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# Studies on Organophosphorus Compounds: The Synthesis of [1,3,2]-Diazaphospholes and [1,3,2]-Oxaazaphospholes 

O. A. Omran<br>H. M. Moustafa<br>Chemistry Department, Sohag, Egypt<br>A number of spiro[cyclopentane (cyclohexane, and cycloheptane)-1,4'-perhydro[1,3,2]diazaphosphole] derivatives (3a-c, 5a-c, 11a-c, and 12a-c) and spiro-[cyclopentane (cyclohexane, and cycloheptane)-1,4'-perhydro[1,3,2]oxaazaphos-phole] derivatives ( $\mathbf{7 a - c}$ ) were prepared via an interaction of 2,4-bis-(4-methoxy-phenyl)-1,3,2,4-dithiaphosphetane-2,4-disulphide (1) with substances containing two functional groups.

Keywords 1-Phenylamino-1-cyanocycloalkanes; 2,4-bis-(4-methoxyphenyl)-1,3,2,4-dithiaphosphetane-2,4-disulphide

## INTRODUCTION

It is well known that 2,4-bis-(4-methoxyphenyl)-1,3,2,4-dithiaphos-phetane-2,4-disulphide (Lawesson's Reagent, [LR]) is a most effective and versatile thiation reagent for different cabonyl compounds. ${ }^{1-5}$ The nucleophiles attack LR at the phosphorus atom and form phosphorus heterocycles. ${ }^{6-9}$ In view of the latest development and also in continuation of our study of phosphorus hetero-cycles, ${ }^{10-13}$ In this article it was of interest to synthesize spiro[1,3,2]diazaphospholes and spiro[1,3,2]oxaazaphospholes from the reaction of LR with other classes of substrates with two functional groups.

## RESULTS AND DISCUSSION

The reaction of 1-phenylamino-1-cyanocyclopentane(cyclohexane, and cyclo-heptane $)^{14}(\mathbf{2 a - c})$ with LR (1) in boiling acetonitrile afforded $2^{\prime}$-(4-methoxy-phenyl)-4'-phenylspiro[cyclopentane (cyclohexane, and

[^0]cycloheptane)-1,4'-perhy-dro[1,3,2]diazaphosphole]-2',5'-disulfides ( $\mathbf{3 a}-\mathbf{c}$ ). The reaction pathway was assumed to proceed via a nucleophilic attack of the amino group on the phosphorus of LR followed by a P-SH addition to the nitrile group and subsequent rearrangement, ${ }^{16}$ which yielded compounds 3a-c (cf. Scheme 1). The IR spectra of compounds 3a-c showed the absence of bands corresponding to cyano groups while exhibiting the characteristic absorption band at 651, 642, and $659 \mathrm{~cm}^{-1}$ for $\mathrm{P}=\mathrm{S}$, respectively. ${ }^{1} \mathrm{H}$ NMR spectra of compounds 3a-c exhibited a singlet at 3.9 ppm corresponding to $\mathrm{OCH}_{3}$ (cf. Table I).


## SCHEME 1

The treatment of compounds 2a-c with sodium metal in ethanol afforded the corresponding amines $4 \mathbf{a}-\mathbf{c}$ in good yield. IR spectra of compounds 4a-c showed the absence of absorption bands corresponding to CN groups and the appearance of new bands corresponding to $\mathrm{NH}_{2}$ groups ( $3163-3412 \mathrm{~cm}^{-1}$ ). The ${ }^{1} \mathrm{H} \mathrm{NMR}\left(\mathrm{CDCl}_{3}\right)$ spectra of compounds $4 \mathbf{a - c}$ are in agreement with the proposed structure (cf. Table I). Compounds 4a-c were allowed to react with LR (1) in anhydrous benzene at $80^{\circ} \mathrm{C}$ to give $2^{\prime}$-(4-methoxyphenyl)-3'-phenylspiro[cyclopentane

| M.P. ${ }^{\circ} \mathrm{C}$ ) |  |  |  | Analytical data Cacd./found |  |  |  | $\operatorname{IR}\left(\mathrm{Cm}^{-1}\right)$ | ${ }^{1}$ HNMR $\partial$ (ppm) |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| no. | solvent | $\begin{gathered} \text { YleId } \\ (\%) \end{gathered}$ | (Mol. Wt.) | C | H | N | S |  |  |
| 3a | $\begin{gathered} 160 \\ \text { ethanol } \end{gathered}$ | 86 | $\underset{(388.48)}{\mathrm{C}_{19} \mathrm{H}_{21} \mathrm{~N}_{2} \mathrm{OPS}_{2}}$ | $\begin{aligned} & 58.74 \\ & 58.50 \end{aligned}$ | $\begin{aligned} & 5.45 \\ & 5.27 \end{aligned}$ | $\begin{aligned} & 7.21 \\ & 7.03 \end{aligned}$ | $\begin{aligned} & 16.50 \\ & 16.23 \end{aligned}$ | $\begin{aligned} & 3212(\mathrm{NH}), 1246 \\ & (\mathrm{C}-\mathrm{O}), 651(\mathrm{P}=\mathrm{S}) \end{aligned}$ | 11.2 (br, 1H, NH), 7.8-6.9 (m, 9H, arom.); 3.9 (s, $3 \mathrm{H}, \mathrm{OCH}_{3}$ ), 1.9-1.4 (m, 8 H , cyclic $\mathrm{CH}_{2}$ ) |
| 3b | $\begin{gathered} 170 \\ \text { ethanol } \end{gathered}$ | 83 | $\underset{(402.51)}{\mathrm{C}_{20} \mathrm{H}_{23} \mathrm{~N}_{2} \mathrm{OPS}_{2}}$ | $\begin{aligned} & 59.68 \\ & 59.30 \end{aligned}$ | $5.75$ | $\begin{aligned} & 6.95 \\ & 6.73 \end{aligned}$ | $\begin{aligned} & 15.93 \\ & 15.72 \end{aligned}$ | $\begin{aligned} & 3266(\mathrm{NH}), 1253 \\ & (\mathrm{C}-\mathrm{O}), 642(\mathrm{P}=\mathrm{S}) \end{aligned}$ | $\begin{aligned} & 12.9(\mathrm{br}, 1 \mathrm{H}, \mathrm{NH}), 7.8-6.7(\mathrm{~m}, 9 \mathrm{H}, \\ & \text { arom.); } 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 1.9-1.2(\mathrm{~m}, \\ & \left.10 \mathrm{H} \text {, cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 3c | $\begin{gathered} 191 \\ \text { ethanol } \end{gathered}$ | 79 | $\underset{(416.53)}{\mathrm{C}_{21} \mathrm{H}_{25} \mathrm{~N}_{2} \mathrm{OPS}_{2}}$ | $\begin{aligned} & 60.55 \\ & 60.21 \end{aligned}$ | $\begin{aligned} & 6.05 \\ & 587 \end{aligned}$ | $\begin{aligned} & 6.72 \\ & 6.58 \end{aligned}$ | $\begin{aligned} & 15.93 \\ & 15.73 \end{aligned}$ | $\begin{aligned} & 3259(\mathrm{NH}), 1239 \\ & (\mathrm{C}-\mathrm{O}), 659(\mathrm{P}=\mathrm{S}) \end{aligned}$ | 11.9 (br, 1H, NH), 7.8-7.0 (m, 9H, arom.); 3.9 (s, $3 \mathrm{H}, \mathrm{OCH}_{3}$ ), 1.9-1.2 (m, 12 H , cyclic $\mathrm{CH}_{2}$ ) |
| 4a | Liquid | 77 | $\begin{gathered} \mathrm{C}_{12} \mathrm{H}_{18} \mathrm{~N}_{2} \\ (190.29) \end{gathered}$ | $\begin{aligned} & 75.74 \\ & 75.27 \end{aligned}$ | $\begin{aligned} & 9.53 \\ & 9.34 \end{aligned}$ | $\begin{aligned} & 14.72 \\ & 14.39 \end{aligned}$ | - | $\begin{gathered} 3359,3261,3203 \\ \left(\mathrm{NH}_{2}+\mathrm{NH}\right) \end{gathered}$ | 11.1 (br, 1H, NH), 7.5-6.9 (m, 5H, arom.); 4.3 (br, $2 \mathrm{H}, \mathrm{NH}_{2}$ ); 2.8 ( $\mathrm{s}, 2 \mathrm{H}$, $\mathrm{CH}_{2}$ ), $1.9-1.2\left(\mathrm{~m}, 8 \mathrm{H}\right.$, cyclic $\mathrm{CH}_{2}$ ) |
| 4b | Liquid | 79 | $\begin{gathered} \mathrm{C}_{13} \mathrm{H}_{20} \mathrm{~N}_{2} \\ (204.31) \end{gathered}$ | $\begin{aligned} & 76.42 \\ & 76.01 \end{aligned}$ | $\begin{aligned} & 9.86 \\ & 9.61 \end{aligned}$ | $\begin{aligned} & 13.71 \\ & 13.49 \end{aligned}$ | - | $\begin{gathered} 3412,3336,3230 \\ \left(\mathrm{NH}_{2}+\mathrm{NH}\right) \end{gathered}$ | 13.0 (br, 1H, NH), 7.5-6.9 (m, 5H, arom.); 4.9 (br, $2 \mathrm{H}, \mathrm{NH}_{2}$ ); 2.8 ( $\mathrm{s}, 2 \mathrm{H}$, $\mathrm{CH}_{2}$ ), 1.9-1.1 (m, 10 H , cyclic $\mathrm{CH}_{2}$ ) |
| 4c | Liquid | 71 | $\begin{gathered} \mathrm{C}_{14} \mathrm{H}_{22} \mathrm{~N}_{2} \\ (218.34) \end{gathered}$ | $\begin{aligned} & 77.01 \\ & 76.60 \end{aligned}$ | $\begin{array}{r} 10.15 \\ 9.97 \end{array}$ | $\begin{aligned} & 12.82 \\ & 12.51 \end{aligned}$ | - | $\begin{gathered} 3389,3298,3163 \\ \left(\mathrm{NH}_{2}+\mathrm{NH}\right) \end{gathered}$ | 11.8 (br, 1H, NH), 7.5-6.9 (m, 5H, arom.); 4.4 (br, $2 \mathrm{H}, \mathrm{NH}_{2}$ ); 2.8 ( $\mathrm{s}, 2 \mathrm{H}$, $\mathrm{CH}_{2}$ ), $1.9-1.0\left(\mathrm{~m}, 12 \mathrm{H}\right.$, cyclic $\mathrm{CH}_{2}$ ) |
| 5a | $\begin{gathered} 216 \\ \text { ethanol } \end{gathered}$ | 61 | $\underset{(358.44)}{\mathrm{C}_{19} \mathrm{H}_{23} \mathrm{~N}_{2} \mathrm{OPS}}$ | $\begin{aligned} & 63.66 \\ & 63.31 \end{aligned}$ | $\begin{aligned} & 6.46 \\ & 6.29 \end{aligned}$ | $\begin{aligned} & 7.81 \\ & 7.57 \end{aligned}$ | $\begin{aligned} & 8.94 \\ & 8.74 \end{aligned}$ | 3286 (NH), 661 (P=S) | 13.0 (br, 1H, NH), 7.5-6.8 (m, 9 H , arom.); 3.9 (s, $3 \mathrm{H}, \mathrm{OCH}_{3}$ ), 2.9 ( $\mathrm{s}, 2 \mathrm{H}$, $\mathrm{CH}_{2}$ ), 1.9-1.3 (m, 8 H , cyclic $\mathrm{CH}_{2}$ ) |
| 5b | $\begin{gathered} 233 \\ \text { ethanol } \end{gathered}$ | 59 | $\mathrm{C}_{20} \mathrm{H}_{25} \mathrm{~N}_{2} \mathrm{OPS}$ <br> (372.46) | $\begin{aligned} & 64.49 \\ & 64.07 \end{aligned}$ | $\begin{aligned} & 6.76 \\ & 6.61 \end{aligned}$ | $\begin{aligned} & 7.52 \\ & 7.33 \end{aligned}$ | $\begin{aligned} & 8.60 \\ & 8.43 \end{aligned}$ | $\begin{gathered} 3299(\mathrm{NH}), 703 \\ (\mathrm{P}=\mathrm{S}) \end{gathered}$ | 10.6 (br, 1H, NH), 7.5-6.8 (m, 9 H , arom.); 3.9 (s, $3 \mathrm{H}, \mathrm{OCH}_{3}$ ), 2.9 (s, 2 H , $\mathrm{CH}_{2}$ ), 1.9-1.2 (m, 10 H , cyclic $\mathrm{CH}_{2}$ ) |
| 5c | $\begin{gathered} 240 \\ \text { ethanol } \end{gathered}$ | 68 | $\begin{gathered} \mathrm{C}_{21} \mathrm{H}_{27} \mathrm{~N}_{2} \mathrm{OPS} \\ (386.49) \end{gathered}$ | $\begin{aligned} & 65.26 \\ & 64.89 \end{aligned}$ | $\begin{aligned} & 7.04 \\ & 6.90 \end{aligned}$ | $\begin{aligned} & 7.24 \\ & 7.05 \end{aligned}$ | $\begin{aligned} & 8.29 \\ & 8.08 \end{aligned}$ | 3271 (NH), 655 (P=S) | $\begin{aligned} & 10.6(\mathrm{br}, 1 \mathrm{H}, \mathrm{NH}) ; 7.5-6.9(\mathrm{~m}, 9 \mathrm{H}, \\ & \text { arom.), } 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 2.9(\mathrm{~s}, 2 \mathrm{H}, \\ & \left.\mathrm{CH}_{2}\right), 1.9-1.1\left(\mathrm{~m}, 12 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \\ & \text { (Continued on next page) } \end{aligned}$ |


| Compound No. | M.P. $\left({ }^{\circ} \mathrm{C}\right)$ <br> Cryst. <br> Solvent | Yield <br> (\%) | Mol. Form. (Mol. Wt.) | Analytical Data Cacd./Found |  |  |  | $\operatorname{IR}\left(\mathrm{Cm}^{-1}\right)$ | ${ }^{1} \mathrm{HNMRR} \partial$ (ppm) |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  | C | H | N | S |  |  |
| $6 \mathbf{}$ | Liquid | 65 | $\begin{gathered} \mathrm{C}_{12} \mathrm{H}_{17} \mathrm{NO} \\ (191.27) \end{gathered}$ | $\begin{aligned} & 75.35 \\ & 74.94 \end{aligned}$ | $\begin{aligned} & 8.95 \\ & 8.77 \end{aligned}$ | $\begin{aligned} & 7.32 \\ & 7.17 \end{aligned}$ | - | $\begin{aligned} & 3412(\mathrm{OH}), 3223 \\ & (\mathrm{NH}) \end{aligned}$ | $\begin{gathered} 10.9(\mathrm{br}, \mathrm{H}, \mathrm{NH}) ; 7.4-6.8(\mathrm{~m}, 5 \mathrm{H} \text {, arom.), } \\ 4.0(\mathrm{~s}, 1 \mathrm{H}, \mathrm{OH}), 3.1\left(\mathrm{~s}, 2 \mathrm{H}, \mathrm{CH}_{2}\right) \\ 1.9-1.2\left(\mathrm{~m}, 8 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{gathered}$ |
| $7 \mathbf{}$ | 163 <br> ethanol | 61 | $\underset{(389.47)}{\mathrm{C}_{19} \mathrm{H}_{20} \mathrm{NO}_{2} \mathrm{PS}}$ | $\begin{aligned} & 58.59 \\ & 58.21 \end{aligned}$ | $\begin{aligned} & 4.91 \\ & 4.77 \end{aligned}$ | $\begin{aligned} & 3.59 \\ & 3.48 \end{aligned}$ | $\begin{aligned} & 16.46 \\ & 16.25 \end{aligned}$ | $\begin{aligned} & 1244(\mathrm{C}-\mathrm{O}), 661 \\ & (\mathrm{P}=\mathrm{S}) \end{aligned}$ | $\begin{aligned} & 7.6-7.0(\mathrm{~m}, 9 \mathrm{H}, \text { arom. }), 4.1(\mathrm{~s}, 2 \mathrm{H}, \\ & \left.\mathrm{OCH}_{2}\right), 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 1.9-1.3(\mathrm{~m}, \\ & \left.8 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 7b | 198 <br> ethanol | 59 | $\underset{(403.49)}{\mathrm{C}_{20} \mathrm{H}_{22} \mathrm{NO}_{2} \mathrm{PS}}$ | $\begin{aligned} & 59.53 \\ & 59.17 \end{aligned}$ | $\begin{aligned} & 5.49 \\ & 5.27 \end{aligned}$ | $\begin{aligned} & 3.47 \\ & 3.38 \end{aligned}$ | $\begin{aligned} & 15.89 \\ & 15.69 \end{aligned}$ | $\begin{aligned} & 1233(\mathrm{C}-\mathrm{O}, 669 \\ & (\mathrm{P}=\mathrm{S}) \end{aligned}$ | $\begin{aligned} & \text { 7.6-7.0 }(\mathrm{m}, 9 \mathrm{H} \text {, arom.), } 4.2(\mathrm{~s}, 2 \mathrm{H} \\ & \left.\mathrm{OCH}_{2}\right), 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 1.9-1.2(\mathrm{~m} \text {, } \\ & \left.10 \mathrm{H} \text {, cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 7c | $\begin{aligned} & 186 \\ & \text { ethanol } \end{aligned}$ | 68 | $\underset{(417.52)}{\mathrm{C}_{21} \mathrm{H}_{24} \mathrm{NO}_{2} \mathrm{PS}}$ | $\begin{aligned} & 60.41 \\ & 60.05 \end{aligned}$ | $\begin{aligned} & 5.79 \\ & 5.61 \end{aligned}$ | $\begin{aligned} & 3.35 \\ & 3.26 \end{aligned}$ | $\begin{aligned} & 15.35 \\ & 15.16 \end{aligned}$ | $\begin{aligned} & 1249(\mathrm{C}-\mathrm{O}), 655 \\ & (\mathrm{P}=\mathrm{S}) \end{aligned}$ | ```7.6-7.0 (m, 9H, arom.), 4.2 (s, 2H, OCH2}),3.9(\textrm{s},3\textrm{H},\mp@subsup{\textrm{OCH}}{3}{}),1.9-1.2(m 12 H, cyclic CH2)``` |
| $8 \mathbf{a}$ | 199 <br> ethanol | 65 | $\begin{gathered} \mathrm{C}_{19} \mathrm{H}_{23} \mathrm{~N}_{3} \mathrm{O} \\ (309.41) \end{gathered}$ | $\begin{aligned} & 73.75 \\ & 73.29 \end{aligned}$ | $\begin{aligned} & 7.49 \\ & 7.33 \end{aligned}$ | $\begin{aligned} & 13.58 \\ & 13.30 \end{aligned}$ | - | $\begin{aligned} & 3276,3209,3148 \\ & (3 \mathrm{NH}), 1649(\mathrm{C}=\mathrm{O}) \end{aligned}$ | $11.2,10.8,10.5(\mathrm{br}, 3 \mathrm{H}, 3 \mathrm{NH}), 7.3-6.7$ (m, 10 H , arom.) $3.3\left(\mathrm{~s}, 2 \mathrm{H}, \mathrm{CH}_{2}\right)$, 1.9-1.2 (m, 12 H , cyclic $\mathrm{CH}_{2}$ ) |
| 8b | $\begin{gathered} 186 \\ \text { dioxan } \end{gathered}$ | 60 | $\begin{gathered} \mathrm{C}_{20} \mathrm{H}_{25} \mathrm{~N}_{3} \mathrm{O} \\ (323.44) \end{gathered}$ | $\begin{aligned} & 74.27 \\ & 73.89 \end{aligned}$ | $\begin{aligned} & 7.79 \\ & 7.63 \end{aligned}$ | $\begin{aligned} & 12.99 \\ & 12.80 \end{aligned}$ | - | $\begin{aligned} & 3295,3200,3147 \\ & (3 \mathrm{NH}), 1644(\mathrm{C}=\mathrm{O}) \end{aligned}$ | $\begin{aligned} & 11.9,10.78,10.1(\mathrm{br}, 3 \mathrm{H}, 3 \mathrm{NH}), 7.3-6.7 \\ & \left(\mathrm{~m}, 10 \mathrm{H}, \text { arom.) } 3.3\left(\mathrm{~s}, 2 \mathrm{H}, \mathrm{CH}_{2}\right)\right. \\ & 1.9-1.1\left(\mathrm{~m}, 10 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 8c | $\begin{gathered} 173 \\ \text { ethanol } \end{gathered}$ | 67 | $\begin{gathered} \mathrm{C}_{21} \mathrm{H}_{27} \mathrm{~N}_{3} \mathrm{O} \\ (337.46) \end{gathered}$ | $\begin{aligned} & 74.74 \\ & 74.37 \end{aligned}$ | $\begin{aligned} & 8.06 \\ & 7.96 \end{aligned}$ | $\begin{aligned} & 12.45 \\ & 12.21 \end{aligned}$ | - | $\begin{aligned} & 3293,3222,3129 \\ & \quad(3 \mathrm{NH}), 1650(\mathrm{C}=\mathrm{O}) \end{aligned}$ | $12.7,10.8,10.5(\mathrm{br}, 3 \mathrm{H}, 3 \mathrm{NH}), 7.3-6.7$ ( $\mathrm{m}, 10 \mathrm{H}$, arom.) 3.3 ( $\mathrm{s}, 2 \mathrm{H}, \mathrm{CH}_{2}$ ), $1.9-1.2\left(\mathrm{~m}, 12 \mathrm{H}\right.$, cyclic $\mathrm{CH}_{2}$ ) |
| 9a | $\begin{gathered} 138 \\ \text { DMF } \end{gathered}$ | 61 | $\begin{gathered} \mathrm{C}_{19} \mathrm{H}_{23} \mathrm{~N}_{3} \mathrm{~S} \\ (325.47) \end{gathered}$ | $\begin{aligned} & 70.11 \\ & 69.75 \end{aligned}$ | $\begin{aligned} & 7.12 \\ & 6.93 \end{aligned}$ | $\begin{aligned} & 12.91 \\ & 12.71 \end{aligned}$ | $\begin{aligned} & 9.85 \\ & 9.69 \end{aligned}$ | $\begin{gathered} 3271,3139(2 \mathrm{NH}), \\ 1410(\mathrm{SH}) \end{gathered}$ | $12.0,10.3$ (br, 2H, 2 NH ), $7.3-6.7$ (m, 8 H, arom.), 4.5 (s, 1H, SH); 3.3 (s, 2H, $\mathrm{CH}_{2}$ ), 1.9-1.2 (m, 8 H , cyclic $\mathrm{CH}_{2}$ ) |
| 9b | $\begin{gathered} 132 \\ \text { ethanol } \end{gathered}$ | 59 | $\begin{gathered} \mathrm{C}_{20} \mathrm{H}_{25} \mathrm{~N}_{3} \mathrm{~S} \\ (339.50) \end{gathered}$ | $\begin{aligned} & 70.75 \\ & 70.44 \end{aligned}$ | $\begin{aligned} & 7.42 \\ & 7.24 \end{aligned}$ | $\begin{aligned} & 12.37 \\ & 12.18 \end{aligned}$ | $\begin{aligned} & 9.44 \\ & 9.29 \end{aligned}$ | $\begin{gathered} 3175,3112 \text { (2 NH), } \\ 1432 \text { (SH) } \end{gathered}$ | $12.9,11.7$ (br, 2H, 2 NH ), $7.3-6.7$ (m, 8 H , arom.), 4.5 (s, 1H, SH); 3.3 ( $\mathrm{s}, 2 \mathrm{H}$, $\left.\mathrm{CH}_{2}\right), 1.9-1.2\left(\mathrm{~m}, 10 \mathrm{H}\right.$, cyclic $\left.\mathrm{CH}_{2}\right)$ |


| 9c | 141 <br> ethanol | 68 | $\begin{gathered} \mathrm{C}_{21} \mathrm{H}_{27} \mathrm{~N}_{3} \mathrm{~S} \\ (353.52) \end{gathered}$ | $\begin{aligned} & 71.34 \\ & 71.01 \end{aligned}$ | $\begin{aligned} & 7.69 \\ & 7.52 \end{aligned}$ | $\begin{aligned} & 11.88 \\ & 11.71 \end{aligned}$ | $\begin{aligned} & 9.06 \\ & 8.89 \end{aligned}$ | $\begin{aligned} & 3307,3169(2 \mathrm{NH}), \\ & 1427 \text { (SH) } \end{aligned}$ | $\begin{gathered} \text { 11.1, } 9.9(\mathrm{br}, 2 \mathrm{H}, 2 \mathrm{NH}), 7.3-6.7(\mathrm{~m}, 8 \mathrm{H}, \\ \text { arom.), 4.3(s, 1H, SH); 3.3(s, 2H, } \\ \left.\mathrm{CH}_{2}\right), 1.9-1.2\left(\mathrm{~m}, 12 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{gathered}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 10a | $\begin{gathered} 203 \\ \text { ethanol } \end{gathered}$ | 65 | $\begin{gathered} \mathrm{C}_{19} \mathrm{H}_{21} \mathrm{~N}_{3} \\ (291.41) \end{gathered}$ | $\begin{aligned} & 78.31 \\ & 77.94 \end{aligned}$ | $\begin{aligned} & 7.26 \\ & 7.09 \end{aligned}$ | $\begin{aligned} & 14.42 \\ & 14.23 \end{aligned}$ | - | $\begin{aligned} & 3209 \text { (NH), } 1621 \\ & (\mathrm{C}=\mathrm{N}) \end{aligned}$ | ```9.7 (br, 1H, NH), 7.5-7.0 (m, 10H, arom.), 3.4 (s, 2H, CH2), 1.9-1.2 (m, 8 H, cyclic CH2)``` |
| 10 b | 211 <br> dioxan | 79 | $\begin{gathered} \mathrm{C}_{20} \mathrm{H}_{23} \mathrm{~N}_{3} \\ (305.44) \end{gathered}$ | $\begin{aligned} & 78.65 \\ & 78.26 \end{aligned}$ | $\begin{aligned} & 7.59 \\ & 7.41 \end{aligned}$ | $\begin{aligned} & 13.76 \\ & 13.56 \end{aligned}$ | - | $\begin{aligned} & 3267(\mathrm{NH}), 1617 \\ & (\mathrm{C}=\mathrm{N}) \end{aligned}$ | ```11.1(br, 1H, NH), 7.5-7.0 (m,10H, arom.), 3.4 (s, 2H, CH2), 1.9-1.2 (m, 10 H, cyclic CH2)``` |
| 10c | $\begin{gathered} 216 \\ \text { ethanol } \end{gathered}$ | 67 | $\begin{gathered} \mathrm{C}_{21} \mathrm{H}_{25} \mathrm{~N}_{3} \\ (319.46) \end{gathered}$ | $\begin{aligned} & 78.95 \\ & 78.56 \end{aligned}$ | $\begin{aligned} & 7.89 \\ & 7.73 \end{aligned}$ | $\begin{aligned} & 13.15 \\ & 13.01 \end{aligned}$ | - | $\begin{aligned} & 3211(\mathrm{NH}), 1609 \\ & (\mathrm{C}=\mathrm{N}) \end{aligned}$ | $\begin{aligned} & 9.9 \text { (br, } 1 \mathrm{H}, \mathrm{NH}), 7.5-7.0(\mathrm{~m}, 10 \mathrm{H}, \\ & \text { arom.), } 3.4\left(\mathrm{~s}, 2 \mathrm{H}, \mathrm{CH}_{2}\right), 1.9-1.2(\mathrm{~m}, \\ & \left.12 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 11a | $\begin{gathered} 249 \\ \text { DMF } \end{gathered}$ | 61 | $\underset{(477.56)}{\mathrm{C}_{26} \mathrm{H}_{28} \mathrm{~N}_{3} \mathrm{O}_{2} \mathrm{PS}}$ | $\begin{aligned} & 65.39 \\ & 65.03 \end{aligned}$ | $\begin{aligned} & 5.91 \\ & 5.78 \end{aligned}$ | $\begin{aligned} & 8.79 \\ & 8.60 \end{aligned}$ | $\begin{aligned} & 6.71 \\ & 6.59 \end{aligned}$ | $\begin{aligned} & 3286(\mathrm{NH}), 1649 \\ & (\mathrm{C}-\mathrm{O}), 661(\mathrm{P}=\mathrm{S}) \end{aligned}$ | $\begin{aligned} & 9.9(\mathrm{br}, \mathrm{H}, \mathrm{NH}) ; 7.5-6.9(\mathrm{~m}, 14 \mathrm{H} \text {, arom.); } \\ & 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right) ; 3.5\left(\mathrm{~s}, 2 \mathrm{H}, \mathrm{CH}_{2}\right) \text {; } \\ & 1.9-1.2\left(\mathrm{~m}, 8 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 11b | $\begin{gathered} 221 \\ \text { ethanol } \end{gathered}$ | 59 | $\begin{gathered} \mathrm{C}_{27} \mathrm{H}_{30} \mathrm{~N}_{3} \mathrm{O}_{2} \mathrm{PS} \\ (491.59) \end{gathered}$ | $\begin{aligned} & 65.97 \\ & 65.60 \end{aligned}$ | $\begin{aligned} & 6.15 \\ & 6.00 \end{aligned}$ | $\begin{aligned} & 8.54 \\ & 8.41 \end{aligned}$ | $\begin{aligned} & 6.52 \\ & 6.38 \end{aligned}$ | $\begin{aligned} & 3213(\mathrm{NH}), 1646 \\ & (\mathrm{C}=\mathrm{O}), 669(\mathrm{P}=\mathrm{S}) \end{aligned}$ | $\begin{aligned} & 10.9(\mathrm{br}, \mathrm{H}, \mathrm{NH}) ; 7.5-6.9(\mathrm{~m}, 14 \mathrm{H}, \\ & \text { arom.); } 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right) ; 3.5(\mathrm{~s}, 2 \mathrm{H}, \\ & \left.\mathrm{CH}_{2}\right) ; 1.9-1.2\left(\mathrm{~m}, 10 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 11c | $\begin{gathered} 272 \\ \text { ethanol } \end{gathered}$ | 68 | $\underset{(505.61)}{\mathrm{C}_{28} \mathrm{H}_{32} \mathrm{~N}_{3} \mathrm{O}_{2} \mathrm{PS}}$ | $\begin{aligned} & 66.51 \\ & 66.15 \end{aligned}$ | $\begin{aligned} & 6.38 \\ & 6.21 \end{aligned}$ | $\begin{aligned} & 8.33 \\ & 8.16 \end{aligned}$ | $\begin{aligned} & 6.34 \\ & 6.17 \end{aligned}$ | $\begin{aligned} & 3287(\mathrm{NH}), 1652 \\ & (\mathrm{C}=\mathrm{O}), 653(\mathrm{P}=\mathrm{S}) \end{aligned}$ | $\begin{aligned} & 10.3(\mathrm{br}, \mathrm{H}, \mathrm{NH}) ; 7.5-6.9(\mathrm{~m}, 14 \mathrm{H}, \\ & \text { arom. }) ; 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right) ; 3.5(\mathrm{~s}, 2 \mathrm{H}, \\ & \left.\mathrm{CH}_{2}\right) ; 1.9-1.1\left(\mathrm{~m}, 12 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 12a | $\begin{gathered} 231 \\ \text { ethanol } \end{gathered}$ | 65 | $\begin{gathered} \mathrm{C}_{26} \mathrm{H}_{28} \mathrm{~N}_{3} \mathrm{OPS}_{2} \\ (493.62) \end{gathered}$ | $\begin{aligned} & 63.26 \\ & 62.85 \end{aligned}$ | $\begin{aligned} & 5.71 \\ & 5.56 \end{aligned}$ | $\begin{aligned} & 8.51 \\ & 8.37 \end{aligned}$ | $\begin{aligned} & 12.99 \\ & 12.70 \end{aligned}$ | 1410 (SH), $658(\mathrm{P}=\mathrm{S})$ | $\begin{aligned} & 7.3-6.9(\mathrm{~m}, 14 \mathrm{H} \text {, arom. }), 4.5(\mathrm{~s}, 1 \mathrm{H}, \mathrm{SH}) ; \\ & 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right) ; 3.5\left(\mathrm{~s}, 2 \mathrm{H}, \mathrm{CH}_{2}\right) \\ & 1.9-1.2\left(\mathrm{~m}, 8 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 12b | 227 <br> dioxan | 79 | $\begin{gathered} \mathrm{C}_{27} \mathrm{H}_{30} \mathrm{~N}_{3} \mathrm{OPS}_{2} \\ (507.65) \end{gathered}$ | $\begin{aligned} & 63.88 \\ & 63.47 \end{aligned}$ | $\begin{aligned} & 5.95 \\ & 5.81 \end{aligned}$ | $\begin{aligned} & 8.27 \mathrm{~s} \\ & 8.09 \end{aligned}$ | $\begin{aligned} & 12.63 \\ & 12.40 \end{aligned}$ | $1429(\mathrm{SH}), 670$ ( $\mathrm{P}=\mathrm{S}$ ) | $\begin{aligned} & 7.5-7.0(\mathrm{~m}, 14 \mathrm{H}, \text { arom. }), 4.3(\mathrm{~s}, 1 \mathrm{H}, \mathrm{SH}), \\ & 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 3.6\left(\mathrm{~S}, 2 \mathrm{H}, \mathrm{CH}_{2}\right) ; \\ & 1.9-1.2\left(\mathrm{~m}, 10 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |
| 12c | $\begin{gathered} 242 \\ \text { ethanol } \end{gathered}$ | 67 | $\begin{gathered} \mathrm{C}_{28} \mathrm{H}_{32} \mathrm{~N}_{3} \mathrm{OPS}_{2} \\ (521.67) \end{gathered}$ | $\begin{aligned} & 64.46 \\ & 64.12 \end{aligned}$ | $\begin{aligned} & 6.18 \\ & 6.03 \end{aligned}$ | $\begin{aligned} & 8.05 \\ & 7.90 \end{aligned}$ | $\begin{aligned} & 12.29 \\ & 12.09 \end{aligned}$ | 1420 (SH), $673(\mathrm{P}=\mathrm{S})$ | $\begin{aligned} & 7.5-7.0(\mathrm{~m}, 14 \mathrm{H}, \text { arom. }), 4.3(\mathrm{~s}, 1 \mathrm{H}, \mathrm{SH}), \\ & 3.9\left(\mathrm{~s}, 3 \mathrm{H}, \mathrm{OCH}_{3}\right), 3.5\left(\mathrm{~S}, 2 \mathrm{H}, \mathrm{CH}_{2}\right) ; \\ & 1.9-1.1\left(\mathrm{~m}, 12 \mathrm{H}, \text { cyclic } \mathrm{CH}_{2}\right) \end{aligned}$ |

[^1](cyclohexane, and cycloheptane)-1,4'-perhydro[1,3,2]diazaphosphole]-$2^{\prime}$-disulfi-des ( $\mathbf{5 a - c}$ ). As to the mechanism for the formation Pheterocycles 5a-c, it is suggested that a nucleophilic attack on LR to give the intermediate, which at elevated temperature looses $\mathrm{H}_{2} \mathrm{~S}$ to give, compounds 5a-c (cf. Scheme 2). The structures of these products were based on spectroscopic data and elemental analysis ( $c f$. Table I).

Compounds 6b,c were synthesized by Desai, ${ }^{15}$ and this synthesis can be carried out in another route via the diazotization of compounds $\mathbf{4 b}, \mathbf{c}$. Also, the diazotization of compound $\mathbf{4 a}$ gave 1anilinocyclopentanemethanol (6a). Compounds 6a-c were then submitted to the reaction with LR in anhydrous benzene to give $2^{\prime}$-(4-methoxyphenyl)-3'-phenylspiro[cyclopentane (cyclohexane, and cycloheptane)-1,4'-perhydro[1,3,2]oxaazaphosphole]-2'-disulfides (7ac) (cf. Scheme 2). The structures of the new products have been elucidated via analytical results and spectroscopic data ( $c f$. Table I).

Moreover, the addition of compounds 4a-c to phenyl isocyanate and/or phenyl isothiocyanate in ethanol at $26^{\circ} \mathrm{C}$ afforded the opened products $\mathbf{8 a - c}$ and $9 \mathbf{9 - c}$, respectively, via an addition of the amino groups of compounds $\mathbf{4 a - c}$ to the isocyanate or isothiocyanate (cf. Scheme 2). The cyclized products 10a-c were obtained by refluxing compounds 8a-c and/or 9a-c in N,N-dimethylaniline. The IR and ${ }^{1} \mathrm{H}$ NMR spectra of these compounds confirm the proposed structures (cf. Table I).

The reaction of compounds 8a-c and/or 9a-c with LR in refluxing $p$-xyl-ene afforded $2^{\prime}(4-$ methoxyphenyl)-3'-phenylspiro[cyclopentane (cyclohexane and cycloheptane)-1,4'-perhydro[1,3,2]diazaphosphole]1 -ylphenylaminomethane-1-ones (11a-c) and $2^{\prime}(4$-methoxyphenyl)-$3^{\prime}$-phenylspiro[cyclopentane (cyclohexane and cycloheptane)-1,4'-perhydro[1,3,2]diazaphosphole]-1-ylphenylaminometha-ne-1-thiones (12a-c), respectively. The same products 11a-c and 12a-c were also obtained through the reaction of compounds 5a-c with phenyl isocyanate and/or phenyl isothiocyanate in boiling benzene. The structure of compounds 11a-c and 12a-c were confirmed on the basis of their elemental and spectral analyses (cf. Table I).

## EXPERIMENTAL

## The Synthesis of 2'-(4-Methoxyphenyl)-4'-phenylspiro [cyclopentane (Cyclohexane and Cycloheptane)-1,4'-Perhydro[1,3,2]diazaphosphole]-2',5'-disulfides (3a-c): General Procedure

2,4-bis-(4-methoxyphenyl)-1,3,2,4-dithiaphosphetane-2,4-disulphide (LR, 1) ( $2.02 \mathrm{~g} ; 0.005 \mathrm{~mole}$ ) and 1-phenylamino-1-cyanocyclopentane


## SCHEME 2

(cyclohexane, and/or cycloheptane) (2a-c) ( 0.01 mol ) were dissolved in acetonitrile ( 80 mL ). The reaction mixture was refluxed for 6 h , concentrated and cooled the precipitate was filtered off, dried, and recrystallized from the suitable solvent to give compounds 3a-c.

## The Synthesis of 1-Anilinocyclopentane(cyclohexane, and Cycoheptane)methyl-amine 14a-c: General Procedure

To a solution of the proper nitrile $\mathbf{2 a - c}(1.0 \mathrm{~g}$ ) in absolute ethanol (10 mL ) was added 1.5 g of clean sodium. When all the sodium reacted ( $10-15 \mathrm{~min}$.), the reaction mixture was cooled to about $20^{\circ} \mathrm{C}$, and 15 mL of conc HCl was added. The reaction mixture was concentrated and cooled and 20 mL of $40 \% \mathrm{NaOH}$ was added dropwise with shaking to the reaction mixture. The formed amine was extracted with chloroform and purified by distillation.

## The Synthesis of 1-Anilinocyclopentane (Cyclohexane and Cycloheptane)methanol 6a-c: General Procedure

To a stirred ice-cooled solution of the proper amine (4a-c) ( 0.01 mole) in 8 mL of conc HCl was added portionwise ( $0.83 \mathrm{~g}, 0.012$ mole) of $\mathrm{NaNO}_{2}$. The mixture was kept at r.t. for 30 min and then poured onto crushed ice. The obtained solution was neutralized with $\mathrm{NH}_{4} \mathrm{OH}$, and the separated oil was extracted with chloroform and purified by distillation to give compounds 6a-c.

> The Synthesis of 2'-(4-Methoxyphenyl)-3'-phenylspiro [cyclopentane(cyclohexane and cycloheptane)-1,4'-Perhydro-[1,3,2]diazaphosphole]-2'-disulfides (5a-c) and 2'-(4-methoxyphenyl)-3'-phe-nylspiro[cyclopentane(cyclohexane and Cycloheptane)-1,4'-perhydro[1,3,2]oxaazaphosphole]-2'disulfides (7a-c): General Procedure

A mixture of the proper amine (4a-c) ( 0.01 mole ) and/or the proper hydroxylderivative (6a-c) ( 0.01 mole) and 2,4-bis-(4-methoxyphenyl)-1,3,2,4-dithiaphosphetane-2,4-disulphide (LR, 1) ( $2.02 \mathrm{~g}, 0.005$ mole) in dry benzene ( 80 mL ) was refluxed for 5 h , concentrated, and cooled. The formed precipitate was filtered off, dried, and recrystallized from the suitable solvent to give compounds $\mathbf{5 a}-\mathbf{c}$ and $7 \mathbf{7 a}-\mathbf{c}$, respectively.

## The Synthesis of Compounds 8a-c and 9a-c: General Procedure

To a stirred solution of the proper amine 4a-c ( 0.01 mole ) in 30 mL of absolute ethanol was added phenyl isocyanate ( $1.19 \mathrm{~g}, 0.01 \mathrm{~mole}$ )
or phenyl isothiocyanate ( $1.35 \mathrm{~g}, 0.01$ mole). The reaction mixture was
stirred at r.t. for 1 hour. The formed precipitate was filtered off, dried, and recrystallized from the suitable solvent to give compounds 8a-c and $9 \mathbf{9 - c}$, respectively.

## The Synthesis of $\mathbf{3}^{\prime}$-Phenyl-2'-phenylaminospiro [cyclopentane(cyclohexane and Cycloheptane)-1,4'-(3', $\mathbf{5}^{\prime}$ dihydroimidazoles)] (10a-c): General Procedure

Compound 8a-c and/or 9a-c ( 0.01 mol ) was refluxed in $\mathrm{N}, \mathrm{N}$ dimethylaniline ( 10 mL ) for 12 h . The solvent was concentrated, cooled, and filtered off. The solid product was recrystallized from the suitable solvent ( $c f$. Table I).

## The Synthesis of 2'-(4-Methoxyphenyl)-3'phenylspiro[cyclopentane(cyclohexane and Cycloheptane)-1,4'-Perhydro[1,3,2]diazaphosphole]-1-ylphenylamino-methane-1-ones (11a-c) or -1-thiones (12a-c): General Procedure

2,4-bis-(4-methoxyphenyl)-1,3,2,4-dithiaphosphetane-2,4-disulphide (LR, 1) ( $2.02 \mathrm{~g} ; 0.005$ mole) and the proper compound 8a-c and/or 9a-c ( 0.01 mol ) were dissolved in p-xylene ( 60 mL ). The reaction mixture was refluxed for 6 h , concentrated, and cooled; the precipitate was filtered off, dried, and recrystallized from the proper solvent to give compounds 11a-c and 12a-c, respectively.

## The Alternate Synthesis of Compounds 11a-c and 12a-c: General Procedure

Compound 5a-c ( 0.01 mol ), phenyl isocyanate and/or pheny isothiocyanate ( 0.01 mol ) and dry benzene ( 70 mL ) were refluxed together for 3 h when colorless crystals separated out, which were filtered and washed with warm benzene to afford compounds 11a-c and 12a-c.

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    Address correspondence to H. M. Moustafa, Chemistry Department, Faculty of Science, Sohag, Egypt. E-mail: hassa20002000@yahoo.com

[^1]:    ${ }^{a}$ Uncorrected
    ${ }^{b}$ Satisfactory microanalysis obtained C, $-0.47 ; \mathrm{H},-0.25 ; \mathrm{N},-0.39 ; \mathrm{S},-0.35$. ${ }^{c}$ Measured by Nicolet FT-IR 710 spectrophotometer.
    ${ }^{d}$ Measured by ${ }^{1}$ HNMR LA 400 MHz (Jeol) Assiut University.

