

Rhodium(II)-Catalyzed Reaction of Salicylaldehyde and Its Derivatives with Diazocarbonyl Compounds

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Abstract—Reactions of salicylaldehyde and its cyclic acetals with diazocarbonyl compounds in the presence of copper and rhodium catalysts have been studied. The reaction direction and product yields are determined by the nature of the initial reactants and catalyst.

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Diazocarbonyl compounds are widely used in the synthesis of polyfunctionalized heterocycles [1–6]. We previously showed that benzaldehyde and its cyclic acetals reacted with ethyl diazoacetate in the presence of $\text{Rh}_2(\text{OAc})_4$ to give ethyl 3-oxo-3-phenylpropanoates and 1,4-dioxanes [7–9]. Under analogous conditions, reactions of phenols with diazo esters afforded the corresponding benzofurans [10].

In this work we studied reactions of salicylaldehyde and its derivatives with diazo carbonyl compounds in the presence of $\text{Cu}(\text{OTf})_2$ and $\text{Rh}_2(\text{OAc})_4$ as catalysts. The initial diazo esters were ethyl diazoacetate (**1a**), ethyl 2-diazo-3-oxobutanoate (**1b**), and diethyl 2-diazomalonate (**1c**), and the aldehyde components were salicylaldehyde (**2**), 2-(1,3-dioxolan-2-yl)phenol (**3**), and 2-(1,3-dioxan-2-yl)phenol (**4**).

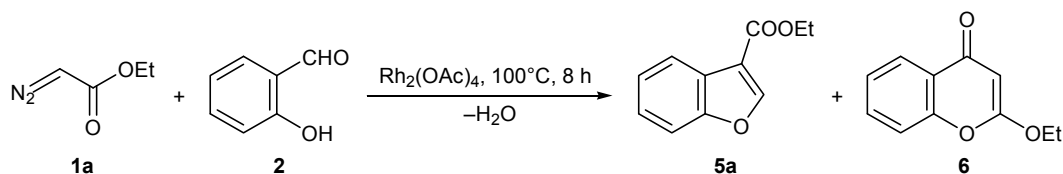
Ethyl diazoacetate (**1a**) reacted with salicylaldehyde (**2**) in the presence of $\text{Rh}_2(\text{OAc})_4$ under solvent-free conditions to give a mixture of ethyl 1-benzofuran-3-carboxylate (**5a**) and 2-ethoxy-4*H*-chromen-4-one (**6**) at a ratio of 2:1 in an overall yield of 55% (Scheme 1). The product composition did not change despite variation of the conditions, such as tempera-

ture, reaction time, and the use of a solvent, and in all cases compound **5a** was formed as the major product. Elevated temperature and prolonged reaction time favored formation of tars, while the overall yield decreased. Benzofuran **5a** was formed as the only product (yield 48%) when the reaction was catalyzed by $\text{Cu}(\text{OTf})_2$.

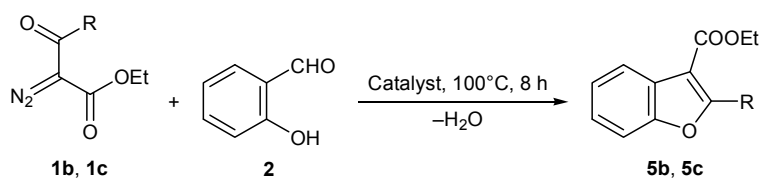
The reactions of ethyl 2-diazo-3-oxobutanoate (**1b**) and diethyl 2-diazomalonate (**1c**) with salicylaldehyde (**2**) in the presence of both $\text{Rh}_2(\text{OAc})_4$ and $\text{Cu}(\text{OTf})_2$ afforded benzofuran **5b** (yield 33%) or **5c** (43%), respectively, as the only product (Scheme 2).

Our results led us to propose a mechanism shown in Scheme 3. Presumably, in the first stage the catalysts coordinates to either the carbonyl group (path *a*) or hydroxy group (path *b*) of salicylaldehyde to form intermediates **A** and **D**, respectively. The reactions of **A** and **D** with ethyl diazoacetate are accompanied by evolution of nitrogen to give intermediates **B** and **E** or **H** (path *c* in the case of ethyl diazoacetate), and the subsequent intramolecular rearrangement and dehydration lead to the formation of benzofuran **5a** or chromene **6**. The proposed mechanism is well consistent with published data [11].

Scheme 1.

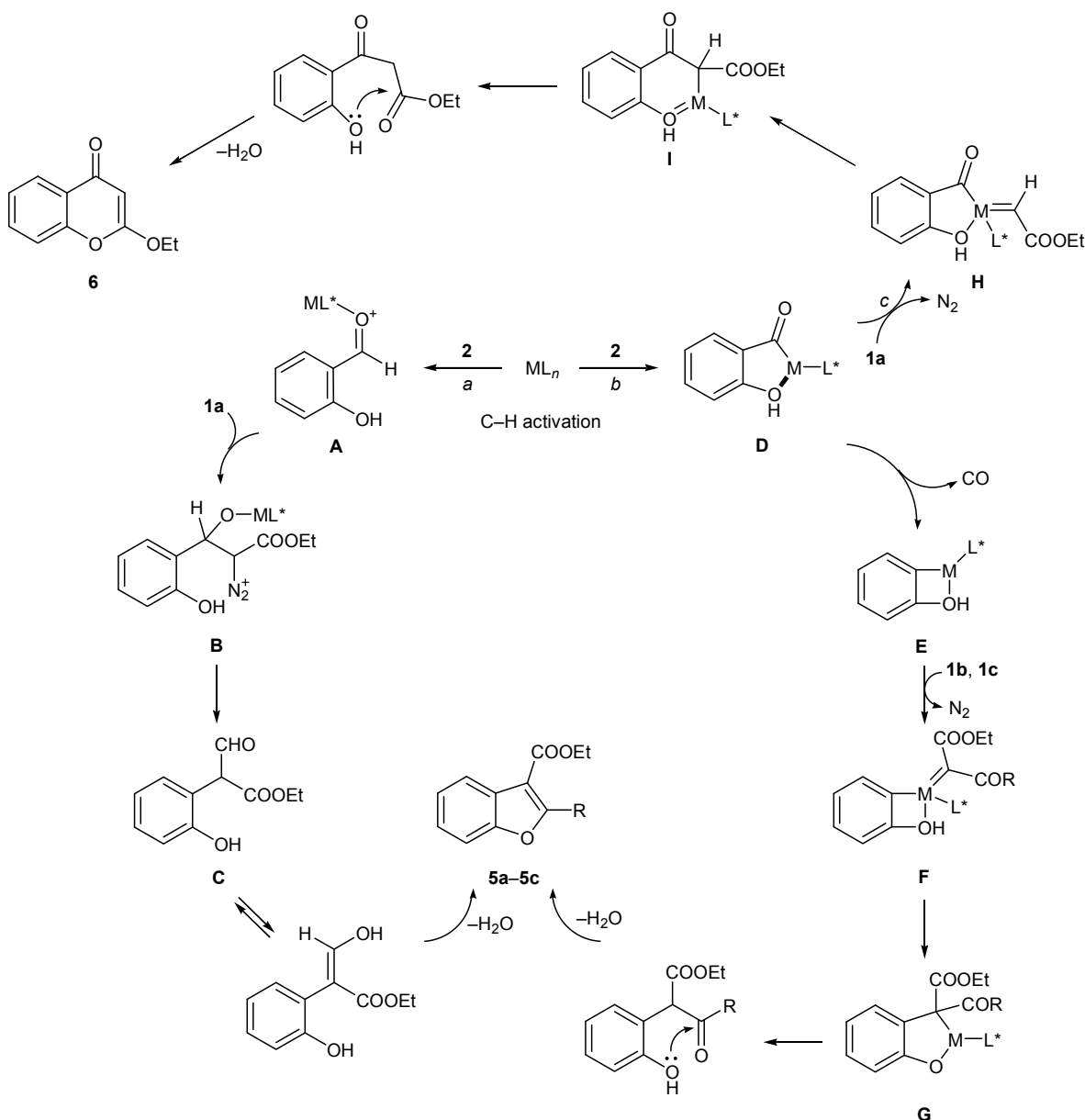


Scheme 2.



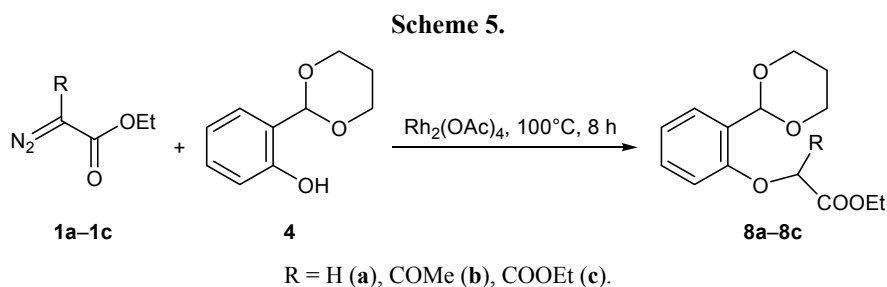
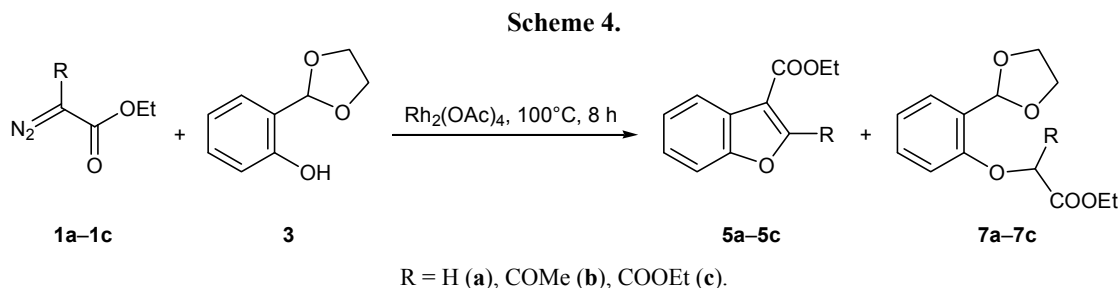
R = Me (b), OEt (c).

Scheme 3.



We also studied the reactions of diazo esters **1a–1c** with cyclic salicylaldehyde acetals **3** and **4**. Acetal **3** reacted with diazo esters **1a–1c** in the presence of $\text{Rh}_2(\text{OAc})_4$ in a non-selective manner to give mixtures of benzofurans **5a–5c** and compounds **7a–7c** resulting

from insertion of intermediate alkoxycarbonylcarbenes into the OH bond of phenol **3** (Scheme 4); the overall yields were 55–80%. Benzofurans **5a–5c** are formed as a result of deacetalization of **3** to salicylaldehyde and its subsequent reaction with the diazo compound.



Copper(II) trifluoromethanesulfonate turned out to be inefficient catalyst in the reactions of diazo esters **1a–1c** with 2-(1,3-dioxolan-2-yl)phenol (**3**). In this case, complete removal of the acetal protection occurred, and the products were salicylaldehyde **2** and benzofurans **5a–5c**.

The reactions of more stable 2-(1,3-dioxan-2-yl)phenol (**4**) [12] with diazo carbonyl compounds **1a–1c** in the presence of both $\text{Cu}(\text{OTf})_2$ and $\text{Rh}_2(\text{OAc})_4$ selectively involved the hydroxy group to give compounds **8a–8c** in moderate yields (Scheme 5).

Thus, salicylaldehyde reacts with diazo esters to afford mixtures of benzofuran and chromene derivatives. The reaction direction and product ratio depends on the nature of the initial diazo compounds.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance-500 spectrometer at 500 and 125 MHz, respectively, using CDCl_3 as solvent and tetramethylsilane as internal standard. The products were analyzed by gas chromatography on a Kristall-2000M chromatograph (Russia) equipped with a thermal conductivity detector; 2000 \times 5-mm column packed with 5% SE-30 on Chromaton N-AW; carrier gas helium; oven temperature programming from 50 to 250 $^\circ\text{C}$ at a rate of 20 deg/min; injector temperature 250 $^\circ\text{C}$, detector temperature 250 $^\circ\text{C}$.

Analytical TLC was performed on Silufol plates (Merck). The products were separated by column chromatography on silica gel (60–100 mesh, Lancaster).

Salicylaldehyde and the catalysts $\text{Rh}_2(\text{OAc})_4$ and $\text{Cu}(\text{OTf})_2$ were commercial products. The solvents used (toluene, hexane, ethyl acetate, *tert*-butyl methyl ether) were purified and dried according to standard procedures [13, 14]. Acetals **3** and **4** were synthesized as described in [15]. Diazo carbonyl compounds **1a–1c** were prepared according to [16].

Reactions of salicylaldehyde (2) and its acetals 3 and 4 with diazo carbonyl compounds 1a–1c (general procedure). A mixture of 20 mg (1.5 mmol) of salicylaldehyde (**2**), 2-(1,3-dioxolan-2-yl)phenol (**3**), or 2-(1,3-dioxan-2-yl)phenol (**4**) containing 1.2×10^{-5} mmol of $\text{Cu}(\text{OTf})_2$ or 3.4×10^{-4} mmol of $\text{Rh}_2(\text{OAc})_4$ was heated to 100 $^\circ\text{C}$, and 1.7 mmol of diazo carbonyl compound **1a–1c** was added over a period of 15 min with vigorous stirring. The mixture was stirred for 8 h on heating. When the reaction was complete (GLC), the mixture was cooled to room temperature, and the products were isolated by column chromatography on silica gel using solvents with increasing polarity.

Ethyl 1-benzofuran-3-carboxylate (5a) [5, 17]. Yield 0.10 g (37%), light yellow liquid, R_f 0.21 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.44 t (3H, CH_2CH_3 , $J = 7.1$ Hz), 4.44 q (2H, OCH_2 , $J = 7.1$ Hz), 7.30 t (1H, 6-H, $J = 7.3$ Hz), 7.44 t (1H, 5-H), 7.52 d (1H, 7-H), 7.60 d (1H, 4-H), 7.68 d (1H, 2-H). ^{13}C NMR spectrum, δ_c , ppm: 14.05 (CH_3), 61.50 (OCH_2), 118.48 (C^7), 118.96 (C^3), 120.26 (C^4), 133.01 (C^6), 133.64 (C^{3a}), 135.88 (C^5), 150.36 (C^2), 156.09 (C^{7a}), 165.21 ($\text{C}=\text{O}$). Mass spectrum: m/z (I_{rel} , %): 190 (5), 162 (60), 145 (100), 134 (10), 118 (32), 89 (38), 78 (5), 63 (19), 51 (3), 43 (4), 39 (6).

Ethyl 2-methyl-1-benzofuran-3-carboxylate (5b) [11]. Yield 0.08 g (33%), light yellow liquid, R_f 0.24 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.33 t (3H, CH_2CH_3 , $J = 7.1$ Hz), 2.38 s (3H, 2- CH_3), 4.44 q (2H, OCH_2 , $J = 7.1$ Hz), 7.59 t (1H, 6-H), 7.64 d (1H, 7-H), 7.80 t (1H, 5-H), 8.05 d (1H, 4-H). ^{13}C NMR spectrum, δ_c , ppm: 13.91 (CH_2CH_3), 13.99 (2- CH_3), 59.08 (OCH_2), 110.77 (C^3), 114.63 (C^7), 119.18 (C^4), 126.15 (C^5), 127.03 (C^6), 129.87 (C^{3a}), 152.78 (C^{7a}), 163.66 (C^2), 167.28 ($\text{C}=\text{O}$). Mass spectrum, m/z (I_{rel} , %): 204 (<1), 190 (90), 162 (98), 145 (100), 134 (14), 118 (50), 89 (76), 78 (10), 63 (28), 51 (3), 44 (16), 32(10).

Ethyl 2-ethoxy-1-benzofuran-3-carboxylate (5c). Yield 0.09 g (43%), light yellow liquid, R_f 0.27 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.26 t (3H, $\text{COOCH}_2\text{CH}_3$, $J = 7.1$ Hz), 1.28 t (3H, 2- OCH_2CH_3 , $J = 7.1$ Hz), 4.18 q (2H, COOCH_2 , $J = 7.1$ Hz), 4.25 q (2H, 2- OCH_2 , $J = 7.1$ Hz), 7.07 d (1H, 7-H), 7.38 t (1H, 6-H), 7.49 t (1H, 5-H), 7.58 d (1H, 4-H). ^{13}C NMR spectrum, δ_c , ppm: 13.94 (COOCH_2CH), 14.02 (2- OCH_2CH_3), 61.63 (COOCH_2), 63.62 (2- OCH_2), 111.83 (C^3), 118.51 (C^7), 118.94 (C^4), 125.17 (C^6), 125.65 (C^5H), 126.01 (C^{3a}), 157.29 (C^{7a}), 165.88 ($\text{C}=\text{O}$), 166.85 (C^2). Mass spectrum, m/z (I_{rel} , %): 234 (14), 207 (8), 190 (40), 179 (38), 162 (100), 145 (36), 133 (70), 120 (52), 105 (24), 89 (16), 77 (36), 63 (10), 51 (6), 44 (8), 32 (5). Found, %: C 66.64; H 5.90. $\text{C}_{13}\text{H}_{14}\text{O}_4$. Calculated, %: C 66.67; H 5.99.

2-Ethoxy-4H-chromen-4-one (6) [11]. Yield 0.06 g (17%), light yellow liquid, R_f 0.34 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.42 t (3H, CH_2CH_3 , $J = 7.1$ Hz), 4.44 q (2H, OCH_2 , $J = 7.1$ Hz), 6.92 s (1H, 3-H), 7.64 d (1H, 8-H), 7.26 t (1H, 7-H), 7.83 t (1H, 6-H), 9.68 d (1H, 5-H). ^{13}C NMR spectrum, δ_c , ppm: 14.10 (CH_3), 61.62 (OCH_2), 115.39 (C^3), 117.19 (C^8), 123.88 (C^{4a}), 126.67 (C^6), 127.84 (C^5), 135.89 (C^7), 156.09 (C^{8a}), 165.21 ($\text{C}=\text{O}$), 173.36 (C^4). Mass spectrum, m/z (I_{rel} , %): 190 (90), 168 (3), 139 (8), 121 (100), 107 (3), 93 (31), 77 (6), 65 (30), 53 (4), 44 (8), 39 (14).

Ethyl [2-(1,3-dioxolan-2-yl)phenoxy]acetate (7a). Yield 0.21 g (54%), light yellow liquid, R_f 0.42 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.28 t (3H, CH_2CH_3 , $J = 7.2$ Hz), 4.18–4.32 m (4H, 4'-H, 5'-H), 4.37 q (2H, OCH_2CH_3 , $J = 7.1$ Hz), 5.31 s (2H, OCH_2CO), 5.84 s (1H, 2'-H), 6.88 d (1H, H_{arom}), 7.02 t (1H, H_{arom}), 7.39 t (1H, H_{arom}), 7.47 d (1H, H_{arom}). ^{13}C NMR spectrum, δ_c , ppm: 14.07

(CH_2CH_3), 61.00 (OCH_2CH_3), 62.76 (C^4 , C^5), 66.73 (OCH_2CO), 110.79 (C^2); 122.60, 125.89, 125.97, 129.90, 135.91, 152.12 (C_{arom}); 167.02 ($\text{C}=\text{O}$). Mass spectrum, m/z (I_{rel} , %): 252 (<1), 250 (4), 235 (18), 190 (66), 162 (100), 145 (70), 133 (22), 121 (15), 118 (30), 105 (14), 89 (20), 77 (25), 63 (12), 51 (12), 43 (76). Found, %: C 61.87; H 6.32. $\text{C}_{13}\text{H}_{16}\text{O}_5$. Calculated, %: C 61.90; H 6.35.

Ethyl 2-[2-(1,3-dioxolan-2-yl)phenoxy]-3-oxobutanoate (7b). Yield 0.08 g (23%), light yellow liquid, R_f 0.34 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.26 t (3H, CH_2CH_3 , $J = 7.3$ Hz), 2.32 s (3H, COCH_3), 3.48–3.52 m (4H, 4'-H, 5'-H), 4.24 q (2H, OCH_2 , $J = 7.2$ Hz), 4.58 s (1H, OCHCO), 5.53 s (1H, 2'-H), 6.72 t (1H, H_{arom}), 6.84 d (1H, H_{arom}), 7.15 t (1H, H_{arom}), 7.84 d (1H, H_{arom}). ^{13}C NMR spectrum, δ_c , ppm: 14.04 (CH_2CH_3), 27.79 (COCH_3), 62.73 (OCH_2), 67.01 (C^4 , C^5), 83.98 (OCHCO), 97.14 (C^2); 110.68, 125.99, 126.22, 126.43, 126.81, 158.53 (C_{arom}); 165.36 ($\text{CO}_2\text{C}_2\text{H}_5$), 200.09 (COCH_3). Mass spectrum, m/z (I_{rel} , %): 294 (<1), 281 (4), 233 (2), 221 (3), 207 (14), 190 (7), 164 (100), 145 (17), 133 (20), 121 (38), 105 (8), 91 (14), 87 (22), 77 (25), 73 (32), 65 (12), 44 (30), 36 (18), 32 (19). Found, %: C 61.18; H 6.09. $\text{C}_{15}\text{H}_{18}\text{O}_6$. Calculated, %: C 61.22; H 6.12.

Diethyl [2-(1,3-dioxolan-2-yl)phenoxy]propane-dioate (7c). Yield 0.08 g (26%), light yellow liquid, R_f 0.31 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.27 t (6H, CH_2CH_3 , $J = 7.2$ Hz), 3.95–4.02 m (4H, 4'-H, 5'-H), 4.26 q (4H, OCH_2 , $J = 7.2$ Hz), 5.27 s (1H, COCHCO), 5.88 s (1H, 2'-H), 6.89 d (1H, H_{arom}), 6.96 t (1H, H_{arom}), 7.34 t (1H, H_{arom}), 7.88 d (1H, H_{arom}). ^{13}C NMR spectrum, δ_c , ppm: 13.95 (CH_2CH_3), 62.24 (OCH_2), 67.05 (C^4 , C^5), 79.09 (COCHCO), 98.95 (C^2); 113.71, 125.74, 125.98, 128.60, 130.03, 159.17 (C_{arom}), 164.27 ($\text{C}=\text{O}$). Mass spectrum, m/z (I_{rel} , %): 324 (<1), 218 (3), 235 (9), 205 (55), 191 (7), 177 (64), 163 (10), 149 (8), 133 (100), 121 (36), 105 (16), 97 (8), 89 (16), 77 (20), 65 (12), 51 (5), 44 (16), 32 (18). Found, %: C 59.21; H 6.15. $\text{C}_{16}\text{H}_{20}\text{O}_7$. Calculated, %: C 59.26; H 6.17.

Ethyl [2-(1,3-dioxan-2-yl)phenoxy]acetate (8a). Yield 0.20 g (49%), light yellow liquid, R_f 0.34 (petroleum ether–ethyl acetate, 8:2). ^1H NMR spectrum, δ , ppm: 1.31 t (3H, CH_2CH_3 , $J = 7.1$ Hz), 2.20–2.26 m (2H, 5'-H), 4.04 t (2H, 4'-H, 6'-H), 4.25 q (2H, OCH_2CH_3), 4.63 s (2H, OCH_2CO), 5.64 s (1H, 2'-H), 6.76 d (1H, H_{arom}), 7.04 t (1H, H_{arom}), 7.26 t (1H, H_{arom}), 7.64 d (1H, H_{arom}). ^{13}C NMR spectrum, δ_c ,

ppm: 14.17 (CH₂CH₃), 25.86 (C^{5'}), 61.29 (OCH₂CH₃), 66.21 (OCH₂CO), 67.60 (C^{4'}, C^{6'}), 96.83 (C^{2'}); 112.25, 127.35, 127.91, 128.34, 129.94, 154.84 (C_{arom}); 168.85 (C=O). Mass spectrum, *m/z* (*I*_{rel}, %): 266 (6), 265 (20), 207 (50), 178 (92), 163 (26), 151 (28), 135 (52), 121 (100), 107 (28), 87 (38), 77 (44), 65 (12), 59 (11), 44 (18). Found, %: C 63.14; H 6.73. C₁₄H₁₈O₅. Calculated, %: C 63.16; H 6.76.

Ethyl 2-[2-(1,3-dioxan-2-yl)phenoxy]-3-oxobutanoate (8b). Yield 0.09 g (25%), light yellow liquid, *R*_f 0.37 (petroleum ether–ethyl acetate, 8:2). ¹H NMR spectrum, δ, ppm: 1.26 t (3H, CH₂CH₃, *J* = 7.1 Hz), 1.88–1.92 m (2H, 5'-H), 2.32 s (3H, COCH₃, *J* = 7.1 Hz), 3.78 t (4H, 4'-H, 6'-H), 4.25 q (2H, OCH₂CH₃), 5.06 s (1H, OCHCO), 5.80 s (1H, 2'-H), 6.71 d (1H, H_{arom}), 7.05 t (1H, H_{arom}), 7.17 t (1H, H_{arom}), 7.41 d (1H, H_{arom}). ¹³C NMR spectrum, δ_C, ppm: 14.05 (CH₂CH₃), 25.76 (C^{5'}), 27.62 (COCH₃), 61.55 (C^{4'}, C^{6'}), 62.40 (OCH₂), 83.34 (OCHCO), 97.03 (C^{2'}); 112.81, 122.56, 125.94, 130.13, 154.02, 158.50 (C_{arom}); 166.03 (CO₂C₂H₅), 202.78 (COCH₃). Mass spectrum, *m/z* (*I*_{rel}, %): 308 (<1), 281 (12), 207 (44), 193 (18), 178 (100), 163 (34), 145 (10), 135 (36), 121 (56), 105 (14), 87 (20), 77 (30), 73 (16), 65 (14), 57 (6), 43 (25), 32 (12). Found, %: C 62.29; H 6.46. C₁₆H₂₀O₆. Calculated, %: C 62.33; H 6.50.

Diethyl [2-(1,3-dioxan-2-yl)phenoxy]propanedioate (8c). Yield 0.14 g (41%), light yellow liquid, *R*_f 0.41 (petroleum ether–ethyl acetate, 8:2). ¹H NMR spectrum, δ, ppm: 1.18 t (6H, CH₂CH₃, *J* = 7.1 Hz), 1.98–2.07 m (2H, 5'-H), 4.08 q (4H, 4'-H, 6'-H, *J* = 7.1 Hz), 4.16 q (4H, OCH₂CH₃, *J* = 7.1 Hz), 5.07 s (1H, COCHCO), 5.91 s (1H, 2'-H), 6.77 d (1H, H_{arom}), 6.98 t (1H, H_{arom}), 7.14 t (1H, H_{arom}), 7.55 d (1H, H_{arom}). ¹³C NMR spectrum, δ_C, ppm: 13.85 and 13.91 (CH₂CH₃), 25.59 (C^{5'}), 62.21 (OCH₂CH₃), 67.40 (C^{4'}, C^{6'}), 78.75 (COCHCO), 96.76 (C^{2'}); 114.88, 123.22, 127.45, 129.20, 129.90, 154.49 (C_{arom}); 165.63 (C=O). Mass spectrum, *m/z* (*I*_{rel}, %): 338 (1), 337 (3.7), 293 (1.5), 279 (1.4), 265 (<1), 251 (1.6), 223 (2.3), 207 (3.5), 191 (<1), 178 (100), 163 (8), 149 (4.3), 133 (8), 121 (46), 105 (8), 93 (5.7), 87 (14), 77 (12), 65 (6), 59 (3.6), 51 (1.6), 41 (2.5). Found, %: C 60.30; H 6.49. C₁₇H₂₂O₇. Calculated, %: C 60.35; H 6.51.

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