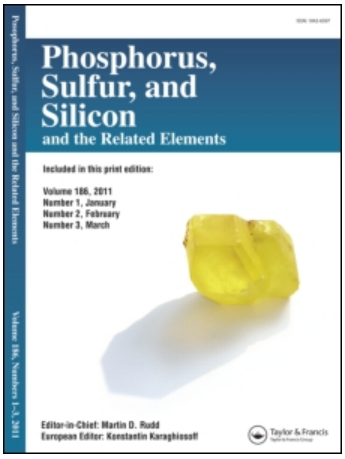

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New Synthesis of Phosphoroheterocycles by Cyclization of Multifunctionalized 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides with Phosphorus Trichloride and Lawesson's Reagent

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NEW SYNTHESIS OF PHOSPHOROHETEROCYCLES BY CYCLIZATION OF MULTIFUNCTIONALIZED 2-(N-(β- OR γ-HYDROXY)ALKYL)-AMINOBENZAMIDES WITH PHOSPHORUS TRICHLORIDE AND LAWESSON'S REAGENT

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Cyclization of multifunctional compounds 2-(N-(β- or γ-hydroxy)-alkyl)aminobenzamides with PCl_3 and Lawesson's reagent have been studied. Both, the reaction of compounds 2-(N-(β- or γ-hydroxy)-alkyl)aminobenzamides with PCl_3 generated chlorinated benzophosphoroheterocycle, 1-[2′- or 3′-chloroalkyl]-3-alkyl-2,3-dihydro-1,3,2-benzodiazaphosphorine-4(1H)-one-2-oxides, and the treatment of 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides with Lawesson's reagent (LR), which could readily produce 5- or 6-membered phosphoroheterocycles, 1,3,2-oxazaphospholidines, or 1,3,2-oxazaphosphorines.

Keywords: 1,3,2-Oxazaphospholidines; 1,3,2-oxazaphosphorines; 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides; benzophosphoroheterocycle; Lawesson's reagent; PCl_3

INTRODUCTION

Organophosphorus compounds are ubiquitous in nature and they have many practical applications in the fields of pharmacy and agriculture. In particular, phosphoroheterocycles have attracted a considerable interest in recent years due to a large variety of biological activities such as fungicidal,[1] herbicidal,[2,3] antitumor, and antiviral properties.[4–7] These significant results prompt us to search continuously for more biologically active phosphoroheterocycles. More recently, new synthesis of some phosphoroheterocycles has met with success

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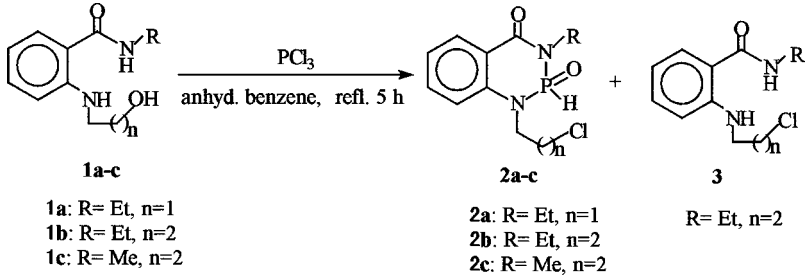


SCHEME 1

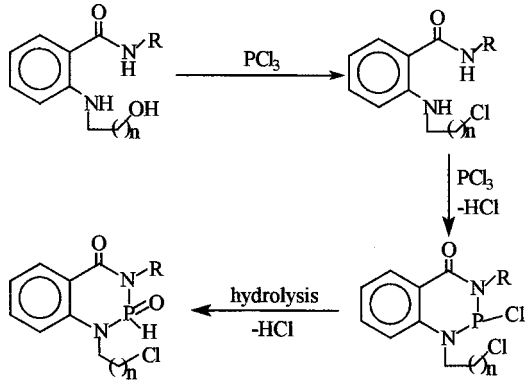
in our laboratory based on the reactions of multifunctional compounds, 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides, with frequently used phosphorus reagents such as PCl_3 and Lawesson's reagent as described in Schemes 1 and 3. In our previous study, we found that multifunctional compounds **1a–c** could be transformed into fused tricyclic phosphoroheterocycles in low to moderate yields under suitable conditions when treated with tris(diethylamino)phosphine.[8] While this article, as a parallel study, focuses on the reactions of 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides with PCl_3 and Lawesson's reagent, resulting in benzoannulated phosphoroheterocycles, 1-[2′- or 3′-chloroalkyl]-3-alkyl-2,3-dihydro-1,3,2-benzodiazaphosphorine-4(1H)-one-2-oxides **2a–c**, 1,3,2-oxazaphosphorines and 1,3,2-oxazaphospholidines **4a–f** respectively.

RESULTS AND DISCUSSION

Reaction of 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides with PCl_3

Starting materials **1a–f** were obtained routinely by the reaction of 2-aminobenzamides with 2-bromoethanol or 3-chloropropanol in refluxing toluene according to the literature.[8] It was found that addition of PCl_3 to the solution of compounds **1a–c** in anhydrous benzene at 40–50°C followed by refluxing for 5 h under N_2 atmosphere resulted in unexpected benzoannulated phosphoroheterocycles **2a–c** and chlorinated compound **3**. The overall yields of **2a–c** were in the range of 39.6–61.5% and the yield of compound **3** was up to 24.6% isolated by column chromatography. The reaction of **1a–c** with PCl_3, to the best of our knowledge, has not been reported to date, although the analogs of **2a–c** can be obtained by treatment of PCl_3 with alternative 2-(2-bromoethyl)aminobenzamides under proper conditions.[9] However,

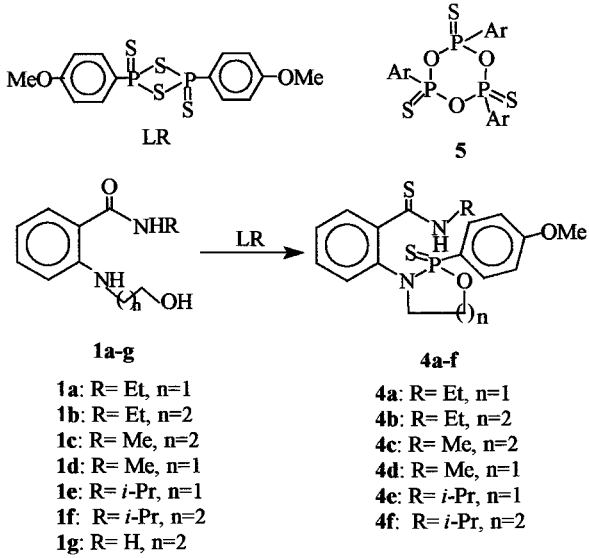
the former reaction might proceed through a different way. As to the mechanism for the formation of compounds **2a–c** and **3**, our understanding is not enough. Anyhow, production of **3** provides important information on the mechanism. Probably chlorination of hydroxy group by PCl_3 occurs first, further cyclocondensation of the chlorinated intermediate with PCl_3 and hydrolysis provides final product with loss of hydrochloride (Scheme 2).



SCHEME 2

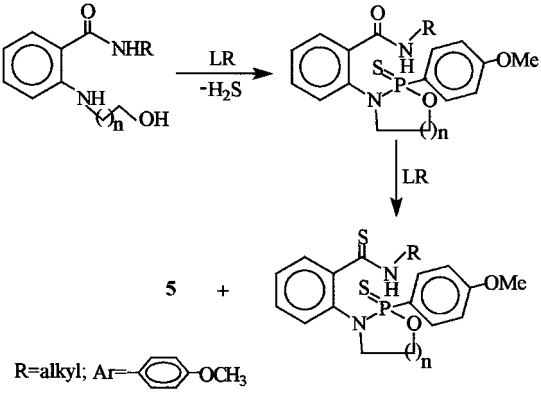
Reaction of 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides with Lawesson's Reagent

Having established the formation of benzoannulated phosphoroheterocycles **2a–c** was feasible, attention was turned toward the compatibility of 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides for the preparation of structurally different phosphoroheterocycle. It was known that Lawesson's reagent (LR) could make diverse bifunctional compounds transform into interesting array of phosphorus-containing heterocycles,[10–14] this leads us to study the reaction of multifunctional compounds **1a–f** with LR. Interestingly, treatment of **1a–f** with an equivalent amount of LR was able to produce O—P—N type phosphoroheterocycles **4a–f** and a trimer **5** concomitantly when carried out at 80–140°C using anhydrous xylene as solvent (Scheme 3). Besides xylene the alternative reaction solvents include toluene and benzene. It should be noted that higher temperature would give higher yield of product and also shorten reaction time effectively. However, the interaction of compound **1g** with Lawesson's reagent in refluxing xylene could not generate desired phosphoroheterocycle, only production of isolable trimer **5** and polymerization were observed during the reaction.



SCHEME 3

Compounds **4a–f** were characterized by spectroscopies and microanalyses (Tables I and II), and **4d** was further confirmed by single crystal x-ray diffraction studies. Characterization of compound **5** by ^{1}H NMR and ^{31}P NMR was consistent with that early reported in the literature,[11] it evidently indicates the existence of oxygen-sulfur exchange process during the reaction. Therefore the mechanism of the reaction is proposed to involve cyclization of compounds **1a–f** with LR in the first step and subsequent thiation of carbonyl group resulting in the final products (Scheme 4). However it is still difficult to tell



SCHEME 4

TABLE I The Experimental and Microanalytical Data of Compounds **4a–f**

Product	Yield (%)[a]	m.p. (°C)	Molecular formula	Formula weight[b]	Elemental analyses found (calcd. %) C	H	N
4a	74	125–127	$C_{18}H_{21}N_2O_2PS_2$	392	55.05 (55.10)	5.14 (5.36)	6.97 (7.14)
4b	61	163–164	$C_{19}H_{23}N_2O_2PS_2$	406	56.37 (56.16)	5.77 (5.67)	6.82 (6.90)
4c	52	130–133	$C_{18}H_{21}N_2O_2PS_2$	392	55.43 (55.10)	5.71 (5.36)	7.44 (7.14)
4d	73	109–112	$C_{17}H_{19}N_2O_2PS_2$	378	53.62 (53.97)	4.89 (5.03)	7.78 (7.41)
4e	44	106–108	$C_{19}H_{23}N_2O_2PS_2$	406	56.60 (56.16)	5.28 (5.67)	6.78 (6.90)
4f	43	122–124	$C_{20}H_{25}N_2O_2PS_2$	420	56.87 (57.14)	6.12 (5.95)	7.04 (6.67)

[a]Yield of pure isolated product.

[b]EI-MS data for **4c**: m/z (%) = 392 (M^+, 1), 359 (100), 328 (12), 220 (3), 173 (20), 145 (19), 117 (13), 108 (13), 77 (18), 63 (9); **4e**: 406 (M^+, 12), 389 (33), 373 (100), 285 (10), 220 (12), 187 (35), 174 (89), 145 (60), 130 (59), 77 (26), 43 (48); **4f**: 420 ((M^+, 7), 387 (100), 328 (25), 299 (7), 187 (16), 157 (41), 144 (39), 131 (33), 108 (29), 77 (26), 63 (13), 43 (25).

which step occurs first because thiation of carbonyl group by Lawesson's reagent usually occurred from 25°C to 110°C,[15,16] and cyclization of Lawesson's reagent with bifunctional compounds containing mobile hydrogens could also take place at 60°C.[17]

In summary, this article presents hereby new methods for the synthesis of different phosphoroheterocycles, 1-[2′- or 3′-chloroalkyl]-3-alkyl-2,3-dihydro-1,3,2-benzodiazaphosphorine-4(1H)-one-2-oxides **2a–c**, 1,3,2-oxazaphosphorines, and 1,3,2-oxazaphospholidines **4a–f**, based on the reactions of multifunctional compounds 2-(N-(β- or γ-hydroxy)alkyl)aminobenzamides with phosphorus reagents such as PCl_3 and Lawesson's reagent. The possible reaction mechanisms are proposed, although our understanding is not enough.

EXPERIMENTAL

All reagents were obtained from commercial suppliers and used without further purification unless otherwise indicated. Xylene and benzene were dried over sodium before use. Lawesson's reagent was prepared according to the literature.[18] Melting points were determined with a model YANACO MP-500 apparatus and are uncorrected. FTIR spectra

TABLE II The FTIR and NMR Data for Compounds **4a–f**

Product	^{31}P NMR (δ ppm)	FTIR (KBr, ν cm^{-1})	^{1}H NMR ($CDCl_3$ as solvent, δ ppm)
4a	85.46	3219 (NH), 1599, 1567, 1515, 1471 (Ar), 1329 (C═S), 1218 (P—O—C), 1061 (Ar—O—C)	1.37 (t, 3H, $^{3}J_{H-H}$ = 6.46 Hz, $CH_2C\underline{H}_3$), 3.68–4.11 (m, 4H, $NC\underline{H}_2CH_3$ + $NC\underline{H}_2CH_2O$), 3.78 (s, 3H, OCH_3), 4.35–4.77 (m, 2H, $NCH_2C\underline{H}_2O$), 7.06 (dd, 2H, $^{4}J_{P-H}$ = 3.58 Hz, $^{3}J_{H-H}$ = 8.68 Hz, H_{arom}), 7.09–7.56 (m, 4H, H_{arom}), 7.94 (dd, 2H, $^{3}J_{P-H}$ = 7.85 Hz, $^{3}J_{H-H}$ = 8.68 Hz, H_{arom}), 9.36 (br, 1H, NH)
4b	78.95	3220 (NH), 1594, 1567, 1501, 1448 (Ar), 1288 (C═S), 1114 (P—O—C), 1049 (Ar—O—C)	1.42 (t, 3H, $^{3}J_{H-H}$ = 6.78 Hz, $CH_2C\underline{H}_3$), 1.84–2.26 (dm, 2H, $NCH_2C\underline{H}_2CH_2O$), 3.64–3.80 (m, 4H, $NC\underline{H}_2CH_3$ + $NC\underline{H}_2CH_2CH_2O$), 3.88 (s, 3H, OCH_3), 4.08–4.52 (dm, 2H, $NCH_2CH_2C\underline{H}_2O$), 7.02 (dd, 2H, $^{4}J_{P-H}$ = 2.98 Hz, $^{3}J_{H-H}$ = 8.44 Hz, H_{arom}), 7.19–7.61 (m, 4H, H_{arom}), 7.87 (dd, 2H, $^{3}J_{P-H}$ = 8.95 Hz, $^{3}J_{H-H}$ = 8.44 Hz, H_{arom}), 9.47 (br, 1H, NH)
4c	79.28	3219 (NH), 1595, 1544, 1500, 1450 (Ar), 1257 (C═S), 1109 (P—O—C), 1050 (Ar—O—C)	1.86–2.24 (dm, 2H, $NCH_2C\underline{H}_2CH_2O$), 3.31 (d, 3H, $^{3}J_{H-H}$ = 4.54 Hz, $NHC\underline{H}_3$), 3.60–3.70 (m, 2H, $NC\underline{H}_2CH_2CH_2O$), 3.86 (s, 3H, OCH_3), 4.03–4.68 (dm, 2H, CH_2O), 6.98–7.92 (m, 8H, H_{arom}), 9.62 (br, 1H, NH)
4d	89.60	3234 (NH), 1595, 1568, 1502, 1452 (Ar), 1259 (C═S), 1113 (P—O—C), 1020 (Ar—O—C)	3.35 (d, 3H, $^{3}J_{H-H}$ = 5.15 Hz, $NHC\underline{H}_3$), 3.71–4.03 (m, 2H, $NC\underline{H}_2CH_2O$), 3.91 (s, 3H, OCH_3), 4.32–4.65 (m, 2H, $CH_2C\underline{H}_2O$), 7.03 (dd, 2H, $^{4}J_{P-H}$ = 3.36 Hz, $^{3}J_{H-H}$ = 8.50 Hz, H_{arom}), 7.06–7.85 (m, 4H, H_{arom}), 7.91 (dd, 2H, $^{3}J_{P-H}$ = 8.12 Hz, $^{3}J_{H-H}$ = 8.50 Hz, H_{arom}), 9.65 (br, 1H, NH)
4e	83.63	3206 (NH), 1593, 1572, 1499, 1455 (Ar), 1256 (C═S), 1107 (P—O—C), 1026 (Ar—O—C)	1.38 (d, 3H, $^{3}J_{H-H}$ = 6.50 Hz, CH_3), 1.56 (d, 3H, $^{3}J_{H-H}$ = 6.86 Hz, CH_3), 3.42–4.87 (m, 5H, NCH_2CH_2O + NCH), 3.89 (s, 3H, OCH_3), 6.51–8.11 (m, 8H, H_{arom}), 9.56 (br, 1H, NH)
4f	78.13	3228 (NH), 1599, 1582, 1459, 1465 (Ar), 1246 (C═S), 1120 (P—O—C), 1036 (Ar—O—C)	1.26 (d, 3H, $^{3}J_{H-H}$ = 6.41 Hz, CH_3), 1.44 (d, 3H, $^{3}J_{H-H}$ = 6.74 Hz, CH_3), 1.72–2.41 (dm, 2H, $NCH_2C\underline{H}_2CH_2O$), 3.79 (s, 3H, OCH_3), 3.58–4.80 (m, 5H, $NC\underline{H}_2CH_2C\underline{H}_2$ + $NC\underline{H}$), 6.64–7.56 (m, 4H, H_{arom}), 6.94 (dd, 2H, $^{4}J_{P-H}$ = 2.84 Hz, $^{3}J_{H-H}$ = 8.60 Hz, H_{arom}), 7.80 (dd, 2H, $^{3}J_{P-H}$ = 7.10 Hz, $^{3}J_{H-H}$ = 8.60 Hz, H_{arom}), 9.28 (br, 1H, NH)

were recorded on a BIO-BAD EXCALIBUR FTS3000 spectrometer. The ^{1}H and ^{31}P NMR spectra were recorded on a BRUKER AC-P200 instrument. Tetramethylsilane (TMS) was used as an internal standard for ^{1}H NMR, and 85% phosphoric acid (H_3PO_4) was used as an external standard for ^{31}P NMR spectroscopy. The nuclei that are deshielded relative to their respective standards are assigned a positive chemical shift. Coupling constants, J, are given in Hz. Elemental analyses were carried out on a Yanaco MT-3 instrument. EI-MS spectra were recorded with a VG-7070E spectrometer. Column chromatography was performed using silica gel H (10–40 μm, Haiyang Chemical Factory of Qingdao).

1-(2′-Chloroalkyl)-3-alkyl-2,3-dihydro-1,3,2-benzodiazaphosphorine-4 (1H)-one 2-oxide 2a and 2b–c (General Procedure)

A suspension of **1a–c** (0.01 mmol) in anhydrous benzene (100 mL) in a 500 mL three-necked flask was stirred at 40–50°C until the mixture became transparent, then an equivalent amount of PCl_3 was added dropwise within 30 min under N_2 at the same temperature. The resulting solution was heated at reflux for another 5 h, then concentrated in vacuo. The crude product was isolated by flash chromatography using EtOAc-petroleum ether ($^{1}/_{3}$ v/v) as eluant ($R_f = 0.2$). Recrystallization of the crude products from Et_2O-petroleum ether ($^{1}/_{5}$ v/v) gave pure products **2a–c**.

2a: White powder, m.p: 99–101°C, yield: 61.5%. Anal. Calcd for $C_{11}H_{14}ClN_2O_2P$ (W = 272.5): C, 48.44; H, 5.14; N, 10.28. Found: C, 48.52; H, 5.08; N, 10.54. ^{1}H NMR ($CDCl_3$/TMS, δppm): 1.32 (t, 3H, $^{3}J_{H-H} = 7.42$ Hz, CH_3), 3.60–4.10 (m, 6H, $C\underline{H}_2CH_3 + NC\underline{H}_2C\underline{H}_2Cl$), 7.87 (d, 1H, $^{1}J_{P-H} = 642.03$ Hz, P–H), 6.90–8.20 (m, 4H, H_{arom}). EI-MS: m/z (%) = 272 (M^+, 22.7), 274 ($M^+ + 2$, 7.6).

2b: White powder, m.p: 47–49°C, yield: 39.6%. Anal. Calcd for $C_{12}H_{16}ClN_2O_2P$ (W = 286.5): C, 50.26; H, 5.58; N, 9.77. Found: C, 50.52; H, 5.18; N, 10.04. ^{1}H NMR ($CDCl_3$/TMS, δppm): 1.34 (t, 3H, $^{3}J_{H-H} = 7.06$ Hz, $CH_2C\underline{H}_3$), 2.23–2.27 (m, 2H, $NCH_2C\underline{H}_2CH_2Cl$), 3.61–4.02 (m, 6H, $C\underline{H}_2CH_3 + NC\underline{H}_2CH_2C\underline{H}_2Cl$), 7.85 (d, 1H, $^{1}J_{P-H} = 641.51$ Hz, P–H), 7.06–8.14 (m, 4H, H_{arom}). ^{31}P NMR ($CDCl_3$/85%H_3PO_4, δppm): 5.59. EI-MS: m/z (%) = 288($M^+ + 2$, 9), 286(M^+, 27), 251 (11), 223 (100), 195 (12), 180 (16), 159 (16), 132 (47), 77 (22).

2c: White powder, m.p: 57°C, yield: 47.8%. Anal. Calcd for $C_{11}H_{14}ClN_2O_2P$ (W = 272.5): C, 48.44; H, 5.14; N, 10.28. Found: C, 48.32; H, 5.48; N, 10.34. ^{1}H NMR ($CDCl_3$/TMS, δppm): 2.19–2.25 (m, 2H, $NCH_2C\underline{H}_2CH_2Cl$), 3.34 (d, 3H, $^{3}J_{P-H} = 8.30$ Hz, CH_3), 3.63 (t, 2H, $^{3}J_{H-H} = 5.95$ Hz, $NCH_2CH_2C\underline{H}_2Cl$), 3.87–3.92 (m, 2H,

$NC\underline{H}_2CH_2CH_2Cl$), 7.82 (d, 1H, $^1J_{P-H} = 643.76$ Hz, P–H), 7.03–8.19 (m, 4H, H_{arom}). ^{31}P NMR ($CDCl_3$/85%H_3PO_4, δppm): 3.49. EI-MS: m/z (%) = 274 (M^+, 8) 272 (M^+, 23), 209 (100), 179 (11), 145 (23), 132 (40), 77 (27).

2-[N-(γ-Chloro)propyl]amino-N-ethyl-benzamide 3

When compound **2b** was isolated by flash chromatography, the fractions ($R_f = 0.7$) were collected and the solvents were further removed at reduced pressure to offer light yellow sticky oil, Yield: 24.6%. Anal. Calcd for $C_{12}H_{17}ClN_2O$ (W = 240): C, 60.00; H, 7.08; N, 11.67. Found: C, 60.23; H, 7.48; N, 11.54. ^1H NMR ($CDCl_3$/TMS, δppm): 1.22 (t, 3H, $^3J_{H-H} = 8.24$ Hz, $CH_2C\underline{H}_3$), 2.10–2.24 (m, 2H, $NCH_2C\underline{H}_2CH_2Cl$), 3.08–4.06 (m, 6H, $NC\underline{H}_2CH_2C\underline{H}_2Cl + C\underline{H}_2CH_3$), 6.38–7.55 (m, 4H, H_{arom}), 8.24 (br, 1H, NH).

EI-MS: m/z (%) = 242 ($M^+ + 2$, 5), 240 (M^+, 14), 177 (29), 132 (100), 120 (7), 105 (6), 77 (11).

3-(*o*-Alkylaminothiocarbonyl)phenyl-1,3,2-oxazaphosphorines and 3-(*o*-Alkylaminothiocarbonyl)phenyl-1,3,2-oxazaphospholidines 4a–f (General Procedure)

A suspension of compound **1a–f** (2.5 mmol) and an equivalent amount of Lawesson's reagent in 50 mL anhydrous xylene were refluxed with stirring under N_2 for 5 h. Then the reaction mixture was filtered. The filtrate was concentrated on a rotatory evaporator at reduced pressure. The residue was chromatographed using a mixture of ethyl acetate-petroleum ether (1/5 v/v) as eluant yielding crude products. Further recrystallization of the crude products from a mixture of methylene chloride and light petroleum(1/3 v/v) afforded compound **4a–f** as yellow powder. The experimental and analytical data for **4a–f** were tabulated in the text and the crystal structure of compound **4d** was illustrated in Figures 1 and 2.

X-ray Diffraction Analysis of Compound 4d

Crystallographic Data for 4d

$C_{17}H_{19}N_2O_2PS_2$, $M_w = 378.43$, Monoclinic, *P*n, a = 9.905 (3), $b =$ 19.091 (5), $c = 20.384$ (5) Å, $\alpha = 90$, $\beta = 99.180$ (5), $\gamma = 90^\circ$, $V = 3805.0$ (17) Å^3, $Z = 8$, $D_x = 1.321$ g/cm^{-3}, and $F(000) = 1584$.

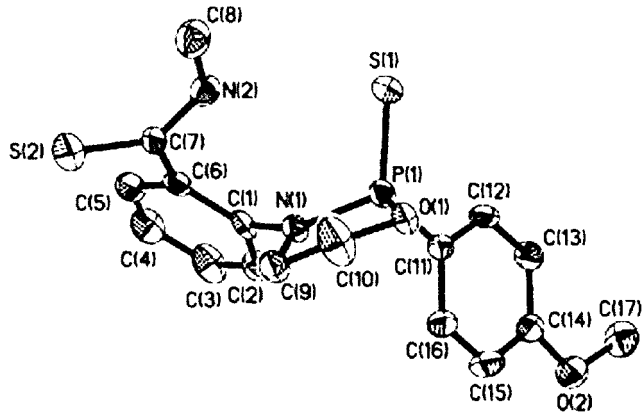


FIGURE 1 Molecular structure of compound **4d** with numbering scheme.

Structure Determination

A colorless crystal of **4d** with approximate dimensions of 0.25 mm × 0.20 mm × 0.20 mm was mounted on a glass fiber in a random orientation. The determination of the unit cell and the data collection were performed with Mo$K\,\sigma$ radiation ($\lambda = 0.71073$ Å) on an BRUKER SMART 1000 diffractometer. A total of 15649 reflections were collected

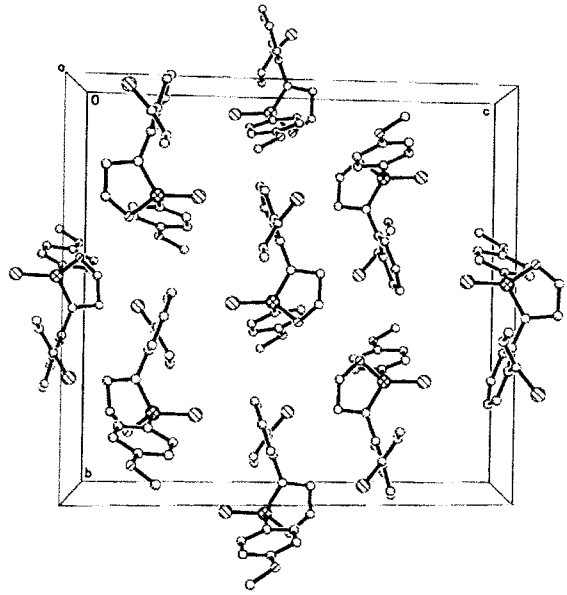


FIGURE 2 Packing of the molecules in a unit cell.

in the range of $1.07^\circ < \theta < 25.03^\circ$ at room temperature, in which 10230 reflections with $I > 2\ \sigma(\mathrm{I})$ were considered to be observed and used in the successive refinements. The correction for Lp factors was applied. The structure was determined by direct method (SHELX-97). Most of the non-hydrogen atoms were located from an E-map; the others were determined with successive differential Fourier syntheses. The structure was further refined by full-matrix least-squares method with anisotropic thermal parameters for all nonhydrogen atoms. The hydrogen atoms were located theoretically and refined with riding model position parameters and fixed isotropic thermal parameters. A full-matrix least-squares refinement gave final $R_1 = 0.0448$, $wR_2 = 0.0861$ (unit weight) with $W = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ and $P = (F_o^2 + 2F_c^2)/3$. The maximum peak in the final difference Fourier maps was 0.277 e/Å^3 and the minimum peak was -0.242 e/Å^3. All calculations were carried out using SHELXS-97.

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