

C H A P T E R - V I

Conductance Studies of Alkali Metal Chlorides and Bromides in Aqueous
Binary Mixtures of Tetrahydrofuran at 25°C

Precise conductance measurements are reported for alkali metal chlorides and bromides, MX ($M^+ = \text{Li, Na, K, Rb and Cs}$; $X^- = \text{Cl and Br}$) in tetrahydrofuran (THF) + water mixtures at 25°C. The limiting molar conductivity, Λ° , the association constant, K_A , and association distance, R, in the solvent mixtures have been evaluated using the 1978 Fuoss Conductance equation. The analysis of data indicates that the electrolytes are almost unassociated at 0.059 mole fraction of THF, whereas at 0.143, 0.273 and 0.500 mole fraction of THF, the association was very strong. The values for alkali metal cations (anion being the same) are found to be in the order : $\text{Li}^+ < \text{Na}^+ < \text{K}^+ < \text{Rb}^+ < \text{Cs}^+$. The results have been explained in terms of ion-solvent interactions and structural changes in the mixed solvents.

Studies on ionic solvation of alkali metal ions of low permittivity are very few. Such studies have been assumed very importance because some of the solvents have their applications in modern technology¹. Tetrahydrofuran (THF), a solvent of low permittivity ($\epsilon = 7.58$), has also been found its probability of applications in high energy batteries and organic syntheses as manifested from the Physico-Chemical studies in this medium^{2,3}. Renard and Justice⁴ studied the conductances of CsCl in THF + water mixtures

to reveal the nature of ionic association and mobility of ions in this mixed solvent system. In this paper, an attempt has been made to ascertain the complete nature of solute-solvent interactions of alkali metal salts (chlorides as well as bromides) in THF + H₂O mixtures through the measurements of their conductances and the results are described below.

Results and Discussion :

The solvent properties of THF+H₂O mixtures are given in Table 1, where ϵ is the dielectric constant, d the density in g cm⁻³, η_0 the viscosity in centipoise and L_0 , the specific conductance in $\Omega^{-1}\text{cm}^{-1}$. The quantity W%, is the weight percent of THF in the aqueous mixtures and X_2 is the corresponding mole fraction. Dielectric constants of solvent mixtures were obtained by extrapolation of ϵ versus Wt% plots, the original values were taken from the work of Justice et al⁴. The measured equivalent conductances and the corresponding molarities at different percentages of THF+H₂O mixtures at 25°C are shown in Table 2. The equivalent conductances at infinite dilution (Λ°) and the association constant (K_A) were calculated using the Fuoss conductance equation⁵.

$$\Lambda = P[\Lambda^\circ(1+RX) + EL] \quad (1)$$

$$P = [1 - \alpha(1 - \gamma)] \quad (2)$$

$$\gamma = 1 - K_A C \gamma^2 f^2 \quad (3)$$

$$-\ln f = \beta k/2(1 + kR) \quad (4)$$

$$\beta = e^2/DR T \quad (5)$$

where the terms have their usual significance. The parameters, K_A and R were obtained from the above equations by finding the values of Λ° and σ which minimize

$$\sigma^2 = \sum_j \left[\Lambda_j(\text{cal}) - \Lambda_j(\text{obs}) \right]^2 / (n-2) \quad (6)$$

for a sequence of R values. Initial Λ° value for the iteration procedure was taken from Shēdlōvsky extrapolations of the data.

A scan using unit increment of R values from 4 to 20, however, gave no significant minima in the $\sigma^2 - R$ curve for CsCl, NaBr, RbBr and CsBr at 0.273 mole fraction of THF, and also for the electrolytes (except LiCl) at 0.500 mole fraction of THF. The computations in these cases were carried out from arbitrarily presetting⁵ of R values at $R = a + d$. Here 'a' is the sum of the crystallographic radii of the ions and 'd' is the average distance corresponding to the side of a cell occupied by a solvent molecule. The distance 'd' is given by

$$d (\text{Å}) = \left(\frac{M}{N\rho} \right)^{1/3} = 1.183 \left(\frac{M}{\rho} \right)^{1/3} \quad (7)$$

where 'M' is the molecular weight of solvent and ρ is its density. For mixed solvents, 'M' is replaced by the mole fraction average molecular weight, M_{av} , which is given by :

$$M_{av} = \frac{M_1 M_2}{(W_1 M_2 + W_2 M_1)} \quad (8)$$

where W_1 is the weight fraction of the first component of molecular weight M_1 . Through, this is an over simplification which ignores possible selective solvation, it at least provides a self-consistent way to obtain an acceptable value for the parameter when a broad range of R values fit the data. The values of Λ° , K_A and R obtained by this procedure are recorded in Table 3.

From Table 1, we see that viscosity (η_o) of the solvent mixture (THF + H₂O) increases rapidly to a maximum at about 0.143 mole fraction or 40 Wt% of THF and thereafter decreases. Such characteristic in the viscosity vs. composition curve is a manifestation of strong specific interaction⁶ between unlike molecules predominated by hydrogen bonding interaction.

Table 3 shows that Λ° values for alkali metal halides increase as the size of the cation increases in any mole fraction of mixed solvent. However, for any particular electrolyte, Λ° continuously decreases with the addition of THF and Walden products are found to be different; viscosity of solvent-mixture does not coincide with the observed trend in Λ° value.

From Table 3, we also see that Λ° value of alkali metal salts of common anion follow the sequence : $Li^+ < Na^+ < K^+ < Rb^+ < Cs^+$. Further, the Λ° for the alkali metal bromides are greater than those for the corresponding alkali metal chlorides. For CsCl, the Λ° -value at 0.500 mole fraction of THF is in close proximity with the value reported by Justice et al⁴. The trend of variation of Λ° values also indicates the relative actual sizes of these ions

as they exist in solution. Thus, the sizes of these cations as they exist in solution follow the order : $\text{Li}^+ \rangle \text{Na}^+ \rangle \text{K}^+ \rangle \text{Rb}^+ \rangle \text{Cs}^+$ and for anions, $\text{Cl}^- \rangle \text{Br}^-$. This shows that Li^+ is the most solvated and Cs^+ is the least one in any mole fraction of THF.

It may be observed from Table 3 that the association constant, K_A , is very low for all the electrolytes at 0.059 mole fraction of THF. This indicates that these salts almost remain unassociated in this composition of the solvent mixtures having high dielectric constant. However, at higher mole fraction of THF ($\epsilon < 57.25$) association occurs in the solvent mixtures (for NaCl, KCl and CsCl ion association takes place above 0.143 mole fraction of THF) and that the K_A values increase with the increase of cationic as well as anionic sizes in all cases (except NaBr at 0.500 mole fraction of THF). Thus we see that CsBr inspite of its greater size is maximum associated than the other bromides at higher percentages of mixed solvents. Fuoss and Coworkers⁷ have found similar trends for many of the alkali metal halides in dioxane-water mixtures.

The values of Walden products ($\Lambda^\circ \eta_o$) for the studied electrolytes pass through a maximum at about 0.059 mole fraction of THF and then decrease continuously. A representative plot for the alkali metal bromides has been given in Fig. 1. Though the variation of the Walden product with solvent composition is difficult to interpret quantitatively, still its variation with solvent composition can be explained by preferential solvation^{8,9} of alkali metal ions by water and THF molecules respectively. At low mole-fraction of

THF, these ions are preferentially solvated by water than THF and the viscosity of the solvent in the vicinity of these ions is lower than that of the bulk solvent (Table 1). Since the bulk viscosity value is used in the calculation of Walden product, the calculated values of $\Lambda^{\circ}\eta_0$ are high upto the point corresponding to viscosity maximum of the solvent mixtures and then the values decrease gradually causing a maximum in the Walden product. The decrease in the Walden products after 0.143 mole-fraction of THF indicates the preferential solvation of alkali metal ions (having the same anion) by THF in THF + Water mixtures. However, this decrease in large part may be due to the Zwanzig¹⁰ solvent relaxation effect also.

Experimental :

Tetrahydrofuran (Merck, India) was kept several days over KOH, refluxed for 24h and distilled over LiAlH_4 . The boiling point (66°C), density (0.8807 g cm^{-3}) and viscosity ($\eta_0 = 0.0046 \text{ P}$) compared well with the literature values¹¹ . The specific conductance of THF was ca. $0.81 \times 10^{-6} \Omega^{-1} \text{ cm}^{-1}$ at 25°C .

Alkali metal Salts (Fluka) were of purum or puriss grade and purified as described earlier¹² . A stock solution for each salt ($\sim 0.1 \text{ M}$) in the appropriate solvent mixture was prepared by weight and the working solutions were obtained by weight dilution. The molar concentration of the solution was calculated from molality and density values. The mixed solvents of THF were prepared accu-

rately by mixing the requisite amounts of THF and water by weights.

Densities and viscosities of solvent mixtures were measured in an Ostwald-Sprengel type pycnometer and suspended-level Ubbelohde viscometer at $25 \pm 0.01^\circ\text{C}$ with a precision of $\pm 3 \times 10^{-5}$ g cm^{-3} and 0.05% respectively.

A Pye-Unicam conductivity meter (PW 9509) was used for measuring the conductances of the solutions at the frequency of 2000 Hz with a dip-type cell of cell constant 0.861 cm^{-1} and having an accuracy of $\pm 0.1\%$. The cell was calibrated with aqueous KCl solutions in the usual way. The measurements were carried out in a thermostatic oil bath maintained at $25 \pm 0.005^\circ\text{C}$. The details of experimental procedure have been described earlier¹³. Several independent solutions were prepared and runs were performed to ensure the reproducibilities of the results. All data were corrected at 25°C with the specific conductance of the solvent. The corrected values were analysed by means of the Fuoss conductance equation⁵.

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TABLE - 1

Physical Properties of THF-Water Mixtures at 25°C.

W%	x_2	ϵ	d_{20}^a	$10^2 \eta_{20}^b$	$10^6 L_{20}^c$
0	0	78.54	0.99707	0.8903	1.01
20	0.059	57.25	0.98668	1.4900	3.20
40	0.143	44.50	0.96640	1.7321	2.60
60	0.273	32.00	0.94600	1.4904	1.35
80	0.500	19.50	0.91592	0.9237	1.18
100	1.000	7.58	0.88072	0.4630	0.81

^aUnits : g cm^{-3} , ^bUnits : cP, ^cUnits : $\Omega^{-1} \text{cm}^{-1}$

TABLE - 2

Equivalent Conductances of Alkali^{metal} halides in THF + H₂O mixtures at 25°C
 [C in mol lit⁻¹ and \wedge in S cm² mole⁻¹]

10^4 C	\wedge	10^4 C	\wedge	10^4 C	\wedge
20% W/W (THF + H ₂ O) mixtures					
LiCl		NaCl		KCl	
394.900	52.70	400.000	69.08	511.142	81.91
345.500	53.16	300.000	70.44	432.505	82.85
296.100	53.60	200.000	72.16	353.867	83.84
246.700	54.10	100.000	72.92	275.230	84.95
197.400	54.70	80.000	74.33	196.593	86.31
148.000	55.45	60.000	75.50	58.977	90.45
93.600	56.20	40.000	76.32	39.318	91.20
493.300	57.20				

$10^4 C$	\wedge	$10^4 C$	\wedge	$10^4 C$	\wedge
	RbCl		CsCl		LiBr
427.883	81.50	579.900	80.99	480.046	58.49
356.569	83.1	483.200	82.62	364.834	59.02
285.255	84.72	418.800	84.01	307.229	59.80
213.941	86.43	322.100	85.93	249.623	60.92
142.627	88.62	225.500	88.31	192.018	62.07
71.313	91.43	161.000	90.18	134.412	63.22
35.656	93.50	96.600	92.43	76.807	64.83
16.000	94.93	64.400	93.87	43.640	65.99
	NaBr		KBr		RbBr
448.103	64.44	420.534	89.96	405.012	82.63
377.350	66.60	319.606	91.75	324.010	85.20
306.597	68.82	269.141	92.46	275.408	87.03
235.844	71.41	218.677	93.55	226.807	88.82

Contd.... (3)

$10^4 C$	\wedge	$10^4 C$	\wedge	$10^4 C$	\wedge
	NaBr		KBr		RbBr
165.090	74.62	168.213	94.60	178.205	90.98
94.337	78.25	117.749	95.92	129.604	93.39
23.584	84.22	67.285	97.61	81.006	96.19
		33.642	99.02	32.401	100.25
	CsBr				
465.377	86.95				
353.686	89.82				
297.841	91.48				
241.996	93.42				
186.151	95.40				
130.305	97.78				
74.460	100.82				
18.615	105.32				

$10^4 C$	^	$10^4 C$	^	$10^4 C$	^
40% W/W (THF + H ₂ O) mixtures					
	LiCl		NaCl		KCl
289.000	36.30	387.100	47.84	205.424	60.31
256.000	37.00	290.300	49.47	179.746	61.17
200.000	38.42	193.500	51.12	154.068	61.92
150.000	39.70	145.100	52.29	128.390	62.83
121.000	40.50	96.700	53.40	102.712	63.83
100.000	41.24	72.500	54.24	77.034	64.92
64.000	42.65	48.300	55.15	51.356	66.27
50.000	43.48	24.100	56.32	25.678	67.97
	RbCl		CsCl		LiBr
225.269	60.12	422.843	60.46	235.210	42.62
197.11	61.16	321.360	62.35	178.759	44.44
168.952	62.10	270.619	63.40	150.534	45.46
140.793	63.21	219.878	64.53	122.309	46.39

Contd.....(5)

$10^4 C$	^	$10^4 C$	^	$10^4 C$	^
	RbCl		CsCl		LiBr
112.634	64.42	169.137	65.82	94.084	47.49
84.476	66.02	118.396	67.31	65.859	48.98
56.317	67.51	67.655	69.15	47.042	50.00
28.158	69.69	16.913	71.95	18.817	52.02
	NaBr		KBr		RbBr
248.012	44.22	250.550	57.62	166.048	60.52
188.489	46.80	190.418	60.20	139.830	61.99
158.727	48.53	160.352	61.51	113.612	63.68
128.966	50.24	130.286	62.96	87.394	65.72
99.205	52.01	100.220	64.59	61.176	67.82
69.443	54.27	70.154	66.77	34.957	70.43
39.682	57.02	40.088	69.02	8.739	74.81
9.920	61.45	10.022	72.82		

Contd.....(6)

10^4C	\wedge	10^4C	10^4C
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CsBr

216.841	59.02
164.799	62.04
138.778	63.65
112.757	65.23
86.736	67.22
60.715	69.60
34.695	72.44
8.673	76.65

$10^4 C$	\wedge	$10^4 C$	\wedge	$10^4 C$	\wedge
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60% W/W (THF + H₂O) Mixtures

LiCl		NaCl		KCl	
158.800	31.53	78.900	37.76	174.704	39.70
100.000	33.50	69.100	38.20	97.834	43.09
64.000	34.95	59.200	38.68	83.858	43.79
30.000	36.95	49.300	39.37	69.881	44.59
19.500	37.60	39.400	40.04	55.905	45.40
16.000	37.92	29.600	40.70	41.929	46.45
9.000	38.66	19.700	41.73	34.941	47.09
5.500	39.27	9.870	42.86	27.952	47.63
				6.988	50.20
RbCl		CsCl		LiBr	
96.981	43.47	131.794	41.85	102.233	39.87
62.068	45.92	79.076	44.82	77.697	41.01

Contd... (8)

$10^4 C$	\wedge	$10^4 C$	\wedge	$10^4 C$	\wedge
	RbCl		CsCl		LiBr
54.309	46.60	68.532	45.62	65.429	41.69
38.792	47.92	47.445	47.45	53.161	42.25
31.034	48.81	36.902	48.43	40.893	43.01
23.275	49.73	26.358	49.50	28.625	43.83
15.517	50.94	15.815	51.22	16.357	45.04
7.758	52.45	5.271	53.12	4.089	46.72
	NaBr		KBr		RbBr
111.719	30.55	119.746	44.57	118.043	41.25
81.927	33.68	86.217	47.22	84.991	44.83
59.583	36.48	67.058	48.92	70.826	46.82
44.688	38.70	52.688	50.42	56.660	48.99
29.791	41.25	43.108	51.64	42.495	51.45
14.896	44.74	28.739	53.25	28.330	54.02

Contd.....(9)

$10^4 C$	\wedge	$10^4 C$	\wedge	$10^4 C$	\wedge
	NaBr		KBr		RbBr
7.448	46.95	14.369	55.80	14.165	57.85
		4.789	58.56	4.721	61.43
	CsBr				
101.442	44.41				
77.096	47.55				
64.923	49.02				
52.750	51.05				
40.577	53.02				
31.212	55.05				
16.230	58.63				
4.057	63.25				

$10^4 C$	^	$10^4 C$	^	$10^4 C$	^
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80% W/W (THF + H₂O) mixtures

	LiCl		NaCl		KCl
300.000	11.10	46.900	23.75	21.781	31.50
169.000	15.30	39.600	24.37	13.939	34.12
100.000	18.25	32.400	25.56	12.197	34.87
90.000	18.80	25.200	27.06	10.454	35.50
80.000	19.25	18.000	28.62	8.712	36.35
60.000	20.40	10.800	30.62	6.969	37.12
25.000	23.00	3.600	33.62	5.227	38.25
10.000	24.80			3.484	39.50
	RbCl		CsCl		LiBr
33.352	24.60	17.928	30.25	79.896	23.51
20.011	29.40	14.668	32.12	22.370	32.01

Contd.....(11)

$10^4 C$	\wedge	$10^4 C$	\wedge	$10^4 C$	\wedge
	RbCl		CsCl		LiBr
17.343	30.70	11.409	34.06	19.175	32.78
14.675	32.00	8.149	36.50	15.979	33.45
12.007	33.40	4.889	39.50	12.783	35.59
9.338	35.00	1.629	44.62	9.587	35.79
6.670	37.00			6.391	36.52
1.334	42.60				
	NaBr		KBr		RbBr
45.066	30.83	21.620	32.01	21.931	31.02
34.250	33.25	16.431	34.88	16.667	34.47
25.237	35.64	13.836	36.02	14.036	35.56
18.026	37.82	11.242	38.17	11.404	37.82
12.618	40.19	8.648	39.44	8.772	40.26
9.013	41.23	6.053	41.83	6.140	42.69

Contd.....(12)

$10^4 C$	^	$10^4 C$	^	$10^4 C$	^
	NaBr		KBr		RbBr
3.605	44.25	3.459	44.20	3.509	45.37
1.802	46.23	0.864	48.22	0.877	49.95
	CsBr				
20.974	29.03				
18.457	31.01				
15.101	33.40				
11.745	35.86				
8.387	39.44				
5.033	43.02				
1.677	49.05				

TABLE - 3

Derived Conductance Parameters

X_2	\wedge°	K_A	R	$\wedge^\circ \eta_0$	σ
Lithium Chloride					
0 ^a	115.12 ±0.01	0.79	5.19	1.025	0.03
0.059	60.88 ±0.06	1.90 ±0.06	11.41	0.907	0.06
0.143	48.57 ±0.08	13.92 ±0.03	12.83	0.841	0.06
0.273	41.38 ±0.04	20.60 ±0.40	16.00	0.617	0.07
0.500	30.40 ±0.17	102.74 ±0.50	13.20	0.281	0.19
Sodium Chloride					
0 ^a	126.53 ±0.01	0.81	5.56	1.126	0.02
0.059	80.44 ±0.023	2.96 ±0.03	10.66	1.198	0.03
0.143	59.76 ±0.04	6.32 ±0.09	11.26	1.035	0.05
0.273	46.01 ±0.05	25.40 ±0.51	15.27	0.686	0.05
0.500	39.04 ±0.49	206.53 ±0.01	7.14	0.361	0.36

Contd.....(1)

x_2	Δ°	K_A	R	$\Delta^\circ \eta_0$	σ
Potassium Chloride					
0 ^a	149.90 ±0.01	0.56	4.65	1.334	0.02
0.059	96.10 ±0.04	2.09 ±0.05	5.99	1.432	0.83
0.143	72.18 ±0.03	9.51 ±0.06	13.00	1.250	0.03
0.273	53.04 ±0.05	24.11 ±0.30	16.50	0.790	0.05
0.500	44.92 ±0.30	228.55 ±74.80	7.43	0.415	0.90
Rubidium Chloride					
0 ^a	153.64 ±0.01	0.26	3.35	1.367	0.02
0.059	98.19 ±0.05	4.67 ±0.04	11.00	1.463	0.07
0.143	74.61 ±0.07	12.35 ±0.16	13.00	1.292	0.07
0.273	55.86 ±0.04	35.64 ±0.34	15.44	0.833	0.04
0.500	46.84 ±0.18	458.73 ±75.29	7.28	0.432	0.94

Contd.....(2)

X_2	Δ°	K_A	R	$\Delta^\circ \eta_0$	σ
Cesium Chloride					
0^a	153.04 \pm 0.02	0.62	3.62	1.362	0.02
0.059	100.42 \pm 0.04	4.52 \pm 0.03	10.00	1.496	0.05
0.143	75.33 \pm 0.05	6.41 \pm 0.07	11.40	1.305	0.06
0.273	56.51 \pm 0.21	31.19 \pm 1.70	7.32	0.842	0.25
0.500	50.89 \pm 0.46	649.80 \pm 36.29	7.84	0.470	0.29
Lithium Bromide					
0^b	116.88	-	-	1.040	-
0.059	70.53 \pm 0.22	2.89 \pm 0.25	6.02	1.051	0.25
0.143	55.35 \pm 0.06	14.86 \pm 0.19	16.60	0.958	0.06
0.273	48.70 \pm 0.04	20.35 \pm 0.30	17.70	0.726	0.05
0.500	45.93 \pm 1.20	265.79 \pm 52.73	7.06	0.424	0.75

Contd.....(3)

x_2	Δ°	A	K_A	R	$\Delta^\circ \eta_0$	σ
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Sodium Bromide

0^b	128.42	-	-	-	1.143	-
0.059	88.82 ± 0.10	11.70 ± 0.11	13.50	1.323	0.10	
0.143	64.57 ± 0.11	28.32 ± 0.41	19.00	1.118	0.12	
0.273	53.33 ± 0.81	120.08 ± 12.00	6.82	0.795	0.65	
0.500	50.94 ± 0.69	224.28 ± 27.60	7.30	0.470	0.73	

Potassium Bromide

0^b	157.77	-	-	-	1.405	-
0.059	103.35 ± 0.06	2.72 ± 0.04	10.80	1.540	0.07	
0.143	75.92 ± 0.08	17.10 ± 0.20	14.20	1.315	0.09	
0.273	61.39 ± 0.11	45.87 ± 0.84	19.00	0.915	0.13	
0.500	52.26 ± 0.66	440.92 ± 42.10	7.62	0.483	0.62	

Contd.....(4)

x_2	Δ°	K_A	R	$\Delta\eta_0$	σ
Rubidium Bromide					
0 ^b	156.37	-	-	1.392	-
0.059	105.98 ±0.06	8.79 ±0.05	11.60	1.579	0.06
0.143	78.03 ±0.11	22.59 ±0.32	13.00	1.352	0.11
0.273	66.56 ±0.65	90.09 ±0.36	7.29	0.992	0.64
0.500	54.95 ±1.03	593.02 ±71.48	7.77	0.507	0.90
Cesium Bromide					
0 ^b	155.51	-	-	1.384	-
0.059	109.41 ±0.04	6.67 ±0.03	9.90	1.630	0.04
0.143	79.83 ±0.08	23.13 ±0.23	17.30	1.383	0.90
0.273	67.75 ±0.05	82.58 ±4.80	7.48	1.010	0.50
0.500	58.54 ±1.71	1001.53 ±140.56	7.96	0.541	-0.98

^a Ref. 9 ^b R.L. KAY and D.F. EVANS, J. Phys. Chem., 1966, 70, 2325.

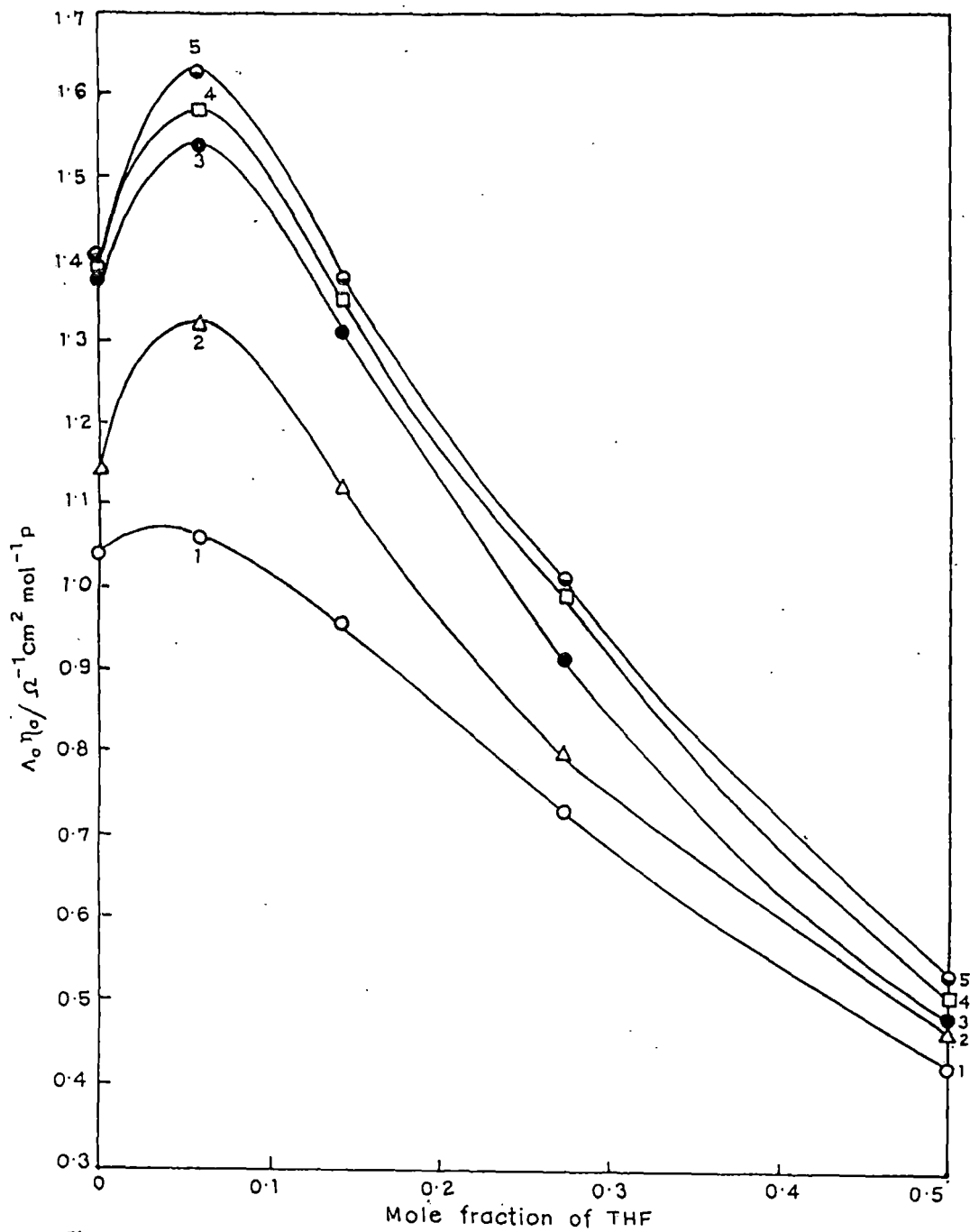


Fig. 1. Variation of Walden products ($\Lambda_0 \eta_0$) with compositions of solvent mixtures: (1) LiBr, (2) NaBr, (3) KBr, (4) RbBr, and (5) CsBr.