

THE ELECTRICAL CONDUCTIVITY OF AQUEOUS SOLUTIONS
OF SODIUM MONOFLUOROPHOSPHATE AND
POTASSIUM HEXAFLUOROPHOSPHATE

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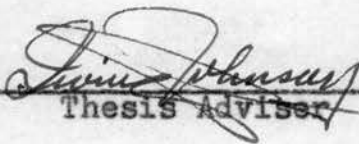
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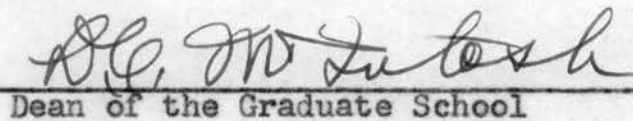
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POTASSIUM HEXAFLUOROPHOSPHATE

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TABLE OF CONTENTS

	Page
I. OBJECTIVE OF RESEARCH	1
II. INTRODUCTION.	2
III. EXPERIMENTAL METHODS.	8
IV. EXPERIMENTAL DATA	11
V. ANALYSIS OF DATA.	14
Potassium Hexafluorophosphate.	14
Sodium Monofluorophosphate	14
VI. CONCLUSIONS	16
VII. BIBLIOGRAPHY.	18
VIII. APPENDIX.	20
Colorimetric Determination of Fluoride	20
Conductometric Titrations.	20

LIST OF TABLES

Table	Page
1. CONDUCTANCE OF SODIUM MONOFLUOROPHOSPHATE	22
2. CONDUCTANCE OF POTASSIUM HEXAFLUOROPHOSPHATE. . . .	23

LIST OF FIGURES

Figure	
1. General Types of Conductance Curves for Strong and Weak Electrolytes	24
2. The Conductance of Potassium Hexafluoro- phosphate	25
3. Determination of the Limiting Equivalent Conductance of Potassium Hexafluorophosphate by the Method of Shedlovsky	26
4. The Conductance of Sodium Monofluorophosphate. .	27
5. Determination of the Limiting Equivalent Conductance of Sodium Monofluorophosphate by the Method of Shedlovsky	28

OBJECTIVE OF RESEARCH

It was the objective of this research to make conductance measurements of aqueous solutions of sodium monofluorophosphate and potassium hexafluorophosphate at 25° C. From these measurements the limiting equivalent conductances of the two salts were to be determined. Deviations of the equivalent conductance with increasing concentration from that predicted by the Onsager equation were to be used to study the behavior of sodium monofluorophosphate and potassium hexafluorophosphate in aqueous solutions.

INTRODUCTION

From measurements of the equivalent conductance of solutions of electrolytes it is possible to characterize the behavior of these electrolytes in solution.

The manner in which conductance data deviate from the Onsager equation classifies the electrolyte as being strong or weak. Further, by using these data, the dissociation constant of the electrolyte can be calculated.

Onsager has shown that for dilute solutions of electrolytes, the equivalent conductance of the solution is given by the equation

$$\Lambda = \Lambda_0 - (A + B\Lambda_0) \sqrt{N}$$

Where Λ_0 is the limiting equivalent conductance, A and B are constants dependent upon temperature and the nature of the solvent, and N is the equivalent concentration of the solution.

The Onsager equation accounts for the observed decrease of equivalent conductance of a solution of an electrolyte with increased concentration by means of two "effects" both of which result in a lowering of ionic mobilities (11).

The constants A and $B\Lambda_0$ of the Onsager equation provide the corrections for these two effects. A accounts for the

"electrophoretic effect" and $B\lambda_0$ for the "time of relaxation effect."

The electrophoretic effect arises from the fact that there is a preponderance of oppositely charged ions surrounding an ion in solution. When a potential is applied to the solution, the surrounding atmosphere of ions moves in a direction opposite to that of the ion being considered. Interionic attraction and friction between associated solvent molecules then impede the motion of the ion. The electrophoretic effect is dependent upon temperature, viscosity, and dielectric constant of the solution.

Around an ion in solution there is a symmetrical space charge. As the ion is moved, the space charge will build up in front of the ion and decay behind it. Since these processes are not instantaneous, there will be an asymmetry of the space charge developed. The asymmetrical space charge exerts an electrostatic attraction in a direction opposite to that of the motion of the ion. This is the time of relaxation effect, which is independent of viscosity since it is purely electrostatic. It is dependent upon dielectric constant, temperature, limiting conductance of the ion, and the charge on the ion.

Rearranging the Onsager equation to the form

$$\lambda'_0 = \frac{\lambda_f \sqrt{AVN}}{1 - BVN}$$

provides a means for evaluating the limiting equivalent conductance of an electrolyte where Λ'_0 is written in place of Λ_0 (2).

It is found that Λ'_0 , which according to theory should be constant, increases with increasing concentration. A graph of Λ'_0 versus N has been found to be linear for many 1-1 and 1-2 electrolytes. This offers a ready method for computing the limiting conductance, Λ_0 , of a strong electrolyte by extrapolation to infinite dilution.

Since the Onsager equation is valid only in the limit of infinitely dilute solutions, deviations from it are noted as the concentration of a solution is increased (11). For a strong electrolyte, the experimental points follow the Onsager equation in the dilute region. As concentration increases, the curve rises above the theoretical slope giving an upward concavity. Solutions of weak electrolytes also follow the theoretical slope in dilute regions, but as concentration increases, the experimental curve falls below the theoretical slope. However, the curve still retains the upward concavity. Figure 1 shows the general types of curves found for strong and weak electrolytes.

A method of estimating the dissociation constant, K , and the limiting conductance, Λ_0 , through the equation

$$\Lambda S_{(z)} = \Lambda_0 - (C\alpha^2 y_{\pm}^2) \Lambda_0 / K$$

appears to be the most satisfactory method when only a single series of conductance measurements is available (2).

A plot of $S(z)$ versus $(c\alpha y_{\pm}^{f/2})$ gives for the negative slope the value Λ_0/K and the intercept the value for the limiting equivalent conductance, Λ_0 .

In the above expression the degree of dissociation, α , is defined by

$$\alpha = \frac{\Lambda}{\Lambda_0} S(z)$$

where $S(z) = 1 + z + \frac{z^2}{2} + \frac{z^3}{8} \dots$

and $z = S_{(\Lambda)} \sqrt{\Lambda c} (\Lambda_0)^{-3/2}$

The values for $S_{(\Lambda)}$ are tabulated by Harned and Owen (1).

The mean activity coefficient, y_{\pm}^f , is defined by the expression

$$\log y_{\pm}^f = -S(f)\sqrt{\alpha c}$$

$S(f)$ also being tabulated by Harned and Owen (3).

By using this equation one can estimate the dissociation constant and compare the values of the limiting conductance with those obtained by the previously described method of Shedlovsky.

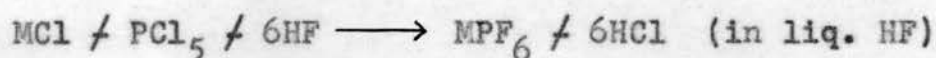
Hill and Audrieth have described a method of preparing sodium monofluorophosphate by fusing a mixture of sodium trimetaphosphate and sodium fluoride.



The fluorophosphates are similar to the corresponding sulfates. In some cases this similarity is so close that the fluorophosphates are isomorphous with the sulfates. Mixed salts of the form $\text{MSO}_4 \cdot (\text{NH}_4)_2\text{PO}_3\text{F} \cdot 6\text{H}_2\text{O}$, where $\text{M} = \text{Ni}$, Cu , Co , Zn , Mn , and Mg have been prepared (12).

The alkali monofluorophosphates are readily soluble in water. The salts are stable in neutral or weakly alkaline solutions. The monofluorophosphates hydrolyze in an acid medium or when heated in an alkaline solution to form the fluoride and orthophosphate (13).

Salts of hexafluorophosphoric acid can be prepared directly by the reaction



Commercially, the salts are prepared by neutralizing the hexafluorophosphoric acid and removing the salt by fractional crystallization (13).

Hexafluorophosphates are similar in solubility behavior to the perchlorates. The salts do not, however, have the same crystal form (9). The hexafluorophosphate ion is symmetrical with the fluorine atoms located octahedrally around the phosphorus atom (8).

Alkali hexafluorophosphates form neutral aqueous solutions and are very stable in neutral and slightly alkaline

solutions. Hydrolysis occurs upon heating with a mineral acid. If the dry alkali salt is heated to a high temperature, it decomposes by the equation $KPF_6 \rightleftharpoons KF + PF_5$ (13).

EXPERIMENTAL METHODS

The conductance measurements of the solutions were made with a conductivity bridge assembly described by Luder (10). The 1000-cycle signal was provided by a Hewlett-Packard model 200A audio oscillator. The signal was amplified with an amplifier constructed after the circuit described in the article by Luder.

The balancing resistance consisted of two decade boxes: a General Radio type 602-G consisting of three decades having steps of 1, 10, and 100 ohms, and another box constructed from General Radio decade resistances 578-E, 510-E and 510-F. The four decade resistances consisted of steps of 0.1, 1000, and two 10,000 ohm assemblies.

The bridge components were shielded wherever possible. Shielded cable was used, and the elements of the balancing circuit were shielded from each other. Additional grounding for the operator was provided since it was found that a sharper null was obtained for the signal when the operator was at ground potential. The tuning knob for the balancing condenser was mounted on a six-inch polystyrene rod. This was done to prevent the proximity of the operator's hand from changing the capacitance of the balancing condenser during the balancing operation.

Two Washburn-type conductivity cells were used. Central Scientific Company type 70015B was used for measurement of the solutions, and type 70015A was used to determine the specific conductance of the conductivity water.

Conductivity water for preparing the solutions was obtained by distilling ordinary distilled water from an alkaline permanganate medium in an all-pyrex system. The water was distilled from a five-liter round-bottom flask equipped with a side-arm. A spray trap was provided between the flask and condenser. The neck of the distilling flask was provided with a nichrome heating element. After aging the system for several weeks by distilling water through the system, conductivity water having a specific conductance of 1 to 1.5×10^{-6} reciprocal ohms was obtained.

The salts used for the investigation were obtained from Ozark-Mahoning Company, Tulsa, Oklahoma. No attempt was made to purify the compounds since they were specially prepared in a high degree of purity.

The sample of potassium hexafluorophosphate was analyzed by precipitation with nitron after the method of Lange and Müller (9). The analysis of sodium monofluorophosphate, which is a difficult procedure, was performed by Mr. W. B. Estill of Ozark-Mahoning Company. The sample was analyzed for fluoride present as the monofluorophosphate ion and for fluoride present as sodium fluoride.

Conductance measurements were made on five series of solutions of sodium monofluorophosphate and three series of solutions of potassium hexafluorophosphate. Each series consisted of four or five solutions of different concentration. The first solution of a series was prepared by adding a known weight of conductivity water to a known weight of salt. Each succeeding solution of the series was prepared by diluting a weighed quantity of the preceding solution with a weighed quantity of conductivity water.

To determine the equivalent concentration of the solutions it was necessary to measure the density of the solutions at the temperature at which the measurements were to be made. A Nicol-type pycnometer was used for this purpose, and the densities were measured at 25° C.

All measurements were made at $25^{\circ} \pm 0.02^{\circ}$ C. Kerosene was used in the constant-temperature bath.

EXPERIMENTAL DATA

The experimental conductance data for sodium monofluorophosphate and potassium hexafluorophosphate are listed in tables 1 and 2.

The values for the equivalent concentration of the solutions were calculated from an expression determined from the density measurements of the solutions and the percentage composition of the solutions. For sodium monofluorophosphate (m.w. 143.97) the equivalent concentration is expressed by

$$\text{Normality} = 0.1389 (\% \text{ weight of } \text{Na}_2\text{PO}_3\text{F})$$

and for potassium hexafluorophosphate (m.w. 184.08)

$$\text{Normality} = 0.05440 (\% \text{ weight of } \text{KPF}_6).$$

The observed specific conductances, κ , of the solutions and solvents were calculated from the expression

$$\kappa = K/R$$

where K is the cell constant and R is the resistance of the solution measured in the cell. Cell number 1, used for measuring the resistance of the solutions, had a cell constant $K_1 = 0.7554 \text{ cm}^{-1}$. Cell number 2, used to measure the resistance of the conductivity water, had a cell constant $K_2 = 0.07212 \text{ cm}^{-1}$.

The equivalent conductance of the solutions, Λ , was calculated from the equation

$$\Lambda = \frac{1000\kappa}{N}$$

The value, κ , in this expression is the specific conductance of the solution corrected for the conductance of the solvent. It is the difference between the observed specific conductance and the specific conductance of the solvent.

The expression, Λ'_0 , used to determine the limiting conductance by the method of Shedlovsky is calculated from the equation

$$\Lambda'_0 = \frac{\Lambda \sqrt{N}}{1 - B/N}$$

where $A = 110.6$ and $B = 0.5265$ for sodium monofluorophosphate, and $A = 60.19$ and $B = 0.2289$ for potassium hexafluorophosphate.

Analysis (in triplicate) of the potassium hexafluorophosphate sample by the nitron method gave the following results:

#1. 99.80%
#2. 99.69%
#3. 99.38%

Avg. 99.62 ± 0.16%

The analysis of sodium monofluorophosphate performed by Mr. Estill was

I. Total Fluoride Analysis

- A. 12.83% F⁻
- B. 13.20% F⁻
- C. 13.10% F⁻

Avg. 13.04~~0.14~~%

II. Na₂PO₃F Analysis1. Fluoride as PO₃F⁼.

- A. 12.86%
- B. 12.95%
- C. 12.70%

Avg. 12.84~~0.06~~%

2. Calculated as % Na₂PO₃F.

- A. 97.44%
- B. 98.12%
- C. 96.23%

Avg. 97.26~~0.69~~%

This analysis of sodium monofluorophosphate shows an impurity of sodium fluoride amounting to 0.58%.

ANALYSIS OF DATA

Potassium Hexafluorophosphate:

A plot of Λ versus \sqrt{N} for potassium hexafluorophosphate is shown in Figure 2. The curve rises above the Onsager limiting slope indicating that potassium hexafluorophosphate is a strong electrolyte.

The value $\Lambda_0 = 130.4$ reciprocal ohms for potassium hexafluorophosphate is determined by extrapolating a plot of Λ_0' versus concentration to infinite dilution. This is shown in Figure 3.

Because of the scattering of points in the dilute range, the graphical extrapolation in Figure 3 results in a deviation of ± 0.5 reciprocal ohm for the value of the limiting equivalent conductance.

Sodium Monofluorophosphate:

Figure 4 is a plot of Λ versus \sqrt{N} for sodium monofluorophosphate. The deviation of the experimental curve from the Onsager equation in the more concentrated regions is characteristic of a strong electrolyte. In the more dilute solutions the experimental points would be expected to fall below the theoretical slope because of the similarity of the monofluorophosphates to the sulfates. Jenkins and Monk have shown that for concentrations below .001

normal the equivalent conductance of sodium sulfate falls below the theoretical value (7).

Analysis of the data for Λ_0 by the Shedlovsky method is shown in Figure 5. This gives a value of 113.5 reciprocal ohms for the limiting conductance of sodium monofluorophosphate.

The extrapolation of Λ_0 versus N is of such a precision that a deviation of ± 0.25 reciprocal ohm for the limiting conductance is obtained. However, the uncertainty of the analysis of the sodium monofluorophosphate sample indicates that the deviation from the value of 113.5 reciprocal ohms for the limiting conductance should be ± 0.5 reciprocal ohm. An impurity of 3% sodium fluoride in the sample would change the limiting equivalent conductance of sodium monofluorophosphate by 0.5%. The value for the limiting conductance of sodium monofluorophosphate, then, will be taken as 113.5 ± 0.5 reciprocal ohms.

An attempt was made to estimate dissociation constants for sodium monofluorophosphate and potassium hexafluorophosphate by the method described in the introduction, but the available data were not of sufficient precision to justify the computation. However, the plot for potassium hexafluorophosphate resulted in a value of 130.4 reciprocal ohms for the limiting conductance which compares well with that obtained using the Shedlovsky method.

CONCLUSIONS

Conductance measurements of aqueous solutions of sodium monofluorophosphate and potassium hexafluorophosphate were made at 25° C. to determine the limiting equivalent conductances and to study the behavior of these salts in aqueous solution.

The limiting equivalent conductance at 25° C. for sodium monofluorophosphate in aqueous solution is 113.5 ± 0.5 reciprocal ohms. For potassium hexafluorophosphate this value of the limiting equivalent conductance is 130.4 ± 0.5 reciprocal ohms.

From the expression for the limiting equivalent conductance of a salt in terms of the limiting ionic conductances

$$\Lambda_0 = \lambda_{+}^{\circ} + \lambda_{-}^{\circ}$$

and the values of the limiting ionic conductances of sodium and potassium, it is possible to calculate the limiting ionic conductances for PO_3F^- and PF_6^- . Harned and Owen list the values (4)

$$\lambda_{\text{Na}^+}^{\circ} = 50.15$$

$$\lambda_{\text{K}^+}^{\circ} = 73.50$$

for 25° C. Hence, the value of the limiting ionic conductance for PO_3F^- at 25° C. is 63.3 ± 0.5 reciprocal ohms, and the value for PF_6^- is 56.9 ± 0.5 reciprocal ohms.

The deviation of the conductance data for potassium hexafluorophosphate from the Onsager equation shows the salt to be highly dissociated in aqueous solution. The conductance data for sodium monofluorophosphate indicates this salt also to be highly dissociated in aqueous solution, its behavior being similar to that of sodium sulfate.

The precision of the conductance measurements is not sufficiently high to permit an estimation of dissociation constants for sodium monofluorophosphate and potassium hexafluorophosphate.

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APPENDIX

Colorimetric Determination of Fluoride:

Hill and Reynolds described a colorimetric analytical method for the determination of monofluorophosphate ion (6). They presented a method based upon the bleaching of titanium peroxide complexes in an acid medium by fluoride ion.

The analysis of sodium monofluorophosphate used in this investigation was attempted by this method. Hill and Reynolds used a Lumetron Model 402EF colorimeter in their investigation. For the analysis in this investigation a Beckman Model DU spectrophotometer was used for the light transmission measurements.

A calibration curve was to be determined for the change in transmission of the solution with the addition of a known quantity of fluoride ion. The author was unable to reproduce a calibration curve using the Beckman DU spectrophotometer. With the reagents specified by Hill and Reynolds for the color reagent it was found that the change of light transmission for the fluoride ion concentration range specified was too small to yield the precision reported by Hill and Reynolds.

Conductometric Titrations:

An attempt was made to determine fluoride by conductometric titration. Samples of sodium fluoride in the presence

of perchloric acid and glass beads were steam-distilled and the distillate titrated conductometrically with a solution of thorium nitrate. The curve obtained from plotting resistance readings versus volume of titrant indicated a break at the equivalence point. However, there was no portion of the curve with sufficient linearity to allow extension to the end-point. Since the method for determining the end-point of a conductometric titration is to extend the linear portions of the curve before and after the equivalence point to the point of intersection, this method had to be abandoned.

TABLE 1
CONDUCTANCE OF SODIUM MONOFLUOROPHOSPHATE

Solution No.	Normality $\times 10^3$	$\chi_{\text{Obs.}} \times 10^3$ Ohms ⁻¹	$\chi_{\text{Solv}} \times 10^6$ Ohms ⁻¹	Equivalent Conductance Ohms ⁻¹	λ Ohms ⁻¹
1	26.13	2.399	1.14	91.77	119.8
2	6.464	0.6558	1.28	101.3	115.0
3	1.915	0.2049	1.28	106.3	113.8
4	0.7901	0.08692	1.28	108.4	113.2
1A	62.62	5.271	1.82	84.14	128.8
2A	18.17	1.720	1.82	94.55	117.8
3A	5.891	0.5997	1.10	101.6	114.7
4A	1.877	0.2015	1.10	106.8	114.2
1B	78.20	6.429	0.904	82.19	132.6
2B	28.72	2.617	1.16	91.08	120.6
3B	7.442	0.7497	1.16	100.6	115.3
4B	1.595	0.1724	1.16	107.3	114.1
1C	106.6	8.469	1.08	79.43	139.5
2C	25.32	2.342	1.11	92.45	120.1
3C	4.339	0.4509	1.11	103.7	114.9
4C	0.7958	0.08775	1.04	109.0	113.76
1D	301.7	20.64	1.09	68.40	181.7
2D	118.9	9.280	1.09	78.04	142.6
3D	63.38	5.327	1.09	84.03	129.0
4D	35.27	3.155	1.09	89.42	122.3
5D	12.37	1.199	1.19	96.84	115.9

TABLE 2
CONDUCTANCE OF POTASSIUM HEXAFLUOROPHOSPHATE

Solution No.	Normality $\times 10^3$	$\chi_{\text{Obs.}} \times 10^3$ Ohms ⁻¹	$\chi_{\text{Solv}} \times 10^6$ Ohms ⁻¹	Equivalent Conductance Ohms ⁻¹	Λ'_b Ohms ⁻¹
1	11.78	1.437	1.35	121.9	131.7
2	3.819	0.4822	1.35	125.9	131.5
3	0.9660	0.1244	1.14	127.6	130.4
4	0.1264	0.01751*	1.14	129.5	130.5
1A	15.33	1.839	1.14	119.9	131.0
2A	3.063	0.3865	1.14	125.6	130.8
1B	6.472	0.8017	1.20	123.7	130.9
2B	2.249	0.2843	1.20	125.9	130.1
3B	1.025	0.1316	1.19	127.2	130.1
4B	0.4164	0.05474	1.19	128.6	130.4

*This solution measured in No. 2 Washburn Cell. $K_2 = 0.07212 \text{ cm.}^{-1}$.

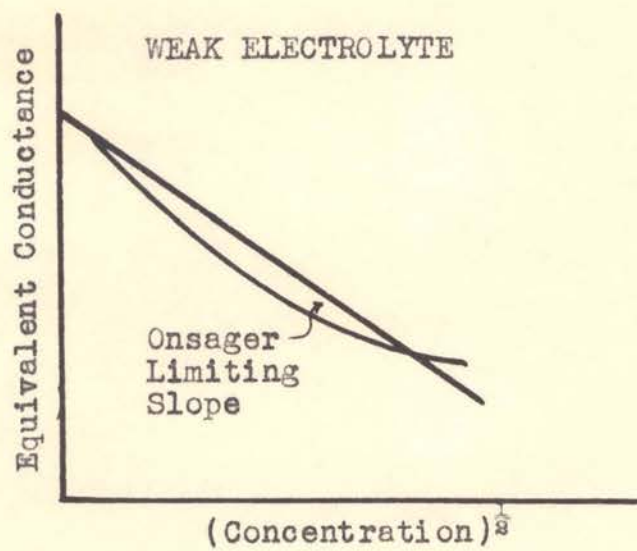
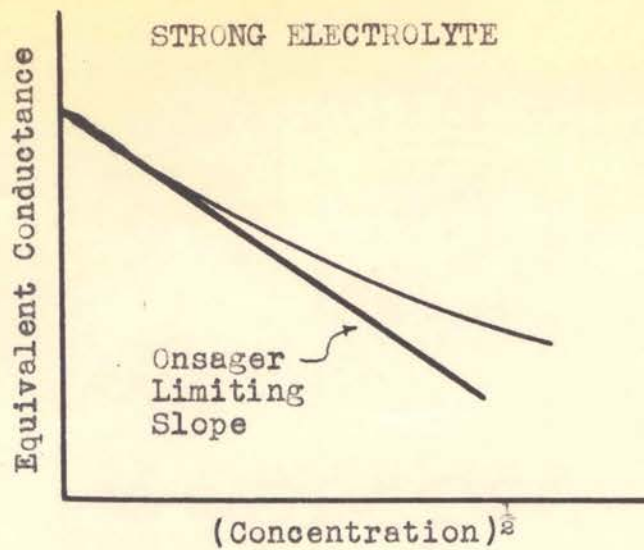


FIGURE 1: General Types of Conductance Curves For Strong and Weak Electrolytes.

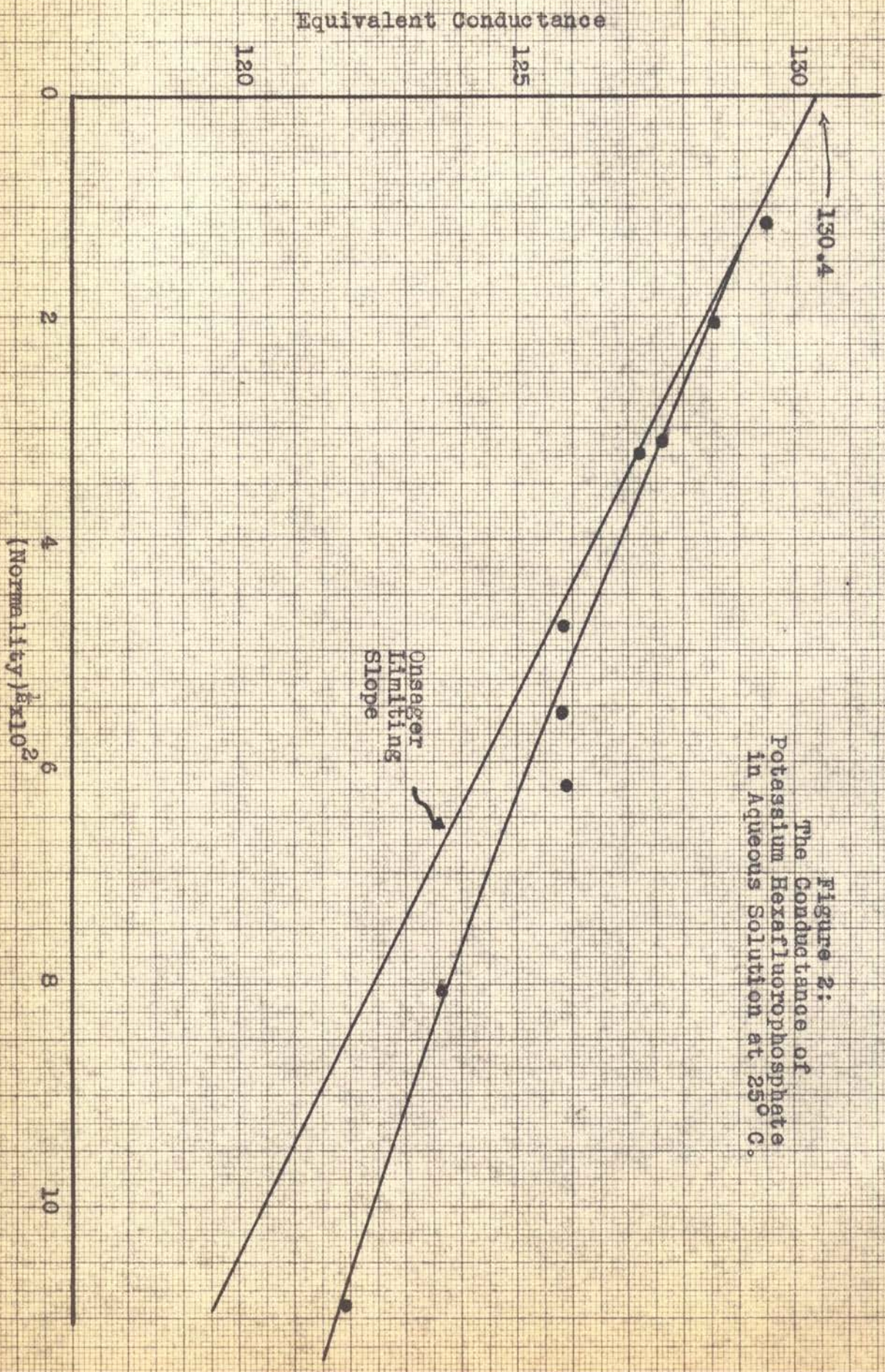


Figure 2:
The Conductance of
Potassium Hexafluorophosphate
in Aqueous Solution at 25° C.

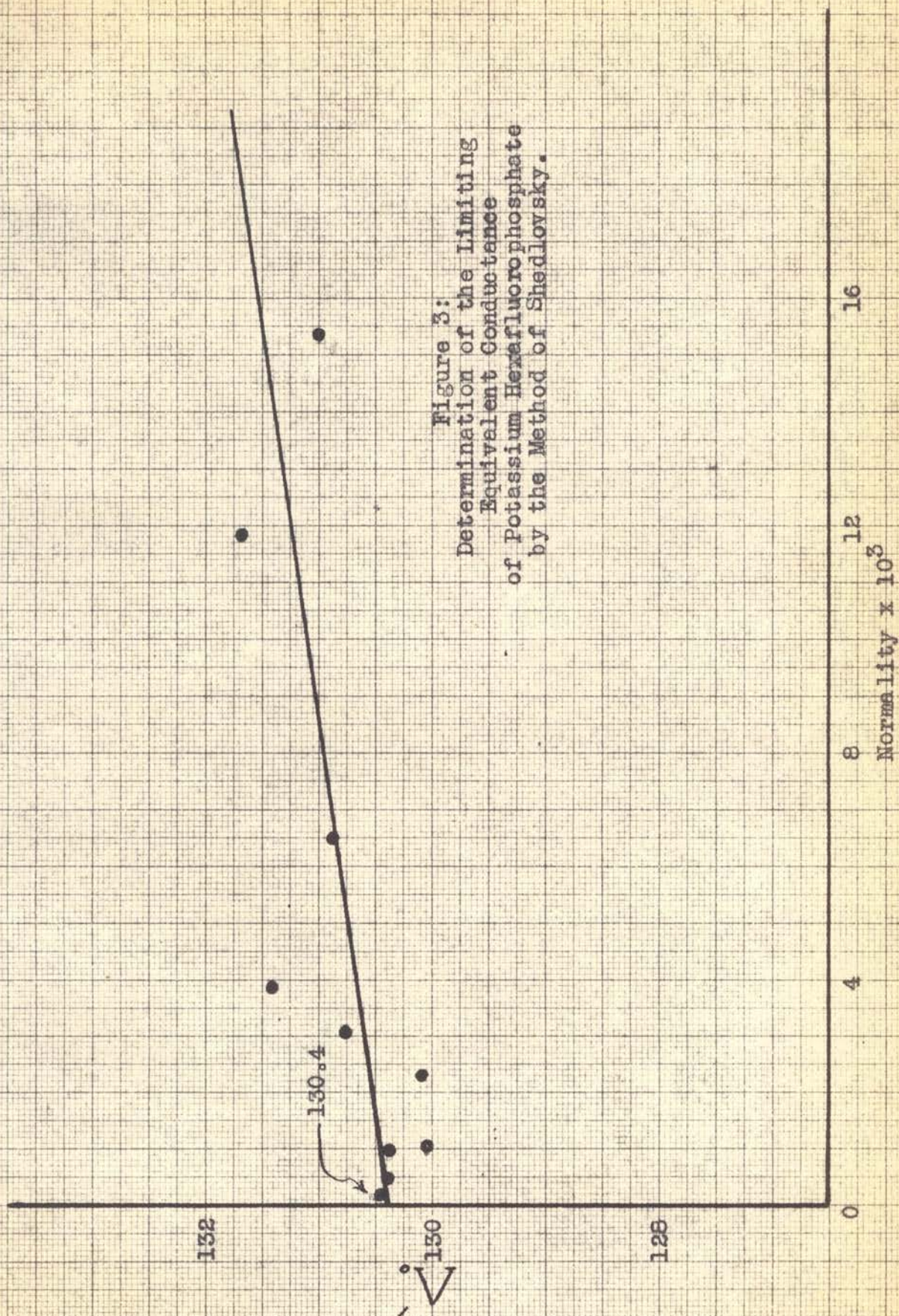
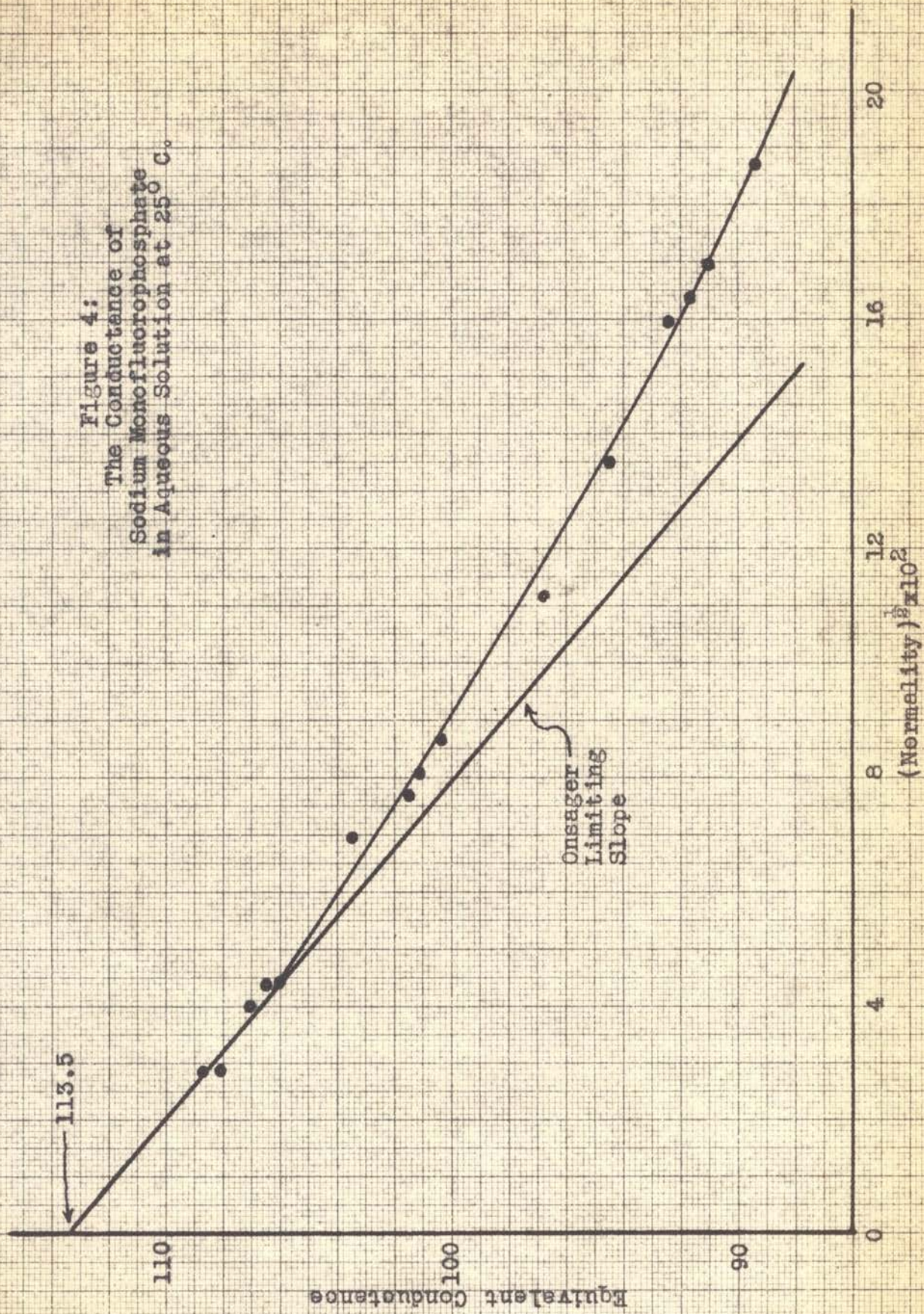


Figure 4:
The Conductance of
Sodium Monofluorophosphate
in Aqueous Solution at 25° C.



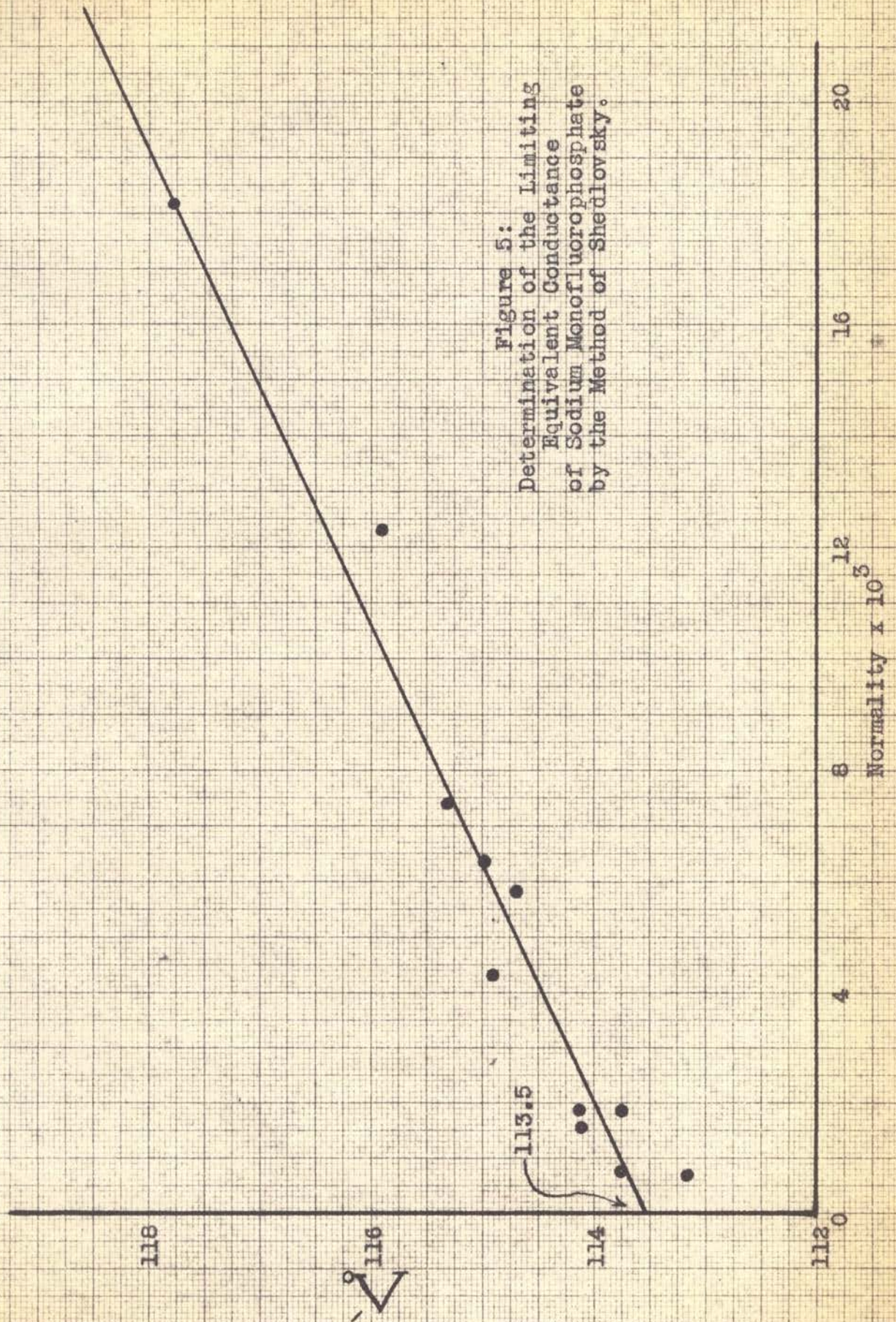


Figure 5:
Determination of the Limiting
Equivalent Conductance
of Sodium Monofluorophosphate
by the Method of Shedlovsky.

VITA

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candidate for the degree of
Master of Science

Thesis: THE ELECTRICAL CONDUCTIVITY OF AQUEOUS SOLUTIONS
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