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Solvents Considered at AIC Annual Meeting in Dallas

JOHN BURKE, MARK ORMSBY, AND DAVID ERHARDT

A Teas Refresher

Solvents are ubiquitous. Not a day goes by when we don't rely on one solvent or another to accomplish some essential task. And yet, who among us hasn't tried in vain to remove one substance from another, guided by rules of thumb such as, "like dissolves like" or vague concepts of solvent "strength." While this approach may often succeed, it also might be risky if, for example, we needed to dissolve one material selectively while leaving other materials completely unaffected. Or, it would be clearly inefficient if, at the same time, we were also trying to control evaporation rates, solution viscosity, material costs, or environmental and health effects.

The selection of a solvent or solvent mixture in the face of complex criteria moves beyond trial and error and, by necessity, must rely on a system that can organize and predict solubility behavior. While this selection could be accomplished empirically by simply testing the effects of specific solvents on specific materials, a universal system that could encompass solubility behavior in general would be immensely useful. While understanding such a system may seem dauntingly complex, the practical application of solubility theory is actually quite straightforward. In fact, many solubility interactions can be pre-

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From the AIC President:

SCMRE Success Close At Hand!

JERRY PODANY

(**ad·vo·cate** n one that defends or maintains a cause)

Outside of the profession, the image most people have of a conservator—if they have one at all—does not include a demonstrator chained to a sculpture to protest its removal from a historical site or an activist marching up and down a demonstration line bellowing objections to the lack of environmental controls at a local archive. It may not even include a strongly worded objection to an unfiltered window in a public museum. These images are reserved for the activists who have captured the public's eye in response to saving our natural resources, not our cultural ones. Our mission, the saving of material culture and all the associated values attached to that material is a clear one, and one, it would seem, that is beyond doubt or reproach. Who would argue, after all, with the preservation of historical artifacts or masterpieces of art? Very few, indeed. But the mission is also a well-kept but unintended secret in the larger context of society. The need for the support of our mission is broader and deeper than simply agreeing with the principle. Conservation, to continue its efforts and to develop more effective approaches in the future, requires basic research, advancing methodologies, and continual practice to "preserve" its mission as well as the objects on which that mission is focused.

The need then for vigilant advocacy on

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dicted on a simple triangular graph.

Solubility Parameters

The solubility character of a material can be described by quantifying the amount of intermolecular attractions that are present in it. Since dissolving something requires the same energy as vaporizing it (in both cases its molecules are separated), we can derive a value called cohesive energy density from a material's heat of vaporization. In 1936, Joel H. Hildebrand, in his landmark book on the solubility of non-electrolytes, proposed the square root of the cohesive energy density as a numerical value indicating the solvency behavior of a specific solvent. It was not until the third edition in 1950 that the term "solubility parameter" was proposed for this value. In looking over a table of Hildebrand solubility parameters, it becomes apparent that by ranking solvents according to solubility parameter a solvent spectrum is obtained, with solvents occupying positions in proximity to other solvents of comparable "strength." For example, if acetone dissolves a particular material, it may likely be soluble in neighboring solvents, like diacetone alcohol or methyl ethyl ketone, since these solvents have similar internal energies. It may not be possible to achieve solutions in solvents further from acetone on the chart, such as ethyl alcohol or cyclohexane-liquids with very different internal energies. Theoretically, there will be a contiguous group of solvents that will dissolve a particular material, while the rest of the solvents in the spectrum will not. A material that cannot be dissolved at all, such as a thermosetting resin, might exhibit swelling behavior in precisely the same way.

Unfortunately, this is not entirely accurate. Theoretically, liquids with similar cohesive energy densities should have similar solubility characteristics, and yet actual behavior in this instance does not bear this out: sometimes a solvent directly in the middle of a solubility range will exhibit poor solubility characteristics. To overcome these observed inconsistencies, Burrell and others began including hydrogen bonding capacity as an additional component of solubility calculations. Even though the cohesive energy density, and therefore Hildebrand solubility parameter, of two materials may be similar, differences in hydrogen bonding could lead to differences in observed solubility behavior.

Three Component Parameters

Additional accuracy was achieved by further distinguishing hydrogen bonding from other kinds of polar interactions, leading to the use of separate values for dispersion forces, polarity, and hydrogen bonding. In 1966, Charles M. Hansen took this three component system further by

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relating all three values to the total Hildebrand value. This was done by first setting the dispersion parameter of a solvent to the Hildebrand value of the nonpolar molecule most closely resembling it (as n-butane would be to n-butyl alcohol), and subtracting this value from the total cohesive energy density. The remainder was then divided between polar and hydrogen bonding contributions, using trial and error experimentation on numerous solvents and polymers to find values that best reflected empirical evidence.

The Teas Graph

To resolve the awkwardness of presenting three component data, Jean P. Teas devised a simple triangular graph in 1968, on which polymer solubility areas could be drawn in their entirety. Because of its clarity and ease of use, the Teas graph found increasing application in problem solving, documentation, and analysis, and is an excellent vehicle for understanding complex solubility behavior.

In order to plot all three parameters on a single planar graph, Teas began with the imaginary assumption that all materials have the same Hildebrand value. Accordingly, solubility behavior would be determined, not by differences in Hildebrand value, but by the relative contributions of the three component forces. This allows the convenience of using percentages rather than unrelated sums.

Hansen derived his parameters from the Hildebrand value: when squares of all three Hansen parameters for a solvent are added together, their sum will be the square of the Hildebrand value for that solvent. Teas parameters, also called fractional parameters, are mathematically derived from Hansen values by calculating the relative amount that each Hansen parameter contributes to the whole.

The intersection of these three values can be easily located on a triangular graph. Overall, the solvents are grouped closer to the lower right apex than the others. This is because determining the dispersion component is the first calculation in assigning Hansen parameters, from which fractional parameters are derived. Unfortunately, this greatly overemphasizes the dispersion force relative to polar forces, especially hydrogen bonding interactions.

It can also be seen that increasing molecular weight within each class shifts the relative position of a solvent on the graph closer to the bottom right apex. This is because, as molecular weight increases, the polar part of the molecule that causes the specific character identifying it with its class, called the functional group, is increasingly "diluted" by progressively larger, nonpolar "aliphatic" molecular segments. This gives the molecule as a whole relatively more dispersion force and less of

the polar character specific to its class. This trend toward less polarity with increasing molecular weight within a class also accounts for the observation that lower molecular weight solvents are often "stronger" than higher molecular weight solvents of the same class, although determinations of solvent strength must really be made in terms of the solvents position relative to the solubility area of the solute.

The only class in which increasing molecular weight places the solvent further away from the lower right corner is the alkanes. This is because the intermolecular attractions between alkanes are due entirely to dispersion forces, and accordingly, Hansen parameter values for alkanes show zero polar contribution and zero hydrogen bonding contribution. Since fractional parameters are derived from Hansen parameters, one would expect all the alkanes to be placed together at the extreme right apex. Observed behavior indicates, however, that different alkanes do have different solubility characteristics, perhaps because of the tendency of larger dispersion forces to mimic slightly polar interactions. For this reason, Teas adjusted the locations of the alkanes to correspond to empirical evidence. Several other solvent locations were also shifted slightly to properly reflect observed solubility characteristics.

Visualizing Solubility

Using a Teas Graph, complex solubility behavior can be described. For example, the solubility of a material can be tested in a variety of solvents and the results color coded onto a Teas graph. This will result in a solubility window being defined, with successful solvents inside the region and non solvents outside. The boundaries of this solubility window can be more accurately defined by drawing a line between two solvents, one inside and the other outside the window, and testing various proportions to find the mixture that just produces solubility. If this procedure is repeated in several locations around the edge of the window, its boundaries may be accurately determined. Interestingly, some composite materials (such as rubber/resin pressure sensitive adhesives, or wax resin mixtures) can exhibit two or more separate solubility windows, more or less overlapping, that reflect the degree of compatibility and the concentration of the original components.

Note that not all the solvents within a solubility window are equal. Contrary to expectations, solvents at the center of a window may not always form the lowest viscosity solutions. And, if differences in evaporation should cause a solvent mixture to drift outside the solubility window, discontinuous films with poor adhesive or optical properties may result.

Solvent Mixtures

Teas graph is particularly useful for creating solvent mixtures for specific applications because the solubility parameters of a mixture can be simply calculated by averaging its components. Solvents can easily be blended to exhibit critical solubility behavior such as dissolving one material but not another. Determining the solubility parameters of a mixture can be done either by calculating from the fractional parameters of the individual solvents, or in the case of a binary mixture, by simply drawing a line between

Further Reading

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its two solvents and measuring their ratio. To calculate the solubility parameters from the individual components, the fractional parameters for each liquid are multiplied by the fraction that the liquid occupies in the blend, and the results for each parameter added together.

What is interesting about visualizing solvent blends on a Teas graph is the control with which effective solvent mixtures can be formulated. For example, two liquids that are non-solvents for a specific polymer can sometimes be blended in such a way that the mixture will act as a true solvent, as long as the line between the solvents passes through the solubility window. This phenomenon is also valuable when selective solvent action is required, such as in dissolving one material while leaving other materials unaffected, particularly if the solubilities of the materials involved are very similar. Another advantage of blending solvents is the ability to design lower toxicity mixtures with similar solubility characteristics, although it should be noted that the biological effects of such substitutions has not been adequately studied.

Solubility parameters in general, and the Teas graph in particular, are useful systems for describing complex solubility behavior. Although rooted in scientific theory, they are empirical systems with limitations in their applicability. It is important to remember, for example, that apparent insolubility does not mean leaching or other subtle changes will not occur, and that differences in evaporation rates and exposure times may have other unwanted effects. It should also be pointed out that the greater the distance between solvents the less accurate will be the results, and that the presence or water or other electrolytes moves behavior out of reach of normal solubility parameter theory. When faced with day-to-day solubility problems, however, the Teas graph—even an internally visualized one—can be a welcome friend.

—John Burke, Head of Conservation, Oakland Museum of California, Oakland, CA. 94647; (510) 238-2258; jb@museumca.org

Solvent Solver Program

A new computer program simplifies the task of finding a mixture of solvents to use as a solvent substitute. The Solvent Solver program makes calculations based on Teas fractional solubility parameters for a variety of solvents used in conservation, including those most commonly used based on the AIC Health and Safety Committee solvent survey (conducted at the 1999 AIC annual meeting) .

The program was written by Mark Ormsby of the National Archives and Records Administration based on ideas from conservators Elissa O'Loughlin (Walters Art Gallery) and Alan Puglia (Harvard Libraries). It is similar to the Teas Time program written by Walter Henry (available at Conservation Online) and also includes some additional features and information.

Solvent Solver allows the user to select up to three solvents to mix together to attempt to replace a target solvent. This solvent substitution may be desirable for a variety of reasons, including the health of the object being treated as well as the conservator doing the treatment. The program lists threshold limit values (TLV) and vapor hazard ratios (VHR) that help in selecting safer solvents.

The program attempts to find a mixture of the solvents that behave similarly to the target solvent based on Teas' solubility parameter model. If the program finds a solution it displays the concentrations needed as well as the TLV and VHR for the mixture.

There is also a more advanced version of the program that provides more flexibility for developing safer mixtures. By making a slightly less accurate match to the target solvent the program can minimize the concentration of the most hazardous of the substitute solvents.

The program is free and will soon be available on the Health and Safety section of the AIC web site. It is also available on floppy disk; please contact Mark Ormsby. The basic version of Solvent Solver requires Microsoft Windows 95 or higher, and the more advanced version requires Microsoft Excel 97. A Macintosh version is not available.

For more information please contact Mark Ormsby at [redacted]

—Mark Ormsby, Physicist, National Archives and Records Administration 8601 Adelphi Road, College Park, MD., 20740-6001

Solvent Grade: Does it Matter?

Solvents come in a number of prices and grades, defined by specific levels of purities listed in the catalogue description. Purity may be quite different for different grades depending on the intended use. For example, solvents used in metals analysis should contain no metals themselves. Food grade solvents should contain no toxic impurities. Unfortunately, there is no "conservation" grade. What grades are suitable for use in conservation?

Several factors should be considered in choosing a solvent grade. The

level of impurities should not affect the solvent action, the amount of residue left behind after evaporation should be low, and the potential health risks should be minimized. Impurities in laboratory grade solvents are usually on the level of parts per thousand or less, and do not affect the solvent properties. You could not distinguish different grades based on their solvent action. Acetone will behave like acetone. Specially dried solvent grades contain only minute traces of water. It generally is not worth it to buy extra dry solvents, because they will quickly pick up water from the air, or simply extract water from objects they are applied to. For example, grain alcohol (95% ethanol and 5% water) would be in equilibrium with air at a relative humidity of about 5%, and will extract water from a hygroscopic material equilibrated to normal RH levels. Dry solvents do not remain that way for long. Amounts of residue in laboratory grade solvents also are low, often less than one part per million. Such levels should be suitable for conservation use. There should always be a grade available that lists the amount of residue, usually at little cost differential. If possible, choose a grade for which the amount of residue is specified.

Some solvents have "impurities" that are purposely added—referred to as denaturants, stabilizers, or preservatives. Compounds such as methanol and kerosene are added to ethanol to make it undrinkable (and avoid the taxes). These additives don't change the solvent properties, but some are less volatile than others. If possible, choose alcohol with a pure volatile denaturant such as methyl isobutyl ketone, rather than a mixture such as kerosene that may contain nonvolatile components. Or, simply use grain alcohol. It may cost a little more, but it contains no denaturants and has multiple uses. The same consideration applies to ethyl ether. Ethers tend to form peroxides, so preservatives are usually added. A preservative such as ethyl alcohol is preferable to the relatively nonvolatile BHT.

Solvents are chosen in large part based on health considerations. Hexane is a central nervous system toxin, so similar solvents such as heptane and isooctane should be substituted. Because hexane is a potential contaminant in a similar compound like heptane, choose a heptane grade in which the amount of hexane impurity is specified. Benzene, a carcinogen, is a typical impurity in toluene, so choose a grade in which the amount of benzene is specified. Similarly, ethanol with a low level of methanol should be used.

The discussion so far has been limited to pure solvents containing one chemical compound. What about mixtures such as petroleum ether? Hydrocarbon mixtures such as petroleum ether or ligroine are generally specified by their boiling point range. Hexane boils at 69°C, and is usually found in substantial amounts in mixtures whose boiling range includes this temperature. Choose a grade whose boiling range starts above 70°C, or better yet just substitute heptane. Similarly, aromatic mixtures should have a minimum boiling point above 80°C, the boiling point of benzene, or mixtures of heptane and toluene with an appropriate polarity can be substituted.

The last "grade" of solvents to be considered is commercial solvents such as those from a hardware store. These generally do not have specifications on the label. The problem is not that a can labeled acetone will not act like acetone, because it takes a large amount of impurities to significantly alter the solvent properties. A greater potential problem is residues. If the amount of residue is not specified, a reasonable amount can be reduced in volume, and the last bit allowed to evaporate on a surface such a mirror where any residue would be easy to see. Again, it is preferable to avoid buying toluene or mineral spirits that do not list the amount of benzene or hexane.

In general, most laboratory grade solvents are suitable for conservation use. If possible, choose grades that list the amounts of residue or hazardous contaminants that might be expected in a specific solvent, and look for the presence of nonvolatile preservatives or additives. Avoid solvents known to cause health problems and mixtures that might contain them, and use less hazardous substitutes. The Solvent Solver program will be helpful in making such choices.

—David Erhardt, Organic Chemist, SCMRE, Smithsonian Institution, Washington, D.C., 20560

EDITOR'S NOTE: *These papers were originally presented as a tripartite lecture at the AIC Annual Meeting in Dallas and were sponsored by the Health & Safety Committee.*

People

Doris A. Hamburg has been appointed as Director of Preservation Programs at the National Archives and Records Administration (NARA). Doris was previously at the Library of Congress where she was head of preventative conservation for six years.

Ellen Pearlstein has taken an 18-month leave from the Brooklyn Museum of Art to work on the research and publication of a textbook on preventive conservation, funded in part by a Kress Publication Fellowship. She can be reached at [redacted] or [redacted]

Theresa Voellinger has taken the position of assistant paper conservator at the National Park Service Department of Conservation at Harpers Ferry Center. Theresa is a graduate of the Buffalo art conservation program and has just completed a two-year, post-graduate, Mellon fellowship at the Balboa Art Conservation Center in San Diego.

Frederick Wallace has just been promoted to chief conservator at the Cincinnati Art Museum.

If you have a listing that you would like to contribute to the People column in the November issue of *AIC News*, email it to Nora McElfish at [redacted] or send a short notice to AIC, Attn: Nora McElfish, 1717 K St., NW, Suite 200, Washington, DC 20006 before October 1, 2001.