Thus, reduction of 1,4-9, 1,5-1, and 1,8-2 dimethoxy- and diethoxyanthraquinones with sodium borohydride, under reaction conditions identical with those employed for other substituted anthraquinones,[7] gave the corresponding 9,10-dihydro-9,10-anthracenediols **10**, **3**, and **4**, respectively, in 90–95% yield[9] (Scheme I). They were generally obtained as a mixture of cis and trans isomers; their composition was not further investigated, because the stereochemistry is irrelevant in the further transformations. These dihydrodiols were used without purification in the next reaction step, because attempted recrystallization frequently led to products that showed carbonyl groups in the infrared spectrum.[10] Addition of lithium or sodium to a solution of dihydrodiols **3a,b** and **4a,b** in a mixture of anhydrous ammonia, tetrahydrofuran, and ethanol gave dihydroanthracenes **5a,b** and **6a,b**, respectively, in 95% yield. As expected, no reduction of the alkoxy-substituted aromatic moieties was observed. Dihydroanthracenes **5** and **6** were smoothly dehydrogenated with an equivalent amount of *p*-chloranil in toluene to give alkoxyanthracenes **7** and **8** in 85% yield.

Reductive elimination of the hydroxyl groups of dihydrodiol **10a,b** was accompanied by reduction of the unsubstituted aromatic ring resulting in the formation of tetrahydroanthracene **11a,b** (95%). Dehydrogenation with the calculated amount of *p*-chloranil gave 1,4-dialkoxyanthracenes **12a,b** (65%). Treatment of dihydrodiol **14a,b**, derived from 2,6-dialkoxyanthraquinone, with alkali metal in liquid ammonia resulted in reduction of both aromatic nuclei with concomitant hydroxyl group elimination to give hexahydroanthracene **15a,b** (70%). Dehydrogenation as above gave 2,6-dialkoxyanthracene **16a,b** (65%).

These results demonstrate that the alkoxyanthraquinone → alkoxyanthracene conversion via the 9,10-dihydroanthracene oxidation stage is an attractive strategy for peri-substituted derivatives. The synthetic sequence may also be employed for the synthesis of other alkoxyanthracenes, although in lower overall yield.

Experimental Section

General. NMR spectra were recorded in $CDCl_3$ with a Varian A-60 spectrometer, using Me_4Si as an internal standard. Mass spectra were recorded on a Perkin-Elmer 137 spectrophotometer. All melting points are uncorrected. Microanalyses were done by Galbraith Laboratories, Knoxville, TN.

General Procedure for Alkoxy-9,10-anthraquinone Reductions. 9,10-Dihydro-9,10-anthracenediols. Sodium borohydride (0.03 mol) was added in small portions to a stirred suspension of the anthraquinone (0.01 mol) in methanol (75 mL) at 0–5 °C. Stirring was continued at this temperature for an additional 2 h. The reaction mixture was poured in water and the resulting precipitate was collected and was washed well with water. The yield of product was 80–95%. The dihydrodiols showed no residual carbonyl absorptions in their infrared spectra and gave satisfactory NMR spectra.

General Procedure for Conversion of 9,10-Dihydro-9,10-anthracenediols to Reduced Anthracenes. Lithium (or sodium) metal (4–8 equiv) was added in small portions to a solution of the 9,10-dihydro-9,10-anthracenediol (0.01 mol) in a mixture of anhydrous ammonia (300 mL), tetrahydrofuran (100 mL), and absolute ethanol (30 mL). The mixture was stirred for an additional 0.5 h. After evaporation of the ammonia, water was added and the resulting mixture was extracted with ether. The ether extracts were washed with water and dried. The ether was removed on a rotary evaporator and the residue was recrystallized from methanol or ethanol. Yields, melting points, and spectroscopic data are collected in Table I.

General Procedure for Aromatization of Reduced Anthracenes. A mixture of the reduced alkoxyanthracene (dihydro, tetrahydro, or hexahydro) (0.01 mol) and *p*-chloranil (slight excess over the theoretical amount) in 100 mL of toluene was refluxed with stirring for 1 h. The mixture was poured in water and was then extracted with ether. The ether extracts were washed successively with aqueous sodium hydrosulfite, potassium hydroxide, and finally water. The residue obtained after evaporation of the solvent was dissolved in benzene and percolated through a short column of Florisil to afford pure alkoxyanthracenes (Table I).

Acknowledgment. Financial support of this work by the National Science Foundation is gratefully acknowledged. We thank Professor M. Szwarc for his support.

Registry No. **1a**, 6448-90-4; **1b**, 22924-22-7; **2a**, 6407-55-2; **2b**, 16294-26-1; **3a**, 75829-87-7; **3b**, 75829-88-8; **4a**, 75829-89-9; **4b**, 75829-90-2; **5a**, 75829-91-3; **5b**, 75829-92-4; **6a**, 75829-93-5; **6b**, 75829-94-6; **7a**, 16294-32-9; **7b**, 75829-95-7; **8a**, 16294-34-1; **8b**, 75829-96-8; **9a**, 6119-74-0; **9b**, 75829-97-9; **10a**, 75829-98-0; **10b**, 75829-99-1; **11a**, 75847-32-4; **11b**, 75847-33-5; **12a**, 13076-29-4; **12b**, 75830-00-1; **13a**, 963-96-2; **13b**, 24884-87-5; **14a**, 75830-01-2; **14b**, 75830-02-3; **15a**, 75930-03-4; **15b**, 75830-04-5; **16a**, 36319-03-6; **16b**, 75830-05-6.

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Preparation of Substituted 2-Pyridones by Thermal Rearrangement of Propargylic Pseudoureas. Improvements in the 2-Pyridone Yields by Variations of the Pseudourea NR_2 Substituent

Larry E. Overman* and Jan P. Roos

Department of Chemistry, University of California, Irvine, California 92717

Received September 30, 1980

We recently reported[1,2] that 3,6-dialkyl- (and -diaryl-) 2-pyridones[3] could be conveniently prepared from propargylic alcohols by the thermal rearrangement of propargylic pyrrolidine pseudoureas (Scheme I). When R^6 was an alkyl group, the overall yields of 2-pyridones obtained in this way were excellent. However, when R^6 was hydrogen or an aryl group, the 2-pyridone yields were not satisfactory, since competing nucleophilic addition of the imino nitrogen to the alkyne to give ultimately a substituted oxazole (see Scheme I) was a dominant reaction pathway.[4] Since intramolecular alkyne addition was not observed in related rearrangements[5] of less basic propargylic trichloroacetimidates, we felt that the 2-pyridone synthesis of Scheme I could be improved by employing pseudoureas *less basic* (*and consequently less nucleo-*

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(2) See also: Overman, L. E. *Acc. Chem. Res.* **1980**, *13*, 218.

(3) 2(1*H*)-Pyridinones.

(4) For other examples of this oxazole-forming reaction and leading references see: Overman, L. E.; Tsuboi, S; Angle, S. *J. Org. Chem.* **1979**, *44*, 2323.

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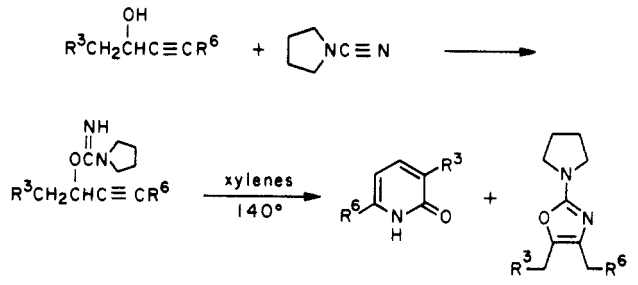
0022-3263/81/1946-0811$01.00/0

Table I. Preparation of Substituted 2-Pyridones and Oxazoles from Propargylic Pseudoureas. Dependence of Product Ratios on the Pseudourea NR_2 Group

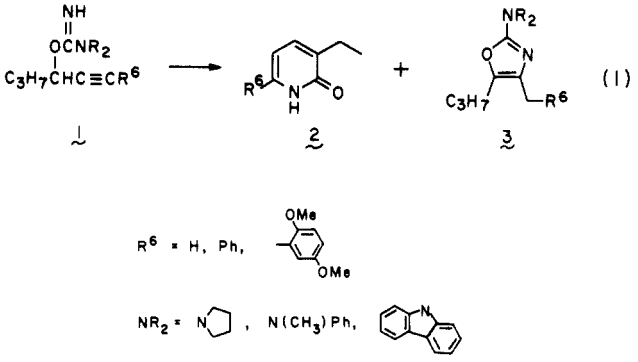
pseudourea[a]				thermal rearrangement[b]			
compd	R^6	NR_2	% yield	temp, °C	time, h	2-pyridone 2, % yield	oxazole 3, % yield
1a[c]	Ph	$N(CH_2)_4$	82[d]	140	20	54	38
1b	Ph	$N(CH_3)Ph$	76	140	28	69	(11)[e]
1c[c]	H	$N(CH_2)_4$	73[d]	140	24	13	31
1d	H	$N(CH_3)Ph$	65	140	20	41	27
1e	H	$N(C_6H_4)_2$[g]	59	205[f]	36	28	
1f	Ar[h]	$N(CH_2)_4$	79[d]	140	24	14	(16)[e]
1g	Ar[h]	$N(CH_3)Ph$	74	140	24	46	39
1h	Ar[h]	$N(C_6H_4)_2$[g]	51	205[f]	12		73

[a] Pseudoureas were chromatographically homogeneous (high-performance LC) unless noted otherwise. [b] Xylene solutions of the pseudourea (0.010 M) and diisopropylethylamine (0.040 M) were heated as indicated. Yields are for isolated pure products. [c] From ref 1. [d] Pseudourea not purified by high-performance LC. Purity ca. 85% by ^1H NMR. Yields are corrected for pseudourea purity. [e] Purity only ca. 90%. [f] Rearrangement conducted in a sealed Fisher-Porter bottle. [g] $N(C_6H_4)_2$ = carbazole. [h] Ar = 2,5-dimethoxyphenyl.

Scheme I



philic)[6] than the pyrrolidine pseudoureas examined previously. In this note we report the results of a comparative study of the rearrangements of a series of propargylic pseudoureas (eq 1) prepared from pyrrolidine (pK_a of BH^+ = 11.3),[7a] *N*-methylaniline (pK_a of BH^+ = 4.8),[7a] and carbazole (pK_a of $BH^+ \simeq -7$).[7b] *N*-Methylaniline pseudoureas are shown to be preferred for preparing, by propargylic pseudourea rearrangement, substituted 2-pyridones which have R^6 = H or aryl.



Results and Discussion

Pseudoureas 1 (see Table I) were prepared by base-catalyzed addition of the starting propargylic alcohol to 1-cyanopyrrolidine,[1,8a] methylphenylcyanamide (*N*-cyano-*N*-methylaniline),[8b] or 9-cyanocarbazole[9,10] by using a procedure we have detailed previously.[1b,11] Pseudoureas prepared from methylphenylcyanamide and 9-cyanocarbazole could be easily purified by preparative high-performance LC on silica gel using mixtures of triethylamine and hexane as the eluent. The more labile[1b] pyrrolidine pseudoureas were not easily purified in this fashion, and crude samples of the pyrrolidine pseudoureas, containing 5–15% of the starting alcohol, were therefore used.

Thermal rearrangements were conducted under identical conditions in xylene in the presence of *N*,*N*-diisopropylethylamine[1b] to give mixtures of substituted 2-pyridones and oxazole products (see Table I). In all cases, products were separated by silica gel chromatography and the crystalline 2-pyridones were further purified by recrystallization. The isolation and purification procedures were kept as similar as possible in experiments in which the pseudourea NR_2 substituent was varied. The *N*-methylaniline and pyrrolidine pseudoureas rearranged at comparable rates in refluxing xylene, and in all cases the 2-pyridone yields were somewhat higher with the less basic *N*-methylaniline pseudoureas. This trend was not, however, continued to the even less basic carbazole pseudoureas, which rearranged conveniently only at 205 °C and gave low yields of 2-pyridone products.

Conclusion

Of the pseudoureas examined to date, those derived from methylphenylcyanamide (*N*-cyano-*N*-methylaniline) are the intermediates of choice for preparing 2-pyridones which have hydrogen or aryl substituents at carbon-6. Further improvements in this 2-pyridone synthesis would still appear possible by examining additional pseudourea derivatives which combine low basicity with small steric size.

Experimental Section[12]

Representative Pseudourea Preparation. 1-(2,5-Dimethoxyphenyl)-1-hexyn-3-yl *N*-Methylaniline-*N*-carboximidate (1g). By method B of ref 1b, 1.17 g (5.0 mmol) of 1-(2,5-dimethoxyphenyl)-1-hexyn-3-ol (4)[13] was condensed with 1.99 g (15

(6) Nucleophilicity typically parallels basicity when the nucleophilic atom is unchanged and steric effects are similar. For a recent study see: Bordwell, F. G.; Hughes, D. L. *J. Org. Chem.* **1980**, *45*, 3314.

(7) (a) Wearst, R. C., Ed. "Handbook of Chemistry and Physics", 59th ed.; Chemical Rubber: Cleveland, 1978; p D199. (b) Chen, H. J.; Hakka, L. E.; Hinman, R. L., Kresge, A. J.; Whipple E. B. *J. Am. Chem. Soc.* **1971**, *93*, 5102.

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(9) Wentrup, C. *Tetrahedron* **1971**, *27*, 367.

(10) We have found that pure 9-cyanocarbazole melts at 103–104 °C and gives a correct combustion analysis of $C_{13}H_8N_2$.

(11) Method B of ref 1b was used. The procedure was identical with that described with the exception that 3 equiv of the cyanamine were employed.

(12) General procedures are summarized in ref 1b. Low-resolution CI mass spectra were determined with a Finnigan model 4000 GC/MS/DS.

mmol) of methylphenylcyanamide (mp 29.5–30.5 °C).[8b] Concentration gave a yellow liquid, TLC analysis (silica gel; 5:1:1 hexane–triethylamine–ethyl acetate) of which showed the presence of 4 (R_f 0.27), methylphenylcyanamide (R_f 0.37), and pseudourea **1g** (R_f 0.20). Immediate purification of this mixture by using a Waters Prep LC-500 and two Prep PAK-500 silica columns, using 5:1:1 hexane–triethylamine–ethyl acetate as the eluent, gave 1.35 g (74%) of pure **1g**: IR (film) 3360, 1630, 1050 cm^{-1}; ^1H NMR (60 MHz, CCl_4) δ 6.7–7.6 (m, Ar H), 5.7 (br t, J = 6 Hz, CHOR), 5.35 (s, NH), 3.75 (s, two OCH_3), 3.25 (s, NCH_3), 0.7-2.2 (m, $CH_2CH_2CH_3$).

Thermal Rearrangement of Pseudourea 1g. Preparation of 3-Ethyl-6-(2,5-dimethoxyphenyl)-2(1*H*)-pyridinone (2g) and 4-[(2,5-Dimethoxyphenyl)methyl]-5-propyl-2-(*N*-methylanilinyl)oxazole (3g). A solution of 350 mg (0.96 mmol) of pseudourea **1g**, 493 mg (3.8 mmol) of *N,N*-diisopropylethylamine, and 96 mL of xylene was degassed[14] and heated at reflux under nitrogen for 24 h. Concentration afforded a yellow semisolid which was chromatographed (silica gel, 1:1 hexane–acetone) to yield two fractions. The first fraction (R_f 0.65, 1:1 hexane–acetone) gave 135 mg (38%) of oxazole **3g**, a light yellow liquid: IR (film) 1740, 1660, 1590, 1390, 1220, 1050 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ 7.5–8.5 (m, Ar H), 3.7 (s, OCH_3 and $ArCH_2$), 3.4 (s, NCH_3), 2.4 (unsymm t, J = 7 Hz, $CH_2CH_2CH_3$), 1.1–1.9 (m, $CH_2CH_2CH_3$), 0.95 (unsymm t, J = 7 Hz, CH_3); mass spectrum (isobutane CI), m/z (relative intensity) 367 (100%, MH^+). The second fraction yielded 114 mg (46%) of 2-pyridone **2g**: mp 131–134 °C; TLC R_f 0.25 (1:1 hexane–acetone). An analytical sample was obtained by recrystallization from 1:2 chloroform–hexane: mp 136–137 °C; IR ($CHCl_3$) 3380, 1640, 1610, 1490, 1040 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ 7.25 (d, J = 7 Hz, C_4H), 6.8–7.2 (m, Ar H) 6.40 (d, J = 7 Hz, C_5H), 3.83 (s, 2 OCH_3), 2.56 (q, J = 7.5 Hz, CH_2CH_3), 1.20 (t, J = 7.5 Hz, CH_2CH_3); ^{13}C NMR ($CDCl_3$) δ 163.1 (C_2), 141.4 (C_6), 136.6 (C_4), 133.8 (C_3), 105.4 (C_5), and other peaks at 154.1, 150.8, 122.4, 115.9, 115.1, 113.2, 56.4, 55.9, 23.2, 12.7; mass spectrum (isobutane CI), m/z (relative intensity) 260 (100%, MH^+). Anal. Calcd for $C_{15}H_{17}NO_3$: C, 69.47; H, 6.61; N, 5.40. Found: C, 69.51; H, 6.69; N, 5.37.

Thermal Rearrangement of Pseudourea 1b. Preparation of 3-Ethyl-6-phenyl-2(1*H*)-pyridinone (2b). A solution of 346 mg (1.13 mmol) of pseudourea **1b**, 583 mg (4.2 mmol) of *N,N*-diisopropylethylamine, and 113 mL of xylene was degassed[14] and heated at reflux under nitrogen for 28 h. Concentration gave a yellow residue which was dissolved in 15 mL of hot acetone and slowly allowed to cool to 0 °C to afford 132 mg (59%) of 2-pyridone **2b**, mp 164–166 °C. Purification of the mother liquor by chromatography on silica gel (4:1 hexane–ethyl acetate) gave an additional batch of **2b** which was recrystallized from ethyl acetate at −15 °C. The total yield of pure 2-pyridone **2b** was 155 mg (69%), mp 164–165 °C (lit.[1b] mp 164–166 °C), identical by TLC, ^1H NMR, and ^{13}C NMR with an authentic specimen.[1b] Careful chromatographic purification of the mother liquor from a comparable reaction which employed 140 mg (0.46 mmol) of **1b** resulted in the isolation of 16 mg (11%) of oxazole **3b**, a yellow oil which was ca. 90% pure by ^1H NMR: ^1H NMR (60 MHz, CCl_4) δ 7.0–7.7 (m, Ph), 3.67 (s, CH_2Ph), 3.45 (s, NCH_3), 2.40 (t, J = 8 Hz, $CH_2CH_2CH_3$), 1.2–1.9 (m, CH_2CH_3), 0.9 (t, J = 7 Hz, CH_3).

Rearrangement of Pseudourea 1d. Preparation of 3-Ethyl-2(1*H*)-pyridinone (2d) and 4-Methyl-5-propyl-2-(*N*-methylanilinyl)oxazole (3d). A solution of 200 mg (0.87 mmol) of pseudourea **1d**, 450 mg (3.5 mmol) of diisopropylethylamine, and 87 mL of xylene was degassed,[14] heated at reflux for 20 h, and concentrated. Purification of the residue by silica gel chromatography (5:1 to 1:1 hexane–acetone) gave in the first fraction 54 mg (27%) of oxazole **3d**, a pale yellow oil (R_f 0.45, 3:1 hexane–acetone). An analytical specimen was obtained by chromatography on silica gel (80:18:2 hexane–acetone–triethylamine): IR (film) 1675, 1590, 1390, 1200, 1035 cm^{-1}; ^1H NMR (60 MHz, CCl_4) δ 6.8–7.4 (m, Ph), 3.45 (s, NCH_3), 2.5 (t, J = 7 Hz, $CH_2CH_2CH_3$), 1.95 (s, CH_3), 1.1–1.9 (m, CH_2CH_3), 0.9 (t, J = 7 Hz, CH_3); mass spectrum (isobutane CI), m/z (relative intensity) 231 (100%, MH^+); mol wt 230.140 (EI mass spectrum, $C_{14}H_{18}N_2O$ requires 230.142). A second fraction yielded a yellow solid (R_f 0.15, 3:1 hexane–acetone) which was recrystallized from 4:1 hexane–acetone to afford 44 mg (41%) of 2-pyridone **2d**, mp 120–121 °C. Recrystallization from hexane–acetone gave an analytical specimen, mp 121–121.5 C (lit.[1b] mp 120–121 °C), whose ^1H NMR spectra was identical with that of an authetic specimen.

Rearrangement of Pseudourea 1h. Preparation of 2-(9-Carbazolyl)-4-[(2,5-dimethoxyphenyl)methyl]-5-propyloxazole (3h). A dry Fisher–Porter bottle was charged with 25 mL of a xylene solution containing 106 mg (0.25 mmol) of pseudourea **1h** and 130 mg (1.0 mmol) of diisopropylethylamine. The bottle was degassed,[14] sealed, and heated at 205 °C for 12 h. Concentration afforded a yellow residue which did not show the presence of 2-pyridone **2h**[1b] by TLC analysis. Crystallization of this residue from acetone (−15 °C) gave 77 mg (73%) of white crystalline oxazole **3h**, mp 118–119 °C. An analytical specimen was obtained by recrystallization from acetone: mp 118.5–119.5 °C; IR ($CHCl_3$) 1590, 1450, 1240, 1045, 810 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ 6.6–8.5 (complex m, Ar H), 3.88 (s, CH_2Ar), 3.82 (s, OCH_3), 3.72 (s, OCH_3), 2.7 (t, J = 7 Hz, $CH_2CH_2CH_3$), 1.5–2.1 (m, $CH_2CH_2CH_3$), 1.0 (t, J = 7 Hz, CH_2CH_3); CI mass spectrum (isobutane), m/z (relative intensity) 427 (100%, MH^+). Anal. Calcd for $C_{27}H_{26}N_2O_3$: C, 76.03; H, 6.14. Found: C, 75.83; H, 6.20.

Thermal rearrangements of pseudoureas **1e** and **1f** were conducted in a similar fashion and the results of these experiments are summarized in Table I.

Acknowledgment. Support from the National Science Foundation (CHE-7901833), the Camille and Henry Dreyfus Foundation (teacher–scholar award to L.E.O.), and the Sloan Foundation (fellowship award to L.E.O.) is gratefully acknowledged. NMR and mass spectra were determined with spectrometers purchased with the assistance of NSF instrumentation grants.

Registry No. 1a, 73267-71-7; **1b**, 75918-66-0; **1c**, 62969-93-1; **1d**, 75918-67-1; **1e**, 75918-68-2; **1f**, 75918-69-3; **1g**, 75918-70-6; **1h**, 75918-71-7; **2b**, 73252-79-6; **2d**, 62969-86-2; **2g**, 75918-72-8; **3a**, 73252-80-9; **3b**, 75918-73-9; **3c**, 73252-77-4; **3d**, 75918-74-0; **3f**, 75918-75-1; **3g**, 75918-76-2; **3h**, 75918-77-3; 4, 75918-78-4; methylphenylcyanamide, 18773-77-8.

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Aromatization of Arene 1,2-Oxides. 1-Cyanobenzene Oxide

Herbert S.-I. Chao and Glenn A. Berchtold*

Department of Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

Received August 4, 1980

Previous investigations of the aromatization of arene 1,2-oxides in our laboratory have provided examples of arene 1,2-oxide aromatization reactions proceeding by all the possible general routes to ortho-substituted phenols and phenol with substituent loss.[1-3] Substituents at C_1 that are electron withdrawing, such as CO_2CH_3 and CHO, favor C_2–O cleavage of the arene 1,2-oxide. The ratio of C_2–O cleavage/C_1–O cleavage of 1-(carbomethoxy)benzene oxide in 1:1 tetrahydrofuran–water is 70:30 at pH 0.1 and 83:17 at pH 7, and C_2–O cleavage results in substituent

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0022-3263/81/1946-0813$01.00/0