was filtered of and the reaction mixture was concentrated under reduced pressure. If deacylation does not occur *in situ,* the following procedure was performed: the residue was dissolved in 30 mL of the corresponding alcohol (EtOH/MeOH) and stirred from 2 h to overnight (temperature varies from room temperature to reflux depending on the substrate). After completion of the reaction the solvent was removed under reduced pressure and the residue was purified by column chromatography.

3-Nitrodihydrofuran-2(3H)-one (***3***): General nitration procedure was used (0.6 mL (5.58 mmol) of α-acetylbutyrolactone (**1**) in CH_2Cl_2 (15 mL)). Quenching and deacylation procedure: for 11 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used. The reaction mixture was filtered, treated with MeOH (2 mL) and concentrated under reduced pressure (30 °C). The residue was purified by column chromatography (*n*-hexane/ethyl acetate 25:1 → 9:1 *v/v*) to give compound **3** (405 mg, 78%) as a orange oil. R_f = 0.16 (*n*-hexane/ethyl acetate = 3:2 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$): 2.82 (dddd, J = 14.0, 9.0, 7.4, 4.8 Hz, 1H), 2.96 (dddd, J = 14.4, 8.5, 7.3, 7.3 Hz, 1H), 4.38 (ddd, J = 9.2, 7.3, 7.3 Hz, 1H), 4.56 (ddd, J = 8.8, 4.9, 4.9 Hz, 1H), 5.38 (dd, J = 9.0, 7.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 27.9, 66.9, 82.2, 167.2; HRMS (ESI): $[M+Na]^+$ calcd for $C_4H_5NO_4Na$: 154.0116, found 154.0106, $[M+K]^+$ calcd for $C_4H_5NO_4K$: 169.9856 found 169.9845.

Dimethyl 2-nitrosuccinate (***5***): General nitration procedure was used (0.82 mL (5.05 mmol) of dimethyl 2-acetylsuccinate (**4**) in CH_2Cl_2 (13.2 mL)). Quenching and deacylation procedure: for 11 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used, the reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 30 mL MeOH and stirred overnight at room temperature. The solvent was removed under reduced pressure and the residue was purified by column chromatography (*n*-hexane → *n*-hexane/ethyl acetate, 100:0 → 9:1 *v/v*) to give compound **5** (569 mg, 75%) as a yellow oil. R_f = 0.49 (*n*-hexane/ethyl acetate = 5:1 *v/v*); ^{1}H spectrum matches with the literature data.[30] ^{1}H NMR (400 MHz, $CDCl_3$): 3.16 (dd, *J* = 17.7, 4.9 Hz, 1H); 3.38 (dd, *J* = 17.7, 9.2 Hz, 1H), 3.75 (s, 3H), 3.87 (s, 3H), 5.57 (dd, *J* = 9.2, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 34.4, 52.8, 54.1, 83.1, 164.2, 168.9. HRMS (ESI): $[M+Na]^+$ calcd for $C_6H_9NO_6Na$: 214.0328, found 214.0322.

Diethyl 2-nitropentanedioate (***7***): General nitration procedure was used (1.0 mL (4.65 mmol) of diethyl 2-acetylpentanedioate (**6**) in CH_2Cl_2 (11.9 mL)). Quenching and deacylation procedure: for 10 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used, the reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 30 mL EtOH and stirred

overnight at room temperature. The solvent was removed under reduced pressure and the residue was purified by column chromatography (*n*-hexane/ethyl acetate 9:1 → 2:1 *v/v*) to give compound **7** (630 mg, 75%) as a colorless oil. R_f = 0.49 (*n*-hexane/ethyl acetate = 3:1 *v/v*); ^{1}H spectrum matches with the literature data.[31] ^{1}H NMR (400 MHz, $CDCl_3$): 1.26 (dd, J = 7.1, 7.1 Hz, 3H), 1.30 (dd, J = 7.1, 7.1 Hz, 3H), 2.39–2.61 (m, 4H), 4.15 (ddd, J = 7.1, 7.1, 7.1 Hz, 2H), 4.29 (ddd, J = 7.1, 7.1, 7.1 Hz, 2H), 5.27–5.32 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 14.3, 25.4, 29.7, 61.1, 63.3, 86.8, 164.3, 171.6. HRMS (ESI): $[M+Na]^+$ calcd for $C_9H_{15}NO_6Na$: 256.0797, found 256.0809.

Ethyl 2-nitropropionate (***9***): General nitration procedure was used (ethyl 2-methylacetoacetate (**8**) purity 95%, 0.75 mL (5.30 mmol) of substrate **8** in CH_2Cl_2 (13.25 mL)). Quenching and deacylation procedure: for 10 mL of the collected solution 10 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used, the reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 30 mL EtOH and stirred for 3 hours at 80 °C (temperature of the oil bath). The solvent was removed under reduced pressure and the residue was purified by column chromatography (*n*-hexane/ethyl acetate 15:1 → 10:1 *v/v*) to give compound **9** (443 mg, 59%) as a yellow oil. R_f = 0.43 (*n*-hexane/ethyl acetate = 5:1 *v/v*). Obtained NMR matches with the literature data.[32] ^{1}H NMR (400 MHz, $CDCl_3$): 1.29 (t, J = 7.1 Hz, 3H), 1.77 (d, J = 7.2 Hz, 3H), 4.26 (q, J = 7.2 Hz, 2H), 5.19 (q, J = 7.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 15.8, 63.1, 83.3, 165.2.

Methyl 2-nitrobutanoate (***11***): General nitration procedure was used (0.72 mL (5.04 mmol) of methyl 2-ethylacetoacetate (**10**) in CH_2Cl_2 (13.28 mL)). Quenching and deacylation procedure: for 11 mL of the collected solution 10 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used, the reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 30 mL MeOH and stirred for 130 minutes at 65 °C (temperature of the oil bath). The solvent was removed under reduced pressure and the residue was purified by column chromatography (*n*-hexane/ethyl acetate 10:1 → 7:1 *v/v*) to give compound **11** (310 mg, 53%) as a yellow oil. R_f = 0.45 (*n*-hexane/ethyl acetate = 5:1 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$): 1.04 (dd, J= 7.4, 7.4 Hz, 3H), 2.14-2.36 (m, 2H), 3.83 (s, 3H), 5.05 (dd, J= 9.3, 5.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 10.3, 24.1, 53.6, 89.4, 165.1; HRMS (ESI): $[M+Na]^+$ calcd for $C_5H_9NO_4Na$: 170.0429, found 170.0419, $[M+K]^+$ calculated for $C_5H_9NO_4K$: 186.0169, found 186.0159. Compound **11** was previously synthesized.[9b]

7-Ethoxy-6-nitro-7-oxoheptanoic acid (***13***) and *7-ethoxy-6-(hydroxyimino)-7-oxoheptanoic acid* (***13a***): General nitration procedure was used (ethyl 2-oxocyclohexanecarboxylate (**12**) purity 95%, 0.85 mL

(5.31 mmol) of substrate **12** in CH_2Cl_2 (13.15 mL)). Quenching and deacylation procedure: for 4 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used, the reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 30 ml mixture of EtOH and water (2:1, *v/v*) and stirred overnight at 45 °C (temperature of the oil bath). The solvent was removed under reduced pressure and the residue was purified by column chromatography (DCM/MeOH 50:1 → 5:2 *v/v*) to give compound **13** (275 mg, 82%) as a yellow-green solid and compound **13a** (33.4 mg, 11%) as a yellow solid. Analytical data for compound **13**: R_f = 0.30 (DCM/MeOH = 30:1 *v/v*); mp 57–59 °C; ^{1}H NMR (400 MHz, $CDCl_3$):1.29 (t, *J* = 7.1 Hz, 3H), 1.40-1.49 (m, 2H) 1.66-1.74 (m, 2H), 2.10-2.19 (m, 1H), 2.24-2.32 (m, 1H) , 2.36-2.39 (m, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 5.09 (dd, *J* = 9.3, 5.4 Hz, 1H), 9-12 (br signal COOH); ^{13}C NMR (100 MHz, $CDCl_3$) 13.9, 23.8, 25.0, 29.9, 33.5, 63.2, 87.9, 164.5, 179.5; HRMS (ESI): $[M+Na]^+$ calcd for $C_9H_{15}NO_6Na$ 256.0797, found 256.0796. Analytical data for compound **13a**: R_f = 0.5 (DCM/MeOH = 15:1 *v/v*); mp 98–100 °C; ^{1}H NMR (400 MHz, $CDCl_3$):1.33 (t, *J* = 7.1 Hz, 3H), 1.62-1.71 (m, 4H) 2.40 (t, *J* = 7.1 Hz, 2H), 2.66 (t, *J* = 7.2 Hz, 2H), 4.28 (q, *J* = 7.1 Hz, 2H), COOH and N-OH protons are not observed; ^{13}C NMR (100 MHz, $CDCl_3$) 14.1, 24.5, 24.6, 25.3, , 33.7, 62.0, 152.0, 163.2, 179.1; HRMS (ESI): $[M+Na]^+$ calcd for $C_9H_{15}NO_5Na$: 240.0848, found 240.0859.

Ethyl 2-nitrohexanoate (***15***): Nitric acid (90+%, 16 µL/min, 0.343 mmol/min) was mixed with sulfuric acid (96%, 103 µL/min, 1.85 mmol/min) at 10 °C using a T-mixer. The resulting flow stream was passed through a 0.03 mL PTFE-tubing and mixed with the ethyl 2-acetylhexanoate (**14**) solution (1.0 mL (5.11 mmol) of substrate **14** in CH_2Cl_2 (15 mL), 0.663 mL/min, 0.212 mmol/min) using second T-mixer at the same temperature. The biphasic mixture was passed through 0.7 mL PTFE-tubing and collected in an Erlenmeyer flask containing stirred suspension of $MgSO_4$ in CH_2Cl_2 (for 11 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used) at room temperature. The reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 30 mL EtOH and stirred overnight at room temperature. The solvent was removed under reduced pressure and the residue was purified by column chromatography (*n*-hexane → *n*-hexane/ethyl acetate, 100:0 → 9:1 *v/v*) to give compound **15** (480 mg, 72%) as a colorless oil. R_f = 0.49 (*n*-hexane/ethyl acetate = 2:1 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$): 0.90 (dd, J = 6.9, 6.9 Hz, 3H); 1.27 (dd, J = 7.1, 7.1 Hz, 3H), 1.32–1.42 (m, 4H), 2.05–2.15 (m, 1H), 2.18–2.31 (m, 1H), 4.25 (ddd, J = 7.1, 7.1, 7.1 Hz, 2H), 5.07 (dd, J = 9.4, 5.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 13.8, 14.0, 22.1, 27.8, 30.1, 63.1, 88.3, 164.8;

HRMS (ESI): $[M+Na]^+$ calcd for $C_8H_{15}NO_4Na$: 212.0899, found 212.0887, $[M+K]^+$ calculated for $C_8H_{15}NO_4K$: 228.0638, found 228.0630. Compound **15** was previously synthesized.[15,33]

Ethyl 2-chloro-2-nitroacetate (***17***): Nitric acid (90+%, 8 µL/min, 0.171 mmol/min) was mixed with sulfuric acid (96%, 52 µL/min, 0.923 mmol/min) at 20 °C using a T-mixer. The resulting acid mixture was passed through a 0.03 mL PTFE-tubing and mixed with the ethyl 2-chloro-3-oxobutanoate (**16**) solution (substrate **16** purity 95%, 0.23 mL (1.6 mmol) substrate **16** in CH_2Cl_2 (7.77 mL), 0.332 mL/min, 0.066 mmol/min) at the same temperature. The biphasic mixture was passed through 0.7 mL PTFE-tubing and collected in an Erlenmeyer flask containing stirred suspension of $MgSO_4$ in CH_2Cl_2 (for 4 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used) at room temperature. The reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 10 ml EtOH and stirred overnight at room temperature. The solvent was gently removed under reduced pressure (room temperature) to give volatile compound **17** as a yellow oil (84%, determined by ^{1}H NMR using mesitylene as internal standard). An analytically pure sample was obtained by column chromatography (*n*-hexane/ethyl acetate 20:1 → 10:1 *v/v*). R_f = 0.60 (*n*-hexane/ethyl acetate = 2:1 *v/v*); Obtained ^{1}H NMR matches with the literature data.[34] ^{1}H NMR (400 MHz, $CDCl_3$): 1.36 (t, *J* = 7.2 Hz, 3H), 4.39 (q, *J* = 6.7 Hz, 2H), 6.23 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 13.9, 64.9, 85.6, 160.7.

Ethyl 2-nitro-3-(4-nitrophenyl)propanoate (***19***), *ethyl 2-nitro-3-(2-nitrophenyl)propanoate (**20**), and ethyl 2-(4-nitrobenzyl)-3-oxobutanoate* (***20a***): Nitric acid (90+%, 16 µL/min, 0.343 mmol/min) was mixed with sulfuric acid (96%, 103 µL/min, 1.852 mmol/min) at 10 °C using a T-mixer. The resulting acid mixture was passed through a 0.03 mL PTFE-tubing and mixed with the ethyl 2-benzylacetoacetate (**18**) solution (0.48 mL (2.26 mmol) of substrate **18** in CH_2Cl_2 (13.5 mL), 0.663 mL/min, 0.106 mmol/min) at the same temperature. The biphasic mixture was passed through 0.7 mL PTFE-tubing and collected in an Erlenmeyer flask containing stirred suspension of $MgSO_4$ in CH_2Cl_2 (for 11 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used) at room temperature. The reaction mixture was filtered and concentrated under reduced pressure. The residue was dissolved in 30 mL EtOH and stirred overnight at 45 °C. The solvent was removed under reduced pressure to give compound **19** (61%), compound **20** (16 %) and compound **20a** (7%). (Due to the tedious purification procedure, the yields were determined by ^{1}H NMR using mesitylene as an internal standard). Analytically pure samples of compound **20** as a yellow oil were obtained using purification by column chromatography

(*n*-hexane/DCM 2:1 → 1:5 *v/v*). Compound **20a** was isolated as a yellow oil from the same column, however was unable to be separated from the impurities according to ^{1}H NMR. An analytically pure sample of compound **19** was obtained by purification using HPLC Hex/*i*PrOH (99:1, *v/v*). Analytical data for compound **19**: R_f = 0.33 (*n*-hexane/DCM 1:4 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$):1.30 (t, *J* = 7.1 Hz, 3H), 3.59 (dd, *J* = 14.8, 5.3 Hz, 1H), 3.68 (dd, *J* = 14.8, 9.5 Hz, 1H), 4.31 (q, *J* = 6.3 Hz, 2H), 5.36 (dd, *J* = 9.4, 5.7 Hz, 1H), 7.42 (d, *J* = 8.3 Hz, 2H), 8.20 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 35.9, 63.8, 88.4, 124.4, 130.1, 141.6, 147.8 163.5; HRMS (ESI): $[M+Na]^+$ calcd for $C_{11}H_{12}N_2O_6Na$: 291.0593, found 291.0585. Analytical data for compound **20**: R_f = 0.63 (*n*-hexane/DCM 1:4 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$): 1.29 (t, *J* = 6.9 Hz, 3H), 3.71 (dd, *J* = 14.3, 9.8 Hz, 1H), 3.90 (dd, *J* = 14.4, 4.9 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 5.66 (dd, *J* = 9.7, 5.0 Hz, 1H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.50 (dd, *J* = 7.8, 7.8 Hz, 1H), 7.59 (dd, *J* = 7.5, 7.5 Hz, 1H), 8.11 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 34.4, 63.5, 88.1, 125.9, 129.6, 129.7, 133.5, 134.2, 148.9, 163.8; HRMS-ESI: $[M+Na]^+$ calcd for $C_{11}H_{12}N_2O_6Na$ 291.0593 found 291.0591. Analytical data for compound **20a**: Obtained NMR matches with the literature data.[35] R_f = 0.20 (*n*-hexane/DCM 1:4 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$): 1.22 (t, *J* = 7.2 Hz, 3H), 2.24 (s, 3H), 3.25 (t, *J* = 7.6 Hz, 2H), 3.78 (t, *J* = 8.0, 1H), 4.12 – 4.21 (m, 2H), 7.36 (d, *J* = 8.8 Hz, 2H), 8.14 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 14.2, 29.7, 33.6, 60.8, 62.0, 123.9, 129.9, 146.2, 147.0, 168.6, 201.3.

Ethyl 2-nitroacetate (**22**): Nitric acid (65%, 31 µL/min, 0.657 mmol/min) was mixed with sulfuric acid (96%, 110 µL/min, 1.972 mmol/min) at -5 °C →-3 °C using a T-mixer (EtOH/N_2 bath was used, volume of the loop before second T-mixer 0.5 mL). The resulting acid mixture was passed through a 0.03 mL PTFE-tubing and mixed with the ethyl acetoacetate (**21**) solution (6.5 mL (49 mmol) of substrate **21** in CH_2Cl_2 (42.5 mL), 0.4 mL/min, 0.4 mmol/min) at the same temperature. The biphasic mixture was passed through 1.8 mL PTFE-tubing and collected in an Erlenmeyer flask containing stirred suspension of $MgSO_4$ in CH_2Cl_2 (40.5 mL of solution was collected, 21 g of $MgSO_4$ was used: for 1 mL of the pumped H_2SO_4 2 g of $MgSO_4$ was used) at room temperature, the reaction mixture was filtered, dried over Na_2SO_4, treated with EtOH (1:1 *v/v*) and concentrated under reduced pressure (30 °C). The residue was purified using Kugelrohr distillation at 100 °C, 11 mbar to give compound **22** (3.8 g, 70%) as a yellow oil. In addition, 22% (determined by NMR using mesitylene as an internal standard) of 3,4-bis(ethoxycarbonyl)-1,2,5-oxadiazole 2-oxide (**22b**)[4c] was observed. Analytical data for compound **22**: ^{1}H NMR (400 MHz, $CDCl_3$): 1.28 (t, J = 7.2 Hz, 3H), 4.27 (q, J = 7.1 Hz, 2H), 5.14 (s, 2H); ^{13}C

spectrum matches with the literature data.[36] ^{13}C NMR (100 MHz, $CDCl_3$): 13.9, 63.3, 76.4, 162.1; HRMS (ESI): $[M+Na]^+$ calcd for $C_4H_7NO_4Na$: 156.0273, found 156.0252.

N,*N*-Diethyl-2-nitroacetamide (**24**), *N*,*N*-diethyl-2-(hydroxyimino)-3-oxobutanamide (**24a**), 3,4-bis(diethylcarbamoyl)-1,2,5-oxadiazole 2-oxide (**24b**): Nitric acid (90+%, 8 µL/min, 0.171 mmol/min) was mixed with sulfuric acid (96%, 52 µL/min, 0.923 mmol/min) at 10 °C using a T-mixer. The resulting acid mixture was passed through a 0.03 mL PTFE-tubing and mixed with *N*,*N*-diethyl-3-oxobutanamide (**23**) solution (0.16 mL (1.01 mmol) of substrate **23** in CH_2Cl_2 (4.84 mL), 0.332 mL/min, 0.066 mmol/min) at the same temperature. The biphasic mixture was passed through 0.7 mL PTFE-tubing and collected in an Erlenmeyer flask containing stirred suspension of $MgSO_4$ in CH_2Cl_2 (for 4 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used) at room temperature. The reaction mixture was filtered and concentrated under reduced pressure. Crude reaction mixture was purified by column chromatography (*n*-hexane/ethyl acetate 5:1 →1:1 *v/v*) to give compound **24** (68.7 mg, 54%) as a yellow oil, compound **24b** (23.4 mg, 10%) as a yellow oil and compound **24a** (16.7 mg, 11%) as a light peach clear solid. Analytical data for compound **24**: R_f = 0.19 (*n*-hexane/ethyl acetate = 2:1 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$): 1.17 (t, *J* = 7.1 Hz, 3H), 1.23 (t, *J* = 7.1 Hz, 3H), 3.24 (q, *J* = 7.2 Hz, 2H), 3.44 (q, *J* = 7.1 Hz, 2H), 5.27 (s, 2H); ^{13}C NMR (100 MHz, CD_3OD): 12.9, 14.2, 42.0, 43.3, 78.3, 163.2; HRMS (ESI): $[M+Na]^+$ calcd for $C_6H_{12}N_2O_3Na$: 183.0746, found 183.0740. Analytical data for compound **24a**: R_f = 0.07 (*n*-hexane/ethyl acetate = 2:1 *v/v*); Obtained NMR matches with literature data.[37] ^{1}H NMR (400 MHz, $CDCl_3$): 1.12 (t, *J* = 7.0 Hz, 3H), 1.22 (t, *J* = 7.6 Hz, 3H), 2.39 (s, 3H), 3.12 (q, *J* = 6.7 Hz, 2H), 3.52 (q, *J* = 7.0 Hz, 2H), 11.64 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 12.7, 14.0, 25.7, 39.3, 42.9, 152.4, 164.2, 195.2. Analytical data for compound **24b:** R_f = 0.31 (*n*-hexane/ethyl acetate = 2:1 *v/v*); ^{1}H NMR (400 MHz, $CDCl_3$): 1.18 – 1.32 (m, 12H), 3.31 (q, *J* = 7.2 Hz, 2H), 3.48-3.55 (m, 4H), 3.59 (q, *J* = 7.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 12.6, 12.7, 14.4, 14.7, 40.5, 41.1, 43.3., 43.8, 111.5, 152.3, 154.8, 156.8; HRMS (ESI): $[M+Na]^+$ calcd for $C_{12}H_{20}N_4O_4Na$: 307.1382, found 307.1372, $[M+K]^+$ calcd for $C_{12}H_{20}N_4O_4K$: 323.1122, found 323.1111.

4-Methoxy-4-oxobutanoic acid (**26**), *methyl 4,5-dioxohexanoate* (**26a**), methyl 4-(hydroxyimino)-5-oxohexanoate (**26b**): General nitration procedure was used (0.38 mL (2.16 mmol) of substrate **25** in CH_2Cl_2 (5.62 mL)). Quenching and deacylation procedure: for 4 mL of the collected solution 5 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used, the reaction mixture was filtered and concentrated under reduced pressure. Crude was purified by column chromatography (*n*-hexane /ethyl acetate 5:1 →1:1

and then DCM → DCM/MeOH 100:1 → 60:1 → 5:1 *v/v*) to give compound **26** (65.4 mg, 34%) as an yellow oil together with compound **26a** (5.6 mg, 3%) as a yellow oil, and compound **26b** (22.0 mg, 9%) as a white amorphous solid. Additionally unreacted starting material was identified by ^{1}H NMR but was not isolated due to tedious purification procedure. Analytical data for compound **26**: Obtained NMR matches with the literature data.[38] ^{1}H NMR (400 MHz, $CDCl_3$): 2.60-2.69 (m, 4H), 3.69 (s, 3H), 8.5-9.5 (br, COOH); ^{13}C NMR (100 MHz, $CDCl_3$): 28.8, 29.1, 52.1, 172.8, 178.4. Analytical data for compound **26a**: R_f = 0.4 (*n*-hexane/ethyl acetate =4:1 *v/v*); ^{1}H NMR (600 MHz, $CDCl_3$): 2.36 (s, 3H), 2.66 (t, *J* = 6 Hz 2H), 3.03 (t, *J* = 6 Hz, 2H), 3.69 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$): 23.8, 27.8, 30.9, 52.1, 172.9, 197.1, 197.6; HRMS (ESI): $[M+Na]^+$ calcd for $C_7H_{10}O_4Na$: 181.0477, found 181.0478. Compound **26a** was previously synthesized.[39] Analytical data for compound **26b**: R_f = 0.37 (DCM/MeOH = 30:1 *v/v*);; ^{1}H NMR (400 MHz, $CDCl_3$): 2.37 (s, 3H), 2.52 (t, *J* = 7.8 Hz, 2H), 2.84 (t, *J* = 7.7 Hz, 2H), 3.67 (s, 3H), 7.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 18.2, 25.4, 30.2, 52.0, 158.8, 173.2, 196.7; HRMS (ESI): $[M+Na]^+$ calcd for $C_7H_{11}NO_4Na$: 196.0586, found 196.0579, $[M+K]^+$ calcd for $C_7H_{11}NO_4K$ 212.0325, found 212.0322.

6-Ethoxy-5,6-dioxohexanoic acid (**28**), *6-ethoxy-5-(hydroxyimino)-6-oxohexanoic acid* (**31**), *6-ethoxy-5-nitro-6-oxohexanoic acid* (**28b**): General nitration procedure was used (0.75 mL (5.06 mmol) of ethyl 2-oxocyclopentanecarboxylate (**27**) in CH_2Cl_2 (13.25 mL)). Quenching and deacylation procedure: for 11 mL of the collected solution 10 g of $MgSO_4$ and 20 mL CH_2Cl_2 were used, the reaction mixture was filtered and concentrated under reduced pressure. Analysis of crude ^{1}H NMR revealed formation of 6-ethoxy-5,6-dioxohexanoic acid (**28**) with the 61% yield determined by ^{1}H NMR using mesitylene as internal standard. Presumably ethyl 1-nitro-2-oxocyclopentanecarboxylate (**29**) was formed, as suggested by the ^{1}H NMR spectrum (^{1}H NMR (400 MHz, $CDCl_3$): 2.05-2.13 (m, 2H), 2.53-2.61 (m, 2H), 2.81 (dt, *J* = 14.5, 7.3 Hz, 1H), 2.96 (dt, *J* = 14.0, 6.8 Hz, 1H), peaks corresponding to EtO-group are overlapping with EtO-group of another compound) which, upon standing, spontaneously undergoes a ring-opening reaction with atmospheric water to give compound **28b**. The residue was purified by column chromatography (*n*-hexane/ethyl acetate 15:1 → 10:1 and then DCM/MeOH 60:1 → 15:1 *v/v*) to give compound **28** (353 mg, 47%) as a yellow oil, oxime **31** (42.2 mg, 5%) as a white solid and compound **28b** (33 mg, 4%) as a white solid. Analytical data for compound **28**: R_f = 0.45 (DCM/MeOH = 15:1 *v/v/v*); Analytical data for the compound **28** matches with the literature data.[40] ^{1}H NMR (400 MHz, $CDCl_3$): 1.37 (t, *J* = 7.1 Hz, 3H), 1.97 (quin, *J* = 7.2 Hz, 2H), 2.44 (t, *J* = 7.2 Hz, 2H), 2.95 (t, *J* = 7.1 Hz, 2H), 4.32 (q, *J* = 7.1 Hz, 2H), 9-11 (br signal COOH); ^{13}C NMR (100 MHz, $CDCl_3$):

14.0, 17.9, 32.6, 38.2, 62.6, 160.8, 179.2, 193.7. Analytical data for compound **31**: R_f = 0.31 (DCM/MeOH = 15:1 *v/v/v*); mp 106–108 °C; ^{1}H NMR: (400 MHz, $CDCl_3$): 1.35 (t, *J* = 7.1 Hz, 3H), 1.96 (p, *J* = 6.9 Hz, 2H), 2.45 (t, *J* = 6.9 Hz, 2H), 2.73 (t, *J* = 7.0 Hz, 2H), 4.30 (q, *J* = 7.1 Hz, 2H), 10-12 (br signal COOH), signal for N-OH proton is not observed; ^{13}C NMR (100 MHz, $CDCl_3$) 14.1, 21.0, 24.3, 33.6, 62.1, 151.9, 163.2, 178.2; HRMS (ESI): $[M+Na]^+$ calcd for $C_8H_{13}NO_5Na$ 226.0691, found 226.0686. Analytical data for compound **28b**: R_f = 0.48 (DCM/MeOH = 15:1 *v/v/v*); mp 47–49 °C; ^{1}H NMR (400 MHz, $CDCl_3$): 1.31 (dd, *J* = 7.1, 7.1 Hz, 3H), 1.67-1.80 (m, 2H), 2.18-2.27 (m, 1H), 2.29-2.39 (m, 1H), 2.46 (t, *J* = 7.1 Hz, 2H), 4.29 (ddd, *J* = 7.1, 7.1 , 7.1 Hz, 2H), 5.12 (dd, *J* = 9.3, 5.5 Hz, 1H), 10-12 (br signal COOH); ^{13}C NMR (100 MHz, $CDCl_3$) 14.0, 20.8, 29.5, 33.0, 63.3, 87.8, 164.4, 178.7; HRMS (ESI): $[M-H]^+$ calcd for $C_8H_{12}NO_6$: 218.0665, found 218.0686.

2. Supporting Information

^{1}H and ^{13}C NMR spectra for all new compounds, optimization experiments to determine minimum amount of desiccant needed for the quenching procedure

3. Author information

Notes

The authors declare no competing financial interest.

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