J. Chem. Eng. Data **1996,** *41,* 231−234

Density, Viscosity, and Electrical Conductivity of x CH_3CONH_2 + $(1 - x)$ $Ca(NO_3)_2 \cdot 4.37H_2O$ from 253.15 K to 348.15 K

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Density, viscosity, and electrical conductivity of x $CH_3CONH_2 + (1 - x)$ $Ca(NO_3)_2 \cdot 4.37H_2O$ systems were measured as functions of temperature ($253.15 \leq T/K \leq 348.15$) and composition ($x = 0.0-0.9188$). Densities and molar volumes show a linear dependence on temperature. Molar volumes and intrinsic volume were found to be additive in nature and intrinsic volume was found to predominantly govern the linear variation of molar volume with composition. The temperature dependence of viscosity and conductivity has been analyzed using the Vogel−Tammann−Fulcher (VTF) equation. The composition dependence of the viscosity and electrical conductivity is primarily governed by the solvated polymeric type ion.

Introduction

Recently attention has been directed to the development of room temperature molten salt electrolytes (Chum and Osteryoung, 1981; Hussey, 1983; Gale and Osteryoung, 1983; Osteryoung, 1987) for a variety of economic applications as conducting solvents for fuel cells and thermal energy storage devices and as media for chemical and electrochemical processing. The majority of research has concentrated on the chloroaluminate melts containing alkylpyridinium, alkylimidazolinium, quaternary ammonium, or carboxylate salts as solvents. The use of chloroaluminate melts, with their relatively low melting points and good acid−base properties have been reviewed recently (McManis et al., 1985).

Molten acetamide−electrolyte binary systems are of considerable interest for energy storage devices owing to their ability to show strong supercooling. They show a large viscosity and high dielectric constant (Berchiesi et al., 1992). Studies on this class of liquid systems involve viscoelastic and ultrasonic relaxation (Berchiesi et al., 1989) at the eutectic composition. Acetamide−calcium nitrate tetrahydrate forms a homogeneous room temperature liquid with very pronounced supercooling (Tripković et al., 1989), and the system has potential application as a reactive medium (Berchiesi et al., 1989). It appears from the published literature that no attempt has yet been made to study the transport properties of acetamide−electrolyte binary systems as a function of composition and temperature. However, Berchiesi et al. (1989) reported the density and viscosity of 0.7644 CH_3CONH_2 + 0.2356 $Ca(NO_3)_2$ in the temperature range 288.15 K to 363.15 K.

In the present paper we report density, viscosity, and electrical conductivity of acetamide−calcium nitrate tetrahydrate melts as a function of temperature and composition.

Experimental Section

Materials. Acetamide (SD, LR grade) was recrystallized twice from hot methanol, and it was subsequently treated with ethyl ether (Perrin et al., 1981). Final drying was made in a vacuum desiccator over P_2O_5. Calcium nitrate tetrahydrate (SM, GR grade) with assay >99.5% was used without further purification. The exact number of moles of water per mole of calcium nitrate was determined through an EDTA titration method (Vogel, 1969) as well as by density comparison with the reported value (Ewing and Mikovsky, 1950) at 323.15 K and corrected as (4.37 ± 0.01) mol.

Preparation of Samples. Appropriate quantities of acetamide and calcium nitrate tetrahydrate were melted at ≃333.15 K in a screw-capped glass tube. All concentrations are expressed in mole fraction.

Density Measurements. Densities (ρ) were measured with a single-stem graduated and precalibrated pycnometer (≃9 cm^3) provided with a well-fitted glass stopper to prevent evaporation. The difference between the duplicate density values of all melt systems was found to be less than ±0.01%.

Viscosity Measurements. The viscosities (η) were measured by using a Schott-Geräte AVS 310 viscosity measuring system equipped with a precalibrated Ubbelohde viscometer (cell constant 0.3099 $mm^2\ s^{-2}$). Measurement of flow times of the melts was generally performed at least on four replicates for each composition and at each temperature, and the results were averaged. The experimental reproducibility between duplicate viscosity values of all melts was with in ±0.4%.

Specific Conductivity. Electrical conductivity (κ) measurements were made by a Wayne Kerr 6425 precision component analyzer operating in the 2.5×10^{-9} to 10 S range with a sensitivity of ±0.01 nS and employing a platinized platinum electrode (cell constant 1.095 cm^{-1}) with a field frequency of 1.5 kHz. The melt was put in a glass tube with a ground joint, and the measuring cell was well fitted to prevent diffusion of moisture. The sample tube was purged with dry nitrogen gas, and the sample was filled under a nitrogen atmosphere.

Temperature Control. All the measurements were made in either a Schott-Geräte CT 1450 or a Lauda RLS6D thermostat which maintained the temperature of the study to ±0.02 K.

Results and Discussion

The measured densities (ρ) of x $CH_3CONH_2 + (1 - x)$ $Ca(NO_3)_2 \cdot 4.37H_2O$ are presented in Table 1 and are found to be a linear function of temperature (Table 2) at a fixed composition. The measured density value of 0.7409 CH_3CONH_2 + 0.2591 $Ca(NO_3)_2 \cdot 4.37\ H_2O$ is found to be around 4.1% less than that reported by Berchiesi et al. (1989). This may be attributed to a difference in the hydrous and anhydrous calcium nitrate used in the two different systems.

0021-9568/96/1741-0231$12.00/0

Table 1. Densities of the x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ Systems as a Function of Temperature and Composition

T/K	ρ/(kg m^{-3})	T/K	ϱ/(kg m^{-3})	T/K	ρ/(kg m^{-3})
$x = 0.0$		$x = 0.1292$		$x = 0.2378$	
309.15	1708.2	308.75	1674.7	309.15	1638.6
311.25	1706.5	311.05	1672.7	311.25	1636.8
313.45	1704.6	313.05	1671.1	313.30	1634.9
315.55	1702.7	315.20	1669.3	315.35	1633.2
317.55	1700.9	317.25	1667.3	317.45	1631.1
319.65	1698.7	319.20	1665.6	319.50	1629.5
321.75	1697.1	321.35	1663.4	321.45	1627.8
323.85	1695.3	323.25	1661.8	323.50	1626.1
326.00	1693.5	325.30	1660.1	325.45	1624.5
328.05	1691.9	327.55	1658.3	327.55	1622.6
330.15	1689.9			329.60	1621.0
				331.75	1619.0
$x = 0.3528$		$x = 0.4544$		$x = 0.6295$	
308.15	1592.0	307.65	1543.7	309.15	1436.6
310.25	1590.5	309.75	1542.0	311.00	1435.2
314.35	1586.9	311.70	1540.5	312.95	1433.5
316.30	1585.2	313.65	1538.8	314.75	1431.9
318.25	1583.2	315.60	1537.0	316.55	1430.4
320.30	1581.7	317.45	1535.4	318.50	1428.6
322.20	1580.0	319.45	1533.4	320.25	1427.2
324.30	1578.3	321.40	1531.9	322.05	1425.7
326.10	1576.8	323.25	1530.3	323.85	1424.2
328.15	1575.0	325.30	1528.7	325.65	1422.8
330.35	1573.4	327.20	1527.2	327.40	1421.2
		329.20	1525.5	329.30	1419.7
$x = 0.7409$		$x = 0.8688$		$x = 0.9188$	
310.70	1349.3	309.45	1219.1	309.50	1155.1
312.50	1347.8	311.15	1217.8	312.05	1153.7
314.35	1346.2	312.85	1216.3	313.65	1152.5
316.10	1344.8	314.50	1215.1	315.25	1151.0
317.90	1343.1	316.20	1213.5	316.75	1149.9
319.55	1341.8	317.80	1212.3	318.35	1148.7
321.35	1340.4	319.45	1211.1	319.95	1147.5
323.10	1339.0	321.10	1209.8	321.50	1146.4
324.75	1337.7	322.75	1208.6	323.10	1145.1
326.55	1336.2	324.40	1207.2	324.65	1143.9
328.20	1334.8	326.00	1206.0	326.25	1142.5
		327.60	1204.5	327.75	1141.4
		329.25	1203.3		

Table 2. Least-Squares-Fitted Values of the Density Equation $\rho = a - b(T - 273.15)$ for x CH_3COHN_2 + (1 − x)$Ca(NO_3)_2{\cdot}4.37H_2O$ Systems

x	a/(kg m^{-3})	b/(kg m^{-3} K^{-1})	σ^a
0.0	1739.7 ± 0.3	0.876 ± 0.007	0.16
0.1292	1706.3 ± 0.3	0.885 ± 0.007	0.14
0.2378	1669.5 ± 0.2	0.861 ± 0.005	0.12
0.3528	1621.9 ± 0.3	0.851 ± 0.007	0.16
0.4544	1573.2 ± 0.3	0.854 ± 0.006	0.14
0.6295	1466.9 ± 0.2	0.842 ± 0.005	0.10
0.7409	1380.3 ± 0.2	0.827 ± 0.005	0.09
0.8688	1247.8 ± 0.2	0.793 ± 0.004	0.09
0.9188	1183.2 ± 0.4	0.765 ± 0.009	0.18

[a] Root mean square deviation in density.

Table 3. Molar Volume of x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ Systems as a Function of Temperature and Composition

	$V \times 10^6$/(m^3 mol^{-1})							
x	283.15 K	293.15 K	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K	353.15 K
0.00	140.3	141.0	141.7	142.4	143.2	143.9	144.7	145.4
0.1292	129.1	129.7	130.4	131.1	131.8	132.5	133.2	133.9
0.2378	119.9	120.5	121.1	121.8	122.4	123.1	123.7	124.4
0.3528	110.3	110.9	111.5	112.1	112.7	113.3	113.9	114.5
0.4544	101.8	102.4	102.9	103.5	104.1	104.7	105.3	105.9
0.6295	87.2	87.7	88.2	88.7	89.2	89.8	90.3	90.8
0.7409	77.8	78.2	78.7	79.2	79.7	80.2	80.7	81.2
0.8688	67.1	67.5	68.0	68.4	68.8	69.3	69.8	70.2
0.9188	62.9	63.4	63.8	64.2	64.6	65.1	65.5	65.9

Table 4. Least-Squares-Fitted Values of the Parameters of Eq 1 and the Values of the Intrinsic Volume, V_o, of x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ Systems

x	$V_o' \times 10^6$/(m^3 mol^{-1})	$b' \times 10^8$/(m^3 mol^{-1} K^{-1})	$\sigma \times 10^{8\ a}$	$V_o \times 10^6$/(m^3 mol^{-1})
0.00	119.5 ± 0.2	7.33 ± 0.06	3.7	134.2 ± 0.1
0.1292	109.4 ± 0.1	6.92 ± 0.05	3.1	123.1 ± 0.1
0.2378	101.6 ± 0.2	6.44 ± 0.05	3.5	114.3 ± 0.1
0.3528	93.3 ± 0.1	6.00 ± 0.02	0.2	105.0 ± 0.1
0.4544	85.2 ± 0.2	5.86 ± 0.05	3.3	96.7 ± 0.1
0.6295	72.5 ± 0.1	5.18 ± 0.05	3.0	82.4 ± 0.1
0.7409	63.8 ± 0.1	4.92 ± 0.05	3.1	73.2 ± 0.1
0.8688	54.4 ± 0.2	4.46 ± 0.05	3.7	62.8 ± 0.1
0.9188	50.9 ± 0.1	4.26 ± 0.05	3.2	58.8 ± 0.1

[a] Root mean square deviation in molar volume.

The molar volumes, V (Table 3), of x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ melts are calculated from their densities and are presented in the form of a linear function of temperature (Table 4):

$$V = V_o' + b'T \tag{1}$$

where V_o' and b' are constant parameters at a particular composition.

The measured values of conductivity (κ) and viscosity (η) of the systems under study are presented in Tables 5 and 6, respectively. The temperature dependence of conductivity and viscosity is non-Arrhenius (≃7% and 15% deviation from linearity from the Arrhenius plot for viscosity and conductivity, respectively). Such a temperature dependence of transport properties is common in concentrated electrolyte and ionic melt systems. The conductivity and viscosity values were, therefore, least-squares-fitted to the Vogel−Tammann−Fulcher (VTF) equation of the form considering the insignificant contribution of the preexponential temperature term:

$$\kappa = A_\kappa \exp[-B_\kappa/(T - T_{o,\kappa})] \tag{2}$$

$$\eta = A_\eta \exp[B_\eta/(T - T_{o,\eta})] \tag{3}$$

where A$_\kappa$, A$_\eta$, B$_\kappa$, and B$_\eta$ are the adjustable parameters, and $T_{o,\kappa}$ and $T_{o,\eta}$ are the ideal glass transition temperatures. The least-squares-fitted values of the parameters of eqs 2 and 3 are presented in Tables 7 and 8. The computed values of T_o obtained for viscosities are slightly higher than those obtained from conductance. The parameters A_κ, A_η, B_κ, and B_η do not vary in a meaningful way with composition. $T_{o,\kappa}$ and $T_{o,\eta}$ vary linearly with composition as shown in Figure 1a.

The ideal glass transition temperature, T_o, for viscosity and conductivity for pure $Ca(NO_3)_2{\cdot}4.37H_2O$ is (200.0 ± 2.0) K which agrees well with the reported T_o = 200 K for $Ca(NO_3)_2{\cdot}4H_2O$ (Angell and Tucker, 1974) obtained from calorimetric studies and is very close to that obtained from conductance (201 K) and viscosity (205 K) studies (Smedley, 1980). On extrapolation of the $T_{o,\kappa,\eta}$ vs x plot (Figure 1a) one obtains $T_{o,\kappa}$ = 186.5 K and $T_{o,\eta}$ = 187.5 K for pure acetamide. Due to a lack of reported T_o values for acetamide we could not make a comparison. However, from the reported empirical relationship between the glass transition temperature, T_g, and the melting temperature, T_m (Sakka and Mackenzie, 1971; Souquet, 1994), one can estimate T_o = 165−189 K for pure acetamide using the approximation $T_o = 0.7 - 0.8T_g$ for molecular liquids (Angell and Rao, 1972). T_o so obtained is in reasonable agreement with the extrapolated T_o value (Figure 1a).

Table 5. Electrical Conductivity Data for x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ Systems

	$\kappa \times 10^3/(S\ cm^{-1})$								
T/K	x = 0.00	x = 0.1292	x = 0.2378	x = 0.3528	x = 0.4544	x = 0.6295	x = 0.7409	x = 0.8688	x = 0.9188
253.15		0.102	0.065	0.074	0.107	0.082	0.079	0.087	
258.15		0.220	0.146	0.160	0.198	0.197	0.157	0.161	
263.15	0.431	0.427	0.296	0.318	0.351	0.309	0.284	0.279	
268.15	0.778	0.766	0.542	0.572	0.599	0.536	0.478		
273.15	1.320	1.291	0.938	0.969	0.951	0.874	0.769	0.706	0.619
278.15	2.075	2.028	1.500	1.525	1.473	1.336	1.165	1.068	0.914
283.15	3.094	3.012	2.260	2.278	2.168	1.958	1.681	1.502	1.296
288.15	4.427	4.280	3.267	3.265	3.080	2.742	2.334	2.037	1.769
293.15	6.112	5.855	4.548	4.492	4.227	3.711	3.129	2.679	2.351
298.15	8.114	7.748	6.121	6.001	5.601	4.882	4.091	3.453	3.030
303.15	10.488	10.008	8.002	7.799	7.242	6.265	5.211	4.347	3.825
308.15	13.230	12.624	10.185	9.861	9.127	7.828	6.503	5.366	4.715
313.15	16.381	15.575	12.702	12.247	11.325	9.625	7.964	6.516	5.692
318.15	19.866	18.888	15.518	14.919	13.761	11.616	9.583	7.783	6.822
323.15	23.802	22.511	18.671	17.837	16.427	13.843	11.356	9.150	8.019
328.15	27.905	26.533	22.123	21.063	19.368	16.220	13.297	10.639	9.329
333.15	32.433	30.776	25.787	24.605	22.538	18.772	15.362	12.264	10.730

Table 6. Viscosity Data for x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ Systems

	$\eta/(mPa\ s)$								
T/K	x = 0.00	x = 0.1292	x = 0.2378	x = 0.3528	x = 0.4544	x = 0.6295	x = 0.7409	x = 0.8688	x = 0.9188
283.65				489.0		335.2	270.7		
284.15			512.8						
284.65					389.6				
285.15	380.4	370.7						133.3	
286.65			404.70						
288.15	296.2	288.5	356.6	332.3	291.5	232.8	190.6	107.9	78.12
293.15	201.7	197.8	239.6	225.5	198.7	161.4	134.5	77.78	56.68
295.65						136.4			
298.15	143.5	141.1	168.4	159.1	141.7	117.2	98.74	57.86	44.43
303.15	105.6	103.9	122.4	115.9	104.3	87.29	74.26	43.93	34.86
308.15	79.93	78.77	91.84	87.53	78.85	66.58	56.92	34.31	28.49
313.15	62.05	61.22	70.63	67.29	61.15	51.99	44.68	27.84	23.53
318.15	49.26	48.63	55.59	53.15	48.53	40.57	35.82	22.58	19.37
323.15	39.83	39.32	44.63	42.62	39.10	33.60	28.99	18.54	16.51
328.15	32.75	32.35	36.39	34.91	32.14	27.37	23.86	15.45	13.93
333.15	27.39	27.09	30.20	28.85	26.75		19.95	13.03	12.16
338.15				24.31	22.55	19.50	16.92	11.13	10.78
343.15				20.66	19.20	16.75		9.559	9.684
348.15				17.69			12.49	8.286	8.677

Table 7. Least-Squares-Fitted Values of the Parameters of Eq 2 for Conductivity for x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ Systems

x	$T_{o,\kappa}$/K	$A_\kappa/(S\ cm^{-1})$	B_κ/K	σ^a
0.0	200.0 ± 1.0	1.626 ± 0.102	520.5 ± 12.0	0.004
0.1292	198.0 ± 1.3	1.622 ± 0.161	535.5 ± 16.7	0.013
0.2378	197.0 ± 1.5	1.805 ± 0.186	576.1 ± 19.8	0.008
0.3528	195.3 ± 1.3	1.698 ± 0.170	581.6 ± 17.4	0.010
0.4544	194.0 ± 1.0	1.239 ± 0.092	561.9 ± 13.9	0.052
0.6295	191.3 ± 1.3	1.240 ± 0.115	592.7 ± 16.8	0.037
0.7409	190.0 ± 2.0	1.019 ± 0.132	597.9 ± 26.3	0.010
0.8688	188.5 ± 1.5	0.691 ± 0.065	581.7 ± 19.0	0.012
0.9188	187.5 ± 1.5	0.637 ± 0.049	593.0 ± 16.4	0.010

[a] Root mean square deviation in ln κ.

Table 8. Least-Squares-Fitted Values of the Parameters of Eq 3 for Viscosity for x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2{\cdot}4.37H_2O$ Systems

x	$T_{o,\eta}$/K	$A_\eta/(mPa\ s)$	B_η/K	σ^a
0.0	201.3 ± 2.8	0.275 ± 0.046	606.1 ± 32.0	0.002
0.1292	199.3 ± 2.0	0.250 ± 0.029	626.3 ± 23.7	0.003
0.2378	197.5 ± 1.5	0.203 ± 0.011	677.3 ± 12.9	0.008
0.3528	196.3 ± 1.5	0.198 ± 0.010	681.5 ± 12.2	0.005
0.4544	194.8 ± 1.0	0.189 ± 0.010	684.8 ± 12.1	0.006
0.6295	192.2 ± 1.5	0.165 ± 0.010	695.3 ± 12.2	0.008
0.7409	191.0 ± 2.5	0.152 ± 0.018	693.5 ± 30.0	0.005
0.8688	189.7 ± 1.3	0.128 ± 0.010	663.4 ± 14.0	0.007
0.9188	188.5 ± 1.3	0.221 ± 0.011	581.5 ± 9.40	0.010

[a] Root mean square deviation in ln η.

The molar volume at 323.15 K as a function of composition of the present system under study is presented in Figure 1b. It is evident that the molar volume, V, varies linearly with x. The extrapolated molar volume (57.6 ± 0.4) × 10^{-6} m^3 mol^{-1} of pure acetamide (Figure 1b) is in reasonable agreement with the value of 57.0 × 10^{-6} m^3 mol^{-1} calculated from density data (Kerridge, 1988). As the system has an inherent tendency to supercool (Berchiesi et al., 1989), the temperature dependence of molar volume may also be represented as

$$V = V_o + b'(T - T_o) \tag{4}$$

where V_o is the intrinsic volume. Comparison of eqs 1 and 4 gives

$$V_o' = V_o - b'T_o \tag{5}$$

The values of the intrinsic volume, V_o, at the different compositions calculated from eq 5 are presented in Table 4 and Figure 1b. From Figure 1b,c it is observed that V_o, V_o', and b' vary linearly with the mole fraction of acetamide. The intrinsic volume, V_o, for pure acetamide from eq 5, keeping T_o = 187 K (Figure 1a), is found to be 52.1 × 10^{-6} m^3 mol^{-1} and is in good agreement with the extrapolated value of V_o = (52.1 ± 0.1) × 10^{-6} m^3 mol^{-1} (Figure 1b). The composition dependence of V_o predominantly governs the linear variation of V with x for x CH_3CONH_2 + (1 − x)

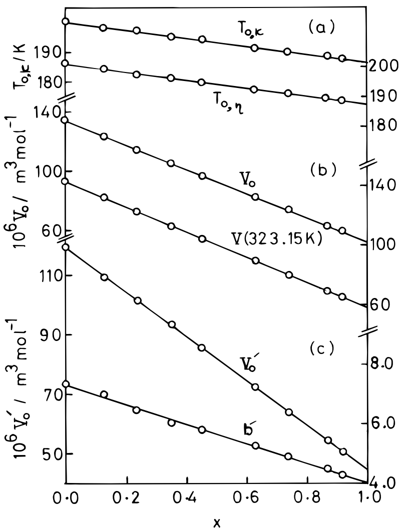

Figure 1. Variation of (a) $T_{o,\kappa,\eta}$, (b) V_o and V (at 323.15 K), and (c) V_o' and b' with x for molten x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2 \cdot 4.37H_2O$ systems. Open circles are observed values, and solid lines represent calculated values.

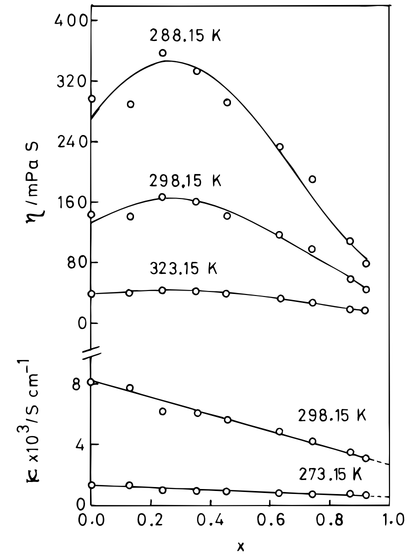

Figure 2. Viscosity and electrical conductivity isotherms for x CH_3CONH_2 + (1 − x) $Ca(NO_3)_2 \cdot 4.37H_2O$ melt systems. Circles are observed values, and solid lines represent calculated values (from a polynomial equation up to second degree for viscosity and one degree for electrical conductivity).

$Ca(NO_3)_2 \cdot 4.37H_2O$ melts. The molar volume is found to be additive within ±0.2%.

The composition dependences of conductivity and viscosity are shown in Figure 2. It is noted that the conductivity decreases linearly with mole fraction, x, whereas viscosity increases with x up to $x \simeq 0.25$ and then decreases. At this moment we are not able to explain why conductivity and viscosity isotherms vary in a different fashion. However, in the NaSCN + CH_3CONH_2 system (Płowiec et al., 1985) Na^+ in the field of polar ($CONH_2$) and alkyl (CH_3) groups of acetamide represents a polymeric type of solvated ion. The polymeric ion is less mobile (Amico et al., 1987) in liquid acetamide in comparison to Na^+ in water. A similar inference may also be drawn in the present system. The mobility of the solvated polymeric Ca^{2+} in the liquid acetamide medium decreases as the concentration of acetamide increases, resulting in the linear decrease in the conductivity with x. In the case of viscosity, solvated polymeric ion with a rigid structure (Berchiesi et al., 1989) governs the viscosity up to $x \simeq 0.25$, and beyond $x \simeq 0.25$ bulk liquid acetamide predominates the flow properties of the system under study. From Figure 2 it is apparent that the viscosity maximum at around $x = 0.25$ decreases as the temperature increases. Such a decrease in the viscosity maximum may be due to the breaking of the rigid polymeric structure of the Ca^{2+} ion with the acetamide with an increase in temperature.

Acknowledgment

The author thanks Dr. Anil C. Ghosh, Director, for encouragement to carry out this work.

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Received for review June 5, 1995. Accepted November 11, 1995.⊗ Grateful thanks are expressed to the Deutsche Gesellschaft für Technische Zusammenarbeit (GTZ) GmbH, Germany, for the gift of the equipment.

JE950132L

⊗ Abstract published in *Advance ACS Abstracts*, January 15, 1996.