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ORIGINAL PAPER

# Novel synthesis of stable 1,5-diionic organophosphorus compounds from the reaction between triphenylphosphine and acetylenedicarboxylic acid in the presence of N–H heterocyclic compounds

**Morteza Ziyaadini · Malek Taher Maghsoodlou · Nourallah Hazeri · Sayyed Mostafa Habibi-Khorassani**

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**Abstract** The addition of triphenylphosphine to acetylenedicarboxylic acid in the presence of N–H heterocyclic compounds such as carbazole, 2-indolinone, 2-benzoxazolinone, 2-mercaptobenzoxazole, 2,4-thiazolidinedione, benzimidazole, isatin, or saccharin leads to stable 1,5-diionic organophosphorus compounds in excellent yields at ambient temperature.

**Keywords** Acetylenedicarboxylic acid · Triphenylphosphine · 1,5-Diionic organophosphorus compounds · N–H heterocyclic compounds · Decarboxylation

## Introduction

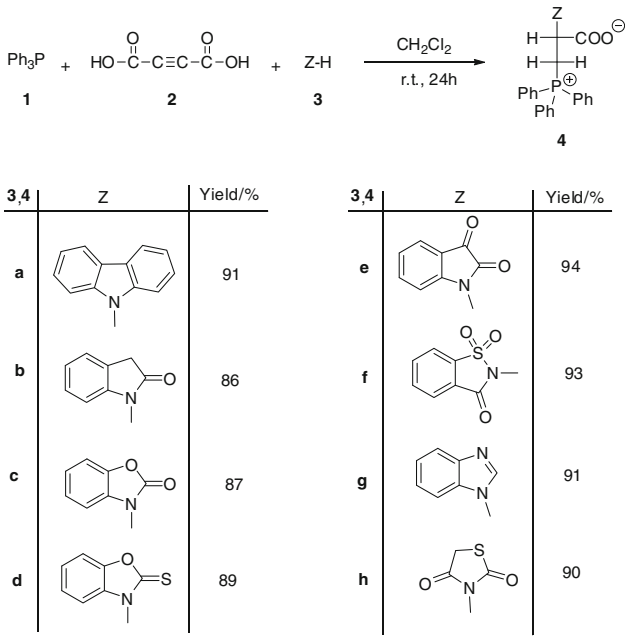
Trivalent phosphorus compounds are known to be nucleophiles, whereas they behave as electron donors toward good electron acceptors either in the ground or excited state [1, 2]. In recent years there has been increasing interest in the synthesis of organophosphorus compounds, i.e., those bearing a carbon atom bound directly to a phosphorus atom [3–15]. This interest has resulted from the recognition of the value of such compounds in a variety of biological, industrial, and chemical synthetic uses [3–5]. A number of reactions have been observed which involve 1,4-diionic phosphorus compounds as elusive transient species [8–10]. In all of these reactions in which this diionic system is postulated, the betaine cannot be isolated but appears to occur as an intermediate on the pathway to an observed product. Researchers have recently described the synthesis of stable diionic compounds from the reaction between triphenylphosphine and electron-deficient acetylenic esters in the presence of CH acids [11–14]. In continuation of our investigation of the reaction between trivalent phosphorus nucleophiles and acetylenic esters in the presence of organic compounds containing NH, OH, or SH groups [16–28], we wish to report a novel and facile one-pot, three-component reaction between triphenylphosphine (**1**), acetylenedicarboxylic acid (ADA, **2**), and N–H heterocyclic compounds **3a**–**3h** for the preparation of the crystalline and stable 1,5-diionic organophosphorus compounds **4a**–**4h** in excellent yields at ambient temperature (Scheme 1).

## Results and discussion

The decarboxylation of carboxylic acids under the influence of the electron-withdrawing triphenylphosphonium group at the α-position has been recently described [29, 30]. In the present three-component reaction, 1,5-diionic organophosphorus compounds are generated via decarboxylation of an intermediate ylide as a key step in the reaction. On the basis of the well-established chemistry of trivalent phosphorus nucleophiles, it is reasonable to assume that compounds **4a**–**4h** result from the initial addition of triphenylphosphine to acetylenedicarboxylic acid and subsequent protonation of the 1:1 adduct **5** by proton source **3** to form **6**. Then the positively charged ion **6** is attacked by the anion of the NH acid to generate phosphorus ylide **7**. Ylide **7** is converted to compound **8**

M. T. Maghsoodlou (✉) · N. Hazeri · S. M. Habibi-Khorassani
Department of Chemistry, Faculty of Science,
University of Sistan and Baluchestan,
P.O. Box 98135-674, Zahedan, Iran
e-mail: mt_maghsoodlou@yahoo.com

M. Ziyaadini
Faculty of Marine Science, Chabahar Maritime University,
Chabahar, Iran




**Scheme 1**

and subsequently to intermediate **9** in a process of H-transfer and elimination of $CO_2$. Ylide **9** can be converted to desired product **4** by another H-transfer (see speculative proposed mechanism in Scheme 2). No product other than **4** could be detected by NMR spectroscopy. The structures of compounds **4a**–**4h** were deduced from the elemental analyses, IR, $^1$H, $^{13}$C, $^{31}$P NMR, and mass spectra. A plausible interpretation of the $^1$H spectral data for compound **4a** is that the signal at $\delta = 5.22$ ppm corresponds to the methine proton with $J_{HP}$ coupling constant 13.1 Hz, whereas signals at 4.76 and 3.80 ppm correspond to the diastereotopic protons of the methylene group, with $J_{HP}$ coupling constants 13.1 and 10.3 Hz, respectively. Vicinal coupling constants are equal to 3.0 and 10.4 Hz, respectively, and the geminal coupling constant −16.1 Hz.

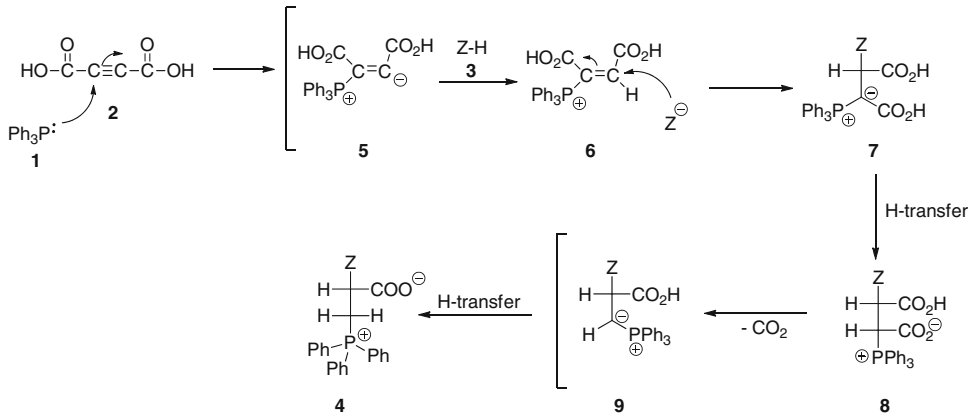
The $^1$H NMR spectra of compound **4a** exhibited two signals for the diastereotopic protons of the methylene group at $\delta = 3.80$ ppm with $^2J_{HH} = 16.1$ Hz, $^2J_{HP} = 10.3$ Hz, $^3J_{HH} = 10.4$ Hz, and $\delta = 4.76$ ppm with $^2J_{HH} = 16.1$ Hz, $^2J_{HP} = 13.1$ Hz, $^3J_{HH} = 3.0$ Hz as two sets of treble doublets. The signal at $\delta = 5.22$ ppm corresponds to the methine proton which appears as treble doublets with $^3J_{HP} = 13.1$ Hz, $^3J_{HH} = 10.4$ Hz, and $^3J_{HP} = 10.3$ Hz (Fig. 1). The $^1$H-decoupled $^{13}$C NMR spectrum of **4a** showed two characteristic doublets at $\delta = 24.42$ ($^1J_{CP} = 53.8$ Hz) and 51.67 ppm ($^2J_{CP} = 5.6$ Hz) for $CH_2$–P (methylene group) and CH–CP (methine group) moieties, respectively. The $^{13}$C NMR spectra are in agreement with the structure of **4a**. Partial assignments of these resonances are given in the "Experimental" section. Furthermore, DEPT spectra of **4a** indicated two chemical shifts for the methylene carbon and methine carbon at $\delta =$ 24.4 and 51.6 ppm, respectively. The $^{31}$P NMR spectra of compound **4a** displayed a signal at $\delta = 20.44$ ppm which is consistent with the presence of a $(Ph)_3P^+$–C grouping [31, 32]. The mass spectrum of **4a** displayed the molecular ion peak at $m/z = 499$.

In summary, the present synthesis of 1,5-diionic organophosphorus compounds offers significant advantages for the synthesis of betaines which are a new category of organophosphorous compounds: not only is the reaction performed under neutral conditions, but also substances can be mixed without any activation or modification.

## Experimental

Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a JASCO FT-IR spectrometer, respectively. $^1$H and $^{13}$C NMR spectra were recorded on Bruker 500 or 400 MHz instruments using $CDCl_3$ as a solvent and TMS as internal standard (500.1 or 400.2 MHz and 125.8 or 100.6 MHz, respectively). Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. In addition, the mass spectra were recorded on a Shimadzu GCMS-QP5050A

**Scheme 2**



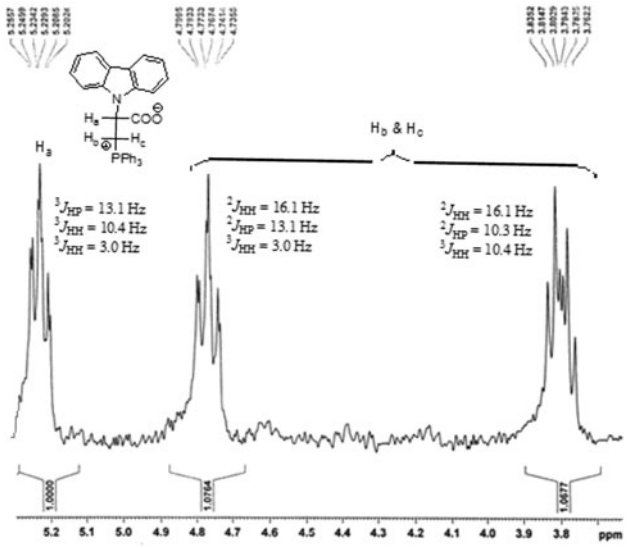



**Fig. 1** Part of the $^{1}$H NMR spectrum of compound **4a**

mass spectrometer operating at an ionization potential of 70 eV. Triphenylphosphine, acetylenedicarboxylic acid, and N–H acid derivatives were purchased from Fuka, Merck, and Acros and used without further purification.

*2-(9H-Carbazol-9-yl)-3-(triphenylphosphonio)propanoate* (**4a**, $C_{33}H_{26}NO_2P$)

To a magnetically stirred solution of triphenylphosphine (1 mmol) and NH acid **3a** (1 mmol) in 10 $cm^3$ $CH_2Cl_2$ was added dropwise a mixture of acetylenedicarboxylic acid (1 mmol) in 1 $cm^3$ $Et_2O$ over 10 min at ambient temperature. After 24 h stirring at ambient temperature the solvent was removed and the residue was crystallized from diethyl ether to yield **4a** as a light yellow powder. Yield 91%; m.p.: 84–87 °C; $^{1}$H NMR ($CDCl_3$, 500.1 MHz): $\delta = 3.80$ (1H, ddd, $^{2}J_{HH} = 16.1$ Hz, $^{3}J_{HH} = 10.4$ Hz, $^{2}J_{HP} = 10.3$ Hz, $CH_2$–P), 4.76 (1H, ddd, $^{2}J_{HH} = 16.1$ Hz, $^{2}J_{HP} = 13.1$ Hz, $^{3}J_{HH} = 3.0$ Hz, $CH_2$–P), 5.22 (1H, ddd, $^{3}J_{HP} = 13.1$ Hz, $^{3}J_{HH} = 10.4$ Hz, $^{3}J_{HH} = 3.0$ Hz, CH–CP), 7.00–7.84 (23H, m, $CH_{arom}$) ppm; $^{13}$C NMR ($CDCl_3$, 125.7 MHz): $\delta = 24.42$ (d, $^{1}J_{CP} = 53.8$ Hz, $CH_2$–P), 51.67 (d, $^{2}J_{CP} = 5.6$ Hz, CH–CP), 109.65, 110.54, 122.45, 124.05, 142.32, 154.04 (s, 12C of $C_{12}H_8N$), 118.11 (d, $^{1}J_{CP} = 86.9$ Hz, $C_{ipso}$), 130.26 (d, $^{3}J_{CP} = 12.7$ Hz, $C_{meta}$), 133.32 (d, $^{2}J_{CP} = 10.0$ Hz, $C_{ortho}$), 134.97 (d, $^{4}J_{CP} = 2.8$ Hz, $C_{para}$), 166.94 (d, $^{3}J_{CP} = 11.6$ Hz, C=O acid) ppm; $^{31}$P NMR ($CDCl_3$, 202.4 MHz): $\delta = 20.44$ ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu} = 1{,}639$ and 1,438 (COO asym and sym) $cm^{-1}$; MS: *m/z* (%) = 499 ($M^+$, 1), 277 ($Ph_3PCH_3$, 15), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 69), 108 (PPh, 25), 77 (Ph, 8), 44 ($CO_2$, 5).

*2-(2,3-Dihydro-2-oxo-1H-indol-1-yl)-3-(triphenylphosphonio)-propanoate* (**4b**, $C_{29}H_{22}NO_3P$)

Light brown powder; yield 86%; m.p.: 123–125 °C; $^{1}$H NMR ($CDCl_3$, 400.2 MHz): $\delta = 2.56$, 3.16 (2H, d, $^{2}J_{HH} = 22.1$ Hz, $CH_2$ of $C_8H_7NO$), 3.66 (1H, ddd, $^{2}J_{HH} = 16.5$ Hz, $^{2}J_{HP} = 10.0$ Hz, $^{3}J_{HH} = 9.7$ Hz, $CH_2$–P), 4.70 (1H, ddd, $^{2}J_{HH} = 16.2$ Hz, $^{2}J_{HP} = 13.7$ Hz, $^{3}J_{HH} = 3.3$ Hz, $CH_2$–P), 5.36 (1H, ddd, $^{3}J_{HP} = 13.6$ Hz, $^{3}J_{HH} = 9.2$ Hz, $^{3}J_{HH} = 3.3$ Hz, CH–CP), 6.89–7.81 (19H, m, $CH_{arom}$) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta = 23.22$ (d, $^{1}J_{CP} = 52.3$ Hz, CH–P), 35.16 (s, $CH_2$ of $C_8H_7NO$), 49.03 (d, $^{2}J_{CP} = 5.0$ Hz, $CH_2$–CP), 110.08, 117.46, 118.36, 122.43, 142.35, 175.05 (s, 7C of $C_8H_7NO$), 118.38 (d, $^{1}J_{CP} = 86.5$ Hz, $C_{ipso}$), 130.21 (d, $^{3}J_{CP} = 13.1$ Hz, $C_{meta}$), 133.51 (d, $^{2}J_{CP} = 10.5$ Hz, $C_{ortho}$), 134.87 (d, $^{4}J_{CP} = 3.0$ Hz, $C_{para}$), 168.09 (d, $^{3}J_{CP} = 12.0$ Hz, C=O acid) ppm; $^{31}$P NMR ($CDCl_3$, 162.0 MHz): $\delta = 21.01$ ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu} = 1{,}700$ (C=O heterocyclic), 1,614 and 1,438 (COO asym and sym) $cm^{-1}$; MS: *m/z* (%) = 463 ($M^+$, 1), 277 ($Ph_3PCH_3$, 2), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 41), 133 ($C_8H_7NO$, 2), 108 (PPh, 17), 77 (Ph, 4), 44 ($CO_2$, 8).

*2-(2-Oxobenzo[d]oxazol-3(2H)-yl)-3-(triphenylphospho-nio)propanoate* (**4c**, $C_{28}H_{22}NO_4P$)

Light yellow powder; yield 87%; m.p.: 103–106 °C; $^{1}$H NMR ($CDCl_3$, 400.2 MHz): $\delta = 3.87$ (1H, ddd, $^{3}J_{HH} = 15.9$ Hz, $^{2}J_{HP} = 10.3$ Hz, $^{3}J_{HH} = 10.4$ Hz, $CH_2$–P), 4.81 (1H, ddd, $^{2}J_{HH} = 16.1$ Hz, $^{2}J_{HP} = 13.1$ Hz, $^{3}J_{HH} = 3.0$ Hz, $CH_2$–P), 5.18 (1H, ddd, $^{3}J_{HP} = 13.3$ Hz, $^{3}J_{HH} = 10.3$ Hz, $^{3}J_{HH} = 3.0$ Hz, CH–CP), 7.45–7.80 (19H, m, $CH_{arom}$) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta = 24.04$ (d, $^{1}J_{CP} = 53.3$ Hz, CH–P), 51.78 (d, $^{2}J_{CP} = 6.0$ Hz, $CH_2$–CP), 109.54, 110.71, 122.34, 124.05, 142.25, 154.09, 159.95 (s, 7C of $C_7H_4NO_2$), 118.12 (d, $^{1}J_{CP} = 86.6$ Hz, $C_{ipso}$), 130.23 (d, $^{3}J_{CP} = 13.1$ Hz, $C_{meta}$), 133.36 (d, $^{2}J_{CP} = 10.1$ Hz, $C_{ortho}$), 134.91 (d, $^{4}J_{CP} = 3.0$ Hz, $C_{para}$), 167.25 (d, $^{3}J_{CP} = 12.1$ Hz, C=O acid) ppm; $^{31}$P NMR ($CDCl_3$, 162.0 MHz): $\delta = 20.30$ ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu} = 1{,}761$ (C=O heterocyclic), 1,638 and 1,438 (COO asym and sym) $cm^{-1}$; MS: *m/z* (%) = 467 ($M^+$, 1), 277 ($Ph_3PCH_3$, 8), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 68), 135 ($C_7H_5NO_2$, 3), 108 (PPh, 13), 77 (Ph, 3), 44 ($CO_2$, 6).

*2-(2-Thioxobenzo[d]oxazol-3(2H)-yl)-3-(triphenylphos-phonio)propanoate* (**4d**, $C_{28}H_{22}NO_3PS$)

Cream powder; yield 89%; m.p.: 97–100 °C; $^{1}$H NMR ($CDCl_3$, 500.1 MHz): $\delta = 3.67$ (1H, ddd, $^{2}J_{HH} = 15.7$ Hz, $^{2}J_{HP} = 8.5$ Hz, $^{3}J_{HH} = 8.4$ Hz, $CH_2$–P), 4.65 (1H, ddd, $^{2}J_{HH} = 15.1$ Hz, $^{2}J_{HP} = 13.8$ Hz, $^{3}J_{HH} = 6.0$ Hz, $CH_2$–P), 5.87 (1H, ddd, $^{3}J_{HP} = 13.5$ Hz, $^{3}J_{HH} = 8.2$ Hz, $^{3}J_{HH} = 6.0$ Hz, CH–CP), 7.47–7.81 (19H, m, $CH_{arom}$) ppm; $^{13}$C NMR ($CDCl_3$, 125.7 MHz): $\delta = 25.43$ (d, $^{1}J_{CP} = 56.5$ Hz, CH–P), 56.06 (d, $^{2}J_{CP} = 3.1$ Hz, $CH_2$–CP), 109.98, 122.34, 124.05, 134.57, 146.75, 156.14 (s, 6C of $C_7H_4NOS$), 118.54 (d, $^{1}J_{CP} = 88.0$ Hz, $C_{ipso}$), 130.02 (d, $^{3}J_{CP} = 12.7$ Hz, $C_{meta}$), 133.43 (d, $^{2}J_{CP} = 9.9$ Hz, $C_{ortho}$), 135.52 (d, $^{4}J_{CP} = 2.6$ Hz, $C_{para}$), 166.58 (d,

$^3J_{CP}$ = 8.2 Hz, C=O acid), 180.26 (C=S) ppm; $^{31}$P NMR ($CDCl_3$, 202.4 MHz): $\delta$ = 20.20 ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu}$ = 1,647 and 1,400 (COO asym and sym), 1,470 (C=S) $cm^{-1}$; MS: *m/z* (%) = 483 ($M^+$, 1), 277 ($Ph_3PCH_3$, 9), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 80), 151 ($C_7H_5NOS$, 17), 108 (PPh, 26), 77 (Ph, 10), 44 ($CO_2$, 5).

*2-(2,3-Dihydro-2,3-dioxo-1H-indol-1-yl)-3-(triphenylphosphonio)propanoate* (**4e**, $C_{29}H_{22}NO_4P$)
Orange powder; yield 94%; m.p.: 148–151 °C; $^1$H NMR ($CDCl_3$, 400.2 MHz): $\delta$ = 3.69 (1H, ddd, $^2J_{HH}$ = 16.1 Hz, $^2J_{HP}$ = 10.3 Hz, $^3J_{HH}$ = 10.4 Hz, $CH_2$–P), 4.82 (1H, ddd, $^2J_{HH}$ = 16.7 Hz, $^2J_{HP}$ = 12.9 Hz, $^3J_{HH}$ = 2.4 Hz, $CH_2$–P), 5.32 (1H, ddd, $^3J_{HP}$ = 13.4 Hz, $^3J_{HH}$ = 11.0 Hz, $^3J_{HH}$ = 2.4 Hz, CH–CP), 7.05–7.84 (19H, m, $CH_{arom}$) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ = 23.90 (d, $^1J_{CP}$ = 53.3 Hz, CH–P), 49.52 (d, $^2J_{CP}$ = 6.0 Hz, $CH_2$–CP), 112.67, 117.87, 123.77, 124.95, 138.54, 149.23, 157.96, 182.33 (s, 8C of $C_8H_4NO_2$), 118.11 (d, $^1J_{CP}$ = 86.5 Hz, $C_{ipso}$), 130.40 (d, $^3J_{CP}$ = 13.1 Hz, $C_{meta}$), 133.55 (d, $^2J_{CP}$ = 10.0 Hz, $C_{ortho}$), 135.21 (d, $^4J_{CP}$ = 2.0 Hz, $C_{para}$), 166.67 (d, $^3J_{CP}$ = 11.1 Hz, C=O acid) ppm; $^{31}$P NMR ($CDCl_3$, 162.0 MHz): $\delta$ = 20.82 ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu}$ = 1,735 and 1,650 (C=O heterocyclic), 1,610 and 1,438 (COO asym and sym) $cm^{-1}$; MS: *m/z* (%) = 479 ($M^+$, 1), 333 (M − $C_8H_4NO_2$, 1), 277 ($Ph_3PCH_3$, 10), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 64), 108 (PPh, 21), 77 (Ph, 17), 44 ($CO_2$, 13).

*2-(1,1-Dioxido-3-oxobenzo[d]isothiazol-2(3H)-yl)-3-(triphenylphosphonio)propanoate* (**4f**, $C_{28}H_{22}NO_5PS$)
White powder; yield 93%; m.p.: 94–98 °C; $^1$H NMR ($CDCl_3$, 500.1 MHz): $\delta$ = 3.88 (1H, ddd, $^2J_{HH}$ = 17.3 Hz, $^3J_{HH}$ = 9.5 Hz, $^2J_{HP}$ = 9.4 Hz, $CH_2$–P), 4.69 (1H, ddd, $^2J_{HH}$ = 16.8 Hz, $^2J_{HP}$ = 13.2 Hz, $^3J_{HH}$ = 6.9 Hz, $CH_2$–P), 4.85 (1H, ddd, $^3J_{HP}$ = 13.5 Hz, $^3J_{HH}$ = 8.9 Hz, $^3J_{HH}$ = 6.7 Hz, CH–CP), 7.53–7.95 (19H, m, $CH_{arom}$) ppm; $^{13}$C NMR ($CDCl_3$, 125.7 MHz): $\delta$ = 24.79 (d, $^1J_{CP}$ = 49.1 Hz, CH–P), 49.99 (d, $^2J_{CP}$ = 4.1 Hz, $CH_2$–CP), 116.33, 117.06, 119.73, 123.42, 131.52, 131.82, 168.85 (s, 7C of $C_7H_4NO_3S$), 118.60 (d, $^1J_{CP}$ = 83.4 Hz, $C_{ipso}$), 130.65 (d, $^3J_{CP}$ = 13.2 Hz, $C_{meta}$), 133.85 (d, $^2J_{CP}$ = 10.6 Hz, $C_{ortho}$), 135.68 (d, $^4J_{CP}$ = 2.7 Hz, $C_{para}$), 166.71 (d, $^3J_{CP}$ = 10.1 Hz, C=O acid) ppm; $^{31}$P NMR ($CDCl_3$, 202.4 MHz): $\delta$ = 18.95 ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu}$ = 1,728 (C=O heterocyclic), 1,638 and 1,438 (COO asym and sym), 1,338 ($SO_2$) $cm^{-1}$; MS: *m/z* (%) = 515 ($M^+$, 1), 277 ($Ph_3PCH_3$, 7), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 74), 108 (PPh, 30), 77 (Ph, 6), 44 ($CO_2$, 18).

*2-(1H-Benzo[d]imidazol-1-yl)-3-(triphenylphosphonio)propanoate* (**4g**, $C_{28}H_{23}N_2O_2P$)
Cream powder; yield 91%; m.p.: 90–92 °C; $^1$H NMR ($CDCl_3$, 400.2 MHz): $\delta$ = 4.03 (1H, ddd, $^2J_{HH}$ = 15.8 Hz, $^3J_{HH}$ = 9.1 Hz, $^2J_{HP}$ = 9.2 Hz, $CH_2$–P), 4.87 (1H, ddd, $^2J_{HH}$ = 16.3 Hz, $^2J_{HP}$ = 13.1 Hz, $^3J_{HH}$ = 4.0 Hz, $CH_2$–P), 5.30 (1H, ddd, $^3J_{HP}$ = 13.4 Hz, $^3J_{HH}$ = 8.6 Hz, $^3J_{HH}$ = 4.1 Hz, CH–CP), 7.34–7.81 (19H, m, $CH_{arom}$), 7.95 (1H, s, CH=N) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ = 27.21 (d, $^1J_{CP}$ = 53.3 Hz, CH–P), 54.98 (d, $^2J_{CP}$ = 5.0 Hz, $CH_2$–CP), 110.46, 119.91, 122.08, 122.90, 128.44, 133.78, 143.21 (s, 7C of $C_7H_5N_2$), 118.50 (d, $^1J_{CP}$ = 87.6 Hz, $C_{ipso}$), 130.18 (d, $^3J_{CP}$ = 13.1 Hz, $C_{meta}$), 133.19 (d, $^2J_{CP}$ = 10.6 Hz, $C_{ortho}$), 134.70 (d, $^4J_{CP}$ = 3.1 Hz, $C_{para}$), 168.83 (d, $^3J_{CP}$ = 10.1 Hz, C=O acid) ppm; $^{31}$P NMR ($CDCl_3$, 162.0 MHz): $\delta$ = 20.87 ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu}$ = 1,639 and 1,400 (COO asym and sym) $cm^{-1}$; MS: *m/z* (%) = 450 ($M^+$, 1), 277 ($Ph_3PCH_3$, 6), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 80), 132 ($C_8H_9N_2$, 5), 108 (PPh, 26), 77 (Ph, 9), 44 ($CO_2$, 2).

*2-(2,4-Dioxothiazolidin-3-yl)-3-(triphenylphosphonio)-propanoate* (**4h**, $C_{24}H_{20}NO_4PS$)
Light yellow powder; yield 90%; m.p.: 94–97 °C; $^1$H NMR ($CDCl_3$, 500.1 MHz): $\delta$ = 3.58, 3.74 (2H, d, $^2J_{HH}$ = 17.1 Hz, $CH_2$ heterocyclic), 3.88 (1H, ddd, $^2J_{HH}$ = 16.0 Hz, $^3J_{HH}$ = 9.5 Hz, $^2J_{HP}$ = 9.4 Hz, $CH_2$–P), 4.76 (1H, ddd, $^2J_{HH}$ = 16.1 Hz, $^2J_{HP}$ = 14.1 Hz, $^3J_{HH}$ = 4.0 Hz, $CH_2$–P), 5.02 (1H, ddd, $^3J_{HP}$ = 14.0 Hz, $^3J_{HH}$ = 9.1 Hz, $^3J_{HH}$ = 4.0 Hz, CH–CP), 7.28–7.82 (15H, m, $CH_{arom}$) ppm; $^{13}$C NMR ($CDCl_3$, 125.7 MHz): $\delta$ = 22.93 (d, $^1J_{CP}$ = 55.6 Hz, CH–P), 33.44 (s, $CH_2$ heterocyclic), 51.28 (d, $^2J_{CP}$ = 5.1 Hz, $CH_2$–CP), 119.10 (d, $^1J_{CP}$ = 87.2 Hz, $C_{ipso}$), 130.33 (d, $^3J_{CP}$ = 12.7 Hz, $C_{meta}$), 133.27 (d, $^2J_{CP}$ = 9.8 Hz, $C_{ortho}$), 134.89 (d, $^4J_{CP}$ = 2.3 Hz, $C_{para}$), 166.61 (d, $^3J_{CP}$ = 10.5 Hz, C=O acid), 171.38, 172.00 (s, 2C=O heterocyclic) ppm; $^{31}$P NMR ($CDCl_3$, 202.4 MHz): $\delta$ = 19.09 ($Ph_3P^+$–C) ppm; IR (KBr): $\overline{\nu}$ = 1,749 and 1,678 (C=O heterocyclic), 1,610 and 1,438 (COO asym and sym) $cm^{-1}$; MS: *m/z* (%) = 449 ($M^+$, 1), 277 ($Ph_3PCH_3$, 12), 262 ($Ph_3P$, 100), 183 ($PPh_2$, 84), 115 ($C_3H_3NO_2S$, 9), 108 (PPh, 71), 77 (Ph, 12), 44 ($CO_2$, 20).

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