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Intramolecular Cyclization of *N*-Arylphosphinimidic Isocyanates – Novel Approach to a 4a,8a-Dihydro-1,3,2λ^5-benzodiazaphosphinin-4(3*H*)-one System

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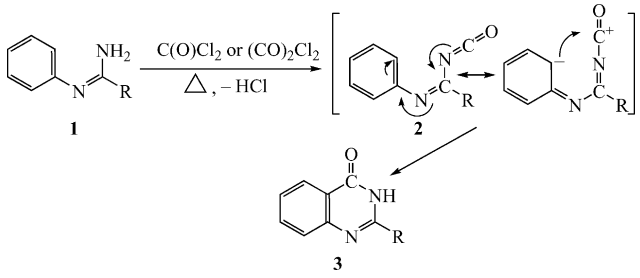
A previously unknown type of 1,3,2λ^5-benzodiazaphosphinin-4(3*H*)-ones was obtained by intramolecular heterocyclization of novel *N*-arylphosphinimidic isocyanates.

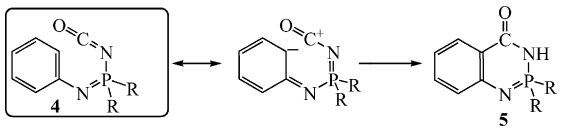
Introduction

Phosphorus-containing heterocycles are of great interest as model compounds for fundamental investigations,[1] ligands in metal-complex catalysis,[2] and physiologically active compounds or precursors for their synthesis,[3] and they are also used in other fields. However, in comparison to sulfur- and nitrogen-containing analogues, the structural diversity of numerous phosphorus-containing heterocycles remains highly limited to date,[4] because of significant limitations in the known methods of their synthesis. Therefore, the search for phosphorus analogues that are effective in the synthesis of other heterocycles is not solely of great theoretical interest but also of practical importance. Our attention focused on widely used reactions involving the intramolecular cyclization of imino isocyanates of type **2** (Scheme 1) leading to the corresponding quinazolin-4(3*H*)-ones **3**. For the first time this conversion was observed when the corresponding amidines **1** were treated with phosgene or oxalyl chloride.[5]

The intermediate **2** cannot be isolated as it undergoes cyclization to 4(3*H*)-quinazolinone (**3**) in situ. Later, other reactions leading to intermediates **2** were found;[6] however, in all cases the corresponding quinazolin-4(3*H*)-ones **3** were the final reaction products, excluding the cases where the heterocyclization process was impossible because of the presence of substituents at both the "*ortho*" positions of the phenyl ring. It could be assumed that the conversion, similar to the one presented in Scheme 1, would be possible when the carbon atom adjacent to the isocyanate group is replaced by a phosphorus atom (see Scheme 2), since electron-donating properties of the phosphinimine fragment and *C*-carbamoylating properties of phosphorus isocyanates are well known.[7–10] The route proposed leads to a previously unavailable type of 1,3,2λ^5-benzodiazaphosphinin-4(3*H*)-one **5** containing an endocyclic P=N double bond (for the synthesis of the closest topological analogues see refs.[11–13]).



Scheme 1.



Scheme 2.

Results and Discussion

It is important to note that compounds **6** belong to the least studied type of phosphorus isocyanates, i.e. phosphin-

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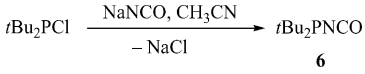
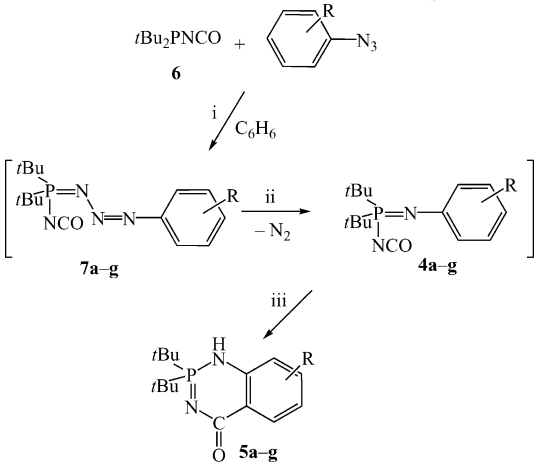
imidic isocyanates. There are only a few representatives of this type of compounds described in the literature,[14–17] and their properties are little studied. For the synthesis of *N*-arylphosphinimidic isocyanates we used an imination reaction with trivalent isocyanate **6**, which exists as a monomer unlike the majority of trivalent phosphorus isocyanates,[18] probably because of the steric effect of the *tert*-butyl groups. The previously unreported compound **6** is a colorless liquid distillable under vacuum (Scheme 3).



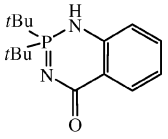
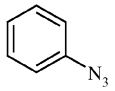
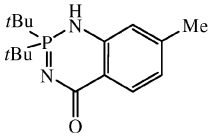
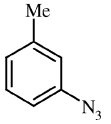
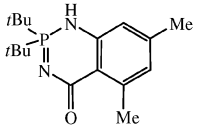
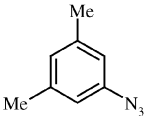
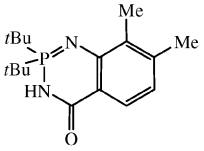
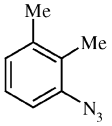
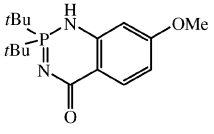
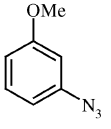
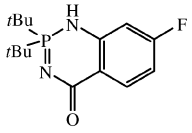
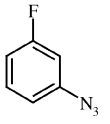
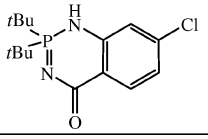
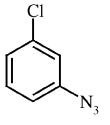
Scheme 3.

We established that the reaction of isocyanate **6** with a set of aryl azides (Scheme 4, Table 1) in benzene at 20–25 °C indeed leads to the formation of the corresponding 2,2-di-*tert*-butyl-4a,8a-dihydro-1,3,2λ^5-benzodiazaphosphinin-4(3*H*)-ones. It can be seen from Table 1 that this is a general reaction which proceeds quite easily as in the case of unsubstituted phenyl azide. In the case of phenyl azide derivatives the reaction rate and yield depend on both the electron effects of the substituents and the steric factors. In the case of halogen-containing aryl azides the reaction slows down and the yields of the cyclization products decrease. According to data on the Staudinger reaction, the



Scheme 4.

Table 1. Compounds **5a**–**g**: structure, reagents, and reaction conditions.

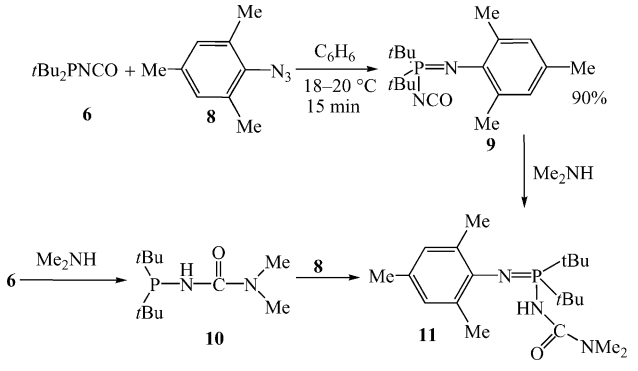
Compound	Product	Starting azide	Yield [%][a]	Reaction time [h]
5a			47	72
5b			50	15
5c			33	48
5d			23	24
5e			52	15
5f			25	96
5g			18	96

[a] Isolated yield.

interaction of isocyanate **6** with azides proceeds via the corresponding triazenes **7** confirmed by ^{31}P NMR spectroscopy, which suggest that steps (i) and (ii) proceed quickly and that the heterocyclization step (iii) is the rate-determining one.

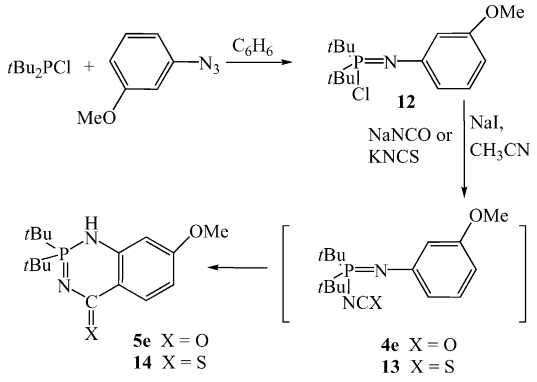
Compounds **5a**–**g** are colorless crystalline substances, slightly soluble in most organic solvents, and melting with decomposition at 300 °C.

The reaction of isocyanate **6** with 2,4,6-trimethylphenyl azide (**8**), which excludes the possibility of further heterocyclization, gives stable phosphinimidic isocyanate **9** in high yield (Scheme 5). Compound **9** is a colorless, distillable liquid that is stable under inert conditions. As with other isocyanates it easily reacts with dimethylamine forming the corresponding *N*-phosphorylated urea **11**, which was also synthesized by another route (Scheme 5). The result obtained is an additional confirmation of the intermediate compounds **4a**–**g** in the transformation of **4**.



Scheme 5.

The introduction of the isocyanate group into chlorophosphinimide can serve as an alternative method for the synthesis of phosphinimidic isocyanates, because prolonged heating of compound **12** in the presence of sodium cyanate gives compound **5e** in high yield; the use of potassium rhodanide in the reaction allowed us to successfully synthesize its thio analogue **14** (Scheme 6).



Scheme 6.

The structure of compound **5e** was confirmed by X-ray diffraction studies (Figure 1).

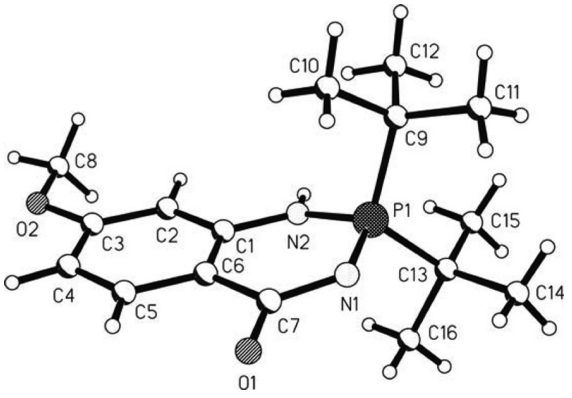


Figure 1. Structure of compound **5e** in the crystal.

Conclusions

Novel di-*tert*-butylphosphinous isocyanate exists as a monomer because of steric factors. It was iminated with a set of aryl azides to give poorly known phosphinimidic isocyanates that spontaneously cyclized into previously unknown 4a,8a-dihydro-1,3,2λ^5-benzodiazaphosphinin-4(3*H*)-one systems. An alternative synthetic approach to 4a,8a-dihydro-1,3,2λ^5-benzodiazaphosphinin(e)-4(3*H*)-(thi)-ones has been proposed.

Experimental Section

General: NMR spectra were recorded with a Varian VXR-300 spectrometer: ^1H NMR (300 MHz) spectra were recorded with TMS as an internal standard; ^{31}P NMR (121 MHz) spectra were recorded with 85% H_3PO_4 as an external standard. IR spectra were recorded with a UR-20 spectrometer for samples in KBr discs. Mass spectra were recorded with an Agilent 1100 Series LC/MSD system. All reactions were carried out under dry argon using Schlenk-type glassware. All solvents, including deuterated solvents used for NMR spectroscopy, were dried and distilled prior to use.

X-ray Crystal Structure Analysis of 5e: The crystals of compound **5e** are monoclinic; at 20 °C a = 17.801(2), b = 7.869(1), c = 12.193(2) Å, β = 100.83(1)°, V = 1677.5(4) Å^3, M_r = 308.35, Z = 4, space group $P2_1/c$, $d_{calcd.}$ = 1.221 g/cm^3, μ(Mo-K_α) = 0.170 mm^{-1}, $F(000)$ = 664. Unit cell parameters and intensities of 8735 reflections (3845 independent, R_{int} = 0.047) were measured with an "Xcalibur-3" diffractometer (Mo-K_α radiation, CCD detector, graphite monochromator, ω-scanning, $2\theta_{max}$ = 55°). The structure was solved by direct methods using the SHELXTL[19] program. Positions of hydrogen atoms were located from difference synthesis of electron density. Hydrogen atoms of butyl groups were refined by the "riding" model with $U_{iso} = 1.5U_{eq}$ of the carrier non-hydrogen atom; the rest of the hydrogen atoms were refined within an isotropic approximation. The structure was refined against F^2 by a full-matrix least-squares procedure with an anisotropic approximation for the non-hydrogen atoms where wR_2 = 0.142 for 3812 reflections [R_1 = 0.061 for 2634 reflections with $F > 4\sigma(F)$, S = 1.099]. CCDC-668559 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Di-*tert*-Butylphosphinous Isocyanate (6): A mixture of *t*Bu$_2$PCl (3.74 g, 20 mmol) and sodium cyanate (1.95 g, 30 mmol) was

stirred at 70 °C for 20 h. The solvent was then evaporated at 25–30 °C/10 Torr. The residue was distilled into a cold receiver (ca. –20 °C), b.p. 22–25 °C/0.05 Torr. The pure product was obtained by additional distillation under the same conditions. Yield 3.18 g (85%). IR (KBr): $\tilde{\nu}$ = 2280 cm^{-1}. ^1H NMR (300 MHz, C_6D_6): δ = 1.06 (d, J = 12.5 Hz) ppm. ^{31}P NMR (121 MHz): δ = 103.3 ppm.

Compounds 5a–g. General Procedure: A solution of the corresponding aryl azide (3 mmol) in C_6H_6 (3 mL) was added to a frozen solution of compound **6** (0.56 g, 3 mmol) in C_6H_6 (3 mL). During the melting of the frozen benzene, the evolution of N_2 was observed; after a few hours, the reaction product began to precipitate. After the time noted in Table 1 (^{31}P NMR control) the reaction mixture was filtered, the product was washed with benzene (3 × 1 mL), and dried in vacuo. For additional analytical and spectroscopic data of compounds **5a**–**g** see Supporting Information.

Compound 5a: ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.23 (d, J = 16.2 Hz, 18 H), 6.81 (t, J = 7.8 Hz, 1 H), 6.98 (d, J = 7.8 Hz, 1 H), 7.98 (t, J = 8.1 Hz, 1 H), 7.81 (d, J = 6.9 Hz, 1 H), 8.06 (d, J = 10.8 Hz, 1 H, NH) ppm. ^{31}P NMR (121 MHz): δ = 52.4 ppm.

Compound 5b: ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.23 (d, J = 15.3 Hz, 18 H), 2.23 (s, 3 H), 6.63 (d, J = 7.9 Hz, 1 H), 6.79 (s, 1 H), 7.68 (d, J = 8.1 Hz, 1 H), 8.10 (d, J = 11.1 Hz, 1 H, NH) ppm. ^{31}P NMR (121 MHz): δ = 51.5 ppm.

Compound 5c: ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.21 (d, J = 15.0 Hz, 18 H), 2.16 (s, 3 H), 2.50 (s, 3 H), 6.45 (s, 1 H), 6.68 (s, 1 H), 7.71 (d, J = 6.6 Hz, 1 H, NH) ppm. ^{31}P NMR (121 MHz): δ = 51.3 ppm.

Compound 5d: ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.22 (d, J = 15.0 Hz, 18 H), 2.09 (s, 3 H), 2.27 (s, 3 H), 6.83 (dd, ^3J = 8.0, ^4J = 2.5, 1 H), 7.34 (t, J = 8.0 Hz, 1 H), 7.47 (br. s, 1 H) ppm. ^{31}P NMR (121 MHz): δ = 38.6 ppm.

Compound 5e: ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.22 (d, J = 15.3 Hz, 18 H), 3.74 (s, 3 H), 6.40 (dd, ^3J = 8.7, ^4J = 2.1, 1 H), 6.53 (d, J = 2.1 Hz, 1 H), 7.73 (d, J = 8.7 Hz, 1 H), 8.23 (d, J = 10.8 Hz, 1 H, NH) ppm. ^{31}P NMR (121 MHz): δ = 52.2 ppm.

Compound 5f: ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.23 (d, J = 14.7 Hz, 18 H), 6.64 (d, J = 11.5 Hz, 1 H), 7.76 (dd, $^3J_{HF}$ = 11.7, $^3J_{HH}$ = 8.7, 1 H), 7.84 (t, $^3J_{HH} \approx {}^4J_{HF}$ = 8.7, 1 H), 8.06 (d, J = 10.8 Hz, 1 H, NH) ppm. ^{31}P NMR (121 MHz): δ = 52.8 ppm.

Compound 5g: ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.22 (d, J = 13.5 Hz, 18 H), 6.87 (d, J = 8.1 Hz, 1 H), 7.03 (s, 1 H), 7.43 (d, J = 8.1 Hz, 1 H), 8.44 (d, J = 10.8 Hz, 1 H, NH) ppm. ^{31}P NMR (121 MHz): δ = 52.8 ppm.

2,2-Di-*tert*-butyl-7-methoxy-1,3,2λ^5-benzodiazaphosphinin-4(3*H*)-one (5e). Procedure 2: A mixture of compound **12** (1.52 g, 5 mmol), sodium cyanate (0.92 g, 6.1 mmol), NaI (10 mg), and CH_3CN (8 mL) was heated and stirred in a pressure tube at 100 °C for 110 h. The solvent was then removed in vacuo, the residue was washed with water (4 × 10 mL), dried, and then washed with C_6H_6 (3 × 20 mL). Yield 1.40 g (90%).

***P*,*P*-Di-*tert*-butyl-*N*′′′-mesitylphosphinimidic Isocyanate (9):** A solution of azide **8** (0.48 g, 4 mmol) in C_6H_6 (4 mL) was added to a frozen solution of compound **6** (0.79 g, 4 mmol) in C_6H_6 (5 mL). During the melting of the frozen benzene, the evolution of N_2 and a strong exothermic effect were observed. The temperature of the reaction mixture was kept in the range 15–20 °C. The reaction was complete after 15 min, and the solvent was removed in vacuo. The product was extracted with pentane (2 × 10 mL), and, after the solvent was removed, the product was purified by vacuum distillation. B.p. 150–160 °C/0.005 Torr. Yield 1.15 g (90%). ^1H NMR (300 MHz, $CDCl_3$): δ = 1.33 (d, J = 15.0 Hz, 18 H), 2.24 (s, 3 H), 2.31 (s, 6 H), 6.83 (s, 2 H) ppm. ^{31}P NMR (121 MHz): δ = 54.9 ppm. MS: m/z = 320 $[M]^+$.

***N*-(Dimethylcarbamoyl)-*P*,*P*-dimethylphosphinous Amide (10):** A solution of dimethylamine (0.2 g, 4.4 mmol) in C_6H_6 (5 mL) was added to a solution of compound **6** (0.7 g, 3.7 mmol) in C_6H_6 (5 mL). After 20 min, the solvent was removed in vacuo, and the product was purified by recrystallization from diethyl ether at –10 °C. M.p. 109–110 °C. Yield 0.67 g (77%). ^1H NMR (300 MHz, $CDCl_3$): δ = 1.14 (d, J = 11.7 Hz, 18 H), 3.00 (s, 6 H), 4.66 (d, J = 8.7 Hz, 1 H) ppm. ^{31}P NMR (121 MHz): δ = 56.5 ppm.

***P*,*P*-Di-*tert*-butyl-*N*-(dimethylcarbamoyl)-*N*′-mesitylphosphinimidic Amide (11). (a):** A solution of dimethylamine (0.1 g, 2.2 mmol) in C_6H_6 (1 mL) was added to a solution of compound **9** (0.4 g, 1.25 mmol) in C_6H_6 (3 mL). After 24 h, the solvent was removed in vacuo, and the product was purified by recrystallization from hexane at –15 °C. Yield 0.35 g (78%). **(b):** A solution of compound **8** (0.43 g, 2.7 mmol) in C_6H_6 (2 mL) was added to a solution of compound **10** (0.62 g, 2.7 mmol) in C_6H_6 (4 mL). The reaction mixture was refluxed for 30 min, and the solvent was then removed in vacuo. After 15 h, the by-product precipitated and was removed, the mother liquor was concentrated in vacuo, and the residue was recrystallized from pentane (3 mL). M.p. 123–124 °C. Yield 0.32 g (33%). ^1H NMR (300 MHz, $CDCl_3$): δ = 1.27 (d, J = 13.6 Hz, 18 H), 2.18 (s, 6 H), 2.22 (s, 3 H), 2.59 (s, 6 H), 6.81 (s, 2 H), 9.05 (s, 1 H) ppm. ^{31}P NMR (121 MHz): δ = 53.9 ppm. MS: m/z = 366 $[M]^+$.

***P*,*P*-Di-*tert*-Butyl-*N*-(3-methoxyphenyl)phosphinimidic Chloride (12):** A solution of 1-azido-3-methoxybenzene (0.88 g, 5.9 mmol) in C_6H_6 (2 mL) was added to a solution of di-*tert*-butylphosphinous chloride (1.06 g, 5.9 mmol) in C_6H_6 (3 mL). After the exothermic reaction had proceeded for 30 min, the solvent was removed in vacuo, and the product was purified by distillation. B.p. 150 °C/0.005 Torr. Yield 1.60 g (90%). ^1H NMR (300 MHz, $CDCl_3$): δ = 1.41 (d, J = 17.1, 18 H), 3.76 (s, 3 H), 6.34 (d, J = 7.2 Hz), 6.5 (s), 6.54 (d, J = 7.8), 7.03 (t, J = 8.1 Hz) ppm. ^{31}P NMR (121 MHz): δ = 51.7 ppm.

2,2-Di-*tert*-butyl-7-methoxy-1,3,2λ^5-benzodiazaphosphinine-4(1*H*)-thione (14): A mixture of compound **12** (0.75 g, 2.5 mmol), potassium rhodanide (0.5 g, 5.1 mmol), NaI (10 mg), and CH_3CN (8 mL) was heated and stirred in a pressure tube at 125 °C for 4 h. The reaction mixture was then filtered, and the residue was washed with CH_3CN (3 × 5 mL). The mother liquor was concentrated in vacuo, and the product was washed with water (3 × 10 mL) and dried under reduced pressure. M.p. 310 °C. Yield 0.45 g (56%). ^1H NMR (300 MHz, [D$_6$]DMSO): δ = 1.24 (d, J = 14.1 Hz, 18 H), 3.77 (s, 3 H, OMe), 6.43 (dd, $^3J_{HH}$ = 9 Hz, 1 H, $^4J_{HH}$ = 2.4 Hz, 6-H) 6.47 (d, $^4J_{HH}$ = 2.4, 1 H, 8-H), 8.45 (d, $^2J_{PH}$ = 9.3 Hz, 1 H, NH), 8.62 (d, $^3J_{HH}$ = 9.0 Hz, 1 H, 5-H) ppm. ^{31}P NMR (121 MHz): δ = 46.0 ppm. MS: m/z = 323 $[M]^+$.

Supporting Information (see footnote on the first page of this article): C,H,N,P analytical data for compounds **5a–g** and crystal structure description for compound **5e**.

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