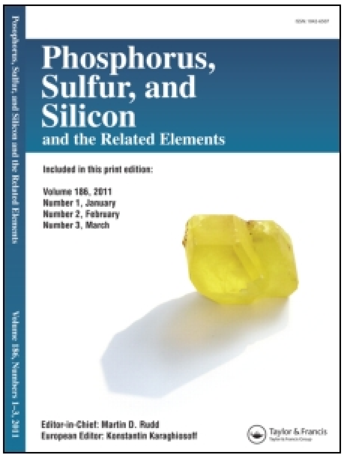

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Further Studies on the Reaction of Stable Phosphorus Ylides with Cyclic *cis*-Disulfides

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To cite this Article Abdou, Wafaa M. and Hennawy, Ibtisam T.(1994) 'Further Studies on the Reaction of Stable Phosphorus Ylides with Cyclic *cis*-Disulfides', Phosphorus, Sulfur, and Silicon and the Related Elements, 89: 1, 105 — 112
To link to this Article: DOI: 10.1080/10426509408020439
URL: http://dx.doi.org/10.1080/10426509408020439

Phosphorus, Sulfur, and Silicon, 1994, Vol. 89, pp. 105–112
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FURTHER STUDIES ON THE REACTION OF STABLE PHOSPHORUS YLIDES WITH CYCLIC *cis*-DISULFIDES

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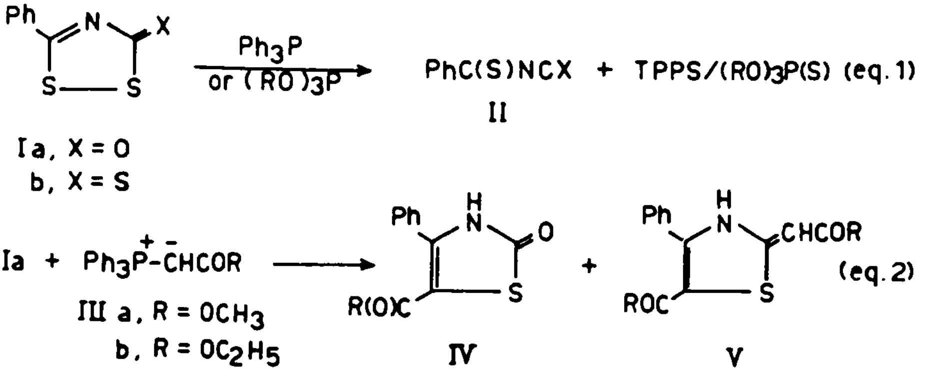
(Received November 24, 1993; in final form March 28, 1994)

The behavior of 5-phenyl-3H-1,2,4-dithiazole-3-thione (**Ib**) toward stable phosphorus ylides: methoxy- (**IIIa**), ethoxycarbonylmethylenetriphenylphosphorane (**IIIb**) and acetonylidenetriphenylphosphorane (**IIIc**) has been studied. The reactions take different paths leading to unusual products, depending only on the nature of the substituents of the ylides used. Structures of the new compounds were established on the basis of elemental analyses and spectroscopic studies. Some reaction mechanisms are proposed.

Key words: Heterocyclic sulfur compounds; *cis*-disulfides; stable phosphorus ylides.

INTRODUCTION

We have recently shown[1] that the reaction of 5-phenyl-3H-1,2,4-dithiazole-3-one, (**Ia**) with stable phosphonium ylides of type **III** reveals rather different reaction routes (Equation (2)) compared to its reaction paths with other III-phosphorus reagents such as triphenylphosphine or trialkyl phosphites[2] (Equation (1)). Since it has been reported[3,4] that the thiocarbonyl group shows an effect on the Wittig reaction courses different from those expected when compared with the carbonyl-analogues, we have extended our study on the behavior of phosphonium ylides of the same species **III** toward the thione-derivative **Ib**, i.e., 5-phenyl-3H-1,2,4-dithiazole-3-thione. This class of compounds is of considerable interest for both biological and chemical behavior studies. Owing to the presence of an asymmetric center at the —S—S— linkage, two additional centers of reactivity may be created.



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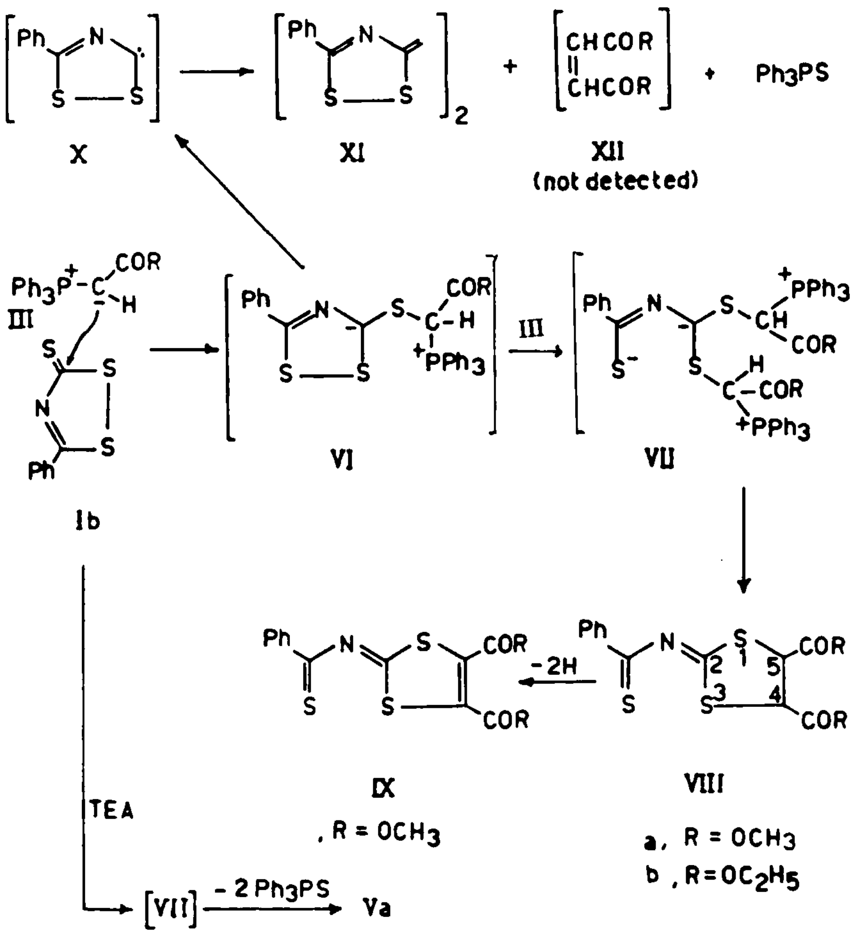
*Author to whom all correspondence should be addressed.

Increasing interest in various biologically active properties of heterocyclic sulfur compounds has prompted investigations of compounds containing this moiety in combination with groups bearing a greater or lesser decrease of electronegativity. A biological evaluation of products **XI** and **XIV** seemed to be of great interest, with regard to the general question, if the biological activity of the known active principle, such as **Ib**,[5-7] is decreased or increased upon its dimerization—after displacement reactions on the thiyl radicals—to **XI** or **XIV**.

RESULTS AND DISCUSSION

In the present systematic study, the reaction of *cis*-disulfide **Ib** with methoxycarbonylmethylenetriphenylphosphorane (**IIIa**), has been investigated. Contrary to the previous report,[1] no evidence of any significant reaction at room temperature, even after 48 h, is found. Addition of a solution of **Ib** in dry tetrahydrofuran (THF) to an excess of (> two molar equivalents) of **IIIa** in the same solvent and an refluxing the reaction mixture, the reactants (TLC) completely disappeared after 18 h. Chromatographic separation of the product mixture afforded two crystalline samples **VIIIa** and **XI**. Triphenylphosphine and triphenylphosphine sulfide also were isolated and identified (Scheme I). Compounds **VIIIa** and **XI** are obtained, equally, irrespective of whether one or two molar equivalents of the phosphonium ylide was used. The identification of the two structures **VIIIa** and **XI** was confirmed by combustion analyses, mass and NMR spectroscopy.

Compound **VIIIa**, the major product, (~52%) was obtained as yellow crystals and formulated as 2-thioacylimino-1,3-dithiole. Its IR indicates the absence of —S—S— linkage, since the sharp and strong band assigned[8] to the *cis*-disulfide stretching vibration at 1255 cm^{-1} in the IR spectrum of **Ia** and **Ib** was absent in the IR spectrum of **VIIIa**. It showed bands at 1703 (C═O, ester), 1605 (C═C, aromatic) as well as strong bands at 1485 and 1425 cm^{-1} which have been assigned to N—C═S and —N═C—S—, respectively. Identification of these latter two moieties are difficult and uncertain, since compounds in which the C═S group is attached to a nitrogen atom show an absorption band in the general C═S stretching region. In addition, several other bands in the broad region of 1563–700 cm^{-1} can be attributed to vibrations involving an interaction between the C═S stretching and C—N stretching.[9] Indeed, structure **VIIIa** could be verified by NMR analysis. In its ^{1}H NMR spectrum, signals appeared at δ 3.93 s, 6H (2 OCH_3). Exhibition of one signal for the two asymmetric methyl groups indicated their equivalency which is probably merely fortuitous.[8] Protons of the established dithiole ring appeared as two doublets each with J_{HH} = 4 Hz at 3.25 1H, (C_4—H) and 4.08 IH, (C_5—H).[9] The spectrum also revealed a multiplet pattern due to the aromatic protons (5H) in the region 7.35–7.85 ppm. In its ^{13}C NMR[10] spectrum, signals were observed at δ 47.21 and 49.8 (2C—C═OR), 56.73 and 58.23 (2 OCH_3), 155.36 (C═N), 168.7 and 169.5 [2C═O] and at δ 206.3 ppm (C═S). Structure **VIIIa** was unambiguously established by its conversion to the 2-thioacylimino-1,3-dithiole derivative **IX** using N—bromosuccinimide. Compound **IX** which shows identical mixed melting points and comparable spectral data with that reported by previous workers,[8] had resulted from the reaction of **Ib** with dimethylacetylenedicarboxylate.



SCHEME I

The second component was also produced in considerable quantities (~20%) and formulated as a symmetric dimer of 5-phenyl-3H-1,2,4-dithiazole (**XI**) which appeared to include —S—S— linkage in its IR spectrum at 1270 cm^{-1}. It also showed a stretching band at 1625 $(>C=C<)$ cm^{-1}. It was clear from the corresponding ^1H NMR, that the aromatic ring was intact and that this component does not contain any carboxylate esters. In addition, its ^{13}C NMR spectrum was entirely consistent with the proposed structure (cf. experimental).

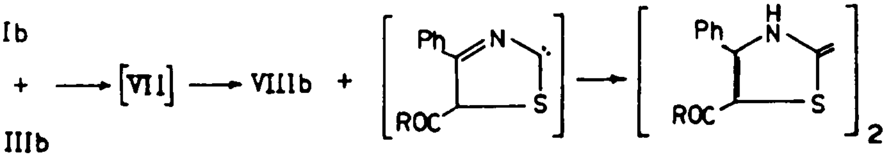
It is worth noting that products parallel to **IVa** and **Va** which have been previously[1] isolated from the reaction of **Ia** with **IIIa** could not be isolated from the present reaction (**Ib** + **IIIa**). Efforts were therefore made to obtain one or both analogous products. Thus, the reaction of **Ib** with **IIIa** was performed using the same experimental conditions, but in the presence of triethylamine, since it has been reported that the strained sulfur-sulfur bond could be readily opened by weak bases such as triethylamine.[11] As expected, the major product (>60%) was the substituted thiazole derivative **Va** which is consistent with melting points and spectral data given in the previous[1] study. Compound **VIIIa** also was isolated in a moderate yield (~16%) while neither compound **IVa** nor **XI** were obtained from this reaction.

The mechanisms proposed in Scheme I can account for the formation of products in the reactions studied. Initial nucleophilic attack by the carbanion center in the ylide **IIIa** on the reactive thiocarbonyl-carbon gives the intermediate **VI** which reacts further in two ways affording finally compounds **VIIIa** and **XI**. Addition of a second phosphorane molecule at the thiyl radical affords another intermediate **VII**. **VIIIa** which probably results from intramolecular-1,3-dipolar cycloaddition of the crowded intermediate **VII** with expulsion of triphenylphosphine. However, the preferable addition reaction of these phosphonium ylides rather than thiocarbonyl olefination or at the —S—S— linkage can be rationalized on the premise that thiones (C=S) are considered to be "superdipolarophiles." Their preeminence was shown by kinetic measurements of their cycloadditions with thiobenzophenone S-methide and diphenyldiazomethane as 1,3 dipoles.[3] This behavior is parallel to the reaction course of similar phosphonium ylides with thiourea[11] or substituted thiourea,[12] previously reported.

On the other hand, even though the mechanism of generating the dimeric product **XI** is not clear, it could be interpreted on the premise that the phosphonium ylide **IIIa** is acting as a Lewis base,[13] catalizing the decomposition of presumably formed intermediate **VI** leading to the formation of the symmetric dimer **XI** via the carbene intermediate **X**[14] with elimination of triphenylphosphine sulfide and the appropriate symmetrical disubstituted ethylene **XII** (not detected). Albeit the basicity of these compounds are less than other phosphorus reagents like triphenylphosphine or trialkylphosphites,[15] there are instances where they are incompatible with sensitive substances.[16,17]

Formation of **Va** in the second reaction arises because of the enhanced ability of —S—S— linkage to be disrupted and extrusion of sulfur due to the effect of triethylamine.[4,15]

We have also investigated the reaction of **Ib** with ethoxycarbonylmethylenetriphenylphosphine (**IIIb**) using the same reaction conditions. Unexpectedly, the reaction was found to proceed by a very different route than that observed for the methoxy analogue **IIIa**, leading to the formation of equimolar quantities of two substances **VIIIb** and **XIVa** (Schemes I and II).



XIII

XIV

a, $R = OC_2H_5$
b, $R = CH_3$

SCHEME II

As expected, the first product (~30%) was identified as 2-thioacylimino-1,3-dithiole derivative (**VIIIb**). This structure is consistent with spectroscopic data available (cf. experimental) and on the basis of comparable arguments with **VIIIa**.

Compound **XIVa** was chromatographically isolated as yellow crystals in ~26% yield. The structure seemed possible for the reaction product, IR data confirmed the absence of the —S—S— linkage and the thiocarbonyl group. It also showed the presence of carbonyl esters at 1725 cm^{-1} and a stretching band at 1622 cm^{-1} attributed to the C═C linkage. The NH stretching vibration band appeared at 3250 cm^{-1}. Its ^1H NMR spectrum revealed that the aromatic ring was intact. It also showed one methylene triplet at 0.9 ppm and a quartet at 3.52 ppm for the two ethoxy groups, the exchangeable (D_2O) protons (NH) appeared as a broad signal at 13.02 ppm. The mass spectrum of compound **XIV** gave a prominent ion peak at m/z 466 (M^+, 11%) consistent with the suggested structure.

Compound **XIVa**, presumably arose from the intermediate **VII**. Expulsion of two moles of triphenylphosphine sulfide led to the formation of **XIVa** via the carbene intermediate **XIII**.

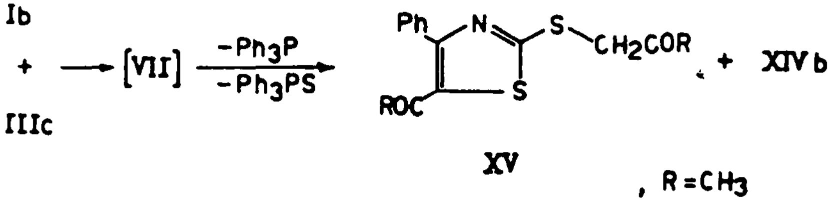
When **Ib** was subjected to reaction with acetonylidenetriphenylphosphorane (**IIIc**, R═CH_3) and subsequently, the product mixture was chromatographed, besides triphenylphosphine and triphenylphosphine sulfide, two crystalline substances were isolated and assigned **XIVb** and **XV** (Schemes I and III). Their product ratio (44:18).

The first component, the major product, was assigned the monosulfide **XV** since it shows elemental analysis, and molecular weight consistent with the molecular formula $C_{14}H_{13}NO_2S_2$. Its IR spectrum exhibited strong absorptions at 1725 and 1715 (2C═O, acetyl) and at 1500 (N═C—S) cm^{-1}. Structure **XV** was indicated by nuclear magnetic resonance. Its ^1H NMR spectrum revealed the presence of signals at δ 1.85 and 1.93 2s, 6H (2 CH_3) and at δ 2.4 ppm s, 2H.(CH_2). Its ^{13}C.NMR gave signals at δ 18.3, 18.9 (2 CH_3), 35.3 (S—CH_2), 152.8, 155.3 [2 C═O] and at 178.05 (N═C—S).

A possible explanation for the course of the reaction of **Ib** with **IIIc** is shown in Schemes I and III.

The second adduct was assigned structure **XIVb** on the basis of comparable spectroscopic data agreement with **XIVa**.

In summary, from this and the previous work,[1] the results allow an interesting conclusion to be drawn. Considering the earlier report,[1] we have been able to isolate the thiazole derivative **V**, only in the presence of triethylamine. On the other hand, in our experiments, the formation of the mono-substituted product **IV** turned out to be irreproducable, whereby we have isolated the symmetric dimer **XI** via the precursor monosubstituted intermediate **VI**. As a consequence, we



SCHEME III

assume a rather different reaction course for **Ib** with **III**, leading primarily to the intermediate **VI** which is quite different from that proposed in the previous work for the reaction course of **Ia** with **III**. As stated in the earlier communication,[1] the initial nucleophilic attack by the carbanion center was at the —S—S— linkage. This seems in our opinion and on the basis of our experimental results unlikely in the case of the reaction of **Ib** with **III**, whereby an addition reaction occurs at the thiocarbonyl group.

It is also worthy to note that the MS analysis of the crude sample of the product mixture (**Ib** + **IIIb**) and (**Ib** + **IIIc**) have indicated the potential formation of the symmetric dimer **XI**, such as (M/z = 358), nevertheless, it could not be isolated under the above experimental conditions.

Data on the biological activity of the new compounds will be published elsewhere.

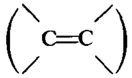
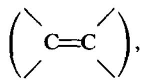
EXPERIMENTAL

All melting points are uncorrected. The IR spectra were recorded in KBr, using Perkin-Elmer Infracord Model 137 and Beckmann Infracord Model 4220. The ^1H and ^{13}C NMR spectra were recorded on Bruker Model WH 90 Spectrometer. The chemical shifts are recorded in δ ppm, using TMS as an internal reference. The mass spectra were performed at 70 eV on MS-50 Kratos (A.E.I.) Spectrometer.

Reaction of 5-Phenyl-3H-1,2-4-dithiazole-3-thione (**Ib**) *with Methoxycarbonylmethylenetriphenylphosphorane* (**IIIa**). To a solution of phosphonium ylide **IIIa**[18] (>0.02 mol) in 20 ml of dry tetrahydrofuran, was added a solution of dithiazole (**Ib**)[8] (2.1 g, 0.01 mol) in 20 ml of the same solvent and the reaction mixture was refluxed for 18 h. The product mixture was evaporated on silica gel under reduced pressure and applied to a silica gel column using petroleum ether containing increasing amounts of chloroform as eluent. Petroleum ether elution afforded colorless needles, mp. 80°C, identified as triphenylphosphine.

Petroleum ether-$CHCl_3$ (up to 8:2 v/v) eluted colorless crystals of triphenylphosphine sulfide (ca 72%) mp 162°C.[19]

Petroleum ether-$CHCl_3$ (up to 1:1 v/v) afforded yellow crystals of **VIIIa** (1.85 g, 52.3%), mp 174°C (ethyl alcohol). For $C_{14}H_{13}NO_4S_3$ (355.46) calculated: 47.30% C, 3.68% H, 3.94% N, 27.06% S; found: 47.62% C, 3.53% H, 3.75% N, 26.93% S. IR spectrum (KBr): 1703 (C=O, esters), 1605 (C=C, aromatic), 1485 (N—C=S), 1425 (N=C—S) cm^{-1}. ^1H NMR spectrum ($CDCl_3$): δ 3.93 s, 6H (2 OCH_3), 3.25 d, 1H, J_{HH} = 4 Hz (C_4—H), 4.08 d, 1H, J_{HH} = 4 Hz (C_5—H), 7.35–7.85 m, 5H (Ar—H). ^{13}C NMR spectrum ($CDCl_3$): δ 47.21, 49.84 (2C—C(O)R); 56.73, 58.23 (2 OCH_3); 156.36 (C=N); 168.7, 169.5 (2 C=O); 206.3 (C=S). Mass spectrum: m/z 355 (M^+, 25%).

Petroleum ether-$CHCl_3$ (up to 3:7 v/v) elution gave 0.6 g (20.3%) of pale yellow crystals of **XI** mp 150°C (acetone). For $C_{16}H_{10}N_2S_4$ (294.53) calculated: 65.24% C, 3.42% H, 9.51% N, 21.81% S; found: 65.06% C, 3.30% H, 9.38% N, 21.56% S. IR spectrum (KBr): 1625 , 1420 (N=C) cm^{-1}. ^{13}C NMR ($CDCl_3$): δ 137.35 , 158.5 (C=N). Mass spectrum: m/z 294 (M^+, 12%).

No reaction was observed, however, in a parallel experiment when the reactants (**Ib** + **IIIa**) were mixed at ambient temperature even after 48 h.

Conversion of **VIIIa** *into* **IX**. A solution of **VIIIa** (0.5 g) in dry dichloromethane (15 ml) was treated with N-bromosuccinimide (200 mg) and the reaction mixture was refluxed for 6 h. The reaction product was then filtered and the filtrate was triturated with n-hexane to give compound **IX** (0.42 g, 85%), mp 153.5°C (cyclohexane); lit.[8] mp 155°C. Mass spectrum: m/z 353 (M^+, 25%).

Reaction of **Ib** *with* **IIIa** *in the presence of triethylamine.* The procedure was the same as above experiment for 15 h refluxing and in the presence of triethylamine (1.5 ml).

Elution with petroleum ether yielded colorless needles mp 80°C, identified to be triphenylphosphine.

Elution with petroleum ether-chloroform (8:2 v/v) eluted colorless crystals of triphenylphosphine sulfide (~72%) mp 162°C.

Elution with n-hexane-acetone (1:1 v/v) yielded yellow substance (1.4 g, 59.5%), mp 143°C (acetonitrile), proved to be compound **Va**, previously reported,[1] (mixed melting points and comparative spectral data). Mass spectrum: m/z 235 (M^+, 15%).

Elution with n-hexane-acetone (4:6) afforded compound **VIIIa** (0.57 g, 16.2%), mixed melting points and comparative IR spectra.

Reaction of **Ib** *with* **IIIb**.[18] The procedure was the same as above experiment for 30 h refluxing.

Elution with petroleum ether yielded colorless needles mp 80°C, identified to be triphenylphosphine.

Elution with petroleum ether-$CHCl_3$ (8:2 v/v) afforded colorless crystals of triphenylphosphine sulfide (~75%) mp 162°C.[19]

Elution with petroleum ether-$CHCl_3$ (1:1 v/v) eluted yellow crystals (1.1 g, 30.5%) of **VIIIb** mp 165°C (ethyl alcohol). For $C_{16}H_{37}NO_4S_3$ (383.51) calculated: 50.11% C, 4.47% H, 3.65% N, 25.08% S; found: 50.33% C, 4.49% H; 3.54% N; 24.88% S. IR spectrum (KBr): 1715, 1710 (2 C=O, esters), 1475 (N—C=S), 1420 (N=C—S) cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$): δ 0.85, 1.3 2t, 6H (2 $C.CH_3$), 3.05 d, 1H, J_{HH} = 4 Hz (C_4—H), 3.85 d, 1H, J_{HH} = 4 Hz (C_5—H); 4.1, 4.4 2q, 4H (2 OCH_2), 7.37–7.85 m, 5H (Ar—H). Mass spectrum: m/z 383 (M^+, 33%).

Petroleum ether-$CHCl_3$ (3:7 v/v) elution yielded pale yellow substance (1.23 g, 26.5%) of **XIVa** mp 187°C (ethyl alcohol). For $C_{24}H_{22}N_2O_4S_2$ (466.578) calculated: 61.78% C, 4.75% H, 6.00% N, 13.74% S; found: 61.88% C, 4.81% H, 5.83% N, 13.63% S. IR spectrum (KBr): 3250 (NH), 1725 (C=O, esters), 1622 (C=C) cm^{-1}, ^{1}H NMR ($CDCl_3$): δ 0.9 t, 6H (2 $C.CH_3$), 3.52 q, 4H (2 OCH_2), 7.36–7.84 m, 10H (2 Ar—H), 13.02 s, 2H (2 NH). Mass spectrum: m/z 466 (M^+, 11%).

Reaction of **Ib** *with* **IIIc**.[20] The procedure was the same as above experiment for 36 h refluxing.

Elution with petroleum ether-$CHCl_3$ (8:2 v/v) yielded colorless crystals of triphenylphosphine sulfide (78%) mp 162°C.[19]

Elution with petroleum ether-$CHCl_3$ (1:1 v/v) eluted orange crystals of **XV** (1.3 g, 44%), mp 148°C (acetonitrile). For $C_{14}H_{13}NO_2S_2$ (291.39) calculated: 57.70% C, 4.49% H, 4.80% N, 22.00% S; found: 57.62% C, 4.45% H, 4.73% N, 21.90% S. IR spectrum (KBr): 1725, 1715 (2 C=O, acetyl), 1500 (N=C—S) cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$): δ 1.85, 1.93 2s, 6H ($2CH_3$), 2.4 s, 2H (CH_2), 7.25–7.74 m, 5H (Ar—H). ^{13}C.NMR spectrum ($CDCl_3$): δ 18.3, 18.9 ($2CH_3$), 35.3 (S—CH_2), 152.8, 155.3 (2 C=O), 178.05 (N=C—S). Mass spectrum: m/z 291 (M^+, 27%).

Elution with petroleum ether-$CHCl_3$ (3:7 v/v) yielded brown crystals of **XIV** b (0.73 g, 18%), mp. 160°C (chloroform). For $C_{22}H_{18}N_2O_2S_2$ (406.53): 65.00% C, 4.46% H, 6.88% N, 15.77% S, found: 65.23% C, 4.38% H, 6.80% N, 15.48% S. IR spectrum (KBr): 3375 (NH), 1705 (C=O, esters), 1615 (C=C) cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$): δ 1.65 s, 6H (2 CH_3), 7.25–7.83 m, 5H (Ar.H), 13.3 broad, 2H (2 NH). ^{13}C NMR spectrum ($CDCl_3$): δ 20.7 ($2CH_3$), 141.2, 144.2 ppm (2 C.C=O), 147.0 (C—C_6H_5), 150.4 (C=C). 162.8 (2 C=O). Mass spectrum: m/z 406 (M^+, 8%).

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