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APPENDICES

APPENDIX A

Determination of Copolymer Compositions of CHN/O Method

Example of calculation

Assume AM : KA = a : b mole %

AM(C_3H_5ON) = a(C_3H_5ON)

KA($C_3H_4O_2$) = b($C_3H_4O_2$)

From CHN/O method C : H : N = 38.191 : 5.750 : 9.242

Carbon $C = [3a+3b]x12 = 38.191$ (1.1)

Nitrogen $N = [a]x14 = 9.242$ (1.2)

From eq. (1.2) $\therefore a = 9.242/14 = 0.660$

From eq. (1.1) $36a + 36b = 38.191$

Instead of a value with 0.660

$36(0.660) + 36b = 38.191$

$23.765 + 36b = 38.191$

$b = (38.191-23.765)/36$

$b = 0.401$ (1.3)

$a + b = 0.660 + 0.401 = 1.061$

$\therefore a : b = 62.21 : 37.79$ %

$\therefore$ Molar ratio of AM : KA = 62.21 : 37.79 %

Table A-1

Determination of Copolymer Compositions of CHN/O Method

Sample No.	Molar ratio of AM : KA in feed	Molar ratio of AM : KA in copolymer	% Carbon	% Hydrogen	% Nitrogen
1	10 : 90	12.10 : 87.89	29.861	4.445	1.405
2	20 : 80	22.43 : 77.57	31.403	4.782	2.932
3	30 : 70	36.75 : 63.25	33.612	4.910	4.803
4	40 : 60	47.15 : 52.85	35.136	4.976	6.443
5	50 : 50	56.88 : 43.12	36.943	5.437	8.172
6	60 : 40	62.21 : 37.79	38.191	5.750	9.242
7	70 : 30	74.43 : 25.56	40.561	6.069	11.741
8	80 : 20	80.95 : 19.05	42.390	6.367	13.343
9	90 : 10	89.24 : 10.76	44.403	6.901	15.410

This data is analyzed by CHNS/O analyser(Perkin Elmer PE2400 Series II).

APPENDIX B

Viscosimetric Methods [83]

Deformation of a polymer sample occurs when an applied force change the sample's shape and size. These deformations can be

(a) Reversible (elastic). When a force is applied to a perfectly elastic polymer sample, a finite deformation occurs. After removal of the force, the elastic sample returns to its original size and shape. The energy applied for the reversible deformation is completely recovered.

(b) Irreversible. When a force is applied to a perfectly viscous sample, the deformation changes with time and the sample flows. The energy applied for the irreversible deformation is not recovered.

Many polymers exhibit an elastic(reversible) deformation for short periods of time and an irreversible deformation if the force is applied for a longer period of time.

The shear stress(τ) in a reversible deformation is given by

$$\tau = F/A \qquad \text{(Appendix B-1)}$$

in dynes per square centimetre(CGS) or newtons per square metre(SI), where

F is the applied force(Figure 1)(in dyne(CGS) or newton(SI),

A is the area(in square centimetres(CGS) or square metre(SI).

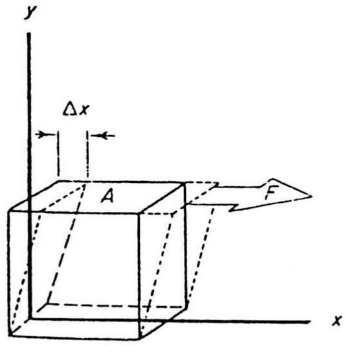


Figure 1 Reversible deformation. A is the area and F is the applied force.

The shear strain(γ) is the amount of deformation given by

$$\gamma = \Delta x / \Delta y \qquad \text{(Appendix B-2)}$$

For an ideal elastic material, the shear strain is directly proportional to the shear stress.

Hooke's law for reversible deformation gives the relation between shear stress(τ) and shear strain(γ):

$$\tau = F/A \qquad = G\gamma \qquad \text{(Appendix B-3)}$$

where G is the shear modulus.

A fluid polymer sample constrained between two plates can be considered as a series of parallel layers(Figure 2). The upper plate moves with a velocity(V)relative to the lower plate under a shearing force(F) per area(A). Particular parallel layers of liquid move with velocities which are proportional to distance of the fluid layer from the lower plate (laminar flow).

Foe ideal viscous fluids, the shear stress is directly proportional to the rate of change of velocity with distance(dV/dy).

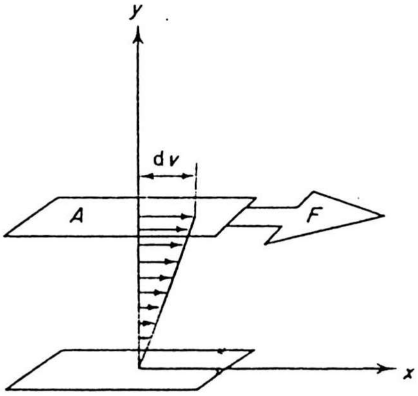


Figure 2 Irreversible deformation. A is the area and F is the applied force.

Hooke's law for irreversible deformation gives the relation between shear stress(τ) and shear strain(γ):

$$\tau = F/A = \eta(dV/dy) \qquad \text{(Appendix B-4)}$$

since

$$(dV/dy) = d(dx/dt)/dy = d(dx/dy)/dt \qquad \text{(Appendix B-5)}$$

eq. Appendix B-4 can be rewritten as

$$\tau = \eta\dot{\gamma} \qquad \text{(Appendix B-6)}$$

where η is the viscosity and $\dot{\gamma}$ is called the shear rate (velocity gradient)(in s^{-1}).

The viscosity (η) is defined for a given shear stress(τ) and velocity gradient(d(dx/dy)/dt) by the equation

$$\eta = \tau / \gamma \qquad \text{(Appendix B-7)}$$

in dyn.s.cm^{-2}(CGS) or N s m^{-2}(SI). The unit (dyn.s.cm^{-2}) is called the POISE. CENTIPOISE is equal to 10^{-2} poise.

The viscosity(η) is independent of the velocity gradient, the fluid is called Newtoniun fluid.

The viscosity(η) is dependent of the velocity gradient, the fluid is called Non-Newtoniun fluid.

Example : *At speed 5.0 rpm, viscosity = 0.25 Pa.s, Spindle No. 14, time for measurement 10 min.*

Factor for shear rate calculation = 0.4 N , N = Spindle speed

$\therefore$ shear rate, $\dot{\gamma}$ = 0.4 x 5 = 2.0 s^{-1}.

From eq.(Appendix B-5) shear strain, $\gamma = \dot{\gamma} \times t$

$\gamma = 2.0\ \text{s}^{-1} \times 10\ \text{min.} \times 60\ \text{s}$

$\therefore \gamma = 1200$

From eq.(Appendix B-6) $\tau = \eta \dot{\gamma}$

$= 0.25\ \text{Pa.s} \times 2\ \text{s}^{-1}$

$= 0.5\ \text{Pa}$

From eq.(Appendix B-3) G $= \tau/\gamma$

G $= 0.5\ \text{Pa} / 1200$

$\therefore$ G $= 4.166 \times 10^{-4}\ \text{Pa}$

shear modulus $= 4.166 \times 10^{-4}\text{Pa}$

APPENDIX C

Determination of Cross-link Density

The effective cross-link density, ρ_x is defined as the concentration of elastically active chains (chains which are deformed by an applied stress) in the polymer network, and is usually reported on the basis of moles of chains per cubic centimeter of dry polymer. Network structure can also be described with a number of closely related terms. For example, when linear polymers are crosslinked, it is often desirable to express cross-linking in terms of the number average molecular weight of the polymer before cross-linking, $\overline{M}_n$, and the number average molecular weight between cross-links, $\overline{M}_c$. These quantities can be related to the cross-link density as follows:

$$\rho_x = [1/\overline{V}\overline{M}_c][1-2\overline{M}_c/\overline{M}_n] \qquad \text{(Appendix C-1)}$$

where $\overline{V}$ = polymer specific volume. The factor $(1-2\overline{M}_c/\overline{M}_n)$ is a correction for network imperfections such as dangling ends. In the case of an ideal network, this factor reduces to one. This correction is difficult to apply to networks formed by copolymerization/ cross-linking reactions since an appropriate value of $\overline{M}_n$ may be difficult to determine. In many cases, however, $\overline{M}_n$ is much greater than $\overline{M}_c$, and equation can be simplified to the following:

$$\rho_x = 1/\overline{V}\overline{M}_c = 1/V_m Z \qquad \text{(Appendix C-2)}$$

where Z = degree of polymerization between cross-links.

Determination the cross-link density by elasticity measurements [68]

When a network polymer is subjected to an external force, it undergoes elastic deformation. The behavior of crosslinked polymers under strain has been the subject of considerable work. The classic reference is by Treloar; Peppas and Barr-howell have reviewed the specific problems related to hydrogel.

The following simple equation characterizes the stress-strain response of ideal networks under uniaxial compression or extension:

$$\tau = F/A_0 = G\,(\lambda - \lambda^{-2}) \qquad \text{(Appendix C-3)}$$

where τ = engineering stress; F = applied force; A_0 = undeformed cross-sectional area of swollen polymer; G = shear modulus; $\lambda = L/L_0$; L = sample length under strain; and L_0 = undeformed sample length. At low strains, a plot of stress versus $(\lambda-\lambda^{-2})$ will yield a straight line whose slope is the modulus. The effective cross-link density may then be calculated from the modulus as follows:

$$\rho'_x = G/(v_2^{1/3}\,RT) \qquad \text{(Appendix C-4)}$$

The cross-link density obtained in this manner is referenced to the swollen polymer volume at the time of compression(ρ'_x). For comparison to theory or to similar polymers swollen to different extents, it is necessary to define the cross-link density in terms moles of chains per unit volume of dry polymer(ρ_x). This conversion is simply:

$$\rho_x = (\rho'_x)/\nu_2 \qquad \text{(Appendix C-5)}$$

Efficiency of the cross-linking reaction can be defined ratio of effective cross-link density, ρ_x to the theoretical cross-linking density, ρ_t. Theoretical cross-link density is not easily defined except for chemical methods, however.. If all of the reagent used forms elastically effective cross-links, then the theoretical cross-linking density, ρ_t is given as:

$$\rho_t = Cf/2 \qquad \text{(Appendix C-6)}$$

where C = cross-linker concentration in the bulk polymer, and f = cross-linker functionality. Cross-linker functionality is defined as the number of network chains connected to the junction. For a bifunctional vinyl monomer such as N,N′ -methylene bisacrylamide, commonly used for copolymerization/cross-linking synthesis of network, f = 4. Theoretical cross-linking density then reduces to 2C.

Equipment for determination of gel moduli are usually based on either compression or extension of the polymer samples. Figure B-1 illustrates the device used to determine cross-link density from equilibrium stress measured after the application of constant strain. Swollen polymer samples are placed in a glass dish beneath a teflon plate attached to a fore transducer and micrometer. The dish contains sufficient solution to prevent drying of the swollen polymer. A constant strain is applied by adjusting the micrometer, which lowers the upper plate and compresses the gel. The force is measured by the transducer and monitored by the computer; when it relaxes to a constant value, the plate is lowered further. After collecting stress-strain data, the modulus and the effective cross-link density are calculated using the equation(Appendix C-3-5).

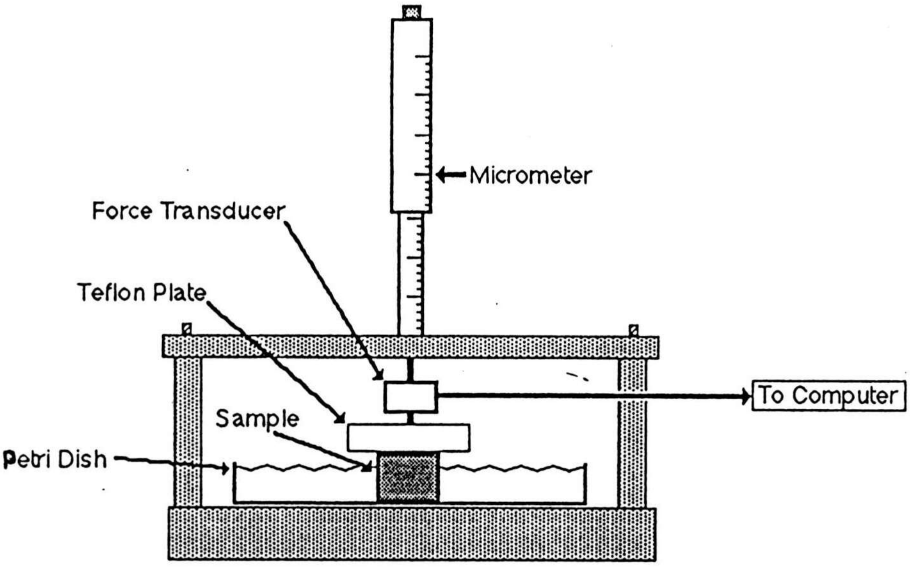
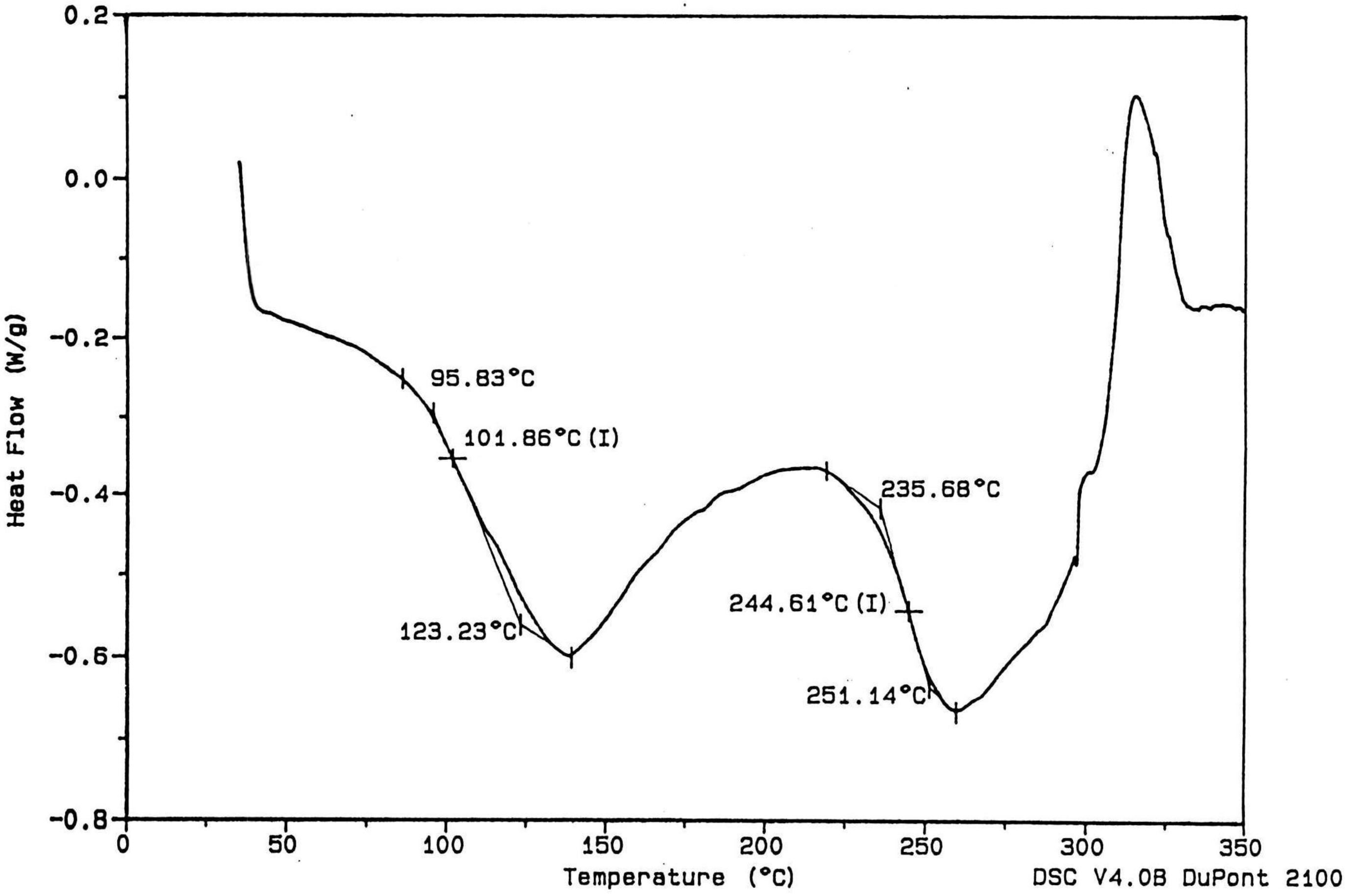


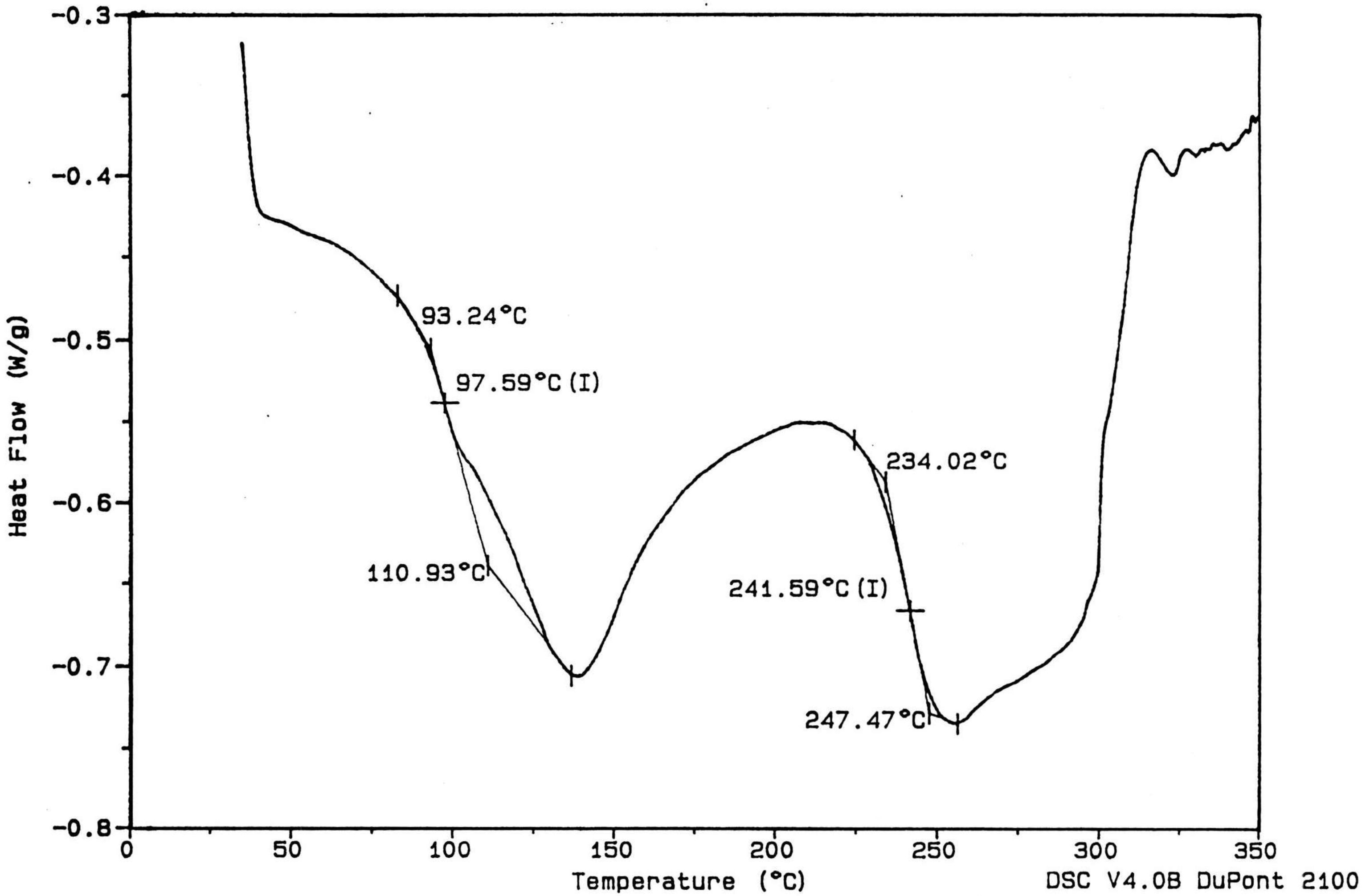
Figure 3 Apparatus for determining ρ_x by uniaxial compression of a gel sample.

APPENDIX D

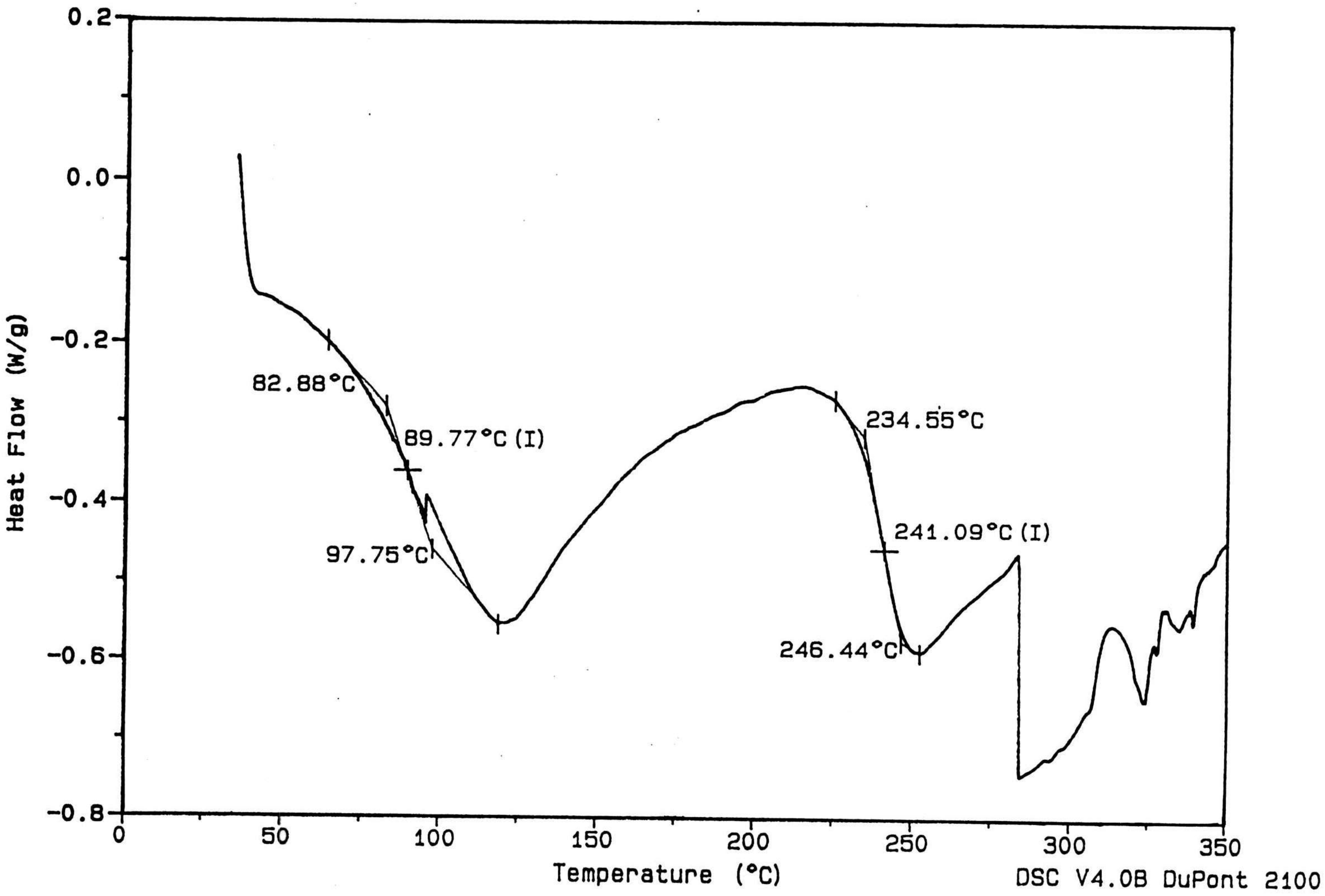
DSC Thermograms of the copolymers



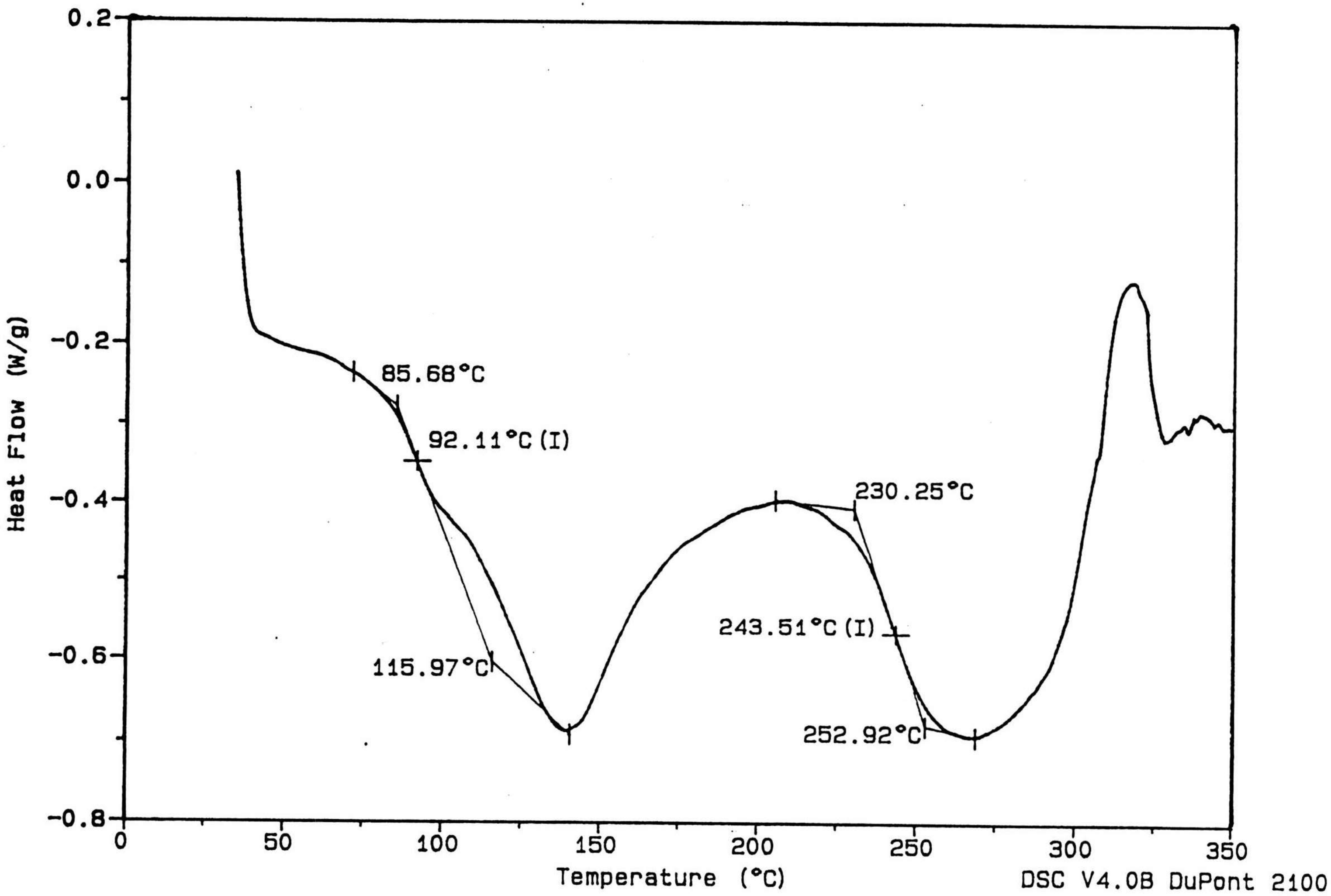
Appendix D-1 DSC Thermogram of the copolymer, synthesized with Span 80, and 0.025% N,N′-MBA



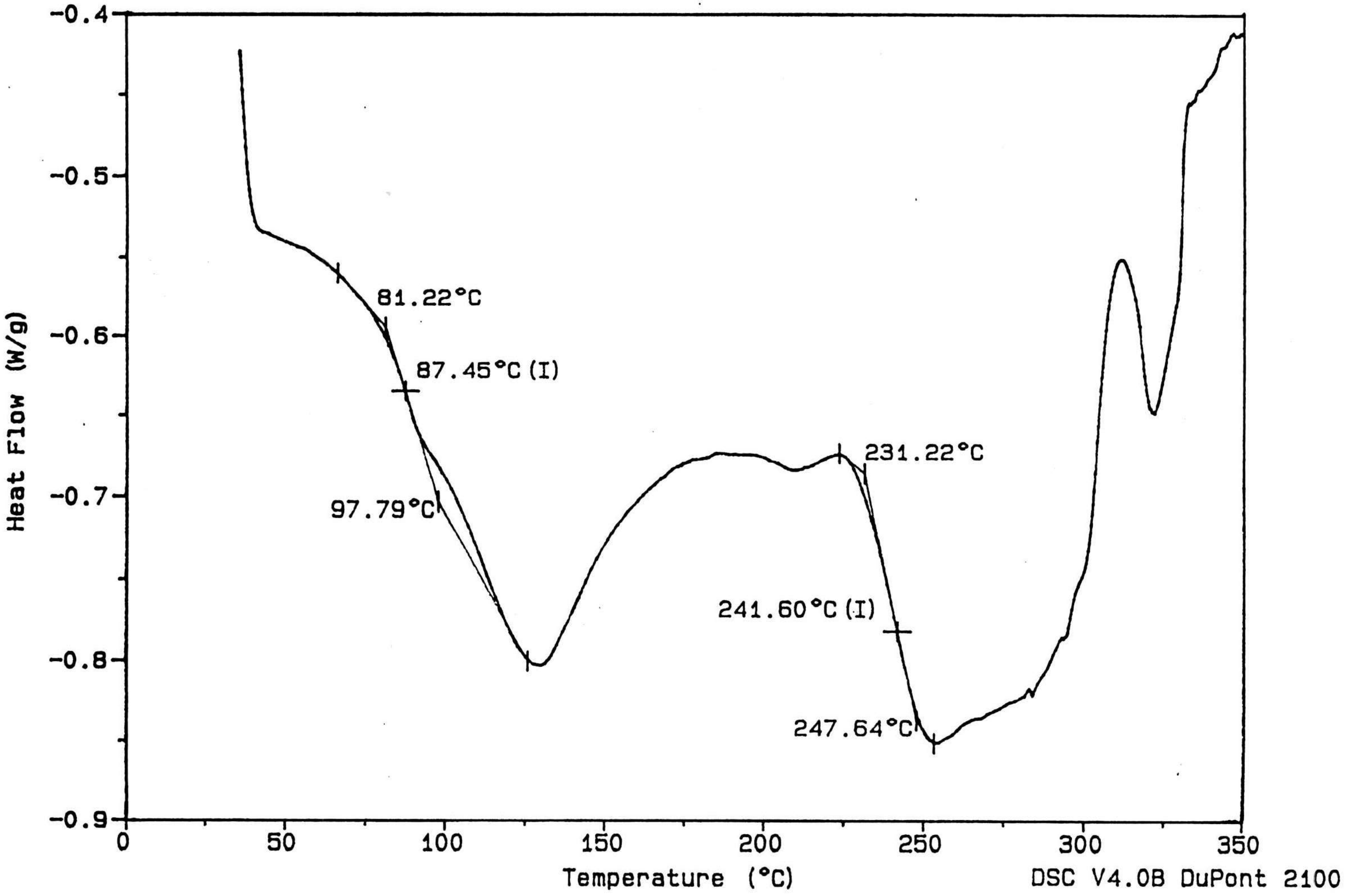
Appendix D-2 DSC Thermogram of the copolymer, synthesized with Span 60, and 0.025% N,N′-MBA



Appendix D-3 DSC Thermogram of the copolymer, synthesized with Span 40, and 0.025% N,N′-MBA



Appendix D-4 DSC Thermogram of the copolymer, synthesized with Span 80, and non cross-linking agent



Appendix D-5 DSC Thermogram of the copolymer, synthesized with Span 80, and 0.075% N,N′-MBA

VITA

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