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Division B

NATIONAL DEFENSE RESEARCH COMMITTEE

OF THE

OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

The Potentiometric Titration of Filtrates from the Bachmann Process (OD-12)

by F. C. Whitmore

OSRD No. 654

Serial No. 276

June 23, 1942

Endorsement (1) from F. C. Whitmore, Chairman, Section B-2 to Roger Adams, Chairman, Division B.

"A procedure is presented which allows the titration of the nitric acid present in Bachmann filtrates, using the glass electrode to determine the end-point. The study was made to determine the minimum amount of base necessary to prevent the appearance of nitric acid in the recovered acetic acid.

No further work is contemplated."

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NATIONAL DEFENSE RESEARCH COMMITTEE
OF THE
OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

Report on "The Potentiometric Titration of
Filtrates from the Bachmann Process."

to

June 22, 1942

by

F. C. Whitmore

OSRD No. 654

Serial No. 276

Copy No. 29

Date: June 23, 1942

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DIVISION B
NATIONAL DEFENSE RESEARCH COMMITTEE
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OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT
Section

Report on "The Potentiometric Titration of
Filtrates from the Bachmann Process."
(OD-12)

by F. C. Whitmore
OSRD No. 654
Serial No. 276

June 23, 1942

Endorsement (1) From F. C. Whitmore Chairman, Section
B-2 to Roger Adams, Chairman, Division B. Forwarding
report and noting:

"A procedure is presented which allows the titration
of the nitric acid present in Bachman filtrates,
using the glass electrode to determine the end-
point. The study was made to determine the minimum
amount of base necessary to prevent the appearance
of nitric acid in the recovered acetic acid.

"No further work is contemplated."

(2) Twenty-three copies forwarded to Dr. Irvin
Stewart, Secretary of the National Defense Research Committee,
as Progress Report under Contract- (B 130, OEMsr 243) with
Pennsylvania State College.

Roger Adams, Chairman
by Harris M. Chadwell
Technical Aide

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THE POTENTIOMETRIC TITRATION OF FILTRATES

FROM THE BACHMANN PROCESS

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ABSTRACT

The details of the study which led to the use of the glass electrode for adjusting the acidity of Bachmann filtrates previous to distillation are presented. Titration data for various synthetic mixtures are given. These data indicated that by adjusting the pH of the solutions to about 2.0 with concentrated ammonium hydroxide, the nitric acid should be converted to ammonium nitrate and thus be retained in the distillation residue. In practice, several Bachmann filtrates were adjusted to various hydrogen ion concentrations and then distilled. In most cases where the pH was 2.0 or above, the ferrous sulfate test was negative and the phenoldisulfonic acid test indicated less than 100 ppm. of nitrate ion in the distillates from these solutions. When the pH was lowered to 1.8 the ferrous sulfate test was still negative but the phenoldisulfonic acid test indicated more than 100 ppm. of nitrate ion in the distillate.

DISCUSSION

In recovering the acetic acid from filtrates obtained in the Bachmann process for the preparation of RDX, it is important for the amount of nitrate ion in the distillate to be very low, preferably less than 100 ppm. Distillation of the diluted filtrates without further treatment does not give a satisfactory distillate. Bachmann titrated the filtrate to the end point of thymolsulfonphthalein (Thymol Blue; pH range, - 1.2 to 2.8) and found that the ferrous sulfate test on the distillate was negative. The difficulty in using this indicator led to the use of the glass electrode.

Potentiometric titrations, with concentrated ammonium hydroxide, were run on synthetic mixtures of nitric acid-acetic acid, hexamethylenetetramine-water, and hexamethylenetetramine dinitrate-30% acetic acid. Similarly solid ammonium nitrate was used to "titrate" 33% acetic acid. The results of all these determinations

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15 MARCH 1961 2.0 not enough free nitric acid was present to appear in the distillate. This was found to work satisfactorily in practice. A series of Bachmann filtrates, adjusted to a pH of 2.0 or above, with concentrated ammonium hydroxide and then distilled, showed less than 100 ppm. of nitrate ion (phenoldisulfonic acid test) in the distillate. Lowering the pH to 1.8 increased the amount of nitrate ion in the distillate to above 100 ppm.

While the results of these studies have been communicated orally to other research groups in this Section, inquiries regarding the details of the procedure cause us to present a complete report at this time. The techniques described herein have been satisfactorily demonstrated at the University of Toronto by repeating the titration of a synthetic acetic acid-nitric acid solution and a typical RDX filtrate diluted to 30% acetic acid.

The advantages of using the glass electrode rather than an indicator include, a) ease of operation, b) relative freedom from observational errors, and c) the possibility of adjusting an entire batch to the desired pH in one operation.

It may be noted here that this titration procedure in its present form is not considered a quantitative method for the determination of nitric acid in acetic acid solutions, but simply a convenient means of adjusting the acidity of Bachmann filtrates to prevent nitrate ion contamination of the acetic acid recovered by distillation.

EXPERIMENTAL

I. Apparatus and Procedure used for the Potentiometric Titrations.

A Beckmann pH meter, Model G, (National Technical Laboratories, Pasadena, California, sold by the Fisher Scientific Company) was used for the potentiometric titration studies reported herein. The titrations were carried out in large beakers using an external shielded glass electrode

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(4919-V3) and a sealed calomel electrode (4919-V5). The potentiometer was first adjusted so that it read a pH of 4.00 when the electrodes were immersed in a buffer mixture of that pH (Harleco, pH reference solution No.4, potassium hydrogen phthalate, $\text{pH} = 4.00 \pm 0.01$ at 25°C ., prepared by the Hartmann-Leddon Company, Philadelphia, Pennsylvania and supplied by the Fisher Scientific Company, Pittsburgh, Pennsylvania).

The electrodes were placed in the solution to be titrated and concentrated ammonium hydroxide was added from a burette, with mechanical stirring. Any changes in the temperature of the solution during the course of the titration were corrected by adjusting the temperature compensator on the potentiometer. As a general practice the readings were taken one to two minutes after adding each milliliter of base. However, as the desired end point was approached this time was increased, since in certain cases it was noticed that as the acidity decreased the readings "drifted" for several minutes after the addition of the ammonium hydroxide before becoming constant. A five minute period of stirring with no change in reading was considered a satisfactory end-point for the end of the titration.

II. Potentiometric Titration of Synthetic Mixtures.

A. Nitric Acid-Acetic Acid.

A sample of 60 ml. glacial acetic acid was diluted to 200 ml. with distilled water to give a 30% acetic acid solution by volume. The pH of this solution was 1.71. On adding 10 ml. of 70% nitric acid the acidity increased to such an extent that the pH could not be read on the instrument used. This mixture was titrated with concentrated ammonium hydroxide (Ammonia Assay = 28%). The data are summarized in Table I., and shown graphically in Figure 1.

From Figure 1 it can be seen that there is a rapid increase in pH per ml. of ammonium hydroxide over the pH range 0.5 to 3.5 with the mid-point of the inflection in the curve lying at a pH of about 2.0. Similar results

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TABLE 1

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POTENTIOMETRIC TITRATION OF SYNTHETIC MIXTURES WITH
CONCENTRATED AMMONIUM HYDROXIDE

Ammonium Hydroxide ml.	HNO ₃ - AcOH pH	HADN- Water pH	HADN- Water pH	HADN- AcOH pH
0.0	-	0.48	0.48	0.21
1.0	-	0.56	0.55	0.30
2.0	-	0.68	0.68	0.42
3.0	0.01	0.82	0.83	0.61
3.5	-	0.95	0.95	0.76
4.0	0.08	1.11	1.10	0.97
4.5	-	1.40	1.36	1.32
5.0	0.12	2.10	2.21	2.00
5.5	-	4.02	4.01	2.38
6.0	0.19	4.50	4.48	2.60
6.5	-	4.70	4.70	2.77
7.0	0.29	4.92	4.92	2.88
8.0	0.39	5.23	5.22	3.07
9.0	0.52	5.60	5.58	3.20
9.5	-	-	5.71	3.28
10.0	0.79	6.00	5.93	3.32
10.5	-	-	6.21	3.38
11.0	1.38	7.00	6.78	3.42
11.5	-	-	7.80	3.48
12.0	2.30	8.30	8.27	3.50
12.5	-	8.4	8.50	-
13.0	2.70	8.66	8.63	3.60
14.0	2.90	-	-	3.68
15.0	3.00	-	-	3.72
16.0	3.20	-	-	3.78
17.0	3.28	-	-	3.87
18.0	3.40	-	-	3.90
19.0	3.44	-	-	-
24.0	3.75	-	-	-
29.0	4.00	-	-	-
32.0	4.10	-	-	-
36.0	4.30	-	-	-
46.0	4.40	-	-	-
50.0	4.72	-	-	-
55.0	4.90	-	-	-
60.0	5.02	-	-	-
65.0	5.20	-	-	-
70.0	5.40	-	-	-
75.0	5.60	-	-	-
80.0	5.90	-	-	-
85.0	6.70	-	-	-
86.0	7.10	-	-	-
87.0	7.50	-	-	-
88.0	7.70	-	-	-

HADN Hexamethylenetetramine dinitrate.

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were obtained over the pH range 6.0 to 7.5 corresponding to the equivalence point of acetic acid. Since in the titration of acid mixtures, the stronger acid should be neutralized first, the first inflection in the curve is due to the neutralization of nitric acid. A calculation of the amount of ammonium hydroxide required to neutralize the nitric acid indicated that 10.66 ml. of ammonium hydroxide is required for the complete neutralization of the nitric acid added. This corresponds to a pH of about 1.2.

$$\text{Ammonium hydroxide ml.} = \frac{10 \times 1.42 \times 0.70 \times 17.03}{63 \times 0.90 \times 0.28} = 10.66 \text{ ml.}$$

B. Hexamethylenetetramine Dinitrate - Water.

To 180 ml. of distilled water 20 g. of hexamethylenetetramine dinitrate was added. The solution was titrated with concentrated ammonium hydroxide. The data for duplicate determinations are given in Table 1 and shown graphically in Figure 2.

The curve obtained by plotting the data from each determination shows two inflections. This indicates in a preliminary way that the "nitric acid" is neutralized in two steps. A calculation shows that 5.1 ml. and 10.2 ml. of concentrated ammonium hydroxide are required for half and complete neutralization of the "nitric acid" respectively. It will be noticed that these points fall very close to the inflections in the curve; the first reflection occurs over a pH range of 1.5 to 4.5 and the second over the range of 6.0 to 8.5. These pH ranges correspond roughly to the ranges determined (Figure 1) for the neutralization of mixtures of nitric and acetic acids.

C. Hexamethylenetetramine Dinitrate - 30% Acetic Acid.

To 180 ml. of 30% acetic acid, 20 g. of hexamethylenetetramine dinitrate was added. The solution was titrated with concentrated ammonium hydroxide. The data are given in Table 1 and shown graphically in Figure 3.

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From Figure 3, it can be seen that the inflection in the curve lies close to the equivalent point for neutralizing half of the "nitric acid". This agrees well with the data obtained when the titration is carried out in water solution (Figure 2). No break in the curve is found for the remaining "nitric acid" when the titration is made in 30% acetic acid. This may indicate that the mononitrate (?) of hexamethylene-tetramine is an acid having a strength equal to or slightly less than acetic acid.

D. The Addition of Ammonium Nitrate to 33% Acetic Acid.

To 30 ml. of glacial acetic acid in a 100 ml. volumetric flask, water was added to bring the solution up to volume. The pH of the solution was determined. A total of 10 g. of ammonium nitrate was added to the acetic acid solution in 1 g. portions; the pH was determined after each addition. The data are shown in Table 2.

TABLE 2
THE EFFECT OF ADDING AMMONIUM NITRATE TO 33% ACETIC ACID

Ammonium Nitrate g.	pH
0	1.69
1	1.68
2	1.68
3	1.67
4	1.66
5	1.65
6	1.64
7	1.63
8	1.63
9	1.62
10	1.62

The data in Table 2 show that the presence of 10% ammonium nitrate in the titration mixture would have little or no effect on the titration.

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E. Summary.

The results may be summarized briefly as follows -

1. Nitric acid may be titrated in acetic acid solution with concentrated ammonium hydroxide to become essentially ammonium nitrate at a pH of 1.0 to 3.5.
2. In water solution hexamethylenetetramine dinitrate appears to behave as a dibasic acid, one half of the acid being neutralized at a pH of 2.0 to 4.0, the remainder being neutralized at a pH of 6.5 to 8.0.
3. In acetic acid solution, one half of the acid in hexamethylenetetramine dinitrate is neutralized at a pH of 1.0 to 2.5, the remainder shows no break in the curve at its equivalence point
4. Ammonium nitrate, up to 10%, has little or no effect on the pH of 33% acetic acid.
5. The above results indicate that the use of the glass electrode is satisfactory for measuring the end point of the neutralization of nitric acid in 30% acetic acid solution.

III. Potentiometric Titration of Bachmann Filtrates.

A. Complete Titration Curve for a Bachmann Filtrate.

A 100 ml. sample (104.5 g.) of filtrate, from a Bachmann RDX preparation run according to directions V-61, was titrated with concentrated ammonium hydroxide. The results are given in Table 3 and shown graphically in Figure 4. As in the curves previously presented in connection with the study of synthetic mixtures, the curve for a Bachmann filtrate shows an inflection over the pH range 1.0-3.0. The mid-point of the inflection lies very close to a pH of 2.0. The calculated equivalence point based on a nitron nitrate determination lies at a pH of 2.8. This is in line with the facts that the nitron method determines total

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TABLE 3

COMPLETE POTENTIOMETRIC TITRATION OF A BACHMANN FILTRATE
WITH CONCENTRATED AMMONIUM HYDROXIDE

The aliquot used for titration was taken from PSC Run No. 3, made according to Bachmann's Directions V-51. Volume - 100 ml. (104.5 g.)

Ammonium Hydroxide ml.	pH	Ammonium Hydroxide ml.	pH	Ammonium Hydroxide ml.	pH	Ammonium Hydroxide ml.	pH
0.0	0.36	10.0	3.89	25.0	4.86	40.0	5.95
1.0	0.78	11.0	3.98	26.0	4.91	41.0	6.10
1.5	1.40	12.0	4.02	27.0	4.98	42.0	6.31
2.0	2.32	13.0	4.10	28.0	5.01	42.5	6.47
2.5	2.63	14.0	4.18	29.0	5.08	43.0	6.67
3.0	2.86	15.0	4.26	30.0	5.13	43.5	6.92
3.5	2.99	16.0	4.31	31.0	5.19	44.0	7.32
4.0	3.12	17.0	4.39	32.0	5.28	44.5	7.61
4.5	3.21	18.0	4.47	33.0	5.31	45.0	7.82
5.0	3.31	19.0	4.50	34.0	5.40	46.0	8.09
5.5	3.40	20.0	4.54	35.0	5.48	47.0	8.28
6.0	3.48	21.0	4.61	36.0	5.52	48.0	8.39
7.0	3.58	22.0	4.68	37.0	5.61	49.0	8.51
8.0	3.69	23.0	4.73	38.0	5.70	50.0	8.61
9.0	3.79	24.0	4.80	39.0	5.81	-	-

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nitrate including both of the nitrate groups in hexamethylenetetramine dinitrate whereas only one is neutralized at this pH in acetic acid solution. The second inflection in the curve occurs over the pH range 6.2 to 8.0 with the mid-point lying close to pH 7.3. This corresponds very closely to the acid-base equivalence point of the solution as determined by ordinary titration with sodium hydroxide using phenolphthalein as the indicator.

B. The Use of pH Measurements in the Recovery of Acetic Acid from Bachmann Filtrates.

Bachmann filtrates were adjusted to various hydrogen ion concentrations, then distilled, and nitrate tests were made on the distillates. The titration data and the results of the nitrate tests are given in Table 4. Typical titration curves are shown in Figure 5. The percent acetic acid recovered in the different runs has been reported before (Report Serial No. 182, from The Pennsylvania State College).

C. Summary.

From an examination of the data presented in Tables 3 and 4 and shown in Figures 4 and 5 it may be seen that -

1. The practical results check the predictions made on the basis of the results obtained with synthetic mixtures.
2. The pH of the original diluted filtrates as determined on aliquots from the same or different runs agree surprisingly well. The results from Runs CU-3-PSC-4 and 5 where less nitric acid was used are shifted in the right direction.
3. The order of magnitude of the volume of ammonium hydroxide required to adjust the filtrates to a given pH, when the amount of filtrate used is considered, is the same in most cases.

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4. It can be seen that in the titration of a series of filtrates from different runs the inflection in the curves appears over essentially the same pH range in all cases, and very closely approximates that determined using synthetic mixtures (1.0 to 2.5).
5. With the exception of two of the early runs, when the pH of the filtrate was 2.0 or above the ferrous sulfate test in the distillates from the filtrates, was negative and the phenoldisulfonic acid test indicated definitely less than 100 ppm of nitrate ion. On adjusting the pH of a filtrate to 1.8 more nitrate ion was found in the distillate.

RECOMMENDATIONS

On the basis of the data presented it is feasible to use the glass electrode for titrating Bachmann filtrates for the purpose of preventing nitrate ion contamination of the distillate, in the process for the recovery of acetic acid. It is recommended that the pH of the filtrates be adjusted to not less than 2.0.

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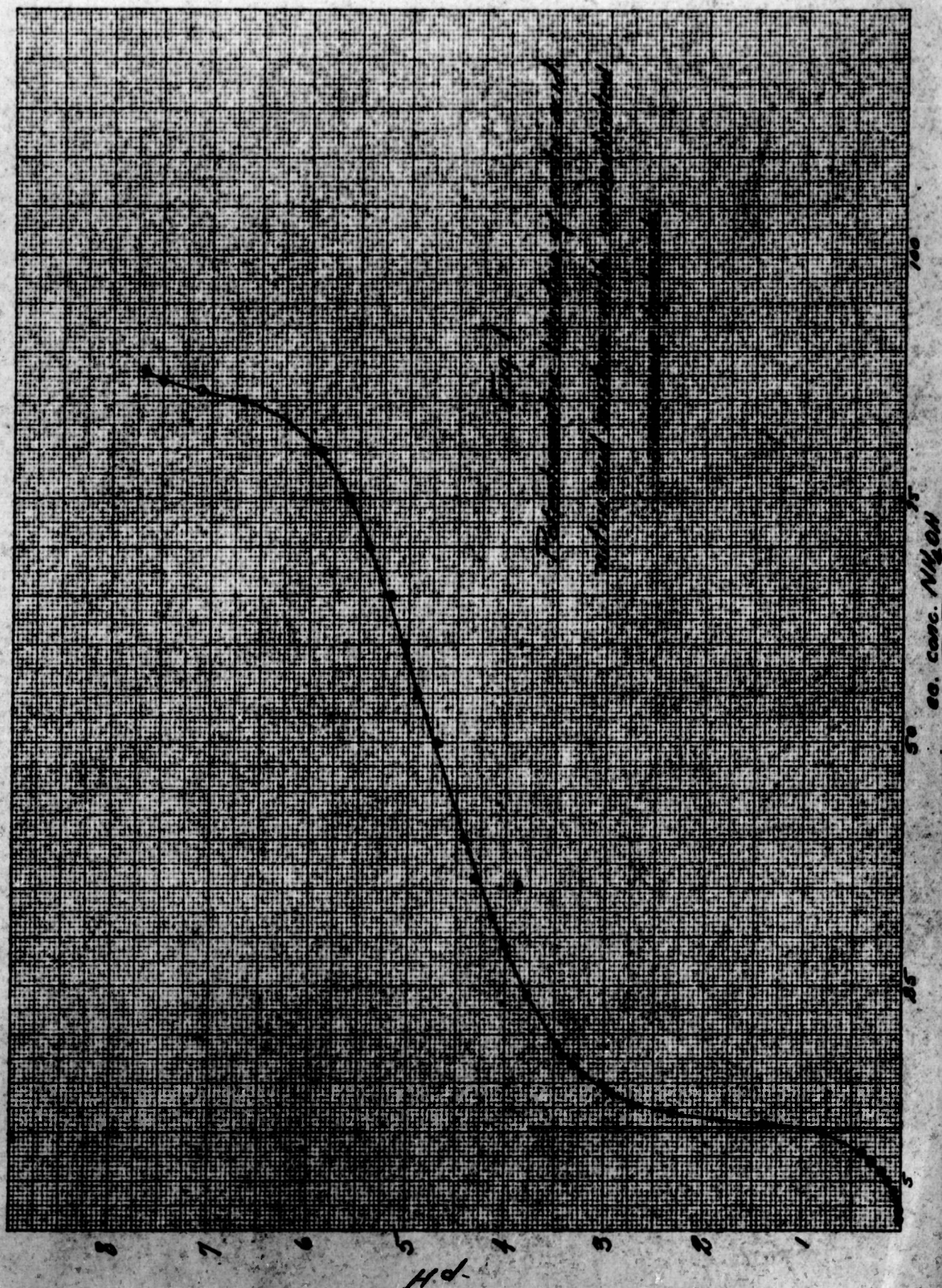
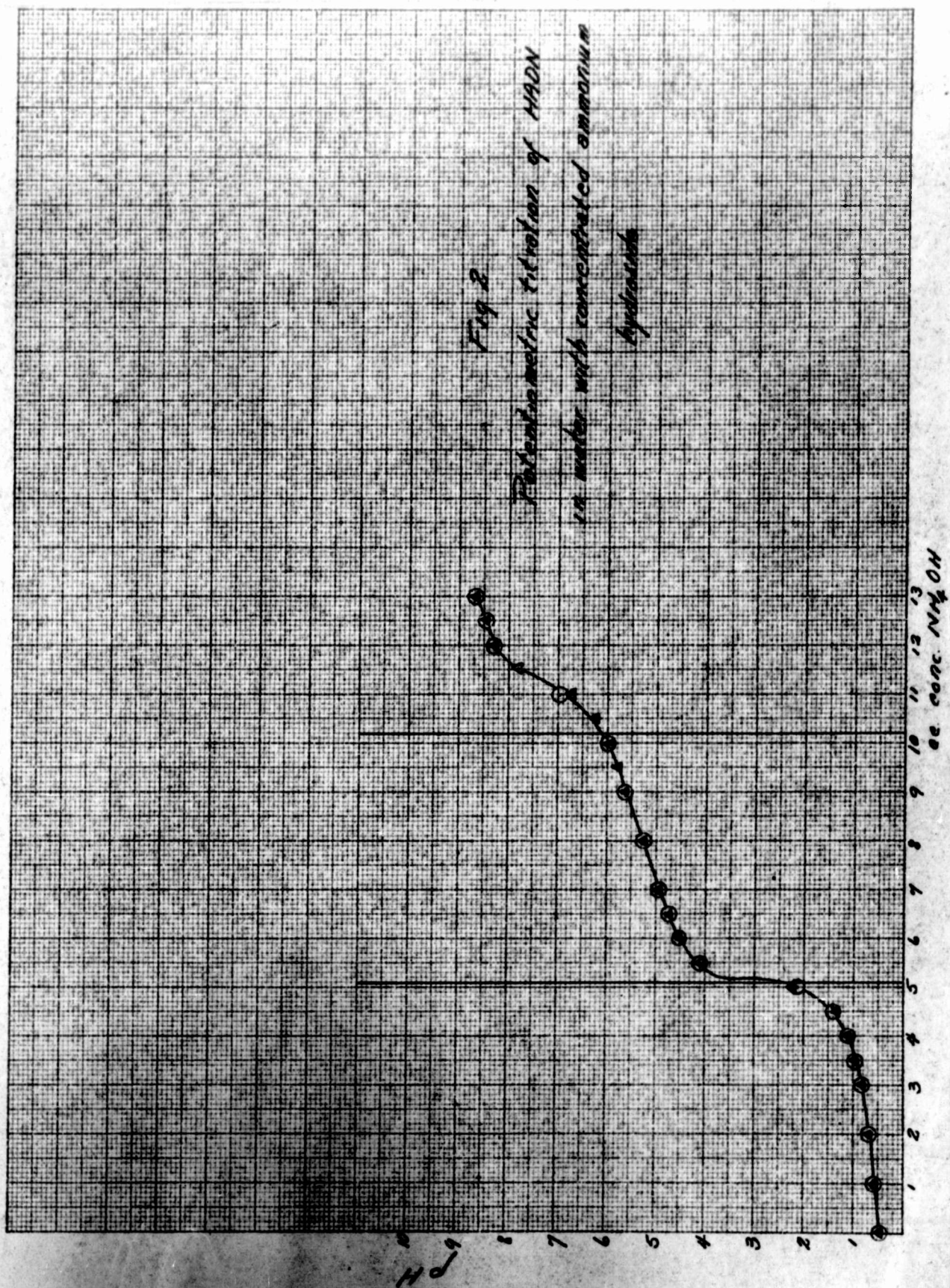


Fig. 1
 Reaction of NH_4OH with H_2O



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Fig 3
Potentiometric titration of
HNO₃ in 30% acetic acid with
concentrated ammonium hydroxide

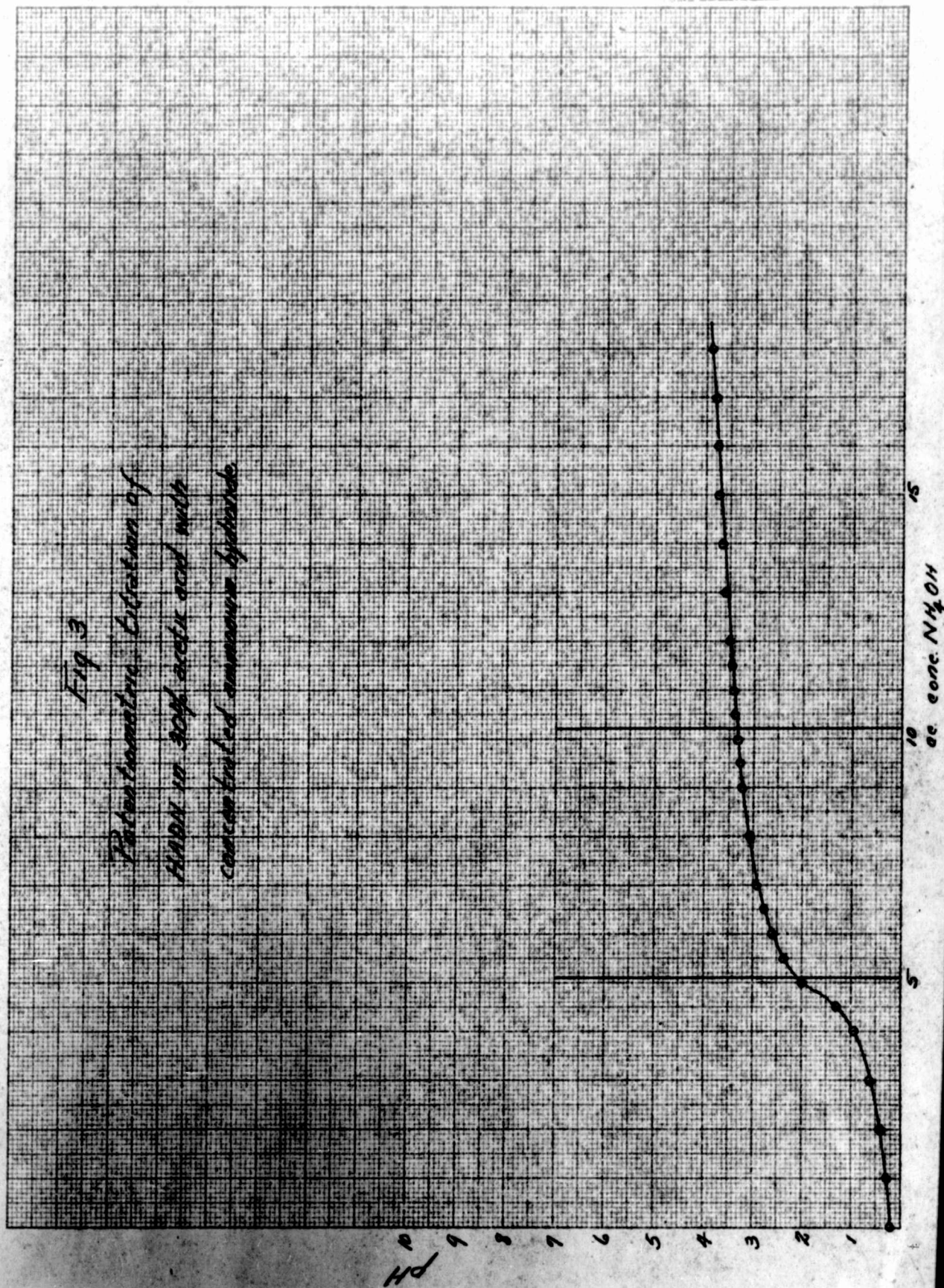


TABLE 4. POTENTIOMETRIC TITRATION OF 506 FILTRATES

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TABLE 4 - CONTINUED

	4	4	7	6	6	8	8	8	8	8	9	10	11	PRC-4 CU-3	PRC-5 CU-3
15.0	2.91	2.91			2.90						1.05	1.23	1.31		
15.5		2.94													
16.0					2.40						1.18	1.38	1.43		
16.25	3.00														
16.80		3.00													
17.0					2.50						1.31	1.53	1.61		
18.0					2.59						1.50	1.71	1.80		
19.0					2.66						1.70	1.90	1.97		
19.4													2.00		
19.5											1.78				
19.8												2.00			
20.0					2.70						1.86				
20.5											1.92				
21.0					2.78										
21.5											2.00				
22.0					2.81										
23.0					2.88										
24.0					2.91										
25.0					2.96										
26.0					2.99										
26.5					3.00										
Final pH before test	3.00	3.00	3.00	3.00	3.00	3.00	2.50	2.25	2.00	1.80	2.00	2.00	2.00	2.00	2.00
NO ₃ ⁻ TEST: FeSO ₄	Pos.	Neg.	Pos.	Neg.	Neg.	Neg.	Neg.		Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.
PHENOL RED ONE DROP NO ₃ ⁻ TEST. PPM	100- 150	1.25	50- 100	<25	<25					50- 100	<100				

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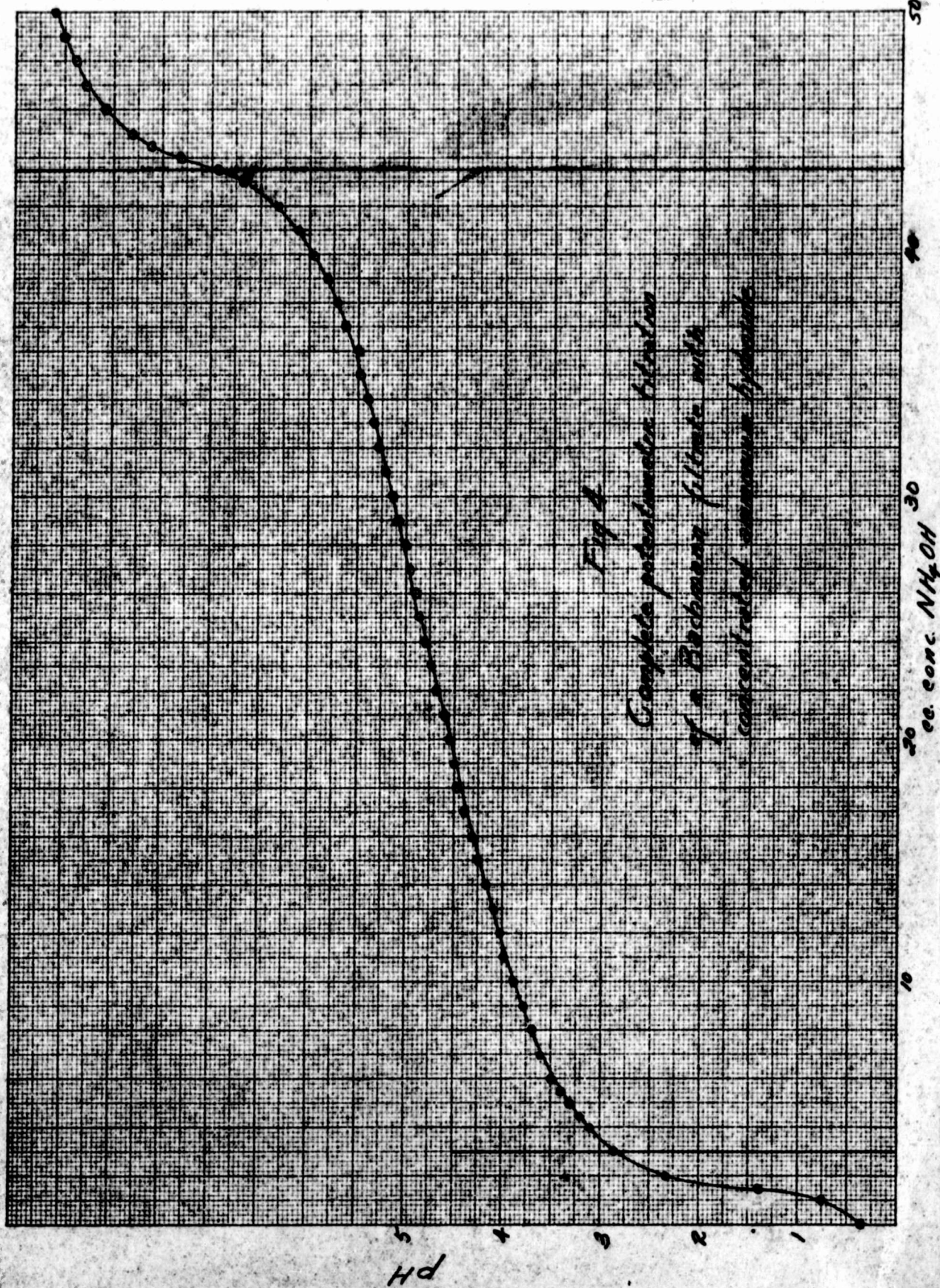
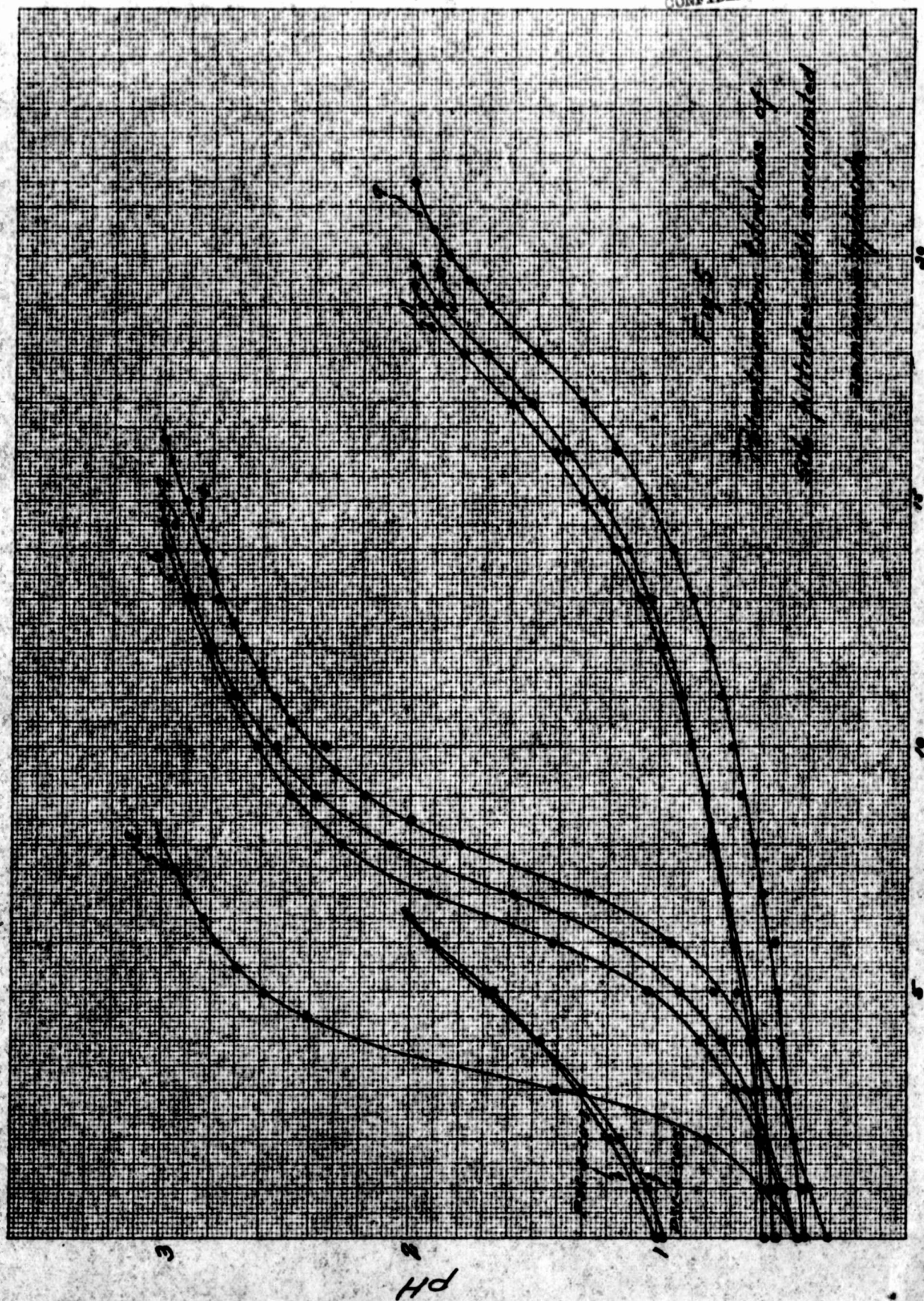


Fig. 4
Complete potentiometric titration
of a Beckmann titration with
concentrated ammonium hydroxide

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