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Synthesis of novel fused thienodiazaphosphorine derivatives from 2-amino-3-cyanothiophenes and Lawesson's reagent

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Synthesis of novel fused thienodiazaphosphorine derivatives from 2-amino-3-cyanothiophenes and Lawesson's reagent

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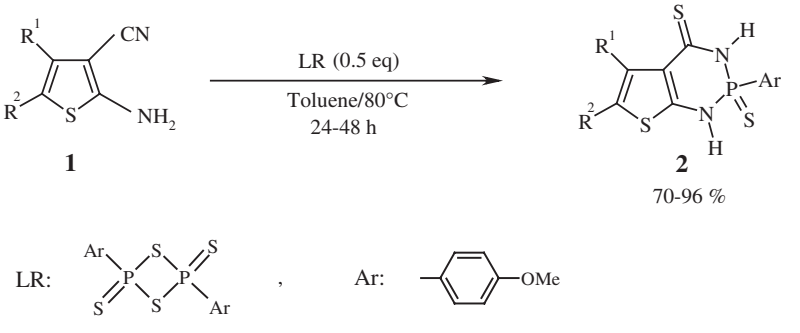
In a simple one-pot procedure, treatment of 2-amino-3-cyanothiophenes with Lawesson's reagent (LR) leads to the new thieno[2,3-*d*][1,3,2]diazaphosphorine-6-thione-2-sulfides in good to excellent yields. A possible reaction mechanism, involving a Dimroth-type rearrangement, is proposed. The structure of obtained products was confirmed by NMR (^{1}H, ^{31}P, and ^{13}C) and IR spectroscopies and by mass spectrometry.



Keywords: thiophenes; diazaphosphorines; Lawesson's reagent; thienodiazaphosphorines; fused heterocycles; Dimroth rearrangement

1. Introduction

Thiophene derivatives and their fused heterocyclic ring systems are associated with a wide range of biological properties including antimicrobial (*1*,*2*), antiviral (*3*), analgesic (*4*), anti-inflammatory (*1*,*5*), and anticancer (*6*,*7*) activities.

On the other hand, compounds bearing 1,3,2-diazaphosphorine ring are known to exhibit antimicrobial (*8*), antiviral (*9*), and antitumor (*10*,*11*) activities.

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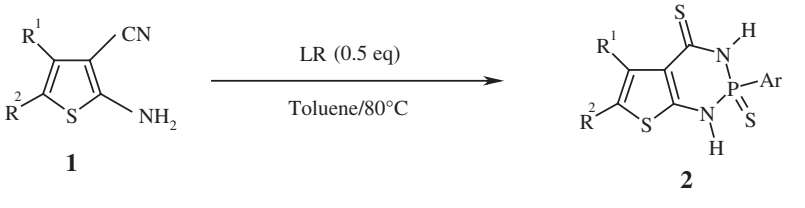
In view of the above, and in continuation of our research program concerning the preparation of new heterocycles with possible biological properties (*12–18*), we report here the synthesis of the new thieno[2,3-*d*][1,3,2]diazaphosphorine-6-thione-2-sulfides which might show enhanced biological activity due to the presence of fused thiophene and diazaphosphorine ring systems.

These compounds were obtained, in a simple one-pot protocol, from easily made 2-amino-3-cyanothiophenes and commercially available Lawesson's reagent (LR), as starting materials.

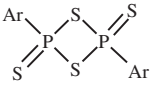
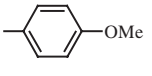
2. Results and discussion

The starting 2-amino-3-cyanothiophenes **1** were easily prepared according to the reported Gewald synthetic procedure (*19*). It was found that reaction of compounds **1** with a stoichiometric amount of LR (0.5 equiv.), performed in toluene, at 80°C, for 24–48 h, led to thieno[2,3-*d*][1,3,2]diazaphosphorine-6-thione-2-sulfides **2** in 70–96% yield (Scheme 1). It is important to note here that the use of excess LR (1 equiv. instead of 0.5) did not enhance the reaction rate but made purification of the products more difficult.

A plausible mechanism for the formation of compounds **2** is depicted in Scheme 2. The transformation is believed to proceed via a nucleophilic attack of the NH_2 group on LR giving rise



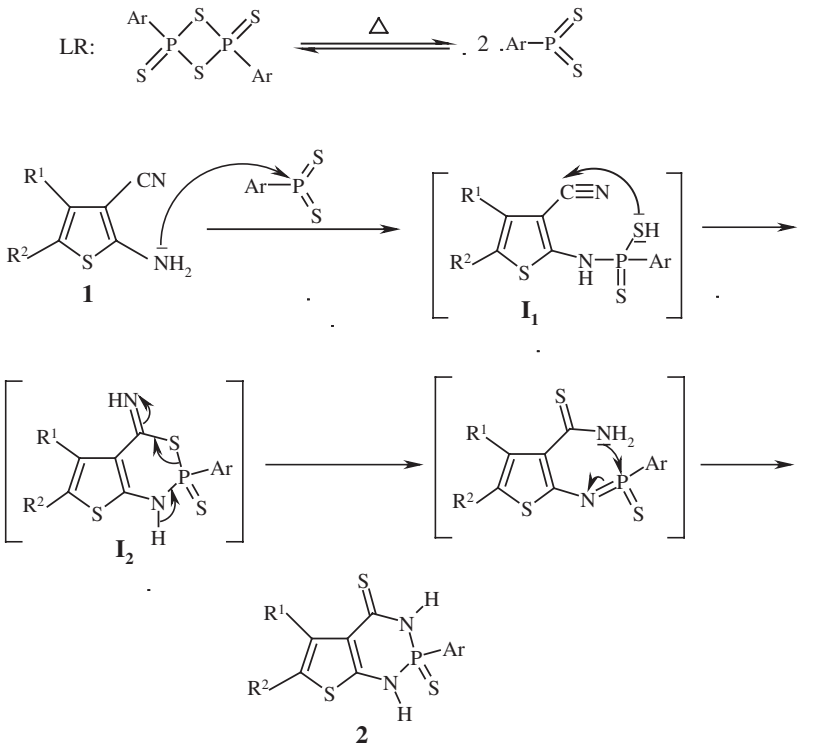
1 **2**

LR:  , Ar: 

	2a	**2b**	**2c**	**2d**	**2e**
R^1	$(CH_2)_4$	$(CH_2)_3$	Ph	CH_3	Ph-CH_2
R^2			H	CH_3	Ph
Yield(%)	96	70	73	87	95

	2f	**2g**	**2h**	**2i**
R^1	CH_3	CH_3	CH_3	CH_3
R^2	$(CH_3)_2CH$	H	Ph-CH_2	Ph
Yield(%)	85	76	92	82

Scheme 1. Synthesis of thieno[2,3-*d*][1,3,2]diazaphosphorine-6-thione-2-sulfides (**2**).



Scheme 2. Reaction mechanism for the synthesis of compounds **2**.

to the intermediate $\mathbf{I}_1$. A subsequent intramolecular cyclization, through the nucleophilic attack of the sulfur atom at the nitrile group, leads to the cyclic intermediate $\mathbf{I}_2$. The latter undergoes a Dimroth-typerearrangement (*20*) that consists in a translocation of endocyclic sulfur and exocyclic nitrogen atoms through a ring-opening–ring-closure sequence, leading to the final products **2**.

This proposed mechanism is consistent with some literature data that report similar rearrangements in reactions of LR with some nitrile derivatives (*21–23*).

The stability of the product is the driving force of the rearrangement; indeed, theoretical RHF/6-31G calculation performed with the Gaussian 03 program showed a stabilization of about 15 kcal/mol in favor of the rearranged product **2**.

The structures of thienodiazaphosphorines **2** were established through IR, NMR (^{1}H, ^{31}P, and ^{13}C) and mass spectral data. The IR spectra of compounds **2** showed a characteristic absorption band around 1100 cm^{-1} attributable to the C=S group. We observed, on the other hand, a broad band in the region 3070–3450 cm^{-1} corresponding to the N−H vibrators. Another band ascribable to the P=S group is present at nearly 700 cm^{-1}.

The ^{1}H NMR spectra showed, in particular, the presence of broad singlets at 10.0–10.5 ppm, corresponding to the protons of NH groups. We observed also a singlet at 3.8 ppm attributable to the OCH_3 protons.

Other evidence for the structure of compound **2** is provided by ^{13}C NMR. Indeed, we find the signals of all carbons and particularly those corresponding to the heterocyclic ring. Of particular interest is the C=S carbon which resonates as a doublet at 185–207 ppm. Such a doublet is characteristic of the coupling with phosphorus with a $^2J_{CP}$ coupling constant of about 9 Hz.

3. Experimental

^{1}H, ^{31}P and ^{13}C NMR spectra were recorded with $CDCl_3$ as the solvent, on a Bruker-300 spectrometer. The chemical shifts are reported in ppm relative to TMS (internal reference) for ^{1}H and ^{13}C NMR and relative to 85% H_3PO_4 (external reference) for ^{31}P NMR. The couplingconstants are reported in Hz. For the ^{1}H NMR, the multiplicities of signals are indicated by the following abbreviations: s: singlet, d: doublet, t: triplet, q: quartet, sept: septuplet, m: multiplet.

Mass spectra were determined on an Agilent 5975B spectrometer, under electronic impact (EI) conditions. IR spectra were recorded on a Perkin Elmer Paragon 1000 PC spectrometer. The progress of the reactions was monitored by TLC. Purification of products was performed by column chromatography using silica gel 60 (Fluka).

3.1. *Synthesis of 2-amino-3-cyanothiophenes (1)*

The starting 2-amino-3-cyanothiophenes **1** were prepared according to the reported procedure (*19*).

3.2. *Synthesis of thieno[2,3-d][1,3,2]diazaphosphorine-6-thione-2-sulfides (2)*

A mixture of 2-amino-3-cyanothiophene **1** (0.01 mol), LR (0.005 mol), and dry toluene (30 ml) was heated at 80°C with stirring for the reaction time (RT) (the reaction was monitored by TLC). The reaction mixture was then concentrated *in vacuo*. The residue obtained was chromatographed on a silica gel column using a mixture of ether and hexane 3:1 as the eluent.

2a: Light brown solid; m.p. = 236°C; r.t. = 24 h; ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 50.1 ppm; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.69–3.21 (m, 8H, cyclic H), 3.77 (s, 3H, CH_3–O), 6.88–7.81 (m, 4H, arom-H), 10.10 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 22.1 ($\underline{C}H_2-CH_2-C{=}C-S$), 22.2 ($\underline{C}H_2-CH_2-CH_2-C{=}C-S$), 24.4 ($\underline{C}H_2-C{=}C-S$), 28.7 ($\underline{C}H_2-(CH_2)_3-C{=}C-S$), 55.1 ($CH_3-O$), 113.4 (d, 16.6 Hz, $\underline{C}{=}C-P$), 118.9 (d, 4.5 Hz, $\underline{C}-C{=}C-P$), 122.9 ($CH_2-\underline{C}{=}C-S$), 126.2 (d, 132.1 Hz, $\underline{C}-P{=}S$), 132.9 (d, 14.3 Hz, $\underline{C}-C{=}S$), 133.4 ($CH_2-\underline{C}-S$), 149.8 (d, 2.3 Hz, $N-\underline{C}-S$), 162.6 (d, 3.0 Hz, $\underline{C}-O-CH_3$), 187.6 (d, 9.1 Hz, C=S); IR (KBr): $\nu_{P=S}$ = 702 cm^{-1}, $\nu_{C=S}$ = 1111 cm^{-1}, ν_{NH} =3076–3469 cm^{-1}; EI-HRMS: calculated for $C_{16}H_{17}N_2OPS_3$, 380.0241 (M^+); found: 380.0243.

2b: Light brown solid; m.p. = 159°C; r.t. = 24 h;^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 50.6 ppm; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.69–3.21 (m, 6H, cyclic H), 3.82 (s, 3H, CH_3-O), 6.77–7.73 (m, 4H, arom-H), 10.17 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 15.3 ($\underline{C}H_2-CH_2-C{=}C-S$), 21.4 ($\underline{C}H_2-C{=}C-S$), 29.7 ($\underline{C}H_2-(CH_2)_2-C{=}C-S$), 55.9 ($CH_3-O$), 113.7 (d, 16.3 Hz, $\underline{C}{=}C-P$), 118.7 (d, 4.7 Hz, $\underline{C}-C{=}C-P$), 125.3 ($CH_2-\underline{C}{=}C-S$), 126.7 (d, 113.2 Hz, $\underline{C}-P{=}S$), 129.2 (d, 14.1 Hz, $\underline{C}-C{=}S$), 137.9 ($CH_2-\underline{C}-S$), 151.0 (d, 3.0 Hz, $N-\underline{C}-S$), 161.5 (d, 3.0 Hz, $\underline{C}-O-CH_3$), 185.2 (d, 9.8 Hz, C=S); IR (KBr): $\nu_{P=S}$ =703 cm^{-1}, $\nu_{C=S}$ =1115 cm^{-1}, ν_{NH} =3072–3350 cm^{-1}; EI-HRMS: calculated for $C_{15}H_{19}N_2OPS_3$, 370.0397 (M^+); found: 370.0395.

2c: Light brown solid; m.p. = 136°C; r.t. = 48 h;^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 67.5 ppm; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.77 (s, 3H, CH_3-O), 6.80–8.29 (m, 10H, arom-H), 10.37 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 55.2 (CH_3-O), 113.5 (d, 15.0 Hz, $\underline{C}{=}C-P$), 122.9 (d, 5.9 Hz, $\underline{C}-C{=}C-P$), 124.9 ($Ph-\underline{C}{=}C-S$), 128.2 (d, 126.8 Hz, $\underline{C}-P{=}S$), 128.9 (d, 12.8 Hz, $\underline{C}-C{=}S$), 133.4 ($Ph-C{=}\underline{C}-S$), 146.0 (d, 2.3 Hz, $N-\underline{C}-S$), 162.2 (d, 3.0 Hz, $\underline{C}-O-CH_3$), 207.5 (d, 9.0 Hz, C=S); Phenyl carbons: 114.0, 127.8, 128.2, 130.6; IR (KBr): $\nu_{P=S}$ = 702 cm^{-1}, $\nu_{C=S}$ = 1111 cm^{-1}, ν_{NH} = 3195–3497 cm^{-1}; EI-HRMS: calculated for $C_{18}H_{15}N_2OPS_3$, 402.0084 (M^+); found: 402.0031.

2d: Light brown solid; m.p. = 174°C; r.t. = 48 h; ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 48.8 ppm;^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.21 (s, 3H, $CH_3-C-C-S$), 2.52 (s, 3H, CH_3-C-S), 3.82 (s, 3H, CH_3-O), 6.85–7.82 (m, 4H, arom-H), 10.23 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 12.6 ($\underline{C}H_3-C-C-S$), 16.4 ($\underline{C}H_3-C-S$), 55.2 (CH_3-O), 113.8 (d, 15.8 Hz, $\underline{C}$=C−P), 120.0 (d, 7.5 Hz, $\underline{C}-C=C-P$), 124.4 ($CH_3-\underline{C}=C-S$), 130.3 (d, 141.9 Hz, $\underline{C}-P=S$), 132.5 (d, 12.6 Hz, $\underline{C}-C=S$), 132.8 ($CH_3-\underline{C}-S$), 157.5 (d, 3.0 Hz, $N-\underline{C}-S$), 162.0 (d, 3.0 Hz, $\underline{C}-O-CH_3$), 206.9 (d, 9.1 Hz, C=S); IR (KBr): $\nu_{P=S}$ = 703 cm^{-1}, $\nu_{C=S}$ = 1111 cm^{-1}, ν_{NH} = 3154–3428 cm^1; EI-HRMS: calculated for $C_{14}H_{13}N_2OPS_3$, 351.9928 (M^+); found: 351.9926.

2e: Light brown solid; m.p. = 182°C; r.t. = 48 h; ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 48.7 ppm;^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.79 (s, 3H, CH_3-O), 4.46 (s, 2H, CH_2), 6.90–7.80 (m, 14H, arom-H), 10.07 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 33.9 (Ph−$\underline{C}H_2$), 55.4 (CH_3-O), 113.8 (d, 16.6 Hz, $\underline{C}$=C−P), 120.3 (d, 5.3 Hz, $\underline{C}-C=C-P$), 125.2 ($CH_2-\underline{C}=C-S$), 126.5 (d, 123.0 Hz, $\underline{C}-P=S$), 132.7 (d, 9.8 Hz, $\underline{C}-C=S$), 134.1 (Ph−$\underline{C}-S$), 152.1 (d, 3.0 Hz, $N-\underline{C}-S$), 162.9 (d, 3.0 Hz, $\underline{C}-O-CH_3$), 188.5 (d, 9.8 Hz, C=S); Phenyl carbons: 113.3, 127.8, 128.0, 128.4, 128.6, 128.7, 129.4, 132.8; IR (KBr): $\nu_{P=S}$ = 702 cm^{-1}, $\nu_{C=S}$ = 1111 cm^{-1}, ν_{NH} = 3043–3448 cm^{-1}; EI-HRMS: calculated for $C_{25}H_{21}N_2OPS_3$, 492.0554 (M^+); found: 492.0574.

2f: Light brown solid; m.p. = 122°C; r.t. = 24 h;^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 49.6 ppm; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.19 (d, 6H, $^3J_{H-H}$ = 6.0 Hz, $(C\underline{H}_3)_2CH$), 2.52 (s, 3H, $CH_3-C=C$), 2.72 (sept, 1H, $^3J_{H-H}$ = 6.0 Hz, $(CH_3)_2C\underline{H}$), 3.81 (s, 3H, CH_3-O), 6.17–7.82 (m, 4H, arom-H), 10.16 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 15.3 ($(\underline{C}H_3)_2CH$), 22.4 ($\underline{C}H_3-C=C$), 28.0 ($(CH_3)_2\underline{C}H$), 55.2 (CH_3-O), 110.4 (d, 15.8 Hz, $\underline{C}$=C−P), 114.1 (d, 4.5 Hz, $\underline{C}-C=C-P$), 124.3 ($CH_3-\underline{C}=C-S$), 123.2 (d, 113.2 Hz, $\underline{C}-P=S$), 132.9 (d, 15.1 Hz, $\underline{C}-C=S$), 133.1 ($CH_3-C=\underline{C}-S$), 151.4 (d, 3.0 Hz, $N-\underline{C}-S$), 162.8 (d, 3.0 Hz, $\underline{C}-O-CH_3$), 188.7 (d, 9.0 Hz, C=S); IR (KBr): $\nu_{P=S}$ = 702 cm^{-1}, $\nu_{C=S}$ = 1119 cm^{-1}, ν_{NH} = 3150–3321 cm^{-1}; EI-HRMS: calculated for $C_{16}H_{19}N_2OPS_3$, 382.0397 (M^+); found: 382.0404.

2g: Light brown solid; m.p. = 162°C; r.t. = 48 h; ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 50.4 ppm; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.31 (s, 3H, $CH_3-C=C$), 3.82 (s, 3H, CH_3-O), 6.75–7.78 (m, 5H, arom-H), 10.26 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 15.2 ($\underline{C}H_3-C=C$), 55.2 (CH_3-O), 114.1 (d, 16.6 Hz, $\underline{C}$=C−P), 120.4 (d, 5.2 Hz, $\underline{C}-C=C-P$), 125.3 ($CH_3-\underline{C}=C-S$), 125.4 (d, 132.1 Hz, $\underline{C}-P=S$), 133.1 (d, 15.1 Hz, $\underline{C}-C=S$), 138.6 ($CH_3-C=\underline{C}-S$), 151.1 (d, 2.3 Hz, $N-\underline{C}-S$), 162.3 (d, 3.2 Hz, $\underline{C}-O-CH_3$), 188.4 (d, 9.0 Hz, C=S); IR (KBr): $\nu_{P=S}$ = 702 cm^{-1}, $\nu_{C=S}$=cm^{-1}, ν_{NH} = 3071–3415 cm^{-1}; EI-HRMS: calculated for $C_{13}H_{13}N_2OPS_3$, 339.9928 (M^+); found: 339.9924.

2h: Light brown solid; m.p. = 141°C; r.t. = 24 h; ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 49.8 ppm; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.31 (s, 3H, $CH_3-C=C$), 2.55 (s, 2H, $C\underline{H}_2-Ph$), 3.77 (s, 3H, CH_3-O), 6.88–7.85 (m, 9H, arom-H), 10.35 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 17.0 ($\underline{C}H_3-C=C$), 33.0 (Ph−$\underline{C}H_2$), 55.4 (CH_3-O), 113.7 (d, 15.8 Hz, $\underline{C}$=C−P), 119.9 (d, 4.8 Hz, $\underline{C}-C=C-P$), 125.1 ($CH_3-\underline{C}=C-S$), 126.3 (d, 102.6 Hz, $\underline{C}-P=S$), 132.4 (d, 12.1 Hz, $\underline{C}-C=S$), 133.3 ($CH_2-\underline{C}-S$), 150.8 (d, 2.6 Hz, $N-\underline{C}-S$), 162.9 (d, 3.1 Hz, $\underline{C}-O-CH_3$), 188.4 (d, 9.0 Hz, C=S); Phenyl carbons:120.7, 125.3, 128.2, 128.9; IR (KBr): $\nu_{P=S}$ = 702 cm^{-1}; $\nu_{C=S}$ = 1111 cm^{-1}, ν_{NH} = 3080–3328 cm^{-1}; EI-HRMS: calculated for $C_{20}H_{19}N_2OPS_3$, 430.0397 (M^+); found: 430.0396.

2i: Light brown solid; m.p. = 102°C; r.t. = 24 h;^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 49.6 ppm; ^{1}H NMR (300 MHz, $CDCl_3$): δ=2.37 (s, 3H, $CH_3-C=C$), 3.73 (s, 3H, CH_3-O), 6.78–7.88 (m, 9H, arom-H), 10.30 (broad s, 2H, 2NH); ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (J_{CP}): 15.2 ($\underline{C}H_3-C=C$), 55.2 (CH_3-O), 113.6 (d, 16.6 Hz, $\underline{C}$=C−P), 122.2 (d,

4.6 Hz, C−C=C−P), 125.3 (CH_3−C=C−S), 127.1 (d, 119.2 Hz, C−P=S), 133.0 (d, 12.1 Hz, C−C=S), 133.5 (CH_3−C=C−S), 152.5 (d, 3.0 Hz, N−C−S), 162.4 (d, 3.1 Hz, C−O−CH_3), 188.1 (d, 9.4 Hz, C=S); Phenyl carbons: 128.2, 128.5, 129.0, 129.4; IR (KBr): $\nu_{P=S} = 702\,cm^{-1}$, $\nu_{C=S} = 1111\,cm^{-1}$, $\nu_{NH} = 3072–3357\,cm^{-1}$; EI-HRMS: calculated for $C_{19}H_{17}N_2OPS_3$, 416.0241 (M^+); found: 416.0244.

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