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The Characterization of Asphalt and Asphalt Recyclability

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INTRODUCTION

The goal of this research program was to construct a simple model and computer programs that will allow at least a qualitative understand of the phase behavior of asphalt (i.e., how asphalt components mix with one another), mixtures of different types of asphalt (i.e., in recycling) and mixtures of asphalt with other materials, such as synthetic polymers.

We have constructed such a model and computer programs (for Macintosh computers) that allow such calculations to be performed easily. The theoretical background and the details of how these calculations should be performed are given in the following section. Because these programs also allow the calculations of the number of hydrogen bonded species, we were also able to perform some calculations that we believe are of considerable use in assessing some of the models that have recently been proposed for asphalt structure. The results of some sample calculations will also be presented in this report.

THE PHASE BEHAVIOR OF ASPHALT MIXTURES

Theoretical Background

In dealing with mixtures of macromolecular species such as those found in asphalt the usual starting point is the Flory-Huggins equation for the free energy. This assumes “weak” interactions only, however, by which we mean London dispersion forces and weak polar forces whose “strength” is of the order of RT or less. Strong interactions, such as hydrogen bonds, introduce a number of difficulties, but in recent work in this laboratory we have developed a lattice model that can also account for these interactions¹. We will not reproduce the fundamental equations in their complete form here, but for clarity of presentation consider a simplified expression for the free energy of mixing;

$$\frac{\Delta G_M}{RT} = \frac{\phi_A}{x_A} \ln \phi_A + \frac{\phi_B}{x_B} \ln \phi_B + \phi_A \phi_B \chi + \frac{\Delta G_H}{RT} \quad (1)$$

where ϕ_A and ϕ_B are the volume fractions of components A and B, χ is the usual Flory-Huggins interaction parameter, limited here to a description of “physical” (non-hydrogen bonding) forces, while $\Delta G_H/RT$ is the contribution to the free energy of mixing from the change in the number and type of hydrogen bonds that occurs as a function of composition. The quantities x_A and x_B are the “degrees of polymerization” of the components A and B, actually equal to the molar volume of these molecules divided by a reference molar volume. If component A is a solvent and B a “polymeric” or macromolecular species, such as those found in asphalt, it is usual to use the molar volume of the solvent (V_A) to define the lattice cell size, so that x_A would be equal to 1. In using association models to describe hydrogen bonding (i.e., the ΔG_H term) it is more convenient (i.e., simplifies the algebra) to use the “repeat” unit of the macromolecules to define the reference volume (V_B) and we therefore use

$$\frac{\Delta G_M}{RT} = \frac{\phi_A}{r} \ln \phi_A + \frac{\phi_B}{x_B} \ln \phi_B + \frac{\phi_A \phi_B \chi}{r} + \frac{\Delta G_H}{RT} \quad (2)$$

where $r = V_A/V_B$. This opens up the question of what is a “repeat” unit for asphalt, or if indeed such a thing can be defined.

Asphalt is, of course, extraordinarily heterogeneous, and in the ordinary sense a polymer repeat unit cannot be defined. That is not what we are doing here, however. As long as the hydrogen bonding functional groups of asphalt are randomly distributed, then all we require is the definition of an average unit or segment per group. This is not for the purpose of defining asphalt to be a polymer of such units, but only a device for computational convenience, as we will show.

We require the quantity V_B for three purposes, first to calculate the combinatorial entropy of mixing (first two terms in equation 2), second, to calculate solubility parameters

and hence χ , third to “scale” the values of the equilibrium constants used to describe hydrogen bonding interactions (ΔG_H term).

In principle we can use any arbitrarily defined reference volume V_B , but it is particularly convenient in calculating ΔG_H to use a value based on the molar volume of a hypothetical repeat unit containing just one oxygen atom, since it is these atoms that in asphalt constitute the hydrogen bonding functional groups. To repeat and emphasize, we are not artificially trying to say that asphalt is a polymer of such units, this definition is simply a way of placing the calculation of all the terms in equation 2 on a common scale.

We will commence our discussion of our calculation methodology by considering group contributions to molar volumes and solubility parameter and then move on to consider the effect of hydrogen bonding.

The calculation of solubility parameters and molar volumes using group contributions.

First, in terms of using the molar volume of an “average repeat unit”, Scott² demonstrated more than 30 years ago that the “physical” interactions of a copolymer of any degree of heterogeneity could be described by a solubility parameter that is a volume fraction average of contributions of its constituents. Accordingly, it would appear to be a trivial task to calculate the solubility parameter of a macromolecule. One only has to consider the groups present in an average repeat unit (e.g., $-\text{CH}_2-$, $-\text{O}-$, $-\text{CH}_3$), refer to tables of molar attraction constant (F) determined by various authors, and use the relationship

$$\delta = \Sigma F_i / V \quad (3)$$

There are two immediate problems in applying this to asphalt: first, we do not know the precise distribution of functional groups; second, there are large differences in the values of the attraction constants reported by different authors and one often does not know the extent of errors involved with the choice of a particular set of parameters. We will first examine this latter problem as it is central to our work.

The group contribution method, originally developed by Small,³ was considerably expanded by Hoy⁴ and van Krevelen.⁵ In previous work where we applied the values tabulated by these last two authors to synthetic polymers we encountered a number of problems and found various inconsistencies, often associated with the use of an insufficient number of compounds to adequately define parameters for particular groups. We therefore decided to calculate our own group contributions using a data set based on the properties of 255 non-hydrogen-bonding organic liquids.⁶ In performing these calculations we had the considerable advantage of access to powerful desktop computers, unavailable when the above quoted work was performed. This allows the calculation of group contributions by matrix methods in a much more interactive matter and lays bare the consequences of unwarranted assumptions and the effects of errors. For a full discussion of these points the reader is referred to our original paper,⁶ but we note in passing that our results are in good agreements with the seminal work of Small,³ who clearly understood the limitations of the method. Of concern to our studies of asphalt are errors, which we will consider shortly, and the recognition of a fundamental assumption. In calculating group contributions the molar attractions constants are determined from the experimentally determined solubility parameters of the model liquids multiplied by their respective molar volumes (i.e., $\Sigma F_i = \delta V$). Accordingly, the subsequent calculation of the solubility parameter for an unknown materials *must also use group contributions to the molar volumes based upon a correlation using the same experimental data set used to determine the factors F_i* . It has been common practice to use molar attraction constants from Hoy or van Krevelen with some arbitrary experimental or calculated molar volume. This is specious. It is necessary to have both molar volume (V^*) and molar attraction (F^*) values from the *same* set of model compounds in order to be consistent in calculating the solubility parameter of a macromolecule.

Given that we have previously determined group contributions to molar attraction constants and molar volumes, their application to asphalt would be trivial, *if we knew the*

distribution of functional groups (i.e., relative proportions of CH₂, CH₃, O, aromatic species, etc.). Unfortunately, this information is not yet available in anything like a complete manner for any asphalt. We therefore adapted a procedure first described by van Krevelen and determined atomic contributions, which only requires a knowledge of the elemental composition and the aromaticity of the material, which is known (or can be easily measured) for many asphalts. The solubility parameter is then calculated on a “per mole of carbon atoms basis” using

$$\delta = \frac{\Sigma(N_i/C)F_i}{\text{molar volume per carbon atom}} \quad (4)$$

where N_i/C denoted the atomic ratios H/C, O/C, etc., and the molar volume per carbon atom was calculated from the molecular weight per carbon atom and atomic contributions.

Using the matrix methods and data set previously employed to calculate group contributions we obtained the results shown in Table I, which includes both the molar attraction constants and molar volume contributions that must be used in concert in order to limit errors. The values listed do not necessarily have intrinsic meaning when considered on their own; for example, carbon atoms are listed as having a negative contribution to molar volume! They are merely *parameters* obtained from a best fit of the experimental data, in the least-squares sense, that when taken together give the best correlation of observed and calculated values.

The values can now be applied to the calculation of solubility parameters by expressing the molar attraction constants and molar volumes on a “per carbon atom” basis:

$$\delta_c = \frac{7.0 + 63.5f_a + 63.5\frac{H}{C} + 106\frac{O}{C} + 51.8\frac{N + S}{C}}{-10.9 + 12f_a + 13.9\frac{H}{C} + 5.5\frac{O}{C} - 2.8\frac{N + S}{C}} \quad (5)$$

The symbol f_a is the fraction of carbon atoms that are aromatic and for application to asphalt. We have arbitrarily assumed that all oxygens behave as ether oxygens and

Table 1. Atomic Group Contributions

group	V^* , cm ³ mol ⁻¹	F^* (cal-cm ³) ^{0.5} mol ⁻¹
carbon	-10.9	7.0
hydrogen	13.9	63.5
ester oxygen	7.5	23.8
ketonic oxygen	23.2	281
ether oxygen	5.5	106
aromatic C=C	23.9	127
nonaromatic C=C	23.4	116
chlorine	23.7	255
nitrile nitrogen	34.4	417
primary nitrogen	0.1	187
secondary nitrogen	-3.8	102
tertiary nitrogen	-2.8	51.8

included the contribution of sulfur atoms (generally small) with nitrogen. The “constitutional” effects, leading to the terms in f_a in eq. 5, were determined as contributions per “pair” of aromatic C atoms. Their contributions per carbon atom are thus half the corresponding values listed in Table I.

When reapplied to the *original data set*, the parameters listed in Table I results in errors of ± 0.6 (cal \cdot cm $^{-3}$) $^{0.5}$ (at the 95% confidence level) in the determination of solubility parameters (compared to errors of ± 0.4 when functional group contributions are used¹). The calculated and observed values are compared in Figure 1. Furthermore, one might anticipate that the deviation between calculated and observed values would be even greater than ± 0.6 when the atomic volume and attraction constants are applied to molecules *not* in this data set (i.e., asphalt).

As part of this project have modified our interactive computer program that allows the calculations of solubility parameters from group contributions so as to also include the ability to calculate parameters from atomic contributions.

Calculation of the molar volume of an “average repeat unit.”

Although there are significant errors in calculating solubility parameters from atomic contributions, they should nevertheless be useful in considering broad trends and the balance of forces that determine phase behavior. In order to calculate χ from solubility parameters we need to define a reference volume for the asphalt that can correspond to *any arbitrarily defined segment*, because we will simply calculate the free energy change *per segment* upon mixing. We can do this for asphalt in two different ways, depending upon whether or not we have molecular weight data available.

For samples where we do not have such data we define a molar volume per oxygen group as V_B , using

$$V_B = \frac{V_m}{O} = \frac{1600}{O'd} \quad (6)$$

where O' is the weight per cent of oxygen in the sample and d is the density.

CALCULATED SOLUBILITY PARAMETER

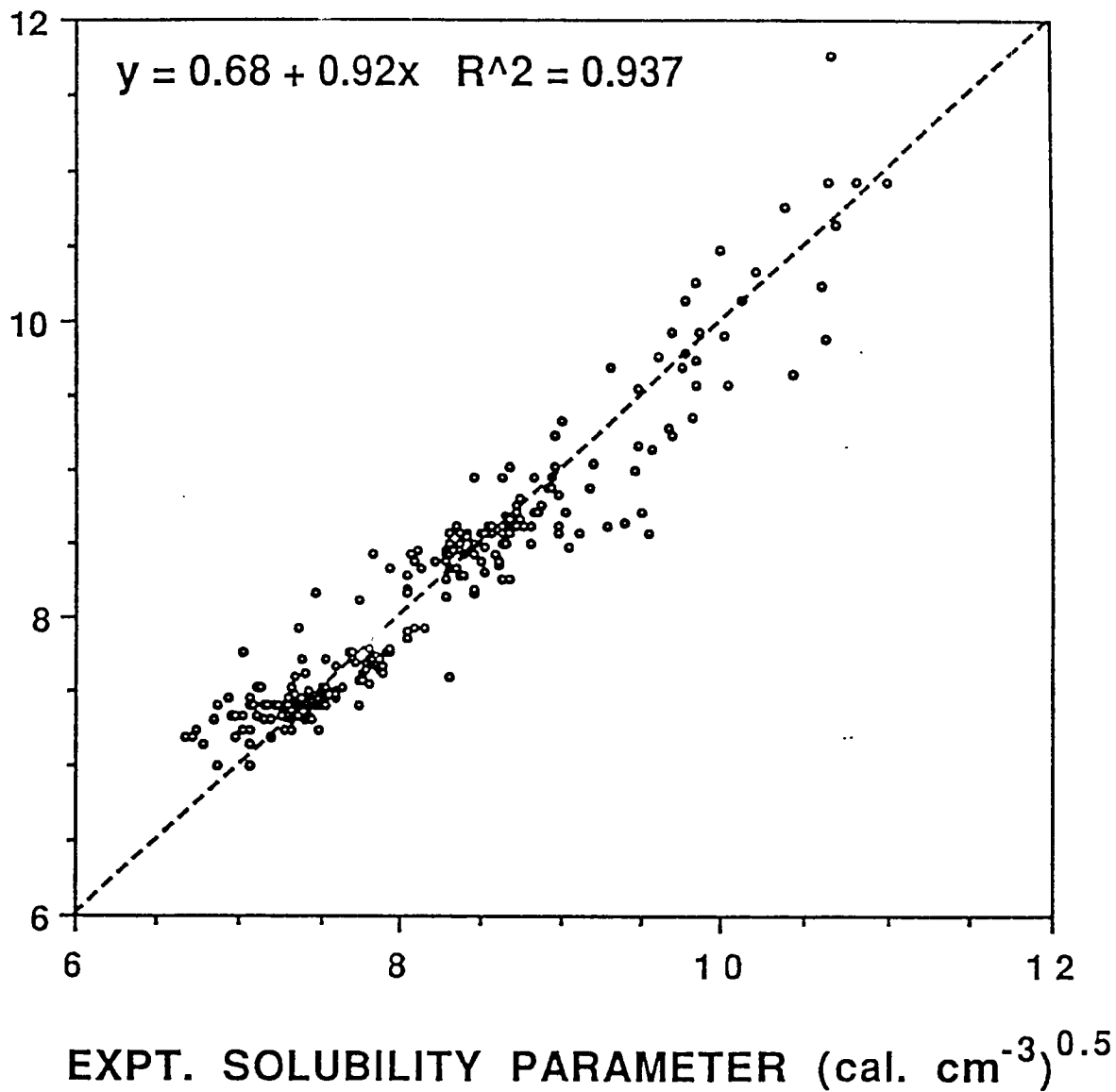


Figure 1. A comparison of experimental solubility parameters to those calculated from atomic contributions.

If we do have molecular weight data available, then we use the average volume per mole of asphalt determined from our group contributions program \bar{V} .

This is equivalent to the volume of an average unit adjusted to a molecular weight of about 100. The precise value of this molecular weight, \tilde{M} is also given by the program and actually ranges between 99 and 101, as a result of round off errors in the calculation. If the number average molecular weight is known, \bar{M}_n , then the volume of a mole of such "average molecules" is simply

$$\frac{\bar{M}_n}{\tilde{M}} \times \bar{V}$$

Then the required average segment molar volume (i.e. per oxygen atom) is given by

$$V_B = \frac{\text{Molar volume/molecule}}{\text{Number of oxygen atoms/molecule}}$$

$$= \frac{\bar{M}_n \bar{V}}{\tilde{M}} \times \frac{1}{N_o} \quad (7)$$

The number of oxygen atoms per molecule, N_o , is simply calculated from the weight percent oxygen from elemental analysis, O' , using

$$N_o = \frac{O'}{100} \times \frac{\bar{M}_n}{16} \quad (8)$$

The χ parameter in equation 2 can now be simply calculated using;

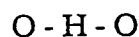
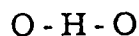
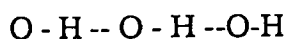
$$\chi = \frac{V_B}{RT} (\delta_A - \delta_B)^2 + F \quad (9)$$

Where F is an empirically determined factor equal to zero for mixing two high molecular weight materials, or two low molecular weight materials, but equal to 0.34 for mixing a high molecular weight polymer with a solvent. It is thought to be related to free volume differences and for asphalt mixtures we will assume its value to be zero.

Equation 9 shows that we calculate χ in terms of a scale of unit volume defined by V_B . We now do exactly the same thing for the ΔG_H term.

The contribution of hydrogen bonding interactions to the free energy of mixing.

In the calculation of the contribution of hydrogen bonding interactions to the free energy of mixing the crucial quantities are equilibrium constants describing the interactions between functional groups (e.g. - OH, COOH etc.). Clearly, we also require a knowledge of the concentration of these functional groups present in the materials being mixed. These quantities can be determined spectroscopically, but for some purposes various simple assumptions are adequate. For example, one contemporary model of asphalt structure proposes that hydrogen bonding associations are crucial. The type of associations that predominate in asphalt would be the self association of hydroxyl and acid groups;



together with OH/ether oxygen, OH/basic nitrogen interactions. Because acid/acid hydrogen bonded pairs are stronger than phenolic OH/OH interactions, we could assume that all oxygen atoms are in acid groups and then calculate the degree of hydrogen bonding. This would give us the maximum degree of association that could occur. We will consider the results of such calculations below; here our task is to outline how these calculations are made.

The equilibrium constants describing various types of hydrogen bonding interactions can be obtained directly or from studies of model systems. It is a consequence of the lattice model¹ that equilibrium constants for a particular functional group determined in one molecule can be transferred to a different molecule with the same functional group by simply adjusting according to the molar volume:

$$K_B^1 V_B^1 = K_B^2 V_B^2$$

And it should now be clear why we define the molar volume of an average segment in terms of the number of oxygen atoms present. In studying a range of polymer molecules containing phenolic and carboxylic acid functional groups we have determined values of $K_B^1 V_B^1$, which are listed in reference 1. Note that for phenols formation of dimers differs from the formation of subsequent h-mers ($K_2 \neq K_B$). Also note that an enthalpy of hydrogen bond formation (which has also been determined) is necessary in order to calculate changes in the equilibrium constants with temperature.

Once the equilibrium constants have been determined or calculated, these are input to a program that calculates ΔG_H . The equation that describes this term and a detailed description of the methodology of these calculations are described in the literature¹. To give a "feel" for the strength of hydrogen bonding interactions, values of K_B , describing the self-association of phenol are of the order of 60 (for a reference molar volume of 100 cm³), while acid groups have a K_B of the order of 300,000! (they self-associate, into pairs, much more strongly). the corresponding values for the enthalpy of these hydrogen bonds are about 5 kcal/mole and 7k cal/mole, respectively.

AVAILABILITY AND USE OF PROGRAMS

The various programs for the calculation of the phase behavior of mixtures, the calculation of solubility parameters and the use of the miscibility guide can be obtained from Technomic Publishing Company, Lancaster, PA 17604. The programs can be purchased with a book "Specific Interactions and the Miscibility of Polymer Blends", which contains a step-by-step guide to the use of the programs (Chapter II). You will also require an update to be able to calculate the solubility parameters of asphalt from atomic contributions. This can be obtained by writing to

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For copyright reasons, the original programs must be purchased first, however.

The program update is for Macintosh computers only and they are all "user friendly". (In other words, you can easily learn to use them by just "playing around".)

RESULTS - SAMPLE CALCULATIONS

Here we will describe the results of three types of calculations that will serve to illustrate the types of things that can now be done. We believe these calculations are more than just examples, however, and serve to throw some insight into asphalt structure and its ability to mix with other materials. Furthermore, they also serve to highlight the type of structural information that needs to be obtained in future calculations if this type of work is to be placed on a more accurate quantitative basis.

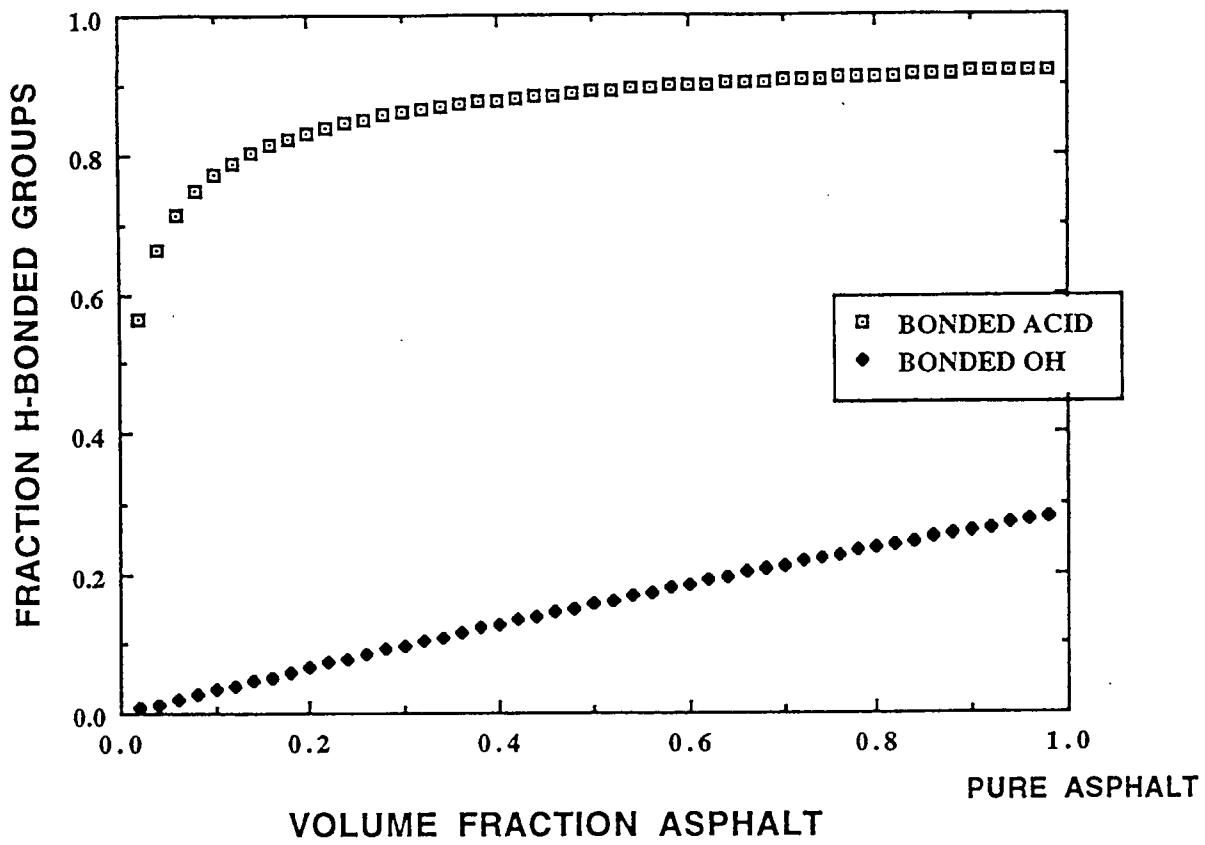
Hydrogen bonding in Asphalt

It has been proposed that hydrogen bonds play a key rôle in the association of asphalt molecules, that such associations can account for various aspects of asphalt physical properties and seriously influence such things as column separations and molecular weight measurements.

At first our calculations might seem to support this model. The plot shown at the top of figure 2 shows the calculated fraction of hydrogen bonded groups for Asphalt AAD/toluene mixtures at 25°C. The concentration of carboxylic acids and phenolic OH groups was taken from measured values and the equilibrium constants describing the self association of these species have been established in our laboratories¹. We assume that phenolic OH groups only hydrogen bond to each other and to $\text{C}=\text{O}$ groups, but not to carboxylic acids. Work on polyamic acids⁷, also performed in these laboratories, indicates that this is a good assumption.

It can be seen from figure 2 that even though the overall concentration of hydrogen bonding functional groups is small, the fraction of such groups that are bonded is significant. This is particularly true for carboxylic acid groups, which strongly self

ASPHALT (AAD) / TOLUENE MIXTURES



ASPHALT (AAD) / TOLUENE MIXTURES

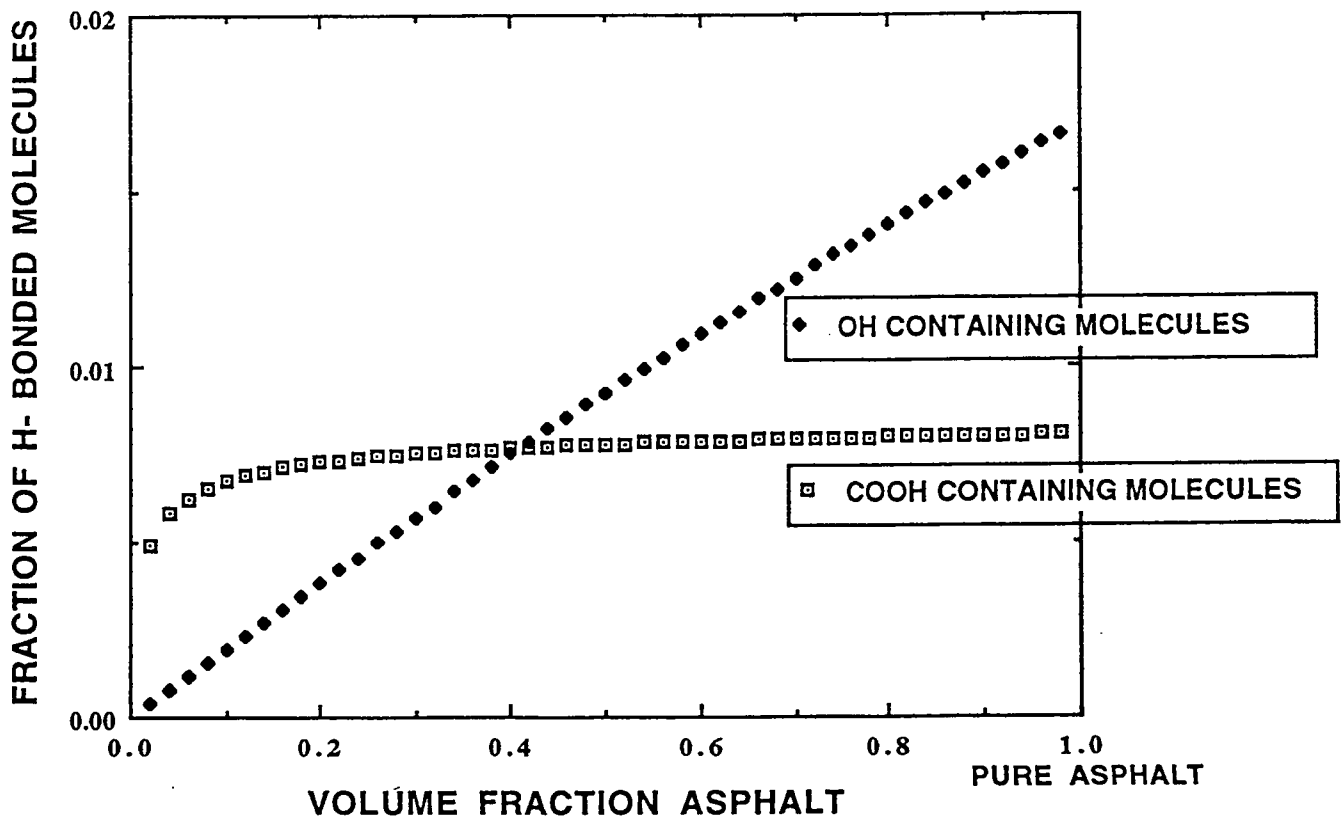


Figure 2. Top; calculation of the fraction of hydrogen bonded functional groups. Bottom; calculation of the fraction of hydrogen bonded molecules.

associate. We calculate that more than 90% of such groups are hydrogen bonded in pure asphalt at any particular instant of time. Even when diluted with a non hydrogen bonding solvent, such as toluene, the fraction of hydrogen bonded COOH groups only drops significantly at concentrations of asphalts below 5%.

However, from measurements of number average molecular weight and oxygen content we can also calculate the fraction of molecules that are hydrogen bonded to one another and this tells a very different story. These results are shown at the bottom of figure 2, where it can be seen that in the pure asphalt only 1% or 2% of the molecules are hydrogen bonded to one another. This is far too small a number to produce any sort of network within the asphalt and suggests that hydrogen bonds play only a secondary rôle in determining most bulk properties.

Similar results were obtained for other asphalts and as an example figure 3 shows the fraction of hydrogen bonded molecules calculated for asphalt AAG.

Asphalt - A single phase or two phase system?

Here our results are equivocal, because we simply do not have good data for the individual structural components of most asphalts. As an example, consider mixing of the strong acid fraction of asphalt G with the neutral fraction. Using measured parameters we calculate the phase diagram shown in figure 4 (between - 100°C and 300°C). It is completely featureless! This says that the two components are miscible over the entire composition range. An increase in the molecular weight of the components would produce the phase diagrams shown in figure 5, however, where the region beneath the inverted U shape curve is two phase. So one of our first conclusions is that we need good molecular weight data!

The second thing we need is good structural data. For the strong acid/neutral components of asphalt A, for example, we did not have any data on the distribution of functional groups. If we assume all the oxygen is present as carboxylic acids, then we get the phase diagram shown in figure 6, which is characteristic of a two phase system.

ASPHALT (AAG) / TOLUENE MIXTURES

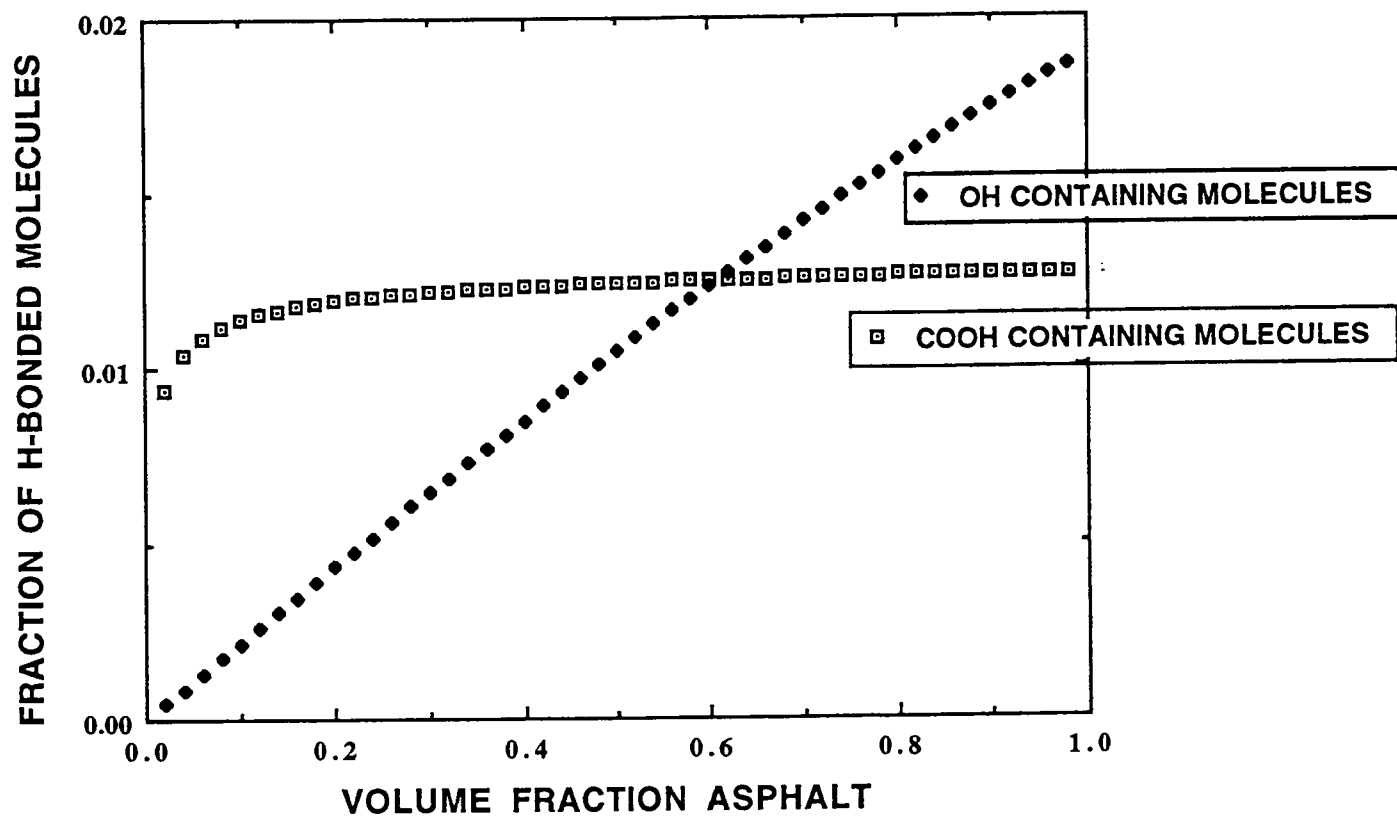


Figure 3. Calculation of the fraction of hydrogen bonded molecules in asphalt AAG.

Poly(neutrals) and Poly(strong acids) Blend

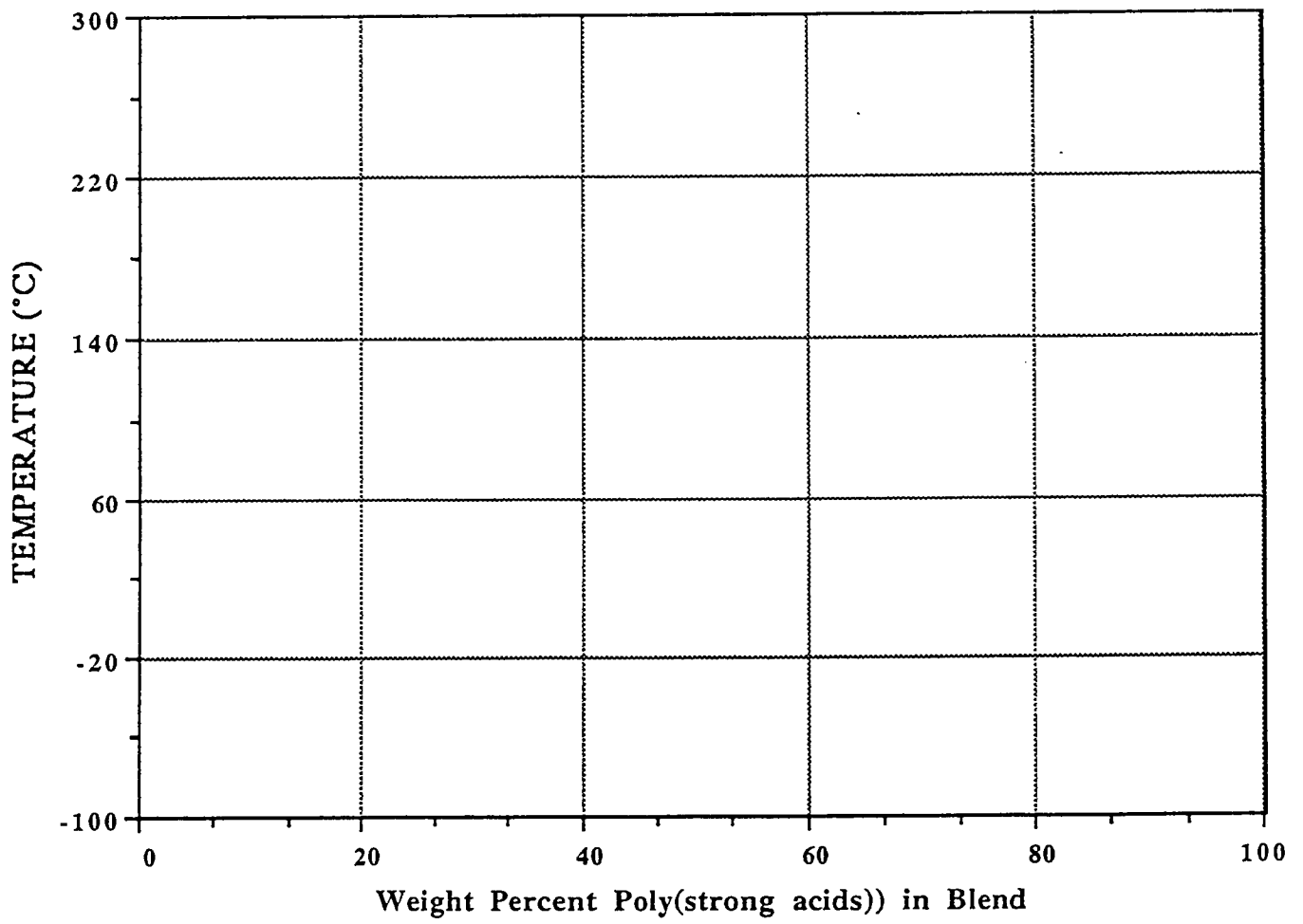


Figure 4. Phase diagram for mixing the neutral and strong acid components of asphalt AAG.

ASPHALT G

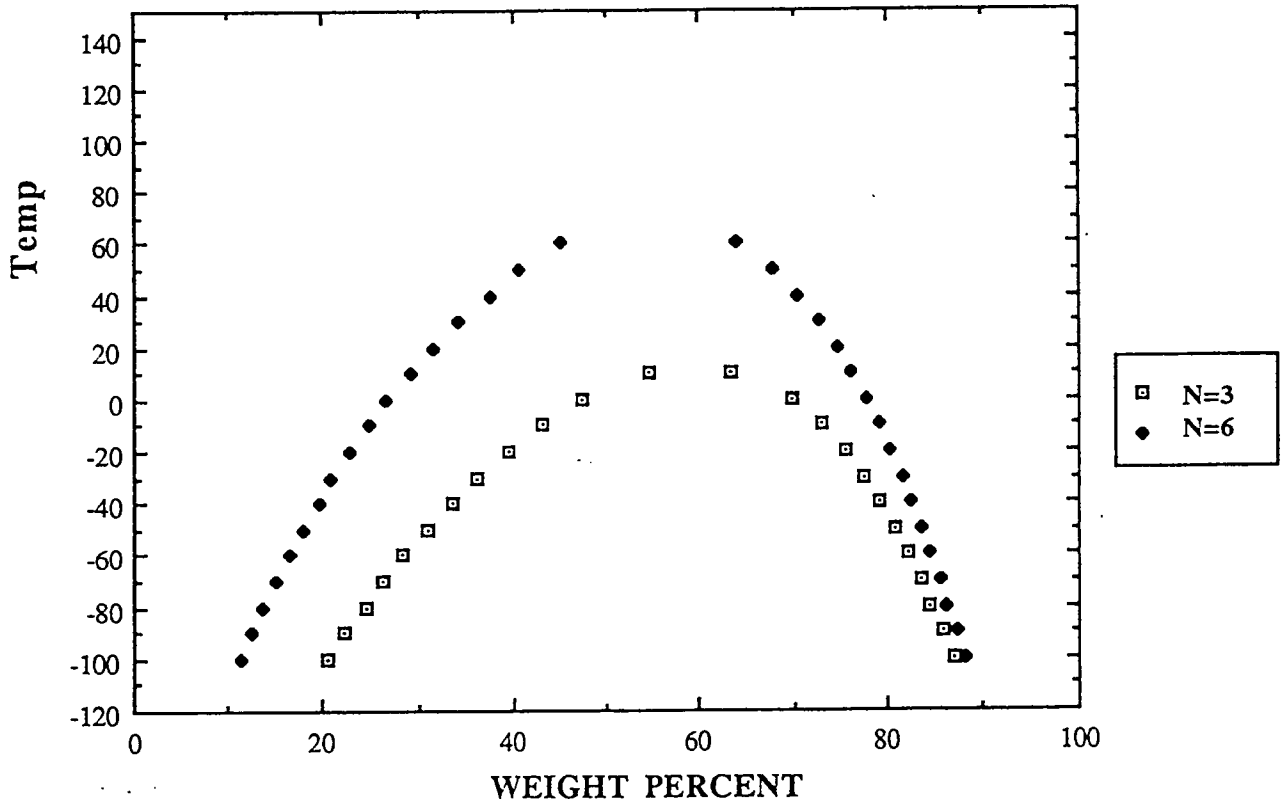


Figure 5. The effect of increasing the molecular weight of the neutral fraction by factors of 3 (N=3) and 5 (N=5).

Poly(neutrals) and Poly(strong acids) Blend

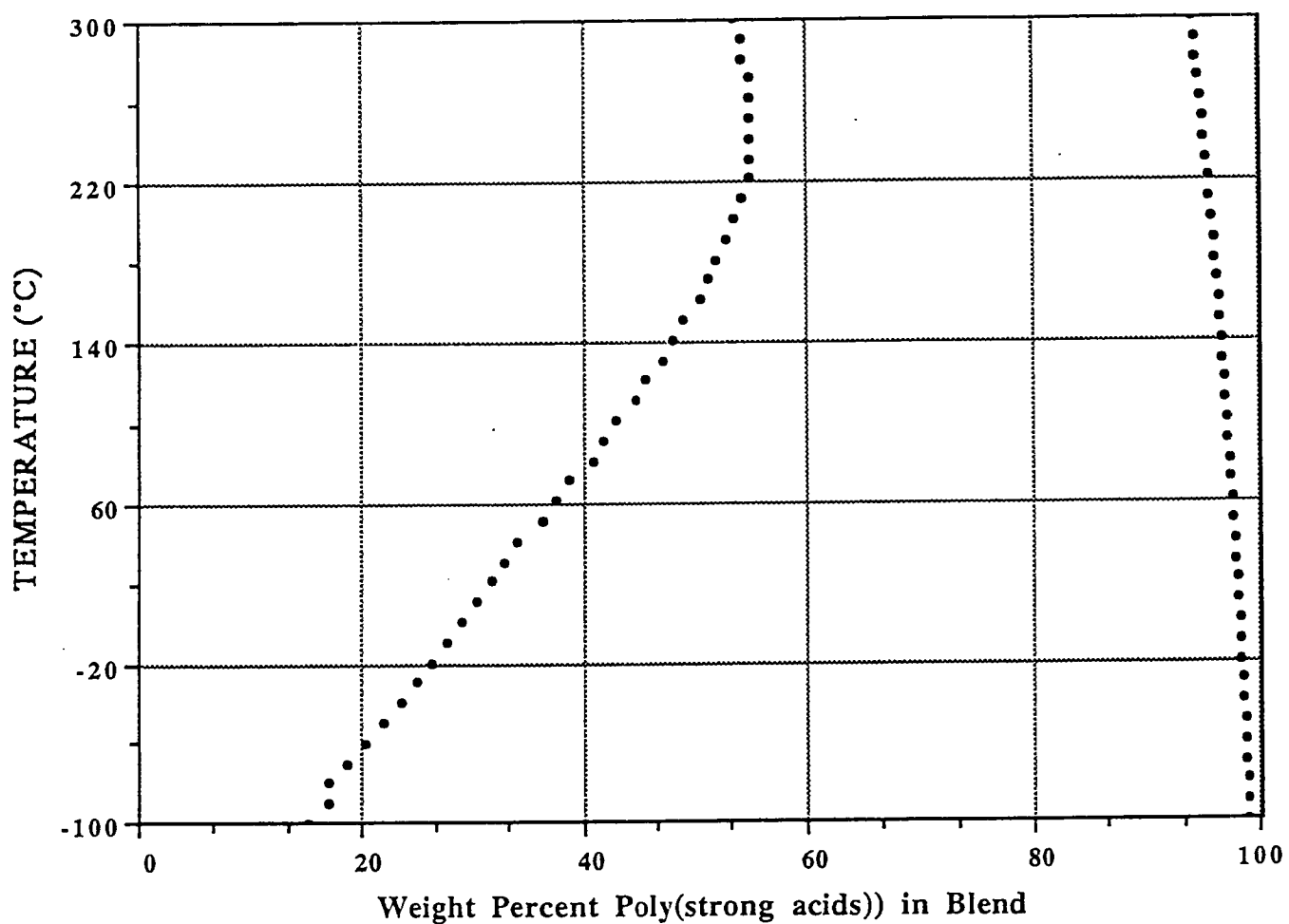


Figure 6. The effect of assuming that all the oxygen atoms in the strong acid fraction are in carboxylic acids groups.

Because acid groups strongly self associate the calculated phase diagram is very sensitive to the acid content. So here again, we cannot draw any reasonable conclusions without more precise analytical data concerning functional group concentrations and distribution.

Asphalt/Asphalt and Asphalt/polymer mixtures

Because more accurate data is available for whole asphalts we can obtain our most useful results for these types of mixtures. We have to assume that the individual asphalts are single phase, but given that assumption we calculate very boring asphalt/asphalt phase diagrams. They are all blank, like in figure 4 indicating that they are single phase systems. A word of caution is in order here, however. A recycled asphalt could well be significantly oxidized and this would, in turn, affect its ability to mix with a “fresh” asphalt. We need to have some reliable numbers that describe the functional group distribution in an oxidized or recycled asphalt in order to be able to perform these calculations.

Mixing asphalts with synthetic polymers is a different story, as the solubility parameters of many common plastics and elastomers are well known. Figure 7, for example, shows a phase diagram for polyethylene/asphalt AAD mixtures. The system is two phase over most of the composition range at ambient temperatures. We would not expect polyethylene to mix significantly even in the single phase regions (i.e., high PE content), however, because this polymer crystallizes to a significant extent (similar results are calculated for other asphalt/polyethylene mixtures).

In contrast, PVC (poly(vinyl/chloride)) is predicted to form single phase mixtures with, for example, asphalt AAG, at most ordinary temperatures, although a phase separation is predicted to occur at low temperatures. This is because the solubility parameter of PVC is much higher than polyethylene and fairly close to that of most asphalts.

In additions to calculating classic phase diagrams (temperature/composition plots) we can calculate so-called miscibility maps, which display single and two phase regions for

Poly(POLYETHYLENE) and Poly(ASPHALT D) Blend

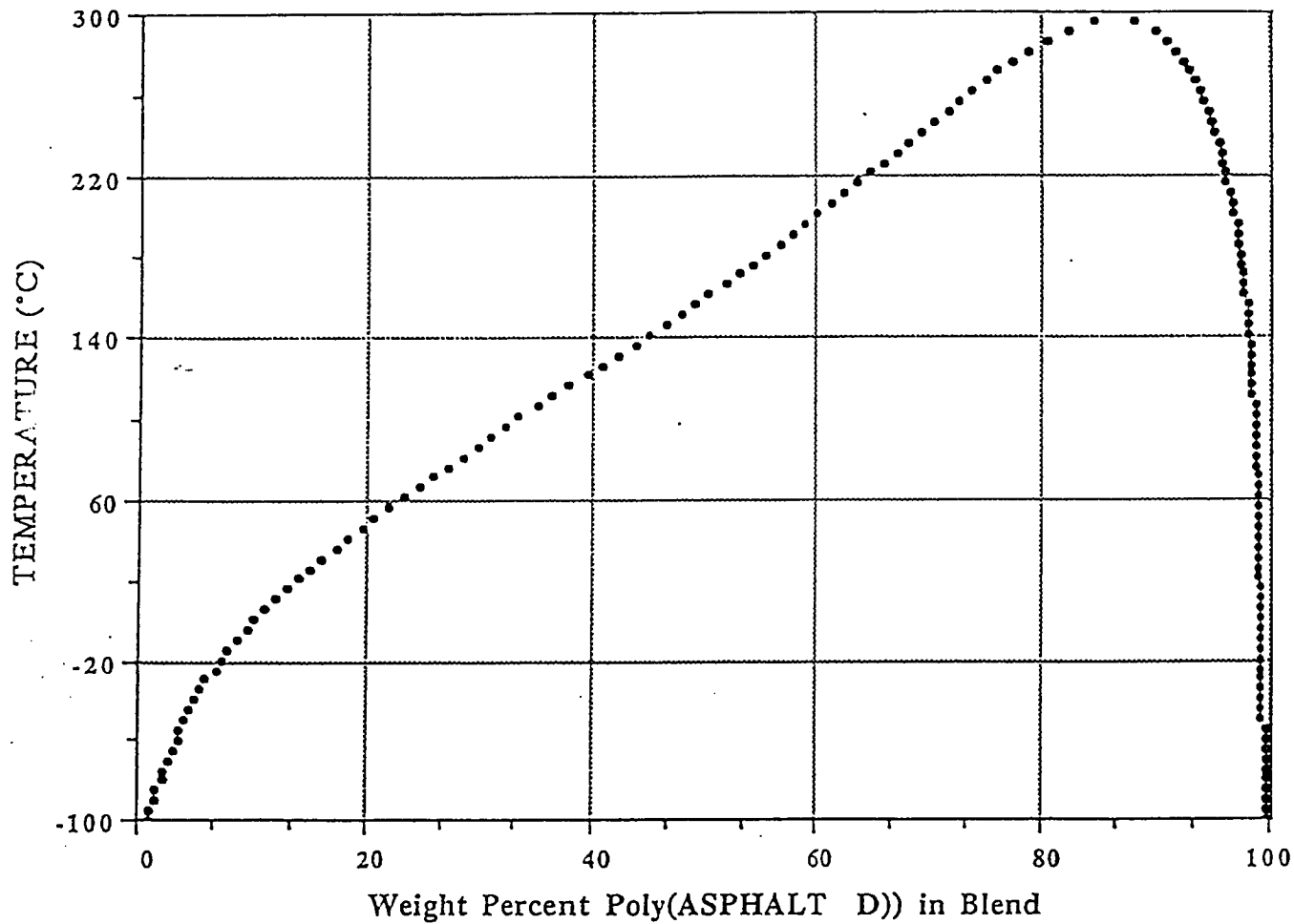


Figure 7. The phase behavior of Asphalt AAD/Polyethylene mixtures.

Poly(PVC) and Poly(ASPHALT G) Blend

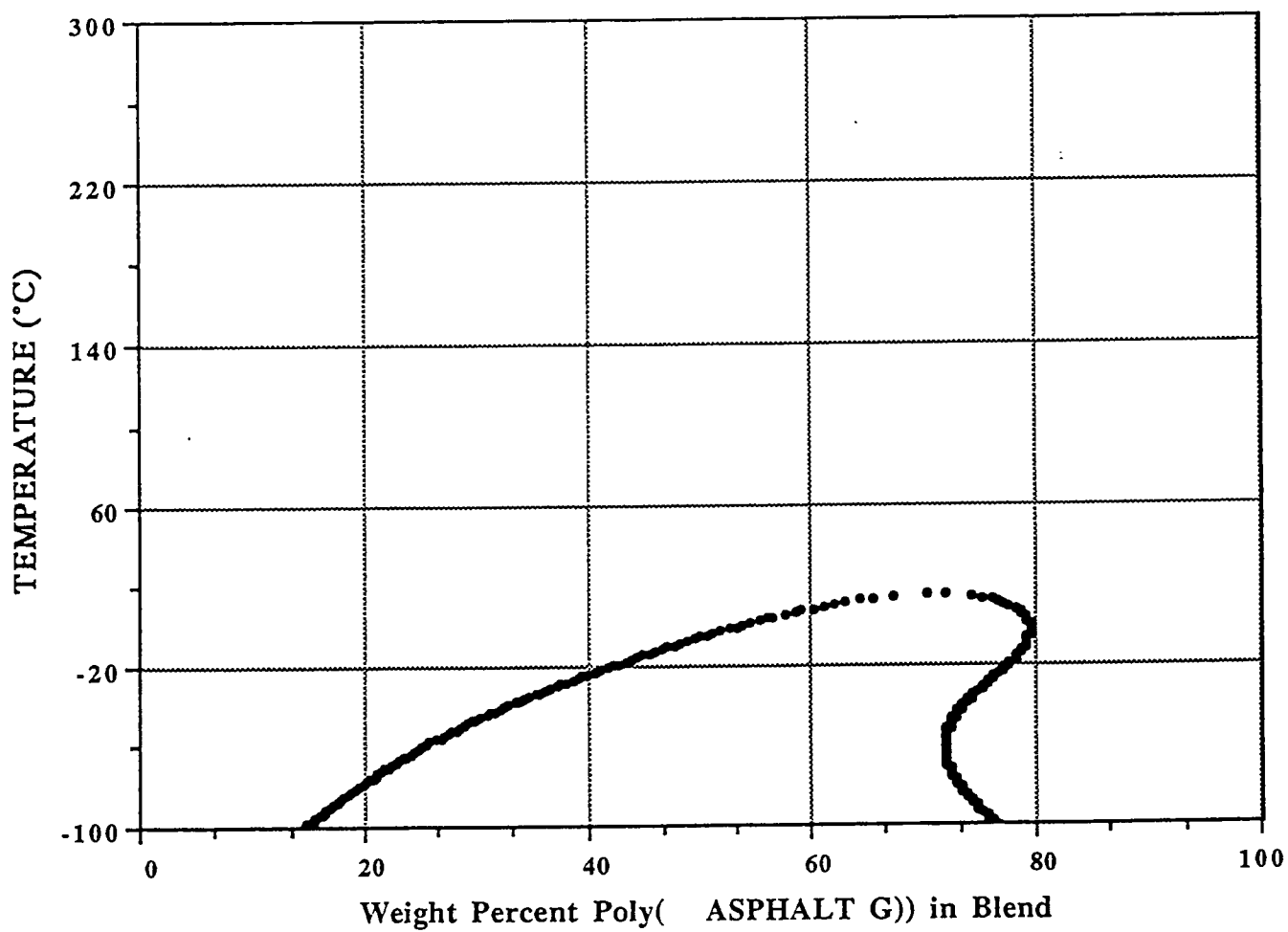


Figure 8. The phase behavior of Asphalt AAG/PVC mixtures.

multicomponent systems for a given temperature. Figure 9 shows such a plot for a random styrene/butadiene copolymer mixed with an asphalt at -20°C . The y axis show the weight of butadiene in the copolymer, while the x-axis is the weight of asphalt in the mixture. The calculated loops enclose two phase regions at the top and bottom of the plot, leaving a single phase region for mixtures where the copolymer has a butadiene content between about 10 and 40%.

As the temperature is raised this miscibility gap widens considerably, as shown in figures 9, 10 and 11. Accordingly, we would only expect those copolymers with a low butadiene content to be miscible with asphalt.

This brings us to the final general point we wish to make. When considering the mixing of asphalt with materials such as plastics the microstructure of the polymer can be crucial. Even if we calculated that natural rubber were miscible with an asphalt for example, (we don't, for the asphalts we have considered, because the solubility parameter of natural rubber is too low), this still would not allow us to use recycled tires to form single phase materials, *because the rubber in these tires is chemically cross linked*. Similarly, kraton, which is a *block copolymer* of styrene and butadiene, would have very different phase behavior to the random copolymers described above. Our calculations indicate that styrene is probably miscible with most asphalts, but butadiene is not. Accordingly, mixing such thermoplastic rubbers with asphalt should lead to a dispersion of small rubber domains within the asphalt. This in turn should improve the impact properties of asphalt significantly (most impact resistant polymers consist of fine dispersions of rubbers in a more rigid matrix material).

Poly(STYRENE-co-BUTADIENE) and Poly(ASPHALT D) Blend

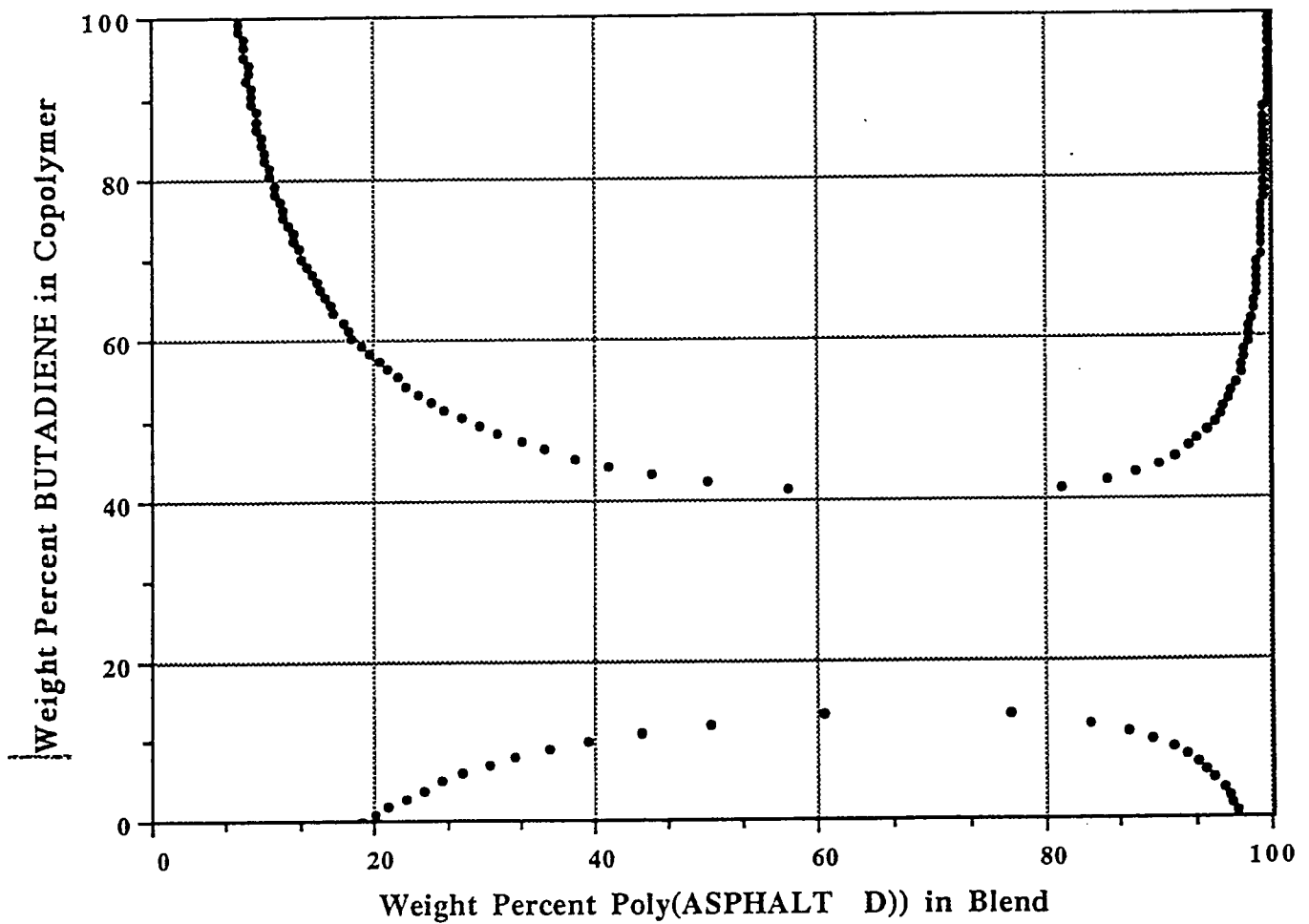


Figure 9. A miscibility map for mixtures of Asphalt AAD with random styrene/butadiene copolymers (-20°C).

Poly(STYRENE-co-BUTADIENE) and Poly(ASPHALT D) Blend

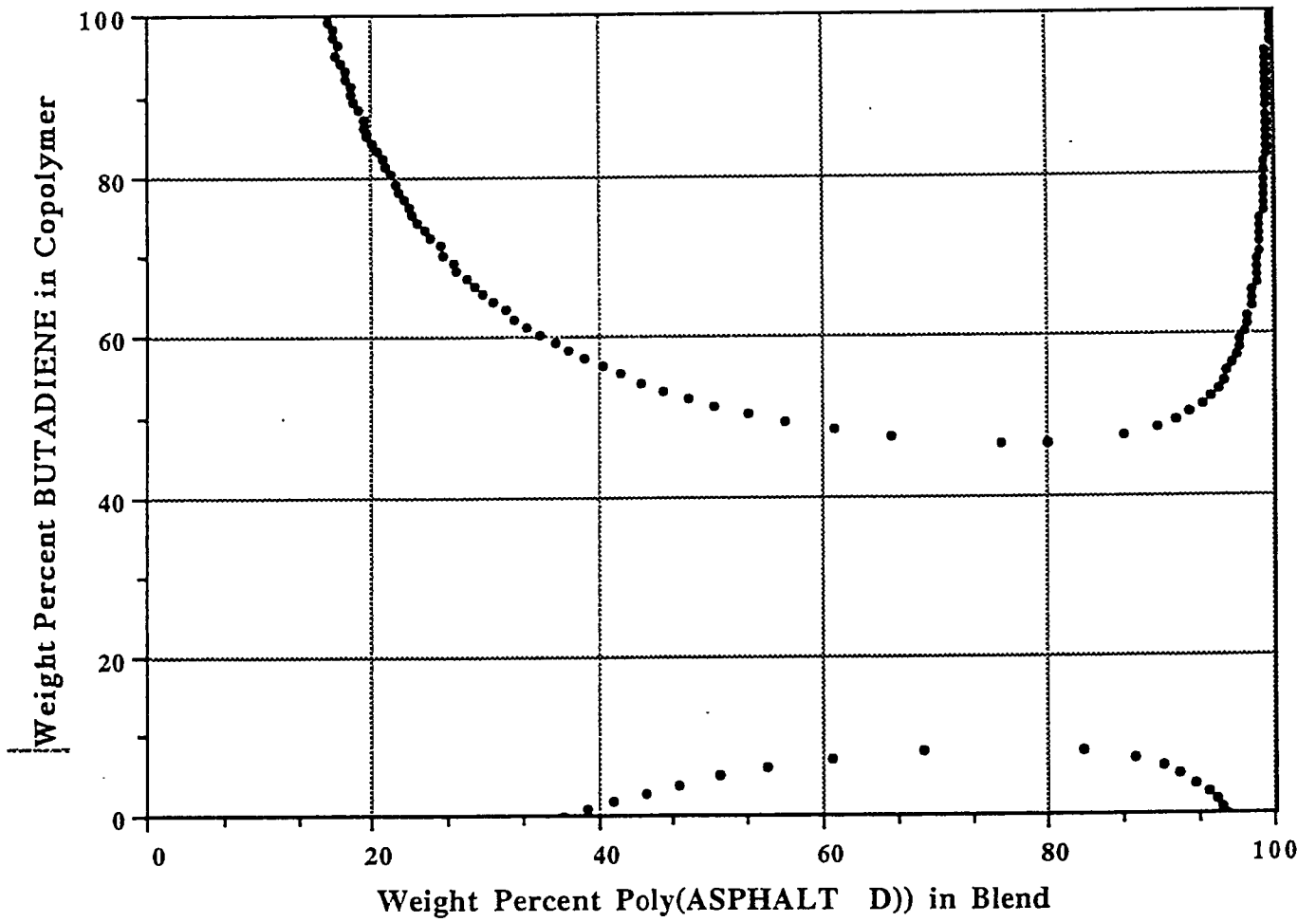


Figure 10. Miscibility map calculated at 25°C.

Poly(STYRENE-co-BUTADIENE) and Poly(ASPHALT D) Blend

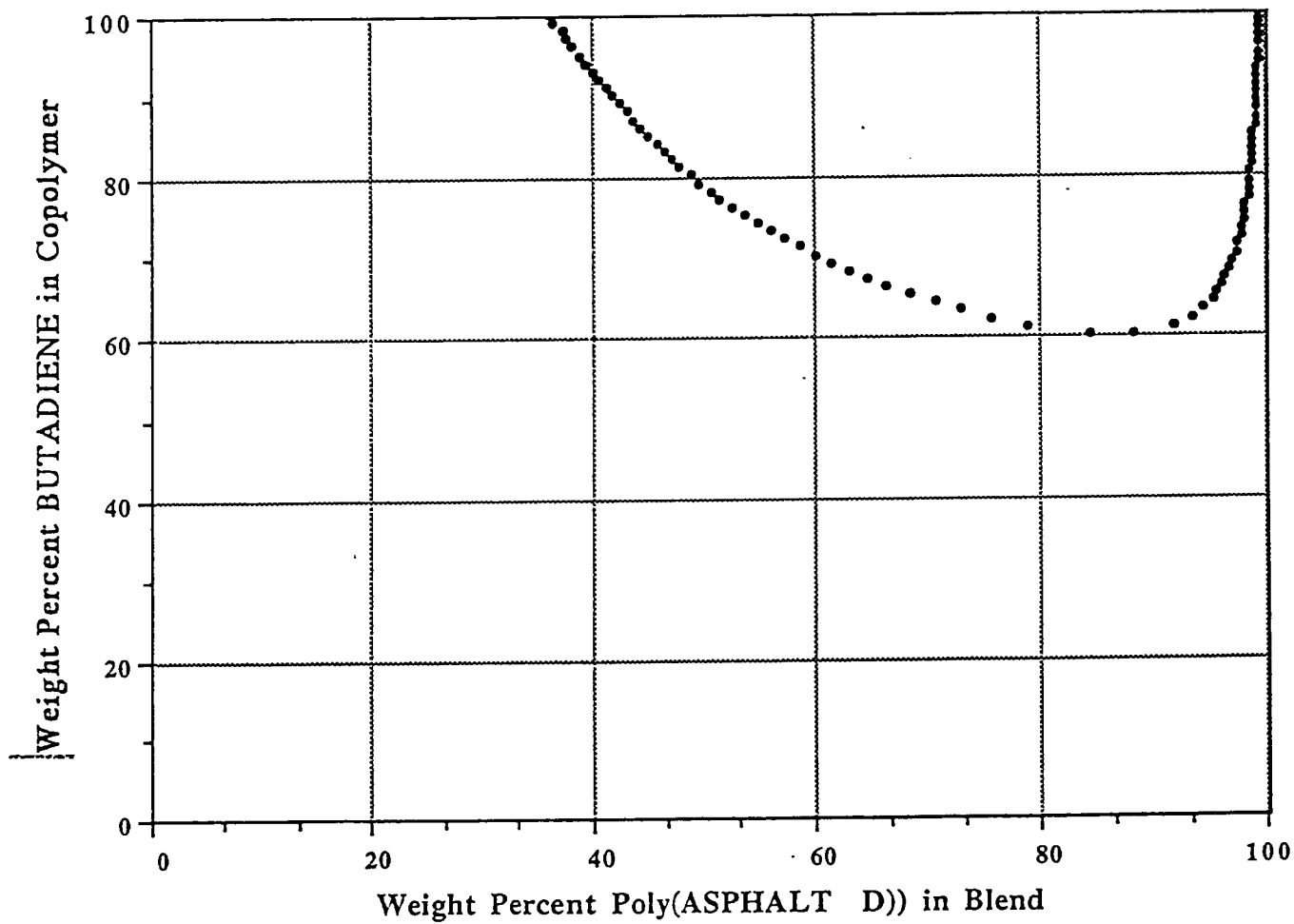


Figure 11. A miscibility map calculated at 100°C.

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