**Convenient Synthesis of ε -Halo- β -ketoesters and γ,γ' -Dibromoalkanones
by Regio- and Chemoselective Reaction of 2-Alkylidenetetrahydrofurans
with Boron trihalides: A 'Ring-Closure / Ring-Cleavage' Strategy**

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Supplementary Information

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2-Alkylidenetetrahydrofurans. The required starting materials were prepared by published procedures: 2-Alkylidenetetrahydrofurans **2k-n** and **2v** were prepared in one step by cyclization of dilithiated 1,3-dicarbonyl compounds **1** with 1-bromo-2-chloroethane (Scheme 3).¹⁰ 2-Alkylidenetetrahydrofurans **2b-e**, containing an alkyl group located at the exocyclic double bond, were prepared in two steps by synthesis of 2-alkylidenetetrahydrofurans and subsequent lithiation and alkylation (Scheme 3).^{10b} The aryl-substituted 2-alkylidenetetrahydrofurans **2f-j** were prepared by bromination of 2-alkylidenetetrahydrofurans and subsequent Suzuki reaction.¹¹ 2-Alkylidenetetrahydrofurans **2o-u** were obtained in one step by cyclization of 1,3-bis-silyl enol ethers with epoxides.¹² Compound **5a** was prepared by cyclization of dilithiated α -acetyl- γ -butyrolactone with 1-bromo-2-chloroethane.¹⁰ Compounds **5b-d** are available by cyclization of the α -acetyl- γ -butyrolactone α -acetyl- γ -butyrolactone derived 1,3-bis-silyl enol ether with epoxides.¹²

General procedure for the reaction of 2-alkylidenetetrahydrofurans with borontribromide or borontrichloride: To a CH₂Cl₂-solution (10 mL/mmol) of the 2-alkylidenetetrahydrofuran (**2**, **5**) (1 equiv.) was added BBr₃ (4 equiv.) at 0 °C. The reaction mixture was allowed to warm to 20 °C over 12 h and was stirred for 6 h at 20 °C. Water (15 mL) was slowly added to the reaction mixture and the organic layer was separated. The aqueous layer was extracted with CH₂Cl₂ (3 × 30 mL). The combined organic extracts were dried (Na₂SO₄), filtered and the filtrate was concentrated in vacuo. The residue was purified by chromatography (silica gel, *n*-hexane/EtOAc = 100:1 → 1:1) to give the 6-bromo-3-oxoalkanoate **3** or 1,7-dibromoheptan-4-one **6**.

6-Bromo-3-oxo-hexanoic acid ethyl ester (3a): Starting with **2a** (0.200 g, 1.28 mmol) and BBr₃ (1.283 g, 5.12 mmol) in CH₂Cl₂ (10 mL), **3a** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 50:1 → 10:1) as a slightly yellow oil (0.229 g, 76%, 7% of enol-form). ¹H NMR (CDCl₃, 300 MHz): δ = 1.29 (t, *J* = 7.2 Hz, 3 H, CH₃), 2.16 (quint, *J* = 6.6 Hz, 2 H, CH₂), 2.77 (t, *J* = 6.9 Hz, 2 H, CH₂), 3.45 (t, *J* = 7.2 Hz, 2 H, CH₂-Br), 3.46 (s, 2 H, CH₂), 4.21 (q, *J* = 7.2 Hz, 2 H, OCH₂), 5.03 (s, 1 H, CH of enol), 12.12 (s, 1 H, OH of enol). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 14.1 (CH₃), 26.17, 32.9 (CH₂), 40.8 (CH₂-Br), 49.3 (CH₂), 61.4 (OCH₂), 90.0 (CH=C of enol), 167.0 (O=C-O), 172.5 (O=C-O of enol), 176.4 (C=CH of enol), 201.6 (C=O). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2982 (w), 2937 (w, C-H), 1743 (s, O=C-O), 1716 (s, C=O), 1649 (w), 1440 (m), 1369 (m), 1320 (s), 1247 (s), 1184 (m), 1116 (m), 1096 (m), 1031 (m), 975 (w), 942 (w), 851 (w), 806 (w), 557 (w). MS (EI, 70 eV): *m/z* (%) = 237 (M⁺, 1), 191 (5), 157 (14), 148 (63), 130 (100), 115 (35), 87 (18). Anal. calcd. for C₈H₁₃O₃Br (237.093): C 40.53, H 5.53; found C 40.13, H 6.11. The exact molecular mass *m/z* = 236.0048 ± 2 ppm [M⁺] for C₈H₁₃O₃Br was confirmed by HRMS (EI, 70 eV).

2-(4-Bromo-butyryl)-decanoic acid ethyl ester (3b): Starting with **2b** (0.070 g, 0.26 mmol) and BBr₃ (0.264 g, 1.04 mmol) in CH₂Cl₂ (5 mL), **3b** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 75:1 → 1:1) as a slightly yellow oil (0.086 g, 95%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.89 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.20 – 1.34 (m, 15 H, 6×CH₂, CH₃), 1.80 – 1.87 (m, 2 H, CH₂), 2.14 (quint, *J* = 6.6 Hz, 2 H, CH₂), 2.62 – 2.82 (m, 2 H, CH₂), 3.43 (t, *J* = 7.2 Hz, 1 H, CH), 3.44 (t, *J* = 6.6 Hz, 2 H, CH₂-Br), 4.20 (q, *J* = 7.2 Hz, 2 H, OCH₂). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 14.2, 14.3 (CH₃), 22.8, 26.4, 27.6, 28.4, 29.3, 29.4, 29.5, 32.0, 33.2 (CH₂), 40.0 (CH₂-Br), 59.4 (CH), 61.5 (OCH₂), 169.9 (O=C-O), 204.5 (C=O). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2956 (m), 2927 (s), 2856 (w, C-H), 1744 (s, O=C-O), 1716 (s, C=O), 1462 (w), 1441 (w), 1406 (w), 1371 (w), 1298 (w), 1250 (w), 1186 (w), 1163 (w), 1123 (w), 1095 (w), 1028 (w). MS (EI, 70 eV): *m/z* (%) = 349 (M⁺, 3), 304 (3), 251 (7), 236 (26), 227 (8),

200 (100), 197 (5), 159 (94), 135 (8), 123 (839), 121 (826), 115 (16). The exact molecular mass $m/z = 348.1300 \pm 2$ ppm [M^+] for $C_{16}H_{29}O_3Br$ was confirmed by HRMS (EI, 70 eV).

2-(4-Bromo-butyl)-undecanoic acid ethyl ester (3c): Starting with **2c** (0.100 g, 0.34 mmol) and BBr_3 (0.338 g, 1.35 mmol) in CH_2Cl_2 (5 mL), **3c** was isolated after chromatography (silica gel, *n*-hexane/EtAOc = 50:1 $\rightarrow$ 20:1) as a slightly yellow oil (0.103 g, 81%). 1H NMR ($CDCl_3$, 300 MHz): δ = 0.88 (t, J = 6.9 Hz, 3 H, CH_3), 1.19 – 1.13 (m, 19 H, CH_3 , $8 \times CH_2$), 1.80 – 1.88 (m, 2 H, CH_2), 2.14 (quint, J = 6.6 Hz, 2 H, CH_2), 2.64 – 2.82 (m, 2 H, CH_2), 3.42 (t, J = 7.5 Hz, 1 H, CH), 3.44 (t, J = 6.6 Hz, 2 H, CH_2 -Br), 4.20 (q, J = 7.2 Hz, 2 H, OCH_2). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 14.30, 14.32 (CH_3), 22.9, 26.4, 27.6, 28.4, 29.49, 29.51, 29.70, 29.73, 29.77, 32.1, 33.2 (CH_2), 39.8 (CH_2 -Br), 59.4 (CH), 61.6 (OCH_2), 169.9 ($O=C-O$), 204.5 ($C=O$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2956 (m), 2927 (s), 2855 (m, C-H), 1744 (s, $O=C-O$), 1716 (s, $C=O$), 1645 (w), 1463 (m), 1407 (w), 1371 (w), 1300 (w), 1251 (m), 1184 (m), 1150 (m), 1128 (w), 1096 (w), 1031 (w). MS (EI, 70 eV): m/z (%) = 377 (M^+ , 6), 256 (5), 252 (8), 229 (81), 199 (6), 183 (7), 150 (100), 121 (29). Anal. calcd. for $C_{18}H_{33}O_3Br$ (377.361): C 57.29, H 8.81; found C 56.92, H 8.15. The exact molecular mass $m/z = 376.1613 \pm 2$ ppm [M^+] for $C_{18}H_{33}O_3Br$ was confirmed by HRMS (EI, 70 eV).

6-Bromo-2-(3-chloro-propyl)-3-oxo-hexanoic acid methyl ester (3d): Starting with **2d** (0.100 g, 0.46 mmol) and BBr_3 (0.463 g, 1.83 mmol) in CH_2Cl_2 (10 mL), **3d** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 50:1 $\rightarrow$ 1:1) as a slightly yellow oil (0.116 g, 84%). 1H NMR ($CDCl_3$, 300 MHz): δ = 1.74 – 1.83 (m, 2 H, CH_2), 2.01 (quint, J = 7.5 Hz, 2 H, CH_2), 2.10 – 2.19 (m, 2 H, CH_2), 2.63 – 2.88 (m, 2 H, CH_2), 3.42 – 3.57 (m, 5 H, CH, CH_2 -Br, CH_2 -Cl), 3.76 (s, 3 H, OCH_3). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 25.6, 26.3, 30.3, 33.1 (CH_2), 39.9 (CH_2 -Br), 44.4 (CH_2 -Cl), 52.8 (OCH_3), 58.2 (CH), 169.9 ($O=C-O$), 203.7 ($C=O$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2956 (m, C-H), 1745 (s, $O=C-O$), 1715 (s, $C=O$), 1639

(w), 1438 (m), 1406 (w), 1369 (w), 1273 (m), 1254 (m), 1203 (m), 1178 (m), 1150 (m), 1102 (w), 1078 (w), 1045 (w). MS (EI, 70 eV): m/z (%) = 300 (M^+ , 1), 269 (2), 263 (3), 237 (2), 222 (3), 205 (2), 177 (5), 150 (98), 148 (100), 129 (2), 123 (26), 118 (5), 116 (11), 112 (6), 101 (4). Anal. calcd. for $C_{10}H_{16}O_3BrCl$ (299.592): C 40.09, H 5.38; found C 39.85, H 5.95. The exact molecular mass $m/z = 297.9971 \pm 2$ ppm [M^+] for $C_{10}H_{16}O_3BrCl$ was confirmed by HRMS (EI, 70 eV).

2-Benzyl-6-Bromo-3-oxo-hexanoic acid methyl ester (3e): Starting with **2e** (0.100 g, 0.43 mmol) and BBr_3 (0.436 g, 1.72 mmol) in CH_2Cl_2 (5 mL), **3e** was isolated after chromatography (silica gel, n -hexane/EtOAc = 50:1 $\rightarrow$ 1:1) as a slightly yellow oil (0.130 g, 96%, 5% of enol-form). 1H NMR ($CDCl_3$, 300 MHz): δ = 2.02 – 2.13 (m, 2 H, CH_2), 2.48 (dt, J = 18.3, 6.6 Hz, 1 H, CH_2), 2.77 (dt, J = 18.3, 6.6 Hz, 1 H, CH_2), 3.18 (d, J = 7.5 Hz, 2 H, CH_2 -Ph), 3.35 (dt, J = 6.6, 1.4 Hz, 2 H, CH_2 -Br), 3.71 (s, 3 H, OCH_3), 3.82 (t, J = 7.5 Hz, CH), 7.15 – 7.30 (m, 5 H, 5 $\times$ CH of Ph), 12.09 (s, 1 H, OH of enol). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 26.2, 32.9, 34.2, 41.0 (CH_2), 52.7 (OCH_3), 60.5 (CH), 102.0 ($C=C-O$ of enol), 126.9, 128.8 (2C), 128.9 (2C) (CH of Ph), 138.1 (C of Ph), 169.5 ($O=C-O$), 172.0 ($O-C=C$ of enol), 203.7 ($C=O$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3029 (w), 3002 (w), 2953 (m, C-H), 1746 (s, $O=C-O$), 1715 (s, $C=O$), 1633 (m), 1604 (w), 1495 (w), 1437 (m), 1405 (w), 1369 (m), 1316 (m), 1255 (m), 1212 (s), 1170 (s), 1105 (m), 1078 (m), 1060 (m), 1031 (w), 983 (w), 751 (m), 701 (m). MS (EI, 70 eV): m/z (%) = 313 (M^+ , 2), 283 (1), 255 (6), 232 (10), 200 (100), 172 (14), 163 (100), 148 (21), 131 (55), 103 (14), 91 (27), 77 (9). The exact molecular mass $m/z = 312.0361 \pm 2$ ppm [M^+] for $C_{14}H_{17}O_3Br$ was confirmed by HRMS (EI, 70 eV).

6-Chloro-3-oxo-2-phenyl-hexanoic acid methyl ester (3f): Starting with **2f** (0.030 g, 0.14 mmol) and BCl_3 (0.55 mL, 1.0 M in heptane, 0.55 mmol) in CH_2Cl_2 (5 mL), **3f** was isolated after chromatography (silica gel, n -hexane/EtOAc = 50:1 $\rightarrow$ 20:1) as a colorless oil (0.030 g,

84%). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.01 (quint, J = 6.6 Hz, 2 H, CH_2), 2.65 (t, J = 6.9 Hz, 2 H, CH_2), 3.53 (t, J = 6.3 Hz, 2 H, $\text{CH}_2\text{-Cl}$), 3.76 (s, 3 H, OCH_3), 4.74 (s, 1 H, CH), 7.27 – 7.38 (m, 5 H, 5 $\times$ CH of Ph). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 26.5, 38.7 (CH_2), 44.5 ($\text{CH}_2\text{-Cl}$), 52.8 (OCH_3), 65.1 (CH), 127.3, 128.7 (2C), 129.0 (2C) (CH of Ph), 134.2 (C of Ph), 169.0 ($\text{O}=\text{C}-\text{O}$), 207.4 ($\text{C}=\text{O}$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3064 (w), 3031 (w), 2963 (m), 2926 (m), 2870 (w), 2857 (w, C–H), 1743 (s, $\text{O}=\text{C}-\text{O}$), 1717 (s, $\text{C}=\text{O}$), 1644 (w), 1603 (w), 1495 (w), 1450 (s), 1440 (s), 1408 (m), 1370 (m), 1350 (m), 1309 (m), 1260 (s), 1205 (s), 1158 (s), 1113 (s), 1095 (s), 1026 (s), 798 (w), 734 (w), 702 (s), 650 (w). MS (EI, 70 eV): m/z (%) = 254 (M^+ , 3), 222 (13), 196 (4), 164 (43), 149 (71), 118 (55), 104 (100), 91 (38), 77 (40). The exact molecular mass m/z = 254.0710 ± 2 ppm [M^+] for $\text{C}_{13}\text{H}_{15}\text{O}_3\text{Cl}$ was confirmed by HRMS (EI, 70 eV).

6-Bromo-3-oxo-2-*p*-tolyl-hexanoic acid methyl ester (3g): Starting with **2g** (0.090 g, 0.39 mmol) and BBr_3 (0.388 g, 1.55 mmol) in CH_2Cl_2 (10 mL), **3g** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 50:1 $\rightarrow$ 5:1) as a colorless oil (0.109 g, 89%, 21% of enol-form). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.09 (quint, J = 6.6 Hz, 2 H, CH_2), 2.36 (s, 3 H, CH_3), 2.67 (t, J = 6.6 Hz, 2 H, CH_2), 3.36 (t, J = 6.6 Hz, 2 H, $\text{CH}_2\text{-Br}$), 3.75 (s, 3 H, OCH_3), 4.69 (s, 1 H, CH), 7.17 – 7.26 (m, 4 H, 4 $\times$ CH), 13.06 (s, 1 H, OH of enol). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 21.3 (CH_3), 26.7, 33.0 (CH_2), 39.6 ($\text{CH}_2\text{-Br}$), 52.8 (OCH_3), 64.8 (CH), 104.4 ($\text{C}=\text{C}-\text{OH}$ of enol), 129.4 (2CH), 129.5 (C), 129.9 (2CH), 138.5 (C), 169.2 ($\text{O}=\text{C}-\text{O}$), 137.8 ($\text{O}=\text{C}-\text{O}$ of enol), 175.6 ($\text{HO}-\text{C}=\text{C}$ of enol), 202.9 ($\text{C}=\text{O}$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3132 (w), 3090 (w), 3026 (w), 3004 (w), 2954 (m), 2923 (m), 2862 (w, C–H), 1749 (s, $\text{O}=\text{C}-\text{O}$), 1717 (s, $\text{C}=\text{O}$), 1644 (w), 1609 (w), 1513 (w), 1437 (m), 1409 (w), 1338 (w), 1307 (w), 1262 (m), 1224 (m), 1205 (s), 1185 (m), 1159 (s), 1114 (w), 1076 (m), 1045 (w), 1020 (w), 814 (w). MS (EI, 70 eV): m/z (%) = 313 (M^+ , 4), 280 (3), 253 (2), 232 (29), 201 (4), 174

(11), 164 (100), 148 (36), 132 (72). The exact molecular mass $m/z = 312.0361 \pm 2$ ppm [M^+] for $C_{14}H_{17}O_3Br$ was confirmed by HRMS (EI, 70 eV).

6-Bromo-2-(4-chloro-phenyl)-3-oxo-hexanoic acid methyl ester (3h): Starting with **2h** (0.100 g, 0.40 mmol) and BBr_3 (0.398 g, 1.58 mmol) in CH_2Cl_2 (10 mL), **3h** was isolated chromatography (silica gel, *n*-hexane/EtOAc = 50:1 $\rightarrow$ 10:1) as a slightly yellow oil (0.102 g, 77%, 22% of enol-form). 1H NMR ($CDCl_3$, 300 MHz): δ = 2.07 – 2.13 (m, 2 H, CH_2), 2.64 – 2.73 (m, 2 H, CH_2), 3.32 – 3.43 (m, 2 H, CH_2 -Br), 3.76 (s, 3 H, OCH_3), 4.72 (s, 1 H, CH), 7.27 – 7.38 (m, 4 H, 4 $\times$ CH of Ph), 13.02 (s, 1 H, OH of enol). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 26.5, 32.9 (CH_2), 39.9 (CH_2 -Br), 53.0 (OCH_3), 64.2 (CH), 103.7 (C=C of enol), 129.3 (2C), 130.9 (2C) (CH of Ph), 133.5, 134.7 (C of Ph), 168.6 (O=C-O), 172.9 (O=C-O of enol), 175.4 (HO-C=C of enol), 202.1 (C=O). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3003 (w), 2954 (m), 2921 (w, C-H), 1751 (s, O=C-O), 1717 (s, C=O), 1645 (s), 1610 (s), 1491 (s), 1438 (s), 1408 (s), 1491 (s), 1438 (s), 1335 (s), 1298 (s), 1272 (s), 1209 (s), 1162 (s), 1091 (s), 1047 (m), 1013 (s), 984 (w), 830 (s), 764 (w), 748 (w), 559 (w), 503 (w). MS (EI, 70 eV): m/z (%) = 334 (M^+ , 6), 302 (21), 221 (5), 184 (89), 148 (100). The exact molecular mass $m/z = 331.9815 \pm 2$ ppm [M^+] for $C_{13}H_{14}O_3BrCl$ was confirmed by HRMS (EI, 70 eV).

6-Bromo-2-(4-hydroxy-phenyl)-3-oxo-hexanoic acid methyl ester (3i): **3i**: Starting with **2i** (0.130 g, 0.5 mmol) and BBr_3 (0.525 g, 2.1 mmol) in CH_2Cl_2 (5 mL), **3i** and **2i'** were isolated after chromatography (silica gel, *n*-hexane/EtOAc = 30:1 $\rightarrow$ 1:1) as brownish and white solids (**3i**: 0.118 g, 72%, 10% of enol-form; **2i'**: 0.027 g, 22%). 1H NMR ($CDCl_3$, 300 MHz): δ = 2.09 (quint, J = 6.9 Hz, 2 H, CH_2), 2.68 (t, J = 6.9 Hz, 2 H, CH_2), 3.35 (t, J = 6.9 Hz, 2 H, CH_2 -Br), 3.76 (s, 3 H, OCH_3), 4.70 (s, 1 H, CH), 5.65 (br, 1 H, OH of Ph), 6.83 (d, J = 8.7 Hz, 2 H, 2 $\times$ CH of Ph), 7.20 (d, J = 8.7 Hz, 2 H, 2 $\times$ CH of Ph), 13.03 (br, 1 H, OH of enol). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 26.3, 32.7 (CH_2), 39.4 (CH_2 -Br), 52.8 (OCH_3), 63.9 (CH),

103.9 (C=C–O of enol), 116.0 (2CH of Ph), 123.7 (C of Ph), 130.6 (2CH of Ph), 156.1 (C–OH of Ph), 169.6 (O=C–O), 175.0 (O–C=C of enol), 203.6 (C=O). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3396 (m, O–H), 3184 (w), 2957 (w, C–H), 1735 (s, O=C–O), 1703 (s, C=O), 1614 (w), 1594 (w), 1516 (s), 1439 (m), 1359 (m), 1335 (w), 1302 (w), 1274 (m), 1249 (m), 1213 (s), 1161 (m), 1095 (w), 991 (w), 832 (w), 557 (w), 528 (w). MS (EI, 70 eV): m/z (%) = 315 (M^+ , 1), 284 (14), 234 (5), 203 (1), 175 (5), 165 (85), 150 (29), 118 (3), 109 (100), 106 (55). Anal. calcd. for $\text{C}_{13}\text{H}_{15}\text{O}_4\text{Br}$ (315.163): C 49.54, H 4.80; found C 49.63, H 5.03.

(Dihydro-furan-2-ylidene)-(4-hydroxy-phenyl)-acetic acid methyl ester (2i'): ^1H NMR (CDCl_3 , 300 MHz): δ = 1.97 (quint, J = 7.2 Hz, 2 H, CH_2), 2.52 (t, J = 7.5 Hz, 2 H, CH_2), 3.68 (s, 3 H, OCH_3), 4.47 (t, J = 6.9 Hz, 2 H, OCH_2), 5.43 (broad s, 1 H, OH of Ph), 6.79 (dd, J = 6.6, 2.1 Hz, 2 H, $2\times\text{CH}$ of Ph), 7.05 (dd, J = 6.6, 2.1 Hz, 2 H, $2\times\text{CH}$ of Ph). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 13.4, 32.1 (CH_2), 51.4 (OCH_3), 74.2 (C–5), 103.0 (C=C–O), 115.2 (2CH of Ph), 129.6 (C of Ph), 131.8 (2CH of Ph), 154.7 (C–OH of Ph), 167.2 (O=C–O), 170.4 (O–C=C). IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3404 (br, O–H), 2955 (w), 2914 (w, C–H), 1684 (s, C=C–O), 1616 (s, C=C–C=O), 1515 (s), 1435 (m), 1338 (w), 1327 (w), 1305 (m), 1276 (s), 1243 (m), 1193 (s), 1170 (s), 1098 (w), 1051 (s), 1025 (w), 999 (m), 846 (m), 596 (w). MS (EI, 70 eV): m/z (%) = 234 (M^+ , 100), 202 (56), 174 (18). The exact molecular mass m/z = 234.0892 ± 2 ppm [M^+] for $\text{C}_{13}\text{H}_{14}\text{O}_4$ was confirmed by HRMS (EI, 70 eV).

6-Bromo-3-oxo-2,4-diphenyl-hexanoic acid methyl ester (3j): Starting with **2j** (0.060 g, 0.20 mmol) and BBr_3 (0.204 g, 0.82 mmol) in CH_2Cl_2 (5 mL), **3j** was isolated after chromatography (silica gel, n -hexane/EtOAc = 75:1 $\rightarrow$ 3:1) as a slightly yellow oil (0.072 g, 96%, 33% of enol-form). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.10 – 2.33 (m, 1 H, CH_2), 2.42 – 2.65 (m, 1 H, CH_2), 3.03 – 3.20 (m, 1 H, CH_2 –Br), 3.21 – 3.43 (m, 1 H, CH_2 –Br), 3.65 (s, 3 H, OCH_3), 3.93 (t, J = 7.2 Hz, 1 H, CH), 4.69 (s, 1 H, CH), 7.00 – 7.38 (m, 10 H, $10\times\text{CH}$ of Ph), 13.22 (s, 1 H, OH of enol). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 29.9 (CH_2), 31.9, 34.9

(CH-Ph), 48.9 (CH₂-Br), 55.9 (OCH₃), 127.2, 128.0, 128.8 (2C), 128.9 (2C), 129.5 (2C), 129.7 (2C) (CH of Ph), 134.1, 137.3 (C of Ph), 167.6 (O=C-O), 206.8 (C=O). IR (KBr, cm⁻¹): $\tilde{\nu}$ = 2956 (w), 2922 (w, C-H), 1750 (s, O=C-O), 1716 (s, C=O), 1494 (w), 1453 (m), 1436 (m), 1346 (w), 1308 (m), 1263 (m), 1206 (m), 1158 (m), 1114 (w), 1098 (w), 1078 (w), 1027 (w), 1013 (w), 704 (s). MS (EI, 70 eV): m/z (%) = 375 (M⁺, 1), 344 (2), 294 (2), 236 (1), 235 (2), 225 (12), 197 (64), 177 (4), 149 (31), 117 (63), 91 (100), 77 (13). HRMS (ESI): calcd. for C₁₉H₂₀O₃Br ([M+1]⁺) = 375.05959; found = 375.05880.

6-Bromo-3-oxo-4-ethyl-hexanoic acid ethyl ester (3k): Starting with **2k** (0.100 g, 0.54 mmol) and BBr₃ (0.549 g, 2.17 mmol) in CH₂Cl₂ (10 mL), **3k** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 100:1 → 1:1) as a yellow oil (0.118 g, 83%, 29% of enol-form) ¹H NMR (CDCl₃, 300 MHz): δ = 0.92 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.29 (t, *J* = 7.2 Hz, 3 H, OCH₂CH₃), 1.51 – 1.63 (m, 1 H, CH₂), 2.84 – 2.91 (m, 1 H, CH), 3.29 – 3.38 (m, 1 H, CH₂-Br), 3.40 – 3.48 (m, 1 H, CH₂-Br), 3.51 (d, *J* = 3.3 Hz, 2 H, CH₂), 4.20 (q, *J* = 7.2 Hz, 2 H, OCH₂), 5.06 (s, 1 H, CH=C of enol), 12.09 (s, 1 H, OH of enol). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 11.2, 14.2 (CH₃), 24.1, 31.5, 32.8 (CH₂), 48.9 (CH₂-Br), 51.6 (CH), 61.5 (OCH₂), 91.0 (CH=C-OH of enol), 167.0 (O=C-O), 172.7 (O=C-O of enol), 178.6 (HO-C=CH of enol), 205.3 (C=O). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2968 (m), 2935 (w, C-H), 1745 (s, O=C-O), 1712 (s, C=O), 1648 (m), 1631 (m), 1461 (w), 1424 (w), 1386 (w), 1369 (w), 1352 (w), 1311 (m), 1234 (s), 1176 (w), 1153 (m), 1117 (w), 1095 (w), 1031 (m). MS (EI, 70 eV): m/z (%) = 265 (M⁺, 2), 236 (1), 186 (5), 179 (8), 158 (59), 143 (7), 130 (5), 114 (55), 97 (5), 87 (42), 70 (100), 55 (30). The exact molecular mass m/z = 264.0361 ± 2 ppm [M⁺] for C₁₀H₁₇O₃Br was confirmed by HRMS (EI, 70 eV).

4-(2-Bromo-ethyl)-3-oxo-heptanoic acid ethyl ester (3l): Starting with **2l** (0.100 g, 0.50 mmol) and BBr₃ (0.505 g, 2.02 mmol) in CH₂Cl₂ (5 mL), **3l** was isolated after

chromatography (silica gel, *n*-hex/EtOAc = 50:1 → 20:1) as a slightly yellow oil (0.135 g, 96%, 12% of enol-form). ¹H NMR (CDCl₃, 300 MHz): δ = 0.92 (t, *J* = 6.9 Hz, 3 H, CH₃), 1.28 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.22 – 1.49 (m, 3 H, CH₂), 1.58 – 1.68 (m, 1 H, CH₂), 1.86 – 1.95 (m, 1 H, CH₂), 2.24 – 2.33 (m, 1 H, CH₂), 2.90 – 2.94 (m, 1 H, CH), 3.29 – 3.37 (m, 1 H, CH₂–Br), 3.40 – 3.48 (m, 1 H, CH₂–Br), 3.51 (s, 2 H, CH₂), 4.19 (q, *J* = 7.2 Hz, 2 H, OCH₂), 5.05 (s, 1 H, C=CH of enol), 12.10 (s, 1 H, OH of enol). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 14.18, 14.24 (CH₃), 20.2, 31.5, 33.3 (CH₂), 35.5 (CH₂–Br), 43.9 (CH), 49.0 (CH₂), 61.6 (OCH₂), 90.9 (CH=C of enol), 167.0 (O=C–O), 172.7 (O=C–O of enol), 178.9 (C=CH of enol), 205.4 (C=O). IR (neat, cm^{–1}): $\tilde{\nu}$ = 2962 (s), 2935 (s), 2873 (w, C–H), 1746 (s, O=C–O), 1712 (s, C=O), 1650 (s), 1629 (s), 1463 (m), 1446 (m), 1424 (m), 1382 (w), 1371 (w), 1311 (m), 1235 (s), 1153 (m), 1097 (w), 1034 (m), 842 (w), 808 (w). MS (EI, 70 eV): *m/z* (%) = 279 (M⁺, 3), 264 (3), 250 (3), 236 (30), 200 (5), 172 (25), 165 (7), 163 (11), 157 (64), 143 (75), 130 (10), 115 (100), 101 (11), 87 (79). Anal. calcd. for C₁₁H₁₉O₃Br (279.173): C 47.33, H 6.86; found C 47.67, H 6.54.

4-(2-Bromo-ethyl)-7-chloro-3-oxo-heptanoic acid methyl ester (3m): Starting with **2m** (0.070 g, 0.32 mmol) and BBr₃ (0.921 g, 1.28 mmol) in CH₂Cl₂ (5 mL), **3m** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 50:1 → 5:1) as a slightly yellow oil (0.082 g, 86%, 33% of enol-form). ¹H NMR (CDCl₃, 300 MHz): δ = 1.62 – 1.98 (m, 4 H, 2×CH₂), 2.16 – 2.33 (m, 2 H, CH₂), 2.92 – 3.01 (m, 1 H, CH), 3.31 – 3.54 (m, 4 H, CH₂–Cl, CH₂–Br), 3.56 (s, 2 H, CH₂), 3.75 (s, 3 H, OCH₃), 5.10 (s, 1 H, C=CH of enol), 12.03 (s, 1 H, OH of enol). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 27.9, 29.6, 31.2, 33.2 (CH₂), 35.36 (CH₂–Br), 43.5 (CH), 44.6 (CH₂–Cl), 48.6 (CH₂), 52.6 (OCH₃), 91.1 (CH=C of enol), 167.4 (O=C–O), 172.9 (O=C–O of enol), 177.8 (C=CH of enol), 204.7 (C=O). IR (neat, cm^{–1}): $\tilde{\nu}$ = 2997 (w), 2955 (s), 2868 (w, C–H), 1750 (s, O=C–O), 1711 (s, C=O), 1655 (s), 1629 (s), 1442 (s), 1404 (m), 1370 (m), 1317 (s), 1238 (s), 1155 (s), 1097 (m), 1050 (m), 1013 (m), 840 (w), 809 (m), 735

(w), 651 (m). MS (EI, 70 eV): m/z (%) = 299 (M^+ , 2), 164 (2), 227 (s), 222 (4), 192 (18), 142 (15), 129 (18), 114 (3), 101 (100). The exact molecular mass $m/z = 297.9971 \pm 2$ ppm [M^+] for $C_{10}H_{16}O_3BrCl$ was confirmed by HRMS (EI, 70 eV).

3-(2-Bromo-ethyl)-2-oxo-cyclododecanecarboxylic acid ethyl ester (3n): Starting with **2n** (0.100 g, 0.36 mmol) and BBr_3 (0.357 g, 1.43 mmol) in CH_2Cl_2 (10 mL), **3n** was isolated after chromatography (silica gel, n -hex/EtOAc = 50:1 $\rightarrow$ 15:1) as a yellow solid (0.113 g, 87%, 5% of enol-form). 1H NMR ($CDCl_3$, 300 MHz): δ = 0.95 – 1.40 (m, 15 H, $6 \times CH_2$, CH_3), 1.41 – 1.69 (m, 3 H, CH_2), 1.72 – 1.85 (m, 1 H, CH_2), 1.88 – 2.05 (m, 1 H, CH_2), 2.09 – 2.18 (m, 1 H, CH_2), 2.28 – 2.52 (m 2 H, CH_2), 3.21 – 3.42 (m, 2 H, CH, CH_2 -Br), 3.44 – 3.49 (m, 1 H, CH_2 -Br), 4.03 (dd, J = 11.7, 3.0 Hz, 1 H, CH), 4.17 (q, J = 7.2 Hz, 2 H, OCH_2), 13.01 (s, 1 H, OH of enol). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 14.3 (CH_3), 20.4, 22.1, 22.2, 23.4, 23.5, 26.4, 26.6, 26.9, 27.9, 31.5 (CH_2), 32.4 (CH_2 -Br), 49.7, 53.7 (CH), 61.8 (OCH_2), 102.0 ($C=C-O$ of enol), 169.9 ($O=C-O$), 174.5 ($O=C-O$ of enol), 177.4 ($O-C=C$ of enol), 206.7 ($C=O$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2929 (s), 2855 (s, C-H), 1735 (s, $O=C-O$), 1701 (s, $C=O$), 1637 (w), 1629 (w), 1619 (w), 1465 (s), 1342 (s), 1371 (w), 1340 (w), 1235 (s), 1185 (m), 1156 (m), 1147 (m), 1125 (m), 1096 (m), 1064 (m), 1027 (m), 982 (w), 944 (w), 854 (w), 836 (w), 746 (w), 728 (w), 702 (w). MS (EI, 70 eV): m/z (%) = 361 (M^+ , 53), 316 (77), 281 (68), 255 (60), 238 (80), 236 (100), 208 (57), 165 (61), 107 (59). The exact molecular mass $m/z = 360.1300 \pm 2$ ppm [M^+] for $C_{17}H_{29}O_3Br$ was confirmed by HRMS (EI, 70 eV).

6-Bromo-3-oxo-heptanoic acid methyl ester (3o): Starting with **2o** (0.060 g, 0.38 mmol) and BBr_3 (0.385 g, 1.54 mmol) in CH_2Cl_2 (5 mL), **3o** was isolated after chromatography (silica gel, n -hexane/EtOAc = 75:1 $\rightarrow$ 3:1) as a slightly yellow oil (0.072 g, 80%, 8% of enol-form). 1H NMR ($CDCl_3$, 300 MHz): δ = 1.74 (dd, J = 6.6, 1.5 Hz, 3 H, CH_3), 1.96 – 2.04 (m, 1 H, CH_2), 2.11 – 2.20 (m, 1 H, CH_2), 2.80 (dt, J = 1.5, 6.6 Hz, 2 H, CH_2), 3.49 (s, 2 H, CH_2), 3.75

(s, 3 H, OCH₃), 4.12 – 4.19 (m, 1 H, CH–Br), 5.07 (s, 1 H, CH=C of enol), 12.04 (s, 1 H, OH of enol). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 26.7 (CH₃), 34.4, 41.3, 49.2 (CH₂), 50.8 (CH–Br), 52.6 (OCH₃), 89.8 (CH=C–O of enol), 167.6 (O=C–O), 174.2 (O=C–O of enol), 178.1 (O–C=CH of enol), 201.7 (C=O). IR (neat, cm^{–1}): $\tilde{\nu}$ = 2957 (m), 2926 (m, C–H), 1750 (s, O=C–O), 1717 (s, C=O), 1652 (m), 1633 (m), 1439 (s), 1408 (s), 1378 (s), 1321 (s), 1257 (s), 1233 (s), 1194 (s), 1172 (s), 1152 (s), 1075 (m), 1012 (m), 963 (w), 887 (w), 840 (w), 655 (w), 587 (w), 530 (w). MS (EI, 70 eV): m/z (%) = 221 (M⁺–Me, 16), 163 (18), 148 (12), 135 (14), 121 (21), 108 (21), 93 (17), 80 (100). HRMS (ESI): calcd. for C₈H₁₄O₃Br ([M+1]⁺) = 237.01187; found = 237.01187.

6-Bromo-3-oxo-octanoic acid methyl ester (3p): Starting with **2p** (0.150 g, 0.88 mmol), BBr₃ (0.883 g, 3.53 mmol) in CH₂Cl₂ (15 mL), **3p** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 75:1 → 1:1) as a slightly yellow oil (0.200 g, 91%, 8% of enol-form). ¹H NMR (CDCl₃, 300 MHz): δ = 1.05 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.82 – 1.92 (m, 2 H, CH₂), 1.94 – 2.06 (m, 1 H, CH₂), 2.15 – 2.24 (m, 1 H, CH₂), 2.80 – 2.87 (m, 2 H, CH₂), 3.49 (s, 2 H, CH₂), 3.75 (s, 3 H, OCH₃), 3.95 – 4.07 (m, 1 H, CH–Br), 5.07 (s, 1 H, CH=C of enol), 12.08 (s, 1 H, OH of enol). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 10.5 (CH₃), 27.7, 32.4, 41.1, 49.1 (CH₂), 52.3 (OCH₃), 59.1 (CH–Br), 89.8 (CH=C–O of enol), 166.5 (O=C–O), 201.3 (C=O). IR (neat, cm^{–1}): $\tilde{\nu}$ = 2971 (m), 2940 (m), 2881 (w, C–H), 1750 (s, O=C–O), 1717 (s, C=O), 1646 (w), 1639 (w), 1456 (m), 1439 (m), 1409 (m), 1378 (m), 1322 (s), 1262 (s), 1247 (s), 1216 (s), 1171 (m), 1116 (w), 1077 (w), 1051 (w), 1015 (w). MS (EI, 70 eV): m/z (%) = 249 (M⁺, 6), 219 (3), 192 (37), 177 (21), 171 (3), 157 (26), 139 (25), 116 (9), 110 (7), 102 (95), 97 (86), 87 (20), 84 (24), 70 (100), 55 (95). Anal. calcd. for C₉H₁₅O₃Br (251.120): C 43.05, H 6.02; found C 43.89, H 6.54.

6-Bromo-3-oxo-decanoic acid methyl ester (3q): Starting with **2q** (0.150 g, 0.76 mmol) BBr_3 (0.766 g, 3.03 mmol) in CH_2Cl_2 (15 mL), **3q** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 75:1 $\rightarrow$ 1:1) as a slightly yellow oil (0.158 g, 75%, 24% of enol-form). ^1H NMR (CDCl_3 , 300 MHz): δ = 0.89 (t, J = 6.9 Hz, 3 H, CH_3), 1.29 – 1.57 (m, 4 H, $2\times\text{CH}_2$), 1.77 – 1.88 (m, 2 H, CH_2), 1.91 – 2.04 (m, 1 H, CH_2), 2.16 – 2.27 (m, 1 H, CH_2), 2.83 (t, J = 7.2 Hz, 2 H, CH_2), 3.48 (s, 2 H, CH_2), 3.75 (s, 3 H, OCH_3), 3.99 – 4.08 (m, 1 H, CH-Br), 5.04 (s, 1 H, CH=C of enol), 12.06 (s, 1 H, OH of enol). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 14.1 (CH_3), 22.2, 29.8, 32.5, 39.3, 41.2, 49.3 (CH_2), 52.5 (OCH_3), 57.6 (CH-Br), 89.7 (CH=C of enol), 167.6 (O=C-O), 173.8 (O=C-O of enol), 177.8 (O-C=CH of enol), 201.8 (C=O). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2958 (m), 2933 (m), 2868 (w, C-H), 1750 (s, O=C-O), 1719 (s, C=O), 1654 (w), 1633 (w), 1438 (m), 1409 (m), 1377 (w), 1361 (w), 1320 (m), 1241 (m), 1197 (m), 1173 (m), 1151 (m), 1101 (w), 1081 (w), 1026 (w). MS (EI, 70 eV): m/z (%) = 279 (M^+ , 2), 205 (5), 199 (35), 167 (7), 139 (2), 129 (15), 116 (100), 101 (49), 85 (34), 60 (45). Anal. calcd. for $\text{C}_{11}\text{H}_{19}\text{O}_3\text{Br}$ (279.173): C 47.33, H 6.86; found C 47.25, H 7.34.

6,7-Dibromo-3-oxo-heptanoic acid ethyl ester (3r): Starting with **2r** (0.100 g, 0.40 mmol) and BBr_3 (0.402 g, 1.61 mmol) in CH_2Cl_2 (10 mL), **3r** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 50:1 $\rightarrow$ 10:1) as a slightly yellow oil (0.123 g, 93%, 7% of enol-form). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.29 (t, J = 7.2 Hz, 3 H, CH_3), 1.93 – 2.05 (m, 1 H, CH_2), 2.51 – 2.62 (m, 1 H, CH_2), 2.80 – 2.86 (m, 2 H, CH_2), 3.48 (s, 2 H, CH_2), 3.62 (dd, J = 7.5, 6.6 Hz, 1 H, $\text{CH}_2\text{-Br}$), 3.86 (dd, J = 7.5, 4.5 Hz, 1 H, $\text{CH}_2\text{-Br}$), 4.21 (q, J = 7.2 Hz, 2 H, OCH_2), 4.19 – 4.27 (m, 1 H, CH-Br), 5.06 (s, 1 H, CH=C of enol), 12.10 (s, 1 H, OH of enol). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 14.3 (CH_3), 30.2, 36.2 (CH_2), 40.5 ($\text{CH}_2\text{-Br}$), 49.5 (CH_2), 51.9 (CH-Br), 61.9 (OCH_2), 90.2 (CH=C-OH of enol), 167.1 (O=C-O), 201.4 (C=O). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2983 (w), 2935 (w, C-H), 1743 (s, O=C-O), 1716 (s, C=O), 1651 (w), 1467 (w), 1436 (w), 1410 (m), 1370 (m), 1318 (s), 1258 (s), 1239 (s), 1177 (m), 1157 (m),

1095 (w), 1029 (m), 566 (w). MS (EI, 70 eV): m/z (%) = 330 (M^+ , 1), 251 (11), 243 (20), 215 (3), 205 (4), 169 (100), 163 (16), 143 (5), 135 (26), 115 (28), 97 (5), 95 (32), 87 (13). Anal. calcd. for $C_9H_{14}O_3Br_2$ (330.016): C 32.76, H 4.28; found C 33.36, H 4.01.

6-Bromo-7-chloro-3-oxo-heptanoic acid methyl ester (3s): Starting with **2s** (0.100 g, 0.52 mmol) and BBr_3 (0.531 g, 2.10 mmol) in CH_2Cl_2 (10 mL), **3s** was isolated after chromatography (silica gel, n -hexane/EtOAc = 75:1 $\rightarrow$ 1:1) as a slightly yellow oil (0.113 g, 80%, 8% of enol-form). 1H NMR ($CDCl_3$, 300 MHz): δ = 1.94 – 2.06 (m, 1 H, CH_2), 2.44 – 2.57 (m, 1 H, CH_2), 2.80 – 2.86 (m, 2 H, CH_2), 3.50 (s, 2 H, CH_2), 3.74 (dd, J = 11.4, 8.4 Hz, 1 H, CH_2 -Cl), 3.76 (s, 3 H, OCH_3), 3.93 (dd, J = 11.4, 4.8 Hz, 1 H, CH_2 -Cl), 4.13 – 4.22 (m, 1 H, CH-Br), 5.07 (s, 1 H, CH=C of enol), 12.07 (s, 1 H, OH of enol). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 29.3, 40.4, 48.3, 49.1 (CH_2), 52.3 (CH-Br), 52.6 (OCH_3), 167.5 ($O=C-O$), 201.2 ($C=O$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2955 (w), 2929 (w, C-H), 1747 (s, $O=C-O$), 1717 (s, $C=O$), 1653 (w), 1633 (w), 1438 (s), 1408 (m), 1375 (w), 1322 (m), 1260 (m), 1165 (m), 1125 (w), 1087 (w), 1013 (w). MS (EI, 70 eV): m/z (%) = 271 (M^+ , 2), 241 (7), 199 (35), 191 (9), 171 (5), 155 (100), 129 (6), 117 (18), 101 (71), 84 (6). The exact molecular mass m/z = 269.9658 ± 2 ppm [M^+] for $C_8H_{12}O_3BrCl$ was confirmed by HRMS (EI, 70 eV).

5-Bromomethyl-3-oxo-heptanoic acid methyl ester (3t): Starting with **2t** (0.100 g, 0.59 mmol) and BBr_3 (0.589 g, 2.35 mmol) in CH_2Cl_2 (10 mL), **3t** was isolated after chromatography (silica gel, n -hexane/EtOAc = 75:1 $\rightarrow$ 5:1) as a slightly yellow oil (0.122 g, 82%, 12% of enol-form). 1H NMR ($CDCl_3$, 300 MHz): δ = 0.91 (t, J = 7.2 Hz, 3 H, CH_3), 1.03 – 1.09 (m, 2 H, CH_2), 2.18 – 2.21 (m, 1 H, CH), 2.56 (dd, J = 18, 5.4 Hz, 1 H, CH_2), 2.78 (dd, J = 18, 7.2 Hz, 1 H, CH_2), 3.47 (s, 2 H, CH_2), 3.50 – 3.58 (m, 2 H, CH_2 -Br), 3.75 (s, 3 H, OCH_3), 5.07 (s, 1 H, CH=C of enol), 12.05 (s, 1 H, OH of enol). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 11.2 (CH_3), 25.5 (CH_2), 36.4 (CH), 38.6 (CH_2), 46.9 (CH_2 -Br), 49.6 (CH_2), 52.6

(OCH₃), 90.8 (CH=C–OH, of enol), 167.6 (O=C–O), 201.7 (C=O). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2966 (m), 2936 (w), 2879 (w, C–H), 1749 (s, O=C–O), 1717 (s, C=O), 1654 (w), 1633 (w), 1454 (m), 1439 (m), 1407 (m), 1377 (w), 1322 (m), 1245 (m), 1156 (m), 1128 (w), 1076 (w), 1012 (w). MS (EI, 70 eV): m/z (%) = 249 (M⁺, 10), 219 (5), 192 (43), 177 (27), 171 (3), 157 (38), 139 (30), 116 (3), 110 (8), 102 (91), 87 (89), 87 (21), 84 (20), 70 (97), 55 (100). Anal. calcd. for C₉H₁₅O₃Br (251.120): C 43.05, H 6.02; found C 43.11, H 6.08.

6-Bromo-3-oxo-5-phenyl-hexanoic acid ethyl ester (3u): Starting with **2u** (0.200 g, 0.86 mmol) and BBr₃ (0.862 g, 3.44 mmol) in CH₂Cl₂ (15 mL), **3u** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 75:1 → 1:1) as a slightly yellow oil (0.223 g, 83%, 9% of enol-form). ¹H NMR (CDCl₃, 300 MHz): δ = 1.25 (t, J = 7.2 Hz, 3 H, CH₃), 3.01 (dd, J = 17.7, 7.2 Hz, 1 H, CH₂), 3.24 (dd, J = 17.7, 5.7 Hz, 1 H, CH₂), 3.88 (s, 2 H, CH₂), 3.56 – 3.66 (m, 3 H, CH₂–Br, CH–Ph), 4.16 (q, J = 7.2 Hz, 2 H, OCH₂), 4.93 (s, 1 H, CH=C of enol), 7.19 – 7.32 (m, 5 H, 5×CH of Ph), 12.09 (s, 1 H, OH of enol). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 14.3 (CH₃), 38.1 (CH₂), 42.4 (CH–Ph), 46.9 (CH₂–Br), 49.9 (CH₂), 61.6 (OCH₂), 91.9 (HC=C–OH, enol form), 127.6, 127.9, 128.9 (CH of Ph), 141.2 (C of Ph), 166.9 (O=C–O), 173.8 (O=C–O of enol), 176.7 (O–C=CH of enol), 200.7 (C=O). IR (neat, cm⁻¹): $\tilde{\nu}$ = 3105 (w), 3086 (w), 3063 (w), 3030 (w), 2983 (m), 2934 (w), 2907 (w, C–H), 1743 (s, O=C–O), 1717 (s, C=O), 1650 (m), 1606 (w), 1495 (w), 1470 (w), 1451 (m), 1408 (m), 1369 (m), 1318 (s), 1247 (s), 1197 (m), 1157 (m), 1096 (m), 1075 (m), 1030 (s), 942 (w), 851 (w), 760 (m), 702 (s), 652 (w), 542 (w). MS (EI, 70 eV): m/z (%) = 313 (M⁺, 1), 267 (1), 232 (88), 225 (9), 203 (6), 199 (2), 187 (6), 185 (26), 158 (15), 145 (100), 141 (4), 129 (2), 114 (69), 103 (74), 87 (11). Anal. calcd. for C₁₄H₁₇O₃Br (313.191): C 53.69, H 5.47; found C 54.10, H 5.68.

6-Bromo-1-phenyl-hexane-1,3-dione (3v): Starting with **2v** (0.100 g, 0.53 mmol) and BBr₃ (0.532 g, 2.13 mmol) in CH₂Cl₂ (10 mL), **3v** was isolated after chromatography (silica gel, *n*-

hexane/EtOAc = 100:1 $\rightarrow$ 50:1) as a yellow solid (0.140 g, 98%, 84% of enol-form, data are listed for enol). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.25 (quint, J = 6.9 Hz, 2 H, CH_2), 2.64 (t, J = 7.2 Hz, 2 H, CH_2), 3.50 (t, J = 6.6 Hz, 2 H, $\text{CH}_2\text{-Br}$), 6.21 (s, 1 H, $\text{CH}=\text{C}$), 7.43 – 7.56 (m, 3 H, 3 $\times$ CH of Ph), 7.87 – 7.90 (m, 2 H, 2 $\times$ CH of Ph), 16.02 (s, 1 H, OH). ^{13}C NMR (CDCl_3 , 150 MHz): δ_{c} = 28.4, 33.2 (CH_2), 37.6 ($\text{CH}_2\text{-Br}$), 96.7 ($\text{CH}=\text{C}\text{-OH}$), 127.2, 128.8, 132.6 (CH of Ph), 135.0 (C of Ph), 183.1 ($\text{HO}\text{-C}=\text{CH}$), 195.7 ($\text{C}=\text{O}$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3439 (br, O–H), 1617 (s, $\text{C}=\text{O}$), 1573 (s), 1490 (m), 1458 (m), 1433 (m), 1359 (w), 1298 (w), 1264 (m), 767 (m), 694 (m). MS (EI, 70 eV): m/z (%) = 269 (M^+ , 15), 189 (3), 175 (5), 162 (53), 147 (100), 120 (3), 105 (58), 77 (48). The exact molecular mass m/z = 268.0099 ± 2 ppm [M^+] for $\text{C}_{12}\text{H}_{13}\text{O}_2\text{Br}$ was confirmed by HRMS (EI, 70 eV).

8-Bromo-3-oxo-oct-6-enoic acid ethyl ester (3w): Starting with **2w** (0.150 g, 0.82 mmol) and BBr_3 (0.833 g, 3.29 mmol) in CH_2Cl_2 (15 mL), **3w** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 50:1 $\rightarrow$ 1:1) as a slightly yellow oil (0.211 g, 98%, 10% of enol-form). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.29 (t, J = 7.2 Hz, 2 H, CH_3), 3.44 (s, 2 H, CH_2), 3.92 (d, J = 6.0 Hz, 2 H, $\text{CH}_2\text{-Br}$), 4.20 (q, J = 7.2 Hz, 2 H, OCH_2), 4.98 (s, 1 H, $\text{CH}=\text{C}$ of enol), 5.72 – 5.77 (m, 2 H, $\text{CH}=\text{CH}$), 12.10 (s, 1 H, OH of enol). ^{13}C NMR (CDCl_3 , 150 MHz): δ_{c} = 14.2 (CH_3), 25.8, 33.0, 41.9 (CH_2), 49.4 ($\text{CH}_2\text{-Br}$), 61.5 (OCH_2), 89.9 ($\text{CH}=\text{C}\text{-O}$ of enol), 127.6, 134.0 ($\text{CH}=\text{CH}$), 167.1 ($\text{O}=\text{C}\text{-O}$), 201.7 ($\text{C}=\text{O}$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2983 (w), 2935 (w, C–H), 1742 (s, $\text{O}=\text{C}\text{-O}$), 1716 (s, $\text{C}=\text{O}$), 1654 (w), 1438 (w), 1410 (w), 1369 (w), 1316 (m), 1247 (m), 1206 (m), 1160 (w), 1113 (w), 1100 (w), 1031 (m), 968 (w). MS (EI, 70 eV): m/z (%) = 263 (M^+ , 3), 218 (1), 183 (100), 176 (7), 148 (2), 137 (42), 135 (1), 130 (9), 119 (6), 114 (19), 110 (6), 95 (61), 87 (23).

1,7-Dibromo-heptan-4-one (6a):^{14a} Starting with **5a** (0.300 g, 1.95 mmol) and BBr_3 (1.950 g, 7.8 mmol) in CH_2Cl_2 (15 mL), **6a** was isolated after chromatography (silica gel, *n*-

hexane/EtOAc = 50:1 $\rightarrow$ 1:1) as a slightly yellow oil (0.365 g, 73%). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.14 (quint, J = 6.6 Hz, 4 H, $2\times\text{CH}_2$), 2.65 (t, J = 6.6 Hz, 4 H, $2\times\text{CH}_2$), 3.45 (t, J = 6.6 Hz, 4 H, $2\times\text{CH}_2\text{-Br}$). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 26.4 (2C), 33.4 (2C) (CH_2), 40.8 (2C) ($\text{CH}_2\text{-Br}$), 208.1 (C=O). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2964 (m), 2918 (w, C-H), 1714 (s, C=O), 1436 (m), 1409 (m), 1375 (m), 1296 (w), 1282 (w), 1253 (m), 1197 (w), 1151 (w), 1091 (w), 1046 (w), 558 (w). MS (EI, 70 eV): m/z (%) = 272 (M^+ , 1), 193 (4), 164 (24), 150 (100), 121 (38), 106 (3), 93 (7).

1,7-Dibromo-octan-4-one (6b): Starting with **5b** (0.020 g, 0.12 mmol) and BBr_3 (0.119 g, 0.48 mmol) in CH_2Cl_2 (3 mL), **6b** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 100:1 $\rightarrow$ 50:1), as a colorless oil (0.030 g, 88%). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.73 (d, J = 6.6 Hz, 3 H, CH_3), 1.92 – 2.03 (m, 1 H, CH_2), 2.08 – 2.18 (m, 1 H, CH_2), 2.18 (quint, J = 6.6 Hz, 2 H, CH_2), 2.65 (t, J = 6.6 Hz, 2 H, CH_2), 2.66 – 2.70 (m, 2 H, CH_2), 3.45 (t, J = 6.6 Hz, 2 H, $\text{CH}_2\text{-Br}$), 4.12 – 4.18 (m, 1 H, CH-Br). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 26.3 (CH_2), 26.6 (CH_3), 33.2, 34.4, 40.8 (CH_2), 40.9 ($\text{CH}_2\text{-Br}$), 51.0 (CH-Br), 208.4 (C=O). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2965 (m), 2923 (m, C-H), 1714 (s, C=O), 1637 (w), 1439 (m), 1410 (m), 1377 (m), 1336 (w), 1283 (w), 1250 (m), 1215 (w), 1182 (m), 1142 (m), 1079 (m). MS (EI, 70 eV): m/z (%) = 286 (M^+ , 3), 205 (4), 178 (1), 166 (45), 164 (45), 148 (49), 121 (16), 83 (30), 55 (100). HRMS (ESI): calcd. for $\text{C}_8\text{H}_{14}\text{OBr}$ ($[\text{M-Br}]^+$) = 205.02281; found = 205.02196.

1,7-Dibromo-nonan-4-one (6c): Starting with **5c** (0.600 g, 3.29 mmol) and BBr_3 (3.300 g, 13.2 mmol) in CH_2Cl_2 (20 mL), **6c** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 100:1 $\rightarrow$ 50:1) as a colorless oil (0.668 g, 68%). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.05 (t, J = 7.2 Hz, 3 H, CH_3), 1.40 – 1.51 (m, 2 H, CH_2), 1.82 – 1.92 (m, 2 H, CH_2), 2.14 (quint, J = 6.6 Hz, 2 H, CH_2), 2.65 (t, J = 6.6 Hz, 2 H, CH_2), 2.68 – 2.73 (m, 2 H, CH_2), 3.45

(t, $J = 6.6$ Hz, 2 H, CH₂-Br), 3.94 – 4.03 (m, 1 H, CH-Br). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 12.1$ (CH₃), 26.3, 32.2, 32.5, 33.2, 40.7, 40.8 (CH₂), 59.5 (CH-Br), 208.4 (C=O). IR (neat, cm⁻¹): $\tilde{\nu} = 2965$ (s), 2935 (m), 2879 (w, C-H), 1717 (s, C=O), 1604 (w), 1440 (s), 1410 (w), 1375 (m), 1312 (s), 1261 (m), 1227 (m), 1199 (s), 1154 (s), 1100 (m), 1066 (m), 1034 (m), 942 (w), 795 (w), 750 (w). MS (EI, 70 eV): m/z (%) = 300 (M⁺, 1), 219 (6), 177 (19), 164 (10), 150 (24), 139 (18), 135 (7), 121 (15), 108 (12), 95 (15), 70 (61), 55 (63), 41 (100). HRMS (ESI): calcd. for C₉H₁₆OBr ([M-Br]⁺) = 219.03846; found = 219.03779.

1,7-Dibromo-8-chloro-octan-4-one (6d): Starting with **5d** (0.200 g, 0.99 mmol) and BBr₃ (0.999 g, 3.95 mmol) in CH₂Cl₂ (15 mL), **6d** was isolated after chromatography (silica gel, *n*-hexane/EtOAc = 75:1 → 1:1) as a colorless oil (0.269 g, 85%). ¹H NMR (CDCl₃, 300 MHz): $\delta = 1.92 - 2.05$ (m, 1 H, CH₂), 2.15 (quint, $J = 6.6$ Hz, 2 H, CH₂), 2.42 – 2.53 (m, 1 H, CH₂), 2.66 (t, $J = 6.6$ Hz, 2 H, CH₂), 2.61 – 2.74 (m, 2 H, CH₂), 3.46 (t, $J = 6.6$ Hz, 2 H, CH₂-Br), 3.73 (dd, $J = 11.4, 8.7$ Hz, 1 H, CH₂-Cl), 3.91 (dd, $J = 11.4, 4.8$ Hz, 1 H, CH₂-Cl), 4.13 – 4.22 (m, 1 H, CH-Br). ¹³C NMR (CDCl₃, 150 MHz): $\delta_c = 26.5, 29.6, 33.4, 40.5$ (CH₂), 40.9 (CH₂-Br), 48.4 (CH₂-Cl), 52.7 (CH-Br), 208.0 (C=O). IR (neat, cm⁻¹): $\tilde{\nu} = 2963$ (w, C-H), 1714 (s, C=O), 1435 (m), 1410 (m), 1375 (m), 1287 (w), 1249 (w), 1180 (w), 1158 (w), 1093 (w), 774 (w). MS (EI, 70 eV): m/z (%) = 321 (M⁺, 4), 285 (5), 241 (16), 213 (11), 199 (47), 171 (10), 164 (12), 161 (6), 150 (100), 133 (32), 121 (35). Anal. calcd. for C₈H₁₃OBr₂Cl (320.451): C 29.99, H 4.09; found C 29.95, H 4.74. HRMS (ESI): calcd. for C₈H₁₃OBrCl ([M-Br]⁺) = 238.98384; found = 238.98305.

1,7-Dibromo-6-methyl-octan-4-one (6e): Starting with *trans*-**5e** (0.100 g, 0.55 mmol) and BBr₃ (0.550 g, 2.2 mmol) in CH₂Cl₂ (5 mL), **6e** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 100:1 → 5:1) as a colorless oil (0.144 g, 87%). The product was obtained as a 1:1 mixture of diastereomers. The analytical data are given for a separated sample of a pure

diastereomer. ^1H NMR (CDCl_3 , 300 MHz): δ = 0.98 (d, J = 6.6 Hz, 3 H, CH_3), 1.68 (d, J = 6.9 Hz, 3 H, CH_3), 2.13 (quint, J = 6.6 Hz, 2 H, CH_2), 2.17 – 2.24 (m, 1 H, CH), 2.41 (dd, J = 17.4, 7.0 Hz, 1 H, CH_2), 2.62 (t, J = 6.6 Hz, 2 H, CH_2), 2.69 (dd, J = 17.4, 6.1 Hz, 1 H, CH_2), 3.45 (t, J = 6.6 Hz, 2 H, $\text{CH}_2\text{-Br}$), 4.31 (dq, J = 6.9, 2.8 Hz, 1 H, CH-Br). ^{13}C NMR (CDCl_3 , 150 MHz): δ_{c} = 15.1, 23.9 (CH_3), 26.5, 33.4 (CH_2), 35.9 (CH), 41.5 (CH_2), 48.7 ($\text{CH}_2\text{-Br}$), 57.8 (CH-Br), 208.6 (C=O). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2969 (s), 2927 (m, C–H), 1713 (s, C=O), 1444 (m), 1409 (m), 1376 (s), 1328 (m), 1273 (m), 1257 (m), 1208 (m), 1143 (m), 1081 (m), 1014 (w), 970 (w), 602 (w). MS (EI, 70 eV): m/z (%) = 300 (M^+ , 6), 219 (7), 177 (21), 164 (66), 150 (65), 148 (74), 121 (26), 97 (31), 85 (7), 70 (100). HRMS (ESI): calcd. for $\text{C}_9\text{H}_{16}\text{OBr}$ ($[\text{M-Br}]^+$) = 219.03846; found = 219.03767.

5-Bromo-pentan-2-one (7):² Starting with α -acetyl- γ -butyrolactone (0.25 mL, 2.34 mmol) and BBr_3 (2.346 g, 9.37 mmol) in CH_2Cl_2 (15 mL), **7** was isolated after chromatography (silica gel, n -hex/EtOAc = 50:1 $\rightarrow$ 1:1) as a slightly yellow oil (0.206 g, 59%). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.12 (quint, J = 6.6 Hz, 2 H, CH_2), 2.17 (s, 3 H, CH_3), 2.65 (t, J = 6.9 Hz, 2 H, CH_2), 3.45 (t, J = 6.3 Hz, 2 H, $\text{CH}_2\text{-Br}$). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2965 (w, C–H), 1715 (s, C=O), 1432 (m), 1413 (m), 1367 (m), 1302 (w), 1282 (w), 1248 (m), 1217 (w), 1196 (w), 1179 (m), 1185 (m).

Synthesis of trans-4,5-Dimethyl-tetrahydro-[2,3']bifuranylidene-2'-one (5e): The synthesis was carried out according to a known procedure for related products:¹¹ Starting with the 1,3-bis-silyl enol ether of α -acetyl- γ -butyrolactone (2.725 g, 10 mmol), *cis*-2,3-epoxybutane (0.883 g, 12 mmol) and TiCl_4 (2.2 mL, 20 mmol) in CH_2Cl_2 (75 mL), **5e** was isolated after chromatography (silica gel, n -hexane/EtOAc = 75:1 $\rightarrow$ 5:1) as a colorless oil (0.669 g, 37%). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.11 (d, J = 6.7 Hz, 3 H, CH_3), 1.38 (d, J = 6.2 Hz, 3 H, CH_3), 1.92 – 2.03 (m, 1 H, CH_2), 2.54 (ddt, J = 18.0, 9.9, 3.0 Hz, 1 H, CH_2), 2.83 – 2.90 (m, 2

H, CH₂), 3.55 (ddt, $J = 18.0, 7.7, 1.7$ Hz, 1 H, CH), 4.01 – 4.10 (m, 1 H, OCH), 4.30 (t, $J = 7.7$ Hz, 2 H, OCH₂). ¹³C NMR (CDCl₃, 150 MHz): $\delta_c = 16.1, 18.8$ (CH₃), 25.0, 37.7 (CH₂), 39.5 (CH), 65.3 (OCH₂), 86.7 (OCH), 92.8 (C=C–O), 168.7 (O–C=C), 173.3 (O=C–O). IR (neat, cm^{–1}): $\tilde{\nu} = 2975$ (w), 2930 (w, C–H), 1733 (s, O=C–O), 1680 (s, C=C–O), 1454 (w), 1389 (w), 1377 (w), 1306 (m), 1262 (m), 1164 (w), 1057 (s), 1023 (s), 1001 (s), 851 (w). MS (EI, 70 eV): m/z (%) = 182 (M⁺, 46), 167 (100), 152 (6), 139 (4), 124 (10), 111 (52), 95 (17). HRMS (ESI): calcd. for C₁₀H₁₅O₃ ([M+1]⁺) = 183.10212; found = 183.10127.

EB0460-1

100%
80%
60%
40%
20%
0%

AKWEISZ

EB075-7

AK WEISZ

Current Date	PARMCC08
NAME	KIDDYS
RYDNO	A
ZRODNO	2
	-

22 Acquisition Parameters
DATE 20040504

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PROBHD 5 mm PBO 13C 1

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SOLV2007	12811

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CS	96	96
DS	96	96

FIGURE NO.	FIGURE
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2017 2019D.4
2018 13.100
2019 6.00

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DI	2.30000000

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***** CHANNEL, F1 *****

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P ₂	0.00

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PL12	15.50

STV 000-1526025

SI 32750
 DE 150.9024957

SSB	0
LT	2.00

1.00
100%

EB0474-1

AKWEISZ

3c

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PROCNO 1

F2 - Acquisition Parameters
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Time_ 4.49
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SOLVENT CCl4
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FIDRES 1.147277 Hz
AQ 0.4350777 sec
RG 812.7
DM 13.300 usec
TS 2.0000000 sec
F2 296.5 K
D1 2.00000000 sec
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SAMPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time_ 4.49
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AQ 0.4350777 sec
RG 812.7
DM 13.300 usec
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F2 296.5 K
D1 2.00000000 sec
D11 0.01000000 sec
MIRAST 0.00000000 sec
MORAK 0.01000000 sec

----- CHANNEL F3 -----

NAME EB0474-1
SAMPNO 2
PROCNO 1

F3 - Acquisition Parameters
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SOLVENT CCl4
NS 162
DS 0
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FIDRES 1.147277 Hz
AQ 0.4350777 sec
RG 812.7
DM 13.300 usec
TS 2.0000000 sec
F3 296.5 K
D1 2.00000000 sec
D11 0.01000000 sec
MIRAST 0.00000000 sec
MORAK 0.01000000 sec

FR0483-2

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Current Data Parameters
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 NUC1: 13C
 P1: 4.00 usec
 PL1: 0.00 dB
 SFO1: 150.9201628 MHz
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 NUC2: 1H
 PCPD2: 100.00 usec
 PL2: 120.00 dB
 PL12: 15.00 dB
 SFO2: 600.1328051 MHz
 F2 - Processing parameters
 S: 32768
 SF: 150.9024957 MHz
 WDW: EM
 SSB: 0
 LB: 2.00 Hz
 GB: 0
 PC: 1.00

FR0478-1

3e

Current Data Parameters
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 EXPTNO: 2
 PROCNO: 1
 F2 Acquisition Parameters
 Date_ Time: 20040427 18.04
 INSTRUM: spect
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 TD: 32768
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 d11: 0.0300000 sec
 MARSRT: 0.0000000 sec
 MCWRT: 0.01500000 sec
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 NUC1: 13C
 P1: 4.00 usec
 PL1: 0.00 dB
 SFO1: 125.9201528 MHz
 ----- CHANNEL f2 -----
 CUPROG2: waltz16
 NUC2: 1H
 PCPD2: 100.00 usec
 PL2: 120.00 dB
 PL12: 20.30 dB
 SFO2: 600.1328053 MHz
 F2 Processing parameters
 SI: 32768
 SF: 500.9925015 MHz
 WDW: HM
 SSB: 0
 LB: 2.00 Hz
 GB: 0
 PC: 1.00

AKWEISZ

Current Data Parameters

NAME: E0465-1

EXPNO: 1

PROCNO: 1

F2 - Acquisition Parameters

Date_: 20040504

Time: 12.57

INSTRUM: spect

PROBHD: 5 mm BBO 13C-1

PULPROG: zgpg30

TD: 32768

SD: 32768

SOLVENT: CDCl3

NS: 128

DS: 4

SWH: 15159.364 Hz

FIDRES: 1.141277 Hz

AQ: 0.432877 sec

RG: 16192

DE: 11.100 usec

TE: 300.2 K

SI: 3.2500000 sec

DI: 0.0100000 sec

MCREST: 0.0000000 sec

MCWAK: 0.0150000 sec

***** CHANNEL f1 *****

NUC1: 13C

P1: 4.00 usec

PL1: 0.00 dB

SP01: 150.9201528 MHz

***** CHANNEL f2 *****

CPDPRG2: waltz16

NUC2: 1H

PL2: 100.00 usec

PL12: 120.00 dB

PL12: 15.00 dB

SFO2: 600.132653 MHz

F2 - Processing parameters

SI: 32768

DF: 150.9201528 MHz

K0: 16

SSB: 2

LB: 2.00 Hz

GB: 0

PC: 1.00

AKWEISZ

EB0463-1

50.18 4.60 3.50 3.40 3.30 3.20 3.10 3.00 2.90 2.80 2.70 2.60 2.50 2.40 2.30 2.20 2.10 2.00 1.90 1.80 1.70 1.60 1.50 1.40 1.30 1.20 1.10 1.00 0.90 0.80 0.70 0.60 0.50 0.40 0.30 0.20 0.10 0.00

50.18 4.60 3.50 3.40 3.30 3.20 3.10 3.00 2.90 2.80 2.70 2.60 2.50 2.40 2.30 2.20 2.10 2.00 1.90 1.80 1.70 1.60 1.50 1.40 1.30 1.20 1.10 1.00 0.90 0.80 0.70 0.60 0.50 0.40 0.30 0.20 0.10 0.00

AKWEISZ

12 C

Current Data Parameters
NAME EB0463 1
EXTNO 2
PROCNO 1

F2 Acquisition Parameters

Date 20040227
Time 12.53
INSTRUM spect
PROBHD 5 mm TBI 1H 31
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 2

SWH 37501.964 Hz
FIDRES 1.147277 Hz
AQ 0.435877 sec
RG 1625.2
GB 1625.2

PC 16.300 msec
DE 6.000 msec
TE 300.2 K

2.0000000 sec
d11 0.0000000 sec
MCALCU 0.0150000 sec
MWMRK 0.0150000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 4.00 msec
PL1 0.00 dB
SFO1 150.9201628 MHz

----- CHANNEL f2 -----
CEPRG2 waltz16
NUC2 1H
PCPD2 150.00 msec
PL2 120.00 dB
PL12 20.10 dB
SFO2 600.137053 MHz

F2 Processing parameters
SI 32768
SF 150.9021523 MHz
WDW RM
SSB 0
GB 2.00 Hz
PC 1.00

EB0464-1

Current	Date	Parameters
NAME	EXPNO	1
EXPNO	PROCNO	2
PROCNO		1
F2 - Acquisition Parameters		
Date_	Time	20040427
Time		11.11
INSTRUM		spec2
PROBHD	5 mm TE1	HH-3.
PULPROG		zgpg
CD		3768
PCPDPRG2		CPD3
SOLVENT		CL3
DS		128
PS		0
SM		37593.964 Hz
NUC1		1.114777 Hz
NUC2		0.435615 Hz
MO		1300 usec
DO		5.00 usec
DE		297.0 K
TE		2.00000000 sec
DEL		0.03000000 sec
MGFTEST		0.00040000 sec
MGWEX		0.07500000 sec
CHANNEL F1		
NUC1		13C
NUC2		4.00 usec
PL1		0.00 dB
SFO1		150.9201628 MHz
CHANNEL F2		
CPDPRG2		waltz16
NUC2		1H
PCPDPRG2		100.00 usec
PL1		120.00 dB
PL2		20.30 dB
SFO2		600.1328053 MHz
F2 - Processing parameters		
SF		375.68
WDW		150.9024900 MHz
SSR		EM
GB		0
GB		2.00 Hz
GB		0
PC		1.00

```
Current Data Parameters
NAME: 151-130373 1a
PAXNO 13
PROXNO 1
USER lane
```

1		2	
Acquisition Parameters		Acquisition Parameters	
Date	2004/01/12	Date	2004/01/12
Time	10:09	Time	10:09
INSTRUM	SPCT	INSTRUM	SPCT
PROBHD	5 mm Midium	PROBHD	5 mm Midium
PULPROG	zgpg2	PULPROG	zgpg2
TD	3268	TD	3268
SD	1.28	SD	1.28
NAME		NAME	
DS	0	DS	0
SWH	21470.14 Hz	SWH	21470.14 Hz
FIDRES	0.065126 Hz	FIDRES	0.065126 Hz
AQ	0.755110 sec	AQ	0.755110 sec
RG	4268	RG	4268
DF	23400000	DF	23400000
DE	32.80 dB	DE	32.80 dB
TE	2996.0 K	TE	2996.0 K
D1	0.00002000 sec	D1	0.00002000 sec
D1.6	0.00000000 sec	D1.6	0.00000000 sec
CPDPRG1	zgpg2	CPDPRG1	zgpg2
PC1	110.00000000 sec	PC1	110.00000000 sec
PL1	0.010000000000000000	PL1	0.010000000000000000
PLS	22.00 dB	PLS	22.00 dB
PI	2.50 sec	PI	2.50 sec
SI	1.00	SI	1.00
SI1	1.00	SI1	1.00
SI1P1	1.00	SI1P1	1.00
SI1P1S	1.00	SI1P1S	1.00
Processing parameters		Processing parameters	
F0=60.136 MHz		F0=60.136 MHz	
F1=75.467578 MHz		F1=75.467578 MHz	
WDW		WDW	
SSB		SSB	
GB		GB	
PC		PC	
LMD		LMD	

Ernst Moritz Arndt Universität Gießen
 ARX 300 Kernspinanz Spektrometer
 1D NMR Spektrum (Protonen/Carbonkoppel)
 1c: Bsp. 18073 1c
 Lösungsmittel: Deuteriochloroform (CDCl₃)
 Referenz: Lösungsmittel 77.0 ppm
 Messtemperatur: 298 K (25 °C)
 Messzeit: 12 Stunden 40 Minuten

2c

Current Data Parameters
 NAME: 18073 1c
 EXPNO: 1
 PROCNO: 1
 F2: Acquisition Parameters
 Date_ Time: 20101112 17:14
 INSTRUM: spect
 PRGNAME: 5fimo Multinu
 F1: 1D
 SOLVENT: CDCl₃
 NS: 16381
 DS: 4
 SWH: 21739.131 Hz
 FIDRES: 0.663126 Hz
 AQ: 0.753110 sec
 RG: 42768
 DW: 21.000 usec
 DE: 32.80 usec
 TE: 298.0 K
 D1: 0.0000000 sec
 D12: 0.0000000 sec
 D16: 0.0000000 sec
 CTDPRT: wait16
 F3: 1.1000000 sec
 F31: 0.0000000 sec
 D15: 22.000000 sec
 P1: 2.50 usec
 SFO1: 75.166117 MHz
 NUC1: 13C
 F2: Processing parameters
 SI: 16384
 SF: 75.467512 MHz
 WDW: 0
 SSB: 0
 LB: 1.20 Hz
 GB: 0
 PC: 1.00

EB0488-1

AKWEISZ

3j

45.6

Current Data Parameters
NAME: 3j
EXPNO: 2
PROCNO: 1

F2 Acquisition Parameters
DATE_: 20010809
TIME: 22.15
INSTRUM: spect
PROBHD: 5 mm TBI
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 512
DS: 4
SWH: 42501.605 Hz
FIDRES: 1.250720 Hz
AQ: 0.3998126 sec
RG: 1628.5
ZG: 0
ZM: 17.200 usec
ZC: 236.7 K
ZD: 2.000000 sec
ZS: 0.000000 sec
MC3EST: 0.0000000 sec
MCKE: 0.01000000 sec

----- CHANNEL f1 -----
NUC1: 13C
P1: 4.00 usec
PL1: 0.00 dB
SFO1: 50.918260 MHz

----- CHANNEL f2 -----
CPROG2: waltz16
NUC2: 1H
PCPD2: 100.00 usec
PL2: 20.00 dB
PL12: 20.30 dB
SFO2: 600.130091 MHz
F2 Processing parameters
SI: 32768
SF: 50.9073927 MHz
WDW: EM
SSB: 0
GB: 0
PC: 1.00

EB0530-1

AKWEISZ

(3k)

Current Data Parameters
 NAME EB0530-1
 EXTNO 2
 PROCNO 1

F2 Acquisition Parameters
 Date_ 20040525
 Time_ 22.36
 INSTRUM spect
 PROBRD 5 mm TAI : H 31
 PULPROG zgpg30
 TD 65536
 TO 17.668
 SOLVENT CHCl3
 NS 128
 DS 4
 SWH 37593.944 Hz
 FIDRES 1.147277 Hz
 AQ 0.438777 sec
 RG 2896.1
 EQ 13.300 usec
 DE 277.0 usec
 WE 2.000000 sec
 DI 0.0100000 sec
 D11 0.0000000 sec
 ACQTIME 0.0000000 sec
 MUMK 0.0200000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 4.00 usec
 PL1 0.00 dB
 SFO1 100.626100 MHz

===== CHANNEL f2 =====
 NUC2 1H
 P2 120.00 usec
 PL2 120.00 dB
 SFO2 500.1328053 MHz

F2 Processing parameters
 SI 32768
 SF 150.9024980 MHz
 WDW EM
 SSB 0
 GB 2.00 Hz
 PC 1.00

AKWEISZ

Current Data Parameters
NAME: EBO472
EXPNO: 2
PROCNO: 1

F2 Acquisition Parameters
Date_ Time: 20040421 14:22
INSTRUM: spect
PROBHD: 5 mm 1H-13
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 129
DS: 0
SWH: 37593.984 Hz
FIDRES: 1.147277 Hz
AQ: 0.4154977 sec
RG: 832.7
DM: 13.300 umsec
DE: 6.00 umsec
TE: 296.6 K
D2: 2.0000000 sec
U2: 0.0200000 sec
MCREST: 0.01500000 sec
NORMK: 0.01500000 sec

***** CHANNEL f1 *****
NUC1: 13C
P1: 4.00 umsec
PL1: 0.00 dB
SFO1: 150.9201628 MHz

***** CHANNEL f2 *****
C12PRG2: waltz16
NUC2: 1H
PC2PR2: 100.00 umsec
PC2: 120.00 dB
PC12: 20.30 dB
SFO2: 600.1329053 MHz

F2 Processing parameters
SI: 32768
SF: 150.9024946 MHz
WFM: LM
SSS: 0
LB: 2.30 Hz
GB: 0
PC: 1.00

AK
WEISZ

CURRENT DATA PARAMETERS	
NAME	EE0673-1
EXPNO	2
PROCNO	1

P2 - Acquisition Parameters
Date_ 20040427

LINE	INSTRUM	PROVID	% CHG '13/1	13-15	APPROX	THE ST
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90						

PUBLISHED	2940
BY	32768
CONVENT	1995

NI	250
D8	0

SM	0.0001
ELINES	1.147277
AO	0.4358777

PO	1625-5
DA	14-330
W	6-22

FR	297.0
DL	2.00000000

1144	0.01000000
MCHE:PA	0.00000000
MCWRK	0.01500000

----- CHAN:PT, F1
METN
130

21	4.00
22	0.00

----- CHANNEL 12 -----

CPDPRG2	W1:TP10
NJC2	1H
CPDPRG2	1.00 00

P.12	20.00
P.12	20.10

SP02 600.1328052

31 32768
SF 150,9024946
EARTH

2000

GB	1.00
PC	1.00

EB0476-1

AKWEISZ

(3n)

172

Current Data Parameters
NAME: EB0476-1
EXPNO: 2
PROCNO: 1

F2 Acquisition Parameters
Date_: 20040427
Time: 15.06
INSTRUM: spect
PROBHD: 5 mm TBI
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 256
DS: 0
SWH: 37593.964 Hz
FIDRES: 1.347277 Hz
AQ: 0.416477 sec
RG: 1025.0
RM: 13.300 uS
R2: 0.0000000
R1: 0.0000000
D1: 2.0000000 sec
D2: 0.0000000 sec
DE: 0.0000000 sec
TE: 297.2 K
DT: 0.0000000 sec
ACQRES: 0.0000000 sec
K1: 0.0150000 sec

===== CHANNEL f1 =====
NUC1: 13C
P1: 4.00 uS
PL1: 0.00 dB
SFO1: 150.9201628 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 100.62 uS
PL2: 120.00 dB
PL12: 20.30 dB
SFO2: 600.132601 MHz

F2 Processing parameters
SI: 32768
SF: 500.132601 MHz
WDW: EM
SSB: 0
GB: 0
CB: 2.00 Hz
PC: 1.00

FB0489-1

30

2c

Current Data Parameters
NAME FB0489-1
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20000427
Time 16.31
INSTRUM spect
PROBHD 5 mm TBI 1H-31
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 37593.984 Hz
FIDRES 1.247277 Hz
AQ 0.4188777 sec
RG 625.5
LW 23.300 usec
TR 6.00 usec
TE 297.0 K
D1 2.50000000 sec
d11 0.01000000 sec
MAGNET 0.01500000 sec
MORF 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 4.00 usec
PL 0.00 dB
SFO1 150.9201628 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 20.30 dB
SFO2 600.1328053 MHz

F2 - Processing parameters
SI 32768
SF 150.9024957 MHz
WDW EM
SS 0
LB 2.00 Hz
GB 0
PC 1.00

AKWEISZ

Bmsr-Moritz-Arndt-Universität Greifswald
 ARX300 - Kernresonanz-Spektrometer
 13C-NMR-Spektrum (Protonenbreitenkoppelt)
 E. Beilur: HB0499-a
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME: BE_HB0499-a
 EXPNO: 13
 PROCNO: 1
 USER: lang

F2 - Acquisition Parameters
 Date_: 20041202
 Time: 13.18
 INSTRUM: spect
 PROBHD: 5 mm Multia
 PULPROG: zgpg
 TD: 32768
 SOLVENT: CDCl₃
 NS: 128
 DS: 0
 SWH: 21739.131 Hz
 FIDRES: 0.665426 Hz
 AQ: 0.7537140 sec
 RG: 32768
 DW: 23.000 usec
 DE: 32.86 usec
 TE: 298.0 K
 D12: 0.0000200 sec
 D16: 30.00 dB
 D1: 2.0000000 sec
 CPDPRG: waltz16
 P31: 110.00 usec
 D11: 0.0300000 sec
 D15: 21.00 dB
 P1: 2.50 usec
 SFO1: 75.476177 MHz
 NUCLEUS: 13C

F2 - Processing parameters
 SI: 32768
 SF: 75.4677545 MHz
 WDW: EM
 SSB: 0
 LB: 1.20 Hz
 GB: 0
 PC: 1.00

EB0496-1

V1 W111111 V1

AKWEISZ

220 200 180 160 140 120 100 80 60 40 20 ppm

Current Data Parameters
 NAME EB0496-1
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040504
 Time_ 12.07
 INSTRUM spect
 PROBHD 5 mm BBO 1H/1
 PULPROG zgpg30
 TD 32768
 SFO1 327.68 MHz
 SOLVENT CDCl3
 NS 34
 DS 4
 SWH 375.91984 MHz
 FIDRES 1.147277 Hz
 AQ 0.4358777 sec
 RG 18390.4
 RW 13.360 MHz
 DE 6.00 MHz
 TE 297.2 K
 DQ 2.0000000 sec
 DE 0.0300000 sec
 ACQRES 0.0000000 sec
 MCNMR 0.0150000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 4.00 MHz
 PL 0.00 dB
 SFO1 150.9201628 MHz
 ===== CHANNEL f2 =====
 CPOBPR2 waltz16
 NUC2 1H
 PCPD2 100.00 MHz
 PL2 120.00 dB
 PL12 15.00 dB
 SFO2 600.128051 MHz
 F2 Processing Parameters
 SI 32768
 GF 150.9024957 MHz
 MDW 0
 SSB 0
 LB 2.00 Hz
 CR 0
 PC 1.00

LB0517-1

AL WEISZ

Current Data Parameters	
NAME	VALUE
EXPNO	2
PROCNO	1

P2 - Acquisition Parameters
Date 20040512

Time	Temperature
11.54	11.54

INSTRUM 5 mm PBO 13C 1
PROBHD

PUF.P803 29dc
TJ 1768

INITIATOR	SOLVENT
7.5 (0.01)	7.5 (0.01)

320

PMG, 1.65 L.L.

PLURIM	1.147277
AQ	0.4358777

RG 16384
17 300

6.00

TR	296.1
71	2.00200000

0.036000000
YTD

MILESST	0.00000000
MCMRZ	0.01500000

1999, 2000, 2001, 2002, 2003, 2004, 2005, 2006, 2007, 2008, 2009, 2010, 2011, 2012, 2013, 2014, 2015, 2016, 2017, 2018, 2019, 2020, 2021, 2022, 2023, 2024, 2025, 2026, 2027, 2028, 2029, 2030, 2031, 2032, 2033, 2034, 2035, 2036, 2037, 2038, 2039, 2040, 2041, 2042, 2043, 2044, 2045, 2046, 2047, 2048, 2049, 2050, 2051, 2052, 2053, 2054, 2055, 2056, 2057, 2058, 2059, 2060, 2061, 2062, 2063, 2064, 2065, 2066, 2067, 2068, 2069, 2070, 2071, 2072, 2073, 2074, 2075, 2076, 2077, 2078, 2079, 2080, 2081, 2082, 2083, 2084, 2085, 2086, 2087, 2088, 2089, 2090, 2091, 2092, 2093, 2094, 2095, 2096, 2097, 2098, 2099, 2100, 2101, 2102, 2103, 2104, 2105, 2106, 2107, 2108, 2109, 2110, 2111, 2112, 2113, 2114, 2115, 2116, 2117, 2118, 2119, 2120, 2121, 2122, 2123, 2124, 2125, 2126, 2127, 2128, 2129, 2130, 2131, 2132, 2133, 2134, 2135, 2136, 2137, 2138, 2139, 2140, 2141, 2142, 2143, 2144, 2145, 2146, 2147, 2148, 2149, 2150, 2151, 2152, 2153, 2154, 2155, 2156, 2157, 2158, 2159, 2160, 2161, 2162, 2163, 2164, 2165, 2166, 2167, 2168, 2169, 2170, 2171, 2172, 2173, 2174, 2175, 2176, 2177, 2178, 2179, 2180, 2181, 2182, 2183, 2184, 2185, 2186, 2187, 2188, 2189, 2190, 2191, 2192, 2193, 2194, 2195, 2196, 2197, 2198, 2199, 2200, 2201, 2202, 2203, 2204, 2205, 2206, 2207, 2208, 2209, 2210, 2211, 2212, 2213, 2214, 2215, 2216, 2217, 2218, 2219, 2220, 2221, 2222, 2223, 2224, 2225, 2226, 2227, 2228, 2229, 2230, 2231, 2232, 2233, 2234, 2235, 2236, 2237, 2238, 2239, 2240, 2241, 2242, 2243, 2244, 2245, 2246, 2247, 2248, 2249, 2250, 2251, 2252, 2253, 2254, 2255, 2256, 2257, 2258, 2259, 2260, 2261, 2262, 2263, 2264, 2265, 2266, 2267, 2268, 2269, 2270, 2271, 2272, 2273, 2274, 2275, 2276, 2277, 2278, 2279, 2280, 2281, 2282, 2283, 2284, 2285, 2286, 2287, 2288, 2289, 2290, 2291, 2292, 2293, 2294, 2295, 2296, 2297, 2298, 2299, 2300, 2301, 2302, 2303, 2304, 2305, 2306, 2307, 2308, 2309, 2310, 2311, 2312, 2313, 2314, 2315, 2316, 2317, 2318, 2319, 2320, 2321, 2322, 2323, 2324, 2325, 2326, 2327, 2328, 2329, 2330, 2331, 2332, 2333, 2334, 2335, 2336, 2337, 2338, 2339, 2340, 2341, 2342, 2343, 2344, 2345, 2346, 2347, 2348, 2349, 2350, 2351, 2352, 2353, 2354, 2355, 2356, 2357, 2358, 2359, 2360, 2361, 2362, 2363, 2364, 2365, 2366, 2367, 2368, 2369, 2370, 2371, 2372, 2373, 2374, 2375, 2376, 2377, 2378, 2379, 2380, 2381, 2382, 2383, 2384, 2385, 2386, 2387, 2388, 2389, 2390, 2391, 2392, 2393, 2394, 2395, 2396, 2397, 2398, 2399, 2400, 2401, 2402, 2403, 2404, 2405, 2406, 2407, 2408, 2409, 2410, 2411, 2412, 2413, 2414, 2415, 2416, 2417, 2418, 2419, 2420, 2421, 2422, 2423, 2424, 2425, 2426, 2427, 2428, 2429, 2430, 2431, 2432, 2433, 2434, 2435, 2436, 2437, 2438, 2439, 2440, 2441, 2442, 2443, 2444, 2445, 2446, 2447, 2448, 2449, 2450, 2451, 2452, 2453, 2454, 2455, 2456, 2457, 2458, 2459, 2460, 2461, 2462, 2463, 2464, 2465, 2466, 2467, 2468, 2469, 2470, 2471, 2472, 2473, 2474, 2475, 2476, 2477, 2478, 2479, 2480, 2481, 2482, 2483, 2484, 2485, 2486, 2487, 2488, 2489, 2490, 2491, 2492, 2493, 2494, 2495, 2496, 2497, 2498, 2499, 2500, 2501, 2502, 2503, 2504, 2505, 2506, 2507, 2508, 2509, 2510, 2511, 2512, 2513, 2514, 2515, 2516, 2517, 2518, 2519, 2520, 2521, 2522, 2523, 2524, 2525, 2526, 2527, 2528, 2529, 2530, 2531, 2532, 2533, 2534, 2535, 2536, 2537, 2538, 2539, 2540, 2541, 2542, 2543, 2544, 2545, 2546, 2547, 2548, 2549, 2550, 2551, 2552, 2553, 2554, 2555, 2556, 2557, 2558, 2559, 2560, 2561, 2562, 2563, 2564, 2565, 2566, 2567, 2568, 2569, 2570, 2571, 2572, 2573, 2574, 2575, 2576, 2577, 2578, 2579, 2580, 2581, 2582, 2583, 2584, 2585, 2586, 2587, 2588, 2589, 2590, 2591, 2592, 2593, 2594, 2595, 2596, 2597, 2598, 2599, 2600, 2601, 2602, 2603, 2604, 2605, 2606, 2607, 2608, 2609, 2610, 2611, 2612, 2613, 2614, 2615, 2616, 2617, 2618, 2619, 2620, 2621, 2622, 2623, 2624, 2625, 2626, 2627, 2628, 2629, 2630, 2631, 2632, 2633, 2634, 2635, 2636, 2637, 2638, 2639, 2640, 2641, 2642, 2643, 2644, 2645, 2646, 2647, 2648, 2649, 2650, 2651, 2652, 2653, 2654, 2655, 2656, 2657, 2658, 2659, 2660, 2661, 2662, 2663, 2664, 2665, 2666, 2667, 2668, 2669, 2670, 2671, 2672, 2673, 2674, 2675, 2676, 2677, 2678, 2679, 2680, 26

13C

	4.00	4.00
P1	4.00	4.00
P13	4.00	4.00

SP01 150.9261626

----- CHANNEL 42 -----

CHDFHC2 2014.2.16

	100
PCEPD2	100.00
PCPD2	100.00

PL2	120.00
PL3	15.00

600.132853

F2 Processing parameters

32768 75

Size	Weight
230	2.5 g
250	2.5 g
270	2.5 g
290	2.5 g
310	2.5 g
 
2370	2.5 g
2390	2.5 g
2410	2.5 g
2430	2.5 g
2450	2.5 g
2470	2.5 g
2490	2.5 g
2510	2.5 g
2530	2.5 g
2550	2.5 g
2570	2.5 g
2590	2.5 g
2610	2.5 g
2630	2.5 g
2650	2.5 g
2670	2.5 g
2690	2.5 g
2710	2.5 g
2730	2.5 g
2750	2.5 g
2770	2.5 g
2790	2.5 g
2810	2.5 g
2830	2.5 g
2850	2.5 g
2870	2.5 g
2890	2.5 g
2910	2.5 g
2930	2.5 g
2950	2.5 g
2970	2.5 g
2990	2.5 g
3010	2.5 g
3030	2.5 g
3050	2.5 g
3070	2.5 g
3090	2.5 g
3110	2.5 g
3130	2.5 g
3150	2.5 g
3170	2.5 g
3190	2.5 g
3210	2.5 g
3230	2.5 g
3250	2.5 g

COLL	TR
0	0
0	0
0	0

CP

1.00
E

EB0497-2

Current Data Parameters
NAME EB0497 2
EXTNO 2
PRGRNO 1

F2 - Acquisition Parameters
Date_ 20040506
Time_ 13.34
INSTRUM spect
PROBHD 5 mm TBI 1H 41
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 37553.084 Hz
FIDUCS 1.147277 Hz
AQ 0.4358777 sec
RG 4597.6
DE 1.100 umet
TE 297.2 K
O1 2.0000000 sec
O11 0.0100000 sec
MIXTURE 0.0000000 sec
MIXPRG 0.0150000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 4.00 umet
PL1 0.00 dB
SFO1 150.9207632 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
EXCPO2 100.00 umet
PL2 120.00 dB
PL12 20.30 dB
SFO2 600.1328051 MHz

F2 Processing parameters
SI 32768
SF 150.9025038 MHz
NUC 1H
HNB 0
IR 2.00 Hz
GB 0
PC 1.00

(35)

Current Data Parameters

NAME EB0522-1

EXPNO 2

PROCNO 1

F2 - Acquisition Parameters

Date_ 20040512

Time 12.13

INSTRUM spect

PROBHD 5 mm 980 13C-1

PULPROG zgpg

TD 32768

SOLVENT CDCl3

NS 16

DS 4

SWH 37593.584 Hz

FIDRES 0.144793 Hz

AQ 0.158777 sec

RG 32768

DE 13.100 usec

TE 255.4 K

DE 6.00 usec

DI 2.0000000 sec

d11 0.0300000 sec

MCREST 0.0000000 sec

MCWEX 0.01500000 sec

***** CHANNEL f1 *****

NUC1 13C

P1 4.00 usec

PL1 0.00 dB

SFO1 150.9201628 MHz

***** CHANNEL f2 *****

CHPROG2 waitz16

NUC2 13C

P2 100.00 usec

PL2 12.00 dB

SFO2 600.1328053 MHz

F2 - Processing parameters

SI 32768

SP 150.9024957 MHz

WDW EM

SSB 0

LB 2.00 Hz

GB 0

PC 1.00

ALY
WEISZ

AKWEISZ

Current Data Parameters
NAME EB0516-1
EXPNO 2
PROCNO 1
F2 Acquisition Parameters
Date_ 20040512
Time 12.23
INSTRUM spect
PROBHD 5 mm BBO 1H-1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 37591.064 Hz
FIDRES 1.14277 Hz
AQ 0.435877 sec
RG 327.68
DD 13.100 usec
DE 1.500 usec
TE 296.5 K
D1 2.0000000 sec
d11 0.0300000 sec
MCREST 0.0000000 sec
MCPRK 6.0150000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 4.00 usec
PL1 0.00 dB
SFO1 100.6201628 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 100.00 usec
PL2 120.00 dB
SFO2 400.1328051 MHz

F2 - Processing parameters
SI 32768
SF 100.6201628 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

EB0495-2

AKWEISZ

3w

Current Data Parameters
NAME: EB0495-2
EXPNO: 1
PROCNO: 1
F2 Acquisition Parameters
Date_ : 20040905
Time : 09:57
INSTRUM : spect
PROBHD : 5 mm TBI 1H 31
PULPROG : zgpg30
TD : 32768
SOLVENT : CDCl3
NS : 33
DS : 0
SWH : 37593.984 Hz
FTHRES : 1.147277 Hz
AQ : 0.4458777 sec
RG : 4597.6
TAU : 13.300 sec
TE : 297.0 K
DE : 2.00000000 sec
D1 : 0.03000000 sec
D11 : 0.00000000 sec
MCRSCT : 0.00000000 sec
MCRMZ : 0.07500000 sec
----- CHANNEL f1 -----
NUC1 : 13C
P1 : 4.00 usec
PL1 : 0.00 dB
SFO1 : 150.9207000 MHz
----- CHANNEL f2 -----
CHUMP2 : 13C
NUC2 : 13C
PCPU2 : 100.00 usec
PL2 : 120.00 dB
PL12 : 20.10 dB
SFO2 : 600.1320000 MHz
F2 - Processing parameters
SI : 32768
SF : 150.9026018 MHz
WDW : EM
SSB : 0
L3 : 2.00 Hz
GB : 0
PC : 1.00

Ernst-Moritz Arndt-Universität Greifswald
 ARX 300 Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbereichskoppelt)
 E-Beitar: EH0577-2
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: TMS = 0.0 ppm
 Mess Temperatur: 298 K (25 °C)

Current Data Parameters
 NAME: 6e, EH0577-2
 EXPNO: 15
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_ Time: 20041109
 Time: 15.49
 INSTRUM: spect
 PROCIID: 5 mm Multinu
 PULPROG: zgpg
 TD: 32768
 SOLVENT: CDCl₃
 NS: 512
 DS: 0
 SWH: 21739.151 Hz
 FIDRES: 0.663426 Hz
 AQ: 0.7537140 sec
 RG: 32768
 DW: 23.600 usec
 DE: 32.86 usec
 TE: 298.0 K
 D12: 0.00002000 sec
 D16: 30.00 usec
 D1: 2.00000000 sec
 C1PDPRG: waltz16
 P31: 110.00 usec
 D11: 0.03000000 sec
 D15: 22.00 usec
 P1: 2.50 usec
 SFO1: 75.476177 MHz
 NUC13: ¹³C
 F2 - Processing parameters
 SI: 16384
 SF: 75.627480 MHz
 WDW: EM
 SSB: 0
 LB: 1.20 Hz
 GB: 0
 PC: 1.00

EB0471-1

220 200 180 160 140 120 100 80 60 40 20 ppm

Current Data Parameters
NAME: EB0471-1
EXTNO: 2
PROCNO: 1

F2 Acquisition Parameters
Date: 20050423
Time: 13.52
INSTRUM: spect
PROBHD: 5 mm TBI J1 11
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 256
DS: 0
SWH: 37691.984 Hz
FIDRES: 0.1317277 Hz
AQ: 0.4258777 sec
RG: 2293.8
DM: 13.300 umm
DE: 6.00 usec
TE: 296.9 K
DT: 2.06000000 sec
d11: 0.03000000 sec
MCREST: 0.00000000 sec
MCWPRG: 0.01500000 sec

===== CHANNEL f1 =====
NUC1: 13C
P1: 4.00 usec
PL1: 0.00 dB
SFO1: 125.9201628 MHz

----- CHANNEL f2 -----
CPFGP2: waltz16
NUC2: 1H
P1PFG2: 100.00 usec
PL2: 120.00 dB
PL12: 20.35 dB
SFO2: 600.1324651 MHz

F2 Processing parameters
SI: 32768
SF: 150.9025125 MHz
WDW: RM
SSB: 0
LB: 2.00 Hz
GB: 0
PC: 1.00

AKWEISZ

Hmsl-Moritz-Arnold-Universität Greifswald
 ARX 300 – Kernresonanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbreitbandentkoppelt)
 H: Bohlur: H10520-1
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Messtemperatur: 298 K (25 °C)

Current Data Parameters
 NAME: BL_10520-1
 EXPNO: 1
 PROCNO: 1
 USER: lang

F2: Acquisition Parameters
 Date_: 20040914
 Time: 12.20
 INSTRUM: spect
 PROBHD: 5 mm Moltum
 PULPROG: zgpg
 TD: 32768
 SOLVENT: CDCl₃
 NS: 640
 DS: 0
 SWH: 21739.131 Hz
 FIDRES: 0.6634261 Hz
 AQ: 0.7837140 sec
 RG: 32768
 DW: 23.800 usec
 DE: 32.86 usec
 TE: 298.0 K
 D12: 0.0000200 sec
 D16: 30.00 dB
 D1: 2.00000000 sec
 CPMPCPG: waltz16
 P31: 110.00 usec
 D11: 0.03000000 sec
 D1.5: 22.00 dB
 P1: 3.00 usec
 SE-OT: 75.4761177 MHz
 NUC1: 13C

F2: Processing parameters
 SI: 16384
 SF: 75.467512 MHz
 WDW: EM
 SSB: 0
 LB: 1.20 Hz
 GB: 0
 PC: 0.80

Ernst-Moritz-Arnt-Universität Greifswald
 AXX300 - Kernspinanz-Spektrometer
¹³C-NMR-Spektrum (Protonenbandenkoppelung)
 E. Bellur: EB595-2b
 Lösungsmittel: Deuteriochloroform, CDCl₃
 Referenz: Lösungsmittel = 77.0 ppm
 Mess Temperatur: 298 K (25 °C)

Current Data Parameters
 NAME: BE_EB595-2b
 EXPNO: 13
 PROCNO: 1
 USER: lang

F2 - Acquisition Parameters

Date: 20040921
 Time: 13.44
 INSTRUM: spect
 PROBHD: 5 mm Maltinu
 PULPROG: zgpg30
 TD: 32768
 SOLVENT: CDCl₃
 NS: 512
 DS: 0
 SWH: 21739.131 Hz
 FIDRES: 0.665426 Hz
 AQ: 0.7537140 sec
 RG: 32768
 DW: 23.000 usec
 DE: 32.85 usec
 TE: 298.0 K
 D12: 0.00002000 sec
 DL6: 30.00 dB
 D1: 2.00000000 sec
 CPDPRG: waltz16
 P31: 110.00 usec
 D11: 0.03000000 sec
 DL5: 22.00 dB
 P1: 2.50 usec
 SFO1: 75.4761177 MHz
 NUCLEUS: ¹³C

F2 - Processing parameters

SI: 16384
 SF: 75.4677525 MHz
 WDW: EM
 SSB: 0
 LB: 1.20 Hz
 GB: 0
 PC: 0.90

EB0562-1

Current Data Parameters
NAME EB0562-1
EXPNO 2
PROCNO 1

F2: Acquisition Parameters
Date_ 20040609
Time 18.13

INSTRUM spect
PROBHD 5 mm CP1 1H 11

PULPROG zgpgc
TD 32768

TD 32768
SOLVENT CDCl3

NS 130
DS 0

SMH 37593.984 Hz
FIDRES 1.147277 Hz

AQ 0.4558777 sec
RG 1625.5

EM 15.800 usec
TE 25.53 K

DE 6.00 usec
WE 2.00 usec

TI 3.0000000 sec
C11 0.01300000 sec

MCBAST 0.00500000 usec
MCMER 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 4.00 usec
PL1 0.00 dB

SFO1 150.9201628 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16

NUC2 1H
P2 100.00 usec
PL2 120.00 dB

P112 20.30 dB
SFO2 600.1340000 MHz

F2: Processing parameters
SC 32768

SP 150.9027602 MHz
WDW EM

SSB 0
GB 0

HN 2.00 Hz
HC 1.00

7d

EB0596-1

7e

3c

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WEISZ

CONTROL DATA INSTRUMENTS
 NAME: FRODOG 1
 EXNO: 2
 PROGNO: 2
 12 Acquisition Parameters
 FID: 200405.1
 TIME: 10.7
 INSTRUM: SPECT
 PRGMD: 5 WE TUI II 15
 PCTPROG: 9999
 TD: 32768
 BRDMKT: CUC13
 NS: 512
 DS: 0
 DMH: 40963.505 Hz
 FIDRES: 1.210720 Hz
 AQ: 0.1978178 sec
 SFO: 200.13009 MHz
 SC: 15.200 usoc
 SW: 20.100 MHz
 TR: 2.0000000 sec
 TR2: 2.0000000 sec
 D1: 1.0100000 sec
 d11: 0.0200000 sec
 MEASST: 0.0100000 sec
 MMSK: 0.0100000 sec
 ----- CHANNEL F1 -----
 NUC1: 13C
 P1: 4.00 usoc
 PL1: 0.30 dB
 SFO1: 100.626100 MHz
 ----- CHANNEL F2 -----
 NUC2: 1H
 P2: 100.00 usoc
 PL2: 10.00 dB
 SFO2: 20.100000 MHz
 SFO2: 500.135009 MHz
 P2: Processing Parameters
 SI: 32768
 SF: 100.626100 MHz
 SSB: 0
 LSSB: 2.00 Hz
 GB: 1.00
 PC: 1.00