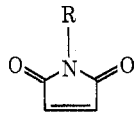
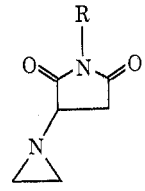
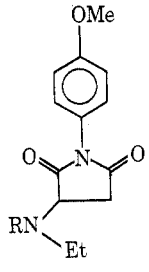
of *N*-(*p*-carbomethoxyphenyl)maleimide (Ic) with aziridine gave the corresponding adduct (IIc) whose pmr spectrum showed the complex high field signals.



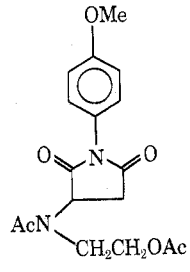
Ia, R = Me
b, R = *p*-MeOC$_6$H$_4$
c, R = *p*-MeOOCC$_6$H$_4$



IIa, R = Me
b, R = *p*-MeOC$_6$H$_4$
c, R = *p*-MeOOCC$_6$H$_4$



IIIa, R = H
b, R = Ac



IV

Experimental Section

Melting points are uncorrected. Infrared spectra were determined in $CHCl_3$ solutions on a Perkin-Elmer 421 spectrophotometer. Ultraviolet spectra were measured in 95% EtOH solutions using a Unicam SP-800 spectrophotometer. Nuclear magnetic resonance spectra were determined using Varian Associates A-60, HA-100, and HR-220 spectrometers. Variable temperature measurements were performed with the aid of V-6040 variable temperature controllers. Chemical shifts are in ppm relative to internal TMS. The elemental analysis were performed by the Alfred Bernhardt Laboratories, West Germany.

N-(*p*-**Methoxyphenyl**)-**3**-(*N'*-**aziridinyl**)**succinimide** (**IIb**).—A solution of 9 g of *N*-(*p*-methoxyphenyl)maleimide (**Ib**) in 250 ml of anhydrous ether and a few drops of pyridine was cooled in an ice bath and treated dropwise under stirring with aziridine, until the yellow solution was completely colorless. A small amount of a pale pink solid that is formed during the reaction was removed by filtration. The solution was washed with water, dried over anhydrous Na_2SO_4, and evaporated. Crystallization from ether–hexane gave 7.5 g (69%) of white prisms, mp 103–104°. The analytical sample showed mp 105–106°; λ_{max} 218, 240, 271 mμ (ε 3000, 7700, 1500); ir bands at 1720 (carbonyl groups) and 1610 and 1590 cm^{-1} (C=C double bonds).

Anal. Calcd for $C_{13}H_{14}O_3N_2$: C, 63.40; H, 5.73; O, 19.49; N, 11.38. Found: C, 63.56; H, 5.83; O, 19.54; N, 11.25.

N-**Methyl-3**-(*N'*-**aziridinyl**)**succinimide** (**IIa**).—Treatment of 1 g of *N*-methylmaleimide (Ia) as in the previous case gave 745 mg (54%) of IIa as prisms: mp 60–61°; λ_{max} 215, 248, 272 mμ (ε 800, 2200, 500); ir band at 1710 cm^{-1} (carbonyl groups).

Anal. Calcd for $C_7H_{10}O_2N_2$: C, 54.54; H, 6.54; O, 20.76; N, 18.17%. Found: C, 54.58; H, 6.63; O, 20.82; N, 18.04%.

N-(*p*-**Carbomethoxyphenyl**)-**3**-(*N'*-**aziridinyl**)**succinimide** (**IIc**).—Treatment of 5 g of Ic as in the previous cases gave 4 g (67%) of IIc as prisms: mp 113–114°; λ_{max} 218, 246 mμ (ε 2900, 11,200); ir bands at 1725 (carbonyl groups) and 1605 cm^{-1} (C=C double bonds); Rast 284, mol wt 274.

Anal. Calcd for $C_{14}H_{14}O_4N_2$: C, 61.31; H, 5.14; O, 23.33; N, 10.21. Found: C, 61.18; H, 5.25; O, 23.35; N, 10.08.

N-(*p*-**Methoxyphenyl**)**succinimide**.—A solution of 300 mg of IIb in 30 ml of tetrahydrofuran was refluxed during 5 hr in the presence of 300 mg of sodium borohydride. The mixture was cooled and filtered and the clear filtrate evaporated to a small volume. Upon addition of hexane, there crystallized 150 mg (60%) of *N*-(*p*-methoxyphenyl)succinimide, mp 151–153°. The analytical sample was obtained as white needles, mp 165–166°. This material was identified by standard procedures with a sample obtained by catalytic hydrogenation of *N*-(*p*-methoxyphenyl)maleimide.[8]

Catalytic Hydrogenation of IIb.—A solution of 500 mg of the compound in 80 ml of ethyl acetate was hydrogenated in the presence of 40 mg of prehydrogenated 10% Pd/C catalyst until the uptake of hydrogen ceased. The catalyst was removed by filtration and the solution concentrated to a small volume. Crystallization from ethyl acetate–hexane gave 432 mg (86%) of IIIa as white prisms mp 112–114°. The analytical sample (ether–hexane) showed mp 115–116°; λ_{max} 218, 237, 272 mμ (ε 4600, 9900, 2100); ir bands at 3310 (amine), 1710 (carbonyl groups), and 1610 and 1590 cm^{-1} (C=C double bonds).

Anal. Calcd for $C_{13}H_{16}O_3N_2$: C, 62.89; H, 6.50; O, 19.33; N, 11.28. Found: C, 62.98; H, 6.46; O, 19.39; N, 11.18.

Acetylation of IIIa.—Treatment of 300 mg of IIIa with Ac_2O–AcONa at room temperature during 12 hr, followed by work-up as usual, gave 197 mg (56%) of IIIb as white prisms: mp 117–118°; λ_{max} 218, 245, 272 mμ (ε 2900, 8700, 2600); ir bands at 1720 (imide carbonyls), 1640 (amide carbonyl), and 1610 cm^{-1} (C=C double bands).

Anal. Calcd for $C_{15}H_{18}O_4N_2$: C, 62.06; H, 6.25; O, 22.04; N, 9.65. Found: C, 62.17; H, 6.21; O, 22.23; N, 9.64.

Treatment of IIb with Acetic Anhydride.—A sample of 500 mg of IIb was treated with Ac_2O–AcONa as described above. Crystallization from ether–hexane gave 335 mg (47%) of IV as white prisms: mp 136–137°; λ_{max} 218, 236, 272 mμ (ε 4700, 8900, 1600); ir bands at 1745 (ester carbonyl), 1720 (imide carbonyls), 1650 (amide carbonyl), and 1610 and 1590 cm^{-1} (C=C double bonds); nmr methoxyl at 3.82 (s), acetyls at 2.15 (s) and 2.08 (s), aromatics at 7.20 (2 H) and 7.01 (2 H), NCH_2CH_2O at 4.22 (2 H) and 3.75 (2 H), and ring protons at 4.07 (1 H) and 3.00 (2 H) ppm.

Anal. Calcd for $C_{17}H_{20}O_6N_2$: C, 58.61; H, 5.79; O, 27.56; N, 8.04. Found: C, 58.56; H, 5.92; O, 27.48; N, 8.12.

Registry No.—IIa, 35740-37-5; IIb, 35740-75-1; IIc, 35740-76-2; IIIa, 35740-77-3; IIIb, 35740-78-4; IV, 35740-79-5.

Acknowledgment.—We are grateful to Professor K. C. Tsou (School of Medicine, University of Pennsylvania), Dr. M. Cohn, and Miss Karen Norton for the 220-MHz measurements, which were done using the facilities provided by NIH Research Grant No. 1 P07 RR-00542-01 from the Division of Research Facilities and Resources.

The Mechanism of Formation of Benzo[*g*]quinolones *via* the Combes Reaction

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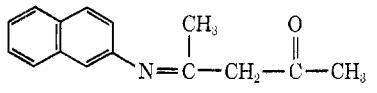
Treatment of the condensation products of 2-aminonapthalene and 1,3-dicarbonyl compounds with H_2SO_4 provides a convenient method of synthesis of benzo[*g*]quinolines.[1-3] The formation of benzo[*g*]quinolines rather than the expected benzo[*f*]quinolines has been explained in two ways: Johnson[2] has proposed that the anil **1** affords linear products because of a larger deactivation of the one position with respect to the three position in the naphthalene ring; Huigsen[3]

(1) W. S. Johnson and F. J. Mathews, *J. Amer. Chem. Soc.*, **66**, 210 (1944).
(2) W. S. Johnson, E. Woroch, and F. J. Mathews, *ibid.*, **69**, 566 (1947).
(3) R. Huisgen, *Justus Liebigs Ann. Chem.*, **564**, 16 (1949).



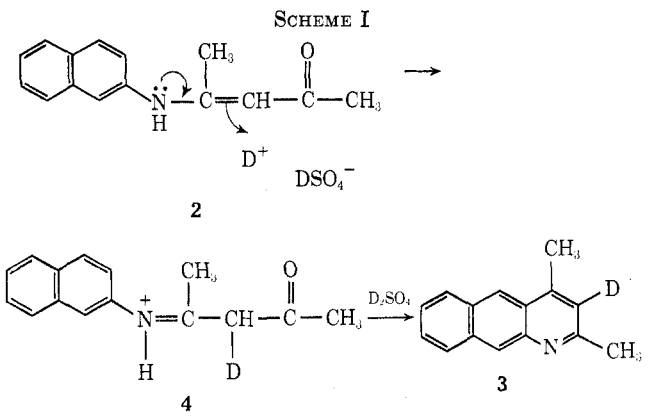
1

has proposed that the enamine **2** is protonated at the one position of the aromatic ring to block the formation of angular products.

The structure of the condensation product of acetylacetone and 2-aminonaphthalene is the enamine **2** as indicated by Huisgen's isolation of 2-acetylaminonaphthalene from the permanganate oxidation of **2**, the nmr spectra of **2**, and the comparison of the uv spectra of **2** with those of other known enamines of the same general structure. The treatment of **2** with D_2SO_4 produced 3-deuterio-2,4-dimethylbenzo[*g*]quinoline (**3**) which was identical except for the H_3 signal with a sample prepared with H_2SO_4. The assignment of the chemical shifts of protons H_3 (δ 6.94), H_5 (8.43), and H_{10} (8.60) is based on electron densities reported for benzo[*g*]quinoline[4,5] and the accepted assignment of chemical shifts in various quinolines.

The lack of incorporation of deuterium into the 10 position of **3** clearly indicates that Huisgen's proposed mechanism is not correct. The formation of **3** most likely proceeds by the mechanism shown in Scheme I. The protonation of **2** to give **4** lends credence to

SCHEME I



Johnson's rationalization of the formation of benzo[*g*]quinolines *via* the Combes reaction.

Experimental Section[6]

2-(2-Naphthyl)amino-2-penten-4-one (**2**).—2-Aminonaphthalene and acetylacetone were condensed as described by Johnson:[1] mp 99° (lit. mp 99°); nmr δ ($CDCl_3$) 2.04 (s, 6 H), 5.1 (s, 1 H), 7.45 (m, 7 H), 12.7 (b s, 1 H); $\lambda_{max}^{i\text{-PrOH}}$ 337 nm (ε 23,000).

2,4-Dimethylbenzo[*g*]quinoline.—The enamine **2** was treated with H_2SO_4 as described by Johnson:[1] mp 92° (lit. mp 93°); nmr δ ($CDCl_3$) 2.62 (s, 3 H), 2.67 (s, 3 H), 6.94 (s, 1 H), 7.43 (m, 2 H), 7.95 (m, 2 H), 8.31 (s, 1 H), 8.56 (s, 1 H).

3-Deuterio-2,4-dimethylbenzo[*g*]quinoline (**3**).—**2** (1 g, 0.044 mol) was treated with 3 g of D_2SO_4 as described by Johnson.[1] The crude material was dried and chromatographed on Brinkman silica gel, eluting with $CHCl_3$. The material so obtained was recrystallized from petroleum ether to give 52% **3**: mp 92° (lit. mp 93°); nmr δ ($CDCl_3$) 2.58 (s, 3 H), 2.65 (s, 3 H), 7.44 (m, 2 H), 7.96 (m, 2 H), 8.31 (s, 1 H), 8.56 (s, 1 H).

Registry No.—**3**, 35666-88-7.

(4) M. J. S. Dewar and G. J. Glecher, *J. Chem. Phys.*, **44**, 759 (1966).
(5) K. Nishimoto and L. S. Foster, *Theor. Chim. Acta*, **4**, 155 (1966).
(6) Melting points were taken with a Thomas-Hoover capillary melting point apparatus and are uncorrected. Nmr spectra were obtained with a Perkin-Elmer R-12A spectrometer and are reported relative to TMS. Uv spectra were recorded using a Perkin-Elmer-Coleman 124 spectrophotometer.

The Synthesis of Azetidine-3-carboxylic Acid[1,2]

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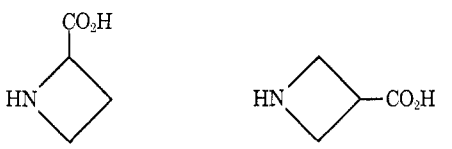
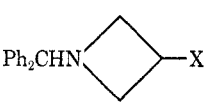
L-Azetidine-2-carboxylic acid (**1**) occurs in nature.[3] It has been shown to inhibit the growth of *E. coli* cultures and various seedlings[4] and to cause abnormalities in growing embryos.[5] The X-ray structure showed the ring to be 11° out of plane and it was postulated that the incorporation of **1** in a polypeptide chain could cause the direction of successive amide bonds in the α helix of the peptide tertiary structure to change by 16°.[6] As an extension of studies on azetidines,[7] it was therefore of interest to synthesize the isomeric azetidine-3-carboxylic acid (**2**).



1 **2**

Chatterjee and Triggle[8] had reported the preparation of the hydrochloride of 1-benzhydrylazetidin-3-ol (**3**) from epichlorohydrin and benzhydrylamine, but gave no experimental details or yields. Application of the procedure described by Gaertner[9] to this reaction gave 60-65% yields of the salt of **3**. Tosylation of **3** gave only 39% of the corresponding ester **4**, and reaction with



3, X = OH
4, X = OTs
5, X = OMs
6, X = OMe
7, X = OEt
8, X = Br
9, X =
10, X = CN
11, X = CO_2H
12, X = CH_2NH_2

(1) Presented in part at the 25th Annual Northwest Regional Meeting of the American Chemical Society, Seattle, Wash., June 1970, Organic Chemistry Abstracts, No. 131, 1970, p 74.
(2) From the Ph.D. thesis of Roger Lok, University of Washington, 1971. Supported in part by the Graduate Research Fund, University of Washington.
(3) L. Fowden, *Biochem. J.*, **64**, 323 (1956); L. Fowden, *Advan. Enzymol.*, **29**, 89 (1967).
(4) L. Fowden and M. H. Richard, *Biochem. Biophys. Acta*, **71**, 459 (1963); E. J. Hewitt and B. A. Notton, *Phytochemistry*, **6**, 1329 (1967).
(5) D. J. Cummings, V. A. Chapman, S. S. Delong, and L. Mondale, *J. Virol.*, **1**, 193 (1967).
(6) H. M. Berman, E. L. McCandy, J. W. Burgner, II, and R. L. Van Etten, *J. Amer. Chem. Soc.*, **91**, 6177 (1969).
(7) A. G. Anderson, Jr., and M. T. Wills, *J. Org. Chem.*, **33**, 3046 (1968), and references cited therein.
(8) S. S. Chatterjee and D. J. Triggle, *Chem. Commun.*, 93 (1968).
(9) V. R. Gaertner, *Tetrahedron Lett.*, 4691 (1966).