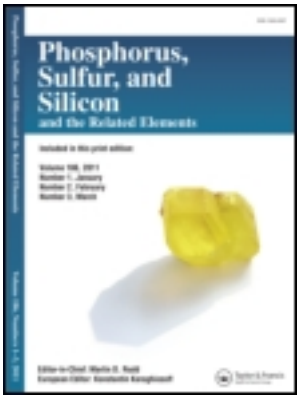

Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gpss20

Synthesis of Functionalized Phosphonates and Chromenes via Catalyst-Free Multicomponent Reactions in Water

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Accepted author version posted online: 30 Aug 2012.Published online: 31 May 2013.

To cite this article: Zinatossadat Hossaini, Faramarz Rostami Charati & Mahboubeh Ghasemian (2013): Synthesis of Functionalized Phosphonates and Chromenes via Catalyst-Free Multicomponent Reactions in Water, Phosphorus, Sulfur, and Silicon and the Related Elements, 188:5, 555-560

To link to this article: http://dx.doi.org/10.1080/10426507.2012.723768

Phosphorus, Sulfur, and Silicon, 188:555–560, 2013

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426507.2012.723768

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Phosphorus, Sulfur, and Silicon and the Related Elements 2013.188:555-560.

SYNTHESIS OF FUNCTIONALIZED PHOSPHONATES AND CHROMENES VIA CATALYST-FREE MULTICOMPONENT REACTIONS IN WATER

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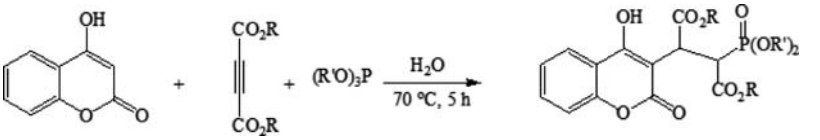
GRAPHICAL ABSTRACT



ABSTRACT *Stable derivatives of phosphonates were prepared using multicomponent reactions of dialkyl acetylenedicarboxylate with 4-hydroxycumarin in the presence of trimethyl or triphenyl phosphite in good yields. Chromene derivatives were produced by using triethyl phosphite and dialkyl acetylenedicarboxylate in the presence of 4-hydroxycumarin in excellent yields.*

Keywords Triphenyl phosphite; dialkyl acetylenedicarboxilates; multicomponent reactions; triethyl phosphite

INTRODUCTION

Multicomponent reactions (MCRs), with three or more reactants combined in a one-pot procedure to give a single product, have become increasingly popular during the last decade.[1–7] They are economically and environmentally advantageous because multistep syntheses produce considerable amounts of waste mainly due to complex isolation procedures often involving expensive, toxic, and hazardous solvents after each step. The developments of MCRs have attracted much attention from the vantage point of combinatorial and medicinal chemistry.[8] Generally, the MCR strategy affords savings in synthetic time and effort, and has significant advantages over conventional two-component reactions in

Received 16 June 2012; accepted 17 August 2012.
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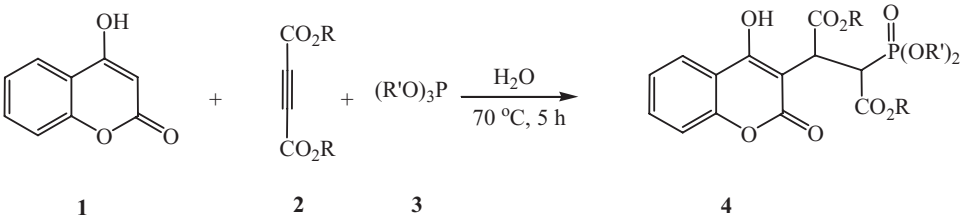
several aspects such as variable and high bond-forming efficiency. With a small set of start materials, very large libraries can be developed within a short time, which can be applied to research on medicinal chemistry. The first MCR was described in 1850 by the Strecker's synthesis of a-amino acids.[9a] Other MCRs have been described (as examples) successfully in Hantsch's synthesis of 1,4-dihydropyridines in 1882,[9b] Biginelli's synthesis of 3,4-dihydropyrimidin-2-ones in 1891,[9c] Mannich's synthesis of b-amino carbonyl compounds in 1912,[9d] Robinson's synthesis of alkaloid tropinone in 1917,[9e] Passerini synthesis of a-acyloxycarboxamide in 1921,[9f] Bucherer–Bergs's synthesis of hydantoins in 1934,[9g] Ugi's synthesis of bis-amide in 1959,[9h] and Pauson–Khand's synthesis of a,b-cyclopentenone in 1977.[9i]

Phosphonates have important applications in flame retardancy,[10,11] organic synthesis,[12] and biological applications.[13] Also, phosphonates have been used as substitutes of the corresponding esters and acids of high biological activity[14,15] and as suitable probes for designing antibodies on the basis of transition state models. A large number of methods have appeared describing novel syntheses of organophosphorus compounds.[16] In this paper, another class of products is chromenes. Chromenes have attracted substantial attention due to their biological activity and their presence in a diversity of significant natural products.[17] The reaction of dialkyl acetylenedicarboxylate and 4-hydroxycumarine in the presence of trimethyl or triphenyl phosphite leads to phosphonate derivatives **4** in high yields[18] (Scheme 1). This type of reactions in the presence of various catalysts has been investigated in literatures.[19–21]



2, 3, 4	R	R'	Yield (%) of **4**
a	Me	Me	85
b	Et	Me	80
c	Me	Ph	72
d	Et	Ph	68

Scheme 1 Reaction of phosphites, activated acetylenes, and 4-hydroxycumarin.

RESULT AND DISCUSSION

The ^{1}H NMR spectrum of **4a** displayed signals for vicinal methine protons at $\delta =$ 3.92 and 5.12, which appeared as two sets of doublet doublets with $^2J_{HP}$ and $^3J_{HP}$ values of 20.4 and 8.7 Hz, respectively. The methoxy groups of the phosphoranyl moiety are diastereotopic and show two separate doublets at $\delta = 2.92$ and 3.72. The hydroxy proton was observed as a broad singlet at $\delta = 8.12$, which disappeared with addition of D_2O.

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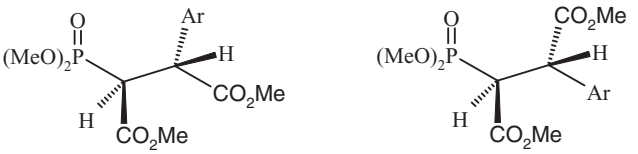
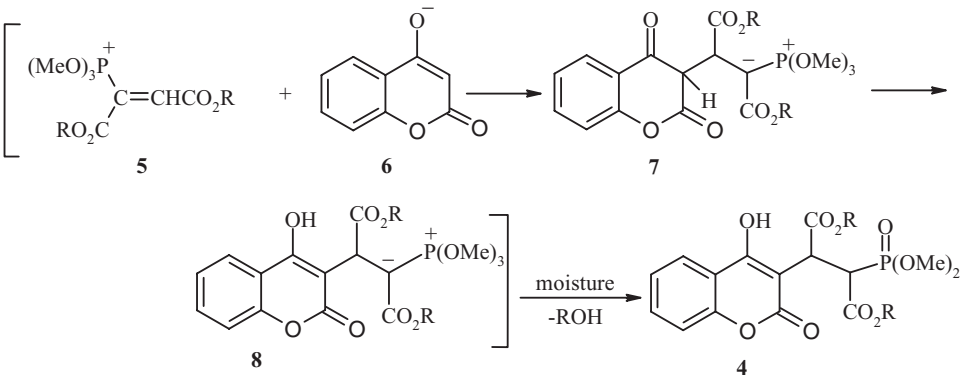


(*2R*, *3S*)-**4** or (*2S*, *3R*)-**4** (*2S*, *3S*)-**4** or (*2R*, *3R*)-**4**

Figure 1 Two diastereomers of **4a** with anti arrangement.

Observation of $^3J_{HH} = 11.7$ Hz for the vicinal methine protons in **4a** specifies the supremacy of anti arrangement. Since compound **4a** possesses two stereogenic centers, two diastereomers with anti HCCH arrangements are possible (Figure 1). The observation of $^3J_{CP}$ of 21.5 Hz for the CO_2Me group and $^3J_{CP}$ of zero for C of naphthalene moiety is in agreement with the (*2R*,*3S*) or (*2S*,*3R*) diastereoisomer.[18]

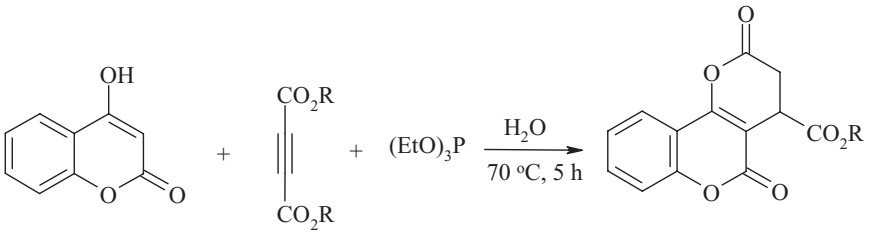
A proposed mechanism for the formation of compound **4** is shown in Scheme 2.[18] Under the reaction conditions, ylide **7** isomerizes to ylide **8** and hydrolysis of **8** leads to phosphonate derivative **4**.



Scheme 2 Proposed mechanism for the formation of **4**.

Under similar conditions, the reaction of dialkyl acetylenedicarboxylate **2** and triethyl phosphite **9** in the presence of 4-hydroxycumarin **1** produces 2,5-dioxo-3,4-dihydro-2*H*,5*H*-pyrano[3,2-c]chromene-4-carboxylate **10** in excellent yield[18] (Scheme 3). Compound **10** is possibly produced through an ylide intermediate similar to **7** (Scheme 2). However, because of the steric reasons, hydrolysis of this intermediate occurs on phosphorus atom and leads to a succinate derivatives, which is lactonized to produce **10**.

In conclusion, we found that the reaction of activated acetylenic compounds with trimethyl phosphite, triethyl phosphite, or triphenyl phosphite in the presence of 4-hydroxycumarin leads to a facile synthesis of some functionalized phosphonates and chromenes in water as green solvent without using any catalyst.



1 **2** **9** **10**

2, 10	R	Yield (%) of **10**
a	Me	87
b	Et	75

Scheme 3 Reaction of triethyl phosphite, activated acetylenes, and 4-hydroxycumarin.

EXPERIMENTAL

Melting points were measured on an Electrothermal 9100 aparatus. Elemental analyses for the C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MATT 8430 spectrometer operating at an ionization potential of 70 eV. IR spectra were measured on a Shimadzu IR-460 spectrometer. ^{1}H, ^{13}C, and ^{31}P spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500.1, 125.8, and 202.4 MHz, respectively. ^{1}H, ^{13}C, and ^{31}P spectra were obtained for solutions in $CDCl_3$ using TMS as internal standard or 85% H_3PO_4 as external standard. All the chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General Procedure for the Preparation of Compounds 4a–d

To a magnetically stirred solution of dialkyl acetylenedicarboxylate **2** (2 mmol) and 4-hydroxycumarin **1** (2 mmol) in H_2O (10 mL) was added trimethyl or triphenyl phosphite **3** (2 mmol). The reaction mixture was then stirred for 5 h at 70 °C. The completion of reaction was confirmed by TLC (EtOAc–hexane 6:1). The resulting precipitate was separated by filtration and was recrystallized from EtOH to afford the pure title compounds.

Dimethyl 2-(Dimethoxyphosphoryl)-3-(4-hydroxy-2-oxo-2H-chromen-3-yl) succinate (4a). Colorless crystals, mp 185–187 °C, 0.70 g, yield 85%. IR (KBr) (ν_{max}/cm^{-1}): 3235, 1732, 1740, 1754 cm^{-1}. Anal. calcd. for $C_{17}H_{19}O_{10}P$ (414.30): C, 49.28; H, 4.62. Found: C, 49.36; H, 4.74%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.92 (3 H, d $^{3}J_{HP}$ 11.2 Hz, MeO), 3.65 (3 H, s, MeO), 3.72 (3 H, d $^{3}J_{HP}$ 11.2 Hz, OMe), 3.85 (3 H, s, MeO), 3.92 (1 H, dd $^{2}J_{HP}$ 20.4 Hz $^{3}J_{HH}$ 11.7 Hz, CH), 5.12 (1 H, dd $^{3}J_{HH}$ 11.7 Hz $^{3}J_{HP}$ 8.7 Hz, CH), 6.95–7.92 (4 H, m, 4 CH), 8.12 (1 H, s, OH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ 43.8 (CH), 48.2 (d $^{1}J_{PC}$ 134.4 Hz, CH), 51.8 (OMe), 52.3 (d $^{2}J_{PC}$ 8.2 Hz, MeO), 53.4 (MeO), 54.0 (d, $^{2}J_{PC}$ 8.2 Hz, MeO), 115.4 (C), 122.4 (CH), 123.8 (C), 125.4 (CH), 126.8 (CH), 127.5 (C), 132.6 (CH), 149.6 (C), 165.2 (C=O), 167.5 (d $^{2}J_{PC}$ 5.4 Hz, C=O), 172.6 (d $^{3}J_{PC}$ 21.5 Hz, C=O). ^{31}P NMR (202 MHz, $CDCl_3$): δ 18.6. MS, *m/z* (%): 414 (M^+, 20), 252 (48), 162 (86), 31 (100).

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Diethyl 2-(Dimethoxyphosphoryl)-3-(4-hydroxy-2-oxo-2H-chromen-3-yl) succinate (4b). White powder, mp 192–194 °C, 0.71 g, yield 80%. IR (KBr) (ν_{max}/cm^{-1}): 3242, 1725, 1738, 1746 cm^{-1}. Anal. calcd for $C_{19}H_{23}O_{10}P$ (442.35): C, 51.59; H, 5.24. Found: C, 51.48; H, 5.18%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.34 (3 H, t, $^3J_{HH}$ 7.4 Hz, Me), 1.38 (3 H, t, $^3J_{HH}$ 7.5 Hz, Me), 3.04 (3 H, d $^3J_{HP}$ 11.6 Hz, MeO), 3.75 (3 H, d $^3J_{HP}$ 11.6 Hz, MeO), 4.02 (1 H, dd $^2J_{HP}$ 21.2 Hz $^3J_{HH}$ 12.4 Hz, CH), 4.21 (2 H, q, $^3J_{HH}$ 7.5 Hz, CH_2O), 4.27 (2 H, q, $^3J_{HH}$ 7.4 Hz, CH_2O), 5.18 (1 H, dd $^3J_{HH}$ 12.0 Hz $^3J_{HP}$ 9.2 Hz, CH), 6.87–7.90 (4 H, m, 4 CH), 8.09 (1 H, s, OH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ 13.4 (Me), 14.2 (Me), 44.0 (CH), 48.8 (d $^1J_{PC}$ 135.4 Hz, CH), 51.7 (d $^2J_{PC}$ 8.5 Hz, MeO), 54.6 (d, $^2J_{PC}$ 8.5 Hz, MeO), 61.7 (CH_2O), 62.3 (CH_2O), 114.7 (C), 122.5 (CH), 124.3 (C), 125.8 (CH), 127.5 (CH), 128.2 (C), 132.4 (CH), 149.1 (C), 164.3 (C=O), 166.8 (d $^2J_{PC}$ 5.8 Hz, C=O), 173.2 (d $^3J_{PC}$ 22.3 Hz, C=O). ^{31}P NMR (202 MHz, $CDCl_3$): δ 17.8.

Dimethyl 2-(Diphenoxyphosphoryl)-3-(4-hydroxy-2-oxo-2H-chromen-3-yl) succinate (4c). Pale yellow crystals, mp 204–206 °C, 0.77 g, yield 72%. IR (KBr) (ν_{max}/cm^{-1}): 3238, 1730, 1738, 1745 cm^{-1}. Anal. calcd for $C_{27}H_{23}O_{10}P$ (538.44): C, 60.23; H, 4.31. Found: C, 60.34; H, 4.42%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.74 (3 H, s, MeO), 3.87 (3 H, s, MeO), 4.12 (1 H, dd $^2J_{HP}$ 21.2 Hz $^3J_{HH}$ 12.2 Hz, CH), 5.23 (1 H, dd $^3J_{HH}$ 12.2 Hz $^3J_{HP}$ 9.2 Hz, CH), 7.14–7.96 (14 H, m, 14 CH), 8.05 (1 H, s, OH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ 44.2 (CH), 49.5 (d $^1J_{PC}$ 135.8 Hz, CH), 52.0 (OMe), 52.8 (MeO), 121.2 (d, $^3J_{CP}$ 6.4 Hz, 2 CH), 122.3 (d, $^3J_{PC}$ 10.2 Hz, C), 123.0 (d, $^3J_{PC}$ 5.6 Hz, 2 CH), 124.6 (CH), 126.2 (CH), 127.4 (CH), 128.2 (CH), 128.8 (CH), 130.4 (m, 4 CH), 130.8 (CH), 132.4 (C), 132.7 (C), 148.6 (d $^2J_{PC}$ 9.5 Hz, C), 150.8 (m, 2 C), 163.5 (C=O), 168.2 (d $^2J_{PC}$ 17.0 Hz, C=O), 175.3 (C=O).

Diethyl 2-(Diphenoxyphosphoryl)-3-(4-hydroxy-2-oxo-2H-chromen-3-yl) succinate (4d). Yellow powder, mp 212–214 °C, 0.76 g, yield 68%. IR (KBr) (ν_{max}/cm^{-1}): 3242, 1738, 1745, 1752. Anal. calcd for $C_{29}H_{27}O_{10}P$ (566.49): C, 61.49; H, 4.80. Found: C, 61.57; H, 4.86%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.36 (3 H, t, $^3J_{HH}$ 7.6 Hz, Me), 1.42 (3 H, t, $^3J_{HH}$ 7.6 Hz, Me), 4.16 (1 H, dd $^2J_{HP}$ 21.5 Hz $^3J_{HH}$ 12.0 Hz, CH), 4.24 (2 H, q, $^3J_{HH}$ 7.6 Hz CH_2O), 4.34 (2 H, q, $^3J_{HH}$ 7.6 Hz, CH_2O), 5.27 (1 H, dd $^3J_{HH}$ 12.5 Hz $^3J_{HP}$ 9.6 Hz, CH), 7.16-8.04 (14 H, m, 14 CH), 8.10 (1 H, s, OH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ 13.2 (Me), 13.8 (Me), 44.6 (CH), 50.2 (d $^1J_{PC}$ 136.4 Hz, CH), 61.2 (CH_2O), 62.3 (CH_2O), 121.5 (d, $^3J_{CP}$ 6.8 Hz, 2 CH), 122.7 (d, $^3J_{PC}$ 10.6 Hz, C), 123.4 (d, $^3J_{PC}$ 6.3 Hz, 2 CH), 125.4 (CH), 126.7 (CH), 128.2 (CH), 128.8 (CH), 129.3 (CH), 130.8 (m, 4 CH), 131.3 (CH), 132.9 (C), 133.5 (C), 149.2 (d $^2J_{PC}$ 10.4 Hz, C), 151.3 (m, 2 C), 164.2 (C=O), 169.3 (d $^2J_{PC}$ 18.4 Hz, C=O), 176.2 (C=O).

General Procedure for Preparation of Compounds 10a–b

To a magnetically stirred solution of dialkyl acethylenedicarboxylate **2** (2 mmol) and 4-hydroxycumarin **1** (2 mmol) in water was added triethyl phosphite **9** (2 mmol) slowly. The reaction mixture was then stirred for 5 h at 70 °C. After completion of reaction (monitored by TLC), the resulting precipitate was separated by filtration and recrystallized from EtOH to afford the pure title compounds.

Methyl 2,5-Dioxo-3,4-dihydro-2H, 5H-pyrano[3,2-c]chromene-4-carboxylate (10a). White powder, mp 130–132 °C, 0.48 g, yield 87%., IR (KBr) (ν_{max}/cm^{-1}): 1735, 1757, 1463 cm^{-1}. Anal. calcd for $C_{14}H_{10}O_6$ (274.23): C, 61.32; H, 3.68. Found: C, 61.44; H, 3.76%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.02 (1 H, dd $^2J_{HH}$ 15.8 Hz $^3J_{HH}$ 7.4 Hz, HCH), 3.28 (1 H, dd $^2J_{HH}$ 15.8 Hz $^3J_{HH}$ 2.8 Hz, HCH), 3.78 (3 H, s, MeO), 4.12 (1 H, dd $^3J_{HH}$ 7.4 Hz $^3J_{HH}$ 2.8 Hz, CH), 7.28-7.76 (4 H, m, 4 CH). ^{13}C NMR (125.7 MHz, $CDCl_3$):

δ 31.8 (CH_2), 42.2 (CH), 52.8 (MeO), 112.7 (C), 121.5 (CH), 122.6 (C), 126.2 (CH), 128.4 (CH), 134.3 (CH), 152.2 (C), 156.8 (C), 160.4 (C=O), 165.4 (C=O), 167.3 (C=O). MS, *m/z* (%): 274 (M^+, 15), 243 (86), 162 (64), 31 (100).

Ethyl 2,5-Dioxo-3,4-dihydro-2H, 5H-pyrano[3,2-c]chromene-4-carboxylate (10b). White powder, mp 138–140 °C, 0.43 g, yield 75%., IR (KBr) (ν_{max}/cm^{-1}): 1738, 1742, 1753 cm^{-1}. Anal. calcd for $C_{15}H_{12}O_6$ (274.23): C, 62.50; H, 4.20. Found: C, 62.38; H, 4.05%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.34 (3 H, t, $^3J_{HH}$ 7.4 Hz, Me), 2.97 (1 H, dd $^2J_{HH}$ 15.4 Hz $^3J_{HH}$ 7.5 Hz, HCH), 3.25 (1 H, dd $^2J_{HH}$ 15.4 Hz $^3J_{HH}$ 3.0 Hz, HCH), 4.15 (1 H, dd $^3J_{HH}$ 7.3 Hz $^3J_{HH}$ 3.0 Hz, CH), 4.24 (2 H, q, $^3J_{HH}$ 7.4 Hz, CH_2O), 7.26-7.78 (4 H, m, 4 CH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ 13.8 (Me), 32.0 (CH_2), 42.5 (CH), 62.4 (CH_2O), 113.4 (C), 121.8 (CH), 123.4 (C), 126.7 (CH), 128.7 (CH), 134.6 (CH), 152.5 (C), 157.0 (C), 161.4 (C=O), 165.8 (C=O), 167.7 (C=O).

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