hours @ 250°C
[3]Room temperature, dry conditions
†Typical properties; not to be construed as specifications
*Trademark of The Dow Chemical Company

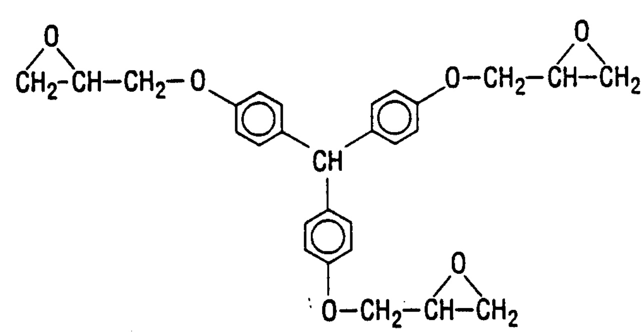


FIGURE 3

T_gs of TACTIX 742/TACTIX 123 Blends

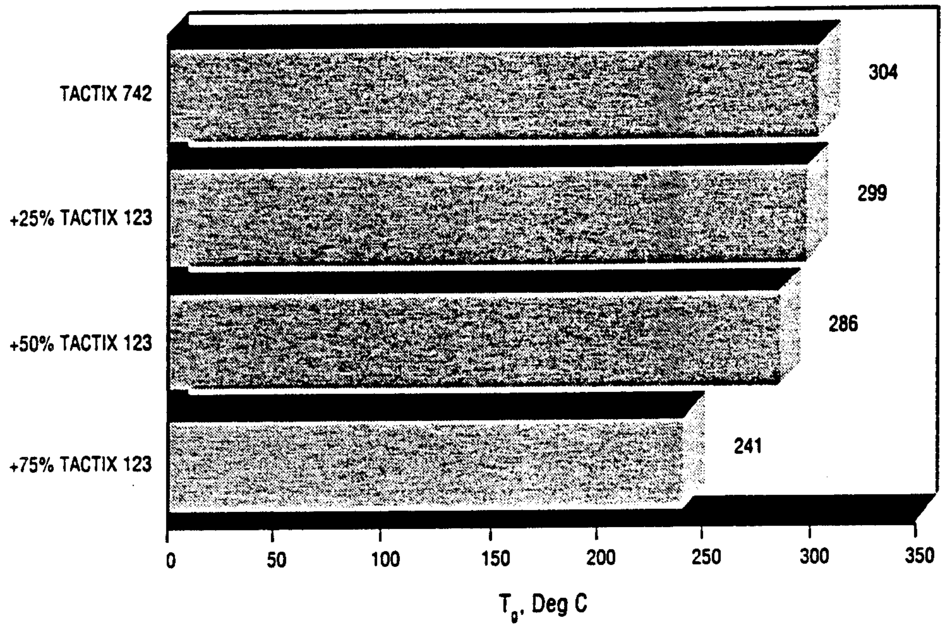
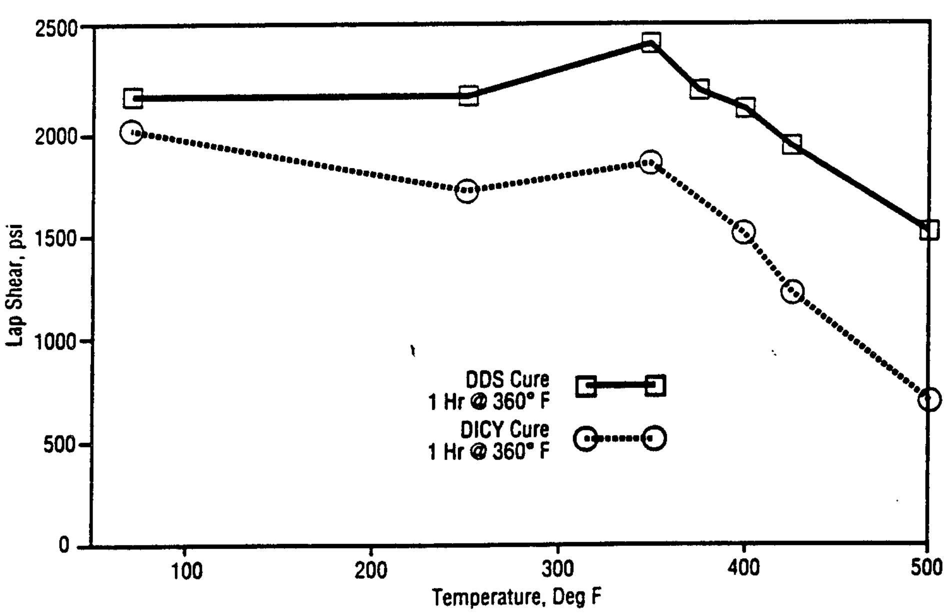


FIGURE 4

Lap Shear of TACTIX 742 Resin Cured With DDS or DICY vs. Temperature



25% by weight has no effect of glass transition temperature, as seen in Figure 3. Even a 50/50 blend of TACTIX 742 and TACTIX 123 lowers T_g by only 14°C. In addition, diluting with liquid bis A resins should increase some mechanical properties such as flexural strength and elongation.

TACTIX 742 resin is used most commonly as an adhesive, especially for applications near high heat zones like aircraft engine nacelles. Its lap shear values approach 2,500 psi at about 350°F when cured with DDS for 1 hour at 360°F (Figure 4). DICY can also be used to cure TACTIX 742 for adhesive applications but gives lower lap shear.

SC:235-86.131
(Supersedes SC:235-85.131)

Technical Bulletin
Shell Chemical Company

EPON® Resin 1031

General description

EPON® Resin 1031 is a solid multifunctional epichlorohydrin/tetraphenylol ethane epoxy resin. It is used to improve the properties of cured epoxy resin systems particularly at elevated temperatures. It finds application in electrical laminates, high performance aerospace composites and adhesives, powder coatings and molding compounds.

Epon resin 1031 features

- An average of greater than three reactive groups per molecule
- Low ionic contaminants
- Low saponifiable chloride
- Easily ground into uniform particle size
- Low melt viscosity
- Stability on storage

Sales specifications

Epoxide equilavent weight	200-240
Viscosity, Gardner-Holdt, 25° C	Z_2-Z_7

Typical properties

Color[1], Gardner	>18
Melt viscosity, at 150° C, poise	15
Saponifiable chloride, %w	0.02-0.06
Sodium, ppm	<10

[1] 80%w solution in MEK

Benefits of using Epon resin 1031

The use of Epon resin 1031 in epoxy resin formulations increases the cross link density of cured systems. This raises the glass transition temperature. At elevated temperatures such systems have greater strength and rigidity and show improved retention of electrical properties and resistance to attack by moisture.

Epon resin 1031 is commonly included in formulations to make laminates for the support of both macro and micro electrical circuits. Formulations using this resin meet the more demanding dimensional stability requirements of multilayer printed circuit boards. The low ionic contaminants in this resin contribute to the excellent resistance to electrical flow. The low saponifiable chloride content shortens cure time with selected curing agents, thus improving handling characteristics and speed of production of the laminates.

Epon resin 1031 is also used in structural composites and adhesives. High performance products are made for aircraft and aerospace use where cured resin systems are used for metal-to-metal bonding and structural components. The excellent adhesive properties of epoxies as well as the retention of other physical properties at higher temperatures are particularly important in these end uses.

The ease of grinding Epon resin 1031 into uniform particle sizes combined with its low melt viscosity make it an excellent candidate for use in epoxy resin powder coatings and molding compounds.

FDA status

Epon resin 1031 is not recommended for food contact use.

Packaging and storage

Epon resin 1031 is supplied in 200 lb. fiber drums. It is also available as a solution of 80%w resin in methyl ethyl ketone or of 70%w resin in acetone.

Handling precautions for systems based on Epon resin 1031

The recommendations for material selection made in this brochure are based upon Shell's experience and research and are believed to be sound technical approaches to the applications or end-uses for which they are presented. However, these recommendations are directed solely toward technical performance and should not be taken as recommendations pertaining to health, safety or the environment.

Epon resin 1031 and the auxiliary materials normally combined with this resin are capable of producing adverse health effects of varying degrees of seriousness ranging from minor skin irritation to serious systemic effects.

These can be minimized and most can be avoided through the observance of proper precautions, use of proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these depends upon responsible action by adequately informed persons. None of these materials should be transported, stored, or used until handling precautions and recommendations are understood by all persons who will work with them.

Questions and requests for information on Shell products should be directed to your Shell Chemical Company Sales Representative or to the nearest Shell Chemical Sales Office. Information on non-Shell products should be obtained from the respective manufacturer or vendor.

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All products purchased from or supplied by Shell are subject to terms and conditions set out in the contract, order acknowledgement and/or bill of lading. Shell warrants only that its product will meet those specifications designated as such herein or in other publications. All other information supplied by Shell is considered accurate but is furnished upon the express condition that the customer shall make its own assessment to determine the product's suitability for a particular purpose. **Shell makes no other warranty, either express or implied, including those regarding such other information, the data upon which the same is based, or the results to be obtained from the use thereof; that any product shall be merchantable or fit for any particular purpose; or that the use of such other information or product will not infringe any patent.**

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February 1989

2.5M

SC:735-86
(Supersedes SC:735-84)

Technical Bulletin
Shell Chemical Company

EPON® Resin DPS-164

General description

EPON® Resin DPS-164 is a solid multifunctional epichlorohydrin/cresol novolac epoxy resin (see molecular structure). It combines the high thermal stability of the novolac backbone with the versatility, reactivity, and chemical resistance of epoxy resins. It is used where improved properties of cured epoxy resin systems are needed, particularly at elevated temperature and where stability of electrical properties under humid conditions are required. It finds application in electrical laminates, molding compounds, high performance aerospace composites, high temperature adhesives, powder coatings, and tooling.

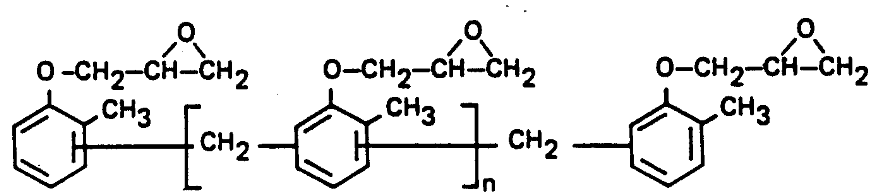
Features

- An average of five reactive epoxide groups per molecule
- Low ionic contaminants
- Low saponifiable chloride
- Easily ground into uniform particle size
- Low melt viscosity
- Stability on storage

Specifications

Epoxide equivalent weight[a] (Shell Method HC-427C-81 Perchloric Acid Method)	200-240
Viscosity[b], Centipoise @ 25°C (ASTM D 445-79)	35-50
Color[b], Gardner (ASTM D 1544-68)	6 max.

[a]Grams of resin containing one gram-equivalent of epoxide.
[b]Determined on a 60%w solution in methyl ethyl ketone.



Where n equals an average of 3

Typical properties	
Vapor pressure, @ 25°C	Negligible
Solubility in water	Negligible
Melt viscosity, Cannon-Fenske cSt, @ 150°C (ASTM D 445-79)	600-1200
Melt viscosity, ICI Cone & Plate, poise @ 150°C	9-14
Weight per gallon, @ 20°C, pounds	10.2
Flash point, Setaflash (ASTM D 3287-73)	>200°F
Bulk density, lbs./ft.^{3}	35-40
Saponifiable chloride, %w	<0.005
Total chloride, %w	<0.15
Free chloride, ppm	<1
Sodium, ppm	<5
Tg (DSC), °C	37-39
Melting point, @ °C (ASTM D 3461, Mettler, 1°C/min.)	82
Water, %w (as manufactured)	0.3 max.

Benefits of using Epon resin DPS-164

The use of Epon resin DPS-164 in epoxy resin formulations increases resistance to attack by moisture, solvents, and environment. In addition, it brings higher glass transition temperature (Tg) and cross-link density to cured systems which provides improved retention of strength, rigidity, electrical, and other properties at elevated temperatures. Epon resin DPS-164 is commonly used in formulations for making structural laminates, electrical printed circuit boards, and electronic molding powders which require higher performance temperatures and greater dimensional stability.

Epon resin DPS-164 is easily ground into powders or blended with other epoxy resins. It is compatible with most materials used with BPA-based epoxies and its low melt viscosity provides ease of handling and good flow characteristics. Its low ionic contamination plus ultra low saponifiable chloride prevents potential electrical interference in sensitive electronic devices. Because of this, DPS-164 is the resin of choice for transfer molding compounds in semiconductors, relays or other active or passive components in the electronic field.

The high functionality of Epon resin DPS-164 shortens cure times and improves handling and speed of production. The resin has excellent adhesive properties needed for bonding both metal and non-metal structural components. Typically, it maintains low weight loss in heat aging of cured systems. For these reasons, DPS-164 is preferred for high temperature adhesives, structural composites, and other high performance products in the aircraft and aerospace industry.

Specification properties

The typical viscosity and epoxide equivalent for Epon resin DPS-164 are mid-range of the sales specifications (see table). It is a very "clean" resin, i.e. low in sodium, free chloride, and other ionics and in total chlorine. In addition, it has low saponifiable chloride which may enhance long term stability as an encapsulant for electrical components and semiconductors.

Handling properties

Epon resin DPS-164 can be handled by melting (melting point ca. 80°C), as a blend with Epon resin 828 [liquid bisphenol A/epichlorohydrin (BPA/ECH) epoxy resin] or other resins, as a powder, or from solution.

Figure 1 shows the viscosity-temperature profile for the neat resin. It has a low melt viscosity of ca. 10 poise at 150°C (302°F). It can be readily blended with other resins, fillers, curing agents, and additives for formulating needs.

Figure 1 also shows viscosities for blends of Epon resin DPS-164 with Epon resin 828. Typically, blends provide reductions in viscosity and cost while maintaining a useful balance of Tg and other performance properties.

Figure 1/**Melt viscosity for EPON® Resin DPS-164 and blends with EPON® Resin 828**

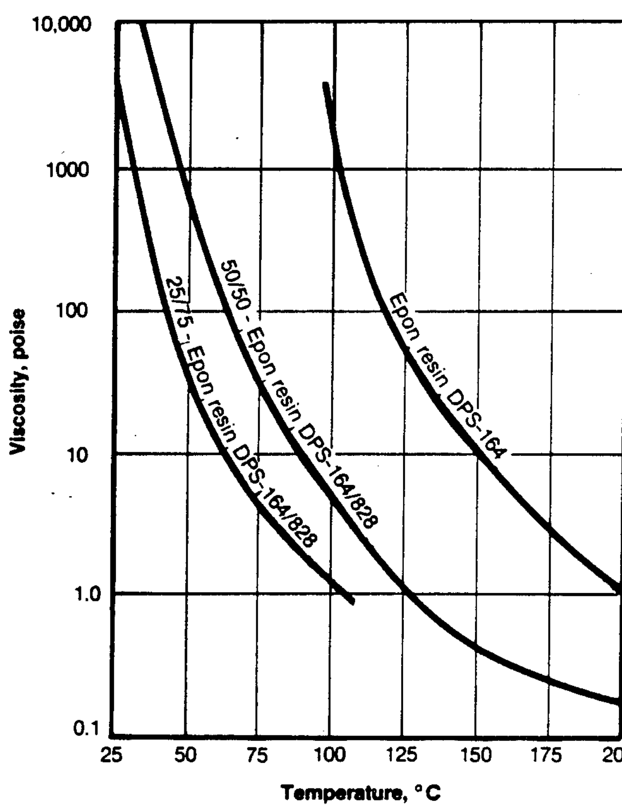
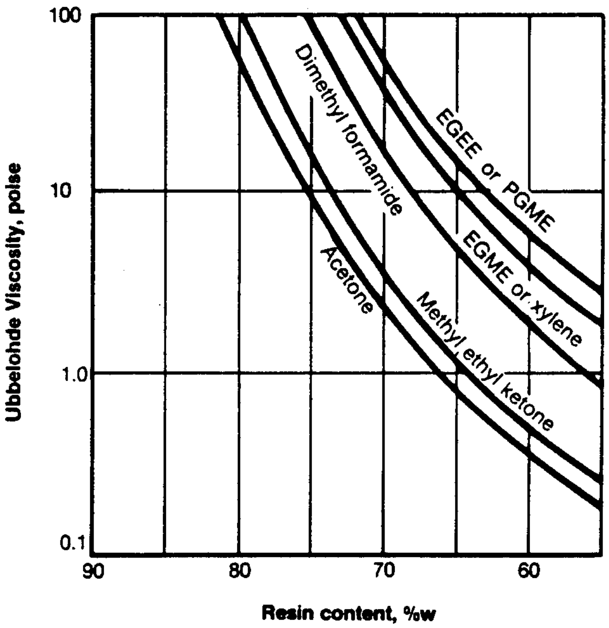


Figure 2/**Viscosities for solutions of EPON® Resin DPS-164 (at 25°C)**



Specific Gravity			
Solvent	**Percent wt. resin**		
	60	70	80
Acetone	1.00	1.05	1.11
MEK	1.01	1.05	1.10
DMF	1.09	1.11	—
Xylene	1.04	1.08	—
Ethylene glycol methyl ether (EGME)	1.09	1.12	—
Ethylene glycol ethyl ether (EGEE)	1.085	1.115	—
Propylene glycol methyl ether (PGME)	1.08	1.11	—

Figure 2 shows solution viscosities for Epon resin DPS-164 with a variety of solvents. Generally, ketones provide lower viscosities at higher resin solids than other solvents. For example, up to 75%w resin may be cut into MEK for 10-20 poise solution viscosity at room temperature, while only 65%w resin can be added to glycol ethers for the same viscosity.

Performance of catalyzed systems

Unfilled casings — Table 1 shows typical properties for Epon resin DPS-164 cured with NADIC Methyl Anhydride (NMA) and diaminodiphenylsulfone (DADS) which are commonly used curing agents in high performance applications. These properties demonstrate superiority of performance over more conventional BPA/ECH epoxies. Epon resin DPS-164 systems exhibit high Tg's and heat deflection temperatures (HDT's) and better retention of properties at elevated temperatures (see Table 1). They

Table 1/**Physical properties for unfilled castings of EPON® Resin DPS-164**

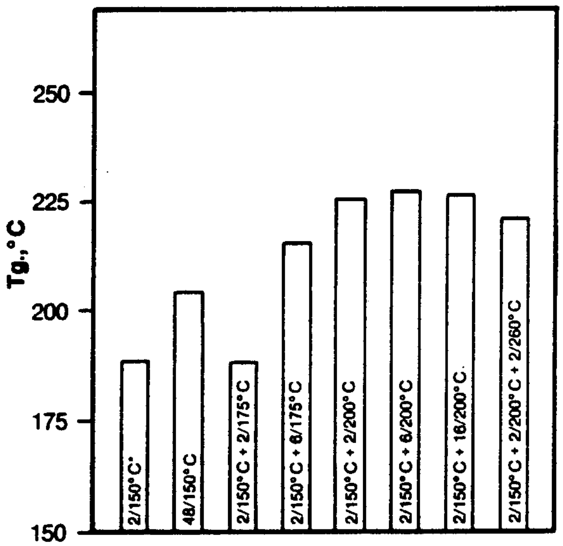
Curing agent Concentration, phr	**NMA 70**[1]	**DADS 25**[2]
Tg, °C (DSC)[3]	230	236
Deflection temperature, °C (ASTM D-648)	225	>225
Density, 25°C, g/ml	1.256	1.198
Tensile strength at break, 23°C, psi	8,200	10,300
Tensile elongation at break, 23°C, %	2.0	2.3
Tensile modulus, psi x 10^6	0.51	0.54
Flexural strength at break, 23°C, psi	18,500	23,000
Flexural modulus, 23°C, psi x 10^6	0.49	0.53
Compressive strength, 23°C, psi		
0.2% Offset	14,000	14,600
Yield	24,200	28,400
Compressive deformation, %		
0.2% Offset	4.5	4.2
Yield	15	17
Compressive modulus, psi x 10^6	0.50	0.52
Dielectric constant		
1 KH_z	3.47	4.46
1 MH_z	3.21	3.75
50 MH_z	3.12	3.60
Dissipation factor		
1 KH_z	0.007	0.014
1 MH_z	0.019	0.021
50 MH_z	0.015	0.031
Dielectric strength (1/8 in), volts/mil	430	560
Volume resistivity, ohm-cm	6 x 10^{16}	3 x 10^{16}
24 Hour water boil, %w gain	0.94	1.43
3 Hour acetone boil, %w gain	-0.03	-0.08
Coefficient of expansion, in/in °C x 10^{-6}		
-10°C to 130°C	22	17
130°C to 230°C	35	30
230°C to 260°C	67	61

[1]Plus 1 phr EMI catalyst; cure, hours/°C = 2/120 + 4/150 + 16/200.
[2]Cure, hours/°C = 4/150 + 16/200.
[3]Differential scanning calorimetry, heating rate 40°C/minute.

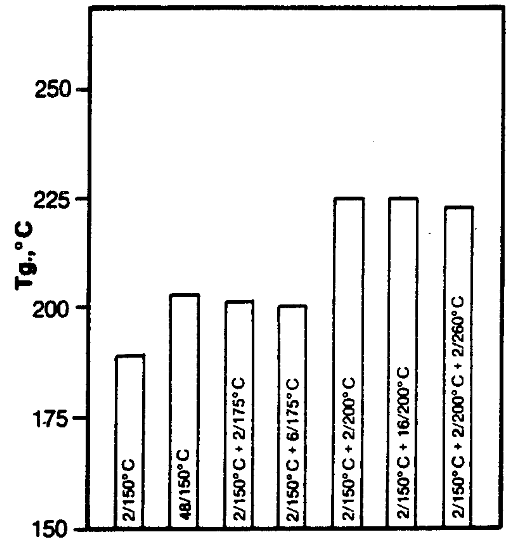
display superior strength and modulus which translates to greater stiffness and rigidity and an ability to withstand higher loadings. They have lower coefficient of linear thermal expansion and excellent electrical properties which makes them an excellent choice for encapsulation use. By contrast, Epon resin DPS-164 systems are less flexible than BPA/ECH epoxies and have relatively lower tensile strength and elongation.

Cure cycles versus Tg — Figure 3 provides guidelines for selecting and optimizing cure cycle requirements. Data generally show that higher cure temperatures of 175°C-200°C are required to

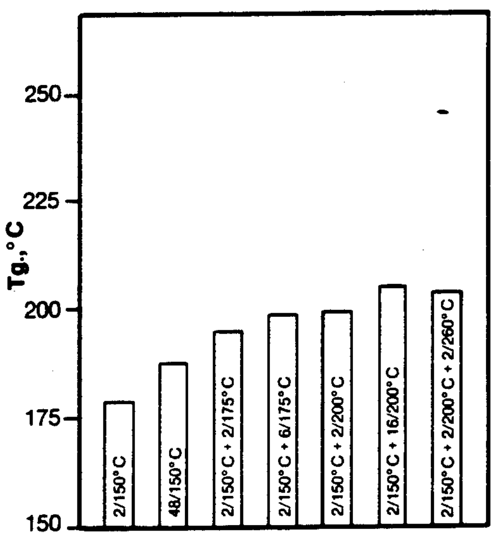
Figure 3/**Effect of cure temperature and time[1] on Tg[2] for EPON® Resin DPS-164 and selected curing agents**



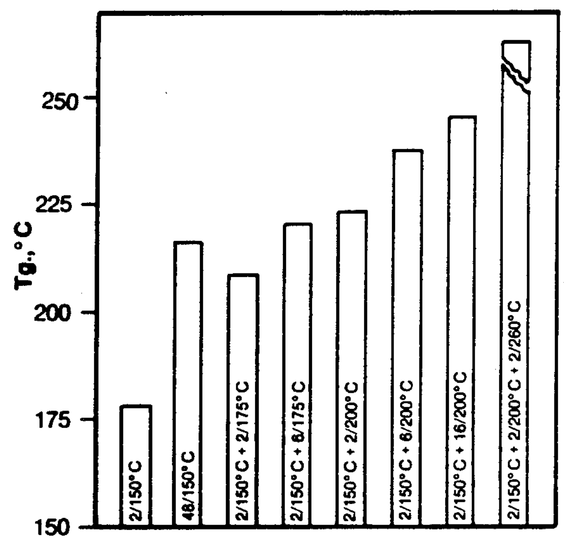
2 - Ethyl, 4-Methylimidazole - 3 phr



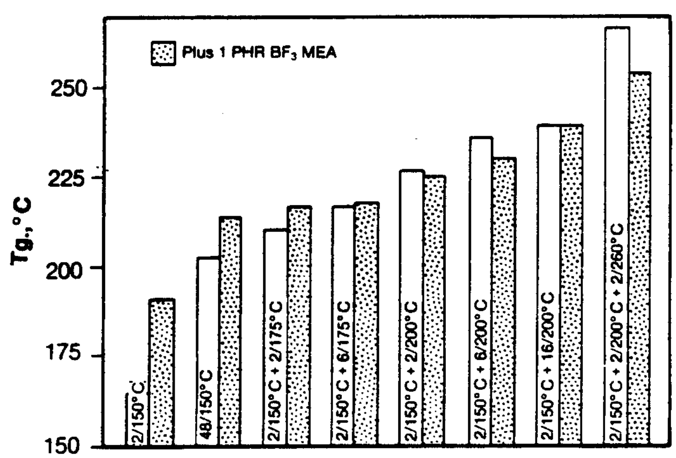
EPON CURING AGENT® Y - 21.5 phr



CRJ 406[3] - 54 phr



Nadic methyl anhydride/EMI - 67/1 phr



Diaminodiphenylsulfone - 20 phr

[1] The cure temperature and time (hours) are noted in each bar of the graphs

[2] The Tg in °C is determined by differential scanning calorimetry, heating rate 40°C/minute

[3] CRJ 406 is a cresylic novolac produced by Schenectady Chemicals Inc. This system is further catalyzed by 0.2 phr 2-methylimidazole

achieve a high Tg. Also, when higher cure temperatures are used, relatively short cure cycles provide full cure to the resin. When low temperature cures are used, Tg's are lower, although still above 150° C, and may be adequate for many uses. Higher Tg's with short cures can be obtained by adding auxiliary catalysts such as boron trifluoride monoethylamine complex (BF_3MEA) and 2-methylimidazole (2-MI) to selected systems. Higher Tg's can also be obtained using longer low temperature cures (e.g., 48 hours at 150° C) without added auxiliary catalysts.

Reactivity — Figure 4 shows gel times for Epon resin DPS-164 and its blends with Epon resin 828 for several curing agents. This information may be useful in adjusting reactivities for resin systems to meet process needs. Generally, gel times are similar to those obtained with BPA/ECH resins. They show a linear decrease with increasing temperature. Adding Epon resin DPS-164 to Epon resin 828 shortens gel times slightly. Gel time can also be shortened by adding an auxiliary catalyst. Among these catalysts, 2-methylimidazole is more effective than benzyldimethylamine on an equal weight basis.

Effect of curing agent stoichiometry on Tg — Figure 5 shows that only slight changes occur in Tg with varying stoichiometry (or concentration) of the curative in most systems, except with DADS which shows a slightly larger effect. The effect is very small or negligible with catalytic curatives such as 2-ethyl-4-methylimidazole and BF_3MEA in the 2-4 phr range and small (although measurable) with curatives which react with the resin such as NMA and CRJ-406 phenolic resin. Overall, the data suggest that Epon resin DPS-164 is less sensitive to variations in curing agent stoichiometry and weighing errors for metering and mixing equipment. However, we recommend careful optimization of both curative concentration and cure cycle for a particular application because this will provide maximum physical and electrical properties and retention of these properties in the end use conditions. For convenience in preparing cost/benefit estimates, point values are also shown for 50/50 blends of Epon resin DPS-164 with Epon resin 828.

Applications

Effect of resin blends on Tg — Figure 6 shows Tg's for blends of Epon resin DPS-164 with Epon resin 828. Generally, Tg's increase linearly with Epon resin DPS-164 content because of its higher functionality. Systems cured with catalysts, aromatic amines, and NMA have similarly high Tg's, indicating usefulness for high temperature adhesives and composites. The system with CRJ-406 phenolic curative has relatively lower Tg (but still >200° C) but appears to be a tougher system that should be especially useful for molding compounds.

Figure 7 gives the Tg's of blends of Epon resin DPS-164 with Epon resin 1123 (solid brominated laminating resin) or 828. In either case Tg can be raised ca. 10°C with 20%w addition of Epon resin DPS-164. Chemical resistance is also improved and hence there is utility in high performance electrical laminate applications. It is noteworthy that the Tg for 100 percent Epon resin DPS-164 cured with dicyandiamide (Figure 7) is high (195° C). This system has potential for adhesives, prepreg, and many structural uses.

Molding powder — Table 2 shows typical properties for Epon resin DPS-164 molding powders using CRJ-406 phenolic curative plus high (silica) filler loading. These filled Epon resin DPS-164 molding powders also display high Tg, flexural strength, and excellent electrical properties.

Table 3 shows thermal properties for molding compounds like those in Table 2 using blends of Epon resin DPS-164 with Epon resin 1002F. Tg can be varied from 122-201°C depending upon needs. HDT varies between 108-190°C and is always 5-15° C lower than the Tg due to stress loading.

Figure 8 demonstrates the low moisture absorption and superiority of Epon resin DPS-164 in steam processing relative to a typical BPA/ECH epoxy molding compound. It is yet another reason for using Epon resin DPS-164 in harsh environments and electronic applications.

Thin film powder coatings — Figure 9 shows the increase in solvent resistance for powder coatings when Epon resin DPS-164 is added to the typical Epon resin 2002 system. Solvent resistance as measured by MEK double rubs can be more than doubled by the addition of up to 17%w Epon resin DPS-164.

Strength retention at high temperature for glass cloth laminates — Table 4 shows excellent high flexural strength properties for glass cloth laminates based upon Epon resin DPS-164 cured using DADS plus BF_3MEA auxiliary catalyst. Also evident are a very high retention of strength (47%) and modulus (77%) at temperatures up to 225° C. This high retention of strength, electrical, and other properties at elevated temperature is due to high crosslink densities obtained with Epon resin DPS-164 systems.

Because most cured resin systems retain a significant proportion of properties up to their glass transition temperature, frequently the needed performance properties can be predicted based upon the Tg for the system selected. Hence, this bulletin provides a large amount of glass transition temperature data on a variety of systems to provide the reader with starting point compositions plus a guide for optimizing these systems.

Figure 4/**Effect of gel temperature on gel time of EPON® Resin DPS-164 systems**

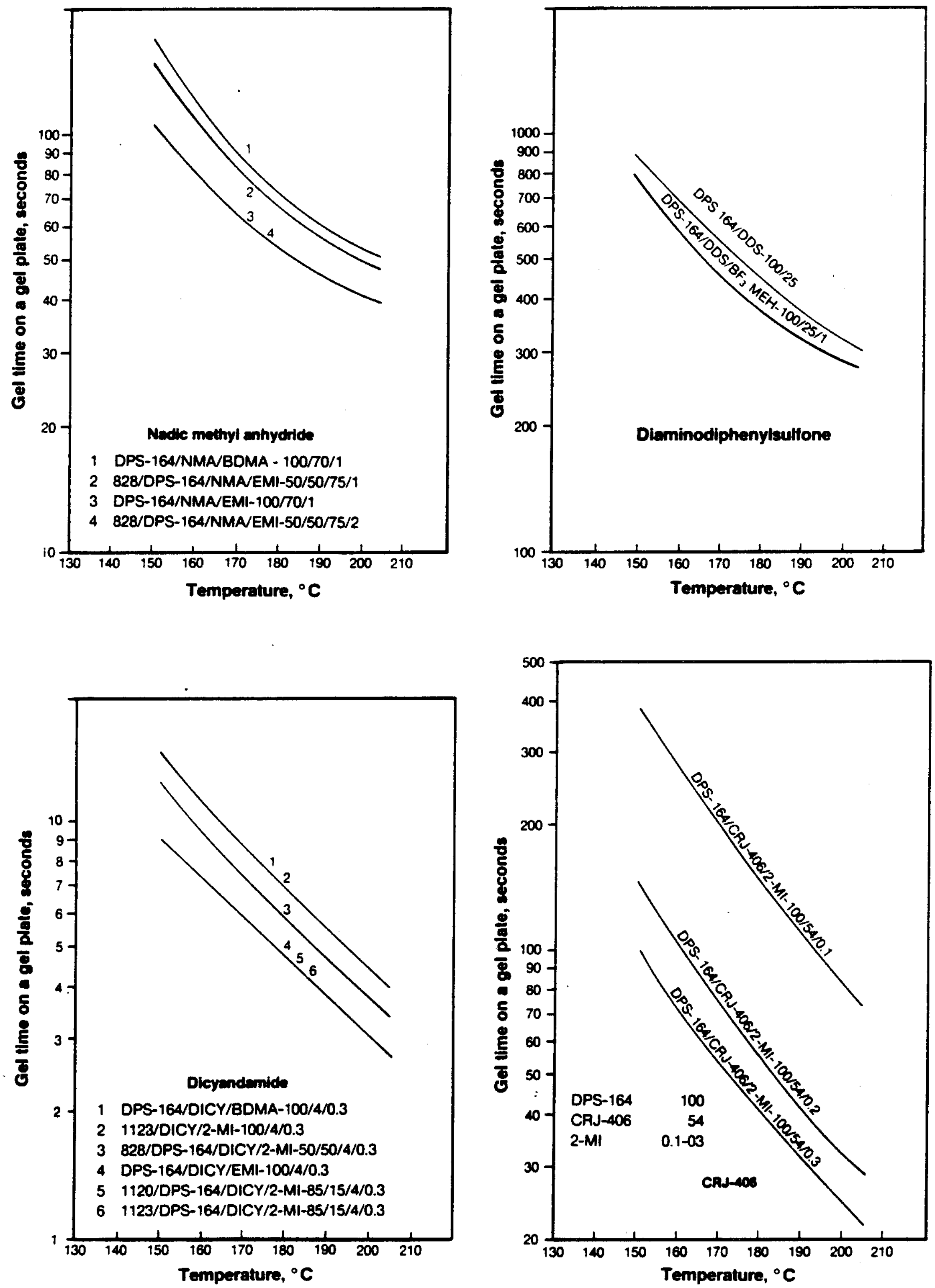
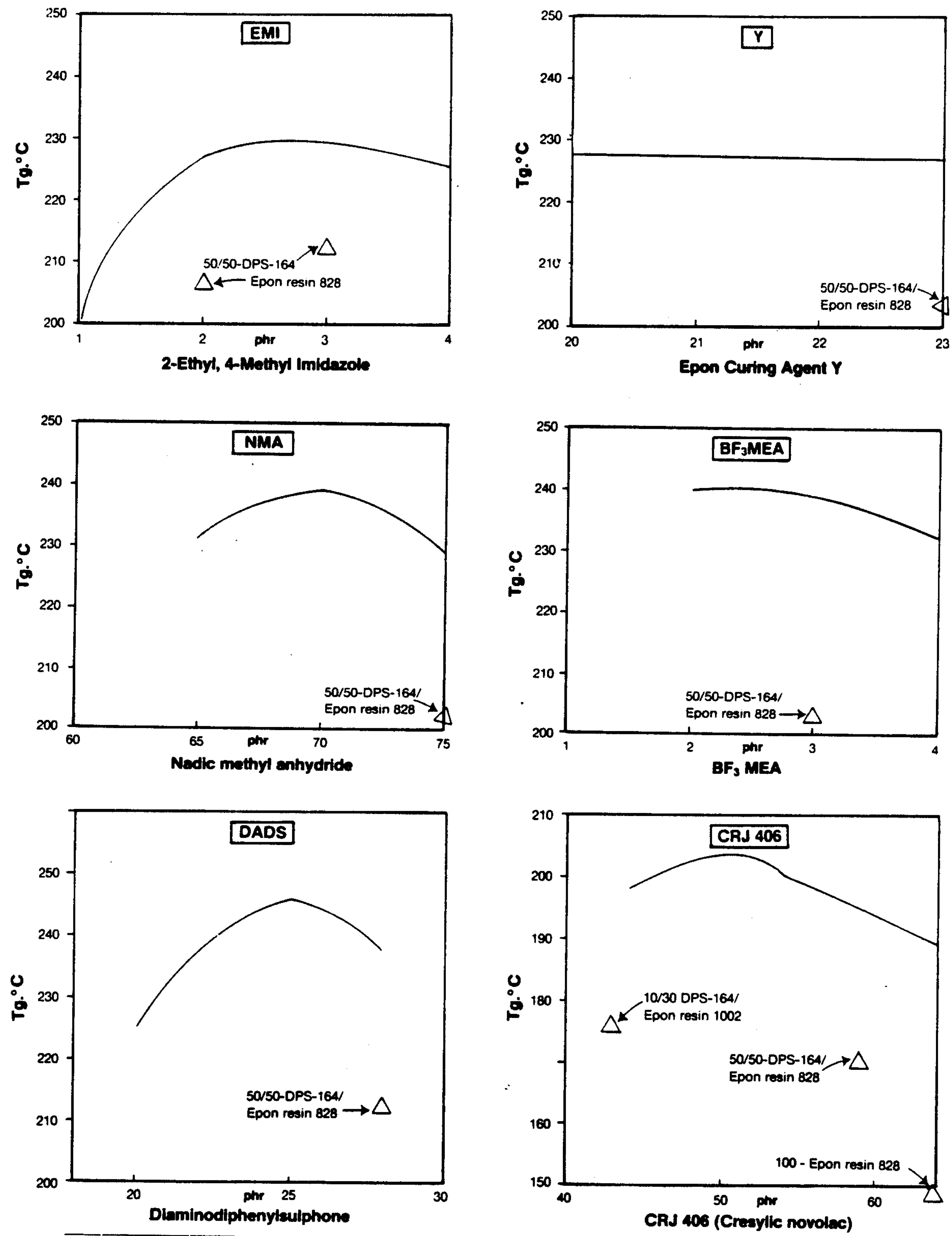
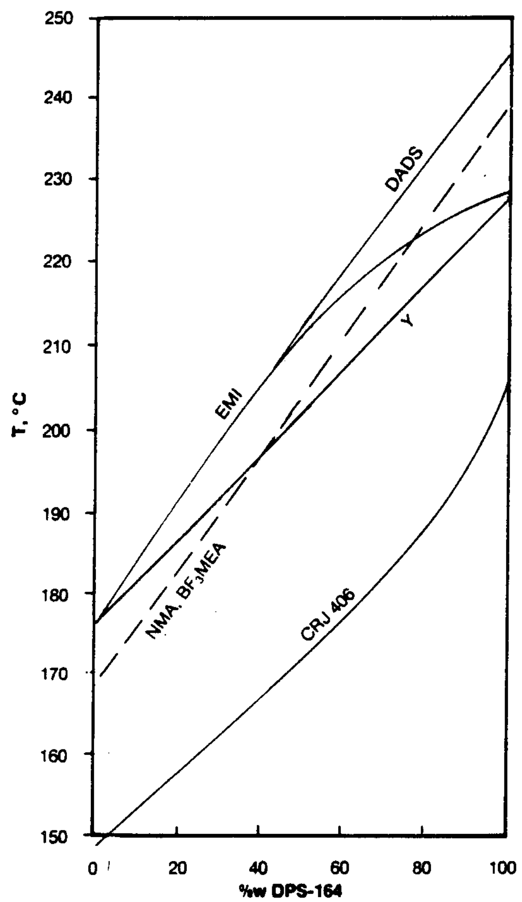


Figure 5/**Effect of curing agent stoichiometry on Tg[1] of EPON® Resin DPS-164 systems**



[1]Tg's by DSC. All systems cured 2 hours @ 150° C plus 2 hours at 200° C

Figure 6/Tg's[1] for blends of EPON® Resin DPS-164 with EPON® Resin 828 with several curing agents



[1]Tg's by DSC. Cure, Hrs/°C = 2/150 + 2/200

Table 2/Properties for EPON® Resin DPS-164 molding powder

Composition	Percent
EPON® Resin DPS-164	19.6
CRJ-406	10.4
2-Methylimidazole	0.08
Silica Flour	70
Properties[1]	
Tg, °C[2]	201
Coefficient of thermal expansion, in/in °C x 10^{-6}	
50-201	32
201-275	79
Flexural strength, 23°C MPa (psi)	123 (17,800)
Flexural modulus, 23°C, MPa (psi)	13,200 (1,900,000)
Weight loss, 120 hours @ 200°C, %w	0.41
Dielectric strength, volts/mil	520
Dielectric constant, 23°C, 1 MHz	3.8
Dissipation factor, 23°C, 1 MHz	.011
Volume resistivity, 23°C, ohm-cm	3.9 x 10^{15}

[1]1/8" molded plaques cured 5 minutes at 150°C under 900 psi molding pressure, then postcured 16 hours at 177°C.

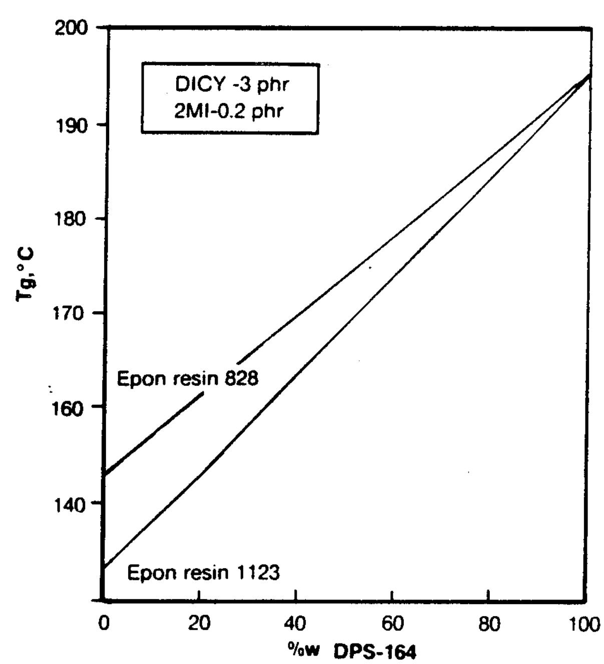
[2]This glass transition value was from tan δ max. as measured by the Rheometrics Mechanical Spectrometer.

Table 3/Effect of EPON® Resin DPS-164 content on HDT and Tg of an electrical molding powder[1]

Resin component				
Epon resin DPS-164	100	70	30	0
Epon resin 1002F	0	30	70	100
HDT, °C	190	177	133	108
Tg, °C	201	182	145	122

[1]These molding powders were 70%w silica flour and 30%w catalyzed resin. The catalyzed resin contained 100 parts EPON® Resins DPS-164 plus 1002F, 0.2 phr 2-methylimidazole accelerator, and varying amounts of CRJ-406 curative as follows 53, 43, 30, and 20 phr respectively.

Figure 7/Tg's[1] for blends of EPON® Resin DPS-164 with EPON® Resin 1123 (Brominated electrical laminating resin) and EPON® Resin 828



[1]Tg's by DSC. Cure, Hrs/°C = 2/150 + 2/200

Figure 8/**Moisture absorption in 15 PSIG steam for an EPON® Resin DPS-164**

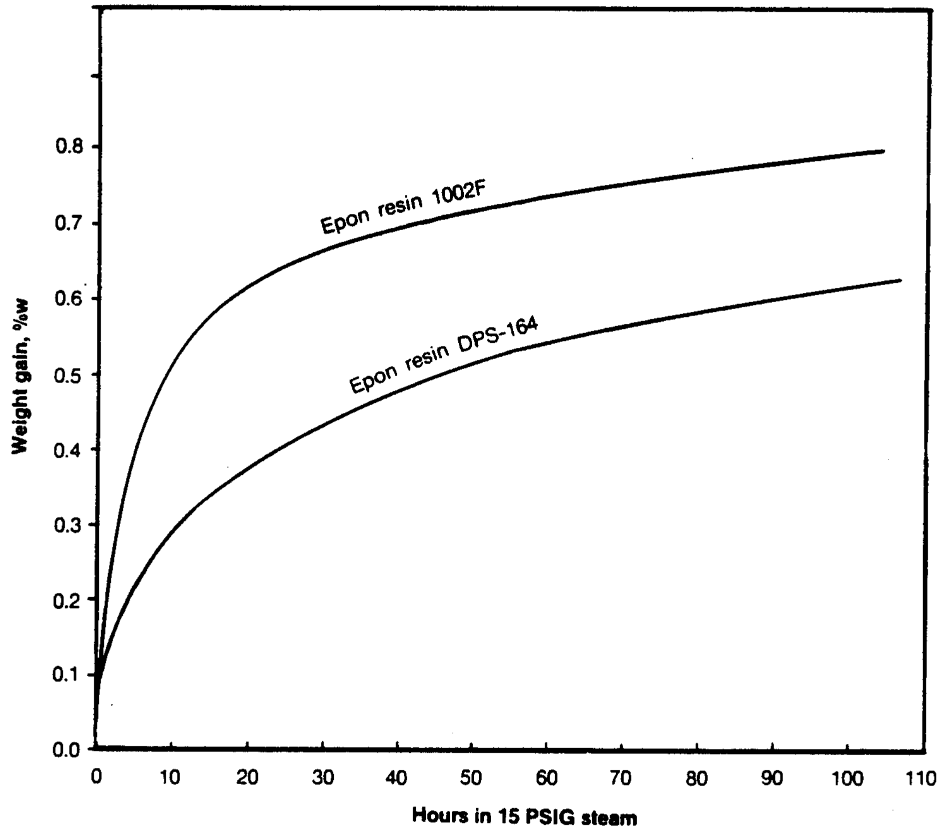


Figure 9/**Increase in solvent resistance with added EPON® Resin DPS-164 in a thin film powder coating**

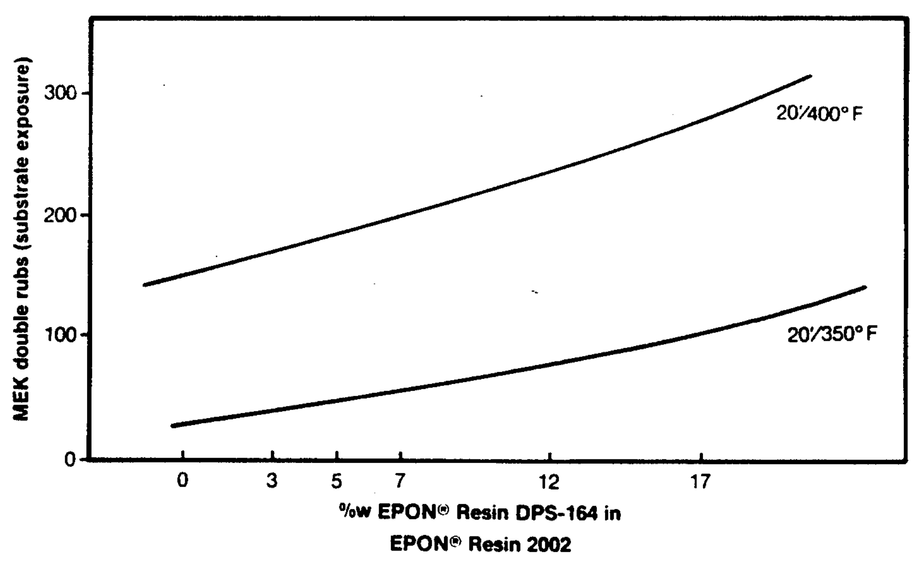


Table 4/**Flexural properties of 8 ply glass cloth laminates**[1]

Composition	
DPS-164	100
DADS	27
BF_3MEA	1

Temperature °C	Flexural strength MPA (psi)	Flexural modulus MPA (psi)	Flexural strain at break, %
23	570 (83,000)	24,000 (3,500,000)	2.4
150	500 (75,000)	22,000 (3,200,000)	2.4
200	340 (49,000)	19,500 (2,800,000)	2.2
225	270 (39,000)	19,000 (2,700,000)	2.2

[1]Dry layup laminates, 35%w resin, cured 8 hours at 175°C.

Identification and Classification

Chemical Abstract Service Registry Number: 29690-82-2

Shell Material Safety Data Sheet Number: 800275-00

Chemical Designation:

- Polyglycidyl ether of ortho cresol novolac

Structural formula base resin: See page 1

Packaging, storage and shipping

- Epon resin DPS-164 is a stable material produced in flake form and packaged in a 200-pound net fiber drum with a bag of desiccant to minimize moisture pickup. This product is slightly prone to sintering or "blocking"; therefore, it should be stored in an area where the temperature does not exceed 80°F and where it is protected against moisture.
- Epon resin DPS-164 is not a hazardous material according to Department of Transportation regulations (Code of Federal Regulations, Title 49).

Handling precautions for systems based on Epon resin DPS-164

The recommendations made in this brochure are based upon Shell's experience and research and are believed to be sound ethical approaches to the applications or end-uses for which they are presented. However, these recommendations are directed solely toward technical performance and should not be taken as recommendations pertaining to health, safety or the environment.

Epon resin DPS-164 and auxiliary materials in these formulations are capable of producing adverse health effects of varying degrees of seriousness ranging from minor skin irritation to serious systemic effects. These can be minimized and most can be avoided through the observance of proper precautions, use of proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these depends upon responsible action by adequately informed persons. None of these materials should be transported, stored, or used until handling precautions and recommendations are understood by all persons who will work with them. Questions and requests for information on Shell products should be directed to your Shell Chemical Company Sales Representative or to the nearest Shell Chemical Sales office. Information on non-Shell products should be obtained from the respective manufacturer or vendor.

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SHELL RESINS

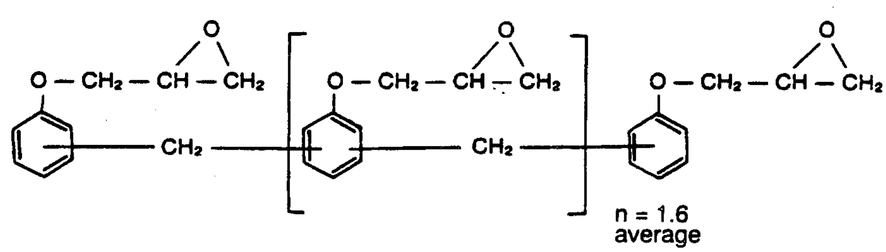
EPON HPT® RESIN 1050
Epoxy Phenolic Novolac Resin

General Description
EPON HPT® Resin 1050 is an epoxy resin manufactured from phenolic novolac resin and epichlorohydrin. The resultant polyfunctional epoxy novolac resin is available in a semi-solid form and as a 75% w solution in acetone (EPON HPT Resin 1050-A-74). When cured with appropriate materials, highly cross-linked compositions exhibiting high chemical resistance, high temperature resistance and dimensional stability are obtained. EPON HPT Resin 1050 may be used as the sole epoxy resin or combined with other epoxy resins such as EPON® Resin 828 (bisphenol A based) or EPON Resin 862 (bisphenol F based) to develop specific application properties.

Features
- Reacts with full range of curing agents.
- Produces high-strength cured systems with high resistance to chemical attack.
- Produces cured systems with elevated temperature stability.
- For FDA regulated food contact use.

End use applications
- Chemical resistant tank linings, flooring and grouts.
- Fiber reinforced pipes, tanks and composites.
- Electrical laminates and encapsulation.
- Tooling, casting and molding compounds.
- Construction, electrical and aerospace adhesives.

Structural formula:



Typical properties

Epoxide equivalent weight[1] (Shell method HC-427-81/ Perchloric Acid Method)	174-180
Viscosity[2] poise @ 52 °C	200-500
Color, Gardner (ASTM D 1544)	2 max.
Weight per gallon, pounds @ 68 °F	10.2
Flash point, (ASTM D3287) Setaflash	Greater than 150 °C

[1]Grams of resin containing one gram-equivalent of epoxide.
[2]Determined with Brookfield LVDT II Spindle #31 Viscometer.

Identification and classification

Chemical Abstract Service Registry Number: 28064-14-4 (EPA/TSCA inventory designation)
Material Safety Data Sheet Number: 1, 32
Chemical designation:

- Formaldehyde, polymer with "Chloromethyl" oxirane and phenol.

Packaging, storage and shipping

- EPON HPT Resin 1050 is a stable semi-solid resin packaged in 55 gallon drums.
- EPON HPT resin 1050 is not a hazardous material according to Department of Transportation regulations (Code of Federal Regulations, Title 49).

FDA status

Several paragraphs in Title 21 of the Code of Federal Regulations permit and regulate the use of epoxy resins such as cured EPON HPT Resin 1050 as indirect food additives in food contact applications.

Curing Agents of EPON resin systems are also regulated under paragraph 175.300 of Title 21 and are subject to the limitations imposed for this section and the general requirements of good manufacturing practices.

For further information on the FDA status of EPON resin products, contact your Shell Chemical Company Sales Representative or Sales Office.

Handling precautions

This product and the auxiliary materials normally combined with it are capable of producing adverse health effects ranging from minor skin irritation to serious systemic effects. Exposure to these materials should be minimized and avoided if feasible through the observance of proper precautions, use of appropriate engineering controls and proper personal protective clothing and equipment, and adherence to proper handling procedures. Each of these preventive measures depends upon responsible action by adequately informed persons. **None of these materials should be used, stored, or transported until the handling precautions and recommendations as stated in the Material Safety Data Sheets (MSDS) for this and all other products being used are understood by all persons who will work with them.** Questions and requests for information on Shell products should be directed to your Shell Chemical Company Sales Office. Information and MSDSs on non-Shell products should be obtained from the respective manufacturer or vendor.

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CIBA-GEIGY

Resins Department

Product Data

ECN 1235, 1273, 1280, 1299

Novolac Epoxy Resins

General

CIBA-GEIGY ECN* resins are derived fron an ortho-cresolformaldehyde novolac which is then reacted with epichlorohydrin to form a polyepoxide. The molecular weight of the novolac determines the molecular weight of the polyepoxy resin whose functionality may range from 2.5 to 5.5.

The major difference between polyepoxides and conventional epoxy resins (derived from bisphenol A) is the availability of more than two reactive groups.

Uses

The materials are recommended for **adhesives** (high temperature), **casting, coatings, electrical** (fluidized bed coatings and molding powder), **laminating** (dry lay-up or vacuum bag laminates), **tooling applications,** and **filament winding.**

These materials are especially recommended in applications where improved thermal properties and high resistance to solvents and chemicals are desired.

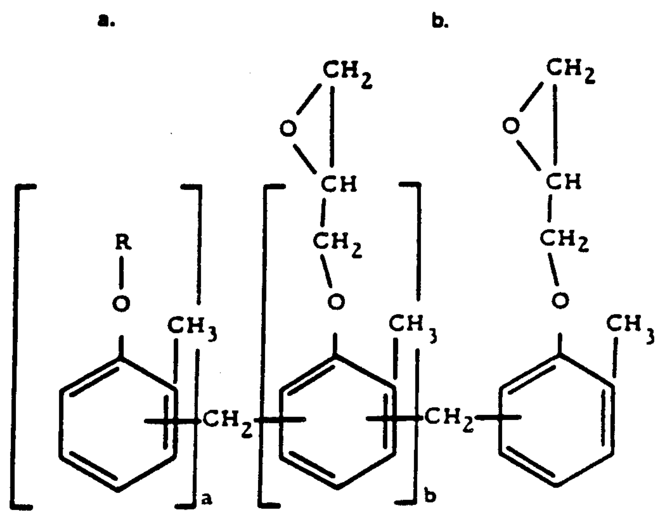
Typical Properties of CIBA-GEIGY ECN Resins

	ECN 1235	ECN 1273	ECN 1280	ECN 1299
Molecular Weight, approx	540	1080	1170	1270
Weight per Epoxide	200	225	230	235
Epoxy Value, Eq /100 gm.	0.500	0.445	0.435	0.425
Melting Point, C.(F)	35(95)	73(164)	80(176)	99(210)
Weight per Gallon, lb.	9.3	9.7	9.8	9.9
Functionality (See Structural Formula)				
a. Ether Type	0.3	1.2	—	1.6
b. Epoxide Type	1.7	3.8	4.1	4.4
Melting Point, °C. (°F.)	35(95)	73(164)	80(176)	99(210)
Weight per Gallon, lb.	9.3	9.7	9.8	9.9

*ECN Epoxy Cresol Novolac

Typical Properties of CIBA-GEIGY ECN Resins (Continued)

Structural Formula:



Note: R represents chlorohydrins, glycols, and/or polymeric ethers.

Handling and Mixing of the Resins

All hardeners used to cure conventional epoxy resins can be used to cure the ECN resins. Generally, CIBA-GEIGY ECN resins exhibit a shorter gel time and cure faster than conventional epoxy resins under the same conditions.

It may be advantageous for particular applications to prepare solutions of these resins. This is easily accomplished since the resins are soluble in many of the more common solvents. For example:

Acetone	Toluene
Cellosolve	Toluene-Cellosolve
Cellosolve, Acetate	Toluene Cellosolve Acetate
Chloroform	Toluene-MIBK
Ethylene Chloride	Xylene
MIBK	

Handling and Mixing of the Resins (Continued)

It is important to consider the different curing characteristics, that is, the rate of cure increase with epoxide functionality. This is illustrated in Table I comparing gel times:

TABLE I

Formulation		Gel Time, Minutes, at 140°C. (284°F.) ECN 1235	ECN 1273	ECN 1299
Hardener 906	89.0 phr*	45	—	—
Accelerator 064	2.0 phr			
Hardener 906	79.5 phr	—	45	—
Accelerator 066	0.5 phr			
Hardener 906	76.0 phr	—	—	50

Table II is designed to act as a guide for the user. The table exhibits the wide variations in reactivities achieved using CIBA-GEIGY ECN 1235 and different hardeners. By proper choice of hardener and cure temperatures, a gel time of one minute to a shelf life of several months can be obtained.

TABLE II

Typical Gel Times Using ECN 1235

Hardener	Phr	Gel Time, Minutes, at 90°C. (194°F.)
Hardener 906✓	89.0	49
Hardener 964✓	133.0	32
Hardener 907#	77.0	65
HET Anhydride	110.0	26
Hardener 972	24.8	32
BF mea	2.0	780
Piperidine	6.0	45
Hardener 950	10.2	2

*Parts by weight per hundred parts of resin
**40 gram mass
✓With Accelerator 064 (DMP-30) 2.0 phr
#With Accelerator 066 (DMP-10) 0.5 phr

Typical Properties of Cured Unreinforced Systems

Physical Properties

ASTM Deflection Temperature, °C. (°F.)

a. Hardener:	Hardener 906
b. Specimen size:	½" x 5" x ⅛"
c. Cure:	1 hr. at 120°C. + 1 hr. at 150°C. + 1 hr. at 180°C. + 12 hr. at 215°C. + 24 hr. at 240°C.

Deflection Temperature

CIBA-GEIGY ECN 1235*	285°C. (545°F.)
CIBA-GEIGY ECN 1275✓	> 300°C. (572°F.)
CIBA-GEIGY ECN 1280	> 300°C. (572°F.)
CIBA-GEIGY ECN 1299	> 300°C. (572°F.)

*With Accelerator 064 (DMP-30), 2.0 phr
✓With Accelerator 066 (DMP-10), 0.5 phr.

Typical Electrical Properties using CIBA-GEIGY ECN 1235

Electrical Properties at 25°C. (77 F.)

Formulation	A		B		C		D	
	Hardener 964	133 phr*	Hardener 907	77 phr	Hardener 906	89 phr	BF_3.mea	5 phr
	Accelerator 064 (DMP-30)	1.5 phr	Accelerator 066 (DMP-10)	0.5 phr	Accelerator α MBDMA†	2 phr		
Cure:	2 hrs. at 93°C. (200 F) - 12 hrs. at 150 C. (302 F)		1 hr at 100°C. (212°F.) -- 4 hrs. at 121 C. (250°F.)		1 hr. at 120°C. (248°F.) — 1 hr. at 150°C. (302°F.) — 12 hrs. at 180°C. (356°F.) — 16 hrs. at 215°C. (419°F.)		¾ hr. at 93°C. (200°F.) — 2 hrs. at 174°C. (345°F.)	

	Condition		Condition		Condition		Condition	
	A**	D***	A	D	A	D	A	D
Dielectric Constant								
60 Hz	2.98	3.03	3.18	3.21	3.42	3.48	3.58	3.62
10^3 Hz	2.95	3.00	3.14	3.18	3.40	3.45	3.54	3.59
10^6 Hz	2.88	2.93	3.03	3.06	3.31	3.36	3.44	3.47
2.5×10^9 Hz	2.83	2.85	2.98	3.00	3.20	3.25	3.34	3.38
Dissipation Factor								
60 Hz	0.0058	0.0051	0.0056	0.0067	0.0041	0.0045	0.0059	0.0063
10^3 Hz	0.0072	0.0071	0.0103	0.0102	0.0056	0.0055	0.0065	0.0069
10^6 Hz	0.0100	0.0105	0.0119	0.0118	0.0142	0.0015	0.0133	0.0148
2.5×10^9 Hz	0.0054	0.0059	0.0077	0.0084	0.0102	0.0113	0.0100	0.0109
Volume Resistivity, 25°C. (77°F.) ohm-cm.	1.4×10^{11}	4.7×10^{16}	2.4×10^{11}	9.5×10^{16}	1.0×10^{16}	—	2.2×10^{11}	1.5×10^{16}

Typical Properties of Cured Reinforced Systems

Formulation	A		B		C		D	
	ECN 1235	100 pbw	ECN 1273	100 pbw	ECN 1299	100 pbw	ECN 1299	100 pbw
	Hardener 906	75.5 pbw	Hardener 906	67.1 pbw	Hardener 906	64.3 pbw	Hardener 905	64.3 pbw
	α MBDMA	2.0 pbw	α MBDMA	2.0 pbw	α MBDMA	1.0 pbw		
Prepreg Characteristics								
Style«-finish					Volan A			
Oven Dwell, min./°F. (°C.)	9/212 (100)		4/212 (100)		3/212 (100)		3.5/212 (100)	
Laminate Construction								
No. of plies					12			
Cure, min./°F./psi.	20/383 (195°C.)/ contact with stops		20/401 (205°C.)/ contact with stops		20/392/95 (206°C.)		20/392/contact with stops	
Postcure, hr./°F. (°C.)	24/419 (215°C.)		24/419 (215°C.)		24/419 (215°C.)		24/419 (215°C.)	
Nominal Thickness, ±0.005 in. (±0.13 mm)	0.103 (2.62 mm)		0.104 (2.64 mm)		0.093 (2.36 mm)		0.102 (2.59 mm)	
Resin Content, %	27		26		24		24	

*parts by weight per hundred parts of resin
†α Methylbenzyldimethylamine

**As is
***After water immersion, 24 hrs. at 25°C. (77°F.)
«J. P. Stevens 181 STYLE or equivalent

/Continued

Properties of Cured Laminates	psi (Kg/cm^2)	psi (Kg/cm^2)	psi (Kg/cm^2)	psi (Kg/cm^2)
Flexural Properties (tested at 25°C.(77°F.)				
Ultimate Flexural Strength	71,800 (5,050)	70,300 (4,900)	67,800 (4,600)	56,400 (3,960)
Elastic Modulus	3.09 x 10^6 (2.17 x 10^5)	2.79 x 10^6 (1.96x10^5)	3.28 x 10^6 (2.32 x 10^5)	2.80 x 10^6 (1.97 x 10^5)
Flexural Properties (tested at 260°C. (500°F.)				
Ultimate Flexural Strength	13,000 (914)	15,800 (1,110)	20,400 (1,430)	22,900 (1,610)
Elastic Modulus	0.96 x 10^6 (6.75 x 10^4)	1.02 x 10^6 (7.17 x 10^4)	1.45 x 10^6 (1.02 x 10^5)	1.12 x 10^6 (7.86 x 10^4)
Flexural Properties (tested at 260°C. (500°F.) after aging 8 days at 260°C.)				
Ultimate Flexural Strength	30,100 (2,110)	16,200 (1,140)	21,700 (1,520)	10,200 (717)
Elastic Modulus	1.89 x 10^6 (1.33 x 10^5)	1.98 x 10^6 (1.39 x 10^5)	2.33 x 10^6 (1.64 x 10^5)	1.17 x 10^6 (8.22 x 10^4)
Compressive Properties				
Ultimate Compressive Strength				
Tested at 25°C (77°F)	70,800 (4,980)	66,500 (4,680)	88,100 (6,200)	56,600 (3,980)
Tested at 260°C (500°F)	22,600 (1,590)	35,800 (2,380)	56,600 (3,980)	42,500 (2,990)
Tested at 260°C (after aging 8 days at 260°C)	39,600 (2,780)	34,900 (2,450)	39,600 (2,780)	34,000 (2,390)
Tensile Properties [Tested at 25°C.(77°F.)]				
Ultimate Tensile Strength	52,100 (3,670)	50,900 (3,580)	—	49,000 (3,450)
Elastic Modulus	3.33 x 10^6 (2.34 x 10^5)	3.22 x 10^6 (2.26 x 10^5)	—	3.32 x 10^6 (2.33 x 10^5)

Handling/Safety Precautions

Caution! May cause irritation and skin sensitization. Do not handle or use until the Safety Data Sheet has been read and understood.
Use only with adequate ventilation.
Do not take internally or taste.
Avoid contact with eyes, skin and clothing.

First Aid

In case of contact:
Eyes: Immediately flush eyes with water for at least 15 minutes. Get medical attention.
Skin: Wash thoroughly after handling, and before eating, drinking or smoking.
Clothing: Wash contaminated clothing before reuse.
Ingestion: If conscious, give water and induce vomiting. Call a physician.
Inhalation: Remove to fresh air. If breathing is difficult, give oxygen and call a physician.

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