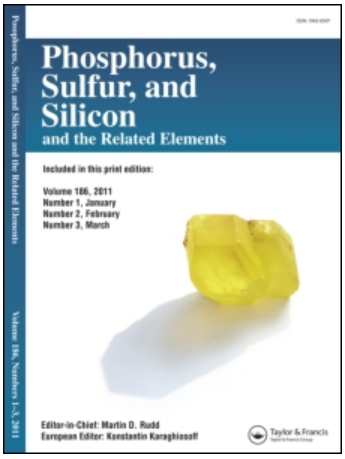

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SYNTHESIS OF POLYFUSED THIEN0(2,3-b)THIOPHENES PART 2: SYNTHESIS OF THIENOPYRIMIDINE, THIENOTHIAZINE, THIENOPYRROLOPIPRAZINE, AND THIENOTHIAZAPHOSPHOLINE DERIVATIVES

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SYNTHESIS OF POLYFUSED THIENO(2,3-b)THIOPHENES PART 2: SYNTHESIS OF THIENOPYRIMIDINE, THIENOTHIAZINE, THIENOPYRROLOPIPRAZINE, AND THIENOTHIAZAPHOSPHOLINE DERIVATIVES

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3,4-Diamino-2,5-dicarbethoxythieno(2,3-b)thiophene (**1**) was reacted with triethylorthoformate to afford compound **2**. The reaction of compounds **2** or **3** with S,S-acetals and N,S-acetals, afforded compounds **4–6**. Treatment of compound **1** with 2,5-dimethoxytetrahydrofuran gave the corresponding bipyrrolyl derivative **7**, which was reacted with hydrazine hydrate to get the corresponding hydrazide derivative **8**. Treating compound **8** with sodium nitrite afforded the corresponding carboazide derivative **9**, which in turn cyclized to compound **10**. Compound **12** was obtained via the reaction of compound **1** with PhNCS or through treating compound **11** with CS_2 $PhNH_2$, and KOH. Also, compound **13** was prepared through treatment of compound **12** with Lawesson's reagent or via the reaction of compound **11** with CS_2 followed by treating the resulting compound **14** with $PhNH_2$. The reaction of compounds **1** or **3** with Lawesson's reagent gave compound **15**. Benzoylation of compound **1** afforded compound **16**, which treated with NH_3 gave compound **17**. Treatment of compounds **16** or **17** with LR furnished compounds **18** or **19**, respectively. The reaction of compound **16** or **17** with P_2S_5 yielded compounds **19** and **20** or **18** and **19**, respectively.

Keywords: PTC; 1; 3-Dithiazine; Lawesson's reagent (LR); Thienopyrimidine; Thienothiazine; Thienopyrrolopiprazine and Thienothiazaphospholine Derivatives

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INTRODUCTION

The biological activity of many heterocyclic compounds containing thiophene ring has reviewed.[1–4] In view of these observation and as a continuation of our previous work,[5,6] we undertook the synthesis of some new heterocyclic compounds containing a thieno(2,3-b)thiophene moiety fused with pyrimidine, thiazine, dithiazine, or thiazaphospholine nucleus.

RESULTS AND DISCUSSIONS

Compound **1** was allowed to react with triethylorthoformate to afford 2,5-dicarbethoxy-3,4-diethoxymethyleneaminothieno(2,3-b)thiophene (**2**). The treatment of compound **1** with hydrazine hydrate gave 3,4-diaminothieno(2,3-b)thiophene-2,5-dicarbohydrazide (**3**).[7]

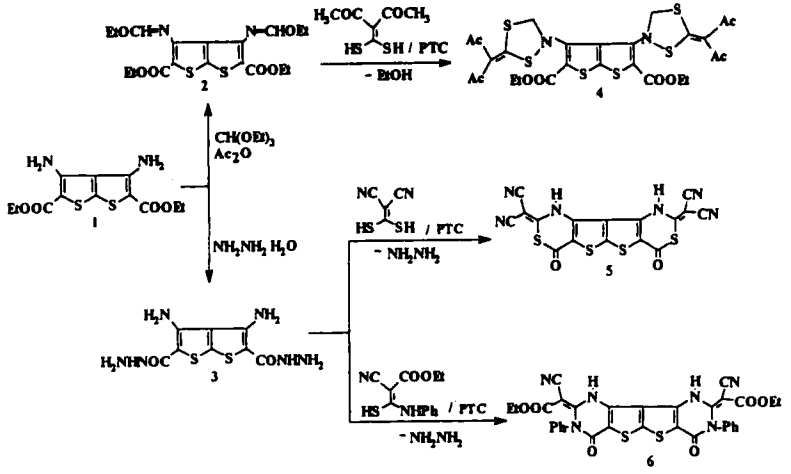
On treating compound **2** with S,S-acetal, which was obtained via reaction of acetylacetone with carbon disulphide under phase-transfer catalysis conditions (PTC), gave 2,5-dicarbethoxy-3,4-di[2′-diacetyl-methylene-4′-(1,3,4)dithiazolyl]thieno(2,3-b)thiophene (**4**). The reaction pathway should proceed through the addition of one SH group of the S,S-acetal at the N=C bond followed by interamolecular cyclization via elimination of an ethanol molecule. Also, treatment of compound **3** with S,S- or N,S-acetals, which were prepared from the reaction of CS_2 with malononitrile or phenylisothiocyanate with ethyl cyanoacetate under PTC conditions, afforded bis[(4-oxo-1(H)thieno(2,3-b)-1′,3′-thiazin-2′-ylidene) malononitrile (**5**) and bis[ethyl(4-oxo-3-phenyl-1(H)thieno(2,3-b)-pyrimidin-2-ylidene) cyanoacetate] (**6**), respectively. The reaction mechanism was postulated to proceed through the nucleophilic attack of the SH group of the S,S-acetal or NH group of the N,S-acetal at the carbonyl group with elimination of a hydrazine molecule followed by intramolecular cyclization through elimination of an H_2S molecule. The IR spectra of compounds **4–7** showed an absorption bands corresponding to $C{=}O_{ester,\ ketone\ or\ acetyl}$, CN and NH groups at 1743 cm^{-1},1710 cm^{-1}, 1689 cm^{-1}, 2100 cm^{-1} and 3210 cm^{-1}, respectively. ^{1}H-nmr spectra were consistent with the proposed structures (cf. Scheme 1, Table I).

TABLE I Analytical and Spectral Data of the New Compounds

Product No.	M.P. (°C)[a]	Yield (%)	Mole. Form. (Mol.wt.)	Analytical Data[b] Cal./Found				IR (Cm^{-1})[c]	^{1}H-NMR ∂ (ppm)[d]
				C	H	N	S		
2	170–2	66	$C_{18}H_{22}N_2O_6S_2$ (426.52)	50.69 50.90	5.20 5.41	6.57 6.75	15.04 15.25	1743 (CO_{ester}), 1621 (C=N), 1108 (C-O-C).	6.0 (s, 2H, 2 =CH), 4.4–4.2(q, 4H, $2CH_2$ ester), 3.7–3.4 (q, 4H,$2CH_2$), 1.5–1.1 (m, 12H, $4CE_3$).
4	140–2	90	$C_{26}H_{26}N_2O_8S_6$ (686.89)	45.46 45.10	3.82 3.51	4.08 4.20	28.00 28.24	3210 (NH), 1743 (CO_{ester}), 1678 (CO_{acetyl})	9.80(s,2H,2NH), 4. 44.1(q,4H, $2CH_2$ ester), 2.3(s,6H,$2CH_3$), 1.3-1.1 (t, 6H, $2CH_3$).
5	211	29	$C_{16}H_2N_6O_2S_4$ (438.49)	43.83 43.61	0.46 0.78	19.19 19.35	29.25 29.44	3328 (NH), 2212 (CN), 1706 (CO).	9.1 (s, 2H, 2NH).
6	185–7	40	$C_{32}H_{22}N_6O_6S_2$ (650.7)	59.07 59.31	3.41 3.72	12.92 12.66	9.86 9.64	3249 (NH), 2210(CN), 1737 (CO_{ester}), 1698 (CO).	7.7–7.0(m,12H,atomatic +2NH), 4.1–4.1(q, 4H,$2CH_2$ ester), 1.4–1.1 (t, 6H, $2CH_3$).
7	320	60	$C_{20}H_{18}N_2O_4S_2$ (414.51)	57.95 57.60	4.38 4.60	6.76 6.47	15.47 15.65	1740 (CO_{ester}).	7.7–6.9 (d, 2H, 2 =CH_α), 6.3–6.0(t,2H, 2 =CH_β), 4.4- 4.1(q, 4H, $2CH_2$ ester), 1.3–1.0 (t, 6H, $2CH_3$).
8	299	69	$C_{16}H_{14}N_6O_2S_2$ (386.46)	49.73 49.89	3.65 3.93	21.75 21.59	16.59 16.71	3340, 3249, 3310 (NH, NH_2), 1688 (CO).	9.2 (s,2H,2NH), 7.7–6.9 (d,2H, 2 =CH_α), 6.3–6.0 (t,2H,2 =CH_β), 5.0–4.5 (br, 4H, $2NH_2$).
9	210–2	80	$C_{16}H_8N_8O_2S_2$ (408.41)	47.05 47.30	1.97 1.70	27.58 27.70	15.70 15.55	2200 (N_3), 1670 (CO).	7.6–7.3 (t, 2H, 2=CH_α), 6.2–5.9(t,2H, =CH_β).
10	322	90	$C_{16}H_8N_4O_2S_2$ (352.4)	54.53 54.20	2.29 2.40	15.90 15.69	18.19 18.34	3224(NH), 1688(CO).	9.0(s,2H,2NH), 6.7(s,1H,=CH_α), 6.6–6.3 (t, 2H, 2 =CH_β)
12	216	70	$C_{22}H_{12}N_4O_2S_2$ (428.49)	61.67 61.41	2.82 2.61	13.08 13.37	14.97 14.77	3320 (NH), 1678(CO), 1154 (CS).	9.5(s,2H, 2 NH), 7.3–6.8 (m, 10H, aromatic).

Product No.	M.P. (°C)[a]	Yield (%)	Mole. Form. (Mol.wt.)	Analytical Data[b] Cal./Found				IR (Cm^{-1})[c]	^{1}H-NMR ∂ (ppm)[d]
				C	H	N	S		
13	261	93	$C_{22}H_{12}N_4S_4$ (460.62)	57.37 57.59	2.63 2.40	12.16 12.34	27.85 27.64	3320 (NH), 1150(CS).	9.0(s,2H, 2 NH), 7.0–6.6 (m, 10H, aromatic).
14	188	30	$C_{10}H_2N_2S_8$ (406.65)	29.54 29.71	0.50 0.74	6.89 6.68	63.08 63.26	3211(NH), 1400 (CS).	9.6 (s, 2H, 2NH)
15	197–9	75	$C_{22}H_{16}P_2N_2O_4S_6$ (626.74)	42.16 42.31	2.57 2.71	4.47 4.68	30.70 30.53	3311(NH), 1710(CO), 655 (P=S).	9.5(s,2H,2NH), 7.8–7.0(m,10H, aromatic), 3.4 (s, 6H,2 OCH_3).
16	165–7	90	$C_{26}H_{22}N_2O_6S_2$ (522.61)	59.75 59.59	4.24 4.55	5.36 5.57	12.27 12.57	3170 (NH), 1735 (CO_{ester}), 1671 (CO).	8.0 (s, 2H, 2NH), 8.0–7.4(m,10H, aromatic), 4.3–4.0 (q,4H, $2CH_2$), 1.3–1.0 (t, 6H, $2CH_3$).
17	109–111	95	$C_{22}H_{16}N_4O_4S_2$ (464.53)	56.88 56.50	3.47 3.69	12.06 12.20	13.80 13.62	3350, 3230, 3190 (NH, NH_2), 1695 (CO).	9.3 (s, 2H, 2NH), 8.0–7.4(m,10H, aromatic), 5.3–5.0 (br,4H, $2NH_2$).
18	154	35	$C_{22}H_{10}N_2O_2S_4$ (462.60)	57.12 57.48	2.18 2.31	6.06 6.32	27.73 27.98	1690 (CO).	8.0–7.5 (m, 10H, arom).
19	243–5	48	$C_{22}H_{10}N_2S_6$ (494.73)	53.41 53.70	2.04 2.30	5.66 5.89	38.90 38.70	1065 (CS).	8.6–8.1 (m, 10H, + arom).
20	176–9	32	$C_{26}H_{22}N_2O_4S_4$ (554.74)	56.30 56.00	4.00 4.30	5.05 5.32	23.12 23.35	310 (NH), 1741 CO), 1134 (CS).	8.8(s,2H,2NH), 7.7–7.0(m,10H, aromatic), 4.4–4.1(q, 4H,$2CH_2$), 1.4–1.1 (t, 6H, $2CH_3$).

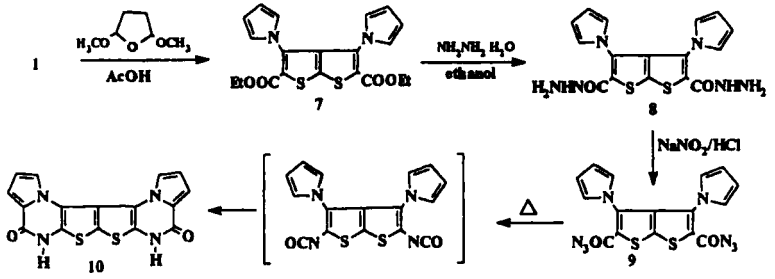
a. Uncorrected.
b. Satisfactory microanalysis obtained C; ± 0.35, H; ± 0.4, N; ± 0.2, S; ± 0.2.
c. Measured by Nicolet FT-IR 710 Spectrophotometer.
d. Measured by a Varian EM 360 L spectrometer at 60 MHZ using TMS as internal standard and DMSO as a solvent.



SCHEME 1

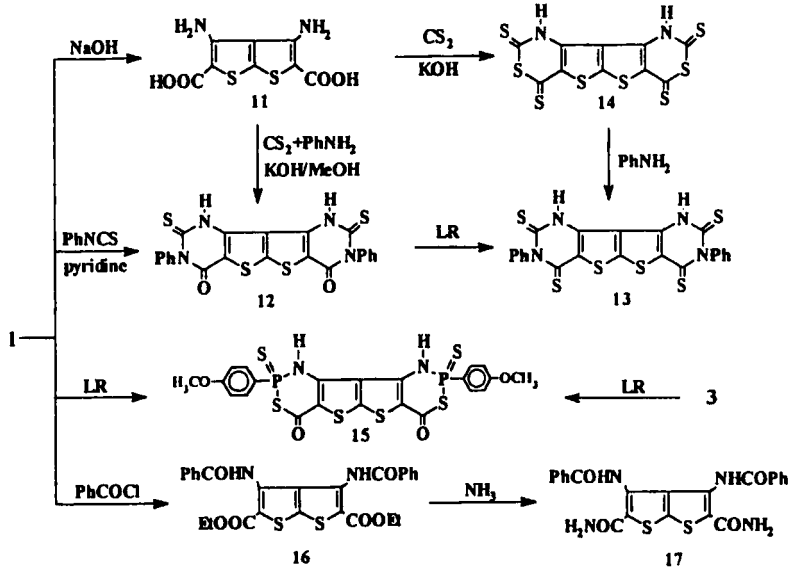
The reaction of compound **1** with 2,5-dimethoxytetrahydrofuran in refluxing acetic acid yielded 2,5-dicarbethoxy-3,4-di(pyrrol-1′-yl)thieno-(2,3-b)thiophene (**7**), which was reacted with hydrazine hydrate to afford 3,4-di(pyrrol-1′-yl)thieno(2,3-b)thiophene-2,5-dicarbohydrazide (**8**) which in turn was converted to 2,5-dicarbazido-3,4-di(pyrrol-1′-yl) thieno(2,3-b)thiophene (**9**) when treated with nitrous acid.[8] IR spectral analysis of compound **9** showed an absorption bands corresponding to N_3 and C=O groups at 2110 cm^{-1} and 1640 cm^{-1}, respectively. This acid azide derivative **9** was easily decomposed at 170°C through a *Curtius rearrangement*[9] and subsequent ring closure to give the corresponding bis[thienopyrrolopiprazine] **10** via the intermediacy of isocyanate derivative (cf. Scheme 2, Table I).

The saponification of compound **1** with sodium hydroxide furnished 3,4-diaminothieno(2,3-b)thiophene-2,5-dicarboxylic acid (**11**),[7] which, upon treatment with aniline, carbon disulphide and potassium hydroxide in 1:2:4:2 molar ratio, underwent cyclization into the corresponding bis[3-phenyl-1(H)-2-thioxothieno(3,2-d)pyrimidin-4-one] (**12**). Another route for the synthesis of compound **12** is the one pot reaction of compound **1** with phenylisothiocyanate in dry pyridine. The interaction of compound **12** with Lawesson's reagent (LR) produced the corresponding



SCHEME 2

bis[4-phenyl-1(H)-thieno(3,2-d)pyrimidine-2,4-dithione] (**13**). Compound **13** also can be obtained on reacting compound **11** with excess of carbon disulphide followed by treating the resulting compound bis[1(H)thieno(3,2-d)(1,3)thiazin-2,4-dithione] (**14**) with aniline. (cf. Scheme 3, Table I).

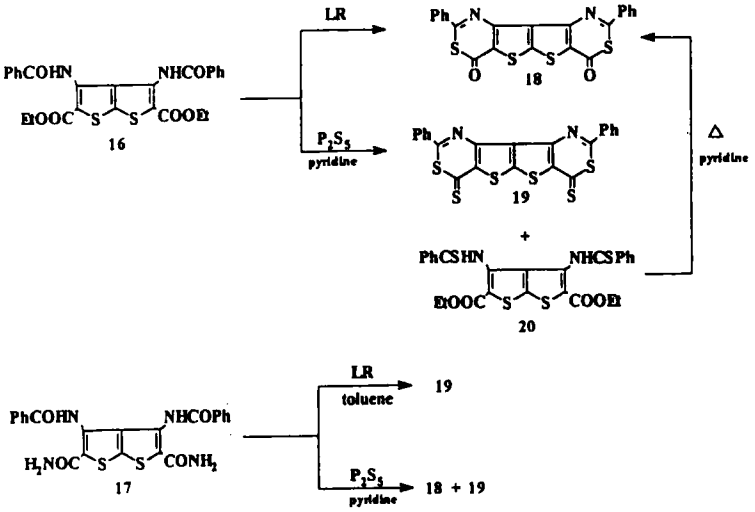


SCHEME 3

The chemistry of Lawesson's reagent (LR) as a thiation reagent has been studied, and several papers describe its ring-closure reactions with substrates containing two functional groups.[6,11–13] Thus, the reaction of compounds **1** or **3** with Lawesson's reagent in boiling toluene yielded bis[theino(2,3-d)(Δ^4-1,3,2)thiazaphospholine (**15**). IR analysis showed the following absorption bands at 3390 cm^{-1}, 1670 cm^{-1} and 1045 cm^{-1} for NH, C=O and P=S groups, respectively (cf. Scheme 3, Table I).

Benzoylation of compound **1** with benzoyl chloride in pyridine furnished 3,4-dibenzoylamino-2,5-dicarbethoxythieno(2,3-b)thiophene (**16**), which in turn was treated with ammonia to give 3,4-dibenzoyl-aminothieno(2,3-b)thiophene-2,5-dicarboxamide (**17**). These compounds were proved to be good starting materials for the synthesis of many polyfused thienothiophenes, such as where compound **16** was allowed to react with Lawesson's reagent to yield bis[4-oxo-2-phenylthieno(2,3-d)(1,3)-thiazine] (**18**). However, the interaction of compound **16** with P_2S_5 in refluxing pyridine gave bis[4-thiono-2-phenylthieno(2,3-d)(1,3)thiazine] (**19**, 48%) and 3,4-thiobenzamido-2,5-dicarbethoxythieno(2,3-b)thiophene (**20**, 32%), respectively.

Compound **17** was allowed to react with Lawesson's reagent or P_2S_5 to afford compound **19** or compounds **18** and **19**, respectively. Compound **20** was converted into compound **18** on heating in dry pyridine (cf. Scheme 4, Table I).



SCHEME 4

EXPERIMENTAL

Synthesis of 2,5-Dicarbethoxy-3,4-diethoxymethyleneaminothieno-(2,3-b)thiophene (2)

To a solution of compound **1** (0.01 mol) in acetic anhydride (10 ml) was added triethylorthoformate (0.02 mol). The reaction mixture was refluxed for 5 h and evaporated *in vacuo*. The residual solid was washed with water and crystallized from ethanol (cf. Scheme 1, Table I).

Synthesis of Compounds 4–6

(General Procedure)

A mixture of a proper active methylene compound (0.04 mol), CS_2 (0.045 mol) or phenylisothiocyanate (0.04 mol), anhydrous potassium carbonate (3 gm), a catalytic amount of TBAB, and dioxan (20 ml) was stirred for 40 minutes at 60°C To the dianionic ambident was added to compounds **2** or **3** (0.02 mol). The reaction mixture was stirred for 6 h at 40°C, filtered, and evaporated *in vacuo*. The residual solid was washed with water, collected by filtration, and crystallized from ethanol (cf. Scheme 1, Table I).

Synthesis of 2,5-Dicarbethoxy-3,4-di(pyrrol-1′-yl)thieno(2,3-b) thiophene (7)

To a solution of compound **1** (0.01 mol) in glacial acetic acid (10 ml) was added 2,5-dimethoxytetrahydrofuran (0.02 mol). The reaction mixture was refluxed for 1 h and evaporated *in vacuo*. The residual solid was washed with water and crystallized from dioxan (cf. Scheme 2, Table I).

Synthesis of 3,4-Di(pyrrol-1′-yl)thieno(2,3-b)thiophene-2,5-dicarbohydrazide (9)

To a solution of compound **8** (0.01 mol) in ethanol (20 ml) was added hydrazine hydrate (0.025 mol). The reaction mixture was refluxed for 3 h and evaporated *in vacuo*. The residual solid was crystallized from benzene (cf. Scheme 2, Table I).

Synthesis of 2,5-Dicarbazido-3,4-di(pyrrol-1′-yl)thieno(2,3-b)thiophene (9)

A solution of compound **9** (0.01 mol) in conc HCl (4 ml) and water (3 ml) was cooled in an ice bath at 0–5°C, whereupon a cold solution of sodium nitrite (0.06 mol) in water (5 ml) was added dropwise while stirring. The reaction mixture was set aside for 3 h, and then the separated solid was filtered, washed with water, dried, and crystallized from benzene (cf. Scheme 2, Table I).

Synthesis of Bis[thienopyrrolopiprazine] derivative 11

A solution of the carbazide compound **10** (0.01 mol) in *o*-dichlorobenzene (20 ml) was heated under reflux for 3 h. The solvent was evaporated *in vacuo* and the residue was triturated with pet. ether (40/60 °C). The separated solid was filtered off and crystallized from dimethylformamide (cf. Scheme 2, Table I).

Synthesis of Bis[4-oxo-3-phenyl-1(H)-2-thioxothieno(3,2-d) pyrimidine] (12)

Method (A)

To a suspension of compound **1** (0.01 mol) in pyridine (10 ml) was added phenylisothiocyanate (0.02 mol). The reaction mixture was refluxed for 10 h. After cooling, it was poured into a mixture of ice – water (200 ml) and HCl (10 ml). The separated solid was collected by filtration, washed with water, dried and crystallized from ethanol (cf. Scheme 3, Table I).

Method (B)

A mixture of compound **11** (0.01 mol) and KOH (0.025 mol) was dissolved in methanol (20 ml) and brought to reflux. A mixture of carbon disulphide (0.05 mol) and aniline (0.02 mol) was added slowly while the reaction mixture was maintained under reflux. On cooling, the precipitated solid was filtered, washed with methanol and dissolved in aqueous potassium hydroxide solution (10 ml, 10%). The solution was filtered, and the product was reprecipitated by addition of dil HCl. The product was filtered off, washed with water and crystallized from ethanol (cf. Scheme 3, Table I).

Synthesis of Bis[1(H)thieno(2,3-d)(1,3)thiazin-2,4-dithione] (14)

A mixture of compound **11** (0.02 mol), carbon disulphide (0.09 mol) and potassium hydroxide (0.06 mol) in methanol (10 ml) was refluxed for 4 h. On cooling, the precipitated solid was filtered, dried, washed with ether, dissolved in water, and the product was reprecipitated by addition of HCl (20 ml). The product was filtered, washed with water and crystallized from ethanol (cf. Scheme 3, Table I).

Synthesis of Bis[3-phenyl-1(H)thieno(2,3-d)pyrimidine-2,4-dithione] (13)

Method (A)

Compound **12** (0.01 mol) was dissolved in dry toluene (100 ml) and Lawesson's reagent (0.02 mol) was added. The reaction mixture was refluxed for 4 h and then filtered while still hot. The filtrate was evaporated to dryness under reduced pressure, and the residue was washed with aqueous sodium hydrogen carbonate (40 ml, 10%) and with water (20 ml). The solid product was dried and crystallized from ethanol (cf. Scheme 3, Table I).

Method (B)

A mixture of compound **14** (0.03 mol), aniline (0.06 mol) and potassium hydroxide (10 ml, 0.08 mol) in methanol (20 ml) was refluxed for 3 h. The solvent was evaporated *in vacuo*, the residue was dissolved in aqueous NaOH (5 ml, 10%), and the product was precipitated by the addition of dil. HCl. The solid product was filtered, washed with water, dried and crystallized from ethanol (cf. Scheme 3, Table I).

Synthesis of 3,4-Dibenzoylamino-2,5-dicarbethoxythieno(2,3-b) thiophene (16)

A solution of compound **1** (0.01 mol) in pyridine (10 ml) was treated with benzoyl chloride (0.02 mol). The reaction mixture was stirred for 10 h and then poured into a mixture of ice cold- water (100 ml) and HCl (10 ml). The separated product was collected by filtration, washed with water, dried, and crystallized from benzene (cf. Scheme 3, Table I).

Synthesis of 3,4-Dibenzoylaminothieno(2,3-b)thiophene-2,5-dicarboxamide (17)

To a solution of compound **16** (0.01 mol) in ethanol (20 ml) was added ammonia solution (10 ml, 50%). The reaction mixture was heated with stirring for 4 h at 40°C, whereby a white precipitate was formed. The precipitant was filtered, washed with water, dried, and crystallized from dioxan (cf. Scheme 3, Table I).

Synthesis of Compounds 15, 18 and 19

A mixture of compound **1, 3, 16** or **17** (0.01 mol) and Lawesson's reagent (0.02 mol) in dry toluene (40 ml) was refluxed for **12** h until no more of the reactants could be detected (*TLC*). The solvent was evaporated to dryness under reduced pressure, and the residue was triturated with ethanol. The separated solid was filtered and crystallized from ethanol (cf. Schemes 3 and 4, Table I).

Reaction of Compounds 16 and 17 with P_2S_5

A mixture of compound **16** or **17** (0.04 mol) and phosphorous pentasulphide (0.09 mol) in dry pyridine (20 ml) was refluxed for 20 h until no more of the reactants could be detected (*TLC*). The reaction mixture was filtered off on hot. The precipitant was boiled with water (100 ml), whereupon a solid product was formed, filtered, dried and crystallized from benzene to give compound **20**. The filtrate was evaporated under reduced pressure to dryness, and the residue was triturated with ethanol to give compounds **20** and **21**, respectively (cf. Scheme 4, Table I).

Conversion of Compound 20 to Compound 18

A suspension of compound **20** (0.01 mol) in pyridine (10 ml) was refluxed for 5 h. The reaction mixture was cooled and poured into a mixture of ice-water (100 ml) and HCl (10 ml). The product was collected by filtration, washed with water, dried, and crystallized from toluene (cf. Scheme 4, Table I).

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