

Three-component reaction of triphenylphosphine, dialkyl acetylenedicarboxylates, and 2-nitrocyclohexanone

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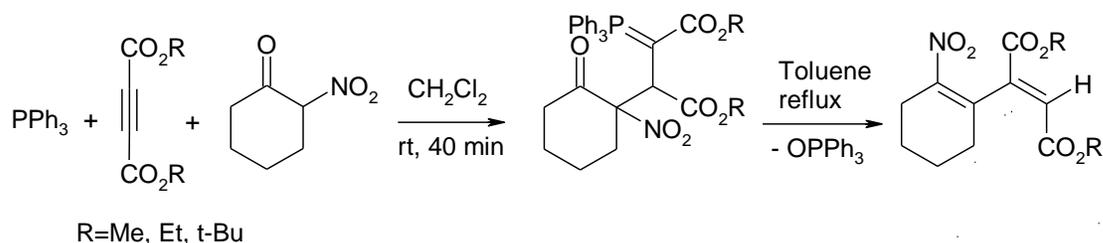
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Abstract

An efficient, one-pot three component synthesis of novel stable phosphorus ylides has been developed from the reaction between triphenylphosphine and dialkyl acetylenedicarboxylates in the presence of 2-nitrocyclohexanone. The resulting phosphorus ylides when heated in toluene afforded dialkyl 2-(2-nitrocyclohex-1-enyl)fumarate derivatives through intramolecular Wittig reaction.

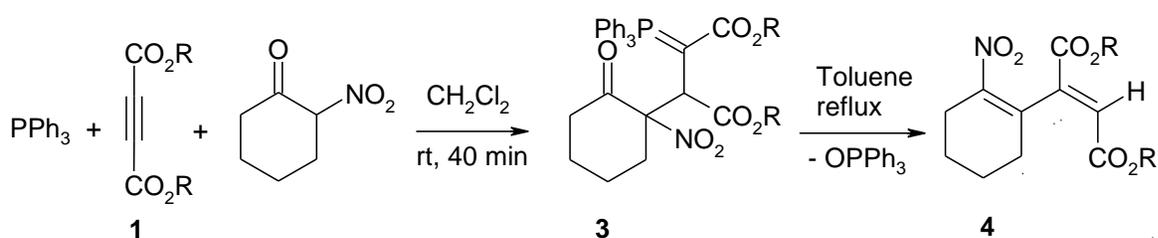


Keywords: Dialkyl acetylenedicarboxylates, triphenylphosphine, 2-nitrocyclohexanone, Wittig reaction, phosphorus ylides

Introduction

In the recent years, multicomponent condensation reactions have become a powerful method for the synthesis of organic compounds, due to the fact that products are formed in a single step, and the diversity can be achieved by simply varying each component.^{1,2}

Recently, the chemistry of organophosphorus is very significant.^{3,4} Phosphorus ylides take part in many reactions of value in organic synthesis.⁵⁻¹⁰ Several methods have been developed for the preparation of phosphorus ylides. These ylides are usually prepared by treatment of a phosphonium salt with a base, and phosphonium salts are usually obtained from the phosphine and an alkyl halide.^{3,11} In recent years, there have been some reports about the synthesis of stable phosphorus ylides.¹²⁻¹⁶ Also, the reaction between triphenylphosphine as a nucleophilic and activated alkynes have already been reviewed.¹⁷⁻¹⁹ In this article, we report the synthesis of new stable phosphorus ylides by reacting triphenylphosphine with dialkylacetylenedicarboxylates in the presence of 2-nitrocyclohexanone. The synthesized phosphorus ylides when refluxed in toluene afforded 2-(2-nitrocyclohex-1-enyl)fumarate derivatives (Scheme 1).



	R	Yield(%) ^a	(Z/E) ^b	4	Yield(%) ^a
a	Me	93	64/36	a	92
b	Et	92	69/31	b	90
c	t-Bu	90	74/31	c	87

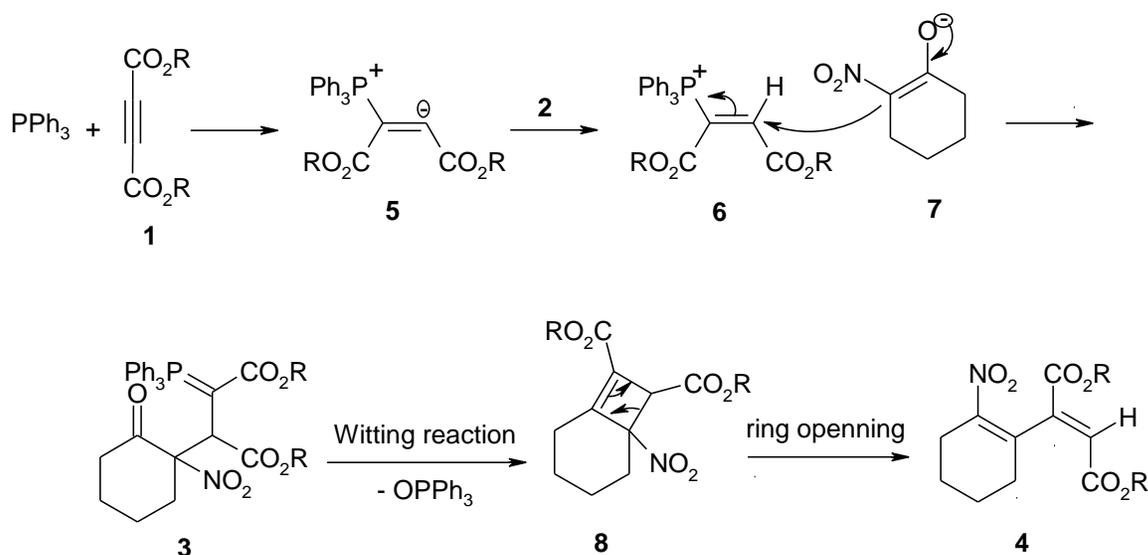
a: Isolated yield

b: Ratios determinate by ¹H NMR spectroscopy

Scheme 1

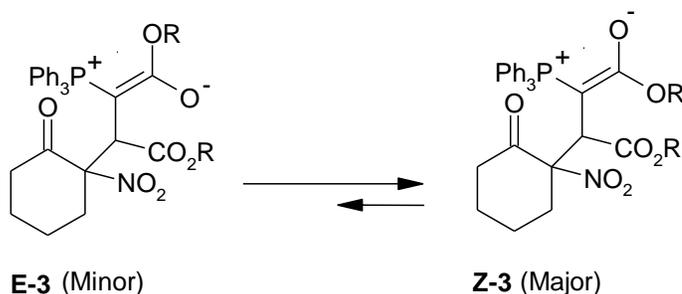
Results and Discussion

On the basis of the chemistry of trivalent phosphorus nucleophiles,^{20,21} the reaction between triphenylphosphine and dialkyl acetylenedicarboxylates in the presence of 2-nitrocyclohexanone led to the corresponding stable phosphorus ylides (Scheme 1). Because of the mechanism proposed in Scheme 2, it is obvious that the reaction between triphenylphosphine and acetylenic esters produces the carbene–ylide intermediate **5**, which is sufficiently stabilized by resonance. Thus, compounds **3** are apparently obtained from the initial addition of triphenylphosphine to the acetylenic ester and concomitant protonation of the carbene–ylide intermediate **5** by compound **2**, followed by attack of the carbon atom of the enolate anion **7** to positively charged ion **6**. This compound undergoes an intramolecular Wittig reaction in boiling toluene to produce intermediate **8**, which is finally converted to **4** by a ring-opening reaction.



Scheme 2

The ^1H and ^{13}C NMR spectra of the crude products clearly indicated the formation of phosphorus ylides **3**. The ^1H , ^{13}C , and ^{31}P NMR spectra of compounds **3a-c** showed a mixture of two rotational isomers (Scheme 3). Assignment of *E*-**3** and *Z*-**3** isomers for each ylide which could be due to strong conjugation of ylide moiety with the adjacent carbonyl group. The ^1H NMR spectrum of **3a** exhibited two singlets at 2.95 and 3.75 ppm arising from two methoxy groups in the *Z* isomer and two singlets at 3.50 and 3.71 ppm for two methoxy groups in the *E* isomer. The shift at 2.95 ppm of the methoxy group in the *Z* isomer is shielded due to the anisotropic effect of a phenyl group of triphenylphosphine. The ^{13}C NMR spectrum of **3a** displays two signals at about 48.8 and 52.0 ppm for the two methoxy groups and a doublet at about 37.7 ppm ($^1J_{\text{PC}}=124.0$ Hz) for P = C group of *Z*-isomer and two signals at about 49.9 and 51.8 ppm for the two methoxy groups and also a doublet at about 38.7 ppm ($^1J_{\text{PC}}=122.3$ Hz) for P = C group of *E*-isomer. Also, ^{31}P NMR spectrum of **3a** displays two signals at about 24.6 and 24.4 ppm which confirmed the presence of two isomers of **3a-Z** and **3a-E**. The ^1H NMR and ^{13}C NMR spectra of **3b, c** were similar to those for **3a** except for the ester moieties, which exhibited characteristic resonances in the appropriate regions of the spectrum.



Scheme 3

The structures of **4a-c** were also deduced from their ^1H , ^{13}C NMR and IR spectral data. The IR spectrum of **4a-c** displayed strong absorption bands at 1720-1650 cm^{-1} for the carbonyl groups. The ^1H NMR spectrum of **4a** showed characteristic signal at about 6.67 ppm for the CH of the olefinic group. Since vinylic proton of **4a**

appeared at frequency higher than 6.5 ppm it seems that it is cis with adjacent esteric group as reported before.²² This result confirms E-geometry of the double bond in compound **4a**. The ¹³C NMR spectrum of **4a** showed 12 distinct resonances in agreement with the proposed structure.

Conclusions

In summary, we have demonstrated a one-pot, simple, and effective procedure for the preparation of stable phosphorus ylides from the reaction between triphenylphosphine and dialkyl acetylenedicarboxylates in the presence of 2-nitrocyclohexanone as *CH*-acid at ambient temperature. The 2-(2-nitrocyclohex-1-enyl)fumarate derivatives were prepared from the synthesized phosphorus ylides via intramolecular Wittig reaction followed by subsequent ring opening process. The present method carries the advantage that, not only is the reaction performed under neutral conditions, but the substances can be mixed without any activation or modifications.

Experimental Section

General. The reagents and solvents used in this work were obtained from Aldrich and Fluka and were used without further purification. IR spectra were recorded on a Shimadzu IR-460 spectrometer. Mass spectra were recorded on Finnigan-Matt 8430 mass spectrometer operating in electron impact mode. ¹H, ¹³C, and ³¹P NMR spectra were obtained from a Bruker DRX (500 and 400) AVANCE instrument with CDCl₃ as solvent. EI-MS: Finnigan-MAT-8430 mass spectrometer, at 70 eV; in m/z. Elemental analyses were performed using a Heraeus CHN-O rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus.

General procedure for the preparation of compounds 3 and 4. To a magnetically stirred solution of triphenylphosphine (0.52 g, 2 mmol) and 2-nitrocyclohexanone (0.29 g, 2 mmol) in CH₂Cl₂ (10 mL), a mixture of dialkyl acetylenedicarboxylate (2 mmol) in CH₂Cl₂ (3 mL) was added dropwise at -5°C over 10 min. The reaction mixture was then allowed to warm up to room temperature and stirred for 40 min. The solvent was removed under reduced pressure and the residue was precipitated in cold diethyl ether then filtered and dried. The products **3a-c** were obtained as white powders. Compounds **3a-c** were refluxed in toluene for 4 h. Upon completion as shown by TLC monitoring, the solvent was removed under reduced pressure and viscous residue was purified by column chromatography [silica gel (230–240 mesh; Merck), hexane/AcOEt 4:1]. The solvent was removed under reduced pressure and products **4a-c** were obtained as yellow oils.

Dimethyl 2-(1-nitro-2-oxocyclohexan-1-yl)-3-triphenylphosphoranylidene succinate (3a). White powder, mp 193–195°C, yield 93%; IR (KBr): 1732, 1631, 1544, 1483, 1433, 1294, 1195, 1113 cm⁻¹; Anal. Calcd for C₃₀H₃₀NO₇P (547.55): C, 65.81; H, 5.52; N, 2.56, Found: C, 65.58; H, 5.33; N, 2.42.

3a-Z-isomer (68%), ¹H NMR (500 MHz): δ = 1.51–1.53 (1H, m, CH), 1.76–1.89 (3H, m, 3 CH), 2.12–2.19 (1H, m, CH), 2.46–2.49 (1H, m, CH), 2.95 (3H, s, OCH₃), 3.05–3.09 (2 H, m, 2 CH), 3.65 (d, ³J_{HP} 18.9 Hz), 3.75 (3H, s, OCH₃), 7.46–7.49 (6H, m, Ar), 7.52–7.56 (3H, m, Ar), 7.64–7.68 (6H, m, Ar); ¹³C NMR (100 MHz): 21.9 (CH₂), 24.2 (CH₂), 31.4 (CH₂), 37.7 (d, ¹J_{CP} 124.0 Hz, P=C), 39.8 (CH₂), 48.7 (d, ²J_{PC} 15.0 Hz, CH), 48.8 (OCH₃), 52.0 (OCH₃), 99.9 (Cq), 127.5 (d, ¹J_{PC} 92.0 Hz, C_{ipso}), 128.5 (d, ²J_{PC} 12 Hz, C_{ortho}), 132.1 (d, ⁴J_{PC} 2.4 Hz, C_{para}), 133.9 (d, ³J_{PC} 9 Hz, C_{meta}), 170.0 (d, ²J_{PC} 12 Hz, C=O, ester), 173.0 (d, ³J_{PC} 7 Hz, C=O, ester), 198.8 (C=O, ketone); ³¹P NMR (161.9 MHz, CDCl₃): δ P 24.6.

3a-E-isomer (32%), ^1H NMR (500 MHz): δ = 1.51-1.53 (1H, m, CH), 1.76-1.89 (3H, m, 3 CH), 2.12-2.19 (1H, m, CH), 2.46-2.49 (1H, m, CH), 3.50 (3H, s, OCH₃), 3.05-3.09 (2H, m, 2 CH), 3.71 (3H, s, OCH₃), 3.73 (d, $^3J_{HP}$ 18.9 Hz), 7.46-7.49 (6H, m, Ar), 7.52-7.56 (3H, m, Ar), 7.64-7.68 (6H, m, Ar); ^{13}C NMR (100 MHz): 21.2 (CH₂), 23.8 (CH₂), 31.3 (CH₂), 38.7 (d, $^1J_{PC}$ 122.3 Hz, P=C), 39.8 (CH₂), 47.4 (d, $^2J_{PC}$ 15.0 Hz, CH), 48.9 (OCH₃), 51.8 (OCH₃), 99.1 (Cq), 127.5 (d, $^1J_{PC}$ 92 Hz, C_{ipso}), 128.5 (d, $^2J_{PC}$ 12 Hz, C_{ortho}), 132.1 (d, $^4J_{PC}$ 2.4 Hz, C_{para}), 133.9 (d, $^3J_{PC}$ 9 Hz, C_{meta}), 165.5 (d, $^2J_{PC}$ 12 Hz, C=O, ester), 170.3 (d, $^3J_{PC}$ 7 Hz, C=O, ester), 198.8 (C=O, ketone); ^{31}P NMR (161.9 MHz, CDCl₃): δ P 24.5.

Diethyl 2-(1-nitro-2-oxocyclohexan-1-yl)-3-triphenylphosphoranylidene succinate (3b). White powder, mp 190-193°C, yield 92%; IR (KBr): 1729, 1631, 1542, 1478, 1434, 1360, 1287, 1196, 1112 cm⁻¹; Anal. Calcd for C₃₂H₃₄NO₇P (575.60): C, 66.77; H, 5.95; N, 2.43, Found: C, 66.59; H, 5.84; N, 2.51.

3b-Z-isomer (74%), ^1H NMR (500 MHz): δ = 0.38 (3H, t, $^3J_{HH}$ 7.1 Hz, CH₃), 1.34 (3H, t, $^3J_{HH}$ 7.1 Hz, CH₃), 1.48-1.51 (1H, m, CH), 1.79-1.89 (3H, m, 3CH), 2.12-2.19 (1H, m, CH), 2.46-2.49 (1H, m, CH), 3.07-3.11 (2H, m, 2CH), 3.34-3.40 (1H, m, OCH₂), 3.64 (d, $^3J_{HP}$ 18.9 Hz, CH), 3.68-3.74 (1H, m, OCH₂), 4.15-4.26 (2H, m, OCH₂), 7.45-7.48 (6H, m, Ar), 7.53-7.57 (3H, m, Ar), 7.68-7.72 (6H, m, Ar); ^{13}C NMR: 13.7 (CH₃), 14.1 (CH₃), 21.8 (CH₂), 24.1 (CH₂), 31.4 (CH₂), 37.4 (d, $^1J_{CP}$ 123.0 Hz, P=C), 39.8 (CH₂), 48.8 (d, $^2J_{PC}$ 15.0 Hz, CH), 57.7 (OCH₂), 60.9 (OCH₂), 99.9 (Cq), 126.9 (C_{ipso}) 128.4 (C_{ortho}), 132.0 (C_{para}), 133.7 (C_{meta}), 169.6 (d, $^2J_{PC}$ 13 Hz, C=O, ester), 172.6 (d, $^3J_{PC}$ 7 Hz, C=O, ester), 198.7 (C=O, ketone); ^{31}P NMR (161.9 MHz, CDCl₃): δ P 24.3.

3b-E-isomer (26%), ^1H NMR (400 MHz): δ = 1.15 (3H, $^3J_{HH}$ 7.1 Hz, CH₃), 1.37 (3H, $^3J_{HH}$ 7.1 Hz), 1.48-1.51 (1H, m, CH), 1.79-1.89 (3H, m, 3CH), 2.12-2.19 (1H, m, CH), 2.46-2.49 (1H, m, CH), 3.07-3.11 (2H, m, 2 CH), 3.34-3.40 (1H, m, OCH₂), 3.72 (d, $^3J_{PC}$ 18.9 Hz, CH), 3.90-3.94 (1H, m, OCH₂), 4.03-4.07 (1H, m, OCH₂), 4.15-4.26 (2H, m, OCH₂), 7.45-7.48 (6H, m, Ar), 7.53-7.57 (3H, m, Ar), 7.68-7.72 (6H, m, Ar); ^{13}C NMR: 14.1 (CH₃), 14.9 (CH₃), 22.0 (CH₂), 24.3 (CH₂), 31.4 (CH₂), 37.4 (d, $^1J_{CP}$ 123.0 Hz, P=C), 39.4 (CH₂), 47.5 (d, $^2J_{PC}$ 14.0 Hz, CH), 58.2 (OCH₂), 61.3 (OCH₂), 99.3 (Cq), 126.9 (C_{ipso}) 128.4 (C_{ortho}), 132.0 (C_{para}), 133.7 (C_{meta}), 169.6 (d, $^2J_{PC}$ 13 Hz, C=O, ester), 172.6 (d, $^3J_{PC}$ 7 Hz, C=O, ester), 198.7 (C=O, ketone); ^{31}P NMR (161.9 MHz, CDCl₃): δ P 24.5.

Di-tert-butyl 2-(1-nitro-2-oxocyclohexan-1-yl)-3-triphenylphosphoranylidene succinate (3c). White powder, mp 186-188°C, yield 90%; IR (KBr): 1731, 1632, 1544, 1481, 1434, 1291, 1115 cm⁻¹; Anal. Calcd for C₃₆H₄₂NO₇P (631.71): C, 68.45; H, 6.70; N, 2.22, Found: C, 68.65; H, 6.84; N, 2.14.

3c-E-isomer (76%), ^1H NMR (500 MHz): δ = 1.20 (9H, s, CMe₃), 1.44 (9H, s, CMe₃), 1.51-1.53 (1H, m, CH), 1.77-1.89 (3H, m, 3 CH), 2.12-2.19 (1H, m, CH), 2.46-2.49 (1H, m, CH), 3.06-3.11 (2H, m, 2 CH), 3.64 (d, $^3J_{HP}$ 18.9 Hz), 7.37-7.65 (15H, m, Ar); ^{13}C NMR: 21.8 (CH₂), 24.0 (CH₂), 28.2 (CMe₃), 28.5 (CMe₃), 31.4 (CH₂), 37.7 (d, $^1J_{CP}$ 123.6 Hz, P=C), 39.6 (CH₂), 48.7 (d, $^2J_{PC}$ 14.9 Hz, CH), 77.3 (CMe₃), 80.1 (CMe₃), 99.7 (Cq), 156.9 (C_{ipso}) 128.4 (C_{ortho}), 132.0 (C_{para}), 133.7 (C_{meta}), 170.1 (d, $^2J_{PC}$ 12 Hz, C=O, ester), 172.8 (d, $^3J_{PC}$ 7 Hz, C=O, ester), 198.9 (C=O, ketone); ^{31}P NMR (161.9 MHz, CDCl₃): δ P 24.1.

3c-E-isomer (24%), ^1H NMR (500 MHz): δ = 1.42 (9H, s, CMe₃), 1.49 (9H, s, CMe₃), 1.51-1.53 (1H, m, CH), 1.77-1.89 (3H, m, 3 CH), 2.12-2.19 (1H, m, CH), 2.46-2.49 (1H, m, CH), 3.06-3.11 (2H, m, 2 CH), 3.73 (d, $^3J_{HP}$ 18.9 Hz), 7.37-7.65 (15H, m, Ar); ^{13}C NMR(100 MHz): 21.2 (CH₂), 23.8 (CH₂), 31.3 (CH₂), 38.7 (d, $^1J_{PC}$ 122.3 Hz, P=C), 39.8 (CH₂), 27.4 (d, $^1J_{CP}$ 15.0 Hz, CH), 99.2 (Cq), 156.9 (C_{ipso}) 128.4 (C_{ortho}), 132.0 (C_{para}), 133.7 (C_{meta}), 165.8 (d, $^2J_{PC}$ 12 Hz, C=O, ester), 170.1 (d, $^3J_{PC}$ 7 Hz, C=O, ester), 198.9 (C=O, ketone); ^{31}P NMR (161.9 MHz, CDCl₃): δ P 24.3.

Dimethyl 2-(2-nitrocyclohex-1-enyl)fumarate (4a). Yellow oil, yield 92%; IR (KBr): 1729, 1631, 1542, 1478, 1434, 1360, 1287, 1196, 1112 cm⁻¹; Mass spectrometry (MS) (EI): m/z (%): 269 (M⁺, 21), 237 (52), 210 (100), 205 (43), 163 (68), 103 (38), 77 (15), 59 (37); Anal. Calcd for C₁₂H₁₅NO₆ (269.26): C, 53.53; H, 5.62; N, 5.20, Found: C, 53.64; H, 5.54; N, 5.31; ^1H NMR (400 MHz): δ = 1.64-1.75 (2 H, m, CH₂), 1.92-2.04 (2 H, m, CH₂), 2.29-2.35 (2 H, m, CH₂), 2.45-2.53 (2 H, m, CH₂), 3.72 (3H, s, OCH₃), 3.74 (3H, s, OCH₃), 6.65 (1H, s, olefinic proton);

^{13}C NMR (100.6 MHz, CDCl_3): δ = 20.8 (CH_2), 24.9 (CH_2), 25.8 (CH_2), 30.9 (CH_2), 52.5 (OCH_3), 53.0 (OCH_3), 124.0 (olefinic carbon, CH), 141.5, 145.6 and 146.8 (quaternary olefinic carbons), 159.1 and 161.8 ($2\text{C}=\text{O}$, ester).

Diethyl 2-(2-nitrocyclohex-1-enyl)fumarate (4b). Yellow oil, yield 90%; IR (KBr): 1729, 1631, 1542, 1478, 1434, 1360, 1287, 1196, 1112 cm^{-1} ; Mass spectrometry (MS) (EI): m/z (%): 297 (M^+ , 37), 268 (62), 251 (62), 224 (100), 177 (56), 149 (38), 103 (42), 73 (65); Anal. Calcd for $\text{C}_{14}\text{H}_{19}\text{NO}_6$ (297.31): C, 56.56; H, 6.44; N, 4.71, Found: C, 56.41; H, 6.49; N, 4.78; ^1H NMR (400 MHz): δ = 1.27 (3H, t, $^3J_{\text{HH}}$ 7.2 Hz, CH_3), 1.32 (3H, t, $^3J_{\text{HH}}$ 7.2 Hz, CH_3), 1.63-1.75 (2 H, m, CH_2), 1.93-2.05 (2 H, m, CH_2), 2.29-2.35 (2 H, m, CH_2), 2.46-2.54 (2 H, m, CH_2), 4.17 (2H, q, $^3J_{\text{HH}}$ 7.2 Hz, CH_2), 4.27 (2H, q, $^3J_{\text{HH}}$ 7.2 Hz, CH_2), 6.66 (1H, s, olefinic proton); ^{13}C NMR (100.6 MHz, CDCl_3): δ = 14.0 (CH_3), 14.3 (CH_3), 20.7 (CH_2), 24.8 (CH_2), 25.8 (CH_2), 31.0 (CH_2), 60.8 (OCH_2), 61.4 (OCH_2), 124.0 (olefinic carbon, CH), 141.4, 145.6 and 147.1 (quaternary olefinic carbons), 159.2 and 161.8 ($2\text{C}=\text{O}$, ester).

Di-tert-butyl 2-(2-nitrocyclohex-1-enyl)fumarate (4c). Yellow oil, yield 87%; IR (KBr): 1729, 1631, 1542, 1478, 1434, 1360, 1287, 1196, 1112 cm^{-1} ; Anal. Calcd for $\text{C}_{18}\text{H}_{27}\text{NO}_6$ (353.42): C, 61.17; H, 7.70; N, 3.96, Found: C 61.32; H, 7.61; N, 4.04; ^1H NMR (400 MHz): δ = 1.39 (9H, s, CMe_3), 1.48 (9H, s, CMe_3), 1.66-1.74 (2 H, m, CH_2), 1.93-2.05 (2 H, m, CH_2), 2.33-2.57 (4H, m, 2 CH_2), 6.63 (1H, s, olefinic proton); ^{13}C NMR (100.6 MHz, CDCl_3): δ = 20.7 (CH_2), 24.8 (CH_2), 25.8 (CH_2), 27.8 (CMe_3), 28.1 (CMe_3), 31.0 (CH_2), 60.8 (OCMe_3), 61.4 (OCMe_3), 124.0 (olefinic carbon, CH), 141.4, 145.6 and 147.1 (quaternary olefinic carbons), 159.2 and 161.8 ($2\text{C}=\text{O}$, ester).

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Supplementary Material

Selected copies of ^1H and ^{13}C NMR spectra of compounds 3 and 4 are given in the Supplementary Material file associated with this manuscript.

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