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Naphtho[1',2':4,5]thieno[3,2-*a*]-4,7-phenanthroline, a novel hexacyclic ring system has been synthesized in four steps. The ^1H and ^{13}C nmr assignments have been made using two-dimensional nmr techniques. The tertiary helical structure was determined by X-ray crystallographic analysis.

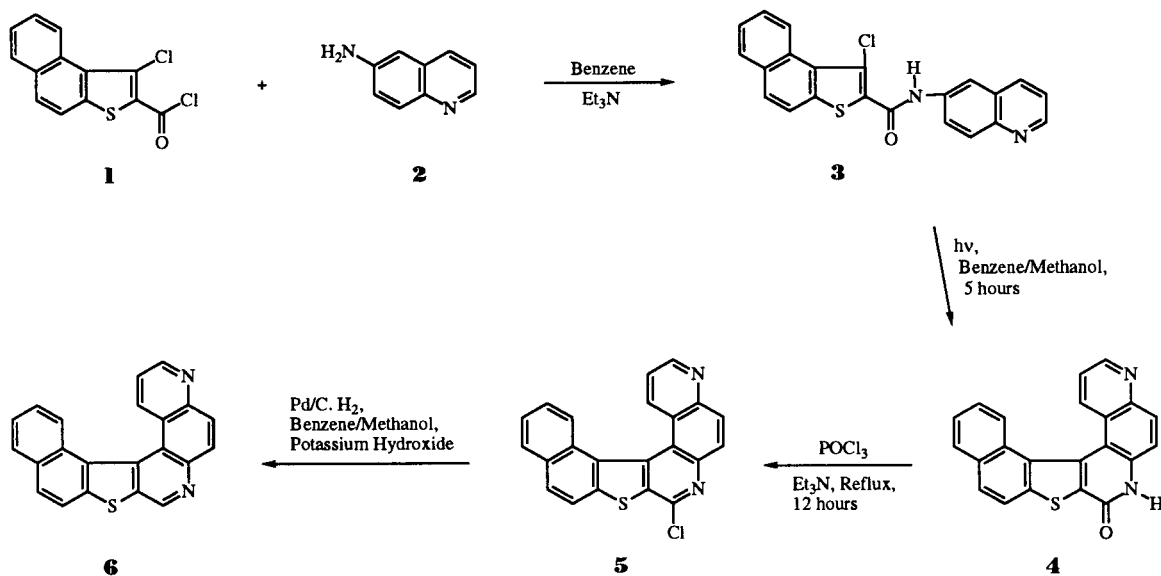
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As a part of an extension of our synthetic work on photo-induced cyclization reactions [1a-i] we now wish to report the synthesis of another hitherto unknown heterocyclic ring system namely naphtho[1',2':4,5]thieno[3,2-*a*]-4,7-phenanthroline (**6**).

The reaction of 1-chloronaphtho[2,1-*b*]thiophene-2-carbonyl chloride (**1**) [3] with 6-aminoquinoline (**2**) provided 1-chloro-*N*-(quinolin-6-yl)naphtho[2,1-*b*]thiophene-2-carboxamide (**3**) in 85% yield. Photocyclization of **3** afforded naphtho[1',2':4,5]thieno[3,2-*a*]-4,7-phenanthroline-8(7*H*)-one (**4**) in 29% yield. Lactam **4** was chlorinated in refluxing phosphorus oxychloride to give 8-chloronaphtho[1',2':4,5]thieno[3,2-*a*]-4,7-phenanthroline (**5**) in 40% yield. Catalytic dechlorination of compound **5** was accomplished with 10% Pd/C in a solution of potassium hydroxide, methanol, and benzene under a hydrogen atmosphere to yield naphtho[1',2':4,5]thieno[3,2-*a*]-4,7-phenanthroline (**6**) in 63% yield.

Spectral assignment of naphtho[1',2':4,5]thieno[3,2-*a*]-4,7-phenanthroline (**6**) was accomplished using data from three two-dimensional nmr experiments [4-6]. As a starting point, data from the COSY experiment was analyzed in order to determine the members of each spin system. Since the furthest downfield proton at 9.48 ppm, appears as a singlet in the proton spectrum and shows no correlations in the COSY spectrum, it is assigned to H-8. At 8.93 ppm a pseudo-doublet shows correlations to 8.20 and 7.05 ppm, constituting a three spin system. The two-spin systems are apparent; the first at 8.47 and 8.36 and the second with members at 8.08 and 8.04. Finally there is one four spin system, 8.07, 7.50, 7.43 and 7.13 ppm. With the protons segregated to their respective spin systems, it is possible to use the data from the one bond proton-carbon correlation technique to pair-up the carbon signals. With all protons and protonated-carbons accounted for, the final step is to assign the non-protonated carbons and inte-

Scheme 1

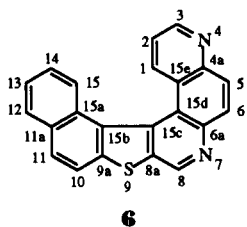


grate the entire structure together. First the phenanthroline moiety is assembled. Using data from the multiple bond proton-carbon correlation experiment, the carbon at 147.31 ppm shows correlations to 8.93 and 8.20, both from the three spin system and from 8.47 ppm, from a two spin system. This suggests that 147.31 can be assigned as C-4a.

With correlations to 7.05 ppm (H-2) and 8.36 ppm (H-5), 124.03 is assigned as C-15e. A four bond correlation from 9.48 (H-8) as well as from 8.47 ppm (H-6) and 8.20 ppm (H-1) allow the assignment of C-15d; C-6a can be assigned in the same manner, showing a correlation to H-8 and H-5. Attachment to the thiophene moiety is shown through correlations between H-8 and carbon signals at 135.41 and 137.07 ppm, assigned on the basis of chemical shifts as C-8a and C-15c respectively.

Correlations of the C and H atoms of the naphthothiophene moiety with the remaining non-protonated carbon atoms can be used to orient the ring with respect to the rest of the molecule. Correlations to the signal at 129.46 ppm are from 8.07, 8.04 ppm and 7.13 ppm. When the correlations to 130.09 ppm from 8.08 and 7.43 ppm are factored in, the assignment can be made as follows: H-10, 8.08 ppm, H-11, 8.04 ppm, H-12, 8.07 ppm, H-13, 7.50 ppm, H-15, 7.43 ppm, leaving H-14 as 7.13 ppm. Carbons can be assigned as C-15a, 129.46, C-15b, 130.09 leaving C-9a as 142.47, verified by the correlation of H-10 and H-11 to this signal. These data are recorded in Table 1.

Table 1
Chemical Shift Data (Deuteriochloroform)



	¹ H	¹³ C		¹ H	¹³ C
1	8.20	137.07	11	8.04	130.89
2	7.05	119.29	11a	—	131.58
3	8.93	149.99	12	8.07	128.91
4a	—	147.31	13	7.50	125.96
5	8.36	130.61	14	7.13	126.07
6	8.47	132.21	15	7.43	127.57
6a	—	145.24	15a	—	129.46
8	9.48	144.88	15b	—	130.09
8a	—	137.07	15c	—	135.41
9a	—	142.47	15d	—	121.74
10	8.08	121.14	15e	—	124.03

The X-ray structural study confirmed the structural formula of compound 6. A list of the atom positional and thermal parameters are found in Table 2. A computer

drawing looking down at the least-squares plane of the molecule (Figure 1) shows the structure with atom labels.

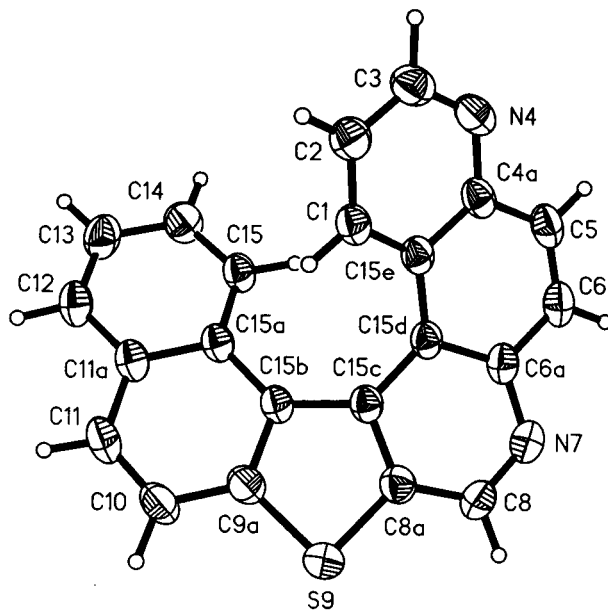


Figure 1

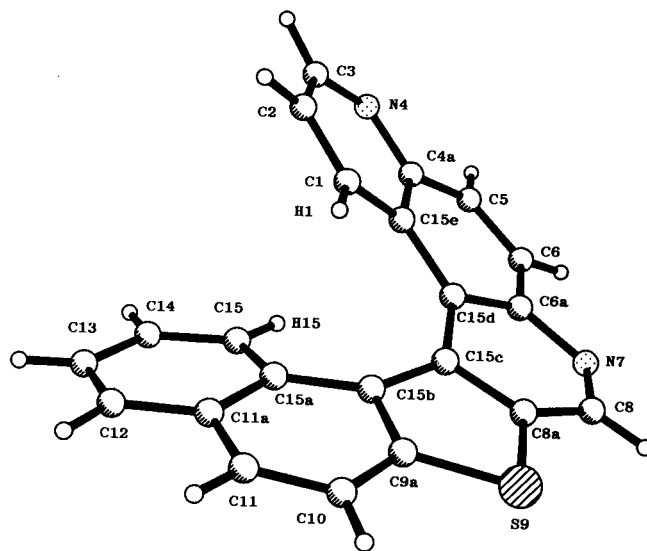


Figure 2

This view of the compound gives the appearance that H1 and H15 are in close proximity but the sideview of the molecule (Figure 2) shows that this is not the case as the molecule has a helical structure as would be expected from other studies of similar fused ring systems. Least-squares plane calculations show that the terminal rings have the smallest distortion from planarity while the inner pyridine ring has the greatest. Least-squares plane data for each plane along with the inter planar dihedral angles are listed in Table 3.

Table 2

Positional ($\times 10^4$) and Isotropic Thermal ($\text{\AA}^2 \times 10^3$) Parameters for Atoms of **6**

atom	x	y	z	U [a]
C1	8536 (2)	4178 (1)	3621 (3)	38 (1)
H1	7987 (19)	4536 (10)	4125(24)	34 (5) [b]
C2	9839 (2)	4074 (1)	4161 (3)	46 (1)
H2	10187 (24)	4351 (12)	5121 (32)	59 (7) [b]
C3	10600 (2)	3565 (1)	3424 (3)	49 (1)
H3	11591 (24)	3453 (11)	3840 (30)	60 (7) [b]
N4	10114 (2)	3153 (1)	2265 (2)	47 (1)
C4a	8800 (2)	3238 (1)	1755 (3)	39 (1)
C5	8241 (2)	2743 (1)	649 (3)	45 (1)
H5	8806 (22)	2394 (12)	208 (29)	47 (6) [b]
C6	6912 (2)	2750 (1)	258 (3)	45 (1)
H6	6508 (21)	2395 (11)	408 (29)	47 (6) [b]
C6a	6038 (2)	3271 (1)	858 (3)	39 (1)
N7	4682 (2)	3173 (1)	571 (3)	45 (1)
C8	3857 (2)	3618 (1)	1178 (3)	44 (1)
H8	2903 (21)	3518 (10)	1056 (27)	41 (5) [b]
C8a	4312 (2)	4223 (1)	1914 (3)	40 (1)
S9	3277 (2)	4863 (1)	2643 (3)	49 (1)
C9a	4664 (2)	5387 (1)	2972 (3)	41 (1)
C10	4565 (2)	6055 (1)	3572 (3)	48 (1)
H10	3713 (24)	6261 (11)	3922 (29)	54 (6) [b]
C11	5692 (2)	6449 (1)	3610 (3)	47 (1)
H11	5667 (22)	6901 (12)	4016 (28)	50 (6) [b]
C11a	6921 (2)	6217 (1)	2920 (3)	40 (1)
C12	8007 (2)	6670 (1)	2706 (3)	46 (1)
H12	7897 (22)	7133 (12)	3114 (29)	49 (6) [b]
C13	9135 (3)	6479 (1)	1884 (3)	51 (1)
H13	9869 (23)	6801 (12)	1711 (30)	58 (7)
C14	9223 (2)	5825 (1)	1227 (3)	48 (1)
H14	10001 (24)	5690 (12)	598 (31)	64 (7) [b]
C15	8197 (2)	5369 (1)	1432 (3)	39 (1)
H15	8247 (19)	4923 (10)	943 (25)	33 (5) [b]
C15a	7030 (2)	5541 (1)	2318 (2)	35 (1)
C15b	5886 (2)	5094 (1)	2494 (2)	34 (1)
C15c	5689 (2)	4386 (1)	2034 (2)	34 (1)
C15d	6596 (2)	3833 (1)	1722 (2)	34 (1)
C15e	7978 (2)	3775 (1)	2335 (2)	35 (1)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor. [b] U values for all hydrogen atoms are regular isotropic U values.

Bond lengths and angles are listed in Table 4. The bond lengths indicate localization of electrons particularly in the N-C bonds. The interior C-C bonds are all somewhat longer than expected for aromatic molecules. This same feature was found in similar structures, for example see the study of 1-methylphenanthro[3,4-b]thiophene [7].

An examination of the planarity data and torsion angles reveals more structural information. Because there are a large number of torsion angles in this structure, only torsion angles about bonds that have some values that deviate by more than 10° from ideality are listed in Table 5.

Table 3

Least-square Plane Data and Dihedral Angles Between Planes for Non-hydrogen Atoms of **6**

Plane 1					
Equation of plane $-3.307x - 12.473y + 5.696z = 5.982$					
Distances of atom from plane in \AA					
C1	0.0104	C3	-0.0206	C4a	0.0327
C2	0.0166	N4	-0.0049	C15e	-0.0342
mean deviation of atoms from plane = 0.0199 \AA					
Plane 2					
Equation of plane $-2.412x - 10.477y + 6.586z = -4.384$					
Distances of atom from plane in \AA					
C4a	0.0249	C6	0.0051	C15d	-0.0884
C5	-0.0507	C6a	0.0659	C15e	0.0432
mean deviation of atoms from plane = 0.0464 \AA					
Plane 3					
Equation of plane $-0.607x - 8.602y + 7.248z = -2.542$					
Distances of atom from plane in \AA					
C6a	-0.0164	C8	0.0493	C15c	-0.1017
N7	-0.0581	C8a	0.0345	C15d	0.0925
mean deviation of atoms from plane = 0.0587 \AA					
Plane 4					
Equation of plane $0.800x - 6.590y + 7.556z = 0.9536 - 0.954$					
Distances of atom from plane in \AA					
C8a	-0.0384	C9a	0.0224	C15c	0.0556
S9	0.0082	C15b	-0.0478		
mean deviation of atoms from plane = 0.0345 \AA					
Plane 5					
Equation of plane $2.446x - 5.887y + 7.388z = 0.2109$					
Distances of atom from plane in \AA					
C9a	-0.0454	C11	0.0521	C15a	-0.0402
C10	-0.0190	C11a	-0.0204	C15b	0.0729
mean deviation of atoms from plane = 0.0417 \AA					
Plane 6					
Equation of plane $4.058x - 6.034y + 6.857z = 1.077$					
Distances of atom from plane in \AA					
C11a	-0.0183	C13	0.0115	C15	-0.0084
C12	0.0021	C14	-0.0081	C15a	0.0213
mean deviation of atoms from plane = 0.0116 \AA					
Dihedral Angles ($^\circ$) Between Planes					
Plane	2	3	4	5	6
1	10.1	22.5	32.8	41.3	48.9
2		12.8	23.1	32.1	40.5
3			10.3	19.5	28.3
4				9.8	19.5
5					10.1

Examination of Table 5 shows that there are only eight bonds that fall into that category. All four torsion angles about bonds C6a-C15d, C15b-C15c, and C15c-C15d deviate by more than ten degrees from 180° . In addition three of the four torsion angles about C15d-C15e, two of the four angles about C9a-C15a and C15a-C15b and one of the two torsion angles about C11-C11a fit into that category. It is of interest that of all these bonds, only one involves a bond (C11-C11a) of the exterior of the helix. The majority of the bonds are on the interior of the helix. Also

Table 4

Bond Lengths and Angles for Non-hydrogen Atoms of **6**

1	2	3	1-2 (Å)	1-2-3 (°)
C2	C1	C15e	1.369 (3)	120.1 (2)
C1	C2	C3		119.1 (2)
C2	C3	N4	1.396 (3)	123.4 (2)
C3	N4	C4a	1.322 (3)	117.9 (2)
N4	C4a	C5	1.368 (3)	117.3 (2)
C15e	C4a	N4	1.421 (3)	122.8 (2)
C5	C4a	C15e	1.426 (3)	119.9 (2)
C4a	C5	C6		120.2 (2)
C5	C6	C6a	1.348 (3)	121.5 (2)
C6	C6a	N7	1.434 (3)	116.1 (2)
C6	C6a	C15d		119.6 (2)
N7	C6a	C15d	1.372 (3)	124.3 (2)
C6a	N7	C8		117.6 (2)
N7	C8	C8a	1.304 (3)	122.2 (2)
C8	C8a	S9	1.402 (3)	125.1 (2)
C8	C8a	C15c		121.8 (2)
S9	C8a	C15c	1.738 (2)	113.0 (1)
C8a	S9	C9a		90.7 (1)
S9	C9a	C10	1.735 (2)	123.3 (2)
S9	C9a	C15b		113.5 (1)
C10	C9a	C15b	1.408 (3)	123.1 (2)
C9a	C10	C11		118.6 (2)
C10	C11	C11a	1.361 (3)	121.1 (2)
C11	C11a	C12	1.425 (3)	120.4 (2)
C11	C11a	C15a		120.3 (2)
C12	C11a	C15a		119.1 (2)
C11a	C12	C13	1.413 (3)	121.3 (2)
C12	C13	C14	1.365 (3)	119.8 (2)
C13	C14	C15	1.399 (3)	120.5 (2)
C14	C15	C15a	1.371 (3)	121.4 (2)
C11a	C15a	C15	1.425 (3)	117.8 (2)
C11a	C15a	C15b		118.2 (2)
C15	C15a	C15b	1.415 (3)	123.7 (2)
C9a	C15b	C15a	1.404 (3)	117.3 (2)
C9a	C15b	C15c		110.7 (2)
C15a	C15b	C15c	1.446 (3)	131.4 (2)
C8a	C15c	C15b	1.404 (3)	111.2 (2)
C8a	C15c	C15d		115.2 (2)
C15b	C15c	C15d	1.457 (3)	133.7 (2)
C6a	C15d	C15c	1.415 (3)	116.2 (2)
C6a	C15d	C15e		117.5 (2)
C15c	C15d	C15e	1.440 (3)	126.1 (2)
C1	C15e	C4a	1.410 (3)	116.3 (2)
C15d	C15e	C1	1.451 (3)	124.0 (2)
C4a	C15e	C15d		119.4 (2)

Table 5

All Torsion Angles of **6** about Bonds which Have Torsion Angles that Deviate by more than 10° from Expected Value

1	2	3	4	1-2-3-4 (°)
C6	C6a	C15d	C15c	169.8 (2)
C6	C6a	C15d	C15e	-15.6 (3)
N7	C6a	C15d	C15c	-11.8 (3)
N7	C6a	C15d	C15e	162.8 (2)
C8	C8a	C15c	C15b	167.2 (2)
C8	C8a	C15c	C15d	-14.0 (3)
S9	C8a	C15c	C15b	-9.2 (2)
S9	C8a	C15c	C15d	169.6 (1)
S9	C9a	C15b	C15a	164.6 (1)
S9	C9a	C15b	C15c	-7.2 (2)
C10	C9a	C15b	C15a	-12.0 (3)
C10	C9a	C15b	C15c	176.2 (2)
C10	C11	C11a	C12	168.9 (2)
C10	C11	C11a	C15a	-6.0 (3)
C11a	C15a	C15b	C9a	11.1 (3)
C11a	C15a	C15b	C15c	-179.1 (2)
C15	C15a	C15b	C9a	-162.1 (2)
C15	C15a	C15b	C15c	7.7 (3)
C9a	C15b	C15c	C8a	10.3 (2)
C9a	C15b	C15c	C15d	-168.2 (2)
C15a	C15b	C15c	C8a	-160.0 (2)
C15a	C15b	C15c	C15d	21.5 (4)
C8a	C15c	C15d	C6a	18.7 (3)
C8a	C15c	C15d	C15e	-155.4 (2)
C15b	C15c	C15d	C6a	-162.8 (2)
C15b	C15c	C15d	C15e	23.1 (3)
C6a	C15d	C15e	C1	-159.4 (2)
C6a	C15d	C15e	C4a	13.6 (3)
C15c	C15d	C15e	C1	14.7 (3)
C15c	C15d	C15e	C4a	-172.3 (2)

these bonds include only one atom of the terminal pyridine ring and no atoms of the terminal benzene ring. This is reasonable based on the least-squares plane data (see Table 5).

There are no unusually short intramolecular contact distances though it is clear from Figures 2 and 3 that without the helical structure this would not be the case. Contact distances that are of interest are H1...H15, 2.694 Å; H15...C1, 2.623 Å; C15a...H1, 2.625 Å; C15b...H1, 2.675 Å; C15...H1, 2.733 Å and C15...C1, 2.953 Å. There are no unusually short intermolecular contact distances.

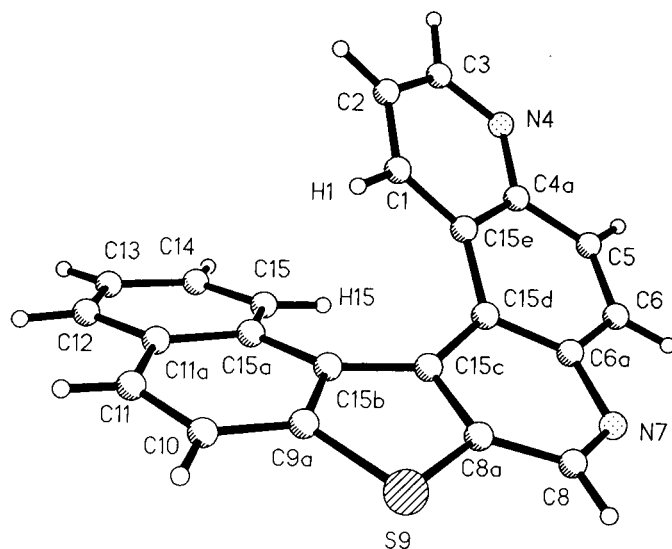


Figure 3

EXPERIMENTAL

Melting points were determined on a Thomas-Hoover melting point apparatus and are uncorrected. The ir spectra were recorded on a Beckman FT 1100 spectrometer as potassium bromide pellets and frequencies are expressed in cm^{-1} . The ^1H and ^{13}C -nmr spectra were obtained on a Bruker AMX 360 spectrometer in the solvent indicated with TMS as the internal standard and chemical shifts reported in ppm (δ) and J values in Hz. Elemental analyses were performed by M-H-W Laboratories, Phoenix, Arizona.

1-Chloro-*N*-(quinolin-6-yl)naphtho[2,1-*b*]thiophene-2-carboxamide (**3**).

A solution of 6-aminoquinoline (**2**) (1.44 g, 10 mmoles) and triethylamine (1.4 ml, 10 mmoles) in benzene (100 ml) was added dropwise to a stirred solution of 1-chloronaphtho[2,1-*b*]thiophene-2-carbonyl chloride (**1**) [**3**] (2.81 g, 10 mmoles) in benzene (100 ml) at room temperature. After the addition was complete, the mixture was stirred for 12 hours at *ca* 80°. Excess solvent was removed under reduced pressure to give a beige colored solid. This solid was suspended in water (15 ml) to remove quaternary salts and then filtered, dried at room temperature to afford **3** in 85% yield. A 0.5 g sample was dissolved in hot benzene (200 ml), the solution was treated with charcoal and filtered, then allowed to cool and stand at room temperature overnight. The precipitate was collected by filtration and dried. Compound **3** was obtained as needles, mp 180-182°; ir (potassium bromide): 3384, 1653, 1548, 818, 746 cm^{-1} ; ^1H -nmr (dimethyl sulfoxide- d_6): δ 10.7 (bs, 1H), 9.4 (m, 1H), 8.8 (bd, 1H, J = 4 Hz), 8.4-7.4 (m, 10H).

Anal. Calcd. for $\text{C}_{22}\text{H}_{13}\text{N}_2\text{ClSO}$: C, 67.95; H, 3.37; N, 7.20. Found: C, 68.02; H, 3.13; N, 7.22.

Naphtho[1',2':4,5]thieno[3,2-*a*]4,7-phenanthroline-8(7*H*)-one (**4**).

A stirred solution of carboxamide **3** (0.5 g, 1.29 mmoles) and triethylamine (0.2 ml, 1.4 mmoles) in a benzene/methanol mixture (3:1) (500 ml) was irradiated for 5 hours with a 450 watt medium pressure Hanovia mercury lamp under a slow stream of air. The material was collected, the solvent was removed under reduced pressure, and washed with water (2 x 10 ml), then dried to give 0.13 g (29%) of **4**. Only one isomer of the possible two isomers was obtained, mp 280-282°; ir (potassium bromide): 3394, 1653, 833, 810 cm^{-1} ; ^1H -nmr (dimethyl sulfoxide- d_6): δ 9.1 (bd, 1H), 8.3 (m, 6H), 7.4 (m, 5H).

This compound was used without further purification because of insolubility.

8-Chloronaphtho[1',2':4,5]thieno[3,2-*a*]4,7-phenanthroline (**5**).

A stirred mixture of naphtho[1',2':4,5]thieno[3,2-*a*]4,7-phenanthroline-8(7*H*)-one (**4**) (0.55 g, 1.4 mmoles) in phosphorus oxychloride (35 ml) and triethylamine (0.2 ml, 1.4 mmoles) was heated at 130-140° for 12 hours, then the excess solvent was removed under reduced pressure. The resulting dark material was cooled to ice bath temperature where residual phosphorus oxychloride was decomposed by the portionwise addition of crushed ice (*ca* 75 g). The precipitate (0.23 g, 40%) was collected by filtration and dried at room temperature. This solid was dissolved in hot benzene (250 ml), the solution treated with charcoal, allowed to cool and stand at room temperature overnight. The precipitate was collected by filtration as thin needles, mp 206-208°; ir (potassium bromide): 1509, 1478, 1023, 828, 805 cm^{-1} ; ^1H -nmr (deuteriochloroform): δ 9.0 (bd, 1H), 8.4 (s, 2H), 8.2-8.0 (m, 3H), 7.6-7.0 (m, 5H).

Anal. Calcd. for $\text{C}_{22}\text{H}_{11}\text{N}_2\text{SCl}$: C, 71.25; H, 2.99; N, 7.55. Found: C, 71.03; H, 3.31; N, 7.42.

Naphtho[1',2':4,5]thieno[3,2-*a*]4,7-phenanthroline (**6**).

The catalyst (10% Pd/C, 0.1 g) was added to a stirred solution of 8-chloronaphtho[1',2':4,5]thieno[3,2-*a*]4,7-phenanthroline (**5**) (0.1 g, 0.27 mmole) in a solution of potassium hydroxide (0.013 g, 0.27 mmole), methanol (100 ml) and benzene (100 ml). The mixture was stirred under a hydrogen atmosphere for 4 days at room temperature. The catalyst was removed by filtration through a Celite pad and the filtrate concentrated to dryness under reduced pressure. The solid residue was dissolved in hot benzene (100 ml) and treated with charcoal then allowed to cool and stand at room temperature overnight. The solid was collected and recrystallized from methylene chloride and methanol by slow evaporation to afford 57 mg (63%) of prisms, mp 218-220°; ir (potassium bromide): 1584, 1491, 836, 805, 730 cm^{-1} ; ^1H -nmr (deuteriochloroform): δ 9.48 (s, 1H), 8.93 (bd, 1H, J = 4.1 Hz), 8.47 (d, 1H, J = 9.2 Hz), 8.36 (d, 1H, J = 9.2 Hz), 8.20 (bd, 1H, J = 8.4 Hz), 8.10-8.01 (m, 3H), 7.50 (t, 1H, J = <1, 7.9 Hz), 7.43 (bd, 1H, J = 8.4 Hz), 7.13 (t, 1H, J = <1, 8.3 Hz), 7.50 (dd, 1H, J = 4.3, 8.4 Hz); ^{13}C -nmr (deuteriochloroform): protonated carbons at δ 149.99, 144.88, 137.07, 132.21, 130.89, 130.61, 128.91, 127.57, 126.07, 125.96, 121.14, 119.29, and quaternary carbons at δ 147.31, 145.24, 142.47, 137.07, 135.41, 131.58, 130.09, 129.46, 124.03, 121.74.

Anal. Calcd. for $\text{C}_{22}\text{H}_{12}\text{N}_2\text{S}$: C, 78.55; H, 3.60; N, 8.33. Found: C, 78.39; H, 3.81; N, 8.08.

X-ray Crystal Structure Determination of **6**.

A suitable crystal of **6** with approximate dimensions 0.40 x 0.44 x 0.50 mm was selected for the X-ray structural study. The crystal was mounted on a Siemens R3m/V automated diffractometer which utilized graphite monochromated $\text{MoK}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). The lattice parameters and the orientation matrix were obtained using a least-squares procedure involving 31 carefully centered reflections ($10.43 < 2\theta < 23.12^\circ$). The material crystallized in the monoclinic space group P2₁/c with $a = 9.914 (3) \text{ \AA}$, $b = 19.727 (6) \text{ \AA}$, $c = 8.063 (2) \text{ \AA}$, $\beta = 91.26 (2)^\circ$ and a volume of $1572 (4) \text{ \AA}^3$. There are 4 molecules in the unit cell ($Z = 4$) with a formula weight of 336.4 and a calculated density of 1.422 Mg/m^3 with a μ of 0.21 mm^{-1} . Data was collected at 20° using a 2θ - θ scan mode to a 2θ limit of 50° . Four quadrants of data were collected ($-11 \leq b \leq 11$, $-23 \leq k \leq 23$, $-9 \leq l < 0$) and the 4837 collected data were merged to 2799 ($R_{int} = 3.2\%$) independent reflections of which 2035 ($F > 6.0\sigma(F)$) were considered observed and used in the structure solution procedure. The solid state structure was obtained using direct methods. All non-hydrogen atoms were obtained from the resulting E-map and were refined anisotropically. It was possible to obtain positions for all hydrogen atoms from difference maps and these atoms were refined isotropically. The average C-H distance was $0.98 (3) \text{ \AA}$. In the final refinements weights based on counting statistics were applied to the data. A full-matrix least-squares refinement was used with the quantity minimized being $\sum w(F_o - F_c)^2$. The resulting R values were $R = 0.0350$ and $R_w = 0.0496$. The goodness-of-fit parameter was 1.27 and the data/parameter ratio was 7.4:1. Structure factors were obtained from The International Tables for X-ray Crystallography [8]. All programs used in the solution, refinement and display of the structure are contained in the program package SHELXTL PLUS [9].

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