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GENERAL ANILINE & FILM CORPORATION

RESEARCH DEPARTMENT

CENTRAL RESEARCH LABORATORY

Preparation of New Arsinic Acids and Esters

Chiddix, M.E. & E.O. Leonard

1965.

This protective marking is cancelled one year after date of report.

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General Aniline & Film Corporation
Central Research Laboratory
Easton, Pennsylvania

June 10, 1965

FINAL REPORT

Prepared For: U.S. Army Biological Laboratories
Fort Detrick
Frederick, Maryland

Contract No.: DA 18-064-AMC-135 (A)

Subject: Preparation of New Arginic Acids and Esters

Project Leader: Dr. M. E. Chiddix

Technical Staff: Dr. E. O. Leonard

Report Period: July 1, 1963 to October 31, 1964

Central Research Laboratory

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Preparation of New Arsinic Acids and Esters

Introduction

Work was started on July 1, 1963 under Contract No. DA 18-064-AMC-135 (A) to prepare arsinic acid compounds containing biologically active moieties such as triple bonds, pyrrolidone rings, aromatic halo, -nitro and -methoxy groups, and hydroxyalkyl groups.

This is the final report issued under this contract. Monthly Letter reports were issued from July 31, 1963 to May 31, 1964. Quarterly Reports were issued each quarter from July 31, 1963 to June 30, 1964 and a Letter Report was issued covering the period July 1, 1964 to September 30, 1964.

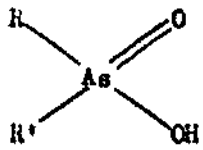
Summary or Abstract

Forty-two new arsinic acids were synthesized and submitted to the United States Army, Biological Laboratories, Fort Detrick, Frederick, Maryland, for screening. Biological data was received on the primary screening results of all compounds presented. Of the forty-two compounds submitted, twenty-four received a rating from 18 to 24 which is considered highly active.

Discussion

The forty-two arsinic acids submitted under this Contract are listed in Table 1 according to functional groups. In Table 2 the compounds are listed according to their activity.

Table 1



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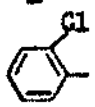
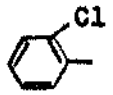
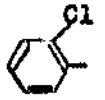
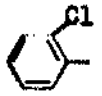
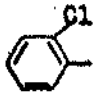
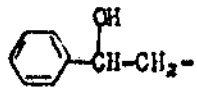
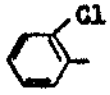
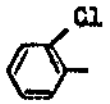
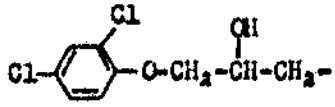
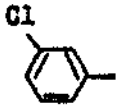
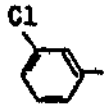
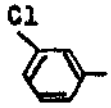
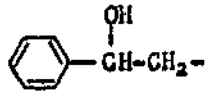
1. 5503-15		$H_2C{=}CH{-}CH_2{-}$	16
2. 5503-114		$HOCH_2{-}CH_2{-}$	21
3. 5503-69		$H_3C{-}\underset{\displaystyle OH}{CH}{-}CH_2{-}$	18
4. 5503-80		$H_3C{-}CH_2{-}\underset{\displaystyle OH}{CH}{-}CH_2{-}$	16
5. 5503-88			20
6. 5503-93		$H_2C{=}CH{-}CH_2{-}O{-}CH_2{-}\underset{\displaystyle OH}{CH}{-}CH_2{-}$	22
7. 5503-94			25
8. 5503-68		$CH_3{-}\underset{\displaystyle OH}{CH}{-}CH_2{-}$	18
9. 5503-72		$CH_3{-}CH_2{-}\underset{\displaystyle OH}{CH}{-}CH_2{-}$	17
10. 5503-75			20

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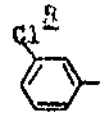
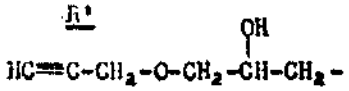
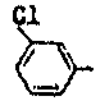
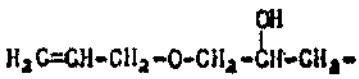
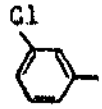
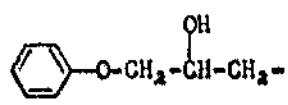
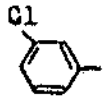
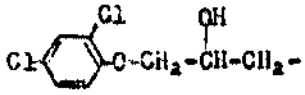
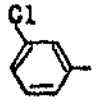
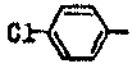
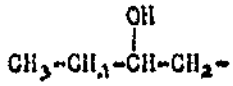
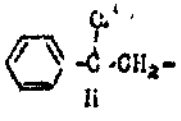
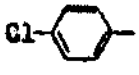
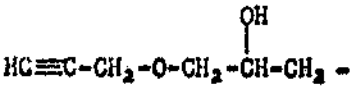
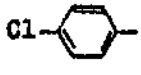
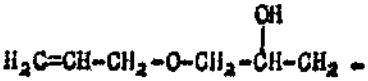
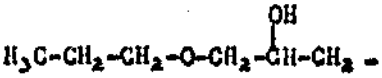
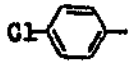
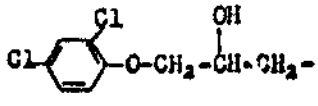
11. 5503-120			23
12. 5503-100			22
13. 5503-99			24
14. 5503-101			24
15. 5503-77			26
16. 5503-111			18
17. 5503-116			18
18. 5503-121			20
19. 5503-121			23
20. 5503-107			22
21. 5503-109			21
22. 5503-108			24

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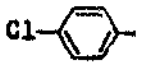
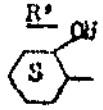
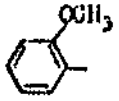
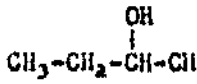
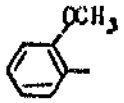
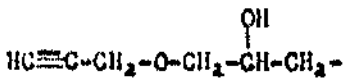
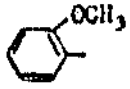
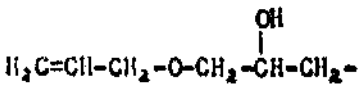
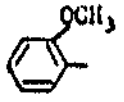
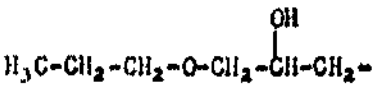
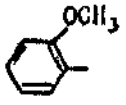
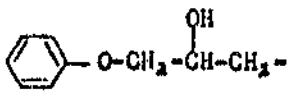
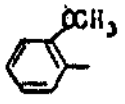
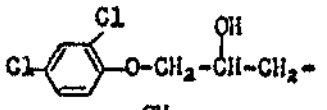
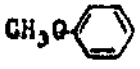
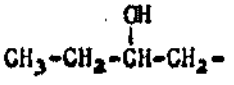
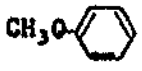
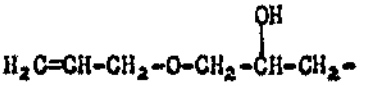
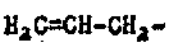
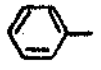
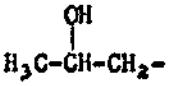
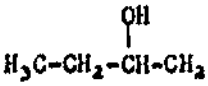
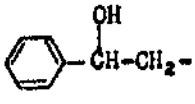
23. 5503-117			26
24. 5503-128			19
25. 5503-125			23
26. 5503-123			22
27. 5503-124			22
28. 5503-127			24
29. 5503-122			24
30. 5503-131			19
31. 5503-133			23
32. 51670-257			15
33. 5503-54			17
34. 5503-71			16
35. 5503-84			20

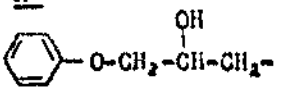
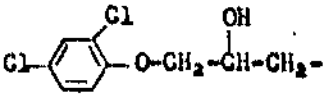
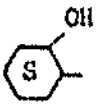
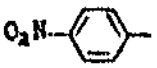
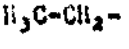
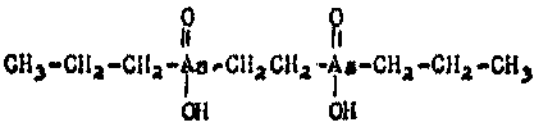
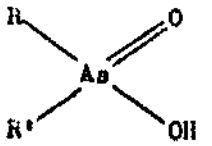
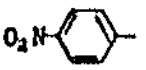
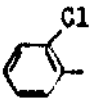
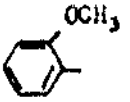
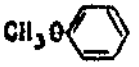
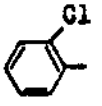
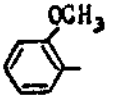
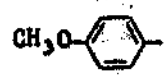
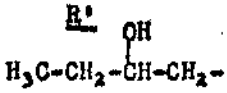
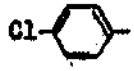
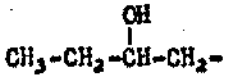
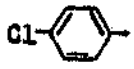
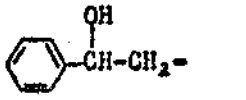
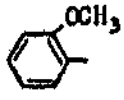
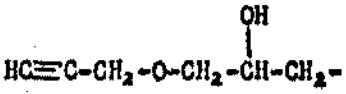
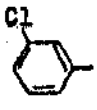
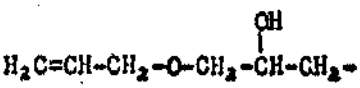
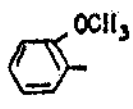
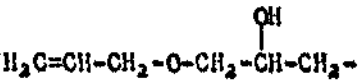
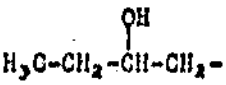
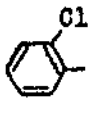
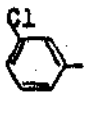
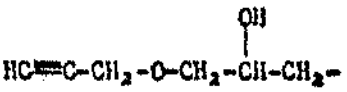
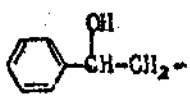
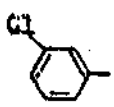
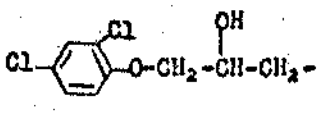
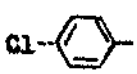
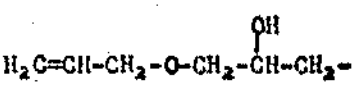
Exp. No.	R	R'	Procedure Page No.
36. 5503-85			23
37. 5503-92			25
38. 5503-76			26
39. 5503-8			25
40. 51670-300			14
41. 51670-260			14
42. 5503-40-B			26

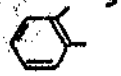
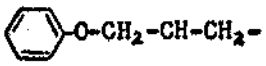
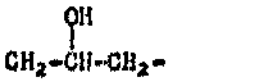
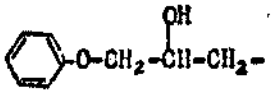
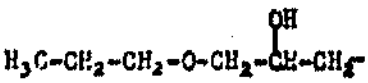
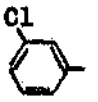
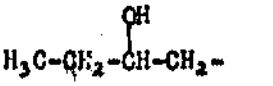
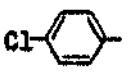
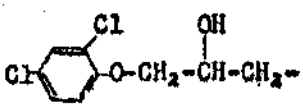
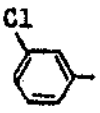
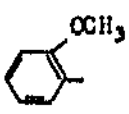
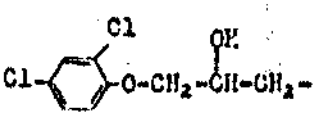
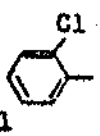
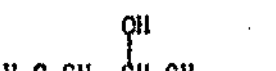
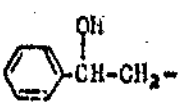
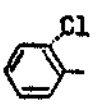
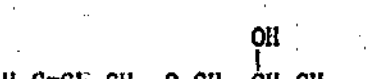
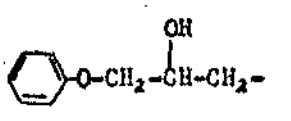
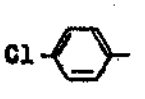
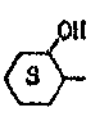
Table 2

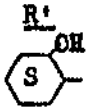
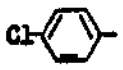
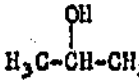
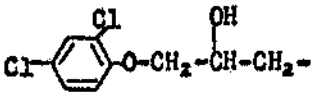
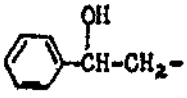
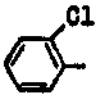
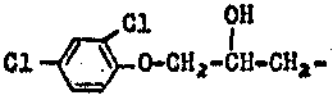
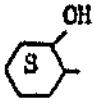
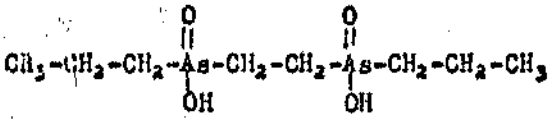


Exp. No.	R	R'	Activity[1]
1. 5503-8		H_3C-CH_2-	24
2. 5503-15		$H_2C=CH-CH_2-$	24
3. 5503-121		$HC\equiv C-CH_2-O-CH_2-\underset{OH}{CH}-CH_2-$	23
4. 5503-128		$H_3C-CH_2-\underset{OH}{CH}-CH_2-$	23
5. 5503-133		$H_2C=CH-CH_2-O-CH_2-\underset{OH}{CH}-CH_2-$	23
6. 5167G-257		$H_2C=CH-CH_2-$	23
7. 5167G-260	$H_3C-CH_2-CH_2-$	$H_2C=CH-CH_2-$	23
8. 5503-69		$H_3C-\underset{OH}{CH}-CH_2-$	22
9. 5503-124		$H_3C-CH_2-CH_2-O-CH_2-\underset{OH}{CH}-CH_2-$	22

[1] 24-18 highly active
17-12 moderately active
11-10 slightly active
9-1 non-effective
24-16 recommended for further screening

Exp. No.		R	R'	Activity
10.	5503-131			22
11.	51670-300	$H_3C-CH_2-CH_2-CH_2-$	$HO-CH_2-CH_2-$	21
12.	5503-116			21
13.	5503-110			21
14.	5503-125			21
15.	5503-100			20
16.	5503-123			20
17.	5503-71			20
18.	5503-114		$HO-CH_2-CH_2-$	19
19.	5503-120			19
20.	5503-84			19
21.	5503-101			18
22.	5503-107			18

Exp. No.	R	R'	Activity
23. 5503-127			18
24. 5503-54			18
25. 5503-99			17
26. 5503-109			17
27. 5503-72			16
28. 5503-108			16
29. 5503-68			15
30. 5503-122			14
31. 5503-80			13
32. 5503-75			12
33. 5503-93			12
34. 5503-85			12
35. 5503-127			11

Exp. No.	R	R'	Activity
36. 5503-77			10
37. 5503-111			10
38. 5503-92			9
39. 5503-88			7
40. 5503-94			7
41. 5503-76			7
42. 5503-40-B			6

A. Purification of Arsinic Acids

Because of the great difficulty of separating water soluble salts from the lower molecular weight, water soluble arsinic acids, the use of a sulfonic acid ion-exchange resin was investigated. It was found that arsinic acids were strongly held to the resin and good separation from salt was possible. A general description of the method used for ion-exchange purifications is as follows. The material to be purified is dissolved in a minimum amount of methyl alcohol, filtered if necessary, and the alcoholic solution carefully applied to a column containing the ion-exchange resin (IR-120 H^+ form). (The resin is previously washed several times with methanol and placed in the column in methanol). The column is then eluted with one to three liters of methanol. The first materials to be eluted are hydrogen halide acids and small amounts of a very dark colored oily material. When the effluent becomes clear, colorless, and neutral, the eluent is changed to a 20% water-80% methanol solution. The water concentration of the eluent is gradually increased to 100%. In several cases the arsinic acid was removed before the system was 100% aqueous. In other cases it was necessary to use a 0.1N NH_4OH solution as the eluent. The removal of product from the column can be easily detected by a pH change of the effluent. When the inorganic acids and impurities mentioned above are removed from the column, the pH of the effluent will return to approximately 7. When the arsinic acid is being removed the pH of effluent drops to pH 3.2 to 4.0. When the pH returns to 7 it is assumed all the arsinic acid is removed. When ammonium hydroxide is used as the effluent, a sharp rise in pH will be noted when the arsinic acid is removed. The product is recovered by removal of the solvent at reduced pressure in a rotating evaporator.

Some arsinic acids were purified by crystallization from a methanol-ether solution in a dry-ice-isopropanol bath.

The samples to be purified were dissolved in up to 500 volumes of acetone. Thorough stirring of this solution usually resulted in precipitation of the desired product. In several instances it was necessary to use a dry-ice-isopropanol bath along with the stirring.

Pure products were obtained by Soxhlet extraction of the final residue with absolute ethanol. Crystallization of the product was induced by addition of acetone to the extracted material after removal of the alcohol.

Purification of solids was accomplished during acidification of the reaction mixture by filtration and thorough washing with acetone.

Impurities were removed by placing the solids obtained in water at 50°C. and filtering while warm. The solids collected on the filter pad were thoroughly washed with water and acetone.

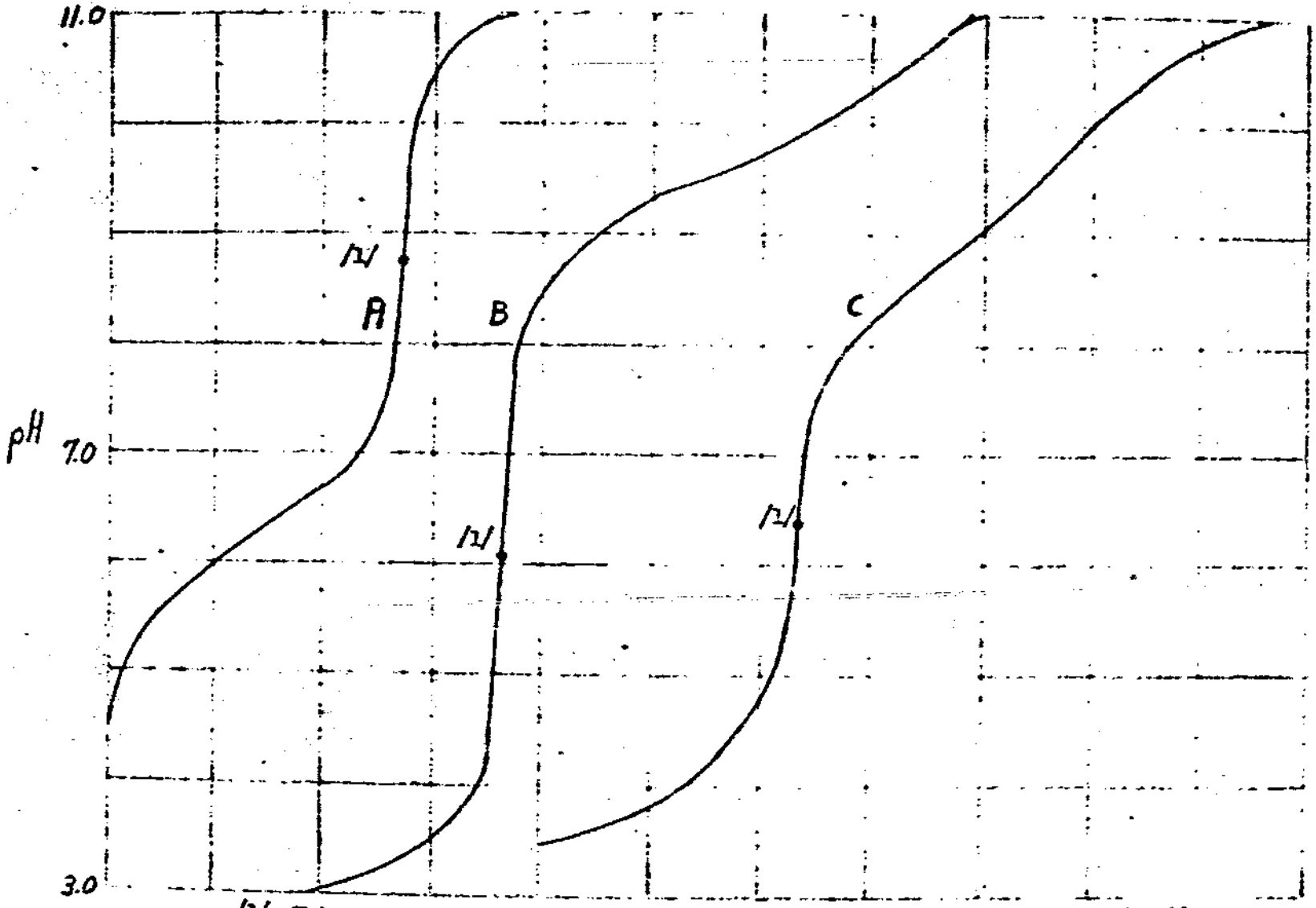
B. Determination of Purity

1. Analysis by Titration

The curves in Figure 1 represent the titration of an arsinic acid (Curve A), an arsonic acid (Curve C), and an unknown compound isolated from the reaction of propargyl bromide with sodium butyl arsonite (Curve B). The curve

Figure 1

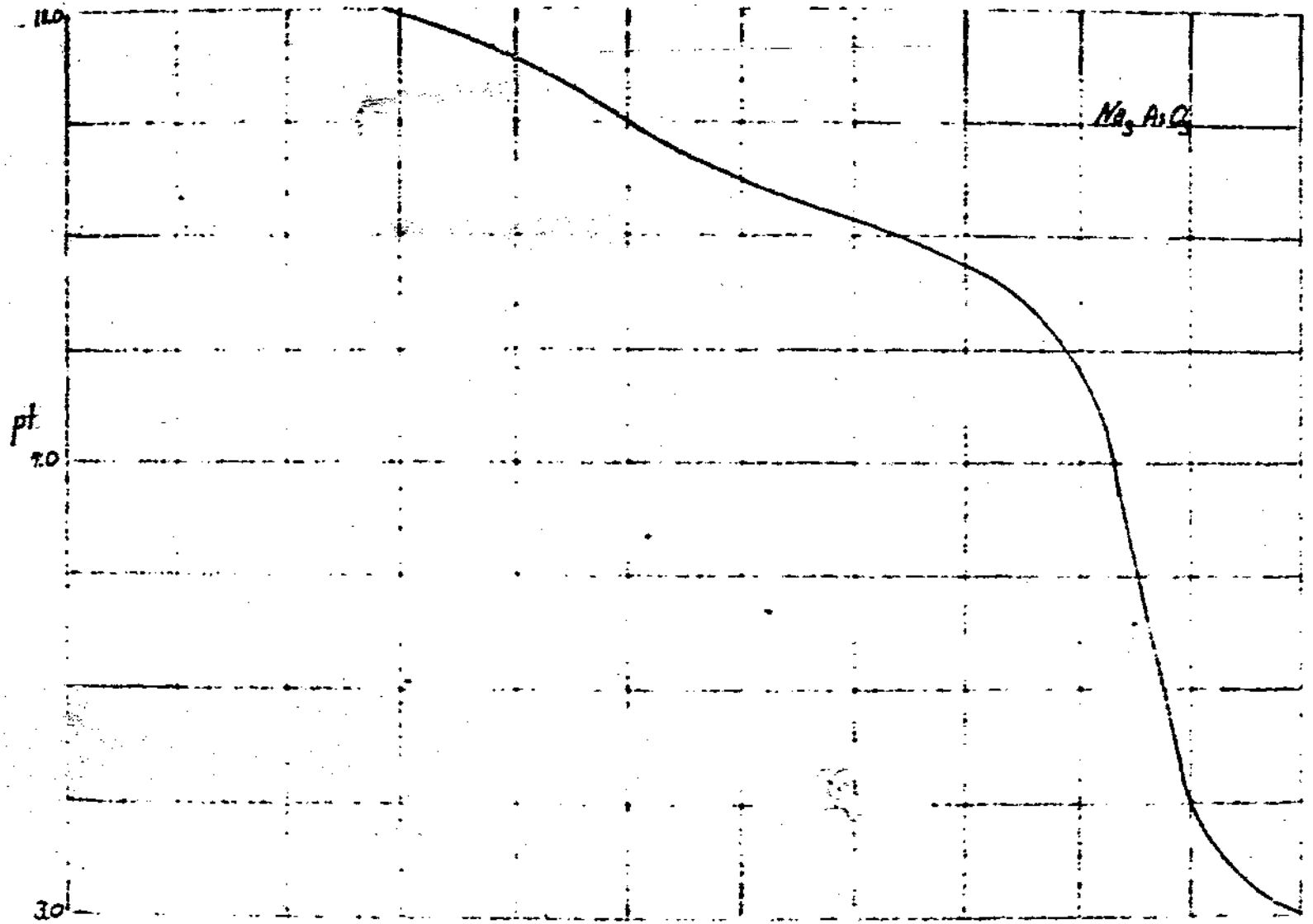
Titration of Arsinic and Arsonic Acids With Standard Sodium Hydroxide



/1/ This point represents the addition of one equivalent of base per mole of acid.

Figure 2

Titration of Trisodium Arsenite With Standard HCl Solution (partial curve)



for allyl propyl arsinic acid (A) shows one sharp break while the unknown compound (B) shows two. Curve C was obtained by titrating benzenearsonic acid with standard sodium hydroxide solution and like B shows two breaks. Figure 2 represents a curve obtained by titrating trisodium arsenite with standard acid solution down to pH 3. From the curves shown in Figures 1 and 2, it can be seen that titration readily distinguishes between arsinic acids, arsonic acids, and sodium arsenite. This method can be used for rapid and quantitative determination of the purity of the product.

2. Determination of Arsonic

Place a weighed sample in a Kjeldahl flask with 10 ml. of conc. H_2SO_4 and heat to boiling. Add 0.5 ml. aliquots of 30% H_2O_2 at intervals until a clear solution is obtained. Cool the solution and dilute to approximately 50 ml. with distilled water. Add 5 grams of KI and heat 1/2 hour on a steam bath. Cool and reduce the iodine with 0.1N Na_2CO_3 to thyodene end point. Neutralize to phenolphthalein with Na_2CO_3, add 0.5 grams $NaHCO_3$, and titrate with 0.1N I_2 to the thyodene end point.

$$\frac{\text{ml } I_2 \times \text{N } I_2 \times 7.491}{\text{wt.} \times 2} = \% \text{ As}$$

C. Synthesis

1. Propyl Arsonic Acid

Three hundred and ninety-six grams (2.0M) of arsenic trioxide was added in small increments to 1200 ml. 10N sodium hydroxide solution. An ice-bath was necessary to maintain a temperature between 65-70°C. during the addition of the arsenic trioxide. The ice-bath was replaced with the heating mantle and the solution heated to 70°C. Four hundred and ninety-two grams (4.0M) of propyl bromide was then added from the dropping funnel at a rate to maintain the temperature of 65-70°C. Upon complete addition of the bromide, the solution was refluxed for 36 hours and an additional 123 g. (1.0M) propyl bromide added. Reflux was continued until titration of an aliquot with standard iodine solution showed the reaction to be 85-90% complete /1/. The reaction solution was cooled to room temperature and filtered. The filtrate was neutralized with concentrated HCl. An ice-bath was necessary to maintain the temperature between 25-30°C. during neutralization. The neutral mixture was again filtered and the filtrate concentrated to about 1/2 volume. The solution was again cooled to room temperature and filtered.

The solids obtained up to this point were primarily NaCl, NaBr and unreacted sodium arsenite. All solids obtained in the following steps contained inorganic salts and product.

The filtrate was acidified with concentrated HCl to pH 4.0. The temperature was kept below 30°C. during acidification. The solution was filtered

/1/ The progress of the reaction can be followed by periodically removing 1 cc. of the reaction solution and titrating the unreacted sodium arsenite with standard iodine solution using starch-iodine paper to determine the end point.

and the filtrate concentrated to about 1/2 volume. It was cooled to room temperature and filtered again. The filtrate was acidified to pH 3.0 and filtered again. By further concentrations and filtrations more product was isolated from the liquid. The greater part of the product was obtained after acidification to pH 4.0 and below. The solids obtained were titrated with standard base to determine the assay of the product in each. The product was purified by recrystallization from a minimum amount of water. Yield: 520 g. (77% of theory), M.P. 125-127°C. /2/

2. Allyl Propyl Arsinic Acid

Allyl propyl arsinic acid was synthesized by the reaction of allyl bromide on sodium propyl arsonite as follows: Propyldichloroarsine (0.34M) was added to 175 ml. of 10N NaOH at room temperature. The resulting solution was cooled to 15°C. and allylbromide (0.5M) was added at a rate to maintain the reaction temperature below 30°C. The solution was then refluxed for 2.5 hours and an additional half mole of allyl bromide added. After standing overnight, the reaction solution was refluxed for two hours, cooled to room temperature and several grams of precipitated salts removed by filtration. The filtrate was extracted with benzene to remove excess allyl bromide, neutralized with concentrated HCl, filtered and concentrated to one-half volume. It was filtered again, and the pH of this filtrate was adjusted to 3.5. At this pH an oil separated from the solution. The oil was separated from the aqueous layer, dried and refrigerated. A small amount of crystallization of the oil occurred after 8 days refrigeration, and these crystals redissolved in the remaining oil during attempts to separate them from the oil by filtration.

The purified product was obtained by the method described in A-1 above and crystallized as described in A-2.

Yield:	10 g.
M.P.:	91-95°C.
Assayed for Arsenic:	Calculated, 39.51%; found, 39.84%.
Neutral Equivalent:	101% of theory.
Bromination:	101% of theory.

Soluble in water, acetone, and alcohol.

3. 2-Hydroxyethyl-n-butyl Arsinic Acid

Ethylene oxide (12 g.) (0.27M) was reacted with n-butyldichloroarsine (51 g.) (0.25M) in 10N sodium hydroxide solution (160 ml.). Acidification and concentration of the reaction solution and ether extraction of an alcoholic solution of the final concentrate resulted in the isolation of a slightly viscous ether solution. Removal of the ether left a very viscous residue which when dissolved in a minimum amount of methanol and diluted with ether yielded 15 g. of a white tacky solid. Concentration of the alcohol-ether filtrate yielded

/2/ The air-dried solid fractions can be titrated with standard base to pH 7 for a close estimate of the assay. One gram of pure product will require 14.38 ml. of 0.5N NaOH.

19 g. of a yellow, viscous oil. Titration of a sample of the oil with standard alkali resulted in a neutral equivalent of 88.8% of theory. A sample of the compound was submitted for arsenic assay, and it was found to contain 33.7% arsenic (Calculated 35.65%). Analyses of this product by gas chromatography showed 3.2% ethylene glycol and smaller quantities of di- and triglycols. These glycols are normal by-products of reactions of ethylene oxide in strong caustic. Calculation of the arsenic content based on the presence of 3.2% ethylene glycol yields a value of 97.5% of theory for the remaining product.

Neutralization Equivalent: 94% of theory (based on the content of impurities)

Soluble in water, alcohol and acetone.

4. Phenyldichloroarsine

Phenyldichloroarsine was synthesized by the method of Quick and Adams./1/

One hundred and ninety grams of phenylarsonic acid was added to 800 ml. concentrated HCl containing one gram of potassium iodine. Sulfur dioxide was passed into the solution until the solution was saturated. Using a separatory funnel, the oily dichloroarsine was separated from the aqueous layer, dissolved in benzene and dried over calcium chloride. Removal of the benzene resulted in a yield of 220 g. of amber liquid. This compound was used without further purification.

5. Allyl Phenyl Arsinic Acid

Fifty-five grams (0.25M) of phenyl dichloroarsine was slowly added to 200 ml. of 10N NaOH at 15°C.-20°C. Thirty-five grams (0.29M) of allyl bromide was added at a rate to maintain the temperature at 20°C. The reaction solution was refluxed for 2 hours and an additional 20 g. of allyl bromide was added. Reflux was continued for an additional hour. The solution was cooled to room temperature and 10 g. of salts filtered off. The pH of the filtrate was adjusted to 7 with concentrated HCl and precipitated salts removed by filtration. The filtrate was then concentrated to approximately two-thirds volume and adjusted to pH 5.5 with concentrated HCl. Precipitated salts were filtered off and the filtrate adjusted to a pH of 3.5. At this pH an oil separated from the solution. The oil was removed from the aqueous layer and dried. Yield - 29 g. Efforts to induce crystallization were unsuccessful. The material was purified by passage through an ion-exchange resin (1R-120 H^+) (Method A-1 above) yeilding 17 g. of pale yellow viscous oil.

Assay for As: Calc'd., 33.18%; Found, 33.4%
Neutralization Equivalent: 97.3% of theory
Bromination: 97% of theory

Soluble in water, acetone and alcohol.

/1/ Quick and Adams, J. Am. Chem. Soc. 44 805 (1922).

6. Allyl o-Chlorophenyl Arsinic Acid

o-Chlorophenyl arsonic acid was prepared by the method of Palmer and Adams /1/ and o-chlorophenyldichloroarsine was prepared by the method of Quick and Adams /2/.

One hundred and twenty grams of sodium hydroxide was dissolved in 180 g. of water and this solution cooled to 0°C. Thirty-three grams (0.12M) of o-chlorophenyldichloroarsine was slowly added and the resulting solution heated to 25°C. Allyl bromide (0.18M) was then added and the reaction solution was heated to reflux for six hours. This solution, after standing overnight, separated into two layers. The organic layer was removed and dissolved in H_2O. The pH of this solution was adjusted to 3 with concentrated HCl. At this pH an oil separated from solution. The aqueous layer was decanted off and the residue dissolved in acetone. Insoluble salts were removed by filtration. Removal of the acetone yielded 13 g. of amber colored liquid which hardened to a waxy solid.

Assay for As: Calc'd., 28.75%; Found, 28.7%
Neutralization Equivalent: 97.5% of theory

Soluble in alcohol and acetone.

7. 2-Hydroxybutyl Phenyl Arsinic Acid

Phenyldichloroarsine (33.5 g.) was added to 140 ml. of 10N NaOH at 25°C. Twenty-nine grams of butylene oxide (1, 2) was added to this solution. The reaction solution was stirred at room temperature for six hours. After standing overnight, the reaction solution was heated at 55°C. for 30 minutes and cooled to room temperature. The product was obtained by acidifying the total reaction solution to pH 3 and concentrating this solution to a thick pasty residue. The residue was filtered, thoroughly washed with acetone and air dried. The dried solid was extracted (Soxhlet) with absolute ethanol. Removal of the alcohol yielded a clear, colorless, viscous liquid. Crystallization was induced by addition of acetone. Yield - 22.5 g.

M.P. 141-143°C.
Assay for As: Calc'd., 29.02%; Found, 29.5%
Neutralization Equivalent: 99.07% of theory

Soluble in water and alcohol. Insoluble in acetone.

8. 2-Hydroxybutyl o-Chlorophenyl Arsinic Acid

o-Chlorophenyl arsonic acid was prepared by the method of Palmer and Adams /1/ and o-chlorophenyl dichloroarsine was prepared by the method of Quick and Adams /2/. The final product, 2-hydroxybutyl o-chlorophenyl arsinic acid

/1/ Palmer and Adams J. Am. Chem. Soc. 44 1356 (1922).
/2/ Quick and Adams J. Am. Chem. Soc. 44 805 (1922).

was synthesized as previously described in Example 7, for the 2-hydroxybutyl phenyl arsinic acid derivative. It was isolated by acidification of the aqueous layer which separated from the reaction solution while standing at room temperature. The pH of the aqueous layer was adjusted to 3 with concentrated HCl and this solution was taken to dryness using a flash evaporator. The solid obtained was extracted (Soxhlet) with absolute ethanol for 24 hours. The cooled alcohol extract was filtered from a small amount of inorganic solids and the filtrate was evaporated to a clear, colorless viscous oil. The solid was purified by crystallization from water. Yield - 3 g.

Sodium o-chlorophenylarsenite (26.4 g.) and butylene oxide (22 g.)
M.P. 150-152°C.
Assay for As: Calc'd., 25.61%; Found, 26.08%
Neutralization Equivalent: 101% of theory

Soluble in hot water or alcohol, insoluble in acetone.

9. 2-Hydroxybutyl m-Chlorophenyl Arsinic Acid

m-Chlorophenylarsonic acid was prepared by the method of Palmer and Adams /1/ and m-chlorophenyldichlorarsine was prepared by the method of Quick and Adams /2/. The final product 2-hydroxybutyl m-chlorophenyl arsinic acid was synthesized as described above for 2-hydroxybutyl phenyl arsinic acid, Example 7. It was isolated by acidification of the organic layer which separated from the reaction solution while standing at room temperature. The pH of the organic layer was adjusted to 3.2 with concentrated HCl. At this pH a large amount of solid precipitated from solution. The solid was removed by filtration, washed thoroughly with water and acetone and air dried. Yield - 25.5 g.

Sodium m-chlorophenylarsenite (26.4 g.) and butylene oxide (22 g.)
M.P. 153-157°C.
Assay for As: Calc'd., 25.61%; Found, 25.8%
Neutralization Equivalent: 103% of theory

Soluble in absolute or aqueous ethanol, insoluble in water or acetone.

10. 2-Hydroxypropyl Phenyl Arsinic Acid

This compound was prepared by the method used to synthesize the 2-hydroxybutyl phenyl arsinic acid derivative, Example 7. Propylene oxide was used in place of butylene oxide. The product was isolated as follows. The reaction solution was filtered from a small amount of precipitated solids, neutralized with concentrated HCl and refiltered. This filtrate was concentrated to 1/2 volume, cooled to room temperature and the precipitated salts removed by filtration. The clear filtrate was acidified to pH 3 with concentrated HCl. At this pH, solid and oil separated from solution. This mixture was filtered and the filtrate concentrated to a pasty residue. The residue was filtered

/1/ Palmer and Adams J. Am. Chem. Soc. 44 1356 (1922).
/2/ Quick and Adams J. Am. Chem. Soc. 44 805 (1922).

using suction and the oily filtrate thus obtained was dissolved in acetone. Precipitation of solid from this acetone solution occurred shortly. This solid was removed by filtration and air dried. Yield - 16 g.

Sodium phenylarsenite (23 g.) and propylene oxide (25.7g.)
M.P. 133-135°C.
Assay for As: Calc'd., 30.78%; Found, 31.76%
Neutralization Equivalent: 95% of theory

Slightly soluble in water and alcohol, insoluble in acetone.

The following three compounds were prepared by the method described above in Example 10.

11. 2-Hydroxypropyl-o-Chlorophenyl Arsinic Acid

Sodium o-chlorophenylarsenite (26.4 g.) and propylene oxide (25.7 g.)
Yield - 6 g.
M.P. 150-154°C.
Assay for As: Calc'd., 26.88%; Found, 27.92%
Neutralization Equivalent: 99.4% of theory

Soluble in water and alcohol, insoluble in acetone.

12. 2-Hydroxypropyl-m-Chlorophenyl Arsinic Acid

Sodium m-chlorophenylarsenite (26.4 g.) and propylene oxide (25.7 g.)
Yield - 4.5 g.
M.P. 146-149°C.
Assay for As: Calc'd., 26.88%; Found, 26.32%
Neutralization Equivalent: 95% of theory

Soluble in water and alcohol, insoluble in acetone.

13. 2-Hydroxypropyl-p-chlorophenyl Arsinic Acid

Sodium p-chlorophenylarsenite (26.4 g.) and propylene oxide (25.7 g.)
Yield - 18 g.
M.P. 123-128°C.
Assay for As: Calc'd., 26.88%; Found, 26.4%
Neutralization Equivalent: 98% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone.

14. 2-Hydroxybutyl p-Chlorophenyl Arsinic Acid

p-Chlorophenyl arsonic acid was prepared by the method of Palmer and Adams /1/ and p-chlorophenyldichloroarsine was prepared by the method of Quick and Adams /2/. The final product, 2-hydroxybutyl p-chlorophenyl arsinic

/1/ Palmer and Adams J. Am. Chem. Soc. 44 1356 (1922).
/2/ Quick and Adams J. Am. Chem. Soc. 44 805 (1922).

acid was synthesized and previously described for the 2-hydroxybutyl phenyl arsinic acid derivative, Example 7. It was isolated from the reaction solution by acidification of the organic layer and the purification was carried out as described above for the purification of the 2-hydroxybutyl m-chlorophenyl arsinic acid derivative. Yield - 13 g.

Sodium p-chlorophenylarsenite (26.4 g.) and butylene oxide (22 g.)
M.P. 155-158°C.
Assay for As: Calc'd., 25.61%; Found 25.2%
Neutralization Equivalent: 95% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

15. 2-Hydroxybutyl o-Methoxyphenol Arsinic Acid

o-Methoxyphenyl arsonic acid was prepared by the method of Palmer and Adams /1/ and o-methoxyphenyldichloroarsine was prepared by the method of Quick and Adams /2/. The final product, 2-hydroxybutyl o-methoxyphenyl arsinic acid was prepared by the method described above for the 2-hydroxybutylphenyl arsinic acid derivative, Example 7 and it was isolated by the method described for the 2-hydroxybutyl m-chlorophenyl arsinic acid, Example 9. Yield - 5.0 g.

Sodium o-methoxyphenylarsenite (24.4 g.) and butylene oxide (21 g.)
M.P. 146-148°C.
Assay for As: Calc'd., 26.0%; Found, 25.8%
Neutralization Equivalent: 104% of theory

Soluble in absolute or aqueous alcohol, insoluble in acetone or cold water.

16. 2-Hydroxybutyl-p-Methoxyphenyl Arsinic Acid

The method for the preparation of the intermediates and the final product, and the isolation of the final product are the same as those described above for the 2-hydroxybutyl-o-methoxyphenyl arsinic acid derivative, Example 15.

Sodium p-methoxyphenylarsenite (24.4 g.) and butylene oxide (22 g.)
Yield - 4 g.
M.P. 144-145°C.
Assay for As: Calc'd., 26.0%; Found 27.4%
Neutralization Equivalent: 104% of theory

Soluble in absolute or aqueous ethanol; insoluble in acetone or cold water.

/1/ Palmer and Adams J. Am. Chem. Soc. 44 1356 (1922).
/2/ Quick and Adams J. Am. Chem. Soc. 44 805 (1922).

17. 2-Phenyl-2-hydroxyethyl Phenyl Arsinic Acid

The synthesis of the intermediates and the final product are the same as those previously described. Styrene oxide was used in place of butylene oxide. At completion of the reaction, 2 layers were present. The upper organic layer contained a large amount of solid. Work-up of the liquids did not yield product. The solid was dissolved in water and the pH slowly adjusted to 3.0. As this pH was approached precipitation of solid occurred. The precipitate was isolated by filtration, washed well with acetone and air dried. Yield - 9.5 g.

Sodium phenylarsenite (23 g.) and styrene oxide (36 g.)
M.P. 158-161°C.
Assay for As: Calc'd., 23.23%; Found, 24.4%
Neutralization Equivalent: 102% of theory

Soluble in aqueous or absolute ethanol, insoluble in acetone or cold water.

18. 2-Phenyl-2-Hydroxyethyl-m-Chlorophenyl Arsinic Acid

The procedures for the synthesis and isolation of this compound are the same as those described for the 2-phenyl-2-hydroxyethyl phenyl arsinic acid, Example 17.

Sodium m-chlorophenylarsenite (26.4 g.) and styrene oxide (36 g.)
Yield - 18 g.
M.P. 181-184°C.
Assay for As: Calc'd. 21.98%; Found, 22.3%
Neutralization Equivalent: 95.3% of theory

Soluble in aqueous or absolute ethanol, insoluble in acetone or cold water.

19. 2-Phenyl-2-hydroxyethyl-p-chlorophenyl Arsinic Acid

The procedure for the synthesis and isolation of this compound are the same as those described for the 2-phenyl-2-hydroxyethyl phenyl arsinic acid derivative, Example 17.

Sodium p-chlorophenylarsenite (26.4 g.) and styrene oxide (36 g.)
Yield - 10 g.
M.P. 158-161°C.
Assay for As: Calc'd., 21.99%; Found, 21.0%
Neutralization Equivalent: 98% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

20. 2-Phenyl-2-Hydroxyethyl o-Chlorophenyl Arsinic Acid

This compound was prepared and isolated by the procedure described above for 2-phenyl-2-hydroxyethylphenyl arsinic acid derivative, Example 17.

Sodium o-chlorophenylarsenite (26.4 g.) and styrene oxide (36 g.)
Yield - 6 g.
Neutralization Equivalent: 95% of theory
Arsenic Assay: Calc'd., 21.99%; Found, 23.21%
M.P. 167-170°C.

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

21. 2-Hydroxyethyl-o-Chlorophenyl Arsinic Acid

This compound was synthesized and isolated as described for the 2-hydroxyethyl-n-butyl arsinic acid derivative, Example 3.

Sodium o-chlorophenylarsenite (26.4 g.) and ethylene oxide (14 g.)
Yield - 6 g.
M.P. 129-132°C.
Assay for As: Calc'd., 28.32%; Found, 27.73%
Neutralization Equivalent: 95% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

22. 3-Propoxy-2-hydroxypropyl p-Chlorophenyl Arsinic Acid

p-Chlorophenyl arsonic acid was prepared by the method of Palmer and Adams /1/.

p-Chlorophenyldichloro arsine was prepared by the method of Quick and Adams /2/.

p-Chlorophenyldichloroarsine (26.4 g.) was added to 140 ml. of 10N NaOH at 25°C. Seventy-four and four tenths grams of 1,2-epoxy-3-propoxypropane was added to this solution. The reaction solution was stirred at room temperature for six hours. After standing overnight, the reaction solution was heated at 55°C. for 30 minutes and cooled to room temperature. It was isolated from the reaction solution by acidification of the organic layer which separated while standing at room temperature. The pH of the organic layer was adjusted to 3.2 with concentrated HCl. At this pH a large amount of solid precipitated from solution. The solid was removed by filtration, washed thoroughly with water and acetone and air dried.

Yield - 4.5 g.
M.P. 103-106°C.
Assay for As: Calc'd., 22.25%; Found, 23.33%
Neutralization Equivalent: 102% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

/1/ Palmer and Adams, J. Am. Chem. Soc. 44, 1356 (1922).
/2/ Quick and Adams, J. Am. Chem. Soc. 44, 805 (1922).

The following nine examples were prepared using the above general procedures.

23. 3-Propoxy-2-hydroxypropyl o-Methoxyphenyl Arsinic Acid

Sodium o-methoxyphenylarsenite (24.4 g.) and 1,2-epoxy-3-propoxypropane (25.3 g.)
Yield - 6 g.
M.P. 119-121°C.
Assay for As: Calc'd., 22.59%; Found, 21.80%
Neutralization Equivalent: 105% of theory

Soluble in absolute or aqueous alcohol, insoluble in acetone or cold water.

24. 3-Allyloxy-2-hydroxypropyl o-Chlorophenyl Arsinic Acid

Sodium o-chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-allyloxypropane (34 g.)
Yield - 5.9 g.
M.P. 124-126°C.
Assay for As: Calc'd., 22.38%; Found, 22.03%
Neutralization Equivalent: 103% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

25. 3-Allyloxy-2-hydroxypropyl m-Chlorophenyl Arsinic Acid

Sodium m-chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-allyloxypropane (34 g.)
Yield - 6 g.
M.P. 115-117°C.
Assay for As: Calc'd., 22.38%; Found, 23.4%
Neutralization Equivalent: 96.7% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

26. 3-Allyloxy-2-hydroxypropyl p-Chlorophenyl Arsinic Acid

Sodium p-chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-allyloxypropane (34.g.)
Yield - 11 g.
M.P. 114-117°C.
Assay for As: Calc'd., 22.38%; Found, 22.4%
Neutralization Equivalent: 104% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

27. 3-Allyloxy-2-hydroxypropyl o-Methoxyphenyl Arsinic Acid

Sodium o-methoxyphenylarsenite (24.4 g.) and 1,2-epoxy-3-allyloxypropane (34 g.)
Yield - 6.5 g.
M.P. 118-119°C.
Assay for As: Calc'd., 22.7%; Found, 22.7%
Neutralization Equivalent: 106% of theory

Soluble in absolute or aqueous alcohol, insoluble in acetone or cold water.

28. 3-Allyloxy-2-hydroxypropyl p-Methoxyphenyl Arsinic Acid

Sodium p-methoxyphenylarsenite (24.4 g.) and 1,2-epoxy-3-allyloxypropane (34 g.).
Yield - 11 g.
M.P. 112-115°C.
Assay for As: Calc'd., 22.7%; Found, 23.0%
Neutralization Equivalent: 103% of theory

Soluble in absolute or aqueous alcohol, insoluble in acetone or cold water.

29. 3-(2-Propynyloxy)-2-hydroxypropyl m-Chlorophenyl Arsinic Acid

Sodium m-chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-propynyloxypropane (33.6 g.)
Yield - 6 g.
M.P. 110-113°C.
Assay for As: Calc'd., 22.52%; Found, 22.36%
Neutralization Equivalent: 105% of theory

Soluble in absolute or aqueous alcohol, insoluble in acetone or cold water.

30. 3-(2-Propynyloxy)-2-hydroxypropyl p-Chlorophenyl Arsinic Acid

Sodium p-chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-propynyloxypropane (33.6 g.)
Yield - 10 g.
M.P. 131-133°C.
Assay for As: Calc'd., 22.52%; Found, 21.57%
Neutralization Equivalent: 104% of theory

Souble in absolute or aqueous alcohol, insoluble in acetone or cold water.

31. 3-(2-Propynyloxy)-2-hydroxypropyl o-Methoxyphenyl Arsinic Acid

Sodium o-methoxyphenylarsenite (24.4 g.) and 1,2-epoxy-3-propynyloxypropane (33.6 g.)
Yield - 4 g.
M.P. 118-119°C.
Assay for As: Calc'd., 22.83%; Found, 23.06%
Neutralization Equivalent: 105% of theory

Soluble in absolute or aqueous alcohol, insoluble in acetone or cold water.

The following compounds, Nos. 32-39 were synthesized and isolated by the method used to obtain 2-phenyl-2-hydroxyethyl phenyl arsinic acid, Example 17.

32. 3-Phenoxy-2-hydroxypropyl-Phenyl Arsinic Acid

Sodium phenylarsenite (23 g.) and 1,2-epoxy-3-phenoxypropane (45 g.)
Yield - 7 g.
M.P. 151-152°C.
Assay for As: Calc'd., 22.28%; Found, 22.94%
Neutralization Equivalent: 102% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

33. 3-Phenoxy-2-hydroxypropyl m-Chlorophenyl Arsinic Acid

Sodium m-chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-phenoxypropane (45 g.)
Yield - 13.5 g.
M.P. 151-153°C.
Assay for As: Calc'd., 20.21%; Found, 20.09%
Neutralization Equivalent: 99% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

34. 3-Phenoxy-2-hydroxypropyl o-Methoxyphenyl Arsinic Acid

Sodium-o-methoxyphenylarsenite (24.4 g.) and 1,2-epoxy-3-phenoxypropane (45 g.)
Yield - 18 g.
M.P. 149-152°C.
Assay for As: Calc'd., 20.46%; Found, 20.31%
Neutralization Equivalent: 105% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone or cold water.

35. 3-(2,4-Dichlorophenoxy)-2-hydroxypropyl m-Chlorophenyl Arsinic Acid

Sodium m-chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-(2,4-dichlorophenoxy)propane (65.7 g.)
Yield - 4 g.
M.P. 176-179°C.
Assay for As: Calc'd., 17.05%; Found, 17.46%
Neutralization Equivalent: 97.6% of theory

Soluble in absolute or aqueous ethanol; insoluble in acetone or cold water.

36. 3-(2,4-Dichlorophenoxy)-2-hydroxypropyl p-Chlorophenyl Arsinic Acid

Sodium p-chlorophenylarsenite (26.4 g.) and 1,2-epxoy-3-(2,4-dichlorophenoxy)propane (65.7 g.)
Yield - 17 g.
M.P. 132-134°C.
Assay for As: Calc'd., 17.05%; Found, 16.6%
Neutralization Equivalent: 105% of theory

Soluble in absolute or aqueous ethanol; insoluble in acetone or cold water.

37. 3-(2,4-Dichlorophenoxy)-2-hydroxypropyl o-Methoxyphenyl Arsinic Acid

Sodium o-methoxyphenyl arsenic acid (24.4 g.) and 1,2-epoxy-3-(2,4-dichlorophenoxy)propane (65.7 g.)
Yield - 7 g.
M.P. 199-203°C.
Assay for As: Calc'd., 17.22%; Found, 16.96%
Neutralization Equivalent 104% of theory

Soluble in absolute or aqueous ethanol; insoluble in acetone or cold water.

38. 3-(2,4-Dichlorophenoxy)-2-hydroxypropylphenyl Arsinic Acid

Sodium phenylarsenite (23 g.) and 1,2-epoxy-3-(2,4-dichlorophenoxy)propane (65.7 g.)
Yield - 27 g.
M.P. 152-155°C.
Assay for As: Calc'd., 18.94%; Found, 18.42%
Neutralization Equivalent: 95.4% of theory

Soluble in absolute or aqueous ethanol; insoluble in acetone or cold water.

39. 3-(2,4-Dichlorophenoxy)-2-hydroxypropyl o-Chlorophenyl Arsinic Acid

Sodium o-Chlorophenylarsenite (26.4 g.) and 1,2-epoxy-3-(2,4-dichlorophenoxy)propane (65.7 g.)
Yield - 11 g.
M.P. 158-161°C.
Assay for As: Calc'd, 17.05%; Found, 17.86%
Neutralization Equivalent: 105%

Soluble in aqueous or absolute ethanol; insoluble in acetone or cold water.

40. Ethyl p-Nitrophenyl Arsinic Acid

This compound was prepared by the use of a modified Bart reaction as described by Doak /1/.

One hundred and twelve grams (0.81M) of p-nitroaniline was placed in 2 L absolute alcohol with 85 g. H_2SO_4 and 194 g. (1.38M) ethyl dichloroarsine. The solution was cooled to 0°C. and diazotized with 57.3 g. (0.83M) $NaNO_2$ in 120 ml. H_2O to an end-point with starch-iodide paper. One gram of cuprous bromide was added and the mixture thoroughly stirred and heated to 60°C. until no more N_2 was evolved. The mixture was cooled to room temperature and filtered. The filtrate was concentrated and the concentrate dissolved in a minimum amount of H_2O. This was passed through an ion-exchange resin (IR-120 H^+). Concentration of the various aqueous fractions collected upon elution first with water and then with dilute aqueous ammonia yielded 41 g. of yellow solid. The fifth fraction eluted with water weighed 6 g. after drying. The solid had the following properties and analysis:

No. 5: M.P. 161-165°C.
Assay for As: Clac'd., 31.06%; Found, 31.0%
Neutralization Equivalent: 101% of theory

Soluble in alcohol, slightly soluble in acetone and water.

/1/ Doak, G. O. J. Am. Chem. Soc. 62 167 (1940).

41. 2-Hydroxycyclohexyl Phenyl Arsinic Acid

This compound was prepared and isolated by the procedure described above for 2-phenyl-2-hydroxyethyl phenyl arsinic acid, Example 17. The solid obtained at pH 3.0 contained some inorganic salts and a small amount of oils, probably glycol. These were removed by placing the solid in water, heating to approximately 50°C. and filtering while hot. The solids collected in the filter funnel were washed well with warm acetone and air dried.

Yield - 13 g.
Neutralization Equivalent: 101% of theory
Arsenic Assay: Calc'd., 26.11%; Found, 26.58%
M.P. 202-207°C.

Soluble in absolute or aqueous ethanol, insoluble in acetone.

42. 2-Hydroxycyclohexyl m-Chlorophenyl Arsinic Acid

This compound was prepared by the procedure described above for 2-phenyl-2-hydroxyethyl phenyl arsinic acid, Example 17. The product was isolated from a lower organic layer. This layer was separated from the reaction mixture and dissolved in about 100 ml. water. This solution was acidified with concentrated HCl to pH 3.0. The precipitated solids were collected by filtration and treated with water and acetone as described above for the 2-hydroxycyclohexyl phenyl arsinic acid, Example 40.

Yield - 22 g.
Neutralization Equivalent: 101% of theory
Arsenic Assay: Calc'd., 23.28%; Found, 23.81%
M.P. 204-206°C.

Soluble in absolute or aqueous ethanol, insoluble in acetone and cold water.

43. 2-Hydroxycyclohexyl p-Chlorophenyl Arsinic Acid

This compound was prepared and isolated by the procedure described above, Example 41.

Yield - 7 g.
M.P. 224-227°C.
Arsenic Assay: Calc'd., 23.51%; Found, 24.37%
Neutralization Equivalent: 105% of theory

Soluble in absolute or aqueous ethanol, insoluble in acetone and cold water.

44. Ethylenebis (Propyl Arsinic Acid) $(CH_3CH_2CH_2-\overset{\overset{O}{\|}}{\underset{OH}{\underset{|}{As}}}-CH_2-CH_2-\overset{\overset{O}{\|}}{\underset{OH}{\underset{|}{As}}}-CH_2CH_2CH_3)$
(5503-40-B)

The following procedure was used in an effort to prepare 2-chloroethyl propyl arsinic acid. However, the ethylenebis acid was the product isolated.

Thirty-five grams (0.18M) propyl dichloroarsine were added to a solution of 44 g. NaOH in 60 ml. H_2O at 25°C. Twenty-six and one-half grams (0.18M) of ethylenechlorobromide were added and the solution refluxed for 15 hours. The solution was then cooled to room temperature and neutralised with concentrated HCl. Precipitated salts were removed by filtration. The filtrate was concentrated to 1/2 volume and salts again removed by filtration. This filtrate was acidified to pH 3 with concentrated HCl and the precipitating solids obtained by filtration. Concentration of the filtrate yielded an additional crop of solids. The last two solids were combined and precipitated from alkaline solution by addition of HCl. Titration of a sample showed a neutralisation equivalent of 102% of theory (as the diarsinic acid).

M.P. 205-209°C.
Arsenic Assay: Calc'd., 45.5%; Found, 47.2% (diarsinic acid)

Slightly soluble in water and alcohol, insoluble in acetone.

Test Methods for Herbicides

Summary of Test Methods

Test:	Vertically directed spray onto potted seedlings
Spray Solution:	Test compound dissolved in Acetone containing 0.5% Tween 20 (unless otherwise noted).
Spray Volume:	12 ml. directed evenly over 3 square feet of area.
Spray Cabinet:	Plexiglass, having bottom area of 3 sq. ft.
Spray Rates:	Applications equivalent to 0.1 and 1 pound per acre.
Replication:	Each rate is applied to 12 pots simultaneously (2 pots of each species). Observations are then made from 2 plants of each broadleaf species and 20 plants of each cereal species.
Species:	Broadleaves* / Cereals**
	Black Valentine Bean / Clinton Oats
	Heavenly Blue Morning-glory / Rice, P.I. 8970
	Scarlet Globe Radish
	Lincoln Soybean
	*Selected 2 plants per pot 7 days after planting **Selected 10 plants per pot 7 days after planting
Age at Treatment:	7 days from planting
Observation:	1 to 2 days; 5 days; 10-14 days after treatment for visual effect as indicated

Scoring:

Rating 1 - No discernable herbicidal activity or very slight effect - symptoms if any are minor, and are probably confounded with environmental effects. Plants appear normal.

Rating 2 - Slight but not marked herbicidal effect - symptoms are detected although their degree of severity is only slight; plants are different from the controls for one or more reasons.

Rating 3 - Moderate or considerable injury to plant tissues differs from (2) in degree of herbicidal activity. Subsequent crop growth would probably be adversely affected through reduced vegetative growth and/or subnormal yield.

Rating 4 - Activity characterized by killing or severe necrosis or other effects which might lead to death before maturity.

Summation of Scoring: The maximum rating for one species at 1 rate over all observed effects is 4. This rating is made for a score of 4 in either "Killing" or "Stunting"; Combinations of other factors may lead to a rating of 4. This is an observer judgement rating. The highest rating for one rate of application is 24 (total of 4 for each of six species rated.) Conversely, a rating of 6 indicates no visible effect.

Significance: Those compounds which receive a rating of 18-24 are considered highly active, those receiving a 12-17 rating are moderately active and those receiving 10-11 are slightly active. Any rating below 10 is considered non-effective. Compounds with an overall rating of 16 or above are recommended for further screening.

Approved by

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