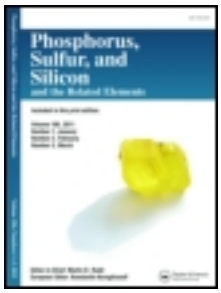


Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gpss20

# Synthesis and Structure of Some 6,7-Dihydro-thiazolo[4,5- d ][1,3,2]diaza-λ 5 -phosphorines

Mikhail A. Rensky [a] , Vladimir S. Zyabrev [a] & Alexander N. Chernega [a]

[a] National Academy of Sciences of Ukraine, Keiv, Ukraine

Version of record first published: 27 Oct 2010.

To cite this article: Mikhail A. Rensky, Vladimir S. Zyabrev & Alexander N. Chernega (2003): Synthesis and Structure of Some 6,7-Dihydro-thiazolo[4,5- d ][1,3,2]diaza-λ 5 -phosphorines, Phosphorus, Sulfur, and Silicon and the Related Elements, 178:10, 2127-2135

To link to this article: http://dx.doi.org/10.1080/713744577

*Phosphorus, Sulfur, and Silicon*, 178:2127–2135, 2003

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500390221591



# SYNTHESIS AND STRUCTURE OF SOME 6,7-DIHYDRO-THIAZOLO[4,5-*d*][1,3,2]DIAZA-$\lambda^5$-PHOSPHORINES

*Mikhail A. Rensky, Vladimir S. Zyabrev, and Alexander N. Chernega*
*National Academy of Sciences of Ukraine, Keiv, Ukraine*

(Received July 24, 2002; accepted March 4, 2003)

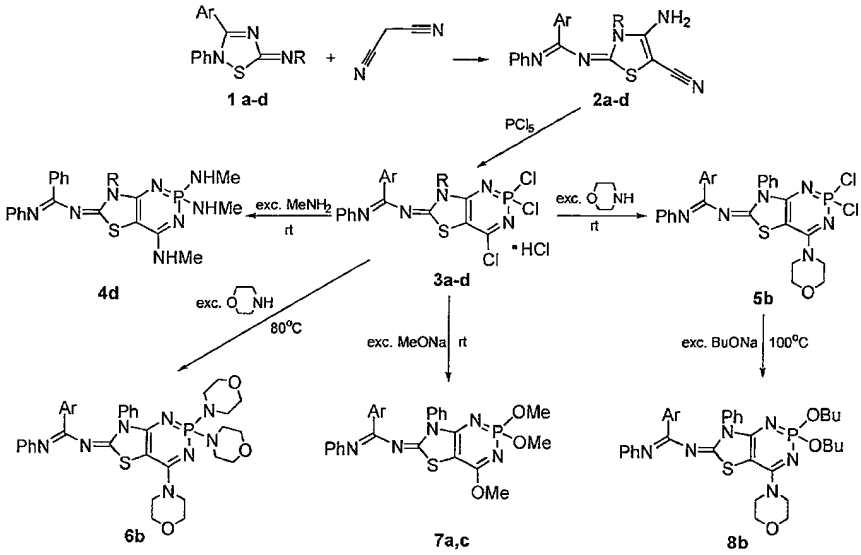
*1,2,4-Thiadiazol-5(2*H*)-imines **1** react with malononitrile under ring cleavage cycloaddition and subsequent ring rearrangement. The products **2** incorporate the enaminonitrile fragment. Treatment of compounds **2** with phosphorus pentachloride results in thiazolo[4,5-*d*][1,3,2]diaza-$\lambda^5$-phosphorine derivatives **3** with a reactive chloro substituent in position 2 and 4 which can be substituted by various nucleophiles. The structure of the trimethoxy derivative **7c** was determined by an X-ray structure analysis.*

*Keywords:* Bicyclic heterocyclic compounds; cycloadditions; rearrangements; substitution

The synthesis of 1,3,2-diaza-$\lambda^5$-phosphorine derivatives has been reported in 1963,[1] and during the ensuing years their chemical, spectral, and pesticidal properties have been intensively studied.[2–8] Some of them exhibited significant antimycotic activity.[9] Nevertheless, fused heterocycles containing the unsaturated 1,3,2-diaza-$\lambda^5$-phosphorine ring have not been reported up to now, whereas bicyclic nitrogen and phosphorus containing compounds are actively screened to search for antitumour agents.[10]

Herein, we describe the first representatives of the thiazolo[4,5-*d*][1,3,2]diazaphosphorine series, which are conveniently prepared from easily available 1,2,4-thiadiazol-5(2*H*)-imines **1** (Scheme 1). We recently revealed that compounds **1** react with malononitrile under ring cleavage cycloaddition and subsequent rearrangement to yield the products

Address correspondence to V. S. Zyabrev, Institute of Bioorganic Chemistry and Petrochemistry, National Academy of Sciences of Ukraine, 1 Murmanskaya St., 02094 Kiev, Ukraine. E-mail: users@bpci.kiev.ua




| 1-8 | a | b | c | d |
|---|---|---|---|---|
| Ar | Ph | 4-$MeC_6H_4$ | 4-$MeOC_6H_4$ | Ph |
| R | Ph | Ph | Ph | $PhCH_2$ |

**SCHEME 1** The synthesis of compounds **2–8**.

**2** which are suitable substrates for further heterocyclizations.[11] One interesting heterocyclization occurs when **2** is treated with phosphorus pentachloride. The reaction proceeds quite regioselectively with participation of the amino and cyano groups as in the case of simple enaminonitriles.[12] The anellation products **3**, containing three reactive chlorine atoms, react differently with amines and alcoholates depending on the nature of the nucleophile and the temperature. These peculiarities of the compounds **3** were exploited with respect to the synthesis of thiazolo[4,5-*d*][1,3,2]diaza-$\lambda^5$-phosphorine derivatives **4–8** bearing various substituents at positions 2 and 4.

The structure of all compounds was verified by IR and $^1$H NMR spectra, whereas the structure of **7c** was determined by an x-ray diffraction analysis (Figure 1, Table I). The P(1)S(1)N(1–3)C(1–3,7) bicyclic system of this compound is almost planar. Deviations from the least-squares plane do not exceed 0.092 Å, the dihedral angle between the 5- and 6-membered ring is only 2.3°. The bond system C(7)=N(4)—C(20)[C(21)]=N(15)—C(14) is approximately coplanar to the

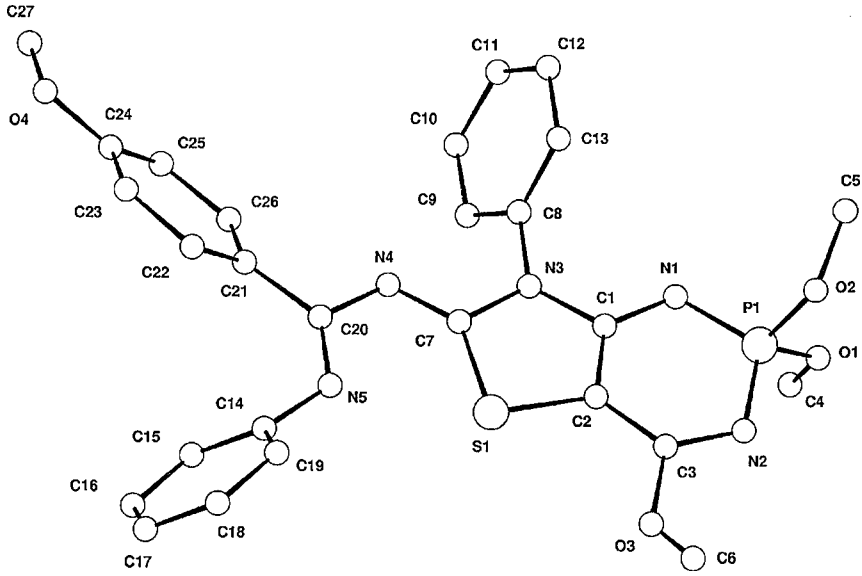



**FIGURE 1** Perspective view and labelling scheme for molecule **7c** (hydrogen atoms are omitted for clarity).

least-squares plane, the corresponding dihedral angle is 7.2°. The sterically unfavourable cisoid conformation of the diazadiene fragment C(7)N(4)C(20)N(5) is probably due to packing forces in the solid state. In a solution, however, **7c** exists as a mixture of two stereoisomers, as evidenced by the $^1$H NMR spectra (see Experimental section). These stereoisomers or conformers can result from syn-anti isomerization at N(4) and N(5), as well as from rotation around the N(4)-C(20) bond. At 90°C, they are rapidly interconverting on the NMR time scale. Similar conformation equilibriums were documented by the $^1$HNMR also for compounds **4d** and **7a**.

## EXPERIMENTAL

All reagents and solvents were commercial products and were used as received unless otherwise indicated. Melting points were determined on a capillary tube apparatus and are uncorrected. IR spectra were taken on a Specord 71IR spectrophotometer in a $CH_2Cl_2$ solution and on a UR 20 spectrophotometer in a KBr disk. $^1$H NMR spectra were recorded on a Varian VXR 300 (300 MHz) and a Varian Gemini 200 (200 MHz) spectrometer. TMS was used as the internal

**TABLE I** Selected Bond Length and Angles in **7c**

| Bond | $d$ (Å) | Angle | $\omega$ (°) |
|---|---|---|---|
| S(1)-C(2) | 1.758(5) | C(2)S(1)C(7) | 90.3(2) |
| S(1)-C(7) | 1.747(5) | O(1)P(1)O(2) | 101.6(2) |
| P(1)-O(1) | 1.541(4) | O(1)P(1)N(1) | 110.5(2) |
| P(1)-O(2) | 1.575(4) | O(2)P(1)N(1) | 112.1(2) |
| P(1)-N(1) | 1.607(4) | O(1)P(1)N(2) | 113.1(2) |
| P(1)-N(2) | 1.602(4) | O(2)P(1)N(2) | 104.2(2) |
| N(1)-C(1) | 1.339(6) | N(1)P(1)N(2) | 114.5(2) |
| N(2)-C(3) | 1.324(6) | P(1)N(1)C(1) | 114.1(4) |
| N(3)-C(1) | 1.375(6) | P(1)N(2)C(3) | 117.6(4) |
| N(3)-C(7) | 1.407(5) | C(1)N(3)C(7) | 115.0(4) |
| N(4)-C(7) | 1.284(6) | C(7)N(4)C(20) | 118.5(4) |
| N(4)-C(20) | 1.394(6) | C(14)N(5)C(20) | 124.8(4) |
| N(5)-C(14) | 1.412(6) | N(1)C(1)C(2) | 127.8(5) |
| N(5)-C(20) | 1.279(6) | N(3)C(1)C(2) | 111.9(4) |
| C(1)-C(2) | 1.384(6) | S(1)C(2)C(1) | 112.6(4) |
| C(2)-C(3) | 1.384(6) | C(1)C(2)C(3) | 120.1(5) |
| C(20)-C(21) | 1.485(6) | N(2)C(3)C(2) | 124.9(5) |
| | | S(1)C(7)N(3) | 110.2(3) |
| | | S(1)C(7)N(4) | 131.3(4) |
| | | N(4)C(20)N(5) | 119.4(4) |
| | | N(4)C(20)C(21) | 111.4(4) |
| | | N(5)C(20)C(21) | 129.1(4) |



standard. Crystallographic measurements of compound **7c** were performed at 18°C on an Enraf-Nonius CAD 4 diffractometer operating in the $\omega$-2$\Theta$ scan mode (the ratio of the scanning rates $\omega/2\Theta = 1.2$). Intensity data were collected within the range $2 < \Theta < 60^\circ$ ($0 < h < 19$, $0 < k < 13$, $-30 < 1 < 30$) using graphite monochromated $CuK_\alpha$ radiation. The intensities of 4329 reflections (3954 unique reflections, $R = 0.023$) were measured. Data were corrected for Lorentz and polarization effects. An empirical absorption correction based on azimutal scan data was also applied.[13] The structure was solved by direct method and refined by full-matrix least-squares technique in an anisotropic approximation usig the CRYSTALS program package.[14] In the refinement, 2579 reflections with $I > 4\sigma(I)$ were used. All hydrogen atoms were located in the difference Fourier maps and included in the final refinement with fixed positional and thermal parameters. Convergence was obtained at $R = 0.056$, $R_w = 0.056$, and GOF = 1.107 (343 refined parameters; obs/variabl. 7.5; the largest and minimal peaks in the final difference map, 0.51 and $-0.37$ e/Å$^3$). The weighting scheme $\omega = l/(0.01F_0^2 + 12\sigma(F_0^2) + 1)$ was used.

## 1,2,4-Thiadiazol-5(2*H*)-imine Hydrobromides 1a–d · HBr

To a stirred solution or a suspension of the appropriate N-imidoylthiourea of the general formula PhN=C(Ar)NHC(S)NHR* (50 mmol) in $CHCl_3$ (50 mL), pyridine (50 mmol) was added in one portion. Then a solution of bromine (50 mmol) in $CHCl_3$ (25 mL) was added dropwise in 40 min. The mixture was stirred for 1 h and the precipitated salt was filtered off and washed with MeOH. The filtrate was concentrated in vacuo and the residue was washed with MeOH to give an additional amount of the product.

Compound **1a · HBr**: yield 90%, m.p. 217°C dec. (AcOH) (lit. 207–208°C,[16] 230–232°C[17]). Found: Br 19.05, N 10.06, S 7.76%. Calcd for $C_{20}H_{15}N_3S \cdot HBr$. Br 19.47, N 10.24, S 7.81%.

Compound **Ib · HBr**: yield 91%, m.p. 216°C dec. (AcOH). Found: Br 19.11, N 9.72, S 7.59%. Calcd for $C_{21}H_{17}N_3S \cdot HBr$: Br 18.83, N 9.90, S 7.56%.

Compound **1c · HBr**: yield 95% m.p. 218°C dec. ($ClCH_2CH_2Cl$). Found: Br 18.03, N 9.63, S 7.24%. Calcd for $C_{21}H_{17}N_3OS \cdot HBr$: Br 18.15, N 9.54, S 7.28%.

Compound **ld · HBr**: yield 93% m.p. 206°C dec. (EtOH). Found: Br 18.31, N 9.97, S 7.52%. Calcd for $C_{21}H_{17}N_3S \cdot HBr$: Br 18.83, N 9.90, S 7.56%.

## Free 1,2,4-Thiadiazol-5(2*H*)-imines 1a–c

To a suspension of salt **1a–c · HBr** (20 mmol) in EtOH (40 mL), triethylamine (22 mmol) was added. The mixture was stirred for 4 h and the product was filtered off.

Compound **1a**: yield 88%, m.p. l38°C (EtOH) (lit. 143–146°C,[17] 142–143°C[18]). Found: N 12.86, S 9.78%. Calcd for $C_{20}H_{15}N_3S$: N 12.76, S 9.73%.

Compound **1b**: yield 94%, m.p. 148°C (EtOH). Found: N 12.32, S 9.47%. Calcd for $C_{21}H_{17}N_3S$: N 12.23, S 9.34%.

Compound **1c**: yield 88%, m.p. 139°C (EtOH). Found: N 11.60, S 8.94%. Calcd for $C_{21}H_{17}N_3OS$: N 11.69, S 8.92%.

## 4-Amino-5-cyano-2,3-dihydro-2-imidoylimino-3-phenylthiazoles 2a–c

A stirred mixture of **1a–c** (20 mmol), malononitrile (20 mmol) and dioxane (10 mL) was heated under reflux for 3 h, cooled, and filtered to

*N-Imidoylthioureas were prepared using the general procedure developed by J. Goerdeler and coworkers.[15]

separate the product which was washed with dioxane and dried at 100°C.

Compound **2a**: yield 88%, m.p. 208°C dec. (MeCN). IR ($CH_2Cl_2$, $cm^{-1}$) 2190 ($\nu$ C≡N), 3380, 3460 ($\nu$ $NH_2$). Found: C 70.08, H 4.65, N 17.53, S 8.02%. Calcd for $C_{23}H_{17}N_5S$: C 69.85, H 4.33, N 17.71, S 8.11%.

Compound **2b**: yield 76%, m.p. 215°C dec. (MeCN). IR (KBr, $cm^{-1}$) 2200 ($\nu$ C=N), 3220–3380 ($\nu$ $NH_2$). $^1H$ NMR (200 MHz, DMSO-D6) $\delta$ 2.18 (s, $CH_3$), 6.69 (d, $^3J_{HH} = 11$ Hz, 2 Har.), 6.84 (s, $NH_2$), 6.92–7.05 (m, 5 Mar.), 7.21 (t, $^3J_{HH} = 11$ Hz, 2 Har.), 7.47–7.60 (m, 5 Har.). Found: C 71.48, H 4.80, N 17.41, S 7.87%. Calcd for $C_{24}H_{19}N_5S$: C 70.39, H 4.68, N 17.10, S 7.83%.

Compound **2c**: yield 75%, m.p. 219°C dec. (dioxane). IR (KBr, $cm^{-1}$) 2200 ($\nu$ C≡N), 3220, 3270, 3340, 3460 ($\nu$ $NH_2$). $^1H$ NMR (200 MHz, DMSO-D6) $\delta$ 3.66 (s, $CH_3$), 6.67–6.73 (m, 4 Har.), 6.83 (s, $NH_2$), 6.95–7.27 (m, 5 Har.), 7.48–7.62 (m, 5 Har.). Found: C 66.91, 4.61, N 16.59, S 7.42%. Calcd for $C_{24}H_{19}N_5OS$: C 67.74, H 4.50, N 16.46, S 7.54%.

## 4-Amino-3-benzyl-5-cyano-2,3-dihydro-2-(N-phenylbenzimidoyl)iminothiazole 2d

To a stirred mixture of salt **1d · HBr** (15 mmol) and malononitrile (15 mmol) in $CH_2Cl_2$ (20 mL), a solution of triethylamine (15 mmol) in $CH_2Cl_2$ (5 mL) was added dropwise. The reaction mixture was stirred for 5 h and the precipitated product was filtered off and washed with water; yield 75%, m.p. 210°C dec. (MeCN). IR ($CH_2Cl_2$, $cm^{-1}$) 2180 ($\nu$ C≡N), 3280–3400 ($\nu$ $NH_2$). Found: C 70.16, H 4.53, N 16.81, S 7.65%. Calcd for $C_{24}H_{19}N_5S$: C 70.39, H 4.68, N 17.10, S7.83%.

## 2,2,4-Trichloro-6,7-dihydro-6-imidoyliminothiazolo-[4,5-*d*][1,3,2]diaza-$\lambda^5$-phosphorine Hydrochlorides 3a–d

A stirred mixture of **2a–d** (4 mmol) and $PCl_5$ (4.2 mmol) in anhydrous chlorobenzene (6 mL) was heated at 100°C for 1 h. After cooling, the precipitate was separated and dried in vacuo to give the product in quantitative yield. Only compound **3b** was analytically pure; m.p. 192°C dec. Found: Cl 25.08, N 11.78, P 5.30, S 5.41%. Calcd for $C_{24}H_{17}Cl_3N_5PS \cdot HCl$: Cl 24.39, N 12.05, P 5.33, S 5.52%.

## 7-Benzyl-6,7-dihydro-2,2,4-tris(methylamino)-6-(N-phenylbenzimidoyl)imino-thiazolo-[4,5-*d*][1,3,2]diaza-$\lambda^5$-phosphorine 4d

Through a suspension of **3d** (1.6 mmol) in anhydrous benzene (10 mL), methylamine was bubbled during 2 h. The resulting mixture was

allowed to stand for a further 10 h then the precipitate was separated and washed with water to obtain the pure product; yield 75%, m.p. 182°C dec. (MeCN). $^1$H NMR (300 MHz, DMSO-$D_6$) $\delta$ 2.33 (dd, $^3J_{HH} = 13$ Hz, $^3J_{HH} = 5$ Hz, 2 $CH_3$); 2.64, 2.76 (d, 1:5, $^3J_{HH} = 3$ Hz, $CH_3$); 4.33 (br. s, 2 NH); 4.98, 5.35 (s, 1:5, $CH_2$); 6.38–7.71 (m, 3 $C_6H_5$). Found: N 21.08, P 5.72, S, 5.91%. Calcd for $C_{27}H_{29}N_8PS$: N 21.20, P 5.86, S 6.06%.

## 2,2-Dichloro-6,7-dihydro-6-(4-methyl-N-phenylbenzimidoyl)imino-4-morpholino-7-phenylthiazolo[4,5-*d*][1,3,2]diaza-λ$^5$-phosphorine 5b

To a suspension of **3b** (1.5 mmol) in anhydrous benzene (8 mL), morpholine (12 mmol) was added in one portion. The reaction mixture was stirred for 2 h then the precipitate was filtered, washed with water, and dried in vacuo over $P_20_5$; yield 72%, m.p. 213°C (MeCN). $^1$H NMR (300 MHz $CDCl_3$) $\delta$ 2.24 (br. s, $CH_3$), 3.76 (br. s, 2 $CH_2$), 3.90 (br. s, 2 $CH_2$), 6.80–7.74 (m, 2 $C_6H_5$, $C_6H_4$). Found: Cl 12.16, N 13.83, S 5.44%. Calcd for $C_{28}H_{25}Cl_2N_6OPS$: Cl 11.91 N 14.11, S 5.38%.

## 6,7-Dihydro-6-(4-methyl-N-phenylbenzimidoyl)imino-2,2,4-trimorpholino-7-phenylthiazolo[4,5-*d*][1,3,2]diaza-λ$^5$-phosphorine 6b

A stirred mixture of **3b** (1.5 mmol) and morpholine (18 mmol) in anhydrous benzene (8 mL) was heated under reflux for 5 h, cooled, and filtered. The filtrate was concentrated in vacuo and the residue was treated with EtOH (10 mL) to crystallize the product; yield 79%, m.p. 174°C (EtOH). $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 2.23 (br. s, $CH_3$), 3.01 (br. s, 4 $CH_2$), 3.58 (br. s, 4 $CH_2$), 3.74 (br. s, 4 $CH_2$), 6.79–7.80 (m, 2 $C_6H_5$, $C_6H_4$). Found: C 61.47, H 6.00, N 16.32, P 4.41, S 4.63%. Caled for $C_{36}H_{41}N_8O_3PS$: C 61.87, H 5.91, N 16.08, P 4.44, S 4.60%.

## 6,7-Dihydro-6-imidoylimino-2,2,4-trimethoxy-7-phenylthiazolo[4,5-*d*][1,3,2]diaza-λ$^5$-phosphorines 7a,c

To a suspension of **3a**,**c** (2 mmol) in dioxane (5 mL), a solution of 4.3 M MeONa/MeOH (1.9 mL) in dioxane (3 mL) was added dropwise. The reaction mixture was stirred for 2 h and filtered. The filtrate was concentrated in vacuo and the residue was treated with MeOH to afford the crystalline product.

Compound **7a**: yield 53%, m.p. 171°C (MeCN). $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 3.62 (d, $^3J_{HH} = 12$ Hz, 2 $CH_3$); 3.83, 3.96 (s, 1:2, $CH_3$);

6.79–7.89 (m, 3 $C_6H_5$). Found: N 13.36, P 5.78, S 6.23%. Calcd for $C_{26}H_{24}N_5O_3PS$: N 13.53, P 5.98, S 6.20%.

Compound **7c**: yield 77%, m.p. 186°C (AcOMe). $^1$H NMR (300 MHz, DMSO-$D_6$, 19°C) $\delta$ 3.57 (d, $^3J_{HP} = 12$ Hz, 2 $CH_3$); 3.67, 3.80 (s, 4:1, $CH_3$); 3.80, 3.91 (s, 1:4, $CH_3$); 6.69–7.79 (m, 2 $C_6H_5$, $C_6H_4$). $^1$H NMR (300 MHz, DMSO-$D_6$, 90°C) $\delta$ 3.57 (d, $^3J_{HP} = 12$ Hz, 2 $CH_3$), 3.72 (s, $CH_3$), 3.88 (s, $CH_3$), 6.75–7.53 (m, 2 $C_6H_5$, $C_6H_4$). Found: N 13.03, P 5.53, S 5.91%. Calcd for $C_{27}H_{26}N_5O_4PS$: N 12.79, P 5.66, S 5.86%. A single crystal for x-ray analysis was prepared by crystallization from MeCN: size $0.09 \times 0.29 \times 0.31$ mm, monoclinic, space group $C2/c$, $Z = 8$, $a = 17.497(3)$, $b = 12.174(3)$, $c = 27.112(1)$ Å, $\beta = 112.26(1)°$, $V = 5344.9$ Å$^3$, $D_c = 1.36$ g·cm$^{-3}$, $\mu = 19.75$ cm$^{-1}$,*.

## 2,2-Dibutoxy-6,7-dihydro-6-(4-methyl-N-phenylbenzimidoyl)imino-4-morpholino-7-phenylthiazolo[4,5-*d*][1,3,2]diaza-$\lambda^5$-phosphorine 8b

To a suspension of **5b** (0.4 mmol) in dioxane (5 mL), a solution of 1.9 M BuONa/BuOH (0.6 mL) was added. The reaction mixture was refluxed for 1.5 h. After cooling, the solvent was evaporated in vacuo and the residue was treated with EtOH (5 mL) to afford the crystalline product; yield 55%, m.p. 126°C (EtOH). $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 0.88 (m, 2 $CH_3$), 1.36 (m, 2 $CH_2$), 1.58 (m, 2 $CH_2$), 2.23 (br. s, $CH_3$) 3.79 (m, 6 $CH_2$), 6.79–7.80 (m, 2 $C_6H_5$, $C_6H_4$). Found: C 64.41, H 6.62, N 12.82, P 4.56, S 4.86%. Calcd for $C_{36}H_{43}N_6O_3PS$: C 64.45, H 6.46, N 12.53, P 4.62, S 4.78%.

## REFERENCES

[1] F. G. Mann, in *The Chemistry of Heterocyclic Compounds*, edited by A. Weissberger and E. C. Taylor (Wiley-Interscience, New York, 1970), 2nd ed., part I, p. 244.
[2] E. A. Romanenko, Yu. P. Egorov, and P. P. Kornuta, *Spectrosc. Lett.*, **6,** 363 (1973).
[3] E. A. Romanenko, Yu. P. Egorov, and P. P. Kornuta, *Spectrosc. Lett.*, **6,** 621 (1973).
[4] E. P. Kornuta, L. I. Deriy, A. I. Kalenskaya, and V. I. Shevchenko, *Zh. Obshch. Khim.*, **46,** 1482 (1976).
[5] P. P. Kornuta and L. S. Kuzmenko, *Zh. Obshch. Khim.*, **48,** 799 (1978).
[6] P. P. Kornuta, L. S. Kuzmenko, and L. N. Markovskiy, *Zh. Obshch. Khim.*, **49,** 2201 (1979).
[7] P. P. Kornuta, L. S. Kuzmenko, and V. I. Kalinin, *Zh. Obshch. Khim.*, **50,** 1313 (1980).

*Full set of crystallographic data has been deposited at the Cambridge Crystallographic Data Centre (CCDC 166902).

[8] E. A. Romanenko, Yu. E. Sapozhnikoy, and Ya. B. Yasman, *Teor. Eksp. Khim.*, **20,** 539 (1984).
[9] V. I. Tolstikova, N. D. Bodnarchuk, B. B. Gavrilenko, and G. I. Derkach, *Fiziol. Aktiv. Veshchestva, Respub. Mezhvedom. Sb.*, N 3, 30 (1971); *C.A.*, **76,** 149504 (1972).
[10] I. Neda, T. Kaukorat, R. Schmutzler, U. Neimeyer, B. Kutscher, J. Pohl, and J. Engel, *Phosphorus, Sulfur, and Silicon*, **162,** 81 (2000).
[11] M. A. Rensky, V. S. Zyabrev, and B. S. Drach, *Zh. Obshch. Khim.*, **72,** 1931 (2002).
[12] P. P. Kornuta, A. I. Kalenskaya, O. P. Lobanov, and V. I. Shevchenko, *Zh. Obshch. Khim.*, **43,** 261 (1973).
[13] A. C. T. North, D. C. Phillips, and F. S. Mathews, *Acta Crystallogr.*, **24A,** 351 (1968).
[14] D. J. Watkin, C. K. Prout, J. R. Carruthers, and P. W. Betteridge, *CRYSTALS*, *Issue 10* (Chemical Crystallography Laboratory, University of Oxford, Oxford, 1996).
[15] J. Goerdeler and D. Weber, *Chem. Ber.*, **101,** 3475 (1968).
[16] G. Barnikow and H. Ebeling, *Z. Chem.*, **12,** 130 (1972).
[17] M. D. Nair and J. A. Desai, *Indian J. Chem.*, **19B,** 335 (1980).
[18] J. Goerdeler and W. Löbach, *Chem. Ber.*, **112,** 517 (1979).