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SYNTHESIS AND CHARACTERIZATION OF NOVEL ALKYL-SUBSTITUTED ARYL DIPHENYLPHOSPHINATE ESTERS

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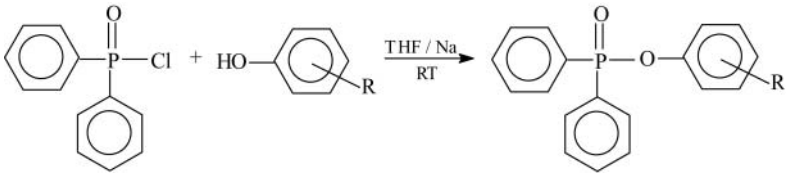
GRAPHICAL ABSTRACT



Abstract *In this work, the reactions of diphenylphosphinic chloride, $(C_6H_5)_2P(O)Cl$, **1**, with the sodium salts of sterically hindered phenol derivatives (**2a-2j**) were investigated. Novel alkyl-substituted aryl diphenyl phosphinate esters $(C_6H_5)_2P(O)OAr$ (**3-12**) were obtained from these reactions. Satisfactory analytical and spectroscopic results were obtained for all the new compounds.*

Keywords Diphenylphosphinic chloride; phenol; phosphinic acid esters; phosphinate

INTRODUCTION

Organophosphorus compounds are an economically important class of chemical compounds. These compounds have found a wide range of applications in the areas of industrial, agricultural, and medicinal chemistry due to their biological and physical properties as well as their utility as synthetic intermediates.[1] Certain organophosphorus compounds are widely used as pesticides, antimicrobicides, or antitumor active substances.[2–7]

Phosphinic acid derivatives are also used in protecting metal surfaces against corrosion,[8] to improve the performance of high-temperature greases,[9] and for flame resistance.[10–12] These derivatives are important and intensively studied compounds.[13–33]

In this study, sterically hindered phenols have been selected as nucleophiles. Sterically hindered phenols are widely used for the preparation of stable phenoxyl radicals and are of

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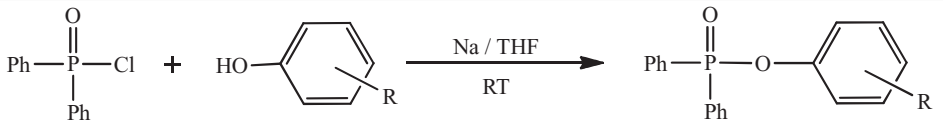
interest as biologically active compounds.[24,25] Steric properties of the substituents also play a major role in determining the biological activity of these chemicals.[26] In particular, phosphorus derivatives of 2,6-di-*tert*-butyl-4-methylphenol are effective antioxidants. They are capable of inhibiting lipid peroxidation and lower the oxidative stress of the organism. Here we report on the synthesis of sterically hindered phenol esters **3-12** of diphenylphosphinic acid. It is anticipated that products **3-12** should show important biological activities.

RESULTS AND DISCUSSION

All compounds **3-12** were synthesized as described in the Experimental section. Diphenylphosphinic chloride **1** was reacted in a 1:1 molar ratio with the sodium salts of phenol derivatives **2a-j** in tetrahydrofuran (THF). Aryl diphenylphosphinate compounds **3-12** were obtained from these reactions (see Scheme 1).



1 **2a-2j** **3-12**

Product	R	Mp °C	Yield %
3	2,4-dimethyl	87-88	40
4	3,4-dimethyl	119-120	38
5	2,6-dimethyl	124-125	28
6	2,4,6-trimethyl	76-77	32
7	4-tert-butyl	168-170	52
8	4-tert-butyl-2-methyl	138-139	43
9	2-tert-butyl-4-methyl	125-126	28
10	2-tert-butyl-6-methyl	76-77	32
11	2,6-di-tert-butyl-4-methyl	149-150	24
12	2,4,6-tri-tert-butyl	196-198	37

Scheme 1 Synthesis and some properties of aryl diphenylphosphinate esters **3–12**.

Products **3-12** were isolated from the reaction mixtures by column chromatography. In all cases, the yields ranged from 52 to 24%. Using diethylether as a solvent instead of THF resulted in significantly lower (from 27 to 9%) yields of products. Because the nature of the reagents and the solvent affect the yield, the steric hindrance also plays an important role in such reactions.

Compounds **3-12** were obtained as white crystals and are stable in air and moisture. The structures of compounds **3-12** were determined initially by ^{1}H-, ^{13}C-, and ^{31}P-NMR spectroscopy, elemental analyses, and infrared spectroscopy. They were also characterized by gas chromatography–mass spectrometry (GC-MS). The structures of compounds **3-12** are consistent with the analytical and spectroscopic data (see Experimental section and Table 1). The composition of all compounds was confirmed by elemental analysis.

Table 1 ^{1}H-, ^{13}C-, and ^{31}P-NMR spectroscopic data (δ ppm, $CDCl_3$) for compounds **3-12**

	^{31}P	^{13}C	^{1}H
3	29.9	147.4 (d, J_{PC} = 8.3 Hz), 133.9, 132.4, 130.5, 128.7 (d, J_{PC} = 6.2 Hz), 127.4, 119.9 (d, J_{PC} = 3.6 Hz), 20.6, 16.8	2.23 (s, 3H, 4-CH_3), 2.28 (s, 3H, 2-CH_3), 7.28–7.94 (m, 10H, arom−H), 6.73–6.96 (m, 3H, ArO−H)
4	30.1	148.8 (d, J_{PC} = 8.3 Hz), 138.1, 132.3 (d, J_{PC} = 2.8), 131.9 (d, J_{PC} = 10.3 Hz), 130.4 (d, J_{PC} = 4.5 Hz), 128.6 (d, J_{PC} = 2.84), 121.8 (d, J_{PC} = 4.7 Hz), 117.7 (d, J_{PC} = 4.8 Hz), 19.9, 19.0	2.16 (s, 3H, 4-CH_3), 2.18 (s, 3H, 3-CH_3), 7.43–7.95 (m, 10H, arom−H); 6.89–6.99 (m, 3H, ArO−H)
5	29.5	148.3 (d, J_{PC} = 9.8 Hz), 133.1, 132.3 (d, J_{PC} = 2.8 Hz), 131.7 (d, J_{PC} = 10.3), 131.3, 130.7 (d, J_{PC} = 2.83 Hz), 129.1 (d, J_{PC} = 1.51 Hz), 128.6 (d, J_{PC} = 7.2 Hz), 124.8 (d, J_{PC} = 1.64 Hz), 18.2	2.05 (s, 6H, 2,6-CH_3), 7.45–7.95 (m, 10H, arom−H), 6.93–6.98 (m, 3H, ArO−H)
6	30.0	146.0 (d, J_{PC} = 9.8 Hz), 134.2 (d, J_{PC} = 1.9 Hz), 133.1, 132.3 (d, J_{PC} = 2.8 Hz), 131.7 (d, J_{PC} = 10.3 Hz), 130.3 (d, J_{PC} = 2.9 Hz), 129.7 (d, J_{PC} = 1.6 Hz), 128.6 (d, J_{PC} = 13.4 Hz), 20.6, 18.1	2.00 (s, 6H, 2,6-CH_3), 2.22 (s, 3H, 4-CH_3), 7.47–7.94 (m, 10H, Ar-H), 6.78 (s, 3H, ArO−H)
7	30.3	148.5 (d, J_{PC} = 8.3 Hz), 147.4, 132.4 (d, J_{PC} = 2.9 Hz), 132.2, 131.9 (d, J_{PC} = 10.4 Hz), 130.4, 128.7 (d, J_{PC} = 13.4 Hz), 126.5, 120.1 (d, J_{PC} = 4.7 Hz), 34.3, 31.4	2.00 (s, 6H, 2,6-CH_3), 2.22 (s, 3H, 4-CH_3), 7.47–7.94 (m, 10H, arom−H), 6.78 (s, 3H, ArO−H)
8	29.8	147.1 (d, J_{PC} = 0.8 Hz), 132.6, 132.4 (d, J_{PC} = 2.8 Hz), 131.8 (d, J_{PC} = 10.4 Hz), 128.7 (d, J_{PC} = 13.4 Hz), 123.8, 119.5 (d, J_{PC} = 3.6 Hz), 34.2, 31.4, 17.2	1.26 (s, 9H, 4-Bu^t), 2.33 (s, 3H, 2-CH_3), 7.88–7.96 (m, 10H, arom−H), 7.01–7.68 (m, 3H, ArO−H)
9	29.5	148.5 (d, J_{PC} = 8.4 Hz), 138.8 (d, J_{PC} = 6.8 Hz), 132.9, 132.3, 132.4 (d, J_{PC} = 2.9 Hz), 131.8 (d, J_{PC} = 10.5 Hz), 130.6, 128.6 (d, J_{PC} = 13.5 Hz), 127.4, 119.8 (d, J_{PC} = 5.2 Hz), 34.5, 30.0, 20.9	1.49 (s, 9H, 2-Bu^t), 2.36 (s, 3H, 4-CH_3), 6.84–7.46 (m, 3H, arom−H)
10	28.2	149.6 (d, J_{PC} = 11.0 Hz), 141.4 (d, J_{PC} = 4.7 Hz), 134.6, 132.7, 132.0 (d, J_{PC} = 2.8 Hz), 131.6 (d, J_{PC} = 10.2 Hz), 128.6 (d, J_{PC} = 10.0 Hz), 125.6, 124.3 (d, J_{PC} = 0.7 Hz), 34.7, 30.5, 21.2	1.19 (s, 9H, 2-C(CH_3)$_3$), 2.15 (s, 3H, 6-CH_3), 7.93–8.00 (m, 10H, arom−H), 6.98–7.57 (m, 3H, ArO−H)
11	29.9	142.8 (d, J_{PC} = 3.6 Hz), 141.7, (d, J_{PC} = 9.4 Hz), 132.5 (d, J_{PC} = 10.0 Hz), 132.1 (d, J_{PC} = 3.0 Hz), 130.1, 128.3 (d, J_{PC} = 13.3 Hz), 36.8, 33.1, 21.0	1.19 (s, 18H, 2,6-Bu^t), 2.31 (s, 3H, 4-CH_3); 7.28–7.63 (m, 10H, arom−H), 7.00 (s, 2H, ArO−H)
12	31.9	145.2 (d, J_{PC} = 1.9 Hz), 142.1 (d, J_{PC} = 3.4 Hz), 141.9 (d, J_{PC} = 9.3 Hz), 132.4 (d, J_{PC} = 10.1 Hz), 132.1 (d, J_{PC} = 2.8 Hz), 130.1, 128.2 (d, J_{PC} = 13.3 Hz), 124.3 (d, J_{PC} = 2.0 Hz), 37.0, 34.4, 33.1, 31.5	1.22 (s, 18H, 2,6-C(CH_3)$_3$), 1.33 (s, 9H, 4-C(CH_3)$_3$), 7.36–7.57 (m, 10H, arom−H); 7.20 (s, 2H, ArO−H)

The characteristic bands in the infrared (IR) spectra of the phosphinates were assigned and are provided in the Experimental section. The P=O stretching vibrations of **3-12** between 1223 and 1238 cm^{-1} are characteristic of phosphinates. In the Fourier transform infrared (FTIR) spectra of compounds **3-12**, P−Ph and P−O−Ph stretching bands are observed between 1436–1440 and 1168–1238 cm^{-1}, respectively. These data are in good agreement with the literature.[21,27–31] In the FTIR spectra of compounds **3-12**, $CH_{(aryl)}$

and $CH_{(aliphatic)}$ stretching bands are observed between 3049–3437 and 2903–2984 cm^{-1}, respectively.

The nuclear magnetic resonance (NMR) data for **3-12** are presented in the Experimental section. The ^{1}H-NMR spectral data of **3-12** are consistent with the structures proposed. The methyl protons resonate at $\delta = 2.28$ (2-CH_3), 2.23 (4-CH_3) (in a 1:1 ratio), 2.18 (3-CH_3), 2.16 (4-CH_3) (in a 1:1 ratio), and 2.05 (2,6-*di*-CH_3) for compounds **3**, **4**, and **5**, respectively. The methyl protons resonate at $\delta = 2.00$ (2,6-*di*-CH_3) and 2.22 (4-CH_3) (in a 2:1 ratio) in the case of compound **6**. Signals are observed at $\delta = 1.26$ (4-Bu^t) for **7**; at $\delta = 1.26$ (4-Bu^t) and 2.33 (2-CH_3) for **8**; at $\delta = 1.49$ (2-Bu^t) and 2.36 (4-CH_3) for **9**; at $\delta = 1.19$ (2-Bu^t) and 2.15 (6-CH_3) for **10**; at $\delta = 1.19$ (2,6-di-Bu^t) and 2.22 (4-CH_3) for **11**; and at $\delta = 1.22$ (2,6-*di*-Bu^t) and 1.33 (4-Bu^t) for **12**. These data are in good agreement with the literature.[32–34] The protons of the aromatic moiety of compounds **3-12** show multiplets in the range of 6.84–7.97 ppm.

All compounds exhibited only a single absorption peak in the ^{31}P-NMR spectrum between 28.2 and 31.9 ppm, characteristic of phosphinate phosphorus.[35–39] The ^{31}P-NMR signal appears at 29.9 for **3**, 30.1 for **4**, 29.5 for **5**, 30.0 for **6**, 30.3 for **7**, 29.8 for **8**, 29.5 for **9**, 28.2 for **10**, 29.9 for **11**, and 31.9 for **12**.

Compounds **3-12** were also characterized by GC-MS. In the GC-MS spectra of compounds **3-10** the molecular ion peaks (M^+) could be observed, except for compounds **11** and **12**. In these cases only the important fragments at m/z 363 and 391 corresponding to the loss of [$(CH_3)_3$] for **11** and [$(CH_3)_3$) + CH_3] for **12** could be detected.

CONCLUSION

In this study, we described the synthesis of the substituted phenyl diphenylphosphinate derivatives **3–12**. Equimolar quantities of the corresponding sodium phenoxide derivatives and diphenylphosphinic chloride were stirred together overnight at an ambient temperature using THF as a solvent. Novel aryl diphenyl phosphinates **3–12** were successfully prepared.

EXPERIMENTAL

All synthetic steps were carried out under an inert atmosphere of N_2 or Ar in pre-dried glassware by using Schlenk techniques.[40] THF was distilled over a sodium/potassium alloy in the presence of benzophenone under a dry argon atmosphere. Starting materials were commercially available and used without further purification. Reactions were monitored by using silica gel 60 F_{254} precoated thin-layer chromatography (TLC) plates (Kieselgel 60, 0.25 mm thickness) and separating conditions were determined. The separation of products was carried out by column chromatography using silica gel (Kieselgel 60, 230–400 mesh; for 3 g crude mixture, 100 g silica gel was used in a column of 3 cm in diameter and 60 cm in length).

The purity of compounds **3–12** was checked by TLC and characterized by elemental analysis; ^{1}H-, ^{13}C-, ^{31}P-NMR; and FTIR spectroscopy. Microanalysis was carried out with a LECO 932 CHNS-O apparatus. Melting points were measured in open capillary tubes with an Electrothermal-9200 melting point apparatus and are uncorrected. IR spectra were recorded with an ATI Unicam Mattson 1000 FTIR spectrophotometer in KBr disks and are reported in cm^{-1} units. ^{1}H-, ^{13}C-, and ^{31}P-NMR spectra were recorded using a Bruker DPX-300 spectrometer operating at 300.13 MHz (^{1}H), 75.47 MHz (^{13}C), and 121.49 MHz (^{31}P).

Chemical shifts (δ) are given in ppm relative to tetramethylsilane (TMS). All data were recorded for solutions in $CDCl_3$. The ^1H- and ^{13}C-NMR chemical shifts were measured using $SiMe_4$ ($\delta = 0$) as an internal standard, and the ^{31}P chemical shifts were measured using 85% H_3PO_4 as an external standard. Mass spectra were recorded with a Shimadzu GCMS-QP2010 gas chromatograph spectrometer.

Aryl Diphenylphosphinate Esters (3-12): General Procedure

To a solution of the respective phenol (4.23 mmol) in THF (15 mL) at 20 °C during 0.5 h, small pieces of Na (0.25 g, 12 mmol) were slowly added with vigorous stirring, and argon was passed over the reaction mixture. The excess of Na was removed by filtration and the solution of the sodium phenoxide was cooled and then frozen with a liquid nitrogen–acetone mixture. To this mixture $(C_6H_5)_2P(O)Cl$ **1** (4.23 mmol) in 10 mL of THF was slowly added and the resulting mixture was allowed to come to an ambient temperature with constant stirring. After the mixture was vigorously stirred (48 h) at room temperature, the precipitated salt (NaCl) was filtered and the solvent was removed under reduced pressure. The resulting white solid was subjected to column chromatography. The residue was chromatographed on silica gel: 100 g (eluent: acetone/*n*-hexane 1:2 [for **3-10**]; ethylacetate/*n*-hexane 1:3 [for **11** and **12**]).

2,4-Dimethylphenoxy Diphenylphosphinoxide (3). White crystals; yield: 0.54 g (40%); m.p. 87–88 °C. (R_f = 0.324 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3274–3059, υ(CH alkyl) 2962–2924, υ(P=O) 1233, υ(P−Ph) 1438, υ(P−O−Ph) 1197 cm^{-1}; MS m/z (%): 322 (M^+, 100). Anal. Calcd. for $C_{20}H_{19}PO_2$: C, 74.60; H, 5.90. Found: C, 74.47; H, 6.01%.

3,4-Dimethylphenoxy Diphenylphosphinoxide (4). White crystals; yield: 0.51 g (38%); m.p. 119–120 °C. (R_f = 0.459 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3437–3054, υ(CH alkyl) 2984–2919, υ(P=O) 1225, υ(P−Ph) 1438, υ(P−O−Ph) 1192 cm^{-1}; MS m/z (%): 322 (M^+, 100). Anal. Calcd. for $C_{20}H_{19}PO_2$: C, 74.60; H, 5.90. Found: C, 74.49; H, 5.98%.

2,6-Dimethylphenoxy Diphenylphosphinoxide (5). White crystals; yield: 0.38 g (28%); m.p. 124–125 °C. (R_f = 0.540 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3436–3057, υ(CH alkyl) 2953–2920, υ(P=O) 1234, υ(P−Ph) 1437, υ(P−O−Ph) 1168 cm^{-1}; MS m/z (%): 322 (M^+, 100). Anal. Calcd. for $C_{20}H_{19}PO_2$: C, 74.60; H, 5.90. Found: C, 74.72; H, 5.86%.

2,4,6-Trimethylphenoxy Diphenylphosphinoxide (6). White crystals; yield: 0.45 g (32%); m.p. 76–77 °C. (R_f = 0.388 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3276–3058, υ(CH alkyl) 2961–2922, υ(P=O) 1235, υ(P−Ph) 1438, υ(P−O−Ph) 1193 cm^{-1}; MS m/z (%): 336 (M^+, 100). Anal. Calcd. for $C_{21}H_{21}PO_2$: C, 75.04; H 6.52. Found: C, 75.28; H, 6.76%.

4-*tert*-Butylphenoxy Diphenylphosphinoxide (7). White crystals; yield: 0.76 g (52%); m.p. 168–170 °C. (R_f = 0.516 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3435–3057, υ(CH alkyl) 2960–2903, υ(P=O) 1228, υ(P−Ph) 1440, υ(P−O−Ph) 1176 cm^{-1}; MS m/z (%): 350 (M^+, 50), 335 (100). Anal. Calcd. for $C_{22}H_{23}PO_2$: C, 75.49; H, 6.57. Found: C, 75.47; H 6.78%.

4-*tert*-Butyl-2-methyl-phenoxy Diphenylphosphinoxide (8). White solid; yield: 0.66 g (43%); m.p. 138–139 °C. (R_f = 0.441 acetone/*n*-hexane 1:2). IR (KBr,): υ(CH aryl) 3435–3057, υ(CH alkyl) 2959–2903, υ(P=O) 1223, υ(P−Ph) 1439, υ(P−O−Ph)

1188 cm^{-1}; MS m/z (%): 364 (M^+, 55), 349 (100). Anal. Calcd. for $C_{23}H_{25}PO_2$: C, 75.52; H, 6.86. Found: C, 75.42; H, 6.68%.

2-*tert*-Butyl-4-methyphenoxy Diphenylphosphinoxide (9). White solid; yield: 0.43 g (28%); m.p. 125–126 °C. (R_f = 0.388 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3435, υ(CH alkyl) 2950–2920, υ(P=O) 1237, υ(P−Ph) 1439, υ(P−O−Ph) 1204 cm^{-1}; MS m/z (%): 364 (M^+, 70), 349 (100). Anal. Calcd. for $C_{22}H_{23}PO_2$: C, 72.52; H, 6.86. Found: C, 75.02; H, 6.92%.

2-*tert*-Butyl-6-methylphenoxy Diphenylphosphinoxide (10). White crystals; yield: 0.49 g (32%); m.p. 122–123 °C. (R_f = 0.388 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3436–3055, υ(CH alkyl) 2962, υ(P=O) 1238, υ(P−Ph) 1439, υ(P−O−Ph) 1204 cm^{-1}; MS m/z (%): 364 (M^+, 60), 201 (100). Anal. Calcd. for $C_{23}H_{25}PO_2$: C, 75.83; H, 6.86. Found: C, 75.92; H, 6.98%.

2,6-Di-*tert*-butyl-4-methylphenoxy Diphenylphosphinoxide (11). White crystals; yield: 0.42 g (24%); m.p. 149–150 °C. (R_f = 0.472 acetone/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3436–3049, υ(CH alkyl) 2954–2907, υ(P=O) 1228, υ(P−Ph) 1436, υ(P−O−Ph) 1196 cm^{-1}; MS m/z (%): 363 [$(M-C(CH_3)_3)^+$, 100]. Anal. Calcd. for $C_{27}H_{33}PO_2$: C, 77.19; H, 7.86. Found: C, 76.99; H, 7.69%.

2,4,6-Tri-*tert*-butylphenoxy Diphenylphosphinoxide (12). White crystals; yield: 0.71 g (37%); m.p. 196–198 °C. (R_f = 0.538 ethyl acetate/*n*-hexane 1:2). IR (KBr): υ(CH aryl) 3436–3057, υ(CH alkyl) 2956–2903, υ(P=O) 1235, υ(P−Ph) 1438, υ(P−O−Ph) 1197 cm^{-1}; MS m/z (%): 391 [$(M-CH_3)_3 + CH_3$) 20], 201 (100). Anal. Calcd. for $C_{30}H_{39}PO_2$: C, 77.97; H, 8.44. Found: C, 77.03; H, 8.48%.

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