

PREPARATION OF BIS(2-p-AMINOPHENOXYETHYL) ETHER
AND RELATED COMPOUNDS

THESIS

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CHAPTER I
INTRODUCTION

This research problem was carried out for the purpose of preparing bis(2-p-aminophenoxyethyl) ether through the necessary intermediates and then coupling the primary amine with dimethylaniline to form an expected azo dye for possible use as an indicator. Also to be investigated was the ability of the primary amine to complex various metal ions. Three of the compounds synthesized are new compounds; therefore, the characterization of their structure is given except in the case of the azo dye. The reason for not giving the characterization of the structure of the dye, bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl) ether, is that diazonium salts react with tertiary amines in alkaline solutions, causing substitution in the position para to the amino group.¹ Therefore, the reaction can proceed to yield only

¹Carl R. Noller, Textbook of Organic Chemistry, p. 378.

the expected dye. Some physical constants of the compounds prepared have been determined during the course of investigation.

The interest in investigating the new amine exists because of the ability of amines in general to complex metal ions and also their ability to form azo dyes which frequently can be used as pH indicators. Both of these properties are of considerable importance. Some amines, when in the presence of certain metal ions, have a tendency to form

complex molecules causing a definite color change. This ability can be used in application to spot tests which are fast becoming an important phase of qualitative analysis. Also most primary amines have the necessary characteristic to react with nitrous acid and form the corresponding diazonium salt which will react with phenols and tertiary amines in weakly acidic, neutral, or alkaline solution, causing substitution in the position para to the hydroxyl or amino group with the production of highly colored azo compounds. Azo dyes have been of considerable importance since the middle of the nineteenth century. Some of these azo dyes have the ability to change colors at different hydrogen ion concentrations; therefore, they can be used as pH indicators.

Riley F. Mogford² has shown that some ether bonds are cleaved

²Riley F. Mogford, "Synthesis and Properties of Ether Derivatives of 2-(2-Ethoxyethoxy)ethanol and Diethylene Glycol," Masters Thesis, Southwest Texas State Teachers College, San Marcos, Texas, 1958, p. 30.

by hydrochloric acid in high concentration at high temperatures; therefore, this situation was avoided throughout the experimentation.

CHAPTER II

EXPERIMENTATION

A. 2,2'-Dibromodiethyl Ether.

2,2'-Dibromodiethyl ether was prepared by the method described by M. H. Palomaa and Arno Kenetti.¹ The physical constants of this

¹M. H. Palomaa and Arno Kenetti, Berichte Der deutschen Chemischen Gesellschaft, Vol. 64B (1931), p. 797.

compound were obtained from P. A. McCusker and J. W. Kroeger.²

²P. A. McCusker and J. W. Kroeger, Journal of the American Chemical Society, Vol. 59 (1937), p. 213.

1. Preparation.

A 162.4-gram (.6 mole) sample of phosphorus tribromide was placed in a 500-ml. three-necked flask which was equipped with a dropping funnel, a stirrer, and a reflux condenser fitted with a calcium chloride drying tube. The three-necked flask was submerged in an ice bath and the temperature of the reaction mixture maintained below 5°C during the addition of the phosphorus tribromide. A mixture of 79.6 grams (.75 mole) of diethylene glycol and 26.9 grams (.34 mole) of pyridine was added dropwise through the funnel. The addition of the diethylene glycol and pyridine mixture required two hours, during

which time the reaction mixture changed from white to pale yellowish-orange. If the reaction mixture is allowed to proceed too fast, hydrogen bromide fumes are forced into the reflux condenser and the addition of the diethylene glycol and pyridine mixture must be stopped until the fumes have redissolved in the reaction medium. After all the diethylene glycol and pyridine mixture had been added, the ice bath was removed and the reaction was allowed to proceed one hour. The mixture was poured into 200 ml. of cold water and the crude product settled as an oil. The aqueous solution was separated, thoroughly extracted with diethyl ether, and the ether extracts were added to the crude product. The solution was washed with two 20 ml. portions each of ten per cent sulfuric acid, ten per cent sodium carbonate, and finally, water. The diethyl ether solution of 2,2'-dibromodiethyl ether was dried over calcium chloride for 24 hours. After the solution was completely dry, the solvent was removed and the crude product distilled under diminished pressure. The product was collected at 112°-113°C at 29 mm pressure. The reported boiling point is 110°C at 26 mm pressure. The yield obtained was 45.5 per cent of the theoretical.

2,2'-Dibromodiethyl ether can be synthesized by an alternate method,³ which was used in an attempt to increase the yield. A mixture

³R. W. Bost and Meldrum B. Winstead, Jr., Journal of the American Chemical Society, Vol. 74 (1952), p. 1822.

of 79.6 grams (.75 mole) of diethylene glycol and 26.9 grams (.34 mole)

of pyridine was placed in a 500-ml. three-necked flask which was equipped with a dropping funnel, a stirrer, and a reflux condenser fitted with a calcium chloride drying tube. The solution was warmed slightly and 162.4 grams (.6 mole) of phosphorus tribromide was added slowly through the dropping funnel. The addition of the phosphorus tribromide required two hours, during which time the reaction mixture became white in color. If the reaction is allowed to proceed too fast, hydrogen bromide fumes are evolved and the reaction mixture must be cooled until the fumes have redissolved in the reaction medium. The reaction was allowed to proceed thirty minutes after all the phosphorous tribromide had been added. The 2,2'-dibromodiethyl ether was separated as previously described. There was no appreciable increase in yield.

2. Physical Data

Boiling Point-----110°C at 26 mm.
 Density-----1.8222 $D_{4}^{27^{\circ}}$
 Index of Refraction-----1.5131 $n_{D}^{27^{\circ}}$
 Color-----Colorless

B. Preparation of Sodium p-Nitrophenolate.

Sodium p-nitrophenolate was prepared by the method described by James Cason and Henry Rapoport⁴ with a few modifications.

⁴James Cason and Henry Rapoport, Laboratory Text in Organic Chemistry, p.131.

A mixture of 15 grams of p-nitrophenolate, 150 ml. of water, 20 ml. of 12N sodium hydroxide, and 20 grams of sodium chloride was placed in a 500-ml. Erlenmeyer flask. The solution was boiled over an open flame until crystallization of sodium p-nitrophenolate from the boiling solution had begun. Then the flask was placed in a water bath and cooled to room temperature. The crystals were filtered through a Büchner funnel and washed with 60 ml. of a solution containing two parts saturated sodium chloride solution and one part water. The purification of the sodium p-nitrophenolate was effected by recrystallization from 125 ml. of 18 per cent sodium chloride solution. The crystals were washed with a solution containing two parts saturated aqueous sodium chloride with one part water. The solid was given a final rinse with 10 ml. of 95 per cent ethyl alcohol and 25 ml. of diethyl ether. The yield obtained was 96 per cent of the theoretical. The crystals of sodium p-nitrophenolate are long golden-yellow needle crystals.

C. Bis(2-p-nitrophenoxyethyl) Ether.

1. Preparation.

A mixture of 46.4 grams (.2 mole) of 2,2'-dibromodiethyl ether, 72.5 grams (.45 mole) of sodium p-nitrophenolate, and 300 ml. of dry methyl alcohol was placed in a three-necked flask fitted with a reflux condenser which was equipped with a calcium chloride drying tube. The solution was refluxed for twenty-four hours. Crystals of bis(2-p-

nitrophenoxyethyl) ether began to appear after approximately twelve hours of the reaction time had lapsed. After the reaction had gone twenty-four hours, the mixture was cooled to room temperature and filtered through a Büchner funnel. The yellow color was removed by repeated washings with small portions of methyl alcohol. The crystals were given a final rinse with water then were recrystallized from ethyl alcohol. The yield obtained was 45.9 per cent of the theoretical.

The reaction time was increased from twenty-four to forty-eight hours with no appreciable increase in yield.

2. Physical Data.

Melting Point-----156^oC

Crystals-----Small white needles

Sodium Fusion-----Negative Bromine

3. Characterization of the Structure of Bis(2-p-nitrophenoxyethyl)

Ether.

Bis(2-p-nitrophenoxyethyl) ether was reduced to the corresponding amine and confirmed by potentiometric titration. The characterization will be discussed under bis(2-p-aminophenoxyethyl) ether.

D. Bis(2-p-aminophenoxyethyl) Ether.

1. Preparation.

A mixture of 13.9 grams (.04 mole) of bis(2-p-nitrophenoxyethyl)

ether and 75 ml. of ethyl alcohol was placed into a 500-ml. three-necked flask fitted with a stirrer and a reflux condenser. The mixture was brought to reflux temperature and 13.4 grams (.24 mole) of iron filings, 50 grams (.48 mole) of concentrated hydrochloric acid, and 80 grams (8.0 mole) of water were added through the reflux condenser, each in approximately 10 per cent quantities and in the order listed at 10 minute intervals. After all the constituents had been added the reaction was allowed to continue for thirty minutes. The reaction mixture was filtered while hot and the filtrate was returned to the cleaned three-necked flask. An additional 5 grams of iron filings and 10 grams of concentrated hydrochloric acid were added to the reaction mixture, which was raised again to reflux temperature. The reaction was allowed to continue one hour, after which time the reaction mixture was filtered while hot, cooled in an ice bath, and kept cool while the solution was made basic with sodium hydroxide. The basic solution was extracted three successive times with 100-ml. portions of benzene. The benzene extract was treated three successive times with 30-ml. portions of 6N hydrochloric acid. The cooled acid extract was made basic with sodium hydroxide. Diethyl ether was used to remove all of the amine from the basic solution. The solvent was removed by distillation under diminished pressure with crystals of bis(2-p-aminophenoxyethyl) ether being formed during the process. The solid was recrystallized from diethyl ether.

Bis(2-p-aminophenoxyethyl) ether can be synthesized by an alternate

method⁵ with a few modifications. A mixture of 15 grams of bis(2-p-

⁵S. M. McElvain, The Characterization of Organic Compounds,
p. 144-145.

nitrophenoxyethyl) ether, 60 grams of iron filings, 35 ml. of 1N ammonium chloride, and 120 ml. of ethyl alcohol was placed in a 500-ml. three-necked flask equipped with a stirrer and a reflux condenser. The material was heated gently on a steam bath until the initial vigorous reaction subsided and then the mixture was refluxed for one hour, after which time 100 ml. of the solvent was removed by distillation. The concentrated residue was cooled to room temperature. The bis(2-p-aminophenoxyethyl) ether was removed from the residue by extraction with benzene and then purified as previously described.

2. Physical Data.

Melting Point ----- 59°C
Crystals ----- Small clear needles

3. Derivatives.

Hydrochloride ----- 227-228°C
Benzoylate ----- 221-222°C
Sulfonamide ----- 133-134°C

4. Characterization of the Structure of Bis(2-p-aminophenoxyethyl) Ether.

The molecular weight of bis(2-p-aminophenoxyethyl) ether was confirmed by potentiometric titration.⁶ A diamine, being basic in a water

⁶Farrington Daniels and Robert A. Alberty, Physical Chemistry, p. 449-451.

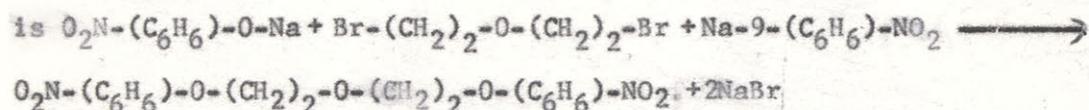
solution, will react with hydrochloric acid to form the hydrochloride of the diamine. Since two molecules of hydrochloric acid will react with one molecule of the diamine, it is possible to titrate a known quantity of the diamine with a standard hydrochloric acid solution and then to calculate the molecular weight from the results. The end point of the titration was determined by a pH meter. The pH will change very slowly during the addition of the hydrochloric acid until almost enough of the acid has been added to react with all of the amine. After this point has been reached, any additional acid will cause the pH to drop rapidly. The point at which the pH takes the sharpest drop per volume of acid added is the end point as can be seen in table I and graphs I and II.

A 0.1411-gram portion of bis(2-p-aminophenoxyethyl) ether and 10 ml. of water were placed in a 50-ml. beaker. To this mixture 0.1003N hydrochloric acid was added from a 5-ml. buret. The hydrochloric acid was added in 1-ml. portions until 9 ml. had been added; then the measured hydrochloric acid was added in 0.05-ml. portions. The pH of the mixture was measured one minute after each addition of the acid.

A 0.1411-gram sample of the amine required 9.74 ml. of the acid to reach the equivalence point, which was determined by reference to Graph II. This graph was prepared by plotting change in pH divided by change in volume against change in volume. The molecular weight of bis(2-p-aminophenoxyethyl) ether is 285.18. The molecular weight found by this procedure was 288.0, an error of 0.97 per cent in molecular weight.

The structure of bis(2-p-aminophenoxyethyl) ether is $\text{H}_2\text{N}-(\text{C}_6\text{H}_6)-\text{O}-(\text{CH}_2)_2-\text{O}-(\text{CH}_2)_2-\text{O}-(\text{C}_6\text{H}_6)-\text{NH}_2$. The intermediate 2,2'-dibromodiethyl ether, $\text{Br}-(\text{CH}_2)_2-\text{O}-(\text{CH}_2)_2-\text{Br}$, was synthesized and verified by physical constants and the intermediate sodium p-nitrophenolate, $\text{Na}-\text{O}-(\text{C}_6\text{H}_6)-\text{NO}_2$, was prepared from pure p-nitrophenol. The two compounds, 2,2'-dibromodiethyl ether and sodium p-nitrophenolate, were used to prepare bis(2-p-nitrophenoxyethyl) ether through the familiar Williamson synthesis of ethers, which involves reacting the sodium alkoxide with the alkyl halide in a typical displacement type reaction.⁷ The reaction

⁷ James Cason, Essential Principles of Organic Chemistry, p. 97-99.



The only other feasible intermediate compound would contain bromine from incomplete displacement. Since a sodium fusion gave a negative bromine test, it was concluded that the product was synthesized.

Bis(2-p-aminophenoxyethyl) ether was prepared from the reduction of the

recrystallized nitro compound and the molecular weight confirmed by potentiometric titration as previously described.

5. Complexation Studies of Bis(2-p-aminophenoxyethyl) Ether
With Various Metal Ions.

The method used to test for bis(2-p-aminophenoxyethyl) ethers ability to complex various metal ions was adapted from the method suggested by Philip W. West.⁸ The metal ions chosen were the ions

⁸Philip W. West, Journal of Chemical Education, Vol. 18 (1941), p. 528.

known to complex with ammonia and ammonia derivatives, which include the amines.

A solution of one per cent bis(2-p-aminophenoxyethyl) ether in ethyl alcohol and an approximate 0.1M water solution of each of the chlorides of cadmium, chromium, cobalt, copper, mercury, nickel, palladium, platinum, silver, and zinc were prepared. Each metal ion was tested for complexation by placing two drops of the respective metal ion solution in each of six depressions of a spot plate. Two drops of the alcoholic amine solution were added to each of three of the depressions with the remaining three being designated as blanks. These blanks were placed in a one to one correspondence with the depressions containing the amine. Two drops of concentrated ammonium hydroxide were added to each of the first pair, the same volume of 3N sodium

hydroxide was added to each of the second pair, and finally, two drops each of both ammonium hydroxide and sodium hydroxide were added to each of the third pair. Any color differences between the blanks and the corresponding depression containing the amine were noted for possible colored complex formation and the results are recorded in Table II. Copper, mercury, palladium, and platinum ions were found to form colored complexes with bis(2-p-aminophenoxyethyl) ether.

E. Bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl) Ether.

1. Preparation

Bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl) ether was prepared by the diazotization of bis(2-p-aminophenoxyethyl) ether and the coupling of the resulting diazo compound (in alkaline solution) with dimethyl aniline to form an azo dye. The method used was adapted from the general procedure suggested by S. M. McElvain.⁹

⁹McElvain, op. cit., p. 143.

A 0.28-gram (.001 mole) portion of bis(2-p-aminophenoxyethyl) ether in a large six-inch test tube was dissolved by the addition of 14 ml. of 15 per cent hydrochloric acid (.006 mole). The acid solution was cooled to 0-5°C in an ice bath and then 0.28 gram (.004 mole) of sodium nitrite dissolved in 3 ml. of cold water was added. The reaction mixture was stirred and maintained below 5°C for ten minutes, after

which time a test with starch-potassium iodide paper confirmed the presence of nitrous acid. The reaction mixture at this point contained the diazonium salt of bis(2-p-aminophenoxyethyl) ether, which was dissolved in the light-green solution. The diazonium salt solution was poured with stirring into a cool (5-10°C) solution of 1 ml. (.008 mole) of dimethylaniline in 8 ml. of 5 per cent sodium hydroxide (.010 mole). The mixture became yellowish-orange in color and the resulting bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl) ether precipitated. The mixture was filtered under light suction through a small Hirsch funnel fitted with a small plate and then rinsed with a saturated water solution of sodium chloride. No attempt was made to purify nor to characterize the product because primary amines have the property of reacting with nitrous acid to form the corresponding diazonium salt which will react with tertiary amines in alkaline solution, causing substitution in the position para to the amino group. Therefore, the only feasible compound would be bis(2-p-(p-dimethylamino-phenylazo)phenoxyethyl) ether.

2. The Use of Bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl)

Ether as a pH Indicator.

The azo dye thus formed was tested to see if any color change would result from adding acid or base to its water solution. A small amount of the azo dye (approximately the size of a match head) was placed in a test tube half-filled with water. Dilute hydrochloric acid

was added to the golden-brown solution, whereupon the solution became light-yellow in color. Sodium hydroxide was then added and the solution returned to a golden-brown. This suggested that the azo dye could possibly be used as a pH indicator.

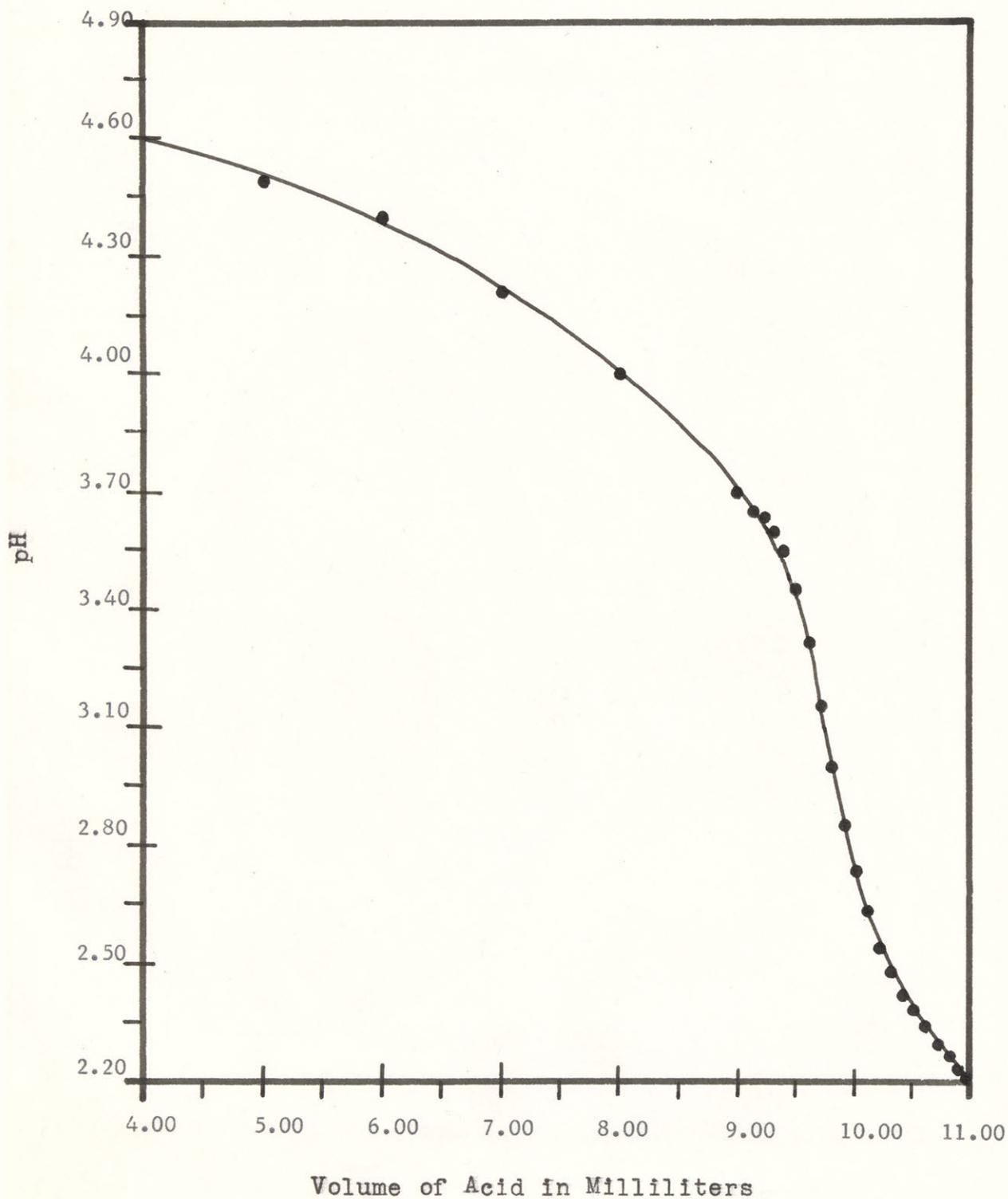
A pH meter was set up, adjusted to a pH of seven with buffer solution, and the electrodes placed in a 100-ml. beaker containing a small amount of bis(2-*p*-(*p*-dimethylaminophenylazo)phenoxyethyl) ether dissolved in 50 ml. of water. The pH was changed gradually by the addition in turn of small portions of approximately 0.1N hydrochloric acid and 0.1N sodium hydroxide solutions. The azo dye was found to act as an indicator and the pH range of the color change was determined. This range was found by the correlation of color with pH and the average was from 3.0 to 4.1, a range of 1.1 pH units. These results were obtained by continuously changing the pH past the indicator's color change from both the acidic and basic sides until an average was determined.

TABLE I
 POTENTIOMETRIC TITRATION OF BIS(2-p-AMINOPHENOXYETHYL) ETHER

Milliliters of acid	pH	Change in pH	Milliliters of acid	pH	Change in pH
1.00	4.75	2.04	9.80	3.00	0.08
2.00	4.74	0.01	9.85	2.92	0.08
3.00	4.66	0.08	9.90	2.85	0.07
4.00	4.60	0.06	9.95	2.79	0.06
5.00	4.48	0.12	10.00	2.74	0.05
6.00	4.40	0.08	10.05	2.69	0.05
7.00	4.20	0.20	10.10	2.64	0.05
8.00	4.00	0.20	10.15	2.59	0.05
9.00	3.70	0.30	10.20	2.54	0.05
9.10	3.65	0.05	10.25	2.50	0.04
9.20	3.65	0.00	10.30	2.48	0.02
9.30	3.60	0.05	10.35	2.45	0.03
9.40	3.55	0.05	10.40	2.42	0.03
9.45	3.50	0.05	10.45	2.40	0.02
9.50	3.45	0.05	10.50	2.38	0.02
9.55	3.40	0.05	10.60.	2.34	0.04
9.60	3.32	0.08	10.70	2.30	0.04
9.65	3.23	0.09	10.80	2.27	0.03
9.70	3.15	0.08	10.90	2.23	0.04
9.75	3.08	0.07	11.00	2.20	0.03

GRAPH I

POTENTIOMETRIC TITRATION
(pH vs. Volume of Acid)



GRAPH II

POTENTIOMETRIC TITRATION
(pH Differential)

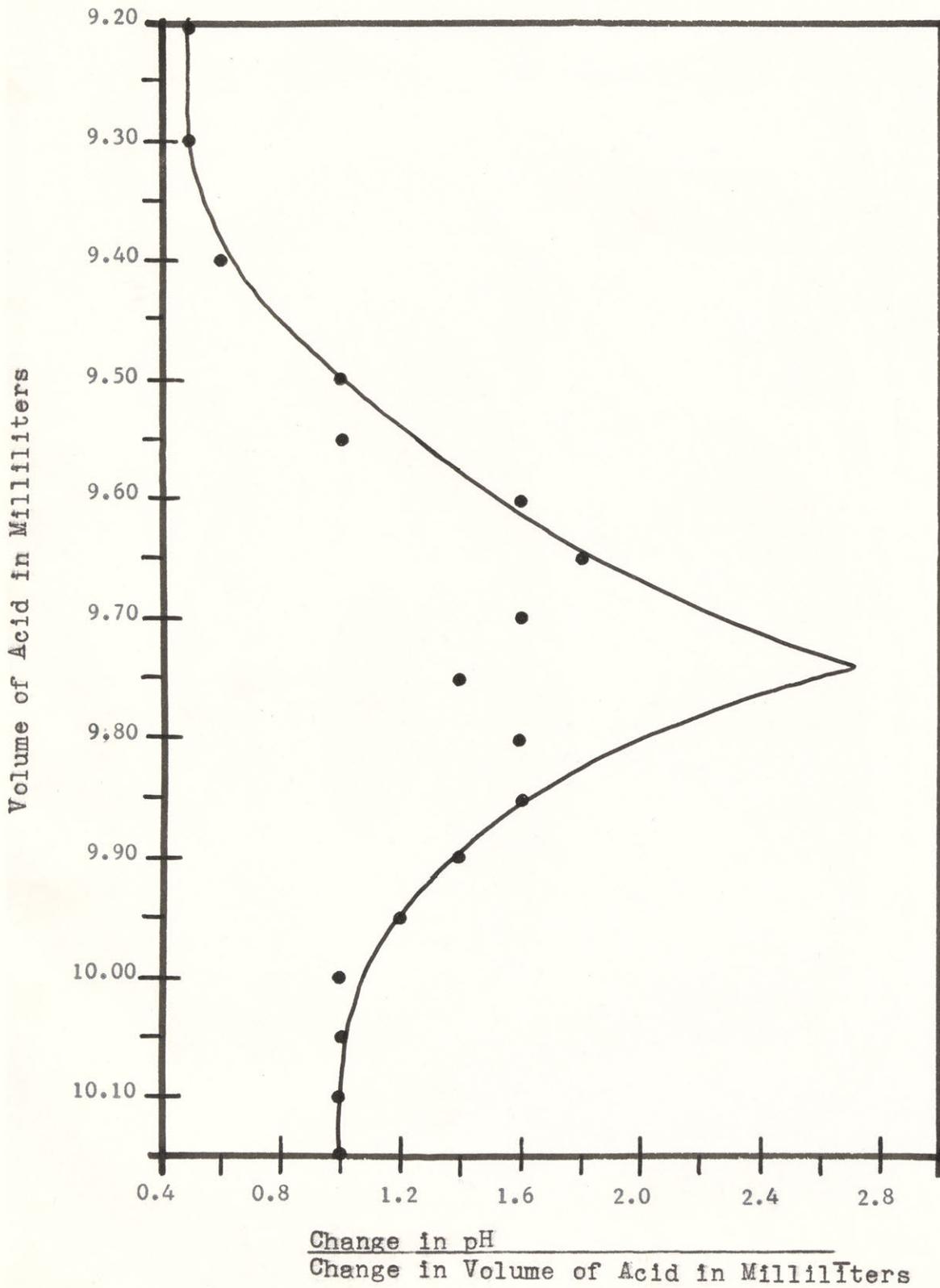


TABLE II

METAL IONS COMPLEXED WITH BIS(2-p-AMINOPHENOXYETHYL) ETHER

Metal ion and Electron Charge	Colored Complex*	Color Change
Cadmium ⁺⁺	-	No color change
Chromium ⁺⁺⁺	-	No color change
Cobalt ⁺⁺	-	No color change
Copper ⁺⁺	+	Brownish-black instead of blue
Mercury ⁺⁺	+	Brown instead of Yellow
Nickel ⁺⁺	-	No color change
Palladium ⁺⁺	+	Dark red instead of orange
Platinum ⁺⁺⁺⁺	+	Brownish-black in- stead of yellow
Silver ⁺	-	No color change
Zinc ⁺⁺	-	No color change

*(+) indicates a colored complex and (-) indicates no colored complex was formed.

CHAPTER III

SUMMARY

This paper was devoted to reporting the preparation and study of bis(2-p-aminophenoxyethyl) ether and related compounds. Bis(2-p-nitrophenoxyethyl) ether, bis(2-p-aminophenoxyethyl) ether, and bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl) ether are new compounds along with the hydrochloride, benzoylate, and sulfonamide of bis(2-p-aminophenoxyethyl) ether. A method for the synthesis of each of these compounds, except the derivatives of bis(2-p-aminophenoxyethyl) ether, and some of their physical constants have been given. 2,2'-Dibromodiethyl ether, sodium p-nitrophenolate, and bis(2-p-nitrophenoxyethyl) ether were prepared and used as necessary intermediates in the synthesis of bis(2-p-aminophenoxyethyl) ether.

Bis(2-p-aminophenoxyethyl) ether was synthesized to study its ability to complex various metal ions and to prepare bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl) ether. The compound bis(2-p-aminophenoxyethyl) ether, was found to complex copper, mercury, palladium, and platinum, a fact which may become useful in spot tests for these metal ions or for their quantitative determination by spectrophotometry. The compound, bis(2-p-(p-dimethylaminophenylazo)phenoxyethyl) ether, was found to contain the characteristics of an azo dye and it also was found that the dye is an acid-base indicator, changing color in the pH range of 3.0 to 4.1. It should be noted that this indicator has

a definite color change, light-yellow to a golden-brown, within a pH range of only 1.1. Nearly every pH indicator in use today changes color over a pH range of 2 units. This indicator may possibly be useful in titrating weak bases with strong acids.

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