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Synthesis of Aromatic Amine Phosphonato Ester Derivatives from the Stereoselective Reaction Between Triphenyl Phosphite and Dimethyl Acetylenedicarboxylate in the Presence of Derivatives of Aromatic Amines

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ABSTRACT: *Aromatic amine phosphonato esters* **4a–d** *were obtained in excellent yields from the 1:1:1 addition reaction between triphenyl phosphite and dimethyl acetylenedicarboxylate in the presence of NH-aromatic amines such as 2-aminobenzophenone, 2-aminoacetophenon, methyl-2-aminobenzoate, and 2-aminobenzonitrile. In the recent works, the assignments of the configuration of* **4a–d** *corresponding to the three-bond carbon-phosphorus coupling,* ^3Jpc*, was determined on the basis of coupling constants by the Karplus equation as* **2R*,3R*** *or* **2S*,3S*** *while they were* **2R*,3S*** *or* **2S*,3R*** *in our previous works in the presence of same solvent.* © 2009 Wiley Periodicals, Inc. Heteroatom Chem 20:240–245, 2009; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20541

INTRODUCTION

In recent years, there has been an increasing interest in the synthesis of organophosphorus compound. This interest has resulted from the recognition of the value of such compounds in a wide range of industrial, biological, and chemical synthetic aspects [1–21]. The successful attack by nucleophilic trivalent phosphorus on a carbon atom is facilitated when the latter is part of, or conjugated with, a carbonyl group, or when it is part of an unsaturated bond otherwise activated [2–4, 22–27]. There are many studies on the reaction between trivalent phosphorus nucleophiles and α,β-unsaturated carbonyl compounds in the presence of a proton source such as alcohol or phenol [2,27,28]. New or improved methods for phosphonate synthesis continue to attract much attention because phosphonates have biologically important properties and serve as natural products, analogues of phosphates, phosphonopeptides, amino acid analogues, and prodrugs. Also the phosphonate esters have physiological activity within the cell [29–34].

RESULT AND DISCUSSION

In the current work, we wish to report a simple, short, and neutral stereoselective synthesis of phosphonate esters from reaction between triphenyl phosphite **1** and dimethyl acetylenedicarboxylate **2** in the presence of NH-aromatic amines

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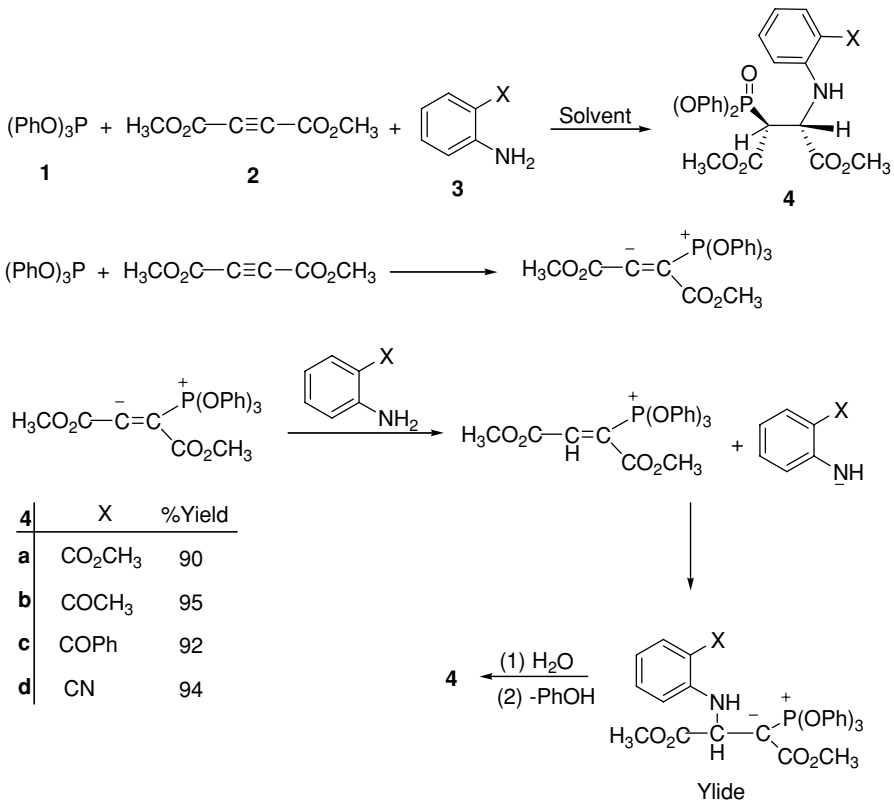
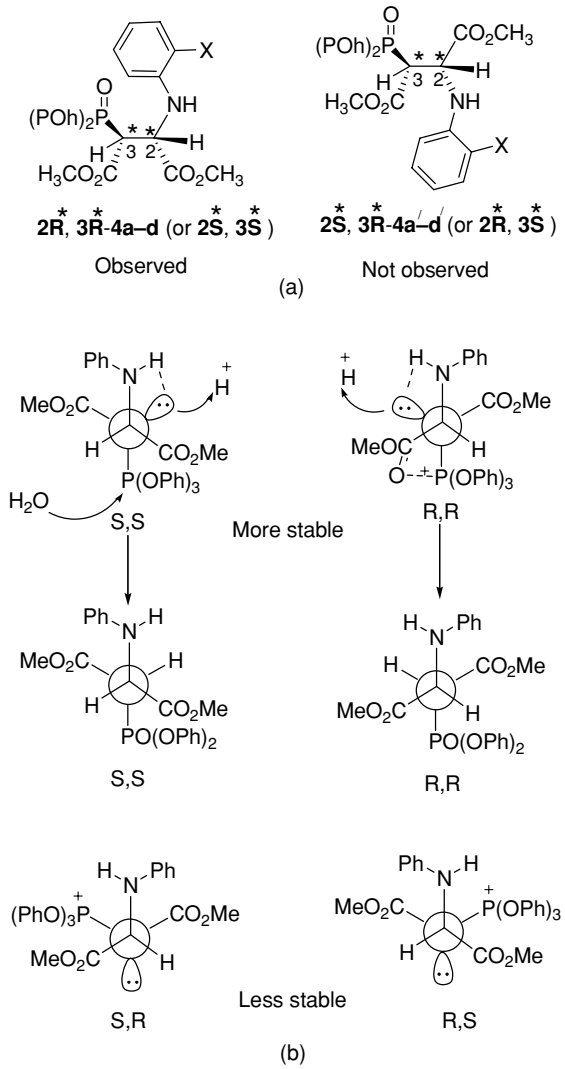


4	X	%Yield
a	CO_2CH_3	90
b	$COCH_3$	95
c	COPh	92
d	CN	94

SCHEME 1

with an orthoelectron-withdrawing group **3**, such as 2-aminobenzophenone, 2-aminoaceto-phenon, methyl-2-aminobenzoate, and 2-aminobenzonitrile for generation of **4a–d** in good yield (see Scheme 1 for the suggested reaction mechanism). The reactions were carried out in a suitable mixture of diethyl ether/*n*-hexane in which the reaction was completed within a few hours. The ^{1}H and ^{13}C NMR spectra of the crude product clearly indicated the formation of phosphonate esters **4a–d**. Any products other than **4a–d** could not be detected by NMR spectroscopy. The structures of compounds **4a–d** were deduced by IR, ^{1}H, ^{13}C, and ^{31}P NMR spectra, mass spectrometry, and also elemental analysis. The mass spectra of compounds **4a–d** displayed molecular ionic peaks at appropriate values, which were consistent with 1:1:1 adducts of NH-aromatic amines, DMAD, and triphenyl phosphite. Although the presence of the ^{31}P nucleus complicates both the ^{1}H and ^{13}C NMR spectra of **4a**, it helps in assignment of the signals by long-range couplings with the ^{1}H and ^{13}C nuclei (see the Experimental section). The ^{1}H NMR (500 MHz) spectra of compound **4a** displayed three sharp lines (δ = 3.69, 3.81, 3.89) arising from methoxy protons, along with signals for methine protons at δ = 4.22 ppm (dd, $^2J_{PH}$ = 24.9 Hz, $^3J_{HH}$ = 4.3 Hz) and δ = 5.08 ppm (ddd, $^3J_{PH}$ = 8.1 Hz, $^3J_{HH}$ = 4.3, $^3J_{HH}$ = 10.6 Hz) which appear as a doublet of doublet and three doublet of doublet, respectively, for the P—CH—CH and P—CH—CH—NH groups. In addition, the NH group appears as a doublet at δ = 8.91 ppm ($^3J_{HH}$ = 10.6 Hz, CH—CH—NH). The vicinal proton–proton coupling constant ($^3J_{HH}$) as a function of the torsion angle can be obtained from the Karplus equation [1,35]. Typically, J_{gauche} varies between 1.5 and 5 Hz and J_{anti} between 10 and 14 Hz. Observation of $^3J_{HH}$ = 4.3 Hz for the vicinal protons in compound **4a** (see the Experimental section) indicates the gauche arrangement for these protons. Since compound **4a** possess two stereogenic centers, two diastereoisomers with gauche arrangements are possible (see Scheme 2a). The three-bond carbon–phosphorus coupling constant, $^3J_{CP}$, depends on the configuration, as expected, being larger for the transoid coupling than cisoid one. The Karplus relation can be derived from the data for organophosphorus compound with tetra- and pentavalent phosphorus [36]. The observation of $^3J_{CP}$ of 16.6 Hz for the ester C=O group (see the Experimental section) is in a good agreement with **2R*,3R*** (**4a**) or its mirror image **2S*,3S*** (**4a**) geometries (see Scheme 2a) [14]. Following configurations that are depicted for **4a–d**



SCHEME 2 (a) Two diastereoisomers with H,H gauche arrangements of aromatic amine phosphonato esters **4a–d** (the recent work). (b) Favor dipole–dipole intraction and also intramolecular hydrogen bond for creation of the stereospecific product (**2R***,**3R*** or **2S***,**3S***).

are assumed to be the predominant rotamers that are relevant to presumably rapid rotation about the C_2—C_3 bond, in NMR time scale. The ^{1}H and ^{13}C NMR spectra of **4b–d** are similar to those of **4a** except for the ester groups, which exhibited characteristic resonances with appropriate chemical shifts (see the Experimental section). The structural assignments made on the basis of the ^{1}H and ^{13}C NMR spectra of compounds **4a–d** were supported by the IR spectra. The carbonyl region of the spectra exhibited two distinct absorption bands for each compound (see the Experimental section). In the recent work under the same conditions with the previous work, assignment of the configuration for **4a–d** lead to **2R***,**3R*** or **2S***,**3S*** arrangement, while they were **2R***,**3S*** or **2S***,**3R*** in our previous investigations [8–14]. It seems that the structural effect in some reactants is an important factor for changing the arrangement of diastereoisomers (see Schemes 2 and 3). At the Arbusove rearrangement under the action of H_2O to phosphorus atom of recent ylide, the first step of hydrolysis, perhaps, is accompanied by very powerful dipole–dipole intraction between the adjacent ester group and phosphonium ion and also intermolecular hydrogen bond between additional hydrogen atom in aromatic amine with negative charge of the ylide region (see Scheme 2b).

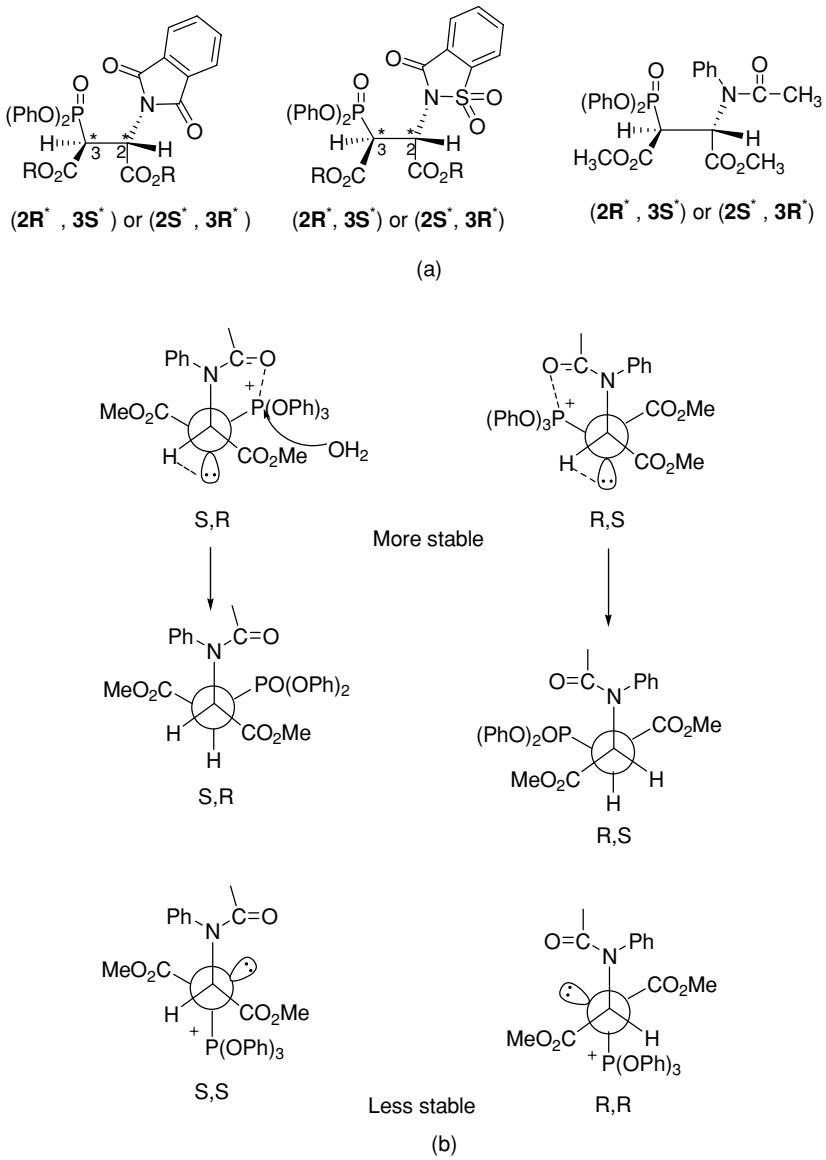
These intramolecular interactions cause a blockage in the molecule; hence, this appears to be a plausible favor for performance of the stereospecific reaction [37]. With report to different NH-acids in our previous work, favor intractions create a different blockage in the molecule (see Scheme 3b) for achievement of other stereospecific reaction with different arrangement **R***,**S*** or **S***,**R*** in comparison with recent works **R***,**R*** or **S***,**S***. In both cases, the results will be changed (a mixture of two diastereoisomers) while the reactions are carried out in the presence with higher dielectric constant solvent; in conclusion, a facil and new diastereoselective synthesis of phosphonato ester is reported. The present method is a simple diastereoselective procedure under mild condition with a good yield.

EXPERIMENTAL

Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a Shimadzu IR-460 spectrometer, respectively. Also, the ^{1}H, ^{13}C, and ^{31}P NMR spectra were obtained on a Bruker DRX-500 Avance instrument with $CDCl_3$ as solvent at 500.1, 125.8, and 202.4 MHz, respectively. In addition, the mass spectra were recorded on a GCMS-QP5050A mass spectrometer operating at an ionization potential of 70 eV. Elemental analysis for C, H, and N were performed on a Heraeus CHN-O-rapid analyzer. Dimethyl acetylenedicarboxylates, triphenl phosphite, 2-aminobenzophenone, 2-aminoacetophenone, methyl-2-aminobenzoat, and 2-amino-benzonitrile were purchased from Fluka and were used without further purifications.

*Preparation of (**2R***,**3R***)-Dimethyl-2-[2-(methoxycarbonyl)phenylamino]-3-(diphenoxy phosphoryl)-butanedioate (**4a**)*

General Procedure. To a magnetically stirred solution of triphenyl phosphite (0.31 g, 1 mmol) and methyl-2-aminobenzoate (0.15 g, 1 mmol) in a



SCHEME 3 (a) Diastereoisomers with H,H arrangement for the phosphonato esters in the previous work. (b) Favor dipole–dipole intraction for generation of other stereospecific product (**2S*, 3R*** or **2R*, 3S***).

mixture of diethyl ether/*n*-hexane (10 mL) was added dropwise a solution of dimethyl acetylenedicarboxylate (0.14 g, 1 mmol) in diethyl ether (5 mL) at −10°C over 10 min. After approximately 24 h stirring at room temperature, the solvent was removed under reduced pressure and product washed with cold diethyl ether (2×5 mL).

White powder. mp = 96–98°C, 0.47 g, 90%. IR (KBr) (ν_{max}, cm^{-1}): 3270 (N—H), 1725 and 1715 (C=O), 1271 (P=O). MS (*m/z*, %): 527 (M^+, 14), 468 (M-CO_2CH_3, 8), 436 (M-CO_2CH_3, and OCH_3, 12), 283 (M-$C_8H_8NO_2$ and OPh, 9), 223 (M-$2CO_2CH_3$ and 2OPh, 13), 77 (Ph, 100). Anal. Calcd for $C_{26}H_{26}NO_9P$ (527): C, 59.18; H, 4.97; N, 2.66%. Found: C, 60.03; H, 4.89; N, 2.54%. ^{1}H NMR (500.1 MHz, $CDCl_3$) δ: 3.69, 3.81, and 3.79 (9H, 3s, $3OCH_3$), 4.22 (1H, dd, $^2J_{PH}$ = 24.9 Hz, $^3J_{HH}$ = 4.3 Hz, P—CH—CH), 5.08 (1H, ddd, $^3J_{PH}$ = 8.1 Hz, $^3J_{HH}$ = 4.3 Hz, $^3J_{HH}$ = 10.6 Hz, P—CH—CH—NH), 6.64–7.89 ($14H_{aro}$, m, $2C_6H_5$ and C_6H_4), 8.91 (1H, d, $^3J_{HH}$ = 10.6 Hz, NH–CH). ^{13}C NMR (125.8 MHz, δ, $CDCl_3$): 47.51 (d, $^1J_{CP}$ = 137.3 Hz, P–CH), 51.64, 53.00, and 53.29 (3s, $3OCH_3$), 54.14 (d, $^2J_{CP}$ = 2.8 Hz, P–C–CH), 112.08, 112.52, and 116.46 (3C, $C_8H_8NO_2$), 120.35 and 120.78 (2d, $^3J_{PC}$ = 4.4 Hz C_{ortho} of $2C_6H_5$), 125.13 and 125.60 (C_{para} of $2C_6H_5$), 129.35 and 129.83 (C_{meta} of $2C_6H_5$), 131.53, 134.44, and 149.61 (3C, $C_8H_8NO_2$), 149.91 (d, $^2J_{CP}$ = 8.2 Hz ,C_{ipso} of $2C_6H_5$), 167.15 (d,

$^2J_{CP}$ = 5.1 Hz, C=O),168.40 (O=C–OCH_3), 171.47 (d, $^3J_{CP}$ = 16.6 Hz, C=O). ^{31}P NMR (202.4 MHz, δ, $CDCl_3$): 13.19 [s, $(PhO)_2P(=O)$].

*(**2R***,**3R***)-Dimethyl-2-[2-acetylphenylamino]-3-(diphenoxyphosphoryl)-butanedioate (**4b**)*

White powder. mp = 103–105°C, 0.49 g, 95%. IR (KBr) (ν_{max}, cm^{-1}): 3250 (N–H), 1730 and 1719 (C=O), 1265 (P=O). MS (*m/z*, %): 511 (M^+, 28), 452 (M-CO_2CH_3, 21), 378 (M-C_8H_8NO, 24), 77 (Ph, 100), 43 ($COCH_3$, 43). Anal. Calcd for $C_{26}H_{26}NO_8P$ (511): C, 61.03; H, 5.13; N, 2.74%. Found: C, 61.17; H, 5.21; N, 2.65%. ^{1}H NMR (500.1 MHz, $CDCl_3$) δ: 2.53 (3H, s, CH_3), 3.71 and 3.91 (6H, 2s, $2OCH_3$), 4.23 (1H, dd, $^2J_{PH}$ = 24.9 Hz, $^3J_{HH}$ = 4.5 Hz, P–CH–CH), 5.13 (1H, ddd, $^3J_{PH}$ = 8.1 Hz, $^3J_{HH}$ = 4.5 Hz, $^3J_{HH}$ = 10.6 Hz, P–CH–CH–NH), 6.66–7.74 ($14H_{aro}$, m, $2C_6H_5$ and C_6H_4), 9.17 (1H, d, $^3J_{HH}$ = 10.6 Hz, NH–CH). ^{13}C NMR (125.8 MHz, δ, $CDCl_3$): 27.93 (CH_3), 47.48 (d, $^1J_{CP}$ = 137.4 Hz, P–CH), 53.03 and 53.28 (2s, $2OCH_3$), 53.72 (d, $^2J_{CP}$ = 2.3 Hz, P–C–CH), 112.65, 115.91, and 119.18 (3C , C_8H_8NO), 120.43 and 120.80 (2d, $^3J_{PC}$ = 4.6 Hz, C_{ortho} of $2C_6H_5$), 125.08 and 125.61 (C_{para} of $2C_6H_5$), 129.36 and 129.84 (C_{meta} of $2C_6H_5$), 132.40, 134.90, and 149.34 (3C , C_8H_8NO), 149.92 (d, $^2J_{CP}$ = 8.8 Hz , C_{ipso} of $2C_6H_5$), 166.83 (d, $^2J_{CP}$ = 5.1 Hz, C=O), 171.28 (d, $^3J_{CP}$ = 16.2 Hz, C=O), 200.62 (O=C–CH_3). ^{31}P NMR (202.4 MHz, δ, $CDCl_3$): 12.14 [s, $(PhO)_2P$=O].

*(**2R***,**3R***)-Dimethyl-2-[2-benzoylphenylamino]-3-(diphenoxyphosphoryl)-butanedioate (**4c**)*

White powder. mp = 93–95°C, 0.53 g, 92%. IR (KBr) (ν_{max}, cm^{-1}): 3280 (N–H), 1736 and 1725 (C=O), 1273 (P=O). MS (*m/z*, %): 573 (M^+, 6), 514 (M-OCH_3, 4), 376 (M-$C_{13}H_{10}NO$, 5), 283 (M-$C_{13}H_{10}NO$ and OPh, 24), 105 (PhCO, 33), 77 (Ph, 100). Anal. Calcd for $C_{31}H_{28}NO_8P$ (573): C, 64.90; H, 4.92; N, 2.44%. Found: C, 65.01; H, 4.99; N, 2.53%. ^{1}H NMR (500.1 MHz, $CDCl_3$) δ: 3.68 and 3.90 (6H, 2s, $2OCH_3$), 4.20 (1H, dd, $^2J_{PH}$ = 25.0 Hz, $^3J_{HH}$ = 4.2 Hz, P–CH–CH), 5.13 (1H, ddd, $^3J_{PH}$ = 8.0 Hz, $^3J_{HH}$ = 4.2 Hz, $^3J_{HH}$ = 10.6 Hz, P–CH–CH–NH), 6.62–7.56 ($19H_{aro}$, m, $3C_6H_5$ and C_6H_4), 9.43 (1H, d, $^3J_{HH}$ = 10.6 Hz, NH–CH). ^{13}C NMR (125.8 MHz, δ, $CDCl_3$): 47.31 (d, $^1J_{CP}$ = 137.5 Hz, P–CH), 53.08 and 53.37 (2s, $2OCH_3$), 54.48 (d, $^2J_{CP}$ = 2.7 Hz, P–C–CH), 113.24, 115.84, and 119.69 (3C, $C_{13}H_{10}NO$), 120.50 and 120.84 (2d, $^3J_{PC}$ = 4.4 Hz, C_{ortho} of $2C_6H_5$), 125.17 and 125.66 (C_{para} of $2C_6H_5$), 127.89 (2C, $C_{13}H_{10}NO$), 129.38 and 129.43 (C_{meta} of $2C_6H_5$), 129.87, 131.00, 134.65, and 134.86 (5C , $C_{13}H_{10}NO$), 149.79 (d, $^2J_{CP}$ = 9.7 Hz, C_{ipso} of $2C_6H_5$), 149.97 and 150.13 (2C, $C_{13}H_{10}NO$), 166.94 (d, $^2J_{CP}$ = 5.3 Hz, C=O), 171.44 (d, $^3J_{CP}$ = 16.8 Hz, C=O), 198.90 (O=C–Ph). ^{31}P NMR (202.4 MHz, δ, $CDCl_3$): 13.43 [s, $(PhO)_2P$=O].

*(**2R***,**3R***)-Dimethyl-2-[2-cyanophenylamino]-3-(diphenoxyphosphoryl)-butanedioate (**4d**)*

White powder. mp = 84–86°C, 0.46 g, 94%. IR (KBr) (ν_{max}, cm^{-1}): 1741 and 1719 (C=O), 1268 (P=O) and 2214 (CN). Anal. Calcd for $C_{25}H_{23}N_2O_7P$ (494): C, 60.71; H, 4.69; N, 5.67%. Found: C, 61.78; H, 4.58; N, 5.60%. ^{1}H NMR (500.1 MHz, $CDCl_3$) δ: 3.75 and 3.94 (6H, 2s, $2OCH_3$), 4.29 (1H, dd, $^2J_{PH}$ = 23.7 Hz, $^3J_{HH}$ = 4.8 Hz, P–CH–CH), 5.01 (1H, ddd, $^3J_{PH}$ = 8.5 Hz, $^3J_{HH}$ = 4.8, $^3J_{HH}$ = 10.7 Hz, P–CH–CH–NH), 5.97 (1H, d, $^3J_{HH}$ = 10.7 Hz, NH–CH). 6.73–7.44 ($14H_{aro}$, m, $2C_6H_5$ and C_7H_5N). ^{13}C NMR (125.8 MHz, δ, $CDCl_3$): 47.14 (d, $^1J_{CP}$ = 137.1 Hz, P–CH), 53.37 and 53.66 (2s, $2OCH_3$), 54.68 (d, $^2J_{CP}$ = 4.2 Hz, P–C–CH), 112.7 (1C, C_7H_5N), 115.47 (CN), 117.35 (1C, C_7H_5N), 120.31 and 120.59 (2d, $^3J_{PC}$ = 4.1 Hz, C_{ortho} of $2C_6H_5$), 125.29 and 126.05 (C_{para} of $2C_6H_5$), 129.46 and 129.76 (C_{meta} of $2C_6H_5$), 149.66 and 149.84 (2d, $^2J_{CP}$ = 8.5 Hz , C_{ipso} of $2C_6H_5$), 167.49 (d, $^2J_{CP}$ = 5.3 Hz, C=O), 170.47 (d, $^3J_{CP}$ = 18.1 Hz, C=O). ^{31}P NMR (202.4 MHz, δ, $CDCl_3$): 10.05 [s, $(PhO)_2P$=O].

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