

This determination of the arsenic to oxygen distance is, so far as the writer is aware, the only basis for an experimental value of the arsenic radius. Taking the oxygen radius as 0.65 Å., the arsenic radius is 1.36 Å. The radius of antimony is found in the same way to be 1.57 Å.

The arrangement of the atoms within the molecule is the same as that of the carbon and nitrogen atoms in hexamethylenetetramine,  $C_6N_4H_{12}$ , as determined by Dickinson and Raymond.<sup>7</sup> The considerations concerning the tetrahedral angles that hold for the latter compound apply also to arsenious and antimonous oxides.

### Summary

The crystal structures of arsenious and antimonous oxides have been determined by means of Laue photographs and spectral photographs. These crystals may be regarded as composed of  $As_4O_6$  and  $Sb_4O_6$  molecules having a diamond arrangement, each molecule corresponding to 1 carbon atom. See Fig. 1. The arrangements are derived from the space group  $O_h^7$ . The sizes of the unit cubes containing 8 molecules of  $As_4O_6$  or  $Sb_4O_6$  are 11.06 Å. for arsenious oxide and 11.14 Å. for antimonous oxide. The shortest distance between arsenic and oxygen atoms is 2.01 Å., and between antimony and oxygen atoms is 2.22 Å.

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[CONTRIBUTION FROM THE MUSPRATT LABORATORY OF PHYSICAL AND  
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## THE EFFECT OF SUCROSE ON THE ACTIVITIES OF CERTAIN IONS

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### Experiments with Potassium Chloride

With the object of investigating the mechanism of chemical change, a method has been adopted in this Laboratory in the case of reactions in aqueous solutions which consists in making use of an artifice, namely, the replacement of various amounts of the water by sucrose, such replacement being attended by measurable alterations in viscosity on the one hand, and in the activities of the participating individuals, (molecules and ions), on the other. On the basis of this simultaneous alteration, information can be obtained regarding the nature of the process taking place in presence of the sucrose. In the first instance, it is necessary to obtain, by direct experiment, a value for only the actual change in activity produced by the sucrose, in order to apply the method. At the same time, the question naturally arises as to the mechanism of the alteration in activity itself, as in the case of an ion, and in a previous communica-

<sup>7</sup> Dickinson and Raymond, *THIS JOURNAL*, **45**, 22 (1923).

tion<sup>1</sup> the attempt was made to account for the alteration in the activity of the chloride ion in terms of the available water present. In the paper referred to, the activities of the chloride ion, in the presence of various amounts of sucrose, were computed from measurements of the electromotive force of cells *with* transport, using silver-silver chloride electrodes. The expression for the e.m.f.'s of such cells is

$$E = 2n_K \frac{RT}{F} \log \frac{Cl_I}{Cl_{II}}$$

where  $Cl_I$  and  $Cl_{II}$  are the activities of the chloride ion in the solutions and  $n_K$  represents the transport number of the potassium ion. It was assumed that in solutions of potassium chloride, the activities of the two ions were equal, even in the presence of sucrose (an extension of the MacInnes<sup>2</sup> postulate which states that in aqueous solutions of potassium chloride the activities of the potassium and chloride ions are equal). Further, it was assumed that the transport number of the potassium ion retained the same value in solutions containing sucrose as in aqueous solutions. It was shown that the general nature of the results obtained justified these two assumptions.

The most important inference drawn from the results obtained by means of the cell with transport was that the potassium and chloride ions are soluble in the water of hydration of sucrose, the activities of the ions being calculable with a fair degree of accuracy on this basis, activity being regarded at the same time as a concentration expressed in terms of available solvent. When the concentration of sucrose was large, however, the observed activities tended to become slightly less than those calculated on the assumption that increase in activity was due to the decrease in the total amount of water present in the solution, owing to the sucrose. These differences may have been due to experimental error. There was also a possibility that the transport number of the potassium ion had slightly, but sensibly, changed and, hence, had influenced the values of the activities calculated from the observed e.m.f.'s. This being the position it was deemed advisable to verify the results already obtained by means of the corresponding cell *without* transport, namely,  $Ag | AgCl | KCl | K_xHg |$   
I

$KCl$  and sucrose  $| AgCl | Ag$ . The e.m.f. of this type of cell is given by the  
II

expression,  $E = \frac{2RT}{F} \log \frac{Cl_I}{Cl_{II}}$ , on the assumption that the activities of the potassium and chloride ions are equal. Any pronounced changes in the transport number of the potassium ion should be demonstrated by a comparison of the results obtained by means of the two types of cell.

<sup>1</sup> Corrnan and Lewis, THIS JOURNAL, 44, 1673 (1922).

<sup>2</sup> MacInnes, *ibid.*, 41, 1086 (1919).

### Experimental Part

The electrical instruments and silver-silver chloride electrodes were as described in a previous paper.<sup>1</sup> A type of flowing amalgam electrode, identical with that used by MacInnes and Parker,<sup>3</sup> was employed. The precautions adopted by these authors were followed, and their statements were completely verified by the present investigation. The potassium amalgam was also prepared according to their recommendations and, provided that air was excluded, it was found to be very satisfactory. The amalgam was kept under pure, dry hydrogen.

All measurements of the cell were carried out at 25°. The concentrations of potassium chloride used were 0.5 *N* and 0.1 *N*, respectively, (moles per liter of solution) and the sucrose was introduced into the decinormal solution of potassium chloride.

The expression from which the activities were calculated was the following,  $E = 2 \times 0.059 \times \log \frac{0.324}{a_{\text{Cl}_{0.1}}}$ , where 0.324 is the activity of the chloride ion in 0.5 *N* potassium chloride and  $a_{\text{Cl}_{0.1}}$  is that in the decinormal solution in the presence of sucrose. Table I contains the values of the observed e.m.f.'s and also the activities calculated from these values by means of the above expression. The values marked with an asterisk were interpolated by a graphical method.

TABLE I

ACTIVITIES OF CHLORIDE ION IN 0.1 *N* SOLUTION IN THE PRESENCE OF SUCROSE

Sucrose per 100 cc. of solution G.	E.m.f. obs.			Av. e.m.f.	$a_{\text{Cl}_{0.1}}$
	Volts	Volts	Volts		
0	0.0738	0.0741	0.0740	0.0740	0.0764
10	.0706	...	...	...	.0817
20	.0674	...	...	...	.0870
30	.0642	...	...	.0642	.0925
40	.0600	.0598	.0602	.0600	.1005
50	.0556	...	...	.0556	.1095
60	.0510	...	...	.0510	.1198
70	.0454	.0456	...	.0455	.1333

In Table II are shown the activities of the chloride ion in decinormal solutions of potassium chloride in the presence of sucrose, calculated from the e.m.f.'s of the cells with and without transport, respectively. In the last column are given the "theoretical" activities,<sup>4</sup>  $a_2$ , of this ion, calculated in terms of total water, on the basis that the activity is a corrected concentration and that sucrose, beyond the fact that it displaces a certain amount of water, is otherwise inert.

<sup>3</sup> MacInnes and Parker, *THIS JOURNAL*, **37**, 1445 (1915).

<sup>4</sup> In order to determine the values of the "theoretical" activities in terms of total water, the compositions of the solutions by weight were found and the concentrations of potassium chloride were expressed as moles per 1000 g. of total water (see the first 3 columns of Table II). The activity coefficients corresponding to these concentrations were obtained from the data of Noyes and MacInnes [*THIS JOURNAL*, **42**, 239 (1920)]. The product of these concentrations and the corresponding activity coefficients gave the "theoretical" activities shown in Table II.

TABLE II  
COMPARISON OF RESULTS OBTAINED BY MEANS OF CELLS WITH AND WITHOUT TRANSPORT,  
RESPECTIVELY

Sucrose per liter of solution G.	Total water per liter of solution of water G.	KCl per 1000 g. of water Moles	Cell <i>with</i> transport		Cell <i>without</i> transport		Theoretical activities	
			$a_{Cl_{0.1}}$	Relative activity	$a_{Cl_{0.1}}$	Relative activity	$a_2$	Relative activity
0	995.05	0.1005	0.0760	1.00	0.0764	1.00	0.0749	1.00
100	933.0	.1072	.0809	1.06	.0817	1.07	.0795	1.06
200	871.6	.1147	.0865	1.14	.0870	1.14	.0850	1.135
300	809.5	.1235	.0921	1.21	.0925	1.21	.0909	1.21
400	748.0	.1337	.0993	1.31	.1005	1.31	.0981	1.31
500	681.7	.1467	.1070	1.40	.1095	1.43	.1068	1.43
600	618.8	.1616	.1158	1.52	.1198	1.57	.1168	1.56
700	554.1	.1805	.1258	1.66	.1333	1.74	.1289	1.72

It was shown previously<sup>1</sup> that the activities obtained by means of a consideration of the cell with transport showed an increase parallel with those of  $a_2$  ("theoretical" activities) calculated in terms of total water, except in the case of solutions containing 60 and 70% of sucrose. In these cases, the observed activities were found to be slightly less than those calculated. This slight divergence does not appear when activities are calculated from the e.m.f.'s of cells without transport. Hence the conclusion that the potassium and chloride ions are soluble in the water of hydration of sucrose is fully substantiated by the results obtained by means of the cell without transport.

### The Transport Number ( $n_K$ ) of the Potassium Ion in the Presence of Sucrose

In Table III are given the ratios of the e.m.f.'s of the two types of cell or, in other words, the values for the transport number of the potassium ion in solutions of potassium chloride, in the presence of sucrose. On account of the fact that the value for  $E_{obs.}$  for 70% sucrose (cell *with* transport), namely, 0.0240, is the mean of several values which exhibited among themselves a variation of 1 mv., the value of the transport number is also an average value, between the limits 0.514 and 0.530, which is a considerable variation for a transport number. This example demonstrates the effect of a comparatively small error in the observed e.m.f. on the value of the transport number obtained. However, the results tend to show that the transport number of the potassium ion does increase slightly in 60 and 70% solutions, but remains practically constant up to 50% sucrose. That is, the assumption made in the previous paper,<sup>1</sup> regarding the constancy of the transport number of the potassium ion, is approximately justified.

It is a somewhat striking conclusion that, up to 50% sucrose solutions, the transport number of the potassium ion, and also of the chloride ion, should hardly alter, even though the viscosity increases 7-fold.

TABLE III  
TRANSPORT NUMBERS OF POTASSIUM ION

Sucrose %	Viscosity of solution	$E_{obs.}$ (cell with transport)	$E_{obs.}$ (cell without transport)	Transport no. of potassium "K"
0	1.00	0.0368	0.0740	0.497
10	1.32	.0352	.0706	.498
20	1.80	.0335	.0674	.497
30	2.63	.0319	.0642	.497
40	4.09	.0300	.0600	.500
50	6.86	.0281	.0556	.505
60	12.77	.0261	.0510	.511
70	25.93	.0240	.0455	.527

### Discussion of Results

Harned<sup>5</sup> has published results which serve to verify the conclusion arrived at during the present investigation regarding the solubility of the chloride ion in the water of hydration of sucrose. He considered cells of the following types: (1)  $H_2 | MeCl (c) \text{ in } HCl (0.1) | Hg_2Cl_2 | Hg$ ; (2)  $H_2 | MeCl (c) \text{ in } HCl (0.1) | HCl (0.1) | H_2$ , where Me represented potassium, sodium or lithium. The concentration of hydrochloric acid in each case was 0.1 M, while that of the alkali chloride was denoted by  $c$ . Hence the total chloride concentration was  $(c + 0.1)$ .

The free energy changes in these 2 cells are given by the following expressions.

$$\text{Cell 1} \quad -(\Delta F)_1 = F(E_0(1) - E(1)) = RT \log \frac{a_{H_2} \cdot a_{Cl(c+0.1)}}{a_{H_{0.1}} \cdot a_{Cl_{0.1}}}$$

$$\text{Cell 2} \quad -(\Delta F)_2 = FE_2 = RT \log \frac{a_{H_2}}{a_{H_{0.1}}}$$

where  $-(\Delta F)$  denotes decrease in free energy,  $E_0$  the e.m.f. of the cell when  $c$  is zero, and  $E$  that when there is salt present;  $a_{H_2}$  denotes the activity of the hydrogen ion in the presence of salt. The value of the e.m.f. of Cell 2 was determined indirectly, in order that liquid-liquid potential differences should not be involved. If we subtract the value of Cell 2 from that of Cell 1, the following expression is obtained.

$$E_0(1) - E(1) - E(2) = \frac{RT}{F} \log \frac{a_{Cl(c+0.1)}}{a_{Cl_{0.1}}}$$

In Table IV is given a summary of the results of Harned for the values of  $\frac{RT}{F} \log \frac{a_{Cl(c+0.1)}}{a_{Cl_{0.1}}}$ , in the cases of potassium, sodium and lithium chlorides, respectively. The value of the logarithmic expression for the same value of  $c$  is practically the same in the cases of all 3 salts. If the chloride ion were insoluble in the water of hydration of the accompanying cation, one would

<sup>5</sup> Harned, THIS JOURNAL, 42, 1808 (1920).

expect that the value of the expression would be greatest in the case of lithium chloride solutions and least in those of potassium chloride, since, according to Washburn,<sup>6</sup> the lithium ion is much more heavily hydrated than the sodium ion, which in turn possesses a greater degree of hydration

TABLE IV  
HARNED'S RESULTS

$c$ (moles) liter		0.2000	0.5000	1.000	2.000	3.000
$\frac{RT}{F} \log \frac{a_{Cl_{0.1}}}{a_{Cl_{1.1}}}$	KCl...	0.0244	0.0404	0.0554	0.0724	0.0834
	NaCl...	.0241	.0401	.0548	.0712	.0821
	LiCl....	.0244	.0400	.0555	.0718	...

than the potassium ion. In 2.00 *M* solutions of these salts the different "effective" concentrations of the chloride ion which would be thus caused, if this ion were insoluble in the water of the hydration of the cations, would certainly affect the value of  $a_{Cl_{(c+0.1)}}$  in such a way that it would be much greater in the solution of lithium chloride than in that of potassium chloride. But this is not so, as the table shows that the logarithmic expression is very slightly greater in the case of potassium chloride, probably due to slight experimental error. Hence, one can only conclude that the chloride ion is soluble in the water of hydration of the accompanying cations, a conclusion which is the analog of that arrived at in the present investigation, for the case of sucrose.

#### Experiments with Barium Chloride

The principle of the method employed for the measurement of the activities of the barium ion in the presence of sucrose is essentially the same as that employed in the case of the potassium and chloride ions. The cell without transport was used, namely, Ag | AgCl | BaCl<sub>2</sub> | Ba<sub>x</sub>Hg | Ba-  
I II  
Cl<sub>2</sub> and sucrose | AgCl | Ag. The e.m.f. of this cell, in which solutions of a uni-bivalent electrolyte are used, is given by the expression

$$E = \frac{3RT}{2F} \log. \sqrt[3]{\frac{Ba_I \cdot Cl_I^2}{Ba_{II} \cdot Cl_{II}^2}}$$

where  $Ba_I$  and  $Cl_I$  are the activities of the barium and chloride ions, respectively, in the first solution and  $Ba_{II}$  and  $Cl_{II}$  are those in the second solution. The value of  $\sqrt[3]{Ba \cdot Cl^2}$  is the mean activity of the ions or, in other words, the activity of the barium chloride. It is evident that, knowing the mean activity in the first solution, a measurement of e.m.f. enables one to calculate the value in the second solution.

In order to obtain the individual barium-ion activities from a knowledge of the mean activities, it can no longer be assumed, as in the case of uni-valent chlorides, that in solutions of the same equivalent concentration the activities of the chloride ion in a uni-univalent and a uni-bivalent

<sup>6</sup> Washburn, THIS JOURNAL, 31, 322 (1909).

chloride, respectively, are the same. Such an assumption would be in contradiction to the results of Lewis and Randall<sup>7</sup> and Brönsted,<sup>8</sup> that the activity coefficient of an ion is a function of the total ion concentration or, in the words of Lewis and Randall, of the "ionic strength" of the solution. However, it is possible to calculate indirectly, on the basis of previous conclusions, the relative increase in the barium-ion activity caused by the presence of sucrose. It is known that the chloride-ion activity, in the presence of sucrose, is inversely proportional to the total amount of water in a given volume of solution. Applying this result to the case of barium chloride solutions, it is evident that the relative activities of the chloride ion can be calculated from a knowledge of the compositions by weight of the solutions. In addition, measurements of the e.m.f.'s of the above cell will furnish a means for the calculation of the relative activities of barium chloride, that is, the mean activities of the ions in the presence of various amounts of sucrose. From the values obtained for barium chloride and chloride ion, respectively, it is an easy matter to calculate the relative activities of the barium ion alone; they are the cubes of the relative activities of the barium chloride divided by the squares of those of the chloride ion itself. From the values thus obtained for the barium ion, information regarding its solubility or insolubility in the water of hydration of sucrose can be obtained in a manner similar to that adopted in the case of solutions of potassium chloride.

In order to assign correct values to the activity of the barium ion in the presence of sucrose, the values of the activity coefficients of barium chloride and chloride ion in 0.1 *M* barium chloride solution in the absence of sucrose, as given by Lewis and Randall<sup>7</sup> and Harned and Brumbaugh,<sup>9</sup> respectively, have been employed and the required activity of barium ion has been calculated from them. Using the value so obtained as that of the barium ion when no sucrose was present, the barium-ion activity in the presence of sucrose can be calculated from a knowledge of the relative activities.

### Experimental Part

The amalgam was prepared by the electrolysis of a saturated solution of barium hydroxide, using a cathode of pure mercury and a platinum anode. On passing a current of 1.5 amp. (current density about 50 milli-amp. per sq. cm.) for about 2 hours while the mercury was constantly stirred, a liquid amalgam was formed containing about 0.1% of barium. This amalgam was separated from the solution and washed several times with small amounts of distilled water. It was then dried in a vacuum, with the help of gentle heat, and finally kept under pure, dry hydrogen.

<sup>7</sup> Lewis and Randall, *THIS JOURNAL*, **43**, 1112 (1921).

<sup>8</sup> Brönsted, *ibid.*, **42**, 761 (1920).

<sup>9</sup> Harned and Brumbaugh, *ibid.*, **44**, 2729 (1922).

The amalgam, which closely resembled potassium amalgam, acquired a dark film on the surface and decomposed slowly in water, and rapidly in acid solution.

The measurements were carried out at 25°, in a manner similar to that used in the case of solutions of potassium chloride, the flowing amalgam electrode being again employed, owing to the reaction between barium and water to form barium hydroxide. The concentrations of barium chloride employed were 0.5 and 0.1 moles per liter of solution, respectively, and into the latter solution were introduced various amounts of sucrose.

It was assumed that, in the expression for the e.m.f. of the cell, namely,

$$E = 0.0885 \log \sqrt[3]{\frac{\text{Ba}_{0.5} \cdot \text{Cl}_{0.5}^2}{\text{Ba}_{0.1} \cdot \text{Cl}_{0.1}^2}}$$

the activity of the barium chloride in 0.1 *M* solution when no sucrose was present was unity. Thus the values obtained when sucrose was present gave the relative increase in the activity of barium chloride caused by the sucrose. In Table V are shown the results of the e.m.f. measurements, together with the calculated relative activities (or the relative increase in activity), in the cases of 0, 40 and 60% solutions of sucrose.

TABLE V  
ACTIVITIES OF BARIUM CHLORIDE IN 0.1 *M* SOLUTION IN THE PRESENCE OF SUCROSE

Sucrose %	<i>E</i> <sub>obs.</sub> (volts)		Average e.m.f.	Relative mean activity $\sqrt[3]{\text{Ba} \cdot \text{Cl}^2}$
	0.1% amalgam	0.01% amalgam		
0	0.0523	0.0526	0.0524	1.00
40	.0427	.0426	.0426	1.28
60	.0348	.0343	.0346	1.59

Expressing the concentration of barium chloride as moles of salt per 1000 g. of water, the relative concentrations can be calculated, as in the case of potassium chloride, assuming that the concentration of salt when no sucrose is present is unity. These relative "corrected" concentrations (*c*<sub>corr.</sub>), in terms of total water, are shown in Col. 3 of Table VI. In the fourth column are shown the observed relative activities.

TABLE VI  
BARIUM-ION ACTIVITIES

Sucrose per liter of solution G.	Total water per liter of solution G.	<i>c</i> <sub>corr.</sub> (in terms of total water)	Relative mean activities (obs.)	Barium-ion activities <i>a</i> <sub>Ba</sub>
0	995.6	1.00	1.00	0.029
400	746.0	1.34	1.28	.037
600	615.0	1.62	1.59	.046

The values of *c*<sub>corr.</sub> are slightly higher than those of the relative activities, but it must be remembered that, as the concentration increases, the activity coefficient decreases slightly. If we allow for this decrease in the



activity coefficient, the values in Cols. 3 and 4 will agree more closely. Since there are no accurate experimental data expressing the activity coefficient of barium chloride as a function of concentration, no attempt has been made to allow exactly for the slight decrease in activity coefficient.

It is evident from the results shown in the above table, that the increase in the mean activity of the ions of barium chloride, caused by the presence of sucrose, can be accounted for by simply allowing for the decrease in the total amount of water. In other words, since the chloride ion has been shown to be soluble in the water of hydration of sucrose, the same conclusion is arrived at in connection with barium ion. Therefore, the values shown in Col. 4 represent not only the relative mean activities of the barium and chloride ions, but also the relative activities of the individual barium and chloride ions, respectively. Hence, by assigning the value 0.029 to the activity of the barium ion in 0.1 *M* aqueous solution, the values shown in the last column of Table VI for the actual barium-ion activities, are obtained.

#### Discussion of Results

Regarding the activity of an ion as the product of the concentration and the activity coefficient, the significance of the foregoing treatment lies in the fact that it throws considerable light on the mode of expressing concentration. It is clear that the availability or non-availability as solvent of the water of hydration of ions or non-electrolytes is of primary importance. In the cases examined during the present investigation, the chloride, potassium and barium ions are apparently soluble in the water of hydration of sucrose. In addition, it has been shown, from a survey of Harned's results,<sup>5</sup> that the chloride ion is soluble in the water of hydration of accompanying cations. On the other hand, Moran and Lewis<sup>10</sup> have shown that the hydrogen ion is apparently insoluble in the water of hydration of sucrose, since the increase in its activity caused by sucrose is not accounted for, even by allowing for the water of hydration. In addition, the fact that lithium chloride causes a greater increase in the hydrogen-ion activity of hydrochloric acid than does potassium chloride or sodium chloride, indicates that the hydrogen ion is insoluble in the water of hydration of salts. In view of this, the hypothesis of MacInnes, as extended by Harned,<sup>5</sup> namely, that in solutions of uni-univalent electrolytes of the same concentration and having an ion in common, the activity of the common ion has the same value in every case, is not strictly true. It cannot be true, for example, in the case of strong monobasic acids at the same concentration, for if it were, in view of what has been said, it would follow that the anions must be hydrated to equal extents, a conclusion that is untenable. However, it must be stated that, in view of

<sup>10</sup> Moran and Lewis, *J. Chem. Soc.*, **121**, 1613 (1922).

the facts and conclusions mentioned, one is led to the conviction that the hydrogen ion stands alone in the sense that it is insoluble in water of hydration, either of an ion or of an undissociated molecule. If this conviction be true, then Harned's extension of the MacInnes postulate should hold for all ions, except the hydrogen ion. The reason for its lack of generality is suggested to be the solubility or insolubility of an ion in the water of hydration of a secondary solute (either an ion or a non-electrolyte).

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### Summary

1. By means of measurements of the e.m.f.'s of potassium chloride concentration cells without transport, containing sucrose, the conclusion arrived at in a previous paper that the potassium and chloride ions are soluble in water of hydration of sucrose has been verified.

2. It has been shown that whereas the transport number of the potassium ion in sucrose solutions remains constant up to 50% sucrose concentration, it appears to increase slightly in 60 and 70% solutions.

3. From a survey of Harned's results, it has been shown that the chloride ion is soluble in the water of hydration of accompanying cations. This conclusion is, therefore, analogous to that arrived at in connection with sucrose.

4. Measurements of the mean activities of barium chloride, in the presence of sucrose, by means of the cell without transport, have shown that the barium ion, as well as the potassium and chloride ions, is soluble in the water of hydration of sucrose.

5. Since the solubility or insolubility of an ion in water of hydration is of primary importance in determining its true concentration, in terms of available water, Harned's extension to any ion of the MacInnes postulate regarding the independent activity of the chloride ion in solutions of univalent chlorides of the same concentration, is only valid for those ions which are soluble in the water of hydration of secondary solutes (either ions or non-electrolytes).

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