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Viscosities and Densities of Highly Concentrated Aqueous MOH Solutions ($M^+ = Na^+, K^+, Li^+, Cs^+, (CH_3)_4N^+$) at 25.0 °C

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The absolute (dynamic) viscosities (η) and densities (ρ) of carbonate-free aqueous tetramethylammonium and alkali metal hydroxides have been determined up to saturation concentrations ([NaOH] ≤ 19.l M, [KOH] ≤ 14.1 M, [LiOH] ≤ 4.8 M, [CsOH] ≤ 14.8 M, and $[(CH_3)_4NOH] \leq 4.2$ M) at 25.00 °C using a Ubbelohde viscometer and a vibrating tube densitometer, respectively. The viscosities are believed to be precise to within 0.1% and the densities to within 5×10^{-6} g cm^{-3}. Densities of isoplethic MOH solutions increase in the order of $(CH_3)_4N^+ < Li^+ < Na^+ < K^+ \ll Cs^+$. Viscosities for [MOH] < 4 M solutions increase in the reverse order, but the viscosities of CsOH solutions become extremely large at very high concentrations. The shape of the density vs concentration function of $(CH_3)_4NOH$ solutions is also quite different when compared with the alkali metal hydroxide solutions. Density data were fitted up to the highest concentrations using the Masson equation. Viscosity vs concentration functions are represented in the form of a fifth-order (empirical) polynomial.

Introduction

The densities of aqueous alkaline hydroxide solutions are available at room temperature from several primary sources.[1−6] Viscosity data relating to the same systems are, however, rarer.[7−10] These data are compiled in various handbooks.[11−13] With only a few exceptions, both density and viscosity data are restricted to relatively low concentrations of the hydroxides. Perhaps the most reliable viscosity data of more concentrated solutions were obtained at temperatures other than 25 °C.[7] Viscosities of concentrated aqueous hydroxide solutions at 25 °C are surprisingly scarce, and serious discrepancies exist among these data, particularly at high concentrations. This is demonstrated in Figure 1, which shows the viscosities of aqueous NaOH solutions from various primary literature sources.

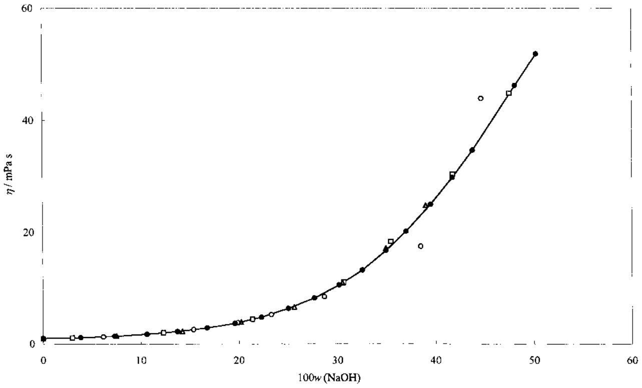

Figure 1. Dynamic viscosities, η, of aqueous NaOH solutions at room temperature as a function the mass fraction of NaOH, w: ○, Klochko and Godneva;[8] △, Baron and Matveeva;[9] □: Baron and Matveeva;[10] ● and —, this work.

Experimental Section

Solution Preparation. Solution series, consisting of samples usually of 100 mL total volume, were each prepared from concentrated (almost saturated) stock solutions of hydroxides by accurately weighing appropriate amounts of the stock solution directly into "A" grade volumetric flasks. These were then filled to the mark with high purity, CO_2-free water (Millipore MilliQ system). Buoyancy corrections were not applied during solution preparation, and thus, the overall accuracy of the concentrations is ~0.1% relative. Concentrated alkali stock solutions were prepared from analytical grade NaOH (98 mass %, Ajax Chemicals, Sydney, Australia), KOH (85 mass % with ca. 10−15 mass % water, Ajax), $LiOH \cdot H_2O$ (99 mol %+, Sigma-Aldrich Chemical Co., Milwaukee, WI), $CsOH \cdot H_2O$ (99.5 mol %+, Sigma-Aldrich), and tetramethylammonium hydroxide pentahydrate, $(TMA)OH \cdot 5H_2O$ (97 mol %+, Sigma-Aldrich). (TMA)OH solutions were also obtained as concentrated analytical grade solutions (25 mass %, Sigma-Aldrich). The approximate concentrations of the stock solutions were the following: NaOH ≈ 20 M; KOH ≈ 14 M; LiOH ≈ 4.8 M; CsOH ≈ 15 M; (TMA)OH ≈ 4.5 M. The carbonate content of the stock solutions (which can be extremely high in commercial samples, even of analytical grade) was minimized using a range of techniques which are described in detail elsewhere.[14]

The concentrations of the base solutions and their carbonate contents were determined by high precision glass electrode potentiometric titrations following the procedures described previously.[15] For these analyses, the concentrated stock solutions were appropriately diluted, as described above, to give a total hydroxide concentration $[OH^-] \approx 0.1$ M and made up to an ionic strength $I = 1$ M (NaCl), where $I = 0.5\sum c_i z_i^2$. Aliquots (10−25 mL) of these solutions were then titrated with standard HCl solutions (BDH Convol, stated accuracy ±0.2%) made up in the same medium. The titrations were evaluated by the Gran method[16] and the ESTA suite of computer programs[17] and have an overall

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Table 1. Densities, ρ, Apparent Molar Volumes, V_ϕ, and Dynamic Viscosities, η, of Aqueous NaOH Solutions at 25.0 °C

c(NaOH) mol L^{-1} [a]	m(NaOH) mol kg^{-1} [b]	100w (NaOH)[c]	100w (Na_2O)[d]	ρ/g cm^{-3}	V_ϕ cm^3 mol^{-1}	η/mPa s
1.002[e]	1.003	3.858	2.990	1.039 258	−2.14	1.1290
1.982[e]	1.986	7.358	5.702	1.077 373	−0.53	1.3837
2.968[e]	2.983	10.662	8.263	1.113 462	0.78	1.7270
3.949[e]	3.991	13.765	10.668	1.147 452	1.19	2.1993
4.955[e]	5.044	16.790	13.012	1.180 355	3.01	2.8361
5.939[e]	6.097	19.606	15.195	1.211 727	3.86	3.6772
6.925[e]	7.184	22.322	17.299	1.240 868	4.80	4.7692
7.960[e]	8.352	25.042	19.408	1.271 462	5.54	6.3355
9.003[e]	9.590	27.725	21.487	1.298 840	6.49	8.2121
10.000[e]	10.814	30.195	23.401	1.324 687	7.25	10.520
10.982[e]	12.079	32.576	25.246	1.348 486	8.02	13.244
12.009[e]	13.441	34.965	27.098	1.373 765	8.65	16.742
12.915[e]	14.706	37.038	28.704	1.394 764	9.23	20.195
14.034[e]	16.354	39.546	30.648	1.419 445	9.93	25.017
15.037[e]	17.911	41.740	32.349	1.441 030	10.50	29.819
15.998[e]	19.498	43.817	33.958	1.460 462	11.06	34.654
18.071[e]	23.203	48.137	37.306	1.501 627	12.11	46.252
19.102[e]	25.292	50.291	38.975	1.519 337	12.69	51.895
0.998[f,g]	0.999	3.841	2.977	1.039 028	−2.08	1.0953
2.007[f,g]	2.010	7.444	5.769	1.078 373	−0.53	1.3732
3.000[f,g]	3.015	10.763	8.341	1.114 922	0.71	1.7406
4.001[f,g]	4.043	13.922	10.790	1.149 474	1.90	2.2320
5.003[f,g]	5.097	16.934	13.124	1.181 616	3.11	2.8733

[a] Concentration expressed in mol of solute/L of solution. [b] Concentration expressed in mol of solute/kg of solvent. [c] Concentration expressed in mass fraction of M′OH. [d] Concentration expressed in mass fraction of M'_2O. [e] Viscosity determination with a IC type tube; efflux time for water is (31.98 ± 0.04) s. [f] Viscosity determination with a 0C type tube; efflux time for water is (321.5 ± 0.40) s. [g] Independent duplication.

accuracy of 0.2% and a carbonate detection limit of 0.05 mol % relative.

Density Measurements. Densities were measured using an Anton Paar DMA O2D vibrating tube density meter. The sample tube was thermostated to (25.00 ± 0.01) °C with a Julabo F33 thermostat. The temperature of the bath was periodically checked and adjusted using a quartz crystal thermometer (Hewlett-Packard, model HP 2804A) which was regularly recalibrated against the triple point of water and is probably accurate to ±0.02 °C. Readings from the densitometer, τ, proportional to the period of oscillation of the tube were taken using a measurement period of 20 s. The difference in density, $\Delta\rho$, between two liquid samples was determined from the relationship

$$\Delta\rho = k(\tau_1^{\,2} - \tau_2^{\,2}) \tag{1}$$

where τ_1 and τ_2 are the readings for the respective liquids. The proportionality constant, k, was determined on a daily basis from the difference in density of air and water. The density of water was taken to be 0.997 042 9 g cm^{-3}, and that of air, to be 0.001 185 0 g cm^{-3}.[18] Calibration parameters were accepted when τ remained stable to six significant figures; otherwise the instrument calibration was repeated until the required reproducibility was achieved. No significant change was observed in k over a period of 8 months indicating that the glass vibrating tube was not being significantly attacked at 25 °C by the strongly caustic solutions.

The samples were delivered to the dry vibrating tube from a caustic-resistant polyethylene syringe. After filling, the tube was carefully checked for the presence of air bubbles and then at least 10 min was allowed for the sample to reach thermal equilibrium. Period measurements were taken and repeated until stable readings were achieved. At least three measurements were performed on each solution, and the observed τ values were then averaged. The precision of the density readings is estimated to be ca. ±5 μg/mL. After measurement of a given sample, the tube was washed with distilled water and dry acetone and then dried with compressed air. The period of the air-filled tube was measured to check the integrity of the cleaning procedure.

Viscosity Measurements. Ubbelohde-type suspended-level capillary viscometers (Schott-Geräte, types 53003 and 53013 with capillary numbers 0c and Ic, respectively, and capacity 15−20 mL) were used for determining solution viscosities. The viscometer tube was held in a Schott-Geräte AVS/S measuring stand fitted with two phototransistors and detectors. The viscometer and stand were connected to a Schott-Geräte AVS 310 measuring unit which allows flow times to be measured automatically to the nearest 0.01 s. The measuring stand and the viscometer were kept in a transparent continuously stirred water bath. The temperature of the water bath was maintained at (25.00 ± 0.005) °C with a locally constructed regulator unit controlling a stainless steel heater with a concentrically located platinum resistance element (1 kΩ, Degussa, Pt-RTD model GR 2141) as the temperature sensor. The apparatus was kept in a thermostated room at (20 ± 2) °C, which provided an adequate heat sink for the PID temperature controller. The temperature in the immediate vicinity of the viscometer bulb was continuously monitored with a Hewlett-Packard quartz crystal thermometer.

The dynamic viscosity of the solutions was determined using:

$$\eta = \eta_0 \frac{(t - \Theta)}{(t_0 - \Theta_0)} \frac{\rho}{\rho_0} \tag{2}$$

where η is the dynamic viscosity, ρ is the density, t is the flow-time, Θ is the flow-time-dependent empirical correction factor (the so-called Hagenbach correction), and the subscript 0 indicates a reference liquid (usually water). The values of Θ were those provided by the viscometer manufacturer unless otherwise indicated.

Water (for which η_0 = 0.8904 mPa s and ρ_0 is as above) was used for the primary calibration of the viscometers.

Table 2. Densities, ρ, Apparent Molar Volumes, V_ϕ, and Dynamic Viscosities, η, of Aqueous KOH Solutions at 25.0 °C

c(KOH) / mol L$^{-1\,a}$	m(KOH) / mol kg$^{-1\,b}$	100w (KOH)^{c}	100w (K$_2$O)^{d}	ρ/g cm^{-3}	V_ϕ / cm^3 mol^{-1}	η/(mPa s)
1.018^{f}	1.029	5.457	4.581	1.046 520	7.53	0.9980
2.003^{f}	2.047	10.302	8.648	1.091 221	9.11	1.1200
3.000^{f}	3.106	14.840	12.457	1.134 401	10.35	1.2656
4.000^{f}	4.204	19.087	16.022	1.175 736	11.47	1.4388
5.003^{f}	5.352	23.095	19.387	1.215 362	12.51	1.6594
6.060^{f}	6.622	27.092	22.741	1.255 184	13.55	1.8760
6.921^{f}	7.724	30.237	25.381	1.284 401	14.63	2.2339
7.997^{f}	9.110	33.827	28.395	1.326 461	14.96	2.5355
9.000^{e}	10.496	37.066	31.114	1.362 377	15.56	2.9927
9.987^{e}	11.959	40.157	33.709	1.395 491	16.26	3.5557
10.981^{e}	13.510	43.118	36.194	1.429 035	16.82	4.2830
12.121^{e}	15.494	46.507	39.039	1.462 389	17.77	5.5439
13.138^{e}	17.253	49.189	41.291	1.498 592	17.98	6.7003
14.152^{e}	19.200	51.861	43.534	1.531 086	18.42	8.5452

[a–f] See Table 1.

Table 3. Densities, ρ, Apparent Molar Volumes, V_ϕ, and Dynamic Viscosities, η, of Aqueous LiOH Solutions at 25.0 °C

c(LiOH) / mol L$^{-1\,a}$	m(LiOH) / mol kg$^{-1\,b}$	100w (LiOH)^{c}	100w (Li$_2$O)^{d}	ρ/g cm^{-3}	V_ϕ / cm^3 mol^{-1}	η/mPa s
0.754^{f}	0.754	1.774	1.106	1.018110	−4.00	1.0924
1.533^{f}	1.531	3.537	2.206	1.038184	−2.90	1.3501
2.269^{f}	2.265	5.146	3.210	1.056117	−2.09	1.6593
3.090^{f}	3.087	6.884	4.294	1.074996	−1.28	2.1037
3.832^{f}	3.835	8.411	5.247	1.091121	−0.60	2.6117
4.854^{f}	4.896	10.495	6.547	1.107717	1.15	3.3124

[a–f] See Table 1.

Table 4. Densities, ρ, Apparent Molar Volumes, V_ϕ, and Dynamic Viscosities, η, of Aqueous CsOH Solutions at 25.0 °C

c (CsOH) / mol L$^{-1\,a}$	m (CsOH) / mol kg$^{-1\,b}$	100w (CsOH)^{c}	100w (Cs$_2$O)^{d}	ρ/g cm^{-3}	V_ϕ / cm^3 mol^{-1}	η/mPa s
1.967^{f}	2.055	23.547	22.133	1.252239	20.23	1.0606
3.899^{f}	4.270	39.028	36.684	1.497613	21.59	1.3560
5.828^{f}	6.769	50.364	47.339	1.734718	23.41	1.8962
7.834^{e}	9.773	59.434	55.863	1.975979	25.03	3.0358
9.979^{e}	13.571	67.045	63.018	2.231253	26.31	6.2607
10.918^{e}	15.418	69.801	65.608	2.344815	26.55	
11.878^{e}	17.561	72.472	68.118	2.457005	27.08	15.2955
14.097^{e}	23.488	77.881	73.203	2.713463	28.24	59.597
14.839^{e}	25.722	79.406	74.636	2.801406	28.40	

[a–f] See Table 1.

Efflux times for water from several hundred independent runs were found to be (31.98 ± 0.04) s (Ic type) and (321.5 ± 0.4) s (0c type). Note that the internal reproducibility of the measurements was better than this (typically ±0.02 and ±0.10 s, respectively); thus, the greater overall standard deviations largely reflect (long term) temperature variation in the bath.

To check the stability of the viscometric system, the t_0 values were regularly determined before and after each measurement. The viscosities of calibrations and test solutions were measured in two or three series, each consisting of 3 to 6 consecutive runs. Measurements were accepted when the internal reproducibility between the consecutive runs was within the values specified in the preceding paragraph. This level of internal reproducibility of the measurements indicate that no significant variations in the solution composition (i.e., adsorption of airborne CO_2(g) or moisture) took place in the duration of the viscosimetric experiments.

For each series of measurements the capillary tube was selected so that the efflux times were roughly between 50 s and 1500 s. Since the flow-time of water in the faster (Ic) tube is outside this range, the Hagenbach correction corresponding to $t_0 = 31.98$ s, i.e., (1.82 ± 0.02) s, was determined using a series of NaOH solutions (4−8 M) for which the viscosities were measured using the slower 0c tube. For these NaOH solutions the efflux times with the Ic tube were high enough to use the tabulated Hagenbach corrections. The reproducibility of the viscosity measurements was found to be within 0.1% on independently prepared samples (see Table 1).

Results and Discussion

The densities and dynamic viscosities of aqueous MOH solutions at 25.00 °C are presented in Tables 1−5 and Figures 2 and 3. Concentrations are expressed in molarities (mol/L of solution) and molalities (mol/kg of solvent) and two further mass percent scales: grams of M′OH and M'_2O per 100 g of solution (the latter is often used in various chemical engineering contexts).

From the data presented it is apparent that the densities of isoplethic hydroxide solutions increase in the order of $(CH_3)_4N^+ < Li^+ < Na^+ < K^+ \ll Cs^+$. The viscosities at concentrations of < 4 M decrease in the reverse order. At very high concentrations, however, the viscosities of CsOH solutions become extremely high. The density vs concen-

Table 5. Densities, ρ, Apparent Molar Volumes, V_ϕ, and Dynamic Viscosities, η, of Aqueous TMAOH Solutions at 25.0 °C ($TMA^+ = (CH_3)_4N^+$)

c(TMAOH) / mol L^{-1} [a]	m(TMAOH) / mol kg^{-1} [b]	100w (TMAOH)[c]	100w (TMA_2O)[d]	ρ/g cm^{-3}	V_ϕ / cm^3 mol^{-1}	η/(mPa s)
0.398[f]	0.413	3.632	3.274	0.999 260	85.84	0.9869
0.806[f]	0.868	7.338	6.614	1.001 672	85.67	1.1130
1.210[f]	1.354	10.990	9.906	1.004 099	85.58	1.2891
1.580[e]	1.832	14.313	12.900	1.006 775	85.25	1.4672
1.990[e]	2.402	17.970	16.197	1.009 915	84.94	1.7494
2.402[e]	3.024	21.615	19.482	1.013 460	84.57	2.1273
2.796[e]	3.668	25.066	22.592	1.017 295	84.16	2.6603
3.376[e]	4.715	30.068	27.101	1.023 977	83.42	4.0777
3.781[e]	5.527	33.514	30.207	1.028 904	82.97	5.7266
4.196[e]	6.432	36.971	33.323	1.035 061	82.34	8.9269

[a–f] See Table 1.

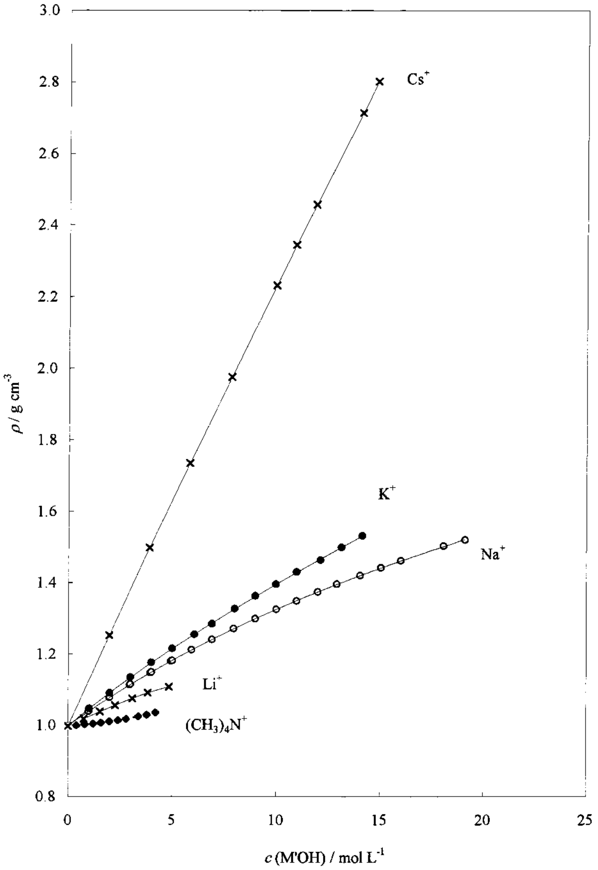

Figure 2. Densities, ρ, of M′OH solutions at 25.0 °C as a function of the molar concentration, c.

tration function of $(CH_3)_4NOH$ solutions behaves unusually in comparison to the other concentrated electrolytes (all having negative deviations from linearity) covered in this study.

Apparent molar volumes (V_ϕ) were calculated using the equation

$$V_\phi = (M/\rho) - 10^3(\rho - \rho_0)/c\rho \qquad (3)$$

where ρ and ρ_0 are the densities of the solution and the solvent, respectively, M is the molar mass of the solute, and c is the molarity concentration of the solution. The corresponding data are presented in Tables 1−5.

Observed density data were modeled in terms of the Masson equation,[11] which can be written as

$$V_\phi = V_\phi^{\ 0} + k_e\sqrt{c} \qquad (4)$$

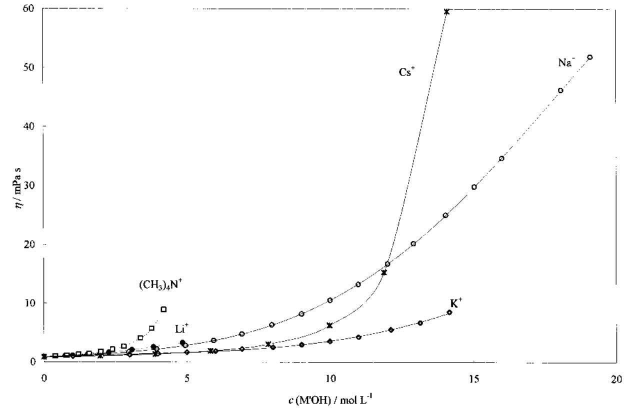

Figure 3. Viscosities, η, of M′OH solutions at 25.0 °C as a function of the molar concentration, c.

where $V_\phi^{\ 0}$ is the standard partial molar volume of the solute in the solvent (at infinite dilution) and k_e is an empirical constant. Despite its well-known theoretical deficiencies, the Masson equation works remarkably well as a simple method for correlating the densities of quite concentrated solutions.[11] This is usually done by combining eqs 3 and 4 to give

$$\rho = \rho_0 + \alpha c + \beta c^{3/2} \qquad (5)$$

where

$$\alpha = 10^{-3}(M - V_\phi^{\ 0}\,\rho_0) \qquad (6)$$

and

$$\beta = -k_e\rho_0 \qquad (7)$$

Introduction of the factor 10^{-3} is necessary to convert between the volume in conventional units of concentration (mol L^{-1}) and density g cm^{-3}. The parameters obtained from a least-squares analysis for α and β are shown in Table 6. The Masson equation gave a satisfactory description up to the highest concentrations for all densities of the electrolytes covered in this study, with an average error of always better than 0.1%.

Viscosity data were modeled first using the Jones−Dole equation as recently modified by Zhang et al.:[19]

$$\eta = \eta_0 + Ac^{1/2} + Bc + Dc^2 + Ec^{7/2} \qquad (8)$$

where η and η_0 are the viscosities of the solution and the solvent, respectively, and A, B, D, and E are constants. According to Zhang et al.[19] this equation is able to describe

Table 6. Calculated Parameters (Obtained by Least-Squares Optimization) in the Numerical Expressions for the Densities and Viscosities of Aqueous Solutions of M′OH (M′ = Li, Na, K, Cs, TMA)

	LiOH	NaOH	KOH	CsOH	TMAOH
		Densities[a]			
α (g cm^{-3} mol^{-1} L)	32.863	47.014	52.060	134.69	1.5090
β (g cm^{-3} mol$^{-3/2}$ L$^{3/2}$)	−4.4586	−4.5044	−3.8457	−3.4250	3.5818
max deviation[d]	0.146	0.106	0.232	0.117	0.087
av deviation[e]	0.081	0.032	0.079	0.052	0.051
		Viscosities[b]			
10^2A (mPa s mol$^{-1/2}$ L$^{1/2}$)	0.17069	0.31671	−0.17848	−1.7756	−0.74362
10^2B (mPa s mol^{-1} L)	1.9860	−11.116	29.305	176.45	158.22
10^2D (mPa s mol^{-2} L^2)	11.713	7.2120	1.7770	−22.370	−54.006
10^4E (mPa s mol$^{-7/2}$ L$^{7/2}$)	−20.006	11.790	7.8696	54.136	841.47
max deviation[d]	0.79	7.23	4.59	9.70	6.83
av deviation[e]	0.44	2.31	1.55	5.51	3.04
		Viscosities[c]			
10^2a (mPa s mol^{-1} L)	21.893	20.275	8.6933	28.356	−43.446
10^2b (mPa s mol^{-2} L^2)	8.7110	2.2961	1.2890	−18.395	−52.967
10^4d (mPa s mol^{-3} L^3)	−415.92	6.1979	1.9984	6755.3	5487.1
10^5e (mPa s mol^{-4} L^4)	1535.9	89.526	8.2422	9362.0	−19.649
10^5f (mPa s mol^{-5} L^5)	−165.66	2.9925	1.1563	490.1	2759.9
max deviation[d]	0.12	1.04	1.18	3.24[f]	1.65
av deviation[e]	0.07	0.30	0.57	1.79[f]	0.63

[a] Using eq 5. [b] Using eq 8. [c] Using eq 9. [d] The largest difference between the observed and calculated values expressed in %. [e] The average difference between the observed and calculated values expressed in %. [f] Point at the highest concentration of CsOH has been omitted.

satisfactorily the viscosities of a number of strong binary electrolytes (NaCl, KCl, and $CaCl_2$) over the entire experimentally accessible concentration range. From the data of Table 6, however, it is apparent that this equation is not satisfactory with our solutions: average deviations were as much as 5.5%, with maximum deviations up to 10%. The agreement between the observed and optimized viscosities were most unsatisfactory at the highest concentrations. Therefore, for practicality, the data were instead fitted using a fifth-order polynomial expression:

$$\eta = \eta_0 + ac + bc^2 + dc^3 + ec^4 + fc^5 \qquad (9)$$

This equation is able to describe almost all the electrolytes over the entire concentration range with a satisfactory average error of ≤ 0.6% (Table 6). The only exception is the $CsOH-H_2O$ system. The irregular behavior of the latter is most likely to be associated with the sudden and sharp upturn of the η vs c(CsOH) function at > 5 M concentrations.

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