



Relative apparent molar enthalpies of dilution of some polyoxyethylene glycol monodecyl ethers ($C_{10}E_n$), of decanol in cyclohexane and heptane, and of $C_{10}E_6$ aqueous mixtures in decane at 298.15 K

Reiji Tanaka*, Minori Adachi¹

Department of Chemistry, Graduate School of Science, Osaka City University, Sugimoto, Sumiyoshi-ku, Osaka 558-8585, Japan

Received 6 November 2002; received in revised form 10 February 2003; accepted 10 February 2003

Abstract

The relative apparent molar enthalpies, L_ϕ , of polyoxyethylene glycol monodecyl ether, $C_{10}E_n$ for $n = 1-8$ and decanol were measured in cyclohexane and heptane at 298.15 K by using a flow calorimeter. Also, the measurements for $C_{10}E_6$ in decane were carried out with addition of small amounts of water. The aggregation of $C_{10}E_n$ proceeds gradually with increasing molality of solutes due to van der Waal's force acting among hydrophilic groups, the OH groups being blocked from forming intramolecular hydrogen bonding. However, by adding a small amount of water the aggregation is strongly enhanced due to the intermolecular hydrogen bonds formed by a bridge of water across the hydrophilic groups. Those results are consistent with the results obtained by heat capacity measurements reported previously.

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Keywords: Aggregation; Reversed micelle; Apparent molar enthalpy; Polyoxyethylene glycol monodecyl ether

1. Introduction

The ternary mixtures composed of surfactant, water and oil have attracted interest since they form thermodynamically stable phases involving self-organized assemblies. In a media of non-polar solvents the surfactants solubilize water forming water–oil (w/o) microemulsions or reversed micelles. It is well known that a surfactant in water forms micelles passing through a CMC. On the other hand, the existence of water is necessary in non-polar media to form

reversed micelles. The study of aggregation process of surfactants in dilute regions helps us to understand the structure and physicochemical properties of self-organized assemblies from the viewpoint of molecular interactions. We have reported experimental results of apparent molar volumes and heat capacities of polyoxyethylene glycol monodecyl ether ($C_{10}E_n$) for $n = 1-8$ and decanol, which corresponds to the compound for $n = 0$, in cyclohexane, heptane, octane, and decane at 298.15 K [1,2]. In the previous studies, we showed the remarkable effect of water on the aggregation process. We also found that the aggregation is strongly influenced by the length of alkane used for solvent as well as the size of hydrophilic groups, and it is enhanced by a small amount of water. In the present paper, we report the results of relative

* Corresponding author. Tel.: +81-6-6605-3126; fax: +81-6-6605-2522.

E-mail address: rtanaka@sci.osaka-cu.ac.jp (R. Tanaka).

¹ Present address: Gunze Ltd., Osaka, Japan.

apparent molar enthalpies, L_ϕ , for $C_{10}E_n$ ($n = 1-8$) and decanol, measured in cyclohexane, heptane and decane at 298.15 K, involving an examination of the effect of added water.

2. Experimental

2.1. Materials

The polyoxyethylene glycol monodecyl ether was purchased from Nikko Chemical Co., and used without further purification. Decanol was Gold Label reagent from Aldrich, and used as received. Cyclohexane, heptane, benzene, toluene, and chlorobenzene were special grade material of Wako Pure Chemicals Co. and fractionally distilled. Decane was from Tokyo Kasei Kogyo Co., and used as received. Water was purified by using osmosis membrane (Millipore Co., Milli-Q Labo). According to glc analysis, the purity of distilled solvents exceeded 99.97 mol.%. The purity of decane was 99.89 mol.%. The mole fraction of water in $C_{10}E_n$ and decanol detected by Karl Fisher's method was <0.02 .

2.2. Calorimetry

A flow micro-calorimeter for measuring enthalpy of mixing was constructed. Fig. 1 shows the design of the calorimeter. A stainless steel tube A (2 mm o.d. and 1.6 mm i.d.) of 1.5 m was tightly wound and soldered around the copper cylinder B (40 mm o.d. and 45 mm height). On a side of this cylinder a square plate of copper (2 mm thickness and 45 mm width) was soldered. A stainless steel tube C (1.5 mm o.d. and 1.2 mm i.d.) was inserted in the tube A and coiled together with tube A for 13 cm length to prevent the backward flowing of the mixed liquids due to diffusion. The liquid are introduced into the calorimeter through the Teflon coupling D after thermostated during they are flowed in the 50 cm tubes which are located on the calorimeter vessel. Stainless steel wire (0.3 mm d) was coiled on two pieces of stainless steel wire (0.6 mm d and 15 mm length). Those of 15 segments E were installed in the tube A to promote mixing. A calibration heater H made of manganese wire (47 Ω) was wound on the copper cylinder alternately with the coil of stainless steel tube A and fixed by using insulating varnish

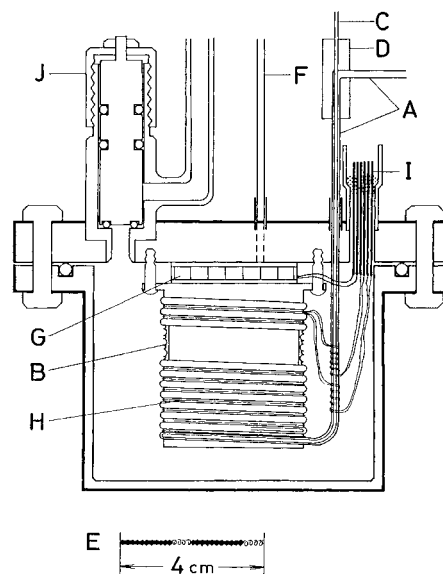


Fig. 1. Schematic diagram of the flow calorimeter for mixing enthalpy.

(General Electric Co., GE 7031). A thermopile G (Komatsumu Electronics Inc., KSM-0671) was sandwiched between the copper cylinder plate and the lid made of brass. The outlet stainless steel tubes F and lead wires I were taken outside through the lid. The assembly was attached to a jacket made of brass, and the air inside of the jacket was evacuated through the valve J to 1 Pa. The calorimeter was immersed in the water bath, and the temperature was controlled constant within ± 0.0002 K. Two HPLC pumps (Waters, Millipore Co., Model 510) were used to make flow of liquids. The flow rate was changed with a step of $0.01 \text{ cm}^3 \text{ min}^{-1}$ keeping the total flow rate to be $0.4 \text{ cm}^3 \text{ min}^{-1}$. The flow rates were calibrated with uncertainty of 0.1% by measuring the time to fill the calibrated volume (1 or 2 cm^3) of pipettes. The electrical signal from the thermopile was monitored with a recorder after amplification. The fluctuation of the base line was $\pm 2 \mu\text{W}$.

The test runs for this calorimeter were carried out by measuring excess molar enthalpies H^E for the systems of [xbenzene + (1 - x)cyclohexane] and [xchlorobenzene + (1 - x)toluene] at 298.15 K. The experimental values were fitted with the smoothing equation:

$$H^E (\text{J mol}^{-1}) = x(1-x) \sum a_i (1-2x)^{i-1}. \quad (1)$$

Table 1
Coefficients a_i for Eq. (1) and the calculated standard deviations s

System	a_1	a_2	a_3	a_4	s (J mol ⁻¹)
x Benzene + (1 - x)cyclohexane	3186.6	-164.84	143.35	-48.56	0.84
x Chlorobenzene + (1 - x)toluene	-474.9	-31.58	8.14	-	0.19

The determined coefficients and the calculated standard deviation for each set are listed in Table 1. More than 60 sets of H^E measurements are listed in the handbook [3] for (benzene + cyclohexane) system. The present results agreed with those reported by Tanaka et al. [4], Elliott and Wormald [5], and Ewing et al. [6] within a deviation of $\pm 0.5\%$. The results for (chlorobenzene + toluene), which was tested for an exothermic system, agreed with those reported by Tanaka and Benson [7] and Kimura and Takagi [8] within a deviation of $\pm 0.5\%$. The experimental values of the tested systems and the deviation plots are shown in a previous report [9].

The experimental results were presented with relative apparent molar enthalpies, L_ϕ , which is defined by the relation:

$$L_\phi = \Delta_{\text{dil}}H_\phi - \Delta_{\text{dil}}H_\phi^\infty \quad (2)$$

where $\Delta_{\text{dil}}H_\phi$ is the apparent molar enthalpy for diluting a solution of a fixed molality m° with solvent, and $\Delta_{\text{dil}}H_\phi^\infty$ the apparent molar enthalpy of dilution extrapolated to the infinitely dilute molality. The molality m° of the solution prepared for dilution process was 0.8–1.8 mol kg⁻¹. The apparent molar enthalpy of dilution $\Delta_{\text{dil}}H_\phi$ was calculated by the equation,

$$\Delta_{\text{dil}}H_\phi = \frac{dQ/dt}{dn/dt} \quad (3)$$

where dQ/dt is the heat evolved per second due to dilution and dn/dt the amount of solute flowing per second. The heat flow dQ/dt was evaluated with constants calibrated for the heat loss that is related to flow rates and the heat capacities divided by volume of the mixture.

3. Results and discussion

The experimental results of L_ϕ for C₁₀E_{*n*} and decanol in cyclohexane, heptane and decane are listed in

Tables 2 and 3, and shown graphically in Figs. 2–4, respectively, along with enlargement for low molalities. The smoothing function,

$$L_\phi \text{ (J mol}^{-1}\text{)} = \sum c_i m^{(i-1)} \quad (4)$$

was fitted to each set of results by the least squares method. In some cases, we could not find a smoothing equation to describe all points with a set of parameters so that two functions were fitted to the observed points which were divided into two overlapping groups. Those curves are presented in figures. The observed points at m° are not plotted.

The curve of L_ϕ versus m for decanol is sigmoid and decreases sharply with increasing m due to

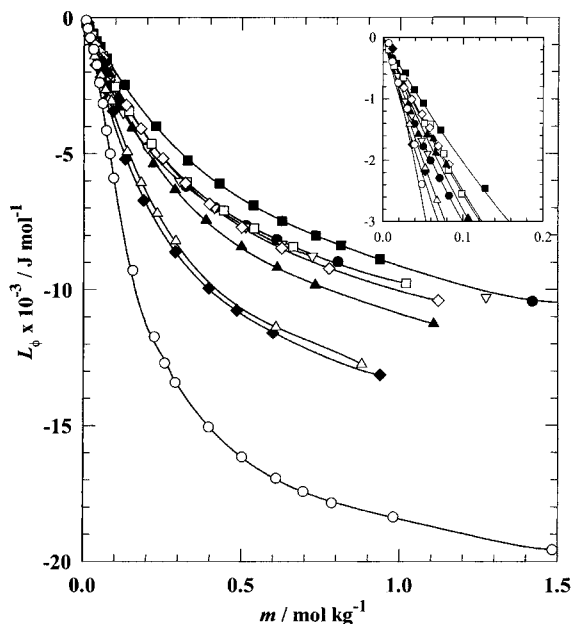


Fig. 2. Relative apparent molar enthalpies, L_ϕ , for C₁₀E_{*n*}, and decanol in cyclohexane at 298 K. Experimental results: (■) $n = 1$; (●) $n = 2$; (▽) $n = 3$; (□) $n = 4$; (◇) $n = 5$; (▲) $n = 6$; (◆) $n = 7$; (△) $n = 8$; (○) decanol. Curves are least square's representation by Eq. (4).

Table 2
Relative apparent molar enthalpies L_ϕ of polyoxyethylene glycol monodecyl ether, $C_{10}E_n$, and decanol in alkane at 298 K

$C_{10}E_1$		$C_{10}E_2$		$C_{10}E_3$		$C_{10}E_4$		$C_{10}E_5$		$C_{10}E_6$		$C_{10}E_7$		$C_{10}E_8$		Decanol	
m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)
In cyclohexane																	
0.0168	-0.42	0.0114	-0.34	0.0120	-0.42	0.0072	-0.20	0.0263	-0.82	0.0339	-1.20	0.0120	-0.19	0.0333	-1.41	0.0070	-0.09
0.0281	-0.58	0.0228	-0.77	0.0185	-0.62	0.0137	-0.47	0.0359	-1.01	0.0444	-1.57	0.0367	-1.74	0.0529	-2.12	0.0116	-0.39
0.0394	-0.85	0.0311	-1.11	0.0276	-1.04	0.0252	-0.66	0.0487	-1.25	0.0556	-1.59	0.0531	-2.18	0.0676	-2.65	0.0190	-0.74
0.0506	-1.07	0.0393	-1.41	0.0363	-1.28	0.0349	-1.00	0.0586	-1.47	0.0631	-1.65	0.0714	-3.01	0.0775	-3.02	0.0304	-1.16
0.0721	-1.50	0.0506	-1.77	0.0484	-1.68	0.0519	-1.40	0.0693	-1.76	0.0663	-1.88	0.0921	-3.44	0.0861	-3.06	0.0397	-1.74
0.1279	-2.46	0.0598	-2.01	0.0559	-1.89	0.0593	-1.41	0.0826	-2.07	0.0795	-2.07	0.1308	-5.20	0.1369	-4.92	0.0487	-2.39
0.2277	-4.00	0.0712	-2.29	0.1235	-3.49	0.0689	-1.86	0.1333	-3.21	0.1067	-2.94	0.1887	-6.72	0.1809	-6.07	0.0591	-3.16
0.3278	-5.25	0.0826	-2.58	0.2009	-4.78	0.0760	-1.89	0.1796	-4.08	0.1515	-4.05	0.2894	-8.61	0.2316	-7.20	0.0707	-4.15
0.4289	-6.10	0.1141	-3.25	0.3027	-6.02	0.0822	-2.16	0.2493	-5.18	0.2190	-5.38	0.3958	-9.95	0.2905	-8.22	0.0823	-5.01
0.5362	-6.91	0.2033	-4.74	0.4128	-7.00	0.0987	-2.55	0.3209	-6.09	0.2877	-6.33	0.4842	-10.76	0.6080	-11.38	0.0943	-5.90
0.6267	-7.48	0.3232	-6.21	0.5118	-7.70	0.1417	-3.44	0.3973	-6.85	0.3867	-7.46	0.5991	-11.59	0.8794	-12.75	0.1549	-9.29
0.7339	-8.03	0.4152	-6.99	0.6267	-8.34	0.2133	-4.63	0.4994	-7.71	0.4984	-8.43	0.9375	-13.14			0.2244	-11.74
0.8128	-8.39	0.5132	-7.65	0.7222	-8.78	0.3253	-6.04	0.6257	-8.48	0.6116	-9.20					0.2572	-12.71
0.9361	-8.89	0.6101	-8.16	1.2717	-10.29	0.4424	-7.10	0.7758	-9.23	0.7318	-9.84					0.2904	-13.42
1.5728	-10.46	0.8033	-8.99			0.5388	-7.75	1.1203	-10.41	1.1056	-11.26					0.3962	-15.06
		1.4199	-10.44			0.6626	-8.43									0.5016	-16.16
						1.0191	-9.78									0.6097	-16.94
																0.6959	-17.43
																0.7859	-17.85
																0.9804	-18.37
																1.4842	-19.57
In heptane																	
0.0181	-0.32			0.0224	-0.60	0.0230	-0.52	0.0199	-0.62	0.0138	-0.54	0.0031	-0.27	0.0819	-3.01	0.0217	-0.97
0.0367	-0.73			0.0314	-0.92	0.0400	-0.93	0.0349	-0.85	0.0208	-0.63	0.0064	-0.38	0.1132	-4.61	0.0248	-1.02
0.0950	-1.61			0.0529	-1.47	0.0718	-1.55	0.0496	-1.12	0.0270	-0.81	0.0110	-0.27	0.1428	-6.01	0.0285	-1.13
0.1530	-2.49			0.0707	-1.85	0.1077	-2.21	0.0739	-1.67	0.0344	-1.06	0.0177	-0.45	0.1989	-7.92	0.0315	-1.46
0.1908	-3.04			0.1077	-2.70	0.1402	-2.74	0.1052	-2.38	0.0363	-0.92	0.0220	-0.60	0.2973	-10.03	0.0333	-1.37
0.2865	-4.18			0.1347	-3.23	0.2081	-3.77	0.1412	-3.11	0.0507	-1.49	0.0257	-0.79	0.4100	-11.39	0.0423	-1.86
0.3720	-5.07			0.1809	-4.01	0.3006	-5.00	0.2037	-4.23	0.0512	-1.57	0.0257	-0.65	0.4989	-12.00	0.0440	-1.81
0.3837	-5.12			0.2495	-4.89	0.3946	-5.96	0.2958	-5.60	0.0679	-2.04	0.0293	-0.77	0.6108	-12.56	0.0501	-2.43
0.4945	-5.94			0.3436	-5.84	0.4951	-6.56	0.3952	-6.66	0.0739	-2.15	0.0338	-0.93	1.6811	-14.71	0.0547	-2.69
0.4961	-6.03			0.4646	-6.73	0.6160	-7.21	0.5061	-7.53	0.0867	-2.83	0.0409	-2.04			0.0586	-2.87
0.5672	-6.44			0.5522	-7.24	0.7195	-7.67	0.6169	-8.18	0.0928	-2.77	0.0545	-2.01			0.0645	-3.14
0.7715	-7.48			0.6609	-7.75	0.8361	-8.08	0.7606	-8.84	0.1120	-3.24	0.0593	-2.22			0.0724	-4.09
0.7920	-7.54			0.7678	-8.20	1.5042	-9.45	1.0841	-9.81	0.1469	-4.19	0.0699	-2.63			0.0769	-4.33
				1.0415	-8.98					0.1888	-5.12	0.0738	-2.83			0.0799	-4.07
										0.2874	-6.93	0.1305	-5.22			0.0828	-4.39
										0.3903	-8.21	0.1527	-5.97			0.0899	-5.38
										0.5111	-9.26	0.1966	-7.26			0.1007	-6.12
										0.8899	11.07	0.3004	-9.32			0.1084	-6.22
												0.4093	-10.61			0.1229	-7.13
												0.4992	-11.34			0.1346	-7.71
												0.6152	-12.02			0.1374	-7.95
																0.1439	-8.25
																0.1613	-9.10
																0.1658	-9.11
																0.2483	-11.79
																0.3315	-13.51
																0.3827	-14.34
																0.5140	-15.65
																0.5811	-16.24
																0.7870	-17.06

Table 3

Relative apparent molar enthalpies, L_ϕ , of mixed solute ($C_{10}E_6 + rH_2O$) in decane at 298 K, where r is the mole ratio of water added to polyoxyethylene glycol monodecyl ether ($C_{10}E_6$)

m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)	m (mol kg ⁻¹)	L_ϕ (kJ mol ⁻¹)
$r = 0$		$r = 0.4$		$r = 0.6$	
0.0374	-1.32	0.0025	-0.59	0.0120	-5.69
0.0400	-1.63	0.0051	-2.10	0.0143	-6.52
0.0544	-1.98	0.0078	-3.48	0.0181	-8.45
0.0707	-2.53	0.0104	-4.02	0.0216	-9.10
0.1221	-4.07	0.0129	-4.64	0.0289	-10.98
0.1592	-5.05	0.0129	-4.64	0.0299	-11.95
0.2032	-6.00	0.0156	-5.31	0.0435	-13.60
0.3047	-7.71	0.0156	-5.31	0.0567	-15.28
0.4071	-8.90	0.0179	-5.45	0.0592	-16.77
0.5237	-9.76	0.0182	-6.17	0.1029	-18.81
0.6327	-10.42	0.0202	-6.92	0.1085	-19.24
		0.0233	-7.81	0.1449	-20.88
		0.0257	-8.35	0.2121	-22.43
		0.0271	-8.15	0.2814	-23.54
		0.0363	-10.10	0.3095	-23.86
		0.0363	-9.88	0.3923	-24.58
		0.0363	-9.88	0.5021	-25.25
		0.0383	-10.68	0.5846	-25.61
		0.0508	-12.32	0.7202	-26.05
		0.0548	-12.31	0.8193	-26.29
		0.0648	-13.32		
		0.0648	-13.61		
		0.0715	-13.89		
		0.0777	-14.70		
		0.0825	-14.83		
		0.0913	-15.41		
		0.1373	-17.77		
		0.1839	-19.26		

intermolecular hydrogen bonds. Stokes and Marsh found by dielectric studies that cyclic associated species of alkanol are formed in non-polar solvents at a small alkanol mole fraction and the self-association occurs passing through a pseudo critical concentration below which only monomers and/or very few dimers exist [10]. This critical concentration appears at a mole fraction below 0.005. On the other hand, by fitting a self-association model to heat capacities measured for binary mixtures of (alkanol + alkane) it was suggested that primary species were tetramers at low mole fraction of alkanol [11,12]. Since the associated species are very few in the infinitely dilute region L_ϕ is kept nearly zero, and then it starts to decrease sharply with increasing m . Therefore, L_ϕ -curve for decanol indicates sigmoidal change. On the other hand, the magnitude of (dL_ϕ/dm) for $C_{10}E_n$ is much smaller

than that of decanol, and L_ϕ decreases monotonously with increasing m . Nakamura et al. found by IR measurements that OH group of $C_{12}E_n$ in CCl_4 forms intramolecular hydrogen bondings with the oxygen of neighboring oxyethylene groups and also with others of a longer polar chain [13]. The present results also imply that the OH groups of $C_{10}E_n$ are blocked due to intramolecular hydrogen bonding so that the van der Waal's force acts predominantly for forming aggregates. The value of L_ϕ decreases in the increasing order of n because attractive force between oxyethylene groups increases. In the previous study of heat capacity measurements, the aggregation of $C_{10}E_n$ with n smaller than 6 proceeds rather moderately compared with that of decanol irrespective of solvent [1,2]. Whereas, we found that the aggregation of $C_{10}E_n$ with $n = 7$ and 8 is remarkable in a solvent of long chain

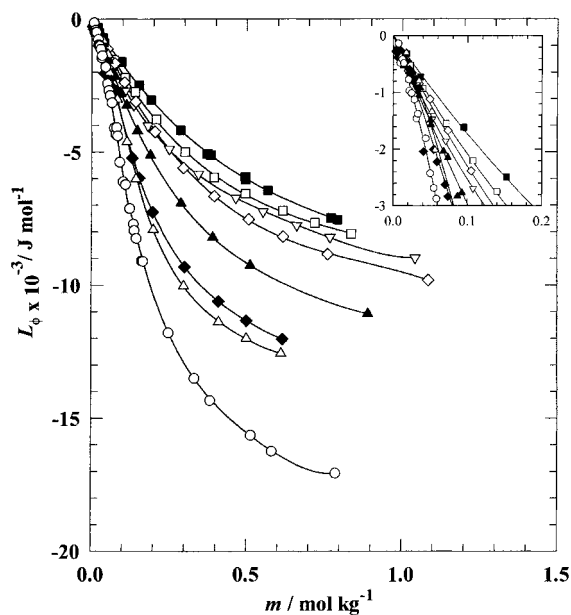


Fig. 3. Relative apparent molar enthalpies, L_ϕ , for $C_{10}E_n$, and decanol in heptane at 298 K. Experimental results: (■) $n = 1$; (▽) $n = 3$; (□) $n = 4$; (◇) $n = 5$; (▲) $n = 6$; (◆) $n = 7$; (△) $n = 8$; (○) decanol. Curves are least square's representation by Eq. (4).

alkane such as octane and decane. This indicates that the aggregation is more enhanced due to larger hydrophilic groups and also a long-chain alkane used for solvent. The values of L_ϕ for $n = 7$ and 8 decrease significantly with increasing m , and the magnitude of (dL_ϕ/dm) is larger in heptane. However, rather surprisingly, it was found that (dL_ϕ/dm) in $C_{10}E_8$ is still smaller than that in decanol instead of its large polyoxyethylene group. Ravey et al. reported that $C_{12}E_n$ takes an extended cylindrical form in alkane and the aggregation number increases in a longer chain alkane [14]. According to their model, the cylindrical form of aggregates is favorable in a solvent of straight chain alkane and the aggregation is drastically promoted by water molecule. Irregular order that is seen between n values of 7 and 8 in cyclohexane and also 3 and 4 in heptane may be attributed to an experimental error since the determination of limiting values of $\Delta_{dil}H_\phi^\infty$ are influenced by few points observed in the infinitely dilute region.

The results for $C_{10}E_6$ measured in decane are shown in Fig. 4 along with those for the mixed solutes ($C_{10}E_6 + rH_2O$), where r is the mole ratio of water

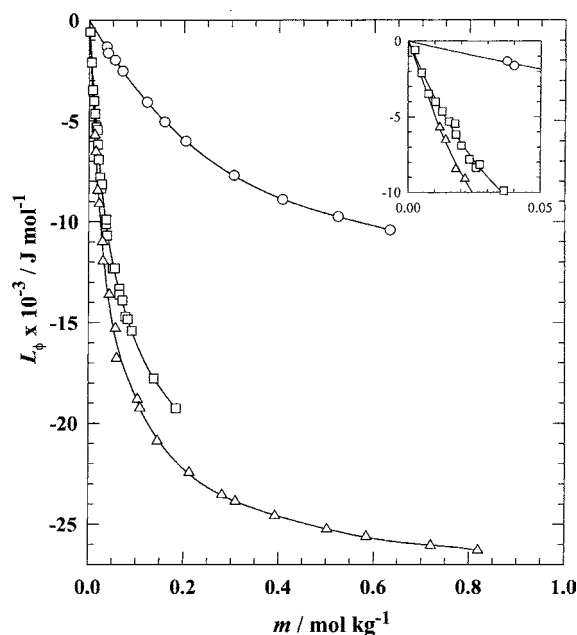


Fig. 4. Relative apparent molar enthalpies, L_ϕ , for $(C_{10}E_6 + rH_2O)$ in decane at 298 K. Experimental results: (○) $r = 0$; (□) $r = 0.4$; (△) $r = 0.6$. Curves are least square's representation by Eq. (4).

added to $C_{10}E_6$. The degree of decrease in L_ϕ for $r = 0$ with increasing m is comparable with that observed in cyclohexane and heptane. However, the effect of water added to the solute is remarkable. The value of L_ϕ decreases abruptly in the dilute region and then the change with m becomes very small reaching a constant value. This sharp decrease of L_ϕ with m shows that water molecules make intermolecular bridges across the oxyethylene groups due to hydrogen bonds so that the aggregation proceeds very sharply with increasing m . The present observation is consistent with the results found by heat capacity measurements [1,2]. The apparent molar heat capacities of $(C_{10}E_6 + rH_2O)$ for $r \geq 0.6$ observed in decane showed a very sharp peak at m below 0.05 mol kg^{-1} suggesting that the aggregation proceeds passing through a pseudo transition around the molality where the peak appears. A sharp peak associated with aggregation process has been found in the partial molar heat capacity or apparent molar heat capacity of alkanols in non-polar solvents [12,15]. It is considered that actual transition occurs at much lower molality than that where a peak appears in apparent molar heat capacity because the peak indicated

in the apparent molar quantity will be shifted to a much lower concentration if the results are presented with partial molar quantity. It is very interesting to see that the added water, that is less than one mole per solute molecule ($r < 1$), acts very effectively to promote aggregation. This observation is compared with the reversed micelle formation of AOT in oil with the help of water, where 2 mol of water are bound tightly to the hydrophilic groups of AOT so that they are not active for forming aggregates [16,17].

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