Triphenylphosphine-promoted *C*-Vinylation of 4-Hydroxyquinolines

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The reaction of dialkyl acetylenedicarboxylates with 4-hydroxyquinoline in the presence of triphenylphosphine (20 mol-%) produces dialkyl (*Z*)- and (*E*)-2-(4-hydroxyquinolin-3-yl)-2-butenedioates in good yields. When the reaction was performed with 2-methyl-4-hydroxyquinoline, similar (*E*)- and (*Z*)-isomers were obtained.

Key words: Acetylenic Ester, *C*-Vinylation, 4-Hydroxyquinoline, 2-Methyl-4-hydroxyquinoline, Triphenylphosphine

Introduction

Due to environmental demands, there has been considerable interest in developing a new catalyst for organic reactions that would be mild, easily available at low-cost, of high performance in transformation and wide applicability. Organophosphorus compounds are widely used in organic synthesis [1, 2]. When they act as a catalyst, 'soft' nucleophilicity is one of their most characteristic features, as shown in the Michael addition, aldol condensation, isomerization of C–C mutiple bonds [3, 4], silylcyanation of aldehydes [5], alcohol addition to methyl propiolate [6], carbonate formation from propargyl alcohol and carbon dioxide [7, 8], and cycloaddition of buta-2,3-dienoates or but-2-ynoates with electron-deficient olefins [9]. In this regard, triphenylphosphine has received increasing attention as a versatile and mild reagent in many occasions for various organic transformations under neutral conditions in recent years [10 – 15]. The addition reaction between electron-deficient acetylenic compounds and nitrogen-containing hetrocycles has been extensively investigated [16]. In continuation of our current interest in the application of triphenylphosphine and activated acetylenes in organic synthesis [17 – 20], we report here a simple one-pot synthesis of functionalized 4-hydroxyquinoline derivatives **3**.

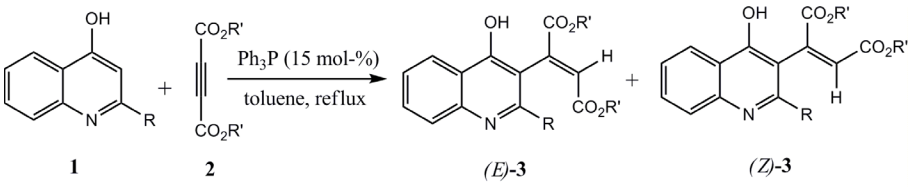
Results and Discussion

The reaction of 4-hydroxyquinoline or 2-methyl-4-hydroxyquinoline and dialkyl acetylenedicarboxylates (**2**) in the presence of Ph_3P (20 mol-%) leads to dialkyl (*Z*)- and (*E*)-2-(4-hydroxyquinolin-3-yl)-2-butenedioates **3a – c** and dialkyl (*Z*)- and (*E*)-2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioates **3d – f** in good yields (Scheme 1).

The reaction of **1** with dialkyl acetylenedicarboxylate **2** in the presence of Ph_3P in boiling toluene was completed within 24 h. The ^{1}H and ^{13}C NMR spectra of the crude products clearly indicated the formation of **3**. The ^{1}H NMR spectra of **3a** exhibited signals for methoxy and methine protons, together with characteristic multiplets for the aromatic protons. The ^{13}C NMR spectrum of (*Z*)-**3a** or (*E*)-**3a** showed 15 distinct resonances in agreement with the proposed structure. Partial assignments of these resonances are given in the Experimental Section. The structural assignments of compounds (*Z*)-**3** and (*E*)-**3** made on the basis of their ^{1}H and ^{13}C NMR spectra were supported by the IR spectra. The carbonyl region of these compounds displayed characteristic absorption bands.

NMR spectroscopy was employed to distinguish between (*Z*)-**3** and (*E*)-**3**. The *Z* and *E* configuration of the olefinic double bond in **3a – f** is based on the chemical shift of the olefinic proton [21]. The ^{1}H NMR spectra of (*Z*)-**3** showed the olefinic proton signal at 6.11 – 6.75 ppm, while the (*E*)-**3** isomer exhibited the olefinic proton at 6.83 – 8.03 ppm.

Although the mechanistic details of the reaction are not clearly known, a plausible rationalization may be advanced to explain the product formation. Pre-

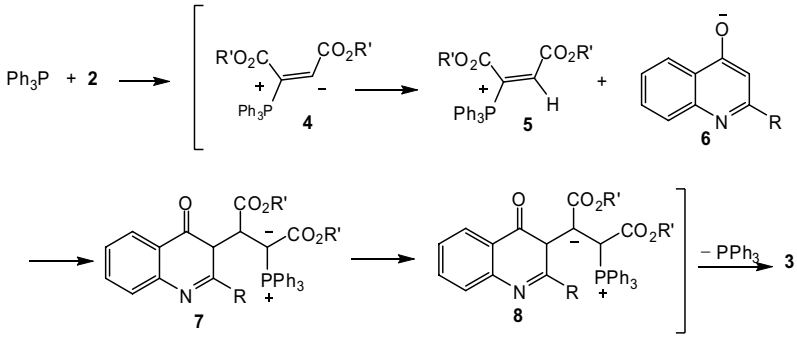


1	R
a	H
b	Me

2	R'
a	Me
b	Et
c	*t*-Bu

3	R	R'	Yield of **3** (%)	*Z*/*E*
a	H	Me	96	1.2
b	H	Et	92	3.1
c	H	*t*-Bu	92	–
d	Me	Me	94	2.1
e	Me	Et	92	1.5
f	Me	*t*-Bu	88	3.2

Scheme 1.



Scheme 2.

sumably, the zwitterionic intermediate [22] **4** formed from Ph_3P and dialkyl acetylenedicarboxylate is protonated by 4-hydroxyquinoline to furnish intermediate **5**, which then is attacked by the conjugate base **6** to produce ylide **7**. This intermediate undergoes proton transfer to furnish the 1,3-diionic structure **8**, which is converted to the final product by loss of Ph_3P (Scheme 2).

Conclusion

We revealed a novel transformation involving 4-hydroxyquinoline derivatives, dialkyl acetylenedicarboxylates, and Ph_3P that results in *C*-vinylation of the aromatic ring at the *ortho* position, affording dialkyl (*Z*)- and (*E*)-2-(4-hydroxyquinolin-3-yl)-2-butenedioates and dialkyl (*Z*)- and (*E*)-2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioates. The present procedure carries the advantage that not only is the reaction performed under neutral conditions, but also the reactants can be mixed without any prior activation or modification.

Experimental Section

General

Melting points were measured on an Electrothermal-9100 apparatus. IR spectra were measured with a Shimadzu IR-460 spectrometer. ^1H and ^{13}C spectra were determined on a Bruker DRX-300 Avance instrument in $CDCl_3$ at 300 and 75 MHz, respectively, with δ in ppm and J in Hz. EI mass spectra (70 eV) were measured on a Finnigan-MAT-8430 mass spectrometer. Elemental analyses (C, H, N) were performed with a Heraeus CHN-O-Rapid analyzer. ^1H and ^{13}C NMR spectra were obtained from solutions in $CDCl_3$ using TMS as internal 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 **3***

To a stirred solution of Ph_3P (0.052 g, 20 mol-%) and **1** (0.48 g, 2 mmol) in toluene (20 mL), a solution of **2** (2 mmol) in toluene (20 mL) was added at r. t. The reaction mixture was refluxed for 24 h. The solvent was removed under reduced pressure, and the residue was separated by column chromatography (SiO_2; EtOAc) to afford the pure title compounds.

*(E)-Dimethyl 2-(4-hydroxyquinolin-3-yl)-2-butenedioate, (E)-**3a***

Yellow powder; yield: 0.25 g (43 %); m. p. 183 – 185 °C. – IR (KBr): ν = 3432 (OH), 1723, 1671 (C=O), 1607, 1556, 1354, 1169 cm^{-1}. – ^1H NMR (300 MHz, $CDCl_3$): δ = 3.71,

3.83 (2 × s, 6 H, OMe), 7.43 (t, J = 6.6 Hz, 1 H, CH), 7.65 (t, J = 8.0 Hz, 1 H, CH), 8.03 (s, 1 H, CH), 8.13 (s, 1 H, CH), 7.86 (d, J = 7.5 Hz, 1 H, CH), 8.29 (d, J = 8.3 Hz, 1 H, CH), 10.88 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 51.98, 53.08 (2 OMe), 115.86 (CH), 119.02 (C), 124.46 (CH), 126.31 (CH), 127.01 (C), 127.32 (CH), 132.76 (CH), 139.53 (C), 141.17 (CH), 142.18 (C), 146.72 (C), 167.27, 170.10 (2 C=O). – MS (EI, 70 eV): m/z (%) = 256 (100) [M–OCH_3]$^+$. – $C_{15}H_{13}NO_5$ (287.27): calcd. C 62.72, H 4.56, N 4.88; found C 62.74, H 4.51, N 4.85.

*(Z)-Dimethyl 2-(4-hydroxyquinolin-3-yl)-2-butenedioate, (Z)-**3a***

Yellow powder; yield: 0.30 g (53 %); m. p. 176 – 178 °C. – IR (KBr): ν = 3437 (OH), 1728, 1673 (C=O), 1630, 1505, 1373, 1154 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.63, 3.72 (2× s, 6 H, OMe), 6.75 (s, 1 H, CH), 7.31 (t, J = 6.8 Hz, 1 H, CH), 7.60 (s, 1 H, CH), 7.63 (t, J = 7.9 Hz, 1 H, CH), 7.86 (d, J = 7.5 Hz, 1 H, CH), 8.21 (d, J = 8.1 Hz, 1 H, CH), 10.80 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 51.73, 52.79 (2 OMe), 113.73 (CH), 118.80 (C), 123.81 (CH), 125.32 (CH), 126.56 (C), 127.24 (CH), 132.42 (CH), 133.17 (C), 140.55 (CH), 141.64 (C), 145.73 (C), 166.15, 168.01 (2 C=O). – MS (EI, 70 eV): m/z (%) = 256 (100) [M–OCH_3]$^+$. – $C_{15}H_{13}NO_5$ (287.27): calcd. C 62.72, H 4.56, N 4.88; found C 62.75, H 4.52, N 4.83.

*(E)-Diethyl 2-(4-hydroxyquinolin-3-yl)-2-butenedioate, (E)-**3b***

Yellow powder; yield: 0.14 g (23 %); m. p. 173 – 175 °C. – IR (KBr): ν = 3431 (OH), 1719, 1674 (C=O), 1607, 1527, 1357, 1165 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.25, 1.31 (2× t, J = 7.1 Hz, 6 H, Me), 4.16, 4.30 (2× q, J = 7.1 Hz, 4 H, OCH_2), 7.42 (t, J = 6.7 Hz, 1 H, CH), 7.52 – 7.70 (m, 2 H, 2 CH), 7.62 (s, 1 H, CH), 8.04 (s, 1 H, CH), 8.30 (d, J = 8.1 Hz, 1 H, CH), 10.76 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 14.28, 14.30 (2 Me), 61.00, 61.94 (2 OCH_2), 116.34 (CH), 118.96 (C), 124.37 (CH), 126.52 (CH), 127.35 (C), 129.33 (CH), 132.56 (CH), 139.78 (C), 141.27 (CH), 142.04 (C), 146.81 (C), 165.77, 167.54 (2 C=O). – MS (EI, 70 eV): m/z (%) = 270 (100) [M–OC_2H_5]$^+$. – $C_{17}H_{17}NO_5$ (315.32): calcd. C 64.75, H 5.43, N 4.44; found C 64.77, H 5.42, N 4.42.

*(Z)-Diethyl 2-(4-hydroxyquinolin-3-yl)-2-butenedioate, (Z)-**3b***

Yellow powder; yield: 0.43 g (69 %); m. p. 177 – 179 °C. – IR (KBr): ν = 3431 (OH), 1725, 1668 (C=O), 1636, 1504, 1354, 1175 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.15, 1.21 (2× t, J = 7.1 Hz, 6 H, Me), 4.09, 4.20 (2× q, J = 7.1 Hz, 4 H, OCH_2), 6.74 (s, 1 H, CH), 7.35 (t, J = 6.8 Hz, 1 H, CH), 7.52 – 7.70 (m, 2 H, 2 CH), 8.10 (s, 1 H, CH), 8.21 (d, J = 8.0 Hz, 1 H, CH), 10.71 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 14.24, 14.27 (2 Me), 60.96, 61.88 (2 OCH_2), 115.77 (CH), 118.57 (C), 123.91 (CH), 125.95 (CH), 126.32 (C), 129.42 (CH), 131.92 (CH), 139.95 (C), 140.09 (CH), 140.67 (C), 146.31 (C), 165.35, 166.41 (2 C=O). – MS (EI, 70 eV): m/z (%) = 256 (100) [M–OCH_3]$^+$. – $C_{17}H_{17}NO_5$ (315.32): calcd. C 64.75, H 5.43, N 4.44; found C 64.75, H 5.47, N 4.41.

*(Z)-Ditert-butyl 2-(4-hydroxyquinolin-3-yl)-2-butenedioate, (Z)-**3c***

Yellow powder; yield: 0.68 g (92 %); m. p. 217 – 219 °C. – IR (KBr): ν = 3435 (OH), 1726, 1706 (C=O), 1626, 1511, 1376, 1151 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.34, 1.46 (2× s, 18 H, C(*Me*)$_3$), 6.64 (s, 1 H, CH), 7.16 (t, J = 6.7 Hz, 1 H, CH), 7.39 (d, J = 8.1 Hz, 1 H, CH), 7.43 (t, J = 6.9 Hz, 1 H, CH), 7.73 (s, 1 H, CH), 8.19 (d, J = 8.1 Hz, 1 H, CH), 10.83 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 27.99, 28.07 (2 C(*Me*)$_3$), 81.26, 81.70 (2 *C*(Me)$_3$), 116.88 (CH), 118.89 (C), 124.21 (CH), 126.59 (CH), 126.63 (C), 129.06 (CH), 132.54 (CH), 134.6 (C), 140.34 (CH), 141.81 (C), 145.40 (C), 165.44 ,167.95 (2 C=O). – MS (EI, 70 eV): m/z (%) = 317 (100) [M–$C(CH_3)_3$]$^+$.– $C_{21}H_{25}NO_5$ (371.43): calcd. C 67.91, H 6.78, N 3.77; found C 67.93, H 6.77, N 3.74.

*(E)-Dimethyl 2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioate, (E)-**3d***

Yellow powder; yield: 0.18 g (30 %); m. p. 177 – 179 °C. – IR (KBr): ν = 3436 (OH), 1717, 1673 (C=O), 1618, 1539, 1437, 1119 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.30 (s, 3 H, Me), 3.62, 3.71 (2× s, 6 H, OMe), 6.99 (s, 1 H, CH), 7.29 (t, J = 7.02 Hz, 1 H, CH), 7.53 (d, J = 7.8 Hz, 1 H, CH), 7.61 (t, J = 7.6 Hz, 1 H, CH), 8.14 (t, J = 8.1 Hz, 1 H, CH), 10.78 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 19.34 (Me), 51.71, 52.77 (2 OMe), 116.02 (CH), 119.09 (C), 123.59 (CH), 124.5 (CH), 126.13 (C), 126.53 (CH), 127.03 (CH), 132.45 (C), 132.77 (C), 141.26 (C), 148.67 (C), 167.29, 168.02 (2 C=O). – MS (EI, 70 eV): m/z (%) = 242 (100) [M–CO_2CH_3]$^+$.– $C_{16}H_{15}NO_5$ (301.1): calcd. C, 63.78, H, 5.02, N 4.65; found C, 63.74, H, 5.05, N 4.62.

*(Z)-Dimethyl 2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioate, (Z)-**3d***

Yellow powder; yield: 0.38 g (64 %); m. p. 182 – 184 °C. – IR (KBr): ν = 3435 (OH), 1716, 1670 (C=O), 1621, 1580, 1477, 1185 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.52 (s, 3 H, Me), 3.70, 3.73 (2× s, 6 H, OMe), 6.28 (s, 1 H, CH), 7.26 (t, J = 7.0 Hz, 1 H, CH), 7.50 (d, J = 7.8 Hz, 1 H, CH), 7.59 (t, J = 7.6 Hz, 1 H, CH), 8.17 (t, J = 8.1 Hz, 1 H,

CH), 10.87 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 19.93 (Me), 51.89, 52.86 (2 OMe), 118.39 (CH), 119.24 (C), 123.89 (CH), 125.34 (CH), 126.29 (C), 126.88 (CH), 129.93 (CH), 132.545 (C), 133.15 (C), 141.70 (C), 148.15 (C), 167.43, 168.02 (2 C=O). – MS (EI, 70 eV): m/z (%) = 242 (100) $[M–CO_2CH_3]^+$.– $C_{16}H_{15}NO_5$ (301.1): calcd. C, 63.78, H, 5.02, N 4.65; found C, 63.80, H, 5.01, N 4.62.

*(E)-Diethyl 2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioate, (E)-**3e***

Yellow powder; yield: 0.24 g (37 %); m. p. 163 – 165 °C. – IR (KBr): ν = 3442 (OH), 1732, 1673 (C=O), 1621, 1580, 1459, 1157 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.11, 1.23 (2× t, J = 7.1 Hz, 6 H, Me), 2.30 (s, 3 H, Me), 4.05, 4.18 (2× q, J = 7.1 Hz, 4 H, OCH_2), 6.99 (s, 1 H, CH), 7.31 (t, J = 7.1 Hz, 1 H, CH), 7.55 (d, J = 7.6 Hz, 1 H, CH), 7.62 (t, J = 7.5 Hz, 1 H, CH), 8.16 (t, J = 8.0 Hz, 1 H, CH), 10.93 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 14.25, 14.33, 19.04 (3 Me), 61.04, 62.01 (2 OCH_2), 116.34 (CH), 118.38 (C), 123.81 (CH), 125.15 (CH), 126.32 (C), 127.82 (CH), 130.33 (CH), 132.44 (C), 140.38 (C), 141.48 (C), 148.19 (C), 165.33, 166.86 (2 C=O). – MS (EI, 70 eV): m/z (%) = 256 (100) $[M–CO_2C_2H_5]^+$.– $C_{18}H_{19}NO_5$ (329.35): calcd. C 65.64, H 5.81, N 4.25; found C 65.61, H 5.84, N 4.26.

*(Z)-Diethyl 2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioate, (Z)-**3e***

Yellow powder; yield: 0.36 g (55 %); m. p. 167 – 169 °C. – IR (KBr): ν = 3431 (OH), 1725, 1676 (C=O), 1636, 1504, 1475, 1175 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.21, 1.27 (2× t, J = 7.1 Hz, 6 H, Me), 2.52 (s, 3 H, Me), 4.16, 4.21 (2× q, J = 7.1 Hz, 4 H, OCH_2), 6.28 (s, 1 H, CH), 7.32 (t, J = 6.9 Hz, 1 H, CH), 7.52 (d, J = 7.5 Hz, 1 H, CH), 7.59 (t, J = 7.7 Hz, 1 H, CH), 8.14 (t, J = 8.1 Hz, 1 H, CH), 10.95 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 14.15, 14.41, 19.35 (3 Me), 61.15, 61.34 (2 OCH_2), 118.00 (CH), 118.45 (C), 124.20 (CH), 125.31 (CH), 126.39 (C), 127.85 (CH), 130.38 (CH), 132.76 (C), 140.60 (C), 142.45 (C), 149.34 (C), 166.02, 167.49 (2 C=O). – MS (EI, 70 eV): m/z (%) = 256 (100) $[M–CO_2C_2H_5]^+$.– $C_{18}H_{19}NO_5$ (329.35): calcd. C 65.64, H 5.81, N 4.25; found C 65.65, H 5.83, N 4.26.

*(E)-Ditert-butyl 2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioate, (E)-**3f***

Yellow powder; yield: 0.13 g (17 %); m. p. 217 – 219 °C. – IR (KBr): ν = 3441 (OH), 1723, 1671 (C=O), 1626, 1541, 1459, 1150 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.28, 1.45 (2× s, 18 H, C(*Me*)$_3$), 2.38 (s, 3 H, Me), 6.83 (s, 1 H, CH), 7.29 (t, J = 7.00 Hz, 1 H, CH), 7.51 (d, J = 7.8 Hz, 1 H, CH), 7.58 (t, J = 7.5 Hz, 1 H, CH), 8.17 (t, J = 8.1 Hz, 1 H, CH), 10.67 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 19.42 (Me), 27.84, 28.15 (2 C(*Me*)$_3$), 81.25, 82.35 (2 *C*(Me)$_3$), 117.51 (CH), 117.71 (C), 123.67 (CH), 125.76 (CH), 125.82 (C), 127.82(CH), 131.71 (CH), 131.86 (C), 139.23 (C), 141.33 (C), 148.68 (C), 164.68, 167.60 (2 C=O). – MS (EI, 70 eV): m/z (%) = 331 (100) $[M–C(CH_3)_3]^+$.– $C_{22}H_{27}NO_5$ (385.45): calcd. C 68.55, H 7.06, N 3.63; found C 68.58, H 7.04, N 3.64.

*(Z)-Ditert-butyl 2-(4-hydroxy-2-methylquinolin-3-yl)-2-butenedioate, (Z)-**3f***

Yellow powder; yield: 0.55 g (71 %); m. p. 208 – 210 °C. – IR (KBr): ν = 3423 (OH), 1741, 1721 (C=O), 1635, 1551, 1478, 1147 cm^{-1}. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.47, 1.51 (2× s, 18 H, C(*Me*)$_3$), 2.49 (s, 3 H, Me), 6.11 (s, 1 H, CH), 7.30 (t, J = 7.00 Hz, 1 H, CH), 7.49 (d, J = 7.8 Hz, 1 H, CH), 7.60 (t, J = 7.5 Hz, 1 H, CH), 8.15 (t, J = 8.1 Hz, 1 H, CH), 10.73 (brs, 1 H, OH). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 19.04 (Me), 27.74, 27.89 (2 C(*Me*)$_3$), 81.07, 82.11 (2 *C*(Me)$_3$), 116.00 (CH), 117.62 (C), 123.45 (CH), 124.23 (CH), 125.82 (C), 127.24 (CH), 131.65 (CH), 131.86 (C), 139.11 (C), 140.73 (C), 147.04 (C), 164.19, 165.97 (2 C=O). – MS (EI, 70 eV): m/z (%) = 331 (100) $[M–C(CH_3)_3]^+$.– $C_{22}H_{27}NO_5$ (385.45): calcd. C 68.55, H 7.06, N 3.63; found C 68.55, H 7.07, N 3.62.

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