Chemical Carcinogenesis: Syntheses of 2,7-Bis-(Acetamido) Fluorenes with Increased Molecular Thickness by 9-Substitution

Fishel, D. L.; Kletecka, G.; Muralidhara, R.
CHEMICAL CARCINOGENESIS; SYNTHESIS OF 2,7-BIS-(ACETAMIDO)FLUORENES WITH INCREASED MOLECULAR THICKNESS BY 9-SUBSTITUTION

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ABSTRACT

The series: 9-methyl, 9-isopropyl, 9-cyclohexyl, 9,9-dimethyl, 9,9-diethyl, and the unsubstituted parent 2,7-bis(acetamido)fluorene were synthesized from the corresponding 9-substituted fluorenes. The two routes employed consisted of either (1) dinitration, reduction to the diamine, and diacetylation, or (2) Friedel-Crafts acylation, then rearrangement via the Schmidt reaction. This provides a series of compounds of use in comparing relative carcinogenicity with changes in chemical reactivity as a function of increased steric hindrance due to altered molecular "thickness."

Preliminary to a study of the donor characteristics in charge-transfer complex formation of potentially carcinogenic compounds, we have synthesized a series of 9-substituted-2,7-bis(acetamido)fluorenes. The parent, 2,7-bis(acetamido)fluorene (fig. 1) should be coplanar by analogy with the hydrocarbon, fluorene (Brown and

Figure 1. 2,7-bis (acetamido) fluorene.

H3CCHN

O

H

O

NHCCCH3

Manuscript received March 1, 1969.

This work is in part derived from the M.S. theses of G. Kletecka, K. S. U. 1963, and R. Muralidhara, K. S. Y. 1965.

Bortner, 1954). Substitution at the 9-position would be expected both to effectively increase molecular thickness and to impose a slight dihedral angle between the planes of the benzene rings. Each of these factors ought to decrease the reactivity for charge-transfer complex formation, assuming a “face-to-face” complex with aromatic electron acceptors.

Of principal interest was a series with increasing steric size of the 9-substituent without great alteration of electron-donor capacities. Chosen for this purpose were the following series of 9-substituents: -hydrogen, -methyl, -ethyl, -isopropyl, -tert-butyl. In addition, the 9,9-dimethyl and 9,9-diethyl-2,7-bis(acetamido) fluorenes were desirable for comparison of the relative contributions of “front” versus “back-side” alignment of the mono-substituted compounds with an electron acceptor. Attempts were also made to prepare a series with 9-substituents varying in electronic character, viz., -alkyl, -halogen, -nitrile or nitro; these latter compounds could not be prepared by the approaches used.

Direct substitution reactions at the 9-position of Figure 1 were considered impractical, owing to its low solubility in most solvents. Accordingly, 9-substituted fluorenes were prepared and converted to the 2,7-bis(acetamido) derivatives by either of two alternate routes: (1) dinitration, reduction, and acetylation, or (2) Friedel-Crafts di-acylation and rearrangement via the Schmidt reaction.

There are many reports of 9-alkylation of fluorene (Schoen and Becker, 1956; Murray and Fritz, 1965; Colonge and Bonnard, 1958; Scherf and Brown, 1961). However, very few methods both give satisfactory yields and are also applicable to substitution with tertiary alkyl groups. The method of Brown and Bluestein (1943) generally gives yields of 50–75% and is applicable to alkylation with both primary and secondary groups. We have also now prepared 9-tert-butylfluorene in 31% yield by this method. Sodium hydride in toluene is as satisfactory as sodium ethoxide in ethanol for the preparation of 9-formylfluorene in the first step of this sequence. A potentially superior method for preparation of 9-tert-butylfluorene is that of Anet and Bavin (1956), who alkylated methyl fluorene-9-carboxylate with tert-butyl bromide in basic solution (94%). This was saponified and decarboxylated in potassium hydroxide/ethylene glycol. However, this conversion was only accomplished with 0.3 g, although in 46% yield.

Bavin’s (1960a) synthesis of 9,9-dimethylfluorene via fluorenyllithium was successfully repeated and was extended to 9,9-diethylfluorene. The yields were maximized at 80–85% by employing low temperatures and reaction times sufficient (overnight) to discharge completely the color of the initially produced fluorenyllithium.

Preparation of 9-alkyl-2,7-dinitrofluorenes has previously been accomplished by Wawzonek, Dufek, and Sial (1956) with fuming nitric acid in acetic acid. This method is satisfactory if the temperature is carefully controlled at 0°C during addition of nitric acid. The reaction was complicated when higher initial temperatures were employed. More than two isomeric dinitro derivatives are formed; at least one of these has a labile nitro group. When these crude mixtures were heated during attempts to recrystallize them, nitrogen dioxide was evolved. The 2,5-dinitro isomer which accompanies the 2,7-dinitro product from reactions at 0°C is more soluble in most solvents used for recrystallization and can thus be easily removed. The yield and analytical data for the preparation of the 2,7-dinitrofluorenes in our laboratory is presented in Table I.

Reduction of the dinitro derivatives to diamines was accomplished with ethanolic hydrazine over a carbon-supported palladium catalyst (Bavin, 1960b). This method is recommended by us over any other (chemical reduction or hydrogenation over nickel catalysts gave intractable mixtures); it requires no special apparatus, is rapid, and gives high yields for reduction of all but highly hindered nitroaromatics. Although Bavin reduced 2-nitrofluorene with hydrazine hydrate, we found it necessary to employ anhydrous hydrazine in absolute ethanol to effect
<table>
<thead>
<tr>
<th>Alkyl Substituents</th>
<th>Yield, %</th>
<th>M.P. °C</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
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<tr>
<td>9-Methyl-</td>
<td>33.4</td>
<td>246-48°</td>
<td>62.22</td>
<td>62.36</td>
<td>3.70</td>
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<tr>
<td>9-isoPropyl-</td>
<td>55.5</td>
<td>183-5°</td>
<td>64.45</td>
<td>64.70</td>
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<td>25.3</td>
<td>192-200°</td>
<td>d</td>
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<td>9-Cyclohexyl-</td>
<td>48.0</td>
<td>225-227°</td>
<td>67.44</td>
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<td>5.36</td>
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<td>9,9-Dimethyl-</td>
<td>74.0</td>
<td>310-311°</td>
<td>63.39</td>
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<td>9,9-Diethyl-</td>
<td>70.0</td>
<td>213-214°</td>
<td>63.37</td>
<td>63.41</td>
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<tr>
<td>9-Bromo-</td>
<td>35.0</td>
<td>250-260°</td>
<td>53.86</td>
<td>53.82</td>
<td>2.45</td>
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<tr>
<td>9-Chloro</td>
<td>60.0</td>
<td>202-203°</td>
<td>53.86</td>
<td>53.82</td>
<td>2.45</td>
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*Recrystallized from ethyl acetate; Bavin (1960a) reports m.p. 246-247°.
*Recrystallized from ethanol.
*Recrystallized from carbon tetrachloride; m.p. obtained in a sealed tube.
*Amount of sample insufficient for elemental analysis; mass spectrum shows parent ion and no appreciable contamination with unsubstituted 2,7-dinitrofluorene.
*Recrystallized from glacial acetic acid.

complete reduction of the dinitrofluorenes. Only 2,7-dinitro-9-tert-butylfluorene could not be reduced in this way to the diamine, presumably due to steric interference towards adsorption on the catalyst surface. The diamines were sensitive to atmospheric oxidation and were converted at once to the diacetyl derivatives.

The elemental analysis of only one derivative, 9-cyclohexyl-2,7-bis(acetamido)fluorene, was not within acceptable limits. That this was an anomaly (either poor

<table>
<thead>
<tr>
<th>9-Substituents</th>
<th>Yield, %</th>
<th>M.P. °C</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
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<td>Calc'd</td>
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<tr>
<td>9-Methyl-</td>
<td>51</td>
<td>308.5-309.5</td>
<td>73.44</td>
<td>73.45</td>
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<td>9-isoPropyl</td>
<td>80</td>
<td>309-310</td>
<td>74.55</td>
<td>74.50</td>
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<td>9-Cyclohexyl</td>
<td>88</td>
<td>271.5-272.5°</td>
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<td>9,9-Dimethyl-</td>
<td>85</td>
<td>315-316°</td>
<td>73.98</td>
<td>73.81</td>
<td>6.54</td>
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<td>9,9-Diethyl-</td>
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<td>9-Chloro</td>
<td>90</td>
<td>285-286°</td>
<td>8.33</td>
<td>8.21</td>
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<tr>
<td>Method B</td>
<td>9-Methyl</td>
<td>63</td>
<td>306-308</td>
<td>9.51</td>
<td>9.51</td>
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<td></td>
<td>9,9-Diethyl</td>
<td>91</td>
<td>255-256</td>
<td>8.74</td>
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</tr>
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</table>

*Monoalkyl derivatives were recrystallized from ethanol or chlorobenzene, m.p.'s were determined in sealed capillaries.
*By reduction of 2,7-dinitro derivatives and acetylation; yields are based on dinitro derivatives.
*Analysis by mass spectrometry; Table III.
*Recrystallized from glacial acetic acid; open tube m.p.
*Hydrogenolysis of 9-halo groups occurred concurrently.
*Schmidt reactions on 2,7-diacetyl derivatives, mixture melting points with authentic material were undepressed; ir spectra, identical.
analytical sample preparation or bad analysis) was shown by confirming the sample identity by mass spectroscopic analysis. There were no peaks above mass 364; the relative ratios for the peaks at m/e 362, 363, and 364 show good agreement with those calculated for contributions due to natural isotope abundance ratios for the formula, C_{23}H_{26}N_{2}O_{2} (molecular ion, m/e 362). Strong fragment peaks at m/e values of 280, 238, and 196, corresponding to loss of C_{6}H_{10}, and successively of two CH_{2}CO fragments, are additional proof of the composition of this substance. Each of these fragmentations occurs by elimination with concomitant 1–3 proton transfer to the 2,7-diaminofluorene moiety. All other peaks are also consistent with the assigned formula. The complete spectrum is given in Table III.

### Table III

<table>
<thead>
<tr>
<th>m/e</th>
<th>Relative Intensity (% of m/e 362)</th>
<th>m/e</th>
<th>Relative Intensity (% of m/e 362)</th>
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<tr>
<td>364</td>
<td>3.4</td>
<td>222</td>
<td>2.6</td>
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<td>363</td>
<td>26.2</td>
<td>221</td>
<td>2.8</td>
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<td>362</td>
<td>100.0</td>
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<td>320</td>
<td>3.3</td>
<td>196</td>
<td>21.0</td>
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<tr>
<td>319</td>
<td>3.0</td>
<td>195</td>
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<td>281</td>
<td>10.0</td>
<td>194</td>
<td>4.3</td>
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<tr>
<td>280</td>
<td>54.3</td>
<td>180</td>
<td>3.2</td>
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<tr>
<td>279</td>
<td>7.4</td>
<td>168</td>
<td>6.5</td>
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<tr>
<td>239</td>
<td>6.9</td>
<td>167</td>
<td>14.2</td>
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<tr>
<td>238</td>
<td>34.7</td>
<td>57</td>
<td>7.6</td>
</tr>
<tr>
<td>237</td>
<td>6.7</td>
<td>55</td>
<td>28.4</td>
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</table>

This first route for preparation of 2,7-bis(acetamido)-fluorenes with other than alkyl 9-substituents was unsatisfactory. For example, both 9-bromo and 9-chlorofluorene were readily converted to 2,7-dinitro derivatives, but the halogen was replaced by hydrogen during reduction. Attempted nitration of either 9-methoxy or 9-carbethoxyfluorene gave mixed isomers that were not separable (fractional recrystallization and column chromatography were tried).

The 2,7-diacylfluorenes, available by Friedel-Crafts diacylation of 9-substituted fluorenes, should be convertible to the corresponding 2,7-bis(acetamido)-fluorene via either Beckmann rearrangement of the bis-oximes or directly by Schmidt reaction. The latter route was successfully employed by us for preparation of the parent 2,7-bis(acetamido)fluorene in good purity and high overall yield. The 9-methyl and 9,9-diethyl derivatives were also prepared as examples of the generality of this route for 9-alkyl-2,7-bis(acetamido)fluorenes.

The best of several techniques used in an attempt to prepare the required diacetyl derivatives essentially free of the monoacetyl compounds is a modification of the method of Dashevskii and Shamis (1964). This employs an acylation mixture previously prepared from acetic anhydride and aluminum chloride dissolved in 1,1,2,2-tetrachloroethane (TCE). However, attempted Friedel-Crafts acylation of fluorenes with 9-halo or 9-alkyloxy substituents gave amorphous high-melting substances.

The specific use of sulfuric acid in the concentration range 80–85% as solvent
and catalyst for Schmidt reactions of this study is based on the observation that maximum reaction rates and conversions of methyl aryl ketones into N-arylacetamides occurs for solvent acidities in this intermediate range (Fishel and Maximovich, 1964). In $\geq 85\%$ sulfuric acid, more than 90% of the ketone is converted to its conjugate acid (Stewart and Yates, 1958). At lower acidities, conversion rates are lower because concentrations of this species are lower. At acidities much greater than 85% sulfuric acid, the concentration of the other reactant, hydrazoic acid in the form of its free base, is effectively diminished by protonation and by unimolecular decomposition. An alternate explanation that has been given (Fishel and Maximovich, 1964) for diminished reaction rates in concentrations of sulfuric acid greater than 85% is the dehydration of the reactive complex (for rearrangement) formed from the ketone-conjugate acid and hydrazoic acid. The dehydrated species was then presumed either not to rearrange or to do so at a much reduced rate. Experiments in which 2,7-diacetylfluorene was subjected to Schmidt reactions over a range of solvent acidities demonstrated considerably lower reaction rates in sulfuric acid with acidities both lower and higher than $\approx 85\%$. Yields of 2,7-bis(acetamido)fluorene were much diminished for reactions of an equivalent amount of hydrazoic acid with 2,7-diacetylfluorene in concentrated sulfuric acid. In the latter experiments, an amount of nitrogen equivalent to the hydrazoic acid used was collected.

One other observation is worthy of note. The reaction of 9-bromofluorene with an equivalent amount of sodium cyanide in DMSO gave, not the expected 9-cyanofluorene, but 9-cyano-9-(9-fluorenyl)fluorene. This probably was formed by initial conversion of part of the 9-bromofluorene to 9-cyanofluorene, a stronger acid than fluorene. Cyanide as base would facilitate loss of the proton and the 9-cyano-9-fluorenyl anion would behave as a nucleophile toward the remaining 9-bromofluorene.

The infrared spectra (KBr pellet) of all 9-alkyl-2,7-bis(acetamido)fluorenes were very similar, differing only as expected for C–H stretching and deformation absorptions.

**EXPERIMENTAL DATA**

**Materials**

Fluorene (Eastman Organic Chemicals) was recrystallized (2X) from petroleum ether (b.p. 110–120°) to a melting point of 115–116°. 9-Chlorofluorene (Aldrich Chemical Company) was purified by elution chromatography using alumina (Fisher, adsorption grade) and petroleum ether, then recrystallized from petroleum ether; m.p. 90–91°. All other reagents and solvents were used as obtained from commercial sources without further purification.

**9-Formylfluorene**

This was synthesized by a modification of the method used by Brown and Bluestein (1943), except that sodium hydride was used as the condensing agent instead of an ethoxide, and in toluene as solvent. The yield by this procedure was 62.3% of purified 9-formylfluorene b.p. 160–163° (2.4 mm). This is comparable
to the yield (65%) reported by Brown and Bluestein when they used sodium ethoxide as the condensing agent in ether solution.

9-Alkylfluorenes

These compounds were prepared by the method of Brown and Bluestein (1943), using the appropriate alkyl halide with 9-formylfluorene and potassium hydroxide in water. Melting points and yields were: 9-methylfluorene, m.p. 45-46°, 50.8%; 9-isopropylfluorene, m.p. 54-55°, 60%; 9-cyclohexylfluorene, m.p. 114-115°, 44%; 9-tert-buty1fluorene, m.p. 97-98°, 31.4%; lit. (Anet and Bavin, 1956) m.p. 101.5°. 9-Tert-butylfluorene had not previously been prepared by this method.

9,9-Dimethylfluorene

This compound was prepared by the method of Bavin (1960a). Best results were obtained by preparing fluorenyllithium at temperatures below 5° before addition of iodomethane. Recrystallization (3X) from ethanol (initially in a dry ice-acetone bath) gave colorless 9,9-dimethylfluorene (85%) m.p. 94-95° lit. (Bavin, 1960a) m.p. 95-96°.

9,9-Diethylfluorene

This compound was prepared in a manner similar to that for 9,9-dimethylfluorene, but in 80% yield, m.p. 29-30°, lit. (Greenow and McNeil, 1956) m.p. 29-30°.

9-Bromofluorene

Fluorene (33.2 g, 0.2 mole) in 250 ml of benzene was heated under reflux with N-bromosuccinimide (36 g, 0.2 mole) for six hours. Succinimide was removed after cooling and the filtrate washed with sodium bicarbonate solution, then dried over magnesium sulfate. Unreacted fluorene was removed from the residue by elution chromatography on alumina with petroleum ether (b.p. 65-80°). The product was recrystallized from petroleum ether to give pure 9-bromofluorene (25 g, 50%), m.p. 102-103° lit. (Fuson and Porter, 1948) m.p. 103-104°.

9-Cyano-9-(9-fluorenyl)fluorene

This compound was obtained during attempted preparation of 9-cyanofluorene. A mixture of 9-bromofluorene (12.25g, 0.05 mole) and sodium cyanide 2.5 g, 0.051 mole) in dimethyl sulfoxide (50 ml) was stirred with heating to 150-180° for six hours. The crude reaction mixture was decomposed in ice and excess ferric chloride solution was added to complex unused cyanide. The solid was collected and washed with water, dissolved in chloroform and extracted with water and with 5% hydrochloric acid (2X). The solution was dried over sodium sulfate and the solvent stripped. The residue was recrystallized from benzene-heptane after decolorization with charcoal, giving colorless prisms (10.6 g, 0.03 mole, 60%) characterized as 9-cyano-9-(9-fluorenyl)fluorene, m.p. 228-230° (sealed tube). The ir spectrum (KBr disk) showed a sharp peak at 2238 cm⁻¹ (-C≡N). The nmr spectrum in deuteriochloroform had a complex aromatic pattern from 2.3-3 r and a singlet at 5.15 with relative intensities of 16:1.


2,7-Dinitrofluorene

This was prepared by direct nitration of the hydrocarbon with fuming nitric acid in glacial acetic acid at 5-10° similar to the method reported by Wawzonek, Dufek, and Sial (1956) for nitration of 9-alkylfluorenes. Trituration of the crude isomeric mixture (with hot cyclohexane) followed by sequential recrystallization from acetic acid and nitrobenzene gave pure 2,7-dinitrofluorene m.p. 295-300°d., lit. (Anantakrishnan and Hughes, 1935) m.p. 295-300°d.
All 9-monomonosubstituted derivatives listed in Table I were prepared in a manner similar to that for 2,7-dinitrofluorene, except that the nitration mixture was warmed to 50° after addition of the nitric acid. The 9,9-dimethylfluorene and 9,9-diethylfluorene were nitrated at —5° with fuming nitric acid in acetic anhydride as solvent. It was necessary to exercise careful control of the temperature to prevent explosive decomposition during nitration under these latter conditions.

2,7-Diaminofluorene

The method of reducing the nitro derivatives listed in Table I is adapted from that of Bavin (1960b) (see also Fieser and Fieser, 1967). Hydrazine (95% + anhyd.) was added slowly to a refluxing suspension of 2,7-dinitrofluorene (10 g, 0.04 mole) and one gram of 8% palladium on carbon in 200 ml. absolute ethanol. A red color developed which gradually faded to straw-yellow as more hydrazine was added. Fresh catalyst (about 0.1g) was added several times during the reduction to maintain vigorous reaction. When reduction was complete, the catalyst was removed by filtration and the solution poured into ice water. 2,7-Diaminofluorene, precipitated as a crude white solid, was filtered, washed with water, and converted to its acetyl derivative without further purification. All of the dinitro derivatives listed in Table I were reduced in the same way and acetylated immediately as described below for 2,7-bis(acetamido)fluorene (Method A).

2,7-Bis(acetamido)fluorene (Method A)

Acetylation of 2,7-diaminofluorene and all of the 9-alkylsubstituted derivatives was accomplished by a standard technique involving rapid successive addition of acetic anhydride and sodium acetate buffer to a water solution of the amine hydrochloride. Recrystallization from ethanol/water and then chlorobenzene gave pure 2,7-bis(acetamido)fluorene (8.9 g, 75%), m.p. 285-286° (sealed tube), lit. (Morgan and Thomason, 1926) 281-282°.

Anal.—Calcd. for C_{17}H_{16}O_{2}N_{2}: C, 72.85; H, 5.75; N, 9.99. Found: C, 72.84; H, 5.85; N, 9.95.

2,7-Diacetylfluorene

One hundred milliliters of an acetylation mixture (Dashevskii and Shamis, 1964) (4.5 equivalents), containing acetic anhydride (16 ml) and aluminum chloride (45 g) in 1,1,2,2-tetrachloroethane (TCE), were added in ten minutes to fluorene (6.0 g, 0.036 mole) in TCE (50 ml) at 20-25° with stirring. After stirring for three hours at 25° and ten minutes on a steam bath, the complex was decomposed with ice and hydrochloric acid, then steam distilled. The solid residue was collected and crystallized from ethanol with decolorization (2X) to give 2,7-diacetylfluorene (6.38 g, 0.026 mole, 71%), m.p. 182-183°, lit. (Ishikawa and Ozawa, 1960) m.p. 183-184°.

Both 9-methyl- and 9,9-diethylfluorene were similarly converted to the 2,7-diacetyl derivatives in 51% and 67% yields respectively. 2,7-Diacetyl-9-methylfluorene had m.p. 127-129° (needles from ethanol).

Anal.—Calcd. for C_{18}H_{16}O_{2}: C, 81.79; H, 6.10. Found: C, 81.99; H, 5.88.

2,7-Diacetyl-9,9-diethylfluorene had m.p. 155-158° (iridescent plates from methanol).

Anal.—Calcd. for C_{21}H_{22}O_{2}: C, 82.32; H, 7.24. Found: C, 82.27; H, 7.19.

2,7-Bis(acetamido)fluorene (Method B)

2,7-Diacetylfluorene (1.82 g, 0.0073 mole) was dissolved with stirring in 45 ml sulfuric acid (85.0%), and sodium azide (1.00 g, 0.015 mole) was added over four hours in portions. The mixture was heated (60°) for 1-2 hours, drowned on ice mixed with excess sodium hydroxide, filtered, washed, and dried, giving crude 2,7-bis(acetamido)fluorene. Recrystallization from N,N-dimethylformamide-
water gave light-yellow crystals of pure 2,7-bis(acetamido)fluorene (1.8 g, 90%), m.p. 285–86°, lit. (Morgan and Thomason, 1926) 281–282°.

The 2,7-diacetyl derivatives of 9-methyl and 9,9-diethylfluorene were also converted to the corresponding bis(acetamido)fluorenes by the Schmidt reaction (Method B). Results are reported in Table II, which also contains data for conversions of the substituted 2,7-dinitrofluorenes to 2,7-bis(acetamido)fluorenes by Method A.

LITERATURE CITED


