Monatshefte für Chemie 138, 1267–1271 (2007)
DOI 10.1007/s00706-007-0711-5
Printed in The Netherlands

Monatshefte für Chemie
Chemical Monthly

A Synthesis of Dialkyl Phosphorylsuccinates from the Reaction of NH-Acids with Dialkyl Acetylenedicarboxylates in the Presence of Trialkyl(aryl) Phosphites

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Received April 9, 2007; accepted (revised) May 13, 2007; published online September 24, 2007

Summary. The reaction of dialkyl acetylenedicarboxylates with trialkyl(aryl) phosphites in the presence of isatin, phthalimide, indole, or pyrrole leads to stable dialkyl(aryl) phosphorylsuccinates in excellent yields.

Keywords. Acetylenic esters; Phosphites; Phosphonates; Isatin; Three-component reaction.

Introduction

Organophosphorus compounds are synthesis targets of interest, not least because of their value for a variety of industrial, biological, and chemical synthetic uses [1–5]. The physical properties and chemical reactivity of phosphate esters interlinks many areas in chemistry and biology. Introduction of a phosphate monoester into a molecule such as a drug candidate enhances the water solubility, hence altering its bioavailability [6–8]. As a result, a large number of methods have appeared describing novel syntheses of organophosphorus compounds [3]. 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 [9–11]. We report the reaction of dimethyl acetylenedicarboxylate (*DMAD*) or di-*tert*-butyl acetylenedicarboxylate (*DTAD*) with a trivalent phosphorus nucleophile such as trimethyl, triethyl, or triphenyl phosphite in the presence of various heterocyclic N-H acids.

The reaction of trialkyl(aryl) phosphites **1** and dialkyl acetylenedicarboxylates **2** in the presence of isatin (**3**) proceeds smoothly in CH_2Cl_2 at ambient temperature to produce dialkyl 2-(dialkoxyphosphoryl)-3-(2,3-dioxo-2,3-dihydro-1*H*-indol-1-yl)succinates **4** in 88–94% yields (Scheme 1).

Results and Discussion

The reactions were carried out by mixing the acetylenic ester with **3**, then the trialkyl(aryl) phosphite was added slowly. The reactions were complete within 24 h. The structures of compounds **4a**–**4f** as 1:1:1 adducts were apparent from their mass spectra, which displayed, in each case, the molecular ion peak at appropriate m/z values. The ^{1}H and ^{13}C NMR spectroscopic data, as well as IR spectra, are in agreement with the proposed structures. The ^{1}H NMR spectrum of **4a** exhibited two doublets readily recognized as arising from the two diastereotopic methoxy ($\delta = 2.85$ ppm, $^3J_{HP} = 11$ Hz and $\delta = 3.67$ ppm, $^3J_{HP} = 11$ Hz) groups. The two singlets at $\delta = 3.71$ and 3.82 ppm belong to the ester methoxy protons. The proton-decoupled ^{13}C NMR spectrum of **4a** showed sixteen distinct resonances in agreement with the proposed structure.

Observation of $^3J_{HH} = 13$ Hz for the vicinal methine protons in **4a** indicates the dominance of

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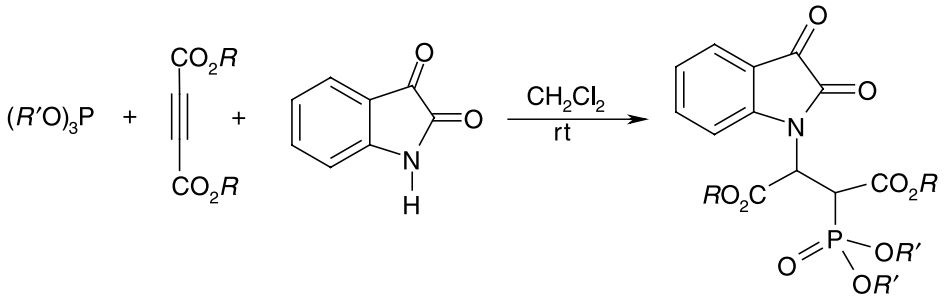


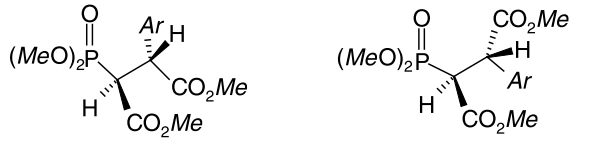
	R′
1a	*Me*
1b	*Et*
1c	*Ph*

	R
2a	*Me*
2b	*t*Bu

3

	R	R′	Yield/%
4a	*Me*	*Me*	89
4b	*Me*	*Et*	94
4c	*Me*	*Ph*	92
4d	*t*Bu	*Me*	89
4e	*t*Bu	*Et*	88
4f	*t*Bu	*Ph*	89

Scheme 1



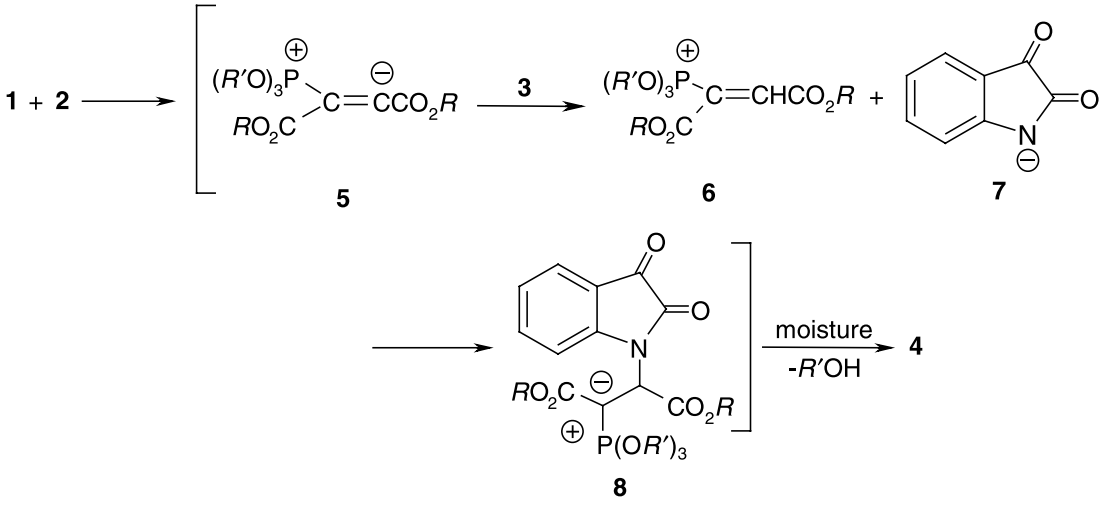
(2*S*,3*R*)-**4a** or (2*R**,3*S**)-**4a** (2*S*,3*S*)-**4a** or (2*R**,3*R**)-**4a**

Scheme 2

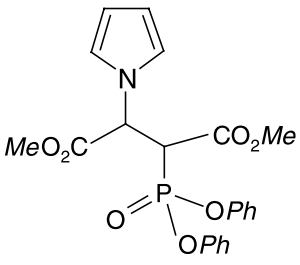
the *anti* arrangement. Since compound **4a** possesses two stereogenic centers, two diastereomers with *anti* HCCH arrangement are possible (Scheme 2).

The observation of $^{3}J_{CP} = 24$ Hz for the carbonyl carbon atom of CO_2Me group is in agreement with the (2*S*,3*R*) or (2*R*,3*S*) diastereomer. However, as long as the coupling constant of at least one other diastereomer is not known, the assignment remains uncertain.

Although we have not established the mechanism of the reaction between trialkyl(aryl) phosphites and dialkyl acetylenedicarboxylates in the presence of isatin in an experimental manner, a possible explanation is proposed in Scheme 3. The first step may involve addition of trialkyl(aryl) phosphites to the acetylenic ester and formation [9–11] of the 1:1 adducts **5** and its subsequent protonation by isatin. Then, the positively charged ion **6** is attacked by the anion of the NH-acid **7** to produce ylide **8**, which is



Scheme 3



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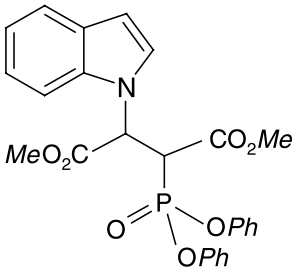

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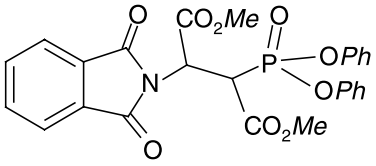

11

Scheme 4

hydrolyzed to give **4**. Since the reactions were carried out under an ordinary atmosphere, the conversion of **8** to **4** is presumably accomplished by the moisture from the air. Also, it is possible that **8** is converted on chromatography to **4** (Scheme 3).

The reaction of triphenyl phosphite with *DMAD* in the presence of pyrrole, indol, or phthalimide led to **9**, **10**, or **11** in excellent yields (Scheme 4).

Observation of $^3J_{HH} = 12.6$ Hz for the vicinal methine protons in **11** indicates the dominance of the *anti* arrangement. The observation of $^3J_{CP} = 22.5$ Hz for CO_2Me group is in agreement with the (2*S*,3*R*) or (2*R*,3*S*) diastereoisomer. The NMR spectral data for compounds **9**–**10** are also in agreement with the (2*S*,3*R*) or (2*R*,3*S*) diastereoisomer.

In summary, the reaction of dialkyl acetylenedicarboxylates with trialkyl(aryl) phosphites in the presence of isatin, phthalimide, indole, or pyrrole provides a simple one-pot synthesis of stable dialkyl(aryl) phosphorylsuccinates of potential synthetic and pharmaceutical interest. The present procedure has the advantage that the reaction is performed under neutral conditions, and the starting material can be used without any activation or modification.

Experimental

General

Compounds **1**–**3** were obtained from Fluka and were used without further purification. Mp: Electrothermal-9100 apparatus. IR spectra: Shimadzu IR-460 spectrometer. ^{1}H, ^{13}C, and ^{31}P NMR spectra: Bruker DRX-500 AVANCE instrument; in $CDCl_3$ at 500.1, 125.7, and 202.4 MHz; δ in ppm, *J* in Hz. EI-MS (70 eV): Finnigan-MAT-8430 mass spectrometer, in *m*/*z*. Elemental analyses (C, H, and N) were performed with a Heraeus CHN-O-Rapid analyzer; their results were found to agree favourably with the calculated values.

*General Procedure for the Preparation of Compounds **4**, **9**, **10**, and **11***

To a stirred solution of 2 mmol **2** and 2 mmol **3** in 10 cm^3 anh. CH_2Cl_2 was added drop-wise a mixture of 2 mmol **1** in 5 cm^3 CH_2Cl_2 at $-5°$ over 10 min. The mixture was then allowed to warm to rt, and stirred for 24 h. The solvent was removed under reduced pressure, and the residue was purified by column chromatography (SiO_2; *n*-hexane/*Ac*O*Et* = 4/1) to afford the pure adducts.

Dimethyl 2-(dimethoxyphosphoryl)-3-(2,3-dioxo-2,3-dihydro-1H-indol-1-yl)succinate (**4a**, $C_{16}H_{18}NO_9P$)

Orange powder, mp 124–126°C; yield 0.71 g (89%); IR (KBr): $\bar{\nu} = 1725$, 1610 cm^{-1}; ^{1}H NMR: $\delta = 2.85$ (d, $^3J_{HP} = 11.0$ Hz, O*Me*), 3.67 (d, $^3J_{HP} = 11.0$ Hz, O*Me*), 3.71 (s, O*Me*), 3.82 (s, O*Me*), 4.30 (dd, $^3J_{HH} = 13.0$, $^2J_{HP} = 21.4$ Hz, CH), 5.69 (dd, $^3J_{HH} = 13.0$, $^3J_{HP} = 9.9$ Hz, CH), 7.21 (t, $^3J_{HH} = 7.2$ Hz, 2CH), 7.51 (t, $^3J_{HH} = 7.5$ Hz, CH), 7.54 (d, $^3J_{HH} = 7.5$ Hz, CH) ppm; ^{13}C NMR: $\delta = 44.5$ (d, $^1J_{CP} = 131.9$ Hz, CH), 51.9 (d, $^2J_{CP} = 7.0$ Hz, CH), 52.7 (d, $^2J_{PC} = 7$ Hz, O*Me*), 53.9 (d, $^2J_{PC} = 7$ Hz, O*Me*), 53.1 (O*Me*), 53.3 (O*Me*), 111.3 (2CH), 117.9 (C), 124.0 (CH), 125.2 (C), 137.9 (CH), 160.9 (C=O), 166.5 (d, $^2J_{CP} = 13.2$ Hz, C=O), 170.5 (d, $^3J_{CP} = 4.0$ Hz, C=O), 181.7 (C=O) ppm; ^{31}P NMR: $\delta = 11.65$ ppm; EI-MS: *m*/*z* (%) = 399 (M^+, 15), 368 (62), 290 (100), 253 (38), 146 (88), 109 (86), 31 (56).

Dimethyl 2-(diethoxyphosphoryl)-3-(2,3-dioxo-2,3-dihydro-1H-indol-1-yl)succinate (**4b**, $C_{18}H_{22}NO_9P$)

Yellow powder, mp 127–129°C; yield 0.80 g (94%); IR (KBr): $\bar{\nu} = 1730$, 1602 cm^{-1}; ^{1}H NMR: $\delta = 1.13$ (t, $^3J_{HH} = 7.1$ Hz, *Me*), 1.15 (t, $^3J_{HH} = 7.1$ Hz, *Me*), 3.71 (s, O*Me*), 3.85 (s, O*Me*), 3.93 (m, OCH_2), 3.97 (m, OCH_2), 4.28 (dd, $^3J_{HH} = 12.2$, $^2J_{HP} = 21.6$ Hz, CH), 5.74 (dd, $^3J_{HH} = 12.2$, $^3J_{HP} = 9.6$ Hz, CH), 7.06 (d, $^3J_{HH} = 7.5$ Hz, CH), 7.14 (t, $^3J_{HH} = 7.5$ Hz, CH), 7.60 (t, $^3J_{HH} = 7.6$ Hz, CH), 7.64 (d, $^3J_{HH} = 7.5$ Hz, CH) ppm; ^{13}C NMR: $\delta = 15.9$ (d, $^3J_{CP} = 6.1$ Hz, *Me*), 16.1 (d, $^3J_{CP} = 6.0$ Hz, *Me*), 43.8 (d, $^1J_{CP} = 130.9$ Hz, CH), 52.7 (d, $^2J_{CP} = 7.0$ Hz, CH), 53.6 (O*Me*), 53.1 (O*Me*), 63.5 (d, $^2J_{CP} = 7.1$ Hz, OCH_2), 63.6 (d, $^2J_{CP} = 7.1$ Hz, OCH_2), 110.9 (2CH), 118.1 (C), 123.9 (CH), 125.5 (C), 138.2 (CH), 160.1 (C=O), 167.5 (d, $^2J_{CP} = 14.2$ Hz, C=O), 170.1 (d, $^3J_{CP} = 10.1$ Hz, C=O),

181.9 (C=O) ppm; ^{31}P NMR: $\delta = 17.28$ ppm; EI-MS: m/z (%) = 427 (M^+, 5), 395 (52), 340 (100).

Dimethyl 2-(2,3-dioxo-2,3-dihydro-1H-indol-1-yl)-3-(diphenoxyphosphoryl)succinate (**4c**, $C_{26}H_{22}NO_9P$)
Yellow crystals, mp 132–134°C; yield 0.96 g (92%); IR (KBr): $\bar{\nu} = 1729$, 1603 cm^{-1}; ^{1}H NMR: $\delta = 3.72$ (s, O*Me*), 3.85 (s, O*Me*), 4.62 (dd, $^3J_{HH} = 12.0$, $^2J_{HP} = 21.1$ Hz, CH), 5.51 (dd, $^3J_{HH} = 12.1$, $^3J_{HP} = 9.2$ Hz, CH), 6.87 (d, $^3J_{HH} = 7.6$ Hz, 2CH), 6.93 (d, $^3J_{HH} = 7.9$ Hz, 2CH), 7.11 (m, 6CH), 7.21 (d, $^3J_{HH} = 7.8$ Hz, CH), 7.52 (t, $^3J_{HH} = 7.8$ Hz, 2CH), 7.54 (d, $^3J_{HH} = 6.9$ Hz, CH) ppm; ^{13}C NMR: $\delta = 44.9$ (d, $^1J_{CP} = 133.1$ Hz, CH), 52.7 (d $^2J_{CP} = 7.2$ Hz, CH), 53.4 (O*Me*), 53.7 (O*Me*), 111.1 (2CH), 118.2 (C), 120.0 (d, $^3J_{CP} = 4.7$ Hz, $2CH_{ortho}$), 120.1 (d, $^3J_{CP} = 4.7$ Hz, $2CH_{ortho}$), 125.5 (C), 124.1 (CH), 125.6 (CH_{para}), 125.7 (CH_{para}), 138.4 (CH), 129.8 (m, $4CH_{meta}$), 149.7 (m, $2C_{ipso}$), 166.6 (d, $^2J_{CP} = 21.0$ Hz, C=O), 158.8 (C=O), 167.7 (d, $^3J_{CP} = 4.7$ Hz, C=O), 181.3 (C=O) ppm; ^{31}P NMR: $\delta = 10.20$ ppm; EI-MS: m/z (%) = 523 (M^+, 5), 430 (54), 376 (54), 285 (100), 147 (92), 92 (56), 77 (92).

Di(tert-butyl) 2-(dimethoxyphosphoryl)-3-(2,3-dioxo-2,3-dihydro-1H-indol-1-yl)succinate (**4d**, $C_{22}H_{30}NO_9P$)
Orange powder, mp 158–160°C; yield 0.86 g (89%); IR (KBr): $\bar{\nu} = 1724$, 1608 cm^{-1}; ^{1}H NMR: $\delta = 1.20$ (*s*, C*Me*$_3$), 1.35 (*s*, C*Me*$_3$), 2.86 (d, $^3J_{HP} = 10.9$ Hz, O*Me*), 3.66 (d, $^3J_{HP} = 10.5$ Hz, O*Me*), 4.35 (dd, $^3J_{HH} = 12.2$, $^2J_{HP} = 21.3$ Hz, CH), 5.67 (dd, $^3J_{HH} = 12.1$, $^3J_{HP} = 9.7$ Hz, CH), 7.11 (d, $^3J_{HH} = 7.3$ Hz, CH), 7.20 (t, $^3J_{HH} = 7.3$ Hz, CH), 7.58 (t, $^3J_{HH} = 7.4$ Hz, CH), 7.54 (d, $^3J_{HH} = 7.4$ Hz, CH) ppm; ^{13}C NMR: $\delta = 27.6$ (C*Me*$_3$), 27.4 (C*Me*$_3$), 44.6 (d, $^1J_{CP} = 132.8$ Hz, CH), 52.1 (d, $^2J_{CP} = 7.1$ Hz, CH), 52.4 (d, $^2J_{PC} = 7.0$ Hz, O*Me*), 53.9 (d, $^2J_{PC} = 7.1$ Hz, O*Me*), 84.7 (*CMe*$_3$), 85.2 (*CMe*$_3$), 111.3 (2CH), 116.9 (C), 123.6 (CH), 124.9 (C), 136.8 (CH), 162.1 (C=O), 167.3 (d, $^2J_{CP} = 21.3$ Hz, C=O), 171.2 (d, $^3J_{CP} = 9.1$ Hz, C=O), 182.3 (C=O) ppm; ^{31}P NMR: $\delta =$ 11.67 ppm; EI-MS: m/z (%) = 483 (M^+, 5), 452 (52), 374 (100), 337 (38), 146 (88), 109 (82), 73 (62), 57 (56), 31 (54).

Di(tert-butyl) 2-(diethoxyphosphoryl)-3-(2,3-dioxo-2,3-dihydro-1H-indol-1-yl)succinate (**4e**, $C_{24}H_{34}NO_9P$)
Yellow crystals, mp 136–138°C; yield 0.91 g (88%); IR (KBr): $\bar{\nu} = 1735$, 1615 cm^{-1}; ^{1}H NMR: $\delta = 1.18$ (t, $^3J_{HH} = 6.7$ Hz, *Me*), 1.21 (t, $^3J_{HH} = 6.7$ Hz, *Me*), 1.25 (s, C*Me*$_3$), 1.30 (s, C*Me*$_3$), 3.92 (m, OCH_2), 4.01 (m, OCH_2), 4.25 (dd, $^3J_{HH} = 12.0$, $^2J_{HP} = 21.0$ Hz, CH), 5.56 (dd, $^3J_{HH} = 11.9$, $^3J_{HP} = 9.5$ Hz, CH), 6.93 (d, $^3J_{HH} = 7.9$ Hz, CH), 7.11 (t, $^3J_{HH} = 7.5$ Hz, CH), 7.55 (t, $^3J_{HH} = 7.7$ Hz, CH), 7.59 (d, $^3J_{HH} = 7.4$ Hz, CH) ppm; ^{13}C NMR: $\delta = 16.2$ (d, $^3J_{CP} = 6.2$ Hz, *Me*), 16.6 (d, $^3J_{CP} = 6.0$ Hz, *Me*), 27.2 (C*Me*$_3$), 27.9 (C*Me*$_3$), 46.2 (d, $^1J_{CP} = 130.8$ Hz, CH), 52.5 (d, $^2J_{CP} = 6.9$ Hz, CH), 63.9 (d, $^2J_{PC} = 7.3$ Hz, O*Me*), 64.4 (d, $^2J_{PC} = 7.2$ Hz, O*Me*), 83.2 (*CMe*$_3$), 84.3 (*CMe*$_3$), 110.5 (2CH), 117.9 (C), 123.8 (CH), 124.5 (C), 138.3 (CH), 157.9 (C=O), 166.9 (d, $^2J_{CP} = 21.1$ Hz, C=O), 169.0 (d, $^3J_{CP} = 14.0$ Hz, C=O), 182.1 (C=O) ppm; ^{31}P NMR: $\delta = 19.04$ ppm; EI-MS: m/z (%) = 511 (M^+, 10), 418 (52), 365 (92), 278 (68), 233 (76), 146 (88), 93 (100), 73 (62), 57 (56).

Di(tert-butyl) 2-(2,3-dioxo-2,3-dihydro-1H-indol-1-yl)-3-(diphenoxyphosphoryl)succinate (**4f**, $C_{32}H_{34}NO_9P$)
Yellow crystals, mp 140–143°C; yield 1.08 g (89%); IR (KBr): $\bar{\nu} = 1726$, 1604 cm^{-1}; ^{1}H NMR: $\delta = 1.21$ (s, C*Me*$_3$), 1.38 (s, C*Me*$_3$), 4.22 (dd, $^3J_{HH} = 11.9$, $^2J_{HP} = 20.9$ Hz, CH), 5.54 (dd, $^3J_{HH} = 12.4$, $^3J_{HP} = 8.9$ Hz, CH), 6.81 (d, $^3J_{HH} = 7.5$ Hz, 2CH), 7.15 (d, $^3J_{HH} = 7.8$ Hz, 2CH), 7.18 (m, 6CH), 7.30 (d, $^3J_{HH} = 7.6$ Hz, CH), 7.61 (t, $^3J_{HH} = 7.7$ Hz, 2CH), 7.63 (d, $^3J_{HH} = 6.7$ Hz, CH) ppm; ^{13}C NMR: $\delta = 27.5$ (C*Me*$_3$), 27.7 (C*Me*$_3$), 46.7 (d, $^1J_{CP} = 132.7$ Hz, CH), 54.2 (d, $^2J_{CP} = 6.9$ Hz, CH), 83.7 (*CMe*$_3$), 84.3 (*CMe*$_3$), 112.5 (2CH), 117.9 (C), 120.5 (d, $^3J_{CP} = 4.6$ Hz, $2CH_{ortho}$), 120.7 (d, $^3J_{CP} = 4.7$ Hz, $2CH_{ortho}$), 124.2 (C), 123.6 (CH), 125.5 (CH_{para}), 125.6 (CH_{para}), 129.8 (m, $4CH_{meta}$), 138.6 (CH), 150.2 (m, $2C_{ipso}$), 157.8 (C=O), 164.1 (d, $^2J_{CP} = 12.2$ Hz, C=O), 165.5 (d, $^3J_{CP} = 9.8$ Hz, C=O), 181.8 (C=O) ppm; ^{31}P NMR: $\delta = 11.68$ ppm; EI-MS: m/z (%) = 607 (M^+, 4), 552 (26), 515(12), 402 (100), 374 (88), 233(16), 147 (76), 92 (58), 77 (38).

Dimethyl 2-(diphenoxyphosphoryl)-3-1H-pyrrol-1-yl)-succinate (**9**, $C_{22}H_{22}NO_7P$)
Pale yellow crystals, mp 98–100°C; yield 0.80 g (90%); IR (KBr): $\bar{\nu} = 1722$ cm^{-1}; ^{1}H NMR: $\delta = 3.72$ (s, O*Me*), 3.75 (s, O*Me*), 4.45 (dd, $^3J_{HH} = 12.4$, $^2J_{HP} = 22.1$ Hz, CH), 4.95 (dd, $^3J_{HH} = 12.4$, $^3J_{HP} = 10.4$ Hz, CH), 6.15 (d, $^3J_{HH} = 7.1$ Hz, 2CH), 7.13 (d, $^3J_{HH} = 7.9$ Hz, 2CH), 7.24 (m, 4CH), 7.35 (d, $^3J_{HH} = 7.8$ Hz, 2CH), 7.45 (t, $^3J_{HH} = 7.8$ Hz, 2CH), 7.52 (d, $^3J_{HH} = 6.9$ Hz, CH), 7.92 (d, $^3J_{HH} = 7.1$ Hz, CH) ppm; ^{13}C NMR: $\delta = 43.9$ (d, $^1J_{CP} = 130.5$ Hz, CH), 50.1 (d, $^2J_{CP} = 7.2$ Hz, CH), 51.3 (O*Me*), 52.4 (O*Me*), 116.3 (2CH), 119.2 (2CH), 120.2 (d, $^3J_{CP} = 4.3$ Hz, $2CH_{ortho}$), 121.3 (d, $^3J_{CP} = 4.7$ Hz, $2CH_{ortho}$), 124.6 (CH_{para}), 125.1 (CH_{para}), 129.8 (m, $4CH_{meta}$), 150.2 (m, $2C_{ipso}$), 164.3 (d, $^2J_{CP} = 22.3$ Hz, C=O), 168.1 (d, $^3J_{CP} = 5.1$ Hz, C=O) ppm; ^{31}P NMR: $\delta = 10.56$ ppm; EI-MS: m/z (%) = 443 (M^+, 5), 377 (96), 350 (52), 233 (100), 210 (58), 93 (86), 66 (88).

Dimethyl 2-(diphenoxyphosphoryl)-3-1H-indol-1-yl)-succinate (**10**, $C_{26}H_{24}NO_7P$)
Pale yellow crystals, mp 110–112°C; yield 0.88 g (90%); IR (KBr): $\bar{\nu} = 1720$ cm^{-1}; ^{1}H NMR: $\delta = 3.64$ (s, O*Me*), 3.89 (s, O*Me*), 4.53 (dd, $^3J_{HH} = 12.2$, $^2J_{HP} = 21.9$ Hz, CH), 4.95 (dd, $^3J_{HH} = 12.2$, $^3J_{HP} = 10.1$ Hz, CH), 6.48 (d, $^3J_{HH} = 6.9$ Hz, CH), 7.03 (d, $^3J_{HH} = 7.4$ Hz, 2CH), 7.11 (d, $^3J_{HH} = 7.5$ Hz, 2CH), 7.23 (m, 6CH), 7.32 (d, $^3J_{HH} = 7.5$, Hz CH), 7.52 (t, $^3J_{HH} = 7.4$ Hz, 2CH), 7.54 (d, $^3J_{HH} = 7.1$ Hz, CH), 7.62 (d, $^3J_{HH} = 6.9$ Hz, CH) ppm; ^{13}C NMR: $\delta = 35.6$ (d, $^1J_{CP} = 139.2$ Hz, CH), 44.9 (d, $^2J_{CP} = 6.8$ Hz, CH), 51.6 (O*Me*), 52.4 (O*Me*), 102.5 (CH), 113.4 (CH), 118.2 (CH), 119.3 (d, $^3J_{CP} = 5.2$ Hz, $2CH_{ortho}$), 119.5 (d, $^3J_{CP} = 4.7$ Hz, $2CH_{ortho}$), 120.5, 121.9, 123.5 (3CH), 125.6, 125.7 ($2CH_{para}$), 129.8 (m, $4CH_{meta}$), 133.5 (C), 139.4 (C), 149.7 (m, $2C_{ipso}$), 166.6 (d, $^2J_{CP} = 14.7$ Hz, C=O), 167.7 (d, $^3J_{CP} = 11.0$ Hz, C=O) ppm; ^{31}P NMR: $\delta = 11.65$ ppm; EI-MS: m/z (%) = 493 (M^+, 10), 462 (72), 377 (54), 233 (98), 116 (78), 93 (100), 31 (96).

Dimethyl 2-(1,3-dioxo-1,3-dihydro-2H-isoindol-2-yl)-3-(diphenoxyphosphoryl)succinate (**11**, $C_{26}H_{22}NO_9P$)
Pale yellow crystals, mp 130–132°C; yield 0.98 g (94%); IR (KBr): $\bar{\nu} = 1727$, 1715 cm^{-1}; ^1H NMR: $\delta = 3.65$ (s, O*Me*), 3.85 (s, O*Me*), 4.62 (dd, $^3J_{HH} = 12.6$, $^2J_{HP} = 20.9$ Hz, CH), 5.94 (dd, $^3J_{HH} = 12.6$, $^3J_{HP} = 9.8$ Hz, CH), 7.36 (d, $^3J_{HH} = 7.6$ Hz, 2CH), 7.41 (d, $^3J_{HH} = 7.9$ Hz, 2CH), 7.56 (m, 6CH); 7.68 (d, $^3J_{HH} = 7.8$ Hz, CH), 7.82 (t, $^3J_{HH} = 7.8$ Hz, 2CH), 7.94 (d, $^3J_{HH} = 6.9$ Hz, CH) ppm; ^{13}C NMR: $\delta = 39.1$ (d, $^1J_{CP} = 136.5$ Hz, CH), 49.8 (d, $^2J_{CP} = 7.6$ Hz, CH), 51.7 (O*Me*), 52.6 (O*Me*), 119.8 (d, $^3J_{CP} = 6.7$ Hz, 4 CH_{ortho}), 123.5 (2CH), 124.6 ($2CH_{para}$), 128.8 (m, $4CH_{meta}$), 134.1 (2CH), 138.7 (2C), 151.3 (m, $2C_{ipso}$), 165.6 (d, $^2J_{CP} = 22.5$ Hz, C=O), 167.9 (d, $^3J_{CP} = 14.5$ Hz, C=O), 170.5 (2C=O) ppm; ^{31}P NMR: $\delta = 11.52$ ppm; EI-MS: m/z (%) = 523 (M^+, 5), 492 (66), 377 (64), 233 (82), 146 (78), 93 (100), 31 (96).

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