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$N \rightarrow C_2 \rightarrow C_3$ Migration of the Dichlorophosphino Group in the Synthesis of Phosphorylated NH-Pyrroles

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ABSTRACT: *A successive $N \rightarrow C_2 \rightarrow C_3$ migration of the dichlorophosphino group has been found to occur in phosphorylation of unsubstituted pyrrole with phosphorus trichloride. As a result of this migration, a number of hitherto unknown C-phosphorylated N-unsubstituted pyrroles have been obtained.* © 2008 Wiley Periodicals, Inc. Heteroatom Chem 19:671–676, 2008; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20495

INTRODUCTION

The literature lacks studies on the phosphorylation of unsubstituted pyrrole, whereas studies on halogenophosphines are scarce. Some evidence was reported for the reactions of potassium pyrrolide [1] and pyrrole in the presence of a base [2] with chlorophosphines, involving a pyrrole nitrogen atom, N-pyrrolylmagnesium bromide with phosphorus halides affording C_2-phosphorylated derivatives [3–7], as well as the reactions of 2,4-dimethylpyrroles and 2,3,5-trimethylpyrroles with chlorophosphites that occur nonselectively at the pyrrole NH group and the C_2 and C_3 atoms [8]. Several $N \rightarrow C_2$ and $C_2 \rightarrow C_3$ migrations of electrophilic groups (C=O, SO_2, P=O, and PBr_2) in pyrroles are also known [9–12].

It looks plausible that the successive $N \rightarrow C_2 \rightarrow C_3$ migration of phosphino groups takes place in phosphorylation of the unsubstituted pyrrole with phosphorus(III) halides. Moreover, a rearrangement of this kind could directly lead to 3-phosphorylated pyrroles, which bear an unsubstituted NH functionality [13] capable of further reactions.

RESULTS AND DISCUSSION

As found, phosphorylation of pyrrole **1** with phosphorus trichloride in dichloromethane in the presence of a base (1 mol of triethylamine or pyridine) proceeds via the following pathway: An initially formed N-phosphorylated intermediate, *N*-pyrrolyldichlorophosphine **2**, undergoes the $N \rightarrow C_2$ migration of the dichlorophosphino group in a matter of hours to give 2-pyrrolyldichlorophosphine **3** in which the $C_2 \rightarrow C_3$ migration of the same

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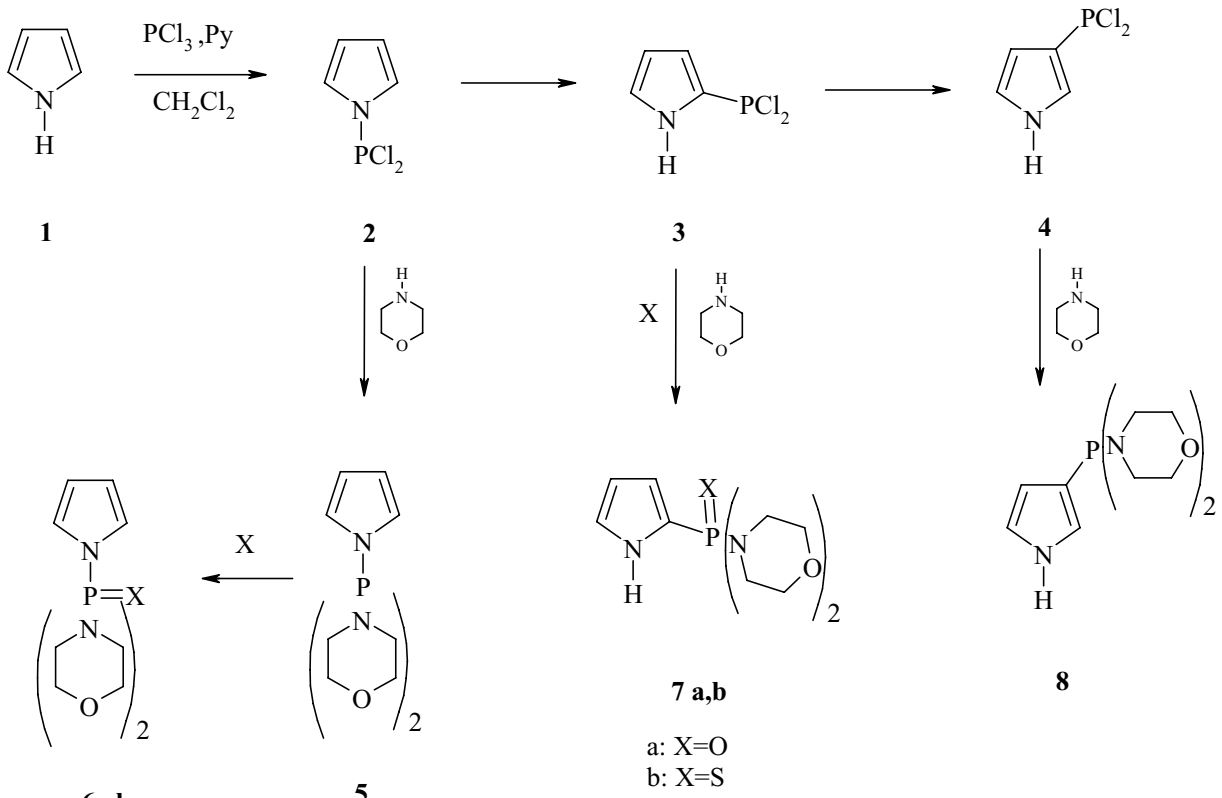
group occurs in a period of 3–5 days, leading to 3-pyrrolyldichlorophosphine **4**. Thus, we have revealed a successive N → C_2 → C_3 migration of the dichlorophosphino group in the pyrrole.

polar benzene, the rearrangement is slowed down significantly and requires 1 month to go to completion. Although in chloroform the migration rate is almost the same as in dichloromethane, but many

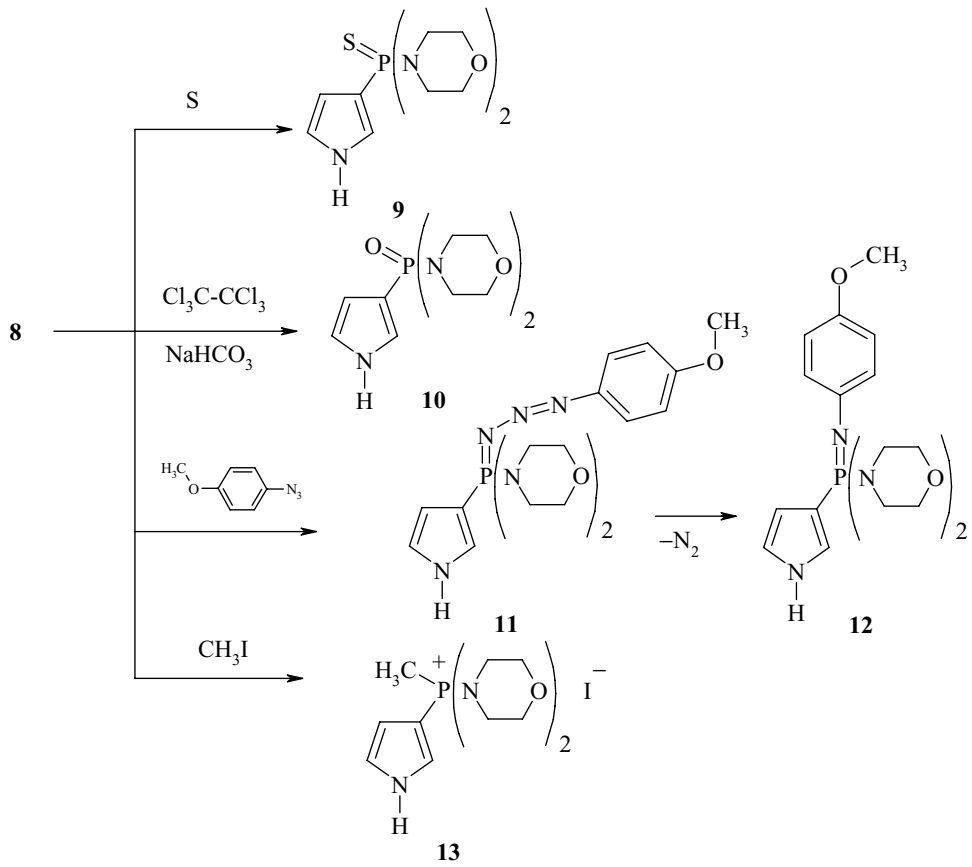


^{31}P NMR monitoring of the reaction between pyrrole **1** and phosphorus trichloride in the presence of triethylamine demonstrates that the signals of the N-phosphorylated intermediate **2** (148.4 ppm), 2-pyrrolyldichlorophosphine **3** (132.7 ppm), and 3-pyrrolyldichlorophosphine **4** (158.7 ppm) appear within 1, 2, and 5 h, respectively, after mixing the reagents. The signal of compound **4** is progressively enhanced and becomes a single one 5 days later. The rearrangement concerned is much accelerated if the reaction is conducted in the presence of pyridine. In this case, only trace amounts of the initial compound **2** can be detected by ^{31}P NMR spectroscopy, whereas **3** becomes the main product already after 1 h of reaction time and it is completely converted to **4** within 4 days.

It should also be noted that the nature of the solvent influences the migration rate of the dichlorophosphino group in the pyrrole ring. In non-

by-products are formed. We failed to carry out the N → C_2 → C_3 migration of the dichlorophosphino group in pyridine. A number of unidentified signals were observed in the ^{31}P NMR spectrum of the reaction mixture.

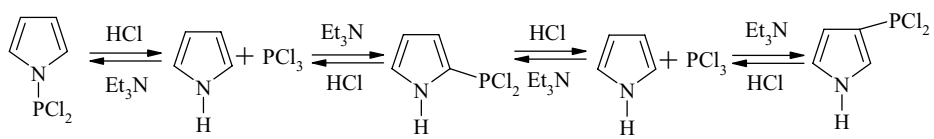
We failed to isolate pyrrolyldichlorophosphine**s** **2**, **3**, and **4** in the pure state because any isolation procedures caused polymerization of products. Two phosphorus(III) compounds, amides **5** and **8**, were isolated in the pure state; their structures were supported by ^{1}H, ^{13}C, and ^{31}P NMR spectra as well as by elemental analysis and high performance liquid chromatography. For unequivocal structural determination of products **2–4**, they were converted to stable phosphorus(V) compounds **6a**,**b**, **7a**,**b**, **9–12**, and phosphonium salt **13**. The phosphorus atom position in products **5**, **6a**,**b**, **7a**,**b**, and **8–13** was firmly established by ^{1}H, ^{13}C, and ^{31}P NMR spectra.



As shown previously, the $C_2 \rightarrow C_3$ migration of the dibromophosphino group in the N-methylpyrrole ring is catalyzed by pyridine hydrobromide [11]. We have also observed the effect of acidic impurities on the $N \rightarrow C_2 \rightarrow C_3$ migration of the dichlorophosphino group. If the reaction is run with freshly distilled phosphorus trichloride in deacidified dichloromethane, the migration rate is strongly reduced. Thus, the rearrangement is probably facilitated by hydrogen chloride present in the reaction mixture:

$$Et_3N^*HCl \rightleftarrows Et_3N + HCl$$

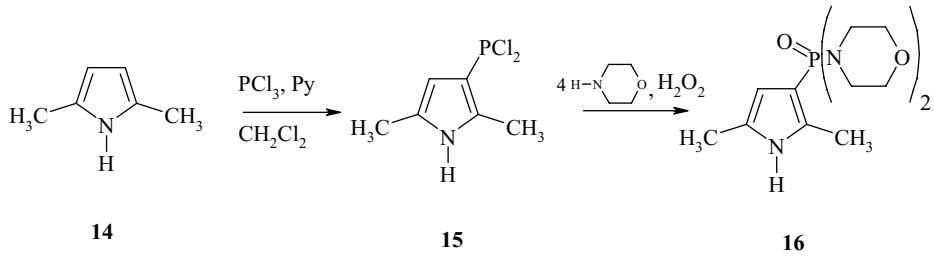
These results suggest the following scheme for the $N \rightarrow C_2 \rightarrow C_3$ migration of the dichlorophosphino group:



Attempted direct phosphorylation of pyrrole **1** with phosphorus tribromide resulted in the formation of polymeric products. On phosphorylation of pyrrole **1** with excess (3 eq.) phosphorus trichloride, only one dichlorophosphino group is introduced into the pyrrole nucleus and its $N \rightarrow C_2 \rightarrow C_3$ migration proceeds in a normal manner, as demonstrated above. It is also notable that N-phosphorylated pyrroles **6 a**,**b** could not be phosphorylated further even in pyridine with phosphorus tribromide, the most reactive phosphorylating agent.

We have also studied the action of phosphorus trichloride on 2,5-dimethylpyrrole **14**. In this case, the phosphorus atom directly attacks the C_3 atom of the pyrrole ring. Accordingly, the ^{31}P NMR spectra show no trace of the signal of the N-substituted product immediately after starting the reaction; instead, a signal at 158.5 ppm is observed that corresponds to 3-pyrrolyldichlorophosphine **15**. This reaction course is attributable to steric constraints at the nitrogen atom caused by two methyl groups; it may also result from the fact that the C_3 atom of

the pyrrole ring is the most nucleophilic center in compound **16**.



14 **15** **16**

CONCLUSION

The study on direct phosphorylation of N-unsubstituted pyrrole with phosphorus trichloride has revealed that the reaction involves a successive $N \rightarrow C_2 \rightarrow C_3$ migration of the dichlorophosphino group. As a result of the rearrangement found, a number of hitherto unknown C-phosphorylated NH-pyrroles have been obtained.

EXPERIMENTAL

^1H, ^{13}C, and ^{31}P NMR spectra were recorded on a Varian VXR-300 spectrometer (at 300, 75, and 121 MHz, respectively, 25°C), with TMS as internal standard for ^1H and ^{13}C signals, and 85% H_3PO_4 as external standard for ^{31}P signals. Liquid chromatography mass spectra were registered on an Agilent 1100 Series LC/MSD instrument.

1H-Pyrrol-1-yl[di(4-morpholyl)]phosphine **5**

To an ice-cooled and stirred solution of pyrrole **1** (0.01 mol) and pyridine (0.01 mol) in CH_2Cl_2 (60 mL), PCl_3 (0.01 mol) was added under dry argon. The reaction mixture was allowed to stand at room temperature for 1 h (until a ^{31}P NMR signal at 148.4 ppm was detected), followed by adding pentane (100 mL), cooling to 0°C, and decanting the pentane layer thrice under argon. To the residue dissolved in benzene, morpholine (0.05 mol) was added with ice-cooling and stirring. The reaction mixture was stirred at room temperature for 2 h, the precipitate of morpholine hydrochloride was filtered off under argon, and the filtrate was evaporated. Product **5** was isolated as a light-colored oil. Yield 78%. ^{31}P NMR ($CDCl_3$): δ 105.9. ^1H NMR ($CDCl_3$): δ 3.05 (m, 8H, N-CH_2), 3.63 (m, 8H, O—CH_2), 6.30 (s, 2H, $H_{3,4}$), 6.88 (s, 2H, $H_{2,5}$). m/z = 270 $[M]^+$. Anal. Calcd for $C_{12}H_{20}N_3O_2P$ (269): C 53.52, H 7.49, N 15.60. Found: C 53.50, H 7.46, N 15.57.

1H-Pyrrol-1-yl[di(4-morpholyl)]phosphine Oxide **6a**

To a stirred solution of amide **5** (0.01 mol) in benzene (30 mL), 50% H_2O_2 (0.03 mol) was added at room temperature. The reaction mixture was stirred for 1 h and then diluted with H_2O (10 mL). The organic layer was separated, dried over Na_2SO_4, and evaporated. Product **6a** was isolated as a light-colored oil. Yield 42%. ^{31}P NMR ($CDCl_3$): δ 9.9. ^1H NMR ($CDCl_3$): δ 3.00 (m, 8H, O—CH_2), 3.49 (m, 8H, N-CH_2), 6.26 (br s, 2H, $H_{3,4}$), 6.99 (br s, 2H, $H_{2,5}$). m/z = 286 $[M]^+$. Anal. Calcd. for $C_{12}H_{20}N_3O_3P$ (285): C 50.52, H 7.07, N 14.73. Found: C 50.50, H 7.09, N 14.70.

1H-Pyrrol-1-yl[di(4-morpholyl)]phosphine Sulfide **6b**

To a stirred solution of amide **5** (0.01 mol) in benzene (30 mL), elementary sulfur (0.01 mol) was added, followed by boiling the reaction mixture for 30 min. After evaporating the reaction mixture, the residue was treated with diethyl ether. The resulting precipitate was filtered off and recrystallized from diethyl ether. Yield 83%. ^{31}P NMR (DMSO-d_6): δ 64.4. ^1H NMR (DMSO-d_6): δ 3.02 (m, 8H, O—CH_2), 3.53 (m, 8H, N-CH_2), 6.33 (br s, 2H, $H_{3,4}$), 7.16 (br s, 2H, $H_{2,5}$).^{13}C NMR (DMSO-d_6): δ 45.95 (s, O—CH_2), 66.74 (s, N-CH_2), 112.65 (d, J_{CP} = 9.0 Hz, $C_{3,4}$), 123.56 (d, J_{CP} = 4.0 Hz, $C_{2,5}$) m/z = 302 $[M]^+$. Anal. Calcd for $C_{12}H_{20}N_3O_2PS$ (301): C 47.83, H 6.69, N 13.94. Found: C 47.81, H 6.67, N 13.91.

1H-Pyrrol-2-yl[di(4-morpholyl)]phosphine Oxide **7a**

To an ice-cooled and stirred solution of pyrrole **1** (0.01 mol) and pyridine (0.01 mol) in CH_2Cl_2 (60 mL), PCl_3 (0.01 mol) was added under dry argon. The reaction mixture was allowed to stand at room temperature for 2.5 h (until a ^{31}P NMR signal

at 132.7 ppm was detected), followed by adding pentane (100 mL), cooling to 0°C, and decanting the pentane layer thrice under argon. To the residue dissolved in benzene, morpholine (0.05 mol) was added with ice cooling and stirring. After 1 h of standing (when the ^{31}P NMR signal at 85.6 ppm appeared), the reaction mixture was filtered to remove the precipitate of morpholine hydrochloride. Hexachloroethane (0.01 mol) was added to the filtrate, and the mixture was left overnight. The resulting phosphonium salt precipitate was filtered off, washed with benzene, and dissolved in CH_2Cl_2 (50 mL). After shaking the solution with 20% aqueous $NaHCO_3$, the organic layer was separated, dried over Na_2SO_4, and evaporated. The product was recrystallized from diethyl ether. Yield 55%. mp 190–192°C. ^{31}P NMR (DMSO-d_6): δ 11.0. ^{1}H NMR (DMSO-d_6): δ 2.96 (m, 8H, O—CH_2), 3.56 (m, 8H, N-CH_2), 6.10 (br s, 1H, H_4), 6.56 (br s, 1H, H_3), 6.99 (br s, 1H, H_5), 11.63 (br s, 1H, NH).^{13}C NMR (DMSO-d_6): δ 44.09 (s, O—CH_2), 66.14 (s, N-CH_2), 108.2 (d, J_{CP} = 11.5 Hz, C_4), 118.46 (d, J_{CP} = 16.5 Hz, C_5), 120.22, 121.56 (d, J_{CP} = 168.5 Hz, C_2), 123.56 (d, J_{CP} = 10.0 Hz, C_3) *m/z* 286 $[M]^+$. Anal. Calcd for $C_{12}H_{20}N_3O_3P$ (285): C 50.52, H 7.07, N 14.73. Found: C 50.50, H 7.04, N 14.70.

1H-Pyrrol-2-yl[di(4-morpholyl)]phosphine Sulfide **7b**

It was obtained similarly to compound **7a**. To a benzene solution of the 2-pyrrolylphosphonous acid amide (with its ^{31}P NMR signal detected at 85.6 ppm), elementary sulfur (0.01 mol) was added, followed by boiling the reaction mixture with a reflux condenser for 30 min. After evaporating the solvent, the residue was recrystallized from diethyl ether. Yield 48%. mp 119–120°C. ^{31}P NMR (DMSO-d_6): δ 62.7. ^{1}H NMR (DMSO-d_6): δ 2.95 (m, 8H, O—CH_2), 3.53 (m, 8H, N-CH_2), 6.15 (br s, 1H, H_4), 6.57 (br s, 1H, H_3), 6.99 (br s, 1H, H_5), 11.63 (br s, 1H, NH). *m/z* 302 $[M]^+$. Anal. Calcd for $C_{12}H_{20}N_3O_2PS$ (301): C 47.83, H 6.69, N 13.94. Found: C 47.81, H 6.67, N 13.91.

1H-Pyrrol-3-yl[di(4-morpholyl)]phosphine **8**

To an ice-cooled and stirred solution of pyrrole **1** (0.01 mol) and pyridine (0.01 mol) in CH_2Cl_2 (60 mL), PCl_3 (0.01 mol) was added under dry argon. The reaction mixture was allowed to stand at room temperature for 5 days (until a ^{31}P NMR signal at 158.7 ppm became a single one), followed by adding pentane (100 mL), cooling to 0°C, and decanting the pentane layer thrice under argon. After dissolving the residue in benzene and cooling the solution with ice, morpholine (0.05 mol) was added with stirring. The reaction mixture was allowed to stand for 1 h (until the ^{31}P NMR signal at 89.5 ppm appeared), and filtered to remove the precipitate of morpholine hydrochloride. The filtrate was evaporated and kept in vacuum. The product was isolated as a light-colored oil. Yield 85%. ^{31}P NMR (DMSO-d_6): δ 87.5. ^{1}H NMR ($CDCl_3$): δ 3.01 (m, 8H, O—CH_2), 3.57 (m, 8H, N-CH_2), 6.06 (br s, 1H, H_4), 6.72 (br s, 1H, H_2), 9.79 (br s, 1H, NH). *m/z* 270 $[M]^+$. Anal. Calcd for $C_{12}H_{20}N_3O_2P$ (269): C 53.52, H 7.49, N 15.60. Found: C 53.51, H 7.48, N 15.59.

1H-Pyrrol-3-yl[di(4-morpholyl)]phosphine Sulfide **9**

To compound **8** (0.01 mol) in benzene (30 mL), elementary sulfur (0.01 mol) was added and the reaction mixture was boiled for 30 min. After evaporating the solvent, the residue was recrystallized from diethyl ether. Yield 68%. mp 127–128°C. ^{31}P NMR (DMSO-d_6): δ 68.9. H^1 (DMSO-d_6): δ 3.04 (m, 8H, O—CH_2); 3.54 (m, 8H, N-CH_2); 6.24 (br s, 1H, H_4); 6.85 (br s, 1H, H_5); 7.14 (br s, 1H, H_2); 11,39 (br s, 1H, N-H). ^{13}C NMR (DMSO-d_6): δ 44,98 (s, O—CH_2); 66.77 (s, N-CH_2); 110,76 (d, J_{CP} = 10.0 Hz, C_4); 112.91; 111.79 (d, J_{CP} = 146.0 Hz, C_3); 120.37 (d,J_{CP} = 14.0 Hz, C_5); 127.52 (d,J_{CP} = 24.0 Hz C_2). *m/z* 302 $[M]^+$. Anal. Calcd for $C_{12}H_{20}N_3O_2PS$ (301.35): N 13.94, P 10.28. Found: N 13.74, P 10.03.

1H-Pyrrol-3-yl[di(4-morpholyl)]phosphine Oxide **10**

To a solution of compound **8** (0.01 mol) in benzene (60 mL), hexachloroethane (0.01 mol) was added and the reaction mixture was allowed to stand at room temperature for 5 h. The resulting phosphonium salt precipitate was filtered off, washed with benzene, and dissolved in CH_2Cl_2 (50 mL). After shaking the solution with 20% aqueous $NaHCO_3$, the organic layer was separated, dried over Na_2SO_4, and evaporated. The residue was recrystallized from diethyl ether. Yield 45%. mp 155–157°C.^{31}P NMR (DMSO-d_6): δ 23.7. ^{1}H NMR (DMSO-d_6): δ 2.93 (m, 8H, O—CH_2), 3.51 (m, 8H, N-CH_2), 6.19 (br s, 1H, H_4), 6.91 (br s, 1H, H_5), 7.11 (br s, 1H, H_2). ^{13}C NMR (DMSO-d_6): δ 44.06 (s, CH_2-O), 66.53 (s, CH_2-N), 109.33; 107.91 (d, J_{CP} = 179,0 Hz, C_3-P), 110.33 (d, J_{CP} = 11.0 Hz, C_4), 119.82 (d, J_{CP} = 14.0 Hz, C_5), 125.33 (d, J_{CP} = 20.0 Hz, C_2). *m/z* 286 $[M]^+$. Anal. Calcd for $C_{12}H_{20}N_3O_3P$ (285.29): C 50.52, H 7.07, P 10.86. Found: C 50.48, H 7.00, P 10.63.

[Di(morpholin-4-yl)][3-(4-methoxyphenyl)triaz-2-enylidene](1H-pyrrol-3-yl)phosphorane **11**

To a solution of compound **8** (0.01 mol) in benzene (60 mL), *p*-methoxyphenyl azide (0.01 mol) was added and the reaction mixture was stirred at room temperature for 12 h, followed by evaporation. The residue was recrystallized from diethyl ether. Yield 72%. ^{31}P NMR (DMSO-d_6): δ 36.6. ^{1}H NMR (DMSO-d_6): δ 3.10 (m, 8H, O—CH_2), 3.55 (m, 8H, N-CH_2), 3.74 (s, 3H, O—CH_3) 6.37 (br s, 1H, H_4), 6.86 (d, J_{HH} = 9.00 Hz, 2H, Ar), 6.95 (br s, 1H, H_5), 7.05 (br s, 1H, H_2), 7.25 (d, J_{HH} = 9.00 Hz, 2H, Ar) *m/z* 419 $[M]^+$. Anal. Calcd for $C_{19}H_{27}N_6O_3P$ (418): C 54.54, H 6.50, N 20.08. Found: C 54.53, H 6.48, N 20.05.

[Di(morpholin-4-yl)](4-methoxyphenylimino)-(1H-pyrrol-3-yl)phosphorane **12**

Compound **11** (0.01 mol) was dissolved in toluene (50 mL) and boiled with a reflux condenser for 5 h. After evaporating the reaction mixture to dryness, the residue was recrystallized from diethyl ether. Yield 93%. mp 152–153°C. ^{31}P NMR (DMSO-d_6): δ 13.5. ^{1}H NMR (DMSO-d_6): δ 3.01 (m, 8H, O—CH_2), 3.52 (m, 8H, N-CH_2), 3.64 (s, 3H, O—CH_3) 6.37 (br s, 1H, H_4), 6.60 (m, 4H, Ar), 6.84 (br s, 1H, H_5), 7.15 (br s, 1H, H_2),^{13}C NMR (DMSO-d_6): δ 44.71 (s, CH_2-O), 54.66 (s, CH_3), 66.29 (d, J_{CP} = 6.0 Hz, C—N), 106.59; 108.02 (d, J_{CP} = 180.0 Hz, C_3-P), 110.83 (d, J_{CP} = 12.0 Hz, C_4), 113.77 (s, C_{Ar}) 119.38 (d, J_{CP} = 15.0 Hz, C_2), 122.99 (d, J_{CP} = 15.0 Hz, C_{Ar}) 125.54 (d, J_{CP} = 19.0 Hz, C_5), 143.55 (s, C_{Ar}), 150.90 (s, C_{Ar}), *m/z* 391 $[M]^+$. Anal. Calcd for $C_{19}H_{27}N_4O_3P$ (390): C 58.45, H 6.97, N 14.35. Found: C 58.44, H 6.96, N 14.33.

Methyl[di(morpholin-4-yl)](1H-pyrrol-3-yl)phosphonium **13**

To a solution of compound **8** (0.01 mol) in benzene (50 mL), CH_3I (0.02 mol) was added at room temperature and the reaction mixture was stirred for 12 h, followed by decantation of the benzene solution and recrystallization of the residue from diethyl ether. Yield 95%. mp 135–136°C.^{31}P NMR (DMSO-d_6): δ 50.4. ^{1}H NMR (DMSO-d_6): δ 2.38 (d, J_{CP} = 15.09 Hz, CH_3), 2.98 (m, 8H, O—CH_2), 3.63 (m, 8H, N-CH_2), 6.56 (br. s, 1H, H_4), 7.17 (br. s, 1H, H_5), 7.68 (br. s, 1H, H_2), *m/z* 286 $[M]^+$. Anal. Calcd for $C_{13}H_{24}N_3O_2P$ (285): C 54.72, H 8.48, N 14.73. Found: C 54.70, H 8.45, N 14.72.

2,5-Dimethyl-1H-pyrrol-3-yl[di(4-morpholyl)]phosphine Oxide **16**

To an ice-cooled solution of 2,5-dimethylpyrrole **16** (0.01 mol) and pyridine (0.01 mol) in CH_2Cl_2 (50 mL), PCl_3 (0.01 mol) was added. The reaction mixture was allowed to stand for 30 min (until a ^{31}P NMR signal at 158.5 ppm was detected), followed by adding morpholine (0.05 mol). After another 2 h of standing at room temperature, 30% H_2O_2 (5 mL) was added to the reaction mixture; it was stirred for 1 h and then diluted with H_2O (40 mL). The organic layer was separated, dried over Na_2SO_4, and evaporated. The residue was recrystallized from acetone. Yield 85%. mp 174–175°C. ^{31}P NMR (DMSO-d_6): δ 25.5. ^{1}H NMR (DMSO-d_6): δ 2.12 (s, CH_3), 2.31 (s, CH_3), 2.93 (m, 8H, O—CH_2), 3.51 (m, 8H, N-CH_2), 5.63 (s, 1H, Pr), 10.84 (br. s, 1H, NH), ^{13}C NMR (DMSO-d_6): δ 11.92 (s, CH_3) 44.57 (s, C-O), 66.49 (d, J_{CP} = 6.5 Hz, C—N), 103.71, 102.30 (d, J_{CP} = 177.5 Hz, C_3-P), 107.68 (d, J_{CP} = 11.5 Hz, C_4), 126.06 (d, J_{CP} = 14.0 Hz, C_5), 134.98 (d, J_{CP} = 21.5 Hz, C_2). *m/z* 314 $[M]^+$. Anal. Calcd for $C_{14}H_{24}N_3O_3P$ (313): C 53.67, H 7.72, N 13.41 P 10.86. Found: C 53.65, H 7.70, P 10.63.

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