CHAPTER III
MATERIAL AND EXPERIMENTAL METHODS

3.1 Materials

Part I Octylphenoxypoly(ethyleneoxy)ethanol with an average of 7 moles of ethylene oxide per mole of octylphenol, $OP(EO)_7$, from Rhone-Poulenc (Igepal CA-620) was the nonionic surfactants used as received in this part. Reagent grade 1,2-dichloroethane, 1,1,1,2-tetrachloroethane, from Fluka Chemika-Biochemika, and 1,1,1-trichloroethane, from J.T. Baker Inc. were used as organic materials in this study. The water was deionized and distilled.

Part II Octylphenoxypoly(ethyleneoxy)ethanol with an average of 9 moles [$OP(EO)_9$: trade name Igepal CA-630] of ethylene oxide per mole of octylphenol from Rhone-Poulenc was the nonionic surfactant used as received in this part. The organic materials and water were used as part I.

3.2 Experimental Methods

Part I Several identical 100-mL separatory funnels containing aqueous solutions with 50 mM $OP(EO)_7$, and an 1.0 mM of organic solute (1,2-dichloroethane, 1,1,1-trichloroethane, and 1,1,1,2-tetrachloroethane). Each set of solutions was placed in an isothermal water bath until equilibrium was reached, generally about 2 days. After phase separation had occurred, the fractional volume of each phase was measured. The $OP(EO)_7$ and organic solute concentrations were measured in both the coacervate and dilute phases.

Part II Ordinary 5-mL equilibrium dialysis cells and transparent regenerated cellulose membranes (6000 dalton molecular weight cutoff) were obtained from Fisher Scientific and used without modification in a technique called semi-equilibrium dialysis (Christian et al., 1985; Uchiyama et al., 1993;Rouse, Sabatini, Deeds, Brown and Harwell, 1995) which can be used to measure solubilization of an organic solute into micelles. When equilibrium dialysis experiments were performed, aqueous solution of 50 mM $OP(EO)_9$ plus 1.0 mM of organic solute was loaded in one compartment of the cell, and pure water was added to the other side. The cells were kept in a desiccator that was submerged in a thermostated bath at 30, 40, and 50 °C until equilibrium was reached, about 24 hours. The surfactant and solute concentrations were measured in both the retentate and permeate sides.

3.3 Analysis

The $OP(EO)_7$ and $OP(EO)_9$ concentrations were measured by using a Cecil Instruments model CE 2000 series UV spectrometer at 224 nm. 1,2-dichloroethane, 1,1,1-trichloroethane, and 1,1,1,2-tetrachloroethane concentrations were analyzed by using Perkin-Elmer model autosystem GC gas chromatography equipped with a flame ionization detector. Perkin-Elmer model HS-40 automatic headspace sampler was used as the sample injection because of the high volatility of organic solutes. The conditions of gas chromatography and headspace sampler used for determination of organic solutes concentration were as follows.

GC conditions for the chloroethanes

	Dichloroethane	Trichloroethane	Tetrachloroethane
Column Type	SUPELCOWAX 10 packed column		
Carrier Gas	Nitrogen, 20 mL/min		
Oven	100 °C isothermal		140 °C isothermal
Injector	150 °C		
Detector	FID, 250 °C		

HS conditions for the chloroethanes

	Dichloroethane	Trichloroethane	Tetrachloroethane
Thermostat Time	10 min		
Sample Temp.	80 °C	70 °C	110 °C
Needle Temp.	100 °C	90 °C	130 °C
Transfer Temp.	100 °C	90 °C	130 °C
Pressurize Time	0.1 min		
Injection Time	0.3 min		
Withdrawal Time	0.3 min		