

Catalytic Asymmetric Direct-type 1,4-Addition Reactions of Simple Esters

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I. Experimental Section

General

¹H and ¹³C NMR spectra were recorded on JEOL JNM-ECA500 and JNM-ECX600 spectrometers in CDCl₃ unless otherwise noted. Tetramethylsilane (TMS) served as internal standard ($\delta = 0$) for ¹H NMR, and CDCl₃ served as internal standard ($\delta = 77.0$) for ¹³C NMR. IR spectra were measured using JASCO FT/IR - 4200 spectrometer. High-performance liquid chromatography was carried out using followed apparatuses; SHIMADZU LC-20AB (liquid chromatograph), SHIMADZU SPD-M20A (Photo diode array detector). Optical rotations were recorded on JASCO P-2100. Column chromatography was conducted on Silica gel 60N (spherical, neutral, Kanto Chem. Co., Inc.) and preparative thin-layer chromatography (PTLC) was carried out using Wakogel B-5F. Potassium bis(trimethylsilyl)amide (KHMDS), sodium bis(trimethylsilyl)amide (NaHMDS), lithium bis(trimethylsilyl)amide (LiHMDS) were purchased from Aldrich Co., Ltd.. Potassium *tert*-butoxide were purchased from Kanto Chemical Co., Ltd..

18-crown-6, amide (**2f**) and ketone (**2g**) were purchased from Tokyo Chemical Industry Co., Ltd.. Toluene, THF and diethyl ether were distilled just before using in the presence of benzophenone and sodium. **L1**¹, **L2**², **S1**¹ and **S2**³ were synthesized by following literatures. Esters (**2a-2e**) were prepared from corresponding acid anhydrides and alcohols. α,β -Unsaturated amides (**1a-1l**), ester (**1m**) and were prepared from corresponding α,β -unsaturated acid chlorides, amines and alcohols. α,β -Unsaturated ketone (**1n**) was prepared by Aldol condensation from pinacolin and benzaldehyde. The yields of **3af**¹, **3ma**⁴, **3na**⁴, and **3ng**⁴ were determined by ¹H NMR analyses of the crude mixture without isolation.

Typical procedure of KHMDS/18-crown-6 catalyzed 1,4-addition reaction of ¹Bu propionate (Table 1, entry 4)

KHMDS (6.0 mg, 3.0×10^{-2} mmol) and 18-crown-6 (8.9 mg, 3.4×10^{-2} mmol) were placed in a dried 5 mL reaction tube with septa inside a glove box filled with argon. The tube was cooled to -40 °C, then toluene (0.6 mL) was added. The reaction mixture was stirred for 1 h at the same temperature for catalyst preparation. After that, ¹Bu propionate (**2a**, 47.2 mg, 0.363 mmol) was added to the reaction mixture by syringe, then *N,N*-dimethylcinnamamide (**1a**, 52.6 mg, 0.300 mmol), which was put in another dried tube inside a glove box, was added to the tube through cannula with extra toluene (0.9 mL). The whole mixture was stirred for 18 h at -40 °C. The reaction was quenched with H₂O (1.0 mL) and extracted with DCM (10 mL) for three times. The combined organic layers were dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the crude product was obtained. The crude product was purified by silica-gel PTLC (Hexane-Ethyl acetate) to afford the desired 1,4-adduct **3aa** in quantitative yield (*syn*-form, 52.2 mg, 0.171 mmol; *anti*-form, 40.0 mg, 0.131 mmol).

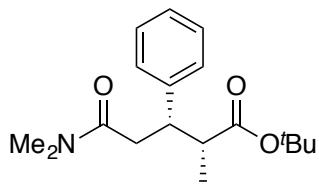
Typical procedure of catalytic asymmetric 1,4-addition reaction of ¹Bu propionate (Table 2, entry 2)

KHMDS (7.9 mg, 4.0×10^{-2} mmol) and **L1** (19.8 mg, 2.23×10^{-2} mmol) were placed in a dried 10 mL flask inside a glove box filled with argon. The flask was cooled to -78

°C, then toluene (0.7 mL) was added. The reaction mixture was stirred for 1 h at the same temperature for catalyst preparation. After that, *t*Bu propionate (**2a**, 64.1 mg, 0.492 mmol) was added to the reaction mixture by syringe, then *N,N*-dimethylcinnamamide (**1a**, 70.0 mg, 0.400 mmol), which was put in another dried tube inside a glove box, was added to the reaction mixture through cannula with extra toluene (1.5 mL). The whole mixture was stirred for 24 h at -78 °C. The reaction was quenched with H₂O (1.0 mL) and extracted with DCM (10 mL) for three times. The combined organic layers were dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the crude product was obtained. The crude product was purified by silica-gel PTLC (Hexane-Ethyl acetate) to afford the desired 1,4-adduct **3aa** in 92 % yield (*syn*-form, 73.7 mg, 0.241 mmol; *anti*-form, 39.3 mg, 0.129 mmol).

tert-butyl

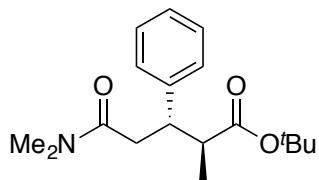
(2*R*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-phenylpentanoate



(3aa-*syn*); colorless oil; IR (neat, cm⁻¹); 2976, 2932, 2361, 1723, 1649, 1495, 1455, 1395, 1367, 1259, 1149, 849, 763, 701, 665; HRMS (DART) calcd for C₁₈H₂₈NO₃ [M + H]⁺ 306.2069, found 306.2054; HPLC analysis using Daicel Chiralpak AD-3 column (Hex/ⁱPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 22.1 min (major), 16.4 min (minor)); [α]_D = -13.77 (c 0.23, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.29-7.18 (m, 5H), 3.38 (td, 1H, J = 10.1, 4.4 Hz), 2.85 (s, 3H), 2.78 (s, 3H), 2.74 (dd, 1H, J = 14.8, 10.0 MHz), 2.67-2.61 (m, 2H), 1.46 (s, 9H), 0.93 (d, 3H, J = 6.9 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 175.1, 170.8, 141.8, 128.2, 128.1, 126.6, 80.4, 46.1, 45.2, 37.9, 37.2, 35.2, 28.0, 16.0.

tert-butyl

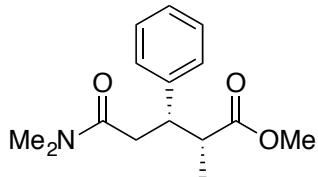
(2*S*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-phenylpentanoate



(3aa-*anti*); colorless oil; IR (neat, cm⁻¹); 2976, 2933, 1724, 1648, 1494, 1455, 1395, 1367, 1253, 1150, 849, 761, 701, 664; HRMS (DART) calcd for C₁₈H₂₈NO₃ [M + H]⁺ 306.2069, found 306.2061; HPLC analysis using Daicel Chiralpak OD-3 column (Hex/ⁱPrOH = 95/5, 0.7 mL/min, 210 nm, t_R = 18.7 min (Major), 16.3 min (minor)); [α]_D = -17.03 (c 0.19, CHCl₃); ¹H

NMR (600 MHz, CDCl₃) δ: 7.26-7.16 (m, 5H), 3.41 (td, 1H, J = 9.1, 5.4 Hz), 2.81 (s, 3H), 2.79 (s, 3H), 2.77-2.73 (m, 2H), 2.66 (dd, 1H, J = 15.1, 8.9 MHz), 1.23 (d, 3H, J = 6.9 Hz), 1.18 (s, 9H); ¹³C NMR (150 MHz, CDCl₃) δ: 174.3, 171.3, 142.4, 128.3, 128.0, 126.5, 79.9, 45.7, 45.4, 37.2, 36.6, 35.4, 27.6, 15.7.

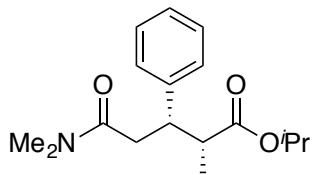
methyl (2*R*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-phenylpentanoate (3ab,



syn/anti = 55:45); colorless oil; HRMS (DART) calcd for C₁₅H₂₂NO₃ [M + H]⁺ 264.1600, found 264.1604; HPLC analysis using Daicel Chiraldak AD-3 column (Hex/ⁱPrOH = 100/1, 1.0 mL/min, 210 nm, t_R = 111.7 min (*anti*, minor),

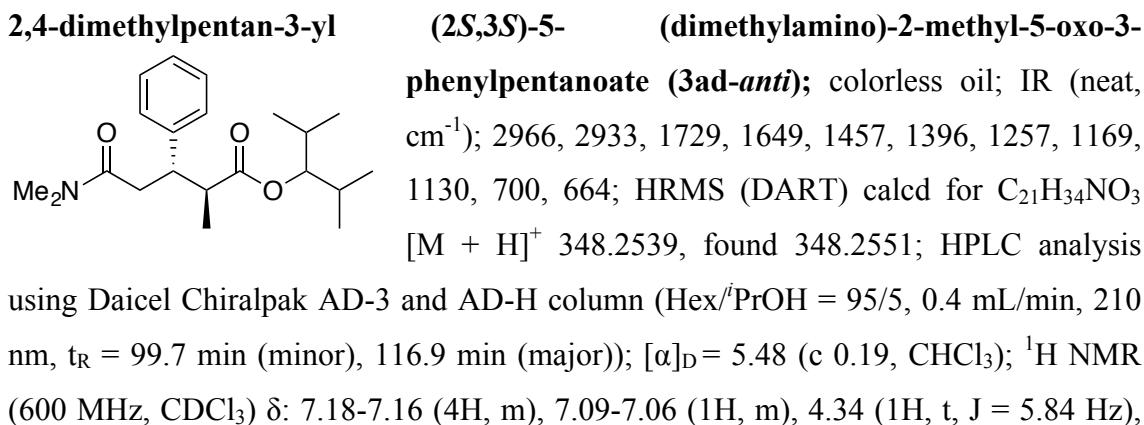
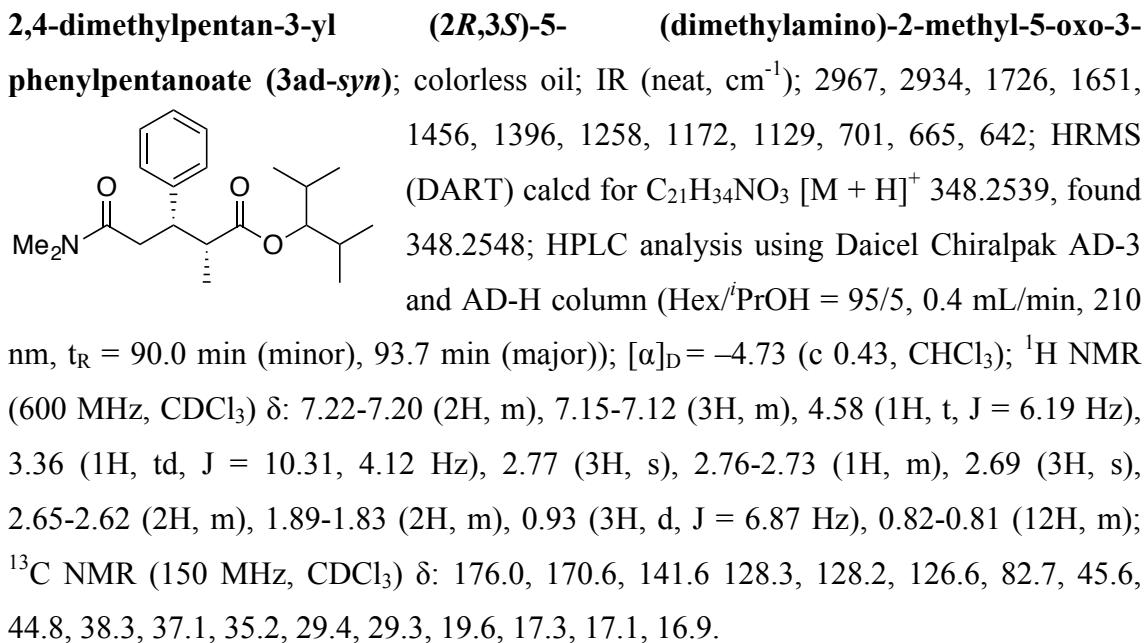
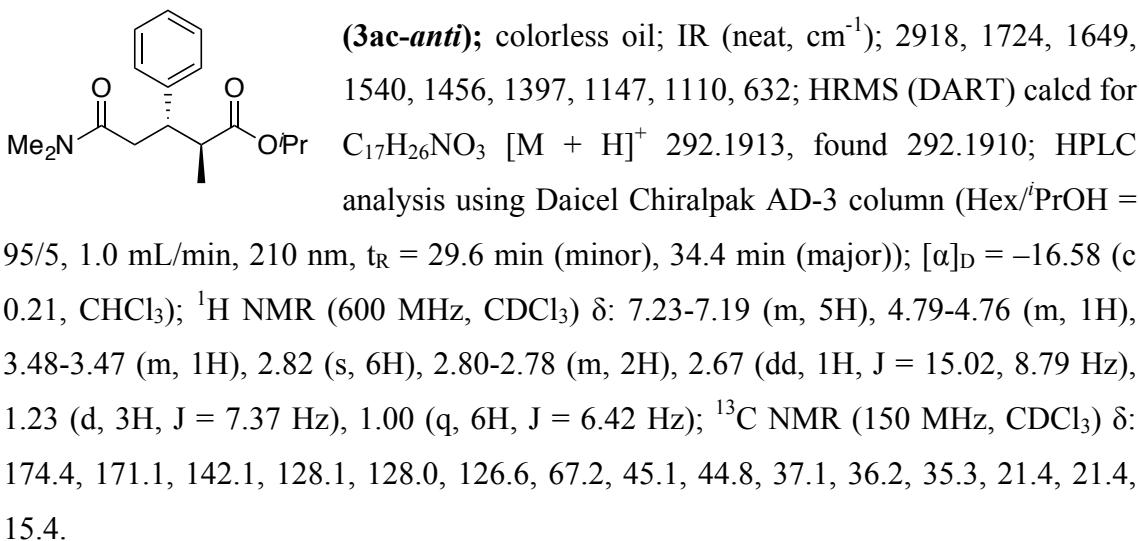
126.8 min (*syn*, minor), 140.2 min (*syn*, major), 149.7 min (*syn*, major)); *syn*-form ¹H NMR (600 MHz, CDCl₃) δ: 7.30-7.17 (m, 5H), 3.70 (s, 3H), 3.48-3.45 (m, 1H), 2.85 (s, 3H), 2.80 (s, 3H), 2.75-2.67 (m, 2H), 2.62 (dd, 1H, J = 15.12, 5.50 Hz), 0.96 (d, 3H, J = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 176.2, 170.7, 141.7, 128.397, 128.0, 126.7, 51.6, 44.9, 44.5, 37.2, 37.1, 35.3, 15.7; *Anti*-form: ¹H NMR (600 MHz, CDCl₃) δ: 7.30-7.17 (m, 5H), 3.57-3.52 (m, 1H), 3.49 (s, 3H), 2.90 (s, 3H), 2.89-2.75 (m, 3H), 2.85 (s, 3H), 1.20 (d, 3H, J = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 175.3, 171.1, 141.9, 128.1, 128.0, 126.6, 51.3, 44.9, 44.8, 37.9, 35.6, 35.4, 14.8.

isopropyl (2*R*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-phenylpentanoate (3ac-syn);



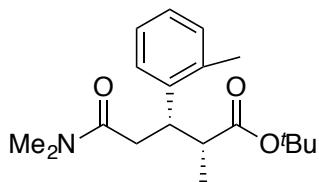
colorless oil; IR (neat, cm⁻¹); 2918, 1722, 1649, 1540, 1456, 1397, 1149, 664; HRMS (DART) calcd for C₁₇H₂₆NO₃ [M + H]⁺ 292.1913, found 292.1907; HPLC analysis using Daicel Chiraldak AD-3 column (Hex/ⁱPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 29.8 min (minor), 33.3 min (major)); [α]_D = -19.65 (c 0.23, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.28-7.27 (m, 2H), 7.21-7.20 (m, 3H), 5.06-5.01 (m, 1H), 3.43 (td, 1H, J = 9.78, 4.35 Hz), 2.86 (s, 3H), 2.78 (s, 3H), 2.75-2.70 (m, 2H), 2.61 (dd, 1H, J = 15.02, 4.25 Hz), 1.25 (d, 6H, J = 6.24 Hz), 0.95 (d, 3H, J = 6.80 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 175.3, 170.7, 141.7, 128.3, 128.1, 126.6, 67.7, 45.2, 45.0, 37.8, 37.1, 35.2, 21.7, 21.6, 15.9.

isopropyl (2*S*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-phenylpentanoate



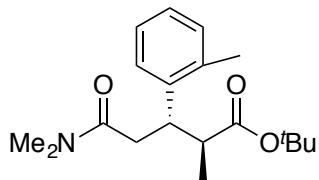
3.47-3.46 (1H, m), 2.93 (1H, dt, $J = 16.27, 7.05$ Hz), 2.73 (3H, s), 2.72-2.70 (1H, m), 2.68 (3H, s), 2.60 (1H, dd, $J = 14.78, 9.28$ Hz), 1.71-1.64 (2H, m), 1.25 (3H, d, $J = 6.87$ Hz), 0.62 (6H, dd, $J = 16.50, 6.87$ Hz), 0.55 (6H, t, $J = 6.19$ Hz); ^{13}C NMR (150 MHz, CDCl_3) δ : 175.2, 171.2, 142.5, 128.2, 128.1, 126.6, 82.4, 44.9, 44.6, 37.1, 36.2, 35.3, 29.3, 29.1, 19.3, 19.2, 17.1, 16.8, 16.2.

tert-butyl (2*R*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-(*o*-tolyl)pentanoate (3ba-*syn*)



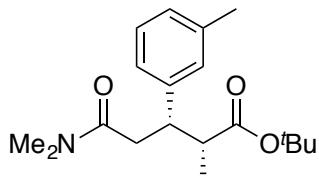
colorless oil; IR (neat, cm^{-1}); 1723, 1651, 1395, 1367, 1259, 1146, 849, 763, 729, 664; HRMS (DART) calcd for $\text{C}_{19}\text{H}_{30}\text{NO}_3$ [$\text{M} + \text{H}$] $^+$ 320.2226, found 320.2221; HPLC analysis using Daicel Chiralpak AD-3 column (Hex/ $i\text{PrOH} = 95/5$, 1.0 mL/min, 210 nm, $t_{\text{R}} = 10.2$ min (major), 12.0 min (minor)); $[\alpha]_D = 3.68$ ($c 0.20$, CHCl_3); ^1H NMR (600 MHz, CDCl_3) δ : 7.15-7.07 (m, 4H), 3.72-3.68 (m, 1H), 2.87 (s, 3H), 2.79-2.76 (m, 4H), 2.63-2.57 (m, 2H), 2.43 (s, 3H), 1.48 (s, 9H), 0.91 (d, 3H, $J = 6.80$ Hz); ^{13}C NMR (150 MHz, CDCl_3) δ : 175.5, 171.0, 140.7, 137.4, 130.3, 126.0, 125.9, 125.6, 80.4, 47.0, 47.0, 38.1, 37.2, 35.3, 28.0, 20.1, 15.9.

tert-butyl (2*S*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-(*o*-tolyl)pentanoate (3ba-*anti*)



colorless oil; IR (neat, cm^{-1}); 2931, 1722, 1649, 1491, 1458, 1395, 1366, 1258, 1150, 1047, 849, 728, 665; HRMS (DART) calcd for $\text{C}_{19}\text{H}_{30}\text{NO}_3$ [$\text{M} + \text{H}$] $^+$ 320.2226, found 320.2230; HPLC analysis using Daicel Chiralpak OD-3 column (Hex/ $i\text{PrOH} = 95/5$, 0.7 mL/min, 210 nm, $t_{\text{R}} = 15.4$ min (minor), 18.9 min (major)); $[\alpha]_D = 8.74$ ($c 0.22$, CHCl_3); ^1H NMR (600 MHz, CDCl_3) δ : 7.13-7.08 (m, 4H), 3.75-3.71 (m, 1H), 2.81 (s, 3H), 2.76 (s, 3H), 2.70-2.66 (m, 3H), 2.42 (s, 3H), 1.24 (d, 3H, $J = 6.80$ Hz), 1.16 (s, 9H); ^{13}C NMR (150 MHz, CDCl_3) δ : 174.5, 171.3, 141.2, 136.8, 130.3, 126.4, 126.1, 125.5, 79.8, 45.7, 40.0, 37.2, 36.1, 35.4, 27.5, 19.7, 15.2.

tert-butyl (2*R*,3*S*)-5-(dimethylamino)-2-methyl-5-oxo-3-(*m*-tolyl)pentanoate (3ca-*syn*)

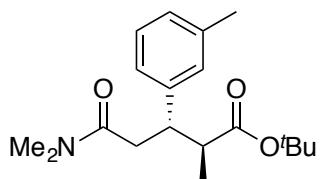


colorless oil; IR (neat, cm^{-1}); 1723, 1650, 1490, 1457, 1395, 1367, 1259, 1149, 105.0, 848, 788, 707, 665;

HRMS (DART) calcd for $C_{19}H_{30}NO_3$ [M + H]⁺ 320.2226, found 320.2237; HPLC analysis using Daicel Chiralpak AD-3 column (Hex/ⁱPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 12.7 min (minor), 22.9 min (major)); $[\alpha]_D$ = -9.35 (c 0.38, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ : 7.16 (1H, t, J = 7.90 Hz), 7.00-6.99 (3H, m), 3.35 (1H, td, J = 9.97, 4.35 Hz), 2.85 (3H, s), 2.79 (3H, s), 2.73 (1H, dd, J = 14.78, 9.97 Hz), 2.66-2.59 (2H, m), 2.31 (3H, s), 1.46 (9H, s), 0.93 (3H, d, J = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ : 175.2, 170.9, 141.7, 137.6, 129.0, 128.1, 127.3, 125.0, 80.3, 46.1, 45.1, 37.9, 37.2, 35.3, 28.0, 21.4, 16.0.

tert-butyl

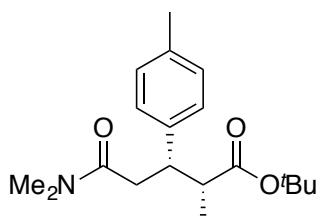
(2S,3S)-5-(dimethylamino)-2-methyl-5-oxo-3-(*m*-tolyl)pentanoate



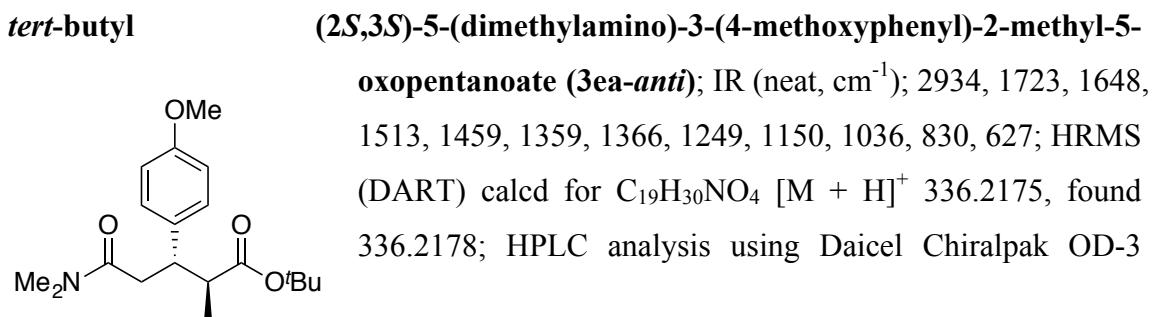
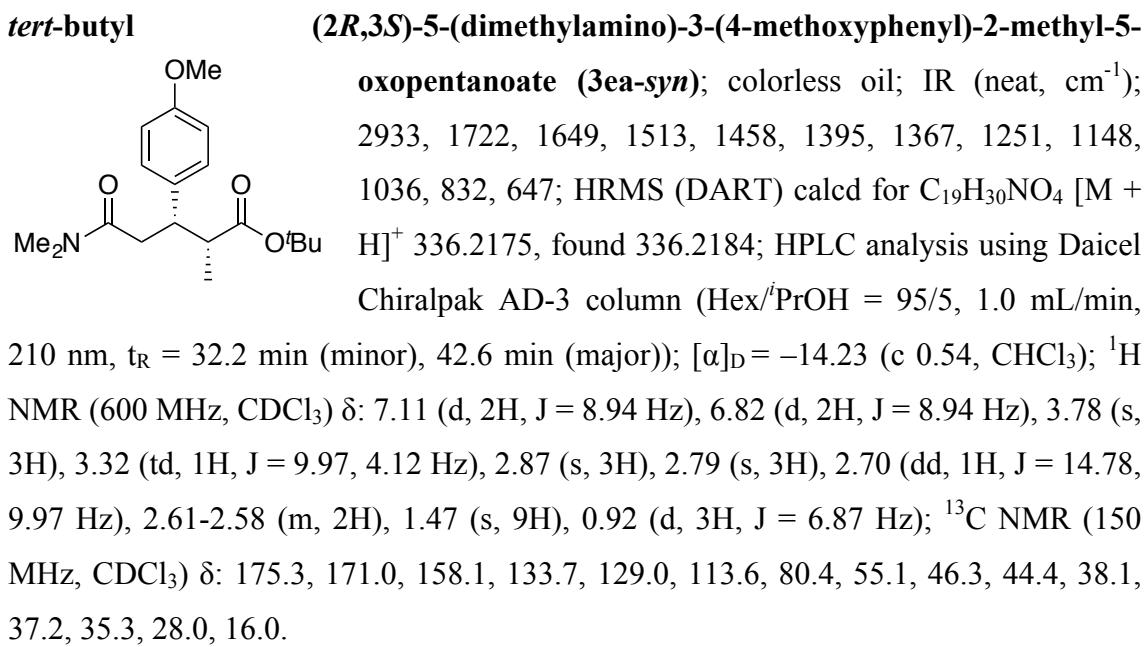
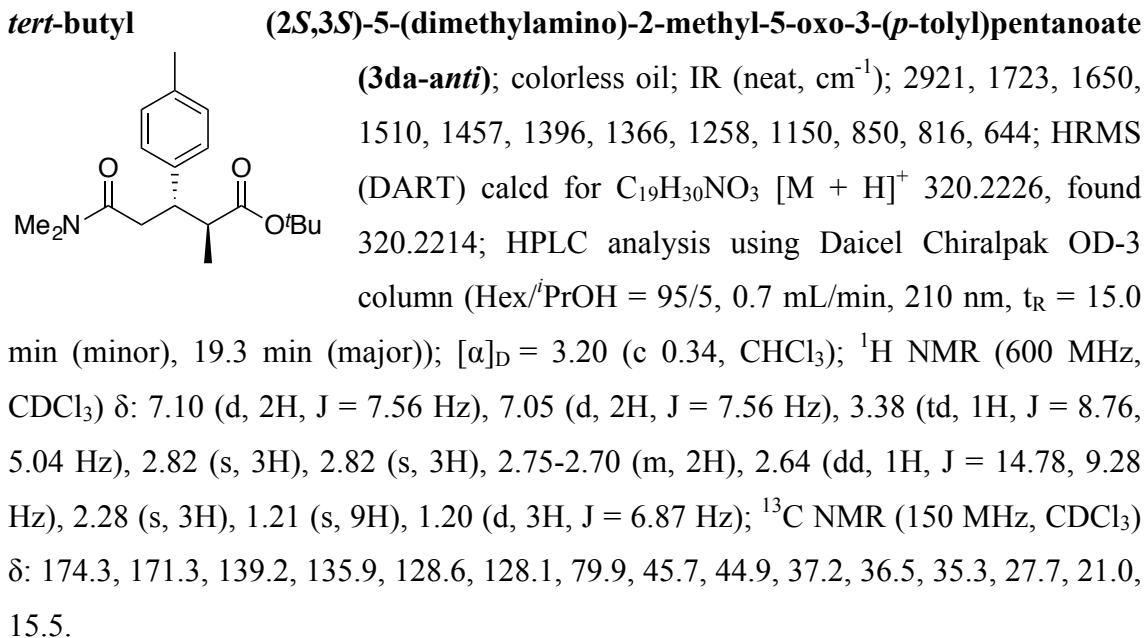
(3ca-anti); colorless oil; IR (neat, cm⁻¹); 2920, 1720, 1650, 1558, 1457, 1396, 1366, 1150, 752, 664; HRMS (DART) calcd for $C_{19}H_{30}NO_3$ [M + H]⁺ 320.2226, found 320.2233; HPLC analysis using Daicel Chiralpak OD-3 column (Hex/ⁱPrOH = 95/5, 0.7 mL/min, 210 nm, t_R = 13.2 min (minor), 16.4 min (major)); $[\alpha]_D$ = 1.37 (c 1.10, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ : 7.14-7.09 (1H, m), 7.03-6.97 (3H, m), 3.38 (1H, td, J = 8.76, 5.27 Hz), 2.82 (3H, s), 2.81 (3H, s), 2.75-2.72 (2H, m), 2.65 (1H, dd, J = 15.12, 8.94 Hz), 2.30 (3H, s), 1.21-1.20 (12H, m); ¹³C NMR (150 MHz, CDCl₃) δ : 174.3, 171.3, 142.3, 137.3, 129.1, 127.8, 127.2, 125.1, 79.8, 45.7, 45.3, 37.2, 36.4, 35.4, 27.6, 21.3, 15.6.

tert-butyl

(2R,3S)-5-(dimethylamino)-2-methyl-5-oxo-3-(*p*-tolyl)pentanoate

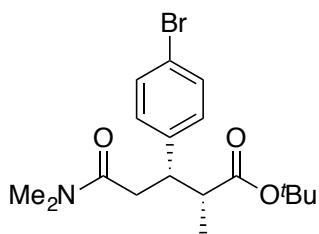


(3da-syn); colorless oil; IR (neat, cm⁻¹); 2931, 1723, 1650, 1512, 1456, 1395, 1367, 1260, 1148, 849, 818, 666; HRMS (DART) calcd for $C_{19}H_{30}NO_3$ [M + H]⁺ 320.2226, found 320.2235; HPLC analysis using Daicel Chiralpak AD-3 column (Hex/ⁱPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 17.7 min (minor), 22.3 min (major)); $[\alpha]_D$ = -15.18 (c 0.55, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ : 7.05 (br s, 4H), 3.31 (td, 1H, J = 9.92, 4.35 Hz), 2.84 (s, 3H), 2.76 (s, 3H), 2.70-2.67 (m, 1H), 2.62-2.57 (m, 2H), 2.27 (s, 3H), 1.44 (s, 9H), 0.90 (d, 3H, J = 6.80 Hz); ¹³C NMR (150 MHz, CDCl₃) δ : 175.3, 170.9, 138.6, 136.0, 128.9, 128.0, 80.3, 46.1, 44.8, 38.1, 37.2, 35.2, 28.0, 21.0, 16.0.



column (Hex/ⁱPrOH = 95/5, 0.7 mL/min, 210 nm, t_R = 23.6 min (minor), 31.7 min (major)); [α]_D = -1.75 (c 0.26, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.07 (d, 2H, J = 8.25 Hz), 6.72 (d, 2H, J = 8.25 Hz), 3.69 (s, 3H), 3.29 (td, 1H, J = 9.28, 4.81 Hz), 2.75 (s, 3H), 2.74 (s, 3H), 2.67-2.63 (m, 2H), 2.55 (dd, 1H, J = 15.12, 8.94 Hz), 1.14-1.13 (m, 12H); ¹³C NMR (150 MHz, CDCl₃) δ: 174.3, 171.3, 158.1, 134.4, 129.2, 113.3, 79.9, 55.1, 45.8, 44.6, 37.2, 36.9, 35.4, 27.7, 15.6.

tert-butyl

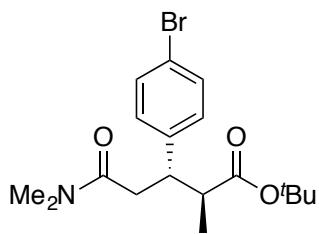


(2*R*,3*S*)-3-(4-bromophenyl)-5-(dimethylamino)-2-methyl-5-

oxopentanoate (3fa-syn); white solid; Mp 86-87 °C; IR (neat, cm⁻¹); 2933, 1723, 1649, 1488, 1396, 1367, 1259, 1149, 1072, 1010, 962, 848, 825, 753, 640, 612; HRMS (DART) calcd for C₁₈H₂₇BrNO₃ [M + H]⁺ 384.1174, found 384.1173; HPLC analysis using Daicel Chiralpak AD-3

column (Hex/ⁱPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 26.2 min (minor), 36.9 min (major)); [α]_D = -1.68 (c 0.31, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.40 (2H, d, J = 8.25 Hz), 7.09 (2H, d, J = 8.25 Hz), 3.37 (1H, td, J = 9.97, 4.12 Hz), 2.90 (3H, s), 2.80 (3H, s), 2.70 (1H, dd, J = 15.12, 10.31 Hz), 2.64-2.56 (2H, m), 1.46 (9H, s), 0.92 (3H, d, J = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 174.8, 170.5, 141.0, 131.3, 129.9, 120.3, 80.6, 46.0, 44.5, 37.7, 37.1, 35.3, 27.9, 16.0.

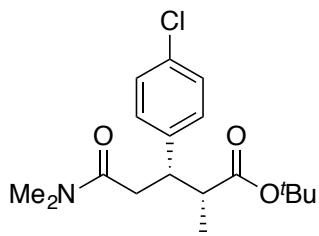
tert-butyl (2*S*,3*S*)-3-(4-bromophenyl)-5-(dimethylamino)-2-methyl-5-oxopentanoate



(3fa-anti); white solid; Mp 96-98 °C; IR (neat, cm⁻¹); 2977, 2934, 1724, 1648, 1488, 1396, 1367, 1258, 1149, 1011, 962, 823, 751, 717, 652; HRMS (DART) calcd for C₁₈H₂₇BrNO₃ [M + H]⁺ 384.1174, found 384.1187; HPLC analysis using Daicel Chiralpak OD-3 column (Hex/ⁱPrOH = 95/5, 0.7

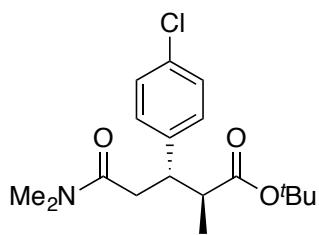
mL/min, 210 nm, t_R = 16.9 min (minor), 21.0 min (major)); [α]_D = 6.15 (c 0.20, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.35 (d, 2H, J = 7.94 Hz), 7.10 (d, 2H, J = 7.94 Hz), 3.38 (td, 1H, J = 8.79, 4.35 Hz), 2.83 (s, 3H), 2.80 (s, 3H), 2.73-2.65 (m, 2H), 2.59 (dd, 1H, J = 15.30, 9.64 Hz), 1.19-1.17 (m, 12H); ¹³C NMR (150 MHz, CDCl₃) δ: 174.0, 170.8, 141.5, 131.0, 131.0, 130.1, 80.2, 45.5, 44.6, 37.2, 36.2, 35.4, 27.6, 15.6.

tert-butyl (2*R*,3*S*)-3-(4-chlorophenyl)-5-(dimethylamino)-2-methyl-5-oxopentanoate



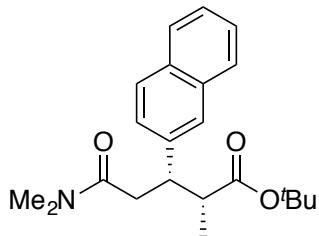
(3ga-syn); white solid; Mp 78-79 °C; IR (neat, cm⁻¹); 2977, 2933, 1723, 1648, 1491, 1457, 1397, 1368, 1260, 1149, 1092, 1052, 1014, 961, 931, 830, 753, 720, 609; HRMS (DART) calcd for C₁₈H₂₇ClNO₃ [M + H]⁺ 340.1674, found 340.1674; HPLC analysis using Daicel Chiraldak AD-3 column (Hex/ⁱPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 24.9 min (minor), 33.6 min (major)); [α]_D = -5.35 (c 0.37, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.25 (2H, d, J = 8.25 Hz), 7.15 (2H, d, J = 8.94 Hz), 3.38 (1H, td, J = 9.97, 4.12 Hz), 2.90 (3H, s), 2.80 (3H, s), 2.71 (1H, dd, J = 15.12, 10.31 Hz), 2.64-2.58 (2H, m), 1.46 (9H, s), 0.92 (3H, d, J = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 174.8, 170.5, 140.5, 132.2, 129.5, 128.4, 80.6, 46.0, 44.5, 37.7, 37.1, 35.3, 28.0, 16.0.

tert-butyl (2*S*,3*S*)-3-(4-chlorophenyl)-5-(dimethylamino)-2-methyl-5-oxopentanoate



(3ga-anti); white solid; Mp 91-93 °C; IR (neat, cm⁻¹); 2976, 2934, 1724, 1645, 1490, 1458, 1397, 1366, 1149, 1047, 1017, 921, 828, 751, 723, 679; HRMS (DART) calcd for C₁₈H₂₇ClNO₃ [M + H]⁺ 340.1674, found 340.1672; HPLC analysis using Daicel Chiraldak OD-3 column (Hex/ⁱPrOH = 95/5, 0.7 mL/min, 210 nm, t_R = 14.6 min (minor), 17.7 min (major)); [α]_D = 7.90 (c 0.85, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.23 (2H, d, J = 8.25 Hz), 7.18 (2H, d, J = 8.25 Hz), 3.41 (1H, td, J = 9.11, 4.58 Hz), 2.85 (3H, s), 2.83 (3H, s), 2.73-2.71 (2H, m), 2.62 (1H, dd, J = 15.12, 9.68 Hz), 1.22-1.20 (12H, m); ¹³C NMR (150 MHz, CDCl₃) δ: 174.0, 170.8, 141.0, 132.2, 129.7, 128.1, 80.2, 45.6, 44.6, 37.2, 36.3, 35.4, 27.7, 15.6.

tert-butyl

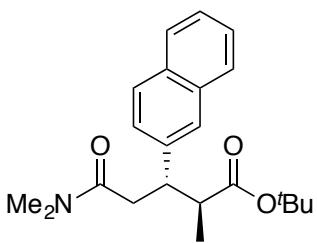


(2*R*,3*S*)-5-(dimethylamino)-2-methyl-3-(naphthalen-2-yl)-5-oxopentanoate (3ha-syn)

white solid; Mp 111-114 °C; IR (neat, cm⁻¹); 2977, 2934, 1722, 1648, 1456, 1395, 1367, 1258, 1148, 1052, 851, 821, 751, 665; HRMS (DART) calcd for C₂₂H₃₀NO₃ [M + H]⁺ 356.2226, found 356.2221; HPLC analysis using Daicel Chiraldak AD-3 column (Hex/ⁱPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 25.7 min (minor), 42.7 min (major)); [α]_D = -3.99 (c

0.48, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.79-7.77 (3H, m), 7.65 (1H, s), 7.44-7.42 (2H, m), 7.36 (1H, dd, J = 8.25, 1.37 Hz), 3.57 (1H, td, J = 10.31, 4.12 Hz), 2.88-2.84 (1H, m), 2.85 (3H, s), 2.76-2.75 (1H, m), 2.75 (3H, s), 2.70 (1H, dd, J = 15.12, 4.12 Hz), 1.48 (9H, s), 0.95 (3H, d, J = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 175.1, 170.8, 139.4, 133.3, 132.4, 127.9, 127.6, 127.5, 127.1, 126.2, 125.8, 125.3, 80.5, 46.1, 45.3, 37.9, 37.2, 35.3, 28.0, 16.2.

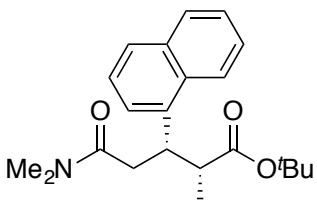
tert-butyl



(2S,3S)-5-(dimethylamino)-2-methyl-3-(naphthalen-2-yl)-5-oxopentanoate (3ha-anti)

white solid; Mp 79-82 °C; IR (neat, cm⁻¹); 2975, 2933, 1723, 1647, 1395, 1366, 1257, 1148, 1055, 853, 819, 749; HRMS (DART) calcd for C₂₂H₃₀NO₃ [M + H]⁺ 356.2226, found 356.2235; HPLC analysis using Daicel Chiralpak OD-3 column (Hex/^tPrOH = 95/5, 0.7 mL/min, 210 nm, t_R = 19.0 min (minor), 23.8 min (major)); [α]_D = 4.51 (c 0.18, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.77-7.75 (3H, m), 7.66 (1H, s), 7.42-7.41 (3H, m), 3.61 (1H, td, J = 8.59, 5.04 Hz), 2.87-2.83 (2H, m), 2.80 (3H, s), 2.79 (3H, s), 2.77-2.76 (1H, m), 1.26 (3H, d, J = 6.87 Hz), 1.13 (9H, s); ¹³C NMR (150 MHz, CDCl₃) δ: 174.2, 171.2, 140.4, 133.2, 132.3, 127.7, 127.5, 127.4, 126.8, 126.8, 125.7, 125.3, 80.0, 45.7, 45.4, 37.2, 36.4, 35.4, 27.6, 15.6.

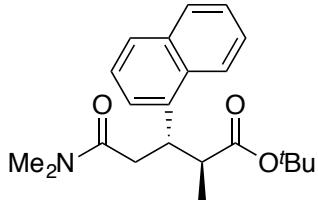
tert-butyl



(2R,3S)-5-(dimethylamino)-2-methyl-3-(naphthalen-1-yl)-5-oxopentanoate (3ia-syn)

colorless oil; IR (neat, cm⁻¹); 2976, 2933, 1722, 1649, 1456, 1395, 1367, 1259, 1150, 950, 848, 799, 781; HRMS (DART) calcd for C₂₂H₃₀NO₃ [M + H]⁺ 356.2226, found 356.2230; HPLC analysis using Daicel Chiralpak AD-3 column (Hex/^tPrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 16.6 min (minor), 20.2 min (major)); [α]_D = 47.06 (c 0.20, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 8.31 (1H, d, J = 10.00 Hz), 7.82 (1H, d, J = 8.25 Hz), 7.71 (1H, d, J = 8.25 Hz), 7.53 (1H, t, J = 7.56 Hz), 7.46-7.42 (2H, m), 7.36 (1H, d, J = 6.87 Hz), 4.39 (1H, br s), 2.92 (1H, br s), 2.84-2.82 (2H, m), 2.81 (3H, s), 2.67 (3H, s), 1.46 (9H, s), 0.92 (3H, d, J = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 175.3, 170.9, 139.1, 133.8, 132.8, 128.6, 127.0, 125.9, 125.4, 125.1, 123.6, 123.2, 80.5, 46.9, 45.3, 38.0, 37.2, 35.3, 28.0, 16.3.

tert-butyl

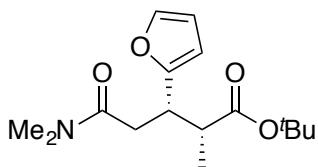


(2*S*,3*S*)-5-(dimethylamino)-2-methyl-3-(naphthalen-1-yl)-5-

oxopentanoate (3ia-anti); colorless oil; IR (neat, cm^{-1}); 2976, 2932, 1723, 1646, 1458, 1396, 1367, 1258, 1152, 1057, 849, 798, 780; HRMS (DART) calcd for $\text{C}_{22}\text{H}_{30}\text{NO}_3$ $[\text{M} + \text{H}]^+$ 356.2226, found 356.2226; HPLC analysis using

Daicel Chiraldpak OD-3 column (Hex/*i*PrOH = 95/5, 0.7 mL/min, $t_{\text{R}} = 19.3$ min (minor), 24.6 min (major)); $[\alpha]_D = 15.65$ (*c* 0.23, CHCl_3); ^1H NMR (600 MHz, CDCl_3) δ : 8.26 (1H, d, *J* = 8.94 Hz), 7.73 (1H, d, *J* = 8.25 Hz), 7.62 (1H, d, *J* = 8.25 Hz), 7.47-7.44 (1H, m), 7.38 (1H, t, *J* = 7.22 Hz), 7.34-7.30 (2H, m), 4.41 (1H, br s), 2.90-2.88 (1H, m), 2.75-2.73 (2H, m), 2.65 (3H, s), 2.64 (3H, s), 1.18 (3H, d, *J* = 6.87 Hz), 1.01 (9H, s); ^{13}C NMR (150 MHz, CDCl_3) δ : 174.4, 171.2, 139.5, 133.9, 131.9, 128.5, 127.0, 126.0, 125.4, 124.9, 123.9, 123.6, 80.0, 45.8, 38.5, 37.2, 35.8, 35.4, 27.5, 14.9.

tert-butyl (2*R*,3*S*)-5-(dimethylamino)-3-(furan-2-yl)-2-methyl-5-oxopentanoate (3ja,



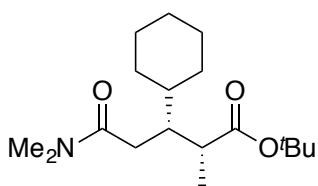
syn/anti = 58:42); colorless oil; HRMS (DART) calcd for

$\text{C}_{16}\text{H}_{26}\text{NO}_4$ $[\text{M} + \text{H}]^+$ 296.1862, found 296.1858; HPLC

analysis using Daicel Chiraldpak AD-3 column (Hex/*i*PrOH = 95/5, 0.7 mL/min, 210 nm, $t_{\text{R}} = 20.8$ min (*syn*, minor), 21.9 min (*anti*, minor), 23.0 min (*anti*, major), 28.4 min (*syn*, major)); *Syn*-form: ^1H NMR (600 MHz, CDCl_3) δ : 7.29-7.29 (1H, m), 6.26-6.26 (1H, m), 6.08-6.07 (1H, m), 3.65-3.62 (1H, m), 2.92 (3H, s), 2.87 (3H, s), 2.84-2.70 (2H, m), 2.53 (1H, dd, *J* = 15.12, 4.12 Hz), 1.44 (9H, s), 1.00 (3H, d, *J* = 6.87 Hz); ^{13}C NMR (150 MHz, CDCl_3) δ : 174.3, 170.7, 154.9, 141.0, 110.0, 106.8, 80.3, 44.1, 38.5, 37.1, 35.4, 35.0, 27.9, 14.7; *Anti*-form: ^1H NMR (600 MHz, CDCl_3) δ : 7.29-7.29 (1H, m), 6.26-6.26 (1H, m), 6.08-6.07 (1H, m), 3.65-3.62 (1H, m), 2.95 (3H, s), 2.90 (3H, s), 2.84-2.70 (3H, m), 1.37 (9H, s), 1.15 (3H, d, *J* = 6.87 Hz); ^{13}C NMR (150 MHz, CDCl_3) δ : 173.8, 170.7, 154.9, 140.9, 110.0, 106.3, 80.1, 43.6, 38.5, 37.1, 35.3, 33.9, 27.8, 14.7.

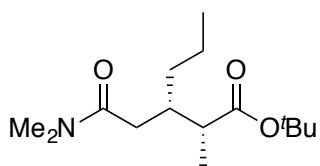
tert-butyl (2*R*,3*R*)-3-cyclohexyl-5-(dimethylamino)-2-methyl-5-oxopentanoate (3ka,

syn/anti = 87:13); colorless oil; HRMS (DART) calcd for



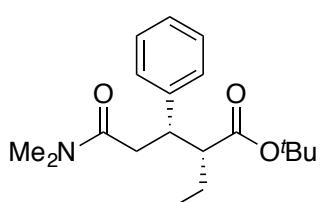
$C_{18}H_{34}NO_3$ [M + H]⁺ 312.2539, found 312.2537; HPLC analysis using Daicel Chiralpak AD-3 column (Hex/ⁱPrOH = 100/1, 0.7 mL/min, 210 nm, t_R = 30.6 min (*anti*, minor), 35.5 min (*anti*, major), 38.4 min (*syn*, minor), 43.2 min (*syn*, major)); *Syn*-form: ¹H NMR (600 MHz, CDCl₃) δ: 2.96 (s, 3H), 2.87 (s, 3H), 2.57-2.53 (m, 1H), 2.47 (m, 1H), 2.20-2.14 (m, 2H), 1.67-1.54 (m, 6H), 1.36 (s, 9H), 1.26-1.25 (m, 2H), 1.13-1.07 (m, 2H), 0.96 (d, 2H, J = 7.56 Hz), 0.94-0.92 (m, 2H); ¹³C NMR (150 MHz, CDCl₃) δ: 175.9, 172.4, 79.8, 41.7, 41.6, 40.3, 37.1, 35.6, 31.2, 28.0, 27.9, 26.7, 26.6, 26.4, 13.4; *Anti*-form: ¹H NMR (600 MHz, CDCl₃) δ: 2.97 (s, 3H), 2.87 (s, 3H), 2.57-2.53 (m, 1H), 2.47 (m, 1H), 2.20-2.14 (m, 2H), 1.67-1.54 (m, 6H), 1.37 (s, 9H), 1.26-1.25 (m, 2H), 1.13-1.07 (m, 2H), 1.03 (d, 2H, J = 7.56 Hz), 0.94-0.92 (m, 2H); ¹³C NMR (150 MHz, CDCl₃, detectable peaks) δ: 175.7, 170.2, 79.8, 32.0, 29.5, 13.4.

tert-butyl (2*R*,3*S*)-3-(dimethylamino)-2-oxoethyl)-2-methylhexanoate (3la,



syn/anti = 52:48); colorless oil; HRMS (DART) calcd for $C_{15}H_{30}NO_3$ [M + H]⁺ 272.2226, found 272.2232; HPLC analysis using Daicel Chiralpak AS-3 column (Hex/ⁱPrOH = 99/1, 0.3 mL/min, 210 nm, t_R = 20.7 min (*syn*, minor), 27.0 min (*anti*, minor), 29.0 min (*syn*, major), 30.6 min (*anti*, major)); *Syn*-form: ¹H NMR (600 MHz, CDCl₃) δ: 2.95 (6H, s), 2.58-2.52 (1H, m), 2.46 (1H, dd, J = 15.81, 5.50 Hz), 2.28-2.25 (1H, m), 2.18 (1H, m), 1.45 (9H, s), 1.36-1.28 (4H, m), 1.05 (3H, d, J = 6.87 Hz), 0.91-0.88 (3H, m); ¹³C NMR (150 MHz, CDCl₃) δ: 175.2, 172.2, 79.9, 42.0, 37.3, 37.2, 35.4, 34.7, 34.3, 28.0, 19.9, 14.1, 12.8; *Anti*-form: ¹H NMR (600 MHz, CDCl₃) δ: 3.02 (6H, s), 2.58-2.52 (1H, m), 2.36 (1H, dd, J = 14.43, 4.81 Hz), 2.28-2.25 (2H, m), 1.45 (9H, s), 1.36-1.28 (4H, m), 1.07 (3H, d, J = 6.87 Hz), 0.91-0.88 (3H, m); ¹³C NMR (150 MHz, CDCl₃, detectable peaks) δ: 175.2, 172.2, 79.9, 41.9, 37.2, 35.4, 34.7, 33.8, 28.0, 20.3, 14.2, 13.4.

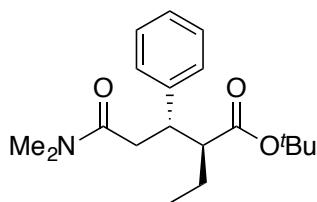
tert-butyl (2*R*,3*S*)-5-(dimethylamino)-2-ethyl-5-oxo-3-phenylpentanoate (3ae-syn);



colorless solid; Mp 75-77 °C; IR (neat, cm⁻¹); 2966, 2934, 1722, 1650, 1456, 1395, 1367, 1267, 1151, 763, 703; HRMS (DART) calcd for $C_{19}H_{30}NO_3$ [M + H]⁺ 320.2226, found 320.2231; HPLC analysis using Daicel Chiralpak AS-3

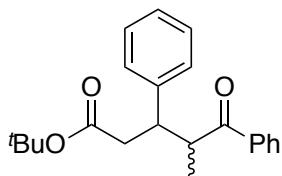
column (Hex/*i*PrOH = 99/1, 1.0 mL/min, 210 nm, t_R = 27.3 min (minor), 54.9 min (major)); $[\alpha]_D$ = -9.96 (c 0.31, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ : 7.28-7.26 (2H, m), 7.19-7.18 (3H, m), 3.37 (1H, td, J = 10.65, 3.67 Hz), 2.82 (3H, s), 2.77-2.74 (1H, m), 2.76 (3H, s), 2.57 (1H, dd, J = 14.78, 3.78 Hz), 2.47 (1H, td, J = 10.48, 3.90 Hz), 1.48 (9H, s), 1.42-1.39 (1H, m), 1.26-1.20 (1H, m), 0.80 (3H, t, J = 7.56 Hz); ¹³C NMR (150 MHz, CDCl₃) δ : 174.5, 170.8, 142.1, 128.2, 128.0, 126.5, 80.5, 54.0, 44.6, 37.8, 37.1, 35.2, 28.0, 24.1, 11.6.

tert-butyl (2*R*,3*S*)-5-(dimethylamino)-2-ethyl-5-oxo-3-phenylpentanoate (3ae-anti);



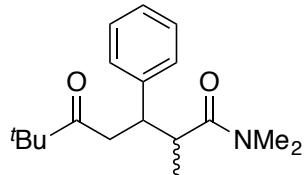
colorless solid; Mp 61-64 °C; IR (neat, cm⁻¹); 2968, 2934, 1723, 1646, 1456, 1395, 1365, 1268, 1152, 755, 701; HRMS (DART) calcd for C₁₉H₃₀NO₃ [M + H]⁺ 320.2226, found 320.2220; HPLC analysis using Daicel Chiralpak AD-3 column (Hex/*i*PrOH = 95/5, 1.0 mL/min, 210 nm, t_R = 18.7 min (major), 19.8 min (minor)); $[\alpha]_D$ = -9.01 (c 0.28, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ : 7.27-7.15 (5H, m), 3.40 (1H, td, J = 9.28, 4.81 Hz), 2.80 (3H, s), 2.76 (3H, s), 2.74 (1H, dd, J = 15.12, 4.81 Hz), 2.63 (1H, dd, J = 15.12, 8.94 Hz), 2.58 (1H, td, J = 10.14, 3.89 Hz), 1.71-1.63 (2H, m), 1.15 (9H, s), 0.94 (3H, t, J = 7.22 Hz); ¹³C NMR (150 MHz, CDCl₃) δ : 173.4, 171.2, 142.4, 128.4, 127.9, 126.5, 79.9, 53.4, 44.6, 37.1, 37.1, 35.3, 26.6, 23.8, 11.9.

tert-butyl (3*R*,4*S*)-4-methyl-5-oxo-3,5-diphenylpentanoate (3mg, dr = 68:32);



colorless oil; HRMS (DART) calcd for C₂₂H₂₇O₃ [M + H]⁺ 339.1960, found 339.1965; major isomer: ¹H NMR (600 MHz, CDCl₃) 8.01-8.00 (m, 2H), 7.59-7.18 (m, 8H), 3.77-3.75 (m, 1H), 3.55-3.53 (m, 1H), 2.64-2.60 (m, 1H), 2.48 (dd, 1H, J = 14.74, 10.77 Hz), 1.19 (s, 9H), 0.98 (d, 3H, J = 6.80 Hz); ¹³C NMR (150 MHz, CDCl₃) δ : 203.6, 170.7, 141.2, 136.8, 133.1, 128.7, 128.2, 128.1, 127.9, 126.7, 80.1, 45.5, 45.0, 40.4, 27.7, 16.6; minor isomer: ¹H NMR (600 MHz, CDCl₃) δ : 7.79-7.78 (m, 2H), 7.59-7.18 (m, 8H), 3.83-3.82 (m, 1H), 3.62-3.59 (m, 1H), 2.77 (dd, 1H, J = 14.74, 5.10 Hz), 2.64-2.60 (m, 1H), 1.24 (d, 3H, J = 6.80 Hz), 1.21 (s, 9H); ¹³C NMR (150 MHz, CDCl₃, detectable peaks) δ : 203.2, 171.2, 136.7, 132.7, 128.4, 128.2, 128.0, 128.0, 126.5, 80.1, 46.0, 44.1, 38.1, 28.1, 14.7.

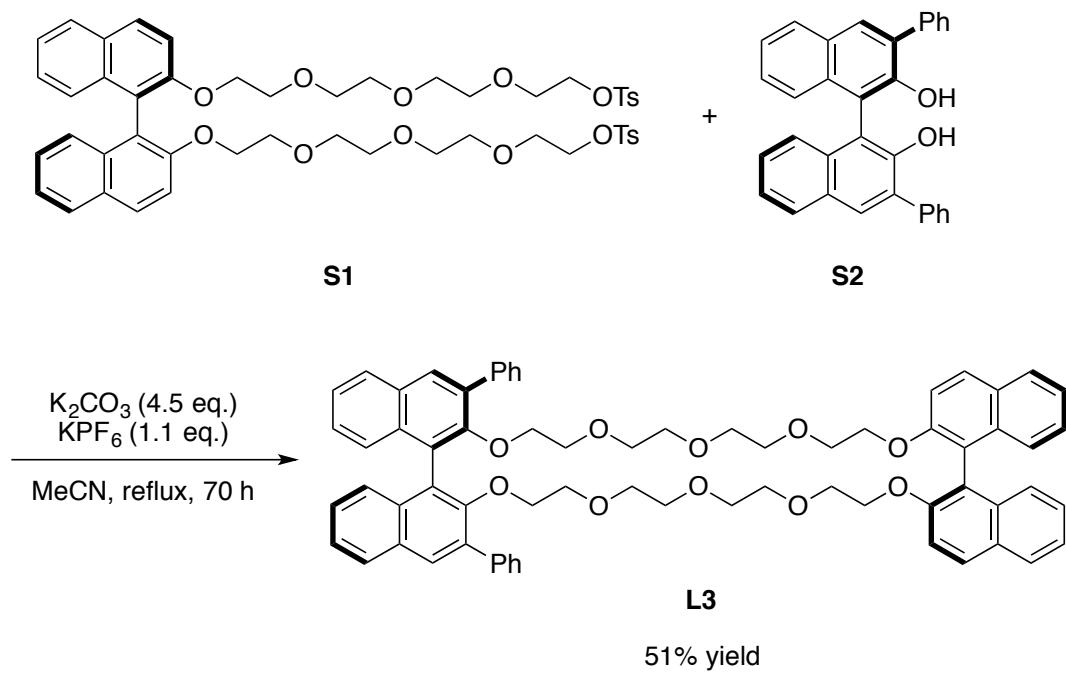
N,N,2,6,6-pentamethyl-5-oxo-3-phenylheptanamide (3nf, dr = 55:45); colorless oil;



HRMS (DART) calcd for $C_{18}H_{28}NO_2$ [M + H]⁺ 290.2120, found 290.2121; major isomer: ¹H NMR (600 MHz, CDCl₃) δ: 7.28-7.13 (5H, m), 3.60 (1H, td, J = 8.59, 3.89 Hz), 3.09-2.97 (2H, m), 2.91 (3H, s), 2.81 (1H, dd, J = 17.18, 4.12 Hz), 2.76 (3H, s), 1.13 (3H, d, J = 6.87 Hz), 1.02 (9H, s); ¹³C NMR (150 MHz, CDCl₃) δ: 213.8, 175.0, 143.3, 128.2, 128.1, 126.3, 44.3, 42.5, 39.8, 37.2, 37.1, 35.5, 26.0, 16.5; minor isomer: ¹H NMR (600 MHz, CDCl₃) δ: 7.28-7.13 (5H, m), 3.50 (1H, td, J = 9.11, 3.89 Hz), 3.17-3.16 (1H, m), 3.09-2.97 (2H, m), 2.95 (3H, s), 2.94 (3H, s), 0.95-0.94 (3H, m), 0.95 (9H, s); ¹³C NMR (150 MHz, CDCl₃) δ: 213.9, 175.3, 142.7, 128.2, 127.8, 126.4, 44.1, 44.1, 40.4, 40.0, 37.7, 35.6, 26.2, 14.3.

Synthesis of L3

Scheme S1

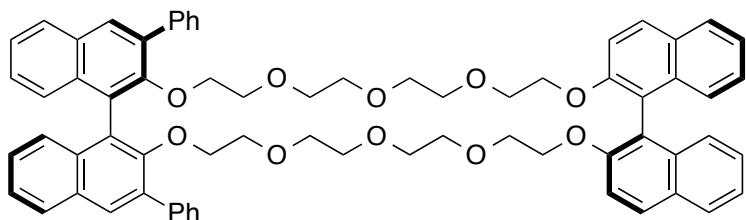


(R)-S1¹ (1.19 g, 1.26 mmol), (R)-S2³ (0.55 g, 1.26 mmol), K₂CO₃ (0.78 g, 5.65 mmol) and KPF₆ (0.25 g, 1.38 mmol) were placed in a flame-dried flask that was

fulfilled with argon, and MeCN (80 mL) was added. The reaction mixture was stirred for 70 h under reflux condition. After that, the reaction mixture was cooled to room temperature followed by filtered through Celite. The filtrate was evaporated under reduced pressure, and DCM (100 mL) was then added. The DCM solution was washed with 1 M HCl aq. (30 mL x 3), 1 M NaOH aq. (30 mL x 2) and brine (30 mL). The organic layer was dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the crude product obtained was purified by silica-gel column chromatography (hexane-ethyl acetate) to afford the desired product **L3** (0.67 g, 51 % yield). The product was dried for 10 h at 100 °C under reduced pressure to remove a trace amount of water.

6,45-diphenyl-8,9,11,12,14,15,17,18,33,34,36,37,39,40,42,43-hexadecahydrotetrapheno[2,1-e₁:1',2'-g₁:2'',1''-n:1''',2'''-p][1,4,7,10,13,18,21,24,27,30]decaoxacyclotetra-

riacontine (L3); white



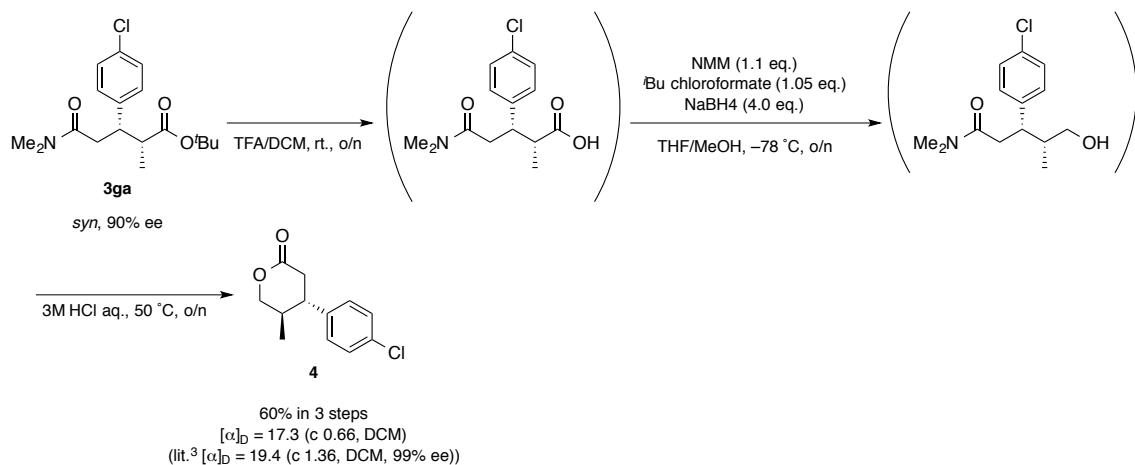
solid; Mp 79-82 °C; IR (neat, cm⁻¹); 3055, 2871, 2361, 2340, 1592, 1507, 1421, 1352, 1128, 892, 807, 751, 700; HRMS (ESI)

calcd for C₆₈H₆₄NaO₁₀ [M + Na]⁺ 1063.4397, found 1063.4374; [α]_D = 30.14 (c 0.58, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ: 7.88 (2H, s), 7.82 (4H, d, J = 8.25 Hz), 7.76 (2H, d, J = 8.25 Hz), 7.68 (4H, d, J = 6.87 Hz), 7.37 (4H, t, J = 7.90 Hz), 7.32-7.31 (4H, m), 7.28 (2H, t, J = 7.56 Hz), 7.24-7.22 (2H, m), 7.19-7.16 (2H, m), 7.14-7.12 (4H, m), 7.06 (2H, d, J = 8.25 Hz), 4.03-3.99 (2H, m), 3.93-3.90 (2H, m), 3.58-3.56 (2H, m), 3.41-3.37 (2H, m), 3.31-3.29 (4H, m), 3.23-3.15 (8H, m), 3.13-3.06 (4H, m), 3.03-2.96 (6H, m), 2.88-2.84 (2H, m); ¹³C NMR (150 MHz, CDCl₃, detectable peaks) δ: 154.4, 153.3, 138.8, 135.2, 134.1, 133.6, 130.7, 130.4, 129.4, 129.4, 129.2, 128.3, 128.0, 127.8, 127.4, 126.3, 126.2, 126.0, 125.8, 125.5, 125.0, 123.6, 120.5, 115.9, 72.0, 70.6, 70.4, 70.3, 70.1, 69.7, 69.6.

Determination of absolute configuration of 3ga

Syn-isomer of **3ga** was transformed into the lactone **4** (Scheme S1). **3ga** was converted into corresponding carboxylic acid by acid treatment; then it was reduced to the alcohol. Finally, it was cyclized under acidic condition to afford the lactone **4**. Through these transformations, the products were obtained as single diastereomers. The absolute configuration of the lactone **4** was determined by comparison of their optical rotations.⁵

Scheme S2



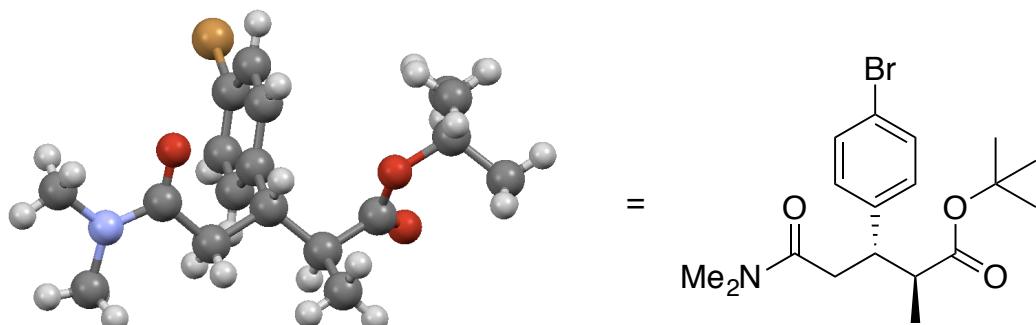
To a solution of **3ga** (83.8 mg, 2.47×10^{-1} mmol, *syn:anti* = >99:1, 90% ee) in DCM (2 mL) was added TFA (2 mL) at 0 °C, then the reaction mixture was warmed to room temperature and stirred for overnight. After stirring, TFA and DCM were removed by evaporator to obtain crude oil. It was dissolved in 1M NaOH aq., and washed with Et₂O for three times. Water layer was acidified with conc. HCl to pH 2, then precipitations appeared was extracted with DCM for five times. The organic layer was evaporated to obtain carboxylic acid as white solid. It was used to next reaction without further purification. To a solution of the carboxylic acid in THF (2.0 mL) were added *N*-methylmorpholine (29.2 μ L, 2.66×10^{-1} mmol, 1.1 eq.) and ⁷Bu chloroformate (33.4 μ L, 2.54×10^{-1} mmol, 1.05 eq.) at 0 °C. After stirring for 1h, the reaction mixture was added to a solution of NaBH₄ (36.6 mg, 9.67×10^{-1} mmol, 4.0 eq.) in THF/MeOH = 7/3 (2.0 mL) at -78 °C through cannula with extra THF (1.5 mL). The reaction mixture was stirred for overnight, then the reaction was quenched by 10% AcOH aq. (1.25 mL) and diluted with H₂O (6 mL). The organic layer was extracted with DCM for three times

and washed with brine, then dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, crude product of the alcohol was obtained. This crude was used for next reaction without purification. To this crude product was added 3M HCl aq. (10 mL), then the reaction mixture was stirred at 50 °C for overnight. After that, the reaction mixture was cooled to room temperature then extracted with DCM for five times. The combined organic layer was washed with brine, then dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the crude product was obtained. The crude product was purified by silica-gel PTLC (Hexane-Acetone) to afford the desired lactone **4** (33.1 mg, 1.47 x10⁻¹ mmol, 60% in 3 steps) as an white solid. The physical data was corresponded with the literature compound reported.⁵

(4*S*,5*R*)-4-(4-chlorophenyl)-5-methyltetrahydro-2*H*-pyran-2-one (4**):⁵**

White solid; [α]_D = 17.3 (c 0.66, DCM) (lit.³ [α]_D = 19.4 (99% ee, c 1.36, DCM)); ¹H NMR (600 MHz, CDCl₃) δ: 7.33 (d, 2H, *J* = 8.25 Hz), 7.12 (d, 2H, *J* = 8.94 Hz), 4.42-4.41 (m, 1H), 4.01 (t, 1H, *J* = 11.00), 2.89 (dd, 1H, *J* = 17.87, 6.19 Hz), 2.75-2.74 (m, 1H), 2.60 (dd, 1H, *J* = 17.87, 11.00 Hz), 2.17-2.15 (m, 1H), 0.82 (d, 3H, *J* = 6.87 Hz); ¹³C NMR (150 MHz, CDCl₃) δ: 170.5, 140.4, 133.0, 129.1, 128.5, 74.1, 44.8, 37.4, 34.6, 14.5.

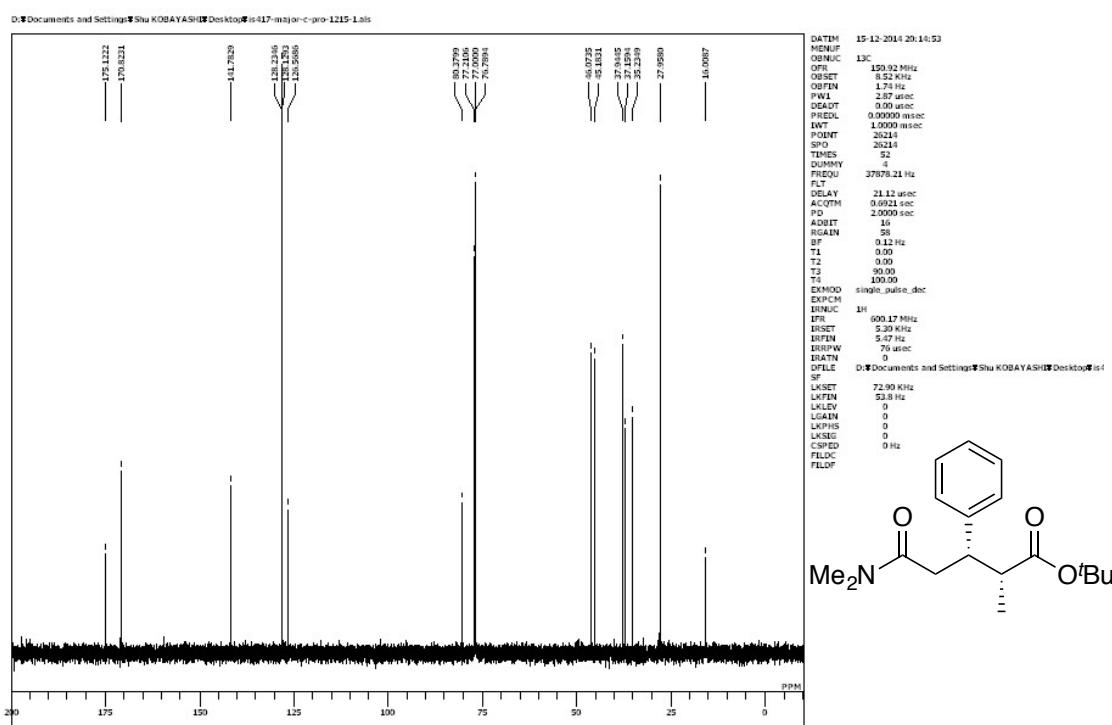
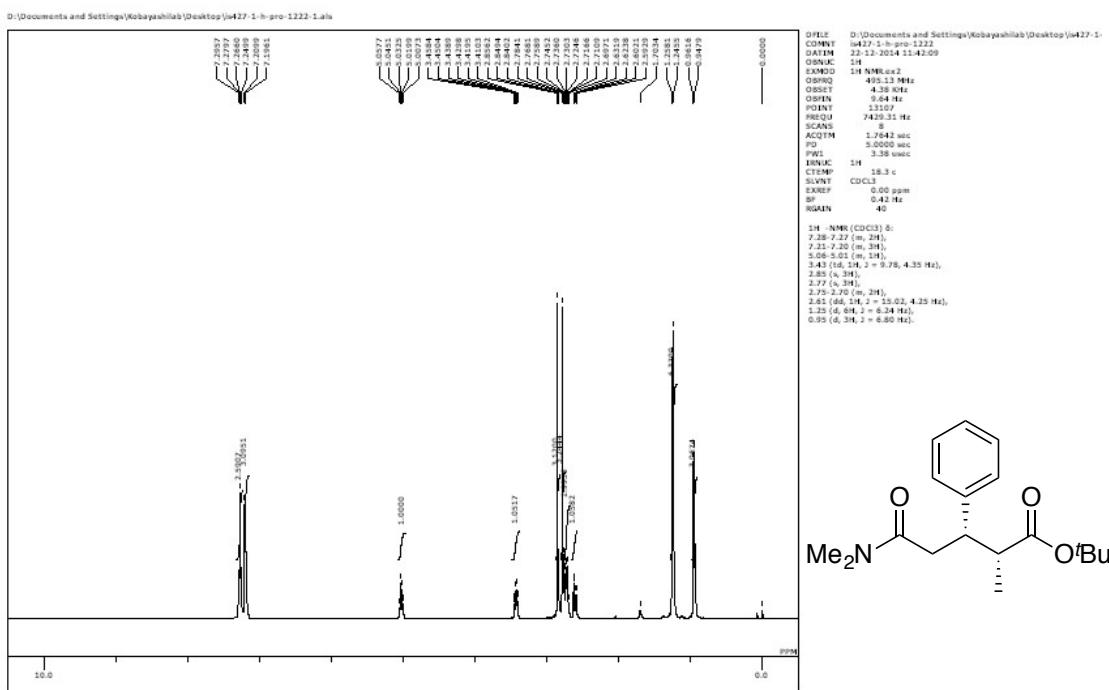
Absolute configuration of **3fa-anti** was determined by by X-ray single crystal structure analysis. The data have been deposited with the Cambridge Crystallographic Data Centre as CCDC 1482367. Copies of the data can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44(0)12238336033 or deposit@ccdc.cam.ac.uk).



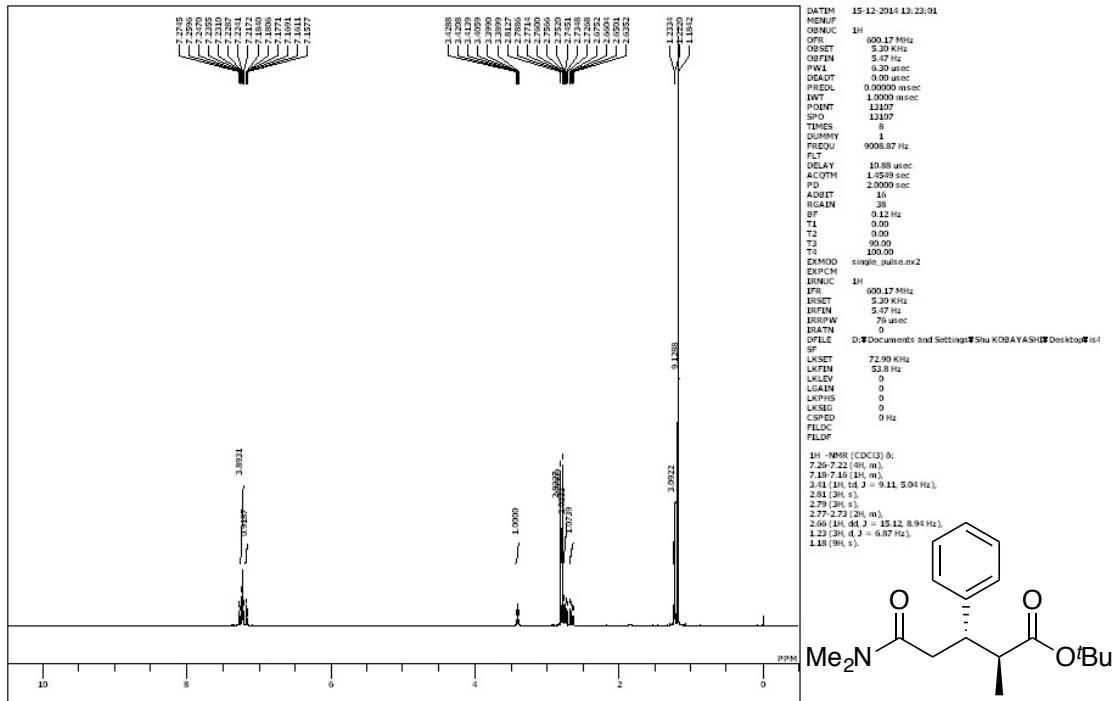
References

- 1 Suzuki, H.; Sato, I.; Yamashita, Y.; Kobayashi, S. *J. Am. Chem. Soc.* **2015**, *137*, 4336.
- 2 Tachibana, Y.; Kihara, N.; Ohga, Y.; Takata, T. *Chem. Lett.* **2000**, *5*, 806.
- 3 Wu, T. R.; Shen, L. X.; Chong, J. M. *Org. Lett.* **2004**, *6*, 2701.
- 4 Bernardi, A.; Dotti, P.; Poii, G.; Scolastico, C. *Tetrahedron* **1992**, *48*, 5597.
- 5 Landa, A.; Maestro, M.; Masdeu, C.; Puente, Á.; Vera, S.; Oiarbide, M.; Palomo, C. *Chem. Eur. J.* **2009**, *15*, 1562.

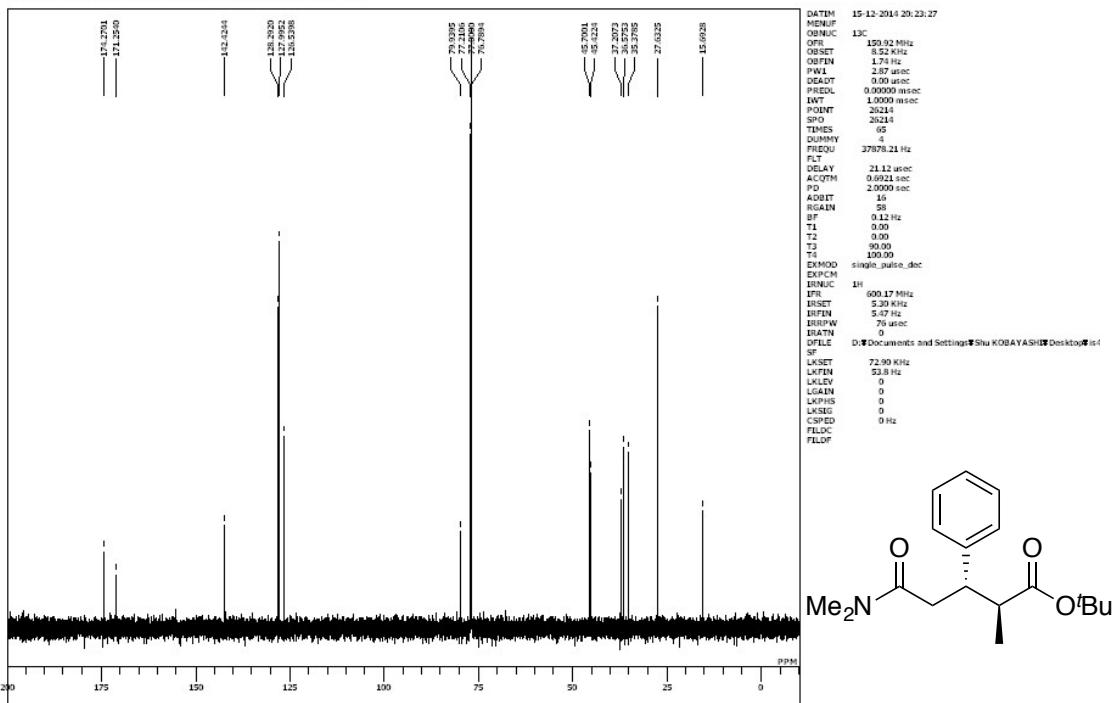
II. NMR and HPLC charts of the products

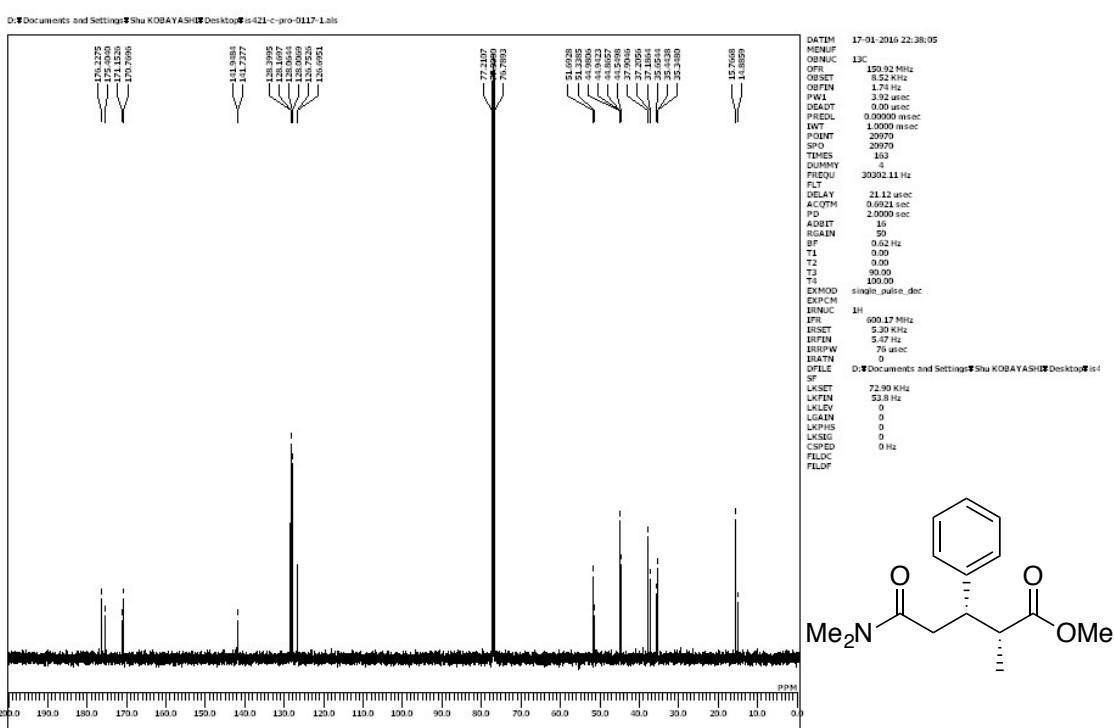
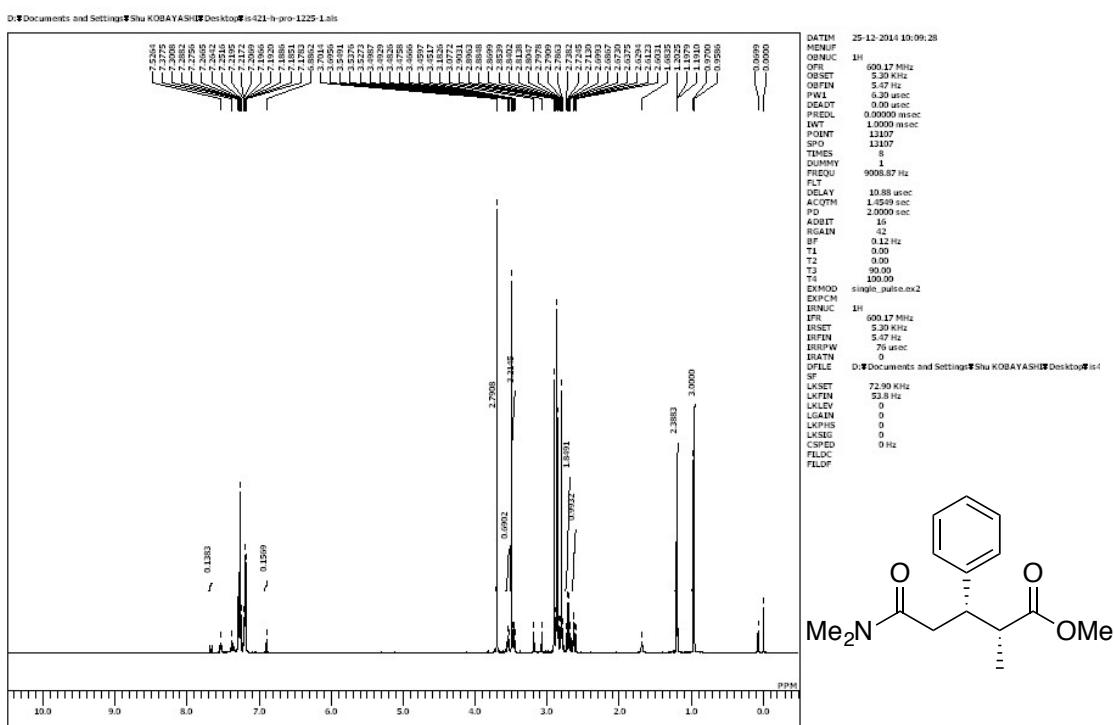


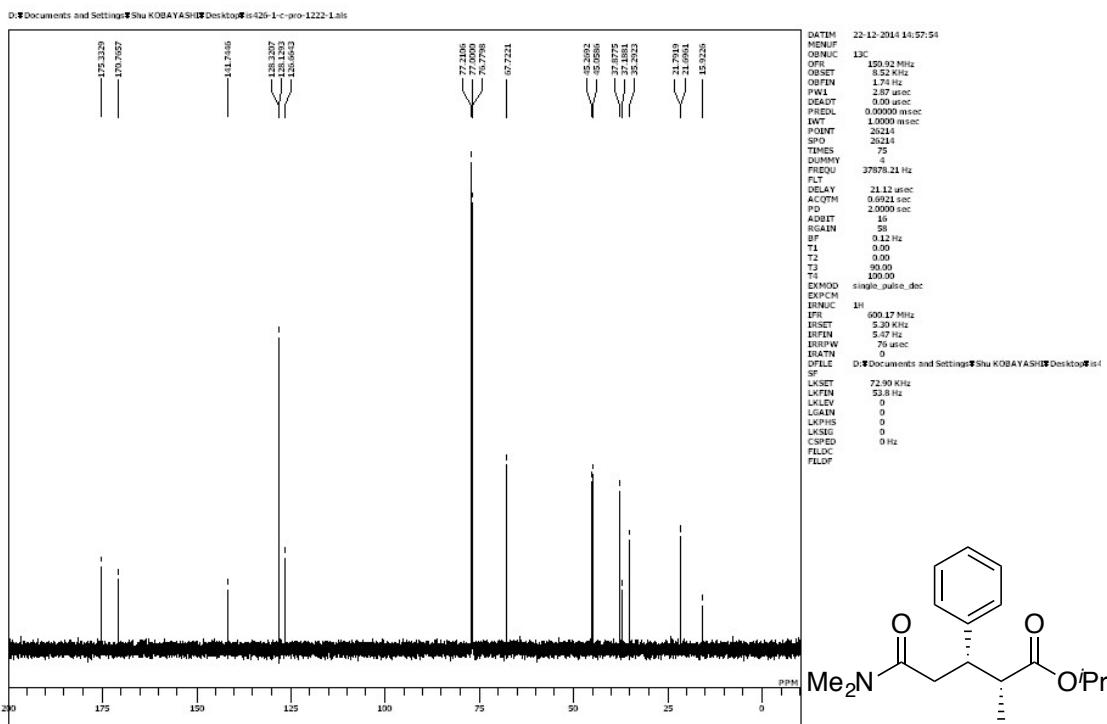
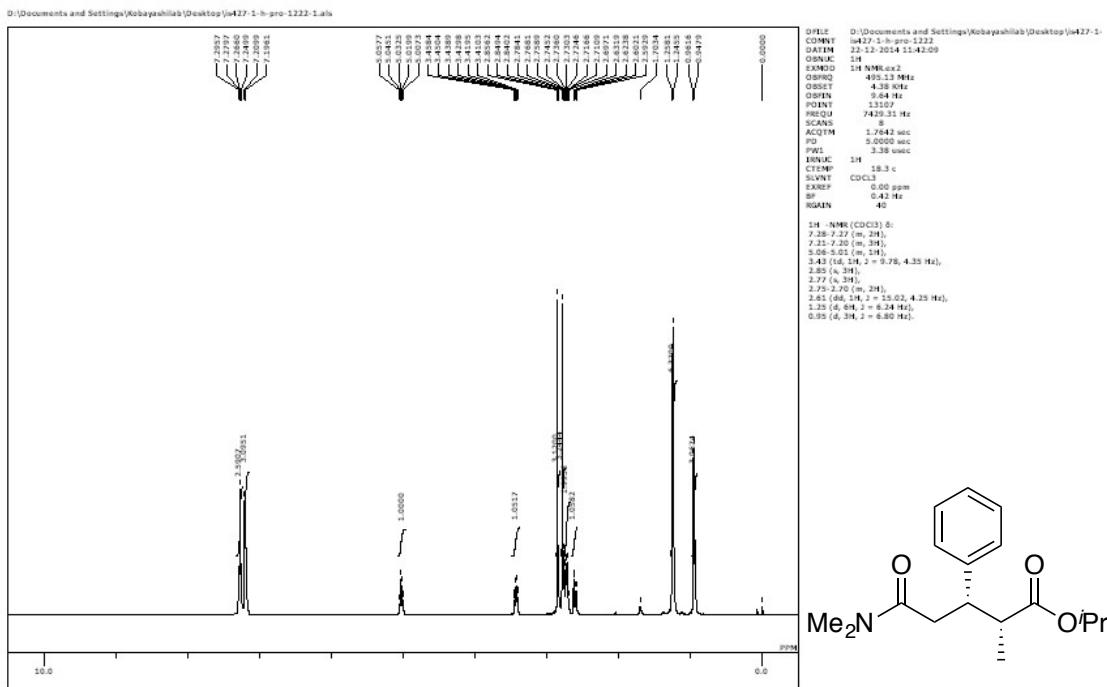
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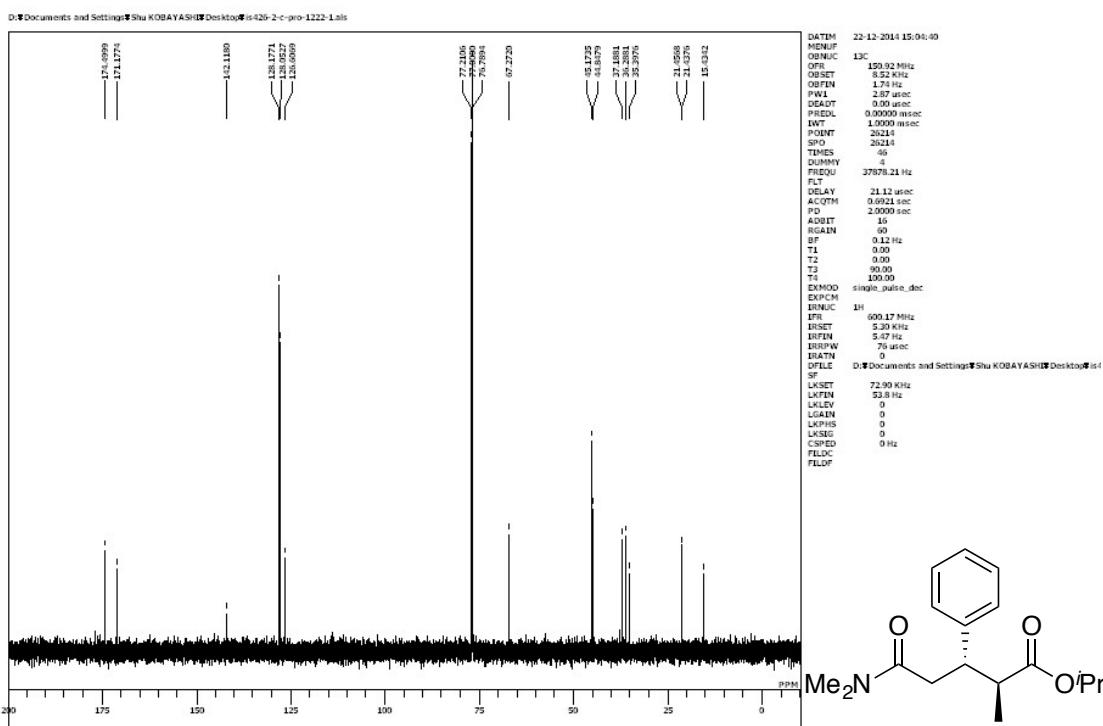
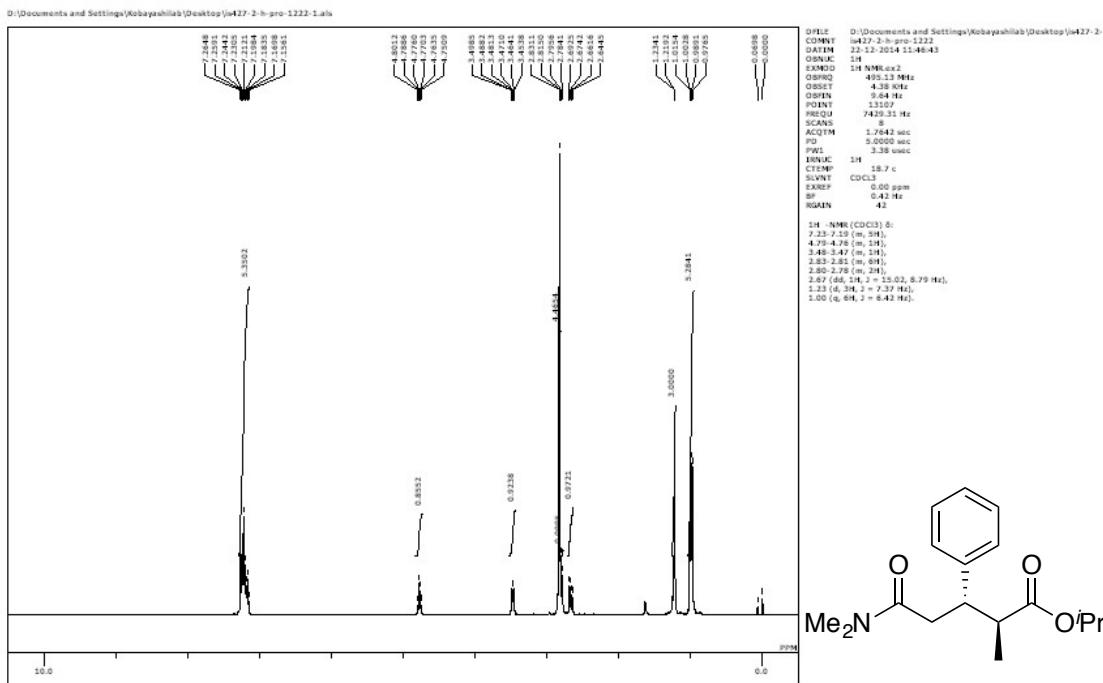


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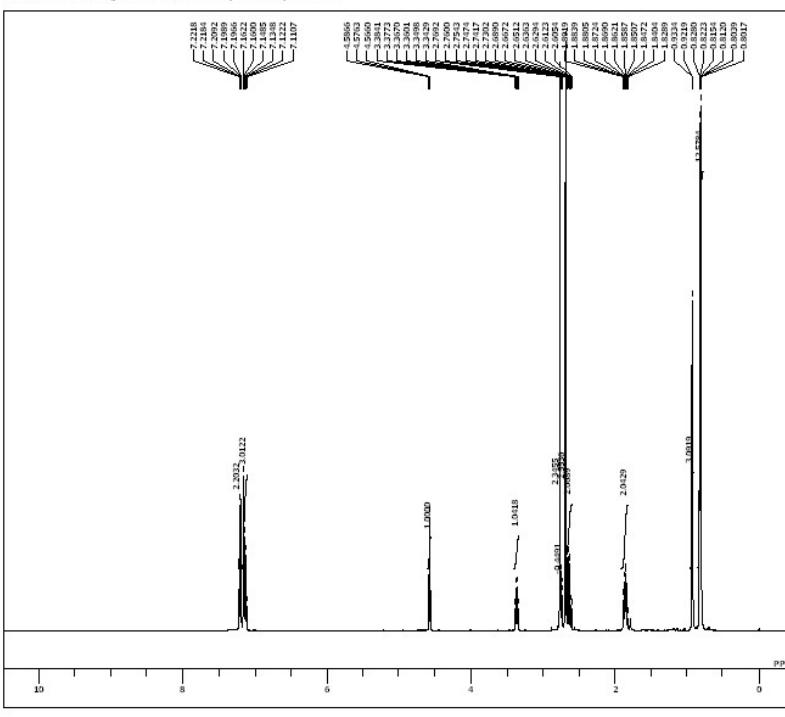








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POINT 13107

SPO 13107
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FIL

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ACQTM 1.4549 sec
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T3 90.00
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ISSET 5.30 kHz
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LXPHS 0
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CSPEQ 0 Hz
FILDF

1H-NMR (CDCl₃): δ:

7.25-7.20 (5H, m),

7.15-7.12 (1H, m),

4.58 (1H, t, J = 10.31, 4.12 Hz),

3.39 (1H, t, J = 10.31, 4.12 Hz),

2.77-2.73 (1H, m),

2.70-2.69 (1H, m),

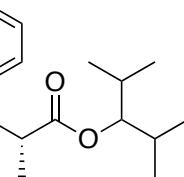
2.69 (3H, s),

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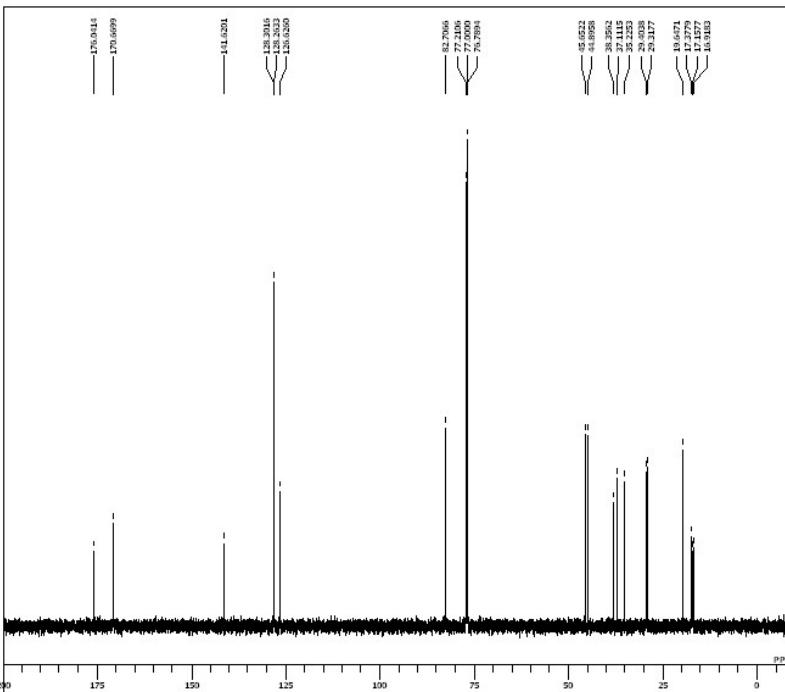
1.99-1.93 (2H, m),

0.92 (3H, d, J = 6.87 Hz),

0.82-0.81 (1H, m).



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POINT 26214

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TIMES 48
DUMMY 4
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FIL

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T3 90.00
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IFR 600.17 MHz
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LKTFN 53.8 Hz
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LXSG 0
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FILDF

13C-NMR (CDCl₃): δ:

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128.8113, 126.2600,

45.6522, 41.8935,

38.3562, 37.3113,

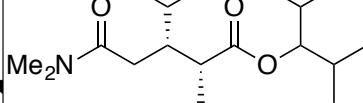
33.3533, 29.2325,

29.2327, 16.9571,

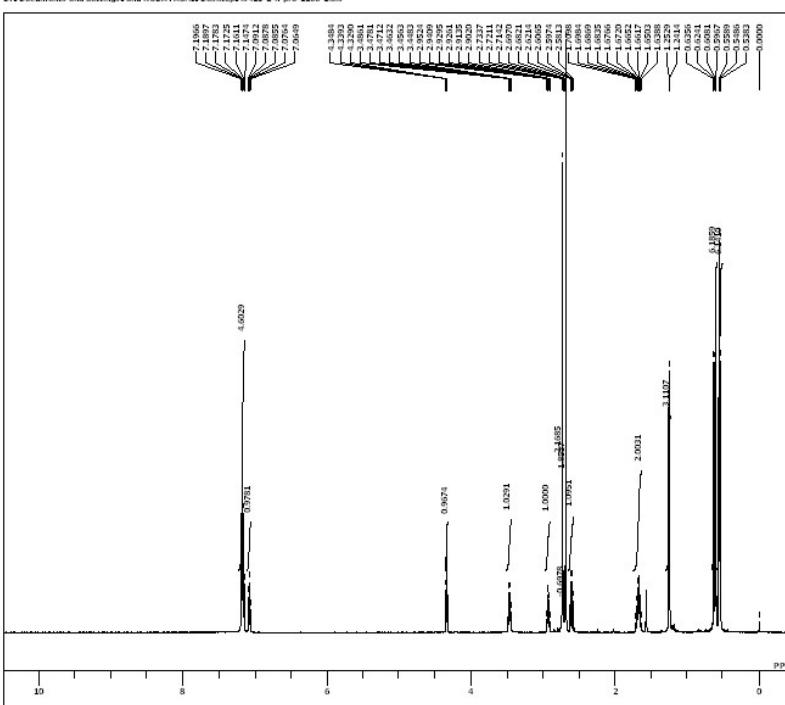
12.7777, 12.7777,

12.7777, 12.7777,

12.7777, 12.7777.



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SPO 12107

TIMES 8

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LGAIN 0

LXPHS 0

LXSG 0

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FILDC

FILDF

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4.38 (m, t, J = 5.84 Hz),

3.47-3.46 (m, m),

2.92 (m, t, J = 16.27, 7.05 Hz),

2.72-2.70 (m, m),

2.68 (m, m),

2.09 (m, d, J = 14.78, 9.28 Hz),

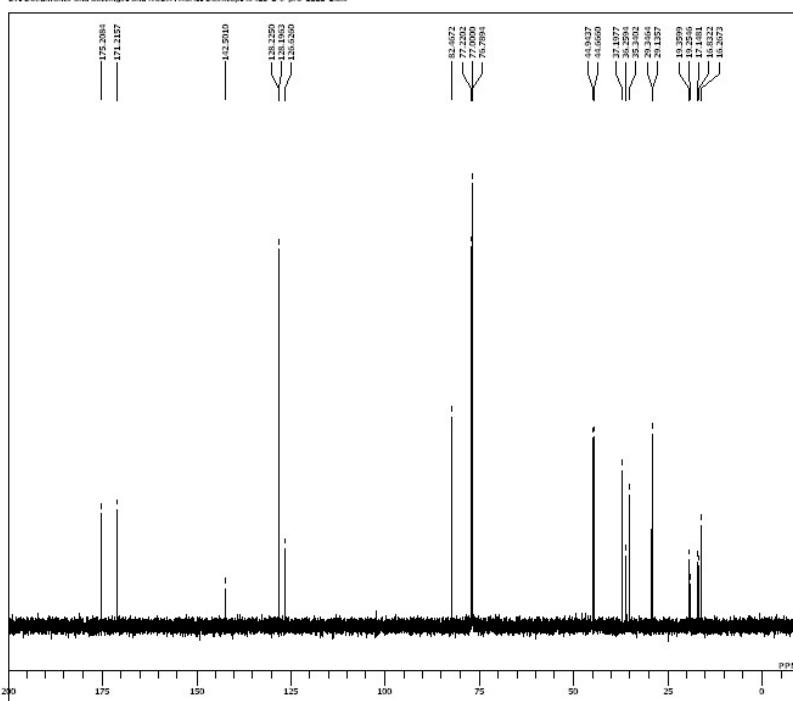
1.71-1.64 (m, m),

1.22 (m, t, J = 6.87 Hz),

0.62 (m, d, J = 16.50, 6.87 Hz),

0.55 (m, t, J = 6.19 Hz).

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13C

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OSET 8.50 Hz

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PREDL 0.00000 msec

INT 1.000 msec

POINT 20214

SPO 20214

TIMES 58

DUMMY 4

FREQU 37878.21 Hz

FLT

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LGAIN 0

LXPHS 0

LXSG 0

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FILDC

FILDF

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7.14 (d, J = 7.05 Hz),

4.38 (m, t, J = 5.84 Hz),

3.47-3.46 (m, m),

2.92 (m, t, J = 16.27, 7.05 Hz),

2.72-2.70 (m, m),

2.68 (m, m),

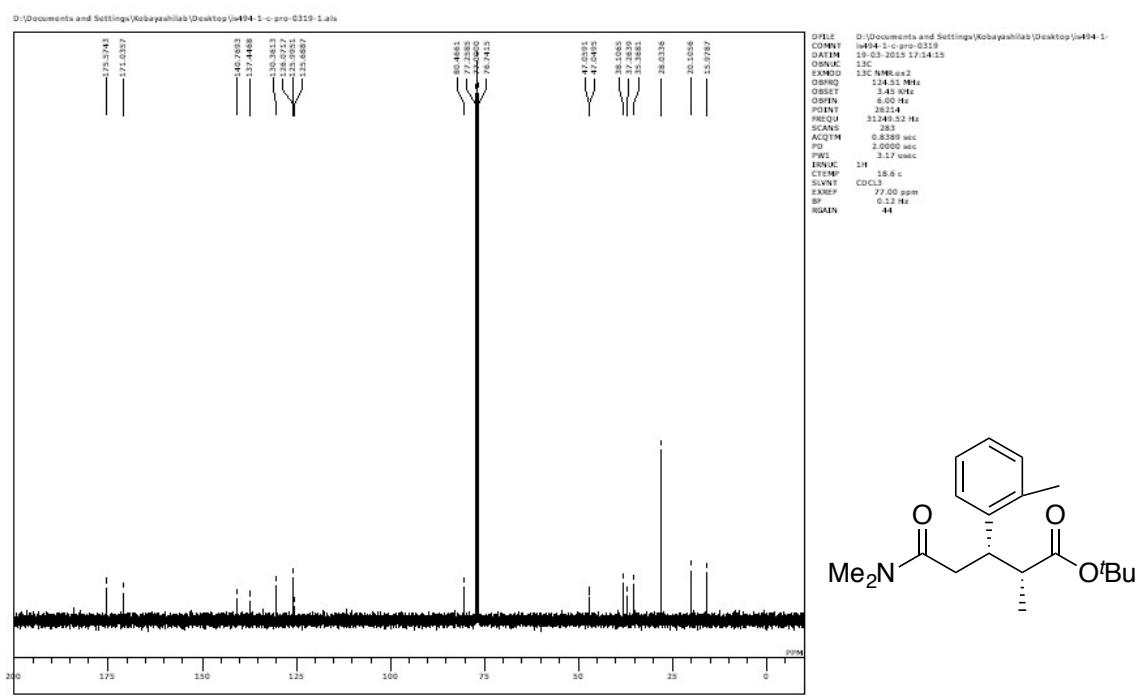
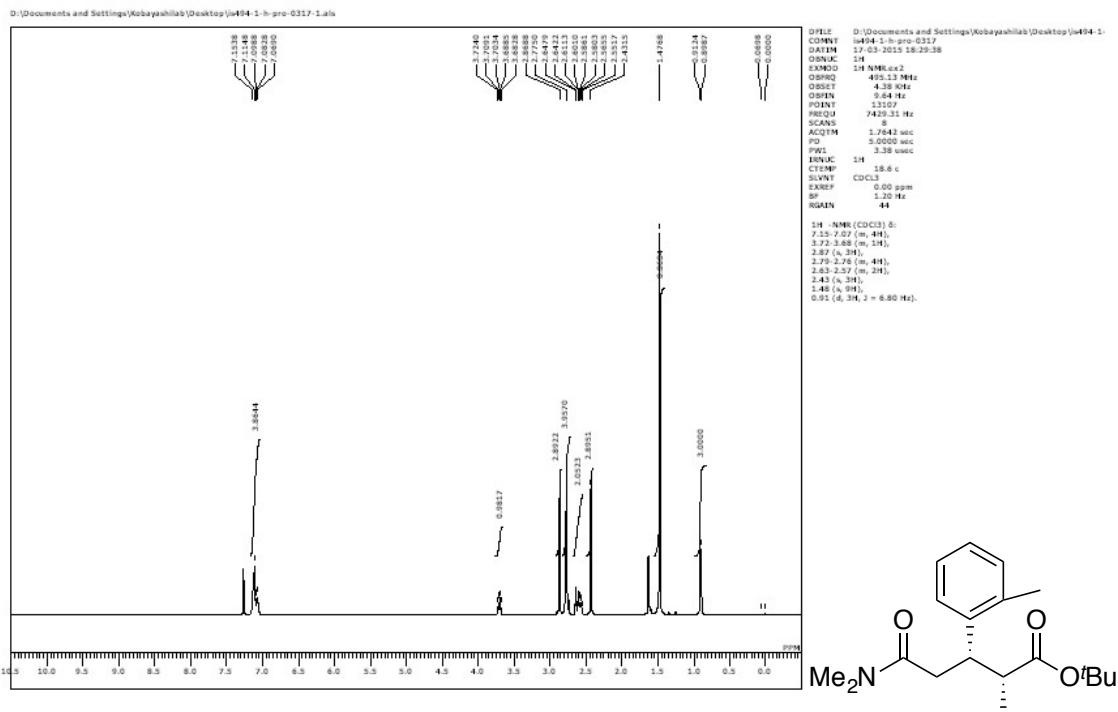
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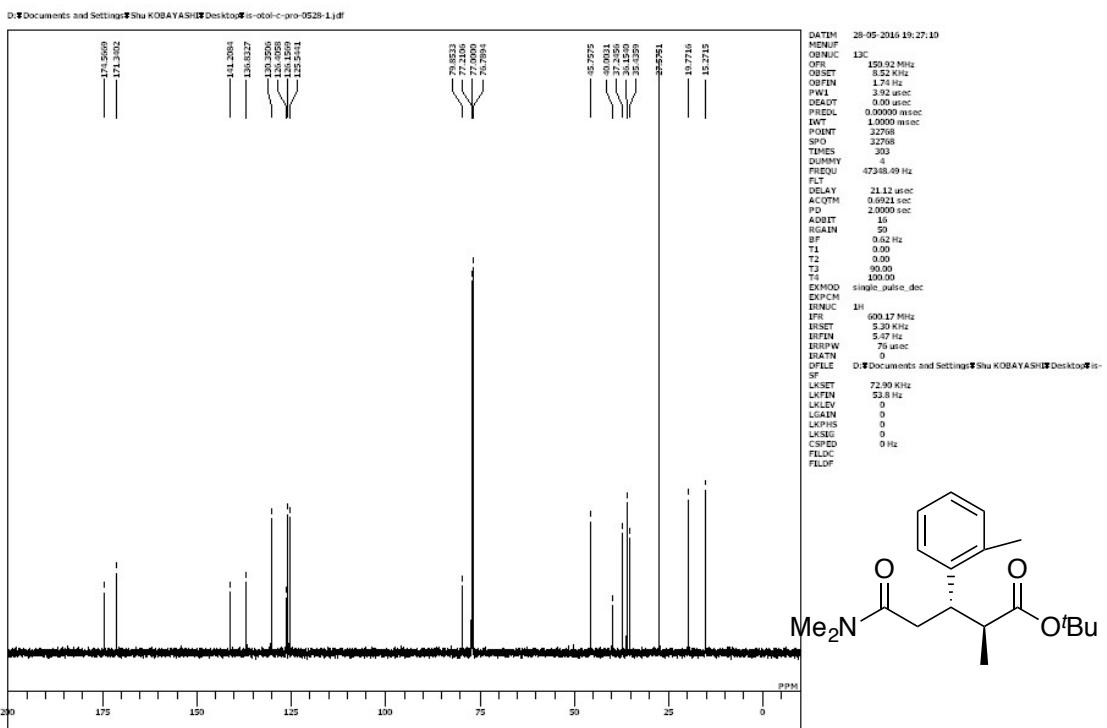
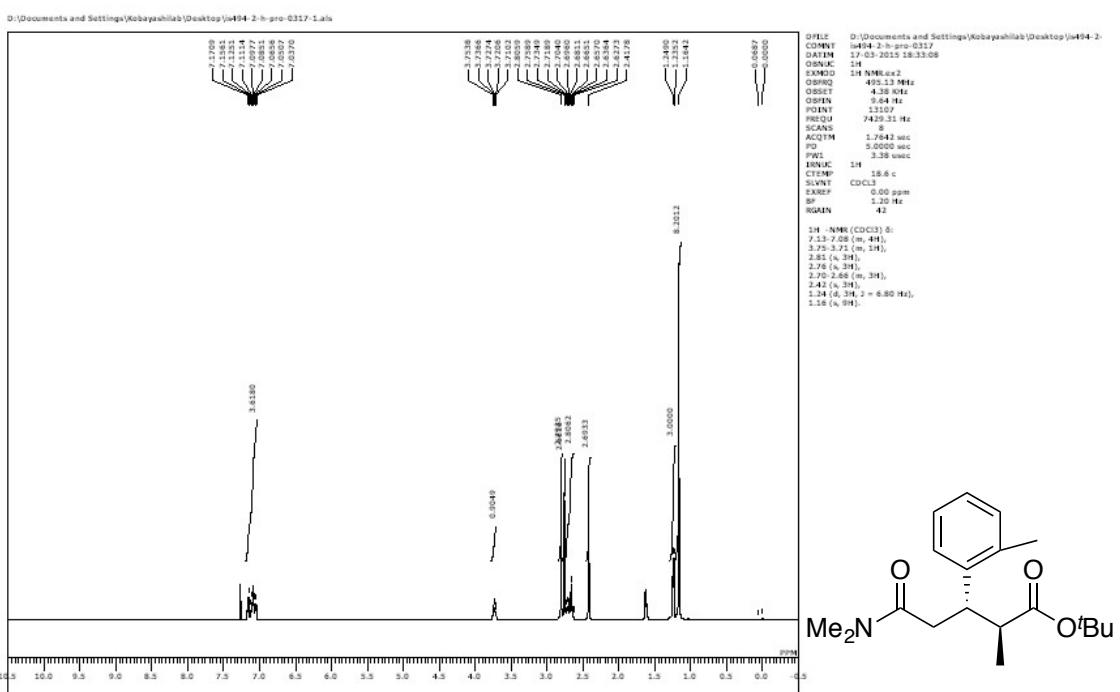
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1.22 (m, t, J = 6.87 Hz),

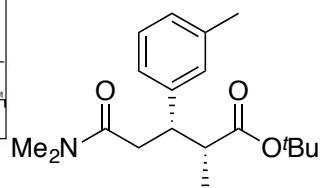
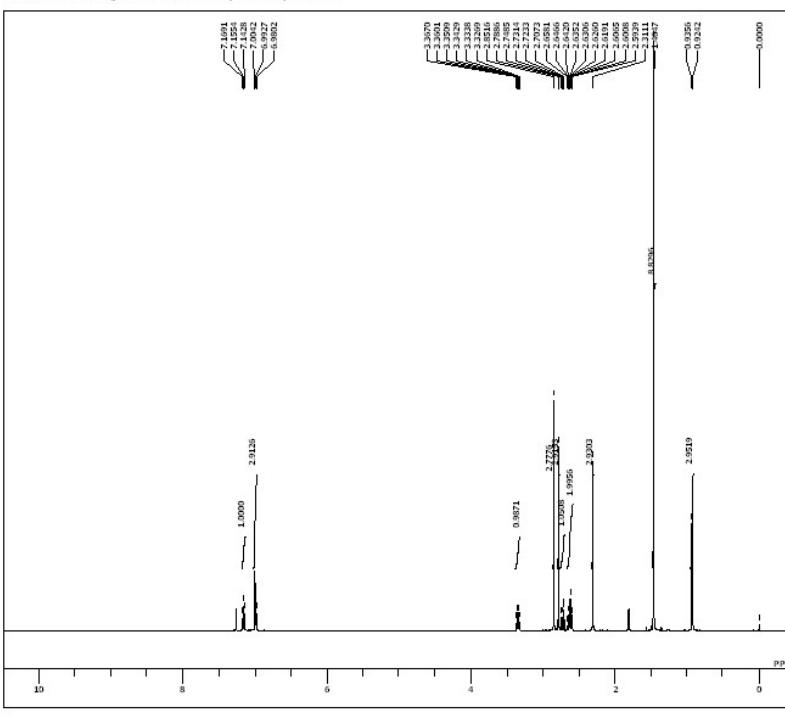
0.62 (m, d, J = 16.50, 6.87 Hz),

0.55 (m, t, J = 6.19 Hz).

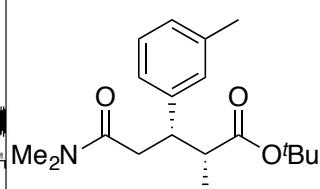
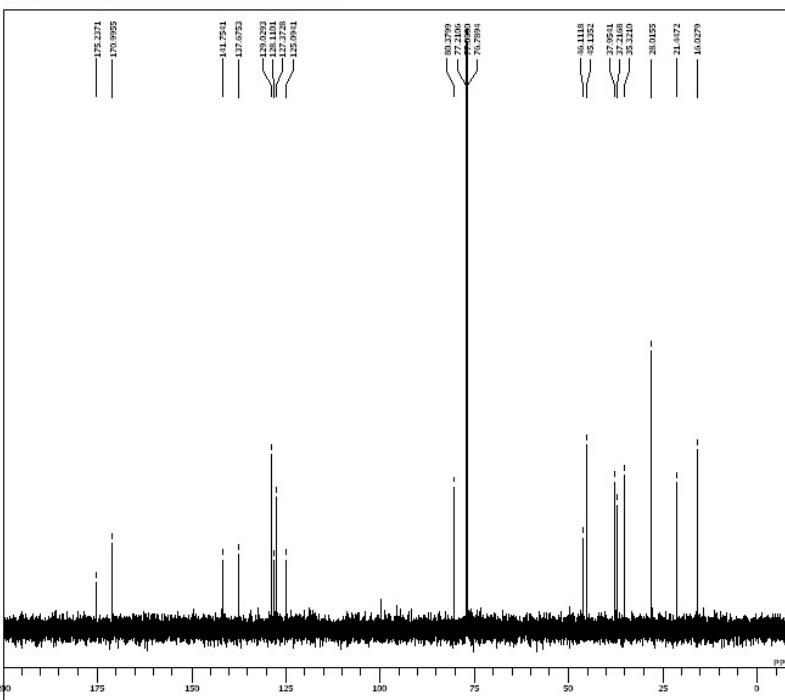




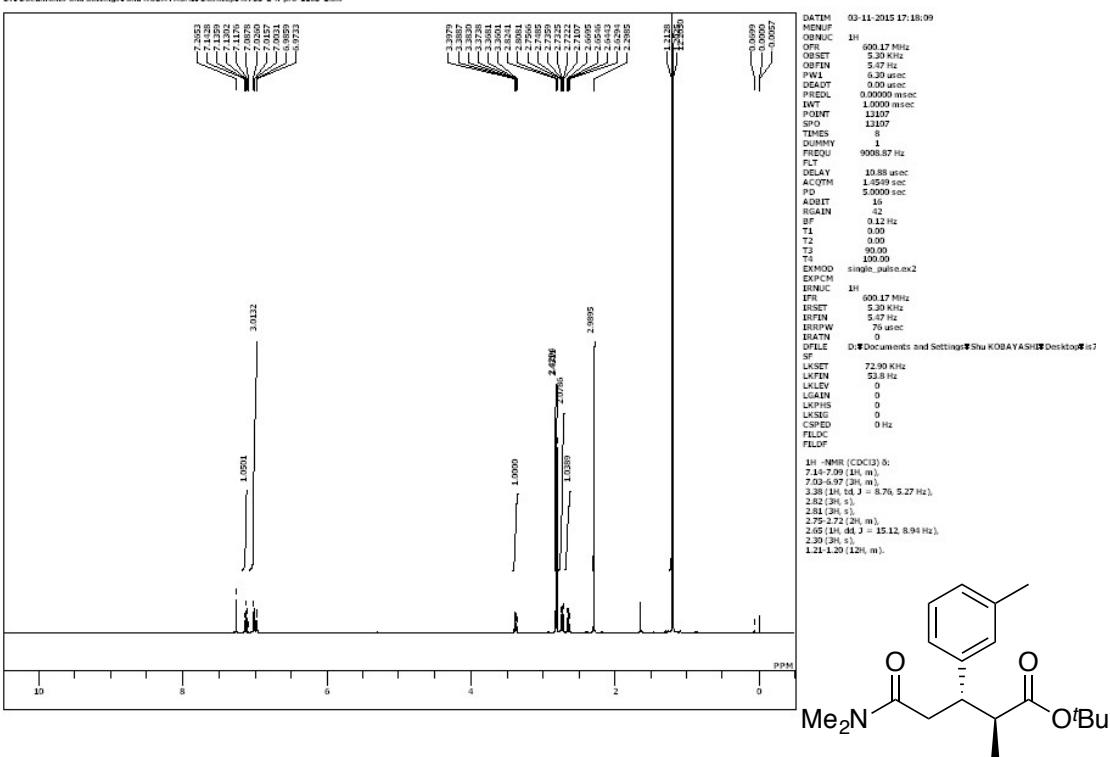
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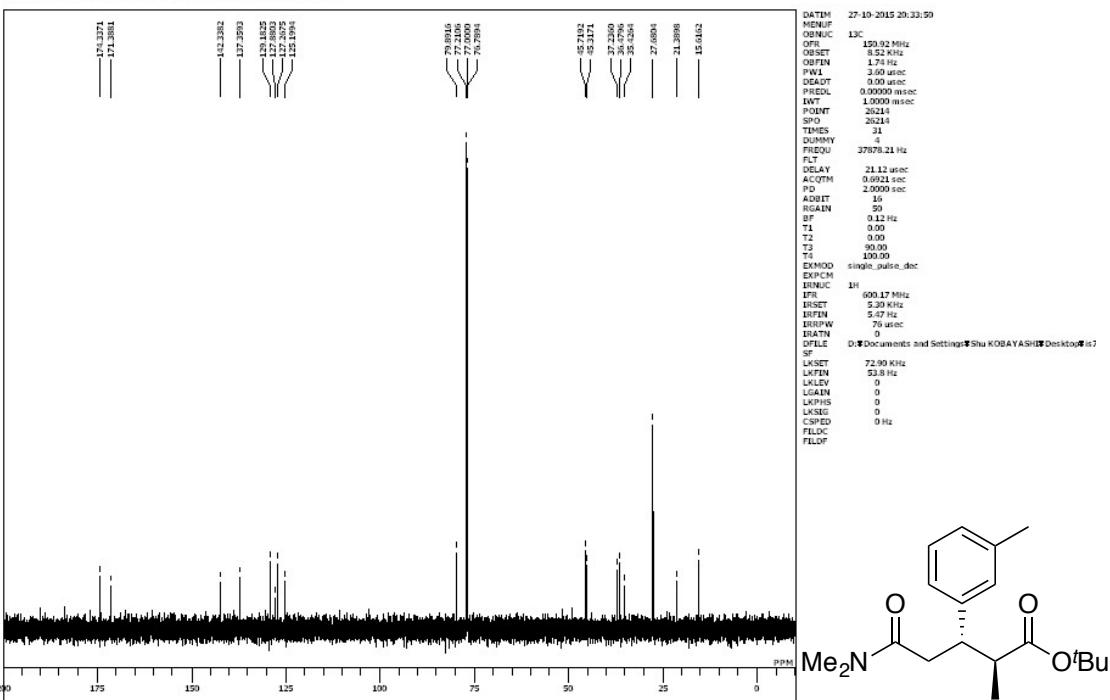
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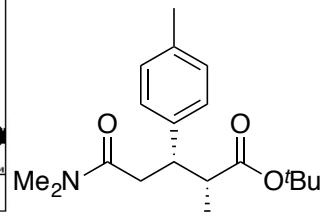
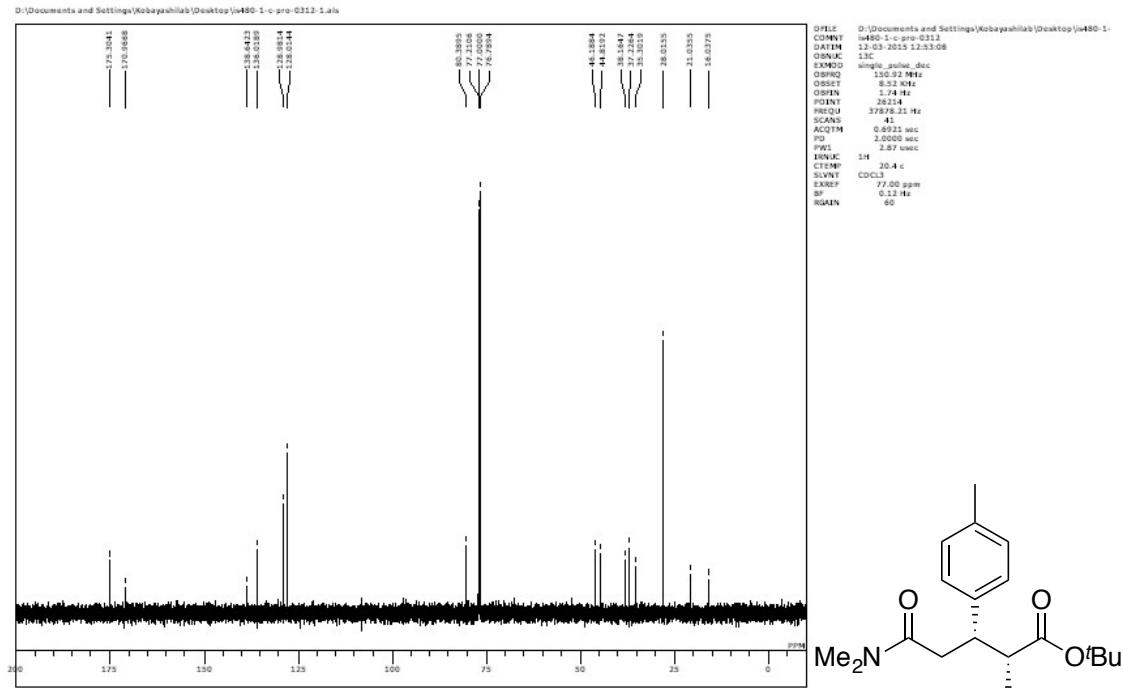
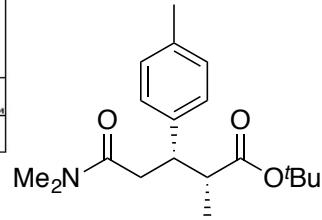
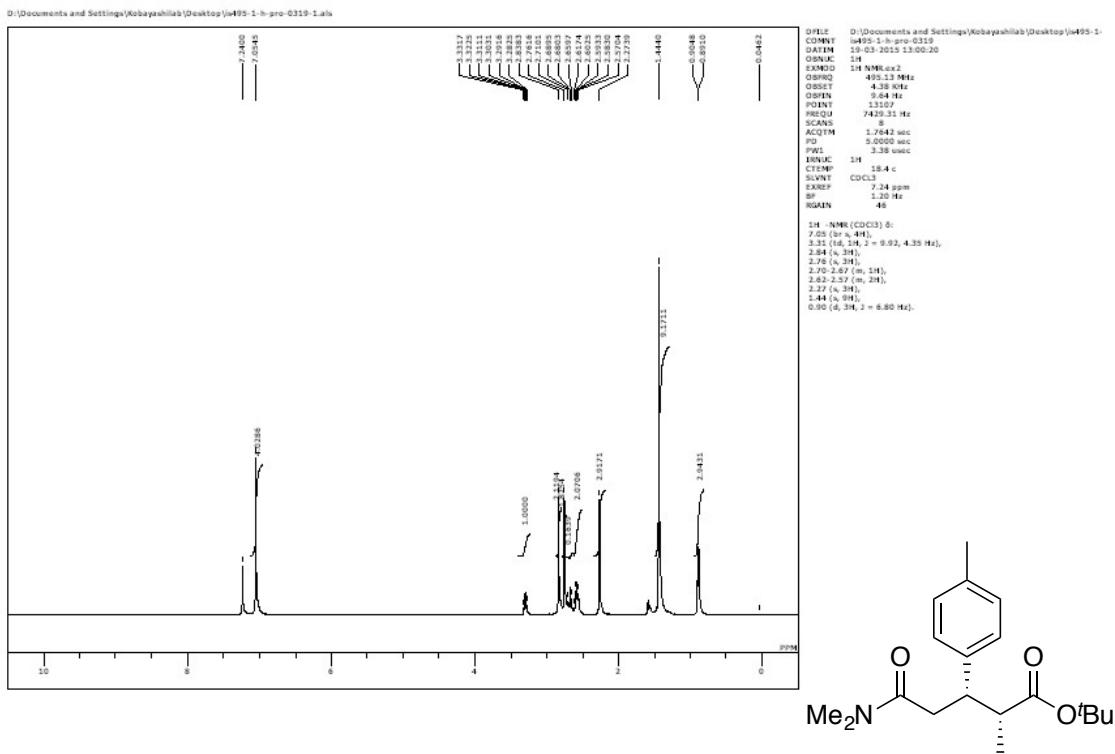


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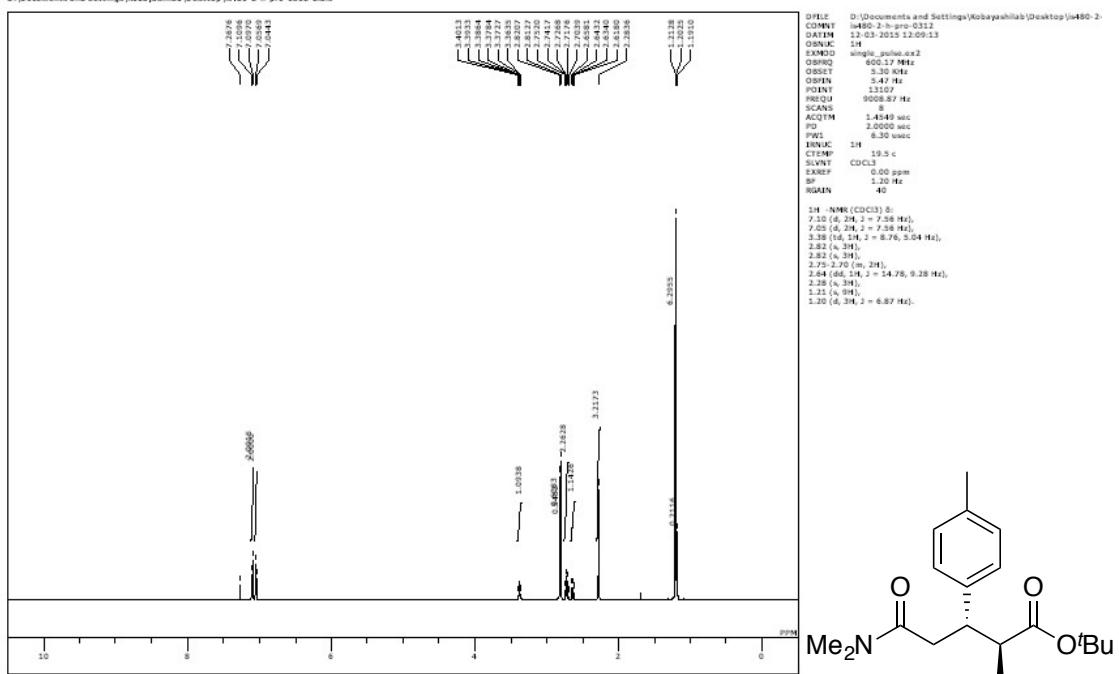


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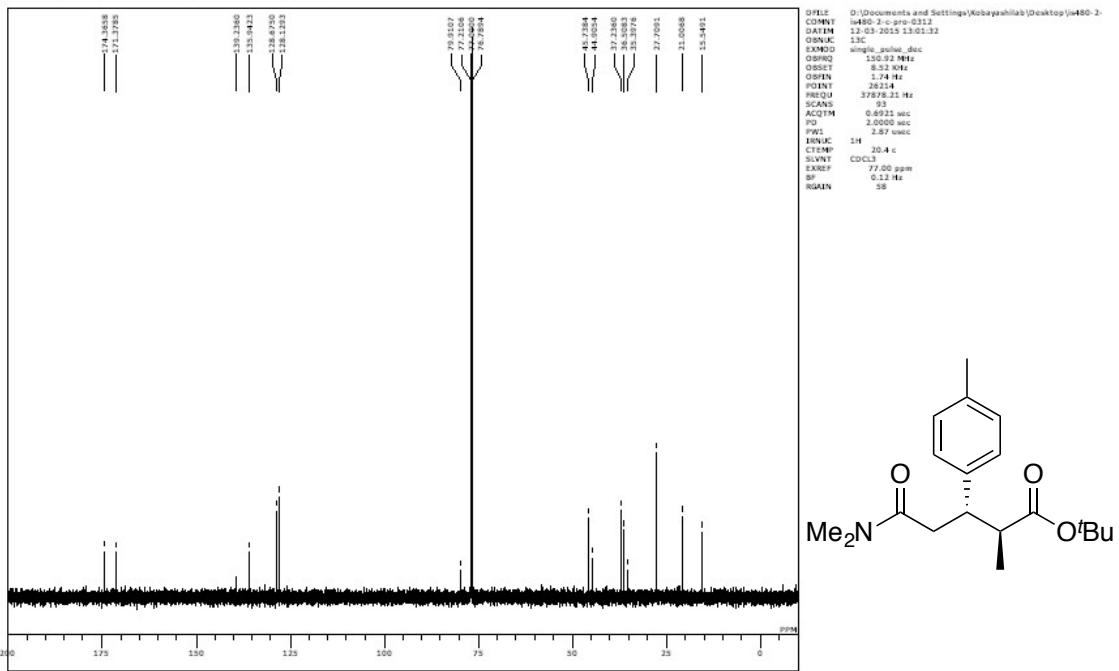




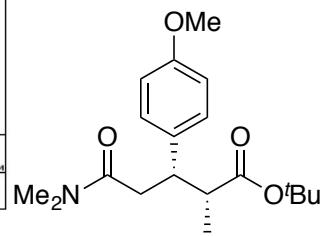
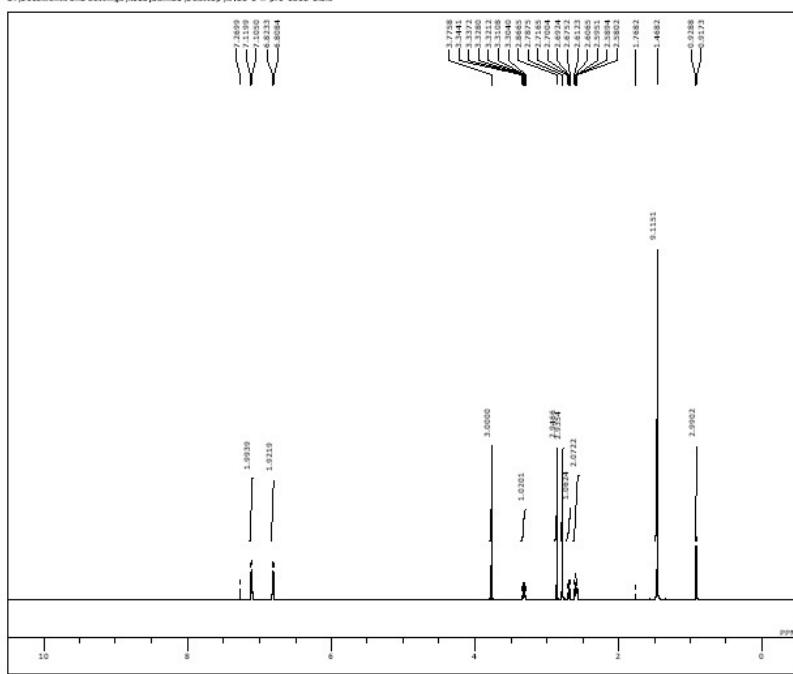
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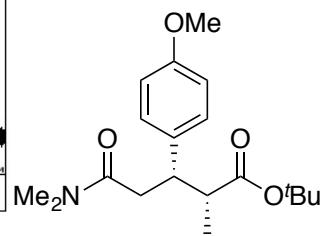
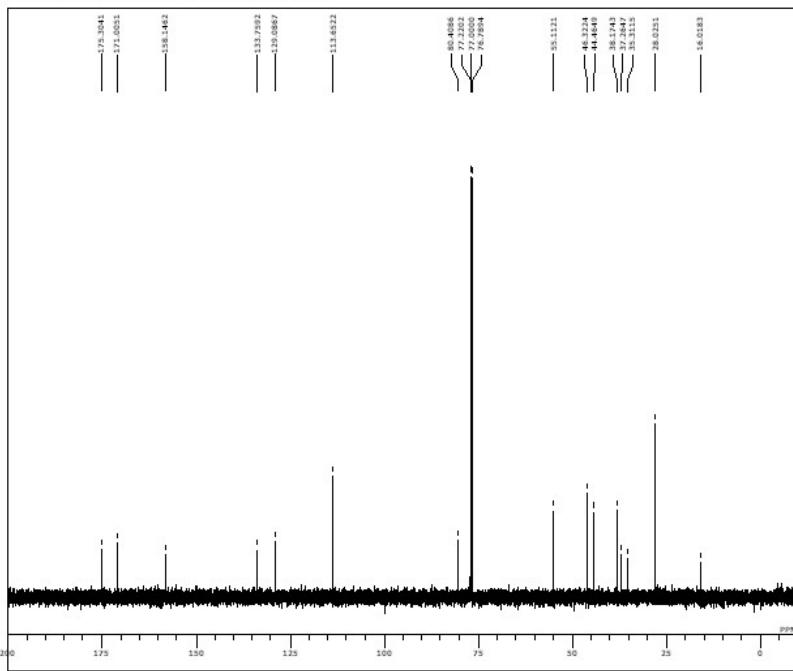
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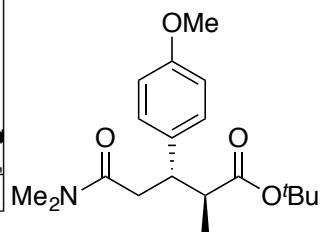
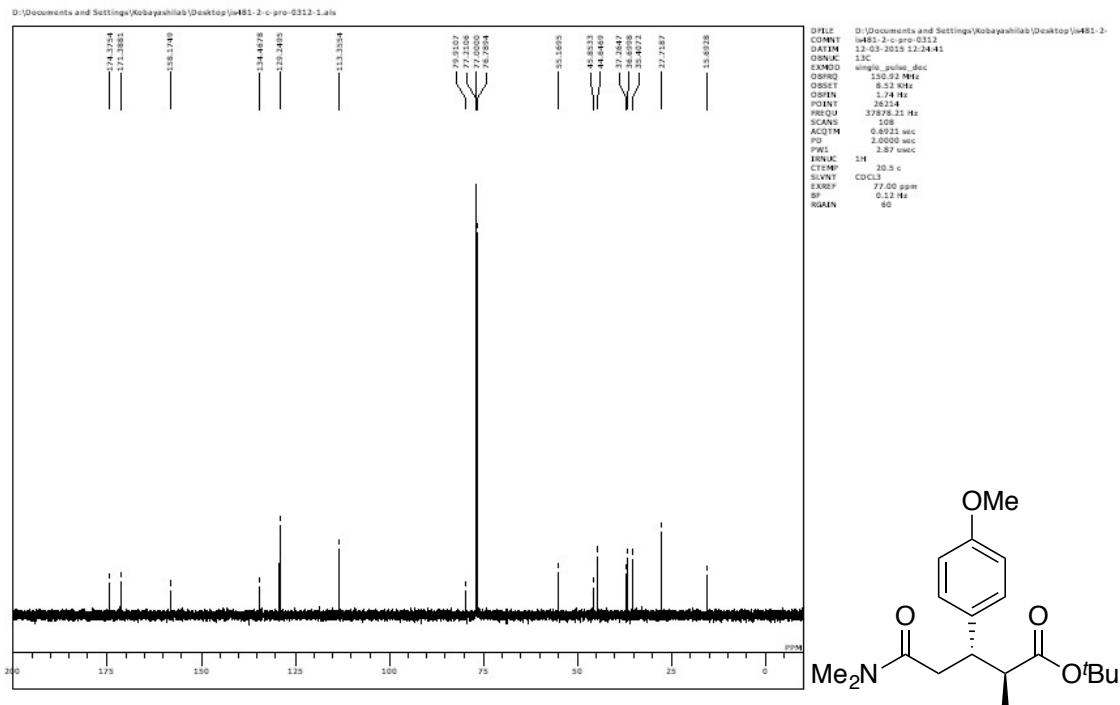
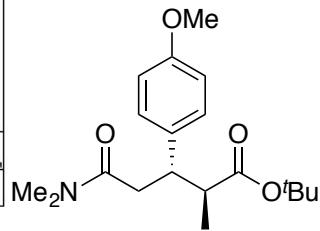
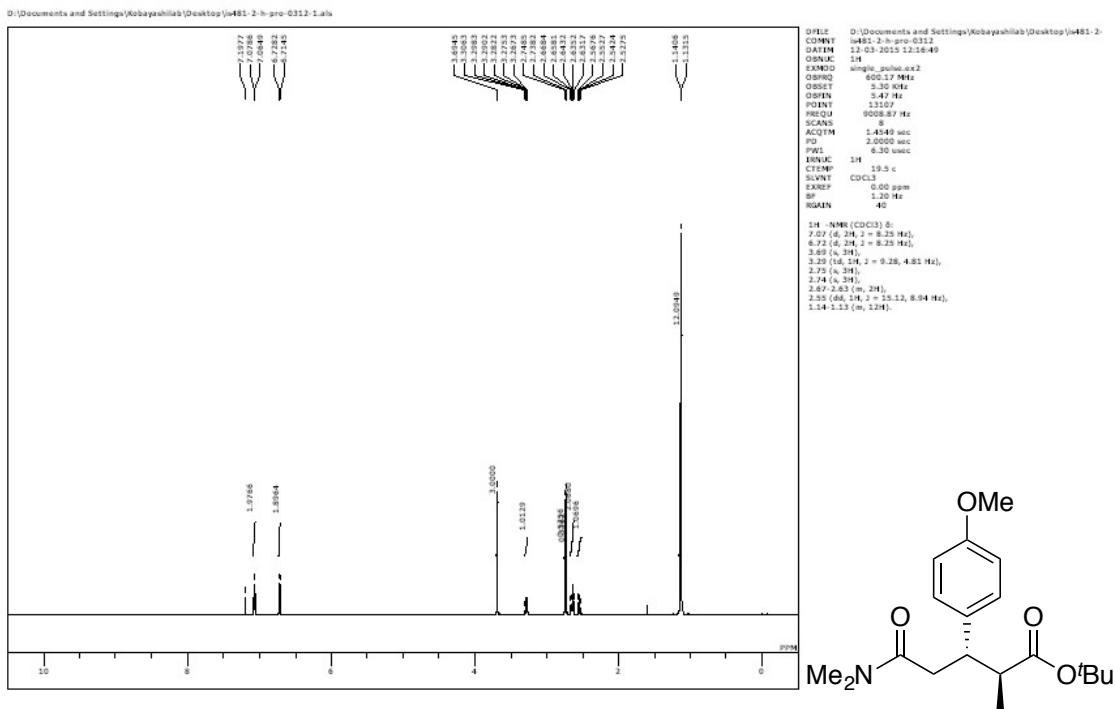


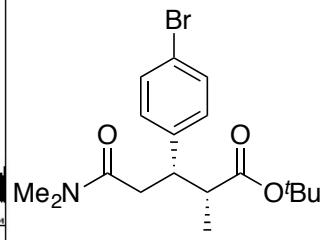
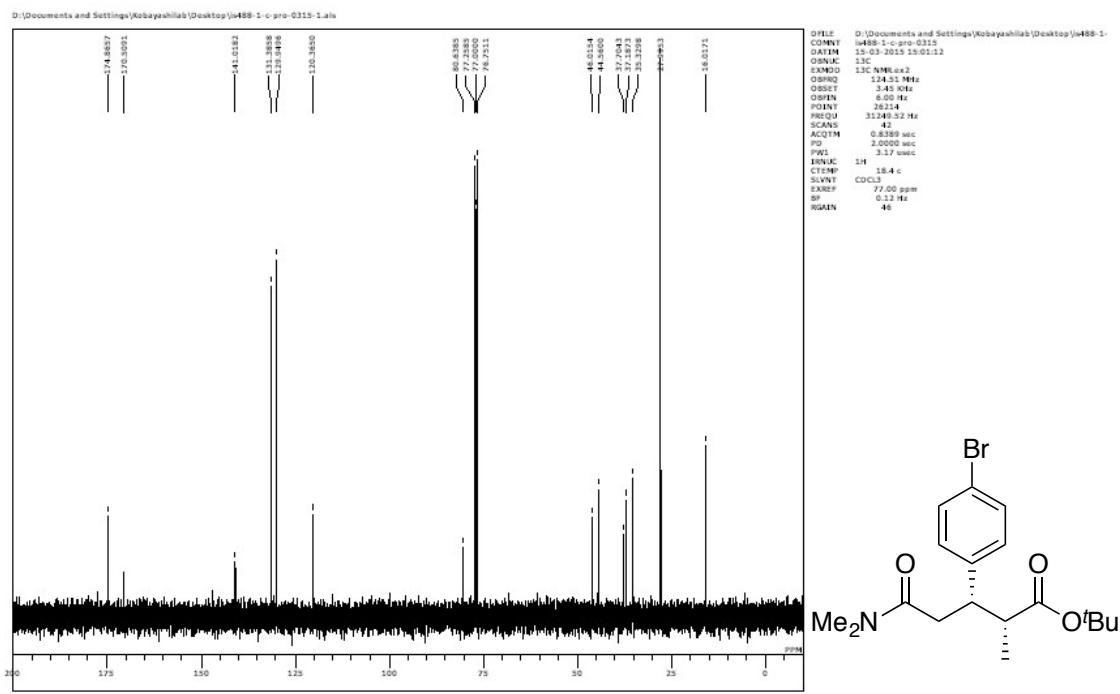
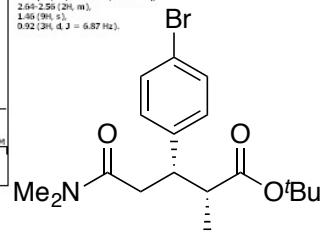
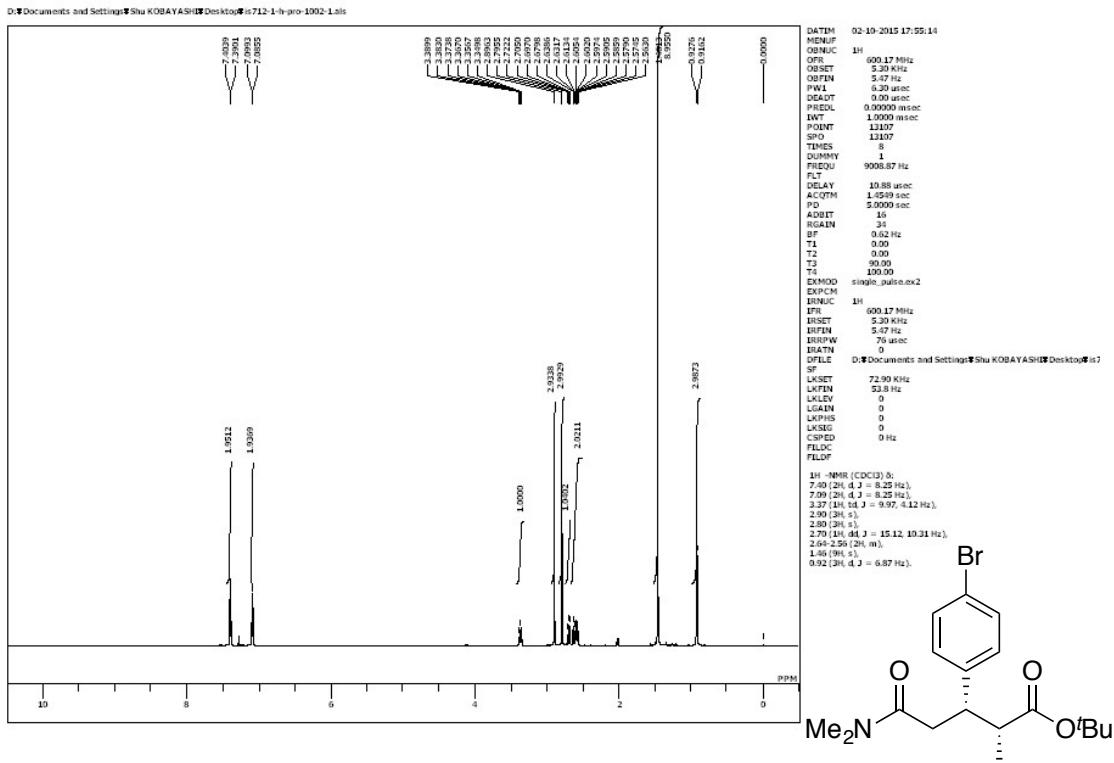
D:\Documents and Settings\Kobayashilab\Desktop\w481-1-h-pro-0312-1.aln

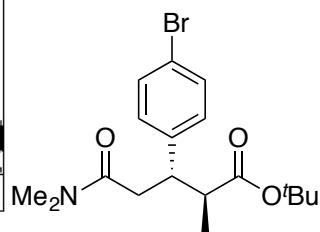
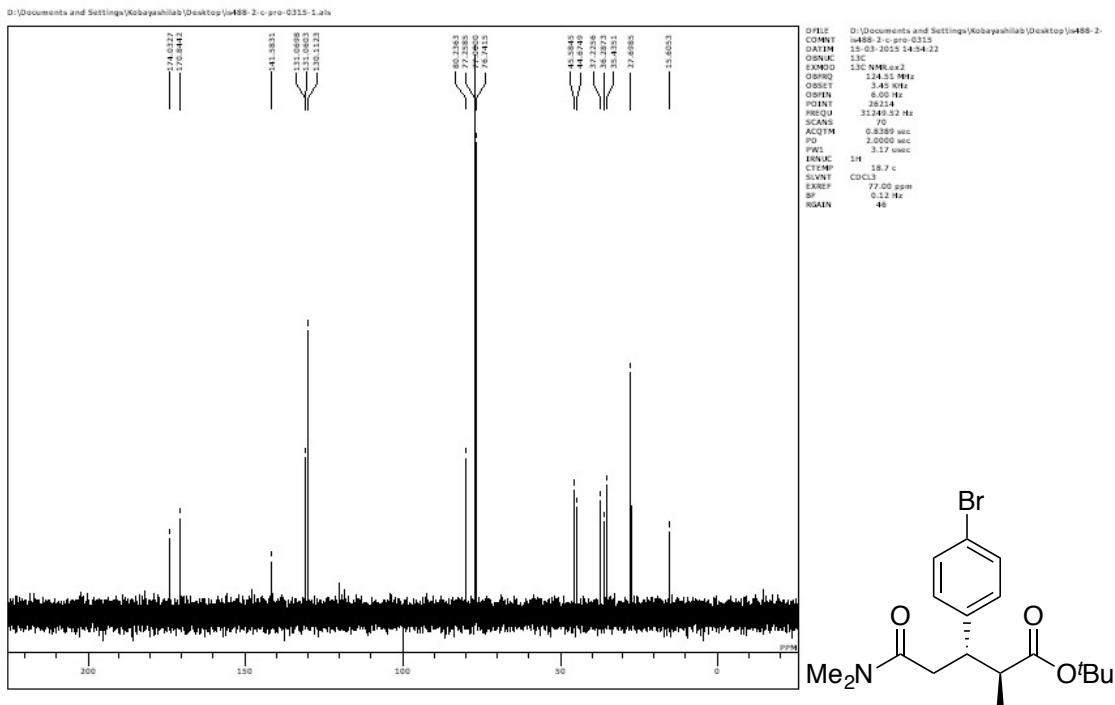
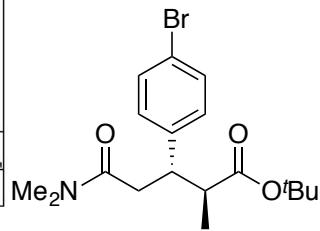
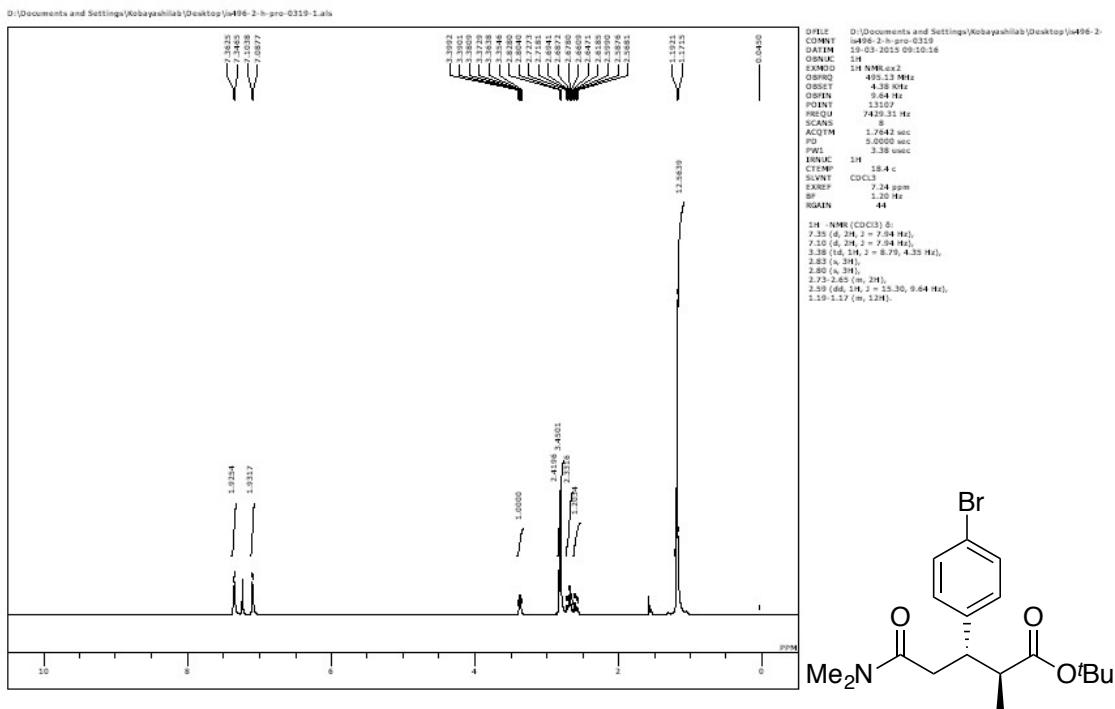


D:\Documents and Settings\Kobayashilab\Desktop\w481-1-c-pro-0312-1.aln

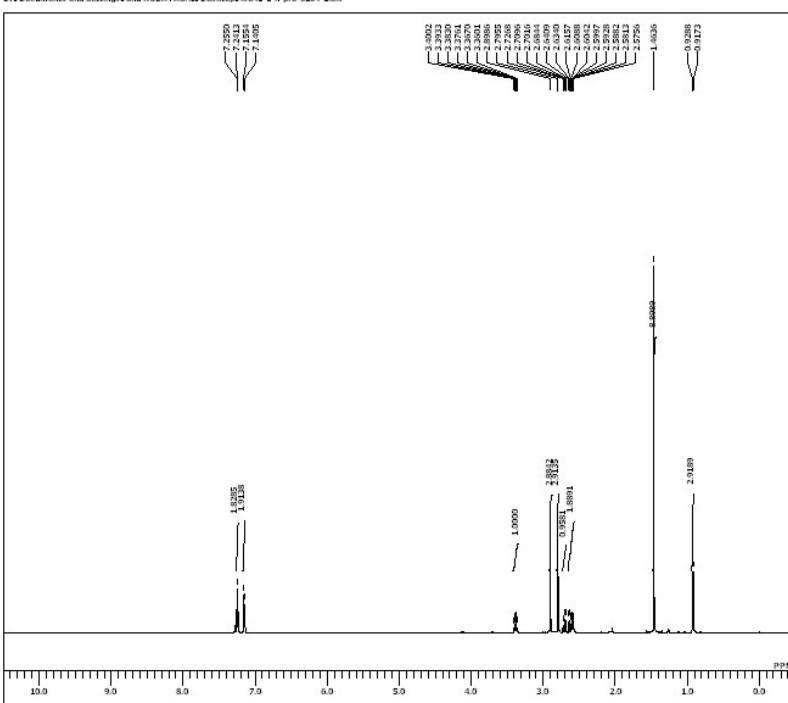








D:\Documents and Settings\Shu KOBAYASHI\Desktop\is841-1-h-pro-0204-1.xls



DATIM 04-02-2015 16:59:43

NUC 1H
OFR 600.17 MHz
OBSET 5.30 kHz
OBPN 5.47 Hz
PW1 0.50 usec
DEADT 0.00 usec
PREDL 0.00000 msec
INT 1.000 msec
POINT 13107

SPO 13107
TIMES 8
DUMMY 1
FREQU 9008.87 Hz

FILT 10.88 usec
ACQTM 1.4549 sec
TD 5.0000 sec
AQBRT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
TE 10.00

EXMOD single_pulse.ex2
EXPCH

DRNUC 1H
IFR 600.17 MHz
OBSET 5.30 kHz
OBPN 5.47 Hz
OBPW 70 usec
OBPN 0

DTIME D:\Documents and Settings\Shu KOBAYASHI\Desktop\is841-1.xls

SF 72.90 kHz

LKSET 53.8 Hz
LKTFN 0
LKLEV 0
LGAIN 0
LKHPS 0
LKSIG 0
CSPEQ 0 Hz
FILDC FILDF

1H NMR (CDCl₃) δ:

7.29 (m, 4H, J = 8.44 Hz),

7.15 (m, 4H, J = 8.44 Hz),

3.38 (m, 5H, J = 9.97, 4.12 Hz),

2.90 (m, 5H),

2.85 (m, 1H),

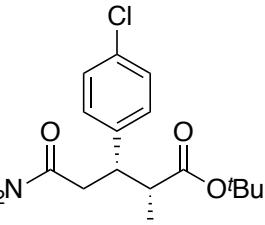
2.71 (m, 4H, J = 15.12, 10.31 Hz),

2.64-2.58 (2H, m),

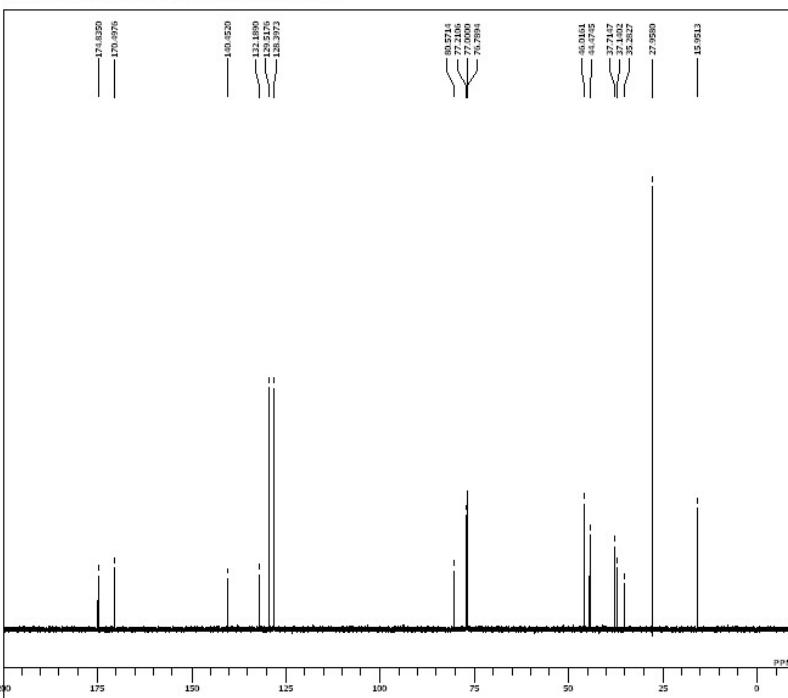
1.46 (m, 1H),

0.92 (m, 4H, J = 6.87 Hz).

0.92 (m, 4H, J = 6.87 Hz).



D:\Documents and Settings\Shu KOBAYASHI\Desktop\is841-1-c-pro-0204-1.xls



DATIM 04-02-2015 17:03:18

NUC 13C
OFR 125.92 MHz
OBSET 4.42 kHz
OBPN 1.74 Hz
PW1 3.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
INT 1.0000 msec
POINT 20214

SPO 20214
TIMES 67

DUMMY 1
FREQU 37878.21 Hz

FILT 21.12 usec
ACQTM 0.0000 sec
TD 2.0000 sec
AQBRT 16
RGAIN 30

BF 0.12 Hz
T1 0.00

T2 0.00

T3 90.00

TE 100.00

EXMOD single_pulse_dec

DRNUC 1H
IFR 600.17 MHz
OBSET 5.30 kHz
OBPN 5.47 Hz
OBPW 70 usec

OBPN 0

DTIME D:\Documents and Settings\Shu KOBAYASHI\Desktop\is841-1.xls

SF 72.90 kHz

LKSET 53.8 Hz

LKTFN 0

LGAIN 0

LKHPS 0

LKSIG 0

CSPEQ 0 Hz

FILDC FILDF

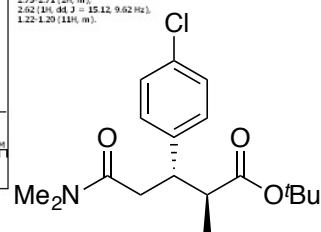
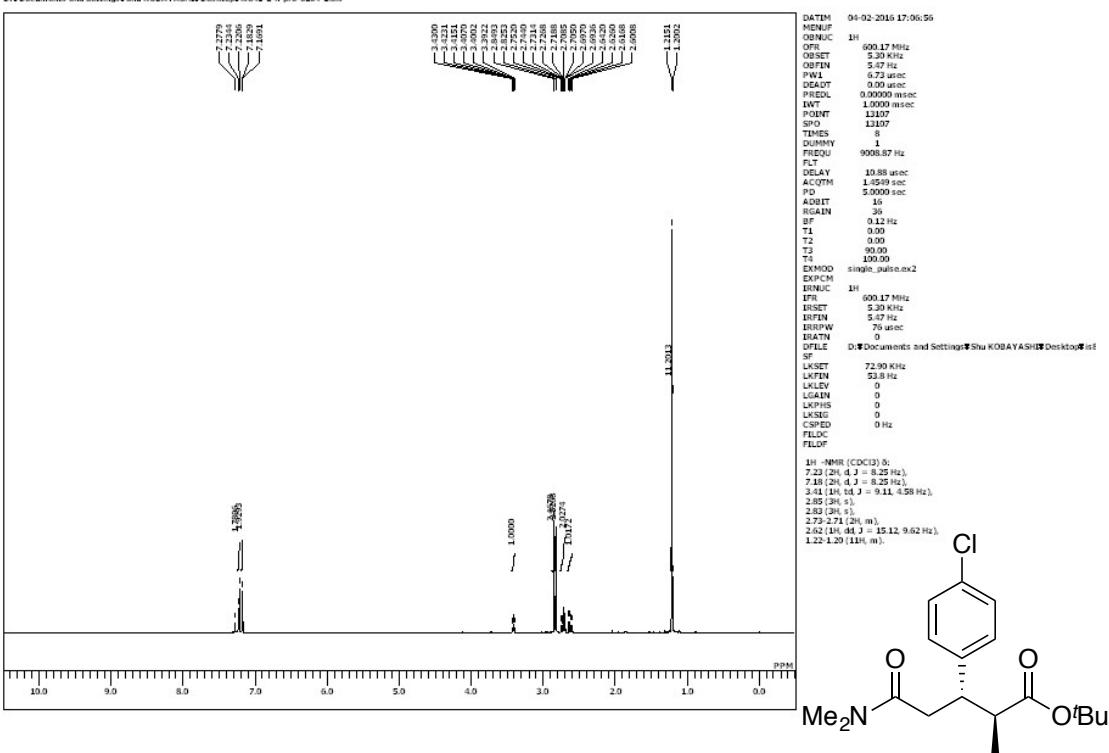
13C NMR (CDCl₃) δ:

175.8950, 170.4976, 140.4520, 132.4890, 128.4175, 128.3573, 92.7111, 92.1140, 77.7000, 75.7984, 65.0164, 44.4715, 37.1932, 35.3237, 27.7850, 15.9513.

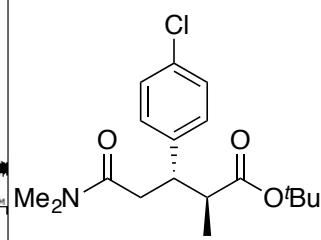
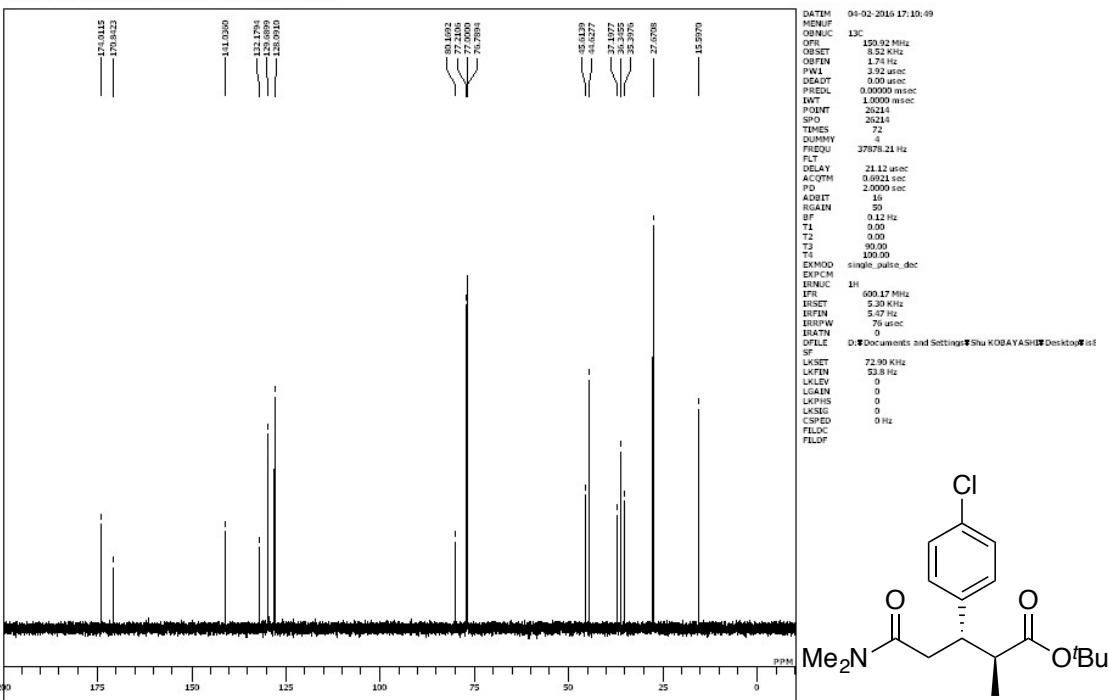
0.92 (m, 4H, J = 6.87 Hz).

0.92 (m, 4H, J = 6.87 Hz

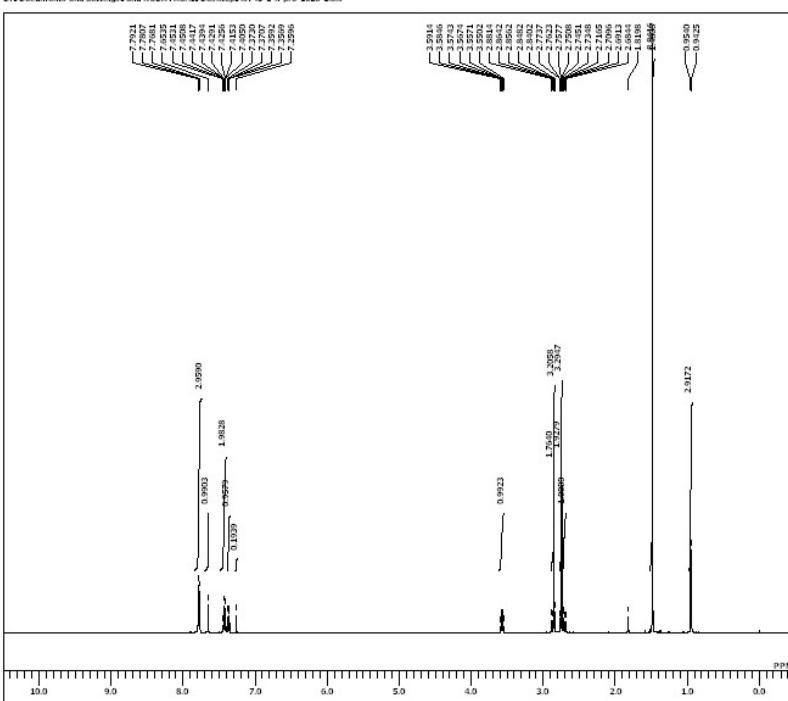
D:\Documents and Settings\Shu KOBAYASHI\Desktop\is841-2-h-pro-0204-1als

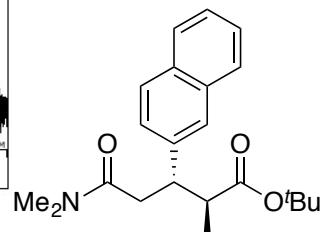
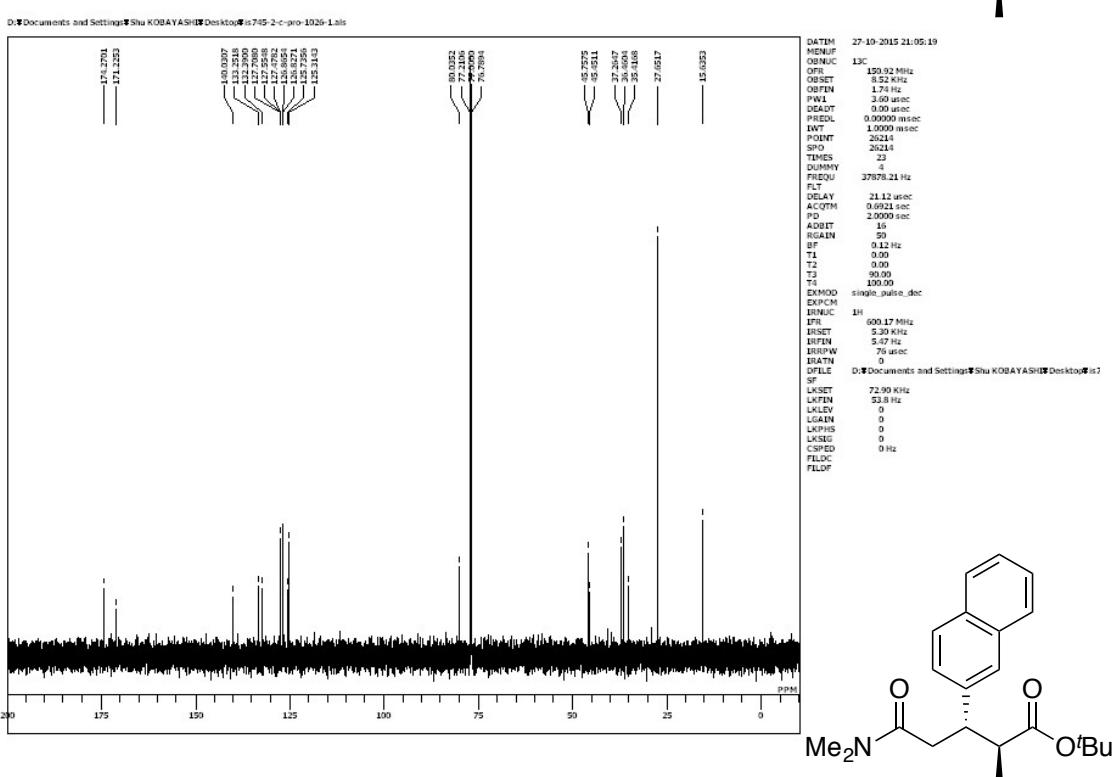
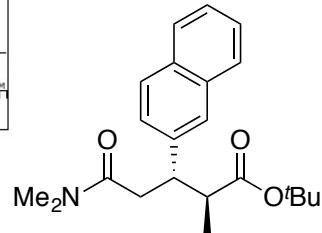
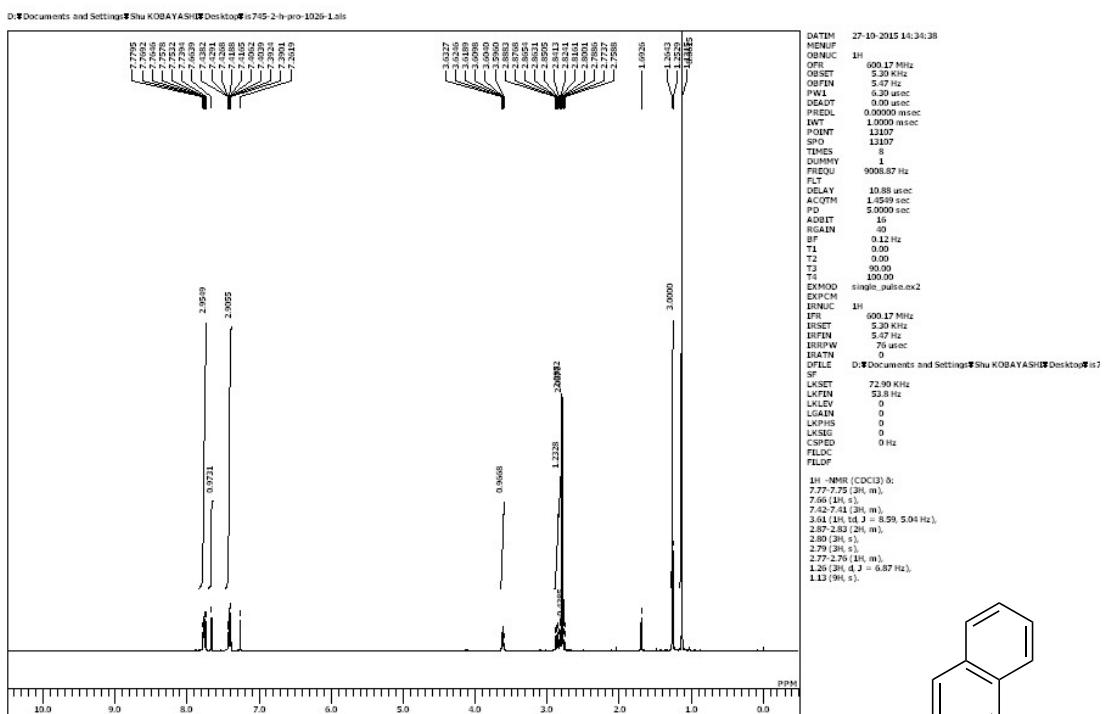


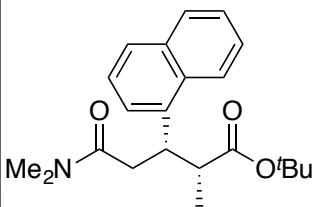
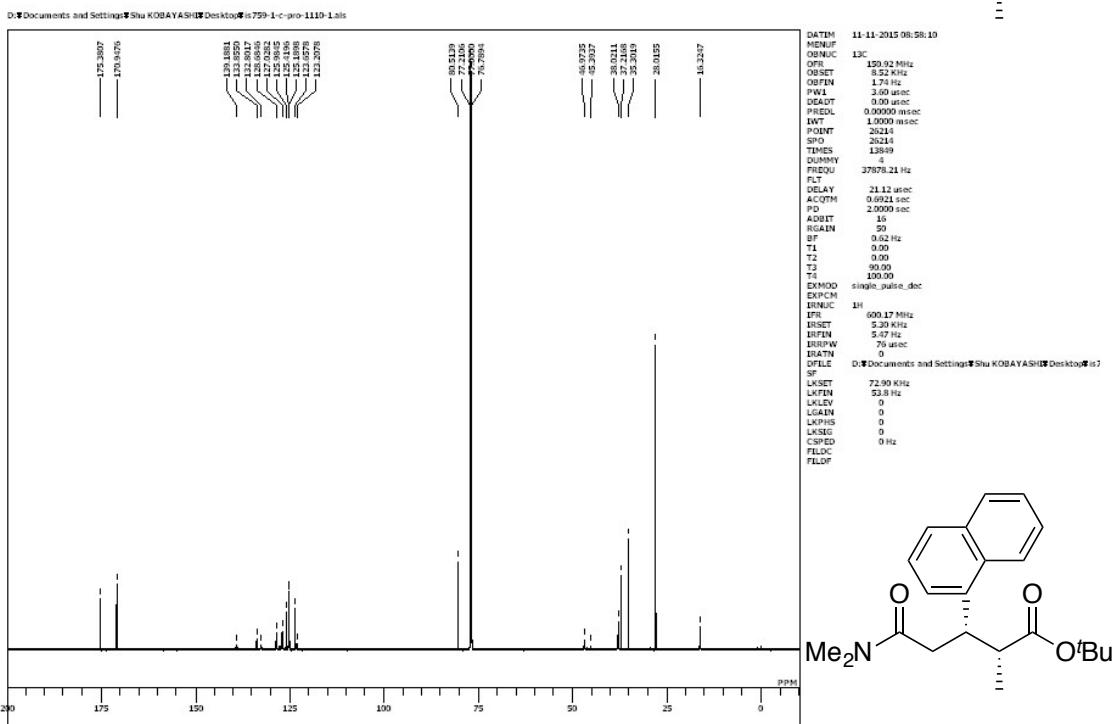
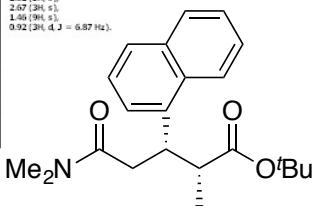
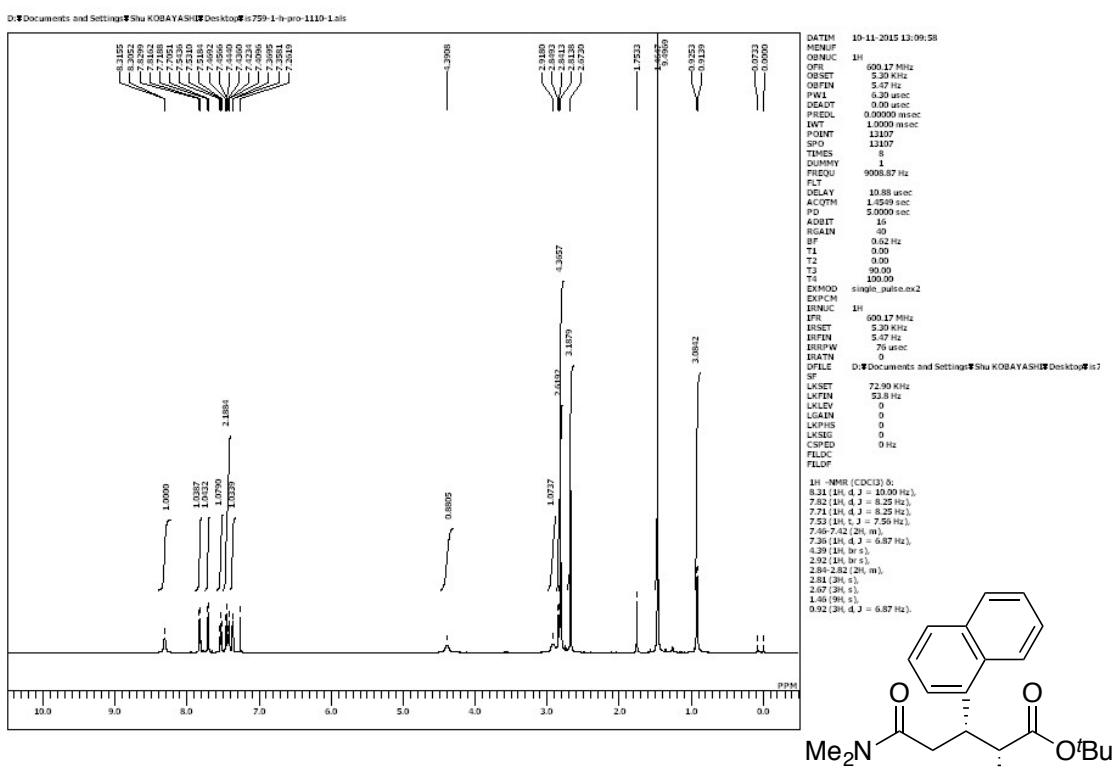
D:\Documents and Settings\Shu KOBAYASHI\Desktop\is841-2-c-pro-0204-1als

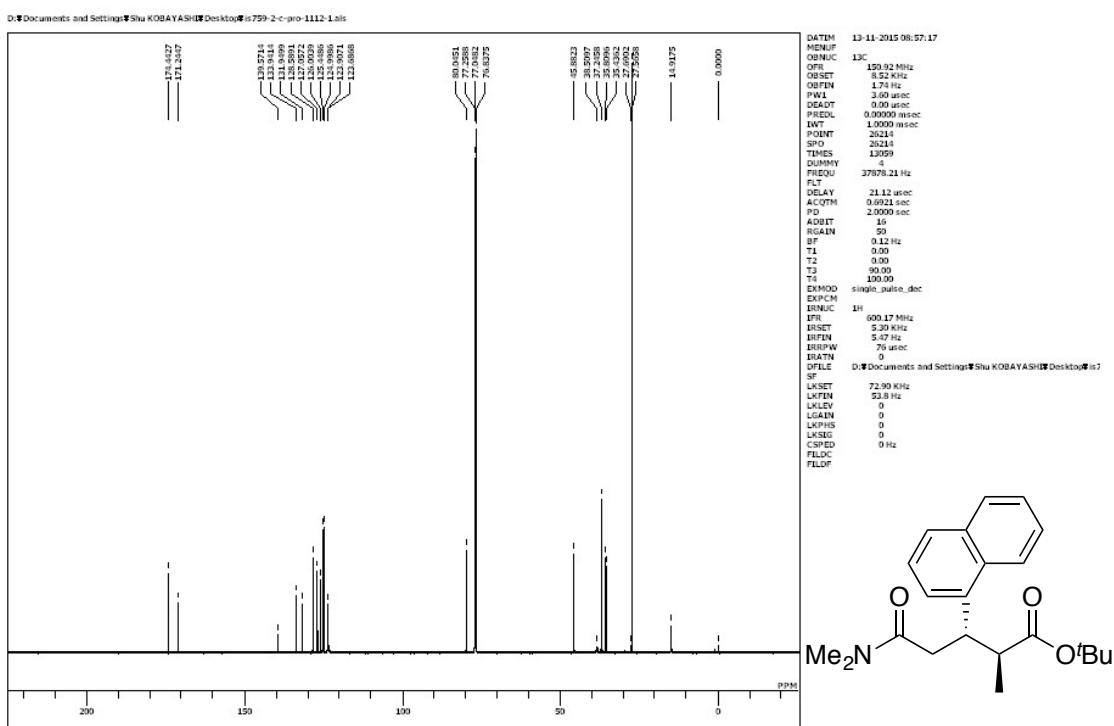
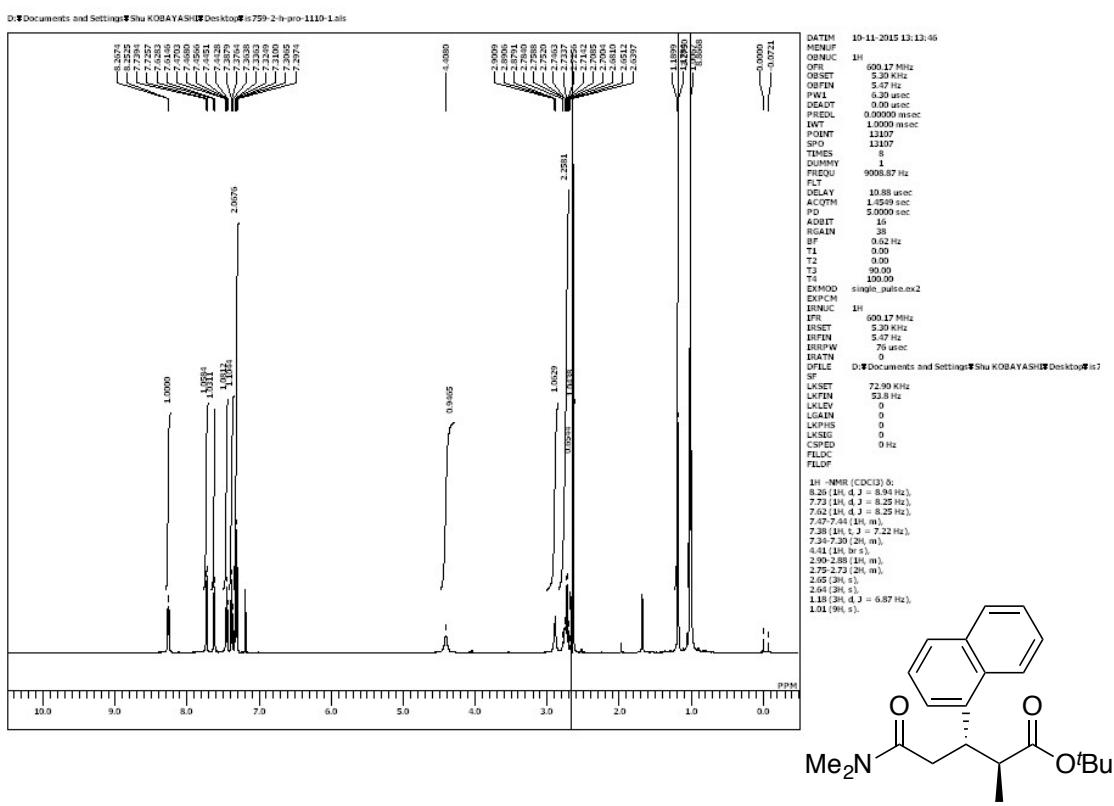


D:\Documents and Settings\Shu KOBAYASHI\Desktop\is745-1-h-pro-1029-1.xls

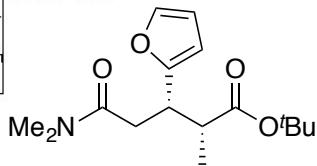
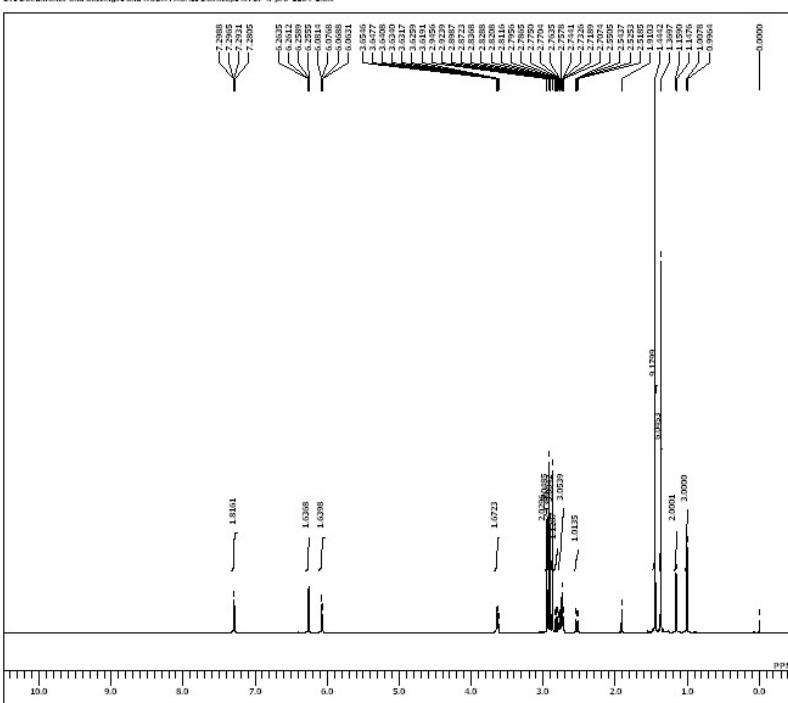




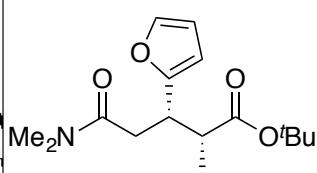
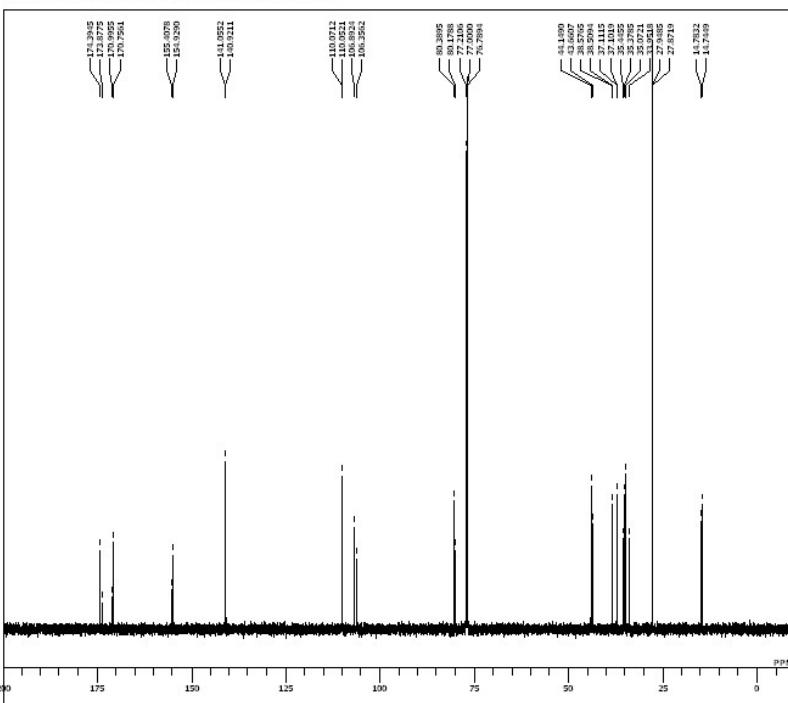


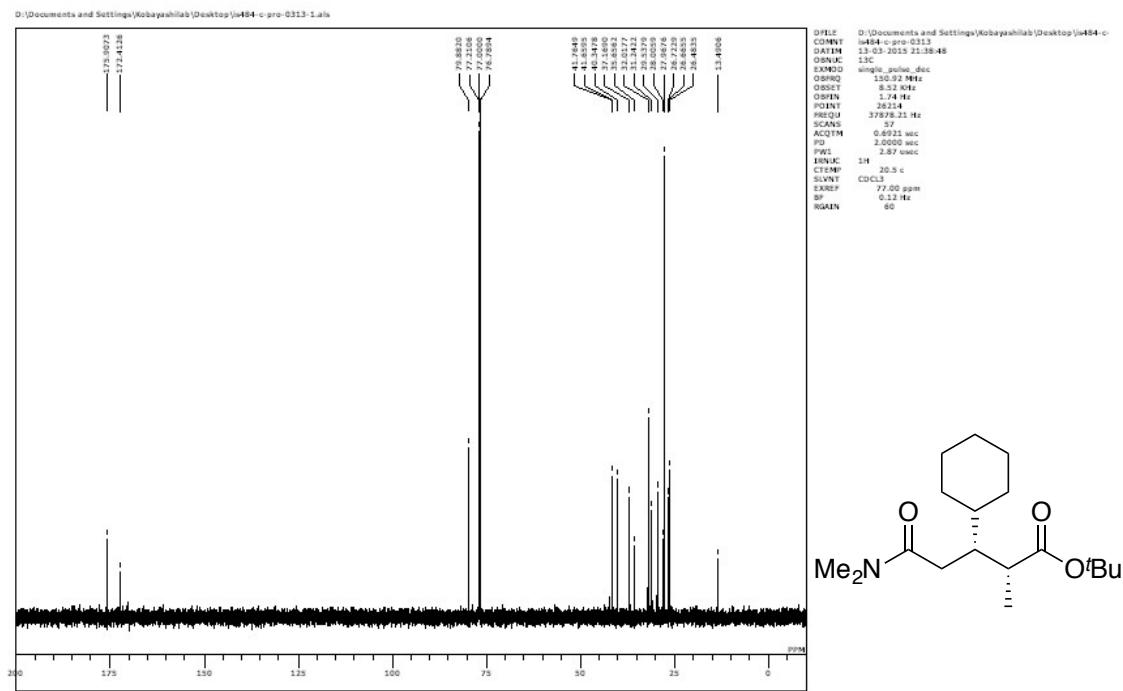
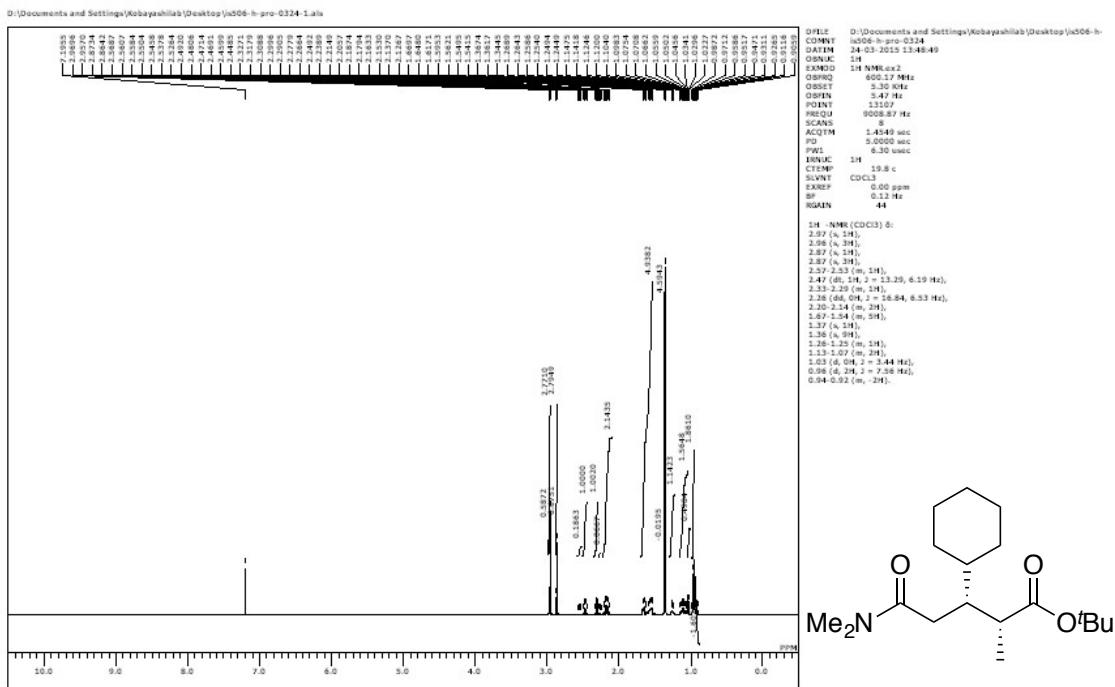


D:\Documents and Settings\Shu KOBAYASHI\Desktop\is757-h-pro-1104-1.xls

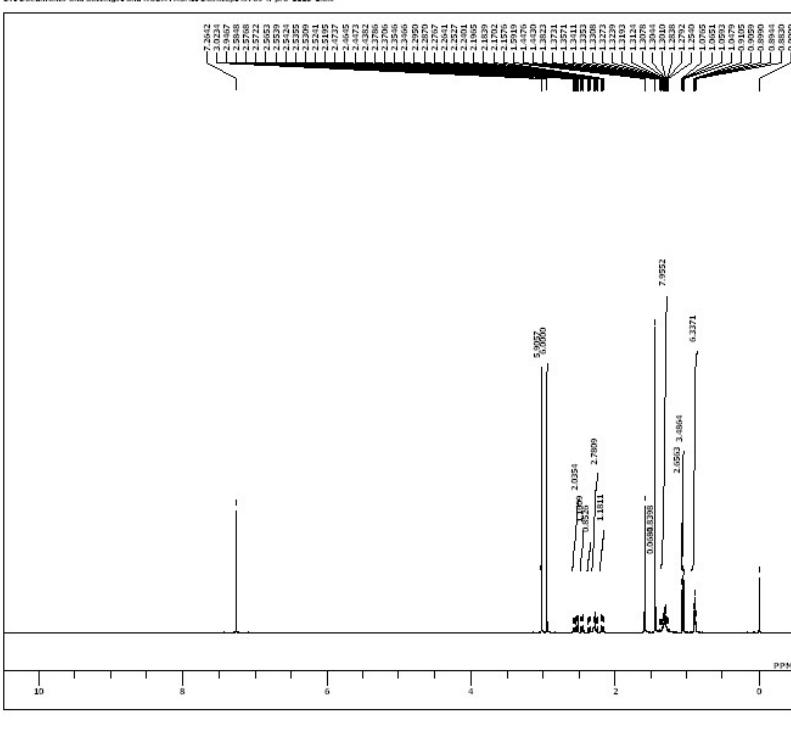


D:\Documents and Settings\Shu KOBAYASHI\Desktop\is757-c-pro-1104-1.xls

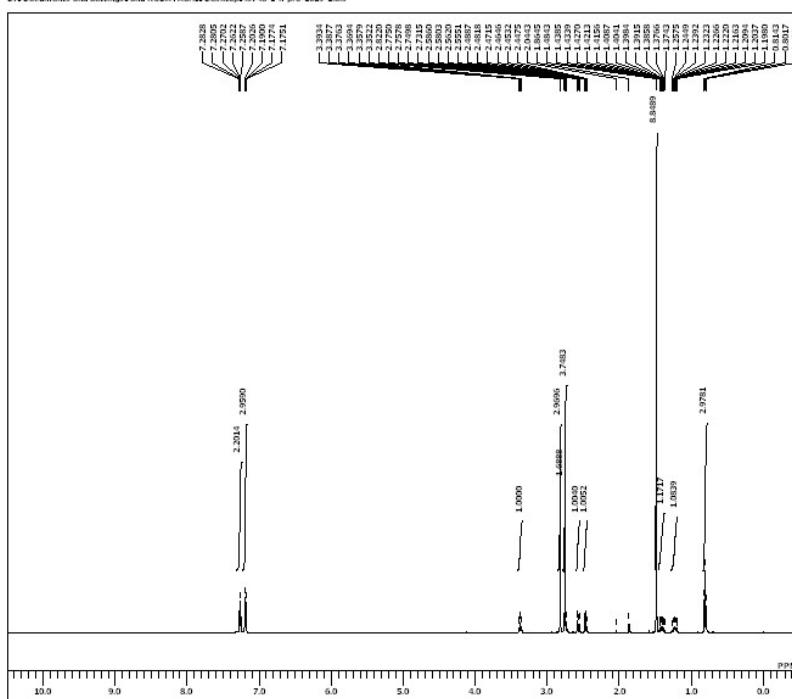




D:\Documents and Settings\Shu KOBAYASHI\Desktop\is769-h-pro-1113-1.xls



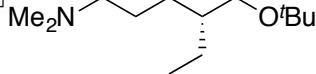
D:\Documents and Settings\Shu KOBAYASHI\Desktop\is748-1-h-pro-1029-1.xls



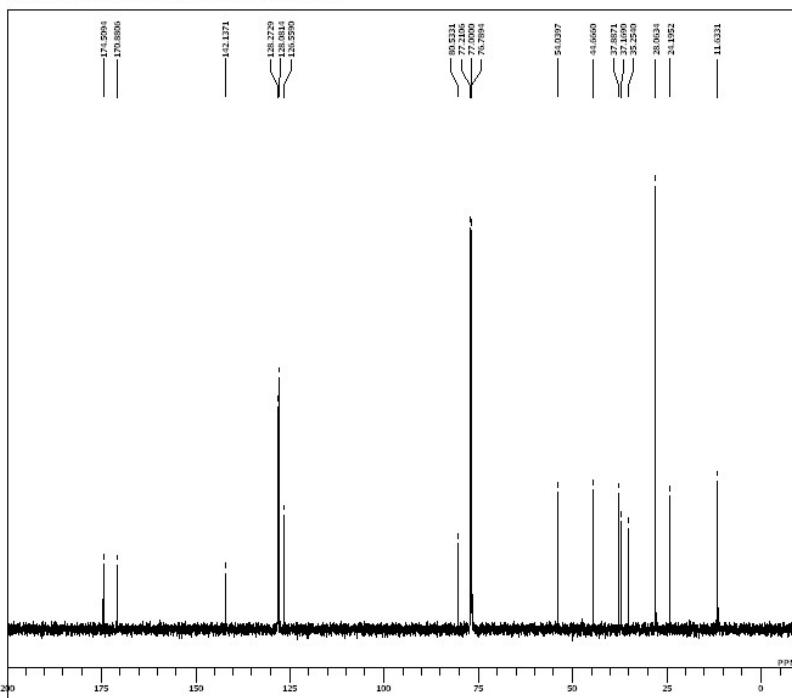
```

DATIM 29-10-2015 10:59:11
MENUF
IRH
OBNUC 1H
IPIR 600.17 MHz
OBSET 5.30 kHz
OBPN 5.47 Hz
PD 0.00 sec
DEADT 0.00 usec
PREDL 0.0000 msec
INT 1.0000 msec
POINT 10485
SPO 10485
TIMES 8
DUMMY 1
FREQU 7206.99 Hz
FILT
DELAY 10.88 usec
ACQTM 1.4549 sec
PD 5.0000 sec
ADBT 16
RGAIN 36
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
TE 10.00
EXMOD single_pulse.seq2
EXPCHM
IRNUC 1H
IPIR 600.17 MHz
OBSET 5.30 kHz
OBPN 5.47 Hz
OBPPW 75 usec
OBATN 0
DTFILE D:\Documents and Settings\Shu KOBAYASHI\Desktop\is7
SF
LKSET 72.90 KHz
LKTFN 53.8 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPEQ 0 Hz
FILDC
FILDF
IH -NH8 (CDCl3) 0;
7.2-7.3 (m, 2H, m);
7.19-7.18 (m, 1H, m);
3.37 (1H, t, J = 10.65, 3.67 Hz),
2.82 (3H, s);
2.77 (1H, t, J = 14.78, 3.78 Hz),
2.57 (1H, d, J = 14.78, 3.78 Hz),
2.50 (1H, d, J = 10.65, 3.67 Hz),
1.48 (9H, s);
1.42-1.39 (1H, m),
1.26-1.20 (1H, m),
0.80 (3H, t, J = 7.56 Hz).

```



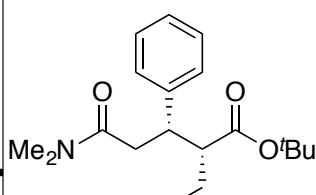
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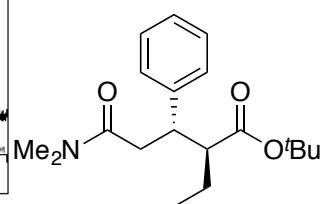
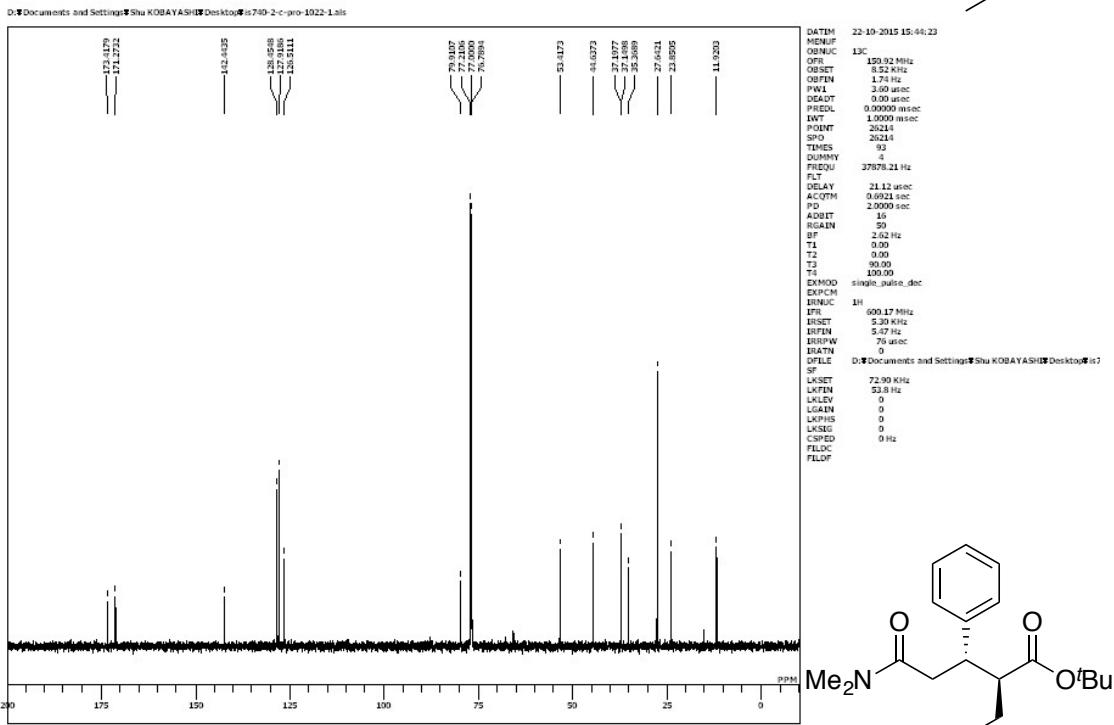
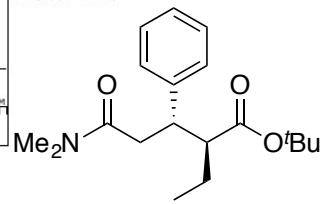
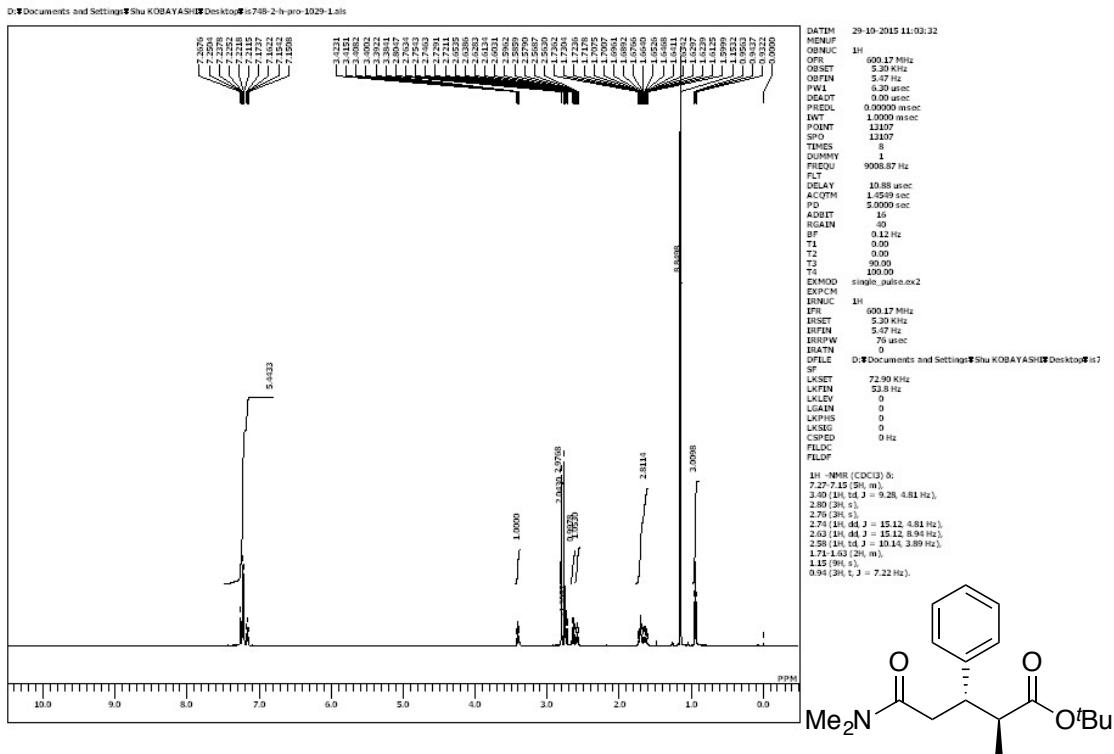


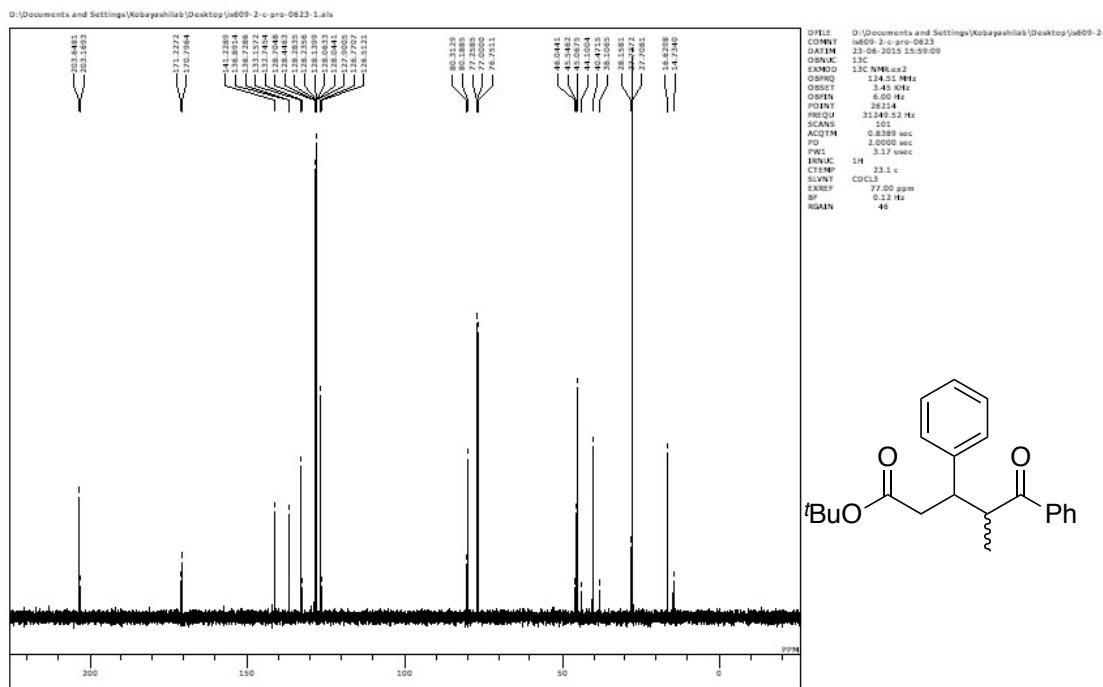
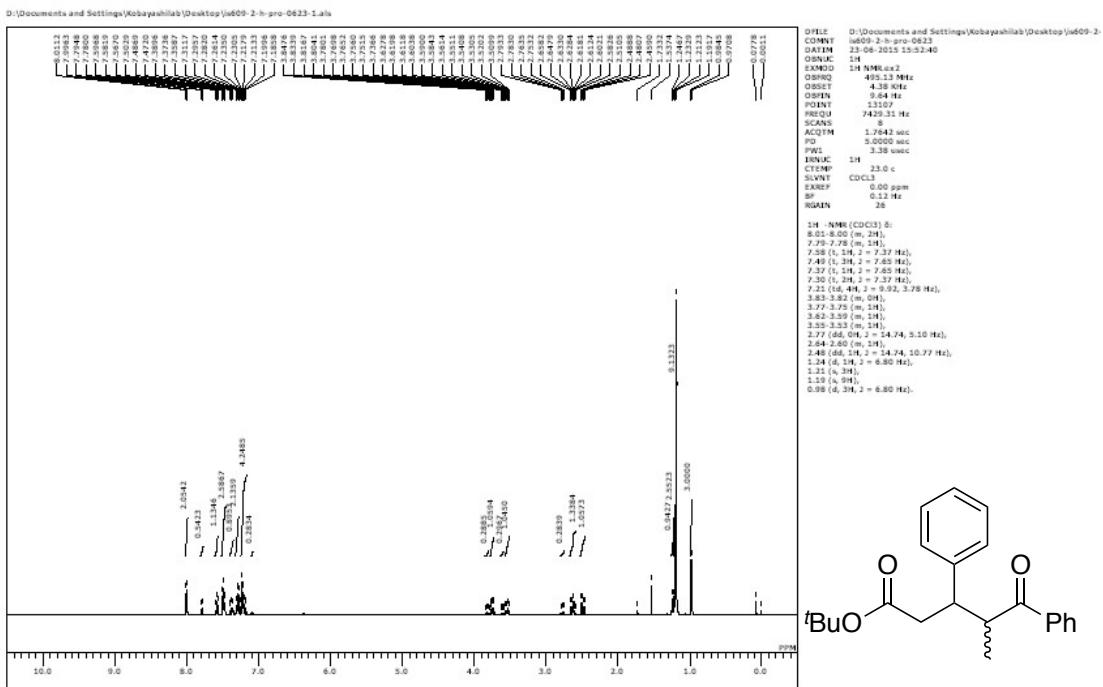
```

DATIM 22-10-2015 15:34:31
MENUF
IRNC 13C
IPIR 120.92 MHz
OBSET 4.44 kHz
OBPN 5.47 Hz
PW1 3.60 usec
DEADT 0.00 usec
PREDL 0.0000 msec
INT 1.0000 msec
POINT 20214
SPO 20214
TIMES 97
DUMMY 4
FREQU 37879.21 Hz
FILT
DELAY 21.12 usec
ACQTM 0.0000 sec
PD 2.0000 sec
ADBT 16
RGAIN 50
BF 2.62 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCHM
IRNUC 1H
IPIR 600.17 MHz
OBSET 5.30 kHz
OBPN 5.47 Hz
OBPPW 75 usec
OBATN 0
DTFILE D:\Documents and Settings\Shu KOBAYASHI\Desktop\is7
SF
LKSET 72.90 KHz
LKTFN 53.8 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPEQ 0 Hz
FILDC
FILDF

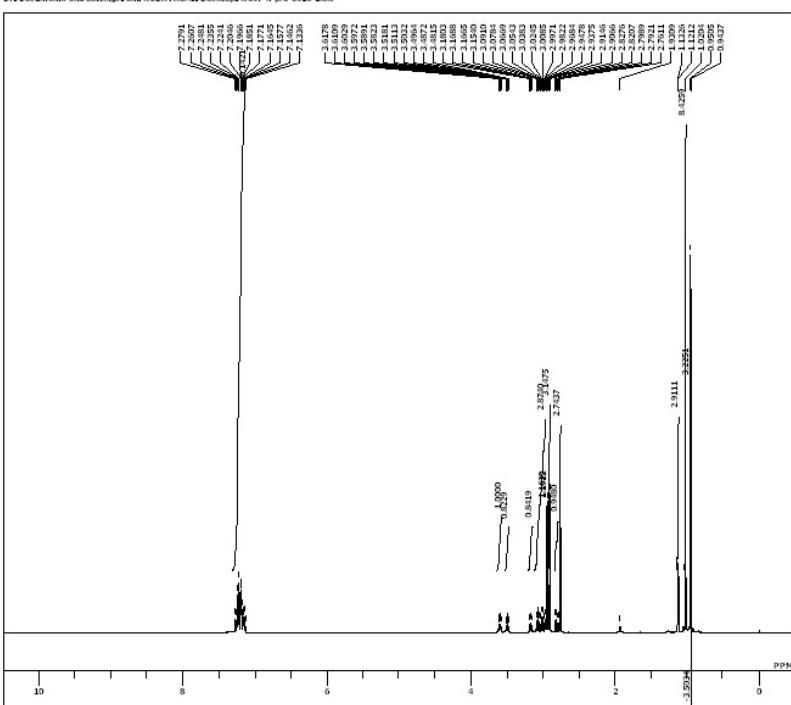
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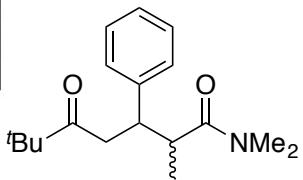
D:\Documents and Settings\Shu KOBAYASHI\Desktop\is097-h-pro-0916-1.xls



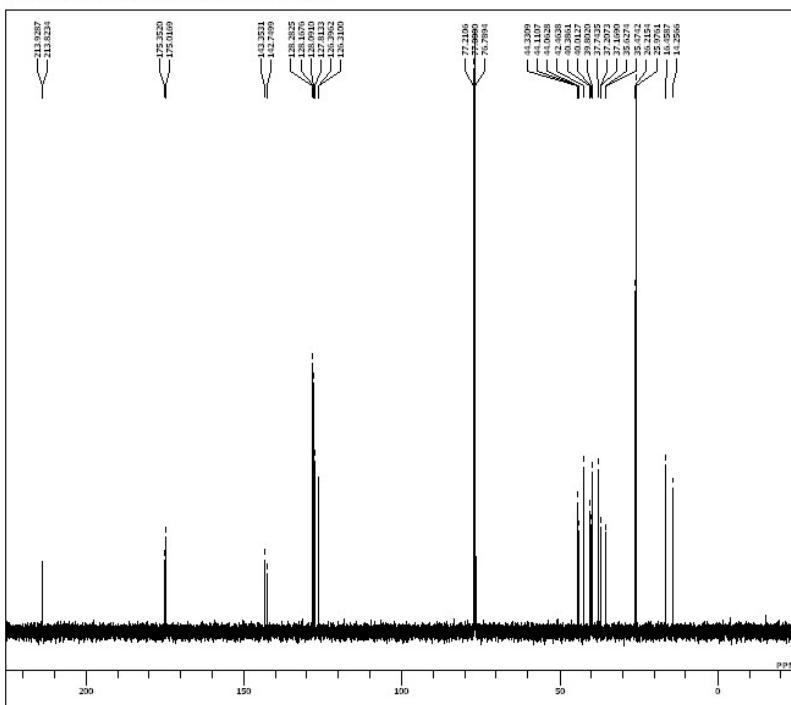
```

DATIM 16-09-2015 16:04:38
MENUFC 1H
DBNUC 1H
DPR 600.17 MHz
DBSET 5.30 kHz
DBTIN 5.47 Hz
PWR 0.5000
DEADT 0.00 usec
PREDL 0.00000 usec
INT 1.0000 usec
POINT 13107
SPO 13107
TD 51200 sec
AQB1T 5
AQB1T 5
RGAIN 34
BF 0.62 Hz
T1 0.00
T2 0.00
T3 90.00
TE 100.00
EXMOD single_pulse.ex2
EXPCLM
DBNUC 1H
DPR 600.17 MHz
DBSET 5.30 kHz
DBTIN 5.47 Hz
DBPW 98.0 usec
DBATN 0
DTFILE D:\Documents and Settings\Shu KOBAYASHI\Desktop\is097-h-pro-0916-1.xls
SF
LKSET 72.90 KHz
LKTFN 53.8 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPEQ 0 Hz
FILDC
FILDF
1H NMR (CDCl3) δ:
7.29 (1H, m), 7.27 (1H, m),
3.90 (1H, d, J = 8.9, 3.89 Hz),
3.59 (1H, t, J = 3.11, 3.89 Hz),
3.17-3.15 (1H, m),
2.98 (1H, m),
2.95 (1H, s),
2.94 (1H, s),
2.93 (1H, s),
2.91 (1H, d, J = 17.18, 4.12 Hz),
2.78 (3H, s),
1.11 (9H, s), 0.9 (3H, m),
1.02 (9H, s),
0.95-0.94 (3H, m),
0.95 (3H, s).

```



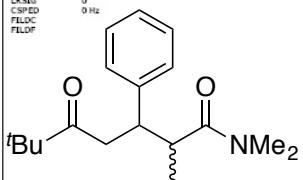
D:\Documents and Settings\Shu KOBAYASHI\Desktop\is097-c-pro-0916-1.xls

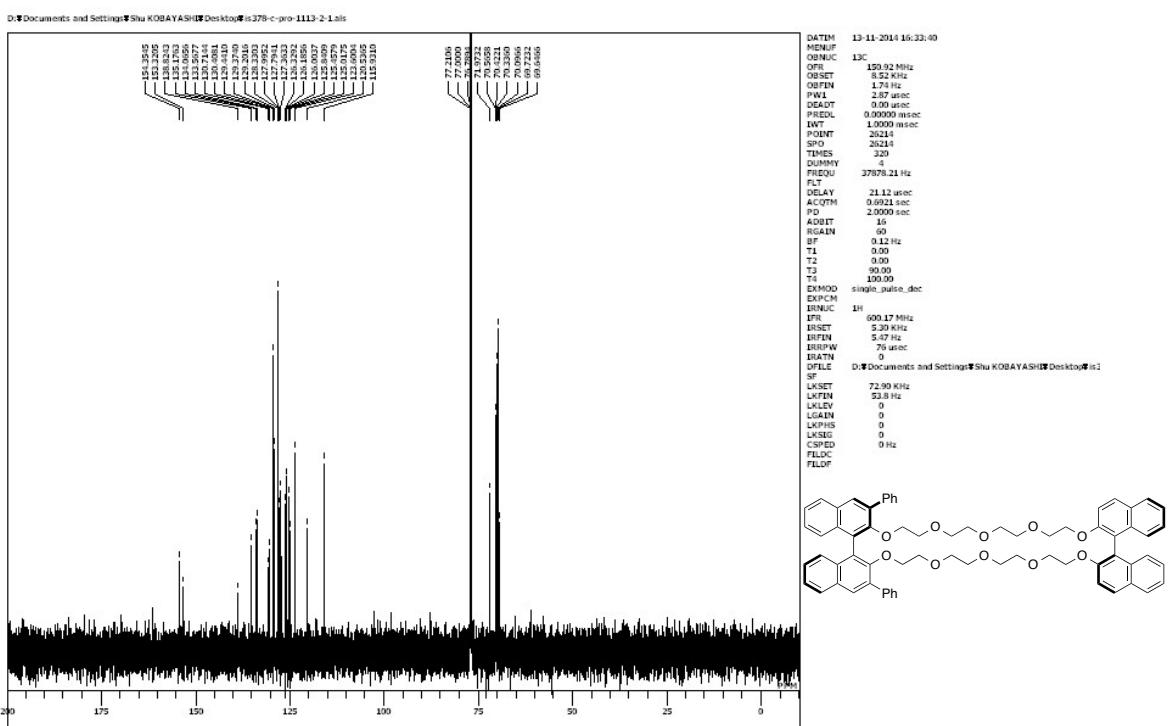
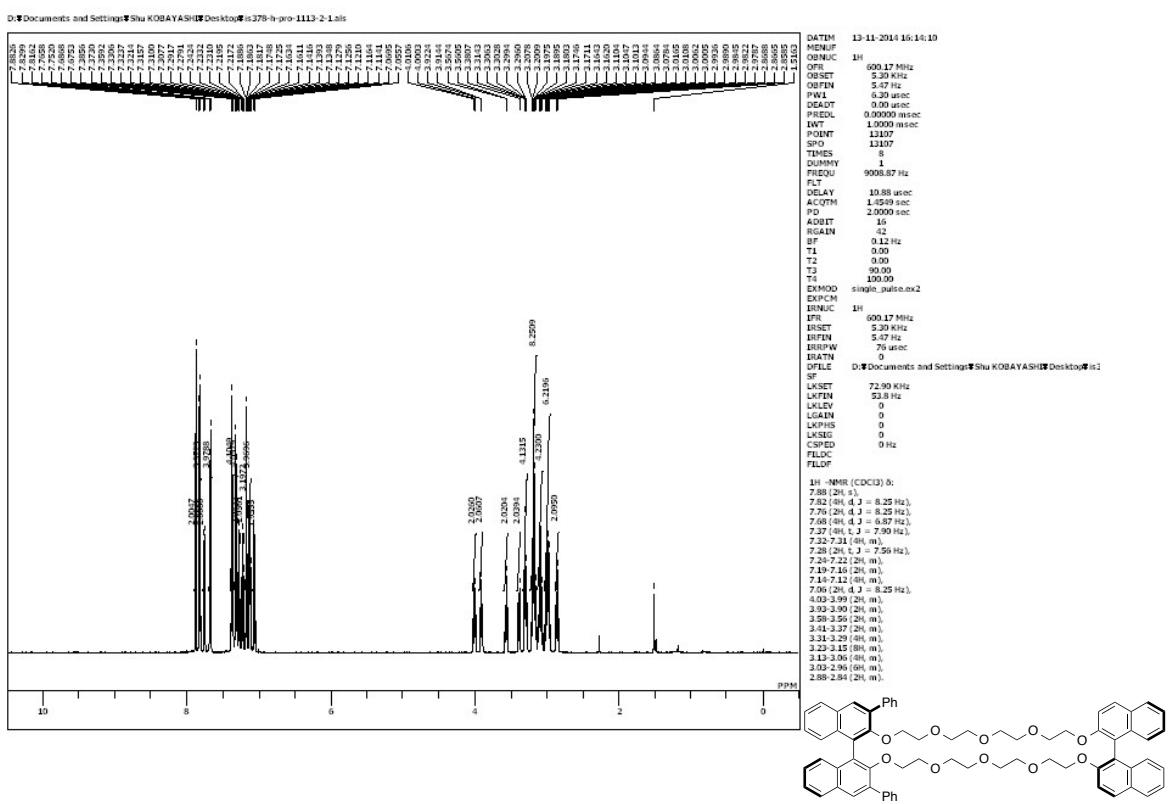


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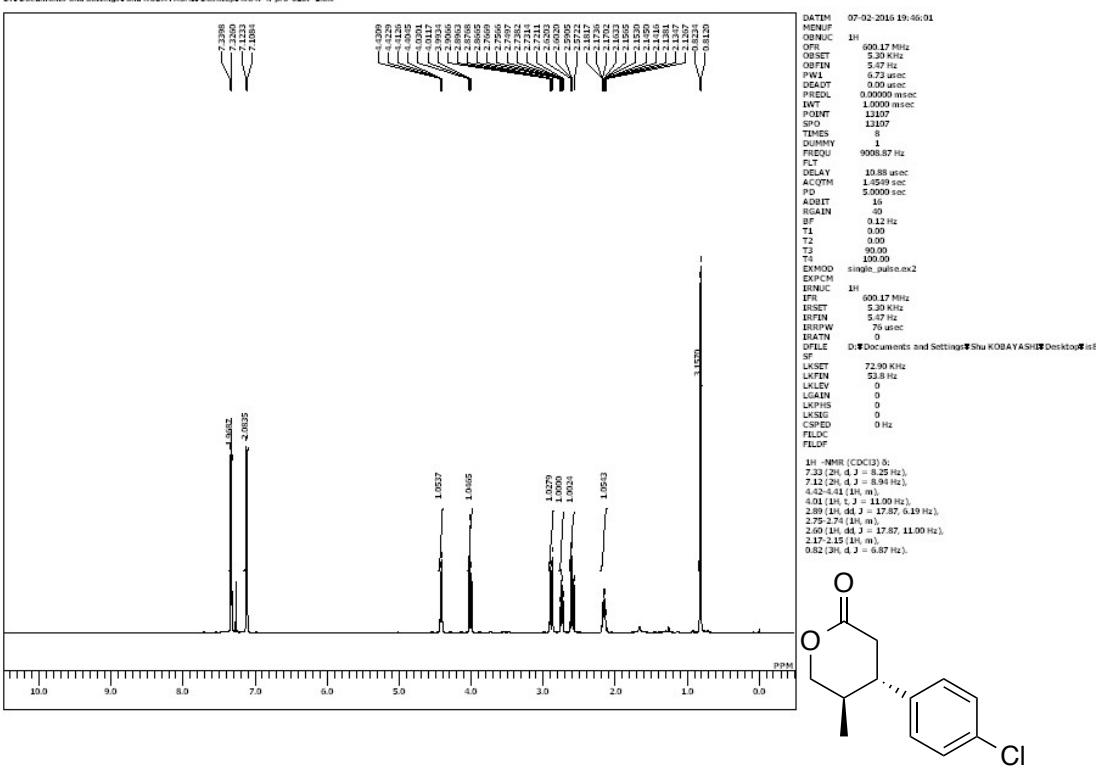
DATIM 16-09-2015 16:08:09
MENUFC 13C
DBNUC 1H
DPR 600.17 MHz
DBSET 8.52 kHz
DBTIN 1.74 Hz
PWR 0.5000
DEADT 0.00 usec
PREDL 0.00000 usec
INT 1.0000 usec
POINT 20214
SPO 20214
TD 51200 sec
AQB1T 5
AQB1T 5
RGAIN 29
BF 0.62 Hz
T1 0.00
T2 0.00
T3 90.00
TE 100.00
EXMOD single_pulse_dec
EXPCLM
DBNUC 1H
DPR 600.17 MHz
DBSET 5.30 kHz
DBTIN 5.47 Hz
DBPW 79.0 usec
DBATN 0
DTFILE D:\Documents and Settings\Shu KOBAYASHI\Desktop\is097-c-pro-0916-1.xls
SF
LKSET 72.90 KHz
LKTFN 53.8 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPEQ 0 Hz
FILDC
FILDF

```

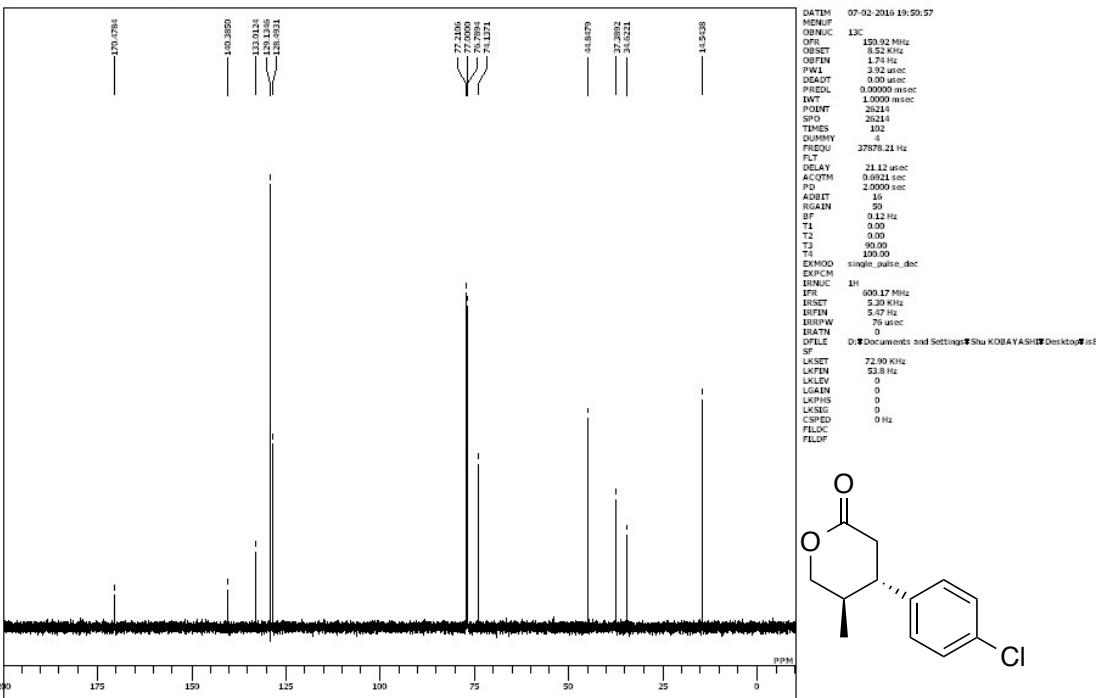




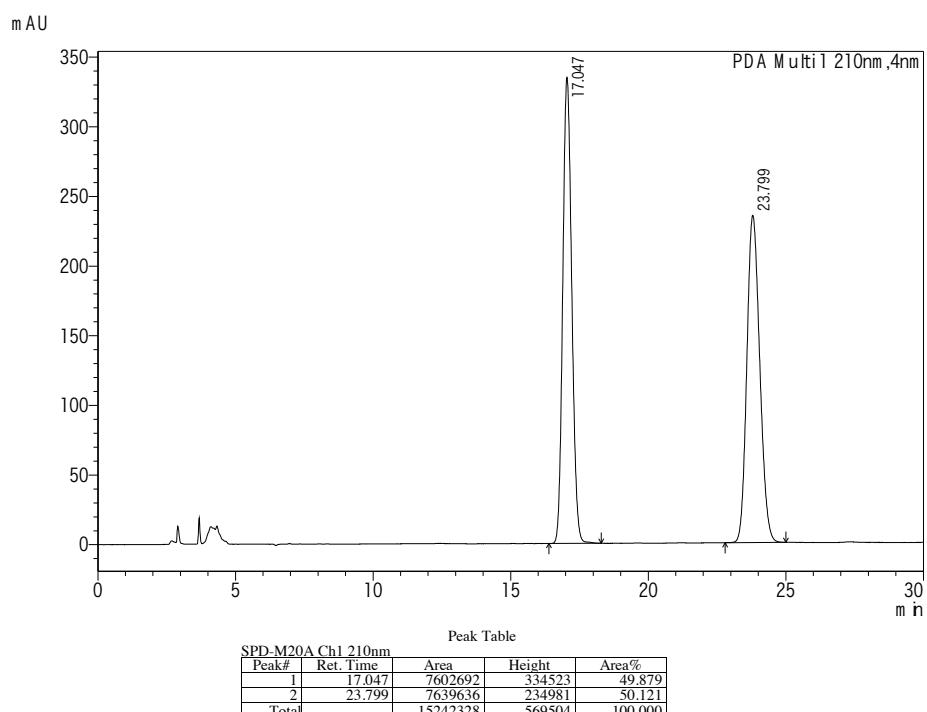
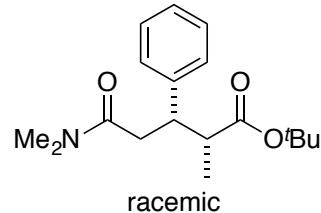
D:\Documents and Settings\Shu KOBAYASHI\Desktop\is847-h-pro-0207-1.xls



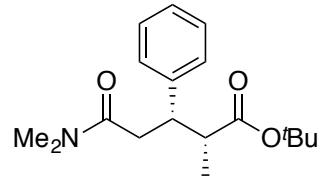
D:\Documents and Settings\Shu KOBAYASHI\Desktop\is847-c-pro-0207-1.xls



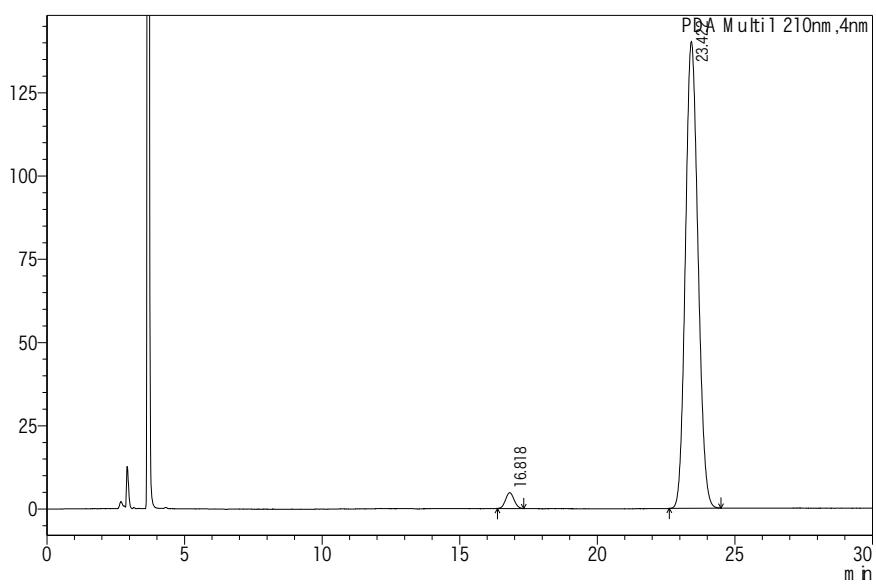
Acquired by :System Administrator
 Sample Name :is353-1-rac-0224
 Sample ID :is353-1-rac-0224
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is353-1-rac-0224.lcd
 Method File :0-1.0-60.tcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/02/24 10:31:20
 Date Processed :2015/02/24 11:44:13



Acquired by : System Administrator
 Sample Name : is456-1-0224
 Sample ID : is456-1-0224
 Tray# : 1
 Vial# : 1
 Injection Volume : 5456-1-0224.lcd
 Data File : 0-1.0-60.tsm
 Method File :
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/02/24 11:45:40
 Date Processed : 2015/02/24 19:03:46

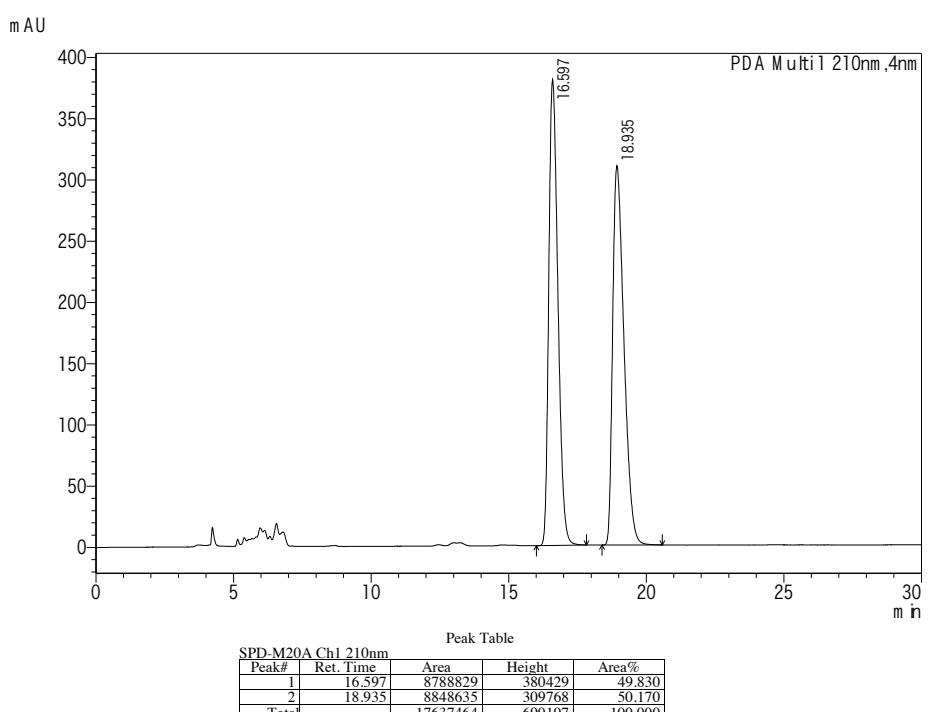
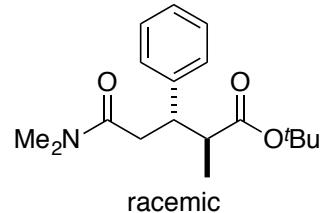


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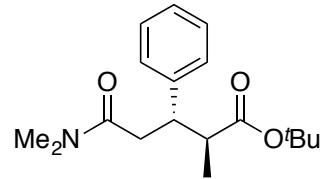
D:\Persona Files\Sato\is456-1-0224.lcd

Acquired by :System Administrator
 Sample Name :is353-2-racem ic-0228
 Sample ID :is353-2-racem ic-0228
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is353-2-racem ic-0228.lcd
 Method File :0-0.7-90.lcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/02/28 10:52:39
 Date Processed :2015/02/28 11:52:01

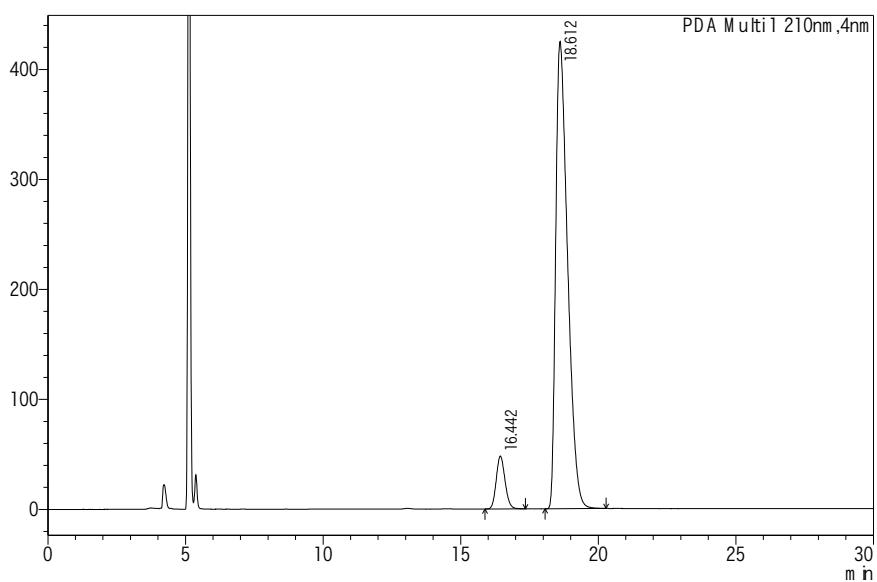


D:\Personal\Files\Sato\is353-2-racem ic-0228.lcd

Acquired by :System Administrator
 Sample Name :s456-2-2nd-0228
 Sample ID :s456-2-2nd-0228
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :s456-2-2nd-0228.lcd
 Method File :0-0.7-90.lcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/02/28 12:57:25
 Date Processed :2015/02/28 17:06:16



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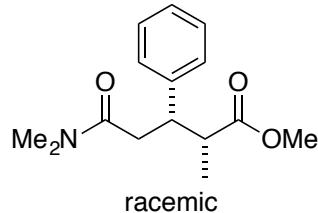


Peak Table

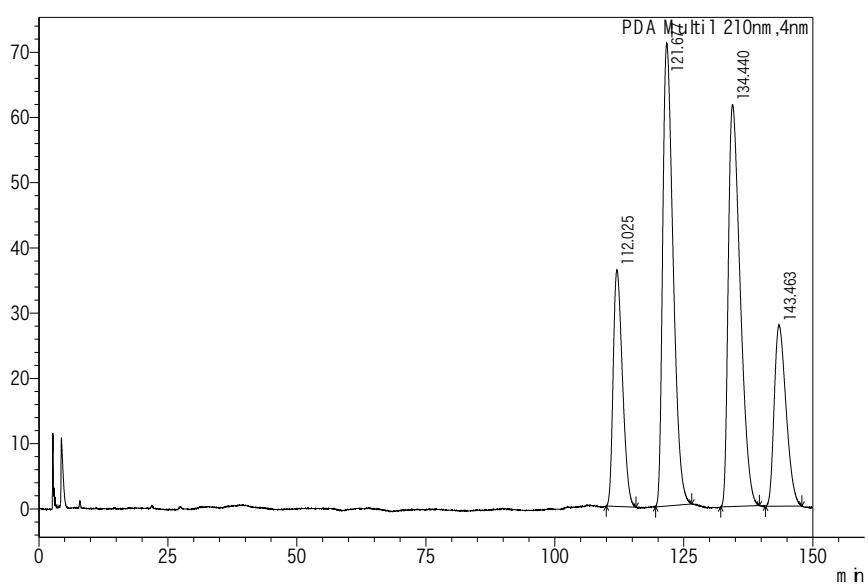
SPD-M20A Ch1 210nm				
Peak#	Ret. Time	Area	Height	Area%
1	16.442	1067869	48057	7.738
2	18.612	12732459	424841	92.262
Total		13800328	472898	100.000

D:\Persona\Files\Sato\s456-2-2nd-0228.lcd

Acquired by : System Administrator
 Sample Name : ls419-1rac-ad3-0316
 Sample ID : ls419-1rac-ad3-0316
 Tray# : 1
 Vial# : 1
 Injection Volume :
 Data File : ls419-1rac-ad3-0316.lcd
 Method File : 0-1.0-60.lcm
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/03/16 16:21:07
 Date Processed : 2015/03/16 20:38:49

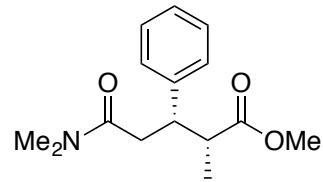


m AU

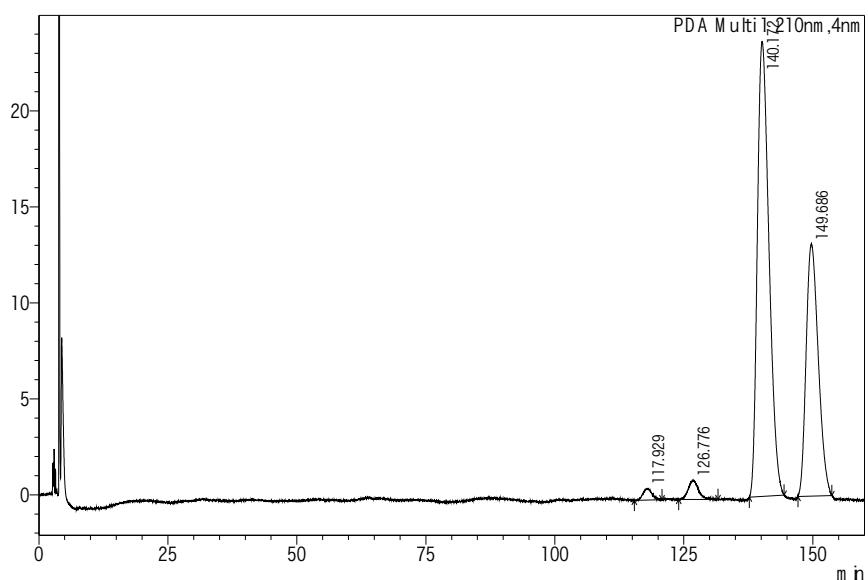


D:\Persona\Files\Save\ls419-1rac-ad3-0316.lcd

Acquired by :System Administrator
 Sample Name :is421-rac-ad3-0316
 Sample ID :is421-rac-ad3-0316
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is421-ad3-0316.lcd
 Method File :0-1.0-60.lm
 Batch File :
 Report Format File :DEFAULT.lrf
 Date Acquired :2015/03/16 18:55:42
 Date Processed :2015/03/16 22:18:51



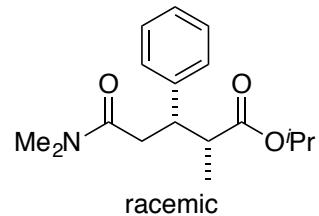
m AU



Peak Table

SPD-M20A Ch1 210nm				
Peak#	Ret. Time	Area	Height	Area%
1	117.929	71288	599	1.173
2	126.776	138398	996	2.277
3	140.172	3714934	23706	61.118
4	149.686	2152700	13143	35.432
Total		6078319	38444	100.000

D:\Persona\Files\Sato\is421-ad3-0316.lcd

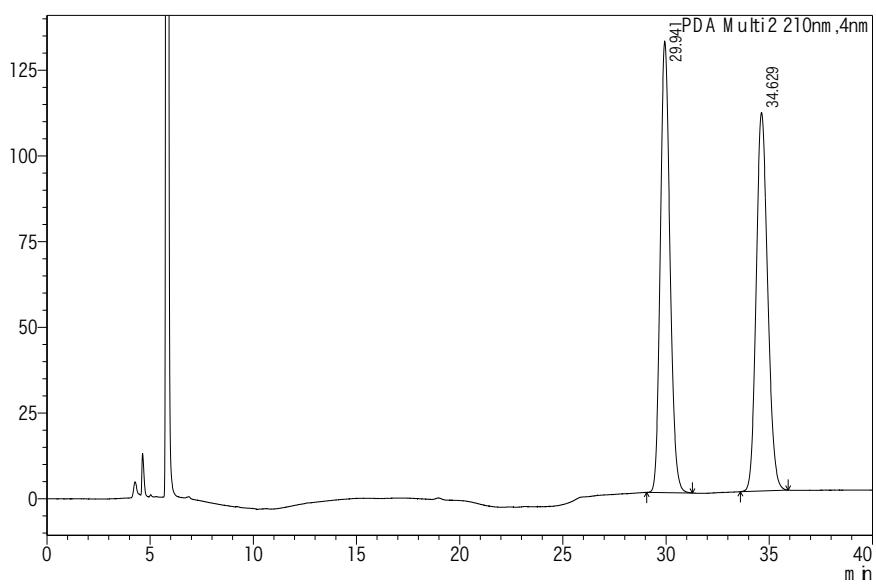


```

Acquired by :System Administrator
Sample Name :is427-1-2nd-ad3-1223
Sample ID   :is427-1-2nd-ad3-1223
Tray#       :1
Vial#       :1
Injection Volume :1
Data File   :is427-1-2nd-ad3-1223.tdf
Method File  :0-0.7-90.tmf
Batch File   :.
Report Form at File :DEFAULT.lsr
Date Acquired :2014/12/23 11:50:53
Date Processed:2014/12/23 13:45:40

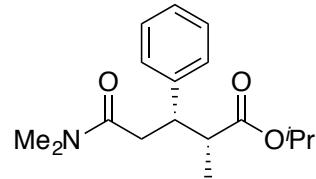
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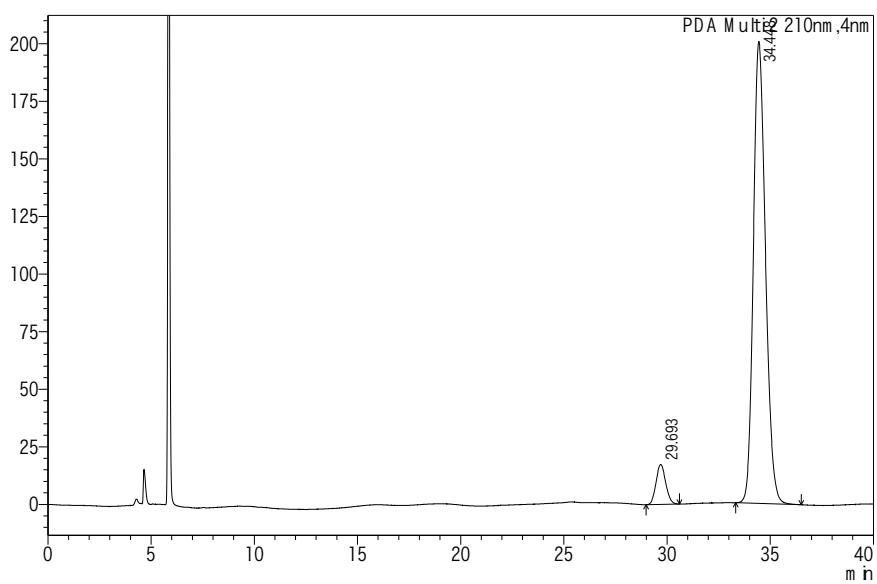


D:\Personals\Files\Sato\is427-1-2nd-ad3-1223.tdf

Acquired by :System Administrator
 Sample Name :is426-1-ad3-1223
 Sample ID :is426-1-ad3-1223
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is426-1-ad3-1223.lcd
 Method File :0-0.7-90.lcm
 Batch File :
 Report Form at File :DEFAULT.lsr
 Date Acquired :2014/12/23 12:47:16
 Date Processed :2014/12/23 13:56:30

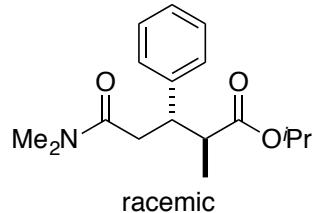


m AU

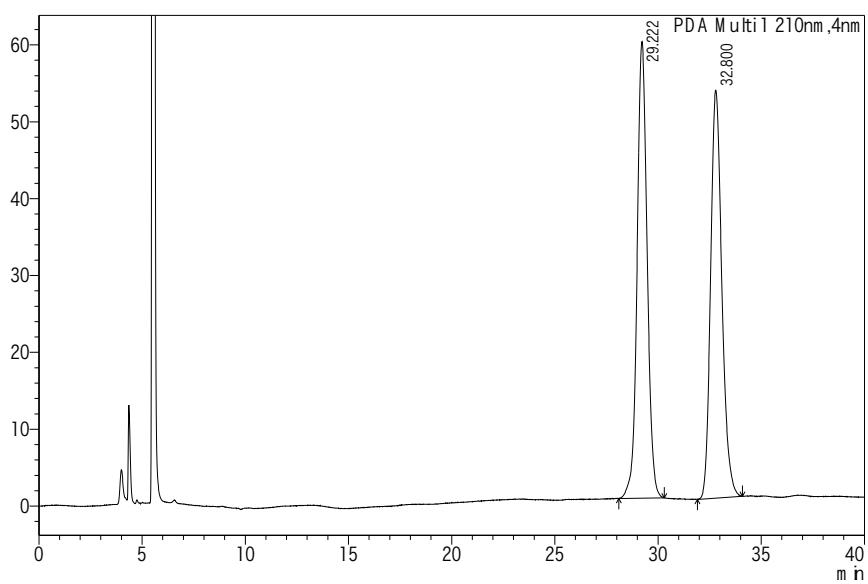


D:\Persona Files\Sato\is426-1-ad3-1223.lcd

Acquired by : System Administrator
 Sample Name : is427-2-ad3-1223
 Sample ID : is427-2-ad3-1223
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.0000000000000001
 Data File : is427-2-ad3-1223.lcd
 Method File : O-0.7-90.lcm
 Batch File :
 Report Form at File : DEFAULT.lsr
 Date Acquired : 2014/12/23 13:29:30
 Date Processed : 2014/12/23 17:14:54

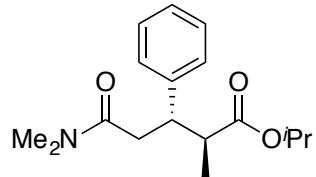


mAU

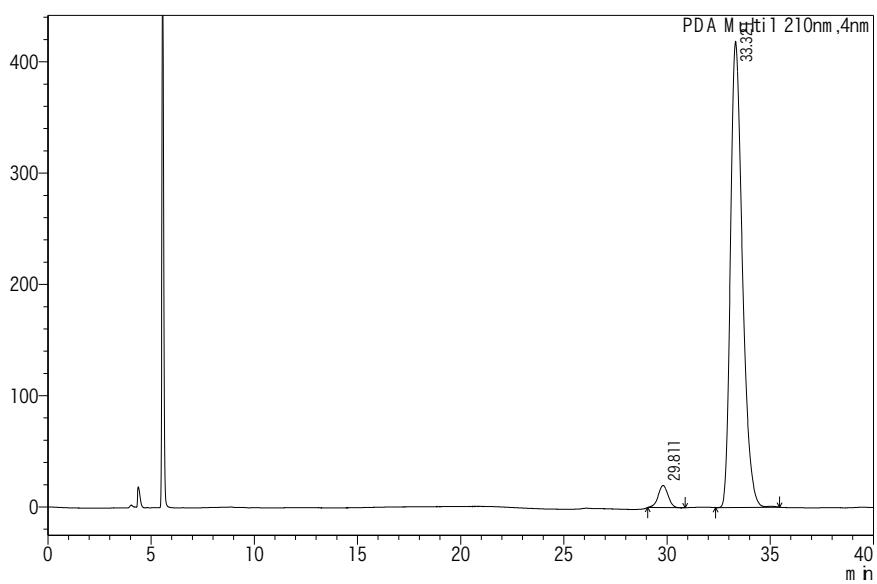


D:\Persona Files\Sato\is427-2-ad3-1223.lcd

Acquired by : System Administrator
 Sample Name : is426-2-ad3-1223
 Sample ID : is426-2-ad3-1223
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.0000000000000001
 Data File : is426-2-ad3-1223.lcd
 Method File : O-0.7-90.lcm
 Batch File :
 Report Form at File : DEFAULT.lsr
 Date Acquired : 2014/12/23 14:24:51
 Date Processed : 2014/12/23 17:14:47

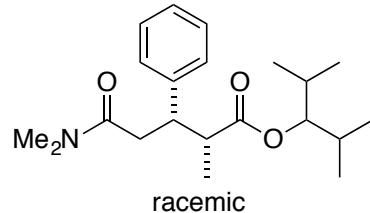


mAU

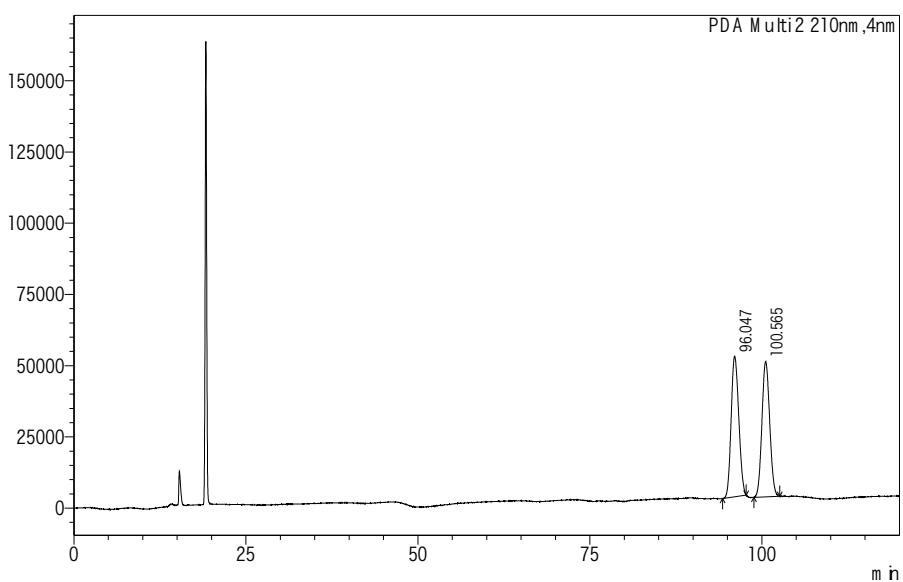


D:\Persona Files\Sato\is426-2-ad3-1223.lcd

Acquired by :System Administrator
 Sample Name :is415-1-ad3adh-0107
 Sample ID :is415-1-ad3adh-0107
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is415-1-ad3adh-0107-1.td
 Method File :0-0.4-120.km
 Batch File :
 Report Format File :DEFAULT.ls
 Date Acquired :2015/01/07 11:24:13
 Date Processed :2015/01/07 13:43:10

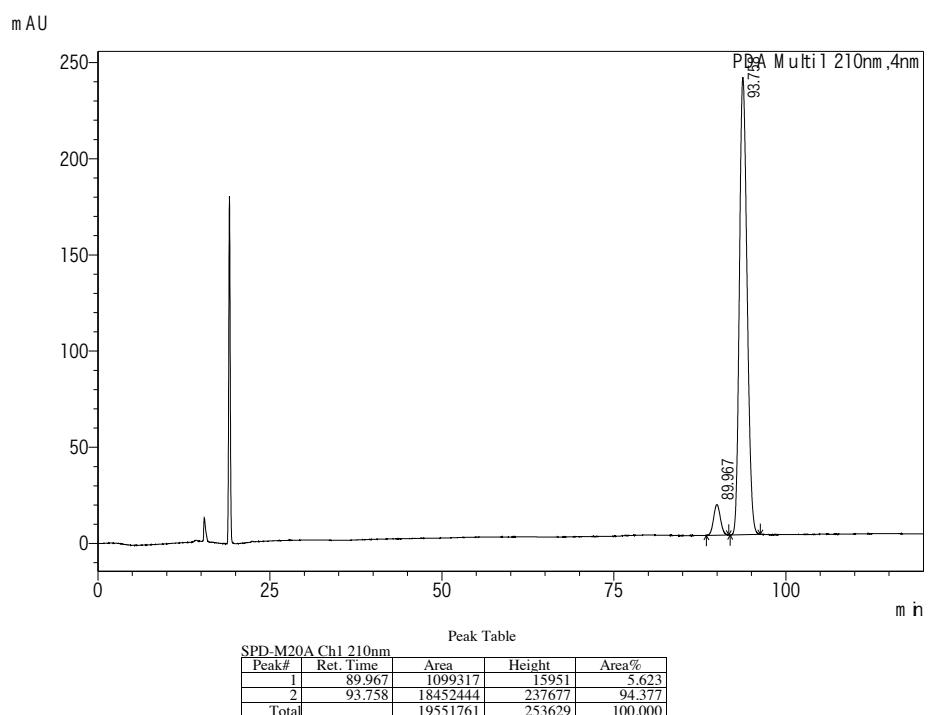
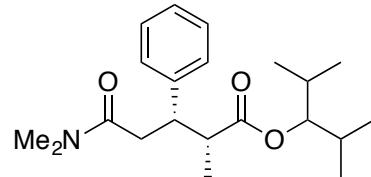


uAU



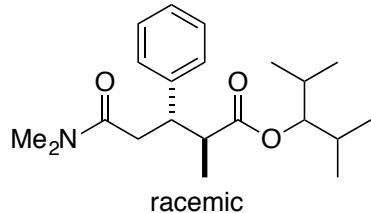
D:\Persona\Files\Sato\is415-1-ad3adh-0107-1.td

Acquired by : System Administrator
 Sample Name : s425-1-ad3adh-0107
 Sample ID : s425-1-ad3adh-0107
 Tray# : 1
 Vial# : 1
 Injection Volume : 0.4-120.0µL
 Data File : s425-1-ad3adh-0107.lcd
 Method File : 0-0.4-120.km
 Batch File :
 Report Format File : DEFAULT.lrf
 Date Acquired : 2015/01/07 13:31:05
 Date Processed : 2015/01/07 15:34:24

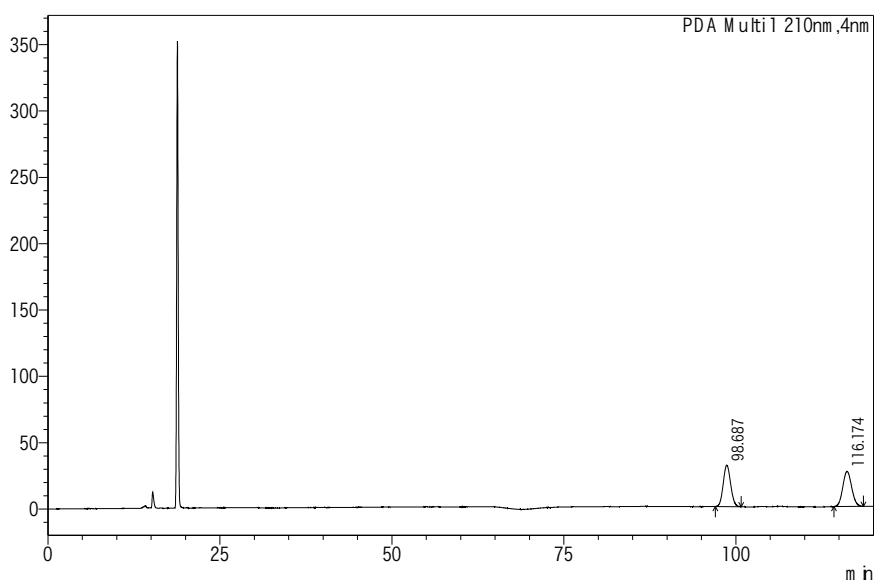


D:\Persona\Files\Sato\s425-1-ad3adh-0107.lcd

Acquired by :System Administrator
 Sample Name :s415-2-ad3adh-0107
 Sample ID :s415-2-ad3adh-0107
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :s415-2-ad3adh-0107.lcd
 Method File :0-0.4-120.km
 Batch File :
 Report Format File :DEFAULT.lrf
 Date Acquired :2015/01/07 15:36:19
 Date Processed :2015/01/07 21:55:52

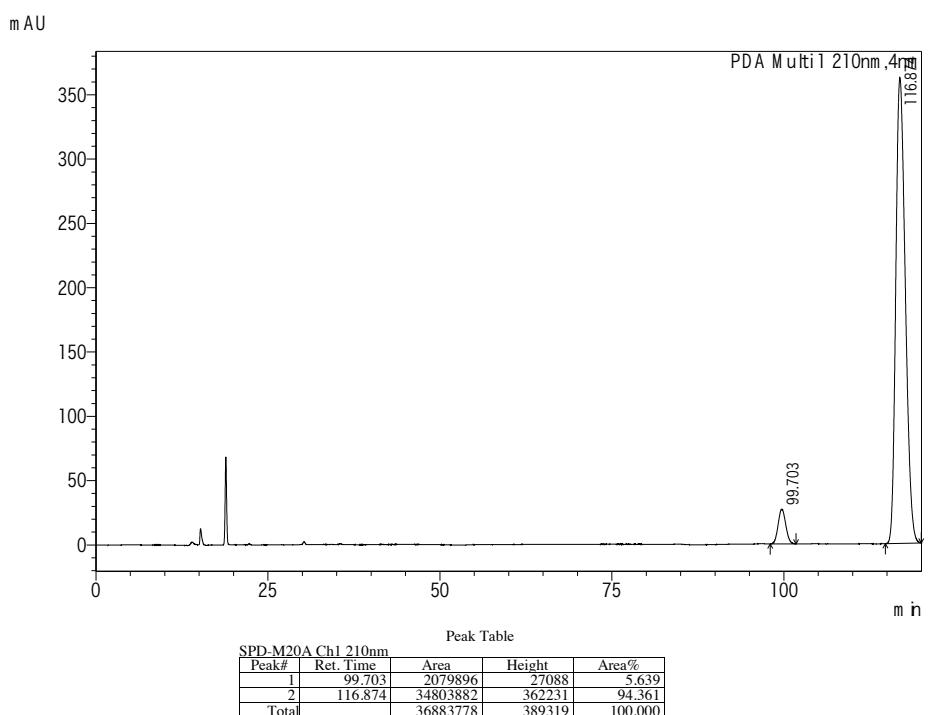
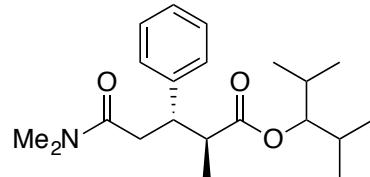


m AU



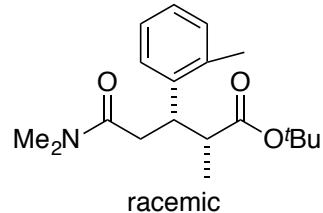
D:\Persona\Files\Sato\s415-2-ad3adh-0107.lcd

Acquired by :System Administrator
 Sample Name :s425-2-ad3adh-0107
 Sample ID :s425-2-ad3adh-0107
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :s425-2-ad3adh-0107.lcd
 Method File :0-0.4-120.km
 Batch File :
 Report Format File :DEFAULT.lrf
 Date Acquired :2015/01/07 18:52:31
 Date Processed :2015/01/07 21:55:19

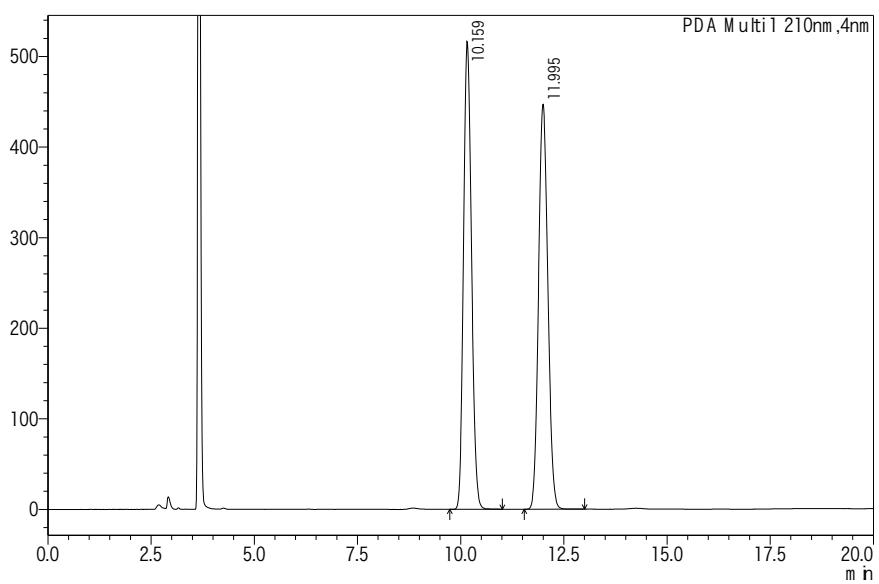


D:\Persona\Files\Sato\s425-2-ad3adh-0107.lcd

Acquired by :System Administrator
 Sample Name :is479-1-o-tol-rac-0317
 Sample ID :is479-1-o-tol-rac-0317
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is479-1-o-tol-rac-0317.tdf
 Method File :0-1.0-60.tmf
 Batch File :
 Report Format File :DEFAULT.rfr
 Date Acquired :2015/03/17 19:46:52
 Date Processed :2015/03/17 20:10:01

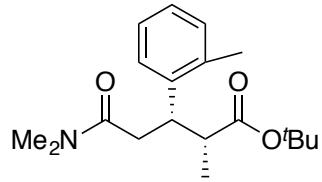


m AU

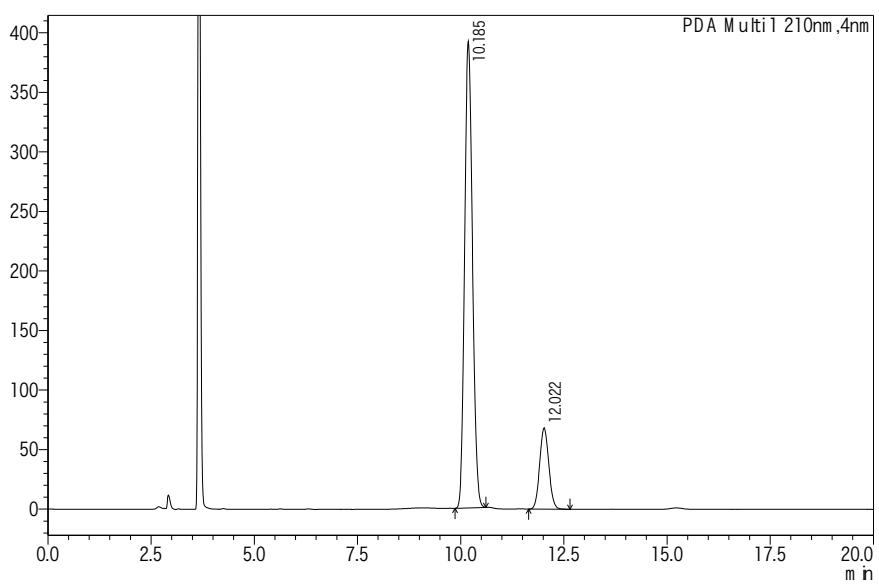


D:\Persona\Files\Sato\is479-1-o-tol-rac-0317.tdf

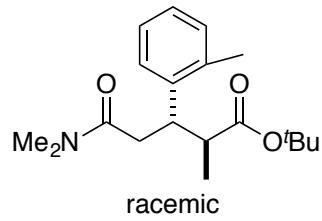
Acquired by : System Administrator
 Sample Name : is494-1-o-to-l-0317
 Sample ID : is494-1-o-to-l-0317
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.0000000000000001
 Data File : is494-1-o-to-l-0317.lcd
 Method File : O-1.0-60.lcm
 Batch File :
 Report Form at File : DEFAULT.lsr
 Date Acquired : 2015/03/17 20:11:24
 Date Processed : 2015/03/17 21:48:08



m AU



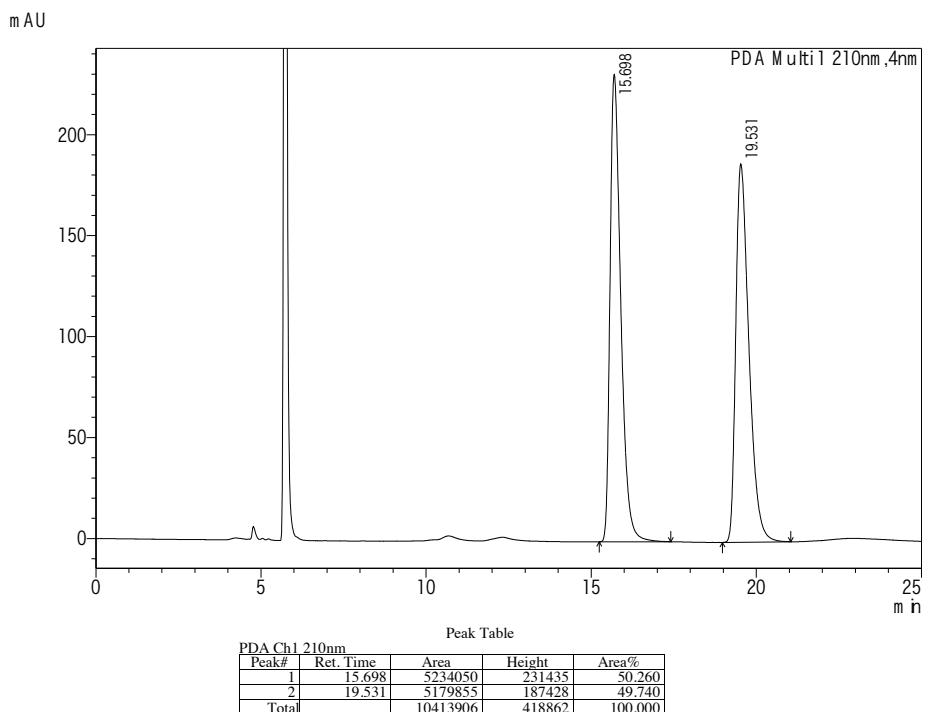
D:\Persona\Files\Sato\is494-1-o-to-l-0317.lcd



```

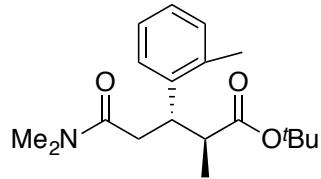
Acquired by : System Administrator
Sample Name : ls479-2-o-tol-rac-0319
Sample ID   : ls479-2-o-tol-rac-0319
Tray#       :
Vial#       :
Injection Volume : 1
Data File   : ls479-2-o-tol-rac-0319.lcd
Method File  : 0-0.7-90.lcm
Batch File   :
Report Form at File : DEFAULT.lsr
Date Acquired : 2015/03/19 14:24:53
Date Processed: 2015/03/19 17:17:39

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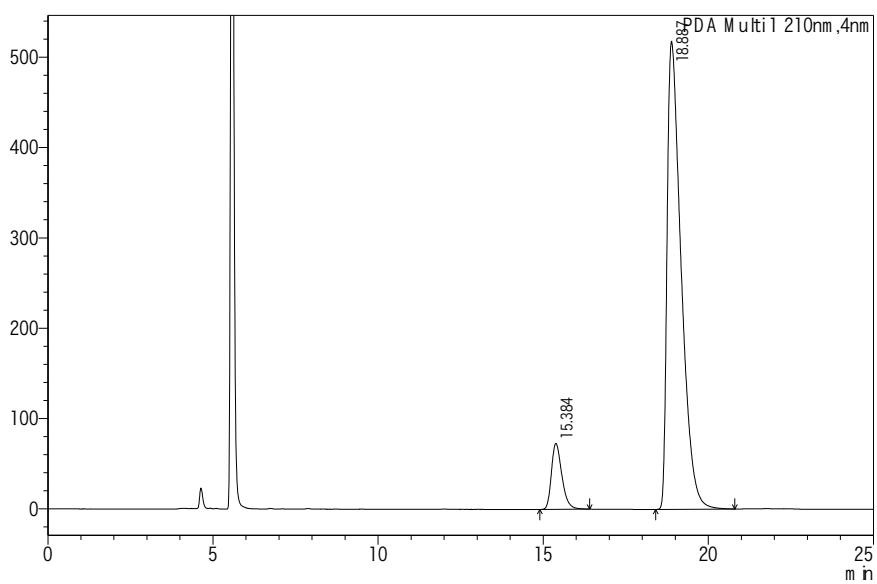


D:\Persona\Files\Sato\ls479-2-o-tol-rac-0319.lcd

Acquired by : System Administrator
 Sample Name : is494-2-o-to-l-0319
 Sample ID : is494-2-o-to-l-0319
 Tray# :
 Vial# :
 Injection Volume :
 Data File : is494-2-o-to-l-0319.lcd
 Method File : 0-0.7-90.lcm
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/03/19 14:54:27
 Date Processed : 2015/03/19 17:17:28



m AU

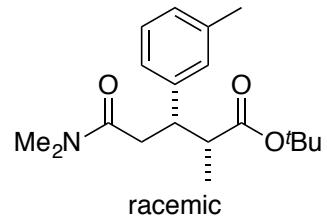


Peak Table

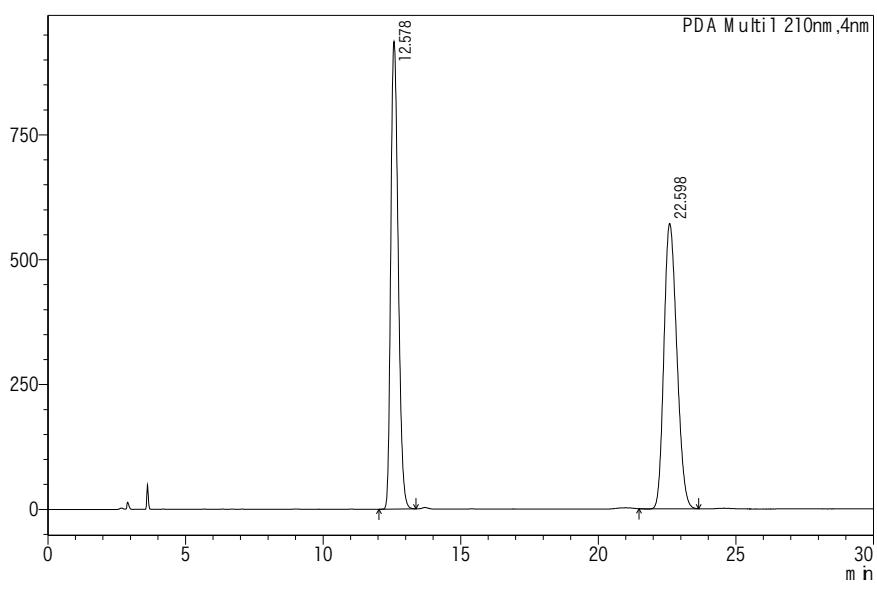
PDA Ch1 210nm					
Peak#	Ret. Time	Area	Height	Area%	
1	15.384	1608787	72982	9.507	
2	18.887	15314115	518027	90.493	
Total		16922902	591010	100.000	

D:\Persona\Files\Sato\is494-2-o-to-l-0319.lcd

Acquired by :System Administrator
 Sample Name :s743-1-rac-1103
 Sample ID :s743-1-rac-1103
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :s743-1-rac-1103.lcd
 Method File :O-1.0-60.tcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/11/03 16:30:44
 Date Processed :2015/11/03 17:59:13

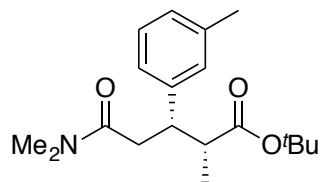


m AU

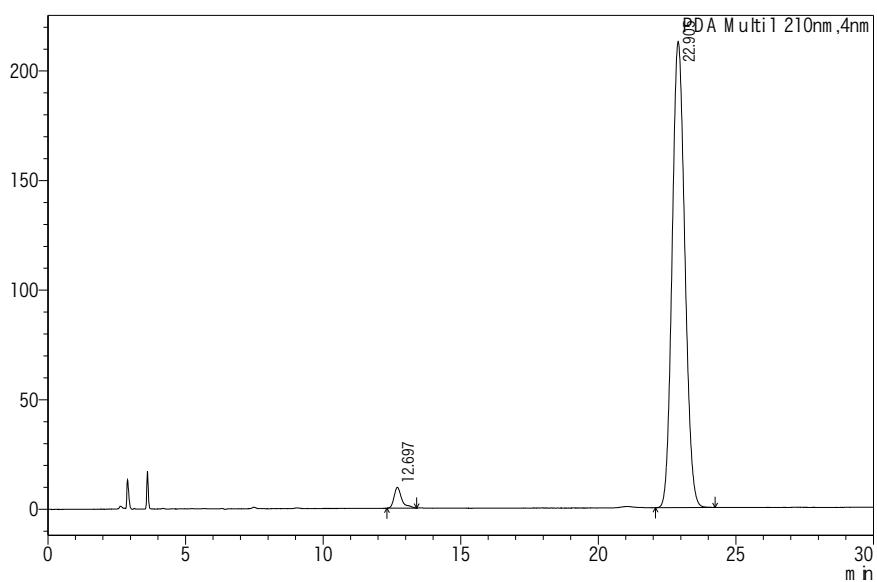


D:\Persona\Files\Sato\s743-1-rac-1103.lcd

Acquired by :System Administrator
 Sample Name :is753-1-1103
 Sample ID :is753-1-1103
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is753-1-1103.lcd
 Method File :0-1.0-60.lcm
 Batch File :
 Report Format File :DEFAULT.lrf
 Date Acquired :2015/11/03 17:11:34
 Date Processed :2015/11/03 17:59:08

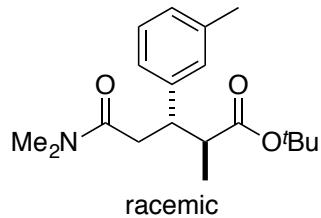


m AU

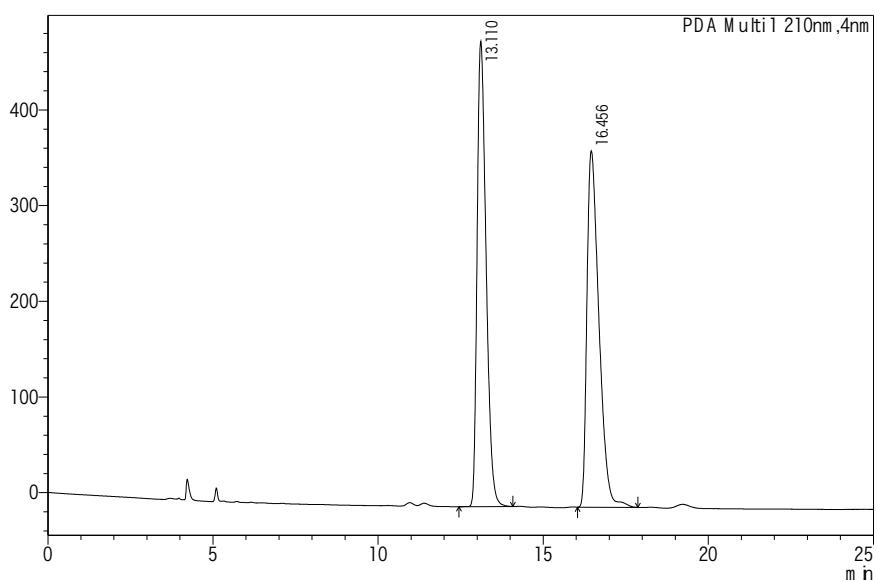


D:\Persona\Files\Sato\is753-1-1103.lcd

Acquired by :System Administrator
 Sample Name :s743-2-rac-1103
 Sample ID :s743-2-rac-1103
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :s743-2-rac-1103.lcd
 Method File :0-0.7-90.lcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/11/03 19:16:41
 Date Processed :2015/11/03 19:56:59

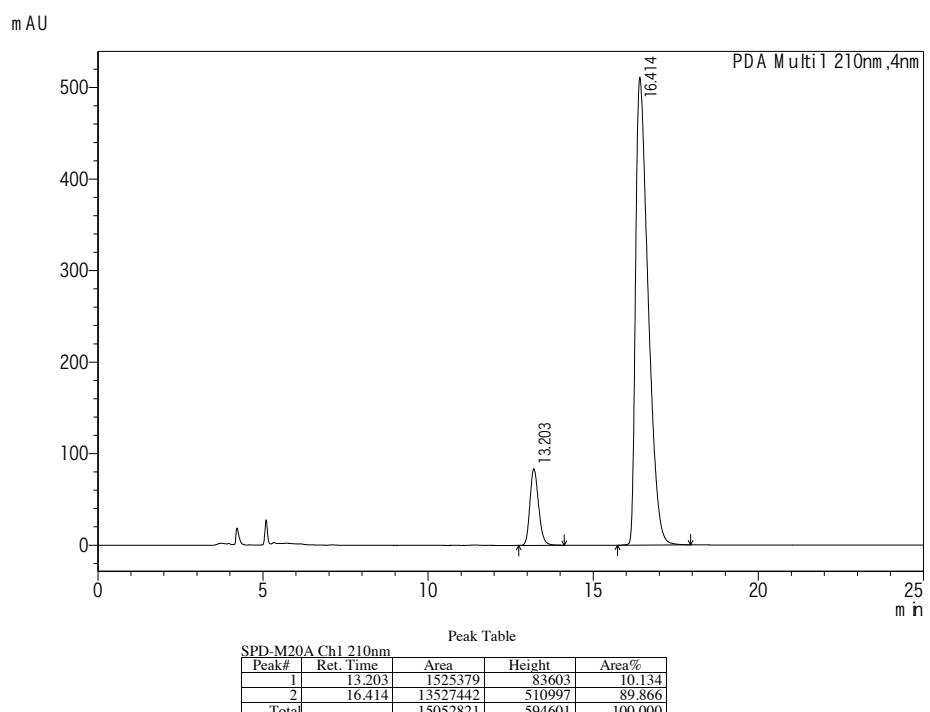
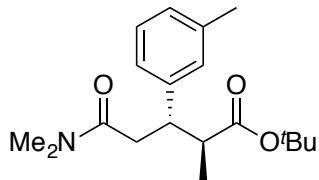


m AU

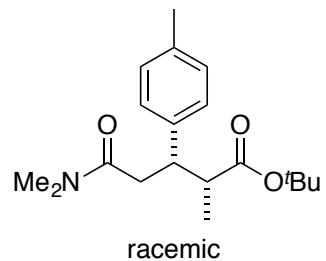


D:\Persona\Files\Sato\s743-2-rac-1103.lcd

Acquired by :System Administrator
 Sample Name :is753-2-1103
 Sample ID :is753-2-1103
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is753-2-1103.lcd
 Method File :0-0.7-90.lm
 Batch File :
 Report Format File :DEFAULT.lrf
 Date Acquired :2015/11/03 19:57:49
 Date Processed :2015/11/03 20:59:37



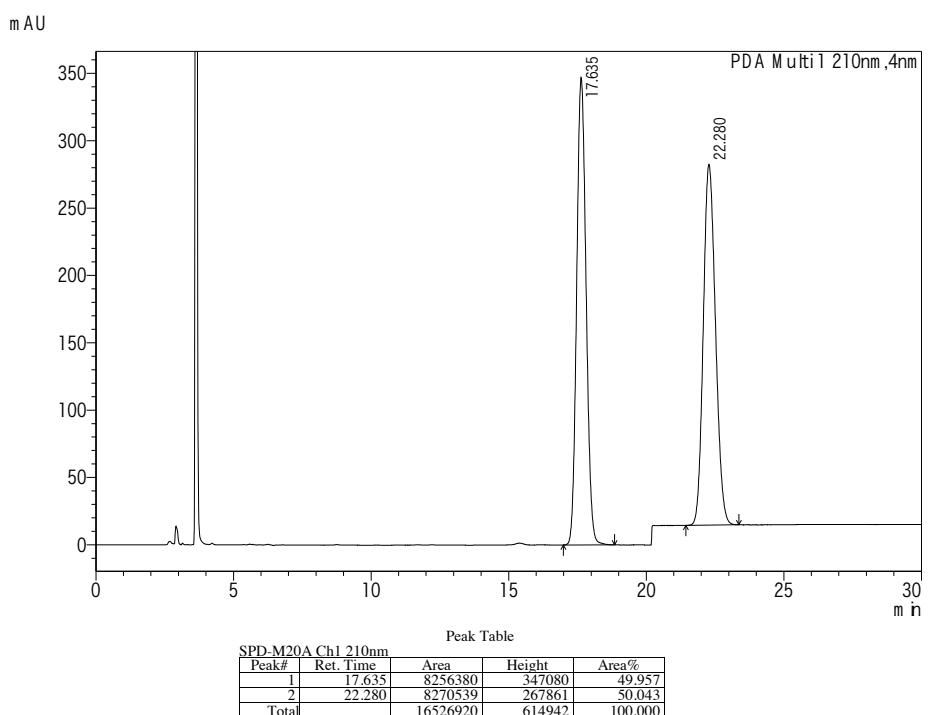
D:\Persona\Files\Sato\is753-2-1103.lcd



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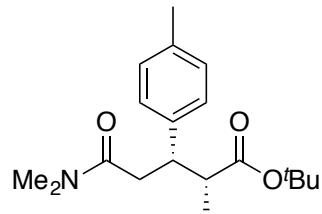
Acquired by :System Administrator
Sample Name :is480-1-p-tol-rac-0319
Sample ID   :is480-1-p-tol-rac-0319
Tray#       :1
Vial#       :1
Injection Volume :1
Data File   :is480-1-p-tol-rac-0319.lcd
Method File :0-1.0-60.tcm
Batch File  :
Report Format File :DEFAULT.lsr
Date Acquired :2015/03/19 13:17:37
Date Processed :2015/03/19 14:30:50

```

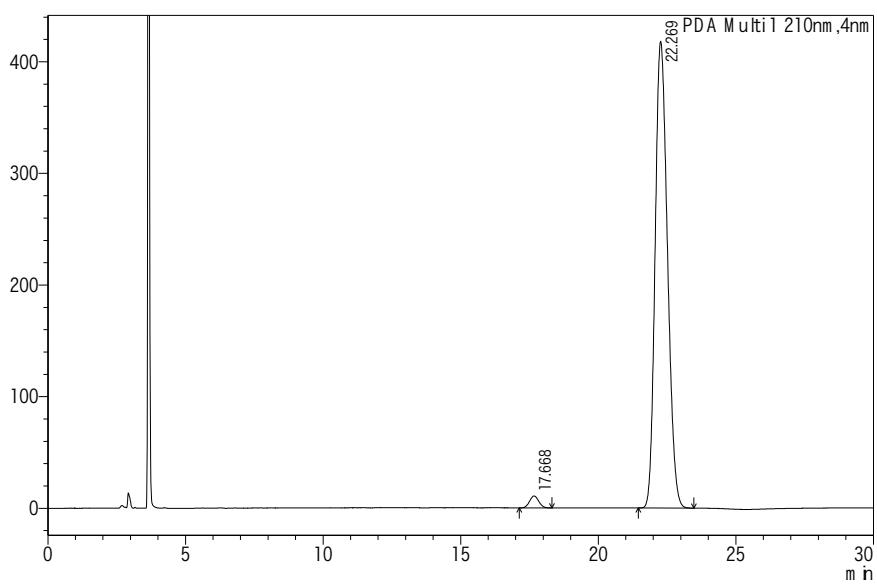


D:\Persona\Files\Sato\is480-1-p-tol-rac-0319.lcd

Acquired by :System Administrator
 Sample Name :s495-1-p-to-l-0319
 Sample ID :s495-1-p-to-l-0319
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :s495-1-p-to-l-0319.lcd
 Method File :O-1.0-60.lcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/03/19 13:56:08
 Date Processed :2015/03/19 14:30:42



mAU



Peak Table

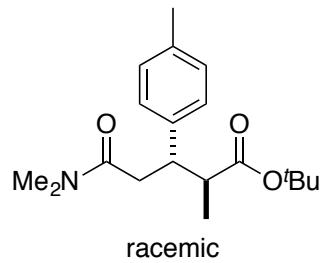
SPD-M20A Ch1 210nm				
Peak#	Ret. Time	Area	Height	Area%
1	17.668	251874	10775	1.896
2	22.269	13029761	417999	98.104
Total		13281635	428774	100.000

D:\Persona\Files\Sato\s495-1-p-to-l-0319.lcd

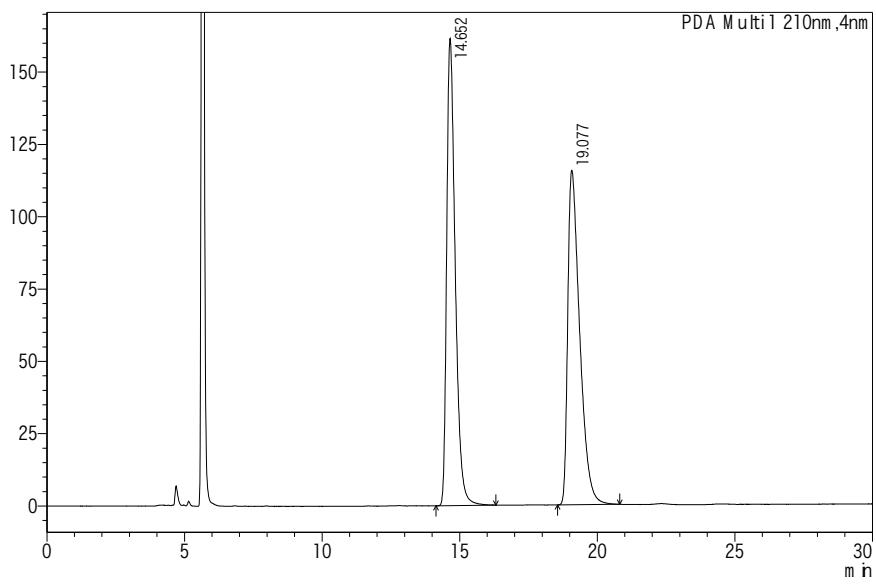
```

Acquired by :System Administrator
Sample Name :is480-2-p-tol-l-rac-0319
Sample ID   :is480-2-p-tol-l-rac-0319
Tray#       :]
Vial#       :]
Injection Volume :]
Data File   :is480-2-p-tol-l-rac-0319.lcd
Method File :0-0.7-90.lcm
Batch File  :]
Report Form at File :DEFAULT.lsr
Date Acquired :2015/03/19 17:09:39
Date Processed :2015/03/19 18:50:42

```



m AU

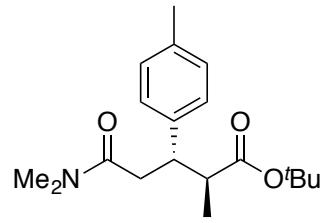


D:\Persona\Files\Sato\is480-2-p-tol-l-rac-0319.lcd

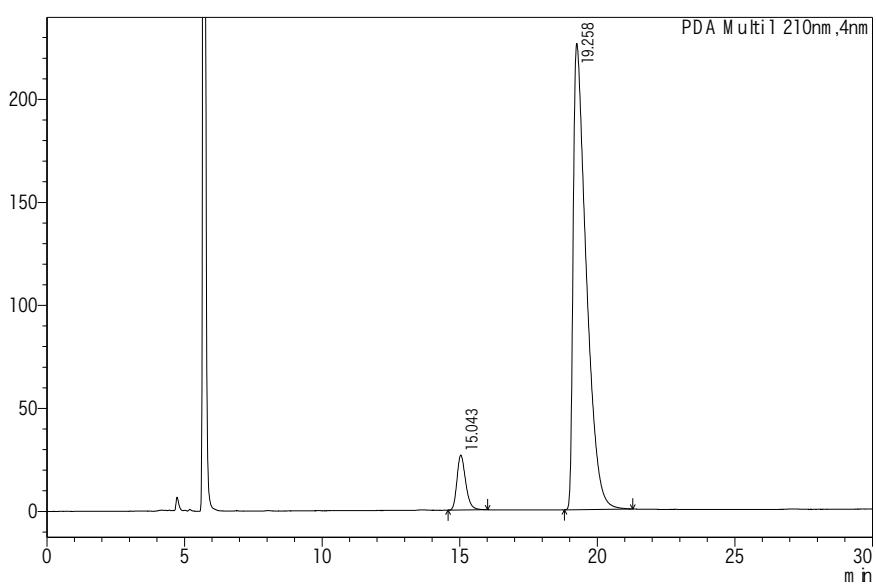
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Acquired by :System Administrator
Sample Name :is495-2-p-to-l-0319
Sample ID   :is495-2-p-to-l-0319
Tray#       :]
Vial#       :]
Injection Volume :1
Data File   :is495-2-p-to-l-0319.lcd
Method File :0-0.7-90.lcm
Batch File  :]
Report Form at File :DEFAULT.lsr
Date Acquired :2015/03/19 17:43:04
Date Processed:2015/03/19 18:50:37

```

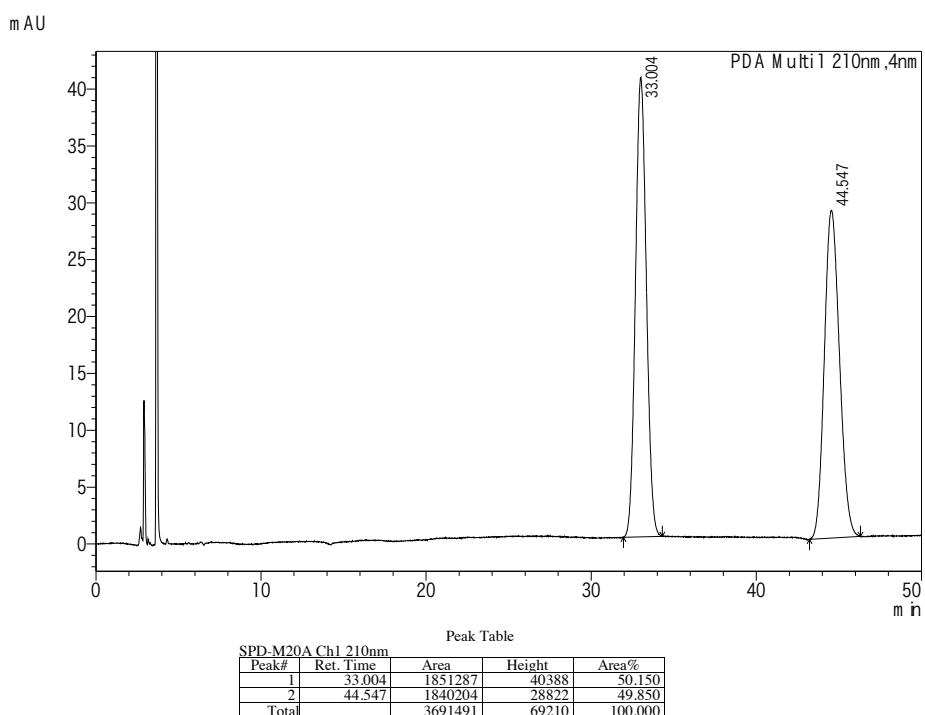
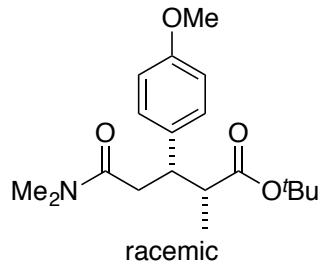


m AU



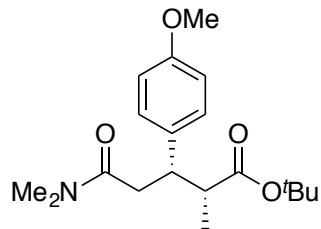
D:\Persona\Files\Sato\is495-2-p-to-l-0319.lcd

Acquired by : System Administrator
 Sample Name : s481-1-p-O Me-rac-0324
 Sample ID : s481-1-p-O Me-rac-0324
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.000 μL
 Data File : s481-1-p-O Me-rac-0324.lcd
 Method File : O-1.0-60.tcm
 Batch File :
 Report Format File : DEFAULT.rfr
 Date Acquired : 2015/03/24 10:54:06
 Date Processed : 2015/03/24 11:54:11

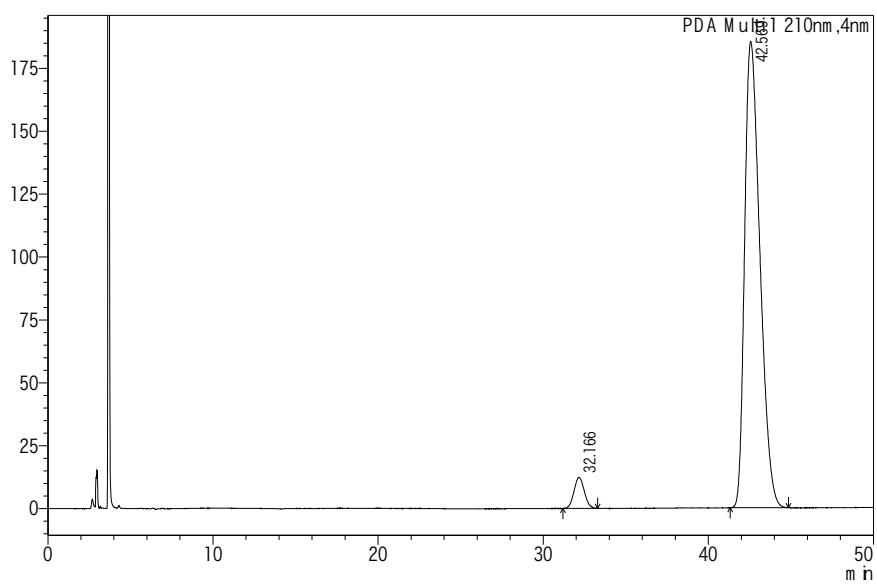


D:\Persona\Files\Sato\s481-1-p-O Me-rac-0324.lcd

Acquired by :System Administrator
 Sample Name :ls503-1-p-0Me-e-0324
 Sample ID :ls503-1-p-0Me-e-0324
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :ls503-1-p-0Me-e-0324.lcd
 Method File :0-1.0-60.tcm
 Batch File :
 Report Format File :DEFAULT.rpr
 Date Acquired :2015/03/24 12:03:00
 Date Processed :2015/03/24 13:19:56



mAU

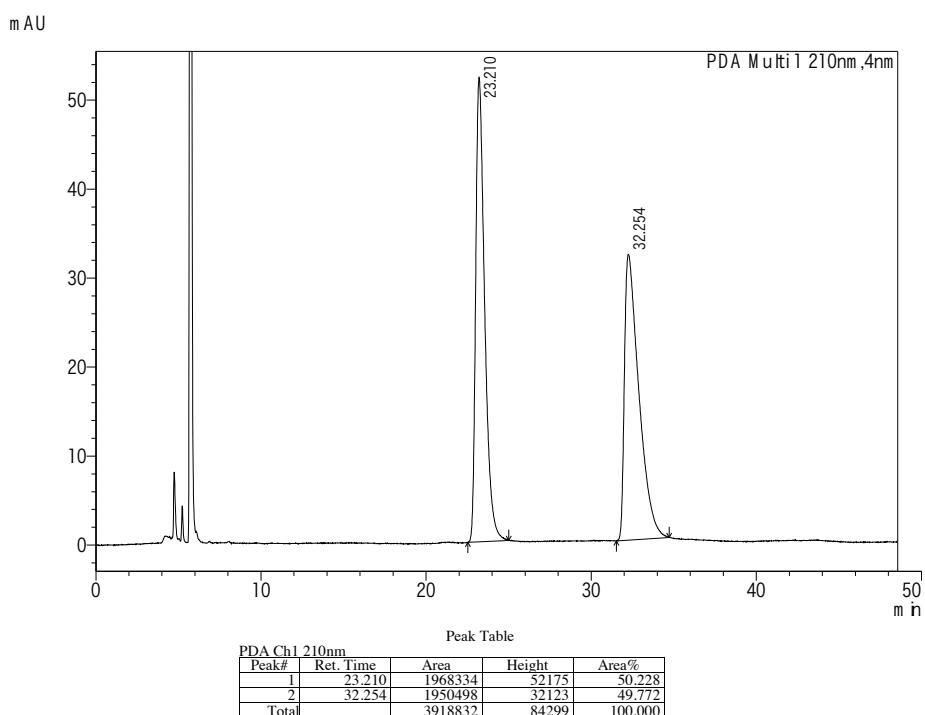
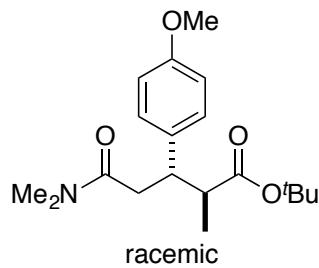


D:\Persona\Files\Sato\ls503-1-p-0Me-e-0324.lcd

```

Acquired by :System Administrator
Sample Name :i481-2-p-OMe-rac-0324
Sample ID   :i481-2-p-OMe-rac-0324
Tray#       :]
Vial#       :]
Injection Volume :i481-2-p-OMe-rac-0324.lcd
Data File   :0-0.7-90.tcm
Method File :DEFAULT.lsr
Report Format File :2015/03/24 15:00:49
Date Acquired :2015/03/24 15:49:27
Date Processed :2015/03/24 15:49:27

```

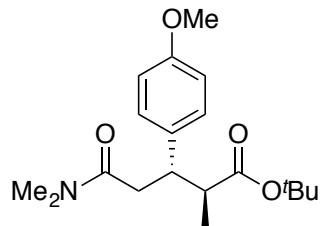


D:\Persona\Files\Sato\i481-2-p-OMe-rac-0324.lcd

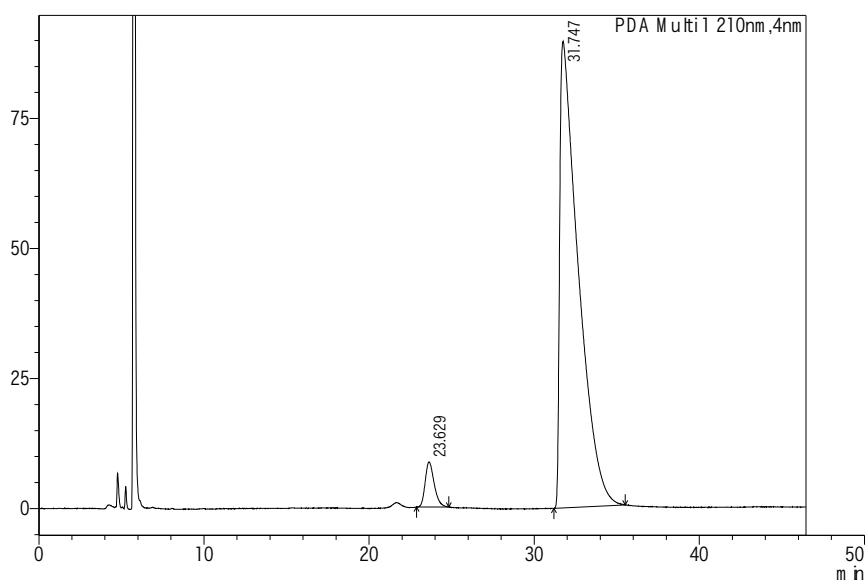
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Acquired by :System Administrator
Sample Name :i503-2-p-O Me-0324
Sample ID   :i503-2-p-O Me-0324
Tray#       :]
Vial#       :]
Injection Volume :i503-2-p-O Me-0324.lcd
Data File   :0-0.7-90.tcm
Method File :DEFAULT.lsr
Report Form at File :2015/03/24 15:51:14
Date Acquired :2015/03/24 16:45:09
Date Processed :2015/03/24 16:45:09

```

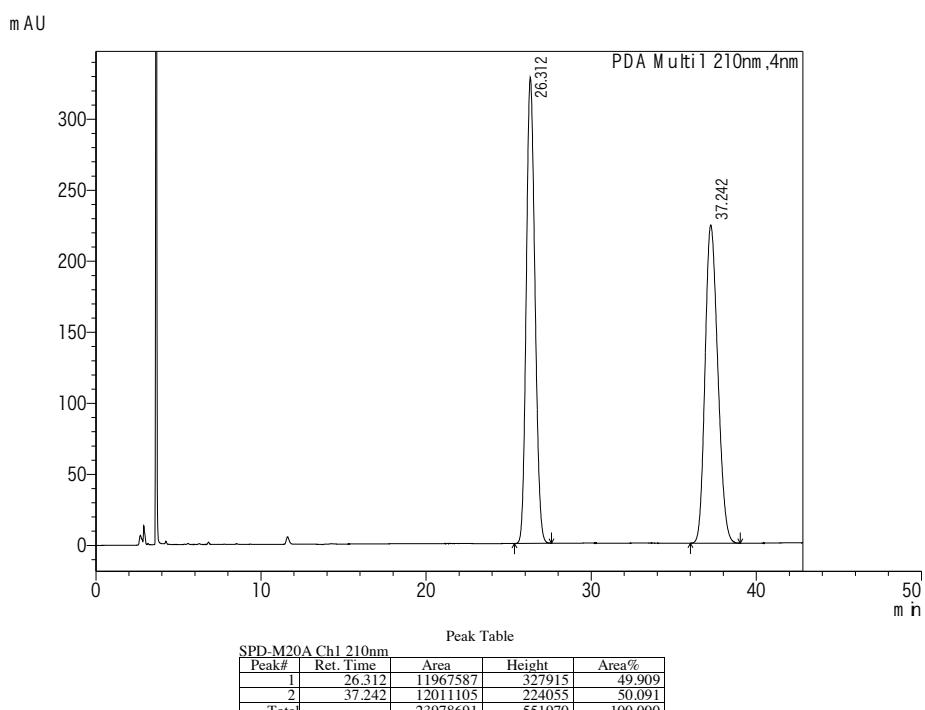
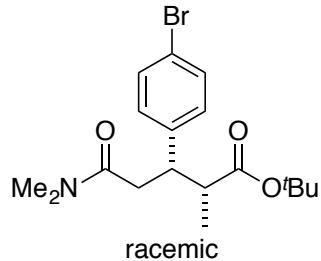


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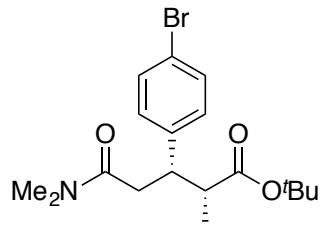
D:\Persona\Files\Sato\i503-2-p-O Me-0324.lcd

Acquired by : System Administrator
 Sample Name : s488-1-p-br-rac-0319
 Sample ID : s488-1-p-br-rac-0319
 Tray# : 1
 Vial# : 1
 Injection Volume : 5488-1-p-br-rac-0319.lcd
 Data File : 0-1.0-60.tdm
 Method File :
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/03/19 11:43:53
 Date Processed : 2015/03/19 12:26:46

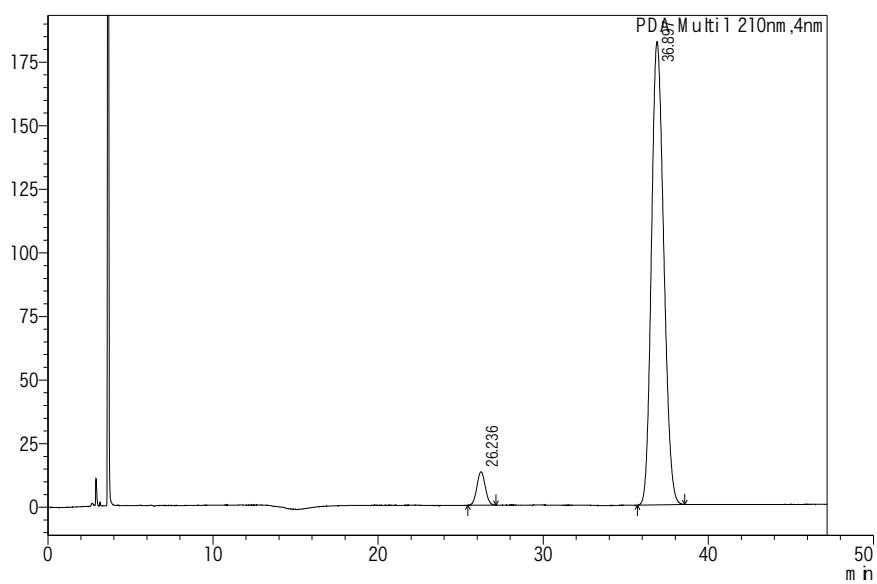


D:\Persona Files\Sato\s488-1-p-br-rac-0319.lcd

Acquired by :System Administrator
 Sample Name :s496-1-p-br-0319
 Sample ID :s496-1-p-br-0319
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :s496-1-p-br-0319.lcd
 Method File :O-1.0-60.lcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/03/19 12:28:31
 Date Processed :2015/03/19 14:30:57



m AU

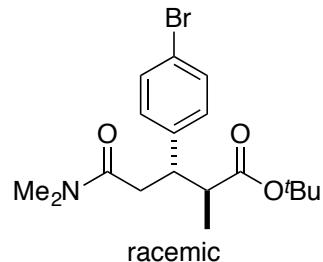


D:\Personal\Files\Sato\s496-1-p-br-0319.lcd

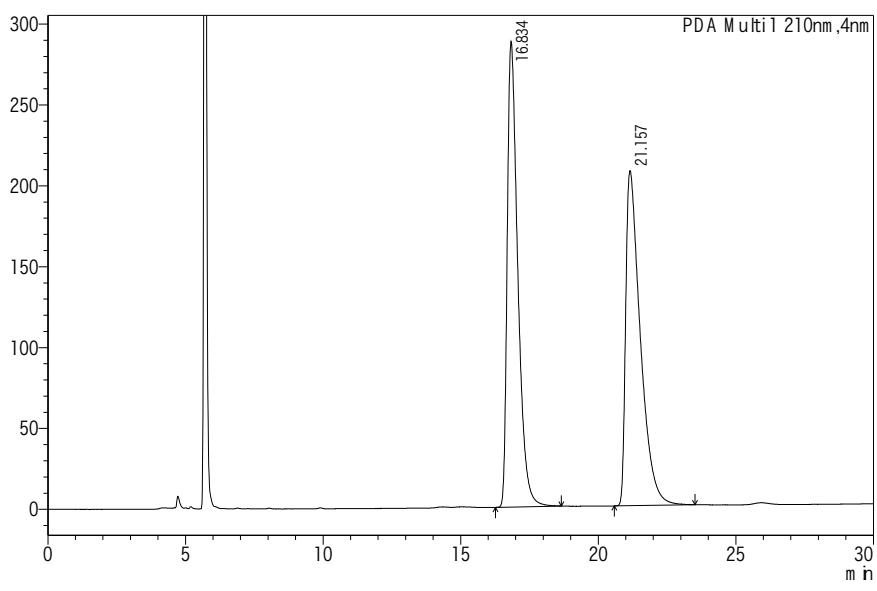
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Acquired by :System Administrator
Sample Name :i488-2-p-br-0319
Sample ID   :i488-2-p-br-0319
Tray#       :]
Vial#       :]
Injection Volume :i488-2-p-br-0319.lcd
Data File   :0-0.7-90.lcd
Method File :DEFAULT.lsr
Report Form at File :2015/03/19 18:46:04
Date Acquired :2015/03/19 19:31:41
Date Processed

```



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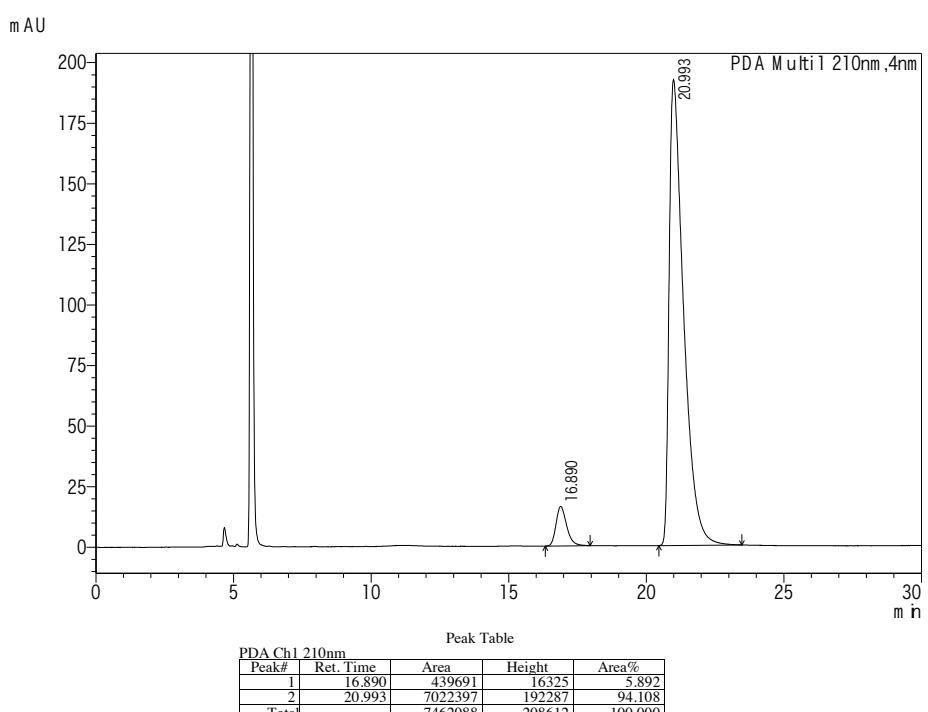
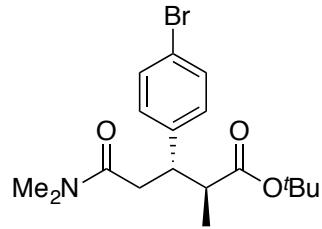


D:\Personal\Files\Sato\i488-2-p-br-0319.lcd

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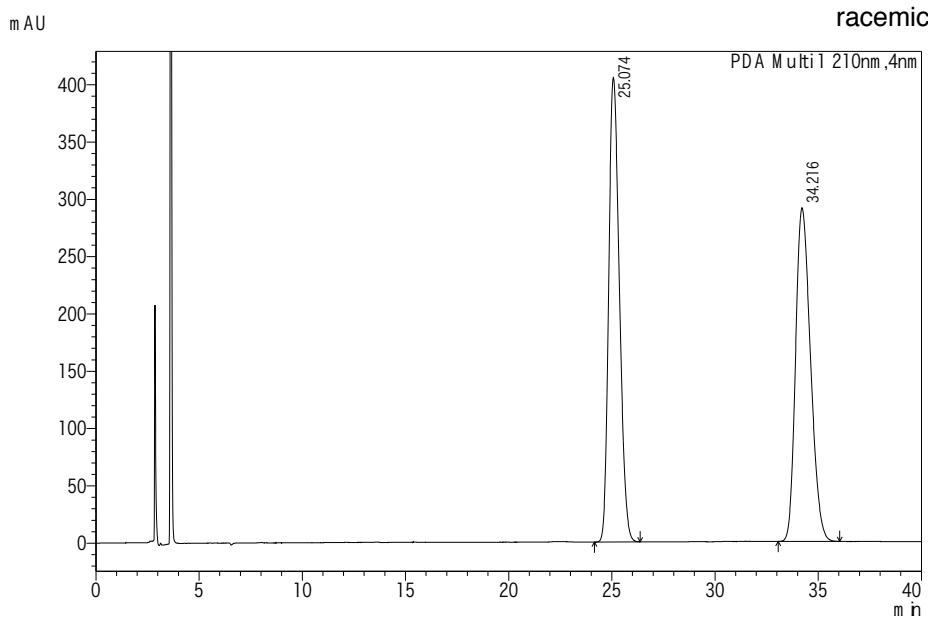
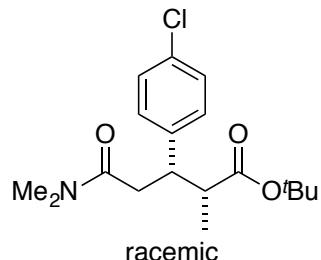
Acquired by :System Administrator
Sample Name :i496-2-p-br-0319
Sample ID   :i496-2-p-br-0319
Tray#       :]
Vial#       :]
Injection Volume :1
Data File   :i496-2-p-br-0319.lcd
Method File :0-0.7-90.lcm
Batch File  :]
Report Form at File :DEFAULT.lsr
Date Acquired :2015/03/19 19:33:31
Date Processed:2015/03/19 20:33:47

```



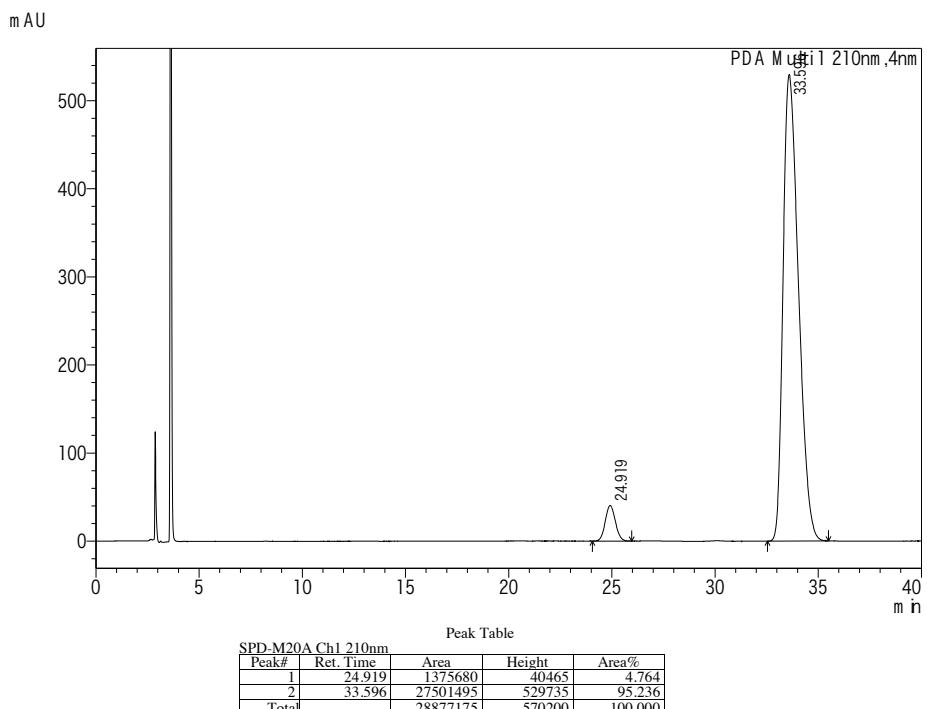
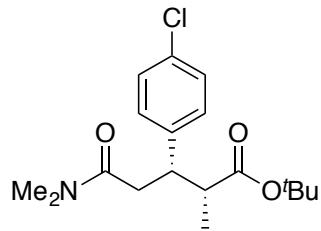
D:\Personal\Files\Sato\i496-2-p-br-0319.lcd

Acquired by :System Administrator
 Sample Name :is841-1-rac-0205
 Sample ID :is841-1-rac-0205
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is841-1-rac-0205.lcd
 Method File :O-1.0-60.tcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2016/02/05 11:48:03
 Date Processed :2016/02/05 16:19:03



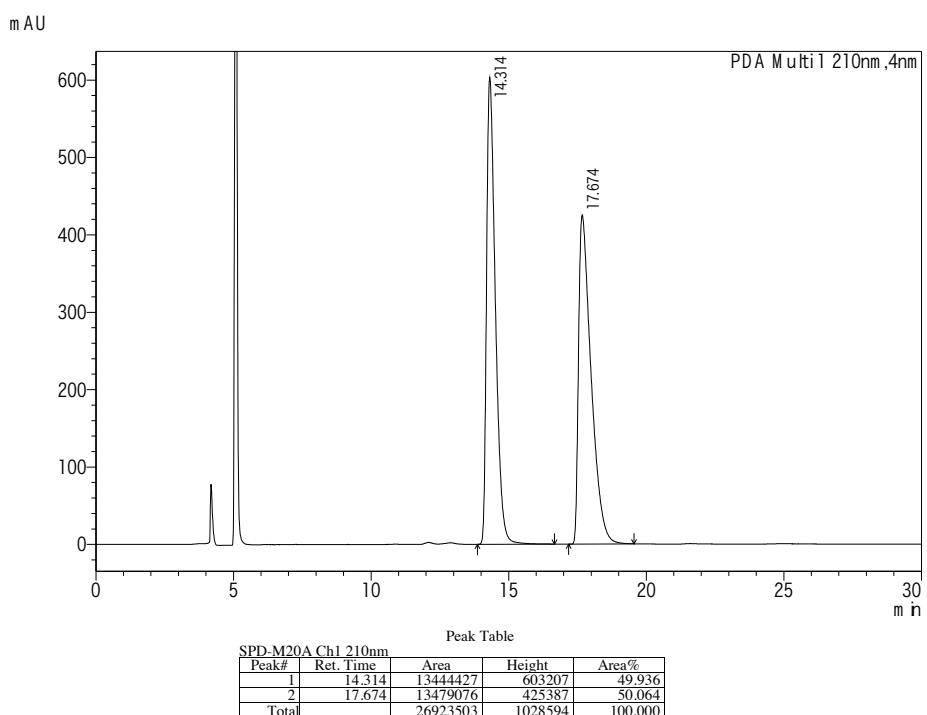
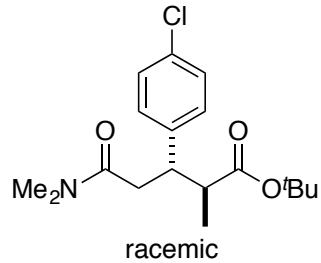
D:\Persona\Files\Sato\is841-1-rac-0205.lcd

Acquired by : System Administrator
 Sample Name : is842-1-0205
 Sample ID : is842-1-0205
 Tray# : 1
 Vial# : 1
 Injection Volume :
 Data File : is842-1-0205.lcd
 Method File : O-1.0-60.tcm
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2016/02/05 12:45:35
 Date Processed : 2016/02/05 17:35:46



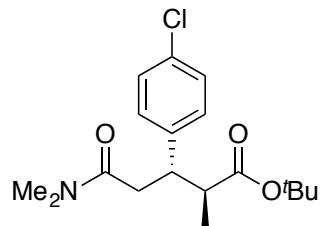
D:\Persona\Files\Sato\is842-1-0205.lcd

Acquired by : System Administrator
 Sample Name : is841-2-rac-0205
 Sample ID : is841-2-rac-0205
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.0000000000000001
 Data File : is841-2-rac-0205.lcd
 Method File : O-0.7-90.lcm
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2016/02/05 16:15:57
 Date Processed : 2016/02/05 17:36:18

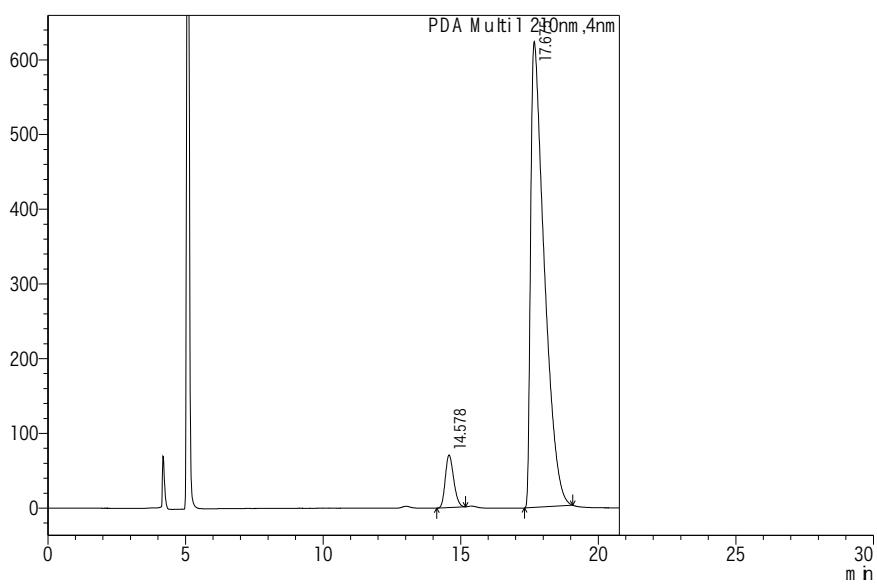


D:\Persona\Files\Save\is841-2-rac-0205.lcd

Acquired by :System Administrator
 Sample Name :is842-2-0205
 Sample ID :is842-2-0205
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is842-2-0205.lcd
 Method File :0-0.7-90.lcd
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2016/02/05 17:14:54
 Date Processed :2016/02/05 17:38:17

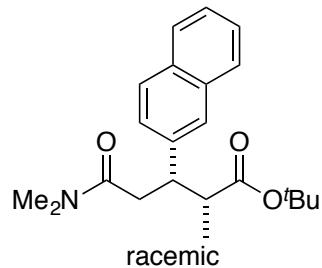


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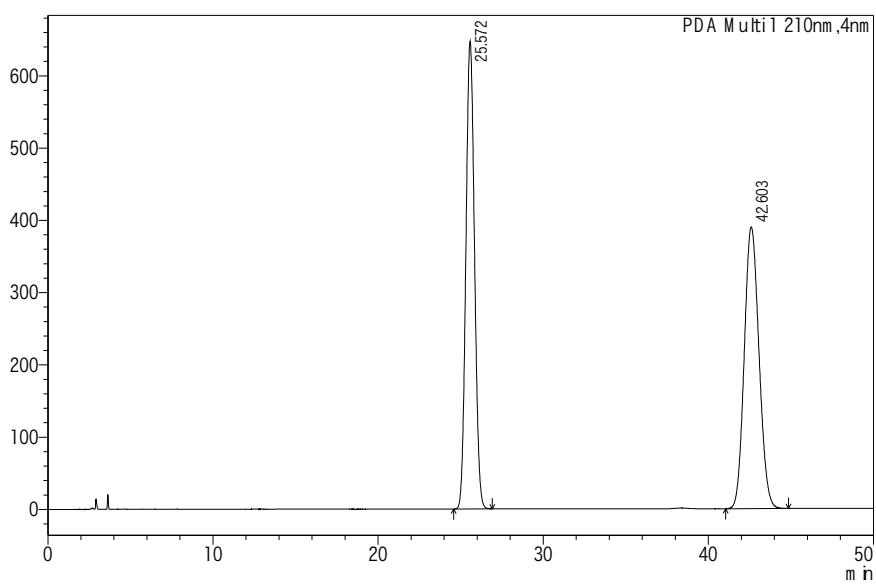


D:\Persona Files\Sato\is842-2-0205.lcd

Acquired by : System Administrator
 Sample Name : s745-1-rac-1105
 Sample ID : s745-1-rac-1105
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.0000000000000001
 Data File : s745-1-rac-1105.lcd
 Method File : O-1.0-60.tcm
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/11/05 14:03:04
 Date Processed : 2015/11/05 16:18:55

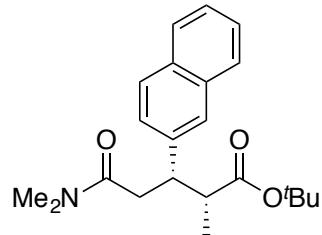


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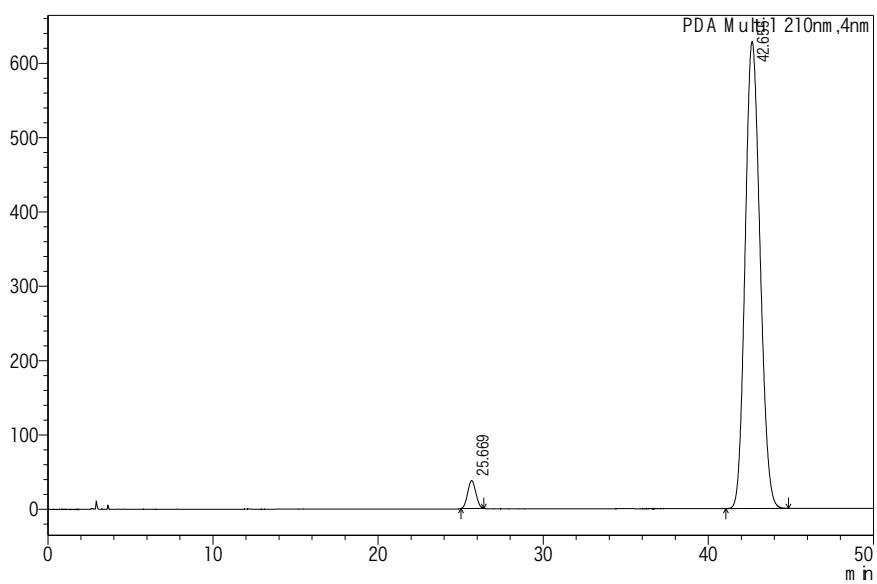


D:\Persona\Files\Sato\s745-1-rac-1105.lcd

Acquired by : System Administrator
 Sample Name : is758-1-1105
 Sample ID : is758-1-1105
 Tray# : 1
 Vial# : -1
 Injection Volume :
 Data File : is758-1-1105.lcd
 Method File : O-1.0-60.lcm
 Batch File :
 Report Format File : for paper.lsr
 Date Acquired : 2015/11/05 15:11:21
 Date Processed : 2016/05/10 13:17:02

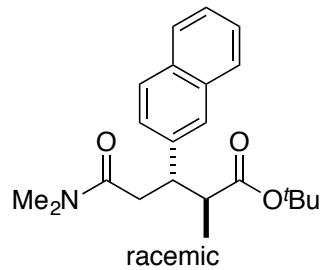


m AU

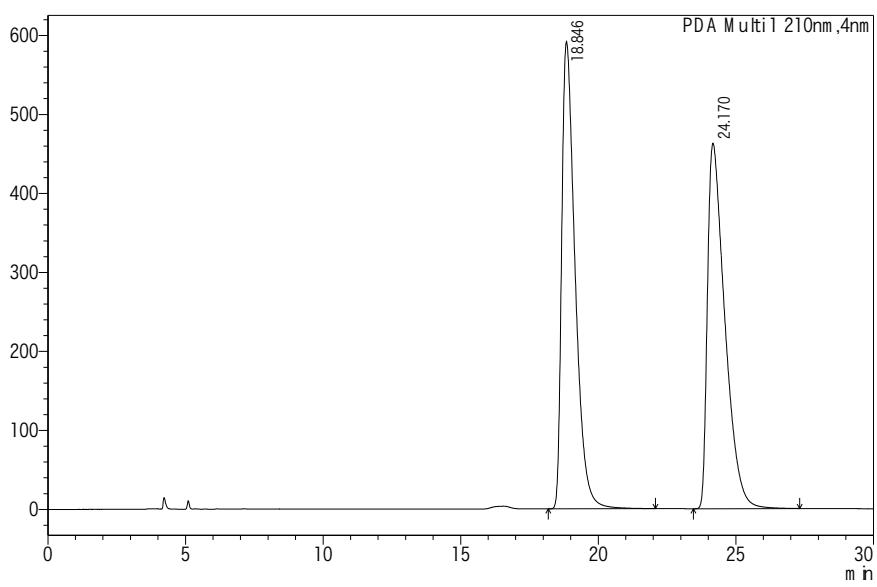


D:\Persona\Files\Sato\is758-1-1105.lcd

Acquired by :System Administrator
 Sample Name :is745-2-rac-1110
 Sample ID :is745-2-rac-1110
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is745-2-rac-1110.lcd
 Method File :0-0.7-90.lcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/11/10 10:37:38
 Date Processed :2015/11/10 12:37:35

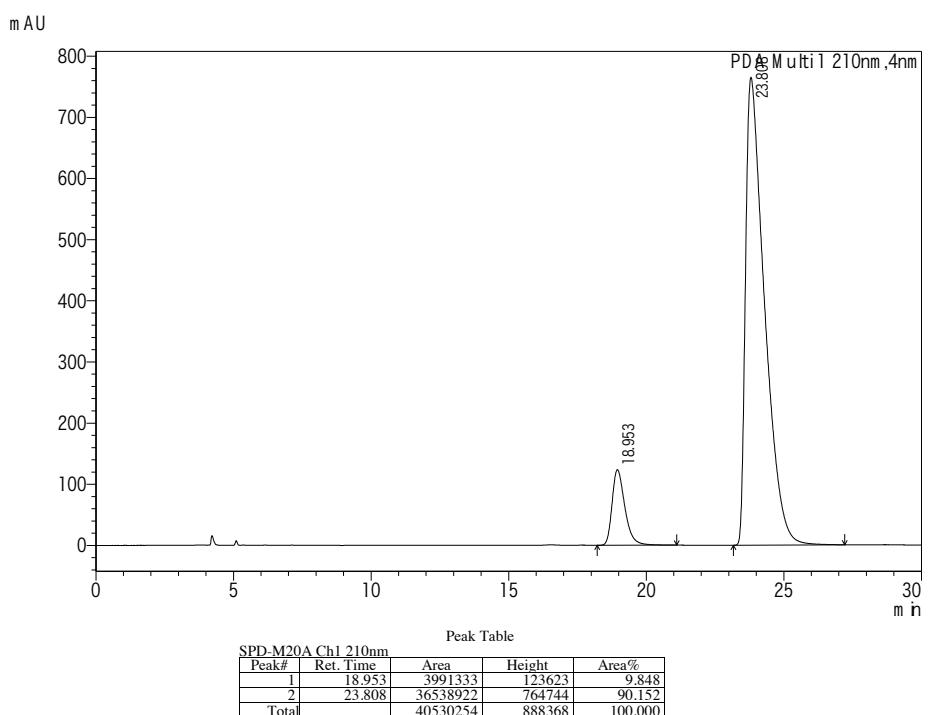
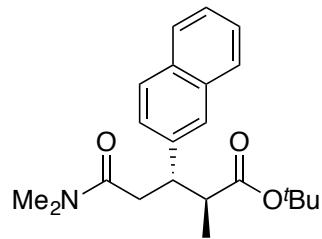


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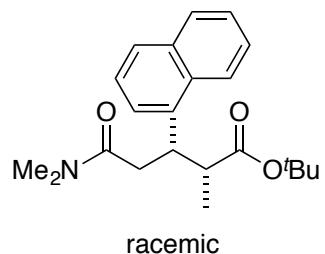


D:\Persona\Files\Sato\is745-2-rac-1110.lcd

Acquired by : System Administrator
 Sample Name : is758-2-1110
 Sample ID : is758-2-1110
 Tray# : 1
 Vial# : -1
 Injection Volume :
 Data File : is758-2-1110.lcd
 Method File : O-0.7-90.km
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/11/10 11:37:15
 Date Processed : 2015/11/10 12:37:28



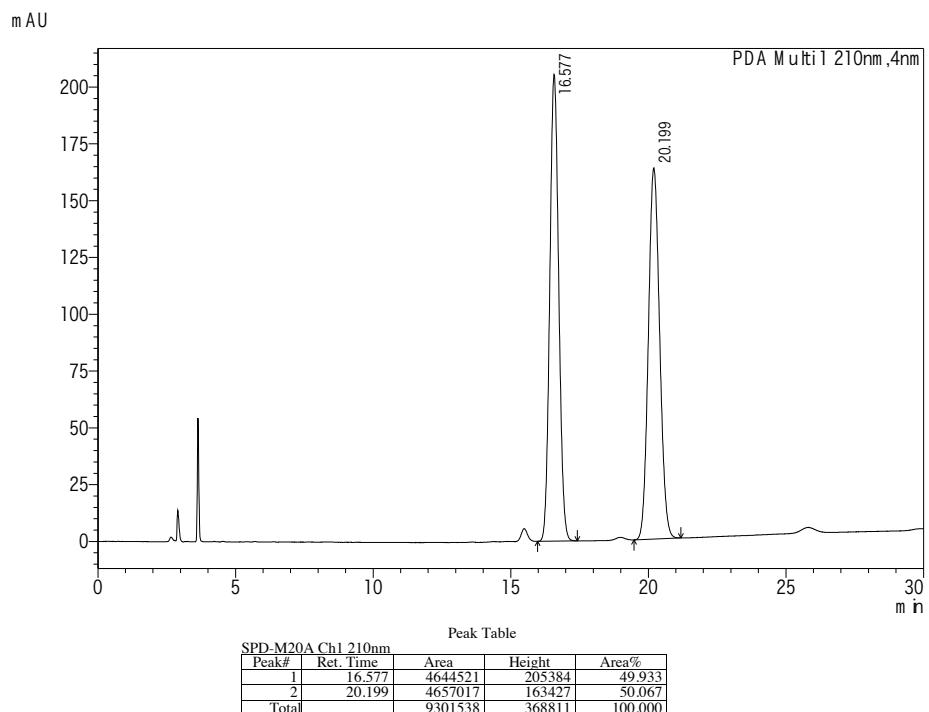
D:\Persona\Files\Sato\is758-2-1110.lcd



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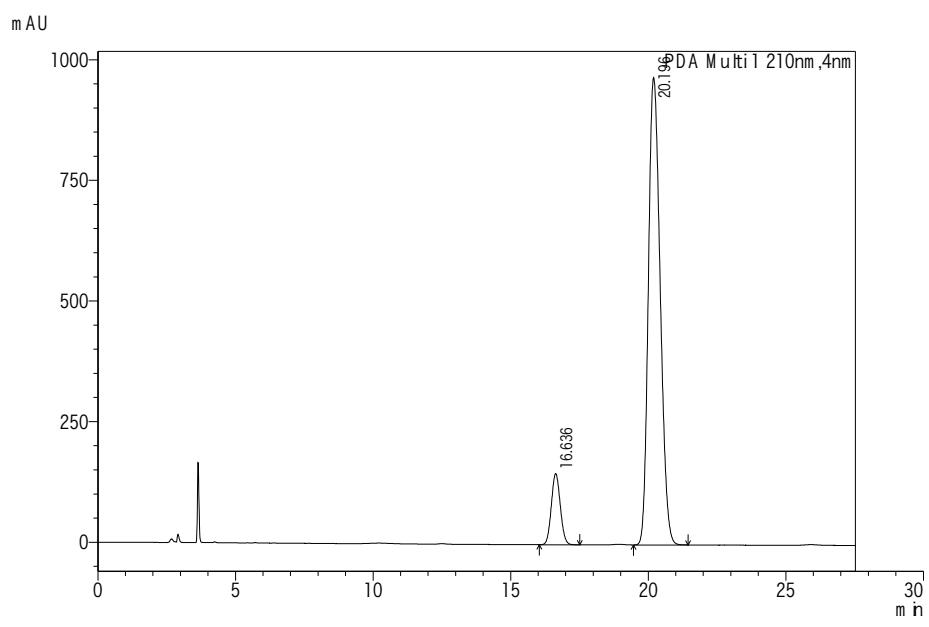
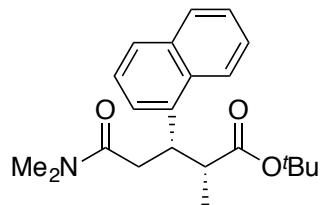
Acquired by :System Administrator
Sample Name :s744-1-rac-1110
Sample ID   :s744-1-rac-1110
Tray#       :1
Vial#       :1
Injection Volume :1
Data File   :s744-1-rac-1110.bdf
Method File :0-1.0-60.km
Batch File  :
Report Format File :DEFAULT.rpr
Date Acquired :2015/11/10 17:05:22
Date Processed :2015/11/10 17:37:57

```



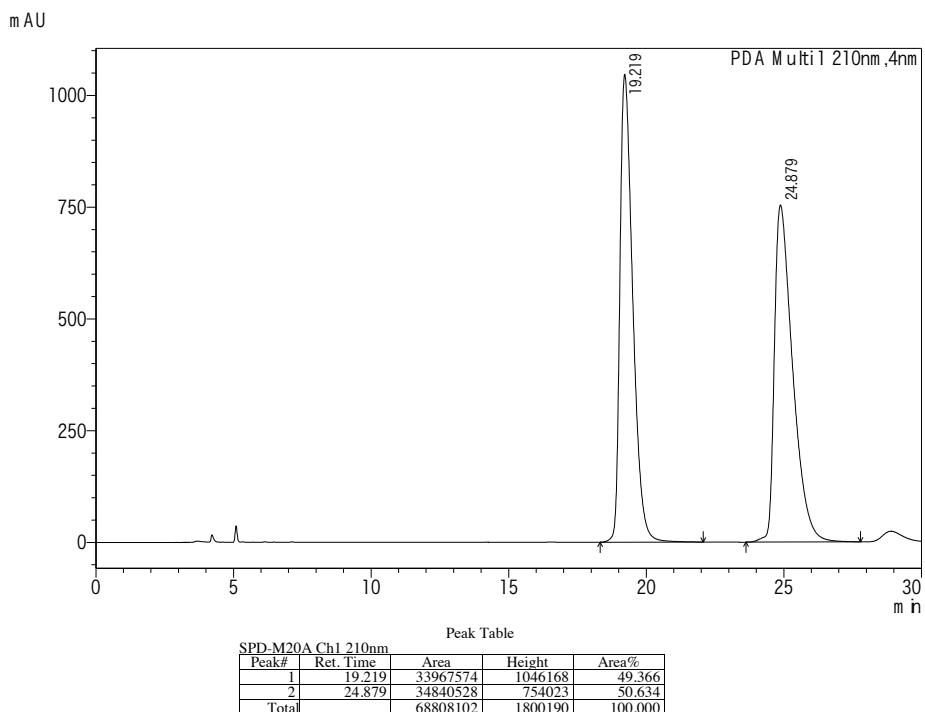
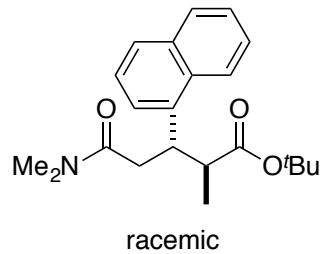
D:\Persona\Files\Sato\s744-1-rac-1110.bdf

Acquired by :System Administrator
 Sample Name :is759-1-1110
 Sample ID :is759-1-1110
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is759-1-1110.lcd
 Method File :O-1.0-60.km
 Batch File :
 Report Format File :DEFAULT.ls
 Date Acquired :2015/11/10 17:38:58
 Date Processed :2015/11/10 18:07:57



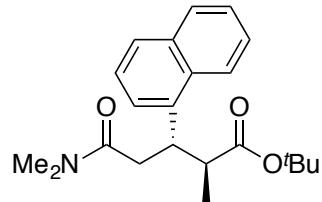
D:\Persona\Files\Sato\is759-1-1110.lcd

Acquired by : System Administrator
 Sample Name : s744-2-rac-1110
 Sample ID : s744-2-rac-1110
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.000000
 Data File : s744-2-rac-1110.lcd
 Method File : O-0.7-90.lcm
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/11/10 12:34:06
 Date Processed : 2015/11/10 16:23:02

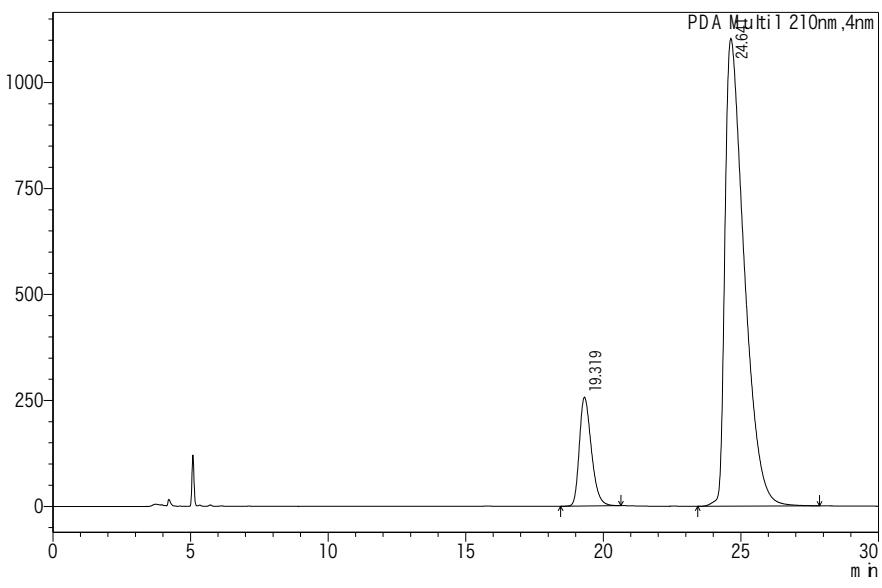


D:\Persona\Files\Sato\s744-2-rac-1110.lcd

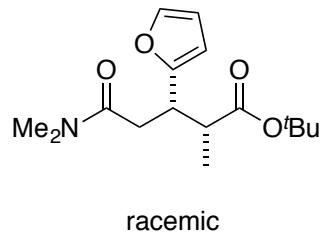
Acquired by : System Administrator
 Sample Name : is759-2-1110
 Sample ID : is759-2-1110
 Tray# : 1
 Vial# : -1
 Injection Volume :
 Data File : is759-2-1110.bdf
 Method File : 0-0.7-90.km
 Batch File :
 Report Format File : DEFAULT.rpr
 Date Acquired : 2015/11/10 13:45:37
 Date Processed : 2015/11/10 16:22:56



mAU

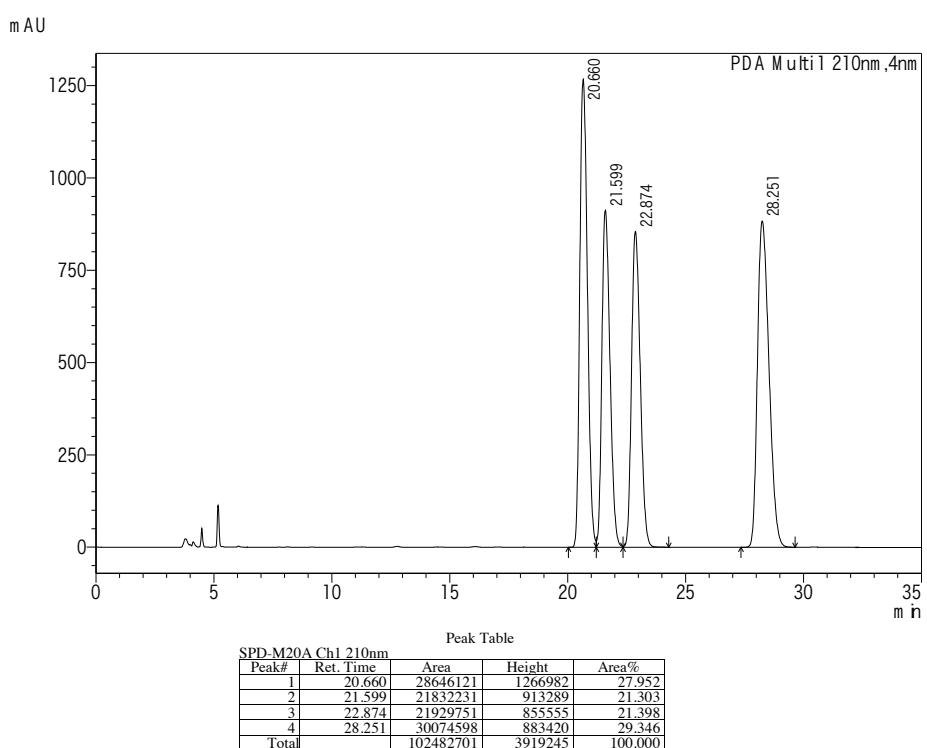


D:\Persona\Files\Sato\is759-2-1110.bdf

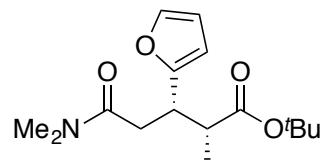


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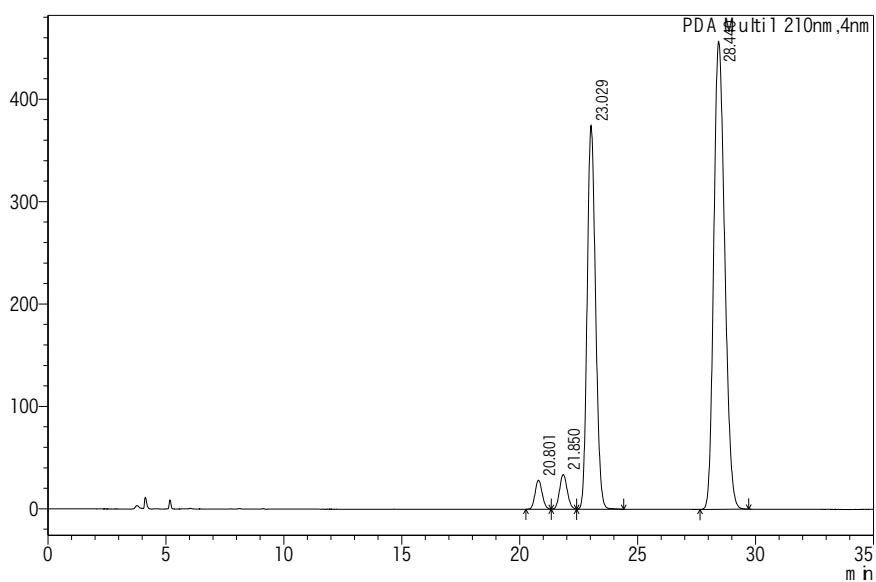
Acquired by :System Administrator
Sample Name :is756-rac-1110
Sample ID   :is756-rac-1110
Tray#       :1
Vial#       :1
Injection Volume :5756-rac-1110.tcd
Data File   :0-0.7-90.tmd
Method File :DEFAULT.lsr
Batch File  :
Report Format File :2015/11/10 18:59:17
Date Acquired :2015/11/10 21:49:41
Date Processed :2015/11/10 21:49:41
  
```



Acquired by : System Administrator
 Sample Name : s757-1110
 Sample ID : s757-1110
 Tray# : 1
 Vial# : 1
 Injection Volume :
 Data File : s757-1110.lcd
 Method File : 0-0.7-90.km
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/11/10 20:11:05
 Date Processed : 2015/11/10 21:49:38



mAU

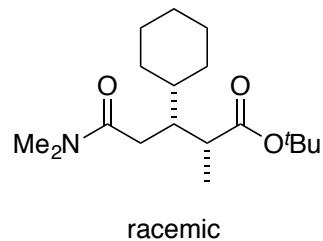


Peak Table

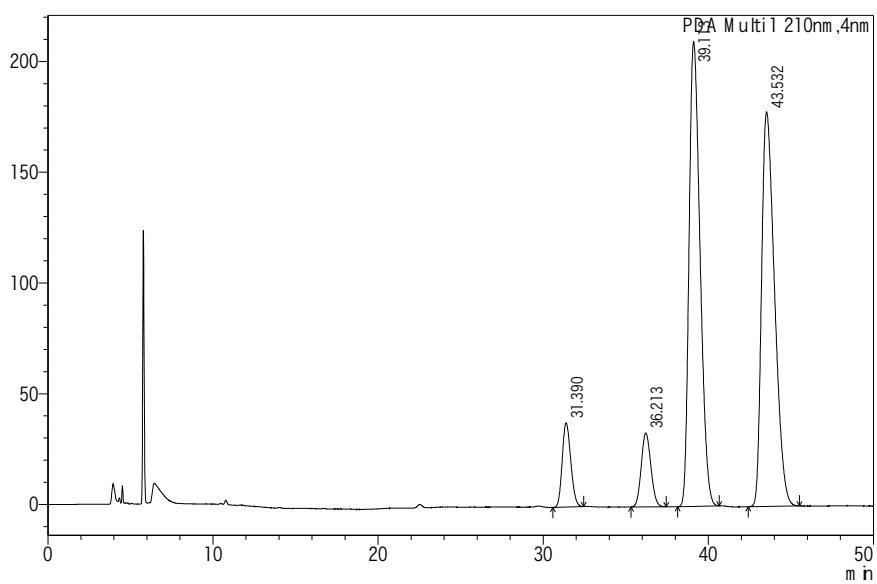
SPD-M20A Ch1 210nm				
Peak#	Ret. Time	Area	Height	Area%
1	20.801	599649	28.384	2.356
2	21.850	773311	33940	3.039
3	23.029	9276552	374718	36.454
4	28.440	14797540	456886	58.150
Total		25447052	893927	100.000

D:\Persona\Files\Sato\s757-1110.lcd

Acquired by : System Administrator
 Sample Name : iS482-cyHex-rac-0324-2nd
 Sample ID : iS482-cyHex-rac-0324-2nd
 Tray# : 1
 Vial# : 1
 Injection Volume : 0.7-180.0mL
 Method File : 0-0.7-180.km
 Batch File :
 Report Format File : DEFAULT.lsr
 Date Acquired : 2015/03/24 16:35:39
 Date Processed : 2015/03/24 20:16:12

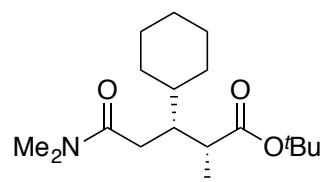


m AU

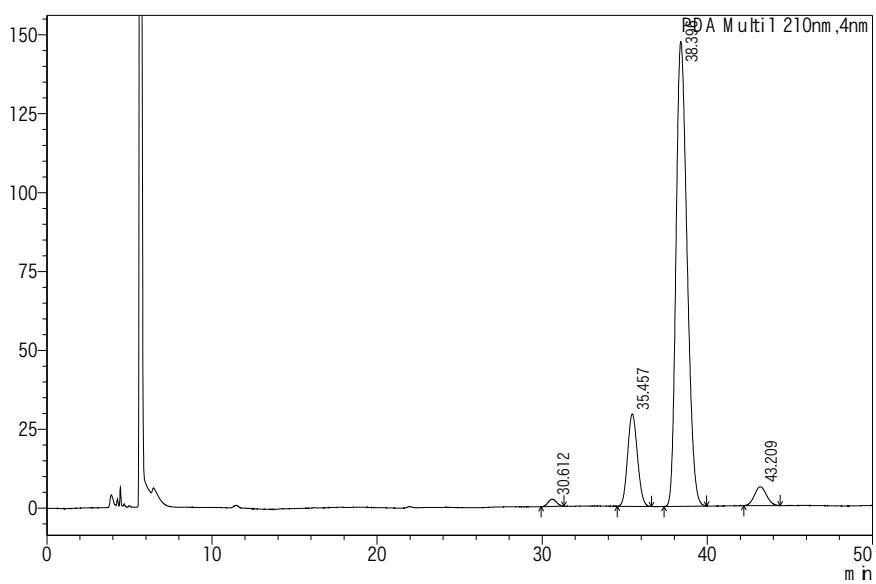


D:\Persona\Files\Sato\iS482-cyHex-rac-0324-2nd.lcd

Acquired by : System Administrator
 Sample Name : i506-cyHex-0324
 Sample ID : i506-cyHex-0324
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.0000000000000001
 Data File : i506-cyHex-0324.lcd
 Method File : O-0.7-180.km
 Batch File :
 Report Format File : DEFAULT.rpr
 Date Acquired : 2015/03/24 19:12:01
 Date Processed : 2015/03/24 20:16:07

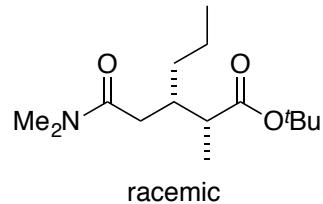


m AU

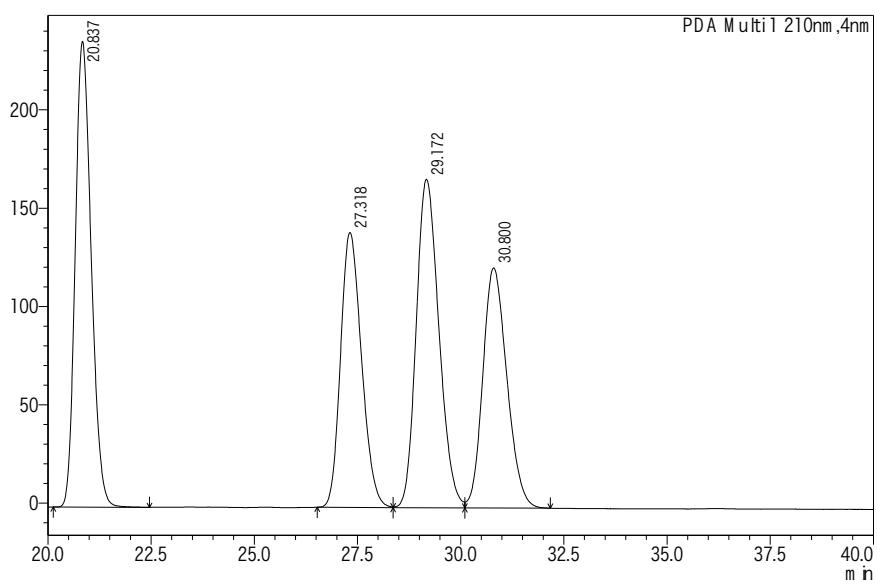


D:\Personal\Files\Sato\i506-cyHex-0324.lcd

Acquired by : System Administrator
 Sample Name : is769-rac-1218
 Sample ID : is769-rac-1218
 Tray# :
 Vial# :
 Injection Volume :
 Data File : is769-rac-1218.tdf
 Method File : 025-200.tcm
 Batch File :
 Report Format File : for paper.lsr
 Date Acquired : 2015/12/18 11:40:20
 Date Processed : 2016/05/10 13:38:22

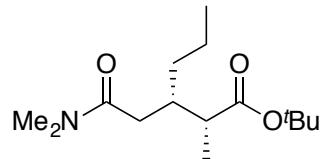


m AU

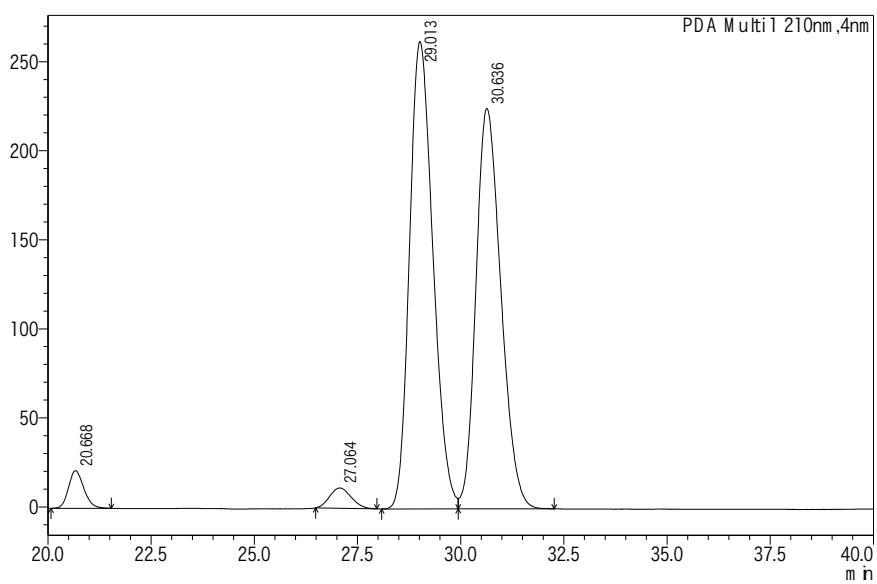


D:\Persona\Files\Sato\is769-rac-1218.tdf

Acquired by : System Administrator
 Sample Name : s773-1218
 Sample ID : s773-1218
 Tray# :
 Vial# :
 Injection Volume :
 Data File : s773-1218.lcd
 Method File : 0.25-200.cm
 Batch File :
 Report Format File : for paper.lsr
 Date Acquired : 2015/12/18 12:36:30
 Date Processed : 2016/05/28 18:51:23

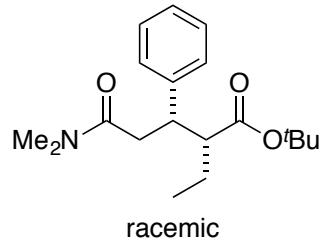


mAU

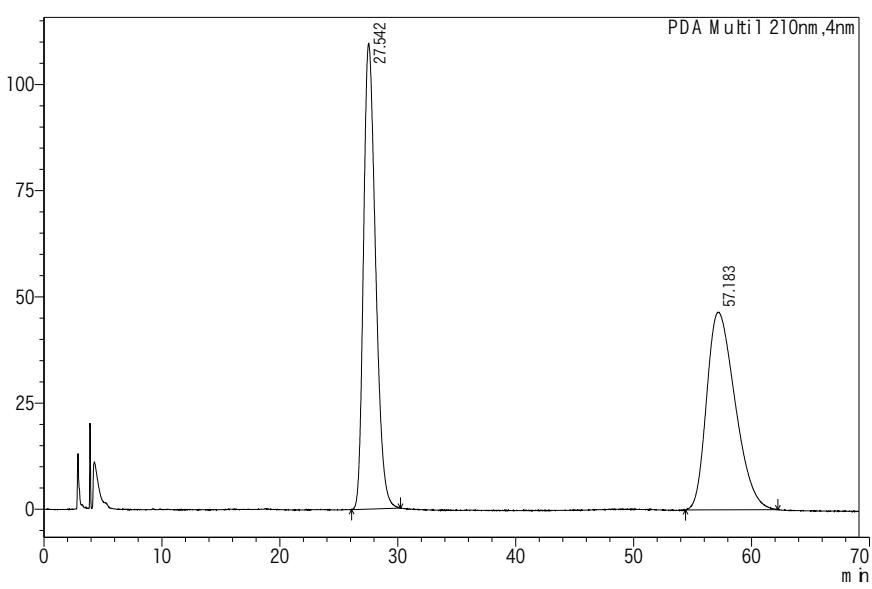


D:\Persona\Files\Sato\s773-1218.lcd

Acquired by : System Administrator
 Sample Name : s740-1-rac-1104
 Sample ID : s740-1-rac-1104
 Tray# : 1
 Vial# : 1
 Injection Volume : 1.0000000000000001
 Data File : s740-1-rac-1104.lcd
 Method File : O-1.0-60.tcm
 Batch File :
 Report Format File : DEFAULT.rfr
 Date Acquired : 2015/11/04 12:08:59
 Date Processed : 2015/11/04 19:59:34

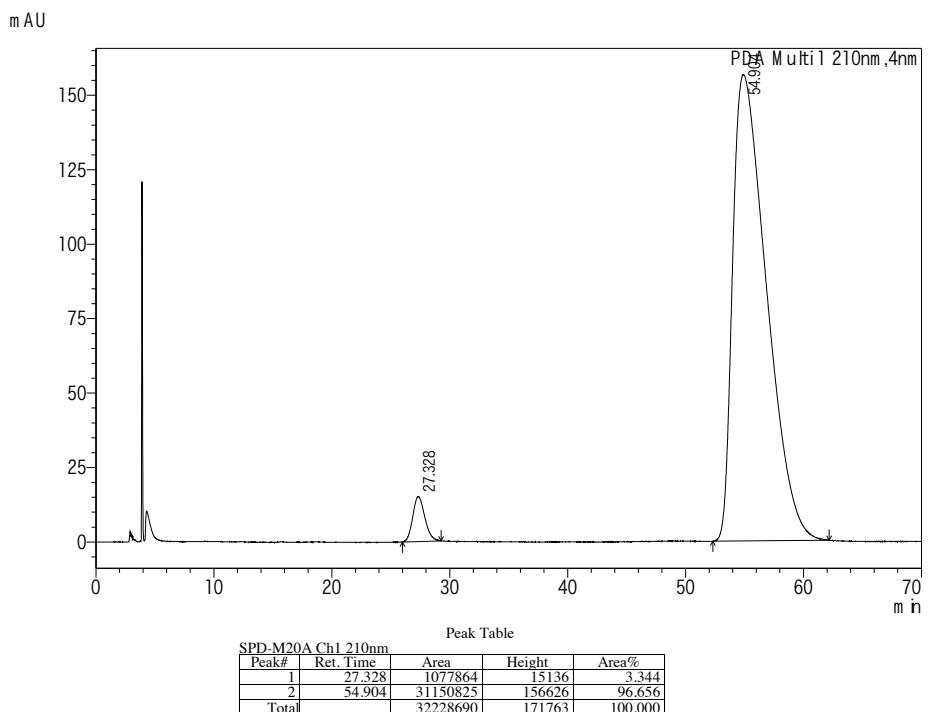
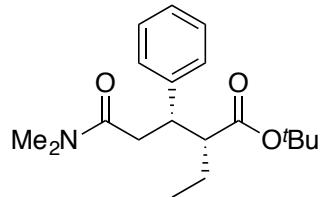


mAU

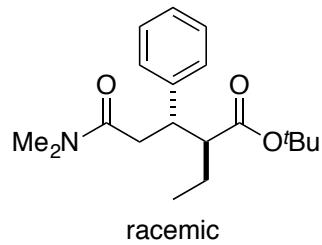


D:\Persona\Files\Sato\s740-1-rac-1104.lcd

Acquired by :System Administrator
 Sample Name :is748-1-1104
 Sample ID :is748-1-1104
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :is748-1-1104.lcd
 Method File :O-1.0-60.lcm
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/11/04 13:20:47
 Date Processed :2015/11/04 14:33:11



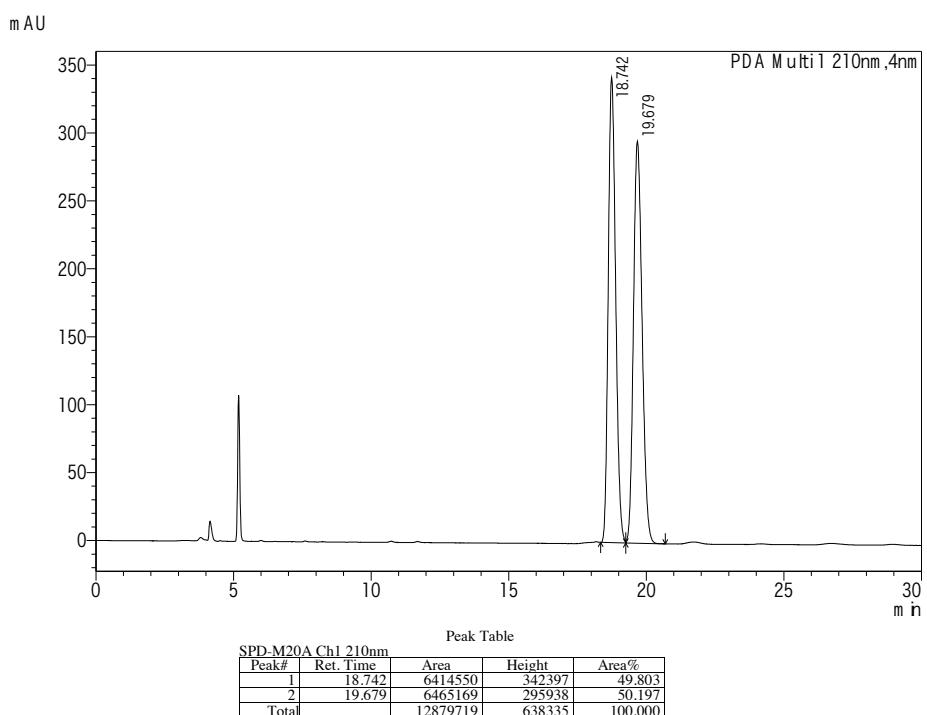
D:\Persona Files\Sato\is748-1-1104.lcd



```

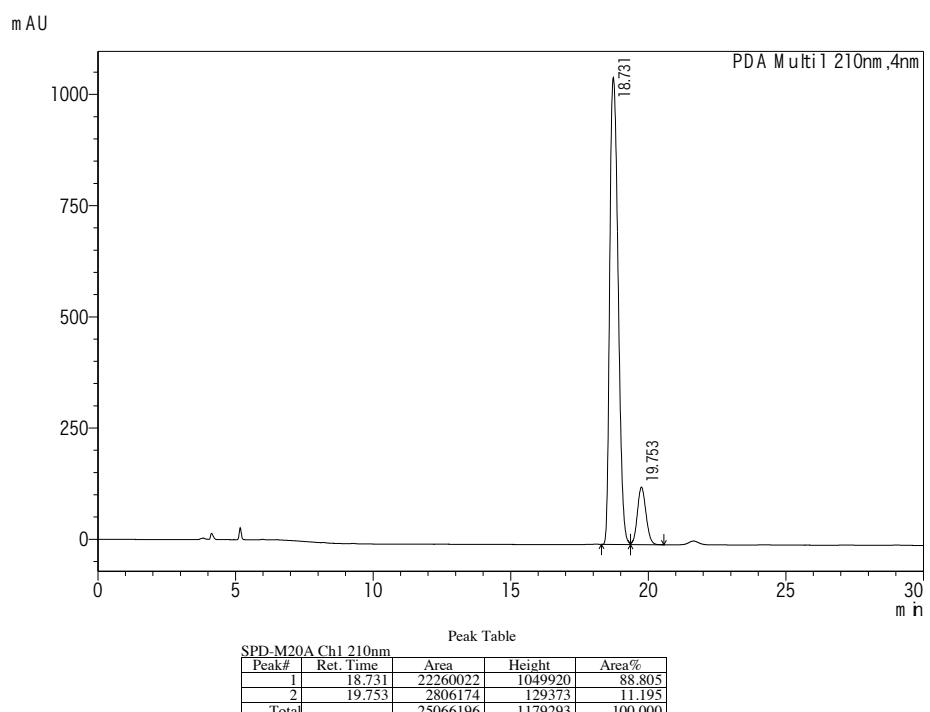
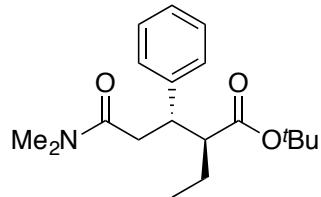
Acquired by :System Administrator
Sample Name :i740-2-1030-2nd
Sample ID   :i740-2-1030-2nd
Tray#       :1
Vial#       :1
Injection Volume :i740-2-1030-2nd.lcd
Data File   :0-0.7-90.lcd
Method File :DEFAULT.lsr
Report Form at File :2015/10/30 12:31:07
Date Acquired :2015/10/30 14:20:04
Date Processed :2015/10/30 14:20:04

```



D:\Persona\Files\Sato\i740-2-1030-2nd.lcd

Acquired by :System Administrator
 Sample Name :S748-2-1030-2nd
 Sample ID :S748-2-1030-2nd
 Tray# :1
 Vial# :1
 Injection Volume :
 Data File :S748-2-1030-2nd.lcd
 Method File :O-0.7-90.lcd
 Batch File :
 Report Format File :DEFAULT.lsr
 Date Acquired :2015/10/30 11:57:22
 Date Processed :2015/10/30 14:19:58



D:\Persona\Files\Sato\S748-2-1030-2nd.lcd