

Stereoselective one-pot synthesis of functionalised phosphonates by three-component reaction between trialkyl(aryl) phosphites, dimethyl acetylenedicarboxylate and indane-1,3-dione or *N,N'*-dimethylbarbituric acid

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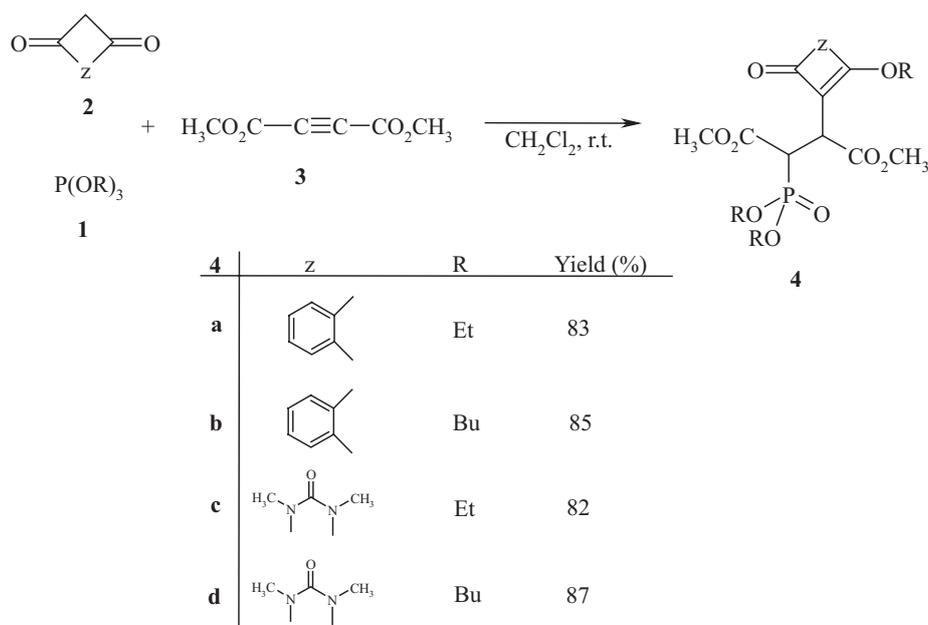
Protonation of the reactive intermediate produced in the reaction between trialkyl(aryl) phosphites and dimethyl acetylenedicarboxylate by CH-acids such as indane-1,3-dione and *N,N'*-dimethylbarbituric acid leads to functionalised phosphonates in good yields.

Keywords: dimethyl acetylenedicarboxylate, phosphites, indane-1,3-dione, *N,N'*-dimethylbarbituric acid, stereoselective synthesis

Organophosphorus compounds, those bearing a carbon atom bound directly to a phosphorus atom, are synthetic targets of interest, at least because of their value for a variety of industrial, biological and chemical synthetic uses.¹⁻³ As a result, a large number of methods have appeared describing novel syntheses of organophosphorus compounds. The 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.¹⁻⁹ There are many studies on the reaction between trivalent phosphorus nucleophiles and α , β -unsaturated carbonyl compounds using alcohols or phenols as reaction adjuncts.¹⁰ The reaction of trimethyl phosphite and dimethyl acetylenedicarboxylate (DMAD) in the presence of alcohols reported to produce phosphite ylide derivatives which are stable at low temperatures, but converted to phosphonate derivatives by warming or by treatment with water.¹¹ There are some other recent reports on the reaction between phosphites and acetylenic esters in the presence of an acidic organic compound, all of them going through a phosphite ylide intermediate.¹²⁻¹⁴ The reaction of trimethyl phosphite and dimethyl acetylenedicarboxylate (DMAD) in the presence of organic CH-acid indane-1,3-dione reported to produce phosphonate derivatives.¹⁵ These products were

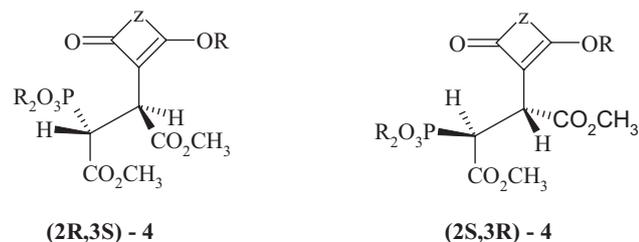
reported to be obtained from a phosphate ylide intermediate by an intramolecular nucleophilic substitution. Similar products were obtained from the reaction between acetylenic esters, triphenyl phosphite and 5,5-dimethylcyclohexan-1,3-dione (dimedone).¹⁶ In order to study the scope of the three-component reaction between active organic CH-acids, acetylenic esters and phosphites and in continuation of our works on the reaction between trivalent phosphorus nucleophiles and acetylenic esters in the presence of organic NH, OH, or CH-acids,^{12,17-19} here we report the results of our study on the reaction between dimethyl acetylenedicarboxylate and phosphites such as triethyl phosphite, tributyl phosphite or triphenyl phosphite in the presence of strong CH-acidic compounds indane-1,3-dione or *N,N'*-dimethylbarbituric acid. Thus, the reaction of dimethyl acetylenedicarboxylate with trialkyl phosphite **1** in the presence of CH-acid leads to the corresponding phosphonates **4a-d** in fairly high yields (Scheme 1). This three-component reaction produces the hitherto unknown butanedioates **4a-d** in 82–87% yields.

All the compounds **4a-d** are stable crystalline solids whose structure is fully supported by elemental analyses and IR, ¹H NMR, ¹³C NMR, ³¹P NMR and mass spectral data. For example, the ¹H NMR spectra of **4a** displayed three triplets (³J_{HH} = 7 Hz) for methyls of the ethoxy groups. Two singlets



Scheme 1

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Scheme 2

were observed at 3.66 and 3.81 ppm, related to two methoxy groups. Protons of three methylenes of ethoxy groups along with the CHP proton resonate between 4.01 and 4.30 ppm as multiplets. The other methine proton resonates at 4.68 ppm as a double doublet ($^3J_{\text{HH}} = 12$ Hz, $^3J_{\text{HP}} = 6$ Hz). The presence of the ^{31}P nucleus in compounds **4a–d** helps in assignment of the signals by long-range couplings with ^1H and ^{13}C nuclei (see Experimental).

The vicinal proton–proton coupling constant ($^3J_{\text{HH}}$) as a function of torsion angle can be obtained from the Karplus equation.^{20,21} Typically, J_{gauche} varies between 1.5 and 5 Hz and J_{anti} between 10 and 14 Hz. Observation of $^3J_{\text{HH}} = 11$ –12 Hz for the vicinal protons in compounds **4a–d** indicates an anti arrangement for these protons. Since compounds **4** possess two stereogenic centres, two diastereoisomers with anti HCCH arrangements are possible (Scheme 2). The three-bond carbon–phosphorus coupling, $^3J_{\text{CP}}$, depends on configuration, as expected, *transoid* couplings being larger than *cisoid* ones. The Karplus relation can be derived from the data for organophosphorus compounds with tetra- and penta-valent phosphorus.²² The observation of $^3J_{\text{CP}}$ of 20–21 Hz for the ester C=O group, and of 2.5–3 Hz for the carbon of CH-acid moiety is in agreement with the (2R,3S) – **4** and its mirror image (2S,3R) – **4** geometries.

On the basis of the well-established chemistry of trivalent phosphorus nucleophiles,^{3–10} it is reasonable to assume that compound **4** results from the initial addition of trialkyl phosphite **1** to dimethyl acetylenedicarboxylate and a concomitant protonation of the 1:1 adduct by indane-1,3-dione or *N,N'*-dimethylbarbituric acid (Scheme 3). Then, the positively charged ion **5** is attacked by the enolate anion **6** of the CH-acid to form phosphite ylide **7**, which is then rearranged

to 1,4-diionic compound **8**. Intermediate **8** converted to the phosphonate ester **4** by intramolecular nucleophilic attack on the alkyl group.

Three component reaction between triphenyl phosphite, DMAD and indan-1,3-dione or *N,N'*-dimethylbarbituric acid afforded phosphonate derivatives **9a** and **9b** respectively (Scheme 4). It is reasonable to assume that compounds **9a** and **9b** are produced in a way similar to the mechanism proposed for the formation of compounds **4a–d**. Here the intermediate phosphite ylide **7** is hydrolysed to the phosphonate **9** and a molecule of phenol is released.

The ^1H NMR spectra of compound **9a** displayed signals for methine protons at 4.07 (dd, $^2J_{\text{HP}} = 21$ Hz, $^3J_{\text{HH}} = 12$ Hz), 4.31 (d, $^3J_{\text{HH}} = 1.5$ Hz), and 4.52 (ddd, $^3J_{\text{HP}} = 5$ Hz, $^3J_{\text{HH}} = 12$ Hz, $^3J_{\text{HH}} = 3$ Hz). Two sharp singlets were observed for methoxy protons at 3.40 and 3.88 ppm. Compound **9a**, as compounds **4a–d** can exist as two diastereomers. The observation of $^3J_{\text{HH}}$ of 12 Hz for vicinal methine protons and of $^3J_{\text{CP}}$ of 21 Hz for the ester C=O group, is in agreement with the (2R,3S)-**9a** and its mirror image (2S,3R)-**9a** geometries (Scheme 5). The NMR spectra of compound **9b** also show only (2R,3S)-**9b** isomer and its enantiomer (The same geometries that were observed for compounds **4a–d**)

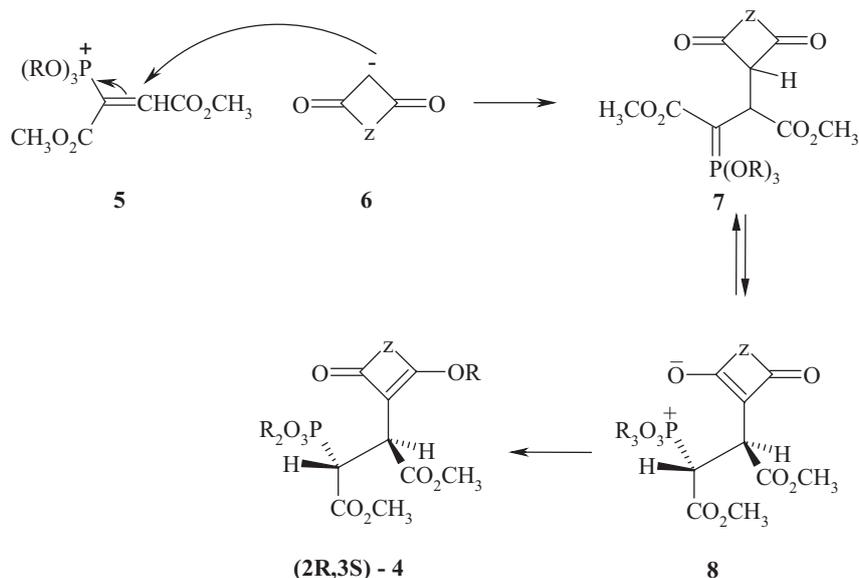
In summary, stable crystalline phosphonates may be prepared stereoselectively by a simple, one-pot three-component reaction between acetylenic esters, phosphites, and indan-1,3-dione or *N,N'*-dimethylbarbituric acid. The present method carries the advantage that not only is the reaction performed under neutral conditions but also that the substances can be mixed without any activation or modification.

Experimental

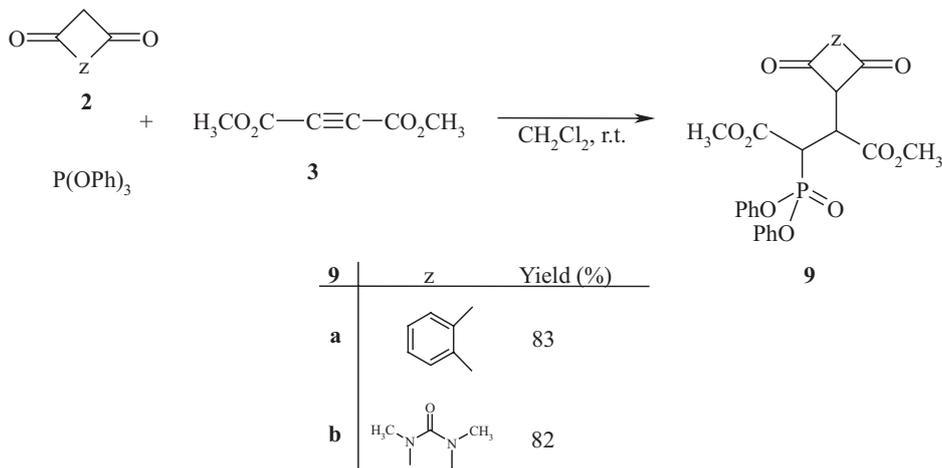
Melting points were determined with an electrothermal 9100 apparatus. Elemental analyses were performed using a Heraeus CHN–O–Rapid analyser. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionisation potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^1H , ^{13}C , and ^{31}P NMR spectra were recorded on Bruker DRX-500 Avance spectrometer at solution in d_6 -DMSO using TMS as internal standard or 85% H_3PO_4 as external standard. The chemicals used in this work purchased from fluka (Buchs, Switzerland) and were used without further purification.

General procedure

To a magnetically stirred solution of CH-acid (2 mmol) and dimethyl acetylenedicarboxylate (2 mmol) in dichloromethane (4 ml)



Scheme 3



Scheme 4

was added dropwise a mixture of trialkyl(aryl) phosphite (2 mmol) in dichloromethane (4 ml) at room temperature over 10 min. The reaction mixture was then allowed to stir for 24 h. The solvent was removed under reduced pressure and the residue was purified by silica gel (Merck silica gel 60, 230–400 mesh) column chromatography using hexane-ethyl acetate as eluent. The solvent was removed under reduced pressure to afford the product.

Dimethyl 2-(3-ethoxy-1-oxoinden-2-yl)-3-(diethylphosphonato)butanedioate (4a): Yellow crystals, m.p. 104–106°C, IR (KBr) (ν_{\max} cm^{-1}): 1725, 1712 (2 C=O, ester); 1619 (C=O). Analyses: Calcd. for $\text{C}_{21}\text{H}_{27}\text{O}_9\text{P}$: C, 55.51; H, 5.99%. Found: C, 55.4; H, 5.6%. MS (m/z , %): 454 (7). ^1H NMR (500 MHz, d_6 -DMSO- Me_4Si): δ 1.01, 1.18 and 1.54 (9 H, 3 t, $^3J_{\text{HH}} = 7$ Hz, 3 CH_3), 3.66 and 3.81 (6 H, 2 s, 2 OCH_3), 4.01–4.19 (5H, m, 2 OCH_2 and CH), 4.21–4.30 (2 H, m, OCH_2), 4.68 (1 H, dd, $^3J_{\text{HP}} = 6$ Hz, $^3J_{\text{HH}} = 12$ Hz, CH), 7.24–7.40 (4 H, m, 4 CH aromatic). ^{13}C NMR (125.8 MHz, d_6 -DMSO- Me_4Si): δ 15.69, 16.21 and 16.51 (3 CH_3), 40.31 (d, $^2J_{\text{CP}} = 2$ Hz, P–C), 45.37 (d, $^1J_{\text{CP}} = 131$ Hz, P–C), 53.23 and 53.13 (2 OCH_3), 63.46 (d, $^2J_{\text{CP}} = 7$ Hz, POCH_2), 63.02 (d, $^2J_{\text{CP}} = 7$ Hz, POCH_2), 68.39 (OCH_2), 104.98 (d, $^3J_{\text{CP}} = 2.4$ Hz, C=C–O), 119.73, 121.17, 130.19, 132.27 (4 CH), 132.88, 140.89 (2 C), 169.78 (d, $^2J_{\text{CP}} = 6$ Hz, C=O), 171.89 (d, $^3J_{\text{CP}} = 21$ Hz, C=O), 172.56 (C=C–O), 195.45 (C=O). ^{31}P NMR (202.5 MHz, d_6 -DMSO): δ 22.41.

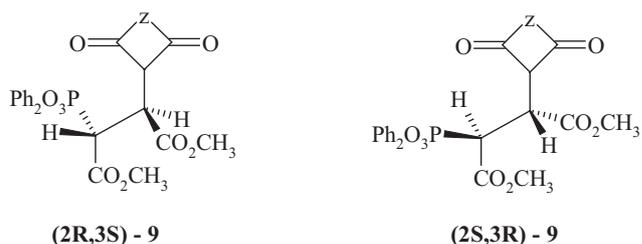
Dimethyl 2-(3-butoxy-1-oxoinden-2-yl)-3-(dibutylphosphonato)butanedioate (4b): Yellow crystals, m.p. 116–118°C, IR (KBr) (ν_{\max} cm^{-1}): 1732, 1719 (2 C=O, ester); 1628 (C=O). Analyses: Calcd. for $\text{C}_{27}\text{H}_{39}\text{O}_9\text{P}$: C, 60.21; H, 7.30%. Found: C, 60.3; H, 7.1%. MS (m/z , %): 538 (6). ^1H NMR (500 MHz, d_6 -DMSO- Me_4Si): δ 0.84, 0.92, and 3.94 (9 H, 3 t, 3 CH_3), 1.26 (4 H, sextet, 2 CH_2), 1.40 (2 H, sextet, CH_2), 1.42 (4 H, quintet, 2 CH_2), 1.65 (2 H, quintet, CH_2), 3.41 and 3.54 (6 H, 2 s, 2 OCH_3), 3.72–3.85 (5 H, m, 2 OCH_2 and CH), 4.10–4.25 (2 H, m, OCH_2), 4.59 (1 H, dd, $^3J_{\text{HP}} = 6$ Hz, $^3J_{\text{HH}} = 11$ Hz, CH), 7.27–7.99 (4 H, m, 4 CH aromatic). ^{13}C NMR (125.8 MHz, d_6 -DMSO- Me_4Si): δ 13.75, 13.73 and 13.95 (3 CH_3), 19.49, 19.52 and 19.57 (3 CH_2), 31.86 and 31.93 (2 d, $^3J_{\text{CP}} = 7$ Hz, 2 CH_2), 32.02 (CH_2), 40.07 (d, $^2J_{\text{CP}} = 2$ Hz, P–C–C), 42.61 (d, $^1J_{\text{CP}} = 127$ Hz, P–C), 49.86 and 52.47 (2 OCH_3), 67.28 and 68.36 (2 d, $^2J_{\text{CP}} = 7$ Hz, 2 POCH_2), 68.77 (OCH_2), 103.36 (d, $^3J_{\text{CP}} = 3$ Hz, C=C–O), 121.59, 123.61, 131.24, 133.26 (4 CH), 133.96, 141.76 (2 C), 170.03 (d, $^2J_{\text{CP}} = 6$ Hz, C=O), 172.15 (d, $^3J_{\text{CP}} = 21$ Hz, C=O), 173.46 (C=C–O), 191.83 (C=O). ^{31}P NMR (202.5 MHz, d_6 -DMSO): δ 23.11.

Dimethyl 2-(3-ethoxy-1-oxo(N,N dimethyl-2-yl)-3-(diethylphosphonato)butanedioate (4c): White powder, m.p. 107–109°C, IR (KBr) (ν_{\max} cm^{-1}): 1725 (C=O, ester), 1631 and 1694 (C=O). Analyses: Calcd. for $\text{C}_{18}\text{H}_{29}\text{N}_2\text{O}_{10}\text{P}$: C, 46.55; H, 6.29; N, 6.03%. Found: C, 46.4; H, 6.2; N, 6.4%. MS (m/z , %): 464 (9). ^1H NMR (500 MHz, d_6 -DMSO- Me_4Si): δ 1.17 (6 H, t, 2 CH_3), 1.48 (3 H, t, CH_3), 3.23 and 3.34 (6 H, 2 s, 2 NCH_3), 3.60 and 3.75 (6 H, 2 s, 2 OCH_3), 3.89–4.01 (4 H, m, 2 OCH_2), 4.12–4.26 (3 H, m, OCH_2 and CH), 4.38 (1 H, dd, $^3J_{\text{HP}} = 6$ Hz, $^3J_{\text{HH}} = 12$ Hz, CH). ^{13}C NMR (125.8 MHz, d_6 -DMSO- Me_4Si): δ 15.73, 16.47 and 16.55 (3 CH_3), 28.43 and 30.56 (2 NCH_3), 40.87 (d, $^2J_{\text{CP}} = \text{Hz}$, P–C–C), 44.72 (d, $^1J_{\text{CP}} = 129$ Hz, P–C), 53.08 and 53.26 (2 OCH_3), 63.07 and 63.23 (2 d, $^2J_{\text{CP}} = 7$ Hz, 2 POCH_2), 72.58 (OCH_2), 98.14 (d, $^3J_{\text{CP}} = 2.5$ Hz, C=C–O), 151.65 (NCON), 159.40 (NCO), 163.64 (C=C–O), 170.03 (d, $^2J_{\text{CP}} = 6$ Hz, C=O), 172.15 (d, $^3J_{\text{CP}} = 20$ Hz, C=O). ^{31}P NMR (202.5 MHz, d_6 -DMSO): δ 22.63.

Dimethyl 2-(3-butoxy-1-oxo(N,N dimethyl-2-yl)-3-(diethylphosphonato)butanedioate (4d): White powder, m.p. 101–103°C, IR (KBr) (ν_{\max} cm^{-1}): 1721 (C=O, ester), 1689 and 1627 (C=O). Analyses: Calcd. for $\text{C}_{24}\text{H}_{41}\text{N}_2\text{O}_{10}\text{P}$: C, 52.55; H, 7.53; N, 5.11%. Found: C, 52.5; H, 7.4; N, 5.7%. MS (m/z , %): 548 (6). ^1H NMR (500 MHz, d_6 -DMSO- Me_4Si): δ 0.84, 0.92 and 1.11 (9 H, 3 t, 3 CH_3), 1.27 (4 H, sextet, 2 CH_2), 1.45 (2 H, sextet, CH_2), 1.58 (4 H, quintet, 2 CH_2), 1.79 (2 H, quintet, CH_2), 3.21 and 3.31 (6 H, 2 s, 2 NCH_3), 3.57 and 3.72 (6 H, 2 s, 2 OCH_3), 3.81–3.92 (4 H, m, 2 OCH_2), 4.05–4.21 (3 H, m, OCH_2 and CH), 4.37 (1 H, dd, $^3J_{\text{HP}} = 6$ Hz, $^3J_{\text{HH}} = 11.5$ Hz, CH). ^{13}C NMR (125.8 MHz, d_6 -DMSO- Me_4Si): δ 13.81, 13.83 and 13.93 (3 CH_3), 18.78, 18.82 and 19.21 (3 CH_2), 28.26 and 30.15 (2 NCH_3), 32.58 and 32.61 (2 d, $^3J_{\text{CP}} = 7$ Hz, 2 CH_2), 32.66 (CH_2), 40.64 (d, $^2J_{\text{CP}} = 2$ Hz, P–C–C), 44.01 (d, $^1J_{\text{CP}} = 130$ Hz, P–C), 52.89 and 53.08 (2 OCH_3), 66.52 and 66.78 (2 d, $^2J_{\text{CP}} = 7$ Hz, 2 POCH_2), 76.31 (OCH_2), 98.01 (d, $^3J_{\text{CP}} = 2.5$ Hz, C=C–O), 151.49 (NCON), 159.37 (NCO), 163.54 (C=C–O), 169.98 (d, $^2J_{\text{CP}} = 6$ Hz, C=O), 171.93 (d, $^3J_{\text{CP}} = 21$ Hz, C=O). ^{31}P NMR (202.5 MHz, d_6 -DMSO): δ 23.17.

Dimethyl 2-(indane-1,3-dione-2-yl)-3-(diphenoxyphosphoryl)butanedioate (9a): Yellow crystals, m.p. 99–101°C, IR (KBr) (ν_{\max} cm^{-1}): 1742, 1727, 1715 (3 C=O); Analyses: Calcd. for $\text{C}_{27}\text{H}_{23}\text{O}_9\text{P}$: C, 62.07; H, 4.44%. Found: C, 62.5; H, 4.3%. MS (m/z , %): 522 (7). ^1H NMR (500 MHz, d_6 -DMSO- Me_4Si): δ 3.40 and 3.88 (6 H, 2 s, 2 OCH_3), 4.07 (1 H, dd, $^2J_{\text{HP}} = 21$ Hz, $^3J_{\text{HH}} = 12$ Hz, P–CH), 4.31 (1 H, d, $^3J_{\text{HH}} = 1.5$ Hz, CH), 4.52 (1 H, ddd, $^3J_{\text{HP}} = 5$ Hz, $^3J_{\text{HH}} = 12$ Hz, $^3J_{\text{HH}} = 1.5$ Hz, P–C–CH), 7.13–7.98 (14 H, m, 14 CH aromatic). ^{13}C NMR (125.8 MHz, d_6 -DMSO- Me_4Si): δ 41.74 (CH), 44.61 (d, $^1J_{\text{CP}} = 141$ Hz, P–C), 51.66 (CH), 53.31 and 53.56 (2 OCH_3), 116.74, 120.93 (2 CH, aromatic), 123.77 (d, $^3J_{\text{CP}} = 5$ Hz, 4 CH_{ortho}), 126.08 (2 CH_{para}), 129.76, 130.29 (2 CH, aromatic), 135.98 (d, $^4J_{\text{CP}} = 8$ Hz, 4 CH_{meta}), 136.21, 142.04 (2 C, aromatic), 150.19 (d, $^2J_{\text{CP}} = 10$ Hz, C_{ipso}), 150.46 (d, $^2J_{\text{CP}} = 9$ Hz, C_{ipso}), 168.35 (d, $^2J_{\text{CP}} = 7$ Hz, C=O), 170.99 (d, $^3J_{\text{CP}} = 21$ Hz, C=O), 196.85 and 198.63 (2 C=O). ^{31}P NMR (202.5 MHz, d_6 -DMSO): δ 22.18.

Dimethyl 2-(N,N dimethyl barbituric acid -2-yl)-3-(diphenoxyphosphoryl)butanedioate (9b): White powder, m.p. 126–128°C, IR (KBr) (ν_{\max} cm^{-1}): 1728, 1699, 1645 (C=O). Analyses: Calcd. for $\text{C}_{24}\text{H}_{25}\text{N}_2\text{O}_{10}\text{P}$: C, 54.14; H, 4.73; N, 5.26%. Found: C, 53.9; H, 4.5; N, 5.6%. MS (m/z , %): 532 (7). ^1H NMR (500 MHz, d_6 -DMSO- Me_4Si): δ 3.24 and 3.26 (6 H, 2 s, 2 NCH_3), 3.70 and 3.88 (6 H, 2 s, 2 OCH_3), 4.34 (1 H, dd, $^2J_{\text{HP}} = 22$ Hz, $^3J_{\text{HH}} = 12$ Hz, P–CH), 4.74 (1 H,



Scheme 5

d, $^3J_{\text{HH}} = 1.5 \text{ Hz}$, CH), 4.78 (1 H, ddd, $^3J_{\text{HP}} = 5 \text{ Hz}$, $^3J_{\text{HH}} = 12 \text{ Hz}$, $^3J_{\text{HH}} = 1.5 \text{ Hz}$, P-C-CH), 7.09–7.36 (10 H, m, 10 CH aromatic). ^{13}C NMR (125.8 MHz, d_6 -DMSO- Me_4Si): δ 29.10 and 29.13 (2 NCH_3), 41.66 (CH), 44.32 (d, $^1J_{\text{CP}} = 145 \text{ Hz}$, P-C), 50.05 (CH), 53.54 and 53.75 (2 OCH_3), 120.84 (d, $^3J_{\text{CP}} = 5 \text{ Hz}$, 4 CH_{ortho}), 126.10 (s, 2 CH_{para}), 130.19 (d, $^4J_{\text{CP}} = 8 \text{ Hz}$, 4 CH_{meta}), 149.92 (d, $^2J_{\text{CP}} = 10 \text{ Hz}$, C_{ipso}), 150.31 (d, $^2J_{\text{CP}} = 8 \text{ Hz}$, C_{ipso}), 151.95 (NCON), 167.0 and 167.48 (2 NCO), 168.26 (d, $^2J_{\text{CP}} = 7 \text{ Hz}$, C=O), 172.77 (d, $^3J_{\text{CP}} = 22 \text{ Hz}$, C=O). ^{31}P NMR (202.5 MHz, d_6 -DMSO): δ 22.34.

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