Synthesis of Indolizines and Pyrrolo[2,1-*b*]azoles from 2-Pyridylmagnesium Bromide and 2-Lithiated Azoles Using Tris(alkylthio)cyclopropenyl Cations as a Three-Carbon Building Block

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A convenient method for the preparation of indolizines **3** and **4** and pyrrolo[2,1-*b*]azoles **7a-e** from 2-pyridylmagnesium bromide and 2-lithiated azoles **6a-e**, respectively, was developed by using tris(*tert*-butylthio)- and tris(isopropylthio)cyclopropenyl cations **1** and **2** as a three-carbon building block.

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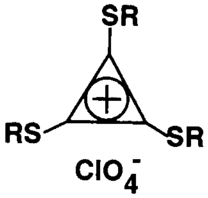
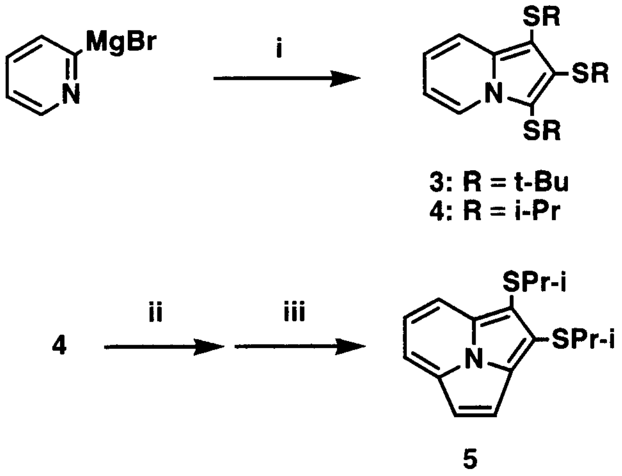
Cyclopropenyl cations, which are highly strained molecules, are of great value as a three-carbon building block in organic synthesis. However, little attention has been drawn to their synthetic use. Recently, it has been reported that the reaction of tris(*tert*-butylthio)cyclopropenylium perchlorate (**1**) with 3-aminopropionitrile and methyl 3-aminopropionate in *N,N*-dimethylformamide containing sodium hydride gives 1,2-dihydropyridines [1]. In relation to this study, we have reported more recently that tris(isopropylthio)cyclopropenylium perchlorate (**2**) reacts readily with anilines, 1-naphthylamines, and the *N*-anion of pyrrole to give quinolines, naphthazepines, and the pyrrolizine derivative, respectively, in high yields [2]. These reactions indicate that the cyclopropenyl cations bearing three alkylthio groups undergo facile ring opening and then intramolecular cyclization at the aliphatic or aromatic carbon atom to give nitrogen heterocycles. To develop a new method for preparing the heterocyclic systems with the cyclopropenyl cations, we carried out the reactions of **1** and **2** with compounds bearing the $-C=N-$ moiety. Herein, we report a convenient method for the preparation of indolizines **3** and **4** and pyrrolo[2,1-*b*]azoles **7a-e** from 2-pyridylmagnesium bromide and 2-lithiated azoles **6a-e**, respectively, using **1** and **2** as a three-carbon building block. A part of the results was reported in our preliminary paper [3].



1: R = t-Bu
2: R = i-Pr

The reaction of the cyclopropenyl cations **1** and **2** with 2-pyridylmagnesium bromide, prepared from 2-bromopyridine and magnesium metal, was carried out in dry tetrahydrofuran (THF) at room temperature for 30 minutes. 1,2,3-Tris(*tert*-butylthio)- and 1,2,3-tris(isopropylthio)indolizines **3** and **4** were obtained in 72 and 99% yields, respectively (Scheme 1). The molecular structure of **3** was determined by the single crystal X-ray analysis (Figure 1 and Table 1). Furthermore, the ^{13}C nmr spectra of **3** and **4** showed eight signals for the indolizine ring carbons at δ 108.5, 111.6, 119.2, 120.1, 120.5, 125.4, 134.1 and 139.7 and at δ 106.0, 111.6, 117.9, 120.3, 124.3, 133.1 and 138.7, respectively, thus corresponding to those of indolizine described previously [4]. The formation of **4** also was elucidated by the fact that **4** is converted efficiently into 1,2-bis(isopropylthio)cycl[3.2.2]azine (**5**) by the reaction of **4** with dimethyl acetylenedicarboxylate (DMAD) in boiling

Scheme 1



i, 1 or 2 / THF / r.t. ; ii, DMAD / Toluene / reflux ; iii, KOH / EtOH and then Cu / Quinoline .

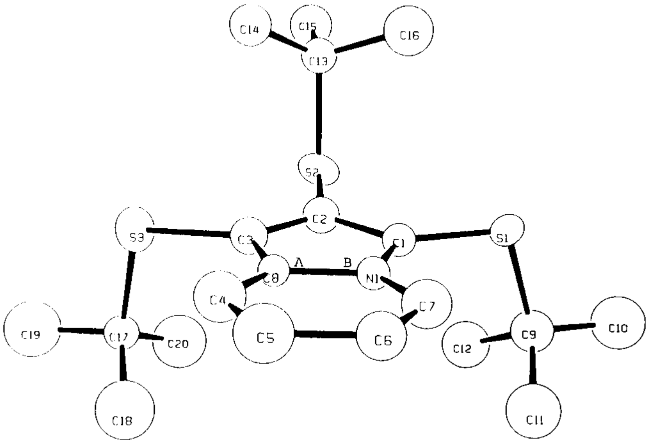


Figure 1. ORTEP drawing of **3**. Two atoms, N1 and C8, disordered over two sites, A and B, with total occupancy of 1.0 for each site. Hydrogen atoms are omitted.

Table 1

Positional Parameters and their Estimated Standard Deviations of **3**

Atom	x	y	z	B(Å^2)
S(1)	0.0787(8)	0.2690(5)	0.8500(8)	3.1(4)
S(2)	-0.3163(8)	0.2544(5)	0.9057(8)	2.9(3)
S(3)	-0.3233(8)	0.2840(5)	1.307(1)	4.0(4)
N(1A)	0.087(2)	0.307(1)	1.162(2)	1.9(5)
N(1B)	-0.011(2)	0.314(1)	1.276(2)	1.6(4)
C(1)	0.001(3)	0.283(2)	1.022(3)	2.1(5)
C(2)	-0.156(3)	0.277(2)	1.047(3)	2.5(6)
C(3)	-0.167(3)	0.288(2)	1.195(3)	2.6(6)
C(4)	0.052(3)	0.346(2)	1.430(3)	4.1(7)
C(5)	0.212(3)	0.371(2)	1.483(3)	5.0(7)
C(6)	0.309(3)	0.360(2)	1.358(3)	3.3(6)
C(7)	0.248(3)	0.332(2)	1.209(3)	3.6(6)
C(8A)	0.0873	0.3069	1.1616	1.9
C(8B)	-0.0113	0.3138	1.2756	1.6
C(9)	0.077(3)	0.143(2)	0.802(3)	3.9(6)
C(10)	0.134(3)	0.124(2)	0.647(3)	6.1(8)
C(11)	0.181(3)	0.119(2)	0.928(3)	5.7(8)
C(12)	-0.093(3)	0.074(2)	0.793(3)	5.0(7)
C(13)	-0.303(3)	0.383(2)	0.888(3)	2.5(6)
C(14)	-0.305(3)	0.445(2)	1.032(3)	3.3(6)
C(15)	-0.453(3)	0.364(2)	0.772(3)	4.3(7)
C(16)	-0.163(3)	0.421(2)	0.803(3)	5.0(7)
C(17)	-0.393(3)	0.152(2)	1.319(3)	2.7(6)
C(18)	-0.271(4)	0.127(2)	1.412(4)	6.7(8)
C(19)	-0.533(4)	0.142(2)	1.407(2)	6.8(9)
C(20)	-0.439(3)	0.087(2)	1.171(3)	6.5(9)

toluene to give dimethyl 3,4-bis(isopropylthio)cycl[3.2.2]azine-1,2-dicarboxylate, followed by the hydrolysis and then decarboxylation of the resulting cyclazine derivative (Scheme 1).

The reaction of **2** with 2-lithiated azoles **6a-e**, prepared from azoles and butyllithium, in dry THF or diethyl ether gave pyrrolo[2,1-*b*]azoles **7a-e** in good yields (Scheme 2). The results are summarized in Table 2.

Scheme 2

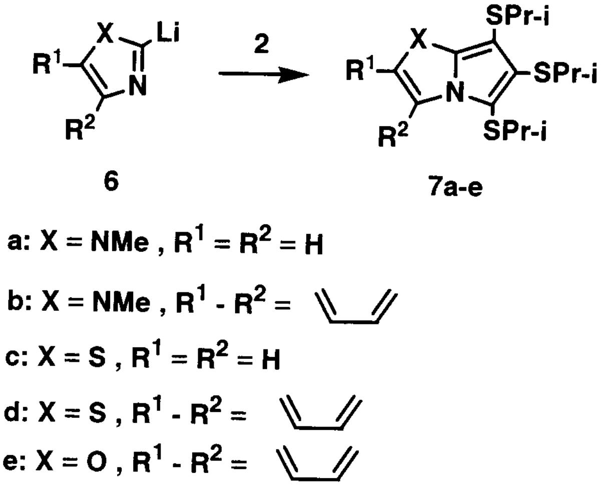


Table 2

Reaction of **2** with 2-Lithiated Azoles

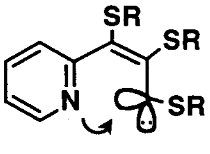
2-Lithiated Azole [a]	Solvent	Temperature °C	Time hours	Product (yield/%) [b]
6a	THF	-70 → rt	1	**7a** (98)
6b	Et$_2$O	-60 → rt	3	**7b** (76)
6c	Et$_2$O	-60 → rt	20	**7c** (71)
6d	THF	-60 → rt	21	**7d** (50)
6e	THF	-78 → rt	1.5	**7e** (77)

[a] The molar ratio of azoles to **2** = 1:1. [b] Isolated yield based on **2**.

The structures of **7a-e** were established by measuring the ^{1}H and ^{13}C nmr spectra (see Experimental). For example, the ^{1}H nmr spectrum of **7a** showed two doublets (2H, δ 6.60 and 7.10) for the imidazole ring protons, a singlet (3H, δ 3.84) for the methyl protons of the NCH$_3$ group, and three doublets (18H, δ 1.17, 1.19 and 1.23) and three septets (3H, δ 3.08, 3.17 and 3.60) for the methyl and methine protons of three isopropylthio groups, respectively. Its ^{13}C nmr spectrum showed six signals (6C, δ 84.8, 105.3, 106.7, 121.2, 131.3 and 139.8) for the pyrrolo[2,1-*b*]imidazole ring carbons and a signals (1C, δ 33.7) due to the NCH$_3$ group. As Table 2 shows, the reaction proceeded smoothly under mild conditions, although, in the cases of 2-lithiated thiazole **6c** and benzothiazole **6d**, the longer reaction time was required for raising the yields of the products. The reaction with 2-lithiated oxazole did not give the intended product, but resulted in the formation of a mixture of unidentified compounds.

The reaction for the formation of **3**, **4**, and **7a-e** is explained to proceed *via* the vinylcarbene intermediates **8** and **9** which are formed by the nucleophilic attack of 2-pyridylmagnesium bromide and 2-lithiated azoles on **1** or **2**. The resulting intermediates **8** and **9** undergo intramolecular cyclization with the nitrogen atom of the pyridine or azole ring to give **3**, **4**, and **7a-e**.

The above results provide the useful method for the preparation of indolizines **3** and **4** and pyrrolo[2,1-*b*]azoles **7a-e** using the cyclopropenyl cations **1** and **2**.



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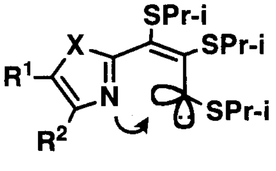

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EXPERIMENTAL

Melting points were determined on a Yanaco MP-S3 melting point apparatus and are uncorrected. The ir spectra were recorded on a Hitachi 215 spectrometer. The ^{1}H nmr (270 MHz) and ^{13}C nmr (68 MHz) spectra were measured on a JEOL JNM-GX 270 FT nmr spectrometer with tetramethylsilane as an internal standard. The uv spectra were obtained on Shimadzu UV-160 spectrophotometer. Mass spectra were obtained at 70 eV with a Finnigan mat TSQ 70 or Shimadzu LKB-9000 spectrometer. Elemental analyses were performed by a Yanaco CHN CORDER MT-3. The single crystal X-ray analysis of **3** was performed by a Rigaku AFC5R diffractometer. Column chromatography was performed on silica gel (Wakogel C-300). All reagents were obtained commercially and used without further purification.

General Procedure for the Preparation of Indolizines **3** and **4**.

1,2-Dibromoethane (282 mg, 1.5 mmoles) and 2-bromopyridine (237 mg, 1.5 mmoles) were added under argon to a suspended solution of magnesium metal (73 mg, 3 mmoles) in dry tetrahydrofuran (5 ml) at room temperature. The mixture was stirred until magnesium metal dissolves completely and **1** (201 mg, 0.5 mmole) or **2** (181 mg, 0.5 mmole) was added to the solution in one portion. The solution was stirred at room temperature for 30 minutes and then a saturated aqueous ammonium chloride solution was added. The mixture was extracted with dichloromethane (2 x 50 ml) and the extract was dried over anhydrous sodium sulfate. The solvent was removed *in vacuo* and the chromatography of the residual oil on silica gel with dichloromethane-hexane (1:3) as the eluent gave **3** (137 mg, 72%) and **4** (168 m, 99%). Compound **4** was converted efficiently into 1,2-bis(isopropylthio)cycl[3.2.2]-azine (**5**). The procedure for the preparation of dimethyl 3,4-bis-(isopropylthio)cycl[3.2.2]azine-1,2-dicarboxylate and the ir, ^{1}H and ^{13}C nmr, uv, and analytical data of **3**, **4**, and dimethyl 3,4-bis-(isopropylthio)[3.2.2]azine-1,2-dicarboxylate have been described in our preliminary paper [3].

Preparation of 1,2-Bis(isopropylthio)cycl[3.2.2]azine (**5**).

Potassium hydroxide (98 mg, 1.75 mmoles) was added to a solution of dimethyl 3,4-bis(isopropylthio)cycl[3.2.2]azine-1,2-dicarboxylate (42 mg, 0.1 mmole) in 95% ethanol (5 ml) and the mixture was stirred under reflux for 2 hours. After the mixture was cooled to room temperature, 12*N* aqueous hydrochloric acid solution (5 ml) was added and the mixture was extracted with dichloromethane (2 x 100 ml). The extract was washed with water (2 x 150 ml), dried over anhydrous sodium sulfate, and concentrated *in vacuo* to give 3,4-bis(isopropylthio)cycl[3.2.2]azine-1,2-dicarboxylic acid as orange solids in quantitative yield. Further purification was performed by recrystallization from tetrachloromethane. This compound was obtained as an orange powder, mp 188-189° dec; ir (potassium bromide): 3100, 2950, 2900, 2510, 1690, 1650, 1475, 1310, 1290, 1240, 1210, 1180, 1120, 1050, 780 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.31 (d, J = 6.7 Hz, 6H, CH(CH_3)$_2$), 1.37 (d, J = 6.7 Hz, 6H, CH(CH_3)$_2$), 3.62 (sep, J = 6.7 Hz, 1H, CH(CH$_3$)$_2$), 3.89 (sep, J = 6.7 Hz, 1H, CH(CH$_3$)$_2$), 8.09 (t, J = 7.9 Hz, 1H, aromatic), 8.23 (d, J = 7.9 Hz, 1H, aromatic), 8.98 (d, J = 7.9 Hz, 1H, aromatic); ^{13}C nmr (deuteriochloroform): δ 22.8, 23.6, 41.3, 43.0, 115.0, 116.2, 118.5, 121.5, 127.9, 128.6, 129.0, 131.3, 131.9, 134.1; uv (acetonitrile): λ max (ϵ) 268 (23400), 430 (12000) nm.

Anal. Calcd. for $C_{18}H_{19}NO_4S_2$: C, 57.27; H, 5.07; N, 3.71. Found: C, 57.03; H, 5.07; N, 3.68.

Copper powder (3 mg, 0.05 mmole) was added under nitrogen to a solution of 3,4-bis(isopropylthio)cycl[3.2.2]azine-1,2-dicarboxylic acid (38 mg, 0.1 mmole) in dry quinoline (2 ml) and the mixture was stirred at 200° for 4 hours. After the mixture was cooled to room temperature, the solid was filtered off. The filtrate was concentrated *in vacuo* and the residue was chromatographed on silica gel eluting with dichloromethane-hexane (1:19) to give **5** as a yellow oil in quantitative yield; ir (neat): 3075, 2970, 2925, 2870, 1615, 1535, 1505, 1465, 1445, 1425, 1390, 1370, 1350, 1345, 1310, 1275, 1240, 1195, 1160, 1050, 1040, 1025, 780 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.27 (d, J = 6.7 Hz, 6H, CH(CH_3)$_2$), 1.51 (d, J = 6.7 Hz, 6H, CH(CH_3)$_2$), 3.34 (sep, J = 6.7 Hz, 1H, CH(CH$_3$)$_2$), 4.10 (sep, J = 6.7 Hz, 1H, CH(CH$_3$)$_2$), 7.27 (d, J = 4.3 Hz, 1H, aromatic), 7.60 (d, J = 4.3 Hz, 1H, aromatic), 7.71 (t, J = 7.7 Hz, 1H, aromatic), 7.86 (d, J = 7.7 Hz, 1H, aromatic), 7.91 (d, J = 7.7 Hz, 1H, aromatic); ^{13}C nmr (deuteriochloroform): δ 23.5, 23.8, 37.6, 40.4, 110.1, 110.3, 110.5, 112.1, 117.4, 121.8, 125.2, 130.3, 133.6, 135.4; uv (acetonitrile): λ max (ϵ) 212 (18800), 228 (18800), 253 (23300), 340 (13100), 408 (6300) nm; ms: m/z 289 (M$^+$).

Anal. Calcd. for $C_{16}H_{19}NS_2$: C, 66.39; H, 6.62; N, 4.84. Found: C, 66.21; H, 6.58; N, 4.67.

General Procedure for the Preparation of Pyrrolo[2,1-*b*]azoles **7a-e**.

A solution of butyllithium (1.6 mmoles) in hexane was added under nitrogen to a solution of azoles (1.5 mmoles) in dry tetrahydrofuran or diethyl ether (10 ml) at −78° to −60° and the mixture was stirred for 1 hour (in the case of *N*-methylbenzimidazole, the mixture was stirred for 4 hours). Quantitative formation of 2-lithiated azoles **6a-e** was ascertained by the ^{1}H nmr analysis of the mixture on treatment with deuterium oxide. Then the cyclopropenyl cation **2** (541 mg, 1.5 mmoles) was added at −78° to −60° and the mixture was allowed to warm to room temperature. After 1-21 hours (Table 2), an aqueous ammonium chloride solution was added and the mixture was extracted with dichloromethane (100 ml). The extract was dried over anhydrous sodium sulfate and concentrated *in vacuo*. The residue was chromatographed on silica gel using hexane-dichloromethane as the eluent.

1,2,3-Tris(isopropylthio)-*N*-methylpyrrolo[2,1-*b*]imidazole (**7a**).

Chromatography using hexane-dichloromethane (7:1) as the eluent gave **7a** as a yellowish oil in 98% yield; ir (neat): 2965, 2925, 2870, 1590, 1465, 1450, 1370, 1270, 1260, 1250, 1160, 1125, 1100, 1050, 740, 700, 680 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.17 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.19 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.23 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 3.08 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.17 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.60 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.84 (s, 3H, NCH_3), 6.60 (d, J = 1.8 Hz, 1H), 7.10 (d, J = 1.8 Hz, 1H); ^{13}C nmr (deuteriochloroform): δ 22.8, 23.1, 23.2, 33.7, 39.3, 40.5, 40.9, 84.8, 105.3, 106.7, 121.2, 131.3, 139.8; uv (acetonitrile): λ max (ϵ) 212 (20200), 255 (10000, sh), 307 (12900) nm; ms: m/z 342 (M$^+$).

Anal. Calcd. for $C_{16}H_{26}N_2S_3$: C, 56.10; H, 7.65; N, 8.18. Found: C, 55.98; H, 7.94; N, 8.00.

1,2,3-Tris(isopropylthio)-*N*-methylpyrrolo[2,1-*b*]benzimidazole (**7b**).

Chromatography using hexane-dichloromethane (5:1) as the eluent gave **7b** as a colorless oil in 76% yield; ir (neat): 3075, 2970, 2930, 2875, 1620, 1580, 1500, 1470, 1390, 1370, 1250, 1160, 1055, 870, 760, 740 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.22 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.23 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.26 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 3.18 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.28 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.68 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.97 (s, 3H, NCH_3), 7.15-7.20 (m, 2H), 7.25-7.28 (m, 1H), 8.29 (d, J = 7.9 Hz, 1H); ^{13}C nmr (deuteriochloroform): δ 22.8 (2C), 23.1, 30.2, 39.3, 40.3, 40.9, 86.2, 108.2, 111.1, 112.3, 119.6, 123.1, 127.0, 132.5, 137.3, 142.0; uv (acetonitrile): λ max (ϵ) 243 (25000), 276 (23400), 341 (8700) nm; ms: m/z 392 (M$^+$).

Anal. Calcd. for $C_{20}H_{28}N_2S_3$: C, 61.18; H, 7.19; N, 7.13. Found: C, 61.25; H, 7.48; N, 6.98.

1,2,3-Tris(isopropylthio)pyrrolo[2,1-*b*]thiazole (**7c**).

Chromatography using hexane-dichloromethane (4:1) as the eluent gave **7c** as a yellowish oil in 71% yield; ir (neat): 3110, 2960, 2930, 2870, 1555, 1475, 1415, 1385, 1370, 1325, 1250, 1160, 1120, 1055, 885, 795, 710 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.20 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.22 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.23 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 3.24 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.32 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.62 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 6.75 (d, J = 4.3 Hz, 1H), 7.63 (d, J = 4.3 Hz, 1H); ^{13}C nmr (deuteriochloroform): δ 23.1, 23.2, 23.3, 39.2, 39.8, 41.2, 105.7, 111.5, 117.4, 120.8, 131.8, 138.2; uv (acetonitrile): λ max (ϵ) 223 (17100), 302 (7900) nm; ms: m/z 345 (M$^+$).

Anal. Calcd. for $C_{15}H_{23}NS_4$: C, 52.13; 6.71; N, 4.05. Found: C, 52.35; H, 7.01; N, 3.82.

1,2,3-Tris(isopropylthio)pyrrolo[2,1-*b*]benzothiazole (**7d**).

Chromatography using hexane-dichloromethane (4:1) as the eluent gave **7d** as colorless crystals in 50% yield, 91.5-92.5° dec; ir (potassium bromide): 3050, 2960, 2930, 2860, 1500, 1485, 1395, 1370, 1270, 1250, 1150, 1050, 910, 740 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.24 (d, J = 6.7 Hz, 12H, $CH(CH_3)_2$), 1.25 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 3.31 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.38 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.67 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 7.29-7.32 (m, 1H), 7.37-7.43 (m, 1H), 7.62-7.66 (m, 1H), 8.79 (m, 1H); ^{13}C nmr (deuteriochloroform): δ 22.7, 23.2, 23.3, 39.3, 39.6, 41.4, 107.0, 115.4, 120.9, 123.7, 124.2, 125.5, 130.9, 133.4, 136.6, 137.8; uv (acetonitrile): λ max (ϵ) 246 (27100), 322 (7300) nm; ms: m/z 395 (M$^+$).

Anal. Calcd. for $C_{19}H_{25}NS_4$: C, 57.68; H, 6.37; N, 3.54. Found: C, 57.45; H, 6.51; N, 3.40.

1,2,3-Tris(isopropylthio)pyrrolo[2,1-*b*]benzoxazole (**7e**).

Chromatography using hexane-dichloromethane (1:1) as the eluent gave **7e** as colorless crystals in 77% yield, mp 69-70°; ir (potassium bromide): 3075, 2970, 2930, 2875, 1630, 1570, 1490, 1470, 1420, 1380, 1370, 1240, 1200, 1160, 1125, 1055, 860, 735 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 1.25 (d, J = 6.7 Hz, 6H, $CH(CH_3)_2$), 1.26 (d, J = 6.7 Hz, 12H, $CH(CH_3)_2$), 3.29 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.30 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 3.63 (sep, J = 6.7 Hz, 1H, $CH(CH_3)_2$), 7.27-7.31 (m, 2H), 7.45-7.49 (m, 1H), 8.06-8.10 (m, 1H); ^{13}C nmr (deuteriochloroform): δ 23.0, 23.1, 23.2, 39.4 (2C), 41.4, 88.8, 111.9, 112.0, 112.5, 123.7, 124.0, 128.2, 131.9, 150.8, 151.8; uv (acetonitrile): λ max (ϵ) 237 (28500), 299 (11300) nm; ms: m/z 379 (M$^+$).

Anal. Calcd. for $C_{19}H_{25}NOS_3$: C, 60.12; H, 6.64; N, 3.69. Found: C, 59.89; H, 6.87; N, 3.59.

Single Crystal X-Ray Analysis of **3**.

A colorless prism crystal of **3** having approximate dimensions of 0.3000 x 0.2000 x 0.1000 mm was mounted on a glass fiber. Crystal data: $C_{20}H_{31}NS_3$; triclinic, space group $P\bar{1}$(#2); a = 8.956(4) Å, b = 14.621(6) Å, c = 8.913(6) Å, α = 99.81(4)°, β = 94.25(5)°, γ = 106.83(3)°, V = 1091(1) Å^3, Z = 2, D_{calc} = 1.161 g/cm^3. Intensity measurements were made with $9.11 < 2\theta < 20.60°$ by using graphite monochromated MoKα radiation at room temperature on a Rigaku AFC5R diffractometer. Of the 2830 reflections which were collected, 2607 were unique (R_{int} = 0.158). The structure was solved by direct methods and refined by using the TEXSAN [5] crystallographic software. In the final stage of refinement, the non-hydrogen atoms were refined either anisotropically or isotropically. Final residuals are R = 0.073 and R_w = 0.065.

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