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Physical Properties of Aqueous Solutions of Sodium Oxalate, Sodium Malonate, and Sodium Succinate, I

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I. Introduction

The disodium salts of oxalic, malonic, and succinic acids are the first members of a family of salts of dibasic acids. Although these salts are rather common, a search of the literature has shown that their physical properties in aqueous solution are not extensively known.

This study is concerned with the determination of six of the more important physical properties of water solutions of these substances including densities, viscosities, surface tensions, refractive indices, electrical conductivities, and freezing point depressions.* Measurements were made on solutions whose concentrations ranged from 0.00500 molal to 0.250 molal for sodium oxalate, and from 0.0100 molal to 1.000 molal for sodium malonate and sodium succinate. The determinations were carried out at three temperatures, 0°, 25°, and 50°C.

In Part I of this report, the experimental data covering all the properties with the exception of conductance will be given. Part II will deal with conductance and a general summary of the investigation.

All three salts are white crystalline solids. Sodium oxalate exists at room temperature with no water of crystallization, while sodium malonate has one mole, and sodium succinate has six moles of water of crystallization.

Interest in this series of compounds arose from the knowledge that the corresponding acids exhibit a type of periodicity in their physical properties, depending on the number of carbon atoms in the chain. This property is well-illustrated by reviewing the melting points and the solubilities of the acids of this series. Table I illustrates this periodic rise and fall of their physical properties.¹

Acid	Number of C atoms	Mpt. °C	Solubility (g./100 g. water)
Oxalic		187	10.2 ²⁰ °
Malonic		135	138160
Succinic		185	6.8 ²⁰ °
Glutaric		97.5	63.9 ²⁰ °
Adipic	6	151	1.4150
Pimelic		105	2.5 ¹⁴ °
Suberic		142	0.14160
Azelaic		106	0.215 °
Sebacic	10	134	0.1170

Table I

Transactions of the Kansas Academy of Science, Vol. 54, No. 2, 1951.

* This study is a continuation of a series of investigations on the physical properties of aqueous solutions of organic salts, the first two of which have been reported in these *Transactions* for December, 1950, and March, 1951.

[233]

It can be seen that the solubilities and the melting points of these acids are determined not only by the number of carbon atoms in the chain, but also by whether the number of carbon atoms is even or odd.

This investigation was carried out to determine if this same periodicity of physical properties is carried out in solutions of the sodium salts of these acids and to supply many data now lacking.

II. Preparation of Materials

A. Sodium oxalate

The sodium oxalate used was prepared by Merck according to U.S. Bureau of Standards specifications, sometimes known as "Sorenson's Primary Standard Sodium Oxalate." No further purification was attempted due to the difficulty of purifying sodium oxalate solutions in glass.^{2, 3}

B. Sodium malonate

This compound had been prepared by the Eastman Kodak Co. and was purified before use. The procedure was as follows:

- 1. A certain volume of hot water was nearly saturated with sodium malonate, and filtered using a Buchner funnel and aspirator, etc., to remove any gross insoluble material;
- 2. This solution was then shaken with absolute alcohol to remove any alcohol-soluble organic material;
- 3. The solution was gently warmed on an electric hot plate while applying the partial vacuum of a water aspirator until a thick syrup was obtained;
- 4. This syrup was allowed to stand until large crystals of salt were formed which were then filtered out;
- 5. This process was repeated until the desired purity was obtained.

Fairly slow crystallization was found to be necessary, and any seeding of the solution with the consequent rapid formation of a mass of small crystals was found to be unsatisfactory. Middle-fraction redistilled water was used for all crystallizations and purifications.

Upon purification, the hydrated salt was oven-dried at 100-120° C., powdered, and redried until anhydrous.

C. Sodium succinate

This salt was likewise dissolved in hot water and filtered to remove any insoluble matter. However, in this case the addition of alcohol, salted-out the sodium succinate and it was then filtered from the solution. This process was repeated until the desired purity was obtained. The purified salt with six moles of water of crystallization was oven-dried at 100-120° C., powdered, and redried until anhydrous.

Both sodium oxalate and sodium succinate can be separated from water solution by the addition of alcohol; however, sodium malonate

cannot be purified in this manner because of its very large solubility in water.

III. Equipment and Procedure

A. Temperature Control

Since the physical properties under study vary with temperature, it was necessary to control the temperature to within \pm 0.01° C. or less.

Because of its higher heat capacity, a liquid (water) bath was used, consisting of a glass-walled tank about 15 inches by 30 inches. The tank was agitated with two motor-driven stirrers, and heated with one 500 watt copper tube heater and two 250 watt light bulbs. The stirrer blades were turned so as to lift the water from the bottom to the top. The heating equipment was arranged to give a uniform distribution of heat. Because the external (room) temperature occasionally exceeded 25° C., it was necessary to equip the tank with a copper cooling coil through which was circulated cold or iced water.

Two meta-static mercury thermoregulators were used in conjunction with a commercial thermostat unit. One thermoregulator was set for 25° C. and the other for 50° C. The setting thermometer had been calibrated against a U. S. Bureau of Standards calibrated thermometer. For the temperature of 0° C. an ice-water system was used in the glass tank.

The water bath temperatures in the inner portion of the bath, and away from the heating units, etc., were as follows:

$$0.00^{\circ} \pm 0.01^{\circ}$$
 C.
25.00° $\pm 0.01^{\circ}$ C.
50.00° $\pm 0.02^{\circ}$ C.

B. Preparation of Solutions

Anhydrous salts were used at all times, and were weighed in weighing bottles or weighing dishes with ground glass tops, then heated, cooled, and reweighed to check for loss of moisture. All weighings were made using calibrated brass weights, and a damped chainomatic balance protected from dust and air currents.

Middle-fraction redistilled water was used for making the solutions. Solutions were made by adding the anhydrous salt to a weighed amount of water, or by adding the salt to an Exax flask of water calibrated at 20° C. No measurable difference was found between the solutions made up in these two ways.

C. Density Determinations

A Parker-type pyknometer was used with one capillary arm, and one large bore arm. The large bore arm was marked at the filling level. The tops of the tubes were closed with ground glass caps to prevent evaporation losses. The pyknometer was standardized at each of the three temperatures using middle-fraction redistilled water. The volume of the capillary arm was determined using a mercury thread which was weighed, and subsequently the volume of each division was calculated.

Before each weighing, the pyknometer was wiped with a damp, lintfree linen cloth, allowed to dry, and then weighed using calibrated brass weights and a damped, chainomatic balance whose arms and chain were calibrated.

D. Viscosity Determinations

An Ostwald viscosimeter was used for these measurements. The large bulb was marked at the filling level, and when temperature equilibrium was reached, the volume of liquid was adjusted to this marking. This method was found to be more satisfactory than adding a definite volume of liquid from a pipette because of the effect of the variation of the room temperature on the volume of liquid added. With this procedure, i.e. adjusting to a definite level, the same volume of liquid was used at all three operating temperatures regardless of the room temperature.

A stopwatch was used to determine the time interval for the passage of the volume of liquid between the bulb markings, and readings were obtained that were reproducible within \pm 0.1 second. A viscosimeter was selected that would give a reading of at least 60 seconds when operating at 50° C. with water as the liquid.

E. Surface Tension Determinations

A length of capillary tubing was calibrated for uniformness of bore with a mercury thread, and then the surface tension of water was measured. Using the calculated radius of the capillary, the actual value of the surface tension of water (as given by that capillary) was determined at the three different temperatures. A capillary tube was chosen that gave values that agreed satisfactorily with the I.C.T. values for water.

The capillary tube was placed in a large tinfoil-covered cork alongside a calibrated thermometer. The cork was fitted into a special test tube approximately 5 cm. internal diameter, and approximately 20 cm. long. The system was immersed within one or two centimeters of the top of the large test tube in the water bath and held rigid with a special clamp.

The capillary meniscus was raised or lowered by means of air pressure on a rubber tubing attached to the ends of the capillary tube. The difference between the heights of the capillary meniscus and the meniscus of the body of solution was read by means of a cathetometer. The readings were reproducible within the accuracy of the cathetometer, i.e., \pm 0.01 cm.

F. Refractive Index Determinations

An Abbé refractometer was used in connection with a pump to

move the water of the bath through the instrument and back into the bath with as little temperature drop as possible. The rate of flow of the water was such that a calibrated thermometer of 0.2° C. markings did not indicate any temperature difference between the water bath and the prisms.

The refractometer was adjusted to read the accepted value for the refractive index of water at the particular temperature in use. The readings for water and solutions were taken under as near identical conditions as possible to nullify any possible temperature difference between water bath and prisms. All values of refractive index reported are for the D line.

G. Freezing Point Depression Determinations

The experimental system consisted of a large, insulated Dewar flask in which was suspended a special test tube with an internal diameter of about 5 cm. A smaller test tube fitted with a cork containing a Beckmann thermometer and a stirrer was suspended inside the large test tube. The general arrangement was that of the Beckmann freezing point depression apparatus.⁵

IV. Densities

Density determinations were made using a Parker-type pyknometer. in all calculations of density, the buoyant effect of air was taken into account.

The expression used for the calculation of the densities was:

$$\mathbf{d}_1 = \frac{\mathbf{W}_1 - \mathbf{W}_p}{\mathbf{W}_w - \mathbf{W}_p} (\mathbf{d}_w - a) + a$$

where d_1 is the density of the liquid at t^o C. in g./ml.

 $d_{\rm w}$ is the density of water at t° C. in g./ml.

- W_1 is the observed weight in grams of the pyknometer filled with liquid at t° C.
- $W_{\rm w}$ is the observed weight in grams of the pyknometer filled with water at t° C.

W_p is the observed weight in grams of the empty pyknometer.

a is the density of air in g./ml.

This is a modified form used for the calculation of density,⁶ and is applicable when the water and liquid content are determined at the same temperature. In this expression we used the average density of air as 0.0012 g./ml.

For an accuracy of $\pm 1 \times 10^{-5}$ in a density determination it is advisable to know the values of the temperature, pressure, and relative humidity at the time of each weighing, and then calculate the density of air. However, since these determinations were concerned with an accuracy in the fourth decimal place only, this effect was not taken into account.

The values of the densities of the solutions of the three salts as thus

determined are given in the following tables. In addition, data already available in the literature are tabulated as well. All values reported are in grams per milliliter.

Molality	0 °	25°	50°
0.250		1.0235	
0.200		1.0181	1.0085
0.100		1.0079	0.9986
0.0500		1.0024	0.9935
0.0250		0.9999	0.9909
0.0100	1.0011	0.9981	0.9893
0.00500	1,0005	0.9976	0.9887
Water ⁷	0.9999	0.9971	0.9881
Literature ⁸			-
0.261		1.0254	
Literature ⁹			
0.231		1.0221	
0.152		1.0137	
0.0750		1.0054	

Table II. Densities of Sodium Oxalate Solutions

Table III. Densities of Sodium Malonate Solutions

Molality	0°	25°	50°
1.000	1.1030	1.0946	1.0831
0.500	1.0546	1.0484	1.0381
0.200	1.0228	1.0184	1.0088
0.100	1.0116	1.0078	0.9986
0.0500	1.0057	1.0024	0.9934
0.0100	1,0009	0.9982	0.9892
Water ⁷	0.9999	0.9971	0.9881
Literature ⁹			
0.508		1.0495	
0.431		1.0417	
0.355		1.0341	
0.282		1.0266	
0.209		1.0191	
0.138		1.0117	
0.0680		1.0044	

Table IV. Densities of Sodium Succinate Solutions

Molality	0°	25°	50°	
1,000	1.0992	1.0910	1.0799	
0.500	1.0528	1.0467	1.0366	
0.200	1.0219	1.0176	1.0082	
0.100	1.0109	1.0074	0.9982	
0.0500	1.0056	1.0022	0.9933	
0.0100	1.0011	0.9980	0.9892	
Water ⁷	0.9999	0.9971	0.9881	
Literature ⁹				
0.537		1.0503		
0.394		1.0368		
0.257		1.0235		
0.126		1.0102		
0.0620		1.0036		

The graphical representation of density variation with concentration of sodium malonate solutions expressed as molality is indicated in Figure I, with temperatures as marked. These curves are of normal form for a molality-density plot, and are nearly straight lines as is true for solutions of sodium oxalate and sodium succinate. As can also be seen, the experimental values in nearly all cases agree quite well with previously reported values.

It is possible to determine an equation for the variation of density



with concentration of the salt for any particular temperature. The usual form of the equation is

 $D_x = D_0 + ax + bx^2 + cx^3$

where D_x is the density at any concentration x,

 D_o is the density at zero concentration, or the density of water at that temperature,

a, b, and c are constants.

These constants, a, b, and c, were calculated from the densities of the 0.250 M, 0.100 M, and 0.0100 M solutions of sodium oxalate, and from the densities of the 1.000 M, 0.500 M, and 0.100 M solutions of sodium malonate and sodium succinate. The concentrations are expressed in moles of solute per 1000 grams of water.

For sodium oxalate at 25° C.

 $D_x = 0.9971 + 0.1031x + 0.07472x^2 - 0.2592x^3$ This equation holds within $\pm 0.1\%$ or less throughout.

For sodium malonate at 25° C.

 $D_x = 0.9971 + 0.1081x - 0.01153x^2 + 0.000889x^3$ This equation also fits the data within \pm 0.1% or less.

For sodium succinate at 25° C.

 $D_x = 0.9971 + 0.1039x - 0.00876x^2 - 0.00122x^3$

This expression fits the data with the same accuracy as the above equations.

V. Viscosities

The viscosity, η_w , of the standard, water, is given by

$$\eta_{\rm w} \equiv c \,\rho_{\rm w} \, t_{\rm w} \tag{1}$$

where η_w is the known viscosity of water ρ_w is the known density of water

t_w is the time of flow for water

c is the calibration constant of the instrument.

The viscosity, η , of the solution is given by

$$\eta \equiv c \rho t \tag{2}$$

where η is the viscosity of the solution

 ρ is the known density of the solution

t is the time of flow for the solution

c is the calibration constant.

Dividing (2) by (1), we obtain

$$\eta \equiv \eta_{\rm w} \; (
ho {
m t}/
ho_{
m w} {
m t}_{
m w})$$

If η_w is expressed as the absolute viscosity in centipoises, then η is obtained as the absolute viscosity of the solution in centipoises, providing ρ_w and ρ , and t_w and t are expressed in the same units.

The expression holds true when the time of flow is relatively large and the difference between the viscosity of the unknown and the viscosity of water is small. Since these conditions were fulfilled experimentally, the equation was used without correction terms.

Tables V, VI, and VII show the absolute viscosities of the various solutions expressed in centipoises.

Molality	0°	25°	50°
0.250		0.993	
0.200	1.942	0.975	0.599
0.100	1.872	0.937	0.575
0.0500	1.838	0.918	0.564
0.0250	1.810	0.908	0.557
0.0100		0.900	0.554
0.00500	1.796	0.898	0.552
Water ¹⁰	1.794	0.895	0.549
Literature ¹¹			-
0.100		0.935	
0.0500		0.914	

Table V. Viscosities of Sodium Oxalate Solutions

Table VI. Viscosities of Sodium Malonate Solutions

Molality	0°	25°	50°
1.000	. 3.110	1.508	0.880
0.500	2.311	1.156	0.694
0.200	1.984	0.970	0.604
0.100	1.887	0.943	0.580
0.0500	1.830	0.918	0.565
0.0100	1.806	0.901	0.555
Water ¹⁰	1.794	0.895	0.549
Literature ¹¹			
0.500		1.119	
0.250		0.990	
0.100		0.924	

240

Molality	0°	25°	50°
1.000	3.554	1.662	0.983
0,500	2.490	1.224	0.735
0,200	2.042	1.014	0.623
0.100	1.919	0.953	0.588
0.0500	1.859	0.925	0.569
0.0100	1.807	0.901	0.554
Water ¹⁰	1.794	0.895	0.549
Literature ¹¹			
0.500		1.229	
0.250		1.050	
0.100		0.956	

Table VII. Viscosities of Sodium Succinate Solutions

Figure II shows the variation of viscosity (in centipoises) with concentration (in molality) at the three experimental temperatures as marked for sodium malonate solutions.





The change of viscosity with molality is almost linear for the dilute solutions of all three salts but as the solutions become more concentrated, i.e. for 1 M sodium malonate and 1 M sodium succinate, the viscosity increases more rapidly. The change of viscosity with concentration can be expressed by an equation such as:

 $\eta_{\rm x} = \eta_{\rm w} + {\rm ax} + {\rm bx}^2 + {\rm cx}^3$

where η_x is the viscosity of the solution at concentration x,

 η_{w} is the viscosity of water,

a, b, and c are constants.

These constants can be determined, and for sodium oxalate at 25° C.

using the data for 0.250 M, 0.100 M, and 0.0100 M solutions, the equation was found to be:

 $\eta_{\rm x} = 0.894 + 0.624 {\rm x} - 2.458 {\rm x}^2 + 6.185 {\rm x}^3$ This expression holds within 0.5% or less for all the data.

For sodium malonate at 25° C., the equation is

 $\eta_{\rm x} = 0.894 + 0.4867 {\rm x} + 0.0225 {\rm x}^2 + 0.1050 {\rm x}^3$

This equation was determined from the data for the 1.000 M, 0.500 M, and the 0.100 M solutions. However, for the 0.200 M solution the equation is only accurate within 2%, and for all other concentrations is accurate within 1% or less.

For sodium succinate at 25° C., using the data for the 1.000 M, 0.500 M, and the 0.100 M solutions, the equation was found to be

 $\eta_{\rm x} = 0.894 + 0.5747 {\rm x} + 0.1485 {\rm x}^2 + 0.0450 {\rm x}^3$

This expression is accurate within 0.2% or less for all experimental data.

VI. Surface Tensions

Because of the difficulty of determining the surface tension of liquids using the du Nouy ring method at other than room temperature, the capillary rise method was used with some success. The selected capillary tube was calibrated with water at the three temperatures, since the experimental values were to be calculated using the ratio of two relationships.

The surface tension, γ_w , of water in a capillary tube of small internal diameter was given by

$$\gamma_{\rm w} = \frac{1}{2} r h_{\rm w} g \rho_{\rm w} \tag{3}$$

where γ_w is the surface tension of water,

r is the radius of the capillary,

g is the acceleration due to gravity,

 $\rho_{\rm w}$ is the density of water, and

hw is the capillary rise for water.

Also the surface tension of a solution can be expressed by a similar relationship

$$\gamma = \frac{1}{2} \operatorname{r} \operatorname{h} \operatorname{g} \rho \tag{4}$$

where γ is the surface tension of the solution,

r is the radius of the capillary,

g is the acceleration due to gravity,

 ρ is the density of the solution, and

h is the capillary rise for the solution.

Now if (4) is divided by (3), we obtain the equation

$$\gamma = \gamma_{\rm w} \; (\frac{h\rho}{h_{\rm w}\rho_{\rm w}})$$

This is a simplified expression omitting any correction due to the

density of the vapor around the tube, etc., since the data is not sufficiently accurate to warrant such considerations.

The use of a cathetometer did permit readings to be made to 0.1 mm., but the difficulty in obtaining and reproducing reasonable results would indicate that the surface tension values are probably the least accurate of all measurements here reported.

Tables VIII, IX, and X show the results of the experimental work. The concentration is expressed as molality and the surface tension is expressed as dynes per cm.

The graphical representation of the variation of surface tension with molality at the various experimental temperatures is shown in Figure III for sodium malonate as a typical case.



Fig. 3.

Molality	0°	25°	50°
0.200	75.9	72.8	68.6
0.100	75.7	72.2	68.1
0.0500	75.7	72.0	67.9
0.0250	75.7	72.0	67.9
0.0100	75.7	72.0	67.9
0.00500	75.7	72.0	67.9
Water ¹²	75.64	71.97	67.91
Literature ¹³			
0.25 (ca)		72.3	68.2

Molality	0°	25°	50°
1.000	79.3	75.0	71.6
1.500		73.1	69.0
0.200		72.4	68.2
0.100		72.2	68.1
0.0500		72.1	68.0
0.0100		72.0	67.9
Water ¹²		71.97	67.91
Table X. Surface Ten	sions of Sodium Succina	te Solut	ions
Table X. Surface Ten Molality	sions of Sodium Succina	te Solut	ions 50°
Table X. Surface Ten Molality 1.000	sions of Sodium Succina	$\frac{25^{\circ}}{75.5}$	ions 50° 72.1
Table X. Surface Ten Molality 1.000 0.500	sions of Sodium Succina 0° 79.5 77.5	25° 75.5 73.5	50° 72.1 69.5
Table X. Surface Ten Molality 1.000 0.500 0.200	sions of Sodium Succina 0° 79.5 77.5 77.5 76.3	25° 75.5 73.5 72.4	50° 72.1 69.5 68.4
Table X. Surface Ten Molality 1.000 0.500 0.200 0.100	sions of Sodium Succina 0° 79.5 77.5 76.3 75.9	25° 75.5 73.5 72.4 72.2	50° 72.1 69.5 68.4 68.1
Table X. Surface Ten Molality 1.000 0.500 0.200 0.100 0.0500	sions of Sodium Succina 0° 79.5 77.5 76.3 75.9 75.9 75.9 75.8	25° 75.5 73.5 72.4 72.2 72.1	50° 72.1 69.5 68.4 68.1 68.0
Table X. Surface Ten Molality 0.000 0.500 0.0200 0.100 0.0500 0.0500 0.0500	sions of Sodium Succina 0° 79.5 77.5 76.3 75.9 75.8 75.8 75.8 75.8 75.7	25° 75.5 73.5 72.4 72.2 72.1 72.0	ions 50° 72.1 69.5 68.4 68.1 68.0 67.9

Table IX. Surface Tension of Sodium Malonate Solutions

At low concentrations the surface tensions of the various solutions are identical (or almost identical) with the surface tension of water at the same temperature. As the concentration increases, the deviation from a straight line becomes larger. However, in all three cases, the curves do approach linearity, especially in the region of lower concentration. **VII. Refractive Indices**

Tables XI, XII, and XIII show the variation of the refractive indices of the various solutions at the indicated temperature with concentration expressed as molality.

The graphical representation of the variation of the refractive index with molality is shown in Figure IV for sodium malonate as typical of all three salts.



		A = 0	= = 0
Molality	0°	25°	50°
0.200	1,3388	1.3370	1.3334
0.100	1.3365	1.3349	1.3316
0.0500	1.3353	1.3337	1.3304
0.0250	1.3346	1.3331	1.3297
0.0100		1.3327	1.3293
0.00500		1.3326	1.3291
Water ¹⁴		1.3325	1.3290
Literature ¹⁵			
0.266		1.3385	

Table XI. Refractive Indices of Sodium Oxalate Solutions

Molality	0°	25°	50°
1.000	1.3580	1.3551	1.3515
0.500	1.3471	1.3448	1.3413
0.200	1.3398	1.3378	1.3343
0.100	1.3370	1.3351	1.3317
0.0500	1.3356	1.3338	1.3305
0.0100	1.3344	1.3328	1.3293
Water ¹⁴	1.3340	1.3325	1.3290

Table XIII. Refractive Indices of Sodium Succinate Solutions

Molality	0°	25°	50°
1.000	1.3601	1.3570	1.3530
0.500	1.3478	1.3455	1.3420
0.200	1.3401	1.3381	1.3346
0.100	1.3373	1.3354	1.3320
0.0500	1.3358	1.3340	1.3307
0.0100	1.3345	1.3329	1.3294
Water ¹⁴	1.3340	1.3325	1.3290

The plots of the refractive index versus molality show the same form as illustrated by the other properties, i.e., linear at low concentrations and a slight curvature at higher concentrations.

VIII. **Freezing Point Depressions**

Using a slightly modified Beckmann freezing point depression apparatus, the freezing points of the various solutions were determined. The values are presented in Table XIV as follows:

Molality	Sodium oxalate	Sodium malonate	Sodium succinate
1.000		4.433	4.583
0.500		2.358	2.491
0.200	0.910	0.926	0.971
0.100	0.501	0.481	0.489
0.0500	0.261	0.254	0.260
0.0250			
0.0100		0.051	0.050
0.00500	0,028		

Table XIV. Freezing Point Depressions C°.

The graphical representation of this data shows that at low concentrations the curves are almost linear, but at higher concentrations a slight curvature is found.

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