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ORIGINAL PAPER

Synthesis of phosphonato esters involving heterocyclic biological bases in a highly diastereoselective and chemoselective route

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Abstract Synthesis of five phosphonato esters has been accomplished via reaction between dimethyl acetylenedicarboxylate and triphenyl phosphite in the presence of biological compounds such as theophylline, 4-hydroxypyrimidine, 2*H*-3,1-benzoxazine-2,4(1*H*)-dione, 2-chloroaniline, or 3-nitroaniline at ambient temperature. The configuration of the compounds was determined on the basis of coupling constants emerging from the Karplus equation.

Keywords Phosphonato esters · Diastereoselective synthesis · Karplus equation · Biological activity

Introduction

Phosphorus compounds containing a P–C bond are not particularly abundant in nature. Their diverse biological activity [1, 2] has for a long time attracted considerable synthetic [3] and pharmacological interest [4]. In recent years, phosphorus–carbon bond formation has drawn much more attention [5–14]. Thus, new reactions are being developed for preparation of organophosphorus compounds such as phosphonates and phosphinates [15–20].

Phosphonate-containing molecules are an important class of active compounds, and their use and synthesis have received an increasing amount of attention during the last two decades [21–26]. The utilities of phosphonates as antibacterial, antiviral, anticancer, and anti-human immunodeficiency virus (HIV) agents [27–37], as well as chelating agents, insecticides, and plant growth regulators [38], are well documented. Because of their biological effects and medicinal and industrial importance, many procedures have been developed for synthesis of phosphonate derivatives [15, 39]. Among them, the synthesis and chemistry of purine and pyrimidine are of interest, because they exhibit useful pharmaceutical and biological properties. Purine and pyrimidine ring systems form the backbone of many important biological molecules, such as nucleic acids, cofactors, and various toxins [40]. Some purine derivatives such as theophylline have long been used as anti-inflammatory drugs and also in therapy for respiratory diseases such as asthma [41, 42].

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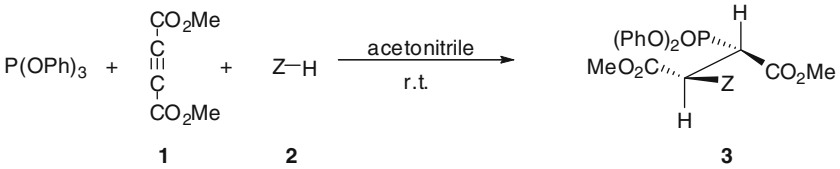
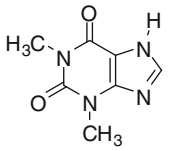
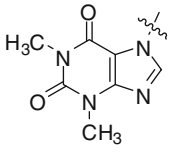
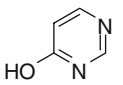
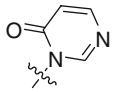
Results and discussions

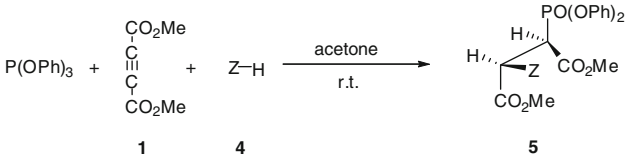
In this work, we describe a simple, short, neutral, and diastereoselective synthesis of phosphonate esters **3a**, **3b**, and **5a**–**5c** at ambient temperature from the reaction between triphenyl phosphite and dimethyl acetylenedicarboxylate **1** in the presence of biological bases such as theophylline, 4-hydroxypyrimidine, 2*H*-3,1-benzoxazine-2,4(1*H*)-dione, 2-chloroaniline, or 3-nitroaniline (Schemes 1, 2).

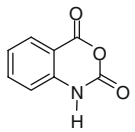
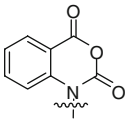
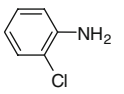
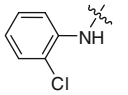
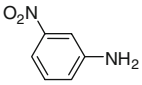
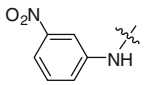
^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra of the crude products clearly indicated formation of phosphonato esters **3a**, **3b**, and **5a**–**5c**. No products other than **3a** and **3b** or **5a**–**5c** could be detected by NMR spectroscopy. The chemical structures of all new phosphonato esters were confirmed by their mass spectra, and infrared (IR) and ^{1}H, ^{13}C, and ^{31}P NMR spectral data. ^{1}H NMR spectra of **3a** and **3b** or **5a**–**5c** displayed two signals for vicinal methine protons which appear as double doublets. The vicinal proton–proton coupling constant ($^{3}J_{HH}$) can help, as a function of the

Scheme 1



	2 (Z-H)	3, Z =	Yield (%)
a			95
b			85



	4 (Z-H)	5, Z =	Yield (%)
a			80
b			90
c			95

Scheme 2

torsion angle, to determine the proton position by the Karplus equation [43–46]. The presence of ^{31}P in **3a**, **3b**, and **5a**–**5c** helps in the assignment of the signals by long-range couplings with ^{1}H and ^{13}C nuclei. The comparison of observed $^{3}J_{CP}$ for the C=O group with the desired values indicates the product geometries. Only one of the two probable diastereoisomers was found in the hydrolysis products. The vicinal proton–proton coupling constant ($^{3}J_{HH}$) as a function of torsion angle can be obtained from the Karplus equation [43–46]. The Karplus relation can be derived from the data for organophosphorus compounds with tetra- and penta-coordinated phosphorus [43–46]. Typically, J_{gauche} varies between 1.5 and 5 Hz, and J_{anti} between 10 and 14 Hz. Observation of $^{3}J_{HH} = 11.0$ Hz for the vicinal protons in compounds **3a** and **3b**, respectively, indicates an anti arrangement for these protons. Since compounds **3** possess two chirality centers, two diastereoisomers with anti H–C–C–H arrangements are possible (Scheme 4). The three-bond carbon–phosphorus coupling $^{3}J_{CP}$ depends on configuration, the transoid coupling being larger than the cisoid one. The $^{3}J_{CP}$ for the P–C–C–C=O moieties are 18.5 and 19.4 Hz, which corresponds to racemic (2*R**,3*S**)-**3a**, **3b**. Observation of $^{3}J_{HH} = 4.2, 4.1$, and 4.9 Hz for the vicinal protons in stereoisomers **5a**–**5c** confirms a gauche arrangement for these protons. Since compounds **5a**–**5c** possess two stereogenic centers, two diastereoisomers [(2*R**,3*R**)–**5a**–**5c** and (2*R**,3*S**)–**5a**–**5c**] with gauche H–C–C–H arrangement are possible (Scheme 5). The $^{3}J_{CP}$ for the P–C–C–C=O moieties are 13.3, 17.2, and 18.1 Hz, which corresponds to racemic (2*R**,3*R**)-**5a**–**5c** geometries.

The work reported here was undertaken in order to determine the possibility of trapping the reactive 1:1 intermediate (Scheme 3) formed in the initial reaction between triphenyl phosphite and dimethyl acetylenedicarboxylate (DMAD) by heterocyclic biological bases, such as theophylline or 4-hydroxypyrimidine. These components produce phosphonato esters **3**, which apparently result from initial addition of triphenyl phosphite to acetylenic ester and concomitant protonation of the 1:1 adduct by heterocyclic biological bases **2** (Scheme 3). Herein, the positively charged ion (phosphonium ion) can be attacked by the Z^- anion (theophylline) through two pathways (**i** and **j**). The result from pathway **i** led to the more stable stereospecific ylide **3i** because of the very powerful dipole–dipole interactions between the positive phosphorus atom and the oxygen atom, which is subsequently converted to the phosphonato ester **3** by nucleophilic attack on the phosphorus atom by water (Scheme 3). In contrast, the less

Scheme 3

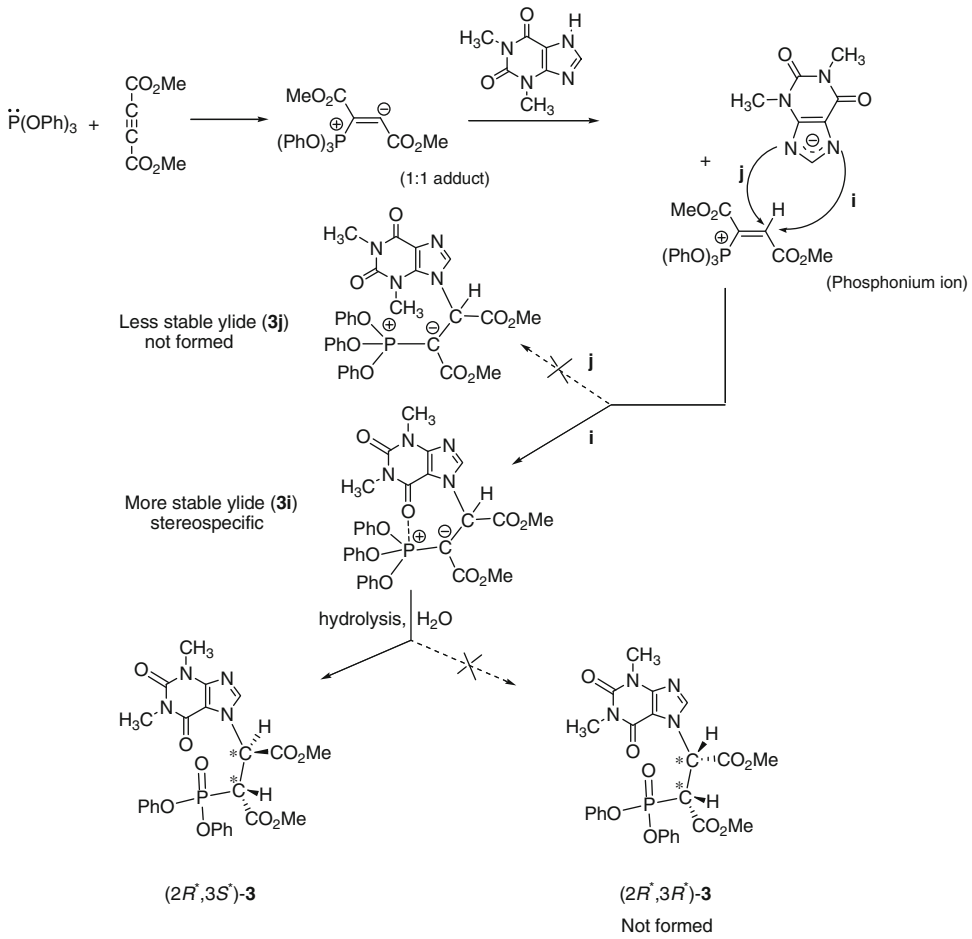


Table 1 Selected NMR data

Cpd.	^{1}H NMR				^{13}C NMR								^{31}P NMR
	δH_2 (ppm)	$^{3}J_{HP}$ (Hz)	δH_3 (ppm)	$^{2}J_{HP}$ (Hz)	δC_1 (ppm)	$^{3}J_{CP}$ (Hz)	δC_2 (ppm)	$^{2}J_{CP}$ (Hz)	δC_3 (ppm)	$^{1}J_{CP}$ (Hz)	δC_4 (ppm)	$^{2}J_{CP}$ (Hz)	δP (ppm)
3a	5.93	5.9	4.87	19.4	166.80	18.5	58.18	4.3	48.43	133.9	166.42	6.9	10.76
3b	5.41	5.6	4.83	20.4	166.89	19.4	53.74	4.2	44.87	134.4	166.64	7.0	10.25
5a	5.12	9.9	4.24	25.0	167.18	13.3	48.64	4.4	43.62	135.6	165.13	5.3	13.11
5b	4.95	8.5	4.19	25.0	171.56	17.2	55.24	4.6	47.45	135.4	167.72	3.9	12.63
5c	5.00	9.0	4.11	24.4	170.72	18.1	53.90	3.4	46.49	136.2	167.04	5.4	12.71

stable ylide **3j**, which lacks dipole–dipole interactions, as a result of pathway **j** could not be formed (Scheme 3).

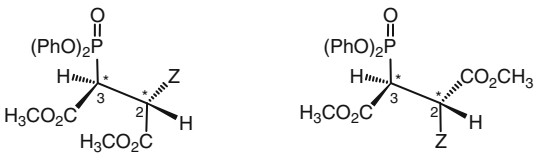
Selected ^{1}H, ^{13}C, and ^{31}P NMR chemical shifts (δ in ppm) and coupling constants (J in Hz) for H-2, H-3, CO_2R, C-2, and C-3 are reported in Table 1 for compounds **3a**, **3b**, and **5a**–**5c** with respect to Schemes 4 and 5 (Scheme 3; Table 1).

These diastereomers were easily separated by removing the solvents under reduced pressure.

In summary, a diastereoselective synthesis of phosphonates has been developed. Our strategy allows preparation of functionalized phosphonato ester derivatives, which are otherwise difficult to make. Particularly attractive is the possibility of access to new phosphonato esters in diastereomerically pure form.

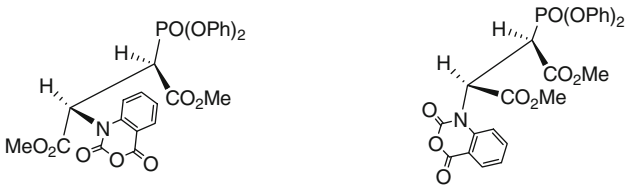
Experimental

All chemicals were commercial products and used without further purification. Melting points were measured on an Electrothermal 9100 apparatus. IR spectra were recorded as



$(2R^*,3S^*)$-**3** $(2S^*,3S^*)$-**3**

Scheme 4



$(2R^*,3R^*)$-**5a** $(2S^*,3R^*)$-**5a**

Scheme 5

KBr pellets on a Shimadzu IR-460 spectrometer. ^{1}H, ^{13}C, and ^{31}P NMR spectra were taken on BRUKER DRX-500, 400, or 300 AVANCE spectrometers with $CDCl_3$ as solvent. Mass spectra were recorded on a Shimadzu GC/MS QP 1100 EX mass spectrometer operating at ionization potential of 70 eV. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer.

General procedure for synthesis of phosphonato esters ***3*** *and* ***5***

To a magnetically stirred solution of 0.31 g triphenyl phosphite (1 mmol) and 1 mmol of the respective base [theophylline, 4-hydroxypyrimidine, 2*H*-3,1-benzoxazine-2,4(1*H*)-dione, 2-chloroaniline, or 3-nitroaniline] in 10 cm^3 acetonitrile was added dropwise 0.142 g dimethyl acetylenedicarboxylate (1 mmol). After approximately 24 h stirring at room temperature, the solvent was removed under reduced pressure and the product washed with cold diethyl ether/*n*-hexane 50:50 (2 × 5 cm^3).

Dimethyl (2R,3S*)-2-(diphenoxyphosphinyl)-3-(1,2,3,6-tetrahydro-1,3-dimethyl-2,6-dioxo-7H-purine-7-yl)succinate* (**3a**, $C_{25}H_{25}N_4O_9P$)

White powder, yield 95%; m.p.: 126–127 °C; IR (KBr): $\bar{\nu}$ = 1,752, 1,738, 1,698 and 1,654 (C=O), 1,224 (P=O) cm^{-1}; ^{1}H NMR (500.1 MHz, $CDCl_3$): δ = 3.28 and 3.51 (2s, 6H, 2N–CH_3), 3.74 and 3.87 (2s, 6H, 2 OCH_3), 4.87 (dd, 1H, $^2J_{PH}$ = 19.4 Hz, $^3J_{HH}$ = 11.0 Hz, P–C*H*–CH), 5.93 (dd, 1H, $^3J_{PH}$ = 5.9 Hz, $^3J_{HH}$ = 11.0 Hz, P–CH–C*H*), 6.89–7.29 (m, 10H, 2 OPh), 7.78 (s, 1H, N=CH) ppm; ^{13}C NMR (125.8 MHz, $CDCl_3$): δ = 27.84 and 29.72 (2q, 2N–CH_3), 48.43 (d, $^1J_{CP}$ = 133.9 Hz, P–*C*H–CH), 53.32 and 53.79 (2q, 2 O*C*H_3), 58.18 (d, $^2J_{CP}$ = 4.3 Hz, P–CH–*C*H), 107.05 (1C, $C_7H_7N_4O_2$), 119.70 (d, $^3J_{PC}$ = 4.7 Hz, C_{ortho} of OPh), 119.82 (d, $^3J_{PC}$ = 4.9 Hz, C_{ortho} of OPh), 125.46 and 125.61 (2d, 2 C_{para} of 2 OPh), 129.64 and 129.80 (2d, 4 C_{meta} of 2 OPh), 143.61, 149.03 (2C, $C_7H_7N_4O_2$), 149.48 (s, $^2J_{PC}$ = 9.3 Hz, C_{ipso} of OPh) and 149.70 (s, $^2J_{CP}$ = 9.1 Hz, C_{ipso} of OPh), 151.30 and 154.90 (2s, 2 *C*=O of $C_7H_7N_4O_2$), 166.43 (s, $^2J_{CP}$ = 6.9 Hz, P–C–*C*=O), 166.80 (s, $^3J_{CP}$ = 18.5 Hz, P–C–C–*C*=O) ppm; ^{31}P NMR (202.4 MHz, $CDCl_3$): δ = 10.76 [s, $(PhO)_2PO$] ppm; EI–MS: *m*/*z* (%) = 556 (M^+, 39), 463 (35), 376 (20), 283 (47), 223 (69), 180 (100), 165 (15), 94 (16), 77 (71).

Dimethyl (2R,3S*)-2-(diphenoxyphosphinyl)-3-(6-oxo-1(6H)-pyrimidinyl)succinate* (**3b**, $C_{22}H_{21}N_2O_8P$)

White powder, yield 90%; m.p.: 82–84 °C; IR (KBr): $\bar{\nu}$ = 1,733 and 1,670 (C=O), 1,206 (P=O) cm^{-1}; ^{1}H NMR (300.1 MHz, $CDCl_3$): δ = 3.77 and 3.85 (2s, 6H, 2 O*C*H_3), 4.83 (dd, 1H, $^2J_{PH}$ = 20.4 Hz, $^3J_{HH}$ = 11.0 Hz, P–C*H*–CH), 5.41 (dd, 1H, $^3J_{PH}$ = 5.6 Hz, $^3J_{HH}$ = 11.0 Hz, P–CH–C*H*), 6.42 (d, 1H, $^3J_{HH}$ = 6.7 Hz, CH of $C_4H_3N_2O$), 6.80–7.40 (m, 10H, 2 OPh), 7.89 (d, 1H, $^3J_{HH}$ = 6.7 Hz, CH of $C_4H_3N_2O$), 8.36 (1H, s, CH of $C_4H_3N_2O$) ppm; ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 44.87 (d, $^1J_{CP}$ = 134.4 Hz, P–*C*H–CH), 52.38 and 52.78 (2q, 2 O*C*H_3), 53.74 (d, $^2J_{CP}$ = 4.2 Hz, P–CH–*C*H), 120.21 (d, $^3J_{CP}$ = 4.4 Hz, 2 C_{ortho} of 2 OPh), 120.4 (d, $^3J_{CP}$ = 4.3 Hz, 2 C_{ortho} of 2 OPh), 124.38 (s, C of $C_4H_3N_2O$), 125.67 and 125.87 (2d, 2 C_{para} of 2 OPh), 129.51 and 129.90 (2d, 4 C_{meta} of 2 OPh), 149.19 (s, $^2J_{CP}$ = 9.6 Hz, C_{ipso} of OPh), 149.55 (s, $^2J_{CP}$ = 9.7 Hz, C_{ipso} of OPh), 153.70 and 156.13 (2s, 2 C of $C_4H_3N_2O$), 166.64 (s, $^2J_{CP}$ = 7.0 Hz, P–C–*C*=O), 166.77 (s, C of $C_4H_3N_2O$), 166.89 (s, $^3J_{CP}$ = 19.4 Hz, P–C–C–*C*=O) ppm; ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 10.25 [s, $(PhO)_2PO$] ppm; EI–MS: *m*/*z* (%) = 472 (M^+, 2), 376 (7), 285 (11), 250 (62), 223 (10), 169 (18), 96 (30), 94 (100), 77 (44).

Dimethyl (2R,3R*)-2-[2,4-dioxo-2H-3,1-benzoxazin-1(4H)-yl]-3-(diphenoxyphosphinyl)succinate* (**5a**, $C_{26}H_{22}NO_{10}P$)

White powder, yield 80%; m.p.: 144–116 °C; IR (KBr): $\bar{\nu}$ = 1,761, 1,731 (C=O), 1,215 (P=O) cm^{-1}; ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 3.71 and 3.89 (2s, 6H, 2 OCH_3), 4.24 (dd, 1H, $^2J_{PH}$ = 25.0 Hz, $^3J_{HH}$ = 4.2 Hz, P–C*H*–CH), 5.12 (br dd, 1H, $^3J_{PH}$ = 9.9 Hz, $^3J_{HH}$ = 4.2 Hz, P–CH–C*H*), 6.70 (t, 1H, J_{HH} = 7.4 Hz, Ar–H), 6.94–7.41 (m, 12H, Ar–H), 7.95 (dd, 1H, J_{HH} = 8.0 Hz, J_{HH} = 1.5 Hz, Ar–H) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = 43.62 (d, $^1J_{PC}$ = 135.62 Hz, P–*C*H–CH), 48.64 (d, $^2J_{PC}$ = 4.4 Hz, P–CH–*C*H), 52.10 and 52.28 (2q, 2 OCH_3), 111.45 and 115.58 (2d, 2 CH of Ar), 119.40 (d, $^3J_{PC}$ = 4.4 Hz, 2 C_{ortho} of OPh), 119.76 (d, $^3J_{PC}$ =

4.5 Hz, 2 C_{ortho} of OPh), 123.16 (d, CH of Ar), 124.18 and 124.64 (2d, 2 C_{para} of 2 OPh), 128.45 (d, CH of Ar), 128.55 and 128.55 (2d, 4 C_{meta} of 2 OPh), 133.18 (s, C of Ar), 148.73 (s, $^2J_{PC}$ = 7.8 Hz, C_{ipso} of OPh), 149.20 (s, $^2J_{PC}$ = 8.1 Hz, C_{ipso} of OPh), 154.63 (s, C of Ar), 165.13 (s, $^2J_{PC}$ = 5.3 Hz, CO), 167.18 (s, $^3J_{PC}$ = 13.3 Hz, CO), 168.16 and 170.33 (2s, CO) ppm; ^{31}P NMR (161.9 MHz, $CDCl_3$): δ = 13.11 [s, $(PhO)_2PO$] ppm.

Dimethyl (2R,3R*)-2-[(2-chlorophenyl)amino]-3-(diphenoxyphosphinyl)succinate* (**5b**, $C_{24}H_{23}ClNO_7P$)
White powder, yield 90%; m.p.: 107–110 °C; IR (KBr): $\bar{\nu}$ = 3,380 (N–H), 1,749, 1,718 (C=O), 1,221 (P=O) cm^{-1}; ^{1}H NMR (300.1 MHz, $CDCl_3$): δ = 3.71 and 3.89 (2s, 6H, 2 OCH_3), 4.19 (dd, 1H, $^2J_{PH}$ = 25.0 Hz, $^3J_{HH}$ = 4.1 Hz, P–C*H*–CH), 4.95 (ddd, 1H, $^3J_{HH}$ = 11.0 Hz, $^3J_{PH}$ = 8.5 Hz, $^3J_{HH}$ = 4.1 Hz, P–CH–C*H*), 5.77 (d, 1H, $^3J_{HH}$ = 11.0 Hz, CH–N*H*), 6.69 (dt, 1H, J_{HH} = 8.0 Hz, J_{HH} = 1.38 Hz, Ar–H), 6.93 (dd, 1H, J_{HH} = 8.3 Hz, J_{HH} = 0.9 Hz, Ar–H), 6.99–7.37 (m, 12H, 2 OPh and Ar–H) ppm; ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 47.45 (d, $^1J_{PC}$ = 135.4 Hz, P–*C*H–CH), 53.07 and 53.34 (2q, 2 OCH_3), 55.24 (d, $^2J_{PC}$ = 4.6 Hz, P–CH–*C*H), 113.27 and 119.04 (2d, 2 CH of Ar), 120.50 (d, $^3J_{PC}$ = 4.5 Hz, 2 C_{ortho} of OPh), 120.55 (d, CH of Ar), 120.78 (d, $^3J_{PC}$ = 4.5 Hz, 2 C_{ortho} of OPh), 125.31 (d, C_{para} of OPh), 129.50 (d, 2 C_{meta} of OPh), 125.69 (d, 2 C_{para} of 2 OPh), 127.14 (d, CH of Ar), 129.23 (s, C of Ar), 129.90 (d, 2 C_{meta} of OPh), 142.65 (s, C of Ar), 149.85 (s, $^2J_{PC}$ = 8.8 Hz, C_{ipso} of OPh), 149.91 (s, $^2J_{PC}$ = 8.6 Hz, C_{ipso} of OPh), 167.72 (s, $^2J_{PC}$ = 3.9 Hz, CO), 171.56 (s, $^3J_{PC}$ = 17.2 Hz, CO) ppm; ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 12.63 [s, $(PhO)_2PO$] ppm; EI–MS: *m/z* (%) = 505 (M^++2, 63), 504 (M^++1, 94), 503 (M^+, 100), 488 (3), 472 (6), 444 (91), 441 (3), 412 (66), 378 (14), 345 (7), 223 (13), 77 (84).

Dimethyl (2R,3R*)-2-(diphenoxyphosphinyl)-3-[(3-nitrophenyl)amino]succinate* (**5c**, $C_{24}H_{23}N_2O_9P$)
Yellow powder, yield 95%; m.p.: 115–117 °C; IR (KBr): $\bar{\nu}$ = 3,309 (N–H), 1,745, 1,725 (C=O), 1,233 (P=O) cm^{-1}; ^{1}H NMR (400.1 MHz, $CDCl_3$): δ = 3.71 and 3.82 (2s, 6H, 2 OCH_3), 4.11 (dd, 1H, $^2J_{PH}$ = 24.4 Hz, $^3J_{HH}$ = 4.9 Hz, P–C*H*–CH), 5.00 (br dd, 1H, $^3J_{PH}$ = 9.0 Hz, $^3J_{HH}$ = 4.9 Hz, P–CH–C*H*), 6.90 (dd, 1H, $^3J_{HH}$ = 9.0 Hz, $^4J_{PH}$ = 2.3 Hz, CH–N*H*), 7.10–7.35 (m, 12H, 2 OPh and Ar–H), 7.46 (t, 1H, J_{HH} = 2.0 Hz, Ar–H), 7.59 (dd, 1H, J_{HH} = 8.1 Hz, J_{HH} = 1.7 Hz, Ar–H) ppm; ^{13}C NMR (100.6 MHz, $CDCl_3$): δ = 46.49 (d, $^1J_{PC}$ = 136.2 Hz, P–*C*H–CH), 52.18 and 52.46 (2q, 2 OCH_3), 53.90 (d, $^2J_{PC}$ = 3.4 Hz, P–CH–*C*H), 106.85, 112.68, and 114.26 (3d, 3 CH of Ar), 119.42 (d, $^3J_{PC}$ = 4.3 Hz, 2 C_{ortho} of OPh), 119.60 (d, $^3J_{PC}$ = 4.5 Hz, 2 C_{ortho} of OPh), 124.51 and 124.60 (2d, 2 C_{para} of 2 OPh), 128.48 (d, 2 C_{meta} of OPh), 128.71 (s, C of Ar), 128.73 (d, 2 C_{meta} of OPh), 145.84 (s, C of Ar), 148.65 (s, $^2J_{PC}$ = 8.4 Hz, C_{ipso} of OPh), 149.01 (s, $^2J_{PC}$ = 8.6 Hz, C_{ipso} of OPh), 156.49 (s, C of Ar), 167.04 (s, $^2J_{PC}$ = 5.4 Hz, CO), 170.72 (s, $^3J_{PC}$ = 18.1 Hz, CO) ppm; ^{31}P NMR (161.9 MHz, $CDCl_3$): δ = 12.71 [s, $(PhO)_2PO$] ppm; EI–MS: *m/z* (%) = 514 (M^+, 3), 482 (1), 376 (12), 345 (4), 251 (8), 223 (43), 138 (100), 92 (91), 65 (95), 41 (7).

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