was obtained from the petroleum ether soluble portion of the neutral fraction; 0.20 g. (4%), b.p. about 80° (5 mm.), $n^{20}D$ 1.4818; $\lambda_{max.}$ 227 mμ, ε 7960. The absorption spectra correspond to a mixture of the α,β- and the β,γ-unsaturated isomer in a ratio of about 2 to 1. Treatment by the hot acid-ethanol method as described before gave the α,β-unsaturated dinitrophenylhydrazone.

4-Isopropyl-1,2-cyclohexanedione 2,4-dinitrophenylosazone was prepared according to Ramirez and Kirby by refluxing 200 mg. of the bromodinitrophenylhydrazone for three hours in 17 ml. of the reagent described.[19] The scarlet crystals, 240 mg. (93%), m.p. 183–185°, were recrystallized from chloroform–ethanol and from chloroform; m.p. 191.5–192° dec., $\lambda_{max.}$ 352 mμ, ε 30,600.

Anal. Calcd. for $C_{21}H_{22}N_8O_8$: C, 49.0; H, 4.3; N, 21.8. Found: C, 49.4; H, 4.2; N, 22.4.

NORTHAMPTON, MASS.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, WEST VIRGINIA UNIVERSITY]

Azabenzazulenes. I. 1-Azadibenz[b,f]azulene

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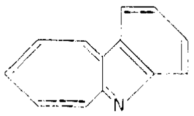
RECEIVED JUNE 23, 1954

The syntheses of the above-mentioned azabenzazulene as well as that of 1-azabenz[b]azulene and 1-azadibenz[b,h]azulene have been accomplished by preparing the appropriate ketones, followed by their conversion to hydroazaazulenes (indoles) and subsequent dehydrogenation.

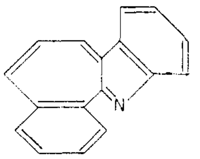
Discussion

In 1950 when an unsuccessful attempt to prepare 1,4-diazatribenz[b,f,h]azulene[2] was started in this Laboratory there were no reported azaazulenes. Since then the preparation of 1-azabenz[b]azulene[3–5] (I), 1-azadibenz[b,h]azulene[5] (II) and 4-azabenz[b]azulene[6] have been reported.

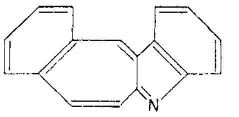
In the meantime our program had been modified to include a study of the syntheses of azabenzazulenes I-III as well as 1-azatribenz[b,e,g]azulene and 1-azatribenz[b,f,h]azulene, the latter two of which will be the subject of a later communication.



I



II



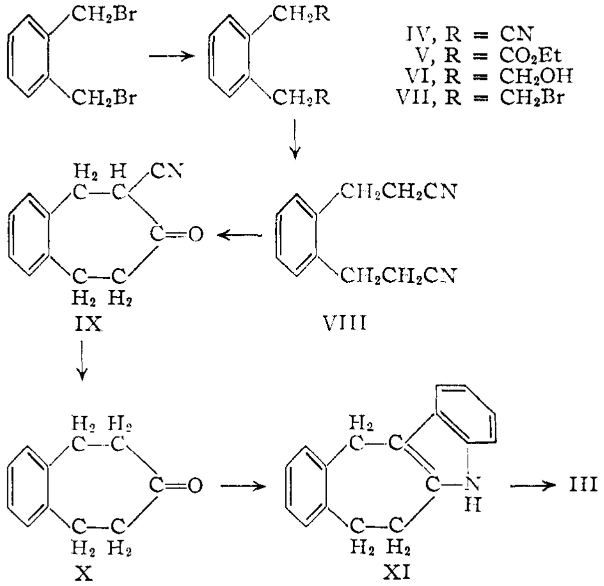
III

The synthetic routes for all the azabenzazulenes of this report may be divided into three phases: (a) synthesis of an appropriate ketone; (b) conversion of the ketone to a hydroazaazulene (indole); (c) dehydrogenation of the hydroazaazulene.

The study for the preparation of 1-azabenz[b]azulene (I) was included because at the time this work was begun there were conflicting reports[3,4] concerning the properties of this compound. The properties observed for I in this work agree with those reported by Anderson and Tazuma[3] as do those later reported by Treibs.[5]

The synthesis of 1-azadibenz[b,h]azulene (II) was completed and its characterization was in the final stages before this compound was reported by Treibs, *et al.*[5] Compound II was obtained by two dehydrogenation procedures including the one used by Treibs. The melting point and color observed for II agree with those reported by Treibs.

The preparation of 7H-5,6,8,9-tetrahydrocycloheptabenzen-7-one (X) was accomplished as outlined in the following diagram by standard paths. All of the compounds of this sequence following V except X are new compounds.



The synthesis of ketone X, in 64% over-all yield by the Ziegler cyclization[7] of VIII to IX followed by its hydrolysis is much superior to the previously used methods for the attempted preparation of X which involved either the Dieckmann reaction[8] or the thorium salt method.[9] It is of interest to note that the Ziegler cyclization product was ketocyano

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(1) From the M.S. thesis of D. O. Steiniger and the Ph.D. Dissertation of Z. B. Papanastassiou, both from West Virginia University, 1954

(2) W. L. Sung, "Attempted Syntheses of Some Derivatives of 4,5,6,7-Dibenzazepin-3-one." Unpublished M.S. thesis, West Virginia University, 1952.

(3) A. G. Anderson and J. Tazuma, THIS JOURNAL, **74**, 3455 (1952).

(4) W. Treibs, *Ann.*, **576**, 110 (1952).

(5) W. Treibs, R. Steinert and W. Kirchoff, *ibid.*, **581**, 54 (1953).

(6) W. Treibs, H. M. Barchet, G. Bach and W. Kirchoff, *ibid.*, **574**, 54 (1951).

(7) K. Ziegler, H. Eberle and H. Ohlinger, *ibid.*, **504**, 94 (1933).

(8) A. F. Titley, *J. Chem. Soc.*, 2571 (1928).

(9) B. Kubota and T. Isemura, *Bull. Chem. Soc. Japan*, **6**, 103 (1931).

compound IX instead of the expected iminocyano compound.

All of the hydroazabenzazulenes (indoles) were prepared from the appropriate ketones and phenylhydrazine in one step in either glacial acetic or aqueous hydrochloric acid.[10] 1,4,7,8-Tetrahydro-1-azadibenz[b,f]azulene (XI) was identified by method of synthesis, elemental analyses and by similarity of its ultraviolet absorption spectrum with that of the two other known hydroazaazulenes prepared in this work.

Dehydrogenation of 1,4,7,8-tetrahydro-1-azadibenzazulene (XI) by two methods yielded the same red solid which is believed to be nearly pure azadibenzazulene (III). Results from micro-analyses, after many purification attempts, varied from sample to sample beyond the limit of experimental error; these results were low for carbon from 0.8 to 5.3% but in fair agreement for nitrogen and hydrogen as compared to the theory for III. However, the neutral equivalent for the red solid as determined by perchloric acid titration was 235 (229.2 is the theory for III) and the perchlorate gave microanalytical results which are in good agreement with the theory for III perchlorate. Also, the red solid reacted with methyl iodide to form a derivative which gave the proper analysis for the methiodide of III. Furthermore, the ultraviolet, visible[11] and infrared[11] spectra of the red solid show the general characteristics which should be expected in the spectra of III. Finally, the ring skeleton for the red solid was established by hydrogenation to give XI from which it was made.

The chloranil dehydrogenation method[5] is superior to the palladium-on-charcoal dehydrogenation method[12] for both the preparation of II and III. Treibs reports[5] that the chloranil method is also superior for the preparation of I.

The maximum absorption for 1,4,5,6-tetrahydro-1-azadibenz[b,h]azulene (XIII) in the ultraviolet region[11] is at 316 mμ log ε 4.42, whereas the other two hydroazaazulenes have their maxima[11] at about 275 mμ with log ε values of about 4.0. These bathochromic and hyperchromic shifts are believed to be due to the increased conjugation which is possible in XIII as compared with the other two compounds.

The ultraviolet spectra curves for azabenzazulenes, I, II and III (Fig. 1) have the same general shapes in the 300 mμ region as do the curves for azulene, benz[b]azulene and benz[g]azulene.[13] Each of the visible absorption spectra[11] for the three azabenzazulenes shows a broad band.

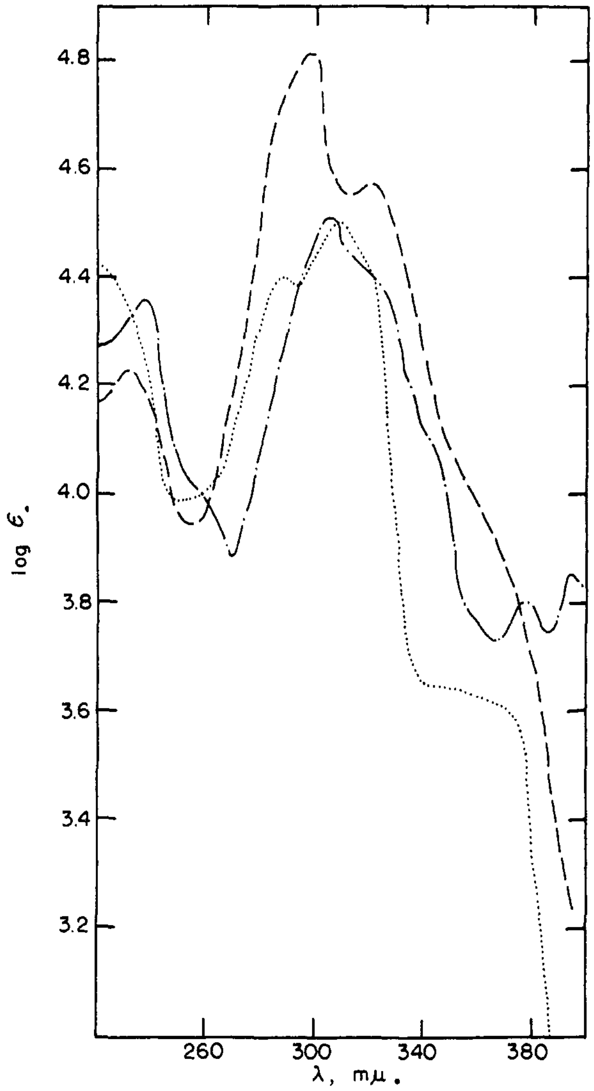

Fig. 1.—Ultraviolet absorption spectra of 1-azabenz[b]-azulene (I), (. . . .), 1-azadibenz[b, h]azulene (II), (·—·—), and 1-azadibenz[b, f]azulene (III), (------) all in cyclohexane.

Azabenzazulene III has been color fast to daylight and the atmosphere when exposed on filter paper during a period of nine months. Compound III was also stable when heated at 170–180° for one-half hour. Less is known about the stability of azaazulene II which was much more difficult to obtain in crystalline form than was III. Azaazulene II apparently is stable when pure and in crystalline form; some solutions of what were believed to be nearly pure II were noted to change from a red to a yellow color on standing for a few hours.

Experimental[14]

1,4,5,6,7,8-Hexahydro-1-azabenz[b]azulene.—Following the method of Rogers and Corson[10] cycloheptanone and phenylhydrazine reacted together in glacial acetic acid. The yield of product recrystallized once from methanol was 74%, m.p. 142–144° (lit. 144°,[15] 141–142°[3]). The absorption spectrum[11] of this product in cyclohexane showed a minimum at 246 mμ log ε 3.59 and a maximum at 275 mμ, log ε 4.05.

1-Azabenz[b]azulene (I).—The apparatus, reagents and technique for the dehydrogenation of 1,4,5,6,7,8-hexahydro-1-azabenz[b]azulene to give I were similar to those used by

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[10] C. U. Rogers and B. B. Corson, This Journal, **69**, 2910 (1947).

[11] The visible and infrared spectra discussed in this article have been deposited as Document number 4351 with the ADI Auxiliary Publications Project, Photoduplication Service, Library of Congress, Washington 25, D. C. A copy may be secured by citing the Document number and by remitting $1.25 for photoprints, or $1.25 for 35 mm. microfilm, in advance by check or money order payable to: Chief, Photoduplication Service, Library of Congress.

[12] A. G. Anderson, J. A. Nelson and J. J. Tazuma, This Journal, **75**, 4980 (1953).

[13] Pl. Plattner, A. Fürst and W. Keller, *Helv. Chim. Acta*, **32**, 2464 (1949).

[14] All temperatures are uncorrected. Unless otherwise indicated all elemental analyses were made by Galbraith Laboratories, Knoxville, Tenn.

[15] W. H. Perkin and S. P. Plant, *J. Chem. Soc.*, 2583 (1928).

Anderson, *et al.*,[16,3] except that 1.4 g. of the hydroazaazulene was added three times as fast to the hot catalyst; yield 0.07 g. (5%), m.p. 137–139° (lit.[3] 140–141°). The visible absorption spectrum[11] of a cyclohexane solution of I showed a maximum with broad shoulders at 500 mμ, log ε 2.62. The infrared spectrum was taken.[11]

5H-6,7,8,9-Tetrahydrocycloheptabenzen-5-one (XII).—A mixture of δ-phenylvaleric acid,[17] m.p. 60°, and 720 g. of polyphosphoric acid[18–20] was frequently swirled and heated to 85°. Within 15 minutes after the 85° temperature was attained a red color developed. After an additional 25 minutes of frequent swirling at 85° the solution was allowed to cool overnight. The solution was poured into 2 l. of ice-water and the resulting mixture was extracted with ether. The extract was washed with dilute sodium hydroxide solution, water, dried, concentrated and the residue distilled to yield 63 g. (93%) of colorless oil, b.p. 120–124° (5–6 mm.); n^{20}_D 1.5642 (lit.[21] n^{19}_D 1.5632).

1,4,5,6-Tetrahydro-1-azadibenz[b,h]azulene (XIII).—During 40 minutes 13.6 g. (0.126 mole) of phenylhydrazine was added to a stirred, refluxing solution of 80 ml. of 10% hydrochloric acid.[10] To this refluxing mixture 19.9 g. (0.124 mole) of ketone XII was added during 2 hours and 20 minutes. As ketone XII was added a brown liquid began to accumulate. After the addition of XII had been completed refluxing was continued for 2.5 hours. The reaction mixture was cooled and ether extracted. The ether extract was washed with water, dried, concentrated and the residue distilled to give 1.6 g. (8%) of XIII, b.p. 113–115° (2–3 mm.) and 20.0 g. (75%) of a viscous, orange oil, b.p. 217–222° (2–3 mm.). This oil was crystallized from methanol to give 14.0 g. (53%) of colorless, clumpy needles, m.p. 98° (lit. 98°,[22] 98°[5]). The absorption spectrum[11] in cyclohexane showed a minimum at 270 mμ, log ε 3.45 and a maximum at 316 mμ, log ε 4.42. The infrared spectrum in carbon tetrachloride showed an absorption band at 2.92 μ which is expected for the N–H group.

Using the glacial acetic acid method[10] a 23% yield of XIII, m.p. 97–98°, was obtained. Using the method of Buu-Hoi and Xuong[22] only a dark-brown resinous material was obtained.

1-Azadibenz[b,h]azulene (II). (a) Palladium-on-Charcoal Dehydrogenation.—The procedure[16,3] was essentially the same as for the preparation of I. Much difficulty was encountered in obtaining the product in crystalline form. A successful technique for obtaining crystals will be described under method b. From 1.48 g. of XIII, 0.31 g. of red oil was obtained which finally yielded a few mg. of deep-red feathery crystals, m.p. 119° (lit.[5] 119.5°). The visible absorption spectrum[11] in cyclohexane showed maxima at 406 mμ, log ε 3.97; 416 mμ, log ε 4.25; 476–504 mμ, log ε 2.90. The infrared spectrum[11] is very similar to the infrared spectrum of I.

b. **Chloranil Dehydrogenation.**—The dehydrogenation was conducted as described[5] except that the solvent for chromatography was methylene chloride and the developers were respectively cyclohexane, cyclohexane–methylene chloride mixtures which were successively richer in the latter and finally methanol. Throughout only one colored zone was present. No other zone was detected by the use of ultraviolet light. The combined eluants were evaporated to dryness and the residue dissolved in cyclohexane. This solution was concentrated by simultaneous use of partial vacuum and heating on a water-bath. A semi-crystalline residue formed on the glass at the surface of the liquid. On refrigeration deep-red feathery crystals formed within an hour. From 2.02 g. of XIII 260 mg. (13%) of II, m.p. 119°, was obtained. This product showed no change in melting point when mixed with the product from method a.

***o*-Bis-(cyanomethyl)-benzene (IV).**—Following the method of Perkin and Titley[23] *o*-bis-(bromomethyl)-benzene was added to a hot aqueous alcoholic suspension of potassium cyanide; m.p. 61–63° (lit.[24] 62–63°), yield 71%.

***o*-Bis-(dicarbethoxymethyl)-benzene (V).**—Dicyanide IV was refluxed with absolute ethyl alcohol saturated with dry hydrogen chloride; yield 74%, b.p. 175–177° (10 mm.) and 144–148° (2–3 mm.), lit.[23] 173–174° (10 mm.), n^{25}_D 1.4959, d^{25}_4 1.093.

***o*-Bis-(β-hydroxyethyl)-benzene (VI).**—Addition of a solution of 121.0 g. (0.48 mole) of V in 700 cc. of dry ether to a solution of 57 g. (1.5 moles) of lithium aluminum hydride in 1800 cc. of dry ether according to the general procedure[25] produced 65.5 g. (81.5%) of a white crystalline powder which after recrystallization from chloroform–carbon tetrachloride mixture was obtained as white needles, m.p. 61–63°.

Anal. Calcd. for $C_{10}H_{14}O_2$: C, 72.26; H, 8.49; mol. wt., 166.2. Found: C, 72.30; H, 8.51; mol. wt.,[26] 164.3.

***o*-Bis-(β-bromoethyl)-benzene (VII).**—A suspension of 65.0 g. (0.39 mole) of VI in 1350 cc. of 48% aqueous hydrogen bromide was refluxed for *ca.* 4 hours and at the same time *ca.* 50 cc. of distillate was slowly collected. The reaction mixture, after cooling, was extracted with methylene chloride and extract was washed, dried, concentrated and the residue was distilled to yield 97.9 g. (86%) of a viscous oil; b.p. 120–121° (1–2 mm.), n^{25}_D 1.5895, d^{25}_4 1.627.

Anal. Calcd. for $C_{10}H_{12}Br_2$: C, 41.14; H, 4.14; molar refractivity, 60.31. Found: C, 41.04; H, 4.22; molar refractivity, 60.51.

***o*-Bis-(β-cyanoethyl)-benzene (VIII).**—A solution of 96.0 g. (0.33 mole) of dibromide VII in 300 cc. of absolute ethyl alcohol was slowly added to a hot stirred solution of 57.0 g. (0.87 mole) of potassium cyanide in 150 cc. of water. Heating and stirring was continued for *ca.* 18 hours after which 250 cc. of the solvent was distilled off and the remaining solution was diluted with water and extracted with ether and then with benzene. Washing, drying and concentrating the combined extracts was followed by distillation of the residue through a short path distilling column. Three fractions were collected totalling 44.0 g. (72%); b.p. 176–183° (1–2 mm.); the distillate solidified on standing, m.p. 41–42°.

Anal. Calcd. for $C_{12}H_{12}N_2$: C, 78.22; H, 6.56. Found: C, 78.16; H, 6.70.

7H-6-Cyano-5,6,8,9-tetrahydrocycloheptabenzen-7-one (IX).—Following the general procedure of Ziegler, *et al.*,[27] in the reaction flask of a high dilution apparatus (similar to the one described by Adams and Kornblum[28]) 500 cc. of a 0.81 *N* ethereal solution of phenyllithium was placed and 100 g. (0.93 mole) of methylaniline was added during 3/4 hour, while the reaction mixture was stirred and heated at 60°. Then a solution of 41.0 g. (0.222 mole) of dinitrile VIII in 300 cc. of ether–benzene mixture was added during 21 hours. The reaction mixture was afterwards cooled and cold water was added to it. The ether layer was separated and the aqueous layer extracted with benzene. Washing, drying and concentrating the combined organic solutions yielded 38.1 g. (92%) of a pale yellow crystalline substance which after recrystallization from methanol produced white crystals, m.p. 134–135°.

Anal. Calcd. for $C_{12}H_{11}ON$: C, 77.82; H, 5.98; N, 7.56. Found: C, 77.80; H, 6.10; N, 7.64.

The 2,4-dinitrophenylhydrazone was prepared as a yellow voluminous precipitate by treating a glacial acetic acid solution of the preceding compound with 2,4-dinitrophenylhydrazine; m.p. 215–216° after one recrystallization from methanol.

Anal. Calcd. for $C_{17}H_{15}O_4N_4$: C, 59.17; H, 4.14; N, 19.17. Found: C, 59.13; H, 3.99; N, 19.28.

Compound IX was further characterized by hydrolysis with 5% potassiun hydroxide solution to produce *o*-bis-

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(17) J. W. Cook, R. Philip and A. R. Somerville, *J. Chem. Soc.*, 164 (1948).

(18) H. R. Snyder and F. X. Werber, THIS JOURNAL, **72**, 2965 (1950).

(19) J. Koo, *ibid.*, **75**, 723 (1953).

(20) The authors wish to thank the Victor Chemical Works, 141 W. Jackson Blvd., Chicago 4, Ill., for an experimental sample of polyphosphoric acid.

(21) R. C. Gilmore and W. J. Horton, THIS JOURNAL, **74**, 5527 (1952).

(22) N. P. Buu-Hoi and N. D. Xuong, *J. Chem. Soc.*, 2225 (1952).

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(25) R. F. Nystrom and W. G. Brown, THIS JOURNAL, **69**, 1197 (1947).

(26) C. L. Ogg, W. L. Porter and C. O. Willits, *Ind. Eng. Chem., Anal. Ed.*, **17**, 394 (1945).

(27) K. Ziegler, H. Eberle and H. Ohlinger, *Ann.*, **504**, 94 (1933).

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(β-carboxyethyl)-benzene, m.p. 172–174° (lit.[29] 171°). Calcd. for $C_{12}H_{14}O_4$: mol. wt., 222.2. Found: mol. wt., 224.9 (by neut. equiv. method).

7H-5,6,8,9-Tetrahydrocycloheptabenzen-7-one (X).—To a 125-cc. flask containing 100 cc. of 76.5% hot sulfuric acid, 9.0 g. (0.048 mole) of cyanoketone IX was added slowly with occasional swirling of the flask. The yellow suspension formed was warmed on a steam-bath until a red solution was obtained (*ca.* 15 minutes). Then the acid solution was poured into the distilling flask of a steam distillation apparatus containing 90 cc. of water. The mixture so formed was heated to boiling and a stream of steam was introduced. A cloudy distillate, *ca.* 250 cc., was collected which on standing at 0° deposited a white crystalline material; yield 5.4 g. (69%), m.p. 42–43° (lit.[9] 41–42°).

1,4,7,8-Tetrahydro-1-azadibenz[b,f]azulene (XI).—Following the method of Rogers and Corson[10] 5.38 g. (0.034 mole) of ketone X in 50 cc. of glacial acetic acid was treated with an equimolecular quantity of phenylhydrazine; the white solid formed was recrystallized from methanol; yield 6.2 g. (83%), m.p. 145°.

Anal. Calcd. for $C_{17}H_{15}N$: C, 87.52; H, 6.48; N, 6.00. Found: C, 87.37; H, 6.60; N, 5.95.

The absorption spectrum[11] in cyclohexane showed a minimum at 246 mμ, log ϵ 3.59 and a broad maximum around 275 mμ, log ϵ 4.05. The infrared spectrum in chloroform showed an absorption band at 2.9 μ which is expected for the N–H group.

1-Azadibenz[b,f]azulene (III). a. Palladium-on-Charcoal.—The apparatus and rate of addition of XI were the same as for the preparation of I and II.[3,16] After the dehydrogenation of 3.4 g. (0.0145 mole) of XI had been completed the products were dissolved in methylene chloride. This solution was extracted with 6 *N* hydrochloric acid solution yielding an orange-yellow extract.

Isolation of the Neutral Fraction.—The remaining methylene chloride solution, after washing and drying was concentrated to dryness yielding 0.80 g. of a solid which after repeated crystallizations from methanol was obtained as a pale-green crystalline substance, m.p. 210–211°. A solution of this substance in methanol exhibited a strong fluorescence in both visible and ultraviolet lights. The infrared spectrum showed an absorption band at 2.9 μ.

Anal. Found: C, 88.20; H, 5.61; N, 5.86.

Isolation of the Basic Fractions.—An orange solid formed as the hydrochloric acid extract was cooled and made alkaline with 7.5 *N* ammonium hydroxide solution. Extraction with methylene chloride yielded a dark red solution which after washing, drying and concentrating (by blowing a stream of nitrogen) left 1.0 g. of a red-orange residue which was dissolved in *ca.* 15 cc. of methylene chloride and chromatographed through a 300 × 22 mm. quartz column packed with activated alumina. The chromatogram was developed with methylene chloride.

Isolation of Benz[a]acridine.—First a pale yellow band was formed exhibiting bluish fluorescence in ultraviolet light. This chromatographic fraction after evaporation of the solvent yielded 0.1 g. of a yellow solid which was recrystallized from benzene–heptane mixture; m.p. 126–128° (lit.[30] 131°). The ultraviolet spectrum of this fraction was nearly identical with the reported ultraviolet spectrum of benz[a]acridine.[31]

Isolation of 1-Azabenz[b,f]azulene (III).—The second band formed in the chromatographic column was broad red-orange zone. It was eluted with methylene chloride and evaporation of the solvent yielded 0.84 g. (24%) of a red-orange powder, m.p. 153–156°, which on recrystallization from methyl ethyl ketone–heptane mixture gave raspberry-red needles, m.p. 150–151°. When the solution of this substance was cooled quickly a red-orange powder was obtained having the same m.p., m.m.p. and ultraviolet spectrum as the raspberry-red needles.

Anal.[32] Calcd. for $C_{17}H_{11}N$: C, 89.05; H, 4.83; N, 6.11. Found: C, 87.63; H, 5.07; N, 6.42.

The trinitrobenzene derivative was prepared by warming a methanol solution of III with trinitrobenzene; m.p. 136–138°.

Anal. Calcd. for $C_{23}H_{14}N_4O_6$: N, 12.65. Found: N, 12.25.

Hydrogenation of the red-orange substance, m.p. 150–151°, yielded a neutral fraction which was identified by m.p., m.m.p., and infrared spectrum to be XI from which the red-orange substance was made.

b. **Chloranil Dehydrogenation.**—One gram (0.0043 mole) of XI and 2.3 g. (0.0095 mole) chloranil in 25 cc. of dry benzene was refluxed for 3 hours. The basic fraction was isolated essentially as described[5]; yield 0.4 g. (40%) of red powder, m.p. 150–150.5°. The analytical sample was recrystallized from methanol–water mixture and dried to constant weight by heating at 180° for 20 minutes under reduced pressure.

Anal. Calcd. for $C_{17}H_{11}N$: C, 89.05; H, 4.83; N, 6.11; neut. equiv., 229.2. Found: C, 88.36; H, 5.22; N, 5.72; neut. equiv., 234.5, 234.8.

As the neutral equivalent was taken with perchloric acid in glacial acetic acid a flocculent orange solid, m.p. 275° dec., separated.

Anal. Calcd. for $C_{17}H_{12}O_4NCl$: C, 61.92; H, 3.67; N, 4.24. Found: C, 61.91; H, 3.95; N, 4.23.

The methiodide derivative was prepared by treating 50 mg. of the red solid in about 10 ml. of benzene with an excess of methyl iodide. The yield was 81 mg. (93%) of an orange powder, m.p. 319–325° dec.

Anal. Calcd. for $C_{18}H_{14}NI$: C, 58.24; H, 3.80; N, 3.77. Found: C, 58.18; H, 3.85; N, 3.78.

The red products obtained from methods a and b showed no change in m.p. on m.m.p. determination and their infrared spectra were alike. The absorption spectrum[11] in cyclohexane showed a maximum with broad shoulders at 458 mμ log ϵ 2.92. The infrared spectrum[11] has been recorded.

Spectra Measurements.—All determinations were made either with a Beckman quartz spectrophotometer, Model DU, a Perkin–Elmer recording spectrophotometer, Model C, or a Baird Associates, Inc., recording spectrophotometer, Model B.

MORGANTOWN, W. VA.

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[29] K. Fries, H. Bestian and W. Kiauditz, *Ber.*, **69**, 715 (1936).

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[31] M. G. Badger, R. S. Pearce and R. Petit, *J. Chem. Soc.*, 3199 (1951).

[32] Analyses by Mr. O. E. Harris, Microanalytical Laboratory, University of Pittsburgh.