The Synthesis of Novel Polycyclic Heterocyclic Ring Systems *via* Photocyclization. **19** [1,2]. Thieno[3',2':4,5]thieno[2,3-*c*]-naphtho[1,2-*f*]quinoline, Thieno[3',2':4,5]thieno[2,3-*c*]naphtho-[1,2-*f*][1,2,4]triazolo[4,3-*a*]quinoline and Thieno[3',2':4,5]-thieno[2,3-*c*]naphtho[1,2-*f*]tetrazolo[1,5-*a*]quinoline

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Photocyclization of 3-chloro-*N*-(3-phenanthryl)thieno[2,3-*b*]thiophene-2-carboxamide (**5**) yielded only one of the two possible structural isomers, thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]quinolin-6(5*H*)-one (**6**), which was further elaborated to afford the unsubstituted ring system **10**, its triazole **11** and tetrazole **12**. The structural confirmation of **10** was achieved by the total assignment of its ^{1}H and ^{13}C nmr spectra by the concerted utilization of two-dimensional nmr spectroscopic methods.

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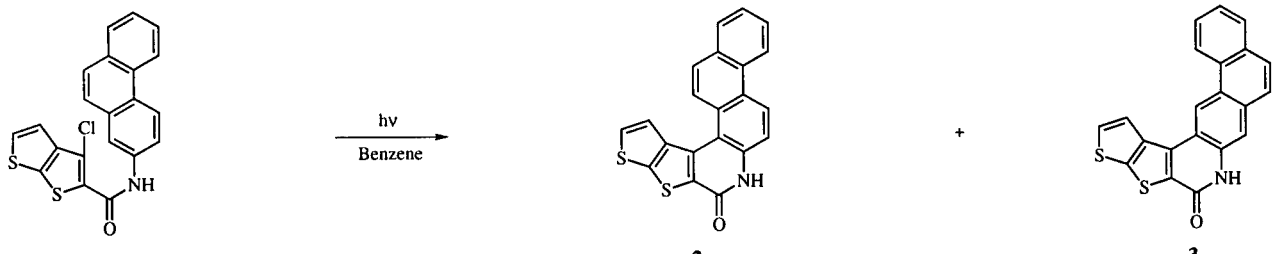
In a recent report, we demonstrated that the oxidative photocyclization of 3-chloro-*N*-(2-phenanthryl)thieno-[2,3-*b*]thiophene-2-carboxamide (**1**) in benzene yielded an isomeric mixture of thieno[3',2':4,5]thieno[2,3-*c*]naphtho-[2,1-*f*]quinolin-8(7*H*)-one (**2**) and thieno[3',2':4,5]thieno-[2,3-*c*]naphtho[1,2-*g*]quinolin-9(8*H*)-one (**3**) [2r] (Scheme 1). Concerning our previous studies [2e,2k,2p], it is of interest to know whether the photocyclization of 3-chloro-*N*-(3-phenanthryl)thieno[2,3-*b*]thiophene-2-carboxamide (**5**) gives a single product or an isomeric mixture of lactams. To this end we describe the synthesis of three novel polycyclic heterocyclic ring systems, thieno-[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]quinoline (**10**), thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*][1,2,4]triazolo-[4,3-*a*]quinoline (**11**) and thieno[3',2':4,5]thieno[2,3-*c*]-naphtho[1,2-*f*]tetrazolo[1,5-*a*]quinoline (**12**), and the confirmation of their structures by two-dimensional nmr spectroscopic methods.

Scheme 1



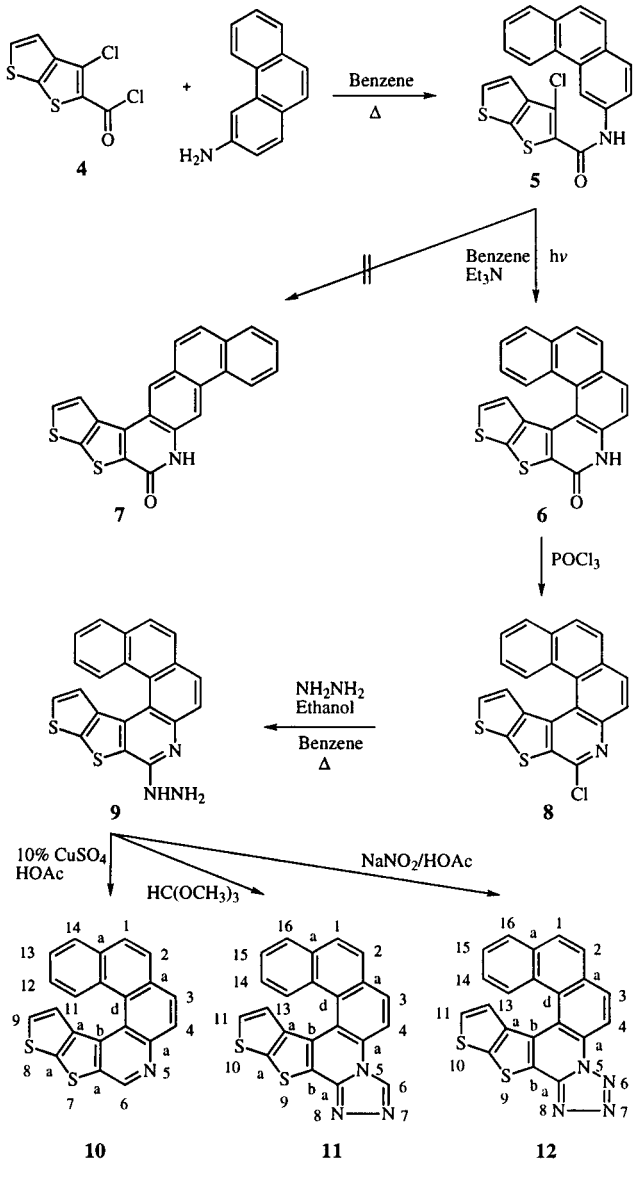
1 **2** **3**

When 3-chlorothieno[2,3-*b*]thiophene-2-carbonyl chloride (**4**) [2a,4,5] was allowed to react with 3-aminophenanthrene [6] in refluxing benzene, 3-chloro-*N*-(3-phenanthryl)thieno[2,3-*b*]thiophene-2-carboxamide (**5**) was obtained in 80% yield. Irradiation of the amide **5** in benzene solution containing triethylamine with a 450 watt Hanovia medium pressure mercury vapor lamp afforded only one of the two possible cyclization products in 86% yield. The structure of the product was tentatively assigned as thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]-quinolin-6(5*H*)-one (**6**) due to the lack of singlets in its proton nmr spectrum. Chlorination of the lactam **6** with phosphorus oxychloride afforded 6-chlorothieno-[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]quinoline (**8**) in 39% yield. Upon hydrazination of the chloride **8** with anhydrous hydrazine in a refluxing mixture of benzene and ethanol, 6-hydrazinothieno[3',2':4,5]thieno[2,3-*c*]naphtho-[1,2-*f*]quinoline (**9**) was obtained in 94% yield. The unsubstituted novel ring system was obtained in 63% yield by refluxing **9** with a mixture of 10% copper sulfate solution and aqueous acetic acid. The hydrazide **9** in refluxing trimethyl orthoformate and ethanol afforded thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*][1,2,4]triazolo[4,3-*a*]quinoline (**11**) in 59% yield, whereas treatment of **9** with sodium nitrite in 50% aqueous acetic acid resulted in a 58% yield of thieno[3',2':4,5]thieno[2,3-*c*]-naphtho[1,2-*f*]tetrazolo[1,5-*a*]quinoline (**12**) (Scheme 2).

NMR Spectroscopy.

The structure of compound **10** was fully supported by

Scheme 2



an in-depth spectral analysis, *i.e.* the total assignments of ^{1}H and ^{13}C spectra of **10** achieved by two-dimensional nmr methods (COSY [7], NOESY [8], HMQC [9], HMQC-TOCSY [10], and HMBC [11]). These spectra attested to the presence of a single compound free of isomers.

The COSY spectrum of **10** exhibited one four-spin and three two-spin systems as expected. The orientations of these spin systems relative to one another were established by the NOESY spectrum of **10**. Thus, all protons of **10** could be assigned through the concerted use of COSY and NOESY. Consequently, the protonated carbons of **10** could be elucidated through the HMQC except C1, C3 and C14 due to the corresponding congested protons. However, C1, C3 and C14 could be differentiated by the HMQC-TOCSY experiment. The assignment of the remaining quaternary carbons requires a long-range heteronuclear chemical shift correlation (HMBC) spectrum of **10**.

The isolated spin, H6 resonating at 9.39 ppm, is the most convenient entry point for the assignment process. Three long-range couplings to H6 are observed for quaternary carbons at 135.5, 138.3 and 145.6 ppm. The carbon resonating furthest downfield at 145.6 ppm exhibits a long-range correlation to H3 and thus is assigned as C4a. The remaining two quaternary carbons show no other long-range couplings and the three-bond coupling from H10-C10b is unreliable based on Karplus relationships. Based on chemical shift arguments, in conjunction with more important T_1 relaxation considerations C6a can be differentiated from C10b. Since T_1 relaxation is dependent on the distance to the nearest proton in an inverse sixth power relationship, C6a is expected to relax much more efficiently than C10b. The less efficient relaxation of C10b will result in a corresponding greater degree of resonance saturation diminshing signal intensity relative to that of C6a. Thus, the quaternary carbon resonating at 135.5 ppm is assigned as C10b, whereas the resonance at 138.3 ppm is assigned as C6a. Given the resonance of H4 at 8.23 ppm, the quaternary carbon resonating at 131.6 ppm reveals another long-range correlation to H1 and thus is assigned to C2a. Hence, the resonance at 119.3 ppm coupling to H4 is attributed to C10c. Likewise, knowing H2 resonating at 7.95 ppm facilitates the assignment of C10d and C14a at 126.1 and 132.1 ppm, respectively. The quaternary carbon C10e resonating at 130.2 ppm can be extracted from its long-range correlations to H1, H12, and H14. To this point the assignment of all the quaternary carbons on the phenanthrene and pyridine moieties are established through the HMBC experiment.

Differentiating the remaining two quaternary carbons on the thienothiophene moiety, C7a and C10a, is not possible because both of them exhibit long-range couplings to H9 and H10. However, in view of the structure of **10** we expect C10a is shielded more than C7a because C10a is located in the helical bay region [12]. Thus, based upon our previous experience [2o,2r] and chemical shift consideration, we assign the carbon resonating at 142.0 ppm as C10a and 142.6 ppm as C7a. The complete chemical shift assignments of **10** are presented in Table 1.

The total assignments of the proton and carbon-13 spectra of the triazole **11** and tetrazole **12** were achieved by utilizing the same strategy just discussed above and are given in Tables 2 and 3.

In conclusion, we have synthesized the novel heterocyclic ring system, thieno[3',2':4,5]thieno[2,3-*c*]naphtho-[1,2-*f*]quinoline (**10**), and its triazole **11** and tetrazole **12**.

Table 1

^1H and ^{13}C NMR Chemical Shift Assignments and Observed Proton-Carbon Multiple-Bond Correlations for Compound **10** in Deuteriochloroform at 298°K at Observation Frequencies of 360.13 and 90.56 MHz, Respectively

Position	δ H	δ C	Two-Bond Correlation	Three-Bond Correlation
1	8.03	128.6		H14
2	7.95	126.0		H3
2a		131.6		H1, H4
3	8.04	128.9		H2
4	8.23	128.9		
4a		145.6		H3, H6
6	9.39	144.4		
6a		138.3	H6	
7a		142.6		H9, H10
9	7.00	125.9	H10	
10	6.03	124.8	H9	
10a		142.0	H10	H9
10b		135.3		H6
10c		119.3		H4
10d		126.0		H2, H3, H11
10e		130.2		H1 H12, H14
11	8.12	129.0		H13
12	7.25	125.9		H14
13	7.56	126.3		H11
14	8.05	128.0		H1, H12
14a		132.1		H2, H11, H13

Table 2

^1H and ^{13}C NMR Chemical Shift Assignments and Observed Proton-Carbon Multiple-Bond Correlations for Compound **11** in Deuteriochloroform Containing Two Drops of Deuterated Trifluoroacetic Acid at 298°K at Observation Frequencies of 360.13 and 90.56 MHz, Respectively

Position	δ H	δ C	Two-Bond Correlation	Three-Bond Correlation
1	8.13	130.3		H16
2	7.98	125.5		H3
2a		132.6		H1, H4
3	8.35	132.1		H2
4	8.40	114.1		
4a		127.5		H3
6	9.98	143.0		
8a		142.0		
8b		120.6		
9a		146.0		H11, H12
11	7.08	128.9	H12	
12	5.86	123.7	H11	
12a		143.0	H12	H11
12b		135.3		
12c		116.7		H4
12d		127.3		H2 H3, H13
12e		129.3		H1, H14, H16
13	8.05	128.8		H15
14	7.29	126.8		H16
15	7.66	128.4		H13
16	8.10	128.5		H1, H14
16a		132.9		H2, H13, H15

Table 3

^1H and ^{13}C NMR Chemical Shift Assignments and Observed Proton-Carbon Multiple-Bond Correlations for Compound **12** in Deuteriochloroform Containing Two Drops of Deuterated Trifluoroacetic Acid at 298°K at Observation Frequencies of 360.13 and 90.56 MHz, Respectively

Position	δ H	δ C	Two-Bond Correlation	Three-Bond Correlation
1	8.06	129.5		H16
2	7.93	125.8		H3
2a		132.3		H1, H4
3	8.23	131.1		H2
4	8.80	115.4		
4a		128.8		H3
8a		143.8		
8b		123.1		
9a		143.1		H11, H12
11	7.02	127.7	H12	
12	5.86	123.8	H11	
12a		143.6	H12	H11
12b		133.8		
12c		116.0		H4
12d		126.9		H2, H3, H13
12e		129.4		H1 H14, H16
13	8.03	128.9		H15
14	7.23	126.3		H16
15	7.61	127.9		H13
16	8.06	128.2		H1, H14
16a		132.7		H2, H13, H15

Their structures have been determined and the proton and carbon nmr spectra unequivocally assigned by the concerted utilization of two-dimensional nmr spectroscopic methods.

EXPERIMENTAL

Melting points were determined on a Thomas-Hoover melting apparatus and are uncorrected. The ir spectra were recorded on a Beckman FT1100 spectrometer as potassium bromide pellets and frequencies are expressed in cm^{-1}. The ^1H nmr spectra of the intermediates were obtained on a JEOL FX-90Q or on a Bruker AMX360 MHz NMR spectrometer in the solvent indicated with TMS as the internal standard and chemical shifts are reported in ppm (δ) and J values in Hz. Analysis (tlc) were performed on Sigma precoated silica gel plates containing a fluorescent indicator. Elemental analyses were performed by M-H-W Laboratories, Phoenix, Arizona.

The ^1H and ^{13}C spectra of **10**, **11** and **12** were acquired on a Bruker AMX360 MHz NMR spectrometer operating at an observation frequency of 360.13 MHz for ^1H and 90.56 for ^{13}C. All experiments were performed using an inverse-geometry 5 mm broad band probe. Pulse widths (90°) for ^1H and ^{13}C were 7.8 and 15.6 μsec, respectively. The COSY spectra were recorded using the Bruker pulse program (*COSY90*) [7]. The NOESY experiments were performed using the Bruker pulse program (*noesytp*) [8]. The HMQC experiments were performed using the Bruker pulse program (*invbdgtp*) with the BIRD sequence optimized for direct couplings (165 Hz $^1J_{CH}$) [9]. The

HMQC-TOCSY were obtained using the Bruker pulse program (*invbmltp*) [10] with decoupling and acquisition initiated simultaneously. The HMBC spectra were obtained using the Bruker pulse program (*inv4lplrnd*) [11] optimized for 10 Hz $^3J_{CH}$ couplings.

3-Chloro-*N*-(3-phenanthryl)thieno[2,3-*b*]thiophene-2-carboxamide (**5**).

A mixture of 2.0 g (8.43 mmoles) of carbonyl chloride **4** [2a,4,5] and 1.63 g (8.43 mmoles) of 3-aminophenanthrene [6] in 50 ml of benzene was heated under reflux for 4 hours. After cooling to room temperature, the mixture was evaporated to dryness *in vacuo*. The solid was recrystallized from benzene to afford 2.65 g (6.73 mmoles, 80%) of amide **5** as yellowish crystals, mp 247-249°; ir (potassium bromide): 3397 (NH stretching), 1653 (C=O stretching); ^{1}H nmr (deuteriochloroform): 50°, δ 7.24-7.93 (m, 9H, ArH), 8.65-8.76 (m, 1H, H-2'), 9.00 (br s, 1H, NH), 9.19 (d, $J_{2',4'}$ = 2.0 Hz, 1H, H-4').

Anal. Calcd. for $C_{21}H_{12}ClNOS_2$: C, 64.03; H, 3.07; N, 3.56; S, 16.28. Found: C, 64.21; H, 3.11; N, 3.34; S, 16.07.

Thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]quinolin-6(5*H*)-one (**6**).

A mixture of 0.50 g (1.27 mmoles) of amide **5**, 0.13 g (1.28 mmoles) of triethylamine in 500 ml of benzene was irradiated with a 450 watt Hanovia medium pressure mercury vapor lamp. A slow stream of air was passed through the solution during the course of the reaction. The precipitate was collected by filtration and washed with water to afford 0.39 g (1.09 mmoles, 86%) of lactam **6**, mp >280°; ir (potassium bromide): 1635 (C=O stretching); ^{1}H nmr (DMSO-d_6): δ 5.84 (d, $J_{9,10}$ = 5.4 Hz, 1H, H-10), 7.10-7.36 (m, 2H, including one doublet at δ 7.25 with $J_{9,10}$ = 5.4 Hz for H-9), 7.48-8.14 (m, 7H, ArH). This compound was used in the next step without further purification because of its low solubility.

6-Chlorothieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]quinoline (**8**).

A mixture of 1.48 g (4.14 mmoles) of lactam **6** and 60 ml of phosphorus oxychloride was heated at 100-110° for four hours. After cooling to room temperature the mixture was poured into 500 ml of ice water with vigorous stirring. The precipitate was collected by filtration, washed with water and dried. The solid was recrystallized from benzene to yield 0.61 g (1.62 mmoles, 39%) of **8** as brown prisms, mp 208-211°; tlc (benzene) R_f 0.52; ir (potassium bromide): 3068 (aromatic CH stretching); ^{1}H nmr (deuteriochloroform): δ 5.92 (d, $J_{9,10}$ = 5.5 Hz, 1H, H-10), 6.96 (d, $J_{9,10}$ = 5.5 Hz, 1H, H-9), 7.20-7.62 (m, 2H, ArH), 7.81-8.18 (m, 6H, ArH).

Anal. Calcd. for $C_{21}H_{10}ClNS_2$: C, 67.10; H, 2.68; N, 3.73; S, 17.06. Found: C, 67.25; H, 2.51; N, 3.52; S, 17.24.

6-Hydrazinothieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]quinoline (**9**).

To a boiling solution of 0.4 g (1.06 mmoles) of chloride **8** in 20 ml of benzene and 40 ml of absolute ethanol 10 ml of anhydrous hydrazine was added dropwise over a period of 30 minutes. The resulting solution was heated at 100-110° for 20 hours. The mixture was concentrated to 5 ml under reduced pressure. After cooling to room temperature the solid was collected by filtration and washed with ice-chilled absolute ethanol to obtain 0.37 g (1.0 mmole, 94%) of hydrazine **9** as a yellow powder, mp 252-254° dec; tlc (benzene) R_f 0.093; ir (potassium bromide): 3356, 3201 (NH stretching), 3093 (aromatic CH stretching); ^{1}H nmr (DMSO-d_6): δ 4.93 (br s, 2H, NH_2), 5.80 (d, $J_{9,10}$ = 5.4 Hz, 1H, H-10), 7.18-7.25 (m, 2H, ArH), 7.51-7.55 (m, 1H, ArH), 7.77 (d, J = 8.3 Hz, 1H, ArH), 7.95-8.09 (m, H, ArH), 8.69 (s, 1H, NH). This compound was used for the next step without further purification because of its low solubility and thermal stability.

Thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]quinoline (**10**).

To a mixture of 0.12 g (0.32 mmole) of hydrazine **9** in 12 ml of glacial acetic acid and 5 ml of water was added dropwise 15 ml of a 10% copper sulfate solution and the mixture was heated under reflux for 20 hours. After cooling to room temperature the mixture was neutralized with a 2*N* sodium hydroxide solution. The solid was collected by filtration and washed with water. The solid was dissolved in 10 ml of benzene and applied to a column chromatography eluted with chloroform/ethyl acetate (5:2). The fractions containing the product were pooled and evaporated to dryness *in vacuo*. The solid was recrystallized from cyclohexane to give 0.068 g (0.20 mmole, 63%) of the unsubstituted product **10** as yellowish shiny crystals, mp 252-254°; ir (potassium bromide): 3050 (aromatic CH stretching).

Anal. Calcd. for $C_{21}H_{11}NS_2$: C, 73.87; H, 3.25; N, 4.10. Found: C, 73.77; H, 3.30; N, 4.07.

Thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*][1,2,4]triazolo[4,3-*a*]-quinoline (**11**).

A mixture of 0.10 g (0.27 mmole) of hydrazine **9**, 20 ml of absolute ethanol and 10 ml of trimethyl orthoformate was heated under reflux for 8 hours. After cooling to room temperature the mixture was evaporated to dryness *in vacuo*. The solid was recrystallized from benzene to give 0.06 g (0.16 mmole, 59%) of triazole **11** as yellow crystals, mp >300°; tlc (chloroform:ethyl acetate, 4:1) R_f 0.18; ir (potassium bromide): 3044 (aromatic CH stretching).

Anal. Calcd. for $C_{22}H_{11}N_3S$: C, 69.27; H, 2.91; N, 11.02. Found: C, 69.45; H, 3.02; N, 10.83.

Thieno[3',2':4,5]thieno[2,3-*c*]naphtho[1,2-*f*]tetrazolo[1,5-*a*]-quinoline (**12**).

To an ice-chilled mixture of 0.10 g (0.27 mmole) of hydrazine **9** in 10 ml of a 50% (v/v) acetic acid was added dropwise a solution of 0.06 g (0.87 mmole) of sodium nitrite in 5 ml of water. The reaction mixture was stirred at room temperature for 20 hours. The solid was collected by filtration and washed with water. The dried solid dissolved in 20 ml of chloroform and applied to a column chromatography eluted with chloroform/-ethyl acetate (5:1). The fractions containing the tetrazole were pooled and evaporated to dryness *in vacuo*. The solid was recrystallized from benzene to afford 0.06 g (0.16 mmole, 58%) of tetrazole **12** as brown prisms, mp >280°; tlc (chloroform) R_f 0.14; ir (potassium bromide): 3080, 3052 (aromatic CH stretching).

Anal. Calcd. for $C_{21}H_{10}N_4S_2$: C, 65.95; H, 2.64; N, 14.65. Found: C, 66.09; H, 2.75; N, 14.46.

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