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Derivatives of Fluorene. VI.[1] Fluorofluorenes I

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New fluorinated derivatives of fluorene, especially of N-2-fluorenylacetamide, have been prepared. These are of interest in cancer research, since blocking of various sites on this carcinogenic molecule with fluorine may lead to significant knowledge about its metabolism and the mechanism of chemical induction of neoplastic change.

In this paper we describe the synthesis of some fluorinated fluorene derivatives. Among these are two compounds, N-2-(5-fluorofluorenyl)-acetamide and N-2-(4-fluorofluorenyl)-acetamide, being used in a carcinogenicity study by Drs. J. A. and E. C. Miller, McArdle Memorial Laboratory, University of Wisconsin. These workers have elucidated the structural features of N-2-fluorenylacetamide necessary for carcinogenicity upon ingestion.[2] Recently, Weisburger and Weisburger,[3] and others have studied the metabolism of this substance. In the rat this compound is transformed to the 1-, 3-, 5-, 7- and 8-hydroxy derivatives. Possible involvement of one or more of these positions in the carcinogenic process is certainly a consideration.[4] On the other hand, based on present evidence, the 4- and 6-positions, apparently not involved in nuclear hydroxylation, cannot be eliminated from consideration as sites of interest in this connection.

Miller and Miller[5] introduced the concept and use of fluorine substitution as a means of detecting biologically active or inactive positions, and have pursued this concept in a study of the carcinogenicity of 4-dimethylaminoazobenzene. These investigators felt that fluorination of various positions in the N-2-fluorenylacetamide molecule would give substances of great value in carcinogenicity studies and reported[6] that N-2-(7-fluorofluorenyl)-acetamide had enhanced carcinogenicity for rat livers. Discussion of this led to collaboration in which, on the part of this Laboratory, it is intended to synthesize all of the remaining monofluoro as well as some difluoro derivatives.

In addition to N-2-(7-fluorofluorenyl)-acetamide, the only other ring substituted fluorofluorene derivatives previously reported are 2-fluorofluorene,[7] 2-fluoro-7-nitrofluorene,[6] 2-amino-7-fluorofluorene[6] and 2-fluorofluorenone.[7] These were made from derivatives with groups in known positions.

The most direct available route to N-2-(5-fluorofluorenyl)-acetamide was from 2,5-dinitrofluorene.[8] This can be monoreduced[9] to a mixture of the two possible aminonitrofluorenes, but there is only a small yield of the isomer which is the more useful for this purpose. Instead, we made 2,5-fluorenediamine[8] and monoacetylated to obtain N-2-(5-aminofluorenyl)-acetamide (established by deamination). Because the maximum yield of good 2,5-dinitrofluorene is $\sim$20%, it was decided to make 2-nitro-5-fluorenamine by slight modification of another known route[10] starting with diphenic acid (from anthranilic acid),[11] effecting cyclodehydration of the latter in polyphosphoric acid to fluorenone-4-carboxylic acid. Wolff–Kishner (Huang-Minlon modification) reduction of the 9-position followed by nitration and a Schmidt reaction gave the nitroamine in 40–43% over-all yields of good quality materials. The diazonium fluoroborate of this amine decomposed with no tar formation, giving 60% yields of 5-fluoro-2-nitrofluorene. Reduction and acetylation gave us the desired compound. 5-Fluoro-2-fluorenamine was deaminated giving 4-fluorofluorene with a melting point of 40°.

One preparation of the 2,4-isomer started with 5-fluoro-2-fluorenamine. Trifluoroacetylation followed by nitration gave N-7-(4-fluoro-2-nitrofluorenyl)-trifluoroacetamide[12] in moderate yield and an isomer, presumably the 6-nitro compound, in small yield. The former substance was hydrolyzed in dilute alkali and deaminated to give 4-fluoro-2-nitrofluorene. This was reduced and acetylated to give N-2-(4-fluorofluorenyl)-acetamide. Deamination of the 4-fluoro-2-fluorenamine gave 4-fluorofluorene identical with the compound obtained above.

The over-all yield of the 2,4-isomer being low, the following method was developed. Esterification of 2-chloro-3,5-dinitrobenzoic acid was followed by an Ullmann reaction with iodobenzene, carried out at a bath temperature of 140–200°. This is considerably lower than the reported temperature[13] and lower than usual for this type of reaction. Hydrolysis and cyclodehydration in polyphosphoric acid gave 2,4-dinitrofluorenone. The latter was reduced to the diamine by Raney nickel and hydrazine hydrate or, more conveniently for larger batches, with stannous chloride in hydrochloric

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(2) J. A. Miller, R. B. Sandin, E. C. Miller and H. P. Rusch, *Cancer Research*, **15**, 188 (1955).

(3) J. H. Weisburger, E. K. Weisburger and H. P. Morris, *Science*, **125**, 503 (1957). Review by Weisburger and Weisburger on N-2-fluorenylacetamide in J. P. Greenstein and A. Haddow (eds.), "Advances in Cancer Research, Vol. V," Academic Press, Inc., New York, N. Y., 1958, pp. 333–431.

(4) G. M. Bonser, *et al.*, *Brit. J. Cancer*, **6**, 412 (1952), *et seq.*

(5) J. A. Miller, E. C. Miller and G. C. Finger, *Cancer Research*, **13**, 93 (1953).

(6) J. A. Miller, R. B. Sandin, E. C. Miller and H. P. Rusch, *ibid.*, **15**, 188 (1955).

(7) E. Bergmann, H. Hoffmann, D. Winter, *Ber.*, **66**, 46 (1933). The oxime of 2-fluorofluorenone, 2,2′-difluorobifluorenylidene and 9,9′-dichloro-2,2′-difluorobifluorenylidene also were reported.

(8) T. L. Fletcher and M. J. Namkung, *J. Org. Chem.*, **23**, 680 (1958).

(9) J. H. Weisburger and E. K. Weisburger, *ibid.*, **21**, 514 (1956).

(10) E. K. Weisburger and J. H. Weisburger, *ibid.*, **20**, 1396 (1955).

(11) E. R. Atkinson, H. G. Lawler, J. C. Heath, E. K. Kimball and E. R. Read, THIS JOURNAL, **63**, 731 (1941).

(12) T. L. Fletcher and M. J. Namkung, manuscript in preparation.

(13) M. S. Lesslie and E. Turner, *J. Chem. Soc.*, 1760 (1930).

acid. Reduction then gave 2,4-fluorenediamine. Both of these diamines are new. By careful technique, as with 2,5-fluorenediamine, a high yield of N-2-(4-aminofluorenyl)-acetamide was obtained, which gave N-2-(4-fluorofluorenyl)-acetamide identical with the above substance obtained from 5-fluoro-2-nitrofluorene.

Experimental[14]

2-Nitro-5-fluorenediazonium Fluoroborate.—To a stirred suspension of 14.7 g. (0.065 mole) of 2-nitro-5-fluorenamine[10] in 60 ml. of 48% fluoroboric acid at 5–10°, 4.5 g. (0.065 mole) of sodium nitrite in 15 ml. of water was added in 30 minutes. Stirring was continued for 15 minutes and the yellow solid was separated on a sintered-glass funnel, washed with 35 ml. of cold fluoroboric acid, two 30-ml. portions of cold ethanol, and several times with ether. The compound was dried overnight on a clay plate at 1 mm. The yield was 19.1 g. (90.5%), dec. 96°.

5-Fluoro-2-nitrofluorene.—The above product (19.1 g.) was mixed with an equal volume of dried Ottawa sand and heated under an air condenser in an oil-bath, held at 80–85°, for 1.5 hours. During this time, the space over the sand was flushed by a gentle stream of air. The temperature then was raised to 125° for 45 minutes at which time fumes were no longer seen at the top of the condenser. The sand was extracted with acetone and the extract boiled with Darco, filtered, and concentrated to 85 ml. The cooled solution deposited 10.6 g. of crude product, m.p. 174–176° (some residue, m.p. ~ 230°), which was extracted with four 75-ml. portions of boiling benzene, leaving 1 g. of residue, m.p. ~ 230°. The benzene was evaporated to small volume and cooled. The crystalline product was washed sparingly with cold benzene and dried to give 8.6 g. (62%), m.p. 180.5–181.5°.

Anal. Calcd. for $C_{13}H_8FNO_2$: C, 68.12; H, 3.52; N, 6.11. Found: C, 68.18; H, 3.53; N, 6.21.

5-Fluoro-2-fluorenamine—Reduction of the preceding compound with Raney nickel and hydrazine hydrate[8] in toluene–ethanol gave 95% yields m.p. 84–87°. Recrystallization from methanol and then benzene gave an analytical sample, m.p. 87–87.5°.

Anal. Calcd. for $C_{13}H_{10}FN$: C, 78.37; H, 5.02; F, 9.57; N, 7.03. Found: C, 78.44; H, 5.03; F, 9.20; N, 7.07.

N-2-(5-Fluorofluorenyl)-acetamide.—5-Fluoro-2-fluorenamine (m.p. 86–87°) was acetylated quantitatively in benzene. The product, m.p. 191–192°, was crystallized from ethanol, m.p. 191.5–192°.

Anal. Calcd. for $C_{15}H_{12}FNO$: C, 74.67; H, 5.01; F, 7.87; N, 5.81. Found: C, 74.83; H, 5.25; F, 8.05; N, 5.84.

Alternatively, the reaction mixture from the above reduction, after filtration from the nickel,[8] was treated with acetic anhydride and warmed. The precipitate (85% from the nitro compound) required one recrystallization from toluene (Darco) to give material with m.p. 190.5–192°. A second crop (10%) was obtained by concentrating the filtrate with a few drops of acetic anhydride.

4-Fluorofluorene.—5-Fluoro-2-fluorenamine (0.6 g., 0.003 moles) was stirred in a mixture of 3.5 ml. of concentrated hydrochloric acid and 5.5 ml. of water and cooled in an ice-bath. A solution of sodium nitrite (0.22 g., 0.0032 mole) in 1.6 ml. of water was added dropwise (0.5 hour) and stirring was continued for a like period. Hypophosphorous acid (11 ml., 50%) was added in small portions in 5 minutes. The mixture was stirred at 0° for 2 more hours, kept at 0–1° for 24 hours, and diluted with water, filtered, and the precipitate washed and dried yielding 0.55 g. of crude product. Recrystallization of 0.15 g. from petroleum ether (b.p. 20–40°) followed by sublimation at 30–35° (0.001 mm.) gave 0.06 g. of white crystals, m.p. 39.5–40.5°.

Anal. Calcd. for $C_{13}H_9F$: C, 84.76; H, 4.93; F, 10.31. Found: C, 84.99; H, 4.97; F, 9.88.

N-2-(5-Fluorofluorenyl)-trifluoroacetamide.—The filtered reaction mixture from the reduction of 8.6 g. of 5-fluoro-2-nitrofluorene was treated with 1 equivalent (7.9 g.) of trifluoroacetic anhydride, heated on the steam-bath for 15 minutes and cooled. The precipitate was recrystallized from benzene. A yield of 93% (10.35 g.) from the nitro compound was recovered in the first crop, m.p. 199–200°.

Anal.[15] Calcd. for $C_{15}H_9F_4NO$: C, 61.02; H, 3.07; N, 4.74. Found: C, 61.35; H, 3.34; N, 4.84.

N-2-(5-Fluoro-7-nitrofluorenyl)-trifluoroacetamide.—The above product (10.35 g.) was dissolved in 150 ml. of hot glacial acetic acid which was then cooled to 45°. Nitric acid (8.5 ml., d. 1.42) was added followed by 1.5 ml. of concentrated sulfuric acid with swirling. The temperature was raised to 60–65° and in a few minutes, with continued agitation, the precipitated solids had gone back into solution. Soon yellow crystals came out. After cooling in an ice-bath, and filtering and washing of the precipitate with small portions of 50% acetic acid and then water, 10 g. of dried crude product was obtained, m.p. 191–232°. This was extracted with 350 ml. of hot benzene and the residue, m.p. 234–239°, was recrystallized from acetone giving 3.8 g. (32%) of N-2-(5-fluoro-7-nitrofluorenyl)-trifluoroacetamide, m.p. 239.5–240.5°. The benzene extract after standing 2 days, had deposited more of the product which, when recrystallized from benzene and then acetone, gave 1 g., m.p. 239–240.5°. Analytical material was obtained from the first crop by one more recrystallization from benzene, m.p. 241–241.5°.

Anal. Calcd. for $C_{15}H_8F_4N_2O_3$: C, 52.95; H, 2.37; N, 8.23. Found: C, 52.87; H, 2.39; N, 8.28.

N-2-(5-Fluoro-x-nitrofluorenyl)-trifluoroacetamide.—The remainder of the above extract was concentrated and the crude crop was recrystallized from acetone to give 1.4 g. (11%). After one more recrystallization this melted at 201.5–202.5°.

Anal. Calcd. for $C_{15}H_8F_4N_2O_3$: N, 8.23. Found: N, 8.24.

5-Fluoro-7-nitro-2-fluorenamine.—To a boiling solution of 3.8 g. (0.011 mole) of N-2-(5-fluoro-7-nitrofluorenyl)-trifluoroacetamide in 70 ml. of ethanol, 70 ml. of boiling 1% aqueous sodium hydroxide was added. Boiling was continued for 5 minutes and precipitated red solids were filtered off and washed with water. Recrystallization from acetone gave 2.1 g. (70%), m.p. 214–215°.

Anal. Calcd. for $C_{13}H_9FN_2O_2$: N, 11.47. Found: N, 11.19.

5-Fluoro-x-nitro-2-fluorenamine.—The above (x-nitro) product was hydrolyzed to give a product melting at 226–228°, presumably the corresponding amine. An attempt to deaminate this failed to give a workable product.

4-Fluoro-2-nitrofluorene.—5-Fluoro-7-nitro-2-fluorenamine (2.1 g., 0.0086 mole) was added slowly with stirring to a mixture of 0.8 g. (0.01 mole) of sodium nitrite in 28 ml. of concentrated sulfuric acid and 2 ml. of water at -5°. After 30 minutes at this temperature, with continuous stirring, 60 ml. of 50% hypophosphorous acid at 0° was added at such a rate that the temperature did not go above 5°. The mixture was kept at 3° for 2.5 days and diluted with water. The precipitate was dried (1.5 g.), m.p. ~ 80° dec., and the filtrate was warmed on the steam-bath for 30 minutes and filtered again to recover 0.45 g. of light tan precipitate, m.p. 145–160°. The 1.5 g. was digested in water on the steam-bath, and a small amount of sodium bicarbonate was added to neutralize the acidic mixture. After filtration and drying, 1.2 g. was recovered. This was extracted with ether and the residue (m.p. 130–150°) recrystallized from alcohol to give 0.7 g. which, together with the 0.45 g. recovered from the original filtrate, was sublimed at 120° and 1 mm., yielding 0.4 g. (20%), m.p. 156–157°.

Anal. Calcd. for $C_{13}H_8FNO_2$: N, 6.11. Found: N, 6.13.

4-Fluoro-2-fluorenamine.—In a typical experiment, 0.2 g. of 4-fluoro-2-nitrofluorene was dissolved in 20 ml. of ethanol and reduced with a small amount of Raney nickel and 0.1 ml. of hydrazine hydrate (100%)[8] by boiling gently, replacing the loss with hot ethanol. No alkaline vapors were detected at the end of 15 minutes and the nickel was filtered

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(14) Melting points were taken on a Fisher–Johns apparatus and are corrected to standards. Microanalyses were done by Schwarzkopf Microanalytical Laboratory, Woodside, N. Y., or by W. Manser, Herrliberg, Switzerland.

(15) The analytical sample was taken from another run and had been recrystallized from ethanol, m.p. 201–201.5°.

off. The colorless solution was boiled down to give a first crop (0.13 g.) of white crystals (71%), m.p. 121–122°. Recrystallization from alcohol gave a sample with m.p. 122–122.5°.

Anal. Calcd. for $C_{13}H_{10}FN$: C, 78.37; H, 5.02; N, 7.03. Found: C, 78.45; H, 4.72; N, 7.22.

A second crop was recovered from the liquor. Acetylation without isolation of the amine led to well over 90% from the nitro compound.

N-2-(4-Fluorofluorenyl)-acetamide.—To 0.03 g. of the above amine 4 drops of acetic anhydride was added. After warming for 10 minutes, the white solid was taken up in a few ml. of ethanol (Darco), filtered and boiled down, adding 1 ml. of hot water, until crystals began to come out. The recovered amide weighed 0.03 g. (80%), m.p. 196–198.5°.

Anal. Calcd. for $C_{15}H_{12}FNO$: C, 74.67; H, 5.01; F, 7.88, N, 5.81. Found: C, 74.47; H, 5.22; F, 7.75; N, 5.99.

Ethyl 2,4-Dinitrodiphenyl-6-carboxylate.—Ethyl 2-chloro-3,5-dinitrobenzoate[13] (73 g., 0.27 mole) was coupled with iodobenzene (100 g., 0.49 mole) by modification of the Ullmann procedure.[13] Activated copper powder[16] (61.5 g.) was added over a period of 25 minutes to the rapidly stirred reactants under reflux while raising the bath temperature from 140–180°. The temperature then was raised gradually (1 hour) to 200°. This was worked up[13] to give a first crop from ethanol of 58 g. (69%), m.p. 83–84.5°, and a second crop of 1.8 g., m.p. 78.5–81.5°. Experience with this reaction had showed us that the temperature reported (250°)[13] gave considerable tar and lower yields.

2,4-Dinitrofluorenone.—2,4-Dinitrodiphenyl-6-carboxylic acid (12.5 g.), from hydrolysis (79–83.5%) of the above ester, was mixed with 90 g. of polyphosphoric acid and heated with occasional stirring in an oven maintained at 150–160° for one hour. The mixture was cooled somewhat and poured over ice. Vigorous stirring gave a finely divided greenish-yellow solid. The dried product weighed 11.1 g. (95%), m.p. 194.5–196.5°. Recrystallization from acetone gave 9.2 g. melting at 196–196.5° (lit.[13] 197°). Larger batches required a longer cyclization time.

2,4-Diaminofluorenone.—A mixture of 76.2 g. (0.282 mole) of 2,4-dinitrofluorenone, 510 g. (2.26 moles) of stannous chloride dihydrate, 235 ml. of ethanol and 530 ml. of concentrated hydrochloric acid in a 4-l. beaker was heated to boiling (caution!), then removed from the heat until the vigor of the reaction had subsided somewhat. It was then boiled for 10 minutes and cooled. The solids were filtered off, and washed with dilute alkali and water. After drying, recrystallization from 1:1 toluene–ethanol gave 55 g. (93%) of bronze-red 2,4-diaminofluorenone, m.p. 221–222°. Recrystallization from the same solvent raised the m.p. to 221.5–222.5°.

Anal. Calcd. for $C_{13}H_{10}N_2O$: C, 74.27; H, 4.80; N, 13.33. Found: C, 74.28; H, 4.96; N, 13.51.

2,4-Fluorenediamine.—To a solution of 30 g. of sodium hydroxide in 320 ml. of diethylene glycol, 45 g. of 2,4-diaminofluorenone and 30 ml. of 85% hydrazine hydrate were added successively and the solution refluxed for 2.5 hours at a bath temperature of 180° and a further 3 hours at 200°. The mixture was cooled and poured into 3 l. of water, filtered and washed. Recrystallization from benzene (Darco) gave 39.4 g. (94%) of glistening white crystals, m.p. 162–163°. Yields ranged from 85–94%.

Anal. Calcd. for $C_{13}H_{12}N_2$: C, 79.56; H, 6.16; N, 14.28. Found: C, 79.81; H, 6.18; N, 14.57.

N,N′-Diacetyl-2,4-fluorenediamine.—To a solution of 0.08 g. of 2,4-fluorenediamine in 2 ml. of pyridine, 4 drops of acetic anhydride was added. The mixture was heated on the steam-bath for 30 minutes, cooled, and stirred vigorously into dilute hydrochloric acid. The white precipitate was dried to give 0.11 g. (~90%), m.p. 290–295.° Two recrystallizations from ethanol gave an analytical sample, m.p. 293–295° (slight softening at 291°).

Anal. Calcd. for $C_{17}H_{16}N_2O_2$: C, 72.84; H, 5.75; N, 9.99. Found: C, 72.40; H, 5.68; N, 9.75.

N-2-(4-Aminofluorenyl)-acetamide.—To a beaker containing a solution of 10 g. (0.051 mole) of 2,4-fluorenediamine in 1 l. of benzene at 60° was added 5 g. (0.049 mole) of acetic anhydride in 50 ml. of benzene dropwise, with thorough mixing on a magnetic stirrer, over a period of 30 minutes. After 10 minutes the temperature had reached 52–54° and a white precipitate began to come out. At the end of the addition, the precipitate was separated and dried yielding 11.3 g., m.p. 202–210°. Recrystallization from ethanol, and filtration when the alcohol had cooled to 35°, gave 6.8 g. (52%), m.p. 209–211°. A sample recrystallized from ethyl acetate showed no change in m.p.

Anal. Calcd. for $C_{15}H_{14}N_2O$: C, 75.60; H, 5.92; N, 11.76. Found: C, 75.67; H, 5.95; N, 11.49.

The residues from evaporation of the benzene and ethanol filtrates were boiled for 5 hours in a 10% solution of concentrated hydrochloric acid in ethanol. The precipitated diamine dihydrochloride was filtered off and taken up in dilute sodium hydroxide and again filtered to give a complete recovery (4.8 g.) of diamine. The latter then was recycled in a 10- or 15-g. batch. The yield in a series of reactions approached 100%.

Deamination of N-2-(4-Aminofluorenyl)-acetamide.—A mixture of 0.8 g. (0.0034 mole) of N-2-(4-aminofluorenyl)-acetamide, 4 ml. of concentrated hydrochloric acid and 8 ml. of water was stirred at room temperature for 30 minutes and then in an ice-bath until the temperature dropped to 0–3°. A solution of 0.26 g. (0.0038 mole) of sodium nitrite in 1.6 ml. of water was added dropwise during 30 minutes and stirring was continued for an equal period, following which 12 ml. of 50% hypophosphorous acid was added in small portions during 5 minutes. After stirring at 0–3° for a further 2 hours the mixture was left at 1° for 15 hours. A 5-ml. portion of hypophosphorous acid was then mixed in and the 1°-temperature was maintained for 9 more hours. After water dilution and neutralization with ammonium hydroxide, the mixture was filtered. The washed and dried precipitate amounted to 0.7 g. Recrystallization from methanol gave 0.51 g. (75%) of white crystals, m.p. 194–195°. The mixture m.p. with authentic N-2-fluorenylacetamide was undepressed.

2-Acetamidofluorene-4-diazonium fluoborate was prepared in the usual way to give a 97% yield, dec. 90°.

N-2-(4-Fluorofluorenyl)-acetamide.—The above salt (31.4 g.) was mixed with 3–4 times its volume of Ottawa sand and heated in an oil-bath at 80° for 7 hours under a water condenser which collected some sublimed product. The decomposition was carried out under a water aspirator vacuum. Two batches were combined and extracted with acetone which was concentrated to 75 ml. This was extracted with benzene leaving a dark tar. The latter was taken up in 30 ml. of acetone and re-extracted with benzene. The two extracts were combined and boiled down to 600 ml. Upon cooling, 26 g. of light brown crystals separated, m.p. 175–190°. Solution in ethyl acetate and filtration left 1 g. of high-melting residue. The filtrate was boiled with Darco, filtered, and evaporated to 250 ml. A first and second crop were combined to give 22 g. of crude product m.p. 196–199°, with previous melting and resolidification at about 180°. The mixture m.p. with the product obtained by reduction and acetylation of 4-fluoro-2-nitrofluorene was undepressed.

Hydrolysis in ethanolic sodium hydroxide gave 4-fluoro-2-fluorenamine identical with the product obtained above, mixture m.p. 124–124.5°.

N-2-(5-Aminofluorenyl)-acetamide.—This monoacetylation was performed in the same manner as for the 2,4-isomer above, m.p. 237.5–239°. Proof of structure was realized by deamination to give a substance, m.p. 191–195°, which mixed with authentic N-2-fluorenylacetamide (m.p. 195°) melted at 192–195°.

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[16] E. C. Kleiderer and R. Adams, THIS JOURNAL, **55**, 4219 (1933). The procedure should be followed explicitly. Prolonged drying on the funnel lowered yields by 20%.