



An investigation of the effects of acid solutions of vanadium in column chromatography
by Theodore Van Vorous

A THESIS Submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree
of Master of Science in Chemistry

Montana State University

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Abstract:

During an investigation of the possibility of using column chromatographic methods for the analysis of Vanadium minerals it was found that the concentration of acid in the test solution determined to a great extent the distance Vanadium will migrate in certain absorbents. The purpose of this thesis is to show the effect of this phenomenon with various absorbents, solvents, and concentrations of acid.

Since the analysis of Vanadium minerals necessarily involves to a great extent the Geochemistry of that element, a brief outline of the Geochemistry of Vanadium has also been included in the introduction.

AN INVESTIGATION OF THE EFFECTS OF ACID SOLUTIONS
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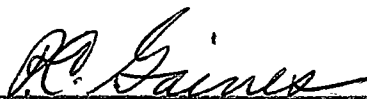
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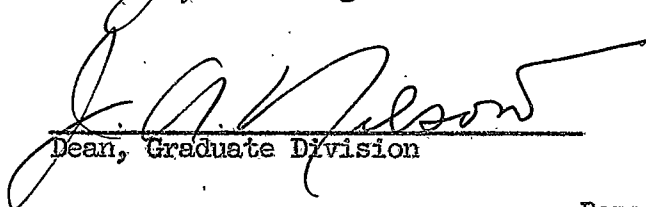
at

Montana State College

Approved:


Head, Major Department


Chairman, Examining Committee


Dean, Graduate Division

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ABSTRACT

During an investigation of the possibility of using column chromatographic methods for the analysis of Vanadium minerals it was found that the concentration of acid in the test solution determined to a great extent the distance Vanadium will migrate in certain absorbents. The purpose of this thesis is to show the effect of this phenomenon with various absorbents, solvents, and concentrations of acid.

Since the analysis of Vanadium minerals necessarily involves to a great extent the Geochemistry of that element, a brief outline of the Geochemistry of Vanadium has also been included in the introduction.

II. INTRODUCTION

Since this study is not only concerned with the effect of acid concentration on the migration of vanadium, but is also somewhat a study of the separation of vanadium from other ions, it is important to know what ions occur with vanadium. For that reason, a brief summary of the geochemical characteristics of vanadium is included in this introduction.

Part A: Geochemistry of Vanadium

The wide distribution of vanadium throughout the earth's crust has been clearly established, not only in ores and coals but in clays, limestones, sandstones and igneous rocks. Vanadium occurs in appreciable amounts in the more basic igneous and metamorphic rocks, up to .08% or more of vanadium trioxide, but seems to be absent or nearly so from the highly siliceous rocks. Some of the igneous and metamorphic rocks can carry up to .13% of vanadium trioxide in ferric aluminous silicates, especially in a biotite separated from a pyroxene gneiss. In general, the search for vanadium should be limited to rocks containing less than 60% silica.

In the upper lithosphere vanadium is oxyphile, a behavior dictated by the fact that it has three stable oxidation states, i.e., tri-, quadri-, quinquivalent vanadium. Because of its position in the periodic table, vanadium resembles phosphorous and titanium in its manner of occurrence but differs from them in several respects. It does not occur in the early pentlandite

assemblages or the late magmatic sulfides. On the other hand, vanadium does become enriched in titaniferous iron ores due to the replacement of titanium. It also concentrates in basic rocks, but has no connection with the phosphorous content in that respect.

In igneous rocks vanadium usually does not form independent minerals, but instead is concealed in the structure of other minerals. An exception is Ardennite, a complex manganese-aluminum-arsenic-vanadium silicate in which the vanadium occurs as VO_4 .

Because of the similar ionic radii of vanadium in its three oxidation states and ferric iron, aluminum and quadrivalent titanium, vanadium has a great many replacement possibilities. Quinivalent vanadium also easily replaces phosphorous in Apatite.

Feldspars are nearly devoid of vanadium, but the pyroxenes, amphibole, and the micas are normally carriers of vanadium. In this respect one mica, roscoelite, can be considered to be a vanadium mineral because much of the aluminum in its structure has been replaced by vanadium.

Vanadium minerals of secondary origin are more common because they become more highly concentrated. Because of the higher redox potentials they occur as vanadates. The minerals might be considered to be mineralogical curiosities because the conditions that form them are seldom met. The vanadium in these

minerals normally comes from enriched ground waters and thermal waters. The chief metals found with these vanadates are calcium, manganese, (ferric) iron, uranium, lead, copper, zinc and bismuth. They may form simple vanadates such as pucherite (bismuth vanadate) and steigerite (aluminum vanadate) or complex vanadates such as descloizite (a lead-copper-zinc vanadate) and carnotite (potassium uranyl vanadate).

Vanadium is found in the biosphere in a number of plants and small sea animals as well as in crude oil. In Persian crude oil, vanadium can occur as high as 2.82%. In any event, it constitutes a poison for cracking catalysts.

In the Geochemical cycle of vanadium several important things are to be noted. Solution and migration takes place at relatively high redox potentials and in the quinquivalent state it has a tendency to form anion complexes. When vanadium is mobilized it moves with the ground water until heavy metals such as copper, zinc, or lead are met and then vanadates will form. This is especially true in the presence of dolomitic and calcitic limestones which give the water and appropriate pH for precipitation. Vanadium will also precipitate in the presence of sulfide ion, a condition often met in crude oil.

When a study was made of the effects of organic material on the concentration of vanadium in clays and shales it was shown that as the organic material percentage went up so did the vanadium percentage. This is true in general for all

sedimentary material containing organic material.

These generalities I have mentioned are the more important characteristics of the Geochemistry of vanadium. They should serve at least as a partial guide to understanding the mode of occurrence of vanadium.

Part B: Considerations of the Thesis

Up to the present time no publications have been mentioned in Chemical Abstracts concerning the effect of acid concentration on the migration of vanadium ions in column chromatography. In addition, very few references were found concerning the use of column chromatography for the separation and determination of vanadium. Of the references available, the most comprehensive was written by T. Ashizawa (B-11). This study covered various solvents, absorbents, and color developing agents that were tried in order to develop inorganic analysis methods for water samples, minerals, and various rocks found in Japan.

Since there was a general lack of information concerning appropriate solvents, absorbents and color developing agents for inorganic column chromatography, a brief study had to be made of these subjects as they apply to vanadium analysis. After usable combinations of the above-named factors were found, experimentation could proceed. During this experimentation, it was observed that in a hydrated calcium silicate (Silene E.F.) absorbent as well as in other similar absorbents there was a

definite tendency for the migration distance of vanadium to be affected by the acid concentration of the test solution. A series of tests was set up to further investigate this fact. Columns were set up in which several possible factors such as vanadium, acid, and other ion concentrations were varied, one at a time. The effect of varying these factors is the basis for this Thesis.

III. APPARATUS

A. Apparatus Setup for Vacuum System

The original equipment used by the author is illustrated in Figure 1. It consisted of a column made of various diameters of soft glass tubing pulled out at the bottom to constrict it and enlarged at the top to allow easy application of test solutions. The diameter of glass tubing was varied in size from 6mm to 1cm (O.D.) to find a suitable size to use. The bottom of the tube was packed with a small wad of glass wool to prevent the absorbent from being pulled out by the vacuum. This column was placed in a cork of suitable size to fit a 500ml suction flask of the conventional type, and connected to a water suction pump. A test tube of suitable length was placed below the tip of the column inside the flask to collect the solvent coming through the column.

It was found that this type of setup had the disadvantage of packing the absorbent too tightly and also drying out at the bottom.

B. Original Setup for Gravity Feed System

To overcome the disadvantages of the suction system, a gravity fed column was tried. The apparatus consisted of a separatory funnel suspended about four feet above the column and connected to it with rubber tubing. The columns were made of 8mm (O.D.) glass tubing about 30cm long, and pulled out at the bottom. The columns were filled with the absorbent in the

form of a slurry to about one half of their length, and the solution to be tested was applied at the top. The columns were then connected to the rubber tubing and a pinch clamp opened to allow the solvent to flow.

Although this system worked well, it had the disadvantage of allowing only one sample to be run at a time unless a number of such setups were used.

C. Apparatus Setup for a Multiple Column System

The multiple column setup (Fig. 3) was essentially the same as the original gravity system except that calcium chloride drying tubes were used in place of a separatory tube. The multi-outlet tube was made in such a way that a source of air or nitrogen pressure could be applied, thus obtaining gravity and gas pressure at the same time on the columns. It was found later that the system flowed sufficiently fast with no gas pressure applied, so this part of the apparatus was removed.

This system allowed twelve columns to be run at once with two six multi-outlet tubes. This greatly increased the speed of the method and also its versatility. It too had a bad point in that the rubber tubing was attacked by the solvents being used. This was remedied by using glass tubing with surgical rubber joints as shown in Fig. 4.

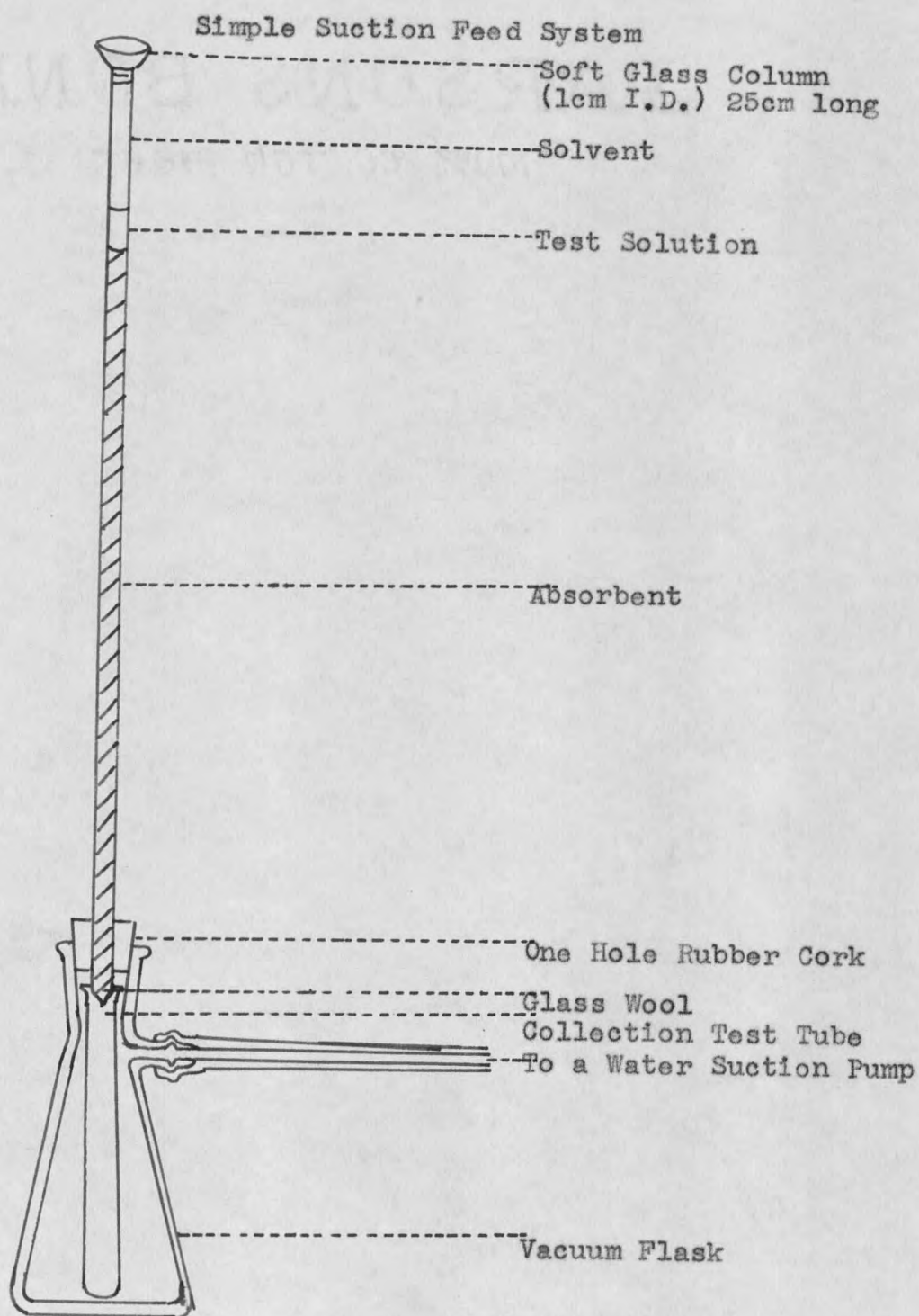


Figure 1

Simple Gravity Feed System

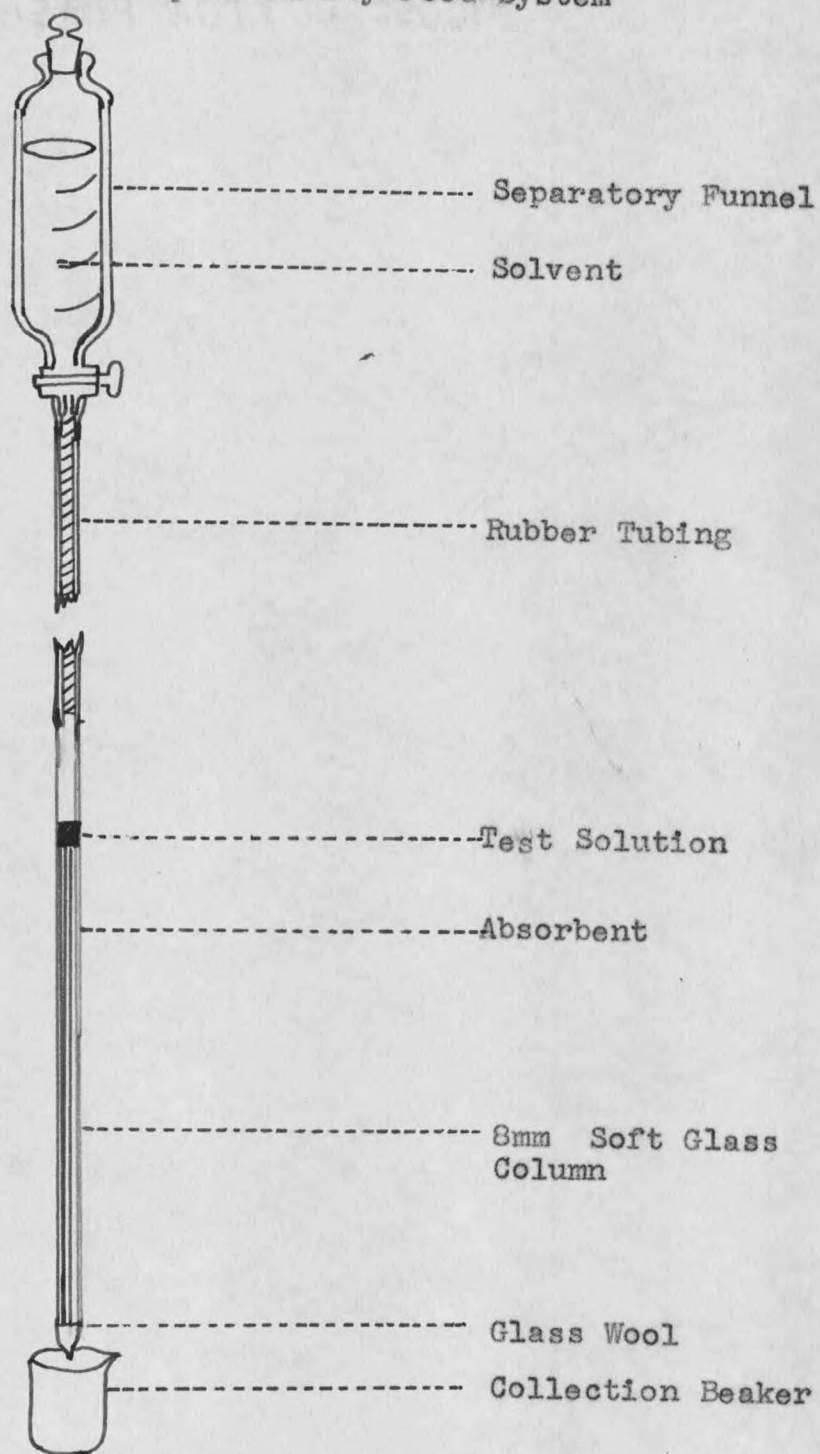


Figure 2

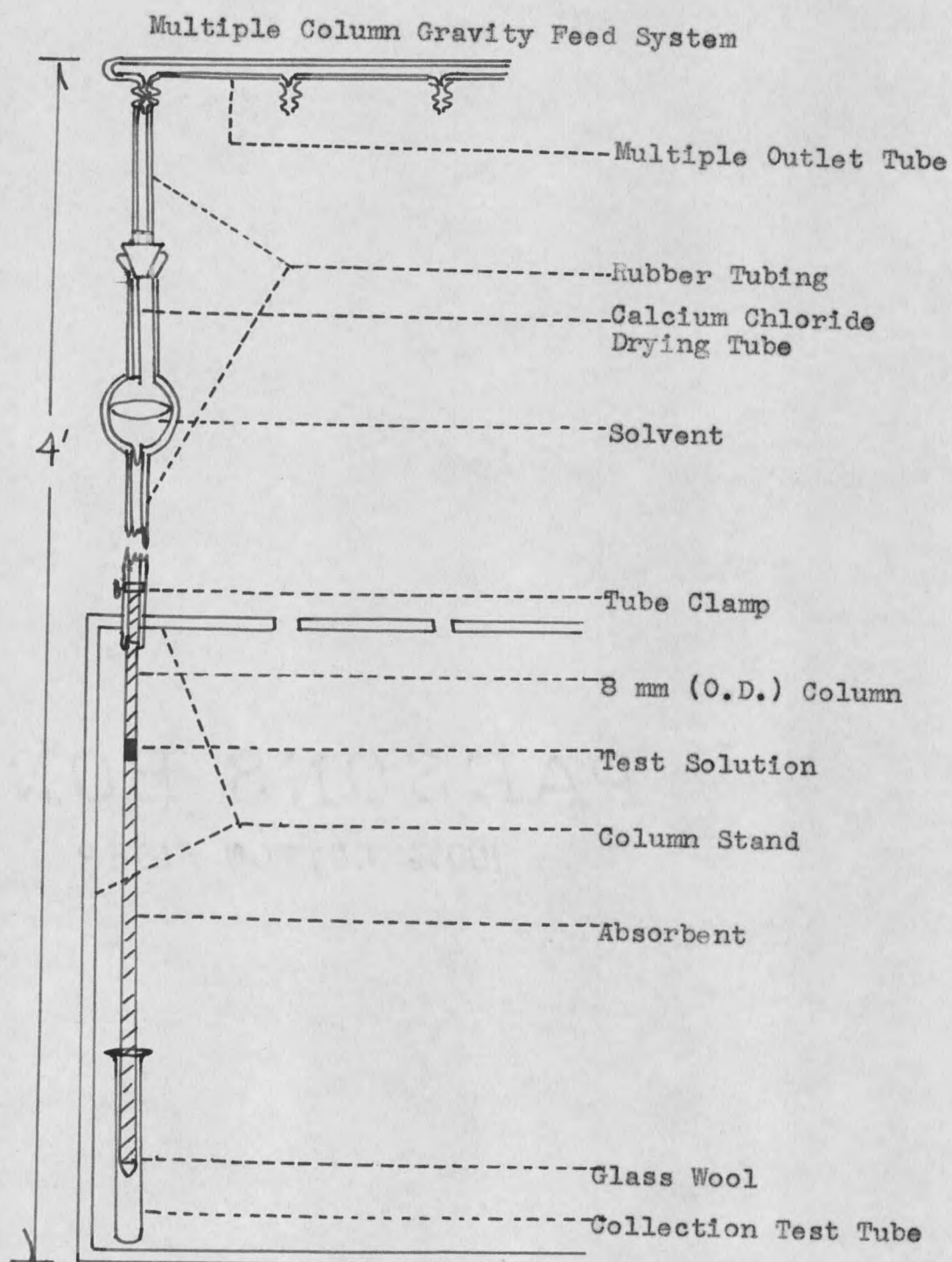


Figure 3

Modified Column Assembly

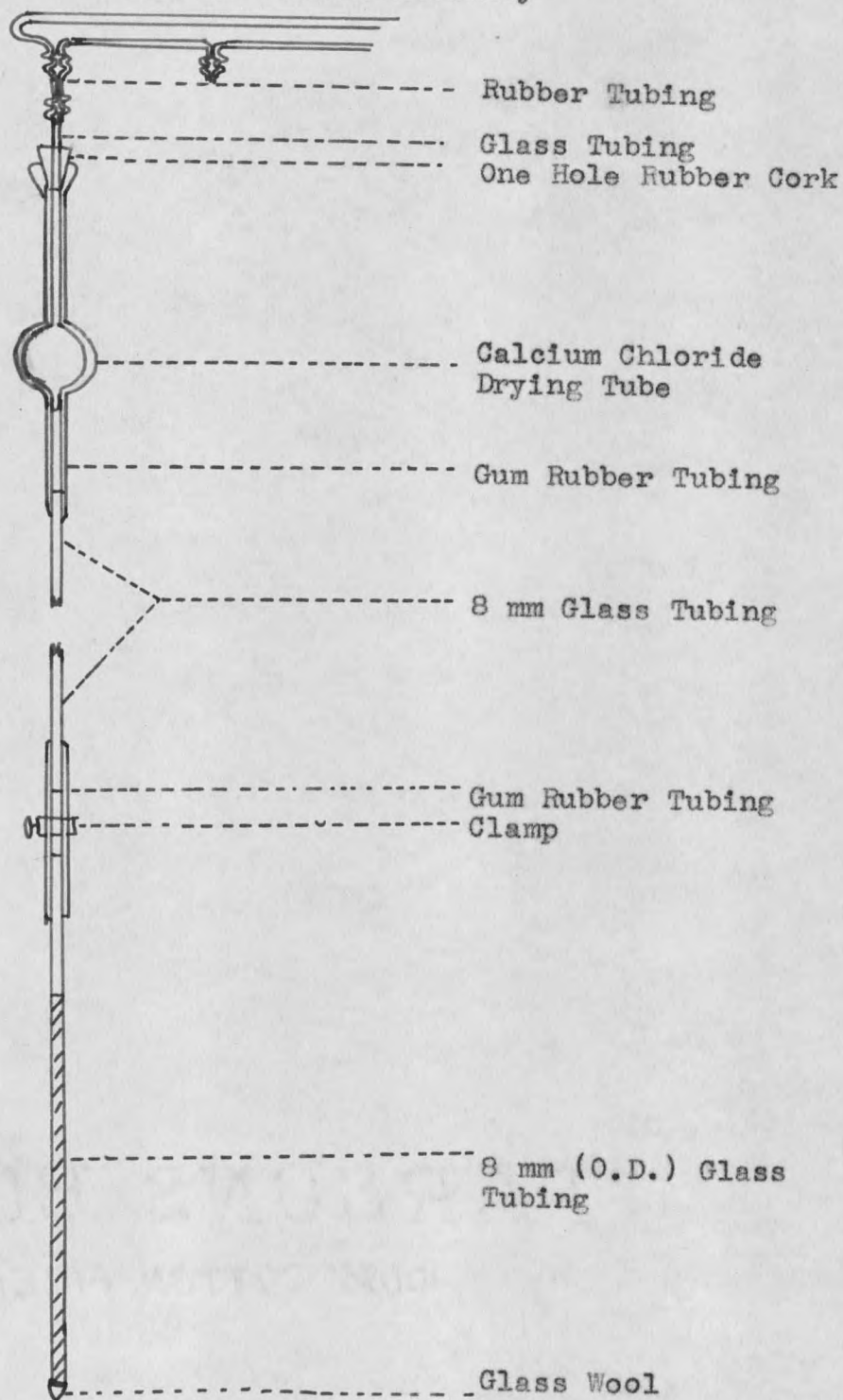


Figure 4

IV. EXPERIMENTAL PROCEDURES

The following conditions and procedures were followed for all of the experimentation except where deviations are specifically noted. These conditions are not listed in any particular order.

1. Except in the early experimentation, the absorbent being used was washed and then poured into the columns as a thick slurry. This was done with a partial vacuum applied to the bottom of the column to insure an even flow and no bubble formation.

2. The absorbents used were as follows:

- a. Aluminum Oxide--Baker Reagent Grade (ignited) thru #100 mesh.
- b. Hydrated Calcium Silicate (Silene E.F.)--Columbia Southern Corp.--thru #100 mesh.
- c. Tricalcium Phosphate--Malinkrodt Reagent Grade--ground to pass #100 mesh.
- d. Bone Ash--unknown analysis for impurities--unknown producer.
- e. Diatomaceous Earth--Tech. Grade
- f. Fullers Earth--
- g. Magnesium Carbonate--Baker C.P.
- h. Calcium Carbonate--Baker C.P.

3. The eluting solvents used were as follows:

- a. Methyl Alcohol - Water (1:1)

- b. Ethyl Alcohol - Water (1:1)
 - c. n-Propyl Alcohol - Water (1:1)
 - d. iso-Propyl - Alcohol (1:1)
 - e. n-Butyl Alcohol - Water (1:1)
 - f. Carbon Tetrachloride
 - g. Ethyl Acetate
 - h. Benzene
 - i. n-Butyl Alcohol - 1N Hydrochloric Acid (1:1)
 - j. n-Butyl Alcohol - 1N Nitric Acid - Oxine (1%)
4. The color-developing agents used were as follows:
- a. Hydrogen Sulfide passed through the column
 - b. 8-Hydroxyquinoline at the correct pH
 - c. Ammonium Polysulfide
 - d. Ultra-violet light (uranium)
 - e. Sodium Thiocyanate .1N soln.
 - f. Ammonium Hydroxide 2N
 - g. Phosphotungstic Acid

5. The solutions containing Vanadium were made up from the following reagents:

- a. Sodium ortho-vanadate--
- b. Sodium meta-vanadate--
- c. Ammonium meta-vanadate--
- d. Vanadium Trioxide

6. Since the absorbents containing carbonates and active forms of silica evolved gas in acidic solutions, the solutions

were kept below a 2N acid condition during the experimentation.

7. In general, the solutions containing Vanadium were made up to contain 1020 micro grams v/ml and diluted to the desired concentration.

8. Since Vanadium will develop a distinct color in absorbents containing active silicates, no color developing solutions were needed with these absorbents.

9. It was found that when absorbents were used, the Vanadium solutions could be diluted to a concentration of 5 micro grams v/ml and still give a distinctly visible colored line.

10. Unless otherwise noted, the columns were run for sixteen (16) hours. This time was chosen for convenience and because very little movement occurred after a longer period of time.

11. When the columns were filled, a line was drawn on each column at a predetermined height so that equal amounts of absorbent could be added. As a check on the consistency of packing, the amount of solvent flow from each column was measured. Also, each batch of solvent was checked with 8-Hydroxyquinoline to see that all of the Vanadium had been retained.

12. A check was made of the ions that might interfere with the experiment. Solutions of phosphate, chlorate, sulfate and several other ions were added to the absorbent slurry to

see what effect they had on the migration distance of the Vanadium ions.

13. The acid concentration was varied between .01 N and 2 N in the second section of the experimentation. Below the .01 N acid concentration no measurable movement was noted in a hydrated calcium silicate (Silene E.F.) absorbent.

V. GENERAL CONSIDERATION OF THE DATA

In Part I of the data, consideration is given to finding an appropriate solvent and absorbent for Vanadium. The solvents that were tried are listed in Table I. Since some work has been done by other authors on the problem of finding a suitable absorbent for Vanadium, this problem will be covered in descriptive form.

Part II of the data deals first with the effects of varying the concentration of Vanadium in the solution being tested. The acidity of the solution varied at the same time that the Vanadium concentration varied. Next the acidity alone was varied between zero and 1 N acid conditions. Lastly the acidity was varied with a constant amount of interfering anion added to the absorbent.

VI. DATA

Part I:

In Table I, data concerning the use of a number of solvents is listed. Although this table shows the effects of these solvents on the migration of vanadium, it does not show other important factors we must know in order to pick a suitable solvent. For example, it does not show that n-Butanol and water move so slowly that the bottoms of the columns dry out and crack. Others, such as methanol and water, move so fast in hydrated calcium silicate that they do not give a good separation of vanadium from the other ions that occur with it. Another factor that had to be considered was that solvents that are acidic in nature could not be used easily because they caused excessive channeling in the absorbents that the author employed. It was found that n-Propanol and water was the best all-around solvent that could be found. For that reason, it was used almost exclusively in Part II of the experimentation.

Aluminum Oxide was the first absorbent to be used. It had to be ignited and then re-ground to pass #100 mesh before it was suitable as a column material. It was found that this absorbent did not give very good separations and dried out easily, causing channeling.

Bone ash was a good absorbent for vanadium as well as other ions, excluding uranium. It did, however, have a very slow solvent flow rate which caused the columns to dry

out easily.

The carbonate absorbents were ineffectual because of the gas evolved when the acidic test solutions were applied.

Hydrated calcium silicate and Diatomaceous Earth had the properties of very good absorbents for vanadium. They ~~did~~ not dry out or channel except at high acid concentrations. They gave good separations of vanadium from other ions such as copper, lead, bismuth and cadmium. They did not, however, retain uranium at all. This fact, of course, has the advantage of completely separating vanadium from uranium in carnotite ore, as an example.

Because of the distinct advantages of hydrated calcium silicate and Diatomaceous Earth over the other absorbents tried, they were used exclusively in the experimentation of Part II.

A literature search revealed that the best color developing reagent for vanadium was 8-Hydroxyquinoline (oxine). This reagent was used to develop color for vanadium whenever this was necessary. It was not needed in hydrated calcium silicate or Diatomaceous Earth because vanadium developed a distinct color without the addition of a separate reagent.

The following lists are the key to the numbers used to indicate the different solvents and vanadium containing compounds used in the experimentation.

Solution Number List:

1. Sodium meta-vanadate
2. Ammonium meta-vanadate
3. Sodium ortho-vanadate
4. Vanadium trioxide

Solvent Number List:

1. Methanol--Water (1:1)
2. Methanol--1 N Nitric Acid (1:1)
3. Ethanol--Water (1:1)
4. Ethanol--1 N Nitric Acid (1:1)
5. n-Butanol--1 N Hydrochloric Acid (1:1)
6. n-Butanol--1 N Nitric Acid--1% oxime
7. n-Butanol--Water (1:1)
8. n-Propanol--1 N Nitric Acid (1:1)
9. n-Propanol--Water (1:1)
10. iso-Propanol--1 N Nitric Acid (1:1)
11. iso-Propanol--Water (1:1)
12. Carbon Tetrachloride
13. Ethyl Acetate
14. Benzene
15. Chloroform

Table I

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1	3	Hydrated Calcium Silicate	1	66	1.0	.6	16
2	"	"	"	33	.5	.3	"
1B	"	"	2	66	1.0	10.	"
2B	"	"	"	33	.5	10.	"
1C	"	"	3	66	1.0	.8	"
2C	"	"	"	33	.5	.3	"
1D	"	"	4	66	1.0	9.0	"
2D	"	"	"	33	.5	9.0	"
1E	"	"	5	66	1.0	6.5	"
2E	"	"	"	33	.5	6.5	"
1F	"	"	6	66	1.0	3.0	"
2F	"	"	"	33	.5	3.0	"

Comments: All of the above solvents developed color in the column without the addition of color developing agents. The color was a dark grey in each case except for solvent number 6, which gave a reddish-brown due to the oxime.

Table I (Cont.)

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1G	3	Hydrated Calcium Silicate	7	66	1.0	2.8	16
2G	"	"	"	33	.5	1.7	"
1H	"	"	8	66	1.0	1.0	"
2H	"	"	"	33	.5	1.0	"
1I	"	"	9	66	1.0	2.6	"
2I	"	"	"	33	.5	1.5	"
1J	"	"	10	66	1.0	1.0	"
2J	"	"	"	33	.5	1.0	"
1K	"	"	11	66	1.0	3.0	"
2K	"	"	"	33	.5	1.7	"
1L	"	"	12	66	1.0	1.0	"
2L	"	"	"	33	.5	1.6	"

Comments: All of the above solvents developed color in the columns without the addition of color developing agents. The color in each case was a dark grey line about 1 to 3mm wide.

Table I (Cont.)

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1M	3	Hydrated Calcium Silicate	13	66	1.0	.9	16
2M	"	"	"	33	.5	.4	"
1N	"	"	14	66	1.0	1.1	"
2N	"	"	"	33	.5	.6	"
1P	"	"	15	66	1.0	1.0	"
2P	"	"	"	33	.5	.5	"

Comments: All of the above solvents developed color in the columns without the addition of color developing agents. The color was grey for #13 and #14 and a yellowish-grey for #15.

Part II

Table II

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
58	4	Hydrated Calcium Silicate	9	66	1.0	3.0	16
59	"	"	"	59	.9	2.5	"
60	"	"	"	52	.8	2.1	"
61	"	"	"	46	.7	1.9	"
62	"	"	"	40	.6	1.7	"
63	"	"	"	33	.5	1.3	"
64	"	"	"	26	.4	1.0	"
65	"	"	"	20	.3	.8	"
66	"	"	"	13	.2	.5	"
67	"	"	"	6.6	.1	.2	"
68	"	"	"	3.3	.05	.1	"
69	"	"	"	0			"

Comments: The base line of vanadium migration in this series varied from a dark green at the highest concentration to a readily distinguishable grey line with 3 micrograms/ml.

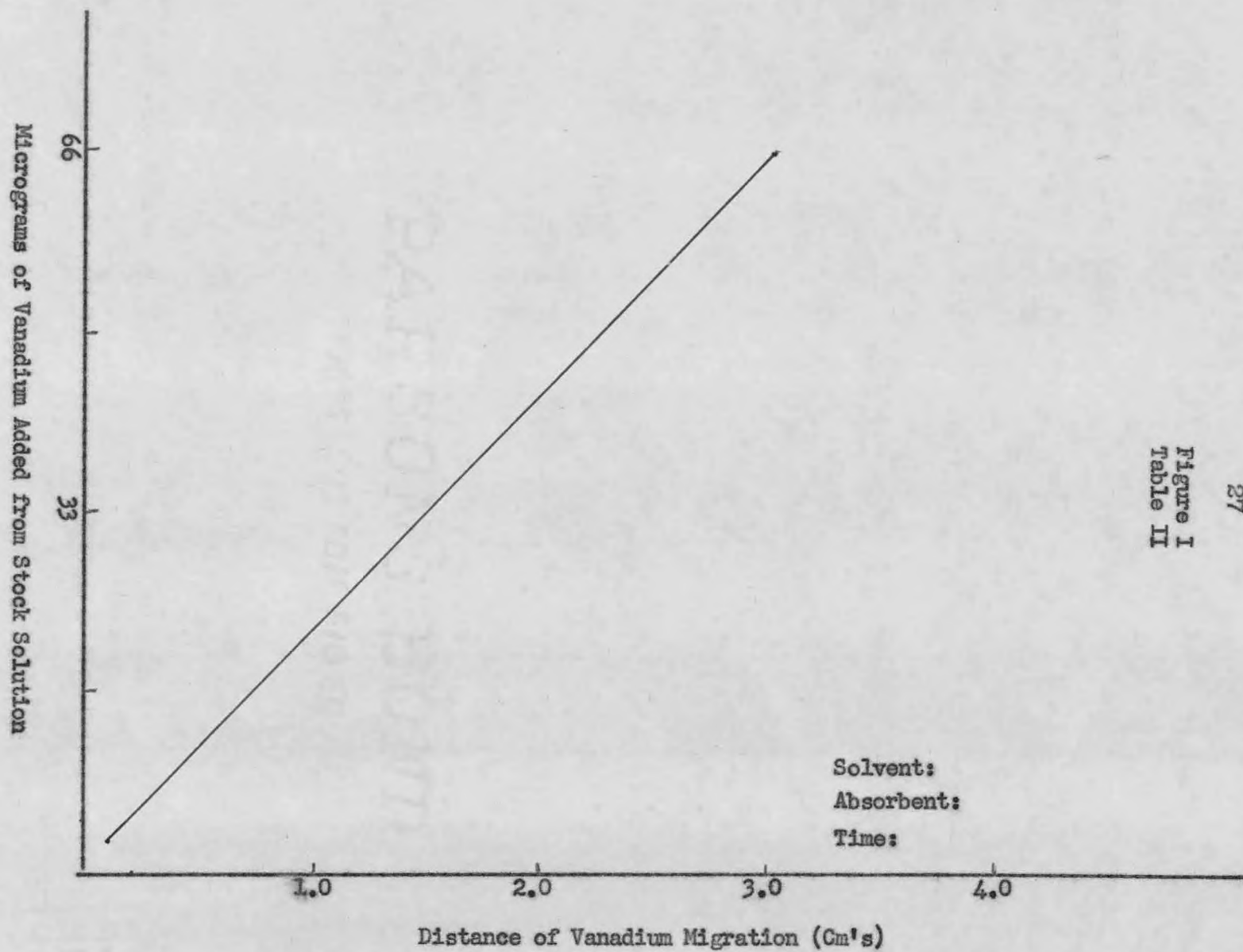
Figure I
Table II

Table III

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
58B	4	Diatomaceous Earth	9	66	1.0	2.8	16
59B	"	"	"	59	.9	2.3	"
60B	"	"	"	52	.8	2.0	"
61B	"	"	"	46	.7	1.8	"
62B	"	"	"	40	.6	1.6	"
63B	"	"	"	33	.5	1.2	"
64B	"	"	"	26	.4	.9	"
65B	"	"	"	20	.3	.7	"
66B	"	"	"	13	.2	.6	"
67B	"	"	"	6.6	.1	.1	"
68B	"	"	"	3.3	.05	"	"
69B	"	"	"	0	"	"	"

Comments: The base line of vanadium migration in this series varied from a dark green at the highest concentration to a yellow at a concentration of 6.6 micrograms/ml.

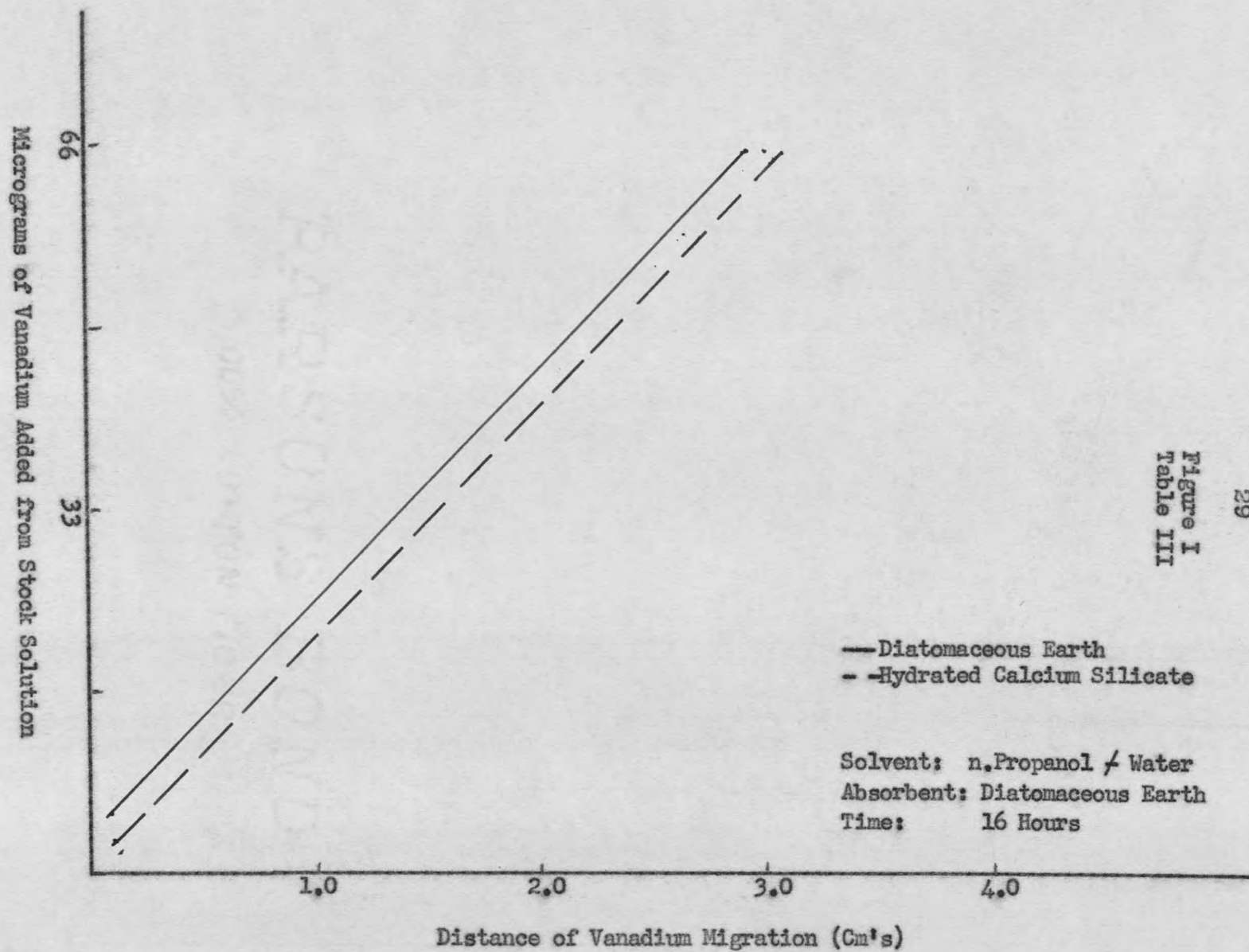
Figure I
Table III

Table IV

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
580	4	Diatomaceous Earth With 2N H_3PO_4	9	66	1.0	2.6	16
590	"	"	"	59	.90	2.1	"
600	"	"	"	52	.80	1.9	"
610	"	"	"	46	.70	1.7	"
620	"	"	"	40	.60	1.4	"
630	"	"	"	33	.50	1.1	"
640	"	"	"	26	.40	.8	"
650	"	"	"	20	.30	.6	"
660	"	"	"	13	.20	.3	"
670	"	"	"	6.6	.10	"	"
680	"	"	"	3.3	.05	"	"
690	"	"	"	0	"	"	"

Comments: The base line of vanadium migration in this series was a yellow-green at the highest concentration down to a hard to distinguish yellow at the lowest concentration.

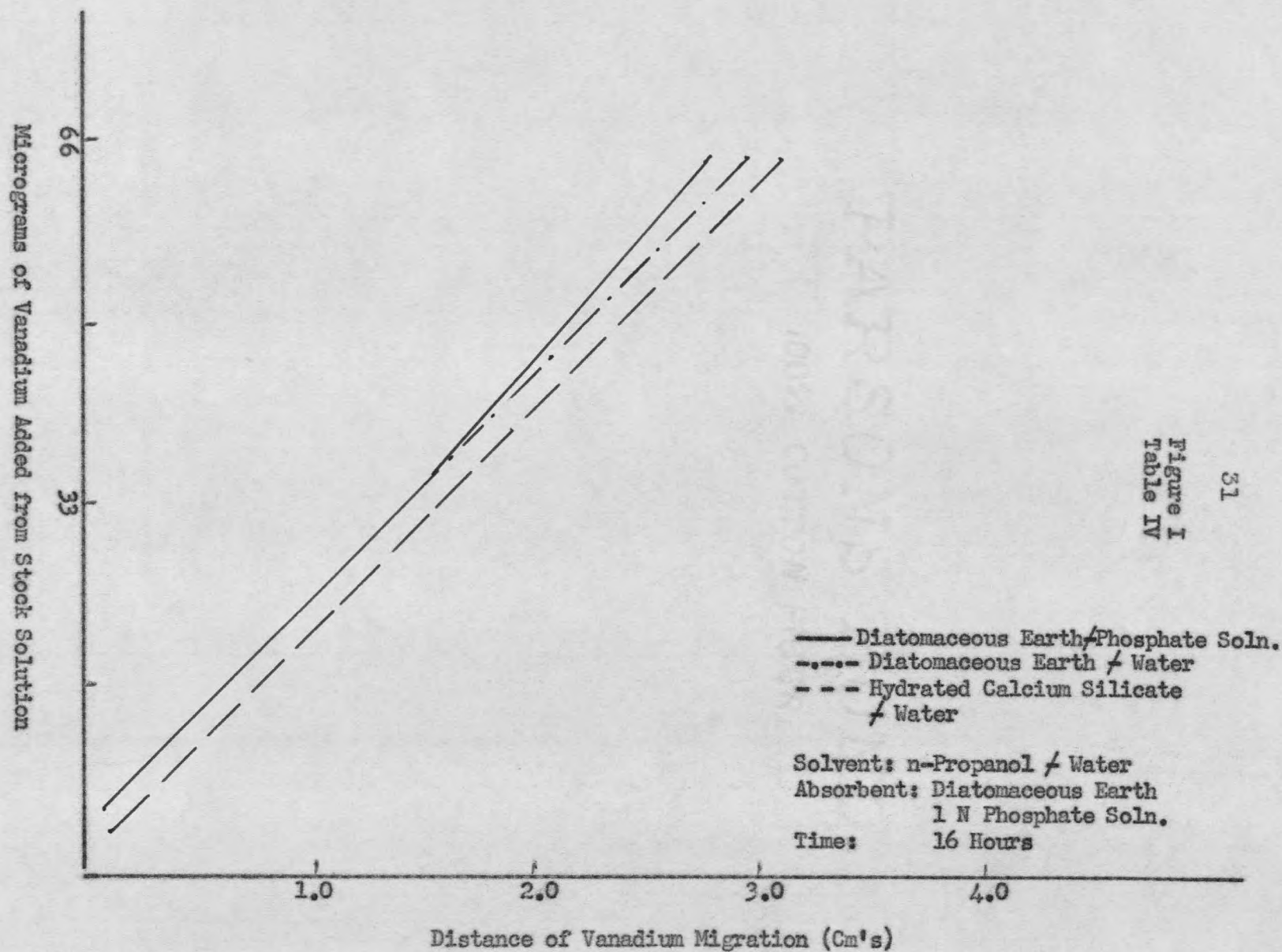
Figure I
Table IV

Table V

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
70	4	Hydrated Calcium Silicate with a constant 100ppm U added	9	66	1.0	3.5	45
71	"	"	"	59	.9	2.9	"
72	"	"	"	52	.8	2.5	"
73	"	"	"	46	.7	2.0	"
74	"	"	"	40	.6	1.6	"
75	"	"	"	33	.5	1.4	"
76	"	"	"	26	.4	1.1	"
77	"	"	"	20	.3	.9	"
78	"	"	"	13	.2	.7	"
79	"	"	"	6.6	.1	.5	"
80	"	"	"	3.3	.05	.3	"
81	"	"	"	-	-	-	"

Comments: The base line of vanadium migration in this series varied from a dark green at the highest concentration to a readily distinguishable grey line at the lowest concentration. The uranium was found in the wash solution because it was not held by this absorbent.

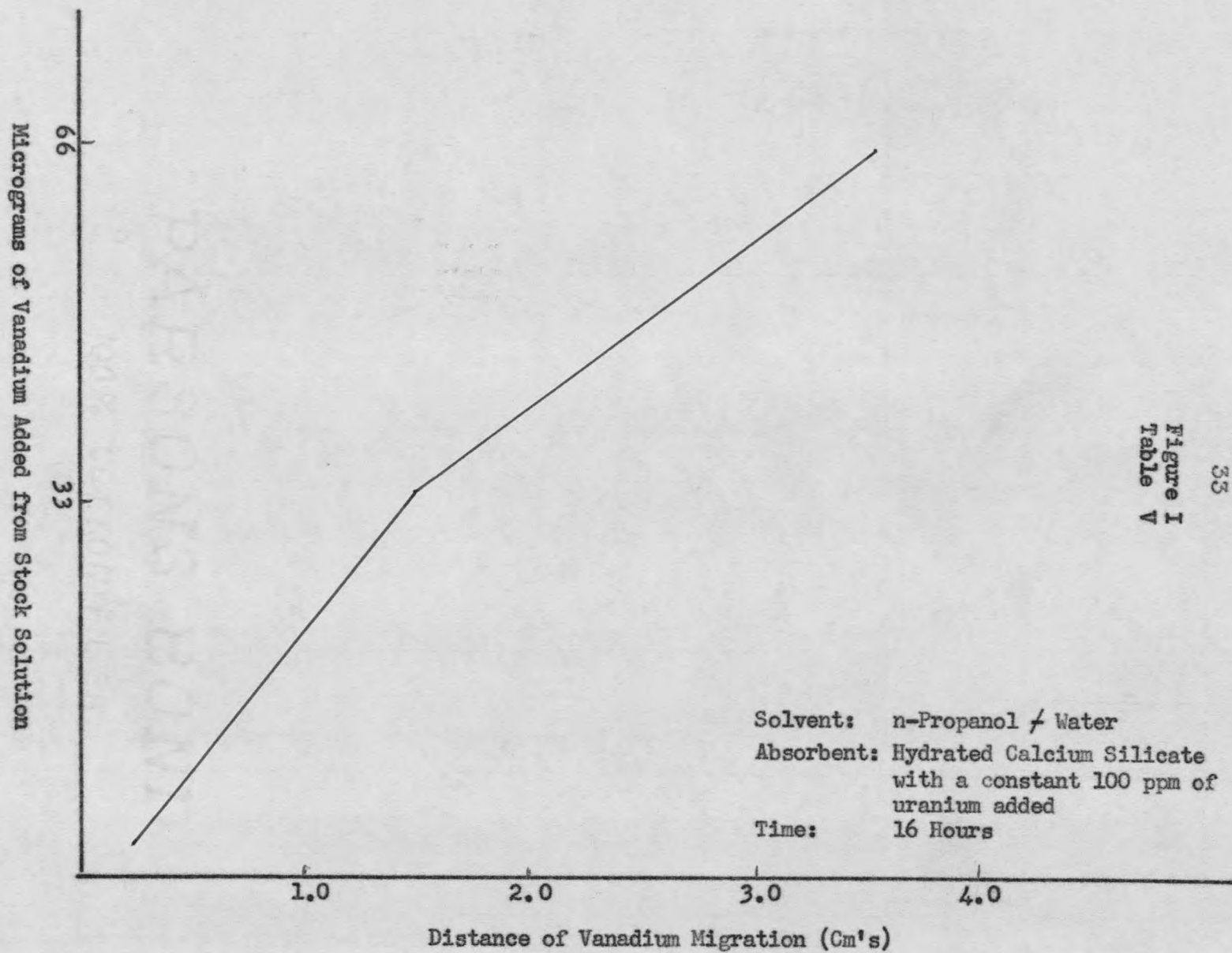
Figure I
Table V

Table VI

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Fluorovanadate in grams/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
102	3	Hydrated Calcium Silicate	9	12.5	.23	.7	16
103	"	"	"	25	.45	1.6	"
104	"	"	"	37.5	.68	2.2	"
105	"	"	"	50	.90	2.9	"
106	"	"	"	67.5	1.12	3.5	"
107	"	"	"	75	1.35	4.1	"
108	"	"	"	87.5	1.58	4.9	"
109	"	"	"	100	1.8	5.4	"
110	"	"	"	125	2.25	6.5	"
111	"	"	"	150	2.7	8.2	"
112	"	"	"	175	3.15	8.9	"
113	"	"	"	200	3.6	10.8	"

Comments: In this series a distinct change in color took place as the concentration went up. The series is grey to pale green to dark green with a grey ring above the green. At the highest concentration the absorbent channeled a great deal, making the measurements vary from what they would be expected to be.

35

Figure I
Table VI

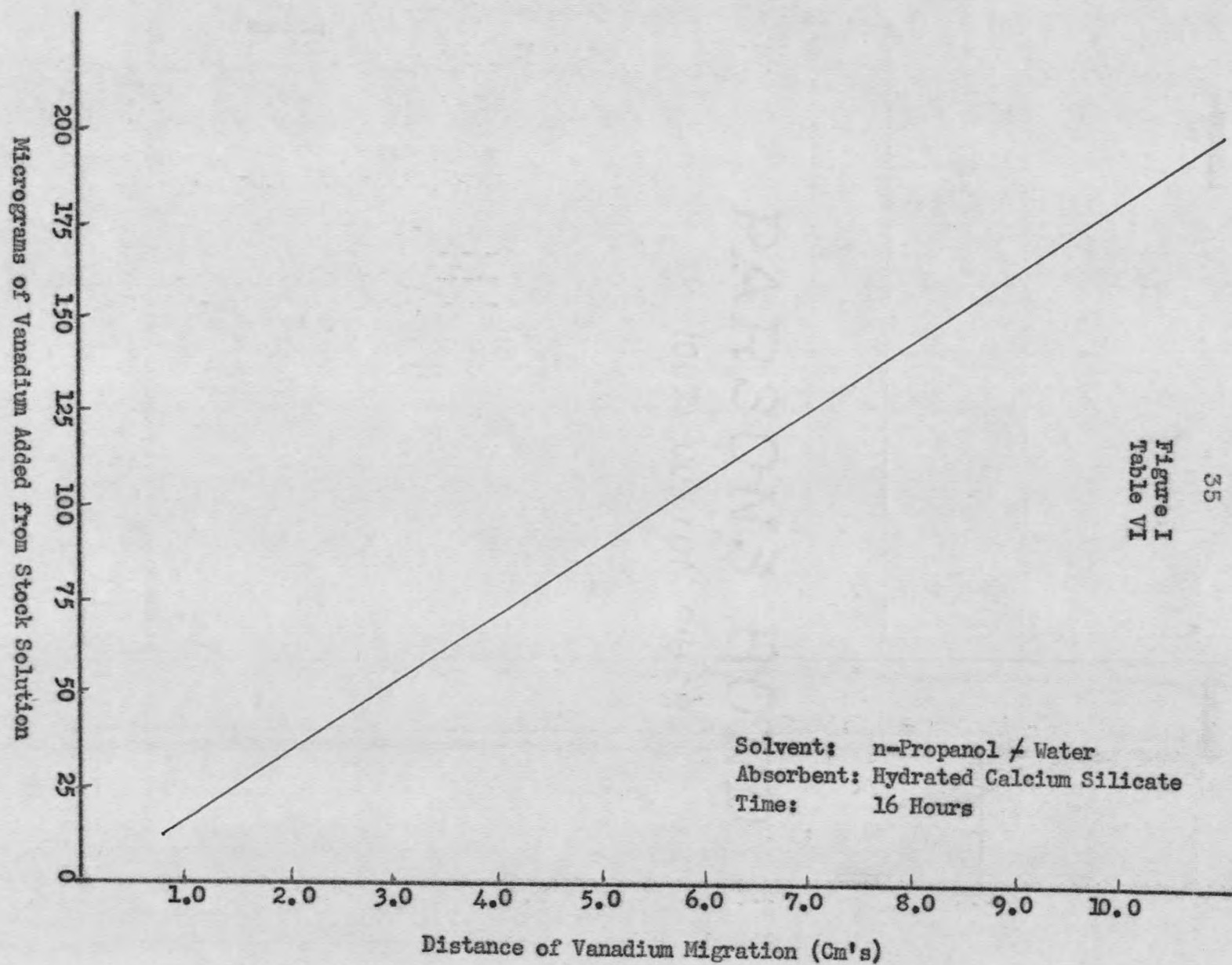


Table VII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
102B	3	Hydrated Calcium Silicate with 1N Phosphate Soln.	9	12.5	.23	.8	16
103B	"	"	"	25	.45	1.6	"
104B	"	"	"	37.5	.68	2.2	"
105B	"	"	"	50	.90	2.6	"
106B	"	"	"	67.5	1.12	3.1	"
107B	"	"	"	75	1.35	3.6	"
108B	"	"	"	87.5	1.58	4.1	"
109B	"	"	"	100	1.8	4.5	"
110B	"	"	"	125	2.25	5.5	"
111B	"	"	"	150	2.7	6.4	"
112B	"	"	"	175	3.15	7.4	"
113B	"	"	"	200	3.6	8.2	"

Comments: The color change in this series was from a dark grey at the lowest concentration to a deep yellow with a green line below it at the highest concentration.

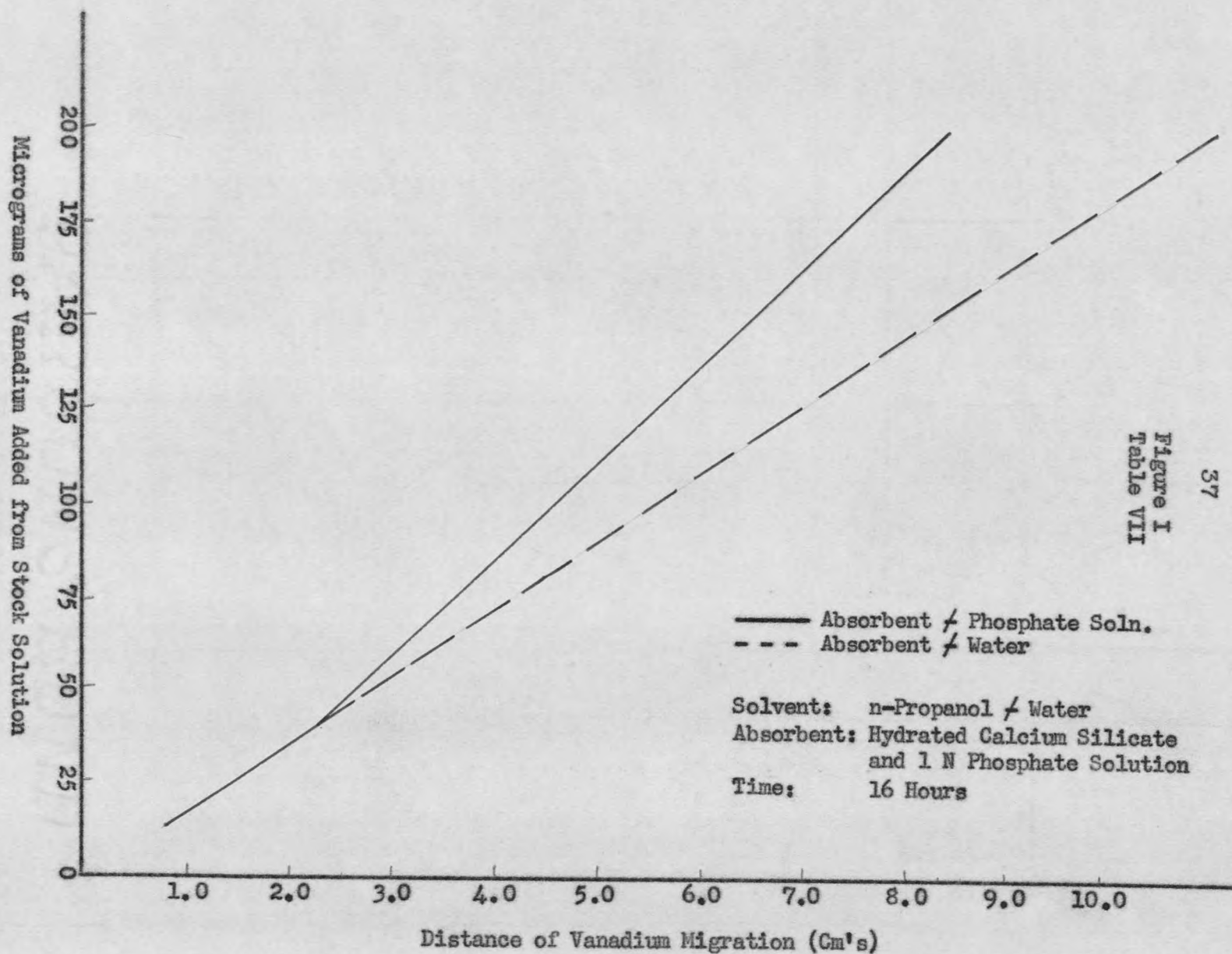
Figure I
Table VII

Table VIII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml. of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1020	3	Diatomaceous Earth	9	12.5	.23	.8	16
1030	"	"	"	25	.45	1.2	"
1040	"	"	"	37.5	.68	1.8	"
1050	"	"	"	50	.90	2.3	"
1060	"	"	"	67.5	1.12	3.0	"
1070	"	"	"	75	1.35	3.7	"
1080	"	"	"	87.5	1.58	4.2	"
1090	"	"	"	100	1.8	4.6	"
1100	"	"	"	125	2.25	5.7	"
1110	"	"	"	150	2.7	7.0	"
1120	"	"	"	175	3.15	8.0	"
1130	"	"	"	200	3.6	9.2	"

Comments: The color change in this series was from a yellow at the lowest concentration to a dark green at the highest concentration. From 1100 up the acid concentration caused channeling.

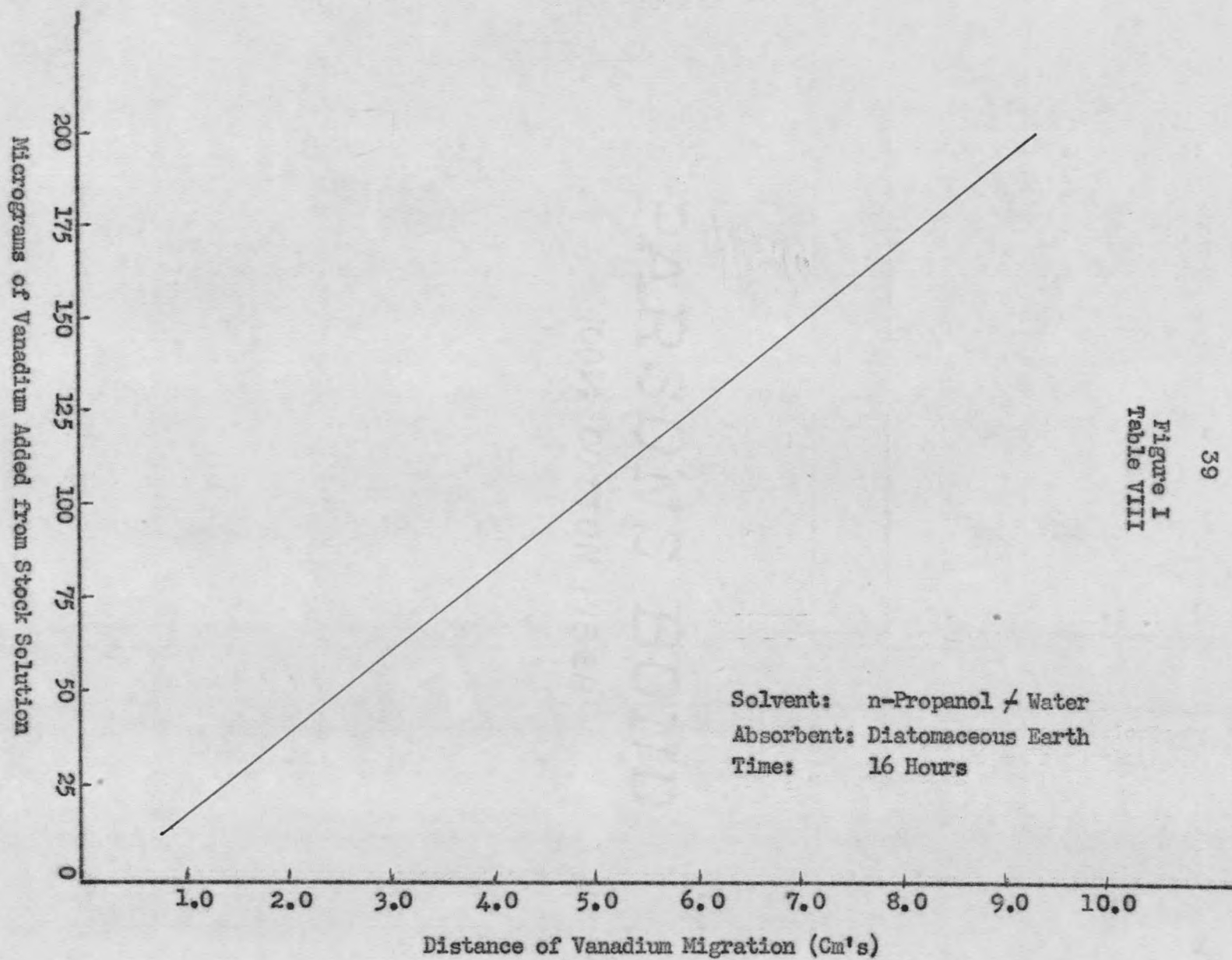
Figure I
Table VIII

Table IX

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
123	2	Hydrated Calcium Silicate	9	10		*2	16
124	"	"	"	20		*2	"
125	"	"	"	30		*2	"
126	"	"	"	40		*2	"
127	"	"	"	50		*2	"
128	"	"	"	60		*2	"
129	"	"	"	80		*2	"
130	"	"	"	100		*2	"
131	"	"	"	125		*2	"
132	"	"	"	150		*2	"
133	"	"	"	175		*2	"
134	"	"	"	200		*2	"
135	"	"	11	10		*1	"
136	"	"	"	30		*1	"
137	"	"	"	50		*1	"
138	"	"	"	70		*1	"

Comments: No color developed in the above tests until 8-hydroxyquinoline was passed through the columns. With this reagent a reddish-brown color developed, deepening in color as the concentration went up.

Table X

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
123B	2	Diatomaceous Earth	9	10	Neutral	.15	16
124B	"	"	"	20		.15	"
125B	"	"	"	30		.15	"
126B	"	"	"	40		.15	"
127B	"	"	"	50		.15	"
128B	"	"	"	60		.15	"
129B	"	"	"	80		.15	"
130B	"	"	"	100		.15	"
131B	"	"	"	125		.15	"
132B	"	"	"	150		.15	"
133B	"	"	"	175		.15	"
134B	"	"	"	200		.15	"
135B	"	"	11	10		.10	"
136B	"	"	"	30		.10	"
137B	"	"	"	50		.10	"
138B	"	"	"	70		.10	"

Comments: No color developed in the above tests until 8-Hydroxyquinoline was passed through the columns. With this reagent an indistinct reddish color appeared that was partially covered by the color of the absorbent.

Table XI

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1230	3	Hydrated Calcium Silicate	9	10	Neutral	.2	16
1240	"	"	"	20		.2	"
1250	"	"	"	50		.2	"
1260	"	"	"	100		.2	"
1270	"	"	"	150		.2	"
1280	"	"	"	200		.2	"
1290	4	"	"	10		.2	"
1300	"	"	"	20		.2	"
1310	"	"	"	50		.2	"
1320	"	"	"	100		.2	"
1330	"	"	"	150		.2	"
1340	"	"	"	200		.2	"
1350	3	"	11	10		.15	"
1360	"	"	"	100		.15	"
1370	4	"	"	10		.15	"
1380	"	"	"	100		.15	"

Comments: No color developed in the above tests until 8-Hydroxyquinoline was passed through the columns.

Table XII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micro-grams/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
123D	3	Hydrated Calcium Silicate	9	10	Neutral	.15	16
124D	"	"	"	20		.15	"
125D	"	"	"	50		.15	"
126D	"	"	"	100		.15	"
127D	"	"	"	150		.15	"
128D	"	"	"	200		.15	"
129D	4	"	"	10		.15	"
130D	"	"	"	20		.15	"
131D	"	"	"	50		.15	"
132D	"	"	"	100		.15	"
133D	"	"	"	150		.15	"
134D	"	"	"	200		.15	"
135D	3	"	11	10		.1	16
136D	"	"	"	100		.1	"
137D	4	"	"	10		.1	"
138D	"	"	"	100		.1	"

Comments: No color developed in the above tests until 8-Hydroxyquinoline was added to the columns.

Table XIII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
144	1	Hydrated Calcium Silicate	9	255	-	-	16
145	"	"	"	"	.01	T	"
146	"	"	"	"	.02	T	"
147	"	"	"	"	.03	T	"
148	"	"	"	"	.04	T	"
149	"	"	"	"	.05	T	"
150	4	"	"	"	-	-	"
151	"	"	"	"	.01	T	"
152	"	"	"	"	.02	T	"
153	"	"	"	"	.03	T	"
154	"	"	"	"	.04	T	"
155	"	"	"	"	.05	T	"

Comments: Color had to be developed with 8-Hydroxyquinoline.
The maximum movement was 1mm.

Table XIV

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
156	1	Hydrated Calcium Silicate	9	255	Neut.	1	16
157	"	"	"	"	.04	.1	"
158	"	"	"	"	.06	.2	"
159	"	"	"	"	.08	.3	"
160	"	"	"	"	.10	.4	"
161	"	"	"	"	.12	.5	"
162	"	"	"	"	.14	.6	"
163	"	"	"	"	.16	.7	"
164	"	"	"	"	.18	.8	"
165	"	"	"	"	.20	.9	"
166	"	"	"	"	.22	1.0	"
167	"	"	"	"	.24	1.1	"

Comments: Acid added in each case was HNO_3 . The line at the base of the vanadium migration was a yellow-green in each case. All of the vanadium seemed to be held at the top of the column in the neutral solution.

Figure I
Tables XIV, XVI, and XVII

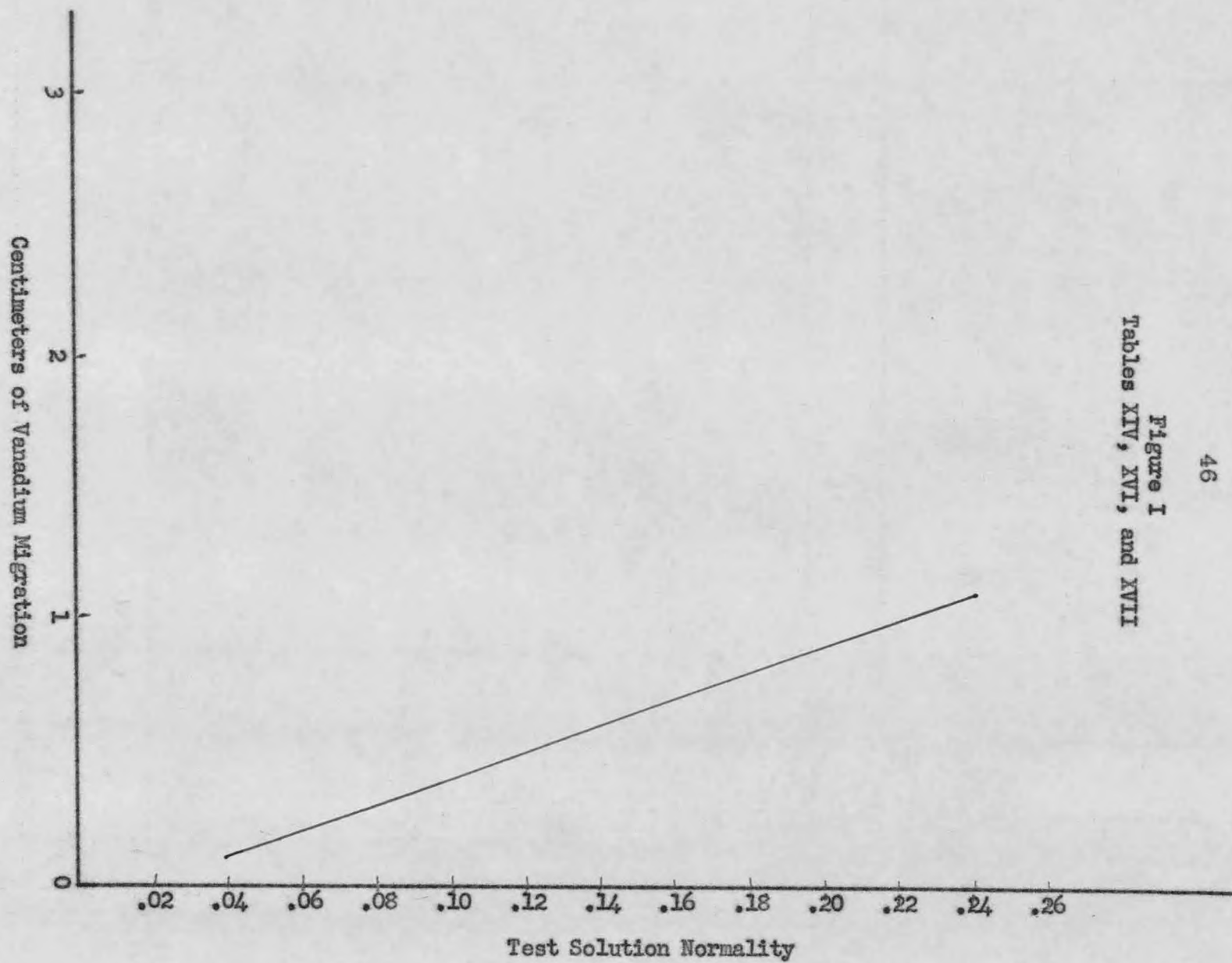


Table XV

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
156B	1	Diatomaceous Earth	9	255	Neut.	1	16
157B	"	"	"	"	.04	1	"
158B	"	"	"	"	.06	5	"
159B	"	"	"	"	.08	.1	"
160B	"	"	"	"	.10	.2	"
161B	"	"	"	"	.12	.3	"
162B	"	"	"	"	.14	.4	"
163B	"	"	"	"	.16	.5	"
164B	"	"	"	"	.18	.6	"
165B	"	"	"	"	.20	.7	"
166B	"	"	"	"	.22	.8	"
167B	"	"	"	"	.24	.9	"

Comments: Acid added in each case was HNO_3 . The line at the base of the vanadium migration was a dark yellow-green in each case.

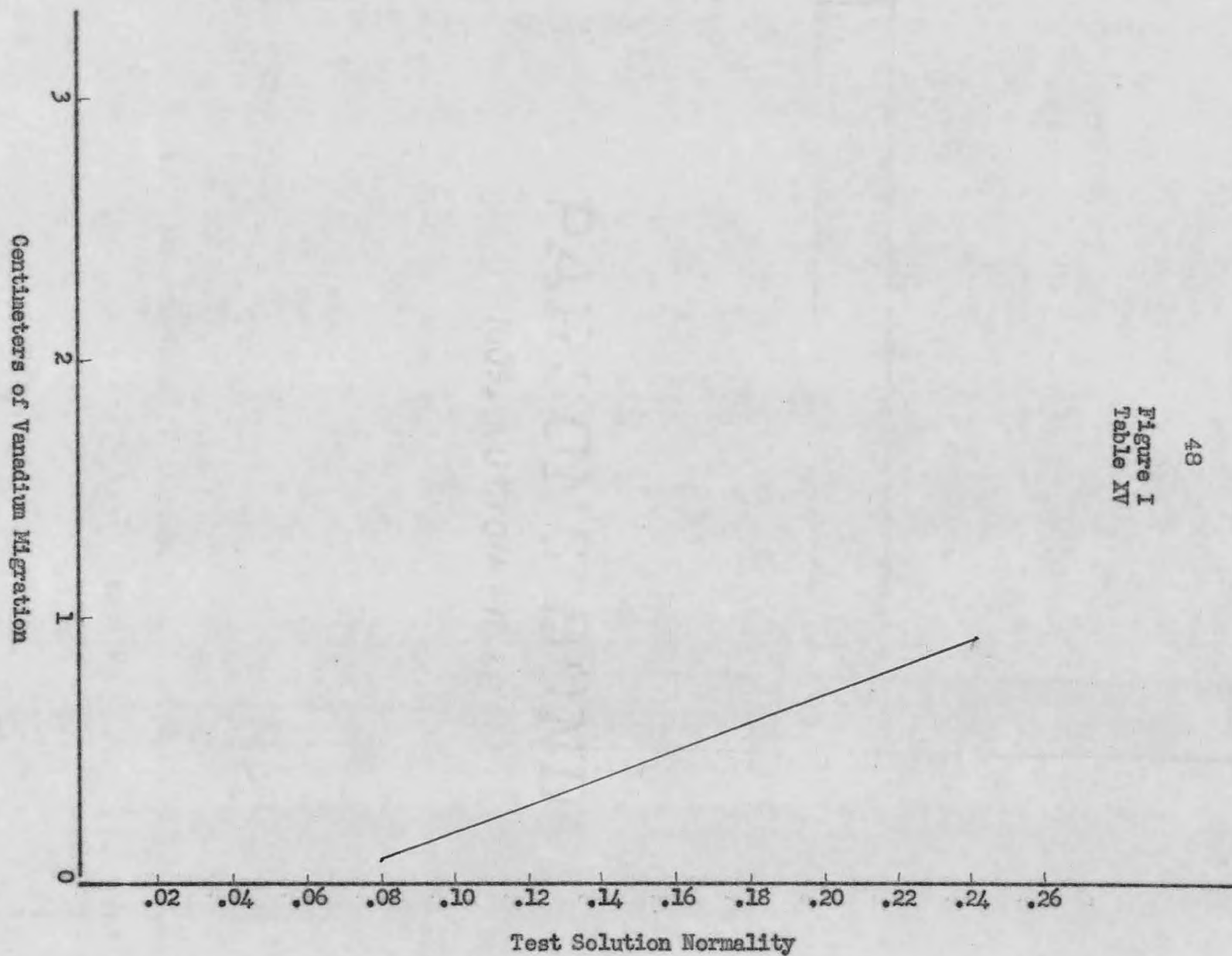
Figure I
Table XV

Table XVI

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1560	1	Hydrated Calcium Silicate with 1 N Phosphate Soln.	9	255	neut.	-	16
1570	"	"	"	"	.04	.1	"
1580	"	"	"	"	.06	.2	"
1590	"	"	"	"	.08	.3	"
1600	"	"	"	"	.10	.4	"
1610	"	"	"	"	.12	.5	"
1620	"	"	"	"	.14	.6	"
1630	"	"	"	"	.16	.7	"
1640	"	"	"	"	.18	.8	"
1650	"	"	"	"	.20	.9	"
1660	"	"	"	"	.22	1.0	"
1670	"	"	"	"	.24	1.1	"

Comments: Acid added in each case was HNO_3 . The line at the base of the vanadium migration was a yellow-green in each case. All of the vanadium seemed to be held at the top of the columns in the neutral solution or at least it did not migrate more than 1 cm.

Table XVII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
156D	1	Hydrated Calcium Silicate with 1 N K_2SO_4	9	255	neut.	-	16
157D	"	"	"	"	.04	.1	"
158D	"	"	"	"	.06	.2	"
159D	"	"	"	"	.08	.3	"
160D	"	"	"	"	.10	.4	"
161D	"	"	"	"	.12	.5	"
162D	"	"	"	"	.14	.6	"
163D	"	"	"	"	.16	.7	"
164D	"	"	"	"	.18	.8	"
165D	"	"	"	"	.20	.9	"
166D	"	"	"	"	.22	1.0	"
167D	"	"	"	"	.24	1.1	"

Table XVIII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micro-grams/ml. of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
156E	1	Hydrated Calcium Silicate with 1 N KClO_4 Soln.	9	255	neut.	-	16
157E	"	"	"	"	.04	.1	"
158E	"	"	"	"	.06	.2	"
159E	"	"	"	"	.08	.3	"
160E	"	"	"	"	.10	.4	"
161E	"	"	"	"	.12	.5	"
162E	"	"	"	"	.14	.6	"
163E	"	"	"	"	.16	.7	"
164E	"	"	"	"	.18	.8	"
165E	"	"	"	"	.20	.9	"
166E	"	"	"	"	.22	1.0	"
167E	"	"	"	"	.24	1.1	"

Table XIX

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
173	1	Hydrated Calcium Silicate	9	255	.24	1.1	16
174	"	"	"	"	.28	1.3	"
175	"	"	"	"	.32	1.4	"
176	"	"	"	"	.36	1.5	"
177	"	"	"	"	.40	1.6	"
178	"	"	"	"	.44	1.7	"
179	"	"	"	"	.48	1.8	"
180	"	"	"	"	.52	2.0	"
181	"	"	"	"	.56	2.1	"
182	"	"	"	"	.60	2.3	"
183	"	"	"	"	.64	2.3	"
184	"	"	"	"	.68	2.4	"
185	"	"	"	"	.72	2.5	"
186	"	"	"	"	.76	2.5	"
187	"	"	"	"	.80	2.8	"
188	"	"	"	"	.90	3.0	"
189	"	"	"	"	1.0	3.2	"

Comments: Acid added was HNO_3 in each case. The line at the base of the vanadium migration was a yellow-green with a bright yellow line 1mm wide above it in each case.

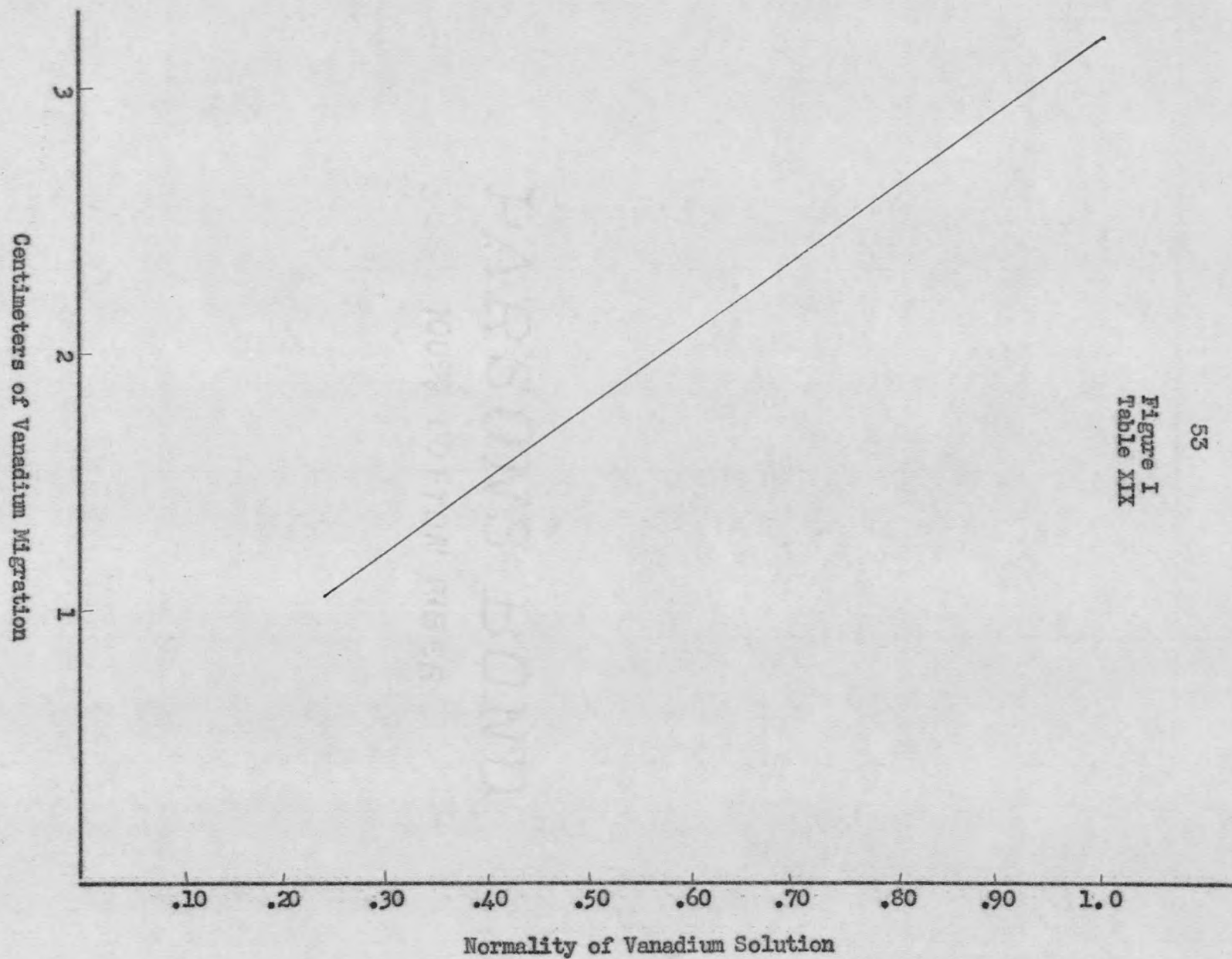
Figure I
Table XIX

Table XX

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
173B	1	Hydrated Calcium Silicate with 1 N Phosphate Soln.	9	255	.24	1.1	16
174B	"	"	"	"	.28	1.2	"
175B	"	"	"	"	.32	1.35	"
176B	"	"	"	"	.36	1.45	"
177B	"	"	"	"	.40	1.6	"
178B	"	"	"	"	.44	1.7	"
179B	"	"	"	"	.48	1.8	"
180B	"	"	"	"	.52	1.9	"
181B	"	"	"	"	.56	2.0	"
182B	"	"	"	"	.60	2.0	"
183B	"	"	"	"	.64	2.15	"
184B	"	"	"	"	.68	2.25	"
185B	"	"	"	"	.72	2.3	"
186B	"	"	"	"	.76	2.35	"
187B	"	"	"	"	.80	2.45	"
188B	"	"	"	"	.90	2.65	"
189B	"	"	"	"	1.00	2.8	"

Comments: The end of the vanadium migration in this series was a pale yellow color in a line about 5mm wide. The color was very distinct even though a pale color.

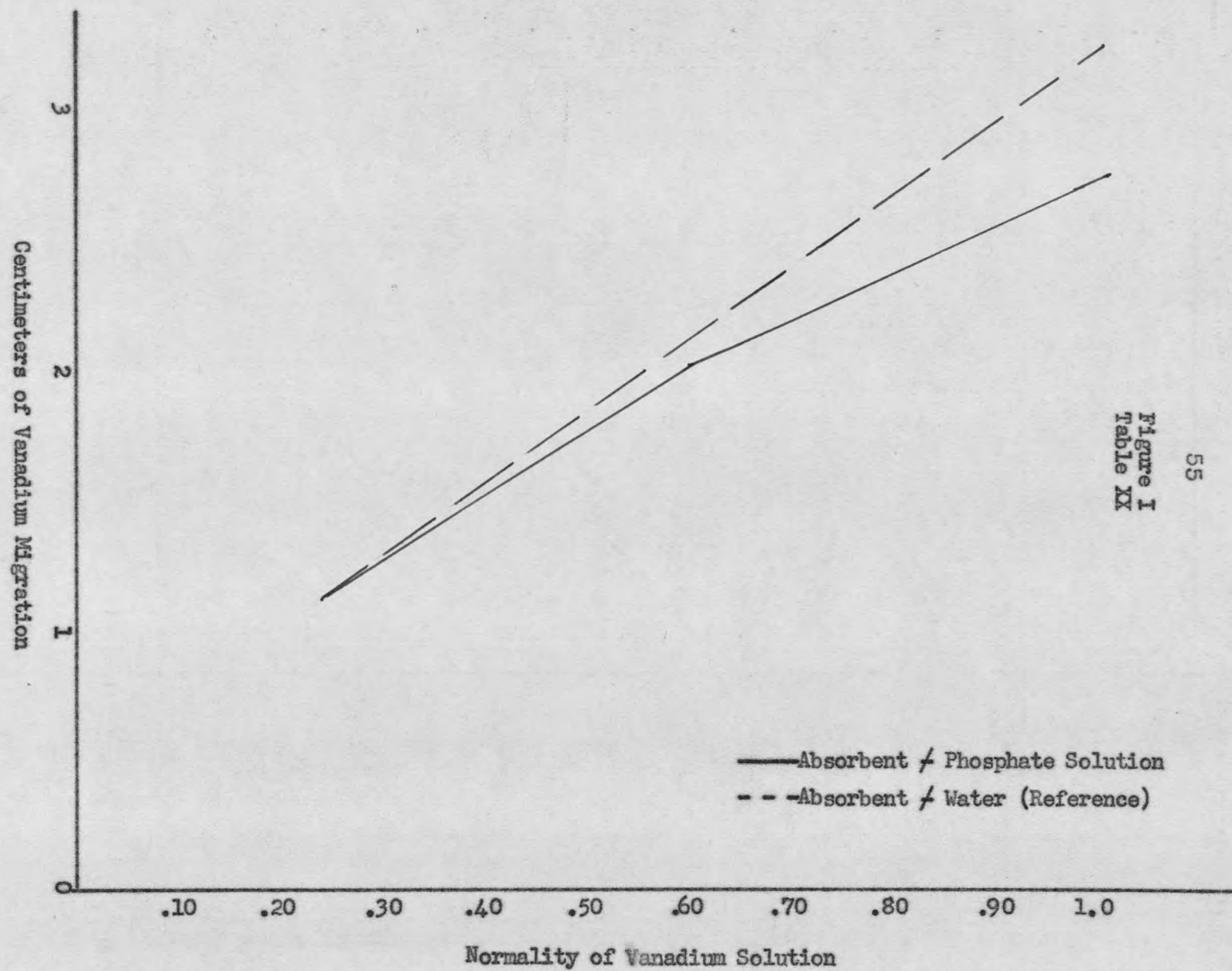
Figure I
Table XX

Table XXI

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1730	1	Hydrated Calcium Silicate with 1 N K_2SO_4	9	255	.24	1.1	16
1740	"	"	"	"	.28	1.2	"
1750	"	"	"	"	.32	1.3	"
1760	"	"	"	"	.36	1.4	"
1770	"	"	"	"	.40	1.55	"
1780	"	"	"	"	.44	1.7	"
1790	"	"	"	"	.48	1.8	"
1800	"	"	"	"	.52	1.95	"
1810	"	"	"	"	.56	2.1	"
1820	"	"	"	"	.60	2.2	"
1830	"	"	"	"	.64	2.3	"
1840	"	"	"	"	.68	2.5	"
1850	"	"	"	"	.72	2.6	"
1860	"	"	"	"	.76	2.7	"
1870	"	"	"	"	.80	2.85	"
1880	"	"	"	"	.90	3.2	"
1890	"	"	"	"	1.0	3.5	"

Comments: The color at the base of the vanadium migration was a green line with a dark yellow above it.

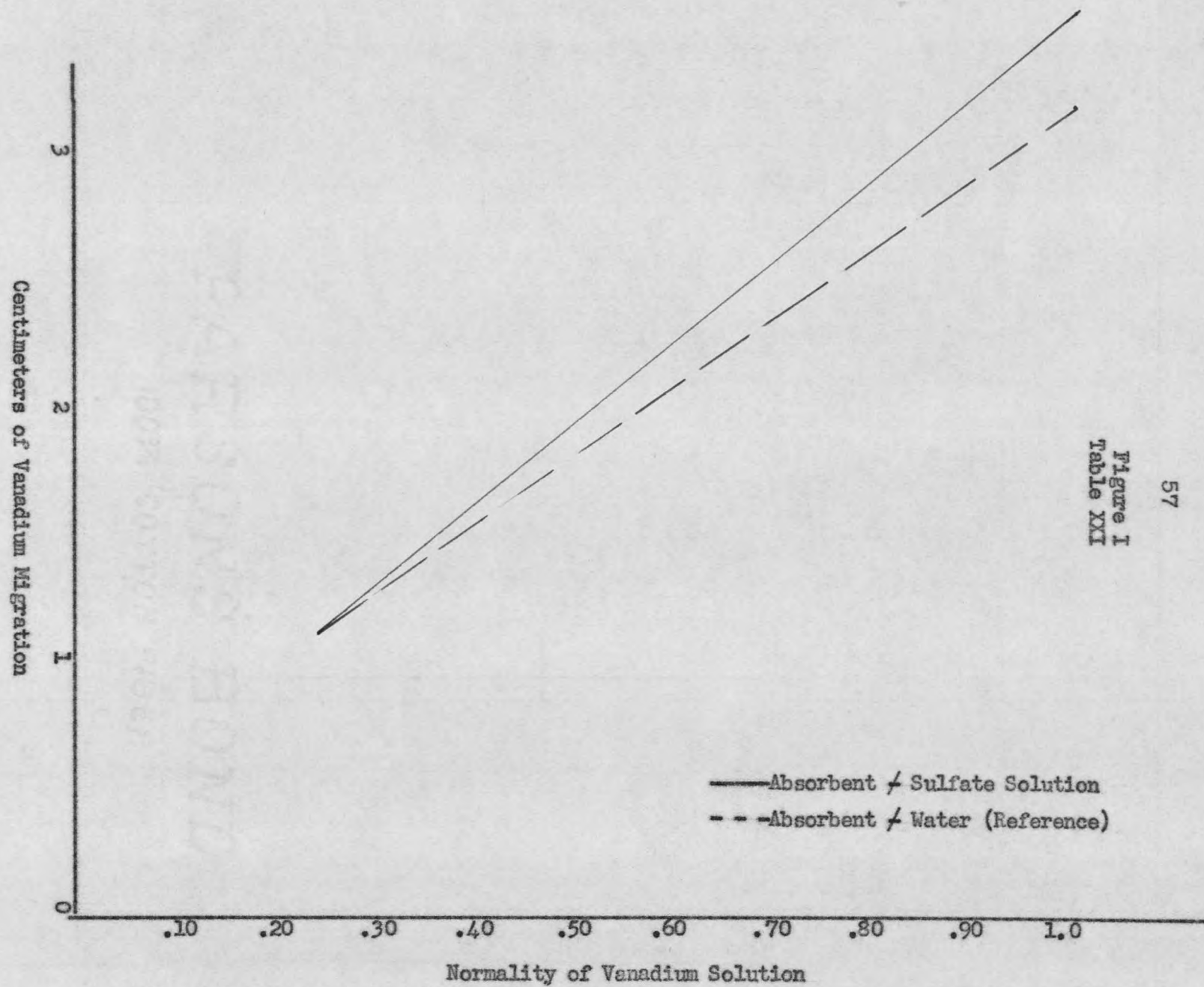
Figure I
Table XXI

Table XXIII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
173D	1	Hydrated Calcium Silicate with 1 N $KClO_4$	9	255	.24	1.1	16
174D	"	"	"	"	.28	1.2	"
175D	"	"	"	"	.32	1.3	"
176D	"	"	"	"	.36	1.4	"
177D	"	"	"	"	.40	1.55	"
178D	"	"	"	"	.44	1.7	"
179D	"	"	"	"	.48	1.85	"
180D	"	"	"	"	.52	2.0	"
181D	"	"	"	"	.56	2.15	"
182D	"	"	"	"	.60	2.3	"
183D	"	"	"	"	.64	2.4	"
184D	"	"	"	"	.68	2.55	"
185D	"	"	"	"	.72	2.7	"
186D	"	"	"	"	.76	2.85	"
187D	"	"	"	"	.80	3.0	"
188D	"	"	"	"	.90	3.3	"
189D	"	"	"	"	1.00	3.75	"

Comments: The color at the base of the vanadium migration was a deep yellow with a tendency toward a green in some of the columns.

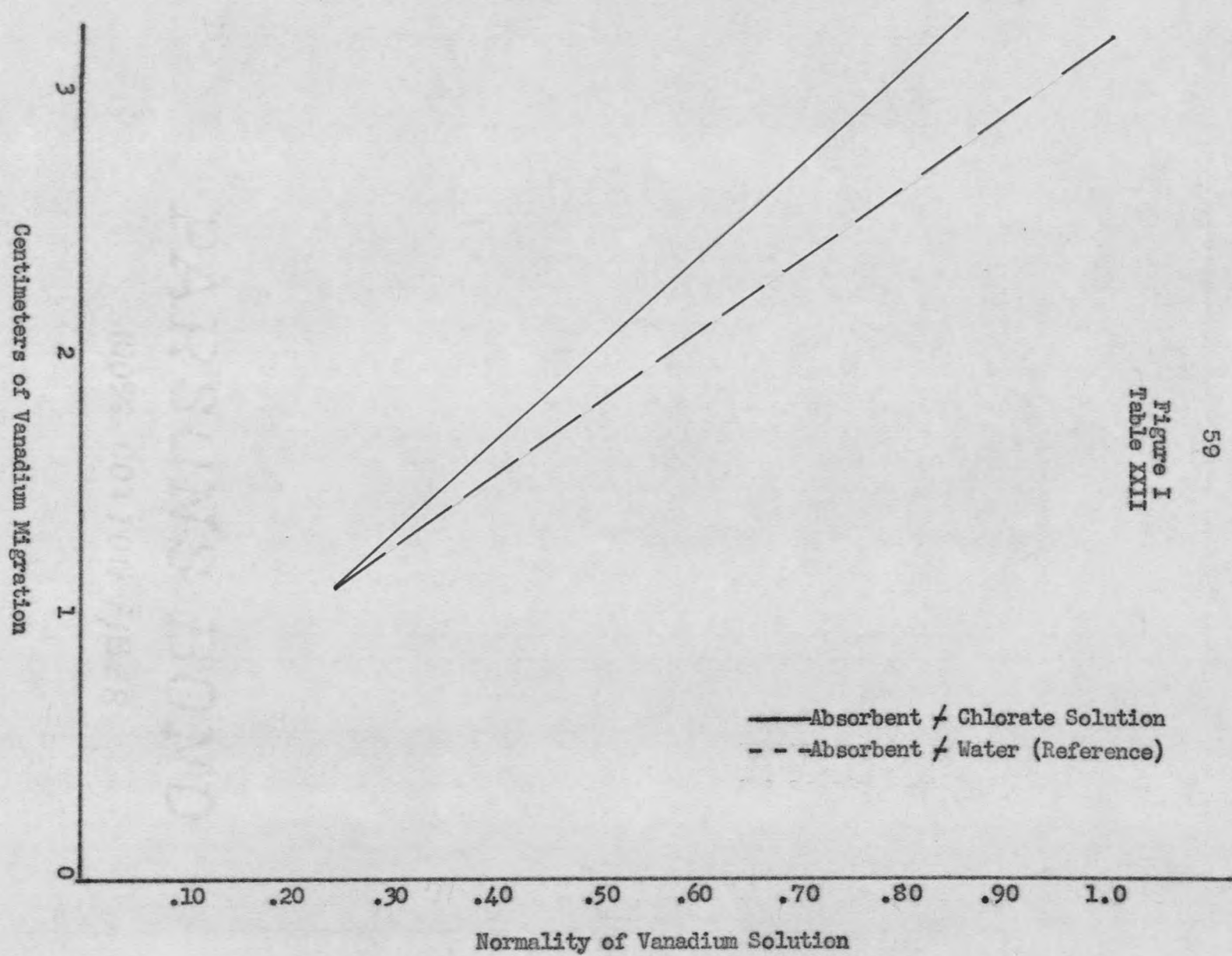
Figure I
Table XII

Table XXIII

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
190	1	Diatomaceous Earth	9	255	.24	.9	16
191	"	"	"	"	.28	1.0	"
192	"	"	"	"	.32	1.1	"
193	"	"	"	"	.36	1.2	"
194	"	"	"	"	.40	1.3	"
195	"	"	"	"	.44	1.35	"
196	"	"	"	"	.48	1.45	"
197	"	"	"	"	.52	1.55	"
198	"	"	"	"	.56	1.6	"
199	"	"	"	"	.60	1.7	"
200	"	"	"	"	.64	1.8	"
201	"	"	"	"	.68	1.9	"
202	"	"	"	"	.72	2.0	"
203	"	"	"	"	.76	2.1	"
204	"	"	"	"	.80	2.2	"
205	"	"	"	"	.90	2.4	"
206	"	"	"	"	1.0	2.6	"

Comments: Acid added in each case was HNO_3 . The line at the base of the vanadium migration was a dark yellow-green in each case.

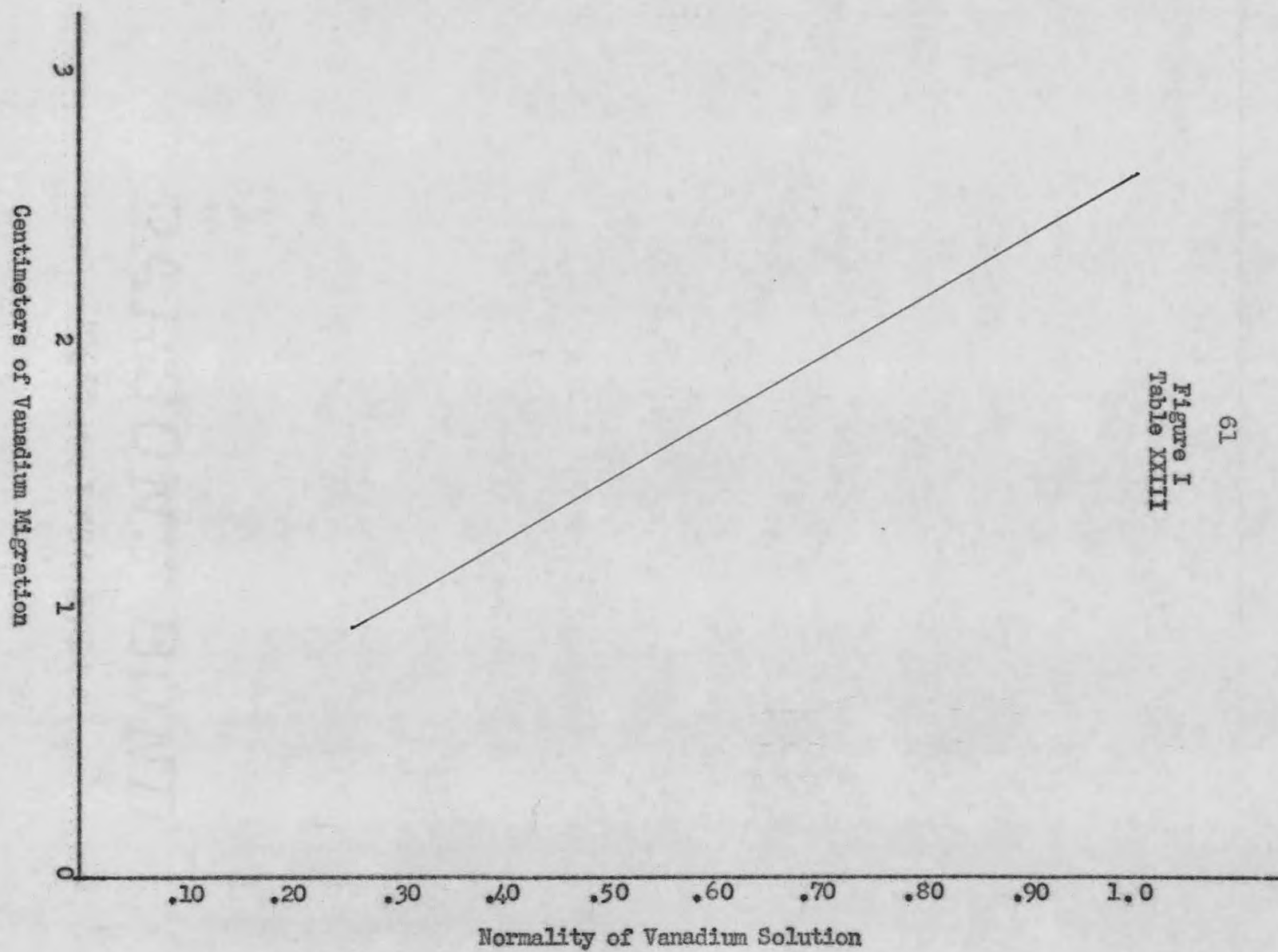
Figure I
Table XIII

Table XXIV

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
190B	1	Diatomaceous Earth with 1 N Phosphate Soln.	9	255	.24	.9	16
191B	"	"	"	"	.28	1.0	"
192B	"	"	"	"	.32	1.1	"
193B	"	"	"	"	.36	1.2	"
194B	"	"	"	"	.40	1.3	"
195B	"	"	"	"	.44	1.35	"
196B	"	"	"	"	.48	1.45	"
197B	"	"	"	"	.52	1.55	"
198B	"	"	"	"	.56	1.6	"
199B	"	"	"	"	.60	1.7	"
200B	"	"	"	"	.64	1.75	"
201B	"	"	"	"	.68	1.8	"
202B	"	"	"	"	.72	1.85	"
203B	"	"	"	"	.76	1.90	"
204B	"	"	"	"	.80	1.95	"
205B	"	"	"	"	.90	2.05	"
206B	"	"	"	"	1.00	2.15	"

Comments: The acid added in each case was Nitric acid. The color at the base of the vanadium migration was a bright yellow.

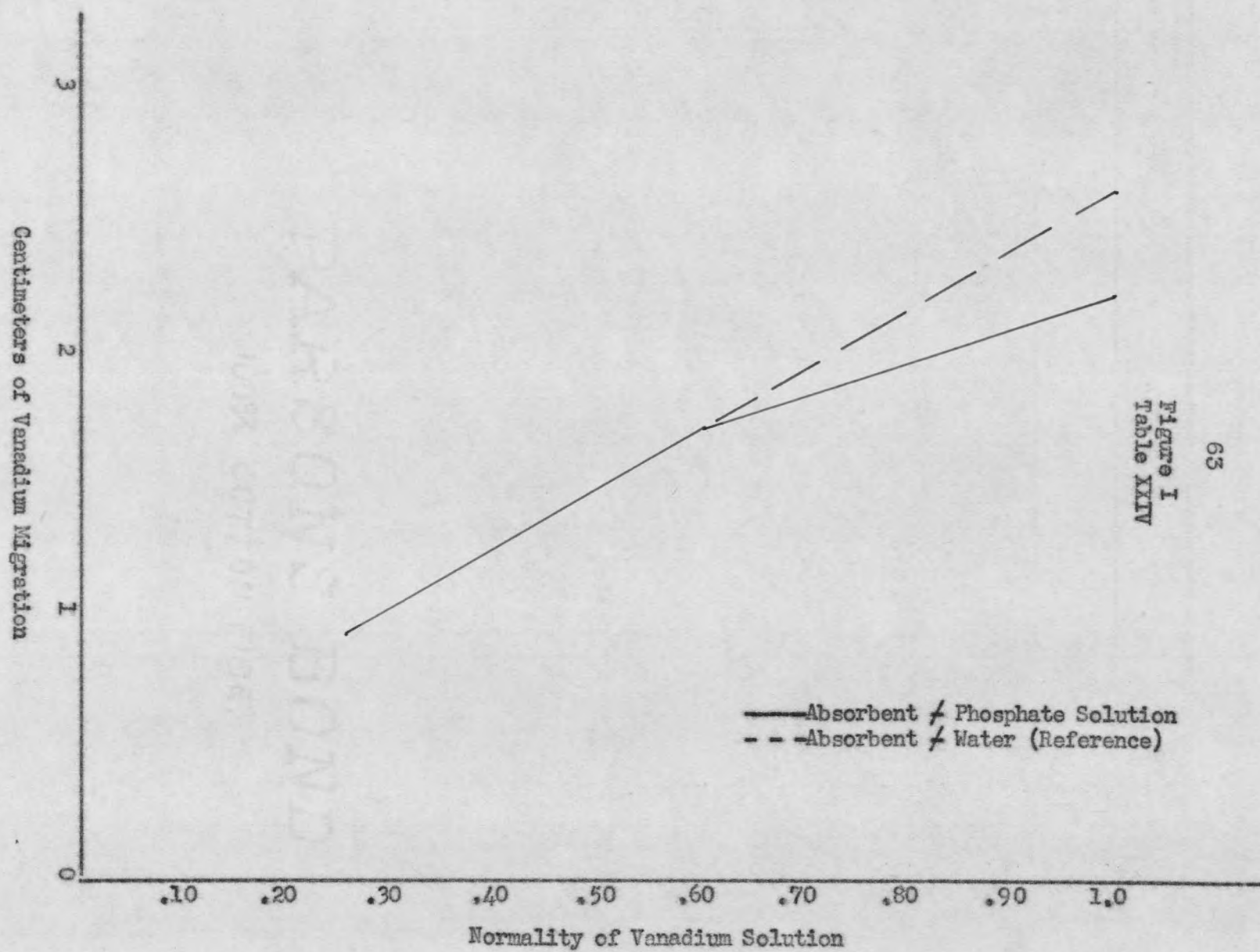
Figure I
Table XXIV

Table XXV

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micrograms/ml of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
1900	1	Diatomaceous Earth with	9	255	.24	.9	16
1910	"	1 N Potassium Sulfate solution added.	"	"	.28	1.0	"
1920	"	"	"	"	.32	1.1	"
1930	"	"	"	"	.36	1.2	"
1940	"	"	"	"	.40	1.3	"
1950	"	"	"	"	.44	1.4	"
1960	"	"	"	"	.48	1.5	"
1970	"	"	"	"	.52	1.6	"
1980	"	"	"	"	.56	1.7	"
1990	"	"	"	"	.60	1.8	"
2000	"	"	"	"	.64	1.95	"
2010	"	"	"	"	.68	2.05	"
2020	"	"	"	"	.72	2.15	"
2030	"	"	"	"	.76	2.25	"
2040	"	"	"	"	.80	2.35	"
2050	"	"	"	"	.90	2.6	"
2060	"	"	"	"	1.00	2.85	"

Comments: The line at the base of the vanadium migration was a dark green line with a dark yellow band above it.

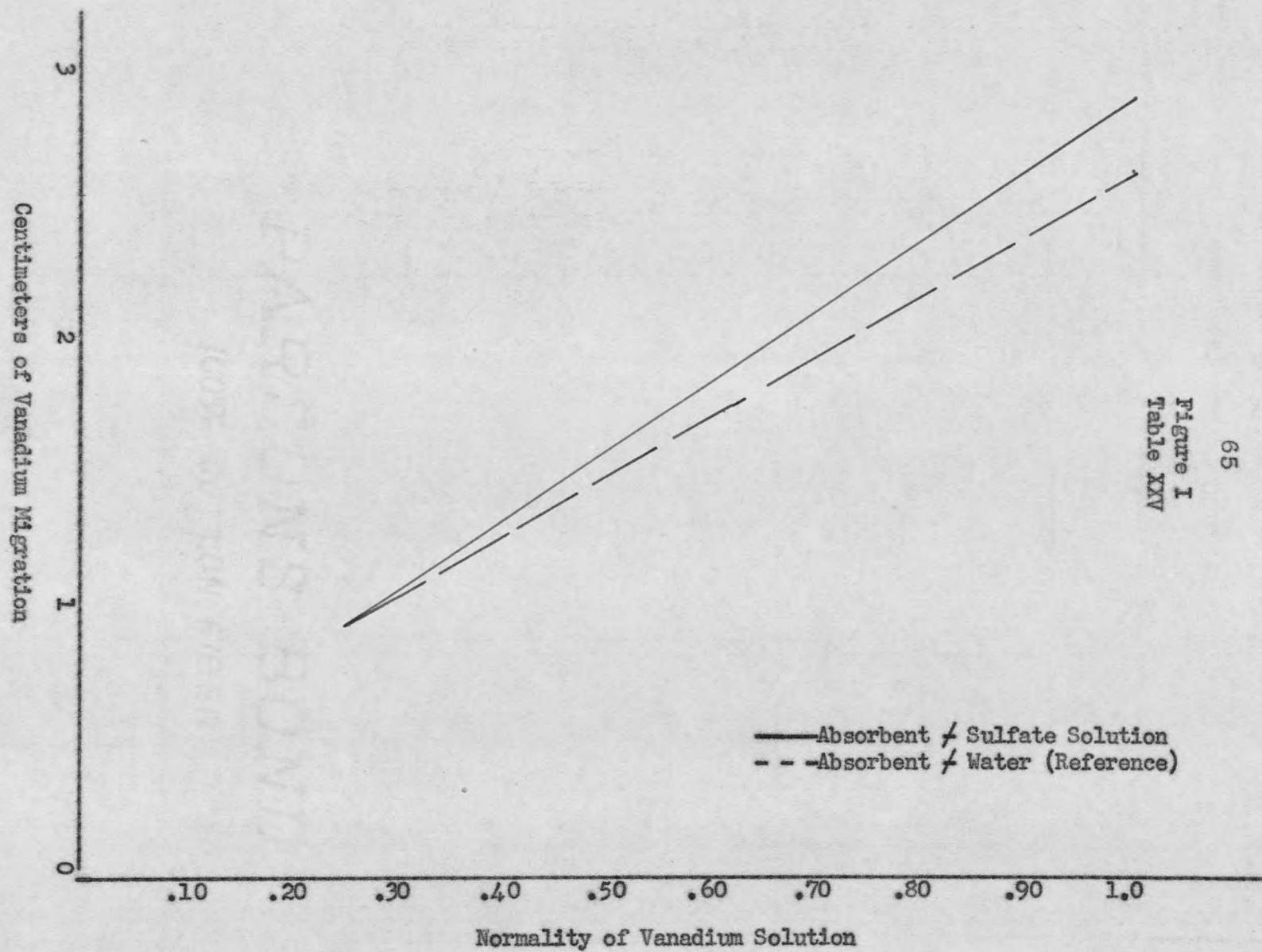
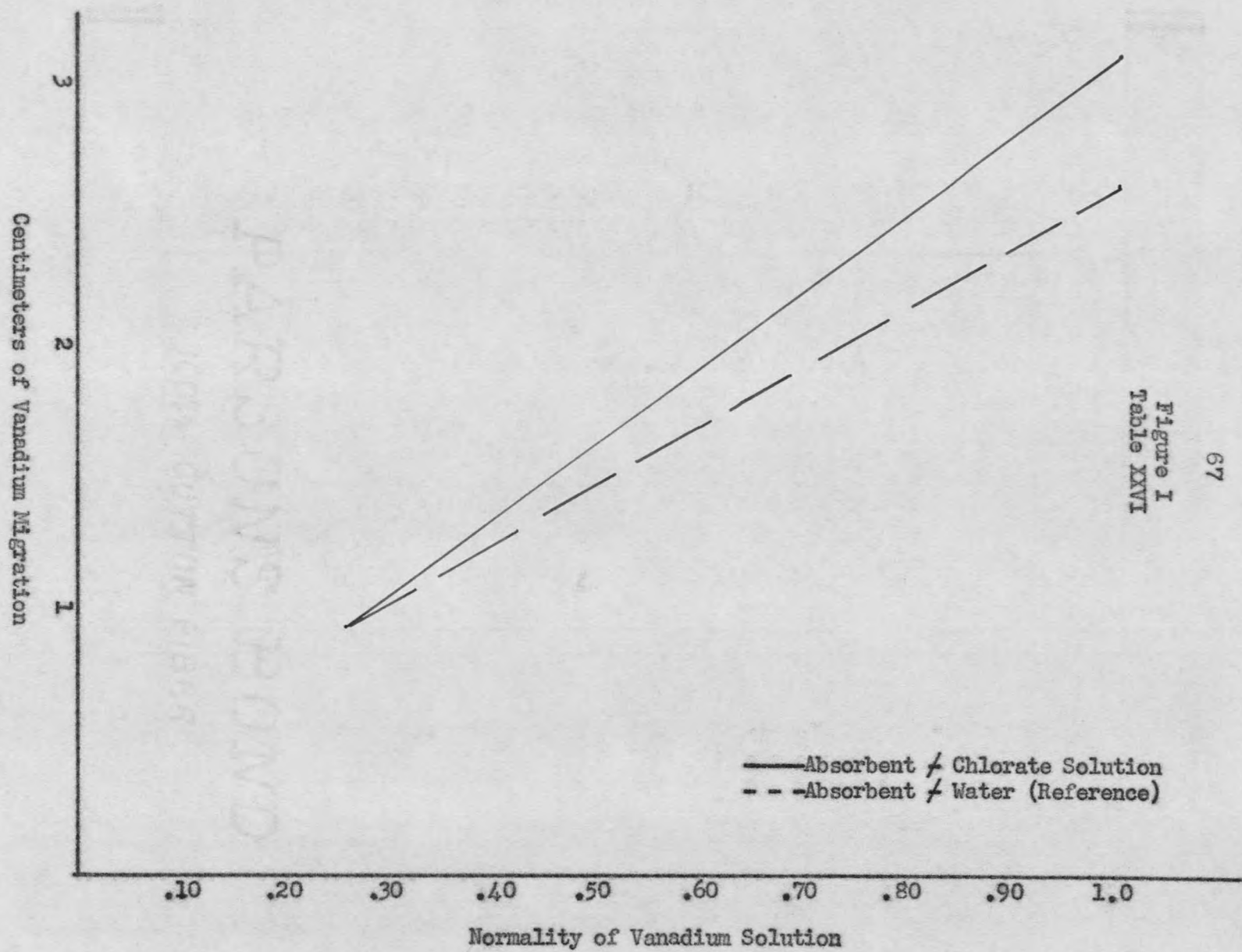
Figure I
Table XV

Table XXVI

Sample Number	Solution Number	Absorbent	Solvent Number	Concentration of Vanadium in Micro-grams/ml. of Soln.	Solution Normality	Distance of Vanadium Migration in Centimeters	Time of Run in Hours
190D	1	Diatomaceous Earth with 1 N Potassium Chlorate solution added	9	255	.24	.9	16
191D	"		"	"	.28	1.05	"
192D	"		"	"	.32	1.15	"
193D	"		"	"	.36	1.25	"
194D	"		"	"	.40	1.4	"
195D	"		"	"	.44	1.5	"
196D	"		"	"	.48	1.6	"
197D	"		"	"	.52	1.75	"
198D	"		"	"	.56	1.85	"
199D	"		"	"	.60	1.95	"
200D	"		"	"	.64	2.1	"
201D	"		"	"	.68	2.2	"
202D	"		"	"	.72	2.3	"
203D	"		"	"	.76	2.4	"
204D	"		"	"	.80	2.55	"
205D	"		"	"	.90	2.8	"
206D	"		"	"	1.00	3.1	"

Comments: The color at the base of the vanadium migration was a deep yellow band about 5 mm wide in each case.

Figure I
Table XXVI

VII. DISCUSSION

The following is a discussion of Part II only.

It is to be noted first that only two absorbents were used in Part II. The reason for this is that the acidity effect was not noted to such an extreme in any of the other absorbents tried by the author.

In Tables II, III and IV the migration of vanadium at any concentration seems to correlate well with the acidity of the solution it is in. The change from a light to a darker color correlates with the vanadium concentration. Silene E.F. seemed to allow a longer migration distance than Diatomaceous Earth at equal concentrations of vanadium. Addition of phosphate ions seemed to slow the vanadium even more, but not in a linear manner. There is a distinct break point at which the phosphate ions slow down the vanadium to a greater extent.

In Table V the addition of a constant amount of uranium ions lengthened the distance of migration a constant amount. This was due to the acidity of the uranium solution. It is to be noted here that none of the uranium was retained by this absorbent.

In Tables IX, X, XI and XII a migration was observed to occur in neutral or slightly alkaline solution, but it was a constant value. This means that the migration distance is definitely correlated with acidity in these absorbents.

Table XIII illustrated that two forms of vanadium in very low acid concentration migrate only a very short distance. The fact that each migrated equal distances led the author to use only one form in the experiments to follow.

Tables XIV through XXVI and the accompanying graphs show the effects of accurately controlled acid concentrations on the migration of vanadium. They also serve to show the effect of interfering anions to a more accurate degree. Note here that the phosphate ions had a depressing effect on vanadium migration while the sulfate and chlorate ions had a raising effect which was somewhat linear in nature.

With these generalities in mind, obtained from the various tables, we can elaborate further on the data. The most evident fact we can see in the data is that the migration distance of vanadium does depend to a great extent on the acidity of the solution being tested. In fact, over the range of zero to 3 N acidic conditions, the distance of movement is a linear expression of acidity. Above this 3 N acid condition the effect is one of leveling off. Evidently the effect can be explained in the following manner. The active silica forms in the absorbents used had a greater tendency to absorb the hydrogen ions than the vanadium ions with which they came in contact. As the hydrogen ions were used in the process of moving down the columns there is a greater chance for the vanadium to be absorbed. This phenomenon is also shown by the

fact, not mentioned previously, that the band of vanadium color development widens slightly as the acidity goes up. In low acidic conditions the hydrogen ions are absorbed more rapidly giving the vanadium ions less opportunity to be absorbed. As the acidity goes up the time required for absorption goes up at a rate dependent on the speed of solvent flow and various other factors. This means that with a change in time in a localized area vanadium has a greater chance of absorption and may be absorbed in a wider band. This of course infers an unequal spread of hydrogen ions in the columns. Since this acidity effect depends on using the hydrogen ions before the vanadium ions can be absorbed, it would necessarily mean that the migration distance of two unequal concentrations of vanadium at equal acid concentrations should be approximately the same. This was found to be the case.

When foreign anions are added to the absorbent, the distance of vanadium migration changes to an observable degree. In the case of phosphate ions, the distance is shortened. This means that the phosphate ions can absorb the hydrogen ions and keep them immobile so that the vanadium ions can be absorbed sooner. It also means that the areas between the absorbent particles that were formerly filled by water molecules are now filled by a combination of water molecules and reactive phosphate ions. The combination of these two absorbent ions would

shorten the migration distance.

With foreign sulfate and chlorate anions added to the absorbent the migration distance is lengthened. This means that these ions are tending to cut down the absorbing power of the silicate ions. The change in migration distance should be variable with the amount of these foreign anions that are added to the absorbent.

VIII. SUMMARY

1. Change of acidity over the range of zero to 3 N conditions causes a linear raise in the migration distance of vanadium.

2. Above 3 N acid conditions, the distance of migration tends to level off.

3. With a constant acidity and constant vanadium concentration, the distance of vanadium migration is dependent to a large degree on the foreign anions present.

4. If the foreign anions present have a tendency to complex with or absorb vanadium, the migration distance will be shortened. If, on the other hand, the foreign anions depress the absorbing power of the absorbent, the migration distance will be lengthened.

5. With no foreign anions present, acidity constant, and vanadium concentration constant, the migration distance of vanadium depends on the rate of solvent flow, type of absorbent, degree of packing and other miscellaneous factors.

6. With the particular absorbents used by the author, n-Propanol and Water (1:1) was found to be the best solvent. It gave the best separation of vanadium from other ions that occur with vanadium.

7. To develop color for vanadium, oxine in absolute Ethanol is the best reagent available.

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