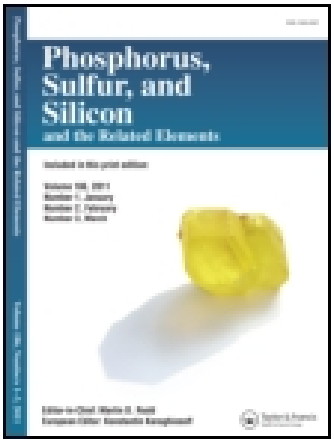

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gpss20

One-Pot Synthesis of β,γ-Unsaturated γ-Lactone Phosphorus Yildes using 2-Nitro Trans-Cinnamaldehyde and Acetylenic Esters in the Presence of Triphenylphosphine

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Accepted author version posted online: 13 Apr 2012.Published online: 20 Jul 2012.

To cite this article: Sakineh Asghari & Marie Osia (2012) One-Pot Synthesis of β,γ-Unsaturated γ-Lactone Phosphorus Yildes using 2-Nitro Trans-Cinnamaldehyde and Acetylenic Esters in the Presence of Triphenylphosphine, Phosphorus, Sulfur, and Silicon and the Related Elements, 187:10, 1195-1201, DOI: 10.1080/10426507.2012.680083

To link to this article: http://dx.doi.org/10.1080/10426507.2012.680083

Phosphorus, Sulfur, and Silicon, 187:1195–1201, 2012

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426507.2012.680083

ONE–POT SYNTHESIS OF β,γ-UNSATURATED γ-LACTONE PHOSPHORUS YILDES USING 2-NITRO TRANS-CINNAMALDEHYDE AND ACETYLENIC ESTERS IN THE PRESENCE OF TRIPHENYLPHOSPHINE

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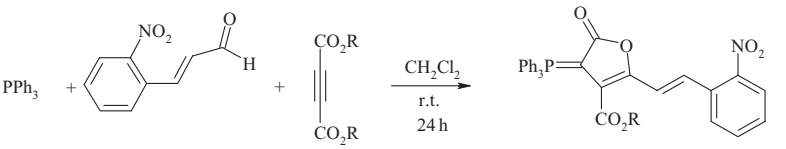
GRAPHICAL ABSTRACT



Abstract *Three-component reaction of triphenylphosphine and 2-nitro trans-cinnamaldehyde with dialkyl acetylenedicarboxylate leads to β,γ-unsaturated γ-lactone phosphorus ylides in moderate yields. The reactions of 2-nitro trans-cinnamaldehyde with alkyl propiolates in the presence of triphenylphosphine produce 2H-pyran derivatives in moderate yields without the formation of any γ-lactone derivatives.*

Keywords Triphenylphosphine; alkyl propiolate; dialkyl acetylenedicarboxylate; β,γ-unsaturated γ-lactone phosphorus ylide; 2*H*-pyran

INTRODUCTION

Lactone derivatives are common structural elements in several natural products that possess antibacterial,[1] cytotoxic[2] and immunosuppressive bioactivities.[3] In particular, γ-lactones exhibit a broad biological profile including strong antibiotic, antihelmintic, antifungal, antitumor, antiviral, and antiinflammatory activities, which makes them interesting lead structures for the development of new drugs.[4,5] Several methods for synthesis of lactones were reported in the literature.[6] Herein, as part of our efforts to the synthesis of novel heterocyclic compounds,[7–13] we report the reaction of 2-nitro trans-cinnamaldehyde **1** and triphenylphosphine in the presence of dialkyl acetylenedicarboxylates **2a–c** that lead to the β,γ-unsaturated γ-lactone phosphorus ylides **3a–c** in moderate yields. The reactions 2of 2-nitro trans-cinnamaldehyde, with alkyl propiolates **4a–b** in the presence of triphenylphosphine lead to 2*H*-pyran derivatives **5a–b** in moderate yields without the formation of any γ-lactone derivatives (Scheme 1).

Received 21 January 2012; accepted 18 March 2012.
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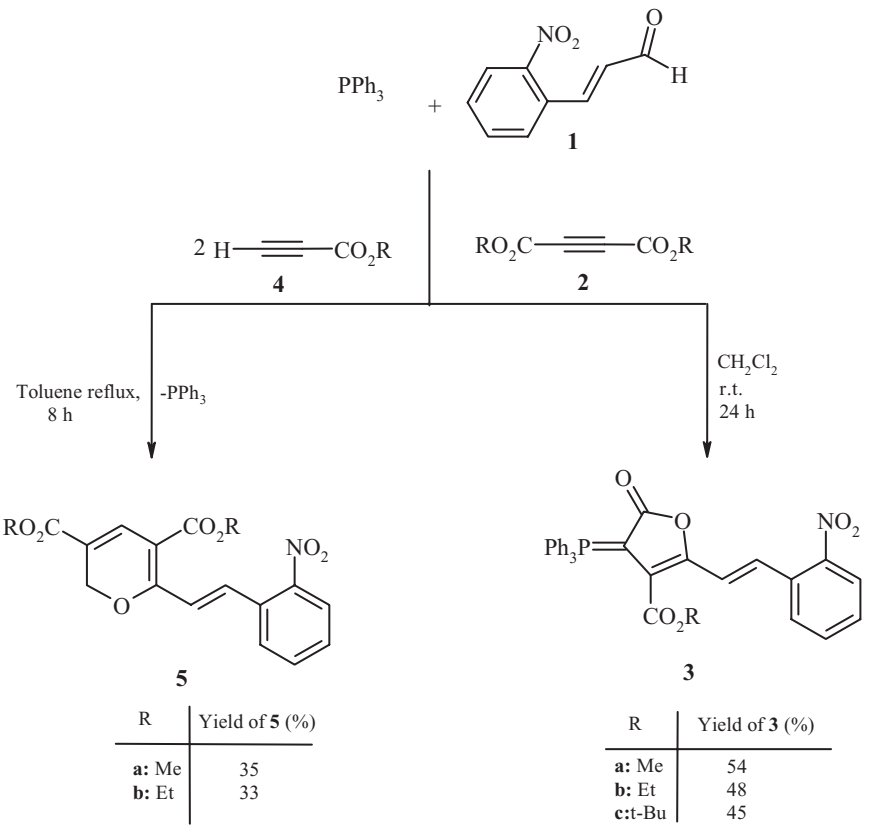


R	Yield of **5** (%)
a: Me	35
b: Et	33

R	Yield of **3** (%)
a: Me	54
b: Et	48
c:t-Bu	45

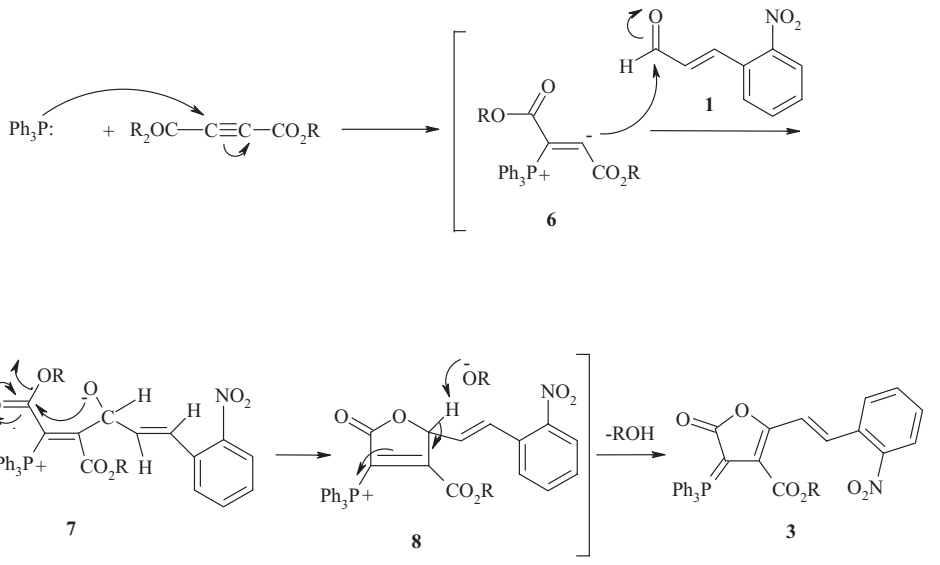
Scheme 1

RESULTS AND DISCUSSION

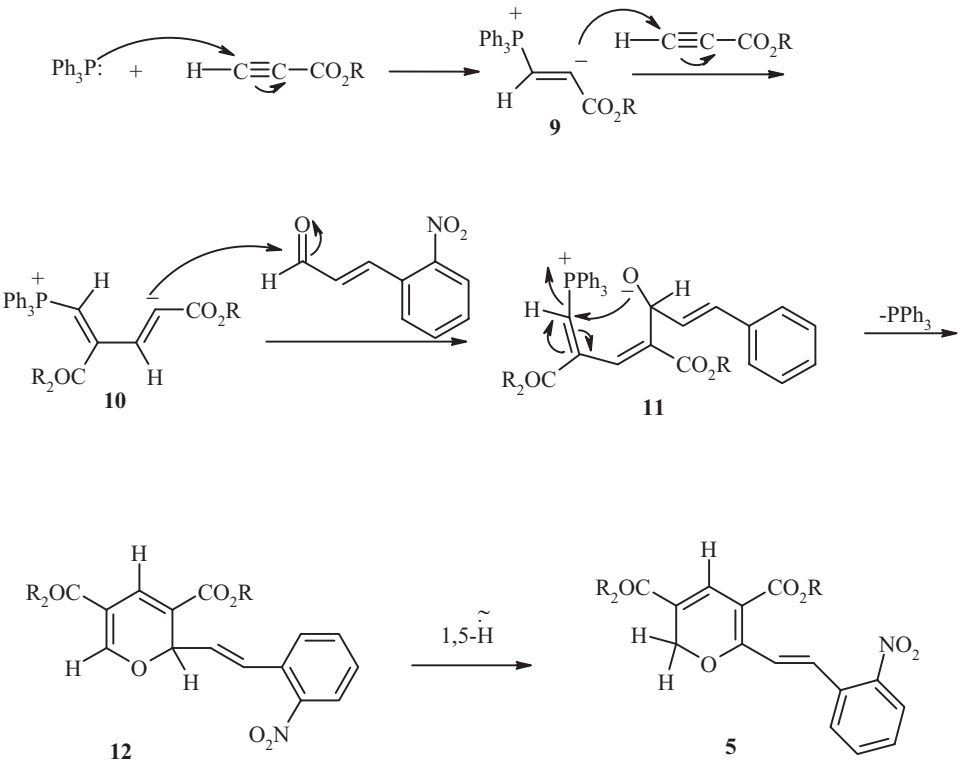
On the basis of the established chemistry of triphenylphosphine,[14–22] it is reasonable to assume that the initial Michael addition of triphenylphosphine to dialkyl acetylenedicarboxilate occurs to form zwitteronic intermediate **6**. The intermediate **6** attacks to the carbonyl group of the compound **1** that leads to the intermediate **7** which cyclizes subsequently to produce the cationic intermediate **8**. Finally, the product **3** is produced by loss of a proton from the intermediate **8** (Scheme 2).

As it is clear from the Scheme 2, the reaction of triphenylphosphine and 2-nitro trans-cinnamaldehyde in the presence of alkyl propiolate did not produce any γ-lactone derivatives. A plausible mechanism for the formation of **5** is displayed in Scheme 3. The Initial addition of triphenylphosphine was added to alkyl propiolate that leads to the reactive zwitterionic intermediate **9**. The intermediate **9** attacks to another alkyl propiolate to produce dipolar species **10**. Compound **5** results from addition of intermediate **10** to 2-nitro trans- cinnamaldehyde **1** and subsequent cyclization reaction of the intermediate **11** and then 1,5-proton transfer of **12** (Scheme 3).

The structures of **3a–c** and **5a–b** were deduced from their ^{1}H, ^{13}C, ^{31}P NMR, and IR spectra. The mass spectra of these compounds displayed molecular ion peaks at the



Scheme 2



Scheme 3

appropriate *m/z* values. Initial fragmentations involve loss of the side chains and scission of the triphenylphosphine.

The ^{1}H NMR spectrum of **3a** displayed a singlet at $\delta = 3.10$ ppm for the methoxy group. The partial assignment of the other signals is given in experimental section. The ^{13}C NMR spectrum of **3a** exhibited a singlet at $\delta = 50.4$ ppm for methoxy group, a doublet at $\delta = 51.5$ ($^1J_{PC} = 161.2$ Hz) for P = C group and four characteristic doublets at about $\delta = 124.6$ ($^1J_{PC} = 95.7$ Hz), 128.9 ($^3J_{PC} = 12.9$ Hz), 132.8 ($^4J_{PC} = 2.9$ Hz), and 133.8 ($^2J_{PC} = 10.4$ Hz) for C_{ipso}, C_{meta}, C_{para}, and C_{ortho} of triphenylphosphine moiety, respectively.

The ^{31}P NMR spectrum of **3a** showed a sharp signal at $\delta = 13.8$ ppm (downfield from 85% H_3PO_4).

The IR spectrum of **3a** displayed characteristic carbonyl stretching vibration (1693 cm^{-1}) and NO_2 asymmetric and symmetric stretching vibrations (1518 and 1377 cm^{-1}).

The ^{1}H and ^{13}C NMR of **3b–c** were similar to those of **3a** except those the substituents in position 3, which showed characteristic resonances in appropriate regions of the spectrum.

The ^{1}H NMR spectrum of **5a** displayed two singlets at $\delta = 3.84$ and 3.86 ppm for two methoxy groups, a singlet at $\delta = 5.01$ ppm for OCH_2, a singlet at $\delta = 7.66$ for vinylic proton of 2H-pyran ring, and a AB quartet system at $\delta = 7.94$ ($^2J_{HH} = 15.0$ Hz) for two vinylic protons. The aromatic protons exhibited characteristic signals in the aromatic region of the spectrum. The ^{13}C NMR spectrum of **5a** showed seventeen sharp lines in agreement with the proposed structure. The IR spectrum of **5a** exhibited carbonyl group absorptions at 1724 and 1712 cm^{-1} and the absorption bonds of NO_2 group at 1518 and 1338 cm^{-1} (see experimental). The mass spectrum of **5a** displayed the molecular ion peak at *m/z* 345 consist of appropriate value for the proposed structure. Initial fragmentations involved loss of the chains (OR, CO_2R, NO_2, O_2N–C_6H_4–CH=CH).

The ^{1}H and ^{13}C NMR spectra of **5b** were similar to those of **5a** except for the alkoxy groups, which exhibited characteristic resonances in appropriate regions of the spectrum.

CONCLUSION

In summary, we have found a facile one-pot, three-component synthesis of highly functionalized γ-lactone and 2H-pyran under neutral conditions without the use of any catalyst. Also, these γ-lactone derivatives have been used as useful building blocks toward efficient preparation of more complex polycyclic lactone derivatives, or other types of compounds. The simplicity of the present procedure also makes it an interesting alternative to complex multistep approaches.

EXPERIMENTAL

2-nitro trans-cinnamaldehyde, triphenylphosphine, alkkyl propiolates, and dialkyl acetylenedicarboxylates were obtained from Fluka (Buchs, Switzerland) and used without further purification. Mp was measured on an Electrothermal 9100 apparatus and are unconnected. IR spectra were measured on a FT-IR Bruker vector 22 spectrometer. Mass spectra were recorded on a Finnigan–Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H, ^{13}C, and ^{31}P NMR spectra were measured with a Brucker DRX-400 AVANCE spectrometer at 400.13, 100.61, and 161.97 MHz, respectively.

General Procedure for Synthesis of Alkyl 2-[(*E*)-2-(2-Nitropheny)vinyl]-5-oxo-4-(Triplenylphosphoranylidene)-4,5-Dihydrofuran-3-Carboxylate 3

To a magnetically stirred solution of triphenylphosphine (0.52 g, 2 mmol) and 2-nitro trans-cinnamaldehyde (0.35 g, 2 mmol) in CH_2Cl_2 (10 mL) was added dropwise a mixture of dialkyl acetylenedicarboxylate (2 mmol) in CH_2Cl_2 (2 mL) at -10 °C for 10 min. The mixture was allowed to stir for 24 h. The solvent was removed under reduced pressure and the viscous residue was purified by silica gel (Mereck silica gel, 230–400 mesh) column chromatography using ethyl acetate: hexane (3:7) as eluent. The solvent was removed under reduced pressure and the products **3a–c** were obtained as red powders.

Methyl 2-[(*E*)-2-(2-Nitrophenyl)vinyl]-5-oxo-4-(Triphenylphosphoranylidere)-4,5-Dihydrofuran-3-Carboxylate (3a)

Red powder; mp165–167 °C; yield 45%, IR (KBr) (ν_{max}, cm^{-1}): 1693 (C=O), 1645 (C=C), 1518 and 1377 (NO_2); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ_H 3.10 (3H, s, OCH_3), 7.34 (^{1}H, td, $^3J = 7.6$ Hz, $^4J_{HH} = 1.2$ Hz, CH), 7.51–7.75 (19 H, m, aromatic, and vinylic protons), 7.91 (1H, dd, $3J_{HH} = 8.2$ Hz, $^4J_{HH} = 0.8$ Hz, CH); ^{13}C NMR (100.61 MHz, $CDCl_3$): δ_C 50.4 (OCH_3), 51.5 (d, $^1J_{PC} = 161.2$ Hz, P=C), 116.5 (C), 119.8 and 123.2 (2CH), 124.6 (d, $^1J_{PC} = 95.7$ Hz, C_{ipso}), 124.8, 127.5, and 127.9 (3CH), 128.9 (d, $^3J_{PC} = 12.9$ Hz, C_{meta}), 132.6 (CH), 132.8 (d, $^4J_{PC} = 2.9$ Hz, C_{para}), 133.1(C), 133.8 (d, $^2J_{PC} = 10.4$ Hz, C_{ortho}), 146.0 and 148.0 (2C), 163.6 and 168.7 (2C=O); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ_P 13.81; MS, *m/z* (%): 550 ($M^+ + 1$, 2), 517 (M^+-CH_3OH, 4), 489 [$M^+ + 1-(CH_3 + NO_2)$, 9], 277 (O^+PPh_3-H, 100), 262 (PPh_3^+, 5), 199 [$M^+-(PPh_3 + CO_2Me + NO)$, 20], 184 [$M^+ + 2-(PPh_3 + CO_2Me + NO_2)$, 31], 77 ($Ph^+$, 24).

Ethyl 2-[(*E*)-2-(2-Nitrophenyl)vinyl]-5-oxo-4-(Triphenylphosphoranylidene)-4,5-Dihydro- Furan-3-Carboxylate (3b)

Red powder, mp 175–177 °C; yield 43%, IR (KBr) (ν_{max}, cm^{-1}): 1700 (C = O), 1550 and 1350 (NO_2); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ_H 0.73 (3H, t,$^3J_{HH} = 7.2$ Hz, CH_3), 3.62 (2H, q, $3J_{HH} = 7.2$ Hz, OCH_2), 7.34 (1H, td, $^3J_{HH} = 7.7$ Hz, $^4J_{HH} = 1.8$ Hz, CH), 7.46–7.74 (19 H, m, aromatic and vinylic protons), 7.91 (IH, dd, $^3J_{HH} = 8.2$ Hz, $^4J_{HH} = 0.8$ Hz, CH); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ_C 13.6 (CH_3), 51.30 (d, $^1J_{PC} = 136.2$ Hz, P=C), 60.0 (OCH_2), 116.7 ((d, $^2J_{PC} = 10.4$ Hz, C), 119.8 and 123.0 (2CH), 124.7 (d, $^1J_{PC} = 95.7$ Hz, C_{ipso}), 124.8, 127.4 and 127.8 (3CH), 128.8 (d, $^3J_{PC} = 13.0$ Hz, C_{meta}), 132.6 (CH), 132.8 (d, $^4J_{PC} = 2.9$ Hz, C_{para}), 133.1(C), 133.9 (d, $^2J_{PC} = 10.4$ Hz, C_{ortho}), 145.9, 148.0 (2C), 163.4, and 168.8 (2C=O); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ_P 13.58; MS, *m/z* (%): 563 (M^+,2), 303 ($M^+ + 2H-ph_3$, 8), 277 ($O^+PPh_3-H^+$, 100), 262 (PPh_3^+, 23), 183 [$M^+-(PPh_3 + CO_2 + C_2H_4 + NO_2)$, 38], 152 [$M^+-(PPh_3 + CH=CH-C_6H_4-NO_2 + H)$, 19], 108 [$M^+-(PPh_3 + OEt + CH=CH-C_6H_4-NO_2)$, 10], 77 ($ph^+$, 32).

Tert-butyl 2-[(*E*)-2-(2-Nitrophenyl)vinyl]-5-oxo-4-(Triphenylphosphoranylidere)-4,5-Dihydrofuran-3-Carboxylate (3c)

Red Powder, mp 140–142 °C; yield 38%, IR (KBr) (ν_{max}, cm^{-1}): 1741 (C=O), 1681 (C=O), 1647 (C=C), 1552 and 1355 (NO_2); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ_H 1.05 (9H, S, CMe_3), 3.33 (1H, td, $^3J_{HH} = 7.8$ Hz and $^4J_{HH} = 1.2$ Hz, CH), 7.45–7.75 (19H, m,

aromatic protons), 7.90 (1H, dd, $^3J_{HH} = 8.0$ Hz and $^4J_{HH} = 1.2$ Hz, CH); ^{13}C NMR (100.6, $CDCl_3$): δ_C 28.0 (C*Me*$_3$), 51.5 (d, $^1J_{PC} = 136.0$ Hz, P=C), 81.03 (O*C*Me$_3$), 113.8 (C), 120.1, 122.2 and 124.8 (3CH), 125.0 (d, $^1J_{PC} = 95.5$ Hz, C_{ipso}), 127.2, and 127.6 (2CH), 128.7 (d, $^3J_{PC} = 12.8$ Hz, C_{meta}), 132.6 (CH), 132.7 (d, $^4J_{PC} = 2.9$ Hz, C_{para}), 133.4(C), 134.1 (d, $^2J_{PC} = 10.3$ Hz, C_{ortho}), 145.0 and 148.6 (2C), 163.2, and 168.9 (2C=O); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ_P 13.31; MS, *m/z* (%): 591 (M^+, 2) 561 (M^+–NO, 5), 535 ($M^+-C_4H_8$, 6), 387 [M^+–($NO_2-C_6H_4-CH=CH + C_4H_8$), 10], 368 [$M^+$–($NO_2-C_6H_4 + CO_2$t–Bu), 13], 262 ($P^+Ph_3$, 100), 183 [$M^+$–($PPh_3 + CO_2 + C_4H_8 + NO_2$), 52], 108 [$M^+$–($NO_2-C_6H_4$–CH=CH + $PPh_3 + C_4H_9$), 34], 77(Ph^+, 22), 57 ($C_4H_9^+$, 18).

General Procedure for Synthesis of Dialkyl 2H-pyran-6-[(*E*)-2-(2-Nitrophenyl) Ethenyl]-3,5-Dicarboxylate 5

To a stired solution of 2-nitro trans-cinnamaldehyde (0.35 g, 2 mmol) and alkyl propiolate 0.34 mL, 4 mmol) was added dropwise a triphenylphosphine (0.52 g, 2 mmol) in toluene (4 mL) at room temperature. The mixture was refluxed for 8 h. The solvent was removed under reduced pressure and the residue was purified by silica gel (Merck silica gel, 230–400 mesh) column chromatography using ethyl acetate: hexane (1:4) as eluent. The solvent was evaporated under reduced pressure and the products **5a–b** were obtained as yellow powders.

Dimethyl-*2H*-pyran-6-[(*E*)-2-(2-Nitrophenyl)Ethenyl]3,5-Dicarboxylate (5a)

Yellow powder; mp170–172 °C; yield 35%; IR (KBr) (ν_{max}, cm^{-1}): 1724 and 1712 (C=O), 1645 (C=C), 1518 and 1338 (NO_2); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ_H 3.84 (3H, s, OCH_3), 3.86 (3H, s, OCH_3), 5.01 (2H, s, OCH_2), 7.54 (1H, t, $^3J_{HH} = 7.5$ Hz, CH), 7.65 (1H, t, $^3J_{HH} = 7.6$ Hz, CH), 7.66 (1H, s, CH), 7.80 (d, $^3J_{HH} = 7.8$ Hz, CH), 7.94 and 7.95 (2H, AB quartet, $^2J_{HH} = 15.0$ Hz, 2CH), 8.08 (1H, d, $^3J_{HH} = 8.0$ Hz, CH); ^{13}C NMR (100.61 MHz, $CDCl_3$): 51.9 ($2OCH_3$), 65.0 (OCH_2), 107.8, and 115.8 (2C), 123.9, 124.9, 129.0, and 129.7 (4CH), 131.5 (C), 132.8, 133.3, and 133.9 (3CH), 147.3 (C), 163.9 ($C-NO_2$), 164.6 and 165.3 (2C=O); MS, *m/z* (%): 345 (M^+, 6), 269 [M^+–(OMe + Me + NO), 20], 221 [M^+–($NO_2 + C_6H_4$ + 2H), 30], 184 [M^+–($NO_2-C_6H_4-CH=CH$ + Me) + 2H, 28], 167 [M^+–($NO_2-C_6H_4-CH=CH + 2CH_3$), 22], 149[$M^+$–($O_2N-C_6H_4 + CO_2Me$ + Me), 100], 59 (CO_2Me^+, 35).

Diethyl-*2H*-pyran-6-[(*E*)-2-(2-Nitrophenyl)Ethenyl]-3,5-Dicarboxylate (5b)

Yellow powder, mp 108–110 °C; yield 33%, IR (KBr) (ν_{max}, cm^{-1}): 1743 and 1707 (C=O), 1648 (C=C), 1552 and 1358 (NO_2); ^{1}H NMR (400.13 MHz, $CDCl_3$): δ_H 1.36 (3H, t, $^3J_{HH} = 7.2$ Hz, CH_3), 1.39 (3H, t, $^3J_{HH} = 7.2$ Hz, CH_3), 4.29 (2H, q, $^3J_{HH} = 7.2$ Hz, OCH_2), 4.32 (2H, q, $^3J_{HH} = 7.2$ Hz, OCH_2), 4.99 (2H, s, OCH_2) 4.5 (1H, td, $^3J_{HH} = 7.8$ Hz, $^4J_{HH} = 1.2$ Hz, CH), 7.62–7.66 (2H, m, 2CH), 7.80 (1H, dd, $^3J_{HH} = 7.8$ Hz, $^4J_{HH} = 0.8$ Hz, CH), 7.92 and 7.95 (2H, AB quartet, $^2J_{HH} = 15.6$ H_z, 2CH), 8.00 (1H, dd, $^3J_{HH} = 8.0$ Hz, $^4J_{HH} = 1.2$ Hz, CH); ^{13}C NMR (100.61 MHz, $CDCl_3$): δ_C 14.3 and 14.4 ($2CH_3$), 60.8, 60.9, and 65.0 ($3OCH_2$), 108.3 and 116.1 (2C), 124.0, 124.8, 129.0, and 129.6 (4CH), 131.6 (C), 132.6, 133.2, and 133.6 (3CH), 148.5 (C), 163.6 ($C-NO_2$), 164.3 and 164.9 (2C=O).

MS, *m/z* (%): 373 (M^+, 15), 343 (M^+-NO, 5), 327 (M^+-NO_2, 31), 299 (M^+-CO_2Et, 18), 281 [$M^+-(NO_2+EtOH)$, 54], 237 [$M^+-(NO_2 + 2OEt)$, 28], 197 [$M^+-(C_2H_4 + O_2N-C_6H_4-CH-CH)$, 36], 151 [$M^+-(CO_2Et + O_2N-C_6H_4-CH=CH+2H)$, 100], 102 [$C_6H_4-CH=CH^+$, 57].

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