[Contribution from the Department of Chemistry, University of Cincinnati]

# SOME NEW FLUORENE ARSENICALS[1]

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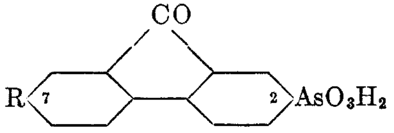
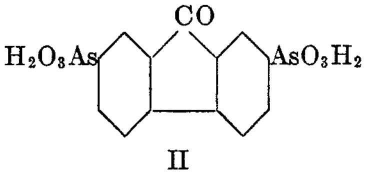
Fluorene arsenicals have been the subject of four papers (1–4). Fluorene, 9-fluorenone, and 9-fluorenol arsenicals have been prepared, all of them derived from fluorene-2-arsonic acid with substituents limited to the 7-position. In several cases, the compounds showed favorable, but not remarkable, pharmacological properties against *Trypanosoma equiperdum*. The more promising of these included 7-acetaminofluorenone-2-arsonic acid, 7-carbamylmethoxyfluorenone-2-arsonic acid, and 7-(carbamylmethylamino)fluorenol-2-arsonic acid. The 2,7-positions, viewed as substituents of the biphenyl system, correspond to the extended *para* (I), a form that would seem to offer the most effective structure type in view of the success of the corresponding benzene analogs.



I

The new arsenicals prepared by us are all fluorenone derivatives. Some extension of the 2,7-series has been made. Fluorenone-2-arsonic acid was obtained from the corresponding amine by a Scheller reaction involving diazotization in acetone medium followed by decomposition in the presence of arsenic trichloride. This is the first application of the Scheller reaction in this series. Conversion of fluorenone-2-arsonic acid to 7-aminofluorenone-2-arsonic acid was accomplished according to the procedures of Morgan and Stewart (2). The aminoarsonic acid was converted through the diazonium salt to the first of the new arsenicals, fluorenone-2,7-diarsonic acid (II). Reduction of the aminoarsonic acid with hypophosphorous acid, followed by treatment with hydrochloric acid, gave 7,7′-diamino-2,2′-arsenofluorenone dihydrochloride (III).



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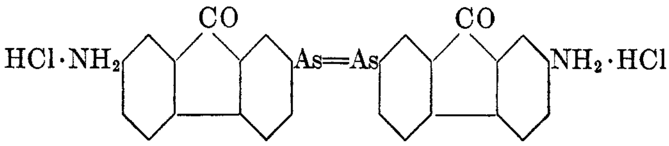

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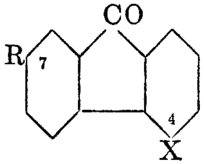
A new parent arsenical, fluorenone-4-arsonic acid (IV), was obtained by a Scheller reaction on the corresponding amine. From this the water-soluble monosodium salt was prepared. The free acid was then reduced to 4-arsenosofluorenone (V) with sulfur dioxide in hydrochloric acid plus a trace of potassium iodide. Treatment of fluorenone-4-arsonic acid with phosphorus trichloride gave the corresponding fluorenone-4-dichloroarsine (VI). Direct nitration of the arsonic acid (IV) yielded 7-nitrofluorenone-4-arsonic acid (VII). This nitroarsonic acid was reduced with alkaline ferrous hydroxide to the aminoarsonic acid (VIII), and the amine acetylated to give the acetamino derivative (IX). Treatment of the supposed 7-aminofluorenone-4-arsonic acid with chloroacetamide gave 7-(carbamylmethylamino)fluorenone-4-arsonic acid (X). Phosphorus trichloride treatment of 7-nitrofluorenone-4-arsonic acid yielded the corresponding dichloroarsine (XI).

### FLUORENONE-4-ARSONIC ACID SERIES

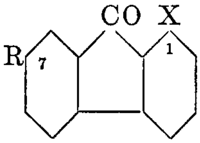


IV. R = H X = $AsO_3H_2$
V. R = H X = AsO
VI. R = H X = $AsCl_2$
VII. R = $NO_2$ X = $AsO_3H_2$
VIII. R = $NH_2$ X = $AsO_3H_2$
IX. R = $CH_3CONH$ X = $AsO_3H_2$
X. R = $CONH_2 \cdot CH_2NH$ X = $AsO_3H_2$
XI. R = $NO_2$ X = $AsCl_2$

Proof of structure for 7-nitrofluorenone-4-arsonic acid was accomplished through the known 7-nitro-4-aminofluorenone prepared from fluorenone-4-carboxylic acid using procedures of Moore and Huntress (5). This 7-nitro-4-aminofluorenone was transformed by us into the arsonic acid by a Scheller reaction, and the nitroarsonic acid was then converted, in the usual manner, into the dichloroarsine. This yielded 7-nitrofluorenone-4-dichloroarsine of known constitution, m.p. 231–232°. The supposed 7-nitrofluorenone-4-dichloroarsine, obtained by direct nitration of fluorenone-4-arsonic acid, melted similarly at 231–232°. A mixed melting point showed no depression.

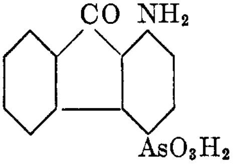
The second new parent arsenical, fluorenone-1-arsonic acid (XII), was prepared from 1-aminofluorenone by a modified Bart reaction using aqueous acetone and alkaline arsenite. A Scheller reaction gave somewhat lower yields. By procedures similar to those employed in the 4-arsonic acid series, the 1-arsonic acid was transformed into 1-arsenosofluorenone (XIII), and fluorenone-1-dichloroarsine (XIV). Direct nitration of XII yielded a mononitroarsonic acid which, from arguments based on analogy, is very probably 7-nitrofluorenone-1-arsonic acid (XV). Work is in progress along the lines of structure proof and will be reported in a later paper. Reduction of this nitro compound with alkaline ferrous sulfate to 7-aminofluorenone-1-arsonic acid (XVI) was followed by conversion, in the usual way, to 7-acetaminofluorenone-1-arsonic acid (XVII) and 7-(carbamylmethylamino)fluorenone-1-arsonic acid (XVIII). Action of phosphorus trichloride on the nitroarsonic acid XV gave the dichloroarsine (XIX).

FLUORENONE-1-ARSONIC ACID SERIES



XII. R = H X = $AsO_3H_2$
XIII. R = H X = AsO
XIV. R = H X = $AsCl_2$
XV. R = $NO_2$ X = $AsO_3H_2$
XVI. R = $NH_2$ X = $AsO_3H_2$
XVII. R = $CH_3CONH$ X = $AsO_3H_2$
XVIII. R = $CONH_2 \cdot CH_2NH$ X = $AsO_3H_2$
XIX. R = $NO_2$ X = $AsCl_2$

Finally, the Bechamp reaction involving direct arsonation with arsenic acid was attempted on 3 amines: 1-aminofluorenone, 2-aminofluorenone, and 4-aminofluorenone. In spite of repeated attempts with a variety of conditions and mole-ratio of reactants, no results were produced with the 2- or 4-amines. It will be noticed that in the case of 2-aminofluorenone, the *para* position is blocked and, at most, very low yields of the 1- or 3-arsonic acid could be expected. In the case of 1-aminofluorenone, however, the *para* position is open for substitution and is also *ortho* to the *ortho-para*-directing phenyl radical. In this case, experiment gave an 11% yield (based on the arsenic acid used) of an aminoarsonic acid which is very probably 1-aminofluorenone-4-arsonic acid (XX).



XX

Structure proof for this compound is in progress. The product gives arsenic contents which are 2–5% too low and is probably contaminated with some arsinic acid ($R_2AsO_2H$). It will be noted that such a structure would appear to offer much in the way of therapeutic properties, since the similarity to many useful arsenicals is apparent. The possibility that the compound may be 1-aminofluorenone-2-arsonic acid cannot be excluded, however, since direct arsonation of 1-aminonaphthalene gives the 2-arsonic acid (6).

The sodium salts of two of the new arsenicals, fluorenone-1-arsonic acid and fluorenone-4-arsonic acid, have been submitted for screening and the pharmacological values will be reported elsewhere.

*Acknowledgement.* The helpful suggestions of Dr. E. F. Farnau of the University of Cincinnati, offered during the last stages of this research, are gratefully recognized.

## EXPERIMENTAL

*Analysis for arsenic.* The procedure of Robertson (7) was employed throughout. The method proved reasonably satisfactory in every case.

*Melting points.* Melting points were obtained with a calibrated thermometer using an aluminum block. Where melting points are not given of the new arsenicals, it may be inferred that such were indefinite or above 300°.

*Fluorenone-2-arsonic acid.* Ten grams (0.0512 mole) of finely divided 2-aminofluorenone was dissolved in 400 ml. of acetone and 11.5 g. (0.112 mole) of concentrated sulfuric acid was added drop-wise with efficient stirring to precipitate the amine sulfate. The mixture was cooled to 5–10° and diazotized with a solution of 5 g. (0.0725 mole) of sodium nitrite in 15 ml. of water. The mixture was stirred for 30 minutes at a temperature not exceeding 10°, and 20 g. (0.0110 mole) of arsenic trichloride was added all at once. After 30 minutes 1 g. of freshly prepared cuprous bromide was added. After nitrogen evolution ceased (30 minutes) the material was poured, with stirring, into 1500 ml. of water and let stand for 2 hours. The insoluble portion was filtered and extracted with a warm (70–80°) solution of 15 g. of sodium bicarbonate in 800 ml. of water. A second extraction was made and the filtrates were combined and treated with decolorizing carbon. The clear yellow filtrate was acidified with 6 *N* hydrochloric acid until distinctly acid to litmus to give a light yellow precipitate of the arsonic acid. The yield was 6.25 g. (40%).

For analysis, purification was made by solution in sodium bicarbonate at 60–70° and acidification with hydrochloric acid. The well-washed material was dried in an oven at 110–115° for 6 to 7 hours or to constant weight.

Anal. Calc'd for $C_{13}H_9AsO_4$: As, 24.6. Found: As, 24.4.

*7-Nitrofluorenone-2-arsonic acid* and *7-aminofluorenone-2-arsonic acid.* These compounds were prepared from fluorenone-2-arsonic acid using procedures of Morgan and Stewart (2).

*Fluorenone-2,7-diarsonic acid.* Ten grams (0.0313 mole) of finely powdered 7-aminofluorenone-2-arsonic acid was dissolved in 100 ml. of water and 5.6 g (0.1 mole) of potassium hydroxide. The solution was cooled to 10° and 2.3 g (0.0334 mole) of sodium nitrite was added and the whole introduced slowly into a mixture of 150 ml. of water and 15 g. (0.146 mole) of concentrated sulfuric acid held at 0°.

An alkaline arsenite solution was prepared from 35 g. (0.626 mole) of potassium hydroxide, 15 g. (0.0760 mole) of arsenious oxide, and 1 g. of copper sulfate in 200 ml. of water. To this, the diazonium solution was added at room temperature in 10-ml. portions with stirring and the mixture was slowly heated on a water-bath to 90° until nitrogen evolution ceased. The filtrate was treated hot with decolorizing carbon, and the resulting clear yellow solution acidified with a slight excess of 6 *N* hydrochloric acid to precipitate the fine yellow-brown diarsonic acid. This was washed well with water and dried to give 9 g. (67%). For purification, the product was dissolved twice in sodium bicarbonate solution with decolorizing treatment in each case and was precipitated with dilute hydrochloric acid. For analysis, a sample of the material was dried in the oven at 110° for 7–8 hours.

*Anal.* Calc'd for $C_{13}H_{10}As_2O_7$: As, 34.7. Found: As, 34.2.

*7,7'-Diamino-2,2'-arsenofluorenone dihydrochloride.* A 1-g. portion of finely divided 7-aminofluorenone-2-arsonic acid was suspended in 10 ml. of glacial acetic acid, and 10 ml. of 30% hypophosphorous acid was added. The mixture was stirred in a water bath at 85–90° for 2½ hours. The buff-orange precipitate was transferred quickly to 100 ml. of boiled distilled water. The mixture was made alkaline with 2 *N* potassium hydroxide, the brown free base was filtered and transferred to 100 ml. of 3 *N* hydrochloric acid, and then filtered again as the buff-orange hydrochloride. The product was washed with dilute hydrochloric acid and dried in a vacuum desiccator over sodium hydroxide to give 0.65 g. (68%) of a light brown material which darkened upon heating for 1 hour at 110°. The compound was analysed as the dihydrochloride.

*Anal.* Calc'd for $C_{26}H_{18}As_2Cl_2N_2O_2$: As, 24.5. Found: As, 23.7.

*Fluorenone-4-arsonic acid.* The modified Scheller procedure used by us in the preparation of fluorenone-2-arsonic acid was employed here on 4-aminofluorenone, of m.p. 138–139°; the yield of light yellow arsonic acid was 45%.

*Anal.* Calc'd for $C_{13}H_9AsO_4$: As, 24.6. Found: As, 24.9.

The monosodium salt was prepared by dissolving the free arsonic acid in a minimum amount of sodium bicarbonate. Dilution with an equal volume of alcohol or acetone resulted, after a time, in the formation of yellow-orange crystals of the salt containing water of hydration. The salt is readily soluble in cold water. After drying at 110° for 5 hours, arsenic analysis indicated the presence of about 2 molecules of water.

*4-Arsenosofluorenone.* A 1-g. portion of finely divided fluorenone-4-arsonic acid was suspended in 25 ml. of 6 *N* hydrochloric acid, and 0.1 g. of potassium iodide was added. On shaking, a slight iodine color was formed. The suspension was saturated at room temperature with sulfur dioxide, with occasional shaking, heated to boiling, and the solution again saturated with the gas and allowed to stand overnight. The fluffy precipitate of the arsenoso compound was filtered off, washed with dilute ammonia and water, and dried. Yield was practically quantitative (about 1 g.).

*Anal.* Calc'd for $C_{13}H_7AsO_2$: As, 27.7. Found: As, 27.6.

*Fluorenone-4-dichloroarsine.* A 1-g. portion (0.00328 mole) of finely divided fluorenone-4-arsonic acid was suspended in 10 ml. of glacial acetic acid and the temperature raised to the boiling point. Heating was interrupted and 1.5 ml. (0.0120 mole) of phosphorus trichloride dissolved in 5 ml. of glacial acetic acid was introduced with swirling. The arsonic acid quickly dissolved to yield a yellow solution. The material was then gently heated to near reflux for 5 minutes and allowed to cool to yield well-formed yellow crystals of the dichloroarsine. Yield 80% (0.85 g.). The melting point after recrystallization from glacial acetic acid was 161–163°.

*Anal.* Calc'd for $C_{13}H_7AsCl_2O$: As, 21.6. Found: As, 21.5.

*7-Nitrofluorenone-4-arsonic acid.* A mixture of 32.5 ml. (0.585 mole) of concentrated sulfuric acid and 37.5 ml. (0.880 mole) of fuming nitric acid ($d$ = 1.50) was cooled in an ice-bath and 15 g. (0.0492 mole) of finely divided fluorenone-4-arsonic acid was introduced with stirring. During the addition, the temperature was not permitted to rise above 10°. The mixture was stirred at room temperature for an additional hour and poured into 500 ml. of ice-water. The precipitated yellow product was filtered, washed, and dried. Yield of crude material was 15.5 g. (90%). Purification was readily accomplished by solution in dilute ammonia, followed by charcoal treatment and precipitation with dilute hydrochloric acid. The pure nitroarsonic acid was dried at 110° for 7 hours for analysis.

*Anal.* Calc'd for $C_{13}H_8AsNO_6$: As, 21.6. Found: As, 21.5.

*7-Nitrofluorenone-4-dichloroarsine.* A 3-g. portion (0.00865 mole) of finely divided 7-nitrofluorenone-4-arsonic acid was converted to the arsine as previously described. The product weighed 2.70 g. (85% yield). Purification was effected by recrystallization from glacial acetic acid; m. p. 231–232°.

*Anal.* Calc'd for $C_{13}H_6AsCl_2NO_3$: As, 20.3. Found: As, 20.5.

*7-Aminofluorenone-4-arsonic acid.* To 200 ml. of water containing 10 ml. of 6 *N* sodium hydroxide was added 16.5 g. (0.0476 mole) of 7-nitrofluorenone-4-arsonic acid and the mixture was stirred and warmed to effect solution. A solution of 100 g. (0.488 mole) of commercial ferrous sulfate (containing about 3 molecules of water of hydration) in 400 ml. of water was made alkaline with 50% sodium hydroxide. To the suspension of ferrous hydroxide was added slowly, with stirring, the solution of the nitroarsonic acid over a period of 30 minutes. The mixture was warmed slowly on the water-bath and maintained at 90° for 1 hour with constant stirring. The material was filtered from the brown-black precipitate of ferric hydroxide and washed with 4 portions of boiling water. The combined deep red filtrates were acidified with 6 *N* hydrochloric acid to the point of precipitation, and precipitation was completed by the addition of a little glacial acetic acid. The precipitated aminoarsonic acid was filtered, washed with water, and dried to give 8.6 g. (56%) of light orange-brown product. For purification, the compound twice was dissolved in hot 3 *N* hydrochloric acid and precipitated by careful neutralization with ammonium hydroxide and acetic acid.

*Anal.* Calc'd for $C_{13}H_{10}AsNO_4$: As, 23.5. Found: As, 23.2.

*7-Acetaminofluorenone-4-arsonic acid.* To 8 ml. of acetic anhydride, 1.5 g. of 7-aminofluorenone-4-arsonic acid was added and the mixture was stirred in a water bath at 75–80° for 2 hours. It was poured into ice-water and the bright orange acetyl derivative was filtered and dried; the yield was practically quantitative. The compound is somewhat soluble in hot glacial acetic acid.

*Anal.* Calc'd for $C_{15}H_{12}AsNO_5$: As, 20.7. Found: As, 20.2.

*7-(Carbamylmethylamino)fluorenone-4-arsonic acid.* A 2-g. portion (0.00532 mole) of 7-aminofluorenone-4-arsonic acid was mixed with 3.7 ml. of 2 *N* potassium hydroxide solution and 5 ml. of water, then diluted with 15 ml. more of water and treated with 0.1 g. of potassium iodide and 1.5 g. (0.0161 mole) of chloroacetamide. The clear wine-red solution was stirred in a boiling-water bath for 2.5 hours to give a red-purple precipitate of the carbamyl derivative. The insoluble portion was washed with water. The dry yield was 2 g. (85%). The compound may be crystallized from alcohol by the addition of ether. Fine purple crystals result. For analysis, the compound was purified through the ammonium salt and dried at 110°.

*Anal.* Calc'd for $C_{15}H_{13}AsN_2O_5$: As, 19.8. Found: As, 19.8.

*Proof of structure of 7-nitrofluorenone-4-arsonic acid.* The known compound, 4-amino-7-nitrofluorenone, was prepared from fluorenone-4-carboxylic acid using the procedures of Moore and Huntress (5). The pure orange-red compound, somewhat soluble in hot alcohol, glacial acetic acid, and acetone melts at 289–290°.

A 4-g. portion (0.0167 mole) of finely divided 4-amino-7-nitrofluorenone was diazotized and treated with arsenic trichloride as previously described. Yield 2.5 g. of product (43%).

The method used previously for the preparation of dichloroarsines was employed on 1.3 g. of the 7-nitrofluorenone-4-arsonic acid of known constitution. Yield was 1.1 g. (80%), which was purified as before by crystallization from glacial acetic acid.

*Anal.* Calc'd for $C_{13}H_6AsCl_2NO_3$: As, 20.3. Found: As, 20.6.

The purified nitrodichloroarsine melted at 231–232° which corresponded to the nitrodichloroarsine prepared from fluorenone-4-arsonic acid. A mixed melting point of the two gave 231–232°.

*Fluorenone-1-arsonic acid.* A 15-g. portion (0.0768 mole) of 1-aminofluorenone (m.p. 118–119°) dissolved in 150 ml. of acetone was poured into a mixture of 100 ml. of 12 *N* hydrochloric acid and 400 ml. of water with efficient stirring. The amine was diazotized at 10° with a solution of 7.0 g. (0.096 mole) of sodium nitrite. After 30 minutes, the mixture was added in small portions to a solution of 15 g. (0.0760 mole) of arsenic trioxide, 15 g. of sodium bicarbonate, 50 g. of potassium hydroxide, and 3 g. of copper sulfate dissolved in 600 ml. of water. The addition required 30 minutes and 6 *N* sodium hydroxide was added as necessary to maintain slight alkalinity. The mixture was let stand 3 hours, slowly heated until nitrogen evolution was completed, filtered, and acidified with 6 *N* hydrochloric acid. The pale yellow solid was filtered, washed, and dried to give 10.5 g. (45%) of the arsonic acid. Purification was accomplished through the sodium salt.

*Anal.* Calc'd for $C_{13}H_9AsO_4$: As, 24.6. Found: As, 24.9.

The monosodium salt was prepared as in the case of fluorenone-4-arsonic acid.

*1-Arsenosofluorenone.* The procedure used in the preparation of the 4-arsenoso compound was repeated. Yield was 95% of light yellow compound.

*Anal.* Calc'd for $C_{13}H_7AsO_2$: As, 27.7. Found: As, 27.9.

*Fluorenone-1-dichloroarsine.* The method of preparation was substantially the same as that used for fluorenone-4-dichloroarsine. The yield of yellow crystalline material was 85%; m.p. 138–140°.

*Anal.* Calc'd for $C_{13}H_7AsCl_2O$: As, 23.1. Found: As, 23.3.

*7-Nitrofluorenone-1-arsonic acid.* Direct nitration of fluorenone-1-arsonic acid was effected as in the case of fluorenone-4-arsonic acid. Yield of yellow product was 90%.

*Anal.* Calc'd for $C_{13}H_8AsNO_6$: As, 21.6. Found: As, 21.1.

*7-Nitrofluorenone-1-dichloroarsine.* Prepared as in the case of the 4-derivative, the yield of yellow crystals was 80%. The product melted at 215–217°.

*Anal.* Calc'd for $C_{13}H_6AsCl_2NO_3$: As, 20.3. Found: As, 20.0.

*7-Aminofluorenone-1-arsonic acid.* 7-nitrofluorenone-1-arsonic acid was reduced to the amine with ferrous hydroxide, using the procedure employed in the 4-arsonic acid series. A light brown compound was obtained in 55% yield.

*Anal.* Calc'd for $C_{13}H_{10}AsNO_4$: As, 23.5. Found: As, 23.1.

*7-Acetaminofluorenone-1-arsonic acid.* A 1.2-g. portion of 7-aminofluorenone-1-arsonic acid was treated at near reflux temperature for 2 hours with 10 ml. of acetic anhydride. Yield of the yellow-orange product was 0.6 g. (42%). Since purification through the ammonium or sodium salt seemed attended with hydrolysis, the compound was recrystallized from glacial acetic acid.

*Anal.* Calc'd for $C_{15}H_{12}AsNO_5$: As, 20.7. Found: As, 20.5.

*7-(Carbamylmethylamino)fluorenone-1-arsonic acid.* The compound was prepared as in the case of the corresponding 4-arsonic acid derivative. Yield of red-purple compound was 72%. For analysis, purification was effected through the ammonium salt.

*Anal.* Calc'd for $C_{15}H_{13}AsN_2O_5$: As, 19.8. Found: As, 19.7.

*The Bechamp reaction on 1-aminofluorenone.* One hundred grams of pure arsenic pentoxide was refluxed with 500 ml. of water for 1 hour. The mixture was cooled and filtered from the small amount of insoluble residue. The clear solution was evaporated with stirring until the concentrated liquor had transformed into a thick paste of crystals. The temperature was kept above 100° but was not permitted to rise above 115°. Upon cooling, the material solidified entirely, and was ground and dried at 110° for 4 hours. It was then quickly transferred to a tightly sealed container.

A 6-g. portion (0.0308 mole) of dry 1-aminofluorenone was placed in a 60-ml. Pyrex test tube and the tube immersed some 1½ inches in an oil-bath. The temperature was elevated to 145° and 3 g. of solid arsenic acid (0.0187 mole, calculated as $H_3AsO_4 \cdot H_2O$) was added over a period of 10 minutes with continual hand stirring. The temperature was then raised to 160–165° and maintained at that level for 2.5 hours. At the end of the first 15–20 minute period, the mixture had thickened considerably and only occasional stirring was then applied. Moisture, collecting on the upper walls of the test tube, was removed by insertion of rolled filter paper from time to time. The material was removed from the oil-bath, cooled, and extracted with four 15-ml. portions of 3 *N* sodium hydroxide. The combined deep yellow filtrates were cooled and acidified with 6 *N* hydrochloric acid until slightly acid. The precipitated orange-yellow aminoarsonic acid (probably 1-aminofluorenone-4-arsonic acid) was filtered off, washed, and dried to give 0.70 g. which corresponded to 11.7% yield based on weight of arsenic acid used. Purification as the sodium or ammonium salt yielded fractions which gave 3–5% low arsenic contents. A better purification was effected by the slow addition, with efficient stirring, of 1 *N* hydrochloric acid to a solution of the sodium salt until about one-third of the material had precipitated. This was then filtered off, and the precipitation was completed by the addition of more hydrochloric acid to the filtrate. The second fraction possesses an arsenic content that comes within 1–2% of the proper value.

## SUMMARY

1. Nineteen new fluorenone arsenicals have been described. The chemical structures of fourteen of these have been established. For the remaining five compounds, the probable structures have been indicated.

2. The Scheller reaction has been applied to the preparation of fluorenone-2-

arsonic acid with an improvement in yield. Acetone is recommended as the solvent for Scheller reactions in preference to alcohol or glacial acetic acid.

3. The Bechamp reaction has been applied, for the first time, to the fluorenone amines, resulting, in the case of 1-aminofluorenone, in a new arsenical.

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