Conductometric, Spectrophotometric, and Viscosity Studies of Some Salts of Cadmium and Cerium Dissolved in Methanol

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Spectrophotometric (2000 to 6000 A), viscosity and conductance data for $CdCl_2$, $CdBr_2 \cdot 4H_2O$, $Cd(C_2H_3O_2)_2 \cdot 2H_2O$, $CeCl_3 \cdot 7H_2O$, $Ce(ClO_4)_3 \cdot 6H_2O$ or $Ce(NO_3)_2 \cdot 6H_2O$ dissolved separately in methanol at selected temperatures between 4.5° and 45° C. are given. Viscosities of water–methanol solutions were measured at 5°, 20°, and 45° C. for 28 water concentrations between 0.00006 and 6.050 moles per liter. All data were processed by a digital computer and an integrated system of FORTRAN programs. The method of least squares was used throughout. Data compatibility tests were made for the viscosity (Jones–Dole, Dawson–Zimmerman, salt molar viscosity), conductance (Onsager, $\lambda = A[\text{salt}] + B$, $R = \mu_{45}/\mu_{20}$), and spectophotometric (Beer's law, collision equation) equations. These data are not compatible with the Jones–Dole, Dawson–Zimmerman, and Onsager equations. The viscosity data for water–methanol solutions are compatible with the salt molar viscosity equations.

WHILE there is extensive literature on the conductance of CdI_2 or bromide dissolved in nonaqueous solvents, the literature on other cadmium salts and cerous salts in non-aqueous solvents appears to be limited. Schrijver and Van Arkel (*21*) measured the conductance of $CdCl_2$ in pyridine-water mixtures. Fredenhagen and Cadenbach (*11*) determined the solubility and conductivity of cerous and cadmium chlorides in anhydrous HF. The conductivities of methanol solutions of $CdBr_2$ (*23*) and CdI_2 (*12, 20. 24–26*) have been reported. Thonnessen (*26*) found that the conductivity of CdI_2-methanol solutions increases with addition of molecular iodine, and suggested that complex formation must cause ionization. Ionic mobilities of cadmium and chloride ions in methanol at 25° C. have been reported (*15, 27*). Viscosity studies of CdI_2 in methanol have also been made (*12, 24*). Evans (*10*) measured the absorption spectra for cadmium chloride in the vapor phase.

de Maine and McAlonie (*4*) reported that *R* (the ratio of the molar conductance at 45° C. to the molar conductance at 20° C.) for first transition group metal chlorides dissolved in methanol is independent of the salt concentration and the degree of hydration. For $ZnCl_2$, *R* increases slowly with increasing salt concentration and is less than one. de Maine and Walsh (*8*) found that *R* for $SnCl_2 \cdot 2H_2O$ and $SnCl_4 \cdot 5H_2O$ in the same solvent is constant. For a majority of the methanol systems investigated to date, plots of the specific conductance *vs.* salt concentration, with temperatures fixed, yielded two intersecting straight lines.

The term *Q* (the ratio of the salt molar viscosity at 25° C. to the salt molar viscosity at 45° C.) is independent of the salt concentration at both temperatures (*5*). Recently (*7*), viscosity data for methanol solutions of chlorides of the first transition metals and tin have been used to test the Jones-Dole (Jones-Talley) (*18*), Dawson-Zimmerman (*1*), and salt molar viscosity (*5*) equations.

This report presents conductance-spectrophotometric-viscosity data for $CdCl_2$, $CdBr_2 \cdot 4H_2O$, $Cd(C_2H_3O_2)_2 \cdot 2H_2O$, $CeCl_3 \cdot 7H_2O$, $Ce(ClO_4)_3 \cdot 6H_2O$ or $Ce(NO_3)_3 \cdot 6H_2O$ dissolved in methanol at selected temperatures between 4.5° and 45.0° C.

EXPERIMENTAL

The materials used and the concentrations studied are shown in Table I. The spectrograde methanol was saturated with dry nitrogen (dew point -60° C.) immediately before densities of several solutions (water-methanol or salt-methanol) at each temperature showed that the calculated Electrical resistances of all methanol-salt solutions were measured at 4.5° to 5.0°, 20°, and 45° C. with a General Radio Co. impedance bridge, Type 1650 A, and Leeds and Northrup cells with constants between 0.1000 and 23.00. Duplicates of selected solutions of each salt showed that resistances are easily reproduced to within 2%. No changes in the resistances of these solutions were observed even after 9 hours.

Flow times for the salt-methanol solutions were measured with the electroviscometer (*6*) and, in some instances, with normal Ostwald-Fenske viscometers at 20° and 45° C. Flow times for 28 water-methanol solutions (water concentration from 0.00006 to 6.050*M*) were measured at 5°, 20°, and 45° C. in the normal way. Densities of the components were used to calculate the density of each solution at each temperature. Direct measurement of the densities were, at most, wrong by 0.2%. Viscosity coefficients (*6*) determined from these densities and flow times were shown by statistical methods to be within 0.57% of the actual value.

The ultraviolet absorption spectra for each solution were measured between 2000 and 6000 A. at 20° and 45° C. with two calibrated, thermostated Beckman Model DU line-operated spectrophotometers, and at room temperature 21° to 24° C.) with a Bausch and Lomb Spectronic 505 with matched 1 cm. glass-stoppered quartz cells with methanol as reference. Maximum error for each individual acceptable absorbance (from 0.060 to 1.200) was estimated to be 2%. The spectra did not change with time.

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Table I. Compounds and Concentration Ranges Used

Compound	Quality	Concn. (Moles/Liter × 10,000) at 20° C.	No. of Samples	Analysis (%, Deviation From Standard)
Methanol	Fisher Spectroanalysed Lot No. 710741	...	...	...
$CdBr_2 \cdot 4H_2O$	Fisher Certified reagent (Contg. 0.30% Cl) Lot No. 783355	25.72–2568.0	12	Br, 1.12%[a]
$CdCl_2$	Fisher Certified reagent (Assayed 99.1 % $CdCl_2$, anhydrous) Lot No. 701577	13.39–339.8	9	Cl, 0.52%[a]
$Ce(NO_3)_3 \cdot 6H_2O$	Fisher Laboratory purified Lot No. 700201	10.09–10096.0	18	Ce, 0.12%[b]
$CeCl_3 \cdot 7H_2O$	G.F. Smith Reagent	4.843–4861.0	18	Cl,–2.62%[a]
$Ce(ClO_4)_3 \cdot 6H_2O$	G.F. Smith Reagent	0.7406–743.3	18	Ce,–1.09%[b]
$Cd(C_2H_3O_2)_2 \cdot 2H_2O$	Fisher Certified reagent Lot No. 794549	15.12–15133.0	18	Cd,–0.40%[c]

[a] Fajan absorption indicator method (*14*, p. 300). [b] Gravimetric oxalate method (*22*, p. 248). [c] Gravimetric sulfide method (*22*, p. 201).

DATA PROCESSING METHOD

All data were processed with an IBM 650 Digital Computer and an integrated system of FORTRAN programs. Incorporated into these programs are corrections for temperature–density–volume–concentration changes and instructions for rejecting all sets of conjugate information containing unacceptable datum. This is the Fail-Safe Procedure. In this work sets of conjugate information were discarded if the specific conductance (input information) was less than 100 times the conductance of the solvent itself; if $(\eta - \eta_0) \leqq (1/16)\eta_0$; or if the measured absorbance was less than 0.060 or greater than 1.200 units. Since the maximum error in η (solution viscosity coefficient) is $(0.57\ \eta/100)$, the condition set means that the maximum error for each $(\eta - \eta_0)$ where η_0 is solvent viscosity coefficient, is nearly 4%.

Ordinates for the equation to be tested are computed from accepted information, and the Self-Judgment Principle is used to determine whether or not the actual input information is compatible with the given equation to within preselected instrument reliability factors. Here input information included flow times, measured absorbances, volumes of solution components before mixing (to calculate concentrations and densities), and specific conductivity (2%); $(\eta - \eta_0)$ [4%, corresponding to a maximum error 0.50 per cent in η (solution viscosity coefficient)], and measured optical densities (2%).

The following equations were tested for data compatibility: viscosity [Jones-Dole (*18*), Dawson-Zimmerman (*1*), and the two salt molar viscosity (*5*) equations]; conductance [Onsager, and the new equations, $\lambda = AC + B$ and $R =$ constant (*8*)]; spectrophotometric [Beer's Law and the collision (*3*) equations]. In these equations C is the salt concentration, λ is specific conductance, and R is the ratio of the molar conductance at upper temperature to the molar conductance at lower temperature. A and B are constants. None of the data discussed here is compatible with the Jones-Dole (*18*), Dawson-Zimmerman (*1*), Onsager or collision (*3*) equations.

RESULTS

Viscosity. Plots of viscosity coefficient *vs.* water concentration (0.00006 to 6.050 moles/liter) for water-methanol solutions are linear for each temperature studied, Figure 1. Each of these straight lines extrapolates [$(H_2O) = 0.0$] to exactly the viscosity coefficient of pure methanol. These new data agree with recent work by Mikhail and Kimel (*19*), and for low water concentrations, with work reported by Hartley and Raikes (*16*). The water molar viscosity values (η_{H_2O}) and Q ($= \eta_{H_2O}$ at $t_1^\circ C/\eta_{H_2O}$ at $t_2^\circ C$; $t_2 > t_1$) are independent of the water concentrations except for high water concentrations at 5° C., Table II.

The new data for salt-methanol solutions are not compatible with either the Jones-Dole (*18*) or Dawson-Zimmerman (*1*) equations. For low salt concentrations $(\eta - \eta_0)/\eta_0\ (C)^{1/2}$ *vs.* $(C)^{1/2}$ does appear to be linear, Figure 2, but the negative values found for the constant, A, are not predicted (*13*) by the Jones-Dole equation. Plots of the salt molar viscosity (*5, 7*) *vs.* salt concentration illustrate the data compatibility with the first salt molar viscosity equation (*5*), Figure 3, found for all temperatures and salts studied. For $CdCl_2$, $CdBr_2 \cdot 4H_2O$, $CeCl_3 \cdot 7H_2O$ and $Cd(ClO_4)_3 \cdot 6H_2O$, Q (η_{salt} at 20° C./η_{salt} at 45° C. is independent of the salt concentration to within the experimental error (4%). For $Cd(C_2H_3O_2)_2 \cdot 2H_2O$ and $Ce(NO_3)_3 \cdot 6H_2O$, Q varied from 1.34 to 1.94 and from 1.41 to 1.76 respectively over the concentration range noted in Table I.

Average Q values, ΔH_Q (calculated with van't Hoff's equation and average Q) and the square root-mean square deviations are given in Table III.

Conductance. The new data are not compatible with Onsager's equation at 5°, 20°, or 45° C., even for limited concentration ranges. At low salt concentrations all cerous salts have molar conductances greater than those of cadmium salts, Figure 4. Plots of specific conductance *vs.* $Ce(ClO_4)_3 \cdot 6H_2O$ concentrations define two intersecting straight lines over the entire concentration range studied. For salt concentrations below 0.0300*M* all salts except $Ce(NO_3)_3 \cdot 6H_2O$ and $CdCl_2$ gave single straight line plots. For high salt concentrations appreciable deviations from linearity were observed for all salts except $Ce(ClO_4)_3 \cdot 6H_2O$.

For $CdCl_2$, $CeCl_3 \cdot 7H_2O$ and $Ce(ClO_4)_3 \cdot 6H_2O$ the ratios R (Molar conductance at t_2°C./molar conductance at t_1°C., $t_2 > t_1$) are independent of the salt concentrations for each pair of temperatures (t_2, t_1) studied. R values for $Ce(NO_3)_3 \cdot 6H_2O$ appear to be constant below 0.200*M*. Between 0.200*M* and 1.002*M*, R increases slowly from 1.222 to 1.512 with temperatures of 45° and 20° C. R values for both $CdBr_2 \cdot 4H_2O$ and $Cd(C_2H_3O_2)_2 \cdot 2H_2O$ increase slowly with concentration, Table IV.

Average R ($t_2 = 45°$; $t_1 = 20°$C.) and ΔH_R values (calculated with the van't Hoff equation and average R values) are given together with the square root-mean square deviations and the number of ratios averaged in Table III. In calculating ΔH_R, we have assumed that each salt behaves as a weak binary electrolyte (*4*). For four of the six salts

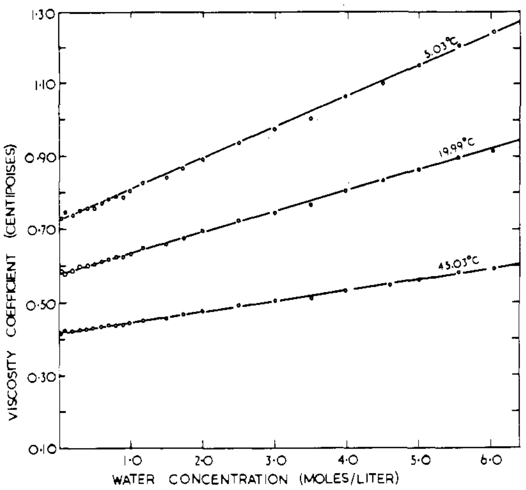

Figure 1. Plot of viscosity coefficient (centipoises) of water–methanol solutions *vs.* concentration of water (moles/liter) at the temperature indicated

Table II. Water Molar Viscosity

Water Conc. (Moles/Liter at 20° C.)	Cp/Mole Liter		
	5.03° C.	19.99° C.	45.03° C.
0.4996	. . .	0.0520	0.0329
0.5995	. . .	0.0517	0.0326
0.6994	0.0743	0.0529	. . .
0.7993	0.0725	0.0526	0.0322
0.8993	. . .	. . .	0.0298
0.9992	0.0728	0.0520	0.0309
1.4988	0.0728	0.0520	0.0302
1.7347	0.0776	0.0548	0.0309
1.9984	0.0792	0.0570	0.0325
2.4980	0.0818	0.0572	0.0326
2.9975	0.0810	0.0554	0.0316
3.4971	0.0776	0.0535	. . .
3.9967	0.0822	0.0563	0.0309
4.4963	0.0836	0.0563	0.0307
4.9959	0.0838	0.0566	0.0303
5.5510	0.0844	0.0571	0.0312
6.0500	0.0835	0.0554	0.0300
Average	0.0815	0.0560	0.0313
Square root-mean square deviation		±0.0011	±0.0009

ΔH_Q (45° C.; 20° C.) = −4.34 (±0.35) kcal.

studied, Table III, ΔH_R is independent of the temperature (between 4.5° and 45° C.) within the experimental error.

Absorption Spectra. Molar absorptivity curves for the six salt-methanol systems at 20° C. are given in Figure 5. None of these salts absorbs light between 4000 and 6000 A. (the upper limit of our measurements). All salts except $Ce(ClO_4)_3 \cdot 6H_2O$ obey Beer's law at both temperatures (20° and 45° C.) and at all wavelengths studied. At fixed temperatures and wavelengths the molar extinction coefficeint for $Ce(ClO_4)_3 \cdot 6H_2O$-methanol solutions decreased by up to 60 per cent as the salt concentration was increased.

The molar absorptivities for $CeCl_3 \cdot 7H_2O$ do not change measurably with the temperature. For $Ce(ClO_4)_3 \cdot 6H_2O$ the molar absorptivities increased by approximately one-fifth between 2200 and 3000 A. on lowering the temperature from 45° to 20° C. With $Ce(NO_3)_3 \cdot 6H_2O$ the same phenomenon was observed at wavelengths below 2600 A. At longer wavelengths the molar absorptivities increased on raising the temperature. Absorptivities of the three cadmium salts studied were smaller at lower temperatures.

DISCUSSION

The new data for concentrated salt-methanol solutions clearly do not fit the Jones-Dole equation (*18*), Figure 2. With dilute solutions the negative values for the Jones-Dole constant, *A*, have no physical meaning. The Dawson-Zimmerman equation (*1*) developed from data for zinc halide-methanol solutions is equally unsatisfactory.

The Einstein equation (*9*) predicts that the salt molar viscosity will be independent of the salt concentration only in the following situations and with species solvated in the normal way:

(a) No polymerization or ionization of the salt occurs.

(b) Salt species + salt species $\rightleftarrows$ complex, where complex concentration is much less than salt concentration.

However, the Einstein equation fails to predict a straight-line dependence with positive slope for the plot of salt molar viscosity *vs.* salt concentration as was found in this work (*7*). de Maine and Russell (*5*, Figure 3) also illustrate the linear dependence of salt molar viscosity on the salt concentration. For four of the six salts studied in this work, constant values were found for *Q* (η_{salt} at 20° C./η_{salt} at 45° C.). Cadmium and cerous salts in methanol have average *Q* values different for each anion, Table III, and different from the approximately constant *Q* (and ΔH_Q) found for all other salt-methanol systems studied (*7*). Thus cadmium and cerous salts may interact differently with methanol than do chlorides of the first-transition metals (*7*) or tin (*5*). Recent work has shown that data for $SnCl_4 \cdot 5H_2O$–isopropyl alcohol–carbon tetrachloride solutions also obey the two salt-molar viscosity equations.

The new viscosity data for water-methanol solutions, Figure 1, Table II, which agree with published work (*15, 18*) suggests that the number of water molecules associated with methanol molecules to form the average water-methanol aggregate is independent of the water concentration between 0.00006*M* and 6.050*M* for each temperature studied. This conclusion is supported by the following facts:

(a) The water molar viscosity is independent of the water concentration at 20° and 45° C. The increased water molar viscosity observed for high water concentrations at 5° C., Table II, can be attributed to increased bonding between water molecules themselves.

(b) The three straight lines shown in Figure 1 extrapolate (at zero water concentration) to exactly the viscosity coefficient for pure methanol (*17*) at 5°, 20°, and 45° C.

Elsewhere we have reported (*2*) situations where small quantities of water do not significantly alter the conductivity of salt-methanol solutions. Walden's rule (*28*) ($\mu\eta$ = constant (*P*)) does not apply to any of the salt-methanol systems discussed here. For each salt, *P* decreased continuously as the salt concentration was increased.

Cadmium salts dissolved in methanol ionize only to a limited extent. For example, with ion mobilities in methanol at 25° C. (*15, 27*) it can be shown that in a 0.01*M* solution only 10% of the cadmium ionizes to form Cd^{++} and Cl^-, or assuming that $CdCl^+$ has approximately the same ion mobility as Ag^+, 20% ionizes to form $CdCl^+$ and Cl^- at 20° C. On the other hand, similar approximate calculations show that in dilute solutions the three cerous salts are more than 25% ionized to form Ce^{+3} and the corresponding anion. Unlike CdI_2 in methanol (*24, 25*) none of the cerous or cadmium salts here studied has a conductance maximum.

Table III. Average R and Q Values for the Salts Dissolved in Methanol

	Conductance, Mho/Mole[a]			Viscosity, Cp[a]		
Salt	No. of Samples	R Values and deviations	ΔH_R (kcal./mole)	No. of samples	Q Values and deviations	ΔH_Q (kcal./mole)
$CdBr_2 \cdot 4H_2O$	11	1.192(1.136–1.271)[c]	2.61	7	1.54(±0.08)	−3.23(±0.38)
$CdCl_2$	6	1.122(±0.012)	1.97[b](±0.28)	4	2.20(±0.11)	−5.84(±0.40)
$Ce(NO_3)_3 \cdot 6H_2O$	11	1.222(±0.021)	2.98[b](±0.26)	7	1.61(1.41–1.76)[c]	−3.52
$CeCl_3 \cdot 7H_2O$	15	1.243(±0.014)	3.23[b](±0.17)	6	1.85(±0.11)	−4.56(±0.45)
$Ce(ClO_4)_3 \cdot 6H_2O$	16	1.341(±0.018)	4.36[b](±0.20)	3	1.18(±0.04)	−1.22(±0.25)
$Cd(C_2H_3O_2)_2 \cdot 2H_2O$	13	1.298(1.73–1.591)[c]	3.88	10	1.62(1.34–1.94)[c]	−3.59

[a] Square-root-mean-square deviations for salts with R and Q independent of salt concentration are given in parentheses. [b] Average ΔH_R between 5° and 45° C. [c] Trend.

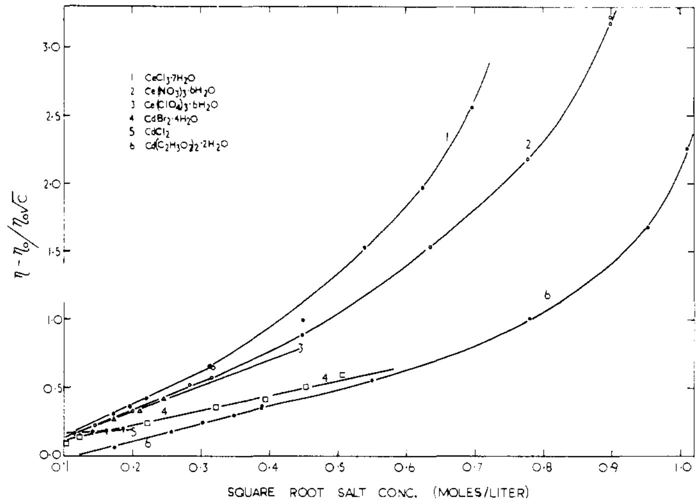

Figure 2. Plots, according to Jones-Dole equation, of viscosity data for emthanol solutions of the indicated salts at 20° C. η and η_0 are the viscosity coefficients of the solution and solvent, respectively

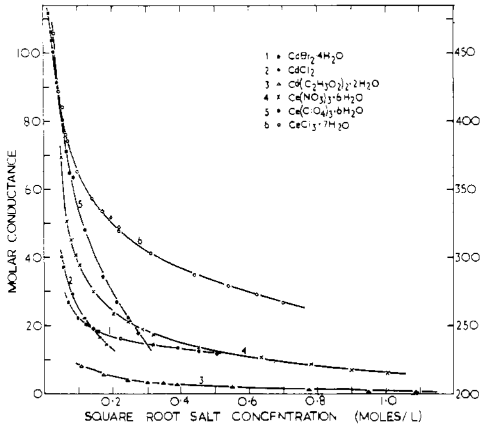

Figure 4. Molar conductance vs. square root of the salt concentration for the indicated salts dissolved in methanol at 20° C. The right ordinate refers to $Ce(ClO_4)_3 \cdot 6H_2O$ data

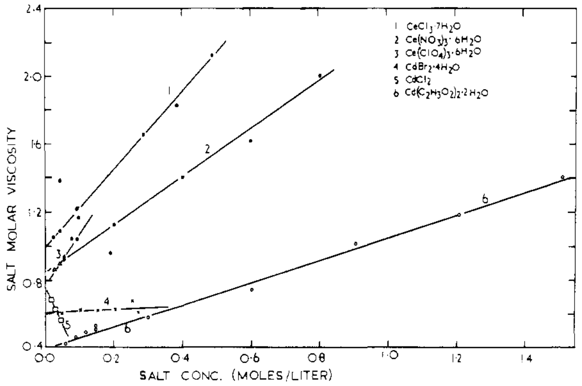

Figure 3. Plot of salt molar viscosity $[=(\eta - \eta_0)/C]$ vs. the salt concentration in moles/liter $[C]$ at 20° C. for the indicated salts. η and η_0 are the viscosity coefficients of the solution and solvent respectively

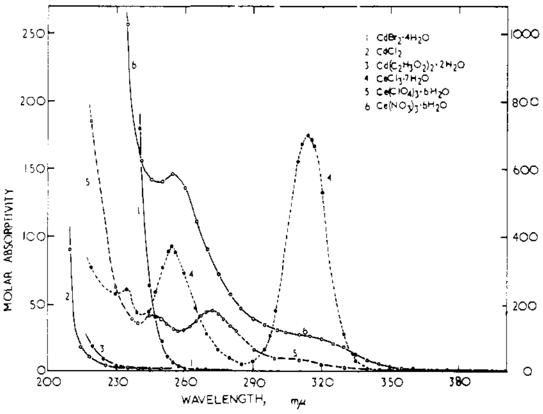

Figure 5. Molar absorptivity for the indicated salts dissolved in methanol at 20° C. The dashed curves ($CeCl_3 \cdot 7H_2O$ and $Ce(ClO_4)_3 \cdot 6H_2O$) are plotted using the ordinates on the right

Table IV. *R* Values for Salt-Methanol Systems

$CdBr_2 \cdot 4H_2O$		$Cd(C_2H_3O_2)_2 \cdot 2H_2O$	
R	Salt conc. (moles/liter)	*R*	Salt conc. (moles/liter)
1.136	0.00515	1.173	0.01209
1.131	0.01029	1.172	0.01512
1.151	0.01543	1.174	0.01512
1.154	0.02058	1.194	0.03024
1.166	0.02572	1.227	0.06048
1.164	0.02572	1.249	0.09072
1.209	0.05143	1.265	0.12096
1.229	0.1028	1.283	0.1512
1.243	0.1542	1.285	0.1512
1.261	0.2055	1.324	0.3024
1.271	0.2568	1.429	0.6050
		1.516	0.9076
		1.591	1.2104
1.192	Average	1.298	

The constant *R* value found for four of the six salts studied indicates that *R* may well have fundamental significance, as mentioned. All conductance equations derived with the assumption that ionization is controlled predominantly or exclusively by the dielectric properties of the medium predict that *R* would be constant only for weak Ostwald electrolytes. The relatively high conductivities found for most salts lay these ideas open to question. Moreover, the ΔH_R values (calculated with the assumption that all salts behave as weak uni-univalent salts) are less than 25% of the expected heats of ionization. The negative ΔH_R values for $ZnCl_2$ (*4*) cannot be explained with accepted theories. There appears to be no simple relation between *R* and *Q*.

It can be shown that for the general reaction: $M_nX_m \rightleftarrows M_nX_{m-1}^{+n} + X^{-n} \rightleftarrows \ldots \rightleftarrows nM^{+m} + mX^{-n}$, Beer's law will be obeyed at all wavelengths where any or all species absorb light if the salt is completely ionized or if the salt is partially ionized and the molar extinction curves for M_nX_m and all positive (or negative) ions are identical and the extinction curves for all negative (or positive) ions are zero. These statements apply regardless of the extent of ion pair formation or the degree of solvolysis of each component. The requirements for partial ionization would not be expected to be met. This applies regardless of the extent of ion-pair formation or the degree of solvolysis of each component. That both the chloride and nitrate of cerium have high conductances and obey Beer's law seem to indicate that both salts ionize completely in methanol at each temperature studied. This is clearly not the case (*15, 27*). A model in which it is supposed that reversible complex formation must precede ionization predicts that Beer's law can be obeyed even if the salt is only partially ionized.

The spectrophotometric data and high molar conductance, Figure 4, for $Ce(ClO_4)_3 \cdot 6H_2O$ indicate that partial polymerization (*i.e.*, to form covalent or ion species) of this salt probably occurs. The high conductivity of its dilute solutions may also be due to methylation with the formation of highly mobile hydrogen ions (*15, 27*).

ACKNOWLEDGMENT

The work was supported by a grant from the Petroleum Research Fund, administered by the American Chemical Society, which is gratefully acknowledged. D.O. Johnston received a Petroleum Research Fund Summer Fellowship during 1961, and a National Defense Education Act Fellowship during the academic year.

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RECEIVED for review October 22, 1962. Accepted May 10, 1963.