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Phosphorylation of *N-iso*-Propyl- and *N-tert*-Butylpyrroles with Phosphorus(III) Halides

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ABSTRACT: *The investigation concerns the effect of a bulky substituent at the pyrrole nitrogen atom on the orientation and regioselectivity of pyrrole phosphorylation with phosphorus(III) halides. As shown, phosphorylation of N-iso-propylpyrrole with phosphorus tribromide or trichloride proceeds nonregioselectively at positions 2 and 3 but it is followed by the 2 → 3 migration of the dihalogenophosphine group which quantitatively yields the 3-isomer. N-tert-butylpyrrole is regioselectively phosphorylated with halogenophosphines at position 3. The tert-butyl substituent at the nitrogen atom does not preclude the binding of even two or three pyrrolyl residues to the phosphorus atom. The key compounds, 3-pyrrolyldihalogenophosphines, were isolated in a pure state, characterized and used to obtain a number of stable phosphorus(V) derivatives*. © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:599–604, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20158

INTRODUCTION

Regioselective electrophilic substitution in the pyrrole ring now remains a challenging open problem, as the orientation of the electrophilic attack is governed by several factors. In spite of the theoretically predicted larger negative charge on the pyrrole 3-carbon atom [1–3], experimental data evidence for a preferable formation of two-substituted isomers [4–7], which are evidently due to a higher stability of the corresponding intermediate state.

Formation of three-substituted pyrrole derivatives results either from the 2 → 3 isomerization occurring under acidic catalysis [8–11] or from the direct attack by a "hard" electrophile, such as trimethylsilyl cation, on the "hard" 3-carbon atom [12].

The orientation and regioselectivity of electrophilic pyrrole phosphorylation depends on the nature of the substituent at the nitrogen atom [13,14], the nature of a phosphorylating agent [15], solvent polarity [13,14,16] etc. The necessary conditions for regioselective 2-phosphorylation of the pyrrole ring with phosphorus tribromide were found previously by [13,14]. Three-phosphorylated pyrroles were obtained by the 2 → 3 migration of the dibromophosphine group studied for *N*-methyl- and *N*-arylpyrrolyldibromophosphines [14,16].

There are precedences in the literature for the electrophilic attack oriented to position 3 of pyrrole which is caused by the steric effect of a bulky substituent at the nitrogen atom [17,18]. One might expect that sufficiently bulky groups like the *iso*-propyl and *tert*-butyl residues should create steric

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hindrances for the attack on position 2 of the pyrrole ring thus inducing 3-phosphorylation.

RESULTS AND DISCUSSION

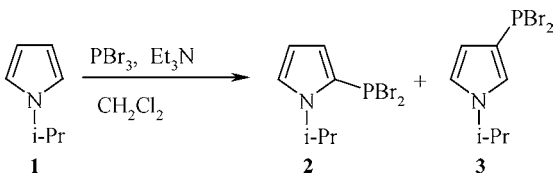
Phosphorylating *N-iso*-propylpyrrole (**1**) with phosphorus tribromide in benzene, we have demonstrated that the steric effect of the *iso*-propyl group does not suffice to completely suppress the electrophilic attack on position 2. Twelve hours after starting the reaction (when it is not yet complete as evidenced by the ^{31}P NMR signal of unreacted phosphorus tribromide in the reaction mixture), the products are constituted by the two-substituted (60%, δ ^{31}P 110.1 ppm) and three-substituted (40%, δ ^{31}P 145.2 ppm) isomers, **2** and **3** [13]. The reaction completes within 24 h to provide the practically unchanged product ratio. During longer duration (for 3–4 months), the 3-isomer is accumulated slowly in the reaction mixture.

An analogous reaction, if conducted in a polar solvent like methylene chloride, involves the rapid 2 → 3 migration of the dibromophosphine group [16] and furnishes *N-iso*-propylpyrrolyl-3-dibromophosphine (**3**) in a pure state. After 2 h of reaction time, the ^{31}P NMR spectral data pointed to 33% of the 2-isomer and 67% of the 3-isomer in the reaction mixture, and after 24 h their respective contributions amounted to 16 and 84%. At 48 h reaction time, the reaction mixture exhibited a single ^{31}P resonance assigned to **3** (Scheme 1).

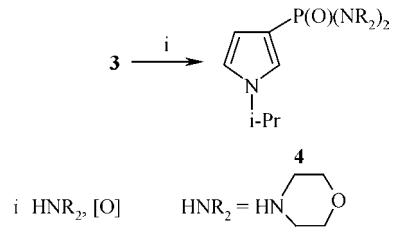
It should be noted that with the *iso*-propyl substituent at the pyrrole nitrogen atom, the 2 → 3 migration of the dibromophosphine group is slowed down significantly as compared to the case of 2-phosphorylated *N*-methyl-substituted pyrrole which undergoes the analogous rearrangement within 2 h [16].

Dibromophosphine **3** was converted into phosphonate **4** in good yield (Scheme 2).

Phosphorylation of *N-iso*-propylpyrrole with phosphorus trichloride in methylene chloride proceeds extremely slowly and also nonregioselectively to provide a mixture of the two-substituted (δ ^{31}P 132.69 ppm) and three-substituted (δ ^{31}P



SCHEME 1



SCHEME 2

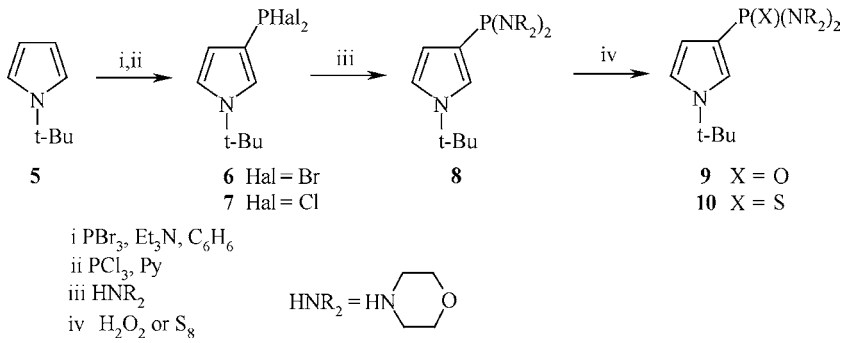
160.23 ppm) products. The reaction takes as long as 3 months to complete; the 2 → 3 migration of the dichlorophosphine group also proceeds completely within this time leading to the accumulation of the 3-isomer.

We have found that phosphorylation of *N-tert*-butylpyrrole (**5**) with both phosphorus tribromide and less reactive phosphorus trichloride proceeds regioselectively at position 3 to furnish the corresponding dihalogenophosphines **6** and **7**. Accordingly, the ^{31}P NMR spectrum of the reaction mixture initially containing *N-tert*-butylpyrrole, phosphorus tribromide, and triethylamine in benzene at room temperature exhibits, at 12 h reaction time, the complete disappearance of the PBr_3 peak (229.2 ppm) and the appearance of the signal from the three-substituted product (145.0 ppm) [13]. No signals upfield from this one are observed at 100–120 ppm, which completely rules out the electrophilic attack on position 2. Phosphorylation of **5** with phosphorus trichloride in benzene is very slow; in pyridine, the reaction requires 24 h to completion. As with phosphorus tribromide, phosphorylation is solely oriented to position 3 (only one ^{31}P NMR signal at 160.5 ppm is detected). The reaction is run with an excess of PCl_3, lest two or three chlorine atoms be substituted.

Dihalogenophosphines **6** and **7** were isolated in a pure state and converted into stable phosphorus(V) derivatives **9** and **10** (Scheme 3).

It is noteworthy that amide **8** is exceedingly reactive toward oxidation. As a result, we failed to isolate it in a pure state (a special technique for solvent deoxygenation was not employed). As soon as 2 h after adding morpholine in the course of the synthesis of amide **8**, the characteristic ^{31}P NMR signal of compound **8** (89.15 ppm) is accompanied by the equally intensive signal of its oxide **9** (21.7 ppm). To obtain sulfide **10**, sulfur should be added before morpholine to the reaction mixture, or else the amide **8** is oxidized by oxygen of the air.

The *N-tert*-butyl substituent in pyrrole causes no hindrances to the introduction of two or three pyrrolyl residues at the phosphorus atom leading to compounds **13–15**. Since the multiple substitution

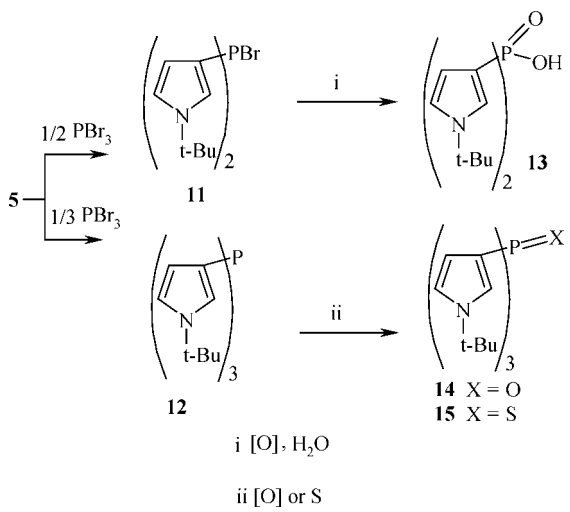


SCHEME 3

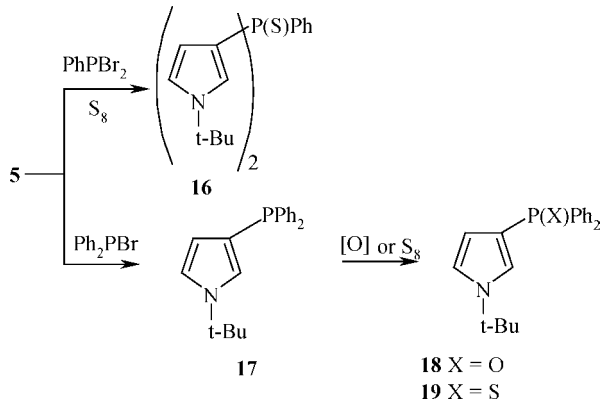
does not take place in benzene even on the boiling, the reaction was conducted in pyridine and it finished within 2 days at room temperature. Starting from dipyrrolylbromophosphine **11** and tertiary phosphine **12**, stable derivatives **13–15** were obtained (Scheme 4).

Phosphorylation of **5** with phenyldibromophosphine and diphenylbromophosphine was run in pyridine, because it does not proceed in a nonpolar solvent, such as benzene. The reaction between **5** and phenyldibromophosphine completes after 24 h, with the reaction mixture abounding with various by-products, as evidenced by the ^{31}P NMR spectra; sulfide **16** was therefore isolated in low yield (see Scheme 5).

With less reactive diphenylbromophosphine, the reaction needs as long as 4 days for completion. The resulting tertiary phosphine **17** is oxidized so readily that the ^{31}P NMR spectrum of the reaction mixture measured after 1 day of reaction time shows two



SCHEME 4



SCHEME 5

signals, one arising from compound **17** (–25.5 ppm) and the other being assigned to the corresponding oxide **18** (20.1 ppm), with the respective relative intensities of 12 and 88%. To obtain sulfide **19, 5** was reacted with diphenylbromophosphine in the presence of sulfur.

CONCLUSION

We have demonstrated that the steric hindrances induced by bulky substituents at the pyrrole nitrogen atom cause a dramatic effect on the orientation and regioselectivity of pyrrole phosphorylation with phosphorus(III) halides. Whereas *N*-methyl- [13] and *N*-arylpyrroles [14] enter into the reaction to give two-phosphorylated derivatives (the 3-isomers are obtainable only by the $2 \rightarrow 3$ migration of the dibromophosphine group), phosphorylation of *N-tert*-butylpyrrole with phosphorus tribromide

and trichloride, and bromophosphines proceed exclusively at position 3 of the pyrrole ring.

EXPERIMENTAL

The ^{1}H, ^{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 ^{1}H and ^{13}C signals, and 85% H_3PO_4 as external standard for ^{31}P signals. The chromato-mass spectra were registered on an Agilent 1100 series LC/MSD instrument.

*1-iso-Propyl-1H-pyrrol-3-ylphosphinyl Dibromide (**3**)*

To a solution of *N-iso*-propylpyrrole **1** (1.09 g, 0.01 mol) in dry methylene chloride cooled to 0°C (30 mL), phosphorus tribromide (2.71 g, 0.01 mol) and triethylamine (1.01 g, 0.01 mol) were added. The reaction mixture was left to stand for 2 days. The course of the reaction was controlled by ^{31}P NMR spectroscopy; the completion of the 2 → 3 rearrangement was marked by a resonance disappearing at 110.10 ppm and another one emerging at 145.24 ppm. After adding dry hexane (10 mL) to the reaction mixture, the resulting precipitate of salts was filtered off in a stream of dry argon. The filtrate was evaporated and held under oil pump vacuum for 2 h. The residue was washed twice with dry hexane and dried. The product appears as a light-colored oil. Yield 85%. ^{31}P NMR ($CHCl_3$): δ 146.09. ^{1}H NMR ($CDCl_3$): δ 1.24 (d, $J_{H,H}$ = 6.6 Hz, 6H, CH_3), 4.09 (m, 1H, CH), 6.39 (br s, 1H, H_4), 6.37 (br s, 1H, H_5), 6.71 (br s, 1H, H_2). Anal. Calcd for $C_7H_{10}Br_2NP$ (299.0): Br 53.51; P 10.37. Found: Br 53.32; P 10.45.

*1-iso-Propyl-1H-pyrrol-3-yl[di(4-morpholyl)]-phosphine Oxide (**4**)*

To an ice-cooled solution of dibromophosphine **3** (0.01 mol) in dry benzene (50 mL), morpholine (0.04 mol) was added. The reaction mixture which was maintained at room temperature for 2 h showed a ^{31}P resonance at 87.87 ppm. After leaving it open for 7 days, the position of the ^{31}P NMR signal changed to 25.00 ppm. Then the precipitate of salts was filtered off, and the filtrate was evaporated. The residue was washed with hexane to give the product as a light-colored oil. Yield 90%. ^{31}P NMR ($CHCl_3$): δ 25.00. ^{1}H NMR ($CDCl_3$): δ 1.45 (d, $J_{H,H}$ = 5.8 Hz, 6H, CH_3), 3.11 (m, 8H, O-Mr), 3.65 (m, 8H, N-Mr), 4.26 (m, 1H, CH), 6.16 (br s, 1H, H_4), 6.77 (br s, 1H, H_5), 7.22 (br s, 1H, H_2). *m/z* 327 $[M]^+$. Anal. Calcd for $C_{15}H_{16}N_3O_3P$ (327.4): N 12.84, P 9.48; Found: N 12.74, P 9.53.

*1-tert-Butyl-1H-pyrrol-3-ylphosphinyl Dibromide (**6**)*

To a solution of *N-tert*-butylpyrrole **5** (1.23 g, 0.01 mol) and triethylamine (1.51 g, 0.015 mol) in dry benzene (100 mL), phosphorus tribromide (2.98 g, 0.011 mol) was added. After 12 h of reaction time when only one signal at 145.00 ppm was observed in the ^{31}P NMR spectrum, the precipitate of triethylamine hydrobromide was filtered off in a stream of dry argon. The filtrate was evaporated and held under oil pump vacuum for 1 h. The residue was treated with dry hexane and filtered off in a stream of dry argon. Yield 83%. mp 48–50°C. ^{31}P NMR ($CHCl_3$): δ 145.21. ^{1}H NMR ($CDCl_3$): δ 1.57 (s, 9H, t-Bu), 6.67 (br s, 1H, H_4), 7.03 (br s, 1H, H_5), 7.43 (br s, 1H, H_2). Anal. Calcd for $C_8H_{12}Br_2NP$ (313.0): Br 51.12, P 9.90. Found: Br 51.42, P 9.72.

*1-tert-Butyl-1H-pyrrol-3-ylphosphinyl Dichloride (**7**)*

To an ice-cooled solution of *N-tert*-butylpyrrole **5** (1.23 g, 0.01 mol) in dry pyridine (30 mL), phosphorus trichloride (1.51 g, 0.011 mol) was added. The reaction mixture was maintained at room temperature for 24 h, then dry hexane (10 mL) was added, and the resulting precipitate of pyridine hydrochloride was filtered off. The filtrate was evaporated and held under oil pump vacuum for 1 h. The product appears as a light-colored oil which was extracted from the residue with dry hexane. Yield 65%. ^{31}P NMR ($CHCl_3$): δ 156.99. ^{1}H NMR ($CDCl_3$): δ 1.49 (s, 9H, t-B), 6.60 (br s, 1H, H_4), 6.97 (br s, 1H, H_5), 7.29 (br s, 1H, H_2). Anal. Calcd for $C_8H_{12}Cl_2NP$ (224.1): Cl 31.70, P 13.84. Found: Cl 31,45, P 13.72.

*1-tert-Butyl-1H-pyrrol-3-yl[di(4-morpholyl)]-phosphine Oxide (**9**)*

To a solution of dibromophosphine **6** (3.13 g, 0.01 mol) or dichlorophosphine **7** (2.24 g, 0.01 mol) in dry benzene (70 mL) cooled to 0°C, morpholine (3.87 g, 0.045 mol) was added. The reaction mixture which was maintained at room temperature for 2 h showed a ^{31}P resonance at 89.15 ppm. On cooling the reaction mixture to 0°C, 50% hydrogen peroxide (12 mL) was added to it. Then it was held at room temperature for 1 h, diluted with water (50 mL), and the organic layer was separated. The benzene solution was dried over sodium sulfate and evaporated under vacuum. The product (light-colored oil) was extracted from the residue with diethyl ether. Yield 65%. ^{31}P NMR ($CHCl_3$): δ 24.40. ^{1}H NMR ($CDCl_3$): δ 1.44 (s, 9H, t-Bu), 3.01 (m, 8H, O-CH_2), 3.54 (m, 8H,

N-CH_2), 6.06 (m, 1H, H_4), 6.78 (m, 1H, H_5), 7.26 (m, 1H, H_2). ^{13}C NMR ($CDCl_3$): δ 30.4 (s, CH_3), 44.2 (s, CH_2-O), 55.5 (s, N-C), 67.0 (s, CH_2-N), 107.7 (d, J_{CP} = 178.0 Hz, C_3-P), 110.1 (d, J_{CP} = 11.3 Hz, C_4), 119.65 (d, J_{CP} = 12.8 Hz, C_5), 125.99 (d, J_{CP} = 20.1 Hz, C_2). *m/z* 341 $[M]^+$. Anal. Calcd for $C_{16}H_{28}N_3O_3P$ (341.4): C 56.30, H 8.21, P 9.09. Found: C 56.18, H 8.25, P 9.16.

*1-tert-Butyl-1H-pyrrol-3-yl[di(4-morpholyl)]-phosphine Sulfide (***10***)*

To a solution of dibromophosphine **6** (3.13 g, 0.01 mol) or dichlorophosphine **7** (2.24 g, 0.01 mol) in dry benzene (70 mL) cooled to 0°C, elementary sulfur (0.48 g, 0.015 mol) and morpholine (3.87 g, 0.045 mol) were added. The reaction mixture was maintained at room temperature for 2 h and then was boiled for 5 min. On cooling, the resulting precipitate was filtered off and the filtrate was evaporated under vacuum. The product appears as a light-colored oil which was extracted from the residue with diethyl ether. Yield 67%. ^{31}P NMR (DMSO-d_6): δ 69.84. ^{1}H NMR (DMSO-d_6): δ 1.55 (s, 9H, t-Bu), 2.94 (m, 8H, O-CH_2), 3.55 (m, 8H, N-CH_2), 6.19 (m, 1H, H_4), 6.98 (m, 1H, H_5), 7.21 (m, 1H, H_2). *m/z* 357 $[M]^+$. Anal. Calcd for $C_{16}H_{28}N_3O_2PS$ (357.5): N 11.76, P 8.68. Found: N 11.68, P 8.61.

*Bis(1-tert-butyl-1H-pyrrol-3-yl)phosphoric Acid (***13***)*

To a solution of *N-tert*-butylpyrrole **5** (2.46 g, 0.03 mol) in dry pyridine (50 mL) cooled to 0°C, phosphorus tribromide (2.71 g, 0.01 mol) and triethylamine (2.02 g, 0.02 mol) were added. The reaction mixture was maintained at room temperature for 2 days, and acetone (10 mL) was added. The reaction mixture was left open for 4 days. After evaporating the solvent, the residue was treated with the mixture methanol: diethyl ether (1:1). The precipitate of the product was filtered off and recrystallized from methanol. Yield 58%. mp 195–196°C. ^{31}P NMR (DMSO-d_6): δ 30.70. ^{1}H NMR (DMSO-d_6): δ 1.49 (s, 9H, CH_3), 6.37 (br s, 1H, C_4), 6.79 (br s, 1, H_5), 7.26 (br s, 1H, H_2), 9.78 (br s, 1H, OH). *m/z* 308 $[M]^+$. Anal. Calcd for $C_{16}H_{25}N_2O_2P$ (308.4): N 9.09, P 10.06. Found N 9.13, P 10.18.

*Tris(1-tert-butyl-1H-pyrrol-3-yl)phosphine Oxide (***14***)*

To a solution of *N-tert*-butylpyrrole **5** (3.69 g, 0.03 mol) in dry pyridine (50 mL) cooled to 0°C, phosphorus tribromide (2.71 g, 0.01 mol) and triethylamine (3.03 g, 0.03 mol) were added. The reaction mixture was maintained at room temperature for 2 days and left open for 7 days. On adding hexane (5 mL), the resulting precipitate was filtered off. The filtrate was evaporated, and the residue was treated with diethyl ether (10 mL). Yield 32%. mp 141–142°C. ^{31}P NMR (DMSO-d_6): δ 11.5. ^{1}H NMR (DMSO-d_6): δ 1.51 (s, 27 H, CH_3), 6.29 (m, 3H, H_4), 6.83 (m, 3H, H_5), 7.27 (m, 3H, H_2). *m/z* 413 $[M]^+$. Anal. Calcd for $C_{24}H_{36}N_3OP$ (413.6): N 10.16, P 7.61. Found: N 10.13, P 7.51.

*Tris(1-tert-butyl-1H-pyrrol-3-yl)phosphine Sulfide (***15***)*

It was obtained analogously to compound **14**. The reaction mixture was additionally treated with elementary sulfur (0.48 g, 0.015 mol) and boiling for 0.5 h. On cooling, hexane (20 mL) was added and the resulting precipitate was filtered off. The filtrate was evaporated under vacuum. The residue was recrystallized from diethyl ether. Yield 43%. mp 185–186°C. ^{31}P NMR (DMSO-d_6): δ 7.70. ^{1}H NMR (DMSO-d_6): δ 1.52 (s, 27 H, CH_3), 6.20 (m, 3H, H_4), 6.93 (m, 3H, H_5), 7.08 (m, 3H, H_2). *m/z* 429 $[M]^+$.Anal. Calcd for $C_{24}H_{36}N_3PS$ (429.6): N 9.79, P 7.23. Found: N 9.69, P 7.08.

*Bis(1-tert-butyl-1H-pyrrol-3-yl)phenylphosphine Sulfide (***16***)*

To a solution of *N-tert*-butylpyrrole **5** (2.46 g, 0.02 mol) in dry pyridine (30 mL), phenyldibromophosphine (2.68 g, 0.015 mol) was added. The reaction mixture was maintained at room temperature for 24 h, and elementary sulfur (0.48 g, 0.015 mol) was added to it. On boiling for 5 min, the mixture was evaporated under vacuum. Water was poured into the residue, and it was stirred with a magnetic stirrer for 10 min. On decanting water, the residue was treated with methanol. The product was recrystallized from methanol. Yield 12%. mp 152–153°C. ^{31}P NMR (DMSO-d_6): δ 20.90. ^{1}H NMR (DMSO-d_6): δ 1.55 (s, 18H, CH_3), 6.17 (m, 2H, H_4), 6.97 (m, 2H, H_5), 7.15 (m, 2H, H_2), 7.39 (m, 3H, Ph), 7.68 (m, 2H, Ph). *m/z* 384 $[M]^+$. Anal. Calcd for $C_{22}H_{29}N_2PS$ (384.5): N 7.29, P 8.07. Found: N 7.21, P 8.00.

*1-tert-Butyl-1H-pyrrol-3-yl(diphenyl)phosphine Oxide (***18***)*

To a solution of diphenylbromophosphine (2.65 g, 0.01 mol) in pyridine (30 mL), a solution of *N-tert*-butylpyrrole **5** (1.23 g, 0.01 mol) in dry pyridine (20 mL) was added. Four days later, the reaction mixture was evaporated under vacuum. The product (light-colored oil) was extracted from the residue with diethyl ether. Yield 45%. ^{31}P NMR (DMSO-d_6): δ 20.90. ^{1}H NMR (DMSO-d_6): δ 1.52 (s, 9H, CH_3), 6.16

(m, 1H, H_4), 7.10 (m, 2H, $H_{2,5}$), 7.59 (m, 10H, Ph). *m/z* 323 $[M]^+$. Anal. Calcd for $C_{20}H_{22}NPO$ (323.4): N 4.33, P 9.59. Found: N 4.38, P 9.52.

*1-tert-Butyl-1H-pyrrol-3-yl(diphenyl)phosphine Sulfide (**19**)*

To a solution of diphenylbromophosphine (2.65 g, 0.01 mol) in pyridine (30 mL), sulfur (0.48 g, 0.015 mol) and a solution of *N-tert*-butylpyrrole **5** (1.23 g, 0.01 mol) in pyridine (20 mL) were added. After holding the reaction mixture at room temperature for 4 days, it was evaporated under vacuum. The residue was treated with water which was then decanted. The product was recrystallized from acetone. Yield 45%. mp 155–156°C. ^{31}P NMR (DMSO-d_6): δ 31.30. ^{1}H NMR (DMSO-d_6): δ 1.53 (s, 9H, CH_3), 6.20 (br s, 1H, H_4), 7.11 (br s, 1H, H_5), 7.18 (br s,1H, H_2), 7.48 (m, 6H, Ph), 7.69 (m, 4H, Ph). ^{13}C NMR (DMSO-d_6): δ 30.1 (s, CH_3), 55.8 (s, N-C), 110.8 (d, $J_{CP} = 110.2$ Hz, C_3-P), 111.7 (d, $J_{CP} = 10.6$ Hz, C_4), 121.4 (d, $J_{CP} = 11.3$ Hz, C_5), 125.7 (d, $J_{CP} = 21.9$ Hz, C_2), 128.3 (d, $J_{CP} = 12.8$ Hz, $C_{2,2'\text{-Ph}}$), 131.0 (d, $J_{CP} = 11.0$ Hz, $C_{4\text{-Ph}}$), 131.1 (s, $C_{3,3'\text{-Ph}}$), 135.0 (d, $J_{CP} = 85.8$ Hz, $C_{Ph\text{-}P}$). *m/z* 339 $[M]^+$. Anal. Calcd for $C_{20}H_{22}NPS$ (339.4): N 4.13, P 9.14. Found: N 4.08, P 9.05.

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