Greek Letters

ϵ = low-frequency dielectric constant

ϵ_∞ = high-frequency dielectric constant (approximated as $1.1 \times n_D{}^2$)

η = viscosity, cP

ρ = density, g/cm^3

μ_0 = dipole moment, Debye units [which are (esu-cm)^2]

α, β, γ, δ = constants in Equations 4 and 5

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Heats of Dilution of Lithium Perchlorate in Anhydrous Acetonitrile, Propionitrile, and Isobutyronitrile at 25 °C

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Heats of dilution of lithium perchlorate in anhydrous acetonitrile, propionitrile, and isobutyronitrile at 25 °C using solution and dilution calorimeters are reported. The results indicate that electrostatic treatments do not consistently predict the behavior of these systems even in the millimolar range, probably because of ion association.

Two recent surveys of thermochemical data for nonaqueous electrolytes (*3, 6*) indicate that determinations of heats of dilution have been confined largely to alcoholic solvents. It is of interest to examine the predictions of the Debye-Hückel model in a negligibly hydrogen-bonded solvent from the point of view of heats of dilution.

Heats of dilution data are reported for lithium perchlorate in acetonitrile, propionitrile, butyronitrile, and isobutyronitrile at 25 °C.

Experimental

Acetonitrile. Fisher ACS reagent grade acetonitrile was purified by a method reported previously (*4, 13*). Alternatively, a purer grade of acetonitrile was used as a starting material (MCB Spectroquality) and was of high purity as received (*2*). The only treatment required was a 6-h period standing over calcium hydride followed by a high vacuum room temperature distillation to remove residual water and dissolved air. After degassing, the void in the flask was refilled with dry nitrogen (Linde) to a pressure of 350–400 torr before warming to room temperature.

An analysis by the Karl Fischer method using the Photovolt "Aquatest II" titrator showed a water content of 30–40 ppm

immediately after distillation and 50–60 ppm after standing in a stoppered flask in the drybox for 24 h. A blank titration showed that 30 ppm was close to the lower limit of detection for this instrument. Therefore, nitriles treated with calcium hydride were considered to have a water content between 0 and 10 ppm if used very soon after distillation.

Propionitrile. Propionitrile was far more sensitive to acid-catalyzed polymerization than acetonitrile, and heating with phosphorus pentoxide was not an efficient method of drying. Instead, propionitrile [Eastman Organic Chemicals or MCB (99% purity)] was first dried in 1-l. batches by standing for 5 days over dried silica gel (Tel-Tale, Davison Chemical Co.), followed by standing over ~30 g phosphorus pentoxide for 2 days. A small amount of polymerization was noted at this point. Drying was completed by refluxing over calcium hydride, followed by fractionation and degassing as described for acetonitrile, except that a 2-ft column with glass packing was used for fractionation. This permitted distillation to be carried out in a fume hood, as the higher nitriles are noticeably toxic (*11*).

The butyronitriles (Eastman Organic Chemicals) were purified by the same procedure as propionitrile.

Lithium perchlorate. Lithium perchlorate (K & K Laboratories) contained approximately 70 ppm halide by potentiometric titration with aqueous silver nitrate and a small amount of unidentified tan-colored, water-insoluble matter. Both of these were removed to within the limits of detection (10 ppm for halide, visibility on sintered glass filter for other matter) by a single recrystallization from water.

Differential thermal analysis of dried lithium perchlorate indicated a melting point range (235–237 °C) above which temperature no salient features appeared on the thermogram

until decomposition commenced at 420–440 °C. These features are important in obtaining a suitably dry salt, since it should not be heated too long near its decomposition point, to avoid further accumulation of oxide and chloride. It is also preferable in this case to conduct most of the drying of the powder well below the melting point to avoid extensive manipulations of crushing and grinding the caked solid prior to filling ampules, when traces of water might be picked up from the drybox atmosphere.

The solid was therefore placed in a covered porcelain dish in a vacuum oven (Thelco Model 29), and the pressure reduced to ~1 torr. The oven temperature was maintained at 100 °C until no further water was collected from the cold trap. The temperature was then raised to 120 °C and held constant for a minimum of 48 h. Glass vessels were deeply etched by the salt at this stage. Pyrex cover glasses were used, but scraping spattered salt off the covers into the main sample is not recommended.

The oven was cooled under vacuum, and the salt removed and crushed as quickly as possible. It was transferred to a vacuum desiccator containing phosphorus pentoxide, evacuated to 0.01 torr, and kept for at least 10 days before unloading into the drybox. Phosphorus pentoxide for this purpose is considered best if slightly wet, though not so much as to be visibly gummy. This causes some coherence so that powder cannot fly up and contaminate the sample during evacuation.

Calorimeters. An LKB 8700 solution calorimeter (LKB Produkter AB, Stockholm-Bromma 1, Sweden) was used for measurements in the four nitriles. Additional measurements in acetonitrile and propionitrile were performed using an LKB 10700 batch microcalorimeter.

The detection system for the LKB 10700 consists of a Keithley Model 150B microvolt amplifier coupled to a Sargent constant-speed integrating chart recorder. A chart speed of 1 in./min gave legibility better than 0.1% for a 50% full scale deflection.

Procedure

LKB 10700. A description of the LKB 10700 batch microcalorimeter has been given recently by Wood and Falcone (14).

The reaction vessel was filled by weight difference using disposable polyethylene syringes as weighing vessels. Each syringe was fitted with a short length (3–6 in.) of 0.3 mm i.d. Teflon tubing which facilitated injection of sample through the filler ports in the reaction vessel. Stock solutions were stored in glass vials and withdrawn through rubber serum caps using the syringes. Losses due to evaporation and wetting of the stock solutions by laboratory air were minimized by drawing the solution up into the syringe needle before disconnecting from the vial.

Successive dilutions were accomplished by removing the solution from one side of the vessel, again using a weighed syringe, and calculating the mass remaining in the other side of the vessel from the total mass removed. Fresh solvent was then added in the same manner, and the sequence continued.

Dilution runs were commenced about 1–2 h after filling the reaction vessel or when a stationary temperature was obtained.

LKB 8700. Ampules were filled under nitrogen. The ampules are machine-blown with thin walls and quite narrow necks, and the following procedure was used in filling them. Dried salt, solvent, and ampules were transferred into the drybox in small desiccators, and the drybox was flushed with dry nitrogen for 30 min. Concentrated stock solutions were prepared by weight. The ampules were weighed and filled using a Pasteur pipet. The filled ampules were weighed, re-

turned to the desiccator, and removed from the drybox for sealing with the dual torch provided with the calorimeter. Sealing was done as quickly as possible to minimize exposure to the atmosphere.

The equilibrium temperature of the calorimeter with the stirrer running was noted in terms of the resistance of the thermistor and the temperature change expected calculated from an earlier rough run. The reaction vessel temperature was then adjusted so that the temperature after the reaction would be as close as possible to the equilibrium temperature. For exothermic reactions the vessel was cooled by placing a drop of pure acetone on the outside wall and passing nitrogen through the bomb until the acetone had all evaporated. Impure acetone and incomplete evaporation before the experiment will lead to significant errors. The calculated interval was always overshot so that cooling curves could be recorded before the experiment, and a brief interval (about 5 min) was allowed before commencement of the cooling curve so that a uniform temperature gradient could be established between the sample and the thermostat.

Temperatures were recorded at intervals of about 30 s for 5–10 min before reaction, which was initiated by breaking the ampule, and for at least 10 min after the reaction appeared to have stopped. Points during reaction were measured as close as possible to the mean temperature. It was most convenient to preset the bridge to the expected mean temperature before breaking the ampule because this temperature was reached in only 5–10 s.

Table I. Coefficients Obtained from Least-Squares Fit

Equation	A_0	A_1	A_2	Δ
		Acetonitrile		
	Set 1: LKB 10700, m_1 = 1.1718, 9 dilutions			
2	2438 ± 70	−3896 ± 430	1595 ± 500	50
	Set 2: LKB 8700, m_1 = 0.8068, 3 dilutions			
1	1184 ± 5	−1513 ± 44	. . .	2
	Set 3: LKB 8700, m_1 = 0.1626, 6 dilutions			
1	759 ± 32	−3890 ± 492	. . .	20
	Final equation: 21 points			
4	−53 ± 41	3959 ± 262	−1560 ± 253	100
		Propionitrile		
	Set 1: LKB 10700, m_1 = 2.167, 6 dilutions			
2	2219 ± 21	−3017 ± 112	1452 ± 108	10
	Set 2: LKB 8700, m_1 = 1.6715, 8 dilutions			
1	1850 ± 72	−2578 ± 486	. . .	50
	Set 3: LKB 8700, m_1 = 0.6040, 7 dilutions			
1	1425 ± 44	−1867 ± 579	. . .	30
	Set 4: LKB 8700, m_1 = 0.2224, 9 dilutions			
1	1472 ± 218	−6554 ± 4101	. . .	190
	Final equation: 28 points			
4	3 ± 24	2451 ± 140	−708 ± 103	70
		Butyronitrile		
	Set 1: LKB 8700, m_1 = 0.8633, 8 dilutions			
1	1607 ± 518	−3821 ± 4229	. . .	370
	Set 2: LKB 8700, m_1 = 0.1884, 5 dilutions			
1	1195 ± 287	−2987 ± 5493	. . .	190
	Set 3: LKB 8700, m_1 = 0.1342, 3 dilutions			
1	1224 ± 55	−3118 ± 1236	. . .	20
	Final equation: 18 points			
4	89 ± 77	2094 ± 462	5 ± 276	220
		Isobutyronitrile		
	Set 1: LKB 8700, m_1 = 1.2804, 7 dilutions			
1	2188 ± 267	−4928 ± 1989	. . .	160
	Set 2: LKB 8700, m_1 = 0.1924, 10 dilutions			
1	1370 ± 204	−6219 ± 3229	. . .	160
	Final equation: 14 points			
4	156 ± 66	3631 ± 570	−1628 ± 485	130

Electrical calibrations were treated in the same way as exothermic heat measurements. Results (for the LKB 8700 calorimeter) were analyzed graphically, using the method of Dickinson (12), by finding the vertical distance between initial and final cooling curves extrapolated to times when 63% (for dilutions) or 50% (for calibrations) of the heat evolution had occurred. Thermistor resistances were plotted directly against time and not converted to temperatures, because electrical calibrations were always arranged to cover the same temperature range as the heat measurements which they accompanied. The error due to nonlinearity of the thermistor resistance with temperature is thus insignificant.

Results

The data were analyzed by the method described by Harned and Owen (5) using a PL/1 least-squares curve fitting program. This program generated values of the coefficients of the equations, their standard deviations, and the overall standard deviation of the estimate. The heats of dilution from the LKB 8700 calorimeter were fitted to the linear equation

$$-\Delta H_{DIL} = A_0 + A_1 m_2^{1/2} \qquad (1)$$

where $-\Delta H_{DIL}$ is the molar heat of dilution from m_1 to m_2; data were grouped by their m_1 values, and groups were fitted separately. Similar fits were made for the LKB 10700 data, except that the larger range of final concentrations covered required an equation of the form:

$$-\Delta H_{DIL} = A_0 + A_1 m_2^{1/2} + A_2 m_2 \qquad (2)$$

From the relation:

$$\phi_L(m_2) = \phi_L(m_1) - \Delta H_{DIL} \qquad (3)$$

it follows that the A_0 in Equations 1 and 2 are the ϕ_L values for the concentration m_1. Thus, provisional or "observed" ϕ_L values were found by difference using Equation 3 for each experimental concentration. These values were smoothed by combining all the observed ϕ_L values in a fit to the equations:

$$\phi_L = A_0 + A_1 m^{1/2} + A_2 m \qquad (4)$$

$$\phi_L = A_0 + A_1 m^{1/2} + A_2 m + A_3 m^{3/2} \qquad (5)$$

$$\phi_L = A_0 + A_1 m^{1/2} + A_2 m + A_3 m^{3/2} + A_4 m^2 \qquad (6)$$

Values which were consistently very high or low were rejected, and a final fit was made to Equation 4 to obtain smoothed ("calculated") values. Coefficients of Equations 1, 2, and 4 are given in Table I. The A_0 values of Equation 4 should be zero, but on combining data a finite value may appear which is expected to be similar in magnitude to the experimental uncertainty as measured by the standard deviation of fit. A_0 was set equal to zero when calculating the smoothed values. Heats of dilution and the observed and smoothed ϕ_L values are given in Tables II–V for the various solvents. The estimated accuracy of these values is about 100 cal mol^{-1}.

Discussion

The water content of the solvents investigated in this study is of the order of 10^{-3} m. In weakly basic solvents like the nitriles, it is to be expected that the salt–water interactions will control the value of ϕ_L when the salt and water concentrations become comparable. The lower concentration limit is quite high and will not allow examination of the linearity of ϕ_L with $m^{1/2}$ at limiting concentrations. However, it seems rea-

Table II. Heats of Dilution in Acetonitrile

m_1	m_2	$-\Delta H_{DIL}$, cal/mol	$\phi_L(m_2)$,[a] cal/mol Obsd	Calcd
	0.9478		2280	2380
0.9478	0.3165	588	1710	1730
	Set 1			
	1.1718		2440	2460
1.1718	0.5607	453	1990	2090
1.1718	0.4378	515	1920	1940
0.5607	0.1830	602	1380	1410
0.4378	0.1559	604	1320	1320
0.1559	0.0534	605	710	830
0.1830	0.0362	693	690	700
0.0534	0.0304	96	620	640
0.0362	0.0152	182	510	460
0.0304	0.0142	169	450	450
	Set 2			
	0.8068		(1180)	
0.8068	0.01814	980	200	510
	0.01138	1025	160	410
	0.00622	1065	120	300
	Set 3			
	0.1626		(760)	
0.1626	0.00709	434	330	320
	0.00594	453	310	300
	0.00479	484	280	270
	0.00364	517	240	230
	0.00270	589	170	200
	0.00139	598	160	150

[a] From Equations 1, 2, and 4.

Table III. Heats of Dilution in Propionitrile

m_1	m_2	$-\Delta H_{DIL}$, cal/mol	$\phi_L(m_2)$,[a] cal/mol Obsd	Calcd
	Set 1			
	2.167		2220	2070
2.167	0.7796	696	1520	1610
2.167	0.6573	714	1510	1520
0.6573	0.2033	452	1050	960
0.2033	0.0782	310	740	630
0.0782	0.0320	249	490	420
0.0320	0.0130	172	320	270
	Set 2			
	1.6715		1850	1980
1.6715	0.03776	1390	460	450
	0.03165	1335	520	410
	0.03049	1440	410	410
	0.02391	1393	460	360
	0.02359	1486	360	360
	0.01601	1468	380	300
	0.01544	1564	290	290
	0.00790	1590	260	210
	0.00786	1676	170	210
	Set 3			
	0.6040		1430	1480
0.6040	0.00895	1267	160	230
	0.00888	1248	180	220
	0.00674	1229	200	200
	0.00604	1304	120	190
	0.00432	1291	130	160
	0.00311	1341	80	130
	0.00231	1329	100	120
	Set 4			
	0.2224		(1470)	
0.2224	0.00569	910	(560)	
	0.00494	936	(540)	
	0.00402	985	(490)	
	0.00307	1354	120	130
	0.00224	1051	(420)	
	0.00200	1420	50	110
	0.00146	1068	(400)	
	0.00123	1453	20	90
	0.00069	1081	(390)	

[a] From Equations 1, 2, and 4.

Table IV. Heats of Dilution in Butyronitrile

m_1	m_2	$-\Delta H_{DIL}$, cal/mol	$\phi_L(m_2)$,[a] cal/mol Obsd	Calcd
3.0110	3.0110		3830	3650
	0.00633	3526	(10)	
Set 1				
0.8633	0.8633		1610	1950
	0.02667	932	680	340
	0.02148	1287	320	310
	0.01911	919	690	290
	0.01679	1354	250	270
	0.01439	894	(710)	
	0.01047	1493	110	220
	0.00747	648	(960)	
	0.00420	1707	(−100)	
Set 2				
0.1884	0.1884		1200	910
	0.00522	936	260	150
	0.00400	994	200	130
	0.00303	1112	80	120
	0.00178	1189	10	90
	0.00127	774	420	80
	0.00109	1267	−70	70
Set 3				
0.1342	0.1342		1220	770
	0.00292	1046	180	210
	0.00200	1101	120	90
	0.00096	1121	100	70

[a] From Equations 1, 2, and 4.

Table V. Heats of Dilution in Isobutyronitrile

m_1	m_2	$-\Delta H_{DIL}$, cal/mol	$\phi_L(m_2)$,[a] cal/mol Obsd	Calcd
Set 1				
1.2804	1.2804		2190	2020
	0.02912	1471	720	570
	0.02697	1249	(940)	
	0.02141	1571	620	500
	0.01882	1349	840	470
	0.01396	1716	470	410
	0.00890	1543	650	330
	0.00703	1909	(280)	
Set 2				
0.1924	0.1924		1370	1280
	0.00677	1009	360	290
	0.00639	675	(700)	
	0.00529	1022	350	260
	0.00510	787	580	250
	0.00409	1050	320	230
	0.00399	916	450	220
	0.00274	1203	(170)	
	0.00272	1057	310	190
	0.00142	901	(470)	
	0.00130	1278	90	130

[a] From Equations 1, 2, and 4.

sonable to regard the coefficient A_1 of Equation 4 as a good approximation to the limiting slope.

Calculated and observed limiting slopes are summarized in Table VI. The data for methanol and ethanol also terminate at fairly high concentrations, and the extrapolation is therefore uncertain. The apparently good agreement of observed and calculated values for acetonitrile may be a coincidence since there is some disagreement over the dielectric constant of acetonitrile (6). Propionitrile is much better characterized in this respect. The calculated values for the butyronitriles must be regarded as rough estimates only, since the temperature dependences of the dielectric constants are not available and were estimated by comparison with data for related compounds (6). Young and Seligmann (15) found a limiting slope of 480 ± 88 cal $l.^{1/2}$ mol$^{-3/2}$ for 11 alkali salts in water, an agreement of 10–15% with the calculated value of 537 cal $l.^{1/2}$ mol$^{-3/2}$. The poorer agreement found for the nitriles (where it is not caused by the need to estimate physical properties of the solvent) could be caused by any of several factors, chiefly the influence of traces of water and ion association.

$NaClO_4$, which has a larger association constant (7) than does $LiClO_4$ (1), seems to have a smaller limiting slope of about 1900 cal kg$^{1/2}$ mol$^{-3/2}$ (8). It is likely that both ion association and solvent ordering make significant contributions to ϕ_L down to the lowest accessible concentrations. This would account for the similar values obtained for the four nitriles and possibly for the low values obtained in methanol and ethanol.

Acknowledgment

Table VI. Calculated and Observed Limiting Slopes for Heats of Dilution of 1:1 Salts at 25 °C

Solvent	Limiting slope, cal kg$^{1/2}$ mol$^{-3/2}$ Calcd	Obsd
Water[a]	517	420
Methanol[b]	4283	2726
Ethanol[b]	7220	4590
Acetonitrile	2789	3960 ± 260
Propionitrile	75	2450 ± 140
Butyronitrile	6937	2090 ± 460
Isobutyronitrile	6289	3630 ± 570

[a] Ref. 9. [b] Ref. 10.

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