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ORIGINAL PAPER

Regioselective vinylation of kojic acid using acetylenic esters in the presence of triphenylphosphine or *tert*-butyl isocyanide

Sakineh Asghari · Maryam Faraji-Najjarkolaee · Mohammad Ahmadipour

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Abstract The 1:1 reactive intermediates generated by addition of *tert*-butyl isocyanide or triphenylphosphine to acetylenic esters were trapped by kojic acid to yield O-vinylated and C-vinylated kojic acid, respectively.

Keywords *tert*-Butyl isocyanide · Triphenylphosphine · Acetylenic ester · Kojic acid · O-vinylation · C-vinylation

Introduction

Kojic acid (5-hydroxy-2-hydroxymethyl-4*H*-pyran-4-one, **1**), a γ-pyrone derivative, is a fungal metabolite produced by many species of Aspergillus and Penicillium. Kojic acid has both antibacterial and antifungal activity [1]. A mixture of ascorbic acid and kojic acid has been patented for use as an anti-browning agent in foods [2]. Kojic acid has potential applicability in the prevention of melanosis in both plant and seafood products [3]. Therefore, the synthesis of kojic acid derivatives is of current interest. The reaction of some of nucleophiles for example isocyanide and triphenylphosphine with acetylenic esters in the presence of ZH-acids (Z = O, S, NH, CH) has been studied [4–23]. Literature study revealed that 4-hydroxy-6-methyl-2*H*-pyran-2-one (**2**), a 2-pyrone derivative, undergoes a three-component reaction with isocyanide and an acetylenic diester [24], as OH-acid, generating an enolate which subsequently attacks the cationic intermediate at its soft site (the negative carbon atom) in a regioselective manner.

S. Asghari (✉) · M. Faraji-Najjarkolaee · M. Ahmadipour
Department of Chemistry, Mazandaran University,
P.O. Box 453, Babolsar, Iran
e-mail: s.asghari@umz.ac.ir

These results motivated us to study this type of the reaction with kojic acid, a 4-pyrone derivative, which is an inexpensive naturally available compound.

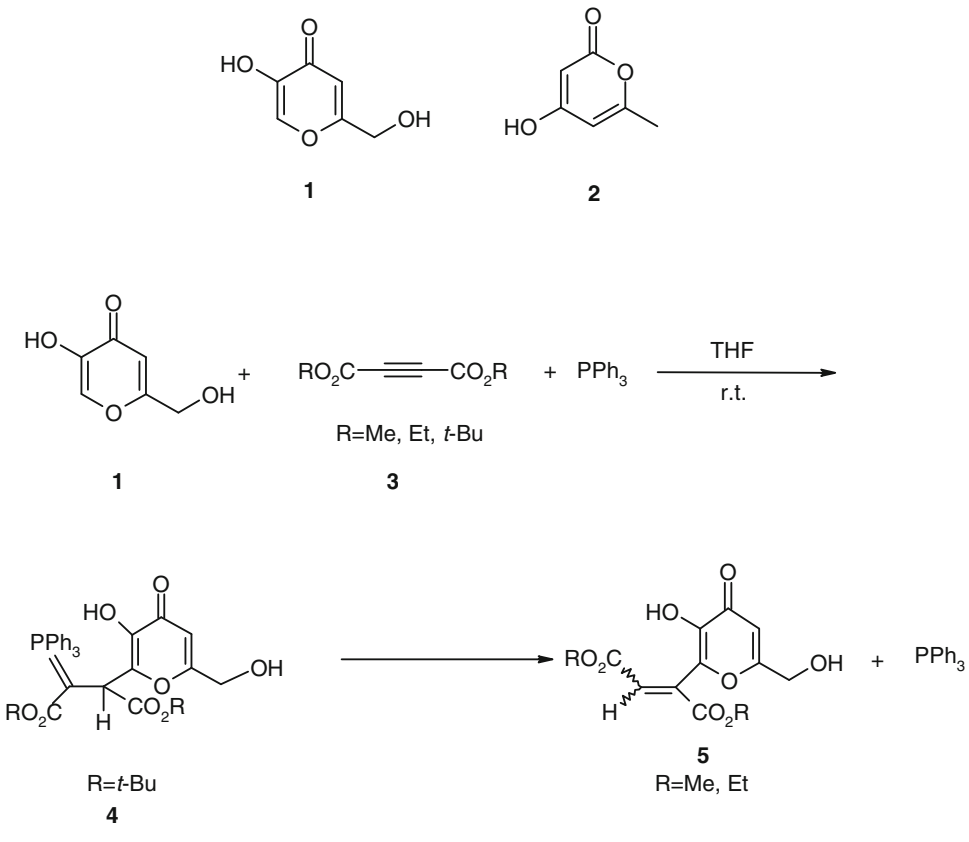
Results and discussion

In this work we studied three-component reactions of kojic acid with acetylenic esters **3** in the presence of *tert*-butyl isocyanide or triphenylphosphine that lead to vinylated derivatives of kojic acid in a regioselective manner, and at the end of the reaction *tert*-butyl isocyanide or triphenylphosphine were recovered. The reaction of kojic acid with dialkyl acetylenedicarboxylates **3** in the presence of triphenylphosphine was carried out in THF at room temperature. The yellow powder separated from the reaction mixture was identified as dialkyl 2-[3-hydroxy-6-(hydroxymethyl)-4-oxo-4*H*-pyran-2-yl]-2-butenedioates **5a**, **5b** for dimethyl and diethyl acetylenic esters, which are C-vinylated derivatives of kojic acid in a regioselective approach. However, this is not the case for di-*tert*-butyl acetylenedi carboxylate, which produced the phosphorus ylide di-*tert*-butyl 2-[3-hydroxy-6-(hydroxymethyl)-4-oxo-4*H*-pyran-2-yl]-3-(triphenylphosphanylidene)succinate (**4c**). When this ylide was heated under reflux in THF at 70 °C in order to obtain the C-vinylated derivative **5c**, the reaction reverted and the starting materials were reisolated (Scheme 1).

However, the reaction of kojic acid with alkyl propiolates **6** in the presence of *tert*-butyl isocyanide in THF at room temperature leads to the O-vinylated kojic acid derivatives **7** in a regioselective manner (Scheme 2).

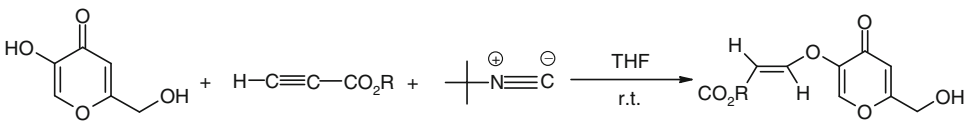
A possible mechanism of C-vinylation of kojic acid in the presence of triphenylphosphine is proposed in Scheme 3. On the basis of the well established chemistry of trivalent phosphorus nucleophiles [24–29], it is reasonable to assume

Scheme 1



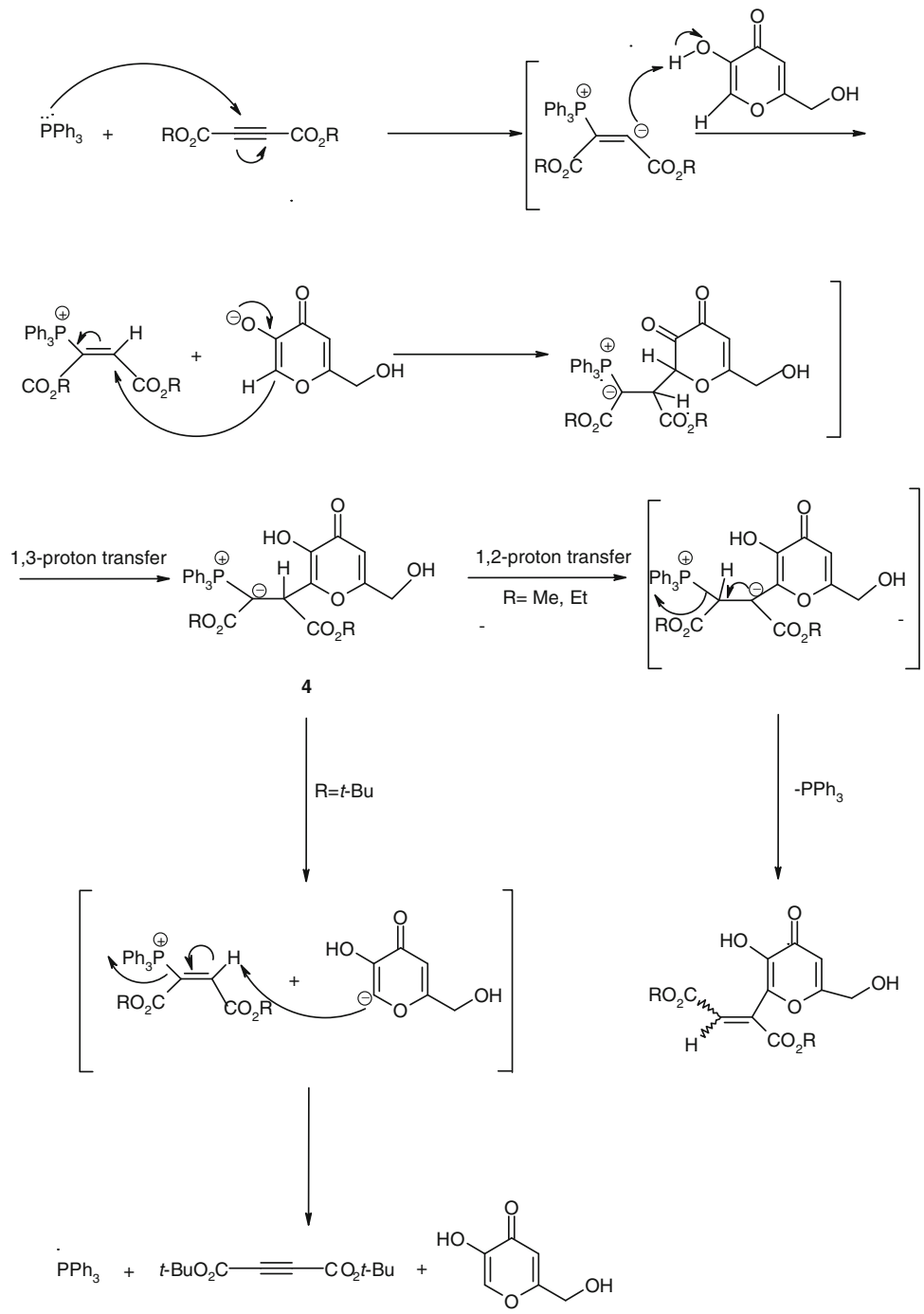
3,4,5	R	yield (%) 4	yield (%) 5
a	M	0	70
b	E	0	75
c	*t*-Bu	90	0

Scheme 2



6, 7	R	yield (%) 7
a	Me	80
b	Et	85

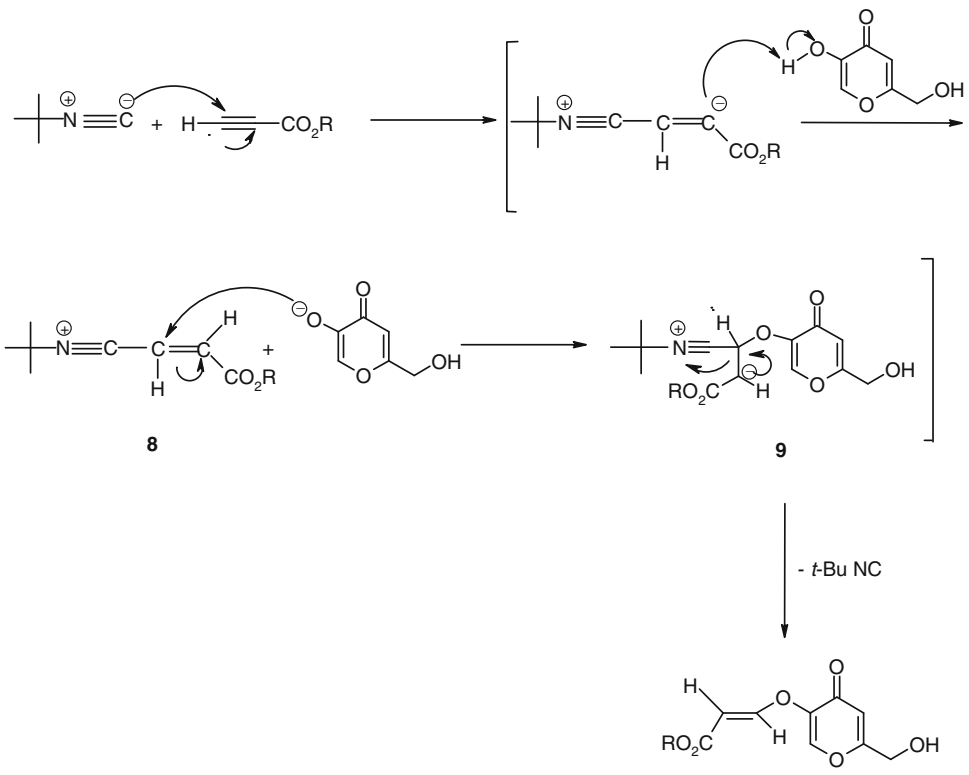
Scheme 3



that phosphorus ylide **4** results from initial addition of triphenylphosphine to the acetylenic ester and concomitant protonation of the 1:1 adduct by kojic acid. The positively charged ion is then attacked by the negative carbon atom (soft site) of kojic acid enolate to form phosphorane **4**. Dimethyl and diethyl phosphorane derivatives (**4a** and **4b**) spontaneously perform a 1,2-proton transfer and then undergo loss of triphenylphosphine to produce C-vinylated derivatives of kojic acid **5a** and **5b**, respectively. But di-*tert*-butyl phosphorane derivative **4c** was not converted to compound **5c**, but reverted to the starting materials under reflux conditions in THF. Although the exact mechanism for this conversion is unknown for us, on the basis of a similar reaction mechanism reported by Kalantari et al. [30], a possible mechanism is suggested (Scheme 3).

A proposed mechanism for formation of O-vinylated derivatives of kojic acid in the presence of *tert*-butyl isocyanide is shown in Scheme 4. On the basis of the well established chemistry of isocyanides [31–35], it is reasonable to assume that alkyl (*E*)-3-[[6-(hydroxymethyl)-4-oxo-

Scheme 4



4*H*-pyran-3-yl] oxy]-2-propenoates **7** result from nucleophilic addition of *tert*-butyl isocyanide to the acetylenic system and subsequent protonation of the 1:1 adduct by kojic acid. The positively charged ion **8** is then attacked by the negative oxygen atom (hard site) of kojic acid enolate and then loss of *tert*-butyl isocyanide produces O-vinylated kojic acid derivatives **7** as shown in Scheme 4.

In order to establish the catalytic activities of *tert*-butyl isocyanide and triphenylphosphine, the reactions were carried out in their absence. The results obtained indicated the reaction did not take place and the starting materials were recovered.

The structures of **4c**, **5a**, **5b**, **7a**, and **7b** were deduced from their ^{1}H, ^{13}C, and ^{31}P NMR, and IR spectra and from their mass spectrometric data. The ^{1}H NMR spectrum of **5a** contained two sharp signals for two methoxy groups ($\delta = 3.70$ and 3.74 ppm), and a singlet for OCH_2 ($\delta = 4.34$), a broad peak for the hydroxy group of CH_2OH ($\delta = 5.6$ ppm), two singlets for two vinyl protons ($\delta = 6.38$ and 6.68 ppm), and another broad peak at approximately 10.5 ppm for the hydroxy group of the 4*H*-pyrone ring. The ^{13}C NMR spectrum of **4a** contained twelve sharp lines in agreement with the proposed structure. The ^{1}H and ^{13}C NMR spectra of **5b** are similar to those of **5a**, except for the signals of the alkoxy groups. The mass spectrum of **5a** contained the molecular ion peak at $m/z = 284$ ($M^{+\cdot}$, 7). Initial fragmentations involved loss of the side chains of the kojic acid moiety.

The ^{1}H NMR spectrum of **4c** contained two sharp signals for two *tert*-butyl groups ($\delta = 0.93$ and 1.51 ppm), a doublet ($^{3}J_{PH} = 17.2$ Hz) at approximately $\delta = 3.68$ ppm for the methine group, an AB system ($^{2}J_{HH} = 14.8$ Hz) at approximately $\delta = 4.22$ and 4.30 ppm for diastereotopic protons of the OCH_2 group, a singlet at $\delta = 6.34$ ppm for a vinylic proton, two broad peaks at $\delta = 4.20$ and 8.30 ppm for two hydroxy groups, and a multiplet at approximately $\delta = 7.47$–7.90 ppm for aromatic protons of the triphenylphosphine moiety.

The ^{13}C NMR spectrum of **4c** contained a doublet ($^{1}J_{PC} = 132.8$ Hz) at $\delta = 40.65$ ppm for the P=C group and a doublet ($^{2}J_{PC} = 13.1$ Hz) at $\delta = 46.36$ ppm for the methine carbon of the $CHCO_2^tBu$ moiety. Other carbons of **3c** gave characteristic signals with appropriate chemical shifts. The ^{31}P NMR spectrum of **4c** contained a sharp singlet at approximately $\delta = 22.53$ ppm. The mass spectrum of **4c** contained the molecular ion peak at $m/z = 630$ ($M^{+\cdot}$, 2). Fragmentation involves loss of one of the side chains (PPh_3, C_4H_8, CO_2^tBu, CH_2OH, and OH).

Assignment of the structures of compounds **5a**, **5b**, and **4c** on the basis of the ^{1}H, ^{13}C NMR, and mass spectra was supported by measurement or their IR spectra. Of special interest are the strong carbonyl absorption bands at 1,744 and 1,717 cm^{-1} and a broad band for the OH group at 3,260 cm^{-1}.

The ^{1}H NMR spectrum of **7a** contained a broad signal for the OH group at $\delta = 2.40$ ppm, two singlets for

methoxy and methylene groups at $\delta = 3.77$ and 4.55 ppm, two singlets for two vinyl protons of the 4*H*-pyrone ring at $\delta = 6.63$ and 7.93 ppm, and two doublets for two vinyl protons at approximately $\delta = 5.54$ and 7.67 ppm ($^3J_{HH} = 12.2$ Hz) showing *E* geometry. The ^{13}C NMR spectrum of **7a** contained ten sharp lines in agreement with the proposed structure. The mass spectrum of **7a** contained the molecular ion peak at $m/z = 226$ ($M^{+\cdot}$, 65). Initial fragmentations involved loss of the side chains of the kojic acid system. The ^1H and ^{13}C NMR spectra of **7b** are similar to those of **7a** except for the alkoxy group, which gave characteristic signals with appropriate chemical shifts.

In summary, the reaction between the zwitterionic intermediate generated from acetylenic esters and nucleophiles such as triphenylphosphine and *tert*-butyl isocyanide with kojic acid leads to vinylated kojic acid via a one-pot, three component regioselective process. This procedure has the advantage that not only is the reaction performed under neutral conditions but also the substances can be mixed without any activation or modification.

Experimental

Dialkyl acetylenedicarboxylates, alkyl propiolates, triphenylphosphine, *tert*-butyl isocyanide, and kojic acid were obtained from Fluka (Buchs, Switzerland) and were used without further purification. Melting points were measured on an Electrothermal 9100 apparatus. ^1H, ^{13}C, and ^{31}P NMR spectra were measured with Bruker DRX-500, 400, and 300 Avance spectrometers. Mass spectra were recorded on a Finnigan-Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. Elemental analyses were performed using a Heraeus CHN–O Rapid analyzer.

*General procedure for the preparation of dialkyl (E)-2-[3-hydroxy-6-(hydroxymethyl)-4-oxo-4H-pyran-2-yl]-2-butenedioates (exemplified by **5a**)*

To a magnetically stirred solution of 0.284 g kojic acid (2 mmol) and 0.524 g triphenylphosphine (2 mmol) in 10 cm^3 THF, 2 mmol of dimethyl acetylenedicarboxylate in 4 cm^3 THF was added dropwise at −10 °C over 10 min. The mixture was stirred at room temperature for 24 h. The solvent was removed under reduced pressure and the residue was purified by recrystallization from ethyl acetate. The solvent was removed under reduced pressure, and the products **5a**, **5b**, and **4c** were obtained as yellow powder.

Dimethyl (E)-2-[3-hydroxy-6-(hydroxymethyl)-4-oxo-4H-pyran-2-yl]-2-butenedioate (**5a**, $C_{12}H_{12}O_8$)
Yellow powder; yield 70%; m.p.: 181–184 °C; $R_f = 0.2$ (*n*-hexane–ethyl acetate 20:80, *v*/*v*); IR (KBr):$\bar{\nu} = 3{,}250$ (OH), 1,750, 1,719, 1,698 (C=O) cm^{-1}; ^1H NMR (300.13 MHz, DMSO-d_6): $\delta = 3.70$, 3.74 (6H, 2s, $2OCH_3$), 4.34 (2H, s, OCH_2), 6.38, 6.68 (2H, 2s, 2CH), 5.6 (1H, bs, OH), 10.5 (1H, bs, OH) ppm; ^{13}C NMR (75.47 MHz, DMSO-d_6): $\delta = 52.1$, 52.6 ($2OCH_3$), 59.5 (OCH_2), 108.7, 118.6 (2CH), 138.8, 139.0, 145.4, 173.9 (4C), 163.0, 163.7 (C=O, ester), 172.3 (C=O, ketone) ppm; MS (70 eV): $m/z = 284$ ($M^{+\cdot}$, 7), 253 ($M^{+\cdot}$–OCH_3, 55), 225 ($M^{+\cdot}$–CO_2Me, 100), 165 ($M^{+\cdot}$–($2CO_2Me + H$), 7), 113 ($M^{+\cdot}$–($C_6H_5O + CH_2O$), 7), 93 ($M^{+\cdot}$–(CH_2OH, $MeO_2CCCCO_2Me + OH$) + 1, 30), 57 ($C_4H_7^+$, 18).

Diethyl (E)-2-[3-hydroxy-6-(hydroxymethyl)-4-oxo-4H-pyran-2-yl]-2-butenedioate (**5b**, $C_{14}H_{16}O_8$)
Yellow powder; yield 75%; m.p.: 124–126 °C; $R_f = 0.3$ (*n*-hexane–ethyl acetate 20:80, *v*/*v*); IR (KBr): $\bar{\nu} = 3{,}620$ (OH), 1,744, 1,716, 1,695 (C=O) cm^{-1}; ^1H NMR (300.13 MHz, DMSO-d_6): $\delta = 1.21$ (3H, t, $^3J_{HH} = 7.1$ Hz, CH_3), 1.24 (3H, t, $^3J_{HH} = 7.1$ Hz, CH_3), 4.14 (2H, q, $^3J_{HH} = 7.1$ Hz OCH_2), 4.23 (2H, $^3J_{HH} = 7$ Hz, OCH_2), 4.34 (2H, s, OCH_2), 5.5 (1H, bs, OH), 6.39, 6.67 (2H, 2 s, 2CH), 10.5 (1H, bs, OH) ppm; ^{13}C NMR (75.47 MHz, DMSO-d_6): $\delta = 13.7$, 14.0 ($2CH_3$), 59.6, 60.9, 61.5 (OCH_2), 108.7,119.0 (2CH), 139.1, 139.2, 145.5, 174.0 (4C), 164.1, 164.2 (2C=O, ester), 168.6 (C=O, ketone) ppm; MS (70 eV): $m/z = 312$ ($M^{+\cdot}$, 4), 267 ($M^{+\cdot}$–OEt, 53), 239 ($M^{+\cdot}$–CO_2Et, 100), 167 ($M^{+\cdot}$–($CO_2Et + CO_2 + C_2H_4$), 27), 142 ($M^{+\cdot}$–EtO_2CCCCO_2Et, 15), 93 ($M^{+\cdot}$–($CH_2OH + EtO_2CCCCO_2Et + OH$), 14).

Di-tert-butyl 2-[3-hydroxy-6-(hydroxymethyl)-4-oxo-4H-pyran-2-yl]-3-(1,1,1-triphenyl-λ^5-phosphanylidene)-succinate (**4c**, $C_{36}H_{39}O_8P$)
White powder; yield 90%; m.p.: 123–126 °C; $R_f = 0.2$ (*n*-hexane–ethyl acetate 20:80, *v*/*v*); IR (KBr): $\bar{\nu} = 3{,}481$ (OH), 1,734, 1,726, 1,680 (C=O) cm^{-1}; ^1H NMR (400.13 MHz, $CDCl_3$): $\delta = 0.93$, 1.51 (18H, 2s, $2CMe_3$), 3.68 (1H, d, $^3J_{PH} = 17.2$ Hz, CH), 4.20 (1H, bs, OH), 4.22, 4.30 (2H, AB system, $^3J_{HH} = 14.8$ Hz, OCH_2), 6.34 (1H, s, CH), 7.47–7.90 (15H, m, aromatic protons), 8.30 (1H, bs, OH) ppm; ^{13}C NMR (100.61 MHz, $CDCl_3$): $\delta = 28.1$, 28.3 ($2CMe_3$), 40.6 (d, $^1J_{PC} = 132.8$ Hz, P=C), 46.4 (d, $^2J_{PC} = 13.1$ Hz, CH), 61.3 (OCH_2), 78.2, 81.4 ($2OCMe_3$), 109.0 (CH), 128.9 (d, $^1J_{PC} = 91.6$ Hz, C_{ipso}), 128.7 (d, $^2J_{PC} = 12.1$ Hz, C_{ortho}), 132.2 (d, $^4J_{PC} = 2.0$ Hz, C_{para}), 133.7 (d, $^3J_{PC} = 9.9$ Hz, C_{meta}), 150.8, 165.3, 174.9 (3C), 170.3 (d, $^3J_{PC} = 9.8$ Hz, C=O), 170.9 (d, $^2J_{PC} = 13.7$ Hz, C=O) ppm; ^{31}P NMR (161.97 MHz, $CDCl_3$): $\delta = 22.53$ ppm; MS (70 eV): $m/z = 630$ ($M^{+\cdot}$, 2), 369 ($M^{+\cdot}$–

PPh_3 + 1, 18), 313 ($M^{+\cdot}$–(PPh_3 + C_4H_8) + 1, 14), 262 ($^+PPh_3$, 100), 211 ($M^{+\cdot}$–(PPh_3 + CO_2^tBu + C_4H_8), 44), 183 ($M^{+\cdot}$–(PPh_3 + $2CO_2^tBu$) + 1, 37), 108 ($M^{+\cdot}$–(PPh_3 + CH_2OH + $^tBuO_2C$–C=$CHCO_2^tBu$ + 2H), 18), 77 (Ph^+, 7), 57 ($C_4H_7^+$, 53).

*General procedure for preparation of methyl (E)-3-[[6-(hydroxymethyl)-4-oxo-4H-pyran-3-yl]oxy]-2-propenoate (exemplified by **7a**)*

To a stirred solution of 0.28 g kojic acid (2 mmol) and 0.17 cm^3 methyl acetylenecarboxylate (2 mmol) in 10 cm^3 THF was added 0.16 g *tert*-butyl isocyanide (2 mmol) dropwise at −10 °C over 10 min. The reaction mixture was then left to warm to room temperature and stand for 24 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 *n*-hexane–ethyl acetate 20:80 as eluent. The solvent was removed under reduced pressure, and products **7a** and **7b** were obtained as white powder.

Methyl (E)-3-[[6-(hydroxymethyl)-4-oxo-4H-pyran-3-yl]oxy]-2-propenoate (**7a**, $C_{10}H_{10}O_6$)
White powder; yield 80%; m.p.: 144–146 °C; R_f = 0.3 (*n*-hexane–ethyl acetate 20:80, *v*/*v*); IR (KBr): $\bar{\nu}$ = 3,395 (OH), 1,710, 1,670 (C=O) cm^{-1}; ^{1}H NMR (500.13 MHz, $CDCl_3$): δ = 2.40 (1H, bs, OH), 3.77 (3H, s, OCH_3), 4.55 (2H, s, OCH_2), 6.63, 7.93 (2H, 2s, 2CH), 5.54, 7.67 (2H, 2d, $^3J_{HH}$ = 12.2 Hz, 2CH) ppm; ^{13}C NMR (125.8 MHz, $CDCl_3$): δ = 51.9 (OCH_3), 60.8 (OCH_2), 102.1, 113.7, 146.3, 166.9 (4CH), 144.8, 159.2 (2C), 167.4 (C=O, ester), 172.8 (C=O, ketone) ppm; MS (70 eV): *m*/*z* = 226 ($M^{+\cdot}$, 65), 211 ($M^{+\cdot}$–CH_3, 7), 195 ($M^{+\cdot}$–CH_2OH, 53), 167 ($M^{+\cdot}$–CO_2Me, 100), 149 ($M^{+\cdot}$–(CO_2Me + OH + H), 87), 125 ($M^{+\cdot}$–MeO_2CCH=CHO, 22), 110 ($M^{+\cdot}$–(MeO_2CCH=CH + CH_2OH), 21), 95 ($M^{+\cdot}$–(CH_2O + MeO_2CCH=CHO), 81).

Ethyl (E)-3-[[6-(hydroxymethyl)-4-oxo-4H-pyran-3-yl]oxy]-2-propenoate (**7b**, $C_{11}H_{12}O_6$)
White powder; yield 85%; m.p.: 92–94 °C; R_f = 0.3 (*n*-hexane–ethyl acetate 20:80, *v*/*v*); IR (KBr): $\bar{\nu}$ = 3,489 (OH), 1,719, 1,674 (C=O) cm^{-1}; ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 1.27 (3H, t, $^3J_{HH}$ = 7.2 Hz, CH_3), 2.25 (1H, bs, OH), 4.17 (2H, q, $^3J_{HH}$ = 7.2 Hz, OCH_2), 6.62, 7.95 (2H, 2s, 2CH), 5.48, 7.61 (2H, 2d, $^3J_{HH}$ = 12.4 Hz, 2CH) ppm; ^{13}C NMR (100.61 MHz, $CDCl_3$): δ = 14.2 (CH_3), 60.8, 61.1 ($2OCH_2$), 102.2, 113.2, 145.9, 166.9 (4CH), 144.5, 159.2 (2C), 166.1 (C=O, ester), 173.4 (C=O, ketone) ppm; MS (70 eV): *m*/*z* = 240 ($M^{+\cdot}$, 52), 209 ($M^{+\cdot}$–CH_2OH, 18), 195 ($M^{+\cdot}$–OEt, 53), 167 ($M^{+\cdot}$–CO_2Et, 100), 149 ($M^{+\cdot}$–(CO_2Me + OH + H), 55), 125 ($M^{+\cdot}$–EtO_2CCH=CHO, 13), 110 ($M^{+\cdot}$–(EtO_2CCH=C!H + CH_2OH), 18).

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