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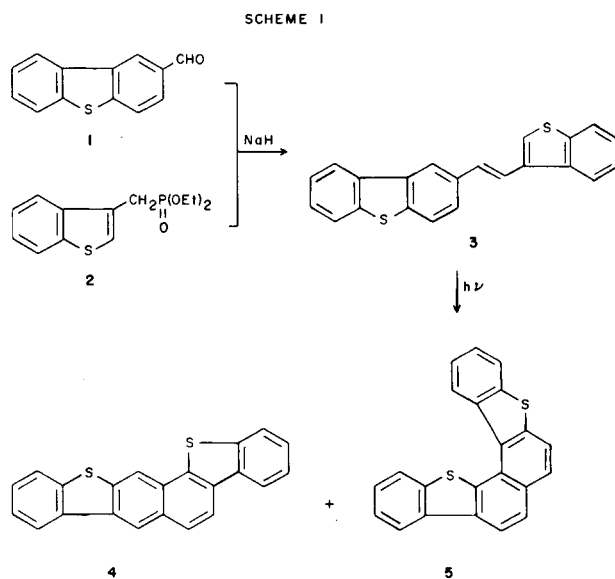
The syntheses of naphtho[1,2-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**4**), naphtho[1,2-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**5**), naphtho[2,1-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**8**), naphtho[2,1-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**9**), naphtho[1,2-*b*:5,6-*b'*]bisbenzo[*b*]thiophene (**14**), naphtho[1,2-*b*:6,5-*b'*]bisbenzo[*b*]thiophene (**17**) and naphtho[2,1-*b*:6,5-*b'*]bisbenzo[*b*]thiophene (**23**) are reported.

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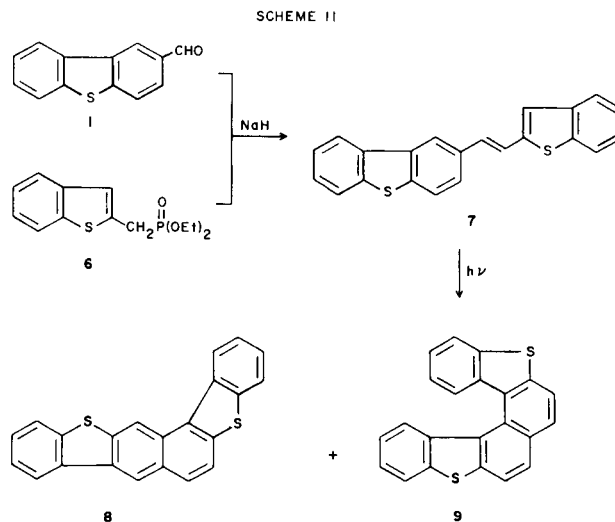
In this paper we report seven novel angular polycyclic aromatic thiophenes containing two thiophene rings with a molecular weight of 340 as part of our continuing study [3-21] directed toward providing all of the possible polycyclic thiophenes suspected of occurring in coal liquids, shale oil or related coal-derived products.

Naphtho[1,2-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**4**) and naphtho[1,2-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**5**) were prepared in a separable mixture in two steps from dibenzothiophene-2-carboxaldehyde (**1**) [22]. A condensation of the aldehyde **1** [22] with diethyl 3-benzo[*b*]thienylphosphonate (**2**) [13] using sodium hydride as the base gave 1-(2-dibenzothiophenyl)-2-(3-benzo[*b*]thienyl)ethene (**3**) as a pale yellow oil in 82% yield. Photocyclization of compound **3** using iodine and air as the oxidants gave a crystalline mixture of naphtho[1,2-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**4**) and naphtho[1,2-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**5**) (Scheme I). Separation of the mixture of compounds **4** and **5** was achieved by column chromatography on a neutral alumina column using hexane followed by benzene as the eluents. Naphtho[1,2-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**5**) eluted first in hexane in pure form as colorless needles in 42% yield. Upon elution with benzene, the very insoluble **4** was obtained as colorless flakes in 23% yield. The structural assignments of **4** and **5** were based on solubility, melting point and nmr. Naphtho[1,2-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**4**) is more linear than compound **5**, therefore compound **4** should have a higher melting point (mp 298°) and a lower solubility in organic solvents than compound **5** (mp 212°). The nmr of compound **4** shows two singlets, one at 8.23 ppm due to the H-7 proton and the second singlet at 9.16 ppm due to the H-13 proton. Compound **5** does not exhibit a singlet in its nmr spectrum thus confirming our structural assignments.

1-(2-Dibenzothiophenyl)-2-(2-benzo[*b*]thienyl)ethene (**7**) was obtained as pale yellow flakes in 69% yield from dibenzothiophene-2-carboxaldehyde (**1**) [22] and diethyl 2-benzo[*b*]thienylphosphonate (**6**) [13] under Wadsworth-



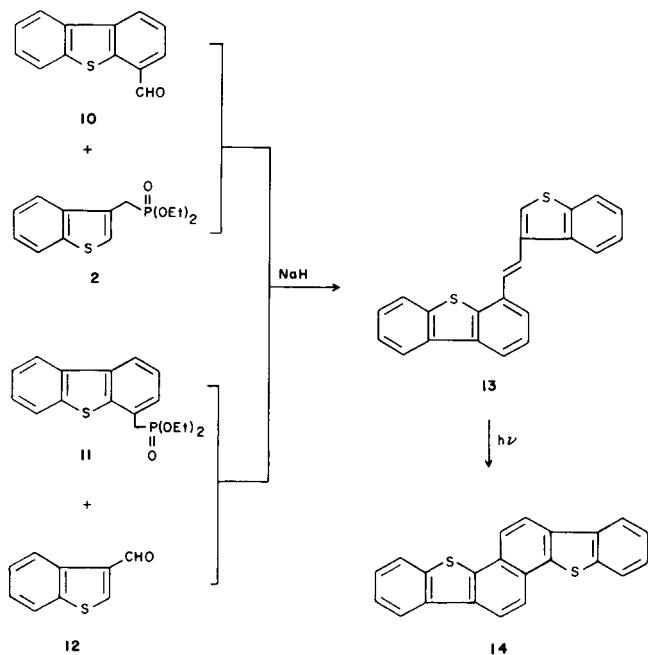
Emmons conditions. Photocyclization of compound **7** gave a crystalline mixture of naphtho[2,1-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**8**) and naphtho[2,1-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**9**) (Scheme II).



phene (**9**) (Scheme II). Separation of the mixture of compounds **8** and **9** was achieved by column chromatography on neutral alumina using hexane followed by benzene as the eluent. Naphtho[2,1-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**9**) eluted first in hexane in pure form as colorless needles in 42% yield. Elution with benzene gave naphtho[2,1-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**8**) as colorless flakes in 16% yield. Structural assignments of compounds **8** and **9** were based on solubility, melting point and nmr. Naphtho[2,1-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**8**) is more linear than compound **9**, therefore, compound **8** should have a higher melting point (mp 345°) and a lower solubility in organic solvents than compound **9** (mp 259°). The nmr of **8** shows two singlets, one at 7.88 ppm due to the H-8 proton and the second singlet at 9.01 ppm due to the H-14 proton. Compound **9** does not exhibit a singlet in its nmr spectrum.

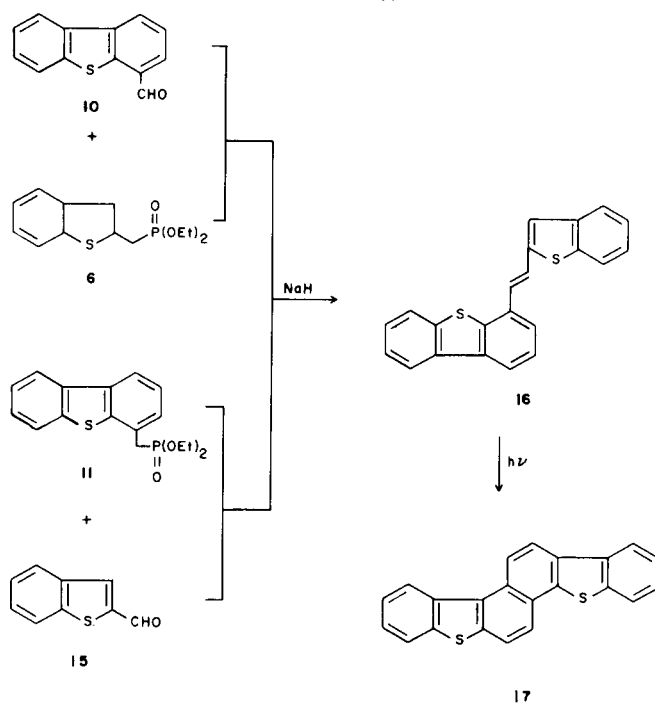
1-(4-Dibenzothieryl)-2-(3-benzo[*b*]thienyl)ethene (**13**) was prepared from the condensation of dibenzothiophene-4-carboxaldehyde (**10**) [23] and diethyl 3-benzo[*b*]thienylphosphonate (**2**) [13] in 52% yield and also from benzo[*b*]thiophene-3-carboxaldehyde (**12**) and diethyl 4-dibenzothierylphosphonate (**11**) in 55% yield. The orange flakes of **13** were irradiated under a 450 watt Hanovia medium pressure mercury lamp for four hours affording naphtho[1,2-*b*:5,6-*b'*]bisbenzo[*b*]thiophene (**14**) as colorless prisms in 42% yield (Scheme III).

SCHEME III

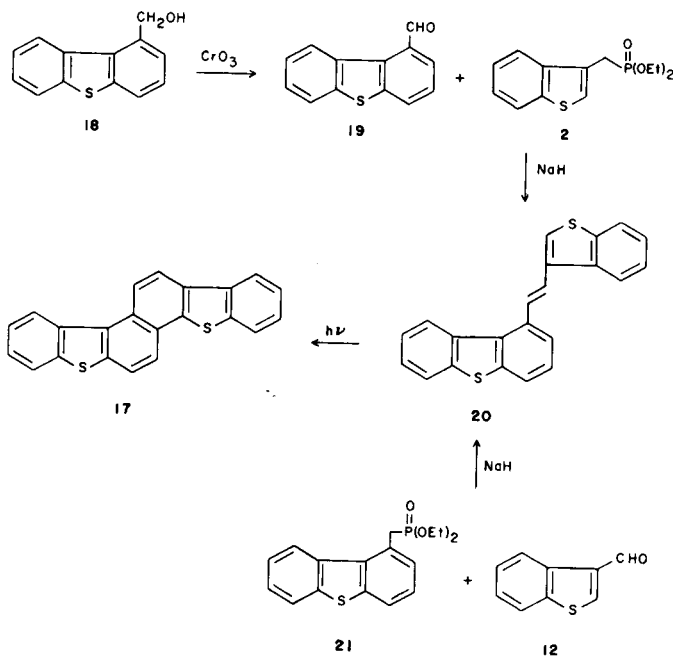


Dibenzothiophene-4-carboxaldehyde (**10**) [23] and diethyl 2-benzo[*b*]thienylphosphonate (**6**) [13] under Wadsworth-Emmons conditions (sodium hydride and 1,2-dimethoxyethane) gave 1-(4-dibenzothieryl)-2-(2-benzo[*b*]thienyl)ethene

SCHEME IV

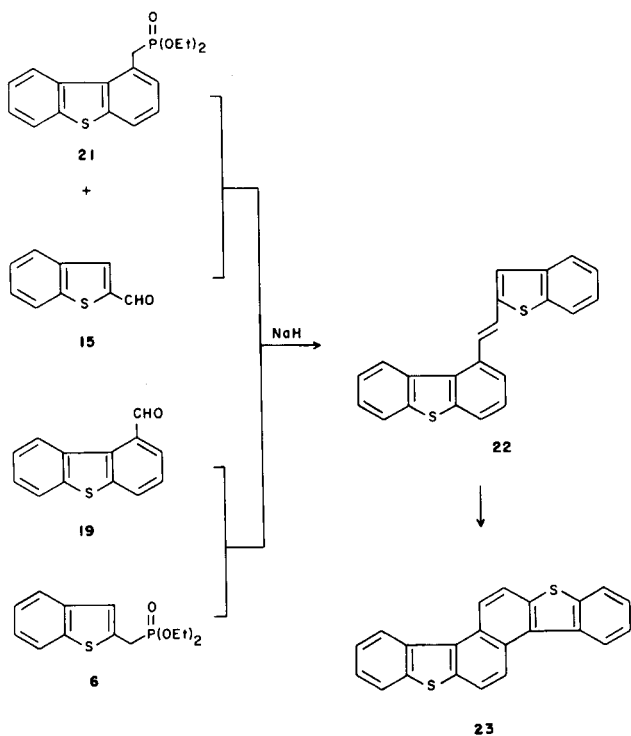


SCHEME V



ene (**16**) in 78% yield. Compound **16** was also obtained from diethyl 4-dibenzothierylphosphonate (**11**) [21] and benzo[*b*]thiophene-2-carboxaldehyde (**15**) in 68% yield. Photocyclization of **16** gave naphtho[1,2-*b*:6,5-*b'*]bisbenzo[*b*]thiophene (**17**) in 79% yield (Scheme IV). Compound **17** was also prepared from a different intermediate as recorded in Scheme V.

SCHEME VI



1-Hydroxymethyldibenzothiophene (**18**) upon oxidation with chromium trioxide-pyridine complex gave dibenzothiophene-1-carboxaldehyde (**19**) in 72% yield. Condensation of the aldehyde **19** with diethyl 3-benzo[*b*]thienylphosphonate (**2**) [13] using sodium hydride as the base afforded 1-(1-dibenzothiényl)-2-(3-benzo[*b*]thiényl)ethene (**20**) in 85% yield as a pale yellow oil. Compound **20** was also obtained in 73% yield *via* the condensation of diethyl 1-dibenzothiénylphosphonate (**21**) [21] and benzo[*b*]thiophene-3-carboxaldehyde (**12**). Photocyclization of **20** gave naphtho[1,2-*b*:6,5-*b'*]bisbenzo[*b*]thiophene (**17**) in 82% yield (Scheme V). Compound **17** was also synthesized from a different intermediate as recorded in Scheme IV.

1-(1-Dibenzothiényl)-2-(2-benzo[*b*]thiényl)ethene (**22**) was prepared in 71% yield from the condensation of diethyl 1-dibenzothiénylphosphonate (**21**) [21] and benzo[*b*]thiophene-2-carboxaldehyde (**15**) and also in 67% yield from diethyl 2-benzo[*b*]thiénylphosphonate (**6**) [13] and dibenzothiophene-1-carboxaldehyde (**18**) under Wadsworth-Emmons conditions. Photocyclization of **22** gave naphtho[2,1-*b*:6,5-*b'*]bisbenzo[*b*]thiophene (**23**) in 22% yield.

Some of these angular polycyclic aromatic thiophenes are being screened against TA-98 and TA-100 in the Ames test (S9 liver homogenate activation) and these results will be published elsewhere.

EXPERIMENTAL

Melting points were determined on a Thomas Hoover melting point apparatus and are uncorrected. The ir spectra were obtained on a Beck-

mann Acculab 2 spectrometer. The ¹H-nmr spectra were obtained on a Varian EM-360A spectrometer and a JEOL FX-90Q spectrometer in deuteriochloroform. Chemical shifts are reported in δ units. Mass spectra were obtained on a Hewlett-Packard model 5980A mass spectrometer. Elemental analyses were performed by MHW Laboratories, Phoenix, Arizona.

1-(2-Dibenzothiényl)-2-(3-benzo[*b*]thiényl)ethene (**3**).

Sodium hydride (50% dispersion in mineral oil, 0.87 g, 0.036 mole) was placed in dry 1,2-dimethoxyethane (100 ml). Sodium hydride was used after washing twice with hexane (50 ml). The slurry was cooled to 20° and diethyl 3-benzo[*b*]thienylphosphonate (**2**) (2.5 g, 0.0088 mole) [13] was added with stirring under a stream of nitrogen. After the addition, the solution was stirred at room temperature for 20 minutes. To the pale yellow solution maintained below 25°, dibenzothiophene-2-carboxaldehyde (**1**) (1.87 g, 0.0088 mole) [22] was added slowly *via* a spatula. The solution was stirred at room temperature for 4.5 hours. The reaction mixture was slowly poured into a large excess of ice-water and the organic layer was separated, dried over anhydrous sodium sulfate and evaporated *in vacuo* affording a yellow oil. Purification was accomplished by column chromatography on silica gel using hexane:benzene (1:2) as the eluent and 2.43 g (82%) of a pale yellow oil was obtained; nmr (deuteriochloroform): δ 7.12-7.91 (m, 2 × ethenyl-*H*, *H*-2', *H*-4', *H*-5', *H*-6', *H*-7' of benzo[*b*]thiophene, *H*-3, *H*-4, *H*-6, *H*-7, *H*-8 of dibenzothiophene, 12H), 7.94-8.28 (m, *H*-9 of dibenzothiophene, 1H, *ArH*), 8.18 (s, *H*-1 of dibenzothiophene, 1H, *ArH*).

Anal. Calcd. for C₂₂H₁₄S₂: C, 77.16; H, 4.12; S, 18.72. Found: C, 77.40; H, 4.32; S, 18.58.

Naphtho[1,2-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**4**) and Naphtho[1,2-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**5**).

A solution of 1-(2-dibenzothiényl)-2-(3-benzo[*b*]thiényl)ethene (**3**) (1.0 g, 0.0029 mole) and iodine (0.05 g) in benzene (360 ml) was irradiated for four hours with a 450 watt Hanovia medium pressure mercury lamp. During the course of the reaction a slow stream of air was passed through the solution. The solvent was evaporated *in vacuo* and the residue was chromatographed on a neutral alumina column using hexane and then benzene as the eluents. Compound **5** eluted first in hexane and 0.42 g (42%) was obtained. Elution with benzene gave pure **4** and 0.23 g (23%) was obtained.

Compound 4.

Colorless flakes of **4** were obtained in 23% yield, mp 298°; nmr (deuteriochloroform): δ 7.08-8.18 (m, *H*-2, *H*-3, *H*-4, *H*-5, *H*-6, *H*-8, *H*-9, *H*-10, 8H, *ArH*), 8.23 (s, *H*-7, 1H, *ArH*), 8.70-8.90 (m, *H*-1, *H*-11, 2H, *ArH*), 9.16 (s, *H*-7, 1H, *ArH*); ms: *m/e* 340 (*M*⁺, 100).

Anal. Calcd. for C₂₂H₁₂S₂: C, 77.61; H, 3.55; S, 18.84. Found: C, 77.54; H, 3.62; S, 18.79.

Compound 5.

Colorless needles of **5** were obtained in 42% yield, mp 212°; nmr (deuteriochloroform): δ 7.18-8.00 (m, *H*-2, *H*-3, *H*-4, *H*-6, *H*-7, *H*-8, *H*-11, *H*-12, 8H, *ArH*), 8.04-8.45 (m, *H*-9, *H*-10, 2H, *ArH*), 8.52-8.82 (m, *H*-13, 1H, *ArH*), 9.01-9.36 (m, *H*-1, 1H, *ArH*); ms: *m/e* 340 (*M*⁺, 100).

Anal. Calcd. for C₂₂H₁₂S₂: C, 77.61; H, 3.55; S, 18.84. Found: C, 77.59; H, 3.42; S, 18.96.

1-(2-Dibenzothiényl)-2-(2-benzo[*b*]thiényl)ethene (**7**).

Sodium hydride (50% dispersion in mineral oil, 0.72 g, 0.03 mole), dry 1,2-dimethoxyethane (85 ml) and 2-benzo[*b*]thiénylphosphonate (**6**) (2.1 g, 0.0074 mole) [13] were treated as described for **3**, followed by dibenzothiophene-2-carboxaldehyde (**1**) (1.6 g, 0.0074 mole) [22] as also described for **3** above. The product was collected by filtration and recrystallized from methanol affording pale yellow flakes in 69% yield, mp 115°; nmr (deuteriochloroform): δ 7.04-7.90 (m, 2 × ethylene-*H*, *H*-2', *H*-4', *H*-5', *H*-6', *H*-7' of benzo[*b*]thiophene, *H*-3, *H*-4, *H*-6, *H*-7, *H*-8 of dibenzothiophene, 12H), 7.93-8.18 (m, *H*-9 of dibenzothiophene, 1H, *ArH*), 8.03 (s, *H*-1 of dibenzothiophene, 1H, *ArH*).

Anal. Calcd. for $C_{22}H_{14}S_2$: C, 77.16; H, 4.12; S, 18.72. Found: C, 77.13; H, 4.08; S, 18.65.

Naphtho[2,1-*b*:7,6-*b'*]bisbenzo[*b*]thiophene (**8**) and Naphtho[2,1-*b*:7,8-*b'*]bisbenzo[*b*]thiophene (**9**).

Compounds **8** and **9** were obtained from compound **7** (1.5 g, 0.0044 mole) and iodine (0.05 g) in a similar manner to the preparation of compounds **4** and **5**. The residue obtained after four hours of photocyclization was chromatographed on a neutral alumina column using hexane followed by benzene as the eluents. Compound **9** eluted first in hexane and upon elution with benzene gave pure **8**.

Compound 8.

Compound **8** was obtained as colorless crystals in 16% yield (0.24 g), mp 345°; nmr (deuteriochloroform): δ 7.11-7.89 (m, H-2, H-3, H-4, H-6, H-7, H-9, H-10, H-11, 8H, *ArH*), 7.88 (s, H-8, 1H, *ArH*), 8.73-8.99 (m, H-1, H-12, 2H, *ArH*), 9.01 (s, H-14, 1H, *ArH*); ms: *m/e* 340 (M^+ , 100).

Anal. Calcd. for $C_{22}H_{12}S_2$: C, 77.61; H, 3.55; S, 18.84. Found: C, 77.32; H, 3.81; S, 18.75.

Compound 9.

Colorless needles of **9** were obtained in 42% yield (0.63 g), mp 259°; nmr (deuteriochloroform): δ 7.02-8.23 (m, H-2, H-3, H-4, H-6, H-7, H-8, H-9, H-11, H-12, H-13, 10H, *ArH*), 8.70-9.15 (m, H-1, H-14, 2H, *ArH*); ms: *m/e* 340 (M^+ , 100).

Anal. Calcd. for $C_{22}H_{12}S_2$: C, 77.61; H, 3.55; S, 18.84. Found: C, 77.59; H, 3.74; S, 18.96.

1-(4-Dibenzothiényl)-2-(3-benzo[*b*]thienyl)ethene (**13**). Method A.

Compound **13** was prepared from diethyl 3-benzo[*b*]thienylphosphonate (**2**) (2.5 g, 0.0088 mole) [13] and dibenzothiophene-4-carboxaldehyde (**10**) (1.87 g, 0.0088 mole) [23] in a manner similar to the preparation of 1-(2-dibenzothiényl)-2-(2-benzo[*b*]thienyl)ethene (**7**) and 1.57 g (52%) of orange flakes was obtained, mp 133°.

Method B.

Compound **13** was prepared from the condensation of diethyl 4-dibenzothiénylphosphonate (**11**) (3.1 g, 0.0093 mole) [21] and benzo[*b*]thiophene-3-carboxaldehyde (**12**) (1.5 g, 0.0093 mole) in a manner similar to the preparation of compound **7** and 1.75 g (55%) of orange flakes was obtained, mp 133°; nmr (deuteriochloroform): δ 7.18-8.32 (m, 2 \times ethenyl-*H* and aromatic-*H* 14H).

Anal. Calcd. for $C_{22}H_{14}S_2$: C, 77.16; H, 4.12; S, 18.72. Found: C, 77.32; H, 4.28; S, 18.60.

Naphtho[1,2-*b*:5,6-*b'*]bisbenzo[*b*]thiophene (**14**).

Compound **14** (1.3 g, 0.0038 mole) and iodine (0.1 g) was photocyclized in a similar manner to the preparation of compounds **4** and **5** and colorless prisms (0.54 g, 42% yield) were obtained, mp >350°; nmr (deuteriochloroform): δ 7.14-8.60 (m, 12H, *ArH*); ms: *m/e* 340 (M^+ , 100).

Anal. Calcd. for $C_{22}H_{12}S_2$: C, 77.61; H, 3.55; S, 18.84. Found: C, 77.52; H, 3.69; S, 18.72.

1-(4-Dibenzothiényl)-2-(2-benzo[*b*]thienyl)ethene (**16**). Method A.

This compound was synthesized from dibenzothiophene-4-carboxaldehyde (**10**) (1.87 g, 0.0088 mole) [23] and diethyl 2-benzo[*b*]thienylphosphonate (**6**) (2.5 g, 0.0088 mole) [13] in a manner similar to the preparation of compound **7** and 2.36 g (78%) of yellow prisms were obtained, mp 154°.

Method B.

Compound **16** was prepared by the condensation of diethyl 4-dibenzothiénylphosphonate (**11**) (3.1 g, 0.0093 mole) [21] and benzo[*b*]thiophene-2-carboxaldehyde (**15**) (1.5 g, 0.0093 mole) in a manner similar to the preparation of compound **7** and 2.16 g (68%) of yellow prisms was obtained, mp 154°; nmr (deuteriochloroform): δ 7.04-8.18 (m, 2 \times ethenyl-*H*, H-3', H-4', H-5', H-6', H-7' of benzo[*b*]thiophene, H-2, H-3, H-4, H-6, H-7, H-8 of dibenzothiophene, 13H), 8.23-8.51 (m, H-9 of dibenzothiophene, 1H,

ArH); ms: *m/e* 340 (M^+ , 100).

Anal. Calcd. for $C_{22}H_{14}S_2$: C, 77.16; H, 4.12; S, 18.72. Found: C, 77.41; H, 4.20; S, 19.01.

Naphtho[1,2-*b*:6,5-*b'*]bisbenzo[*b*]thiophene (**17**). Method A.

Compound **16** (1.1 g, 0.0032 mole) was photocyclized in a manner similar to the photocyclization of compounds **4** and **5** and colorless prisms were obtained in 79% yield (0.86 g), mp 292°.

Method B.

Compound **17** was prepared by photocyclizing **20** (1.0 g, 0.0029 mole) in a manner similar to the preparation of compounds **4** and **5** and colorless prisms were obtained in 82% yield (0.81 g), mp 292°; nmr (deuteriochloroform): δ 7.13-8.59 (m, 12H, *ArH*); ms: *m/e* 340 (M^+ , 100).

Anal. Calcd. for $C_{22}H_{12}S_2$: C, 77.61; H, 3.55; S, 18.84. Found: C, 77.36; H, 3.74; S, 18.63.

1-(1-Dibenzothiényl)-2-(3-benzo[*b*]thienyl)ethene (**20**). Method A.

Chromium trioxide (5.7 g, 0.057 mole) was carefully added in small portion to 100 ml of dry pyridine. After stirring at room temperature for 30 minutes, 1-hydroxymethyl-dibenzothiophene (2.5 g, 0.012 mole) dissolved in 30 ml of dry pyridine was added to a suspension of chromium trioxide. After stirring at room temperature for two and a half hours, the reaction mixture was filtered to remove the inorganic compounds and washed with chloroform (3 \times 150 ml). The filtrate was washed several times with 20% hydrochloric acid and then with 10% aqueous sodium carbonate solution. The chloroform layer was dried over anhydrous sodium sulfate and evaporated *in vacuo* giving 1.8 g (72%) of a tan oil; ir (potassium bromide): 1695 cm^{-1} (C=O). The unstable crude aldehyde was used in the next reaction without further purification.

Compound **20** was synthesized from diethyl 3-benzo[*b*]thienylphosphonate (**2**) (2.5 g, 0.0088 mole) [13] and dibenzothiophene-1-carboxaldehyde (**19**) (1.87 g, 0.0088 mole) in a manner similar to the preparation of 1-(2-dibenzothiényl)-2-(3-benzo[*b*]thienyl)ethene (**3**) and a pale yellow oil was obtained in 85% yield (2.5 g).

Method B.

Compound **20** was prepared from diethyl 1-dibenzothiénylphosphonate (**21**) (3.1 g, 0.0093 mole) [21] and benzo[*b*]thiophene-3-carboxaldehyde (**12**) (1.5 g, 0.0093 mole) in a manner similar to the preparation of compound **3** and 2.15 g (73%) of pale yellow oil was obtained; nmr (deuteriochloroform): δ 7.00-8.46 (m, 2 \times ethenyl-*H*, aromatic-*H*, 14H).

Anal. Calcd. for $C_{22}H_{14}S_2$: C, 77.16; H, 4.12; S, 18.72. Found: C, 77.32; H, 4.30; S, 18.61.

1-(1-Dibenzothiényl)-2-(2-benzo[*b*]thienyl)ethene (**22**). Method A.

Compound **22** was prepared from diethyl 1-dibenzothiénylphosphonate (**21**) (3.1 g, 0.0093 mole) [21] and benzo[*b*]thiophene-2-carboxaldehyde (**15**) in a similar manner to the preparation of compound **7** and 2.26 g (71%) of pale yellow prisms was obtained, mp 150°.

Method B.

Compound **22** was prepared from the condensation of dibenzothiophene-1-carboxaldehyde (**19**) (1.87 g, 0.0088 mole) and diethyl 2-benzo[*b*]thienylphosphonate (**6**) (2.5 g, 0.0088 mole) [13] in a manner similar to the preparation of compound **7** and 2 g (67%) of pale yellow prisms was obtained, mp 150°; nmr (deuteriochloroform): δ 7.03-8.02 (m, 2 \times ethenyl-*H*, H-3', H-4', H-5', H-6', H-7' of benzo[*b*]thiophene, H-2, H-3, H-4, H-6, H-7, H-8 of dibenzothiophene, 13H), 8.09-8.45 (m, H-1 of dibenzothiophene, 1H, *ArH*).

Anal. Calcd. for $C_{22}H_{14}S_2$: C, 77.16; H, 4.12; S, 18.72. Found: C, 77.36; H, 4.29; S, 18.68.

Naphtho[2,1-*b*:6,5-*b'*]bisbenzo[*b*]thiophene (**23**).

Compound **22** (1.5 g, 0.0044 mole) was photocyclized in the presence of iodine (0.01 g) in a similar manner to the preparation of compounds **4** and **5** and colorless prisms (0.33 g, 22%) were obtained, mp 293°; nmr (deuteriochloroform): δ 7.12-8.05 (m, H-1, H-2, H-3, H-5, H-6, H-8, H-9,

H-10, H-12, H-13, 10H, ArH), 8.30-8.63 (m, H-4, H-11, 2H, ArH); ms: m/e 340 (M⁺, 100).

Anal. Calcd. for C₂₂H₁₂S₂: C, 77.61; H, 3.55; S, 18.84. Found: C, 77.52; H, 3.73; S, 19.06.

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