

a solution of **2c** (1.4 g, 6.7 mmol) in cyclohexane (20 ml) and TMEDA (1.6 ml) at 10°. The solution was allowed to stand at 0° for 65 hr before addition of ethyl iodide (30 mmol, 2.3 ml), then stirred for 2 hr at ambient temperature. The reaction mixture was quenched by water and worked up in conventional manner to provide an oil. Glpc analysis on column B at 160° showed 25% *trans-2e*<sup>23</sup> (*t<sub>R</sub>* 5.3 min) together with 65% unreacted starting material (*t<sub>R</sub>* 8.0 min). A pure sample of *trans-2e* was obtained from glpc. Variable-temperature nmr studies confirming the structure and providing evidence regarding its conformation are reported elsewhere.<sup>8,23</sup>

*Anal.* Calcd for C<sub>15</sub>H<sub>20</sub>: C, 91.47; H, 8.53. Found: C, 91.28; H, 8.87.

**2,4a,9,10-Tetrahydrophenanthrene (3a).**—Lithium-ammonia reduction of 9,10-dihydrophenanthrene (5 mmol, 0.9 g) was quenched with water after 2 hr and the product was isolated by extraction (as rapidly as possible) with ether. Glpc analysis on column B at 175° indicated 62% **3a** (*t<sub>R</sub>* 3 min), 23% **2a** (*t<sub>R</sub>* 4 min), 2% *as*-octahydrophenanthrene (*t<sub>R</sub>* 1.6 min), and minor products. Although **3a** underwent spontaneous reversion to **2a** on standing, it was sufficiently stable to obtain nmr and uv spectra of a sample trapped off the gas chromatograph: nmr δ 7.1 (m, 4, aromatic), 6.4 (d, 1, *J* = 10 Hz, vinyl), 5.8 (m, 2, vinyl), and 2.7 and 2.2 (complex multiplets, 7 protons); uv max (95% ethanol) 234 mμ (ε 1200).

Analogous reaction conducted in the absence of ferric chloride provided **3a** (32%), further reduction products (49%), and recovered **2a** (19%).

**4a-Methyl-2,4a,9,10-tetrahydrophenanthrene (9).**—To a solution of phenanthrene (20 mmol, 2.6 g) in THF (100 ml) and ammonia (250 ml) was added lithium (5 equiv, 0.69 g). The dark green-blue color was discharged 5 min later by methyl bromide. Conventional work-up provided an oil, glpc analysis of which on

column B at 125° showed 93% **9** (*t<sub>R</sub>* 7.2 min). Distillation through a short Vigreux column gave 1.5 g of **9** as a colorless oil: bp 109°; nmr data cited in Results.

*Anal.* Calcd for C<sub>15</sub>H<sub>16</sub>: C, 91.78; H, 8.22. Found: C, 91.52; H, 8.36.

Catalytic hydrogenation of **9** (838 mg, 4.27 mmol) in 95% ethanol (10 ml) over a platinum catalyst (10 mg of PtO<sub>2</sub>) in a "Vortex Low Pressure Hydrogenation Apparatus" (J. B. Thompson Co.) at 28 lb and 30° for 18 hr resulted in absorption of 1.4 equiv of hydrogen. Two products were isolated from glpc (column B at 100°). The first (*t<sub>R</sub>* 10 min) proved to be **10** (60%): nmr (CCl<sub>4</sub>) δ 7.0 (4 aromatic protons), 5.4 (1 vinyl), 2.8 (m, 2, benzylic), 2.6–1.5 (m, 8), and 1.4 (s, 3, CH<sub>3</sub>).

*Anal.* Calcd for C<sub>15</sub>H<sub>18</sub>: C, 90.85; H, 9.15. Found: C, 90.38; H, 9.38.

The second product of hydrogenation (*t<sub>R</sub>* 7 min) was **11** (40%), whose nmr spectrum corresponded with that recently reported by Boekelheide and Sturm.<sup>11</sup>

*Anal.* Calcd for C<sub>15</sub>H<sub>20</sub>: C, 89.93; H, 10.06. Found: C, 89.80; H, 10.05.

**Registry No.**—**1a**, 85-01-8; **1b**, 883-20-5; **1c**, 3674-75-7; **1d**, 604-83-1; **1e**, 15810-14-7; **2c**, 22139-41-9; *cis-2d*, 22140-64-3; *trans-2d*, 22140-65-4; *cis-2e*, 22155-45-9; *trans-2e*, 22140-66-5; **5d**, 22139-42-0; **6d**, 22139-43-1; **9**, 22139-44-2; **10**, 22139-45-3; 9-bromo-10-ethylphenanthrene, 17918-49-9.

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## A Preparation of 9,10-Dicyanoanthracene and 9,10-Dicyanophenanthrene by Cyanation of the Respective 9-Cyanoarenes<sup>1</sup>

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Carbanion intermediates formed by reaction of sodium cyanide with 9-cyanoanthracene and 9-cyanophenanthrene are oxidized in high yield by sodium 9,10-anthraquinone- $\alpha$ -sulfonate in dimethyl sulfoxide to give the 9,10-dinitriles, and in the former case the yield is almost theoretical; the cyanoarenes are themselves prepared in excellent yield from the corresponding bromo compounds with cuprous cyanide in dimethyl sulfoxide. Whereas the 9-cyanoanthracene-cyanide mixture shows signs of electron exchange and is oxygenated, the 9-cyanophenanthrene-cyanide mixture exhibits neither of these properties.

Sodium 9,10-anthraquinone- $\alpha$ -sulfonate ( $\alpha$ -SAS) recently has been shown to be a most expedient reagent for the abstraction of hydride ions from certain preformed carbanions;<sup>2</sup> such carbanions have been generated in dipolar aprotic solvents, especially dimethyl sulfoxide (DMSO), by the facile reaction of cyanide ion with hydrocarbons possessing a polarized carbon-carbon double bond. The attack of cyanide ion upon a derivative of an aromatic system, 9-nitroanthracene, was the subject of a previous publication from this laboratory,<sup>3</sup> and, because of the remarkable reactivity exhibited by this compound, we have now examined the reaction of sodium cyanide with the 9-cyano derivatives of anthracene and phenanthrene. The incidental formation of the 9,10-dinitriles of anthracene<sup>3</sup> and phenanthrene<sup>4</sup> in experiments where the corresponding cyano nitro compounds were prepared in aprotic

solvents with alkali metal cyanides provided a precedent for the choice of the particular cyanoarenes as substrates.

Treatment of DMSO solutions<sup>5</sup> of the 9-cyanoarenes, under nitrogen, with sodium cyanide in the presence of  $\alpha$ -SAS, resulted in the immediate formation of an intense magenta color. This color has been evident in previous cyanation reactions employing this oxidizing agent,<sup>2</sup> and its generation is considered due to the formation of the hydroquinone ion. Its appearance therefore provides a visual indication that an oxidation-reduction process is taking place and that, in turn, there has been a reaction between the substrate and sodium cyanide. This color indication is particularly helpful if a solution of the substrate and sodium cyanide does not develop any color of its own, as was found to be the case with 9-cyanophenanthrene.

The cyanation of 9-cyanoanthracene with sodium cyanide and  $\alpha$ -SAS was accomplished very smoothly, and a 94% yield of the 9,10-dinitrile was achieved from

(1) Grateful acknowledgement is made to the U. S. Army Research Office [grants DA-ARO(D)-G679 and G857] for the support of this work.

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(3) R. G. Landolt and H. R. Snyder, *ibid.*, **33**, 403 (1968).

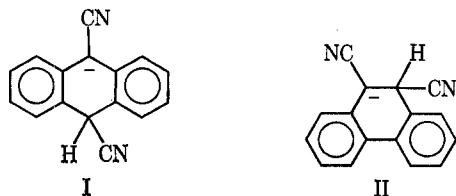
(4) W. L. Mosby, *ibid.*, **24**, 421 (1959).

(5) DMSO was chosen for the solvent as it is superior to DMF for dissolving  $\alpha$ -SAS.

a 2-hr experiment run at an optimum temperature of 80°. 9-Cyanophenanthrene was more difficult to cyanate, and a reaction time of some 70 hr was necessary to obtain a maximum yield of the dicyanophenanthrene (74%). Attempts to shorten this time by raising the temperature were unsuccessful, as the reaction began to yield tarry side products. At 130° no dinitrile was isolated at all, and a 60% recovery of unchanged 9-cyanophenanthrene was realized after 40 hr. At this high temperature, it is quite possible that considerable reaction takes place between the quinone and sodium cyanide, at the expense of cyanation of the 9-cyanophenanthrene.

As mentioned above, solutions containing only 9-cyanophenanthrene and sodium cyanide in DMSO or dimethylformamide (DMF) remained colorless under nitrogen, and work-up of the solutions after as long as 40 hr at 80° afforded only the unchanged nitrile in 90% yield. Such solutions were found unreactive to oxygen, and the same experiments run in the presence of air yielded a similar recovery of starting material. In contrast, however, solutions of 9-cyanoanthracene in the same solvents, under nitrogen, rapidly became a deep red-purple when sodium cyanide was added, and from these solutions similar amounts of 9-cyanoanthracene and 9,10-dicyanoanthracene (30–35%) were obtained after 17 hr. In the presence of air, the color of the solutions was darker, becoming quite black within a minute of adding the sodium cyanide, and work-up of a DMF solution after 1 hr at 80° yielded 46% 9,10-dicyanoanthracene as well as 12% 9-cyano-10-anthranol.

The accepted mechanism for the reaction of molecular oxygen with carbanions is a free-radical chain process involving one-electron transfer.<sup>6</sup> Electron transfer is also widely recognized to occur between carbanions and their parent unsaturated systems, and leads to the formation of products of nucleophilic substitution.<sup>7,8</sup> If it is assumed then that cyanide ion attack on the cyanoarenes results in the initial formation of the resonance-stabilized carbanions I and II, the observed differences in the behavior of the two cyanoarene-



cyanide mixtures would indicate that carbanion I readily undergoes electron transfer whereas carbanion II does not. If this is the case, the successful oxidation of the 9-cyanophenanthrene-cyanide mixture by  $\alpha$ -SAS may reflect a mechanism involving a true hydride transfer, rather than an electron transfer. Lead dioxide, which is frequently used as an electron abstractor<sup>9</sup> and which has been used with good success for oxidizing certain other cyano carbanions in aprotic

solvents,<sup>2</sup> was found to be virtually ineffective for oxidizing a cyanide solution of 9-cyanophenanthrene.

Factors known to influence the ease of electron transfer between a carbanion and its parent unsaturated system include the electron affinity of the parent system<sup>10</sup> and the relative stabilities of the carbanion and corresponding radical species.<sup>11</sup> Anthracene has a high electron affinity, particularly when compared with phenanthrene,<sup>12</sup> and its anion radical has been shown to be generated extremely readily.<sup>6</sup> It is therefore not surprising that the cyanide solutions of 9-cyanoanthracene show signs of radical activity; the presence of a nitrile group in an arene is known to greatly enhance its electron affinity.<sup>13</sup> It has been reported that basic DMSO solutions of dihydrophenanthrene show a very poor rate of reaction with molecular oxygen, compared with similar solutions of dihydroanthracene,<sup>14</sup> and it could be that the nitrile group in the related structure of carbanion II provides the extra stabilization necessary<sup>15</sup> to prevent this carbanion from undergoing any electron abstraction by oxygen or 9-cyanophenanthrene. A mixture containing 9-cyanophenanthrene and sodium cyanide in the presence of anthracene as an electron acceptor did develop a pink color under nitrogen, but work-up of this experiment after 40 hr gave an 84% recovery of the unchanged nitrile and only a trace (<1%) of 9,10-dicyanophenanthrene. Although carbanion I is also stabilized by a nitrile group, this is evidently not sufficient to offset the influence of the strong electron-accepting power of 9-cyanoanthracene, and the presence of two phenyl substituents is also likely to stabilize the radical intermediate rather than carbanion I.<sup>11</sup>

The isolation of 9,10-dicyanophenanthrene from cyanation of 9-cyanophenanthrene appears to be its first chemical preparation<sup>16</sup> in good yield. 9,10-Dicyanoanthracene has been frequently prepared by dibromination of anthracene and subsequent reaction of the dibromo compound with cuprous bromide.<sup>17</sup> Our preparation of this dinitrile requires an extra step when started with anthracene, since 9-bromoanthracene gives only a trace amount (<1%) of the dinitrile on treatment with sodium cyanide and  $\alpha$ -SAS. However, if 9-cyanoanthracene is available, the direct cyanation of this compound removes the previous need to brominate it first.<sup>18</sup> The preparation of 9-cyanoanthracene and 9-cyanophenanthrene from the bromo compounds with cuprous cyanide in refluxing DMSO is an improvement on various previous methods which have

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(12) N. S. Hush and J. A. Pople, *Trans. Faraday Soc.*, **51**, 600 (1955).

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(15) The ease of oxidation of a variety of substituted compounds in basic solution has been found to decrease with the increasing capacity of the substituents to conjugate with and thereby stabilize the carbanion intermediates.<sup>11</sup>

(16) M. V. Sargent and C. J. Timmons have previously isolated the dinitrile in 60% yield by a photolysis of  $\alpha, \alpha'$ -dicyano-*trans*-stilbene [*J. Chem. Soc.*, 5544 (1964)].

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(6) G. A. Russell, *Amer. Chem. Soc., Div. Petrol. Chem., Reprints*, **10** (2), A-7 (1965).

(7) G. A. Russell and W. C. Danen, *J. Amer. Chem. Soc.*, **90**, 347 (1968), and references cited therein.

(8) The cyanation of certain hydrocarbons under nitrogen by sodium cyanide has been interpreted in terms of electron-transfer processes.<sup>2</sup>

(9) L. F. Fieser and M. Fieser in "Reagents for Organic Synthesis," John Wiley & Sons, Inc., New York, N. Y., 1967, p 533.

entailed much longer reaction times and have employed pyridine,<sup>19</sup> DMF,<sup>20</sup> N-methylpyrrolidone,<sup>21</sup> and dry conditions.<sup>22</sup> DMSO has already been noted to be superior to these solvents in promoting nucleophilic displacement from naphthyl halides with copper salts.<sup>23,24</sup>

In the overall preparation of the 9,10-dinitriles of anthracene and phenanthrene from their 9-bromo compounds, with cuprous cyanide and then with sodium cyanide, a striking example of how a nitrile group is introduced into an aromatic system by two quite different mechanisms is provided. Although an aryl halide-cuprous salt substitution is considered to require the initial formation of a complex, involving bonding between the copper atom and the halogen of the aryl halide,<sup>24</sup> nucleophilic attack of an aromatic system by an alkali metal salt is recognized to proceed through a charged intermediate<sup>25</sup> such as I or II. The introduction of a substituent in place of hydrogen in a polarized, unsaturated system through nucleophilic attack and then oxidation of the charged intermediate is a novel concept.<sup>2</sup> The cyanations of 9-cyanoanthracene and 9-cyanophenanthrene are believed to be the first instances of the use of this kind of substitution to prepare aryl derivatives in high yield.

### Experimental Section<sup>26</sup>

**Materials.**—Commercially available analytical grade DMF and DMSO were employed, after they had been stored over Linde Type 4a Molecular Sieves for at least 2 weeks. Finely divided sodium cyanide (98%) and  $\alpha$ -SAS were dried for 24 hr at 110° under vacuum, and the sodium cyanide was stored over calcium sulfate in a tightly closed container. Cuprous cyanide was used in powder form without special drying. 9-Bromoanthracene (Aldrich, mp 95–99°) was recrystallized from hexane (bp 60–68°) to give pale yellow crystals, mp 103–105° (lit.<sup>27</sup> mp 98–99°), and a private sample of 9-bromophenanthrene was similarly recrystallized to give colorless crystals, mp 64–65.5° (lit.<sup>28</sup> mp 65–66°).

**General Procedure for Cyanation Reactions with Sodium Cyanide.**—A mixture of substrate and  $\alpha$ -SAS in DMSO was magnetically stirred under dry nitrogen in a 100-ml, three-necked flask equipped with a gas inlet tube dipping into the liquid, a condenser, and a calcium sulfate drying tube. The reaction mixture was heated and sodium cyanide was added after 1 hr. On work-up, the reaction was quenched with 10 ml of water (degassed with nitrogen) and the contents of the flask were poured into 300 ml of water containing 50 ml of a saturated solution of ammonium chloride. The aqueous mixture was filtered hot to retain the quinone in solution.

**Cyanation of 9-Cyanoanthracene. Preparation of 9,10-Dicyanoanthracene.**—Upon addition of sodium cyanide (0.40 g, 0.008 mol) to 9-cyanoanthracene (0.61 g, 0.003 mol) and  $\alpha$ -SAS (1.51 g, 0.005 mol) in DMSO (80 ml) at 80°, an intense magenta color developed, and a yellow, crystalline material separated. The reaction was worked up after 1.75 hr and the solid product was recrystallized from chloroform to yield golden yellow crystals (0.64 g, 94%) of 9,10-dicyanoanthracene. The melting point (335°) and infrared spectrum were in agreement with those previously reported for this compound.<sup>3</sup>

**Cyanation of 9-Cyanophenanthrene. Preparation of 9,10-Di-**

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(24) R. G. R. Bacon and H. A. O. Hill, *J. Chem. Soc.*, 1097 (1964).

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(26) Melting points are uncorrected and were determined with a Kofler microstage apparatus. Infrared spectra in potassium bromide disks were run on a Perkin-Elmer 521 infrared spectrophotometer.

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(28) W. E. Bachmann, *J. Amer. Chem. Soc.*, **56**, 1363 (1934).

**9-cyanophenanthrene.**—A mixture of 9-cyanophenanthrene (0.61 g, 0.003 mol), sodium cyanide (0.90 g, 0.018 mol), and  $\alpha$ -SAS (2.79 g, 0.009 mol) in DMSO (200 ml) was heated at 80° for 68 hr. The intensely magenta reaction mixture afforded a colorless solid product which was recrystallized from benzene to give colorless needles (0.50 g, 74%) of 9,10-dicyanoanthracene, mp 296–298° (lit.<sup>4</sup> mp 290–292°). An analysis and infrared spectrum confirmed the identity of this product.

An attempted cyanation of 9-cyanophenanthrene (0.61 g, 0.003 mol) with sodium cyanide (0.40 g, 0.008 mol) and lead dioxide (1.92 g, 0.008 mol) in DMF for 22 hr at 100° gave a tan solid which was chromatographed on silica gel. Elution with cyclohexane-benzene (1:1) gave 0.45 g (75%) of unchanged starting material, and elution with benzene gave 0.06 g (9%) of 9,10-dicyanoanthracene.

**Attempted Cyanation of 9-Cyanophenanthrene in the Presence of Anthracene.**—A mixture of 9-cyanophenanthrene (0.61 g, 0.003 mol), anthracene (0.53 g, 0.003 mol), and sodium cyanide (0.40 g, 0.008 mol) in DMF (80 ml) was heated at 80° for 40 hr under nitrogen. The color of the mixture was faintly pink after 5 hr but by the end of the reaction the color was a faint green. The colorless solid product was chromatographed on silica gel. Elution with cyclohexane gave 0.38 g of anthracene. Elution with cyclohexane-benzene (3:1) yielded 0.51 g (84%) of slightly impure 9-cyanophenanthrene, and elution with benzene gave 0.003 g (<1%) of 9,10-dicyanoanthracene.

**Reaction of 9-Cyanoanthracene with Sodium Cyanide. A. In the Presence of Air.**—A stream of dry air was bubbled through a mixture containing 9-cyanoanthracene (0.61 g, 0.003 mol) and sodium cyanide (0.30 g, 0.006 mol) in DMF (80 ml) at 80°. The mixture became black within 1 min and remained so until the reaction was quenched after 50 min. A brilliant yellow precipitate was collected (0.38 g) leaving a filtrate possessing a strong yellow-green fluorescence. Recrystallization of the solid product from chloroform yielded 0.31 g (46%) of 9,10-dicyanoanthracene, and from the aqueous solution, 0.08 g (12%) of 9-cyano-10-anthranol [mp 258–265° from methanol (lit.<sup>3</sup> mp 255–265°)] was isolated by ether extraction and precipitation of the ammonium salt.<sup>3</sup> The identity of the cyanoanthranol was verified by comparison of its melting point and infrared spectrum with those of an authentic sample.

**B. In the Absence of an Oxidizing Agent.**—A mixture of 9-cyanoanthracene (0.61 g, 0.003 mol), sodium cyanide (0.40 g, 0.008 mol), and DMF (80 ml) was agitated at 80° for 17 hr under nitrogen. A red-purple color developed immediately and gradually darkened until it became quite black. A solution of the yellow solid product in chloroform was evaporated onto a small quantity of silica gel, and the impregnated silica was added to the top of a silica gel column. Elution with cyclohexane-benzene (1:1) gave 0.18 g (30%) of unchanged 9-cyanoanthracene as pale yellow needles, and elution with benzene alone yielded deep yellow needles of 9,10-dicyanoanthracene (0.24 g, 35%).

Similar reactions run in DMF for 45 hr and in DMSO for 17 hr provided comparable yields of the same compounds.

**9-Cyanoanthracene.**—A mixture of 9-bromoanthracene (0.51 g, 0.002 mol) and cuprous cyanide (1.79 g, 0.020 mol) in DMSO (80 ml) was refluxed under nitrogen for 2 hr. The clear yellow-orange solution was poured into a mixture of ammonium hydroxide and water (500 ml, 1:1). Thorough agitation and warming of the aqueous mixture caused a pale yellow precipitate to separate which was filtered dry (0.40 g). The product was dissolved in benzene and put on a short silica gel column. Elution with cyclohexane-benzene (3:1) yielded pale yellow needles of 9-cyanoanthracene [0.37 g, 91%, mp 175–176° (lit.<sup>19</sup> mp 174–175°)].

A reaction in which 9-bromoanthracene (0.77 g, 0.003 mol) in DMSO (80 ml) was treated with sodium cyanide (0.80 g, 0.016 mol) and  $\alpha$ -SAS (1.51 g, 0.005 mol) at 80° for 8 hr yielded a light tan solid which was chromatographed on silica gel to give 0.60 g (78%) of unchanged starting material by elution with cyclohexane and 5 mg of slightly impure 9,10-dicyanoanthracene by elution with cyclohexane-benzene (1:1).

**9-Cyanophenanthrene.**—This compound was prepared in a manner analogous to that used to prepare 9-cyanoanthracene. From 0.51 g (0.002 mol) of 9-bromophenanthrene, a colorless solid product was obtained which, on recrystallization from hexane-benzene (bp 60–68°), gave colorless needles of 9-cyanophenanthrene [0.375 g, 93%, mp 108–110° (lit.<sup>22</sup> mp 110°)].

**Registry No.**—9,10-Dicyanoanthracene, 1217-45-4; 9,10-dicyanoanthracene, 2510-54-5.