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SOLUBILITY CHARACTERISTICS OF A METHACRYLATE COPOLYMER

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POLYMER RESEARCH BRANCH

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ABSTRACT

Solubility maps in several solubility coordinate systems have been constructed to determine regions of solubility for a methacrylate copolymer. The general methodology followed was that of ASTM D 3132 Test Method for Solubility Range of Resins and Polymers. Over 90 individual solvents and solvent mixtures were tested as solvents for the copolymer in the concentration range 5% - 7.5% w/v. A database on the solubility characteristics of common solvents was created to store and utilize the information. Solvent maps were formulated using the solubility parameter-hydrogen bond index-dipole moment system, the Hansen solubility parameter system, and the Teas fractional cohesion parameter system. Each system is primarily empirical in nature, but has varying degrees of theoretical justification.

Key words

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INTRODUCTION

A semi-dilute (8g/dL) multi-component solution is under investigation in which the solute is a high molecular weight copolymer serving as a viscoelastic additive and the three cosolvents are components which undergo further reaction. It is of critical importance that the isobutylmethacrylate-co-t-butylaminoethylmethacrylate (iBMA/tBAEMA) copolymer remain soluble during the reaction process to ensure complete reaction. The end-use requirements mandate that the copolymer remain soluble in the resulting reaction product in order to provide the desired viscoelastic properties. In addition, the copolymer must remain soluble in the reactants for long periods of time before use. Thus, the solubility of the copolymer in the original reactants and in the final reaction product is critical.

The entire solubility range (as determined by two-dimensional plots of various solvent properties) of the copolymer was determined to assure that the coordinates of the ternary solvent system and of the final reaction product, the compositions of which are proprietary, lie well within the boundaries of the soluble portion of the solubility phase diagram.

The general solubility test method outlined in ASTM D 3132[1] has been used to assess the solubility range of the copolymer in various organic solvents and solvent mixtures. The solvent mixtures, however, have not been limited to those listed in the test method. The resulting solubility data has been plotted using Hansen cohesion parameters and Teas fractional cohesion parameters in addition to the Hildebrand solubility parameter/dipole moment/hydrogen bond index method described in the ASTM method.

The Hildebrand solubility parameter, (δ), is defined[2] as the square root of the cohesive energy density, or cohesive pressure,

$$\delta = c^{1/2} = (-U/V)^{1/2} \tag{1}$$

where U is the internal energy of the molecule and V is its molar volume. This can be envisioned as the amount of energy required to disperse the molecules in the liquid state, similar to what occurs in vaporization. This solubility parameter is also referred to as a cohesion parameter,[3] and may be experimentally determined from the molar enthalpy of vaporization and the molar volume *via* $U = \Delta H - RT$. The units of δ are generally $(\mathrm{MPa})^{1/2}$ or $(\mathrm{cal/cm^3})^{1/2}$.

The ASTM D 3132 method is based on correlating the Hildebrand parameter with the hydrogen bonding ability and the dipole moment of a solvent to determine regions of solubility and immiscibility for polymers. One method of quantifying the hydrogen bonding ability of a solvent (hydrogen bond index, γ) was developed by Crowley et. al.[4] and subsequently has been designated with the subscript "C". This parameter is defined by

$$\gamma_C = \Delta\nu/10 \tag{2}$$

where $\Delta\nu$ is the spectroscopic shift (in wavenumbers) observed for the O-H band in the IR spectrum of an alcohol in a solvent of interest, relative to the spectrum of the neat alcohol.

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[1] ASTM D 3132-84 *Standard Test Method for Solubility Range of Resins and Polymers*, American Society for Testing and Materials. Philadelphia, PA, 1984.

[2] HILDEBRAND, J. H., and SCOTT, R. L. *Solubility of Non-Electrolytes.* 3rd Edition, Reinhold, New York, 1950, Dover, New York, 1964.

[3] BARTON A. F. M. *CRC Handbook of Solubility Parameters and Other Cohesion Parameters.* CRC Press, Inc, Boca Raton, Florida, 1983.

[4] CROWLEY, J. D., TEAGUE, G. S., Jr., and LOWE, J. W. *A Three-dimensional Approach to Solubility.* J. Paint Technol., v. 38, 1966, p. 269.

The stronger the hydrogen bonding between the solvent and the hydroxyl hydrogen, the weaker the O-H bond in the alcohol, and the lower is the frequency of the vibration. The third parameter used in the ASTM method is the dipole moment of the solvent (in Debye units).

Hildebrand's original solubility parameter[2] was intended for non-polar, non-associating molecules, but has been used for all molecules anyway. Hansen[5] extended the Hildebrand solubility concept to a three-component solubility parameter composed of dispersive (non-polar), polar, and hydrogen bonding contributions. The total solubility parameter (δ_t) is essentially equivalent to the Hildebrand parameter, although minor differences exist due to the values being determined by different methods. The Hansen components are related to one another through the equation

$$\delta_t^2 = \delta_d^2 + \delta_p^2 + \delta_h^2 , \tag{3}$$

where the subscripts d, p, and h refer to the dispersive, polar, and hydrogen bonding components, respectively. The dispersive contribution of a molecule is usually estimated by comparing the δ value of a polar molecule with the δ value of a non-polar molecule of similar size and shape (homomorph method[6]). This dispersive contribution is then subtracted from the total solubility parameter, and the remainder can be divided into hydrogen bonding and polar contributions using various semi-empirical approaches.

Although Hansen's approach is somewhat more theoretical in nature, in terms of practical applications (i.e., determining solubility ranges for polymers), it does not appear to offer any significant advantage over that of the ASTM Hildebrand parameter-hydrogen bond index system. The only advantage to either system would probably be the availability of data for the particular solvents of interest.

Another three-component cohesion system was developed by Teas[7] using *fractional* cohesive parameters. This system is based on the idea that the total cohesion parameter is composed of the same three components as proposed by Hansen, but the fraction of the total cohesion parameter describing each of the three components is significant, as opposed to the actual value of the cohesion parameter. In other words, the total cohesion parameter for all liquids is the same, but the contribution arising from the three forces (polar, dispersive, and hydrogen bonding) differs from molecule to molecule. One advantage of this method is that the three components can be plotted on a triangular chart in two dimensions, whereas the other systems described require three dimensions (or two-dimensional contour plots) to view the effects of three parameters simultaneously. However, there is little theoretical justification for this type of analysis, considering the variation in the total solubility parameters, as can be seen in Appendix A.

---

[5] HANSEN, C. M. *The Three-dimensional Solubility Parameter - Key to Paint Component Affinities. I. Solvents, Plasticizers, Polymers and Resins.* J. Paint Technol., v. 39, 1967, p. 104.

[6] BROWN, H. C., BARBARAS, G. K., BERNEIS, H. L., BONNER, W. H., JOHANNENSEN, R. B., GRAYSON, M., and NELSON, K. L. *Strained; Homomorphs. 14. General Summary.* J. Am. Chem. Soc., v. 75, 1953, p. 1.

[7] TEAS, J. P. *Graphic Analysis of Resin Solubilities.* J. Paint Technol., v. 40, no. 19, 1968, p. 516.

EXPERIMENTAL

Materials

The iBMA/tBAEMA copolymer was obtained from Polysciences (Lot #CM1-120). The composition of the copolymer was determined to be 77:23 from ^{13}C NMR and an average molecular weight of 2.9×10^6 has been estimated by aqueous GPC and light scattering.

The following solvents were used individually and in the solvent mixtures:

Acetone, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.
Acetonitrile, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.
n-Butanol, certified grade. Fisher Scientific. Used as received.
2-Butoxyethanol, 99%. Aldrich Chemical Company, Inc. Used as received.
Cyclohexane, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.
Cyclohexanone, technical grade. Distilled before use.
1,2-Dichloroethane, certified grade. Fisher Scientific. Used as received.
Dichloromethane, distilled in glass (UV grade). Caledone Laboratories, Ltd. Used as received.
Diisopropyl ether, anhydrous, 99%. Aldrich Chemical Company, Inc. Used as received.
Dimethylsulfoxide, certified grade. Fisher Scientific. Used as received.
Ethanol, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.
2-Ethyl-1-hexanol, 99%. Aldrich Chemical Company, Inc. Used as received.
Ethylene Carbonate, 98%. Aldrich Chemical Company, Inc. Used as received.
n-Heptane, distilled in glass (UV grade). Burdick & Jackson Laboratories, Ltd. Used as received.
n-Hexane, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.
Methanol, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.
N-Methylpyrrolidone, GC grade. Burdick & Jackson Laboratories, Ltd. Used as received.
Nitrobenzene, certified grade. Fisher Scientific. Used as received.
Nitromethane, 96%. Aldrich Chemical Company, Inc. Used as received.
n-Pentane, GC grade. Caledon Laboratories, Ltd. Used as received.
Phenol. Aldrich Chemical Company, Inc. Used as received.
n-Propanol, Certified ACS grade. Fisher Scientific. Used as received.
Pyridine, reagent grade. British Drug House, Ltd. Used as received.
Tetrahydrofuran, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.
Toluene, distilled in glass (UV grade). Caledon Laboratories, Ltd. Used as received.

Procedure

The copolymer (0.15 ± 0.01 g) was weighed into 4 mL glass vials fitted with teflon-lined caps. The solvent components were delivered into the vials individually via burets or graduated pipets (the total volume of solvent added was 2 to 3 mL, to keep the copolymer solution concentration between 5 and 7.5 g/dL). The vials were then tightly capped and put into a drum tumbler rotating end-over-end (approximately 14 rpm) for 48 to 72 hours. (The ASTM method specifies 48 hours at 1 to 5 rpm, however, at the higher mixing rate used in this study, some of the more viscous solutions required more mixing time.) After mixing, the solutions were analyzed visually for solubility and characterized as follows:

Soluble (S):	Transparent solutions of one phase.
Borderline (B):	One phase which appears cloudy or turbid.
Insoluble (I):	Two distinct phases.

The solution composition (by volume percent of solvent components) was recorded along with the solubility designation for each mixture. The solubility parameters were calculated for each mixture and plotted in the three different coordinate systems previously discussed.

RESULTS AND DISCUSSION

Database

A solubility database has been created for the individual solvent components used in this study. This database contains the following information:

Solvent names (up to five names or abbreviations).
Molar volume (g/ml).
Hildebrand solubility parameter $(\mathrm{cal/cm^3})^{1/2}$.
Hydrogen bond index (Crowley parameter, wavenumbers).
Dipoloe moment (Debye units, 3.336×10^{-30} C-m).
Hansen dispersion solubility parameter $(\mathrm{cal/cm^3})^{1/2}$.
Hansen polar solubility parameter $(\mathrm{cal/cm^3})^{1/2}$.
Hansen hydrogen bonding solubility parameter $(\mathrm{cal/cm^3})^{1/2}$.
Teas fractional dispersion cohesion parameter (x100, Unitless).
Teas fractional polar cohesion parameter (x100, Unitless).
Teas fractional hydrogen bonding cohesion parameter (x100, Unitless).

This information is listed in Appendix A for the solvents used in this study. The solubility data stored in this database are used to calculate the solubility constants for the solvent mixtures based on the solubility constants of the individual components and their volume fraction in the mixture. The calculated solubility constants are defined as

$$S = (\Phi_1 s_1 + \Phi_2 s_2 + \Phi_3 s_3) / \Sigma \Phi_i \qquad (4)$$

where S is the calculated solubility constant for the mixture of i solvent components (e.g., S could be the Hildebrand solubility parameter, the Hansen polar parameter, etc.), s_i is the solubility constant for each solvent component and Φ_i is its corresponding volume fraction. The volume fraction of component i is defined by

$$\Phi_i = V_i x_i / (V_1 x_1 + V_2 x_2 + V_3 x_3). \qquad (5)$$

The single and multi-component solvents used in this study are listed in Appendix B, along with the experimentally determined solution solubilities (soluble, borderline, or insoluble), as well as the solubility constants as calculated from Equations 4 and 5. The different solubility phase diagrams or *maps* were constructed from the information in Appendix B, as described in the following section. Most of the solvent mixture compositions were obtained from a previous solubility study[8] on a similar polymer, poly(methylmethacrylate-co-isobutylacrylate-co-ethylacrylate), and others were designed to fill in the undefined areas in the diagram.

Solubility Phase Diagrams

Figures 1 through 7 show two-dimensional plots of the various solubility parameters indicating general areas of solubility and insolubility. Figure 1, in which the hydrogen bond index is plotted against the Hildebrand parameter, is the most common type of solubility map. The particular solvents and solvent mixtures in this study were chosen such that a closed solubility region would be determined on this type of plot (i.e., when areas on the plot were undefined, solvent mixtures with the appropriate coordinates were tested). Because the coordinates were chosen for a particular solubility system, the solubility regions on the other maps are not as clearly defined. Figures 2 and 3 show the dipole moment versus the Hildebrand solubility parameter and the hydrogen bond index, respectively. Figures 4 through 6 show the various Hansen solubility parameters (dispersion, hydrogen bonding, and polar) plotted in two dimensions.

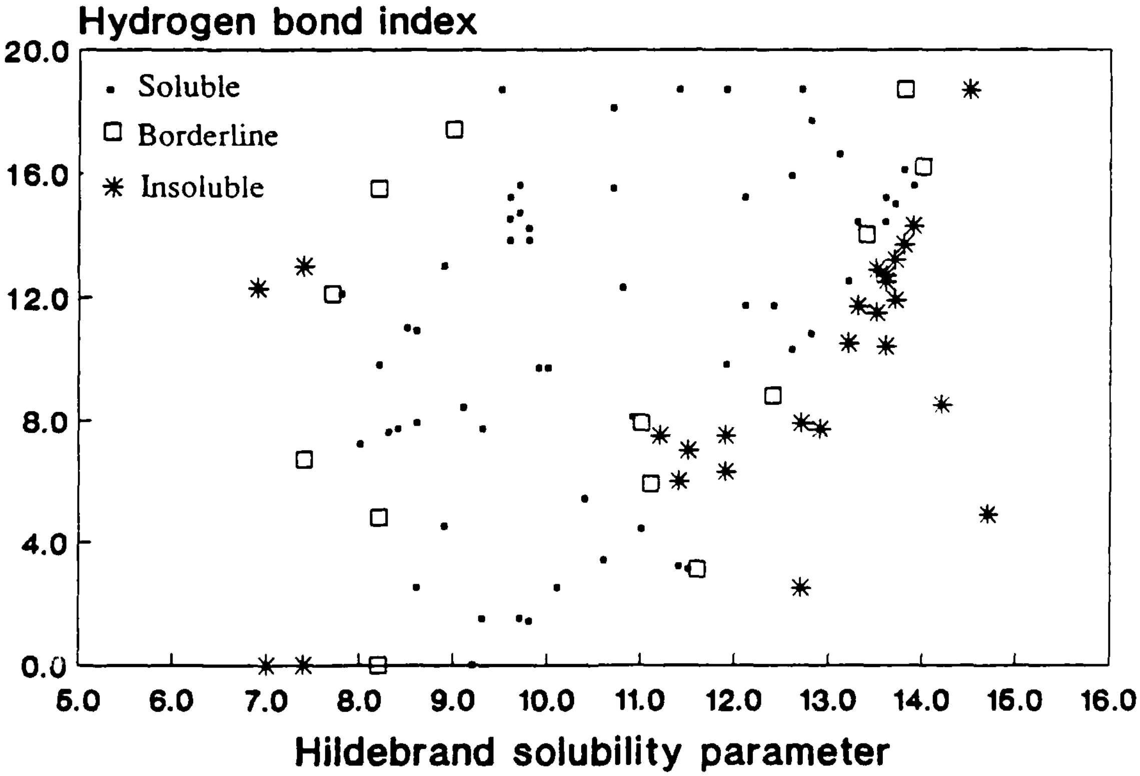

Figure 1. Plot of hydrogen bond index versus Hildebrand solubility parameter.

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[8] SHUELY, W J., and SCOTT, L. G. *The Polymer Solubility Phase Diagram for the Methylmethacrylate Copolymer Acryloid K125*. ARCSL-TM-80008, 1980.

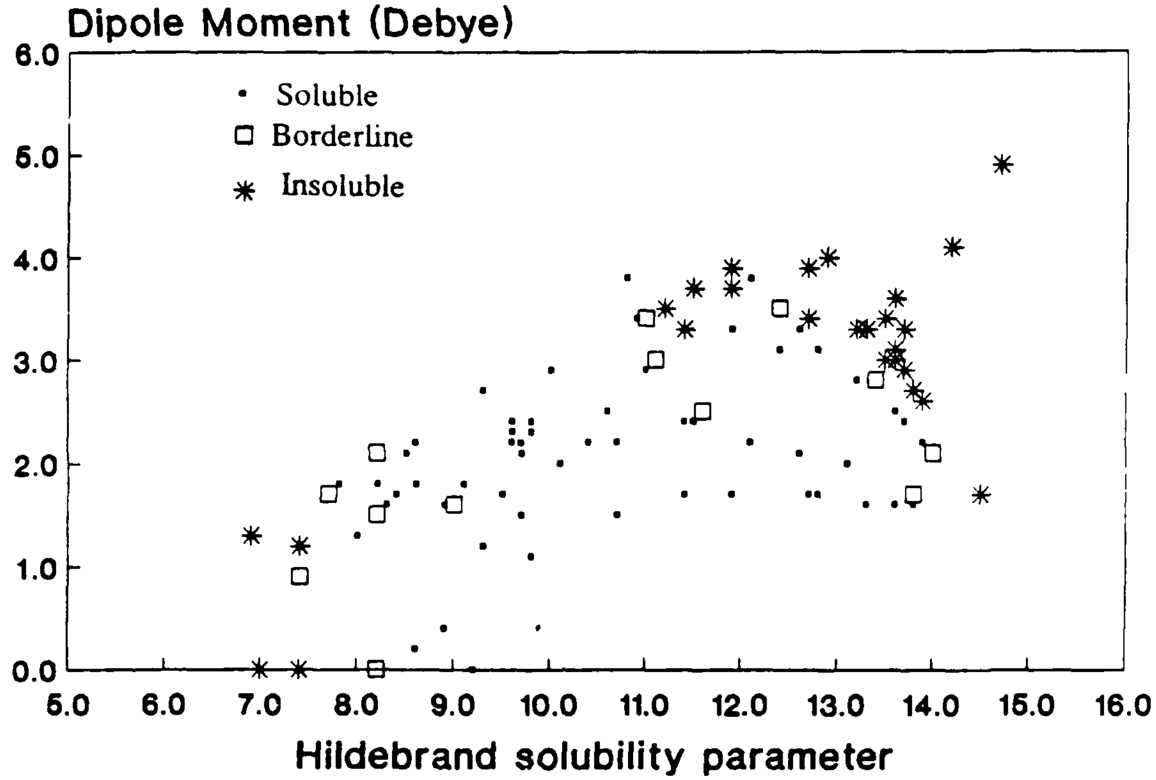

Figure 2. Plot of dipole moment versus Hildebrand solubility parameter.

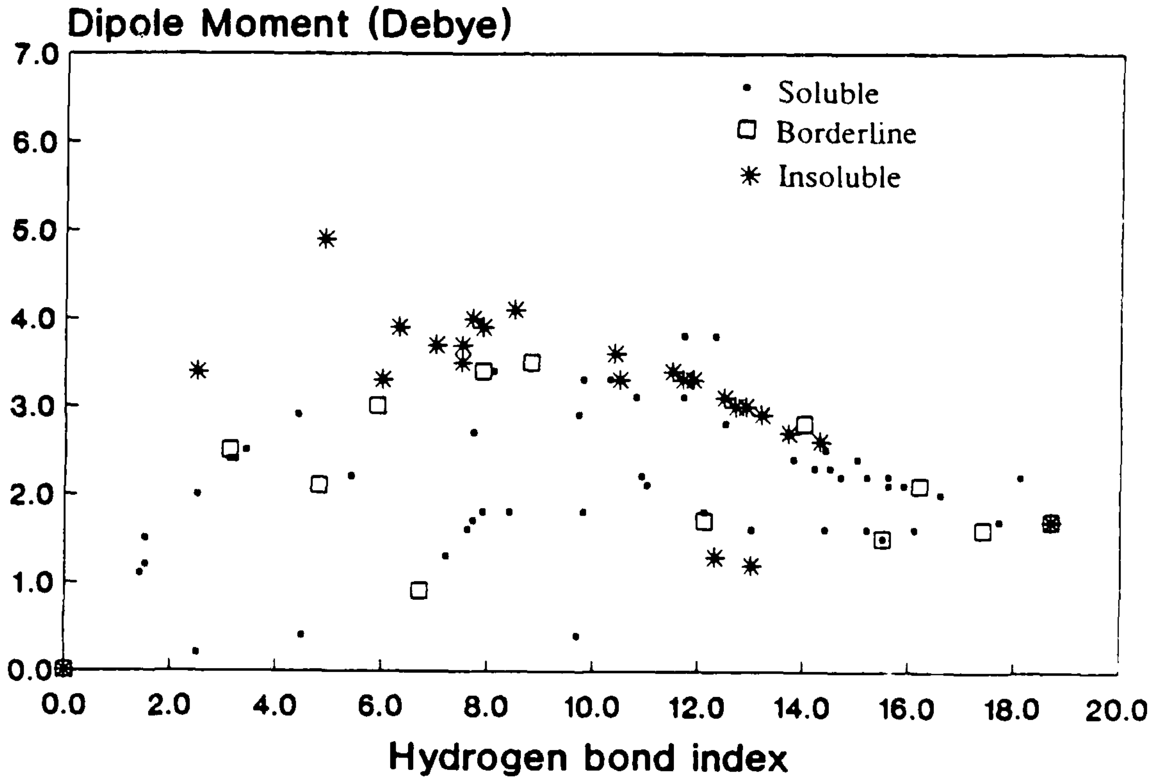

Figure 3. Plot of dipole moment versus hydrogen bond index.

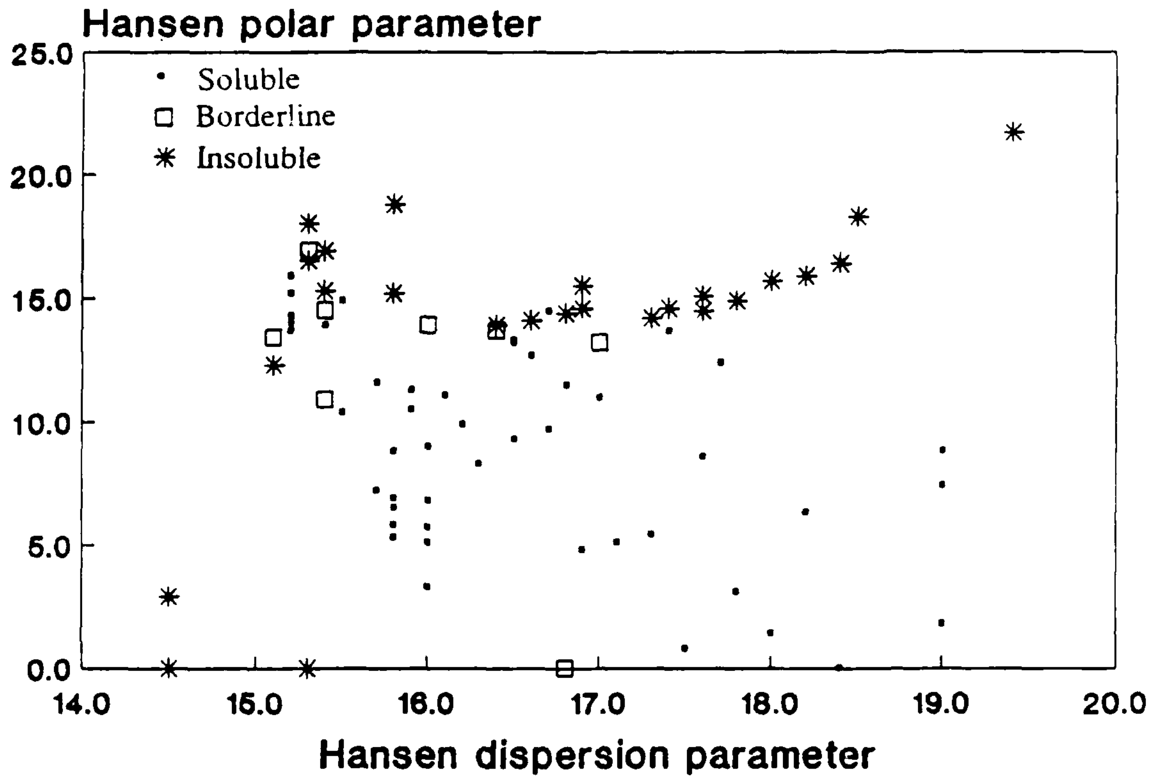

Figure 4. Plot of Hansen polar parameter versus dispersion parameter.

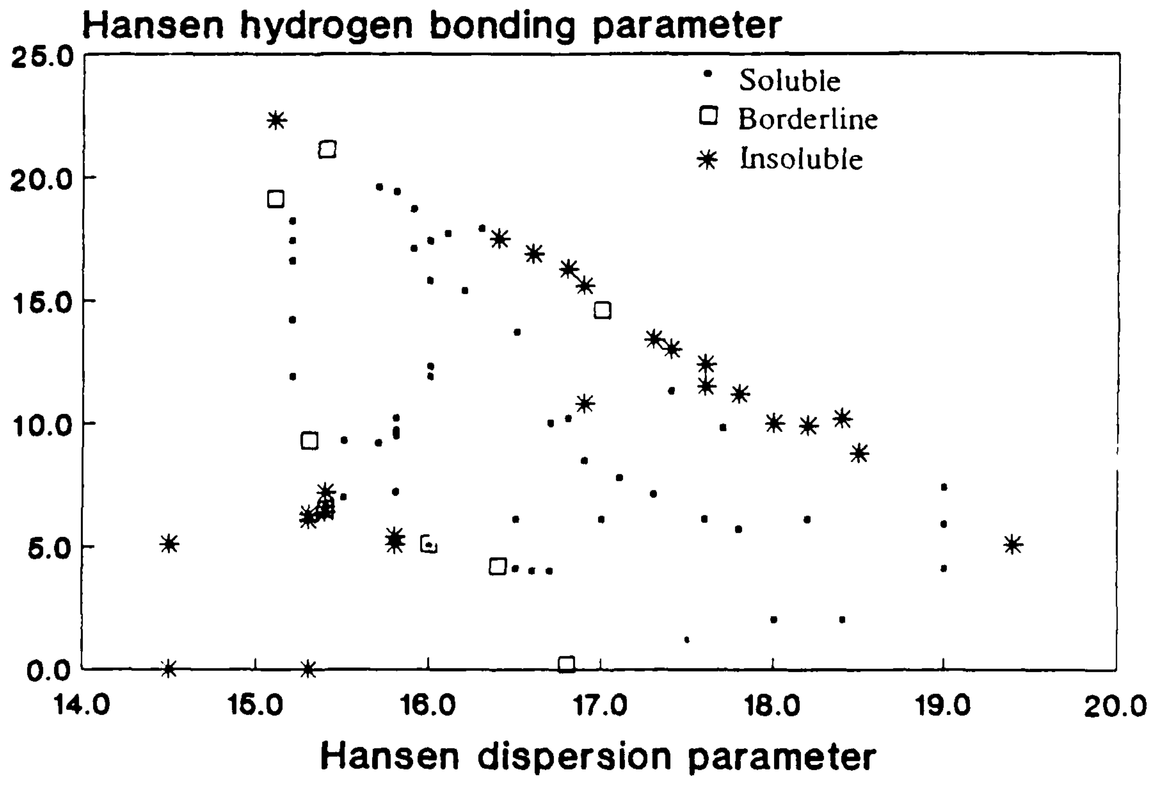

Figure 5. Plot of Hansen hydrogen bonding parameter versus dispersion parameter.

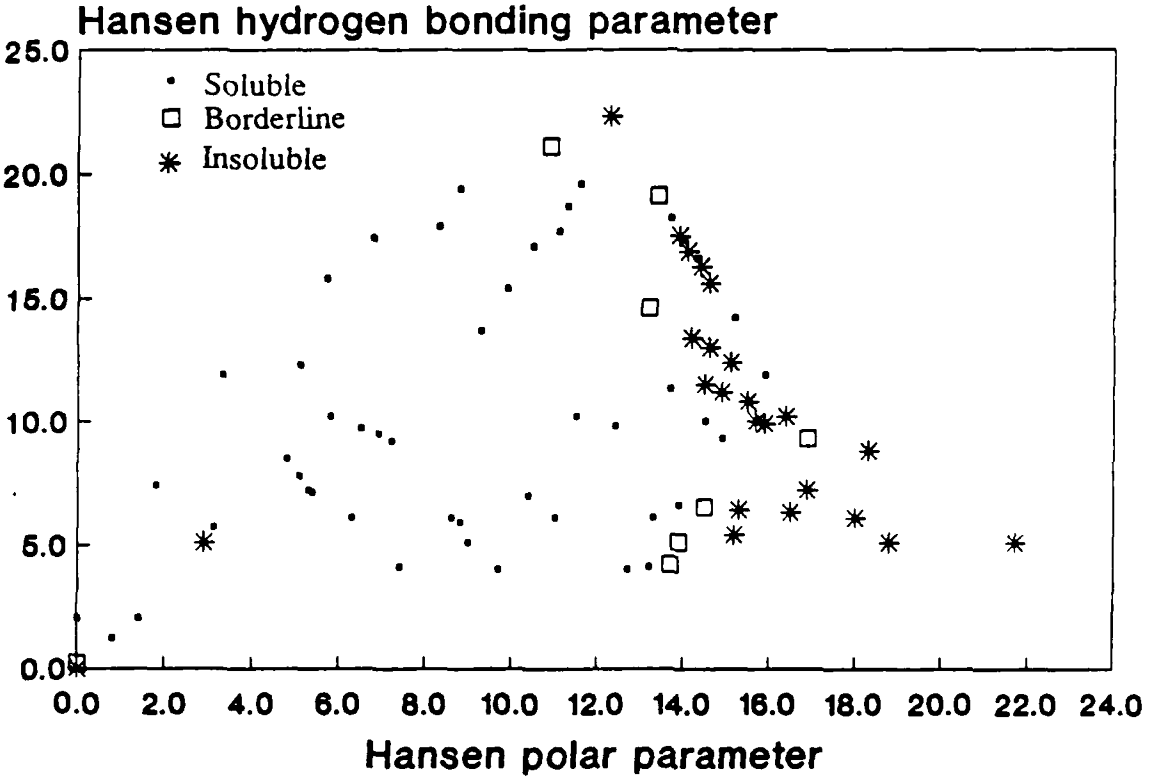

Figure 6. Plot of Hansen hydrogen bonding parameter versus polar parameter.

Three-dimensional solubility maps are probably the most appropriate way to analyze a solubility region, although they are difficult to construct. By adding a third dimension to Figure 1 (the dipole moment), Figures 1 through 3 could be combined for a single map. The same type of analysis could be applied to the Hansen cohesion system with Figures 4 through 6. Alternatively, contour plots can be drawn on two-dimensional maps. This requires more planning in selecting the solvent mixtures, however, noting that the third dimension values must be at certain fixed values. Neither three-dimensional nor contour plots have been constructed for this study.

Figure 7 shows the Teas three fractional cohesion parameters plotted on a triangular chart in two dimensions, as explained in the Introduction. This type of analysis appears to be almost three-dimensional in nature, but since the three-fractional parameters sum to unity, only two are actually independent variables (e.g., $f_h = 1 - f_d - f_p$). The presence of the third variable, however, changes the values of the other two and, thus, it has some influence on the plot. The points on a triangular plot such as this are determined by constructing lines of constant solubility for each *axis* by drawing lines at equal increments along the axis parallel to the adjoining axis, as shown in Figure 8. The intersection of these *iso* lines for each axis represents the point of interest. The point corresponding to ethyl ether with the coordinates (f_d, f_p, and f_h) equal to (0.64, 0.13, and 0.23) is indicated in Figure 8 by a circle.

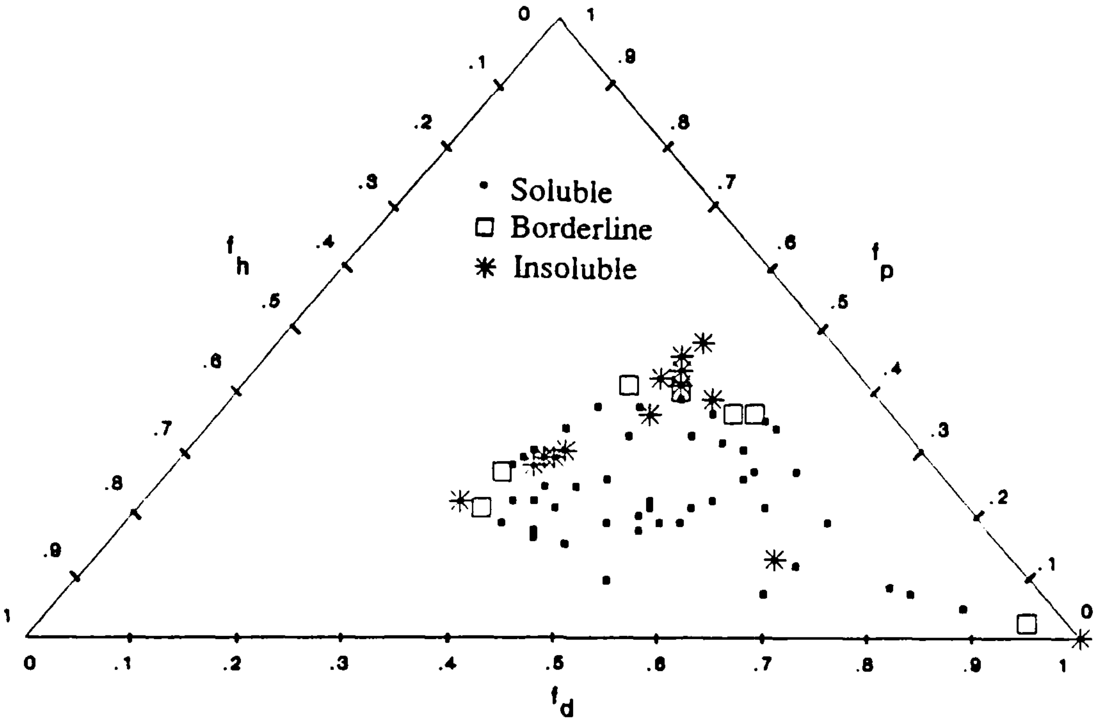

Figure 7. Plot of Teas fractional cohesion parameters on triangular plot.

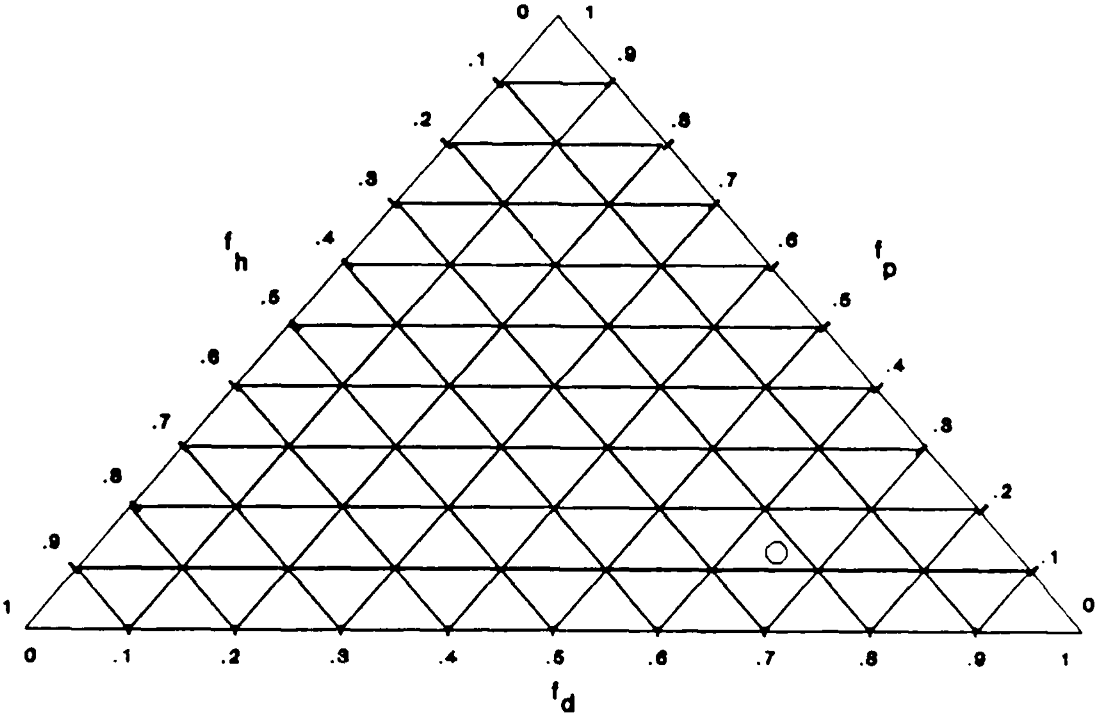

Figure 8. Plot of Teas fractional cohesion contours.

The experimental points in Figure 7 appear to cluster in the lower right hand corner of the plot, corresponding to a high dispersive content and little polar or hydrogen bonding contribution, and, indeed, this is to be expected for most liquids. A solubility border is roughly indicated by the line extending from (0.3, 0.2, and 0.5) to (0.4, 0.5, and 0.1). The borders along the other two axes are not as clearly defined.

Figure 9 shows the general solubility region, as depicted in Figure 1, along with the previously determined[9] duPont solubility map for poly(isobutytlmethacrylate). The area of overlap for the two solubility regions is large, presumably because the isobutylmethacrylate component is in large excess (75%) in the copolymer. The two components of the copolymer are both methacrylate esters, differing only in their alkyl groups and one of the alkyl groups contains an amino group. Despite the similarities in the solubility maps, there is an area characterized by a high solubility parameter and intermediate to high hydrogen bonding, in which only the copolymer is soluble. Solvents whose coordinates lie in this region are dimethylformamide, N,N-dimethylacetamide and N-methylpyrrolidone. According to the duPont[9] map, poly(iBMA) would not be soluble in these solvents; however, it is known* that poly(iBMA) is soluble in both DMF and N-methylpyrrolidone. The reason for this discrepancy can probably be attributed to the three-dimensional nature of the solubility diagram. The third parameter of interest with regard to the solubility map is the dipole moment. The duPont solubility map[9] for the iBMA polymer was determined for solvents of low to intermediate dipole moment. Apparently, the solubility of poly(iBMA) is enhanced at higher values of the dipole moment. This can be envisioned as larger contours at increasing dipole moment values. In other words, poly(iBMA) is probably not soluble in liquids which have the same solubility parameter and hydrogen bond index as DMF, but a smaller dipole moment.

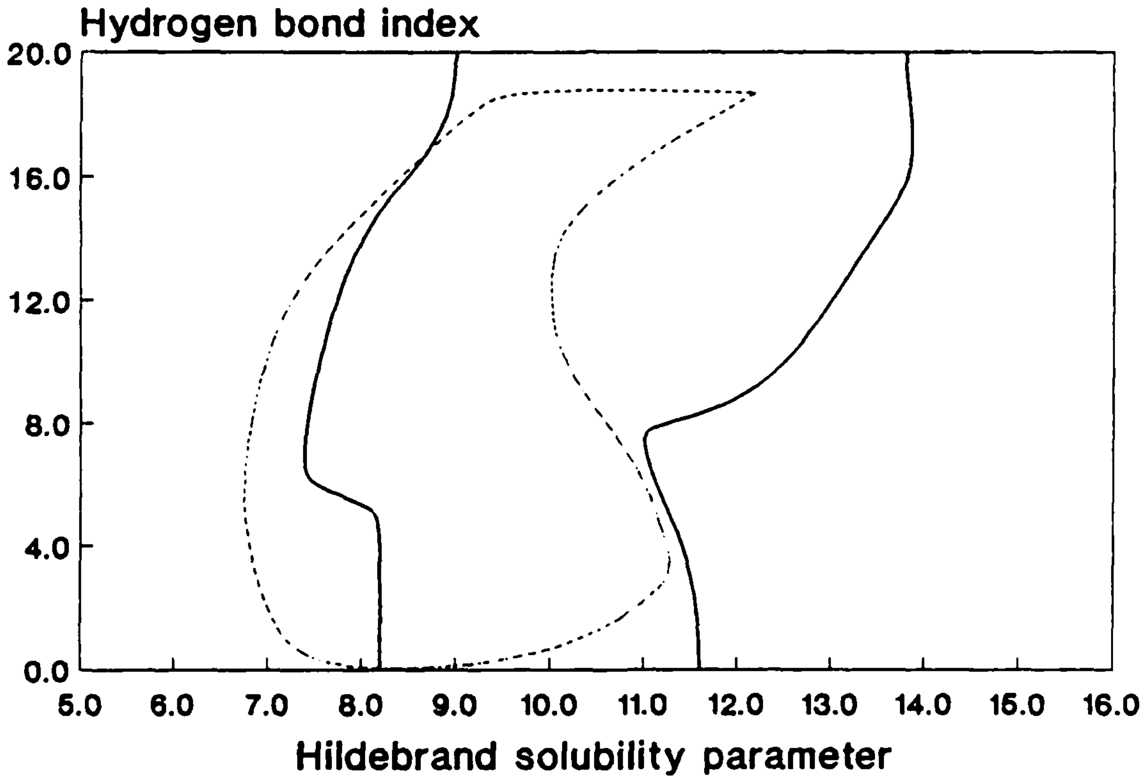

Figure 9. Plot of solubility contours for copolymer (—) and iBMA (....).

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* HONG, S. H. and SHUELY, W. J. U.S. Army Chemical Reaserch, Development, and Engineering Center, Aberdeen Proving Ground, MD, Private Communication.

[9] duPont de Nemours, E. I. and Company. *Solvent Formulating Maps for Elvacite Acrylic Resins.* Bulletin PA-12-174, A-88130, Wilmington, DE, 1974.

Both the iBMA homopolymer and the iBMA/tBAEMA copolymer are soluble in most alcohols, which typically have high hydrogen bond indices and intermediate solubility parameters (e.g., n-propanol, with a solubility parameter of 11.9 and a hydrogen bonding index of 18.7). However, only the copolymer is soluble in ethanol, which has the same hydrogen bond index but a higher solubility parameter (12.7). Methanol, on the other hand, which has the same hydrogen bonding ability but an even higher solubility parameter of 14.5, is not a solvent for either the copolymer or the poly(iBMA). Even though the alcohols have similar structure, apparently the increasing cohesion as the size of the n-alkyl groups decreases, changes the solubility properties significantly.

Calculation of Solubility Parameters

Since polymers usually cannot be volatilized without decomposing, solubility parameters cannot generally be calculated from heats of vaporization. Group contribution methods have been developed to calculate solubility parameters of polymers and have worked quite well. Using the values of Hoy,[10] the solubility parameters of the two homopolymers poly(iBMA) and poly(tBAEMA) can be calculated by the following equation

$$\delta = d \, \Sigma \, G \, / \, M \qquad (6)$$

where d is the density of the polymer, G is the group molar contribution for each functional group in the polymer repeat unit, and M is the molecular weight of the repeat unit.

Figure 10 shows the repeat units of the copolymer and the group molar contributions for each functional group. Assuming that the densities of the two homopolymers and the copolymer are the same (estimated to be 1.1 g/mL), the calculated solubility parameters for the polymers are as follows:

poly(iBMA): 8.9

poly(tBAEMA): 9.2

poly(iBMA-co-tBAEMA), 75/25: 9.0

As is apparent from Figure 9 the copolymer has a slightly larger solubility parameter than the isobutylmethacrylate polymer, probably due to a higher solubility parameter for the tBAEMA component. This is confirmed by the above calculations. These calculations are probably not too accurate, but the general trends can be seen. Indeed, if the tBAEMA homopolymer and the copolymer have higher densities than the iBMA polymer, then the differences between the calculated solubility parameters of the iBMA homopolymer and the copolymer would be even greater.

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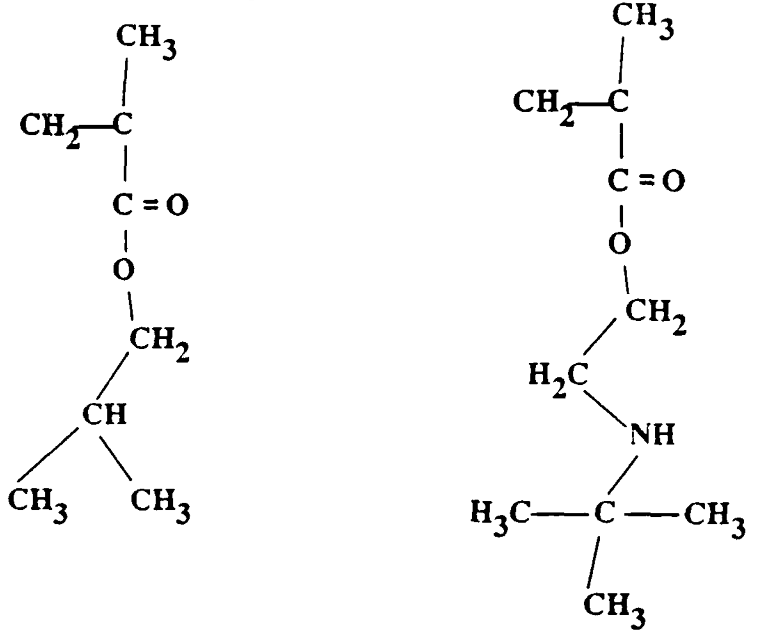
[10] HOY, K. L. J. Paint Technology, V. 42, 1970, p. 76.


iBMA, M = 142.20 g/mol

tBAEMA, M = 185.27 g/mol

iBMA	Group Contributions, G	tBAEMA
3	$-CH_3$ (147.3)	4
2	$-CH_2-$ (131.5)	3
1	>C< (32.03)	2
1	-COO- (326.58)	1
1	-CH< (180.03)	0
0	-NH- (180.03)	1
1149.50		1554.37

Figure 10. Repeat unit structures for copolymer and group molar contributions to solubility parameter.

CONCLUSIONS

The solubility map for poly(iBMA-co-tBAEMA) of composition 77:23 has been determined for the coordinates of Hildebrand solubility parameter and hydrogen bond index. In addition, portions of the solubility regions have been determined in the Hansen solubility parameter system, as well as in the Teas fractional cohesion parameter system. The range of solvents for this copolymer is significantly greater than that of the homopolymer iBMA which is the co-monomer in greater abundance. The amino group in the tBAEMA unit apparently enhances the solubility of the copolymer in solvents of high solubility parameter and hydrogen bonding ability in which a simple alkylmethacrylate, such as poly(iBMA), is insoluble.

Solubility maps, or phase diagrams, can be used successfully to determine areas of solubility for polymers in selected solvents and solvent mixtures. The determination of an *exact* borderline for solubility/insolubility is not feasible using this method and, obviously, any solvent chosen for solution studies based on location on a solubility map should be checked for solubility individually. A general solubility region, however, can be constructed to serve as a guideline for solvent selection, and this should be particularly helpful in the selection of cosolvents in which one or more of the solvents individually is not a solvent. The method used in this study to detect solubility of a polymer solution (visual inspection) is only an indication of macroscopic solubility and is not necessarily an indication of solubility on the molecular level; i.e., polymer solutions can form microgel, which is transparent and would not be detected visually. A more sophisticated test for solubility (such as light transmission) would produce a more reliable test method.

A disadvantage of the solubility test method as applied in this study is the time-consuming, tedious nature of the work. On the other hand, the individual tasks are simple to do, and this makes this type of study an excellent choice for automation/robotics. The processes of weighing out a powder, titrating small volumes of liquid, and using some type of sensor to test for solubility are all tasks well suited for a laboratory robotics system. Possibilities for solubility sensors include machine vision, measuring light transmission to determine transparency, and sampling the solution to monitor concentration.

Future work in our laboratory may be directed toward automating this procedure.

ACKNOWLEDGEMENTS

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The author also wishes to thank Mr. Andrew Sullivan for assistance in polymer solution preparation.

APPENDIX A. SOLUBILITY PARAMETERS FOR COMMON SOLVENTS (FROM REFERENCE 3)

	SP*	HBI*	DM*	δ_d^{**}	δ_p^{**}	δ_h^{**}	δ_t^{**}	$100f_d^{\#}$	$100f_p^{\#}$	$100f_h^{\#}$
Acetone	10.0	9.7	2.9	7.6	5.1	3.4	9.8	47.0	32.0	21.0
Acetonitrile	11.9	6.3	3.9	7.5	8.8	3.0	11.9	39.0	45.0	16.0
Benzene	9.2	0.0	0.0	9.0	0.0	1.0	9.1	78.0	8.0	14.0
n-Butanol	11.4	18.7	1.7	7.8	2.8	7.7	11.3	43.0	15.0	42.0
2-Butanone (MEK)	9.3	7.7	2.7	7.8	4.4	2.5	9.3	53.0	30.0	17.0
2-Butoxyethanol	8.9	13.0	1.6	7.8	2.5	6.0	10.2	46.0	18.0	36.0
Chloroform	9.3	1.5	1.2	8.7	1.5	2.8	9.3	67.0	12.0	21.0
Cyclohexane	8.2	0.0	0.0	8.2	0.0	0.1	8.2	94.0	2.0	4.0
Cyclohexanone	9.7	11.7	2.7	8.7	3.1	2.5	9.6	55.0	28.0	17.0
1,2-Dichloroethane	9.8	1.4	1.1	9.3	3.6	2.0	10.2	67.0	19.0	14.0
Dichloromethane	9.7	1.5	1.5	8.9	3.1	3.0	9.9	59.0	21.0	20.0
Dimethylacetamide	10.8	12.3	3.8	8.2	5.6	5.0	11.1			
Dimethylformamide	12.1	11.7	3.8	8.5	6.7	5.5	12.1	41.0	32.0	27.0
Dimethylsulfoxide	12.9	7.7	4.0	9.0	8.0	5.0	13.0	41.0	36.0	23.0
1,4-Dioxane	9.9	9.7	0.4	9.3	0.9	3.6	10.0	67.0	7.0	26.0
Ethanol	12.7	18.7	1.7	7.7	4.3	9.5	13.0	36.0	18.0	46.0
Ethyl acetate	9.1	8.4	1.8	7.7	2.6	3.5	8.8	51.0	18.0	31.0
Ethyl ether	7.4	13.0	1.2	7.1	1.4	2.5	7.6	64.0	13.0	23.0
Ethylene carbonate	14.7	4.9	4.9	9.5	10.6	2.5	14.4			
2-Ethylhexanol	9.5	18.7	1.7	7.8	1.6	5.8	9.9	50.0	9.0	41.0
n-Heptane	7.4	0.0	0.0	7.5	0.0	0.0	7.5	100.0	0.0	0.0
n-Hexane	7.3	0.0	0.0	7.3	0.0	0.0	7.3	100.0	0.0	0.0
Isopropyl ether	6.9	12.3	1.3							
Methanol	14.5	18.7	1.7	7.4	6.0	10.9	14.5	30.0	22.0	48.0
N-Methylpyrrolidone	11.3	10.0[1]	4.1	8.8	6.0	3.5	11.2	48.0	32.0	20.0
Nitrobenzene	9.8	2.8	4.3	9.8	4.2	2.0	10.8	52.0	36.0	12.0
Nitromethane	12.7	2.5	3.4	7.7	9.2	2.5	12.3	40.0	47.0	13.0

	SP^*	HBI^*	DM^*	δ_d^{**}	δ_p^{**}	δ_h^{**}	δ_t^{**}	$100f_d^{\#}$	$100f_p^{\#}$	$100f_h^{\#}$
n-Pentane	7.0	0.0	0.0	7.1	0.0	0.0	7.1	100.0	0.0	0.0
Phenol	14.5	18.7	1.7	8.8	2.9	7.3	11.8	46.0	15.0	39.0
n-Propanol	11.9	18.7	1.7	7.8	3.3	8.5	12.0	40.0	16.0	44.0
Pyridine	10.7	18.1	2.2	9.3	4.3	2.9	10.6	56.0	26.0	18.0
Tetrahydrofuran	9.1[2]	8.6[2]	1.8	8.2	2.8	3.9	9.5	55.0	19.0	26.0
Toluene	8.9	4.5	0.4	8.8	0.7	1.0	8.9	80.0	7.0	13.0

*ASTM parameters
SP - Hildebrand solubility parameter, $(cal/cm^3)^{1/2}$
HBI - hydrogen bonding index, Crowley units
DM - dipole moment, Debye units

**Hansen solubility parameters, $(cal/cm^3)^{1/2}$
δ_d - dipsersive component
δ_p - polar component
δ_h - hydrogen bonding component
δ_t - total solubility parameter

#Teas fractional cohesion parameters, unitless
f_d - dispersive component
f_p - polar component
f_h - hydrogen bonding component

[1] estimated (listed in Barton's Handbook as "moderate")

[2] from Ref. 9

APPENDIX B. EXPERIMENTALLY DETERMINED COPOLYMER SOLUBILITIES

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d##	δ_p##	δ_h##	100*f_d#	100*f_p#	100*f_h#
1	100	Isopropyl ether	I	6.9	12.3	1.3						
2	100	Pentane	I	7.0	0.0	0.0	14.5	0.0	0.0	100.0	0.0	0.0
3	100	Heptane	I	7.4	0.0	0.0	15.3	0.0	0.0	100.0	0.0	0.0
4	45 45 10	Isopropyl ether Heptane Cyclohexanone	B	7.4	6.7	0.9						
5	100	Ethyl ether	I	7.4	13.0	1.2	14.5	2.9	5.1	64.0	13.0	23.0
6	70 30	Isopropyl ether Cyclohexanone	B	7.7	12.1	1.7						
7	67 33	Isopropyl ether Cyclohexanone	S	7.8	12.1	1.8						
8	35 35 30	Isopropyl ether Heptane Acetone	S	8.0	7.2	1.3						
9	100	Cyclohexane	B	8.2	0.0	0.0	16.8	0.0	0.2	94.0	2.0	4.0
10	30 30 40	Isopropyl ether Heptane Nitrobenzene	B	8.2	4.8	2.1						

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d##	δ_p##	δ_h##	100*f_d#	100*f_p#	100*f_h#
11	48	Isopropyl ether	S	8.2	9.8	1.8						
	12	Heptane2										
	40	Acetone										
12	50	2-Ethylhexanol	B	8.2	15.5	1.5						
	50	Isopropyl ether										
13	50	2-Ethylhexanol	B	8.2	15.5	1.5						
	50	Isopropyl ether										
14	30	Isopropyl ether	S	8.3	7.6	1.6						
	30	Heptane										
	40	Acetone										
15	28	Isopropyl ether	S	8.4	7.7	1.7						
	28	Heptane										
	45	Acetone										
16	50	Isopropyl ether	S	8.5	11.0	2.1						
	50	Acetone										
17	56	Toluene	S	8.6	2.5	0.2	17.5	0.8	1.2	86.2	4.8	9.0
	44	Cyclohexane										
18	25	Isopropyl ether	S	8.6	7.9	1.8						
	25	Heptane										
	50	Acetone										
19	45	Isopropyl ether	S	8.6	10.9	2.2						
	55	Acetone										
20	100	Toluene	S	8.9	4.5	0.4	18.0	1.4	2.0	80.0	7.0	13.0

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d ##	δ_p ##	δ_h ##	100*f_d #	100*f_p #	100*f_h #
21	100	2-Butoxyethanol	S	8.9	13.0	1.6	16.0	5.1	12.3	46.0	18.0	36.0
22	80 20	2-Ethylhexanol Isopropyl ether	B	9.0	17.4	1.6						
23	100	Tetrahydrofuran	S	9.1	8.4	1.8	15.8	5.3	7.2	51.0	18.0	31.0
24	100	Benzene	S	9.2	0.0	0.0	18.4	0.0	2.0	78.0	8.0	14.0
25	100	Chloroform	S	9.3	1.5	1.2	17.8	3.1	5.7	67.0	12.0	21.0
26	100	2-Butanone	S	9.3	7.7	2.7	16.0	9.0	5.1	53.0	30.0	17.0
27	100	2-Ethylhexanol	S	9.5	18.7	1.7	16.0	3.3	11.9	50.0	9.0	41.0
28	70 30	Cyclohexanone 2-Ethylhexanol	S	9.6	13.8	2.4	17.3	5.4	7.1	53.5	22.3	24.2
29	60 40	Cyclohexanone 2-Ethylhexanol	S	9.6	14.5	2.3	17.1	5.1	7.8	53.0	20.4	26.6
30	50 50	Cyclohexanone 2-Ethylhexanol	S	9.6	15.2	2.2	16.9	4.8	8.5	52.5	18.5	29.0
31	100	Dichloromethane	S	9.7	1.5	1.5	18.2	6.3	6.1	59.0	21.0	20.0
32	45 55	Acetone 2-Ethylhexanol	S	9.7	14.7	2.2	15.8	6.5	9.7	48.7	19.3	32.0
33	35 65	Acetone 2-Ethylhexanol	S	9.7	15.6	2.1	15.8	5.8	10.2	49.0	17.0	34.0

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	$\delta_d^{\#\#}$	$\delta_p^{\#\#}$	$\delta_h^{\#\#}$	$100{*}f_d^{\#}$	$100{*}f_p^{\#}$	$100{*}f_h^{\#}$
34	100	1,2-Dichloroethane	S	9.8	1.4	1.1	19.0	7.4	4.1	67.0	19.0	14.0
35	55 45	Acetone 2-Ethylhexanol	S	9.8	13.8	2.4	15.7	7.2	9.2	48.4	21.7	30.0
36	50 50	Acetone 2-Ethylhexanol	S	9.8	14.2	2.3	15.8	6.9	9.5	48.5	20.5	31.0
37	100	Dioxane	S	9.9	9.7	0.4	19.0	1.8	7.4	67.0	7.0	26.0
38	100	Acetone	S	10.0	9.7	2.9	15.5	10.4	7.0	47.0	32.0	21.0
39	80 20	Dichloromethane Acetonitrile	S	10.1	2.5	2.0	17.6	8.6	6.1	55.0	25.8	19.2
40	50 50	Toluene Acetonitrile	S	10.4	5.4	2.2	16.7	9.7	4.0	59.5	26.0	14.5
41	60 40	Dichloromethane Acetonitrile	S	10.6	3.4	2.5	17.0	11.0	6.1	51.0	30.6	18.4
42	50 50	Methanol Isopropyl ether	S	10.7	15.5	1.5						
43	100	Pyridine	S	10.7	18.1	2.2	19.0	8.8	5.9	56.0	26.0	18.0
44	100	Dimethylacetamide	S	10.8	12.3	3.8	16.8	11.5	10.2			
45	46 54	Acetonitrile Acetone	S	10.9	8.1	3.4	15.4	13.9	6.6	43.3	37.9	18.7

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d ##	δ_p ##	δ_h ##	100*f_d #	100*f_p #	100*f_h #
46	40 60	Dichloromethane Acetonitrile	S	11.0	4.4	2.9	16.5	13.3	6.1	47.0	35.4	17.6
47	53 47	Acetonitrile Acetone	B	11.0	7.9	3.4	15.4	14.5	6.5	42.7	38.9	18.3
48	75 25	Acetonitrile Toluene	B	11.1	5.9	3.0	16.0	13.9	5.1	49.2	35.5	15.3
49	64 36	Acetonitrile Acetone	I	11.2	7.5	3.5	15.4	15.3	6.4	41.9	40.3	17.8
50	65 35	Nitromethane Toluene	S	11.4	3.2	2.4	16.6	12.7	4.0	54.0	33.0	13.0
51	83 17	Acetonitrile Toluene	I	11.4	6.0	3.3	15.8	15.2	5.4	46.0	38.5	15.5
52	100	n-Butanol	S	11.4	18.7	1.7	16.0	5.7	15.8	43.0	15.0	42.0
53	68 32	Nitromethane Toluene	S	11.5	3.1	2.4	16.5	13.2	4.1	52.8	34.2	13.0
54	80 20	Acetonitrile Acetone	I	11.5	7.0	3.7	15.3	16.5	6.3	40.6	42.4	17.0
55	71 29	Nitromethane Toluene	B	11.6	3.1	2.5	16.4	13.7	4.2	51.7	35.3	13.0
56	100	Acetonitrile	I	11.9	6.3	3.9	15.3	18.0	6.1	39.0	45.0	16.0

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d##	δ_p##	δ_h##	$100 \cdot f_d$#	$100 \cdot f_p$#	$100 \cdot f_h$#
57	90 10	Acetonitrile n-Propanol	I	11.9	7.5	3.7	15.4	16.9	7.2	39.1	42.1	18.8
58	72 28	Acetonitrile n-Propanol	S	11.9	9.8	3.3	15.5	14.9	9.3	39.3	36.9	23.8
59	100	n-Propanol	S	11.9	18.7	1.7	16.0	6.8	17.4	40.0	16.0	44.0
60	100	Dimethylformamide	S	12.1	11.7	3.8	17.4	13.7	11.3	41.0	32.0	27.0
61	50 50	Methanol Cyclohexanone	S	12.1	15.2	2.2	16.5	9.3	13.7	42.5	25.0	32.5
62	80 20	Acetonitrile Methanol	B	12.4	8.8	3.5	15.3	16.9	9.3	37.2	40.4	22.4
63	34 33 33	Ethylene Carbonate Ethanol Cyclohexanone	S	12.4	11.7	3.1	17.7	12.4	9.8			
64	32 24 44	Ethylene Carbonate Methanol Acetone	S	12.6	10.3	3.3	16.7	14.5	10.0			
65	60 40	Methanol Cyclohexanone	S	12.6	15.9	2.1	16.2	9.9	15.4	40.0	24.4	35.6
66	100	Nitromethane	I	12.7	2.5	3.4	15.8	18.8	5.1	40.0	47.0	13.0
67	8 92	Acetone Dimethylsulfoxide	I	12.7	7.9	3.9	18.2	15.9	9.9	41.5	35.7	22.8

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d##	δ_p##	δ_h##	100*f_d#	100*f_p#	100*f_h#
68	100	Ethanol	S	12.7	18.7	1.7	15.8	8.8	19.4	36.0	18.0	46.0
69	64 36	Acetonitrile Methanol	S	12.8	10.8	3.1	15.2	15.9	11.9	35.8	36.7	27.5
70	80 6 14	Ethanol Dichloroethane Phenol	S	12.8	17.7	1.7	16.3	8.3	17.9	39.3	17.6	43.1
71	100	Dimethylsulfoxide	I	12.9	7.7	4.0	18.4	16.4	10.2	41.0	36.0	23.0
72	70 30	Methanol Cyclohexanone	S	13.1	16.6	2.0	15.9	10.5	17.1	37.5	23.8	38.7
73	40 30 30	Ethylene Carbonate Acetone Methanol	I	13.2	10.5	3.3	16.9	15.5	10.8			
74	50 50	Methanol Acetonitrile	S	13.2	12.5	2.8	15.2	15.2	14.2	34.5	33.5	32.0
75	46 45 9	Ethylene Carbonate Ethanol Cyclohexanone	I	13.3	11.7	3.3	17.6	14.5	11.5			
76	75 25	Methanol Dichloroethane	S	13.3	14.4	1.6	16.1	11.1	17.7	39.3	21.3	39.5
77	34 66	Ethylene Carbonate Ethanol	B	13.4	14.0	2.8	17.0	13.2	14.6			

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d##	δ_p##	δ_h##	100*f_d#	100*f_p#	100*f_h#
78	36	n-Propanol	I	13.5	11.5	3.4	17.8	14.9	11.2			
	12	Ethanol										
	53	Ethylene Carbonate										
79	58	Ethanol	I	13.5	12.9	3.0	17.3	14.2	13.4			
	42	Ethylene Carbonate										
80	60	Ethylene carbonate	I	13.6	10.4	3.6	18.0	15.7	10.1			
	40	n-Propanol										
81	45	Ethylene Carbonate	I	13.6	12.5	3.1	17.4	14.6	13.0			
	55	Ethanol										
82	45	Methanol	I	13.6	12.7	3.0	16.9	14.6	15.6	36.1	29.7	34.3
	55	Dimethylsulfoxide										
83	65	Methanol	S	13.6	14.4	2.5	15.2	14.3	16.6	33.1	30.0	36.8
	35	Acetonitrile										
84	80	Methanol	S	13.6	15.2	1.6	15.9	11.3	18.7	37.4	21.4	41.2
	20	Dichloroethane										
85	49	Ethylene carbonate	I	13.7	11.9	3.3	17.6	15.1	12.4			
	51	Ethanol										
86	50	Methanol	I	13.7	13.2	2.9	16.8	14.4	16.3	35.5	29.0	35.5
	50	Dimethylsulfoxide										
87	70	Methanol	S	13.7	15.0	2.4	15.2	14.0	17.4	32.7	28.9	38.4
	30	Acetonitrile										

Solvent Mixtures (vol. %)			Sol.*	SP**	HBI**	DM**	δ_d ##	δ_p ##	δ_h ##	100*f_d #	100*f_p #	100*f_h #
88	55	Methanol	I	13.8	13.7	2.7	16.6	14.1	16.9	34.9	28.3	36.7
	45	Dimethylsulfoxide										
89	85	Methanol	S	13.8	16.1	1.6	15.7	11.6	19.6	35.6	21.6	42.9
	15	Dichloroethane										
90	40	Ethanol	B	13.8	18.7	1.7	15.4	10.9	21.1	32.4	20.4	47.2
	60	Methanol										
91	60	Methanol	I	13.9	14.3	2.6	16.4	13.9	17.5	34.4	27.6	38.0
	40	Dimethylsulfoxide										
92	75	Methanol	S	13.9	15.6	2.2	15.2	13.7	18.2	32.3	27.7	40.0
	25	Acetonitrile										
93	80	Methanol	B	14.0	16.2	2.1	15.1	13.4	19.1	31.8	26.6	41.6
	20	Acetonitrile										
94	74	Ethylene carbonate	I	14.2	8.5	4.1	18.5	18.3	8.8			
	26	Ethanol										
95	100	Methanol	I	14.5	18.7	1.7	15.1	12.3	22.3	30.0	22.0	48.0
96	100	Ethylene Carbonate	I	14.7	4.9	4.9	19.4	21.7	5.1			

*Sol. - Experimentally determined solubilities
S - Soluble
B - Borderline
I - Insoluble

Solvent Mixtures (vol. %)	Sol.*	SP**	HBI**	DM**	δ_d##	δ_p##	δ_h##	100*f_d#	100*f_p#	100*f_h#

**ASTM parameters
SP - Hildebrand solubility parameter, $(cal/cm^3)^{1/2}$
HBI - hydrogen bonding index, Crowley units
DM - dipole moment, Debye units

##Hansen solubility parameters, $(MPa)^{1/2}$
δ_d - dipsersive component
δ_p - polar component
δ_h - hydrogen bonding component
δ_t - total solubility parameter

#Teas fractional cohesion parameters, unitless
f_d - dispersive component
f_p - polar component
f_h - hydrogen bonding component

[1] estimated (listed in Barton's Handbook as "moderate")

[2] from Ref. 9

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1 Committee on Marine Structures, Marine Board, National Research Council,
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Director, U.S. Army Materials Technology Laboratory, Watertown, MA 02172-0001
2 ATTN: SLCMT-TML
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U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
SOLUBILITY CHARACTERISTICS OF A
METHACRYLATE COPOLYMER -
Marie K. Potts

Technical Report MTL TR 90-28, May 1990, 28 pp-
illus, D/A Project: FI8BF8J2FIAW,
AMCMS Code: 61110271A0011

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Key Words

Solubility
Copolymers
Methacrylates

Solubility maps in several solubility coordinate systems have been constructed to determine regions of solubility for a methacrylate copolymer. The general methodology followed was that of ASTM D 3132 Test Method for Solubility Range of Resins and Polymers. Over 90 individual solvents and solvent mixtures were tested as solvents for the copolymer in the concentration range 5% - 7.5% w/v. A database on the solubility characteristics of common solvents was created to store and utilize the information. Solvent maps were formulated using the solubility parameter-hydrogen bond index-dipole moment system, the Hansen solubility parameter system, and the Teas fractional cohesion parameter system. Each system is primarily empirical in nature, but has varying degrees of theoretical justification.

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