ACYLATIVE DESYMMETRIZATION OF GLYCEROL DERIVATIVES BY CHIRAL DMAP DERIVATIVES

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Supporting Information

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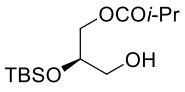
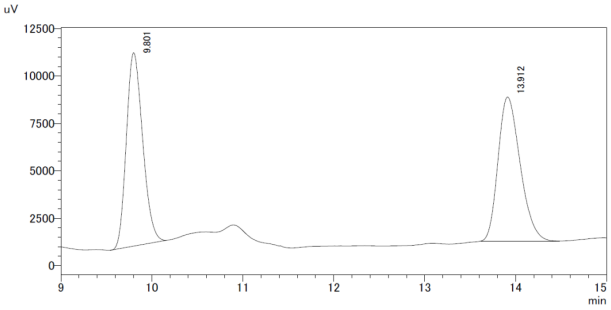
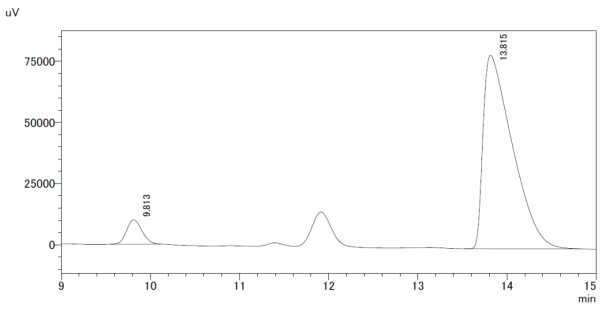
Characterization of the products 3a–e and 4a and 4c–e

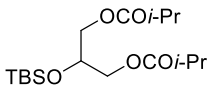
(*R*)-2-((*tert*-butyldimethylsilyl)oxy)-3-hydroxypropyl isobutyrate (3a)



According to general procedure, substrate **2a** (41.3 mg, 0.200 mmol) with a catalyst **1e** (0.2 μmol), Et_3N (30.6 μL, 0.220 mmol), and $(i\text{-PrCO})_2O$ (36.4 μL, 0.220 mmol) in toluene (4 mL) at –60 ˚C gave **3a** (47.5 mg, 0.172 mmol, 86% yield, 94:6 er), diacylate **4a** (2.9 mg, 0.00837 mmol, 4% yield) and recovered **2a** (1.8 mg, 0.0872 mmol, 4% yield). Colorless liquid. Enantiomeric ratio was determined by HPLC with DAICEL CHIRALPAK® IB N-3 (hexane/*i*-PrOH = 98/2, v/v, flow rate = 0.5 mL/min, 30 °C, UV = 205 nm), T_R = 9.8 min (minor) and T_R = 13.8 min (major), 94:6 er; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.14–4.03 (m, 2H), 3.93 (quin, *J* = 5.0 Hz, 1H), 3.65–3.51 (m, 2H), 2.56 (sep, *J* = 6.9 Hz, 1H), 1.96 (dd, *J* = 7.1, 5.7 Hz 1H), 1.18 (d, *J* = 6.9 Hz, 6H), 0.91 (s, 9H), 0.12 (s, 3H), 0.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.2, 70.7, 64.9, 64.0, 34.1, 25.9, 19.1 (2), 18.1, –4.6, –4.7; IR (neat) 3460, 2932, 1732, 1472, 1258, 1194 cm^{-1}; HRMS (ESI$^+$) $[M+Na]^+$ calculated for $C_{13}H_{28}O_4NaSi$ 299.1649, found 299.1649; $[\alpha]^{20}_D$ –10.5 (*c* 0.99, $CHCl_3$, 94:6 er).

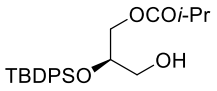
Racemic sample of **3a**				**(*R*)-3a**			
							
Peak	**Ret. Time**	**Area**	**Area %**	**Peak**	**Ret. Time**	**Area**	**Area %**
1	9.801	126588	49.659	1	9.813	118820	6.173
2	13.912	128328	50.341	2	13.815	1805883	93.827

2-((*tert*-butyldimethylsilyl)oxy)propane-1,3-diyl bis(2-methylpropanoate) (4a)

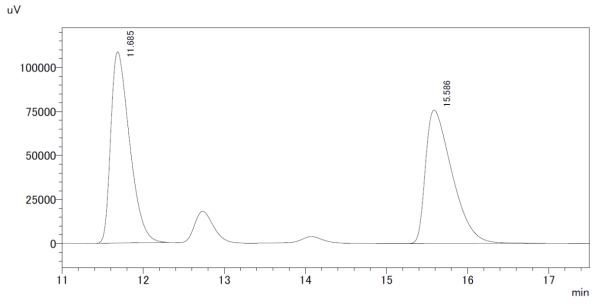
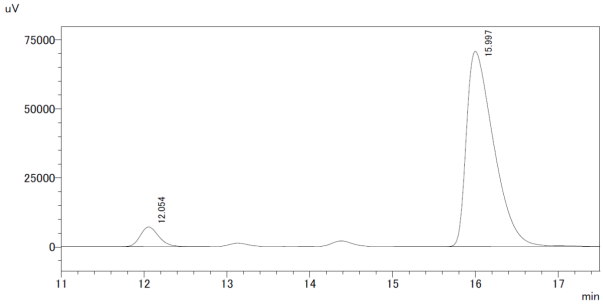


Colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.16–3.97 (m, 5H), 2.56 (sep, *J* = 6.9 Hz, 2H), 1.17 (d, *J* = 6.9 Hz, 12H), 0.88 (s, 9H), 0.10 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 68.5, 65.4, 34.1, 25.8, 19.1 (2), 18.1, –4.7; IR (neat) 2959, 1738, 1472, 1256, 1153 cm^{-1}; HRMS (ESI$^+$) $[M+Na]^+$ calculated for $C_{20}H_{24}O_3Na$ 369.2068, found 369.2055.

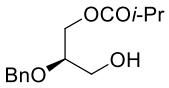
(*R*)- 2-((*tert*-butyldiphenylsilyl)oxy)-3-hydroxypropyl isobutyrate (3b)



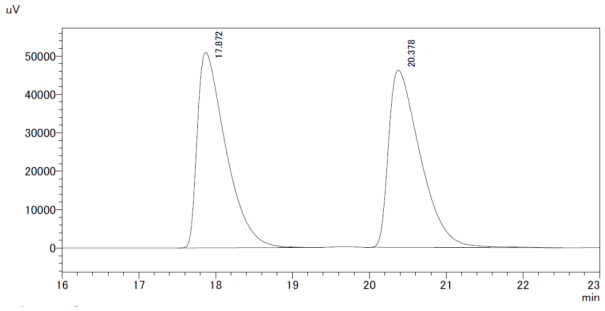
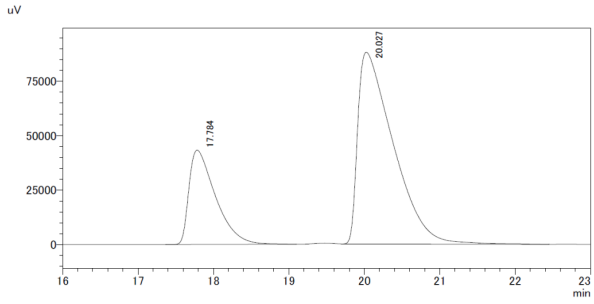
According to general procedure, substrate **2b** (66.1 mg, 0.200 mmol) with a catalyst **1e** (0.2 μmol), Et_3N (30.6 μL, 0.220 mmol), and $(i\text{-PrCO})_2O$ (36.4 μL, 0.220 mmol) in toluene (4 mL) at –60 ˚C gave **3b** (56.0 mg, 0.140 mmol, 70% yield, 94:6 er) and recovered **2b** (19.8 mg, 0.0599 mmol, 30% yield). Yellow liquid. Enantiomeric ratio was determined by HPLC with DAICEL CHIRALPAK® IB N-3 (hexane/*i*-PrOH = 97/3, v/v, flow rate = 0.5 mL/min, 30 °C, UV = 254 nm), T_R = 12.1 min (minor) and T_R = 16.0 min (major), 94:6 er; ^{1}H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 7.73–7.63 (m, 4H), 7.49–7.34 (m, 6H), 4.15 (dd, *J* = 11.4, 5.0 Hz, 1H), 4.06 (dd, *J* = 11.4, 5.0 Hz, 1H), 3.95 (quin, *J* = 5.0 Hz, 1H), 3.54 (bs, 2H), 2.42 (sep, *J* = 7.3 Hz, 1H), 1.97 (bs, 1H), 1.10 (d, *J* = 7.3 Hz, 3H), 1.10 (d, *J* = 7.3 Hz, 3H), 1.08 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 177.3, 135.9, 135.8, 133.4, 130.1, 130.0, 128.0, 127.8, 71.8, 64.7, 63.6, 34.0, 27.0, 19.4, 19.1, 19.0; IR (neat) 3472, 2932, 1736, 1472, 1427 cm^{-1}; HRMS (ESI$^+$) $[M+Na]^+$ calculated for $C_{23}H_{32}O_4NaSi$ 423.1962, found 423.1973; $[\alpha]^{23}_D$ –8.0 (*c* 0.97, $CHCl_3$, 94:6 er).

Racemic sample of **3b**				**(*R*)-3b**			
							
Peak	**Ret. Time**	**Area**	**Area %**	**Peak**	**Ret. Time**	**Area**	**Area %**
1	11.685	1751443	50.351	1	12.054	110970	6.412
2	15.586	1727032	49.649	2	15.997	1619787	93.588

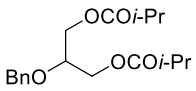
(*R*)- 2-(benzyloxy)-3-hydroxypropyl isobutyrate (3c)



According to general procedure, substrate **2c**[1] (36.4 mg, 0.200 mmol) with a catalyst **1e** (0.2 μmol), Et_3N (30.6 μL, 0.220 mmol), and $(i\text{-PrCO})_2O$ (36.4 μL, 0.220 mmol) in toluene (4 mL) at –60 ˚C gave **3c** (40.3 mg, 0.160 mmol, 80% yield, 73:27 er), diacylate **4a** (3.0 mg, 0.00931 mmol, 5% yield) and recovered **2c** (4.8 mg, 0.0263 mmol, 13% yield). Colorless liquid. Enantiomeric ratio was determined by HPLC with DAICEL CHIRALPAK® IB N-3 (hexane/*i*-PrOH = 95/5, v/v, flow rate = 0.5 mL/min, 30 °C, UV = 254 nm), T_R = 17.8 min (minor) and T_R = 20.0 min (major), 73:27 er; ^{1}H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 7.41–7.28 (m, 5H), 4.73 (d, *J* = 11.4 Hz, 1H), 4.61 (d, *J* = 11.4 Hz, 1H), 4.24 (d, *J* = 5.0 Hz, 2H), 3.78–3.68 (m, 2H), 3.68–3.59 (m, 1H), 2.58 (sep, *J* = 7.0 Hz, 1H), 2.07–1.97 (m, 1H), 1.18 (d, *J* = 7.0 Hz, 3H), 1.18 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 177.2, 138.0, 128.6, 128.0, 127.9, 72.3, 62.9, 62.1, 34.0, 19.1; IR (neat) 3458, 2974, 2878, 1742, 1454, 1061 cm^{-1}; HRMS (ESI^+) $[M+Na]^+$ calculated for $C_{20}H_{24}O_3Na$ 275.1254, found 274.1243; $[\alpha]^{22}_D$ –24.3 (*c* 0.98, $CHCl_3$, 73:27 er).

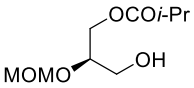
Racemic sample of **3c**				**(*R*)-3c**			
							
Peak	**Ret. Time**	**Area**	**Area %**	**Peak**	**Ret. Time**	**Area**	**Area %**
1	17.872	1310915	49.718	1	17.784	1058367	26.881
2	20.378	1325797	50.282	2	20.027	2878831	73.119

2-(benzyloxy)propane-1,3-diyl bis(2-methylpropanoate) (3c)

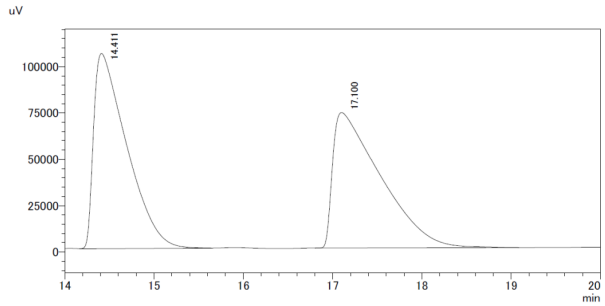
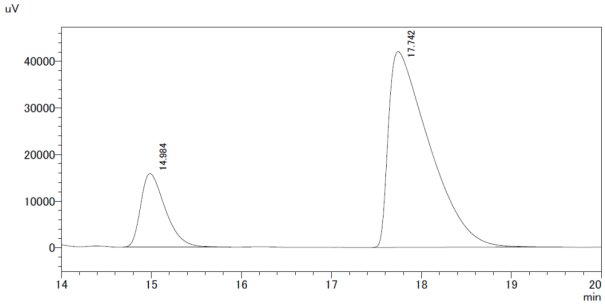


Colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 7.40–7.27 (m, 5H), 4.66 (s, 2H), 4.26 (dd, *J* = 11.5, 5.1 Hz, 2H), 4.16 (dd, *J* = 11.5, 5.1 Hz, 2H), 3.84 (quin, *J* = 5.1 Hz, 1H), 2.57 (sep, *J* = 7.0 Hz, 2H), 1.18 (d, *J* = 7.0 Hz, 6H), 1.17 (d, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 177.0, 138.0, 128.6, 128.0, 127.9, 74.7, 72.2, 63.2, 34.1, 19.1 (2); IR (neat) 2974, 1730, 1472, 1389, 1155 cm^{-1}; HRMS (ESI^+) $[M+Na]^+$ calculated for $C_{20}H_{24}O_3Na$ 345.1672, found 345.1672.

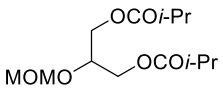
(*R*)-3-hydroxy-2-(methoxymethoxy)propyl isobutyrate (3d)



According to general procedure, substrate **2d** (27.2 mg, 0.200 mmol) with a catalyst **1e** (0.2 μmol), Et_3N (30.6 μL, 0.220 mmol), and $(i\text{-PrCO})_2O$ (36.4 μL, 0.220 mmol) in toluene (4 mL) at –60 ˚C gave **3d** (27.6 mg, 0.134 mmol, 67% yield, 82:18 er), diacylate **4d** (6.4 mg, 0.0232 mmol, 12% yield) and recovered **2d** (5.4 mg, 0.0397 mmol,20% yield). Colorless liquid. Enantiomeric ratio was determined by HPLC with DAICEL CHIRALPAK® IB N-3 (hexane/*i*-PrOH = 95/5, v/v, flow rate = 0.5 mL/min, 30 °C, UV = 205 nm), T_R = 15.0 min (minor) and T_R = 17.7 min (major), 82:18 er; ^{1}H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 4.78 (s, 2H), 4.23 (dd, *J* = 11.4, 5.5 Hz, 1H), 4.17 (dd, *J* = 11.7, 5.5 Hz, 1H), 3.89–3.79 (m, 1H), 3.69 (dd, *J* = 11.9, 3.2 Hz, 1H), 3.62 (dd, *J* = 11.9, 6.4 Hz, 1H), 3.43 (s, 3H), 2.73–2.50 (m, 2H), 1.18 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 177.2, 96.7, 77.8, 63.4, 62.7, 55.8, 34.0, 19.1, 19.0; IR (neat) 3445, 2974, 1732, 1472, 1032 cm^{-1}; HRMS (FAB^+) $[M+Na]^+$ calculated for $C_9H_{18}O_5Na$ 229.1046, found 229.1055; $[\alpha]^{20}_D$ –22.6 (*c* 0.98, $CHCl_3$, 82:18 er).

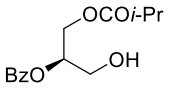
Racemic sample of 3**d**				**(*R*)-3d**			
							
Peak	**Ret. Time**	**Area**	**Area %**	**Peak**	**Ret. Time**	**Area**	**Area %**
1	14.411	2709205	49.882	1	14.984	299076	18.392
2	17.100	2722023	50.118	2	17.742	1327041	81.608

2-(methoxymethoxy)propane-1,3-diyl bis(2-methylpropanoate) (4d)

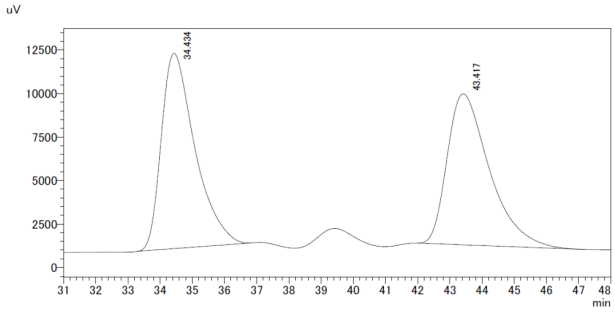
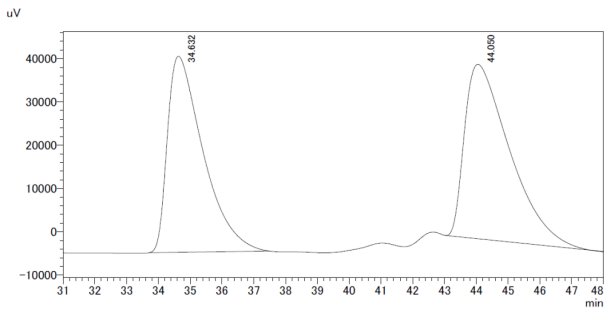


Colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 4.72 (s, 2H), 4.24 (dd, *J* = 11.7, 5.2 Hz, 2H), 4.15 (dd, *J* = 11.7, 5.2 Hz, 2H), 4.02 (quin, *J* = 5.2 Hz, 1H), 3.40 (s, 3H), 2.58 (sep, *J* = 7.3 Hz, 2H), 1.18 (d, *J* = 7.3 Hz, 12H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 176.9, 96.1, 72.8, 63.4, 55.7, 34.0, 19.1, 19.0; IR (neat) 3649, 2976, 1734, 1558, 1456, 1034 cm^{-1}; HRMS (FAB^+) $[M+Na]^+$ calculated for $C_{13}H_{24}O_6Na$ 299.1465, found 299.1478.

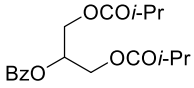
(*R*)- 1-hydroxy-3-(isobutyryloxy)propan-2-yl benzoate (3e)



According to general procedure, substrate **2e**[2] (39.2 mg, 0.200 mmol) with a catalyst **1e** (0.2 μmol), Et_3N (30.6 μL, 0.220 mmol), and $(i\text{-PrCO})_2O$ (36.4 μL, 0.220 mmol) in CPME (4 mL) at –60 ˚C gave **2e** (32.5 mg, 0.122 mmol, 61% yield, 53:47 er), diacylate **4e** (4.9 mg, 0.0146 mmol, 7% yield) and recovered **2e** (9.6 mg, 0.0489 mmol,24% yield). Colorless liquid. Enantiomeric ratio was determined by HPLC with DAICEL CHIRALCEL® OD-H (hexane/*i*-PrOH = 98/2, v/v, flow rate = 1.0 mL/min, 30 °C, UV = 254 nm), T_R = 34.6 min (minor) and T_R = 44.1 min (major), 53:47 er; ^{1}H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 8.05 (d, *J* = 7.3 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 5.34 (quin, *J* = 5.0 Hz, 1H), 4.46 (dd, *J* = 12.1, 5.0 Hz, 1H), 4.42 (dd, *J* = 12.1, 5.0 Hz, 1H), 3.88 (d, *J* = 5.0 Hz, 1H), 3.86 (d, *J* = 5.7 Hz, 1H), 2.59 (sep, *J* = 6.9 Hz, 1H), 2.16 (t, *J* = 5.7 Hz, 1H), 1.17 (d, *J* = 6.9 Hz, 3H), 1.16 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 177.2, 166.3, 133.5, 129.9, 129.7, 128.6, 73.2, 62.2, 61.6, 34.0, 19.0 (2); IR (neat) 3482, 2974, 2878, 1717, 1452 cm^{-1}; $[\alpha]^{24}_D$ +2.47 (*c* 0.98, $CHCl_3$, 53:47 er) (lit[3]. $[\alpha]^{20}_D$ –0.48 (*c* 1.0, $CHCl_3$, 58% ee, (*R*) configuration).

Racemic sample of 3**e**				**(*R*)-3e**			
							
Peak	**Ret. Time**	**Area**	**Area %**	**Peak**	**Ret. Time**	**Area**	**Area %**
1	34.434	813940	50.638	1	34.632	3535108	46.967
2	43.417	793418	49.362	2	44.050	3991701	53.033

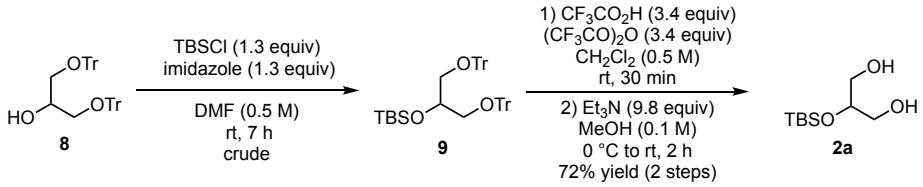
2-(benzoyloxy)propane-1,3-diyl bis(2-methylpropanoate) (4e)



Colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 8.07–7.99 (m, 2H), 7.62–7.55 (m, 1H), 7.49–7.42 (m, 2H), 5.58–5.51 (m, 1H), 4.40 (dd, *J* = 11.9, 4.1 Hz, 2H), 4.34 (dd, *J* = 11.9, 6.0 Hz, 2H), 2.57 (sep, *J* = 6.9 Hz, 2H), 1.16 (d, *J* = 6.9 Hz, 6H), 1.14 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 176.6, 166.7, 133.4, 129.8, 129.6, 128.5, 69.9, 62.1, 33.9, 19.0; IR (neat) 2976, 2878, 1744, 1719, 1472 cm^{-1}.

Synthesis of substrates 2a, 2b, and 2d

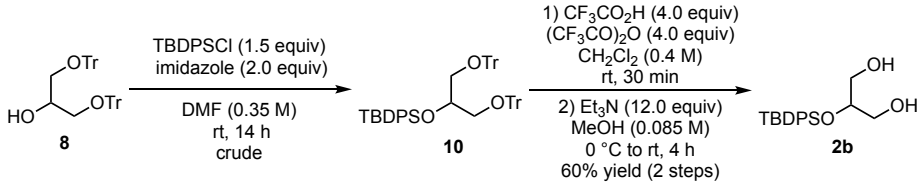
2-((*tert*-butyldimethylsilyl)oxy)propane-1,3-diol (2a)



To a solution of **8**[4] (5.76 g, 9.99 mmol) and imidazole (0.889 g, 13.1 mmol) in dry DMF (20.0 mL) was added TBSCl (1.97 g, 13.1 mmol) at rt. After stirred for 7 h, the reaction mixture was quenched with H_2O (100mL), then extracted with EtOAc (150 mL×2), washed with H_2O (100mL×2) and brine (100mL×1). The organic layer was dried over $MgSO_4$, concentrated *in vacuo* to give the crude product **9**. The crude product was used the next reaction without further purification.

To a solution of crude product **9** in CH_2Cl_2 (21.3 mL) was added CF_3CO_2H (2.60 mL, 34.0 mmol) and $(CF_3CO)_2O$ (4.80 mL, 34.1 mmol) at rt and the reaction mixture was stirred for 30 min. Then the reaction mixture was cooled at 0 °C and added Et_3N (14.2 mL, 102 mmol) and MeOH (100 mL). After stirred at rt for 2 h, the reaction mixture was evaporated under reduced pressure. Then the residue was dissolved in CH_2Cl_2 (100 mL) and washed with brine (100mL×1). The purification of the crude product by flash column chromatography on silica gel (eluent: hexane/EtOAc = 1:1, v/v) gave **2a** (1.49 g, 7.23 mmol, 72% yield (2 steps)) as a colorless solid. Detailed analytical data of **2a** was identical to the literature[5].

2-((*tert*-butyldiphenylsilyl)oxy)propane-1,3-diol (9b)

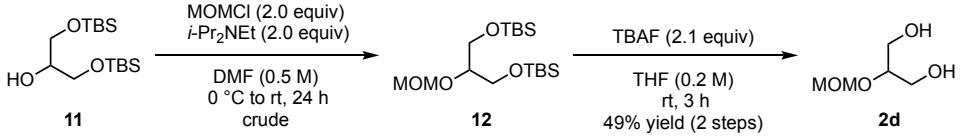


To a solution of **8** (2.88 g, 4.99 mmol) and imidazole (0.691 g, 10.2 mmol) in dry DMF (14.3 mL) was added TBDPSCl (2.00 mL, 7.69 mmol) at rt. After stirred for 14 h, the reaction mixture was quenched with H_2O (30mL), then extracted with EtOAc (50 mL×2), washed with H_2O (50mL×2) and brine (50mL×1). The organic layer was dried over $MgSO_4$, concentrated *in vacuo* to give the crude product **10**. The crude product was used the next reaction without further purification.

To a solution of crude product **10** in CH_2Cl_2 (12.5 mL) was added CF_3CO_2H (1.60 mL, 20.9 mmol) and $(CF_3CO)_2O$ (2.80 mL, 19.9 mmol) at rt and the reaction mixture was stirred for 30 min. Then the reaction mixture was cooled at 0 °C and added Et_3N (8.4 mL, 60.3 mmol) and MeOH (58.8 mL). After stirred at rt for 4 h, the reaction mixture was evaporated under reduced pressure. Then the residue was dissolved in CH_2Cl_2 (30 mL) and washed with brine (30mL ×1). The crude product was purified by flash column chromatography on silica gel (eluent: hexane/EtOAc = 3:1 to 1:1, v/v) followed by recrystallization from toluene/hexane gave **2b** (0.983 g, 2.97 mmol, 60% yield (2 steps)) as a colorless solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71–7.76 (m, 4H), 7.48–7.36 (m, 6H), 3.85 (quin, J = 4.8 Hz, 1H), 3.64 (d, J = 4.6 Hz, 2H), 3.63 (d, J = 4.6 Hz, 2H), 1.84–1.76 (m, 2H), 1.09 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.8, 133.5, 130.1, 128.0, 73.4, 64.1, 27.1, 19.4; IR (KBr) 3466, 2895, 1472, 1119, 1053 cm^{-1}; HRMS (ESI$^+$) $[M+Na]^+$ calculated for $C_{19}H_{26}O_3SiNa$

353.1543, found 353.1543; mp 75.5–76.2 °C.

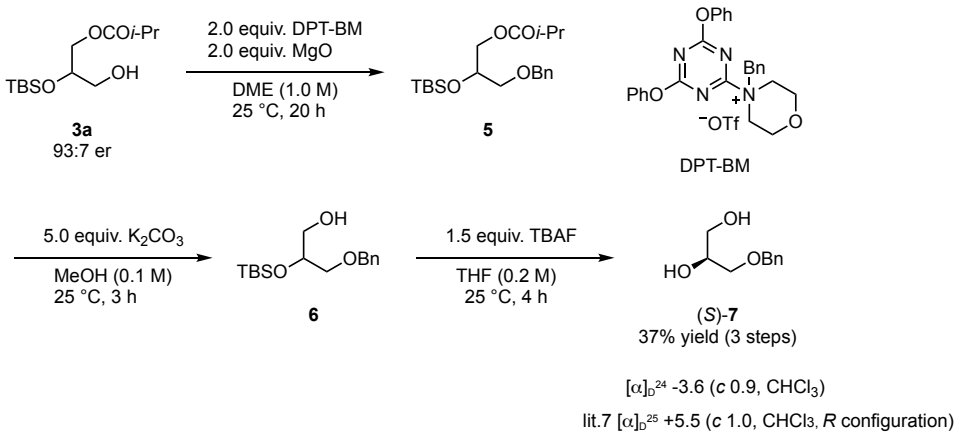
2-(methoxymethoxy)propane-1,3-diol (2d)



To a solution of **11**[6] (1.60 g, 4.99 mmol) and *i*-Pr_2NEt (1.74 mL, 9.99 mmol) in dry DMF (10.0 mL) was added MOMCl (0.760 mL, 10.0 mmol) at 0 °C. After stirred at rt for 24 h, the reaction mixture was quenched with H_2O (30mL), then extracted with Et_2O (40 mL×2), washed with H_2O (60mL×2). The organic layer was dried over $MgSO_4$, concentrated *in vacuo* to give the crude product **12**. The crude product was used the next reaction without further purification.

To a solution of crude product **12** in THF (11.2 mL) was added TBAF (1.0 M in THF, 10.3 mL, 10.3 mmol) at rt. After stirred at rt for 3 h, the reaction mixture was quenched with saturated aqueous NH_4Cl (30 mL), then extracted with EtOAc (40mL×3). The organic layer was dried over $MgSO_4$, concentrated *in vacuo*. However, the target compound **2d** was in aqueous layer, so the aqueous layer was concentrated *in vacuo* to give the crude product. The crude product was purified by flash column chromatography on silica gel (eluent: EtOAc to EtOAc/MeOH = 10:1 v/v) gave **2d** (0.331 g, 2.43 mmol, 49% yield (2 steps)) as a colorless liquid. ^1H NMR (400 MHz, $CDCl_3$, 0.03% TMS) δ 4.79 (s, 2H), 3.74–3.60 (m, 5H), 3.47 (s, 3H), 2.58–2.51 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, 0.03% TMS) δ 97.0, 81.7, 62.9, 55.8; IR (neat) 3381, 2891, 1466, 1213, 1026 cm^{-1}; HRMS (ESI^+) $[M+Na]^+$ calculated for $C_5H_{12}O_4Na$ 159.0628, found 159.0620.

Determination of the absolute configuration of 3a



To a solution of (*R*)-**3a** (83.0 mg, 0.300 mmol) in DME (0.3 mL) was added DPT-BM (259.5 mg, 0.597 mmol) and MgO (24.2 mg, 0.600 mmol) at rt. After stirred for 20 h, the reaction mixture was diluted with Et_2O and washed with saturated aqueous $NaHCO_3$ (10 mL×2), H_2O (10mL×1) and brine (10 mL×1). The organic layer was dried over $MgSO_4$, concentrated *in vacuo.* The residue was passed through a short pad of silica gel (eluent: hexane/EtOAc = 10:1, v/v) to give the crude product **5**. The crude product was used the next reaction without further purification.

To a solution of crude product **5** in MeOH (3.0 mL) was added K_2CO_3 (209 mg, 1.51 mmol) at room temperature. After stirred for 3 h, the reaction mixture was quenched with H_2O (5 mL) and extracted with EtOAc (10 mL×2). The organic layer was dried over $MgSO_4$, concentrated *in vacuo* to give the crude product **6**. The crude product was used the next reaction without further purification.

To a solution of crude product **6** in THF (1.5 mL) was added TBAF (1.0 M in THF, 0.450 mL, 0.450 mmol) at rt. After stirred at rt for 4 h, the reaction mixture was quenched with saturated aqueous NH_4Cl (10 mL), then extracted with EtOAc (10mL×3). The organic layer was dried over $MgSO_4$, concentrated *in vacuo* to give the crude product. The purification of the crude product by flash column chromatography on silica gel (eluent: hexane/EtOAc = 1:1 to 1:3, v/v) gave (*S*)-**7** (20.3 mg, 0.111 mmol, 37 % yield (3 steps)) as a colorless oil. Detailed analytical data of (*S*)-**7** was identical to the literature[7]. Enantiomeric ratio was determined by HPLC with DAICEL CHIRALPAK® IB N-3 (hexane/*i*-PrOH = 90/10, v/v, flow rate = 0.5 mL/min, 30 °C, UV = 254 nm), T_R = 16.3 min (major) and T_R = 19.2 min (minor), 93:7 er; $[\alpha]^{24}_D$ –3.6 (*c* 0.95, $CHCl_3$, 93:7 er). [lit.[7] $[\alpha]^{25}_D$ +5.5 (*c* 1.0, $CHCl_3$, (*R*) configuration).]

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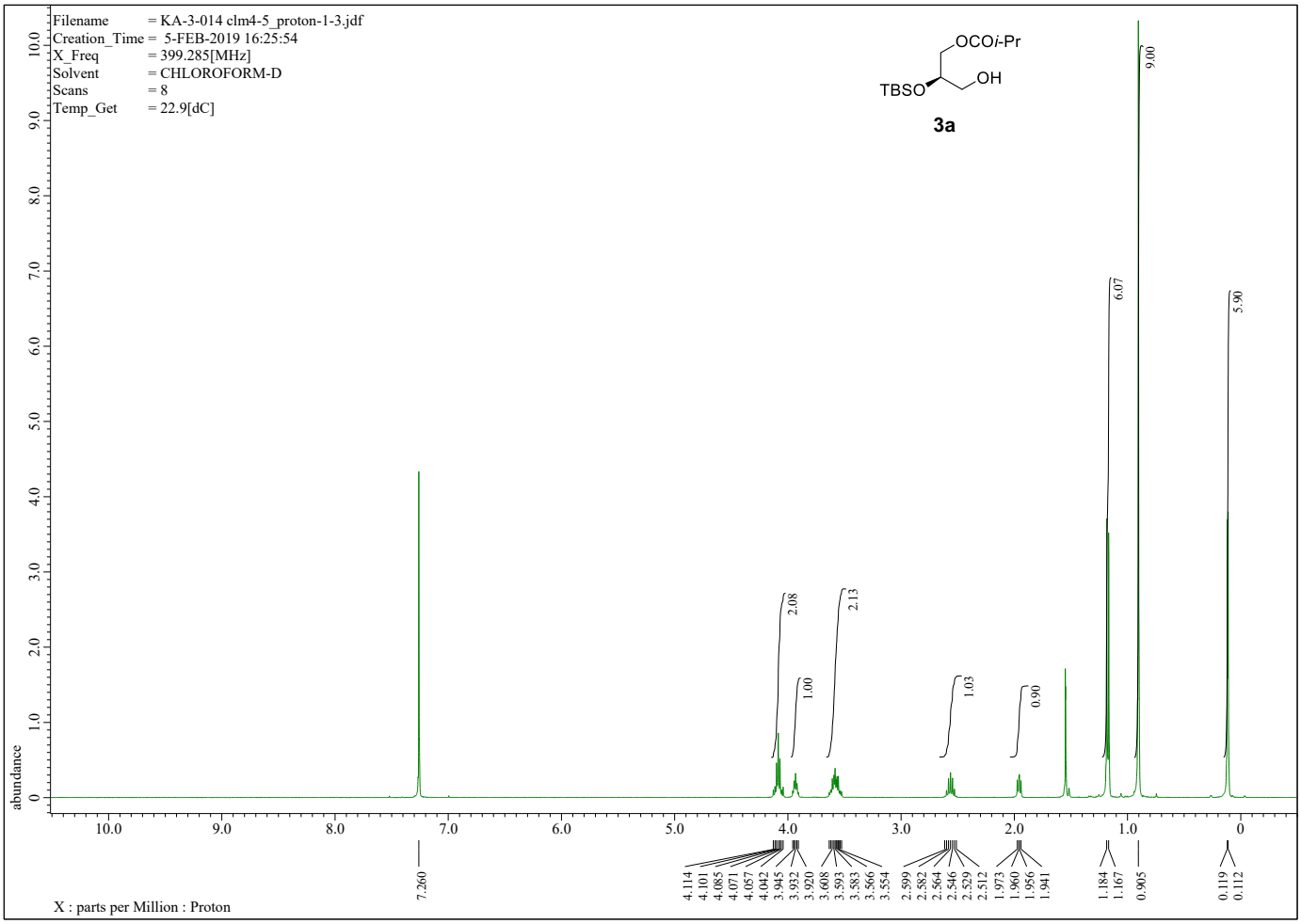
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abundance
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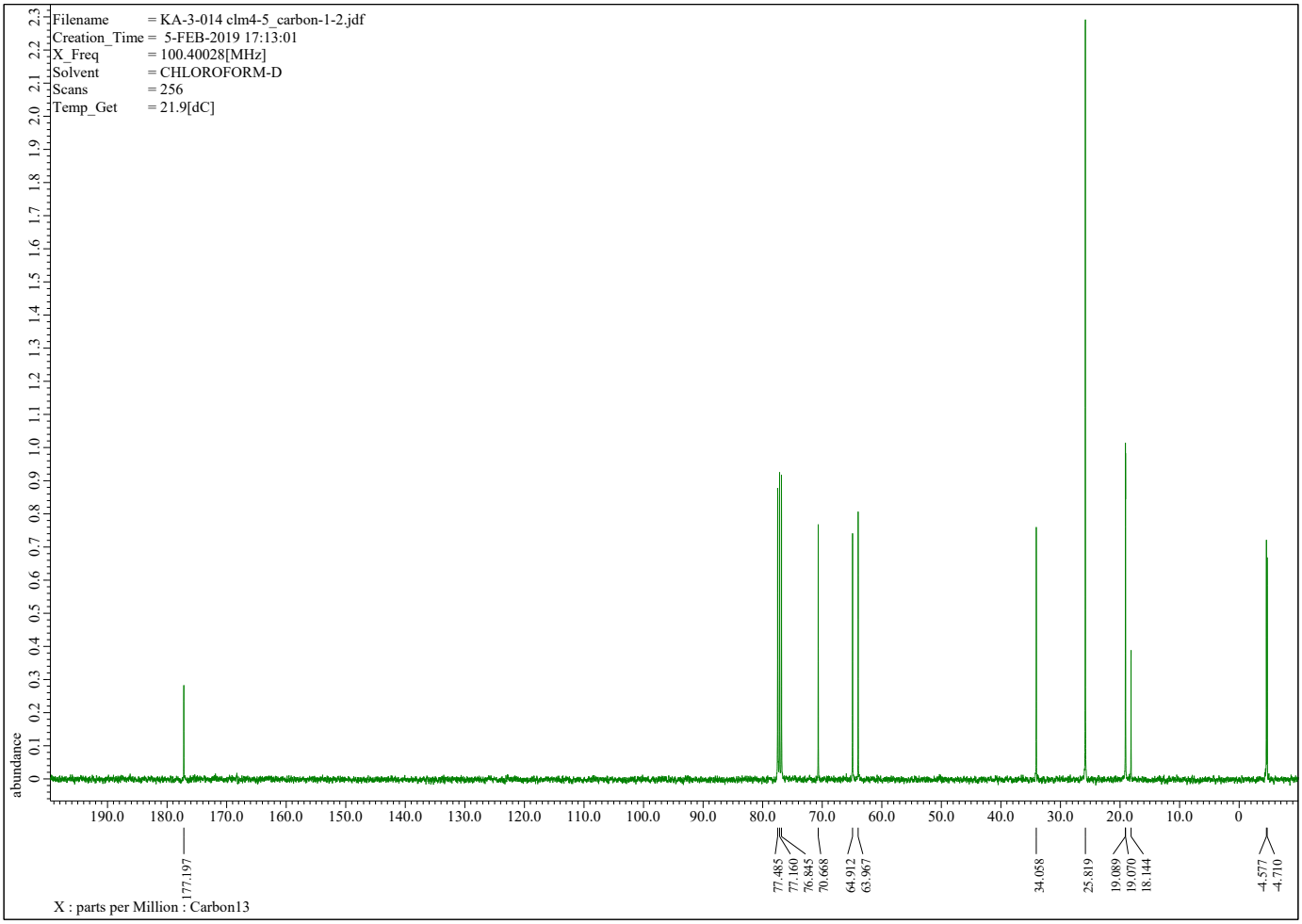
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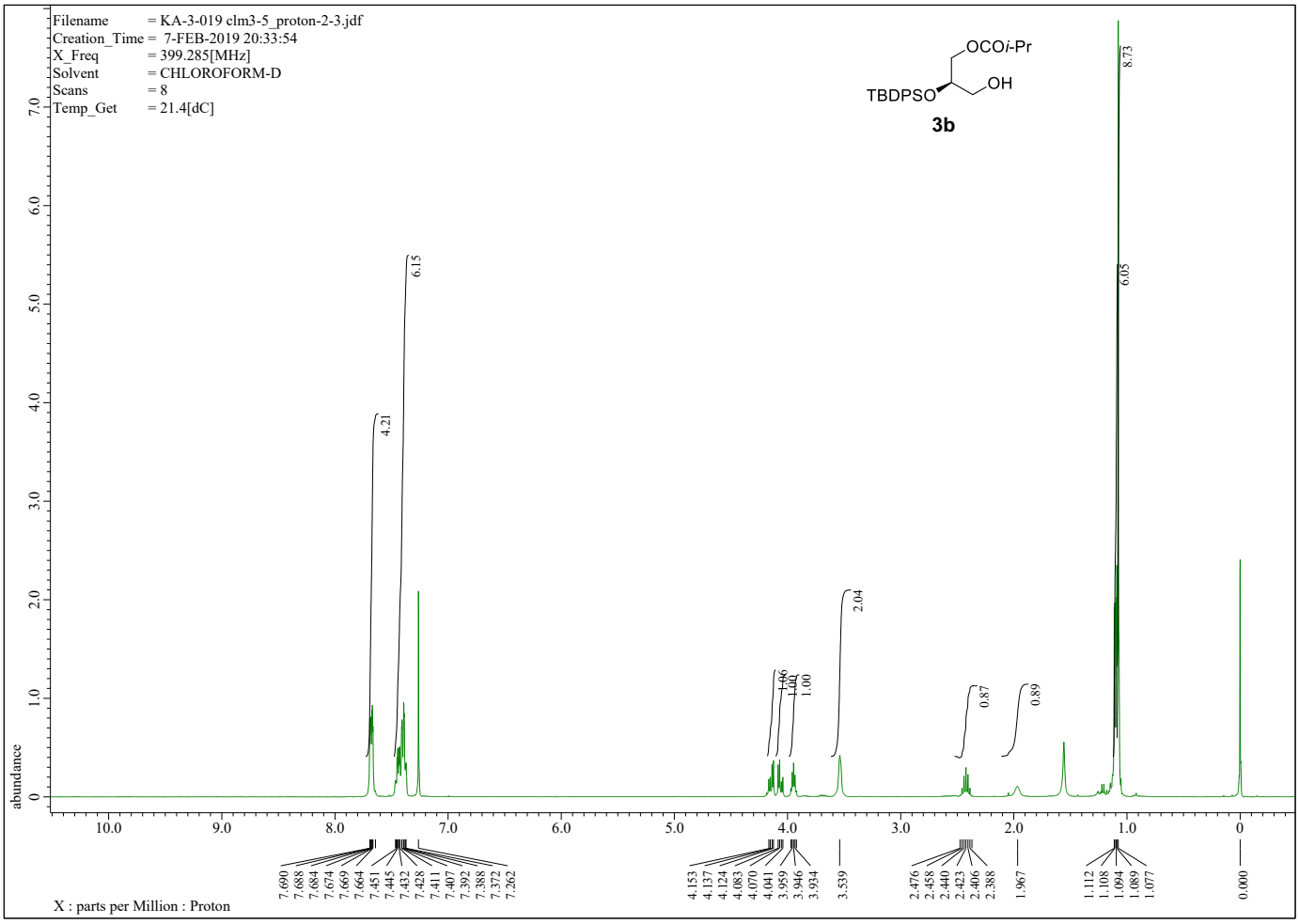
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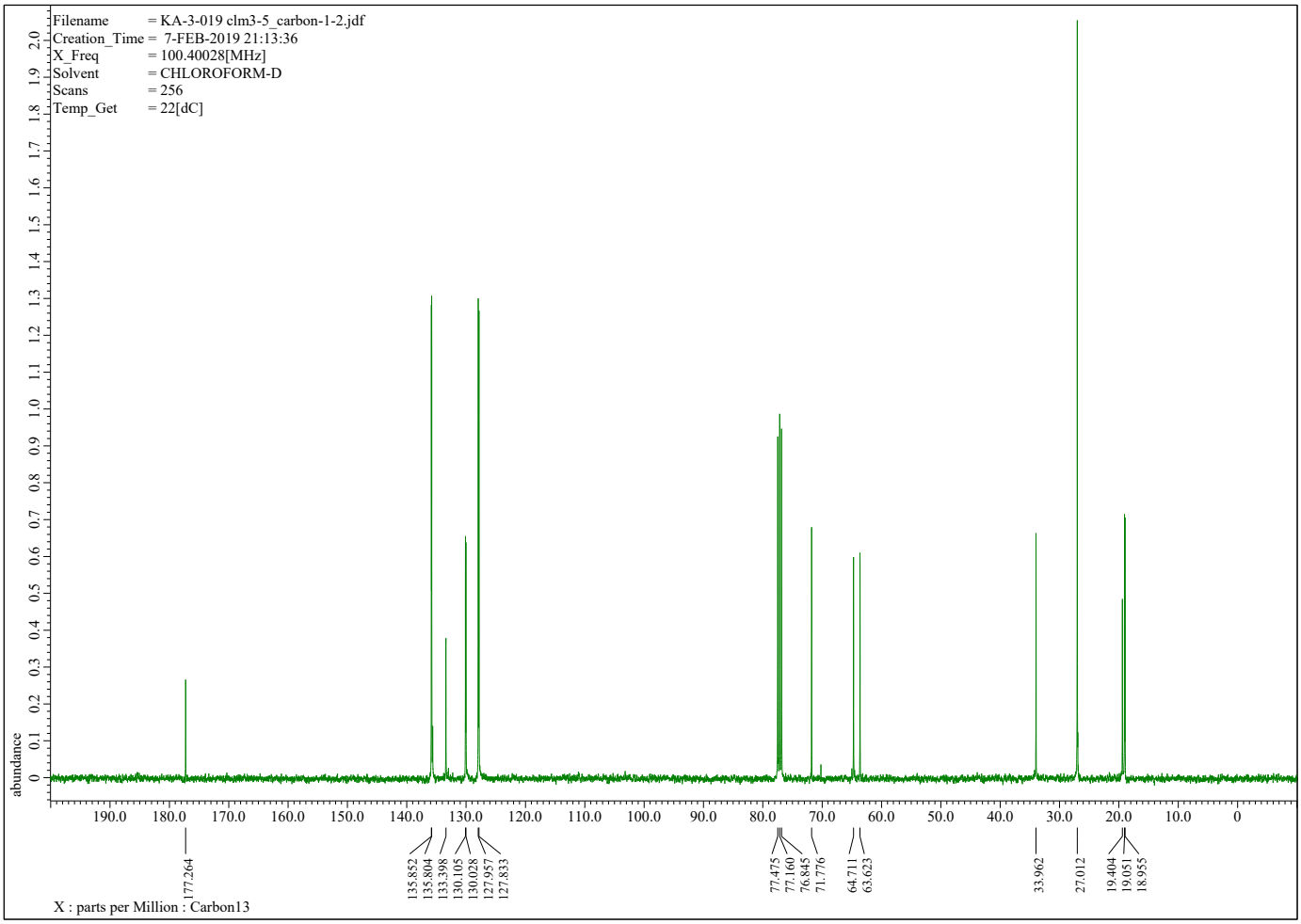
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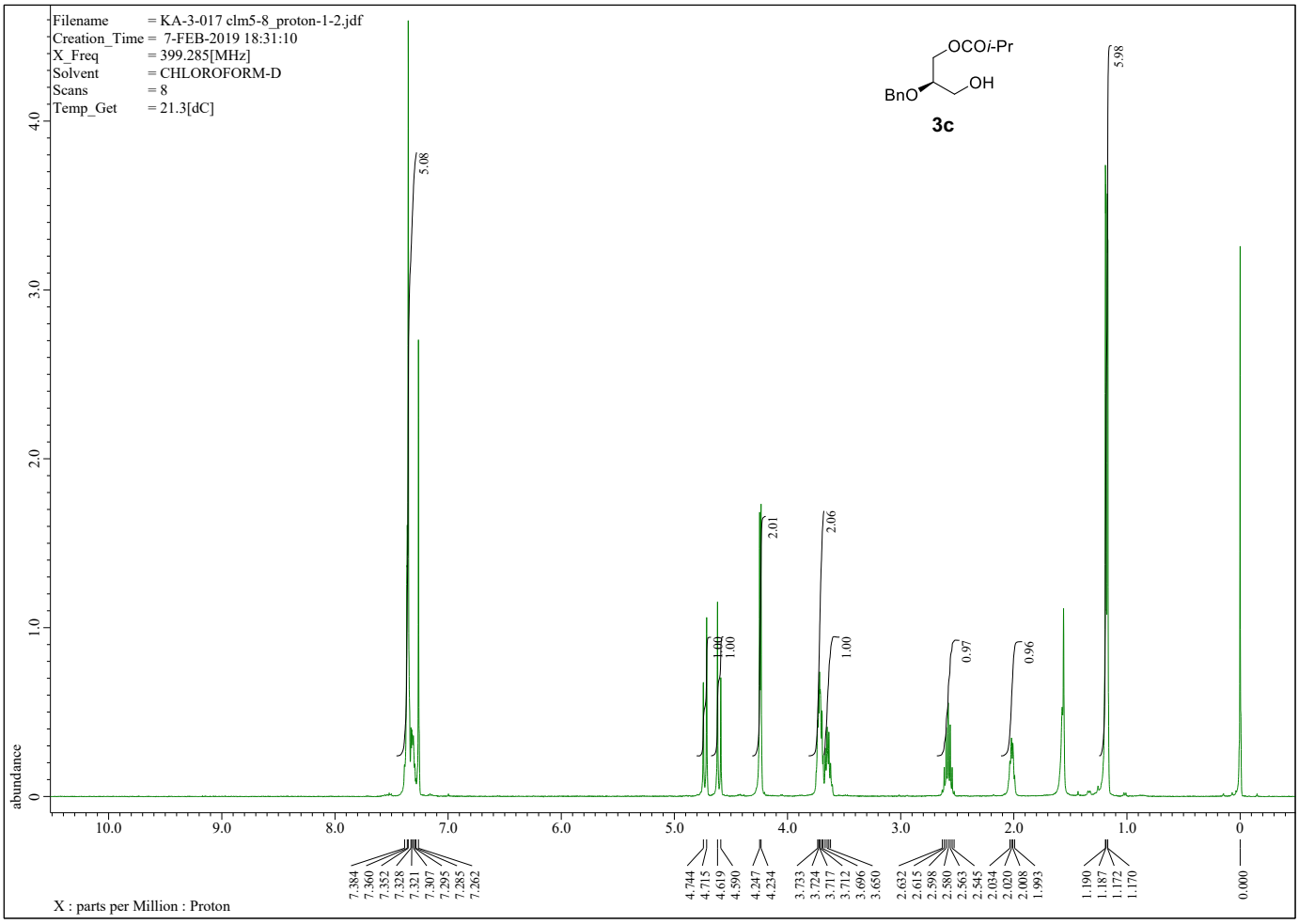
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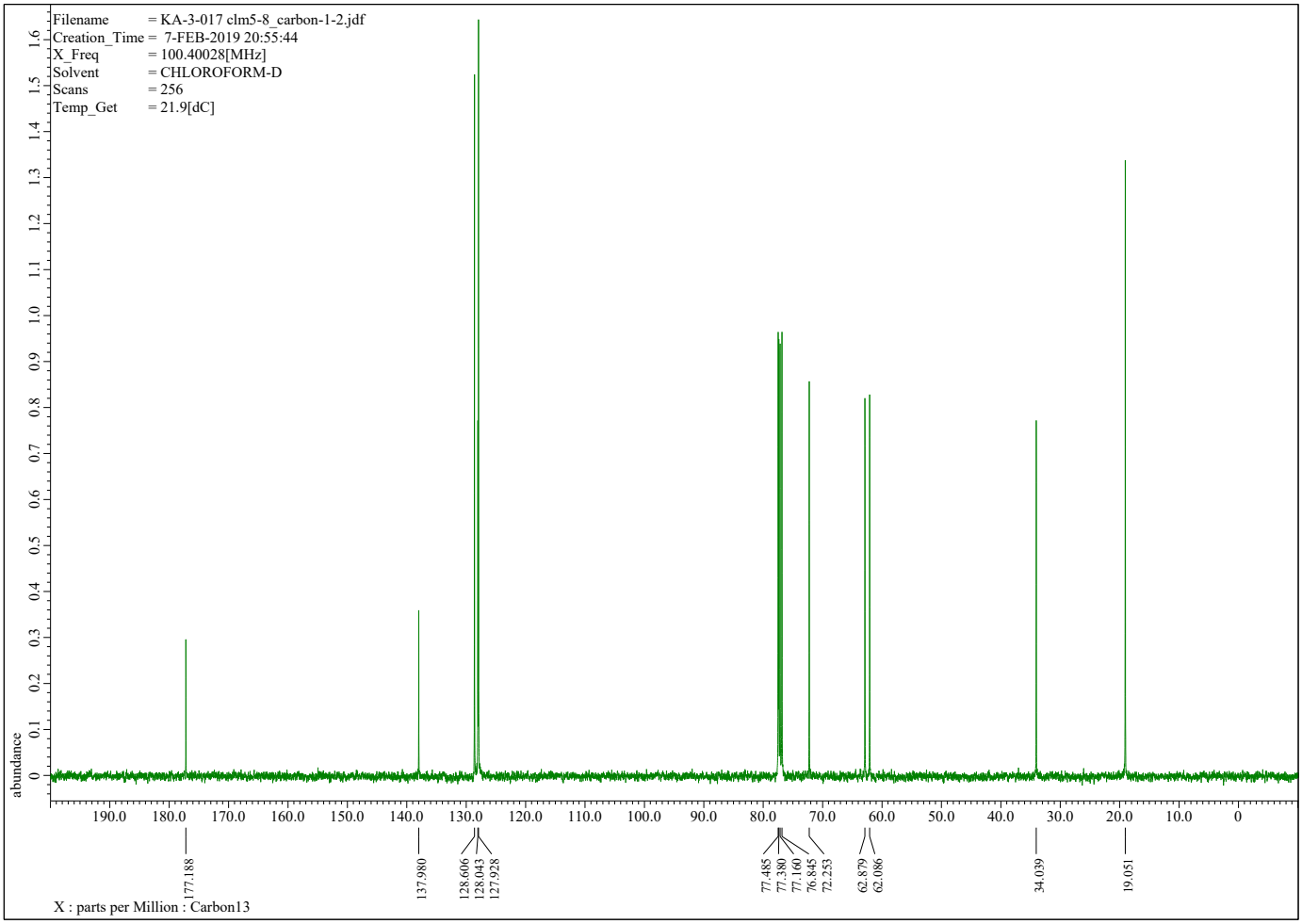
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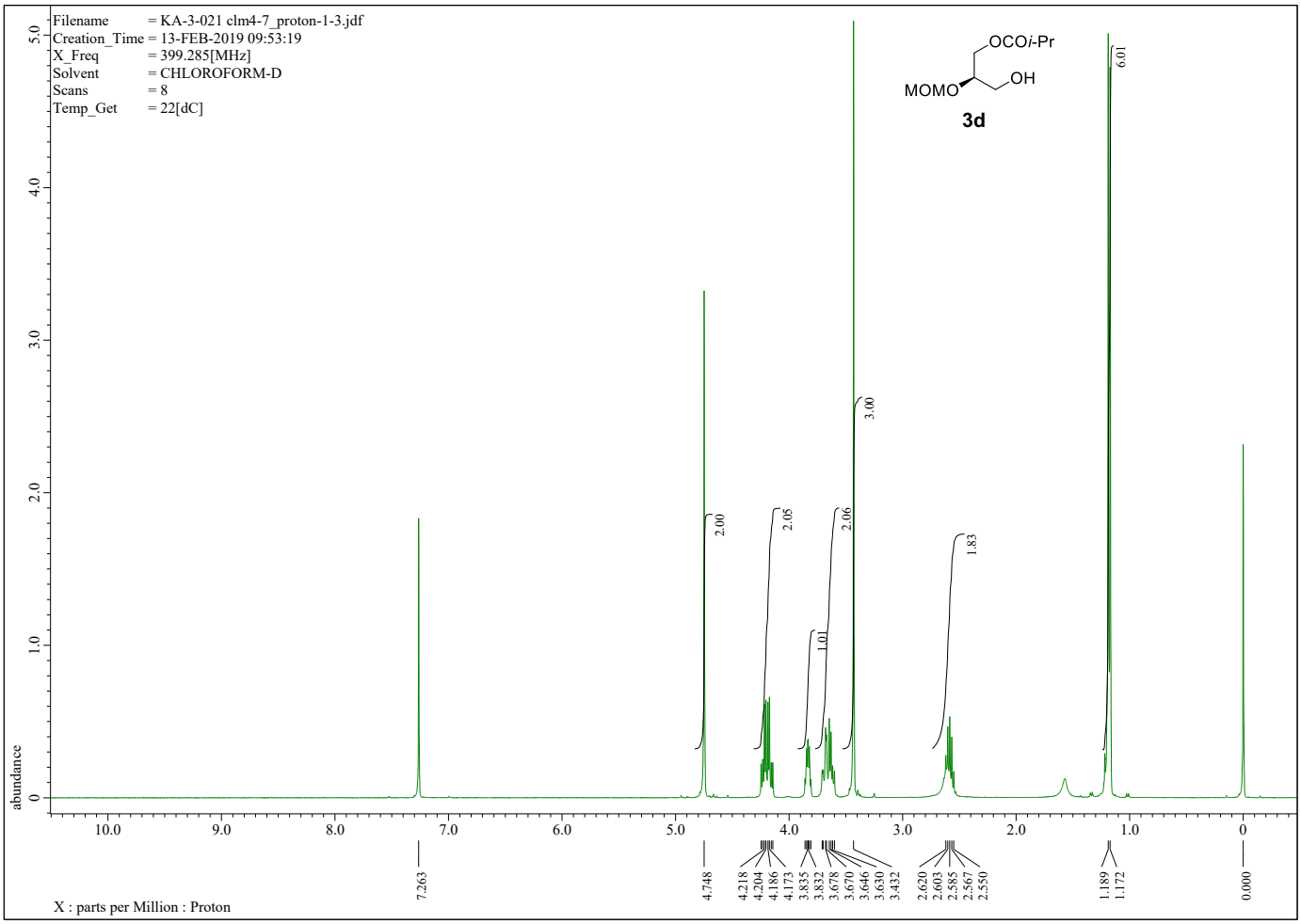
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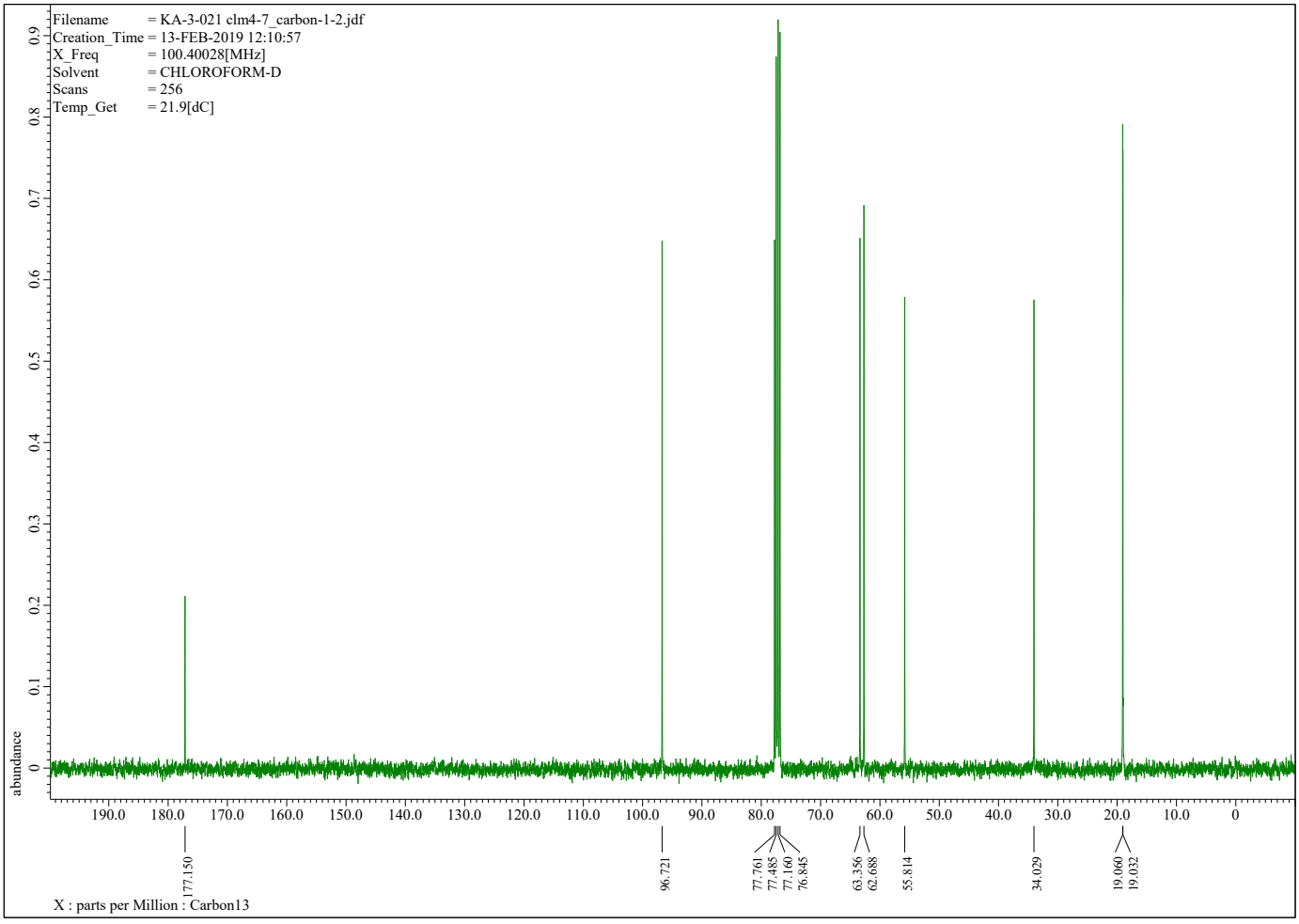
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