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MICROEMULSION OF MOLTEN SALTS

DO REN CHANG

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13. ABSTRACT (Maximum 200 words) A non-aqueous, microemulsion system, consisting of molten salts with sodium dodecyl sulfate (SDS), 1-pentanol, and decane, was studied with electrical-conductivity measurements in the molten salt-rich emulsion phase. The molten salts are a nitrate mixture of ethylenediamine/ammonium/potassium of 50/42.5/7.5 by weight, respectively. The continued addition of alcohol to a fixed ratio of salts/surfactant/oil initially causes the conductivity to decrease and then to rise again, and the solution exhibits a change from turbid to clear to increasing turbidity. This may indicate a structural change from a globular droplet to a bi-continuous microemulsion form. Other properties of microemulsions related to these measurements will be discussed.				
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1. INTRODUCTION

A solution phenomenon termed "microemulsion" by Schulman et al. (Hoar and Schulman 1943; Schulman, Stoerckenius, and Prince 1959) was used to describe a change in turbid emulsion solutions of oil, water, and surfactant into transparent solutions when alcohol was added. More recently, this concept has stimulated broad interest both theoretically and experimentally (Prince 1977; Robb 1982; Andelman et al. 1987; Widom 1984, 1986; De Gennes and Taupin 1982; Talmon and Prager 1977, 1978, 1982; Scriven 1976, 1977; Di Meglio, Dvolaitzky, and Taupin 1985; Chen et al. 1986; Borkovec et al. 1988; Lindman et al. 1980; Lichterfeld, Schemling, and Strey 1986; Kaler et al. 1983; Kaler, Davis, and Scriven 1983; Winterfield 1981; Dvolaitzky et al. 1980; Shah and Hamlin 1971; Calje, Agerof, and Vrij 1977). With the availability of certain modern instruments, great progress has been made in elucidating the microstructure of microemulsions. It is generally believed that there are three broad classifications of microemulsions—oil-in-water, water-in-oil, and bi-continuous. When there are small fractions of oil in water or of water in oil, the structure can be considered to exist as globular, tubular, or lamellar micelles, and when the volumetric fractions of oil and water are comparable, one expects random, bi-continuous microemulsions (Talmon and Prager 1978; Scriven 1976, 1977; Kaler et al. 1983; Kaler, Davis, and Scriven 1983; Winterfield 1981; Lazabat et al. 1982). This explains why such a small amount of surfactant takes up such a large amount of oil and water. The oil-in-water, microemulsion-containing surfactant micelles are swollen with oil, and the water-in-oil microemulsion containing inverted surfactant micelles are swollen with water. Friberg and Buraczewka (1977) have shown inverse micellar solutions to be identical to microemulsions. Kaler et al. (1983) have even demonstrated that the microemulsion changes from a water-rich, lower phase at low salinity to an intermediate-density, middle phase, and then to an oil-rich, upper phase as salinity increases in a system of brine/octane/ TRS surfactant/tertiary amyl alcohol. Borkovec et al. (1988) have seen similar behavior in other systems. Shinoda (Shinoda 1983; Shinoda and Lindman 1987) referred to the microemulsion phase as a surfactant or surfactant-rich phase, which seemed quite proper. Biais et al. (1986) considered a microemulsion, from vapor pressure measurement, as a three-pseudophase system—oil pseudophase, membrane pseudophase, and water pseudophase. Each pseudophase obeys equilibrium classical thermodynamical principles, with negligible interfacial tension between microdomains. Theoretical work in ternary and pseudoternary systems has shown that the middle phase is a microemulsion (Andelman et al. 1987; Widom 1984, 1986).

Most of the work in microemulsions has been done in aqueous systems. Only a few nonaqueous systems have been reported (Peyrelasse, Moha-Ouchane, and Boned 1988, 4155; Friberg, Rong, and Ward 1988; Langmuir 1988; Almgren, Swarup, and Lofroth 1985; Fletcher, Gralal, and Robinson 1984). Here we report experimental studies on nonaqueous microemulsions of molten salts. The molten salts are a mixture of ethylene diamine dinitrate, ammonium nitrate, and potassium nitrate, with the composition 50/42.5/7.5 by weight percent, respectively. This mixture has a eutectic melting point around 104° C. Because the system is solid at room temperature, it was convenient to examine it under a microscope. Thus, this study may shed some light on the microstructure of microemulsions.

2. EXPERIMENT

2.1 Chemicals. Ethylene diamine dinitrate was obtained by straightforward nitration of ethylene diamine with nitric acid, washed with methanol, and dried. Reagent grade ethylene diamine, nitric acid, ammonium nitrate, potassium nitrate, 1-pentanol and decane were obtained from Fisher Scientific, and sodium dodecyl sulfate (SDS) was obtained from the Stepan Company. These were used without further purification.

2.2 Procedure. Melt 10 g of salt mixture in an oil bath at 120° C, while its electrical conductivity is measured with a Beckman Conductivity Bridge, RC-16D, at low frequency (85 Hz) to avoid electrophoresis. The value so obtained for each run will be used as a reference for the subsequent measurements. Two definite paths are followed; they both have dilute surfactant, cosurfactant, and oil in the concentrated, molten salts: Path A—a given amount of SDS and decane in 10 g of molten salts is titrated with 1-pentanol. Path B—a given amount of SDS and pentanol in molten salts is titrated with decane. Conductivities are then compared with the molten salt alone and shown in Figures 1a–1c. Associated with each addition of pentanol or decane is an observation of the visual appearance of the solutions.

3. RESULTS AND DISCUSSION

To a single-phase molten salt, one adds surfactant to form a micellar solution, which is in equilibrium with the micellar aggregate of the surfactant-rich phase. The conductivity versus the SDS

concentration plot is shown in Figure 2. The critical micellar concentration (CMC) at 120° C is found to be about 0.03 M or 0.6 wt%. This is an interesting comparison with the CMC of SDS in other solvents (Almgren, Swarup, and Lofroth 1985; Ramadan et al. 1985). It was about 0.008 M in water at 30° C; 0.03 M in hydrazine at 45° C; and 0.0008 M in formamide at room temperature, but near 0.22 M at 60° C. Thus, the SDS CMC in molten salt is at least four times greater than in water. This will have some effects on the phase diagram as will be shown later. Micelles seem more difficult to form in molten salt than in water. However, due to high concentration of counter ions in molten salt, it will effectively screen the head group repulsion of SDS. The surfactant film curvature is easily flattened with small amounts of oil penetration. Therefore, the three-phase triangle is highly unsymmetric and biased toward the molten salt corner of this "pseudoternary" system. When oil is added to the micellar solution of molten salts, the uptake of oil drastically decreases the curvature of the surfactant film and also increases micellar concentration. If alcohol is added, the rigidity of the surfactant film is reduced (Andelman et al. 1987; De Gennes and Taupin 1982; Di Meglio, Dvolaitzky, and Taupin 1985) and the surface tension of the film is also lowered (Almgren and Swarup 1983; Ruckenstein 1978, 1979, 1980; Ruckenstein and Krishnan 1980). Thus, it facilitates the making and breaking of the micelles. This favors the random, disordered phase; the droplet size becomes smaller, and the solution become clearer. This explains why the conductivity of the solution decreases as alcohol is added. Further addition of alcohol at some particular amount of surfactant causes the cluster of micelles to become infinitely connected, and conductivity goes down to a very low value. The microemulsion droplets aggregate to form the bi-continuous, lamellar form. This is a percolation phenomenon (Dvolaitzky et al. 1980; Lagourette et al. 1979; Chatenay et al. 1985; Lagüe 1979), that is, a transition between two types of structure (De Gennes and Taupin 1982; Talmon and Prager 1977, 1978, 1982). The conductivities go up and the solution appears turbid, as shown in Figure 1. That the addition of the alcohol induces a percolation transition has been demonstrated by Chen et al. (1986). The general features of this path are that conductivities initially decrease to some minimum value, then increase as alcohol is added. Accompanying this trend, the solution is changed from turbid to clear to turbid. This is the traditional Schulman (Hoar and Schulman 1943; Schulman, Stoerckenius, and Prince 1959) approach by emulsifying oil and water using surfactant and then adding alcohol to reach to the transparent solution. Because the surfactant must be present almost exclusively at the interface, the amount of surfactant available will control the area of contact between the oil and molten salt regions and, therefore, determine the extent of microemulsion.

The experimental results of Figure 1c showed that approximately 0.4 g SDS and approximately 0.6 ml of oil are needed to have percolation near zero in conductivity. Thus, an oil percolation threshold, i.e., the volume ratio of oil to total solution, can be obtained, which is approximately 0.10 ± 0.02 . Because molten salt has a density of 1.48 g/ml, this threshold value is slightly lower than 0.16 (Kaler, Bennett, et al. 1983; Kaler, Davis, et al. 1983; Winterfield 1981), and theory predicted between 0.15 - 0.33 depending on lattice structure (Kirkpatrick 1971, 1971; Webman, Jortner, and Cohen 1975), and about 0.2 by experiments of others (Borkovec, Eicke, Hammerich, and Gupta 1988). Actually, a percolation threshold is a complex function (Peyrellase, Moha-Ouchane, and Boned 1988) of temperature and molar ratios of conductor/surfactant. Stronger interaction between clusters is believed to reduce the percolation threshold (Safran, Webman, and Grest 1985). Because 0.4 g SDS is needed to saturate the interface created between molten salt and oil, following De Gennes and Taupin (1982), of the inverted micelle model, if N_s is the number of surfactant molecules; Σ is the area per head group; v is the number of droplets of radius r , and V is the volume of molten salt, then we can calculate Σ if r is known using the following equations:

$$v \ 4\pi \ r^2 = N_s \ \Sigma \quad \text{and} \quad V = \frac{4\pi}{3} \ r^3 \ v ,$$

thus,

$$\Sigma = \frac{3V}{rN_s} .$$

The molten salts have a density of 1.48 g/ml; therefore, $V = 6.8$ cc for a 10-g sample, and it is reasonable to assume the droplet diameter is about 800 Å, that is, approximately 1/6 the wavelength of visible light, when the solution becomes transparent. Therefore, Σ is calculated to be about 60 Å². This is in excellent agreement with the experimental limiting value of 66 Å² at high water content (Dvolaitzky 1980), and 60 Å² for various counter ions (Almgren and Swarup 1983).

We can also estimate interfacial film make-up from the amount of surfactant, alcohol, and oil at the percolation bi-continuous region of Figure 1c. In terms of molecular ratios this is about 1 surfactant/2 alcohol/4 oil.

Following path B, the fixed ratio of molten salt/SDS/pentanol is diluted with oil. As mineral oil is added, the solution changes from an upper surfactant-rich phase to a three-phase region with a

middle surfactant-rich phase and finally to a lower surfactant-rich phase. Further addition of mineral oil changed it into the gel form. However, if decane is used as the oil component, the final stage of the lower surfactant-rich phase does not achieve gel formation at the concentration of the decane that produced gel formation when mineral oil was used. This means that the three-phase region is probably extended to the pseudoternary oil corner for decane. Bennett et al. (1982), Borkovec et al. (1988), and Guering and Lindman (1985) have observed a change from O/W \rightarrow bi-continuous \rightarrow W/O as salinity increases in a different system. However, we believe that an inversion phenomenon does not occur in our system, and it is very similar to the system (Guering and Lindman 1985) SDS/butanol/toluene/water even though an inversion does occur in SDS/butanol/toluene/brine as the salinity of the brine is varied (Belloce et al. 1979).

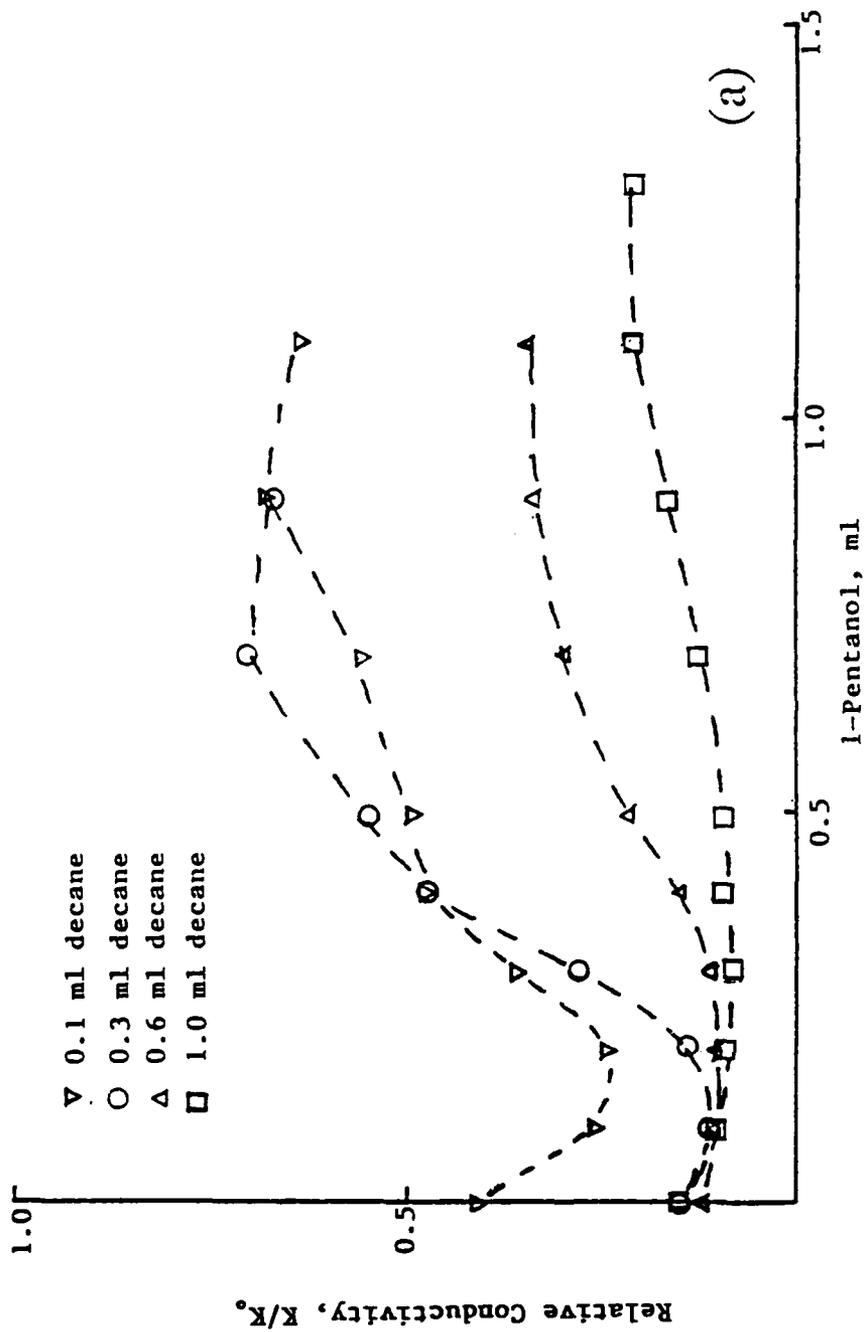
A typical result for conductivity measurement along path B is shown in Figure 3. The conductivity drops and then rises again for mineral oil, indicating that the lower bi-continuous, surfactant-rich phase has been reached.

Based on paths A and B, a partial pseudoternary-phase diagram on a weight-percent basis at 120° C was constructed and is shown in Figure 4. In this multicomponent system, we treat the molten salts as a single component, then from the tetrahedron quaternary system—molten salt, surfactant, cosurfactant and oil—a plane is cut at the fixed weight ratio of molten salt/surfactant equals 25. This is our pseudoternary-phase diagram. It shows a three-phase region with lamellar bi-continuous phase as one of the three phases, and it is highly asymmetric with its apex toward the molten salt corner. This indicates that the solubilities of the surfactant in the molten salt and in the oil have significant differences (Andelman et al. 1987; Cates et al. 1988). A greater amount dissolves in molten salt; therefore, one corner of the three-phase triangle does not extend to the molten salt pseudoternary corner. Thus, lamellar bi-continuous, molten salt micellar and near-pure oil phases make up the three-phase triangle. In the two-phase region, there is a coexistence between molten salt micellar solution and lamellae. The boundary curve in Figure 4 was established through conductivity measurements along path A, where it signalled the transition to the bi-continuous form.

A typical micrograph of molten salts/pentanol/decane/SDS micellar solution is shown in Figure 5. The globular, tubular, and bi-continuous structures can be seen; this may indicate equilibrium among different forms. Lindman et al. (1980) indicated that some microemulsions are continuous in both oil and water.

4. CONCLUSION

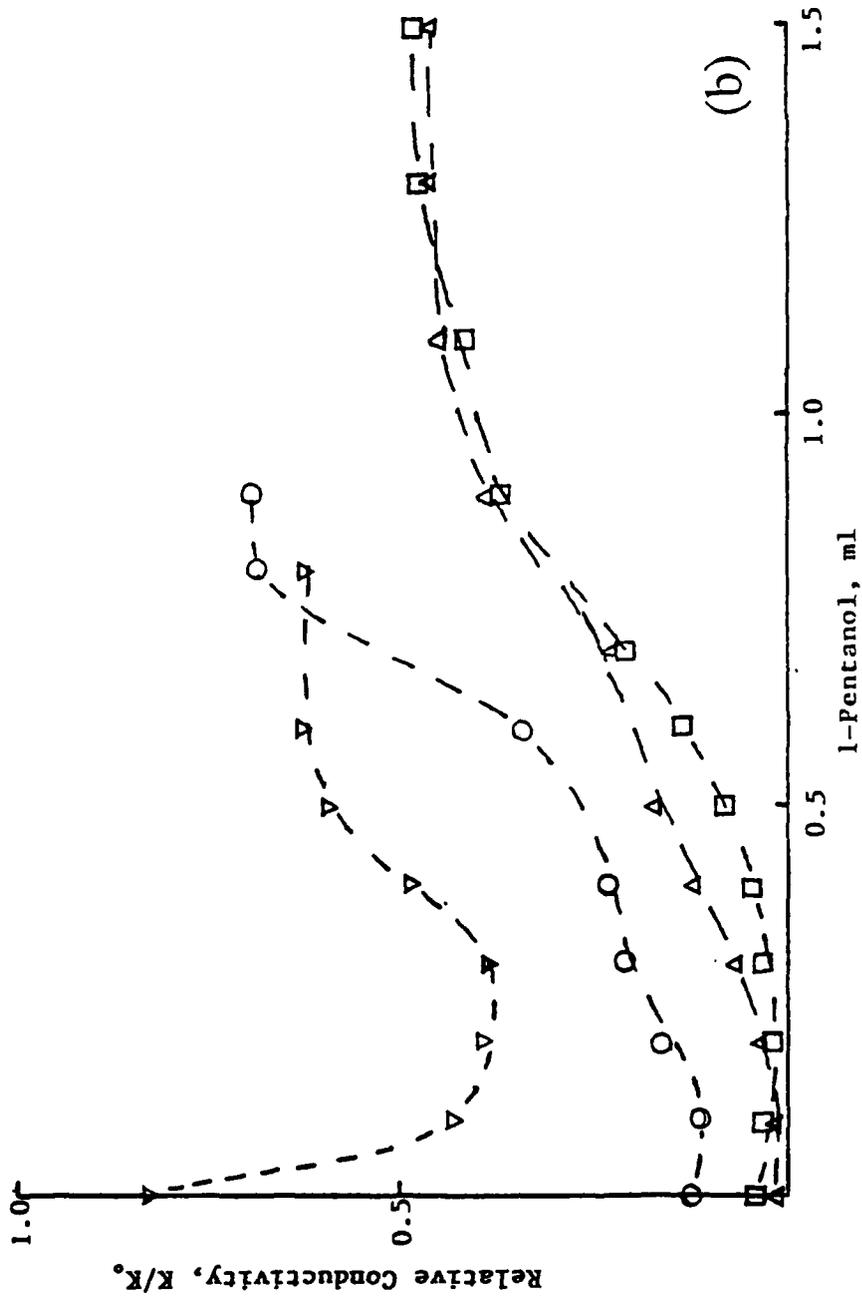
Electrical conductivity measurements have provided some information about percolation phenomena, head group area of surfactant molecules, and interfacial film make-up. The system studied involves the three-phase triangle with the coexistence of lamellae, oil, and molten salt micellar solution, while the two-phase region involves the coexistence of lamellae and molten salt micellar solution. The product solidified at room temperature, which provided a convenient way to examine the structure under a microscope. These are quite different from aqueous microemulsion systems. It would be desirable to have complementary techniques such as surface tension, viscosity, diffusion and vapor pressure measurement, and a complete phase diagram.



(a)

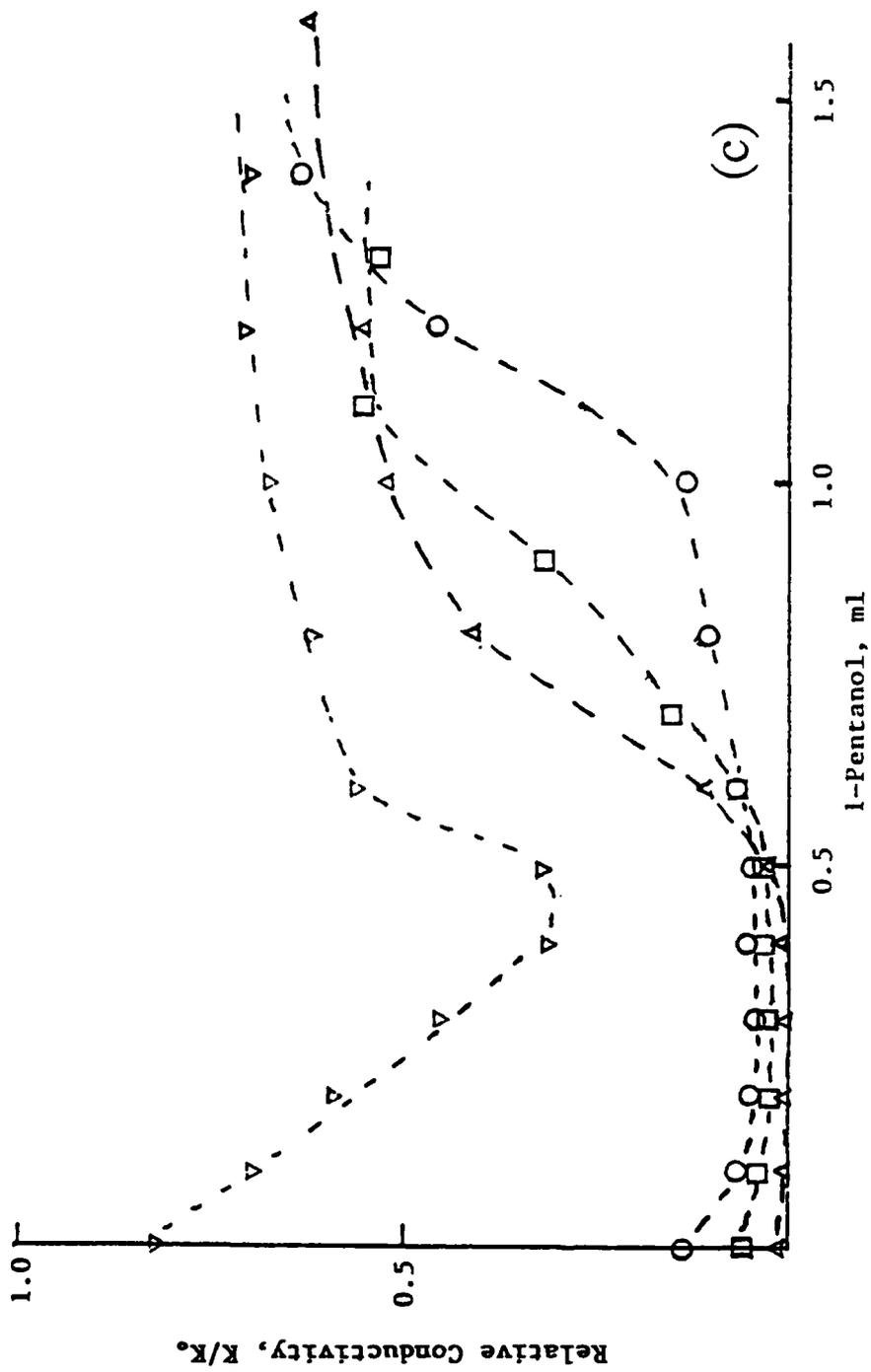
(a) For Salt/Surfactant Weight Ratio 10/0.1.

Figure 1(a). Electrical Conductivity at Fixed Salt/Surfactant Ratio, but With Different Oil Level, Where K and K₀ Are, Respectively, the Specific Conductivity at the Point of Concern and at the Molten Salt Alone. Titration Is With 1-Pentanol.



(b) For Salt/Surfactant = 10/0.2 (Same Symbols for Oil as in (a)).

Figure 1(b). Electrical Conductivity at Fixed Salt/Surfactant Ratio, but With Different Oil Level, Where K and K₀ Are, Respectively, the Specific Conductivity at the Point of Concern and at the Molten Salt Alone. Titration Is With 1-Pentanol.



(c) For Salt/Surfactant = 10/0.4 (same Symbols as in (a)).

Figure 1(c). Electrical Conductivity at Fixed Salt/Surfactant Ratio, but With Different Oil Level, Where K and K_0 Are, Respectively, the Specific Conductivity at the Point of Concern and at the Molten Salt Alone. Titration Is With 1-Pentanol.

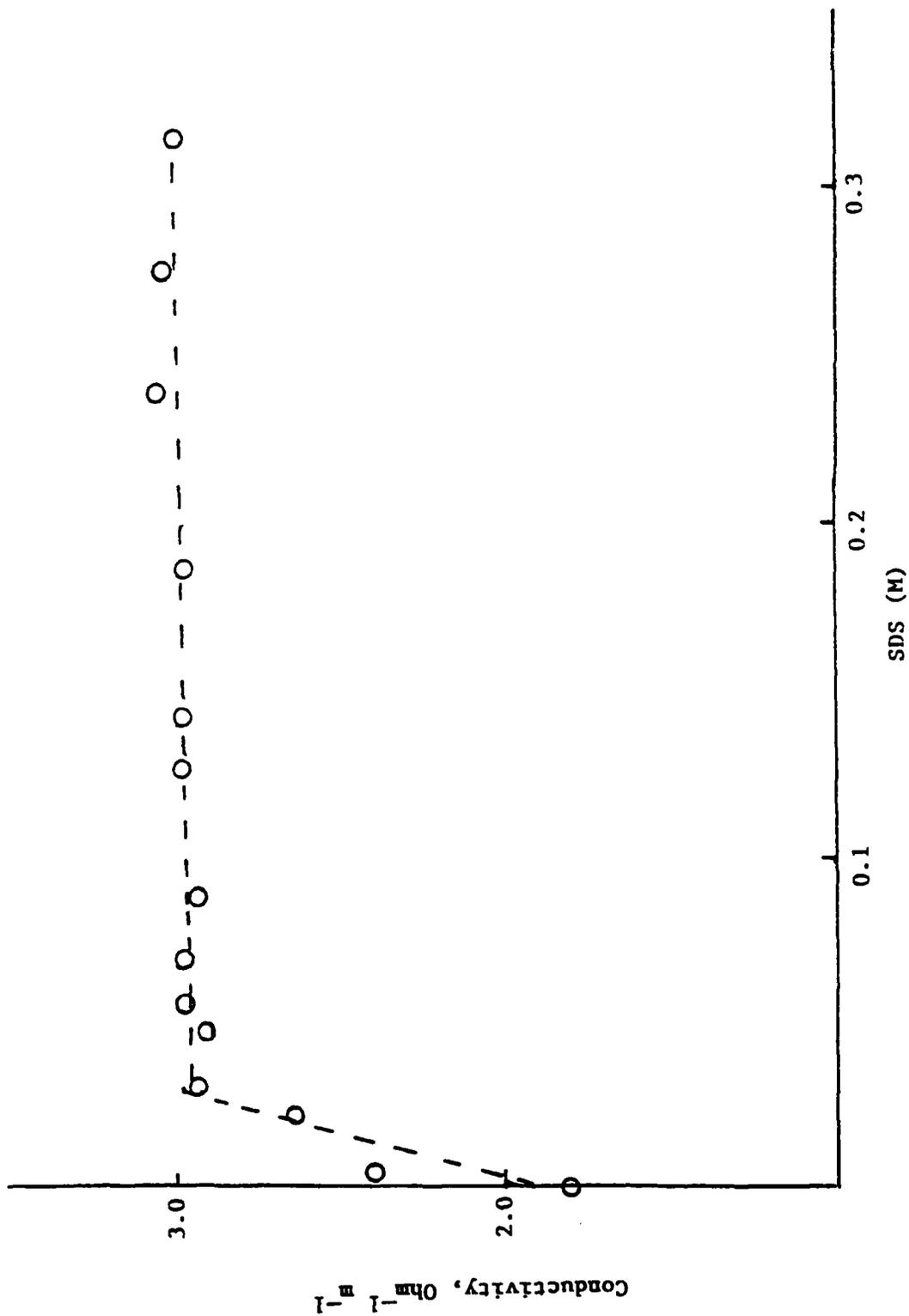


Figure 2. Electrical Conductivity vs. SDS Concentration in Molten Salts.

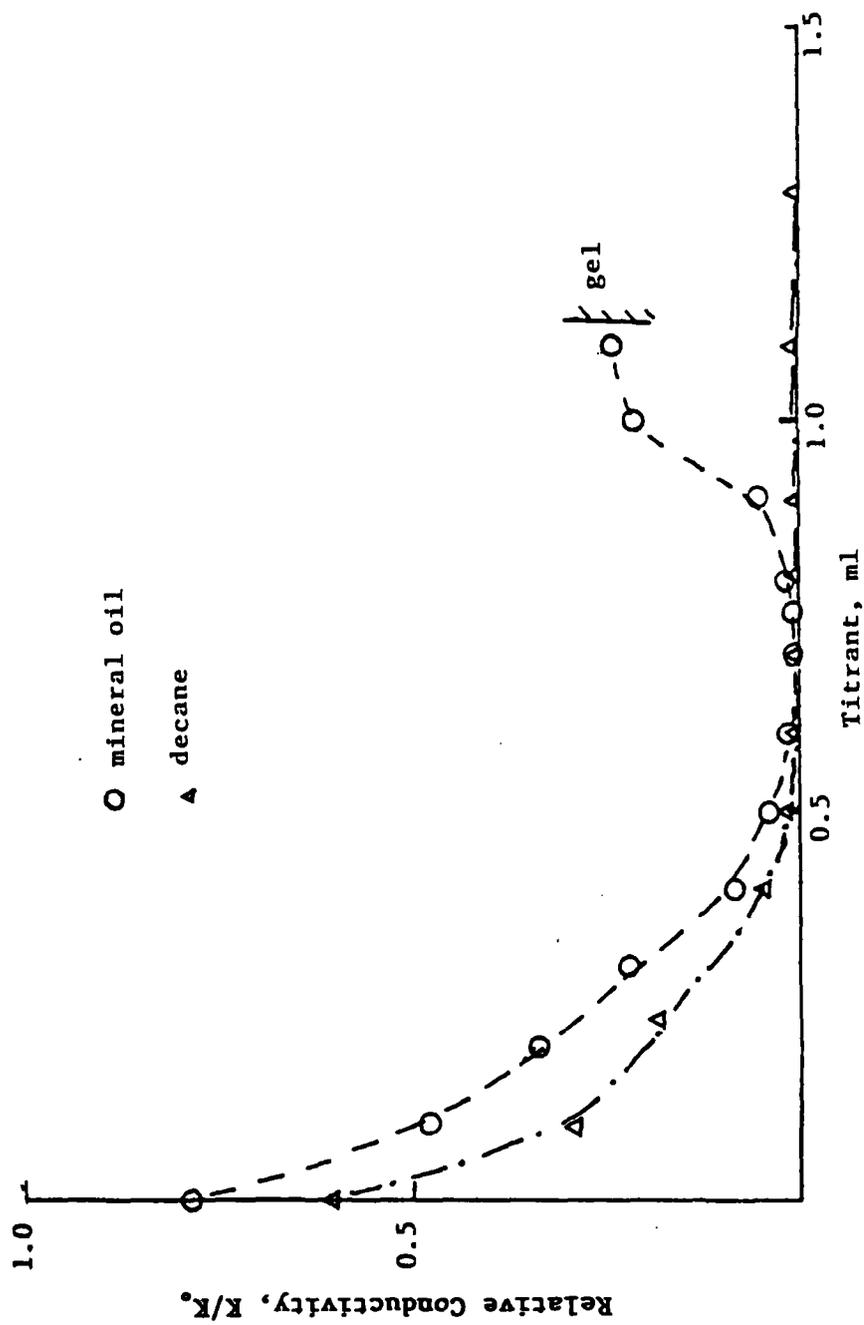


Figure 3. Electrical Conductivity at Fixed Ratio of Salt/Surfactant/Alcohol = 10 g/0.4 g/0.4 ml. Titration Is With Oil.

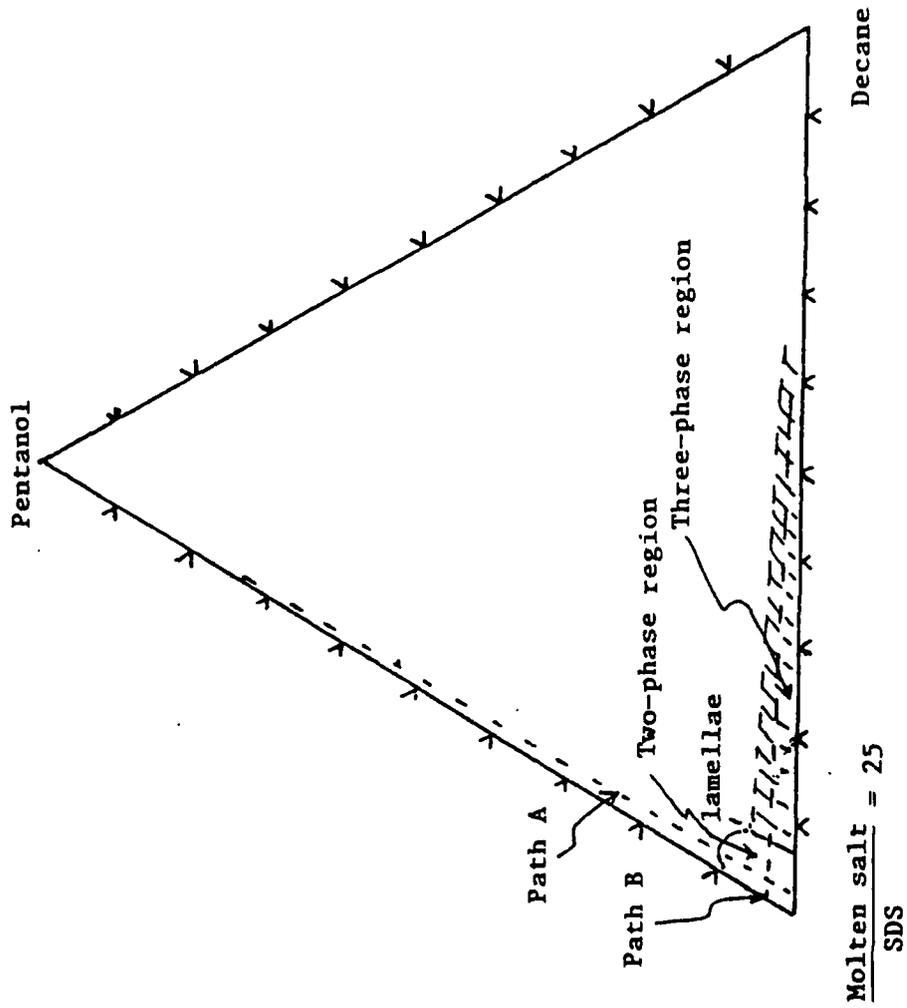


Figure 4. Partial Pseudoternary Phase Diagram.

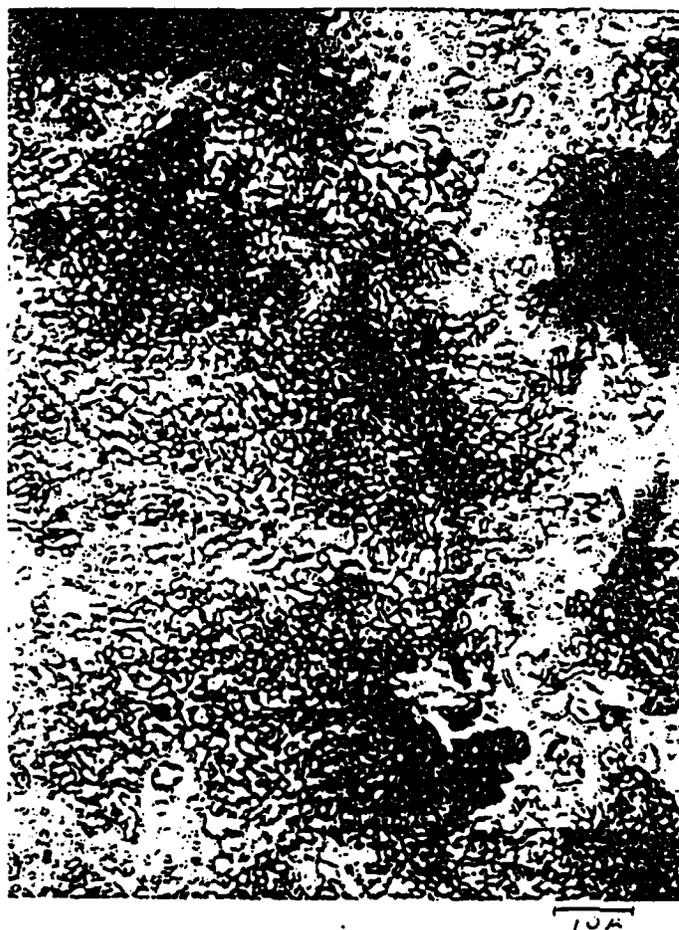


Figure 5. Micrograph of Molten Salts/SDS/Pentanol/Decane System.

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