ELASTIC VISCOUS OLEATE SYSTEMS CONTAINING KCl. XIII 1)

- 1. Influence of the pH and of the temperature on the G— C_{KCl} and n— C_{KCl} curves.
- 2. Substitution of K or Cl in KCl by other cations or anions.
- 3. The $G-C_{KCI}$ and $n-C_{KCI}$ curve for chemically pure K-oleate.

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1. Introduction and methods.

The influence of the KCl concentration on the elastic behaviour has first been investigated in part VI of this series. In the parts VI — XII we have studied with direct or indirect methods, the effects of added organic substances on the $G-C_{KCl}$, $1/\Lambda-C_{KCl}$, $\lambda-C_{KCl}$ and $n-C_{KCl}$ curves. The latter subject is no longer continued in the present communication, in which we will discuss the further investigation of the $G-C_{KCl}$ and $n-C_{KCl}$ curves, as is indicated in the title. We started from Na-oleate, neutral powder from Baker 3) (remarks on this preparation in part X, section 4, small print and section 7) and in section 5 from chemically pure oleic acid. The methods are the usual ones: rotational oscillation, contrivance to excite them (see part VII), marking with electrolytic H₂ (see part X). The experiments were performed in completely filled spherical vessels of 110 or 500 ml capacity, except in the case of the experiments with chemically pure oleic acid, where for economical reasons we had to be content with half filled 110 ml vessels (see part X, section 8, for the conflict between the strive for economical use of the preparations and for smaller experimental errors).

2. Influence of the pH on the $G - C_{KCI}$ and $n - C_{KCI}$ curves.

In all previous parts of this series the elastic oleate systems contained a small constant concentration of KOH (usually 0.05 N) to ensure a pH

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¹⁾ Part I has appeared in these Proceedings 51, 1197 (1948); Parts II—VI in these Proceedings 52, 15, 99, 363, 377, 465 (1949); Parts VII—XII in these Proceedings 53, 7, 109, 233, 743, 759, 975 (1950).

²⁾ Publication no. 8 of the Team for Fundamental Biochemical Research (under the direction of H. G. Bungenberg de Jong, E. Havinga and H. L. Booij).

³⁾ A generous gift of Na-oleate from The Rockefeller Foundation provided the means for the experiments described in this paper.

higher than 12, which excludes all hydrolysis of the oleate. From experiments on the behaviour of dilute oleate systems, performed in other fields of research 4), this precaution did seem to be the indicated one as it could be feared that oleic acid set free by partial hydrolysis would but complicate the elastic behaviour. The aim of the following experiment was to control this expectation. Four 2.4 % stock oleate solutions were made, containing respectively 0.1 N KOH, 0.01 N KOH, no added KOH and 0.00388 N HCl. In ERLEMEYER flasks (with rubber stoppers), a series of mixtures were made with each of these stock solutions according to the receipt: 70 ml stock solution + 70 ml KCl solution (of known and respectively increasing concentrations). After vigorous shaking, a series of 110 ml spherical Pyrex vessels were completely filled (till into the neck) with the above mixtures and the vessels placed overnight in a thermostate of 20°, to become free of enclosed air bubbles. After measuring T and n, the vessels were placed overnight in a thermostate of 30° and the measurements were repeated. Of each series three mixtures with different KCl concentrations were taken every time to determine with a glass electrode (Coleman) the pH at room temperature and the average of these results was taken as the "pH" of the series 5).

The results have been plotted in the figs. 1A and 1B, which show that when the pH is lowered, both the $G-C_{\rm KCl}$ and the $n-C_{\rm KCl}$ curves are shifted in the direction of smaller KCl concentrations and that at the same time the maximum of the n curve decreases (i.e. the damping increases). Compare the survey below.

Lowering of the pH has therefore the same effect as addition of organic substances showing the "action type A" (see part VI), or — using the nomenclature proposed in part XI of this series — exerting a KCl-sparing influence on the oleate system.

Now, when the pH is lowered, oleic acid is set free by hydrolysis and a KCl-sparing influence exerted by the oleic acid is just what we would expect. The unionised oleic acid possesses the same physical chemical character (a not too small carbon chain carrying an unionised polar group at one end) as the *n*-primary alcohols (of not too short a length of the carbon chain), which typically exert a KCl-sparing action (Compare parts VI and VIII).

If we compare fig. 1A and 1B with figure 2 in part VI, showing the KCl sparing influence of n-hexylalcohol, there is still a difference. In the case

⁴⁾ Coacervation of oleate at sufficiently high KCl concentration, see H. G. Bungenberg de Jong, H. L. Booij and G. G. P. Saubert, Protoplasma 29, 536 (1938); viscosity of the elastic viscous oleate system at smaller KCl concentrations, see H. G. Bungenberg de Jong and G. W. H. M. van Alphen, these Proceedings 50, 849 (1947).

⁵) We are aware that the calculated pH values may be systematically erroneous, because of difficulties inherent with the glass electrode at high pH. But they give at least an impression of the order of magnitude of the pH differences between the four series of experiments.

of n-hexylalcohol the G value corresponding to the maximum of the n curve remains practically the same when this n curve is shifted by n-hexylalcohol in the direction of smaller KCl concentrations.

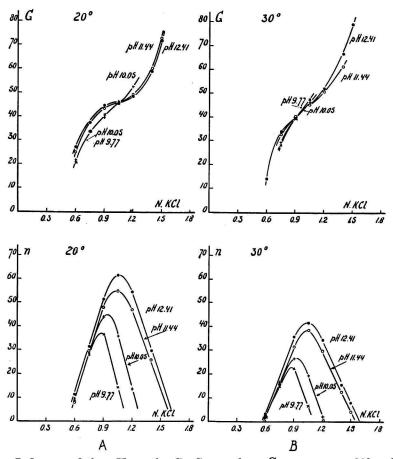


Fig. 1. Influence of the pH on the $G-C_{\rm KCl}$ and $n-C_{\rm KCl}$ curves at 20° and 30°.

In fig. 1, however, the G value corresponding to the maximum of the n curve decreases considerably when the n curve is shifted in the direction of smaller KCl concentrations, as a consequence of the lowering of the pH. See the survey below.

	temp.	рН 12.41	рН 11.44	pH 10.05	pH 9.77
KCl conc. (moles/l) corresp. to max. of the n curve	20°	1.06	1.04	0.95	0.87
	30°	1.03	1.02	0.93	0.87
n at the max. of the n curve	20°	61.5	54.5	45	37
	30°	41.5	38.5	37	23
G at the max. of the n curve	20°	46.5	45.5	42.5	39
	30°	44.5	44	41	38.5

This difference is easily explained if we consider that G depends on the oleate concentration. If n-hexylalcohol is added at a pH > 12 the oleate concentration is not altered. When the pH is lowered oleic acid is produced at the cost of oleate. Thus the actual oleate concentration diminishes.

As G has been shown to be approximately proportional to the square of the oleate concentration (see part III) a percentually small hydrolysis may therefore have a relative large effect on G.

3. Influence of the temperature of the $G-C_{KCI}$ and $n-C_{KCI}$ curves and analogous curves with other salts.

The experiments have been performed with completely filled 500 ml vessels. The endconcentration of Na-oleate (Baker's) was 1.2 % and 0.05 N KOH was present to prevent partial hydrolysis of the oleate. The elastic systems were measured consecutively at 15°, 20°, 25°, 31.3° and 15° (the vessels remained standing overnight in a thermostate of that temperature whereby the measurements were performed the following day). In fig. 2 we give the results.

Comparing the two series of measurements at 15°, we see that the elastic properties have changed to a small extent during the four days in which we worked at higher temperatures (compare dotted curves in fig. 2). This change (probably due to a chemical alteration of the oleate) is, however, small compared with the difference in position of the curves

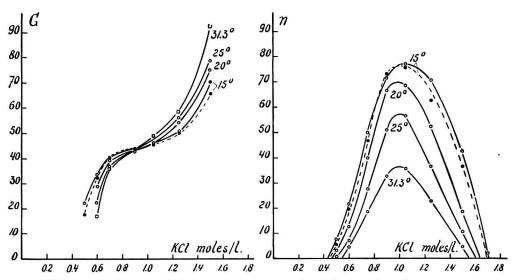


Fig. 2. Influence of the temperature on the $G-C_{KCl}$ and $n-C_{KCl}$ curves.

at 15° , 20° , 25° and 31.3° . Therefore these different positions of the G and n curves give expression to a reversible change of the elastic proporties with the temperature. We may formulate these changes as follows:

1. Dependent on the KCl concentration the change of the shear modulus with temperature has different signs. At increase of the temperature G

decreases at 0.5, 0.6 and 0.75 N KCl and G increases at 1.05, 1.25 and 1.5 N KCl. The G curves of different temperature intersect between 0.9 and 1.0 N KCl.

- 2. At all KCl concentrations n decreases (consequently the damping increases) at increase of the temperature. The maximum of the n curve becomes lower (compare the survey below) and the footpoints of the n curve draw nearer to one another (the left footpoint shifts to the right and the right footpoint to the left).
- 3. At increase of the temperature, the KCl concentration corresponding to that of minimum damping (maximum of the n curve) shifts slightly in the direction of smaller KCl concentrations. The shear modulus corresponding to the maxima of the n curves is, however, practically not changed by this shift of the maximum, to the left. Compare the survey below.

temp	15°	20°	25°	31.3°	15°
KCl concen. (moles/l) corresp. to max. of the n curve	1.06	1.01	0.99	0.98	1.02?
n at the maxim. of the n curve	77	70	57	36	76
G at the maxim. of the n curve	46.3	45.1	46.4	46.6	45.2

The experiments in the sections 2 and 4 have been performed at two temperatures, so that the effect of the temperature could also be seen. Taking into account the reduced accuracy of these experiments with smaller vessels (110 ml), there is still no reason to doubt that the above three points hold very generally. Compare for instance fig. 4, in which this is evident for the six salts with different anions. If we had made analogous graphs for the remaining experiments in the sections 2 and 4, these three points would also have been plainly visible.

4. Substitution of Cl or K in KCl by other ions.

The right footpoint of the curve representing n as a function of the KCl concentration, lies just before the KCl concentration at which coacervation sets in. Supposing this to hold for other K salts too, the determination of their coacervation limits will give us the concentrations below which elastic systems may be expected. We started with a 4% solution of Baker's oleate containing 0.167 N KOH and made in test tubes, a series of mixtures according to the receipt: 3 ml stock oleate solution + x cc stock K salt solution + (7 - x) ml H_2O , which gives mixtures containing, apart from a variable concentration of the investigated K salt, the usual oleate concentration (1.2%) and the usual KOH

concentration (0.05 N). After closing the tubes with rubber stoppers, the contents were mixed thoroughly by shaking; the test tubes were put in a thermostate of 25° and after standing overnight the volume of the coacervate layer was read. The results are represented in fig. 3, which gives

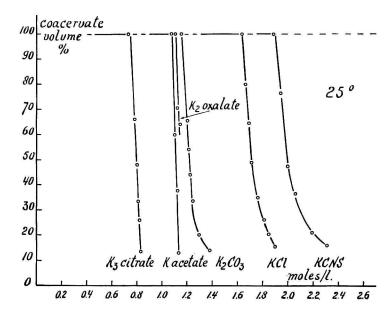


Fig. 3. Determination of the coacervation limit for six potassium salts at 25°.

the volume of the coacervate layer (in percents of the total volume of the mixture) as a function of the salt concentration (in moles/1).

The sequence of the curves in fig. 3 corresponds in the main to the lyotropic series of the anions.

With the same salts we investigated, at 20° and 30° , in completely filled 110 ml vessels, the elastic properties of the 1.2 % oleate systems containing 0.05 N KOH.

For the preparation of the mixtures in the series with potassium oxalate (because of its too small solubility), we could not start from the usual 2.4 % stock oleate solution, but had to use a more concentrated one, viz. a 4 % solution (containing 0.167 N KOH) of which 45 ml were mixed with x ml stock potassium oxalate solution + (105 - x) ml $\rm H_2O$. The end concentration of oleate and KOH is then the same as in the other salt series.

The results obtained with each of the salts apart, have been represented in the diagrams of fig. 4, which show that the typical elastic systems are not restricted to KCl but can also be realized with other K salts. The shape of the $G-C_{\rm salt}$ curves and $n-C_{\rm salt}$ curves is the usual one and in general the same effects of temperature as are discussed in section 3 hold here too.

Fig. 5 enables us to compare the relative positions of the G and n curves

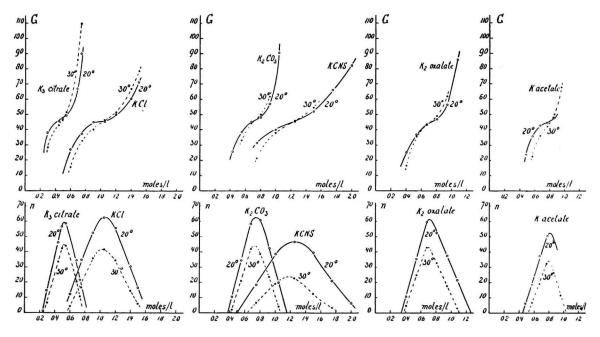


Fig. 4. $G-C_{\rm salt}$ and $n-C_{\rm salt}$ curves for six potassium salts at two temperatures.

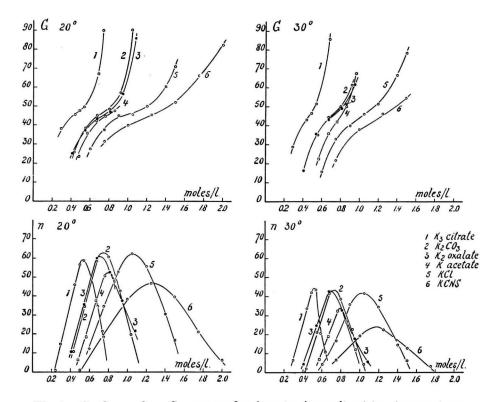


Fig. 5. $G-C_{\rm salt}$ and $n-C_{\rm salt}$ curves for six potassium salts at two temperatures.

with different K salts at the same temperatures. These diagrams show that the order in which the following G and n curves rank from left to right viz.

citrate \(\) carbonate \(\) chloride \(\) thiocyanate

is the same as found for the coacervation limit in fig. 3. The curves for acetate, oxalate and carbonate lie close together. Their relative position is not only different from that in fig. 3, but is even different in the upper and lower graphs of fig. 5.

The next survey gives the salt concentration of the maximum of the n curves, the values of n at the maxima and the values of G corresponding to these maxima:

	temper.	K ₃ - citrate	K_2 - oxalate	K ₂ CO ₃	K- acetate	KCl	KCNS
salt con- centr. moles/l	20° 30°	0.52 0.52	0.71 0.70	0.76 0.73	0.80 0.79	1.06 1.03	1.24 1.16
n	20°	59	61	63	52	63	47.5
	30°	44	43	43	34	42	24
G	20°	49	44	46.5	45.5	45.5	45
	30°	49	44	46.5	44.5	45	43.5

At the comparison of the numerical values of G and n in the above survey, we must take into consideration that the different salt series have not been investigated simultaneously and that for the series with oxalate another stock oleate solution had to be used (see above).

Therefore we may not consider as being with certainty different those G and n values which differ only a few units in G or in n.

We therefore get as a general impression that at the same temperature the n values at the maxima of the n curve are equal in the case of citrate, oxalate, carbonate and chloride, but that the acetate and the thiocyanate ion exert in addition, a lowering influence on the maximum of the n curve. The G values corresponding to the maxima of the n curves are in general also equal (apart from a too high value for citrate 6) which may not be real).

The possibility to realize elastic oleate systems with NaCl proved to be restricted to a narrow tract of temperatures (in practice $25^{\circ} - 35^{\circ}$). At too low temperatures (e.g. 15° , 20°) no stable elastic systems are formed because of transgression of the solubility of the Na-oleate ("curd"-forma-

⁶) We must add the remark that the elastic systems containing citrate were perfectly clear, whereas those with the other salts showed a marked tubidity. It looks as if citrate, by complex formation, removes a contaminating cation present in Baker's cleate, which is responsible for the formation at room temperature of the above turbidity.

tion); at too high temperatures (e.g. 40° and higher) the elastic phenomena disappear because of the highly increased damping.

The actual experiments served at the same time the aim to investigate the influence of the partial replacement of KCl by NaCl. Four series of experiments (1.2 % oleate systems, completely filled 110 ml vessels) were made, one with only KCl, one with only NaCl and two, whereby for the preparation of the mixtures, stock solutions containing KCl+NaCl were used, in which the molar proportions were 2:1 and 1:2 respectively. The results have been plotted in fig. 6 in which the abscissa represent the total salt concentrations (moles NaCl/l+ moles KCl/l). The curves for

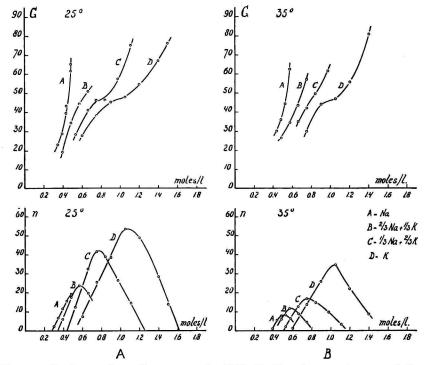


Fig. 6. $G-C_{\text{salt}}$ and $n-C_{\text{salt}}$ curves for KCl, NaCl and two mixtures of these salts (K:Na=2:1 and 1:2) at two temperatures.

NaCl in this figure show the difficulties announced above. At 25° curd formation sets in just at or just before the maximum of the n curve, at 35° , however, though the maximum of the n curve could be passed, the damping is now so high that there are only 8 turning points left. The difficulties are smaller in the next curve (Na: K=2:1), the maximum of the n curve being realizable at 25° , though at higher total salt concentration curd formation still occurred.

The next survey gives the total salt concentrations corresponding to the maxima of the n curves, the values of n at these maxima and the values of G corresponding to the maxima of the n curves.

	temp.	NaCl	2 NaCl + 1 KCl	1 NaCl + 2 KCl	KCl
total salt concentra- tion moles/l	25° 35°	? 0.51?	0.58 0.58	0.77 0.75	1.06 1.04
n	25° 35°	≥ 19 8	24 12	41.5 17	53.5 35
G	25° 35°	? 38?	45 35.5	46 42	48 46.5

We perceive from this survey that in substituting K by Na, both the salt concentration at the maximum of the n curve (= minimum damping) and the value of n at this maximum decrease considerably, whereas G corresponding to the maximum of the n curve also decreases, though much less pronounced.

Using the data of the above survey, the three diagrams of fig. 7 have been constructed. The diagrams A and B give the values of G and of 1/n (an approximate measure for A) as functions of the ratio KCl/KCl + NaCl.

Diagram C gives the concentration of KCl apart (abscissa) and of NaCl apart (ordinates), this being the KCl and NaCl present in the total salt concentration mentioned in the survey for the position of the maximum of the n curve (for the calculation we used here the averages of the values given for 25° and 35°).

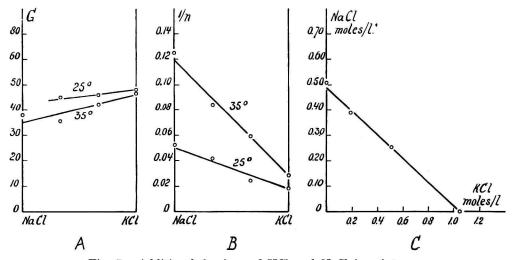


Fig. 7. Additive behaviour of KCl and NaCl in mixtures.

The three diagrams of fig. 7, suggest that in essential points KCl and NaCl behave additively in mixtures.

We may summarize the results obtained hitherto in this section as follows:

- 1. By substitution of Cl or of K in KCl by other ions the G and n curves are generally shifted in the direction of lower or higher concentrations.
- 2. In the case of substitution of Cl by other ions (leaving out of consideration here acetate and thiocyanate which show additional effects) the numerical values of G and n at characteristic points of the G curve (inflexion point) and n curve (maximum) are not altered.
- 3. In the case of substitution of K by Na, however, the above numerical values assume other values.

For a theory of the elastic, KCl containing oleate systems, the above difference between anions and cations seems to be of great importance.

We now turn to the question if elastic oleate systems may be obtained with the chlorides of the other alkalimetals. With LiCl this was not possible; already at small concentrations a fine precipitation was formed (somewhat similar to that with CaCl₂). With RbCl and CsCl, however, elastic systems could be obtained. The quantities of the latter salts at our disposal (a few grams) excluded to investigate those elastic systems in the usual way. By a micromethod ⁷) we could determine approximately the range of saltconcentrations in which the elastic system is present. For comparison we determined this range for KCl with the same method too.

The following results were obtained at approx. 20°.

The values obtained with this method for KCl do not differ more than 0.05 moles/1 from the values one obtains with the macromethod at the same temperature. For NaCl at 25° the elastic properties just begin at about 0.28 moles/1, and at 20° this limit will be still lower (compare footpoints to the left of the n curve for NaCl in fig. 6).

Thus, in the case of the alkalications the concentrations needed to obtain elastic systems increase in the order

$$Na \langle K \langle Rb \langle Cs \rangle$$

From the number of drops added, the dropweight, the weighted quantity of salt, its spec. grav. and its molecular weight we calculated the approximate salt concentration given in the survey above for the two limits of the elastic systems.

⁷⁾ For determination of both limits 100 mgr of the salt was weighted in a small glassvessel. With a drippingpipette we added increasing amounts of a 1.2 % Naoleate solution (containing 0.05 N KOH). After each addition the contents were thoroughly mixed with a pointed glassrod and it was observed under the microscope if the system was still coacervated. Thus we determined the coacervationlimit, which was taken as the upperlimit of the region of elastic systems. These systems have the pecular property that by pulling out of them the glassneedle threads are formed; compare these Proceedings 50, 1227 (1947). At further successive additions of the oleate solution this property becomes at first pronounced, thereafter it diminished. As the lower limit of the region of elastic systems we took that added amount of oleate solution where we could just no longer observe this property.

We would expect Li at the beginning of this series, but, as already stated, the observation of this is prevented by precipitation.

5. The $G - C_{KCI}$ and $n - C_{KCI}$ curve for potassium oleate prepared from pure oleic acid.

In all previous experiments on the elastic behaviour of KCl containing oleate systems we used, because of practical and economical reasons, commercial Na-oleate preparations (see part X, section 7). Most experiments were performed with 1,2 % oleate systems, which is approximately 40 millimoles/1. Thus we never have worked with Na* free systems, but always with such containing a mixture of K* and Na*.

At the KCl (+ KOH) concentration corresponding to the minimum damping (Merck's oleate 1.43 N KCl + 0.05 N KOH; Baker's oleate 1.05 N KCl + 0.05 N KOH) the Na concentration is small compared to the K concentration, viz. the ratio Na/K + Na is 2.6 % and 3.5 % respectively. Taking into account the additive behaviour of KCl and NaCl found in section 4, one is inclined to conclude that this small fraction of Na ions will scarcely alter the elastic behaviour which is to be expected if instead of Na-oleate we had used K-oleate at the preparation of the elastic systems 8).

This conclusion is based on the assumption, that the Na ions from Na oleate are freely interchangeable with K ions and thus no unforeseen effects are present.

Therefore, it seemed desirable to investigate if starting from oleic acid and using KOH to dissolve it, one obtains with KCl the usual $G-C_{\rm KCl}$ and $n-C_{\rm KCl}$ curves.

The actual experiments to control this, served another aim at the same time. The commercial oleate preparations which were used cannot be considered as being chemically pure, as they contain other fatty acids as well (saturated e.g. palmitate and unsaturated ones).

One may therefore ask if the elastic phenomena shown by these oleate preparations are not possibly due to for instance palmitate (BAKER's oleate contains relatively much palmitate, compare part X, section 7), instead of to the oleate itself.

We used for the following experiment chemically pure oleic acid 9).

⁸⁾ In the case of a 4 % substitution of K by Na one calculates a decrease of G from $48 \rightarrow 47.8$ dyne/cm² and a shift of the KCl concentration corresponding to the minimum damping from $1.06 \text{ N} \rightarrow 1.03 \text{ N}$, which are differences of the same order as the experimental errors or even smaller. The influence of a 4 % substitution would only be detectable in the change of n (at the maximum of the n curve), viz. at 25° from $53.5 \rightarrow 49.9$ and at 35° from $35 \rightarrow 30.8$ (calculated from the additivity of 1/n).

⁹⁾ We are much indebted to Mr. C. DE BOCK for preparing a sample of chemically pure cleic acid.

Commercial oleic acid was freed by mercuri acetate from saturated fatty acids, the resulting "oleic acid" distilled in vacuo, the Li-salts of the resulting "oleic

It was dissolved in KOH and to the stock solution of K-oleate, the usual excess of KOH was added to ensure a sufficiently high pH (> 12).

As only a relatively small quantity of pure oleic acid was available and part of it had to be used for more fundamental investigations which are to be published in a next part of this series, we had to use as economical a method as possible. We thus used the so called simplified technique with half filled 110 ml vessels, described in part X. Fig. 8 gives the result at

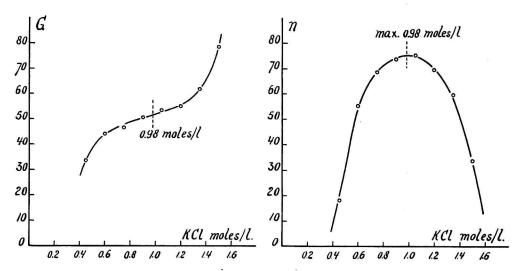


Fig. 8. $G-C_{KCI}$ and $n-C_{KCI}$ curves for potassium oleate prepared from chemically pure oleic acid at 17.8° .

17.8° for an oleate concentration of 37.5 millimoles/1. The $G-C_{\text{KCl}}$ and $n-C_{\text{KCl}}$ curves which were obtained have the usual form and quantitatively do not differ so very much of the analogous curves with the (palmitate containing) Na-oleate from Baker (the KCl concentration corresponding to the minimum damping is \pm 0.98 N; G at this KCl concentration is 50; n at the maximum of the n curve is 74.3).

We may conclude from this experiment that the elastic phenomena studied hitherto, whereby commercial Na oleate preparations were used, are not due to impurities (e.g. palmitate) in these preparations, nor were they bound to appear only in the presence of an amount of Na ions, equivalent to the oleate ions.

6. Summary.

1. The influence of the pH, the temperature and the substitution of each of the ions of KCl by others, on the curves representing G (shear

acid" recrystallized three times from 50 % ethanol to free it from higher unsaturated fatty acids and the oleic acid which was set free was finally distilled in vacuo. Melting point 13°. (See S. H. Bertram, Rec. Trav. Chim. 46, 397 (1927) and H. W. Scheffers, Rec. Trav. Chim. 46, 293 (1927).

modulus) and n (approximate measure for $1/\Lambda$; $\Lambda = \text{logarithmic decrement}$) as functions of the KCl (or in general: of the salt) concentration, has been investigated.

- 2. The effects, in consequence of the lowering of the pH, can be explained by partial hydrolysis of the oleate, the undissociated oleic acid which is formed exerting a KCl-sparing action on the elastic oleate system.
- 3. Increase of the temperature has only a slight influence on the KCl concentration corresponding to the maximum of the n curve, decreases G at KCl concentrations lower than the above-mentioned one and increases G at KCl concentrations higher than the above-mentioned one. At all KCl concentrations n decreases considerably at increase of temperature.
- 4. Other potassium salts (citrate, oxalate, acetate, carbonate, thiocyanate) give analogous $G-C_{\rm salt}$ and $n-C_{\rm salt}$ curves, which show practically the same values of G and of n (n is only lower with acetate and thiocyanate, which is attributed to a secondary effect) at the maximum of the n curve. The sequence of the curves in $G-C_{\rm salt}$ and $n-C_{\rm salt}$ diagrams is in the main that of the lyotropic series of the anions.
- 5. By substituting K (in KCl) partially or wholly by Na, the salt concentration corresponding to the minimum damping is displaced towards lower values, the value of G at the maximum of the n curve is sensibly decreased and the value of n at this maximum is strongly decreased. In mixtures KCl and NaCl behave additively.
- 6. Also with RbCl and CsCl elastic systems may be obtained, not, however, with LiCl. The minimum concentrations needed, as well as the coacervation limits (not realisable with Na) increase in the order

$$Na \langle K \langle Rb \langle Cs \rangle$$

7. Starting from chemically pure oleic acid, and using KOH to dissolve it, we obtained with KCl, typical elastic systems. This shows that the elastic systems obtained from commercial Na oleate preparations are not due to the presence of an equivalent amount of Na ions nor of other, contaminating fatty acids, e.g. palmitate.

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