

A Systematic Approach to the Behavior of Metal Soap-Oil Systems

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ABSTRACT

Application of the solubility parameter concept to micellar and crystalline metal soap dispersions in low-polarity solvents can give some order to effects of the solvent in these systems. The ASTM dropping point temperature has been shown to be a function of solubility parameter, and it is proposed that dropping points can be estimated when solubility parameters are known. A new method for estimating the solubility parameter of an oil, based on its interaction with soap, has also been suggested.

PROBLEM STATUS

This is an interim report; work on this problem is continuing.

AUTHORIZATION

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A SYSTEMATIC APPROACH TO THE BEHAVIOR OF METAL SOAP-OIL SYSTEMS

INTRODUCTION

Soap-oil systems exhibit a variety of phase equilibria and solubility characteristics which depend upon both the soap and the solvent. Even upon consideration of a particular soap species, the effect of changes in solvent character are understood only qualitatively. These changes are not simply related to the usual physical properties such as density, boiling point, viscosity, or dielectric constant. The inability to correlate solvent effects in soap-oil systems with suitable solvent characteristics has resulted in a large body of rather poorly organized information on soap-solvent systems. Moreover, estimating solvent effects on the solubility and phase behavior of soaps in untried oils has remained more art than science.

A substantial degree of order can be achieved if the effect of solvent in these systems is assessed by use of the Hildebrand solubility parameter concept (1). The solubility parameter is a measure of the intermolecular forces in a liquid. More specifically it is the square root of the energy of vaporization per cubic centimeter. Solubility parameters may be determined experimentally from heats of vaporization, internal pressures, surface tension, and solubility data. Estimates may also be made from the Hildebrand rule (which relates boiling point and heat of vaporization), from critical constants in equations of state, and from optical data (1). It may be instructive to review the correlations between soap-solvent behavior and the solubility parameter of the solvent which have been recently reported in order to illustrate the utility of the solubility parameter concept as applied to soap-oil systems. It is convenient to classify soap-oil systems into two types – micellar dispersions and crystalline dispersions – and to consider these in some detail before reporting new data on the correlation between the dropping point of lithium soap greases and the solubility parameter of the oil used in the grease formulation.

MICELLAR DISPERSIONS

Most oil-soluble soaps behave experimentally like extremely viscous liquids (2). This is well illustrated in Fig. 1 for the lithium dinonylnaphthalene sulfonate/dimethyl siloxane heptamer system. This system has a critical solution temperature of 47°C, above which soap and solvent are miscible in all proportions. This phase diagram is characteristic of a liquid-liquid pair showing limited miscibility. Soaps appearing to be liquidlike in their phase relations with oils are really mixtures of branched-chain isomers and homologs as exemplified by aliphatic carboxylates (3), arylstearates (4), and dinonylnaphthalene sulfonates (2). For most commercially important oil-soluble soaps – for example, the petroleum sulfonates – the critical solution temperature in ordinary oils lies well below the temperature of use, and soap and oil are infinitely miscible. The soap is of course present as a dispersion of nonaqueous micelles in which the ionic portions of the molecule are concentrated in a polar core screened from intimate contact with the oil by the hydrocarbon tails of the soap molecules which form the exterior of the micelle. Consequently, the solubility behavior of the dinonylnaphthalene sulfonates parallels that of the parent hydrocarbon (dinonylnaphthalene) with one exception. The sulfonates are completely miscible with nearly all solvents that are miscible with dinonylnaphthalene, but in addition, they dissolve completely in polar solvents whose functional groups interact strongly with the polar head of the soap molecule (5).

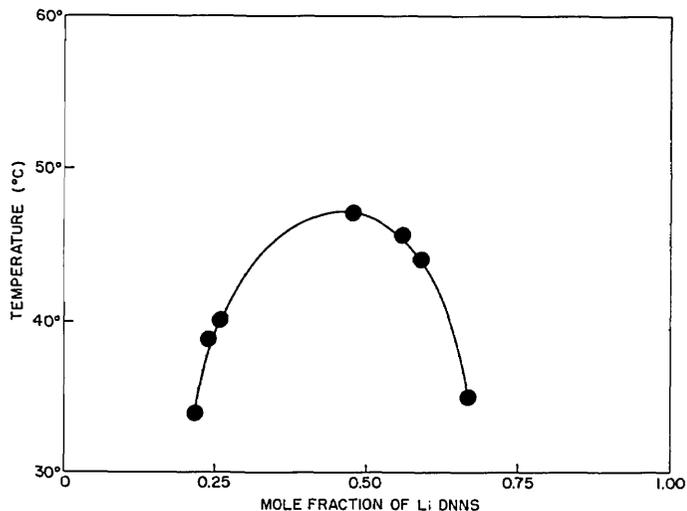


Fig. 1 - Mutual solubility of lithium dinonylnaphthalene sulfonate (LiDNNS) and dimethyl siloxane heptamer

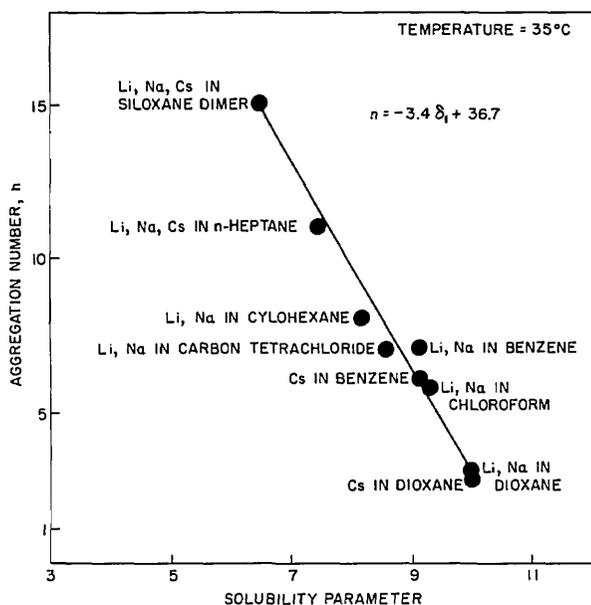


Fig. 2 - The dependence of the aggregation number of dinonylnaphthalene sulfonate micelles upon the solubility parameter of the solvent

The dependence of the aggregation number of soap micelles on the solubility parameter of the solvent is shown in Fig. 2. Micelles of sulfonates tend to be large in solvents having weak intermolecular forces (low solubility parameter) and small in solvents having stronger intermolecular forces (higher solubility parameters). This variation in micelle size has been explained on a semitheoretical basis (2); it is sufficient to suggest here that the soap micelle appears to adjust its size so that the overall solubility parameter of the micelle as viewed from the solvent approaches that of the solvent. In this way the excess free energy of mixing of micelle and solvent becomes nearly zero as the parameters approach a match. For solutions of the dinonylnaphthalene sulfonates in solvents having low intermolecular forces, the soap micelle presents a tightly packed outer surface (high aggregation number) consisting primarily of low-energy methyl groups. In solvents having

higher intermolecular forces, more of the hydrocarbon radical is exposed (low aggregation number) allowing a greater degree of interaction between the polar core and the solvent molecules.

CRYSTALLINE DISPERSIONS

Metal soaps having straight-chain nonpolar radicals are most likely to yield crystalline dispersions because their molecular geometry permits close packing of the molecules in the crystal state. This results in strong coulombic interactions between polar heads plus additional van der Waals interactions between the nonpolar tails. Straight-chain alkali carboxylates are examples of this type. Bondi (6) first suggested the utility of the solubility parameter concept as an index to the swelling behavior of soap crystals in solvents. Figure 3 shows the degree of swelling of sodium stearate as a function of solvent solubility parameter using Bondi's data. Similar curves are obtained when the sedimentation volume data of Mardles and Clarke (7) are plotted against solubility parameter (8). In addition, critical solution temperatures of zinc stearate (8,9) and lead stearate (8) show characteristic curves having minima when plotted against solubility parameter, as in Fig. 4. These effects are qualitatively similar to those observed for high polymer-solvent systems (10). The dissolution of crystalline soap in a nonpolar solvent first involves swelling of the crystal lattice by penetration of solvent molecules between nonpolar tails. As the critical solution temperature is approached, the combined effects of swelling and increased thermal loosening of the lattice overcome the lattice forces, and the swollen soap disintegrates or swells without limit. The maximum of Fig. 3 and the minimum of Fig. 4 might be interpreted as apparent solubility parameters of the concerned soap molecules if one continues to pursue the soap-polymer analogy. However, this interpretation may require modification as the body of data on soaps increases. It is of interest to note that solvents having solubility parameters much lower than 6.5 (silicones) or higher than 10.5 (lower homologs of nitroparaffins and nitriles) will not mix with the soaps even at the melting point of the pure soap.

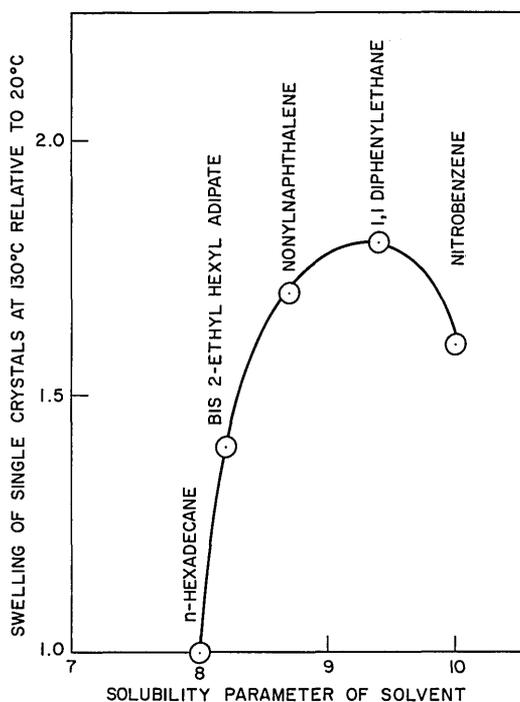


Fig. 3 - Relative swelling volume of sodium stearate versus solubility parameter

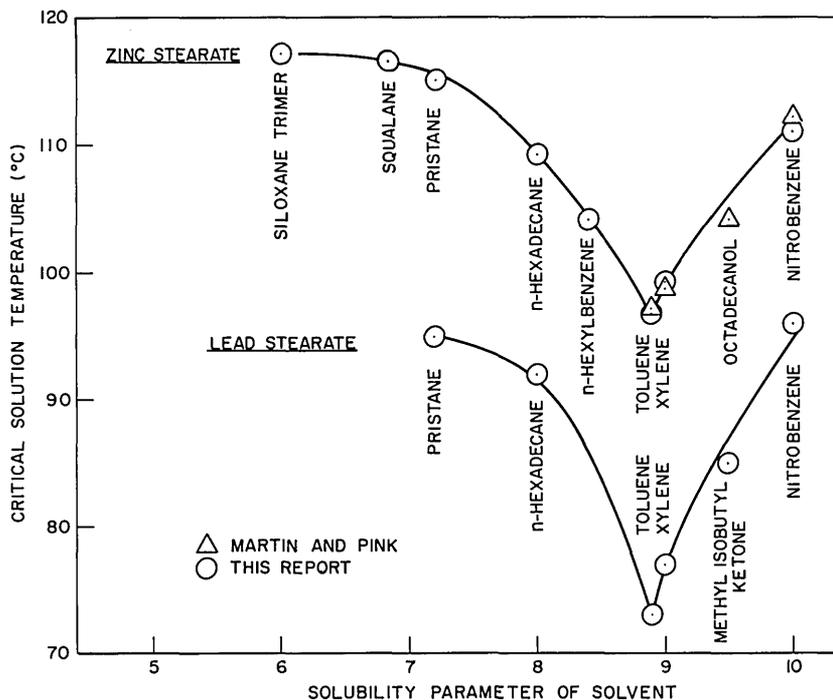


Fig. 4 - Critical solution temperatures of zinc and lead stearates versus solubility parameter

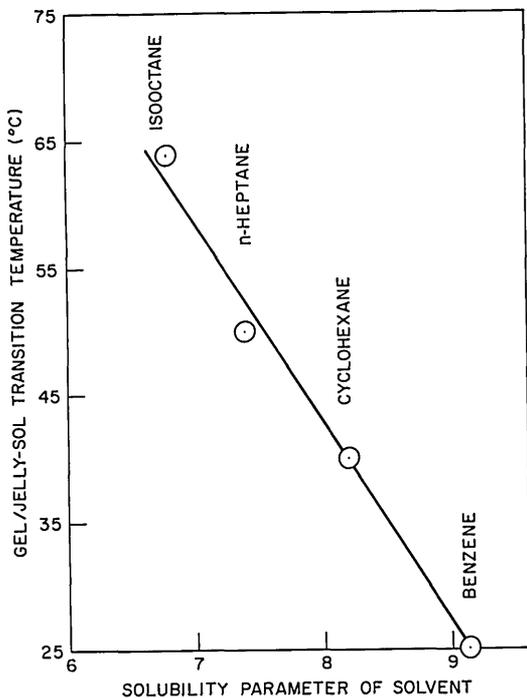


Fig. 5 - Gel/jelly-or-sol transition temperatures of aluminum dilaurate/hydrocarbon systems versus solubility parameter

The influence of solvent solubility parameter on soap swelling and critical solution temperatures suggests that there should be a general correlation between soap phase transitions and solubility parameter. Phase transitions are of some importance in grease formulation in that alterations in appearance and consistency frequently accompany them. Figure 5 plots the data of McBain et al. (11) showing the transition temperatures of anhydrous aluminum dilaurate gels into jellies or sols against the solvent solubility parameter.

It has been shown in the study of lithium greases that at least three major phase transitions in the solid state of the soap occur as the grease is heated (12). Lithium grease formulations retain their form to within several degrees of the final solid state phase transition between the waxy and liquid crystal phases. Since this last phase transition lies only a few degrees above the ASTM dropping point temperatures (12), the dropping point test was used to investigate the working range of greases made from a commercial lithium soap as solvent solubility parameter was varied.

EXPERIMENTAL PROCEDURE

Materials

The soap used in this investigation was a commercial lithium stearate of the purity commonly used in grease manufacture. It was composed of a mixture of lithium soaps, approximately 51% palmitate, 43% stearate, 4% oleate, and 2% myristate. Sodium soaps were present only in trace amounts. The soap was dried over phosphorus pentoxide in an evacuated desiccator.

The solvents used were percolated over Florisil, as necessary, to remove polar impurities and were dried over Linde molecular sieve material.

Technique

Sedimentation volume increases were determined using the technique described by Mardles and Clarke (7). One gram of the dried soap was placed in a glass tube of 1 cm I.D. and its volume measured after settling with the aid of vibration. Dried solvent (20 ml) was then added and the soap brought into suspension by shaking. The soap was allowed to settle under isothermal conditions at 110°C, and the sedimentation volume increase was determined after 24 hours.

Critical solution temperatures of zinc stearate-oil systems were determined by a method similar to that of Tughan and Pink (13). A 0.2-g portion of soap was added to 2 ml of solvent. The solutions were warmed at a rate of 1°C per minute when close to their critical solution temperatures (CST). CST's were reproducible to within 1°C. The apparatus consisted of a 5-ml test tube jacketed by an electrically heated 50-ml test tube vented to the atmosphere.

The lithium soap greases were prepared from the soap and those solvents having sufficiently high boiling points. One gram of the dried soap was mixed with 10 ml of the dried solvent in a test tube, which was then immersed in an oil bath preheated to 175°C. The temperature was further increased until solution occurred. The solution was chilled by pouring it onto a metal plate at room temperature.

The dropping points of these greases were determined by the ASTM standard test method D 566-42 (14).

RESULTS AND DISCUSSION

Grease Dropping Points and Critical Solution Temperature

Figure 6 shows the relation found between grease dropping point and solvent solubility parameter (δ) for systems of commercial lithium stearate and the indicated oils. The appearance of the dropping point curve strongly resembles the critical solution temperature curves of Fig. 4 for the zinc and lead stearates. However, the minimum for the lithium stearate dropping point curve is displaced 0.5 δ units, occurring at 9.4 rather than 8.9. The solution temperatures of 1 g/10 ml mixtures of lithium soap and oil are shown in Fig. 7. The minimum in this curve also lies close to that observed for the dropping point curve at $\delta = 9.4$. The displacement of the minimum from $\delta = 8.9$ to 9.4 may be an indication of the increased polarity of the polar group in lithium stearate over that of the zinc and lead stearates (8).

If the grease dropping point is taken as a measure of the final solid state phase transition of the lithium soap (12) (waxy to liquid crystal), then the data suggest that the

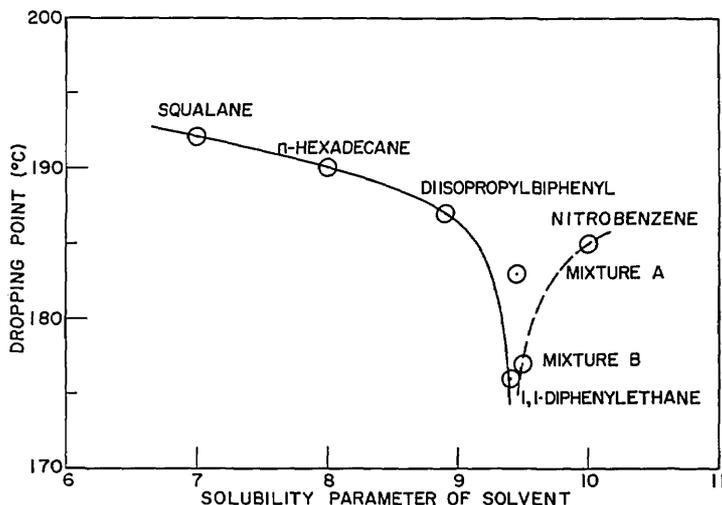


Fig. 6 - Lithium stearate grease dropping points versus solubility parameter; mixture A = diisopropylbiphenyl + nitrobenzene; mixture B = 1,1-diphenylethane + nitrobenzene

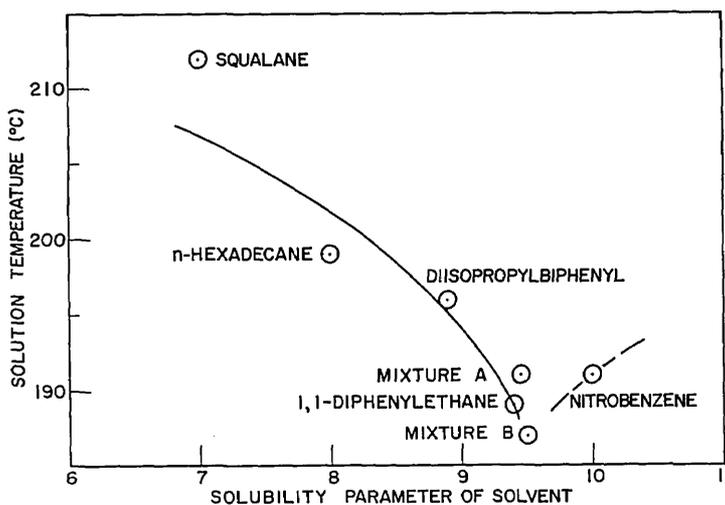


Fig. 7 - Lithium stearate solution temperatures versus solubility parameter; mixtures A and B as in Fig. 6

solvent exerts a parallel effect on both the CST and the last solid state transition. Phase transitions (waxy to liquid crystal) of lithium stearate-mineral oil systems have been shown to be a function of the molecular weight of the oil (15). Also, grease dropping points have been shown to be a function of oil viscosity within a given oil family (16). While it is not possible to assign definite δ values to the oils used in these experiments, the trends in the data appear to correspond to the changes in solubility parameter which are to be expected with increasing molecular weight (or oil viscosity) in a given oil type.

Commercial oils may be expected to have solubility parameters in the range 7 to 9. Paraffinic oils would tend to low values, while highly aromatic oils would tend to high values

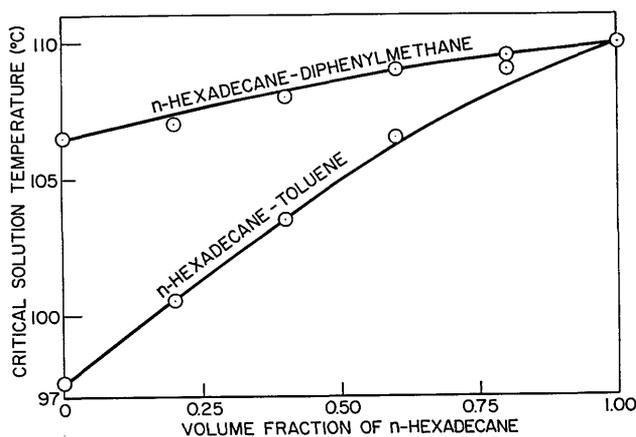


Fig. 8 - Critical solution temperatures of zinc stearate in n-hexadecane-toluene and n-hexadecane-diphenylmethane mixtures

in this range. The left leg of the grease dropping point curve is the section which is of interest in grease manufacture.

The prediction of grease dropping points requires (a) a calibration curve of dropping point versus the solubility parameter of known solvents and (b) a convenient method of estimating the solubility parameter of unknown commercial oils. In general, the methods of determining δ mentioned earlier apply to pure solvents and are not necessarily directly applicable to commercial oils which are mixtures of many species. Although the solubility parameter of a binary solvent system can be determined from a knowledge of the solubility parameter and volume fraction of the components (17), the difficulties involved in applying this calculation to commercial oils are obvious.

Estimation of Solvent δ From Zinc Stearate CST's

Figure 4 suggests that the CST's of zinc stearate-oil systems could be used to estimate the δ of an oil by assigning it a δ which will place it on the curve already established for known solvents with zinc stearate. Although other soaps might also be used, zinc stearate in particular offers the advantages of low temperatures of measurement and low water sensitivity. It is to be expected that nearly all commercial oils will have δ 's corresponding to the left leg of the curve. Such an empirically determined solubility parameter will serve as a useful guide in understanding and predicting various interactions between the oil and soaps.

Some insight into the effect of solvent mixtures on zinc stearate CST's is provided by studies of binary solvents of known composition. Figure 8 demonstrates this effect in two systems, n-hexadecane-toluene and n-hexadecane-diphenylmethane, as the volume fraction of n-hexadecane is varied. The lower curve results from a solvent pair chosen from the left leg of the CST- δ curve; the upper curve results from a solvent pair lying on opposite legs of the CST- δ curve.

These curves appear to indicate that the CST observed for solvent mixtures is approximately a volume fraction addition of the CST's of each component in the solvent mixture. For binary solvent mixtures made from components lying on one leg of the CST- δ curve, the CST's observed are close to those predicted for the solubility parameter of the mixed solvent. For a binary mixture of oil components taken from opposite legs of the zinc stearate CST- δ curve, no minimum CST was found corresponding to an average δ of

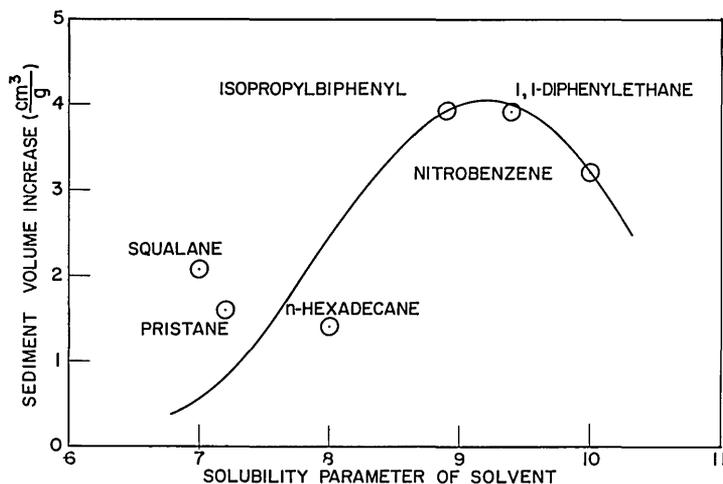


Fig. 9 - Sedimentation volume of lithium stearate versus solubility parameter

8.9 computed for a n-hexadecane volume fraction of 0.36. These observations suggest that the components of mixed solvents act independently in swelling the soap crystallite. Each component molecule seems to enter the soap lattice on the basis of its own δ and its statistical availability in the mixture.

Because of the difference in minima between the zinc stearate curve and the lithium stearate curve, lithium stearate solution temperatures should be used to estimate oil solubility parameter when oil components having parameter values greater than 8.9 are suspected in the oil mixture. It should be recognized, however, that the solubility parameter concept is most validly applied to systems composed of two components - that is, a pure solute plus a pure solvent. It cannot be expected to account for all the possible interactions between molecules of widely varying cohesive forces as when oil mixtures become very complex.

Estimation of Solvent δ From Sedimentation Volumes

The sedimentation volume data of Mardles and Clarke (7) when plotted against the solubility parameter of the oil (8) could provide another possible method to determine δ . Figure 9 shows the "calibration curve" obtained from the indicated oils of known δ . This method appears to be useful only when soap-oil density differences and oil viscosities are comparable (see squalane point). It could not be applied safely to rating commercial solvents of widely varying viscosities.

Application to Commercial Oils

As an example of the applicability of the solubility parameter concept to the study of grease properties. Table 1 lists some commercially available oils and compares the predicted and the experimental grease dropping points of the grease formations prepared from them. The oils chosen for test have solubility parameters between 7 and 9 and differ widely in chemical type and viscosity. In spite of these differences, the agreement between prediction and experiment is excellent. On the other hand, the correlation reported between grease dropping point and viscosity characteristics mentioned earlier failed when the oil type was varied (16). Since the solubility parameter concept allows an estimation of solvent effects in soap-oil systems, it now becomes possible to make a

Table 1
Prediction of Lithium Grease Dropping Points

Oil*	Zinc Stearate CST	Empirical Solub. Param.	Drop. Pt. (Predicted)	Drop. Pt. (Observed)
Navy pet. oil 1065	119°C	< 7	194°C	199°C
Paraffin oil 125/135	117°C	7.0	192°C	193°C
Light mineral oil	112°C	7.8	190°C	190°C
Medium mineral oil	113°C	7.6	191°C	189°C
Bis (2-ethylhexyl) sebacate	115°C	7.4	191°C	189°C
Navy oil 1065 + diphenylmethane 50 - 50 mixture	-	8.9†	186°C	184°C
Diphenylmethane	106°C	9.3	181°C	181°C

*Dried over molecular sieves.

†From lithium stearate solution temperature curve.

more significant assessment of any additional effect of such factors as oil viscosity, oil volatility, molecular weight, and rate of cooling on the ultimate characteristics of the grease.

The right legs of the dropping point and CST curves shown include solvents of rapidly increasing polarity which may or may not conform to the relation indicated by simple solubility parameter considerations, depending on their degree of interaction with soap polar groups. Water, and to a lesser extent the lower alcohols, would exhibit effects deviating widely from those predicted from their solubility parameters, or from the solubility parameter of mixed solvents in which they appear. It is well known that three-component systems - particularly those containing two polar additives - may exhibit unusual phase behavior (18). Nitroparaffins (2,5) and nitriles, however, appear to interact very little with soap polar groups, and their effects on grease dropping points are expected to lie closer to that projected for the solubility parameter of the mixed solvent when they appear in such systems.

SUMMARY

1. The solubility parameter has been used as an index of the effects of the solvent in soap-oil systems.
2. A correlation exists between solubility parameter and certain effects, such as swelling behavior, sedimentation volume, and critical solution temperature.
3. The ASTM dropping points of greases made from a commercial lithium stearate have been shown to be a function of the solubility parameter of the oil used in the grease formulation.
4. Because of the relations mentioned above, it has been suggested that the solubility parameter can be estimated when the CST is determined, and that the dropping point can be estimated from the solubility parameter.

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