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Green synthesis of phosphoryl pyran employing multicomponent reaction of alkyl bromides

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Abstract: An effective one-pot synthesis of dialkoxyphosphoryl-2-oxo-2H-pyran derivatives by three-component reaction of alky bromides and dialkylacetylenedicarboxylates in the presence of trialkyl phosphite is described. The reactions were performed under solvent-free conditions at 50ºC and neutral conditions and provided good yields of products.

Keywords:2H-pyran, Trialkyl phosphite, Dialkylacetylenedicarboxylates, Alky bromide, Three-component reaction, Green chemistry.

Introduction

One present significant area of new synthetic chemistry is the expansion of effective workable procedures that diminish the necessary reagents, solvents, cost, time, and separation courses for the preferred conversion and also minimize the creation of waste[1]. Whereas the multi component reaction (MCR) tactic is identified as a strong method in the direction of this end, a catalytic reaction involving a MCR would be more beautiful to attain this purpose. Also, Phosphorus compounds are not mostly plentiful in nature but they have various biological activities and have interested remarkable synthetic and pharmacological significance [2, 3]. Phosphonates have chief uses in flame retardancy [4, 5], organic synthesis [6] and biological applications [3, 7]. Phosphonates have been applied as substitutes of the corresponding esters and acids of high biological activity and as appropriate probes for planning antibodies due totransition state models [8, 9]. A large number of methods have showed investigating new synthesis of organophosphorus compounds [10-13]. This procedure is performed under solvent free conditions and green conditions but more of procedure that is reported in the literature was performed in the hazardous solvents such as CH_2Cl_2 and $CHCl_3$ [14].

Also, 2*H*-pyrans derivatives are a class of important heterocycles with a wide range of biological properties [15]such as spasmolytic, diuretic, anticoagulant, anti-cancer, and anti-anaphylactic activity [16].Moreover they can be used as cognitive enhancers, for the treatment of neurodegenerative diseases, including Alzheimer's disease, amyotrophic lateral sclerosis, Parkinson's disease, Huntington's disease, AIDS associated dementia and Down's syndrome as well as for the treatment of schizophrenia and myoclonus [17]. Therefore, in this report we explain the synthesis of dialkoxy phosphoryl-2-oxo-2*H*-pyrans under solvent-free conditions through the reaction of trivalent phosphorus nucleophile **1** with dialkylacetylenedicarboxylate**2** and alkyl bromides **3** (Scheme **1**).

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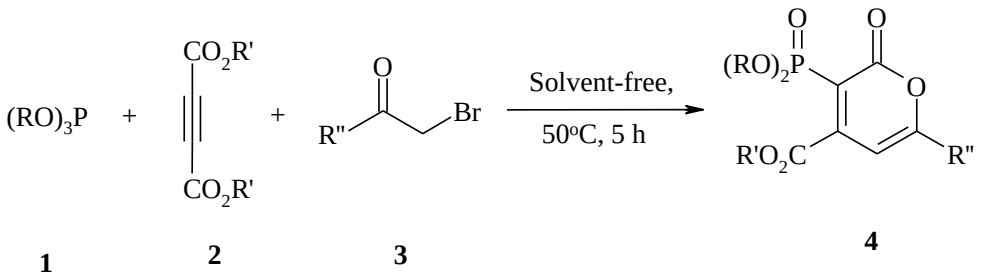


1 **2** **3** **4**

1, 2, 3, 4	R	R'	R"	Yield (%) of **4**
a	Me	Me	CO_2Et	87
b	Me	Et	CO_2Et	85
c	Et	Me	CO_2Et	80
d	Et	Et	CO_2Et	87
e	Me	Me	4-MeO-C_6H_4	85
f	Me	Me	4-Me-C_6H_4	87
g	Me	Me	4-Br-C_6H_4	80
h	Me	Me	4-NO_2-C_6H_4	75

Scheme 1. Synthesis of phosphonate derivatives

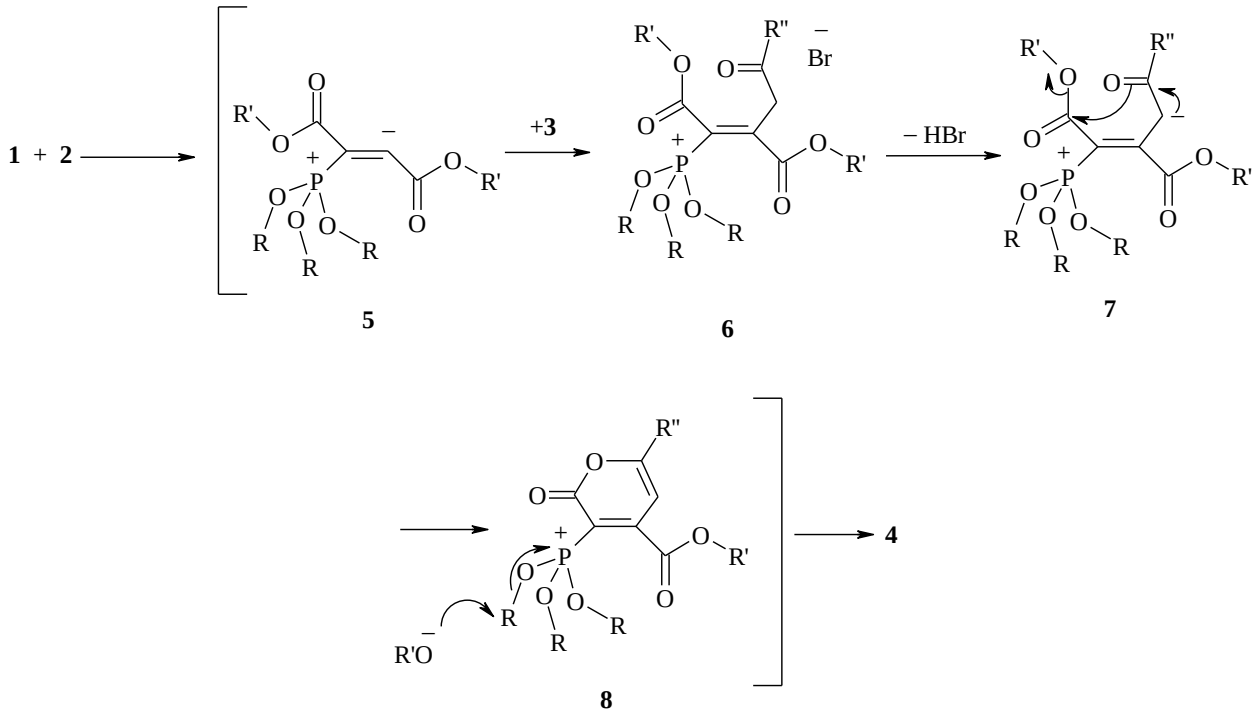
Result and discussion

To choosing the solvent, we checked several solvent systems, such as CH_3CN, H_2O, CH_2Cl_2, $CHCl_3$, Et_2O, *n*-hexane and solvent-free conditions for the synthesis of compound **4a** (Table **1**). The results were shown the quantity of preferred product was set up to be greater in solvent-free condition.

Table 1: Solvent and temperature optimization

Entry	**Solvent**	**Temp.(C)**	**Yeild(%)**
1	CH_3CN	r.t.	50
2	CH_3CN	70	68
3	H_2O	r.t.	48
4	H_2O	80	50
5	CH_2Cl_2	r.t.	35
6	$CHCl_3$	r.t.	38
7	Et_2O	r.t.	-----
8	*n*-Hexane	r.t.	-----
9	Toluene	r.t.	70
10	Toluene	80	75
11	Solvent-free	r.t.	75
12	**Solvent-free**	**50**	**87**

The ^{1}H NMR spectrum of **4a** showed one singlet for methoxy protons at (δ3.78 ppm) and one singlet for methin proton at (δ 7.62 ppm). The two methoxy groups of the phosphoranyl moiety are display one doublets at 3.75 (d $^3J_{HP}$ 11.5 Hz). The ^{13}C NMR spectrum of **4a** showed one doublets for two methoxy groups of the phosphoranyl moiety at 52.4 (d, $^2J_{PC}$ = 8.2 Hz) and resonance of methin group at 121.4 (d, $^3J_{PC}$ = 23.2 Hz, CH), along with resonance of carbonyl groups at 159.4 (d, $^2J_{PC}$ = 5.4 Hz, C=O), 162.4 (C=O), 167.2 (d, $^3J_{PC}$ = 24.3 Hz, C=O) ppmin agreement with the proposed structure. ^{31}P NMR signals was found atδ = 17.8 ppm. On the basis of the well established chemistry of trivalent phosphorus nucleophiles [18-22]it is reasonable to guess that phosphoryl-2-oxo-2*H*-pyrans **4** results from initial addition of trialkyl phosphite to the acetylenic compound and subsequent attack of the resulting anion **5** to the carbon of alkyl bromides to yield intermediate **6.** Intermediate **6** losethe HBr and produce intermediate **7** which apparently cyclizes, under the reaction conditions employed, to generate the phosphonate **4** (Scheme **2**).



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

In summary the three component reaction of trialkyl phosphite, dialkylacetylenedicarboxylate and alkyl bromides capably giving phosphoryl-2-oxo-2*H*-pyran derivatives under solvent-free conditions in good yield. The advantages of these reactions involve good yield, easy reaction workup procedures and solvent-free conditions as green conditions.

Experimental

All chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification. Melting points were measured on an Electrothermal 9100 apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN–O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MAT 8430 spectrometer operating at an ionization potential of 70 eV.

IR spectra were measured on a Shimadzu IR-460 spectrometer. ^{1}H, and ^{13}C NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500.1 and 125.8 MHz, respectively. ^{1}H, and ^{13}C, spectra were obtained for solutions in $CDCl_3$ using TMS as internal standard or 85% H_3PO_4 as external standard.

General procedure for preparation of compounds 4

To a stirred mixture ofalkyl bromides **3** (2 mmol) and dialkylacetylenedicarboxylate**2**(2 mmol) wasaddedtrialkylphosphite**1** (2 mmol) at 50ºC. The reaction mixture was stirred for 5 h at 50 ºC. After completion of reaction (monitored by TLC), 15 mL H_2O was poured into the reaction mixture, and the solid residue was filtered and washed by cold diethylether (Et_2O) to afford **4**.

*6-Ethyl-4-methyl-3-(dimethoxyphosphoryl)-2-oxo-2H-pyran-4,6-dicarboxylate (**4a**)*:

Yellow powder, m.p. 185-187°C,Yield: 0.58 g (87%). IR (KBr) (ν_{max}/cm^{-1}): 1742, 1737, 1565, 1478, 1263, 1157 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ1.27 (3 H, t, $^{3}J_{HH}$ =7.5 Hz, Me),3.75 (6 H, d $^{3}J_{HP}$ 11.5 Hz, 2 MeO), 3.87 (3 H, s, MeO), 4.22 (2 H, q, $^{3}J_{HH}$ =7.5 Hz, CH_2O), 7.62 (1 H, s, CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 13.8 (Me), 52.4 (d, $^{2}J_{PC}$ = 8.2 Hz, 2 MeO), 53.7 (MeO), 61.4 (CH_2O), 118.4 (d $^{1}J_{PC}$ = 142.4 Hz, C), 121.4 (d, $^{3}J_{PC}$ = 23.2 Hz, CH), 148.5 (C), 152.3 (d, $^{2}J_{PC}$ = 9.4 Hz, C), 159.4 (d, $^{2}J_{PC}$ = 5.4 Hz, C=O), 162.4 (C=O), 167.2 (d, $^{3}J_{PC}$ = 24.3 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 17.8. MS, *m/z* (%): 334 (M^+, 15), 303 (58), 31 (100). Anal. Calcd for $C_{12}H_{15}O_9P$(334.22): C 43.13, H 4.52;Found: C 43.24, H 4.63%.

*Dithyl-3-(dimethoxyphosphoryl)-2-oxo-2H-pyran-4,6-dicarboxylate (**4b**)*:

Yellow powder, m.p. 178-179°C,Yield: 0.59 g (85%) IR (KBr) (ν_{max}/cm^{-1}): 1745, 1738, 1574, 1483, 1276, 1195 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ1.28 (3 H, t, $^3J_{HH}$ =7.4 Hz, Me),1.32 (3 H, t, $^3J_{HH}$ =7.3 Hz, Me),3.78 (6 H, d $^3J_{HP}$ 11.8 Hz, 2 MeO), 4.23 (2 H, q, $^3J_{HH}$ =7.4 Hz, CH_2O), 4.26 (2 H, q, $^3J_{HH}$ =7.3 Hz, CH_2O), 7.67 (1 H, s, CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 14.0 (Me), 14.2 (Me), 51.8 (d, $^2J_{PC}$ = 8.5 Hz, 2 MeO), 61.5 (CH_2O), 62.3 (CH_2O), 119.2 (d $^1J_{PC}$ = 140.4 Hz, C), 122.1 (d, $^3J_{PC}$ = 22.8 Hz, CH), 149.2 (C), 151.8 (d, $^2J_{PC}$ = 8.7 Hz, C), 158.4 (d, $^2J_{PC}$ = 6.2 Hz, C=O), 163.2 (C=O), 166.5 (d, $^3J_{PC}$ = 22.3 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 18.4. MS, *m/z* (%): 348 (M^+, 10), 317 (64), 45 (88), 31 (100). Anal. Calcd for $C_{13}H_{17}O_9P$(348.24): C 44.84, H 4.92;Found: C 44.72, H 4.83%.

*6-Ethyl-4-methyl-3-(diethoxyphosphoryl)-2-oxo-2H-pyran-4,6-dicarboxylate (**4c**)*:

Yellow powder, m.p. 172-174°C,Yield: 0.58 g (80%) IR (KBr) (ν_{max}/cm^{-1}): 1747, 1742, 1587, 1495, 1234, 1147 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ1.23 (3 H, t, $^3J_{HH}$ =7.5 Hz, Me),1.27 (3 H, t, $^3J_{HH}$ =7.5 Hz, Me),1.34 (3 H, t, $^3J_{HH}$ =7.4 Hz, Me), 3.87 (MeO), 4.12 (2 H, m, CH_2O), 4.22 (2 H, m, CH_2O), 4.28 (2 H, q, $^3J_{HH}$ =7.5 Hz, CH_2O), 7.72 (1 H, s, CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 13.7 (Me), 14.0 (d, $^3J_{PC}$ = 21.8 Hz, Me), 14.3 (d, $^3J_{PC}$ = 21.8 Hz, Me), 52.3 (MeO), 61.4 (CH_2O), 62.3 (d, $^2J_{PC}$ = 9.8 Hz, CH_2O), 63.2 (d, $^2J_{PC}$ = 9.8 Hz, CH_2O), 118.4 (d $^1J_{PC}$ = 138.7 Hz, C), 124.2 (d, $^3J_{PC}$ = 23.4 Hz, CH), 148.6 (C), 152.3 (d, $^2J_{PC}$ = 9.4 Hz, C), 156.2 (d, $^2J_{PC}$ = 6.5 Hz, C=O), 163.7 (C=O), 167.2 (d, $^3J_{PC}$ = 22.7 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 18.7. MS, *m/z* (%): 362 (M^+, 15), 331 (88), 317 (82), 45 (100), 31 (100). Anal. Calcd for $C_{14}H_{19}O_9P$(362.23): C 46.42, H 5.29;Found: C 46.53, H 5.34%.

*Diethyl-3-(diethoxyphosphoryl)-2-oxo-2H-pyran-4,6-dicarboxylate (**4d**)*:

Yellow powder, m.p. 168-170°C,Yield: 0.65 g (87%) IR (KBr) (ν_{max}/cm^{-1}): 1740, 1737, 1592, 1486, 1264, 1183 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ1.25 (3 H, t, $^3J_{HH}$ =7.3 Hz, Me),1.30 (3 H, t, $^3J_{HH}$ =7.3 Hz, Me),1.36 (3 H, t, $^3J_{HH}$ =7.4 Hz, Me),1.42 (3 H, t, $^3J_{HH}$ =7.5 Hz, Me), 4.15 (2 H, m, CH_2O), 4.26 (2 H, m, CH_2O), 4.32 (2 H, q, $^3J_{HH}$ =7.5 Hz, CH_2O), 4.40 (2 H, q, $^3J_{HH}$ =7.5 Hz, CH_2O),7.78 (1 H, s, CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 13.8 (Me), 14.2 (Me), 14.8 (d, $^3J_{PC}$ = 19.2 Hz, Me), 15.3 (d, $^3J_{PC}$ = 19.3 Hz, Me), 60.4 (CH_2O), 61.2 (CH_2O), 62.0 (d, $^2J_{PC}$ = 10.2 Hz, CH_2O), 62.8 (d, $^2J_{PC}$ = 10.2 Hz, CH_2O), 116.3 (d $^1J_{PC}$ = 139.4 Hz, C), 124.7 (d, $^3J_{PC}$ = 23.5 Hz, CH), 149.2 (C), 153.4 (d, $^2J_{PC}$ = 9.8 Hz, C), 159.4 (d, $^2J_{PC}$ = 6.8 Hz, C=O), 164.2 (C=O), 167.5 (d, $^3J_{PC}$ = 21.2 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 19.4. MS, *m/z* (%): 376 (M^+, 15), 331 (86), 45 (100). Anal. Calcd for $C_{15}H_{21}O_9P$(376.29): C 47.88, H 5.63;Found: C 74.92, H 5.74%.

*Methyl-3-(dimethoxyphosphoryl)-6-(4-methoxyphenyl) 2-oxo-2H-pyran-4-carboxylate (**4e**)*:

Yellow powder, m.p. 175-177°C, Yield: 0.63 g (85%). IR (KBr) (ν_{max}/cm^{-1}): 1732, 1692, 1585, 1426, 1254, 1125 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.67 (6 H, d, $^3J_{HP}$ = 12.2 Hz, 2 MeO), 3.78 (3 H, s, MeO), 3.89 (3 H, s, MeO), 6.87 (1 H, s, CH), 7.34 (2 H, d, $^3J_{HH}$ = 7.8 Hz, 2 CH), 7.62 (2 H, d, $^3J_{HH}$ = 7.8 Hz, 2 CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 52.4 (MeO), 53.2 (d, $^2J_{PC}$ = 8.7 Hz, 2 MeO), 55.6 (MeO), 100.2 (d, $^3J_{PC}$ = 22.4 Hz, CH), 113.2 (2 CH), 115.4 (d $^1J_{PC}$ = 143.2 Hz, C), 124.2 (2 CH), 136.4 (C), 153.5 (d, $^2J_{PC}$ = 9.8 Hz, C), 156.2 (C), 159.6 (d, $^2J_{PC}$ = 6.3 Hz, C=O), 161.4 (C), 164.2 (d, $^3J_{PC}$ = 23.8 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 20.3. MS, *m/z* (%): 368 (M^+, 20), 337 (78), 107 (48), 31 (100). Anal. Calcd for $C_{16}H_{17}O_8P$(368.28): C 52.18, H 4.65;Found: C 52.24, H 4.76%.

*Methyl-3-(dimethoxyphosphoryl)-6-(4-methylphenyl)-2-oxo-2H-pyran-4-carboxylate (**4f**)*:

Yellow powder, m.p. 183-185°C, Yield: 0.63 g (87%). IR (KBr) (ν_{max}/cm^{-1}): 1738, 1687, 1574, 1453, 1284, 1163 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.36 (Me), 3.72 (6 H, d, $^3J_{HP}$ = 11.8 Hz, 2 MeO), 3.85 (3 H, s, MeO), 6.92 (1 H, s, CH), 7.28 (2 H, d, $^3J_{HH}$ = 7.6 Hz, 2 CH), 7.58 (2 H, d, $^3J_{HH}$ = 7.6 Hz, 2 CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 22.3 (Me), 51.2 (d, $^2J_{PC}$ = 9.6 Hz, 2 MeO), 52.7 (MeO), 98.6 (d, $^3J_{PC}$ = 20.7 Hz, CH), 115.8 (d $^1J_{PC}$ = 139.4 Hz, C), 123.7 (2 CH), 128.4 (2 CH), 135.2 (C), 137.5 (C), 152.7 (d, $^2J_{PC}$ = 10.4 Hz, C), 158.4 (d, $^2J_{PC}$ = 7.0 Hz, C=O), 162.3 (C), 165.6 (d, $^3J_{PC}$ = 22.7 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 21.4. MS, *m/z* (%): 352 (M^+, 15), 321 (64), 91 (56), 31 (100). Anal. Calcd for $C_{16}H_{17}O_7P$(352.28): C 54.55, H 4.86;Found: C 54.42, H 4.73%.

*Methyl-3-(dimethoxyphosphoryl)-6-(4-Bromophenyl)-2-oxo-2H-pyran-4-carboxylate (**4g**)*:

Yellow powder, m.p. 174-176°C, Yield: 0.67 g (80%). IR (KBr) (ν_{max}/cm^{-1}): 1742, 1693, 1587, 1462, 1278, 1198 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.75 (6

H, d, $^3J_{HP}$ = 12.2 Hz, 2 MeO), 3.87 (3 H, s, MeO), 6.87 (1 H, s, CH), 7.48 (2 H, d, $^3J_{HH}$ = 7.5 Hz, 2 CH), 7.67 (2 H, d, $^3J_{HH}$ = 7.5 Hz, 2 CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 51.3 (d, $^2J_{PC}$ = 10.2 Hz, 2 MeO), 52.6 (MeO), 95.4 (d, $^3J_{PC}$ = 21.2 Hz, CH), 116.2 (d $^1J_{PC}$ = 140.3 Hz, C), 124.2 (C), 127.7 (2 CH), 131.4 (2 CH), 139.2 (C), 153.5 (d, $^2J_{PC}$ = 11.7 Hz, C), 157.6 (d, $^2J_{PC}$ = 8.5 Hz, C=O), 161.7 (C), 166.5 (d, $^3J_{PC}$ = 21.8 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 22.3. Anal. Calcd for $C_{15}H_{14}BrO_7P$(417.14): C 43.19, H 3.38;Found: C 43.27, H 3.42%.

*Methyl-3-(dimethoxyphosphoryl)-6-(4-nitrophenyl)-2-oxo-2H-pyran-4-carboxylate (**4h**)*:

Yellow powder, m.p. 188-190°C, Yield: 0.57 g (75%). IR (KBr) (ν_{max}/cm^{-1}): 1739, 1696, 1588, 1485, 1263, 1178 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.78 (6 H, d, $^3J_{HP}$ = 11.5 Hz, 2 MeO), 3.83 (3 H, s, MeO), 6.84 (1 H, s, CH), 7.24 (2 H, d, $^3J_{HH}$ = 7.8 Hz, 2 CH), 7.48 (2 H, d, $^3J_{HH}$ = 7.8 Hz, 2 CH) ppm.^{13}C NMR (125.7 MHz, $CDCl_3$):δ 51.7 (d, $^2J_{PC}$ = 9.8 Hz, 2 MeO), 53.2 (MeO), 96.7 (d, $^3J_{PC}$ = 19.8 Hz, CH), 116.4 (d $^1J_{PC}$ = 142.3 Hz, C), 124.3 (2 CH), 127.8 (2 CH), 145.2 (C), 147.6 (C), 153.5 (d, $^2J_{PC}$ = 10.8 Hz, C), 159.3 (d, $^2J_{PC}$ = 8.6 Hz, C=O), 164.6 (C), 167.8 (d, $^3J_{PC}$ = 23.2 Hz, C=O) ppm. ^{31}P NMR (202 MHz, $CDCl_3$): δ 21.6. Anal. Calcd for $C_{15}H_{14}NO_7P$(383.25): C 47.01, H 3.68, N 3.65;Found: C 47.14, H 3.74, N 3.75%.

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