Pot and time economies in the total synthesis of Corey lactone

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SUPPORTING INFORMATION

Experimental procedures and Characterization data

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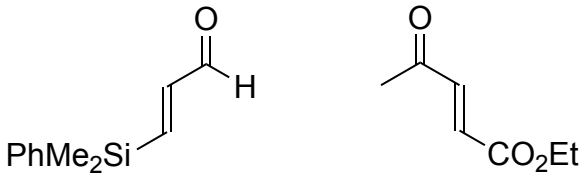
1. Materials and Methods

General Methods.

General Remarks: All reactions were carried out under argon atmosphere and monitored by thin-layer chromatography using Merck 60 F254 precoated silica gel plates (0.25 mm thickness). Specific optical rotations were measured using a JASCO P-1020 polarimeter and a JASCO DIP-370 polarimeter. FT-IR spectra were recorded on a JASCO FT/IR-410 spectrometer and a Perkin Elmer spectrum BX FT-IP spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on an Agilent-400 MR (400 MHz for 1H NMR, 100 M Hz for ^{13}C NMR) instrument. Data for 1H NMR are reported as chemical shift (δ ppm), integration multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, ddd = doublet of doublet of doublets, dt = double of triplets, m = multiplet), coupling constant (Hz), Data for 13C NMR are reported as chemical shift. High resolution ESI-TOF mass spectra were measured by Themo Orbi-trap instrument. HPLC analysis was performed on a HITACHI Elite LaChrom Series HPLC, UV detection monitored at appropriate wavelength respectively, using Chiralpak ID (0.46 cm × 25 cm) and Chiralpak IF (0.46 cm × 25 cm). Melting-point apparatus was Yanaco MP-J3.

Materials.

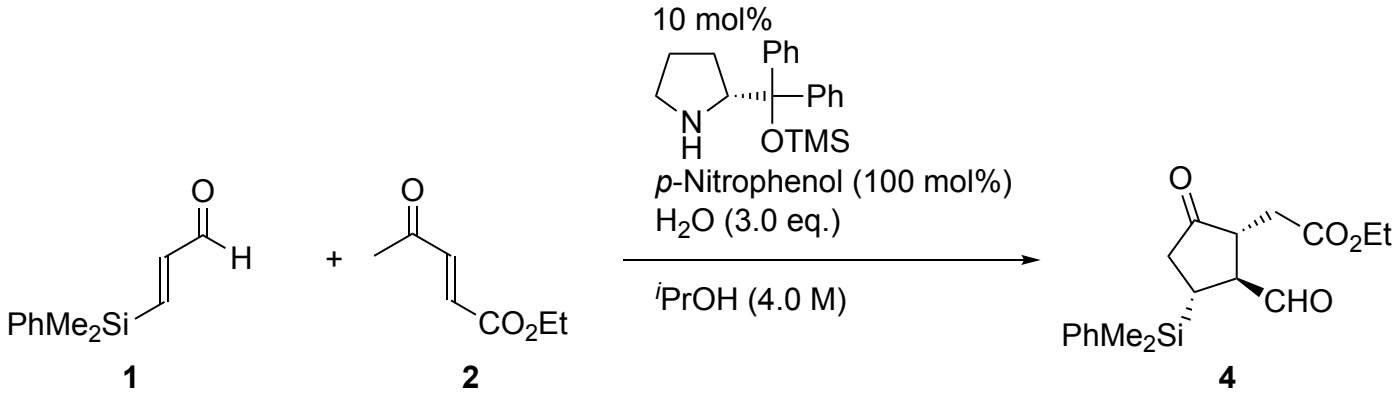


1 **2**

The aldehyde [1] **1** (CAS number [11948-92-1]) and ethyl 4-oxo-2-pentenoate [2] **2** (CAS number [10150-93-3]) were commercially available.

2. Experimental Procedures

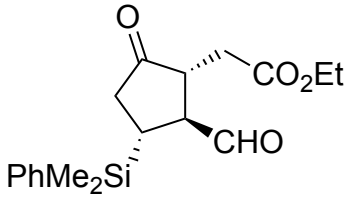
2.1. Formal (3+2) cycloaddition reaction



1 **2** **4**

To a solution of aldehyde **1** (32.4 mg, 0.18 mmol) and ethyl 4-oxo-2-pentenoate **2** (21.3 mg, 0.15 mmol) in ^{i}PrOH (75 μL), H_2O (8.1 μL, 0.45 mmol), diphenylprolinol silyl ether (4.88 mg, 0.015 mmol), *p*-nitrophenol (20.8 mg, 0.15 mmol) were added at room temperature. After stirring the reaction mixture at this temperature for 1 h, the reaction mixture was directly purified by column chromatography on silica gel (^{n}Hexane: EtOAc = 6:1) to give the target compound (42.4 mg, 0.128 mmol) in 85% yield (single isomer).

Ethyl 2-((1*R*,2*S*,3*R*)-3-(dimethyl(phenyl)silyl)-2-formyl-5-oxocyclopentyl)acetate (4)



Yield: 85% (42.4 mg)

Physical State: colorless oil

^{1}H NMR ($CDCl_3$) δ 0.365 (s, 3H), 0.381 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H), 1.73 (ddd, *J* = 8.8, 12.0, 13.2 Hz, 1H), 2.18 (dd, *J* = 13.2, 18.8 Hz, 1H), 2.49 (ddd, *J* = 1.2, 8.8, 18.4 Hz, 1H), 2.53 (dd, *J* = 6.8, 17.6 Hz, 1H), 2.62 (dd, *J* = 4.0, 17.6 Hz, 1H), 2.69 (dddd, *J* = 1.2, 4.0, 6.4, 12.0 Hz, 1H), 2.82 (dt, *J* = 3.6, 12.0 Hz, 1H), 4.06 (dq, *J* = 1.6, 7.2 Hz, 2H), 7.36-7.41 (m, 3H), 7.48-7.51 (m, 2H), 9.43 (d, *J* = 3.2 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 215.5, 201.1, 171.3, 135.6, 133.8, 133.8, 129.8, 128.1, 128.1, 60.9, 55.9, 47.5, 38.7, 32.7, 22.7, 14.0, -4.43, -4.65

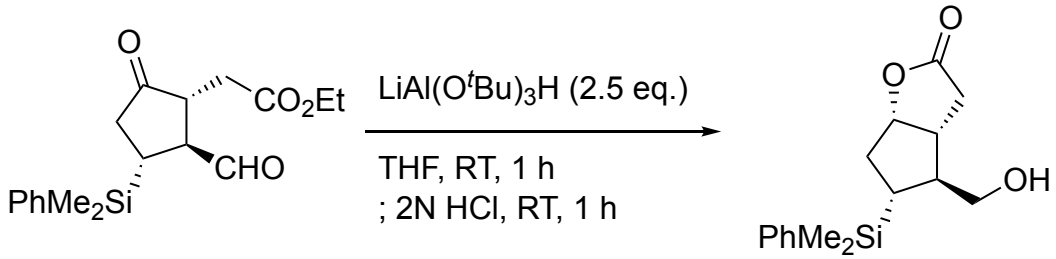
HRMS (ESI): $[M+Na]^+$ calcd for $C_{18}H_{24}O_4SiNa$: 355.1336, found: 355.1340

IR(neat)ν 1725, 1427, 1375, 1253, 1189, 1113, 1026, 835, 818, 775, 736, 701 cm^{-1}

$[\alpha]_D^{26}$ -59.30 (*c* 3.0, $CHCl_3$)

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.50

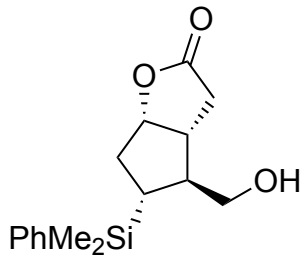
2.2. Reduction & Lactonization



5 **8**

To a solution of aldehyde **5** (33.2 mg, 0.10 mmol) in THF (300 μL), $LiAl(O^tBu)_3H$ (63.5 mg, 0.25 mmol) was added at room temperature. After stirring the reaction mixture at this temperature for 1 hour, 2N HCl (150 μL) was added to the solution at room temperature. After stirring the reaction mixture at this temperature for 1 h, the reaction mixture was quenched by aq. $NaHCO_3$ (5 mL). Upon completion, H_2O (2 mL) was added and the mixture was extracted with EtOAc (3 ×5 mL). The combined organic extracts were washed with aq. $NaHCO_3$ (5 mL) and sat. NaCl solution (5 mL). Then, the combined organic extracts were dried with anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. After concentration in vacuo, the reaction mixture was purified by column chromatography on silica gel (^{n}Hexane: EtOAc = 2:1) to give the target compound in 91% yield (26.4 mg, 0.091 mmol).

(3a*R*,4*S*,5*R*,6a*S*)-5-(Dimethyl(phenyl)silyl)-4-(hydroxymethyl)hexahydro-2H-cyclopenta[b]furan-2-one



Yield: 91% (26.4 mg)

Physical State: colorless oil

^{1}H NMR ($CDCl_3$) δ 0.283 (s, 3H), 0.320 (s, 3H), 1.23 (ddd, *J* = 8.4, 12.0, 19.2 Hz, 1H), 1.63-1.76 (m, 3H), 2.34 (dd, *J* = 7.2, 14.4 Hz, 1H), 2.37 (dd, *J* = 2.0, 15.6 Hz, 1H), 2.59-2.63 (m, 1H), 2.70 (dd, *J* = 9.2, 18.0 Hz, 1H), 3.32 (dd, *J* = 6.4, 11.2 Hz, 1H), 3.53 (dd, *J* = 3.6, 11.2 Hz, 1H), 4.88 (dt, *J* = 4.0, 7.2 Hz, 1H), 7.32-7.37 (m, 3H), 7.46-7.86 (m, 2H)

^{13}C NMR ($CDCl_3$) δ 177.3, 137.6, 133.5, 133.5, 129.4, 128.1, 128.1, 85.6, 63.7, 50.2, 44.5, 35.7, 34.7, 27.6, -3.69, -5.23

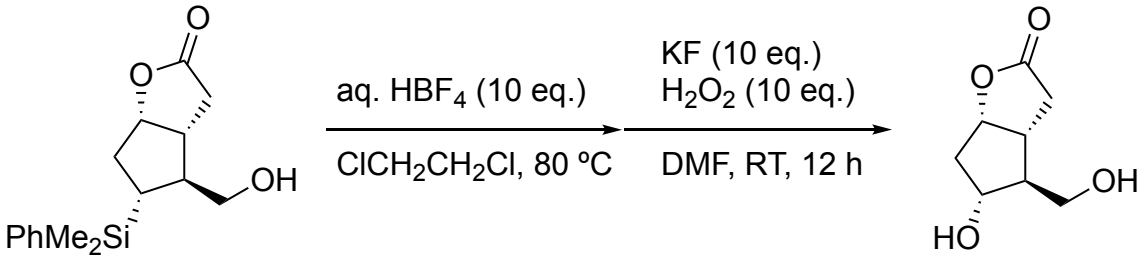
HRMS (ESI): $[M+Na]^+$ calcd for $C_{16}H_{22}O_3SiNa$: 313.1230, found: 313.1235

IR(neat)ν 3441, 3069, 2956, 1748, 1427, 1251, 1171, 1112, 1037, 910, 818, 740, 648 cm^{-1}

$[\alpha]_D^{26}$ -10.05 (*c* 0.90, $CHCl_3$)

R_f (^{n}Hexane: EtOAc = 2:1, color reagent: Hanessian's stain reagent): 0.45

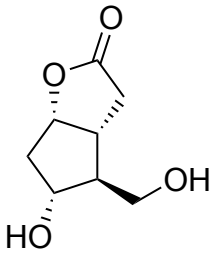
2.3. Tamao-Fleming oxidation



8

To a solution of compound **8** (29.0 mg, 0.10 mmol) in $ClCH_2CH_2Cl$ (300 μL), aq. HBF_4 (160 μL, 1.0 mmol, 47 wt. % in H_2O) was added at 80 ºC. After stirring the reaction mixture at this temperature for 4 h, the reaction mixture was quenched by aq. $NaHCO_3$ (1.0 mL). Upon completion, H_2O (2 mL) was added and the mixture was extracted with EtOAc (3 ×5 mL). The combined organic extracts were washed with aq. $NaHCO_3$ (5 mL) and sat. NaCl solution (5 mL). Then, the combined organic extracts were dried with anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. To a solution of the crude in DMF (300 μL), KF (56 mg, 1.0 mmol) and aq. H_2O_2 (35 μL, 1.0 mmol, 35 wt. % in H_2O) were added at room temperature. After stirring the reaction mixture at this temperature for 12 h, Me_2S (35 μL) was added to the solution at room temperature. After concentration in vacuo, the reaction mixture was directly purified by column chromatography on silica gel ($CHCl_3$: MeOH = 20:1, 10:1) to give the target compound in 86% yield (14.8 mg, 0.086 mmol).

(3a*R*,4*S*,5*R*,6a*S*)-5-Hydroxy-4-(hydroxymethyl)hexahydro-2H-cyclopenta[b]furan-2-one



Yield: 86% (14.8 mg)

Physical State: White solid (m.p. 115~117 ºC)

^{1}H NMR ($CDCl_3$) δ 1.98-2.06 (m, 2H), 2.42 (td, *J* = 6.8, 14.8 Hz, 1H), 2.53 (dd, *J* = 1.2, 18.0 Hz, 1H), 2.62 (dtd, *J* = 2.0, 7.2, 10.4 Hz, 1H), 2.81 (dd, *J* = 10.0, 18.0 Hz, 1H), 3.62 (dd, *J* = 7.2, 10.4 Hz, 1H), 3.74 (dd, *J* = 5.2, 10.4 Hz, 1H), 4.18 (q, *J* = 6.0 Hz, 1H), 4.93 (dt, *J* = 2.8, 6.8, 1H)

^{13}C NMR ($CDCl_3$) δ 177.2, 83.6, 75.4, 63.5, 55.2, 40.6, 39.5, 35.3

HRMS (ESI): $[M+Na]^+$ calcd for $C_8H_{12}O_4Na$: 195.0628, found: 195.0628

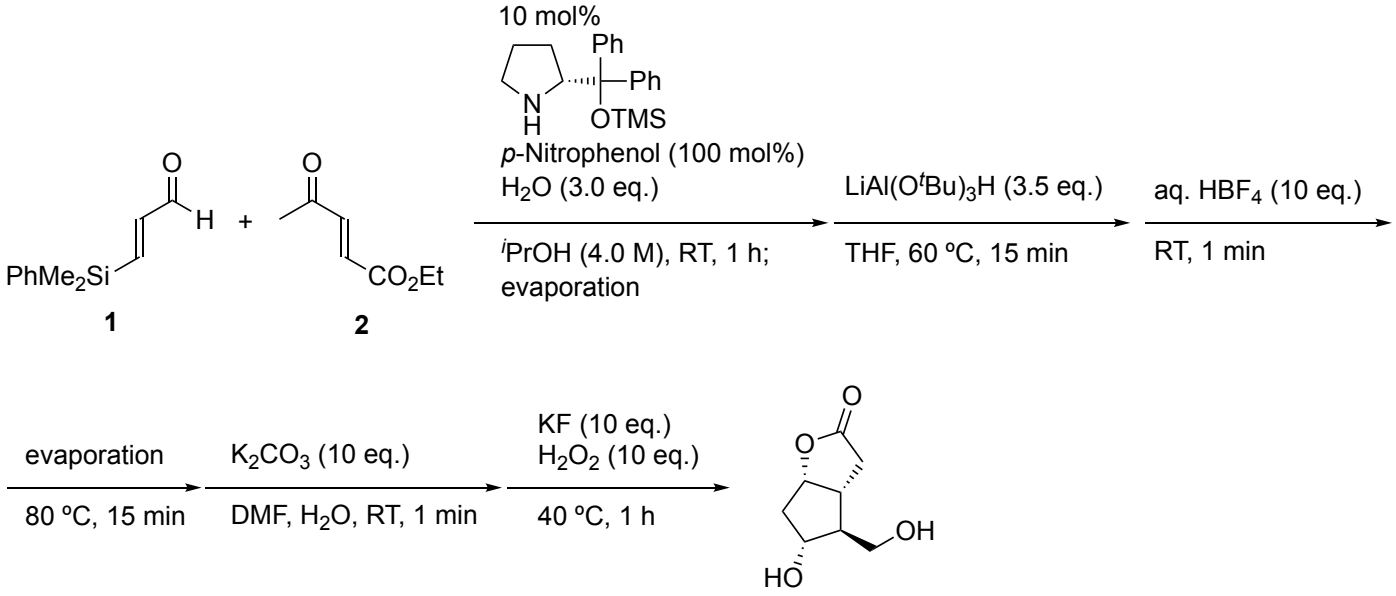
IR(neat)ν 3419, 2925, 1760, 1634, 1336, 1071, 668 cm^{-1}

$[\alpha]_D^{26}$ -41.87 (*c* 0.40, MeOH)

Literature data [3]: $[\alpha]_D$ -43.4 (c 1.12, MeOH)

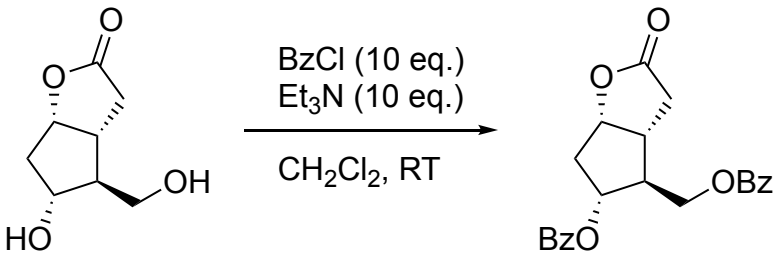
R_f (CH_2Cl_2: MeOH = 9:1, color reagent: Hanessian's stain reagent): 0.45

2.4. One pot procedure of Corey lactone diol



To a solution of 3-(dimethylphenylsilyl)propenal **1** (2.28 g, 12 mmol) and ethyl 4-oxo-2-pentenoate **2** (1.42 g, 10 mmol) in ^{i}PrOH (2.5 ml), H_2O (540 μL, 30 mmol), diphenylprolinol silyl ether (325 mg, 1.0 mmol), *p*-nitrophenol (1.39 g, 10 mmol) were added at room temperature. After stirring the reaction mixture at this temperature for 1 h, the reaction mixture was concentrated in vacuo. After the crude material was dissolved in THF (20 ml), $LiAl(O^{t}Bu)_3H$ (8.89 g, 35 mmol) was added to the solution at 60 ºC. After stirring the reaction mixture at this temperature for 15 min, aq. HBF_4 (10.6 ml, 100 mmol, 47 wt. % in H_2O) was added to the solution at room temperature. After stirring the reaction mixture at this temperature for 1 min, the reaction mixture was the resulting solution was concentrated in vacuo (80 ºC, 15 min). After the crude material was dissolved in DMF-H_2O = (2:1) (30 ml), K_2CO_3 (13.8 g, 100 mmol) was added to the solution at room temperature. Then, KF (5.6 g, 100 mmol) and aq. H_2O_2 (3.5 ml, 100 mmol, 35 wt. % in H_2O) were added at 40 ºC. After stirring the reaction mixture at this temperature for 1 min, Me_2S (2.3 ml) was added to the solution at room temperature. After concentration in vacuo, the reaction mixture was directly purified by column chromatography on silica gel ($CHCl_3$: MeOH = 20:1~ 10:1) to give the target compound in 50% yield (865 mg, 5.0 mmol).

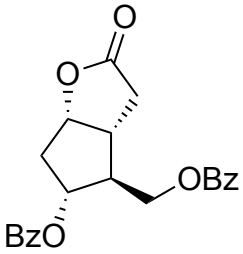
2.5. Dibenzoylation of Corey lactone diol and determination of *ee* value



To a solution of Corey lactone diol (8.1 mg, 0.050 mmol) in CH_2Cl_2 (100 μL), Et_3N (69 μL, 0.50 mmol), benzoyl chloride (64 μL, 0.50 mmol), were added at room temperature. After stirring the reaction mixture at this temperature for 30 min, the reaction mixture was quenched by aq. $NaHCO_3$ (1.0 mL). Upon completion, H_2O (2 mL) was added and the mixture was extracted with EtOAc (3 ×5 mL). The combined organic extracts were washed with aq. $NaHCO_3$ (5.0 mL) and sat. NaCl solution (5 mL). Then, the combined organic extracts were dried with anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. After concentration in vacuo, the reaction mixture was purified by column chromatography on silica gel (^{n}Hexane : EtOAc = 3:1) to give the target compound in 80% yield.

The enantiomeric ratio was determined by HPLC using CHIRALPACK IF (^{n}Hexane / ^{i}PrOH = 9:11; flow rate 1.0 ml/min, major isomer t_R = 8.23 min, minor isomer t_R = 8.91 min) (>99% *ee*).

((3a*S*,4*R*,5*S*,6a*R*)-5-(benzoyloxy)-2-oxohexahydro-2H-cyclopenta[b]furan-4-yl)methyl benzoate



Yield: 80% (15.2 mg)

Physical State: White solid (m.p. 115.2~116.5 ºC)

^{1}H NMR ($CDCl_3$) δ 2.40 (td, *J* = 0.80, 15.6 Hz, 1H), 2.57-2.68 (m, 3H), 2.87-2.93 (m, 1H), 2.97 (dd, *J* = 10.0, 17.2 Hz, 1H), 4.40 (d, *J* = 10.8 Hz, 2H), 5.13 (dt, *J* = 1.6, 10.0 Hz, 1H), 5.47 (td, *J* = 4.0, 6.0 Hz, 1H), 7.42-7.46 (m, 4H), 7.54-7.60 (m, 2H), 7.99-8.02(m, 4H)

^{13}C NMR ($CDCl_3$) δ 176.2, 166.3, 165.9, 133.4, 133.4, 129.7, 129.7, 129.7, 129.7, 129.6, 129.6, 128.6, 128.6, 128.5, 128.5, 84.0, 64.4, 64.4, 51.6, 40.6, 38.3, 35.8

HRMS (ESI): $[M+H]^+$ calcd for $C_{22}H_{20}O_6H$: 381.1333, found: 381.1332

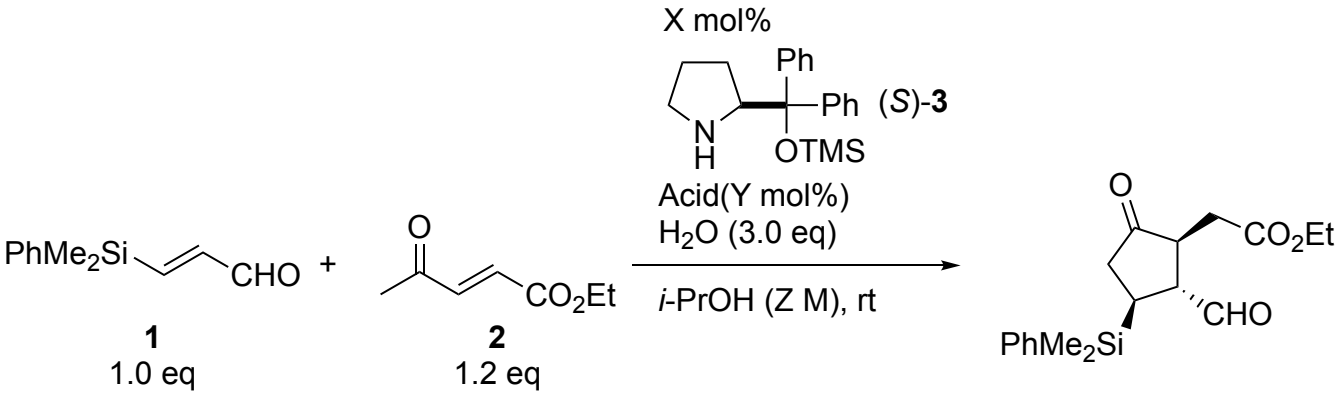
IR(neat)ν 1772, 1718, 1415, 1315, 1272, 1176, 1111, 1070, 1026, 711 cm^{-1}

$[\alpha]_D^{26}$ -70.08 (*c* 0.85, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK IF (hexane/ ^{i}PrOH = 9:11; flow rate 1.0 ml/min, major isomer t_R = 8.23 min, minor isomer t_R = 8.91 min) (>99% *ee*).

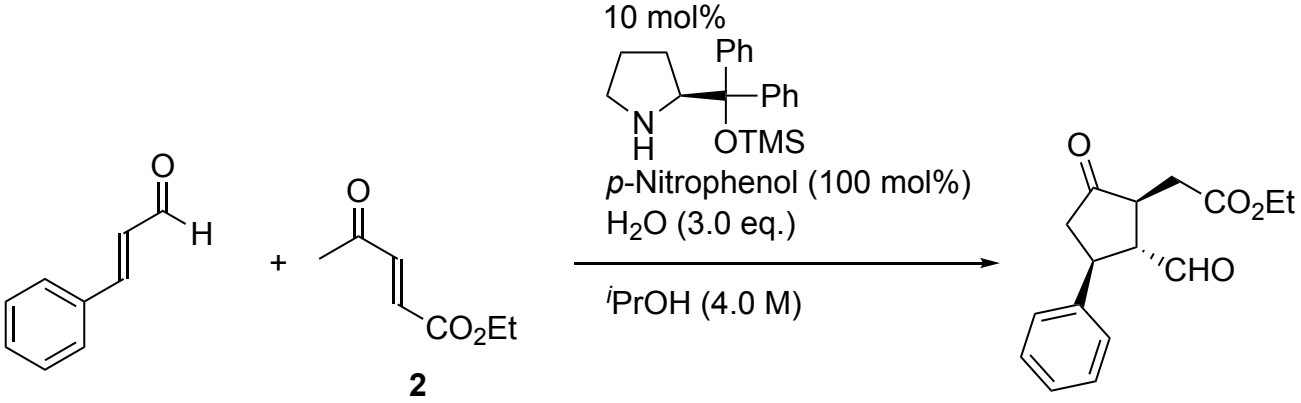
R_f (^{n}Hexane: EtOAc = 1:1, color reagent: Hanessian's stain reagent): 0.45

2.6. Optimization and typical procedure of formal (3+2) cycloaddition reaction



Entry	Catalyst [X mol%]	Acid [Y mol%]	*i*-PrOH [Z M]	Time [h]	Yield [%][b]
1	10	$PhCO_2H$ (100 mol%)	0.25	24	62
2	10	2,4,6-Trichlorophenol (100 mol%)	0.25	24	52
3	10	*p*-nitrophenol (100 mol%)	0.25	24	65
4	10	*p*-nitrophenol (50 mol%)	1.0	6	82
5	10	*p*-nitrophenol (50 mol%)	2.0	4	83
6	10	*p*-nitrophenol (50 mol%)	4.0	3	85
7	10	*p*-nitrophenol (50 mol%)	8.0	2	78
8	10	*p*-nitrophenol (100 mol%)	4.0	1	85
9	5	*p*-nitrophenol (100 mol%)	4.0	8	90
10	2.5	*p*-nitrophenol (100 mol%)	4.0	24	79

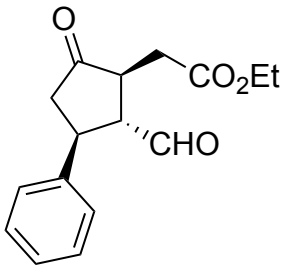
[a] Unless otherwise shown, reactions were performed by employing α,β-unsaturated aldehyde **1** (0.15 mmol), ketoe **2** (0.18 mmol), organocatalyst (*S*)-**3** (X mol%) and acid (Y mmol) in *i*-PrOH (Z M) at room temperature for the indicated time. [b] Isolated yield.



To a solution of cinnamaldehyde (19.6 mg, 0.15 mmol) and ethyl 4-oxo-2-pentenoate **2** (42.6 mg, 0.30 mmol) in ^{i}PrOH (75 μL), H_2O (8.1 μL, 0.45 mmol), diphenylprolinol silyl ether (4.88 mg, 0.015 mmol), *p*-nitrophenol (20.8 mg, 0.15 mmol) were added at room temperature. After stirring the reaction mixture at this temperature for 10 h, the reaction mixture was directly purified by column chromatography on silica gel (^{n}Hexane: EtOAc = 6:1) to give the target compound (40.3 mg, 0.147 mmol) in 98% yield (single isomer).

2.7. Compounds information

Ethyl 2-((1*R*,2*S*,3*R*)-2-formyl-5-oxo-3-phenylcyclopentyl)acetate (Table 1, entry 1)



Yield: 98% (40.3 mg)

Physical State: White solid (m.p. 82.0~84.0 ºC)

^{1}H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.2 Hz, 3H), 2.66-2.75 (m, 2H), 2.81-2.87 (m, 2H), 2.91 (q, *J* = 10.8 Hz, 1H), 3.40 (dt, *J* = 2.0, 11.2 Hz, 1H), 3.50 (dt, *J* = 8.0, 11.6 Hz, 1H), 4.14 (dq, *J* = 1.6, 7.2 Hz, 2H), 7.27-7.40 (m, 5H), 9.69 (d, *J* = 1.6 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.7, 200.9, 171.5, 140.1, 129.0, 129.0, 127.6, 127.2, 127.2, 61.1, 59.9, 46.7, 45.8, 42.4, 32.5, 14.1

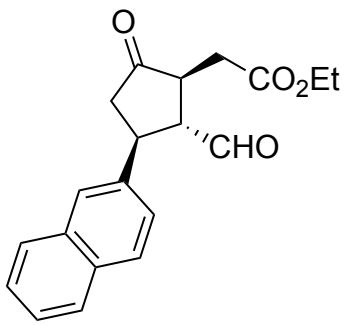
HRMS (ESI): $[M+Na]^+$ calcd for $C_{16}H_{18}O_4Na$: 297.1097, found: 297.1100

IR(neat)ν 2982, 1726, 1497, 1455, 1406, 1376, 1197, 1031, 763, 702 cm^{-1}

$[\alpha]_D^{26}$ +92.83 (*c* 3.2, $CHCl_3$)

R_f (^{n}Hexane: EtOAc =3:1, color reagent: Hanessian's stain reagent): 0.45

Ethyl 2-((1*R*,2*S*,3*R*)-2-formyl-3-(naphthalen-2-yl)-5-oxocyclopentyl)acetate (Table 1, entry 2)



Yield: 84% (40.9 mg)

Physical State: White solid (m.p. 73.0~75.0 ºC)

^{1}H NMR ($CDCl_3$) δ 1.28 (t, *J* = 7.2 Hz, 3H), 2.72-3.00 (m, 5H), 3.54 (dt, *J* = 2.4, 11.2 Hz, 1H), 3.64 (dt, *J* = 8.0, 11.6 Hz, 1H), 4.16 (dq, *J* = 1.2, 7.2 Hz, 2H), 7.46-7.53 (m, 3H), 7.78-7.89 (m, 4H), 9.72 (d, *J* = 2.0 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.7, 200.9, 171.6, 137.4, 133.4, 132.8, 129.2, 127.7, 127.7, 126.5, 126.3, 126.2, 124.8, 61.1, 59.7, 46.7, 45.8, 42.7, 32.6, 14.1

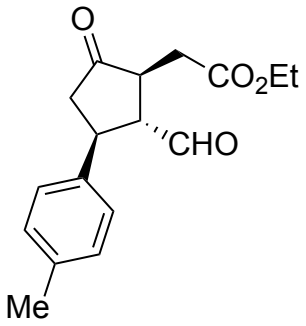
HRMS (ESI): $[M+Na]^+$ calcd for $C_{20}H_{20}O_4Na$: 347.1254, found: 347.1258

IR(neat)ν 1726, 1727, 1508, 1406, 1396, 1405, 1396, 1375, 1301, 1269, 1238, 1197, 1154, 1030, 860, 822, 751, 478 cm^{-1}

$[\alpha]_D^{26}$ +79.98 (*c* 1.2, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.35

Ethyl 2-((1*R*,2*S*,3*R*)-2-formyl-5-oxo-3-(*p*-tolyl)cyclopentyl)acetate (Table 1, entry 3)



Yield: 91% (39.5 mg)

Physical State: White solid (m.p. 80.5~83.1 ºC)

^{1}H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.2 Hz, 3H), 2.34 (s, 3H), 2.67 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.69-2.75 (m, 1H), 2.81-2.90 (m, 2H), 3.35 (dt, *J* = 2.0, 11.2 Hz, 1H), 3.43 (dt, *J* = 8.0, 11.2 Hz, 1H), 4.14 (dq, *J* = 1.6, 7.2 Hz, 2H), 7.17 (d, *J* = 7.6 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 9.68 (d, *J* = 2.0 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.8, 201.1, 171.3, 137.4, 137.0, 129.7, 129.7, 127.1, 127.1, 61.0, 60.0, 46.7, 45.8, 42.1, 32.5, 21.0, 14.1

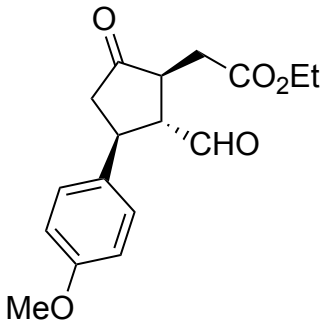
HRMS (ESI): $[M+Na]^+$ calcd for $C_{17}H_{20}O_4Na$: 311.1254, found: 311.1256

IR(neat)ν 1726, 1238, 1192, 1030, 817, 503, 490, 467, 434, 418, 406 cm^{-1}

$[\alpha]_D^{26}$ +76.44 (*c* 2.8, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.40

Ethyl 2-((1*R*,2*S*,3*R*)-2-formyl-3-(4-methoxyphenyl)-5-oxocyclopentyl)acetate (Table 1, entry 4)



Yield: 88% (40.2 mg)

Physical State: White solid (m.p. 95.4~98.1 ºC)

^{1}H NMR ($CDCl_3$) δ 1.24 (t, *J* = 7.2 Hz, 3H), 2.63 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.65 (dd, *J* = 3.2, 16.8 Hz, 1H), 2.78-2.89 (m, 3H), 3.32 (dt, *J* = 1.6, 11.2 Hz, 1H), 3.40 (dt, *J* = 8.0, 11.2 Hz, 1H), 3.79 (s, 3H), 4.12 (dq, *J* = 1.6, 7.2 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 9.66 (d, *J* = 1.6 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.8, 201.1, 171.5, 158.9, 131.9, 128.2, 128.2, 114.4, 114.4, 61.0, 60.1, 55.3, 46.7, 45.9, 41.7, 32.5, 14.1

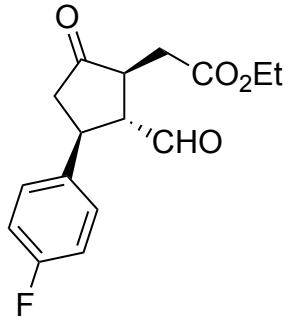
HRMS (ESI): $[M+Na]^+$ calcd for $C_{17}H_{20}O_5Na$: 327.1202, found: 327.1207

IR(neat)ν 2981, 1745, 1726, 1613, 1516, 1376, 1298, 1252, 1182, 1157, 1033, 832 cm^{-1}

$[\alpha]_D^{26}$ +56.93 (*c* 3.8, $CHCl_3$)

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.19

Ethyl 2-((1*R*,2*S*,3*R*)-3-(4-fluorophenyl)-2-formyl-5-oxocyclopentyl)acetate (Table 1, entry 5)



Yield: 81% (35.5 mg)

Physical State: yellow oil

^{1}H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.2 Hz, 3H), 2.65 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.72 (dd, *J* = 4.4, 17.2 Hz, 1H), 2.81 (dddd, *J* = 0.80, 4.4, 5.6, 11.6 Hz, 1H), 2.89 (dd, *J* = 5.6, 17.6 Hz, 1H), 2.89 (ddd, *J* = 0.80, 8.8, 18.8 Hz, 1H), 3.36 (dt, *J* = 2.0, 11.6 Hz, 1H), 3.47 (dt, *J* = 8.0, 11.6 Hz, 1H), 4.14 (dq, *J* = 2.0, 7.2 Hz, 2H), 7.06 (d, *J* = 8.8 Hz, 2H), 7.32 (dd, *J* = 5.2, 8.4 Hz, 2H), 9.68 (d, *J* = 2.0 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.4, 200.7, 171.5, 162.1 (d, $J_{C\text{-}F}$ = 246 Hz), 135.9 (d, $J_{C\text{-}F}$ = 3.0 Hz), 128.8 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.8 (d, $J_{C\text{-}F}$ = 8.0 Hz), 116.1 (d, $J_{C\text{-}F}$ = 21 Hz), 116.1 (d, $J_{C\text{-}F}$ = 21 Hz), 61.1, 60.1, 46.7, 45.8, 41.6, 32.5, 14.1

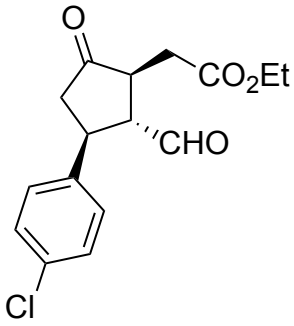
HRMS (ESI): $[M+Na]^+$ calcd for $C_{16}H_{17}FO_4Na$: 315.1003, found: 315.1009

IR(neat)ν 2983, 1747, 1727, 1605, 1513, 1376, 1350, 1226, 1197, 1160, 1097, 1030, 838, 737 cm^{-1}

$[\alpha]_D^{26}$ +69.17 (*c* 1.0, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.30

Ethyl 2-((1*R*,2*S*,3*R*)-3-(4-chlorophenyl)-2-formyl-5-oxocyclopentyl)acetate (Table 1, entry 6)



Yield: 85% (39.5 mg)

Physical State: White solid (m.p. 70.4~72.9 ºC)

^{1}H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.2 Hz, 3H), 2.65 (dd, *J* = 11.6, 18.8 Hz, 1H), 2.72 (dd, *J* = 3.6, 17.2 Hz, 1H), 2.81 (dddd, *J* = 1.2, 3.6, 4.8, 10.8 Hz, 1H), 2.89 (ddd, *J* = 1.2, 8.0, 18.4 Hz, 1H), 2.65 (dd, *J* = 5.2, 17.6 Hz, 1H), 3.37 (dt, *J* = 2.0, 11.2 Hz, 1H), 3.46 (dt, *J* = 8.0, 11.6 Hz, 1H), 4.14 (dq, *J* = 2.0, 7.2 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 9.68 (d, *J* = 2.4 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.2, 200.5, 171.5, 138.7, 133.4, 129.2, 129.2, 128.6, 128.6, 61.1, 59.8, 46.6, 45.6, 41.6, 32.5, 14.1

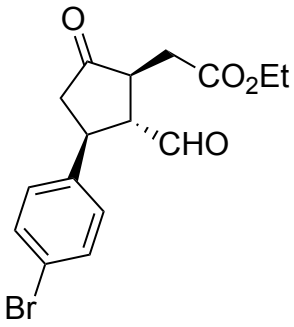
HRMS (ESI): $[M+Na]^+$ calcd for $C_{16}H_{17}ClO_4Na$: 331.0708, found: 331.0714

IR(neat)ν 2981, 1727, 1494, 1414, 1376, 1414, 1376, 1350, 1196, 1092, 1030, 1014, 830 cm^{-1}

$[\alpha]_D^{26}$ +57.31 (*c* 1.1, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.32

Ethyl 2-((1*R*,2*S*,3*R*)-3-(4-bromophenyl)-2-formyl-5-oxocyclopentyl)acetate (Table 1, entry 7)



Yield: 83% (43.8 mg)

Physical State: White solid (m.p. 86.4~88.3 ºC)

^{1}H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.2 Hz, 3H), 2.65 (dd, *J* = 12.0, 19.2 Hz, 1H), 2.71 (dd, *J* = 4.0, 17.2 Hz, 1H),

2.80 (dddd, *J* = 1.2, 3.2, 4.8, 10.4 Hz, 1H), 2.88 (ddd, *J* = 1.6, 8.0, 18.4, Hz, 1H), 2.90 (dd, *J* = 5.6, 17.2 Hz, 1H), 3.36 (dt, *J* = 2.0, 11.2 Hz, 1H), 3.44 (dt, *J* = 8.0, 11.6 Hz, 1H), 4.13 (dq, *J* = 2.0, 7.2 Hz, 2H), 7.23 (d, *J* = 8.8 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 9.67 (d, *J* = 2.4 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.2, 200.5, 171.5, 139.2, 132.2, 132.2, 129.0, 129.0, 121.4, 61.1, 59.8, 46.7, 45.6, 41.8, 32.5, 14.1

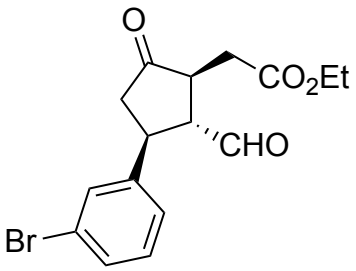
HRMS (ESI): $[M+Na]^+$ calcd for $C_{16}H_{17}BrO_4Na$: 375.0202, found: 375.0202

IR(neat)ν 3463, 1745, 1726, 1490, 1375, 1239, 1196, 1157, 1074, 1029, 1010, 825 cm^{-1}

$[\alpha]_D^{26}$ +35.95 (*c* 2.8, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.32

Ethyl 2-((1*R*,2*S*,3*R*)-3-(3-bromophenyl)-2-formyl-5-oxocyclopentyl)acetate (Table 1, entry 8)



Yield: 74% (39.1 mg)

Physical State: yellow oil

^{1}H NMR ($CDCl_3$) δ 1.27 (t, *J* = 7.2 Hz, 3H), 2.66 (dd, *J* = 11.6, 18.4 Hz, 1H), 2.66 (dd, *J* = 11.6, 18.4 Hz, 1H), 2.72 (dd, *J* = 3.2, 17.2 Hz, 1H), 2.81 (dddd, *J* = 1.2, 3.2, 5.6, 11.2 Hz, 1H), 2.89 (ddd, *J* = 1.2, 8.0, 18.8 Hz, 1H), 2.90 (dd, *J* = 5.6, 17.2 Hz, 1H), 3.38 (dt, *J* = 2.0, 11.2 Hz, 1H), 3.44 (dt, *J* = 8.0, 11.2 Hz, 1H), 4.14 (dq, *J* = 1.6, 7.2 Hz, 2H), 7.23 (d, *J* = 7.6 Hz, 1H), 7.28 (td, *J* = 1.6, 9.6 Hz, 1H), 7.43 (td, *J* = 2.0, 7.6 Hz, 1H), 7.49 (d, *J* = 2.0 Hz, 1H), 9.69 (d, *J* = 2.0 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.0, 200.4, 171.4, 142.5, 130.8, 130.7, 130.3, 126.0, 123.1, 61.2, 59.6, 46.7, 46.0, 42.0, 32.5, 14.1

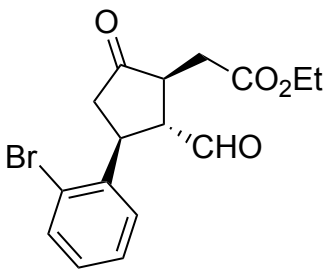
HRMS (ESI): $[M+Na]^+$ calcd for $C_{16}H_{17}BrO_4Na$: 375.0202, found: 375.0202

IR(neat)ν 1726, 1375, 1239, 1195, 1092, 1074, 1029, 787, 695, 442, 431, 413 cm^{-1}

$[\alpha]_D^{26}$ +115.6 (*c* 1.0, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.36

Ethyl 2-((1*R*,2*S*,3*R*)-3-(2-bromophenyl)-2-formyl-5-oxocyclopentyl)acetate (Table 1, entry 9)



Yield: 82% (43.2 mg)

Physical State: yellow oil

^{1}H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.2 Hz, 3H), 2.55 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.74 (dd, *J* = 5.2, 19.2 Hz, 1H), 2.85-2.92 (m, 2H), 2.99 (dd, *J* = 8.4, 18.4 Hz, 1H), 3.43 (dt, *J* = 2.8, 11.2 Hz, 1H), 4.04 (dt, *J* = 8.4, 11.6 Hz, 1H), 4.14 (dq, *J* = 2.0, 7.2 Hz, 2H), 7.16 (ddd, *J* = 1.6, 7.2, 8.0 Hz, 1H), 7.37 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.49 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.60 (dd, *J* = 1.2, 8.0 Hz, 1H), 9.69 (d, *J* = 2.4 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.2, 200.1, 171.6, 139.3, 133.3, 129.0, 128.4, 127.7, 124.8, 61.1, 59.4, 46.3, 44.7, 40.6, 32.5, 14.1

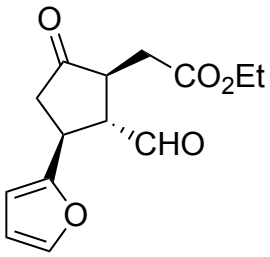
HRMS (ESI): $[M+H]^+$ calcd for $C_{16}H_{17}BrO_4H$: 353.0383, found: 353.0386

IR(neat)ν 1747, 1727, 1473, 1375, 1239, 1196, 1157, 1026, 757 cm^{-1}

$[\alpha]_D^{26}$ +19.29 (*c* 3.0, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.30

Ethyl 2-((1*R*,2*S*,3*R*)-2-formyl-3-(furan-2-yl)-5-oxocyclopentyl)acetate (Table 1, entry 10)



Yield: 79% (31.5 mg)

Physical State: yellow oil

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 3H), 2.68-2.76 (m, 2H), 2.80-2.88 (m, 3H), 3.31 (dt, *J* = 2.0, 10.4 Hz, 1H), 3.59 (dt, *J* = 8.4, 11.2 Hz, 1H), 4.17 (dq, *J* = 1.6, 7.2 Hz, 2H), 6.17 (d, *J* = 0.80, 3.2 Hz, 1H), 6.33 (d, *J* = 2.0, 3.2 Hz, 1H), 7.36 (dd, *J* = 0.80, 2.0 Hz, 1H), 9.84 (d, *J* = 2.0 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 211.7, 200.5, 171.2, 153.2, 142.2, 110.4, 106.2, 61.0, 57.6, 46.4, 42.2, 35.2, 32.6, 14.0

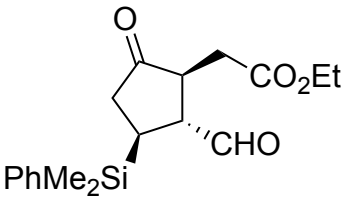
HRMS (ESI): $[M+Na]^+$ calcd for $C_{14}H_{16}O_5Na$: 265.1071, found: 265.1076

IR(neat)ν2983, 1748, 1730, 1409, 1376, 1346, 1240, 1195, 1154, 1014, 742 cm^{-1}

$[\alpha]_D^{26}$ +112.7 (*c* 0.86, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.25

Ethyl 2-((1*R*,2*S*,3*R*)-3-(dimethyl(phenyl)silyl)-2-formyl-5-oxocyclopentyl)acetate



Yield: 90% (45.1 mg)

Physical State: colorless oil

^{1}H NMR ($CDCl_3$) δ 0.365 (s, 3H), 0.381 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H), 1.73 (ddd, *J* = 8.8, 12.0, 13.2 Hz, 1H), 2.18 (dd, *J* = 13.2, 18.8 Hz, 1H), 2.49 (ddd, *J* = 1.2, 8.8, 18.4 Hz, 1H), 2.53 (dd, *J* = 6.8, 17.6 Hz, 1H), 2.62 (dd, *J* = 4.0, 17.6 Hz, 1H), 2.69 (dddd, *J* = 1.2, 4.0, 6.4, 12.0 Hz, 1H), 2.82 (dt, *J* = 3.6, 12.0 Hz, 1H), 4.06 (dq, *J* = 1.6, 7.2 Hz, 2H), 7.36-7.41 (m, 3H), 7.48-7.51 (m, 2H), 9.43 (d, *J* = 3.2 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 215.5, 201.1, 171.3, 135.6, 133.8, 133.8, 129.8, 128.1, 128.1, 60.9, 55.9, 47.5, 38.7, 32.7, 22.7, 14.0, -4.43, -4.65

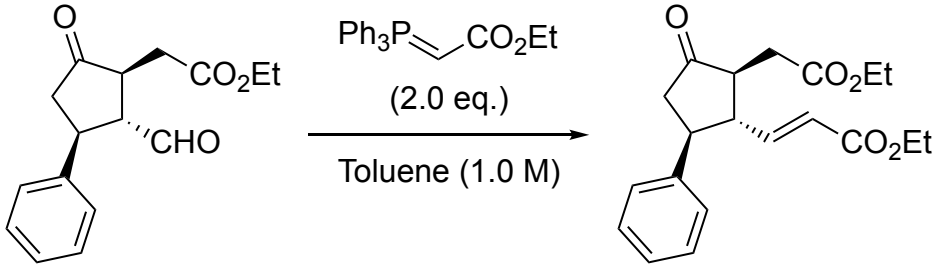
HRMS (ESI): $[M+Na]^+$ calcd for $C_{18}H_{24}O_4SiNa$: 355.1336, found: 355.1340

IR(neat)ν 1725, 1427, 1375, 1253, 1189, 1113, 1026, 835, 818, 775, 736, 701 cm^{-1}

$[\alpha]_D^{26}$ +59.42 (*c* 3.8, $CHCl_3$)

R_f (*n*Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.50

2.8. Typical procedure of Wittig reaction and determination of *ee* value

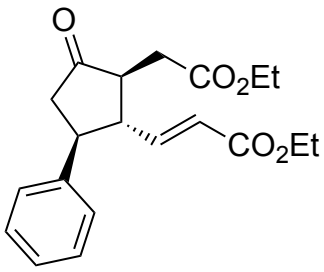


To a solution of aldehyde (27.4 mg, 0.10 mmol) in toluene (100 μL), Wittig reagent (69.6 mg, 0.20 mmol) were added at room temperature. After stirring the reaction mixture at this temperature for 1 h, the reaction mixture was directly purified by column chromatography on silica gel (*n*Hexane : EtOAc = 3:1) to give the target compound (32.7 mg, 0.095 mmol) in 95% yield (single isomer).

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 31.8 min, minor isomer t_R = 43.5 min) (>99% *ee*).

2.9. Compounds information

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-2-(2-ethoxy-2-oxoethyl)-3-oxo-5-phenylcyclopentyl)acrylate



Yield: 95% (32.7 mg)

Physical State: White solid (m.p. 85.0~87.1 ºC)

^{1}H NMR ($CDCl_3$) δ 1.22 (t, *J* = 7.2 Hz, 3H), 1.25 (t, *J* = 7.2 Hz, 3H), 2.48 (dddd, *J* = 1.2, 4.8, 5.2, 11.6 Hz, 1H), 2.56 (dd, *J* = 4.8, 17.6 Hz, 1H), 2.61 (dd, *J* = 12.4, 18.8 Hz, 1H), 2.77 (dd, *J* = 5.2, 17.6 Hz, 1H), 2.83 (ddd, *J* = 1.2, 8.0, 18.8 Hz, 1H), 3.04 (dd, *J* = 8.8, 11.2 Hz, 1H), 3.18 (dt, *J* = 8.0, 12.0 Hz, 1H), 4.08-4.16 (m, 4H), 5.64 (d, *J* = 15.6 Hz, 1H), 6.78 (dd, *J* = 8.8, 15.6 Hz, 1H), 7.23-7.25 (m, 3H), 7.29-7.33 (m, 2H)

^{13}C NMR ($CDCl_3$) δ 214.5, 171.3, 165.8, 147.1, 140.2, 128.8, 128.8, 127.3, 127.3, 127.2, 123.7, 60.9, 60.5, 51.6, 51.5, 46.5, 45.6, 31.7, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{20}H_{24}O_5Na$: 367.1516, found: 367.1520

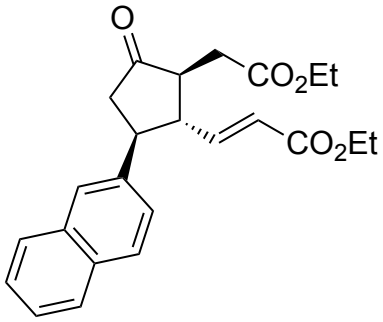
IR(neat)ν 1744, 1726, 1372, 1308, 1269, 1250, 1227, 1188, 1154, 1033 cm^{-1}

$[\alpha]_D^{26}$ +91.61 (*c* 1.2, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 31.8 min, minor isomer t_R = 43.5 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc =3:1, color reagent: Hanessian's stain reagent): 0.45

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-2-(2-ethoxy-2-oxoethyl)-5-(naphthalen-2-yl)-3-oxocyclopentyl)acrylate



Yield: 90% (35.5 mg)

Physical State: White solid (m.p. 95.1~97.8 ºC)

^{1}H NMR ($CDCl_3$) δ 1.21 (t, *J* = 7.2 Hz, 3H), 1.28 (t, *J* = 7.2 Hz, 3H), 2.55 (dddd, *J* = 0.80, 4.4, 5.2, 11.2 Hz, 1H), 2.62 (dd, *J* = 4.4, 17.2 Hz, 1H), 2.73 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.83 (dd, *J* = 5.2, 17.2 Hz, 1H), 2.91 (ddd, *J* = 0.80, 8.0, 18.4 Hz, 1H), 3.19 (dd, *J* = 9.2, 11.2 Hz, 1H), 3.36 (dt, *J* = 8.0, 12.0 Hz, 1H), 4.06-4.20 (m, 4H), 5.66 (dd, *J* = 0.4, 15.6 Hz, 1H), 6.78 (dd, *J* = 8.8, 15.6 Hz, 1H), 7.34 (dd, *J* = 2.0, 8.8 Hz, 1H), 7.44-7.51 (m, 2H), 7.68 (m, 1H), 7.79-7.84 (m, 3H)

^{13}C NMR ($CDCl_3$) δ 214.4, 171.4, 165.7, 147.1, 137.6, 133.4, 132.6, 128.7, 127.7, 126.3, 126.3, 125.8, 125.0, 123.8, 61.0, 60.4, 51.6, 51.5, 46.8, 45.7, 31.7, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{24}H_{26}O_5Na$: 417.1673, found: 417.1673

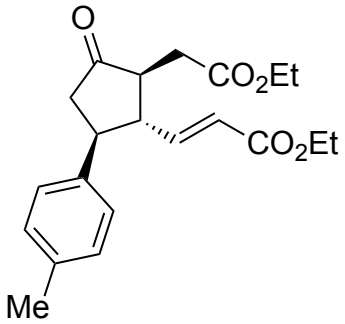
IR(neat)ν 3460, 1744, 1725, 1655, 1372, 1306, 1271, 1230, 1191, 1154 cm^{-1}

$[\alpha]_D^{26}$ +96.47 (*c* 2.6, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 30.2 min, minor isomer t_R = 50.9 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.35

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-2-(2-ethoxy-2-oxoethyl)-3-oxo-5-(*p*-tolyl)cyclopentyl)acrylate



Yield: 92% (32.8 mg)

Physical State: White solid (m.p. 91.5~93.2 ºC)

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 6H), 2.36 (s, 3H), 2.49 (dddd, *J* = 0.80, 4.4, 5.2, 11.6 Hz, 1H), 2.58 (dd, *J* = 4.4, 17.2 Hz, 1H), 2.59 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.76 (dd, *J* = 5.2, 17.2 Hz, 1H), 2.82 (ddd, *J* = 1.2, 8.0, 18.8 Hz, 1H), 3.02 (dd, *J* = 8.8, 11.2 Hz, 1H), 3.16 (dt, *J* = 8.0, 12.0 Hz, 1H), 4.09-4.18 (m, 4H), 5.67 (dd, *J* = 0.4, 15.6 Hz, 1H), 6.79 (dd, *J* = 8.8, 15.6 Hz, 1H), 7.13 (s, 4H)

^{13}C NMR ($CDCl_3$) δ 214.6, 171.4, 165.8, 147.3, 137.1, 136.8, 129.5, 129.5, 127.2, 127.2, 123.6, 60.9, 60.5, 51.7, 51.6, 46.1, 45.7, 31.7, 21.0, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{21}H_{26}O_5Na$: 381.1673, found: 381.1684

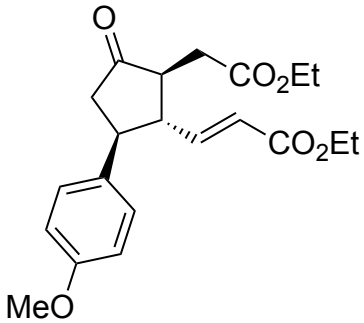
IR(neat)ν 1746, 1731, 1655, 1373, 1349, 1308, 1269, 1249, 1228, 1214, 1185, 1155, 1096, 1034, 818 cm^{-1}

$[\alpha]_D^{26}$ +131.4 (*c* 0.34, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/ ^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 23.7 min, minor isomer t_R = 31.5 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.40

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-2-(2-ethoxy-2-oxoethyl)-5-(4-methoxyphenyl)-3-oxocyclopentyl)acrylate



Yield: 85% (31.8 mg)

Physical State: yellow solid (m.p. 105~106 ºC)

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 3H), 1.26 (t, *J* = 7.2 Hz, 3H), 2.49 (dtd, *J* = 0.80, 4.8, 11.6 Hz, 1H), 2.57 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.58 (dd, *J* = 4.8, 17.2 Hz, 1H), 2.78 (dd, *J* = 4.8, 16.8 Hz, 1H), 2.82 (ddd, *J*

= 1.2, 8.0, 18.4 Hz, 1H), 2.99 (dd, *J* = 9.2, 11.2 Hz, 1H), 3.15 (ddd, *J* = 8.0, 11.2, 12.0 Hz, 1H), 3.79 (s, 3H), 4.09-4.17 (m, 4H), 5.66 (dd, *J* = 1.2 , 15.6 Hz, 1H), 6.79 (dd, *J* = 8.8, 15.6 Hz, 1H), 7.13 (s, 4H)

^{13}C NMR ($CDCl_3$) δ 214.7, 171.4, 165.8, 158.6, 147.3, 132.2, 128.3, 128.3, 123.7, 114.2, 114.2, 60.9, 60.5, 55.3, 51.9, 51.6, 45.8, 45,7, 31.7, 14.2, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{21}H_{26}O_6Na$: 397.1622, found: 397.1627

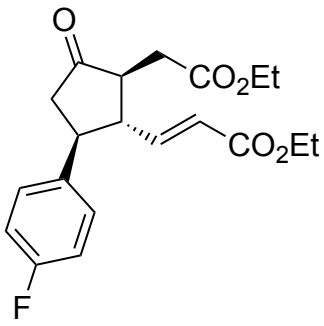
IR(neat)ν 2982, 1745, 1730, 1655, 1515, 1373, 1308, 1252, 1226, 1183, 1035, 832 cm^{-1}

$[\alpha]_D^{26}$ +85.60 (*c* 0.57, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 37.9 min, minor isomer t_R = 58.1 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.19

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-2-(2-ethoxy-2-oxoethyl)-5-(4-fluorophenyl)-3-oxocyclopentyl)acrylate



Yield: 92% (33.2 mg)

Physical State: White solid (m.p. 100~102 ºC)

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 3H), 1.26 (t, *J* = 7.2 Hz, 3H), 2.48 (dddd, *J* = 0.80, 4.8, 5.2, 11.2 Hz, 1H), 2.58 (dd, *J* = 4.8, 17.2 Hz, 1H), 2.59 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.81 (dd, *J* = 5.2, 17.2 Hz, 1H), 2.83 (ddd, *J* = 0.80, 8.0, 18.8 Hz, 1H), 3.01 (dd, *J* = 9.2, 10.8 Hz, 1H), 3.18 (ddd, *J* = 8.0, 11.2, 12.0 Hz, 1H), 4.09-4.18 (m, 4H), 5.65 (dd, *J* = 0.80 , 15.6 Hz, 1H), 6.77 (dd, *J* = 9.2, 15.6 Hz, 1H), 7.02 (t, *J* = 8.8 Hz, 2H), 7.21 (dd, *J* = 5.2, 8.8 Hz, 2H)

^{13}C NMR ($CDCl_3$) δ 214.2, 171.3, 165.7, 161.8 (d, $J_{C\text{-}F}$ = 245 Hz), 146.8, 135.9 (d, $J_{C\text{-}F}$ = 3.0 Hz), 128.8 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.8 (d, $J_{C\text{-}F}$ = 8.0 Hz), 123.9, 115.8 (d, $J_{C\text{-}F}$ = 21 Hz), 115.8(d, $J_{C\text{-}F}$ = 21 Hz), 60.9, 60.5, 51.8, 51.5, 45.8, 45.6, 31.6, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{20}H_{23}FO_5Na$: 385.1421, found: 385.1417

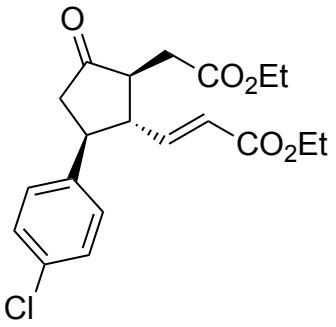
IR(neat)ν 1745, 1720, 1655, 1512, 1373, 1308, 1270, 1226, 1186, 1159, 1095, 1033, 837 cm^{-1}

$[\alpha]_D^{26}$ +72.19 (*c* 1.0, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 23.7 min, minor isomer t_R = 41.9 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.30

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-5-(4-chlorophenyl)-2-(2-ethoxy-2-oxoethyl)-3-oxocyclopentyl)acrylate



Yield: 81% (30.7 mg)

Physical State: White solid (m.p. 103~105 ºC)

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 3H), 1.26 (t, *J* = 7.2 Hz, 3H), 2.47 (dtd, *J* = 0.80, 4.8, 11.6 Hz, 1H), 2.57 (dd, *J* = 4.4, 17.6 Hz, 1H), 2.58 (dd, *J* = 12.4, 18.8 Hz, 1H), 2.80 (dd, *J* = 4.8, 17.6 Hz, 1H), 2.83 (ddd, *J* = 0.80, 8.0, 18.8 Hz, 1H), 3.01 (dd, *J* = 9.2, 11.2 Hz, 1H), 3.17 (dd, *J* = 8.0, 12.0 Hz, 1H), 4.07-4.19 (m, 4H), 5.65 (dd, *J* = 0.80, 15.6 Hz, 1H), 6.77 (dd, *J* = 9.2, 15.6 Hz, 1H), 7.18 (d, *J* = 8.4 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 214.0, 171.3, 165.6, 146.7, 138.8, 132.9, 129.0, 129.0, 128.7, 128.7, 124.0, 61.0, 60.5, 51.7, 51.5, 45.9, 45.4, 31.6, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{21}H_{26}ClO_6Na$: 401.1126, found: 401.1125

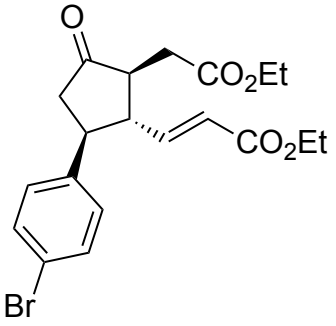
IR(neat)ν 1745, 1720, 1495, 1373, 1308, 1272, 1249, 1227, 1187, 1155, 1092, 1033 cm^{-1}

$[\alpha]_D^{26}$ +80.34 (*c* 1.0, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 19.5 min, minor isomer t_R = 34.3 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.32

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-5-(4-bromophenyl)-2-(2-ethoxy-2-oxoethyl)-3-oxocyclopentyl)acrylate



Yield: 96% (40.6 mg)

Physical State: White solid (m.p. 105~107 ºC)

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 3H), 1.26 (t, *J* = 7.2 Hz, 3H), 2.47 (dtd, *J* = 1.2, 4.8, 11.2 Hz, 1H), 2.57 (dd, *J* = 4.8, 17.6 Hz, 1H), 2.58 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.81 (dd, *J* = 4.8, 17.6 Hz, 1H), 2.83 (ddd, *J* = 0.80, 8.0, 18.8 Hz, 1H), 3.02 (dd, *J* = 9.2, 11.2 Hz, 1H), 3.16 (dt, *J* = 8.0, 12.0 Hz, 1H), 4.07-4.19 (m, 4H),

5.66 (dd, *J* = 0.80 , 15.6 Hz, 1H), 6.77 (dd, *J* = 9.2, 15.6 Hz, 1H), 7.12 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H)

^{13}C NMR ($CDCl_3$) δ 214.0, 171.3, 165.6, 146.7, 139.2, 131.9, 131.9, 129.0, 129.0, 124.0, 121.0, 61.0, 60.5, 51.6, 51.5, 46.0, 45.4, 31.6, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{20}H_{23}BrO_6Na$: 445.0621, found: 3445.0634

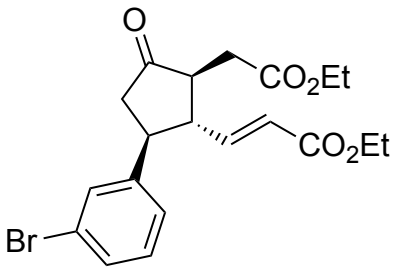
IR(neat)ν 1724, 1373, 1308, 1273, 1227, 1187, 1034, 1010 cm^{-1}

$[\alpha]_D^{26}$ +67.76 (*c* 1.6, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 20.6 min, minor isomer t_R = 36.8 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.32

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-5-(3-bromophenyl)-2-(2-ethoxy-2-oxoethyl)-3-oxocyclopentyl)acrylate



Yield: 94% (39.7 mg)

Physical State: yellow oil

^{1}H NMR ($CDCl_3$) δ 1.26 (t, *J* = 7.2 Hz, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 2.48 (dtd, *J* = 0.80, 4.8, 11.2 Hz, 1H), 2.58 (dd, *J* = 4.4, 17.2 Hz, 1H), 2.58 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.80 (dd, *J* = 5.2, 17.2 Hz, 1H), 2.84 (ddd, *J* = 0.80, 8.0, 18.8 Hz, 1H), 3.04 (dd, *J* = 8.8, 11.2 Hz, 1H), 3.15 (dt, *J* = 8.0, 12.0 Hz, 1H), 4.09-4.18 (m, 4H), 5.68 (dd, *J* = 0.80 , 15.6 Hz, 1H), 6.77 (dd, *J* = 8.8, 15.6 Hz, 1H), 7.16-7.20 (m, 2H), 7.37-7.40 (m, 2H)

^{13}C NMR ($CDCl_3$) δ 213.8, 171.3, 165.6, 146.5, 142.7, 130.4, 130.3, 130.3, 126.1, 124.0, 122.8, 61.0, 60.6, 51.5, 51.4, 46.2, 45.5, 31.6, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{20}H_{23}BrO_6Na$: 445.0621, found: 445.0628

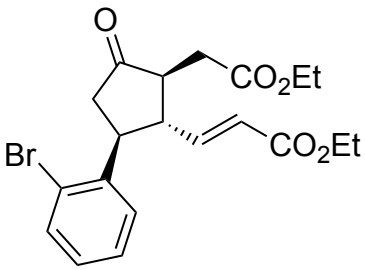
IR(neat)ν 1745, 1724, 1655, 1476, 1373, 1349, 1308, 1270, 1249, 1227, 1187, 1155, 1095, 1074, 1033, 787, 695 cm^{-1}

$[\alpha]_D^{26}$ +57.39 (*c* 1.2, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 23.8 min, minor isomer t_R = 40.0 min) (>99% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.30

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-5-(2-bromophenyl)-2-(2-ethoxy-2-oxoethyl)-3-oxocyclopentyl)acrylate



Yield: 93% (39.2 mg)

Physical State: yellow oil

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 2.41 (dd, *J* = 12.4, 18.8 Hz, 1H), 2.54 (dddd, *J* = 0.80, 4.0, 5.2, 11.6 Hz, 1H), 2.61 (dd, *J* = 4.0, 17.2 Hz, 1H), 2.81 (dd, *J* = 5.2, 17.2 Hz, 1H), 2.97 (ddd, *J* = 0.80, 8.0, 18.8 Hz, 1H), 3.20 (dt, *J* = 9.2, 11.2 Hz, 1H), 3.80 (ddd, *J* = 8.0, 10.8, 12.0 Hz, 1H), 4.10-4.18 (m, 4H), 5.72 (dd, *J* = 0.80 , 15.6 Hz, 1H), 6.83 (dd, *J* = 8.8, 15.6 Hz, 1H), 7.11 (td, *J* = 4.4, 8.4 Hz, 1H), 7.32 (d, *J* = 4.0 Hz, 2H), 7.56 (d, *J* = 8.0 Hz, 1H)

^{13}C NMR ($CDCl_3$) δ 213.8, 171.4, 165.6, 146.5, 139.5, 133.2, 128.6, 128.1, 127.2, 125.3, 123.8, 61.0, 60.5, 51.4, 50.3, 44.7, 44.6, 31.5, 14.1, 14.1

HRMS (ESI): $[M+Na]^+$ calcd for $C_{20}H_{23}BrO_5H$: 423.0802, found: 423.0807

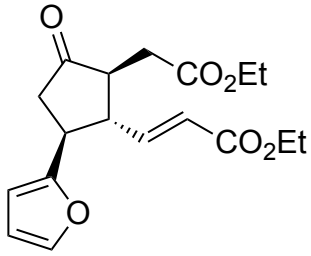
IR(neat)ν 1745, 1720, 1655, 1473, 1373, 1308, 1274, 1249, 1228, 1188, 1156, 1096, 1031, 757 ßßcm^{-1}

$[\alpha]_D^{26}$ +37.77 (*c* 1.6, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 26.3 min, minor isomer t_R = 39.0 min) (96% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.30

Ethyl (*E*)-3-((1*S*,2*R*,5*R*)-2-(2-ethoxy-2-oxoethyl)-5-(furan-2-yl)-3-oxocyclopentyl)acrylate



Yield: 80% (26.6 mg)

Physical State: yellow oil

^{1}H NMR ($CDCl_3$) δ 1.25 (t, *J* = 7.2 Hz, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 2.50 (dddd, *J* = 1.2, 4.8, 5.2, 11.2 Hz, 1H), 2.58 (dd, *J* = 4.8, 17.2 Hz, 1H), 2.67 (dd, *J* = 12.0, 18.8 Hz, 1H), 2.71 (dd, *J* = 5.2, 17.2 Hz, 1H), 2.79 (ddd, *J* = 1.2, 8.0, 18.4 Hz, 1H), 3.04 (dd, *J* = 9.2, 11.2 Hz, 1H), 3.30 (dt, *J* = 8.0, 11.2 Hz, 1H), 4.08-4.20 (m, 4H), 5.78 (dd, *J* = 0.80 , 15.6 Hz, 1H), 6.10 (d, *J* = 3.6 Hz, 1H), 6.29 (dd, *J* = 2.0, 3.2 Hz, 1H), 6.88 (dd, *J* = 8.8, 15.6 Hz, 1H), 7.33 (dd, *J* = 0.80 , 2.0 Hz, 1H),

^{13}C NMR ($CDCl_3$) δ 213.5, 171.1, 165.7, 153.3, 146.9, 141.9, 123.9, 110.2, 106.2, 60.9, 60.5, 51.3, 49.4, 42.5,

39.5, 31.7, 14.1, 14.0

HRMS (ESI): $[M+Na]^+$ calcd for $C_{18}H_{22}O_6Na$: 335.1490, found: 335.1490

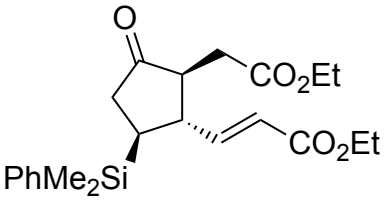
IR(neat)ν 2982, 1730, 1656, 1373, 1308, 1270, 1245, 1188, 1154, 1096, 1034, 1011, 737 cm^{-1}

$[\alpha]_D^{26}$ +105.7 (*c* 0.80, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, minor isomer t_R = 43.9 min, major isomer t_R = 72.2 min) (96% *ee*).

R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.25

Ethyl (*E*)-3-((1*R*,2*R*,5*R*)-5-(dimethyl(phenyl)silyl)-2-(2-ethoxy-2-oxoethyl)-3-oxocyclopentyl)acrylate



Yield: 90% (36.2 mg)

Physical State: White solid (m.p. 111~112 ºC)

^{1}H NMR ($CDCl_3$) δ 0.323 (s, 3H), 0.328 (s, 3H), 1.17 (t, *J* = 7.2 Hz, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.48 (ddd, *J* = 8.4, 11.6, 13.2 Hz, 1H), 2.11 (dd, *J* = 13.2, 18.8 Hz, 1H), 2.30 (dddd, *J* = 1.2, 5.2, 6.0, 9.2 Hz, 1H), 2.40 (ddd, *J* = 1.2, 8.4, 18.4 Hz, 1H), 2.43 (dd, *J* = 5.2, 17.2 Hz, 1H), 2.50 (dd, *J* = 6.0, 17.2 Hz, 1H), 2.58 (q, *J* = 11.6 Hz, 1H), 4.02 (dq, *J* = 1.6, 7.2 Hz, 2H), 4.15 (dq, *J* = 1.6, 7.2 Hz, 2H), 5.74 (d, *J* = 15.6 Hz, 1H), 6.68 (dd, *J* = 9.6, 15.6 Hz, 1H), 7.32-7.35 (m, 3H), 7.43-7.45 (m, 2H)

^{13}C NMR ($CDCl_3$) δ 216.4, 171.2, 165.6, 149.3, 136.3, 133.8, 133.8, 129.4, 129.4, 127.9, 122.6, 60.7, 60.3, 52.4, 47.3, 39.2, 31.7, 27.5, 14.1, 14.0, -4.15, -4.25

HRMS (ESI): $[M+Na]^+$ calcd for $C_{22}H_{30}O_5SiNa$: 425.1755, found: 425.1758

IR(neat)ν 1734, 1653, 1428, 1371, 1348, 1253, 1179, 1113, 1040, 820, 737, 702 cm^{-1}

$[\alpha]_D^{26}$ +98.79 (*c* 2.1, $CHCl_3$)

The enantiomeric ratio was determined by HPLC using CHIRALPACK ID (hexane/^{i}PrOH = 10:1; flow rate 1.0 ml/min, major isomer t_R = 15.0 min, minor isomer t_R = 19.2 min) (>99% *ee*).

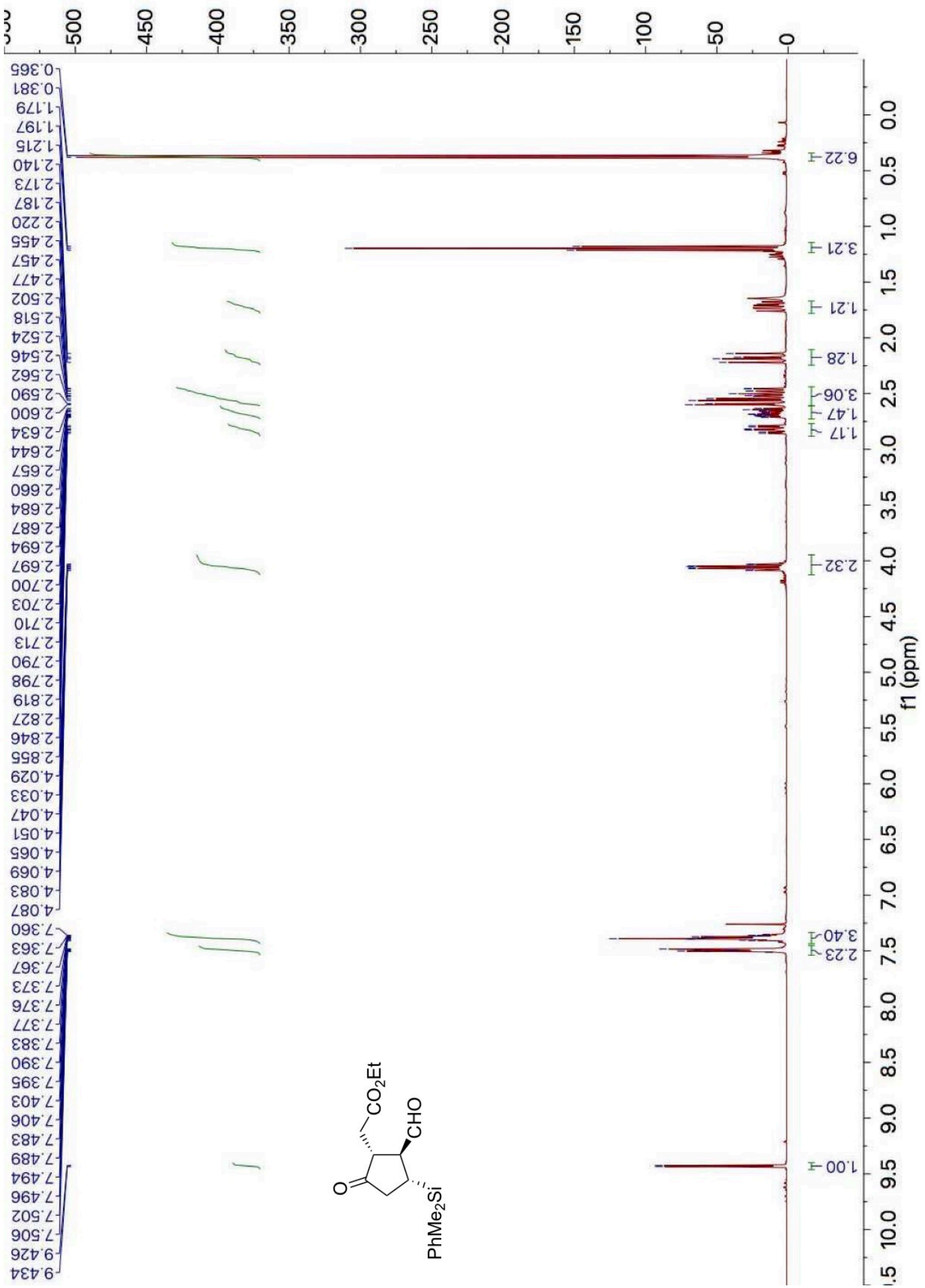
R_f (^{n}Hexane: EtOAc = 3:1, color reagent: Hanessian's stain reagent): 0.50

3. References

[1] M. G. McLaughlin and M. J. Cook, *Chem. Commun*., 2011, **47**, 11104; R. Ostwald, P.-Y. Chavant, H. Stadtmüller and P. Knochel, *J. Org. Chem*. 1994, **59**, 4143.

[2] F. Yin, A. Garifullina and F. Tanaka, *Org. Biomol. Chem*., 2017, **15**, 6089.

[3] C. A. González-González, A. Fuentes-Benítes, E. Cuevas-Yáñez, D. Corona-Becerril, C. González-Romeroa and D. González-Calderón, *Tetrahedron Lett*., 2013, **54**, 2776.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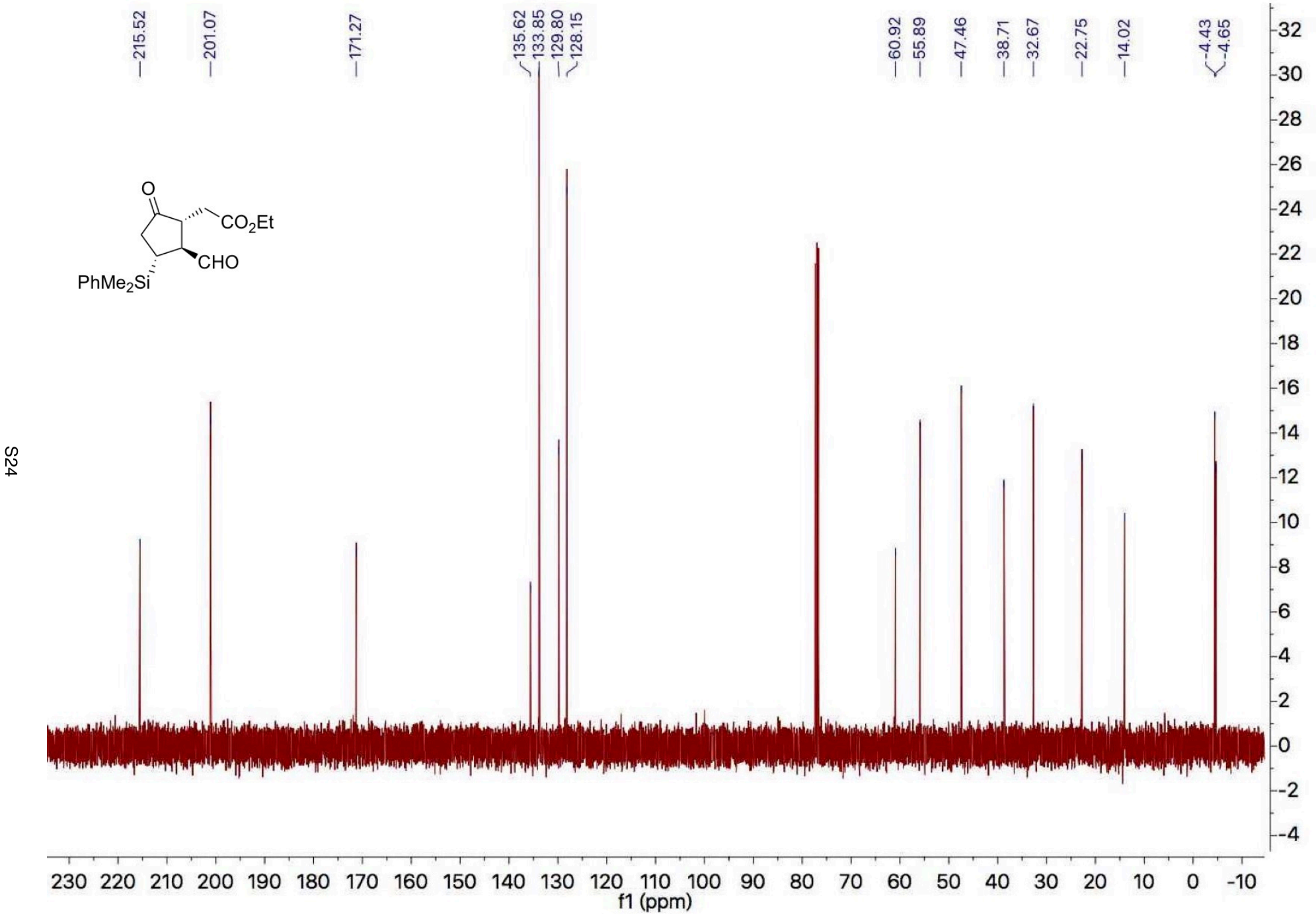
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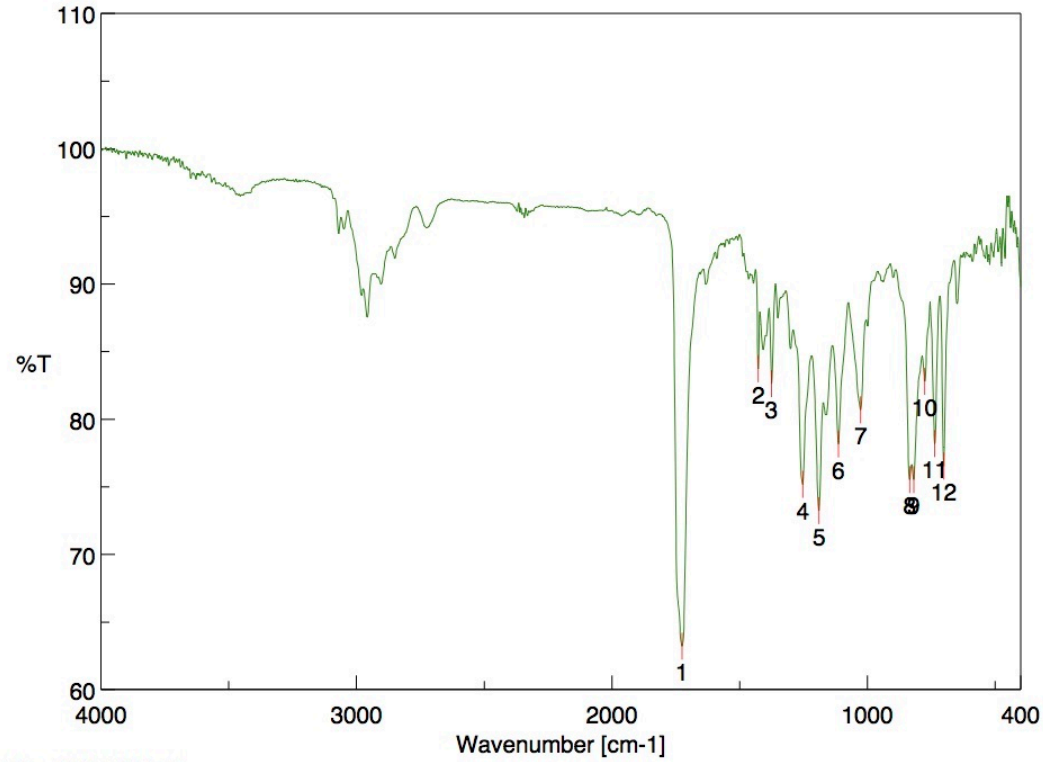
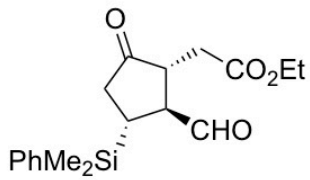
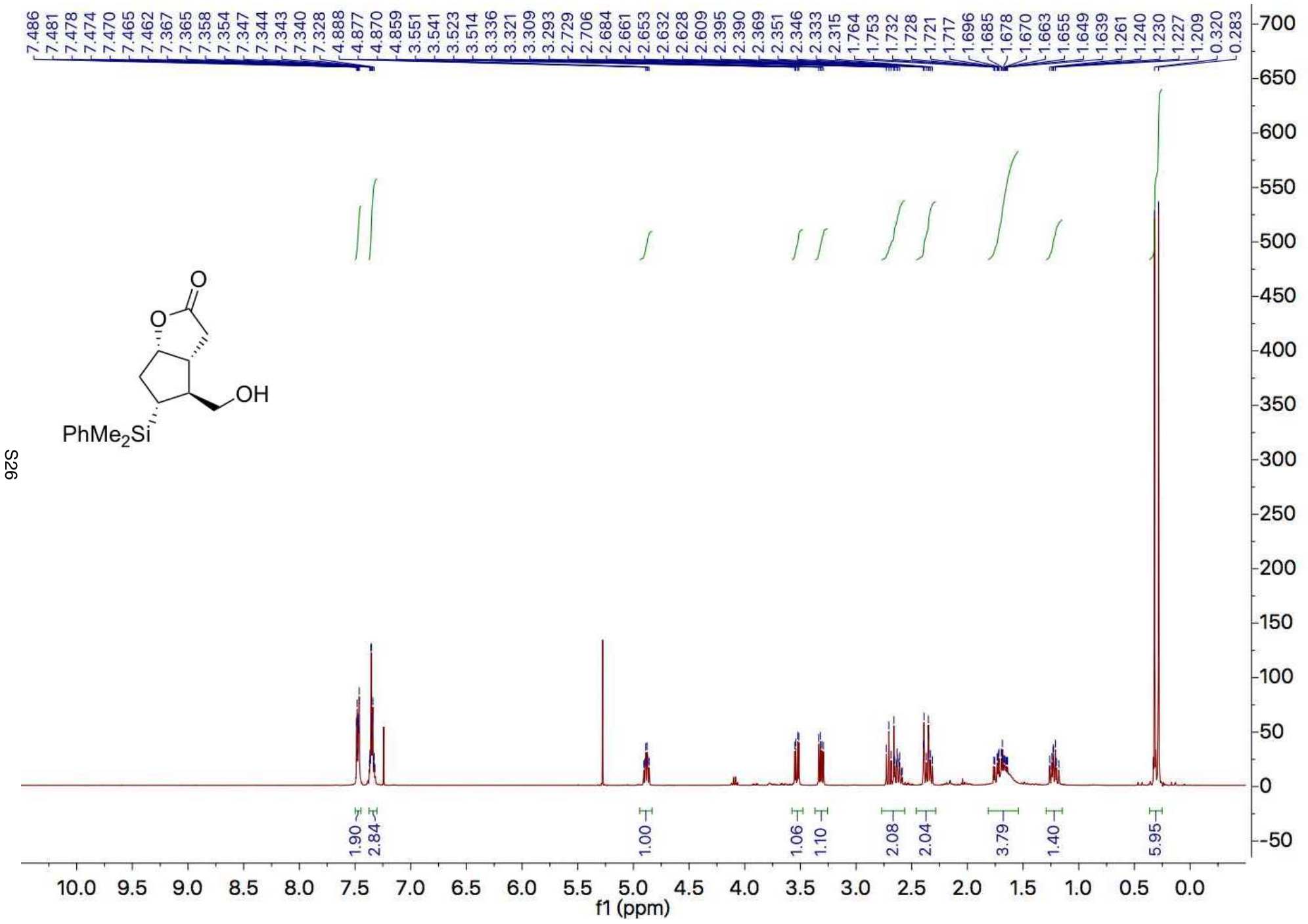
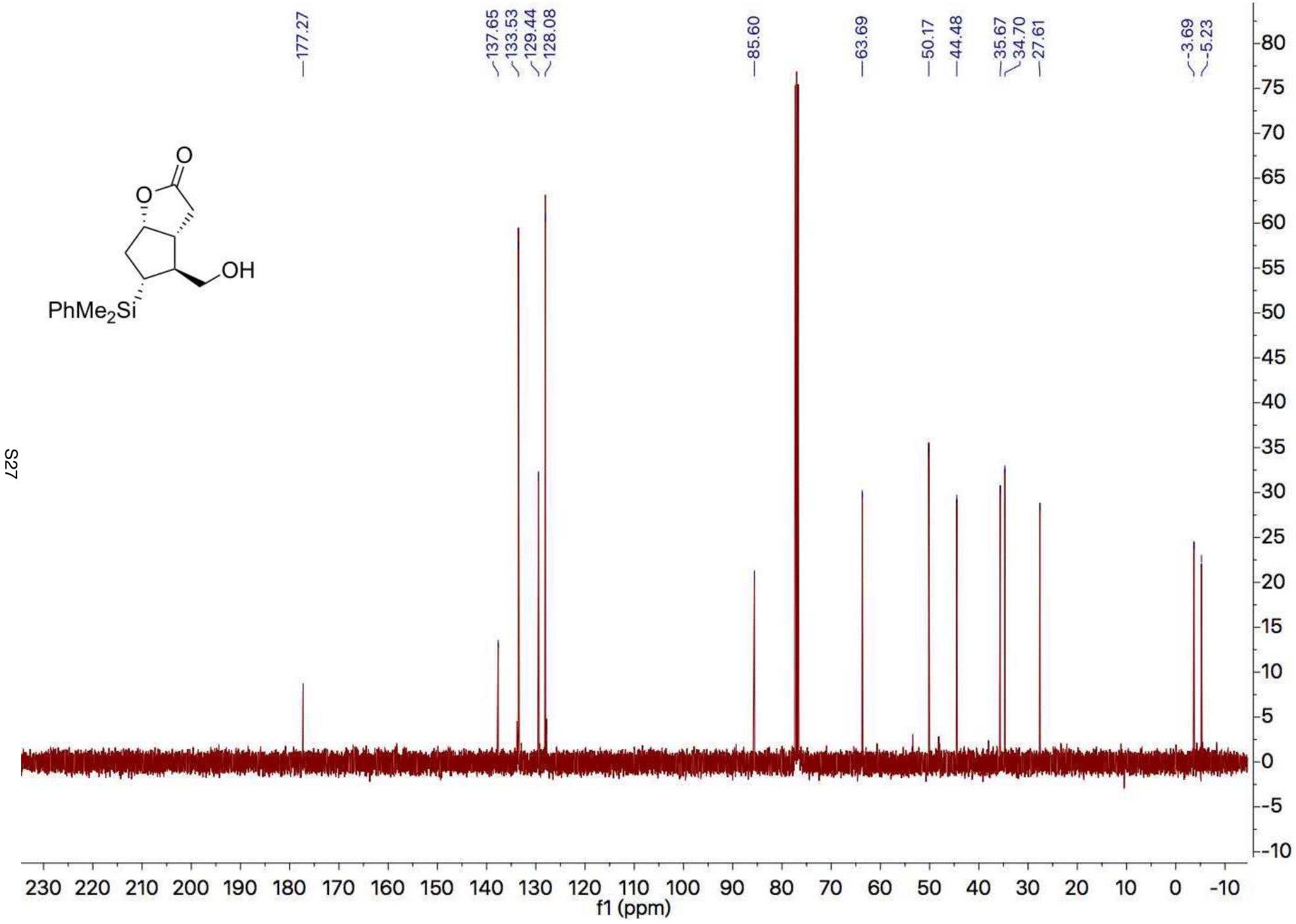


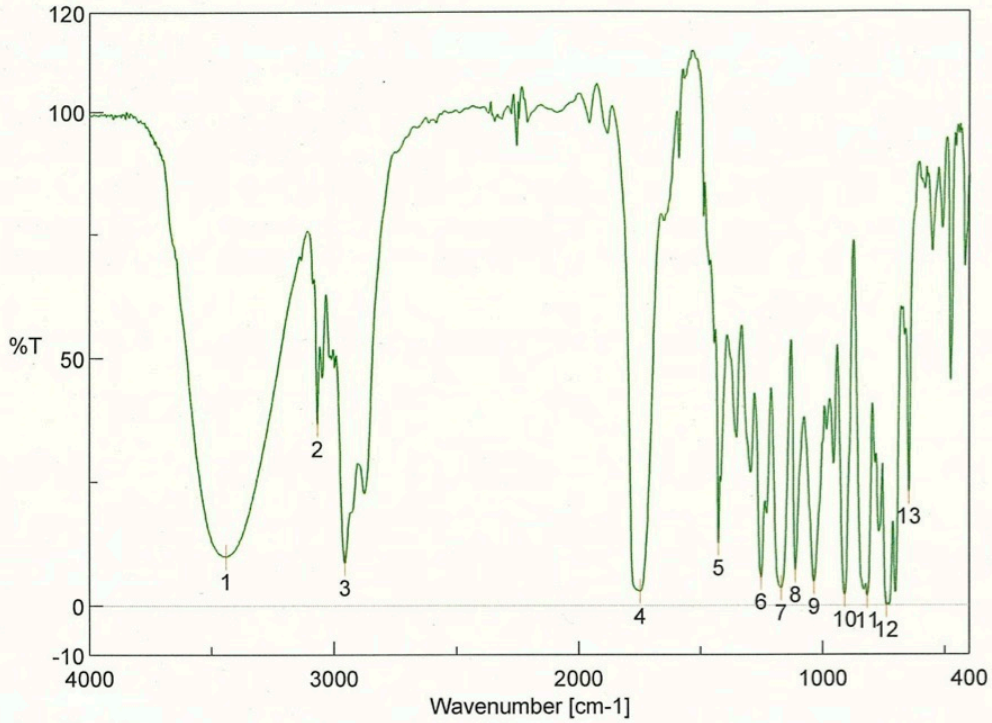
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1725.01	63.2039	2	1427.07	83.7189
3	1375	82.6205	4	1253.5	75.1643
5	1189.86	73.2347	6	1113.69	78.17
7	1026.91	80.6785	8	835.026	75.5319
9	818.634	75.5323	10	775.244	82.79
11	736.674	78.1806	12	700.998	76.5157



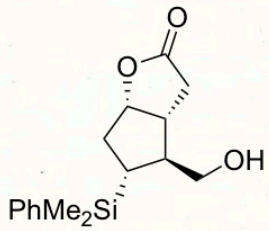

7.486 7.481 7.478 7.474 7.470 7.465 7.462 7.367 7.365 7.358 7.354 7.347 7.344 7.343 7.340 7.328 4.888 4.877 4.870 4.859 3.551 3.541 3.523 3.514 3.336 3.321 3.309 3.293 2.729 2.706 2.684 2.661 2.653 2.632 2.628 2.609 2.395 2.390 2.369 2.351 2.346 2.333 2.315 1.764 1.753 1.732 1.728 1.721 1.717 1.696 1.685 1.678 1.670 1.663 1.655 1.649 1.639 1.261 1.240 1.230 1.227 1.209 0.320 0.283
O
O
OH
PhMe2Si
1.90
2.84
1.00
1.06
1.10
2.08
2.04
3.79
1.40
5.95
f1 (ppm)

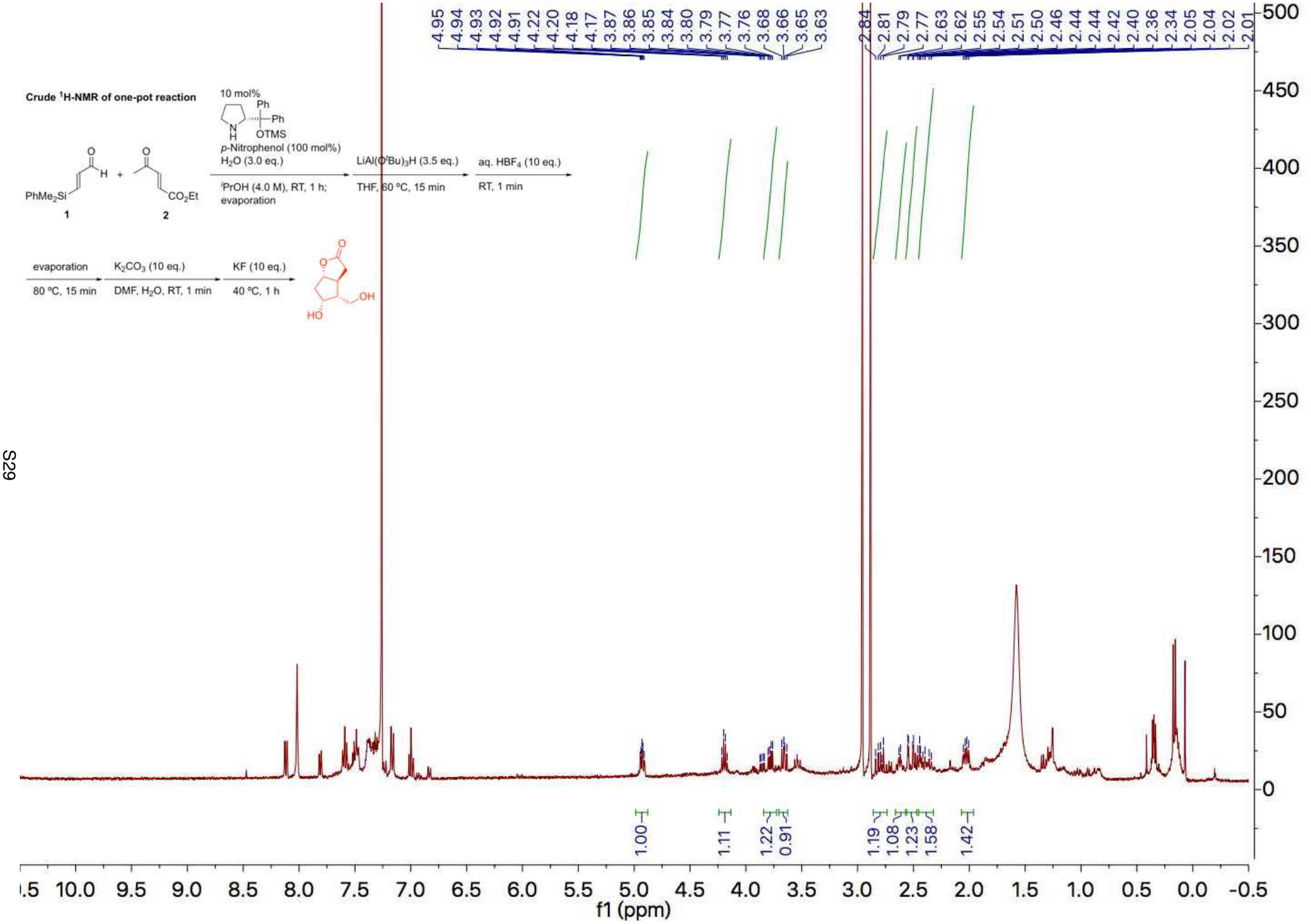

177.27
137.65
133.53
129.44
128.08
85.60
63.69
50.17
44.48
35.67
34.70
27.61
3.69
5.23
O
O
OH
PhMe2Si
f1 (ppm)

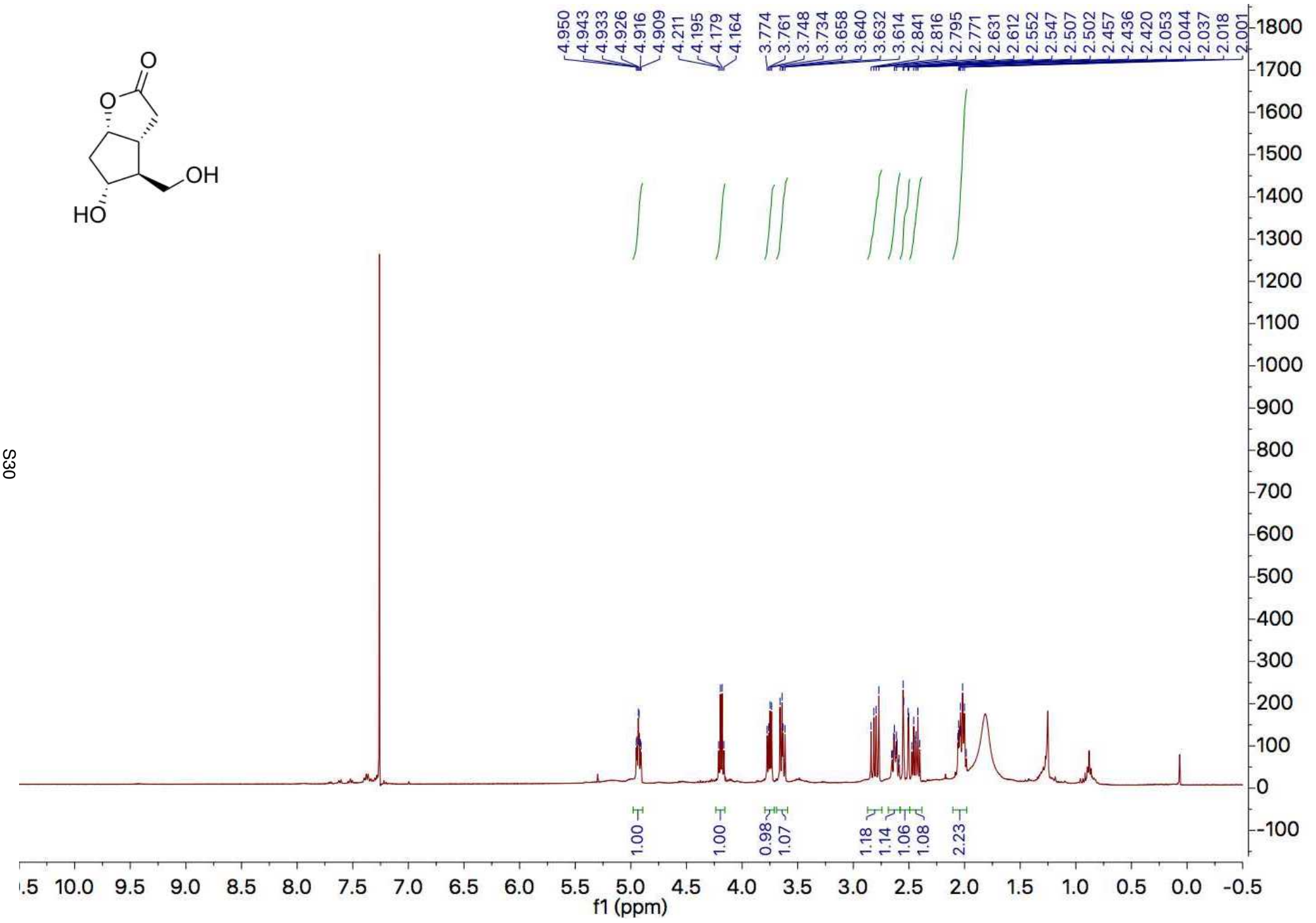


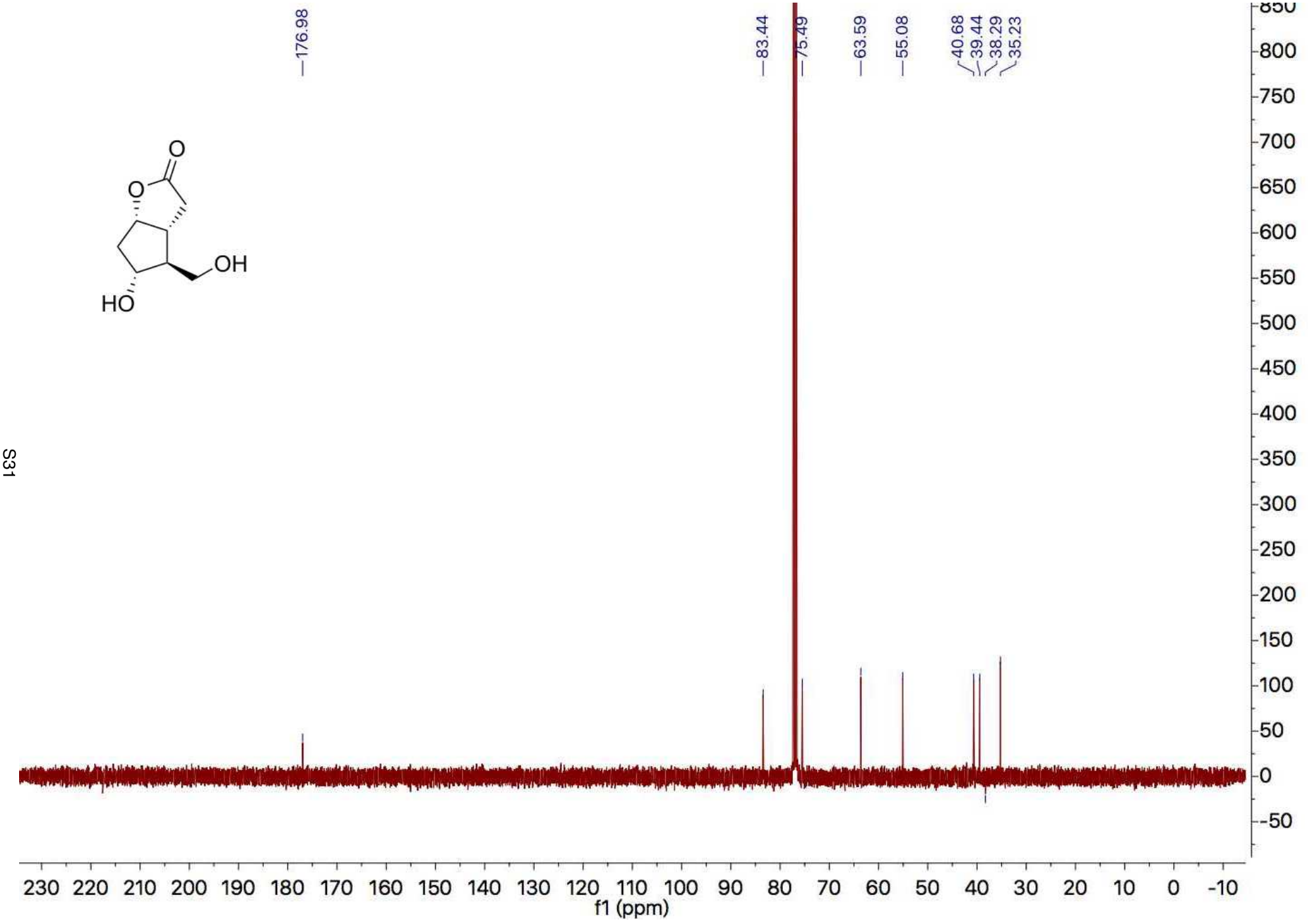
[ピーク検出結果]

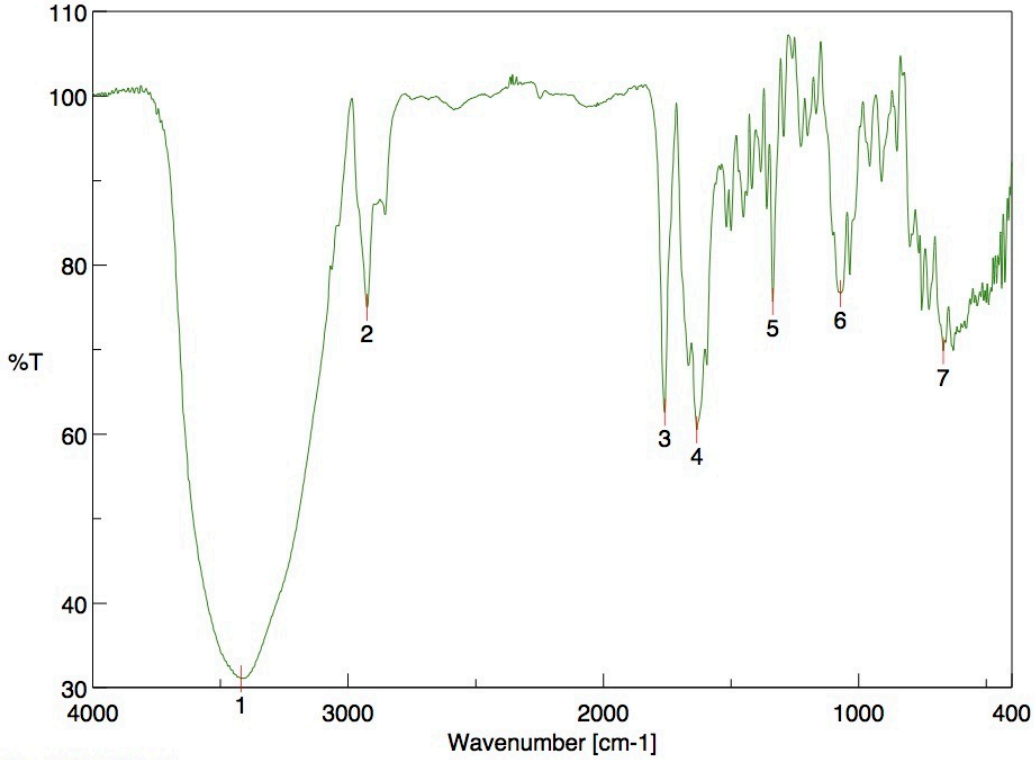
No.	位置	強度	No.	位置	強度
1	3441.35	9.8041	2	3069.16	36.6294
3	2956.34	8.57681	4	1748.16	2.81947
5	1427.07	12.614	6	1251.58	5.68628
7	1170.58	3.53032	8	1111.76	7.15859
9	1036.55	4.74536	10	910.236	2.13359
11	817.67	1.89086	12	739.567	−1.76516E−038
13	647.965	22.8932			




Crude ^{1}H-NMR of one-pot reaction
10 mol%
Ph
Ph
N
H
OTMS
p-Nitrophenol (100 mol%)
H_2O (3.0 eq.)
^{i}PrOH (4.0 M), RT, 1 h; evaporation
$LiAl(O^tBu)_3H$ (3.5 eq.)
THF, 60 °C, 15 min
aq. HBF_4 (10 eq.)
RT, 1 min
$PhMe_2Si$
CO_2Et
1
2
evaporation
80 °C, 15 min
K_2CO_3 (10 eq.)
DMF, H_2O, RT, 1 min
KF (10 eq.)
40 °C, 1 h
OH
HO
4.95 4.94 4.93 4.92 4.91 4.22 4.20 4.18 4.17 3.87 3.86 3.85 3.84 3.80 3.79 3.77 3.76 3.68 3.66 3.65 3.63
2.84 2.81 2.79 2.77 2.63 2.62 2.55 2.54 2.51 2.50 2.46 2.44 2.44 2.42 2.40 2.36 2.34 2.05 2.04 2.02 2.01
1.00 1.11 1.22 0.91 1.19 1.08 1.23 1.58 1.42
f1 (ppm)

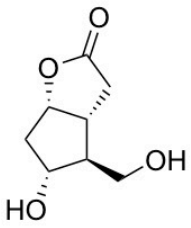

O
O
OH
HO
4.950
4.943
4.933
4.926
4.916
4.909
4.211
4.195
4.179
4.164
3.774
3.761
3.748
3.734
3.658
3.640
3.632
3.614
2.841
2.816
2.795
2.771
2.631
2.612
2.552
2.547
2.507
2.502
2.457
2.436
2.420
2.053
2.044
2.037
2.018
2.001
1.00
1.00
0.98
1.07
1.18
1.14
1.06
1.08
2.23
f1 (ppm)

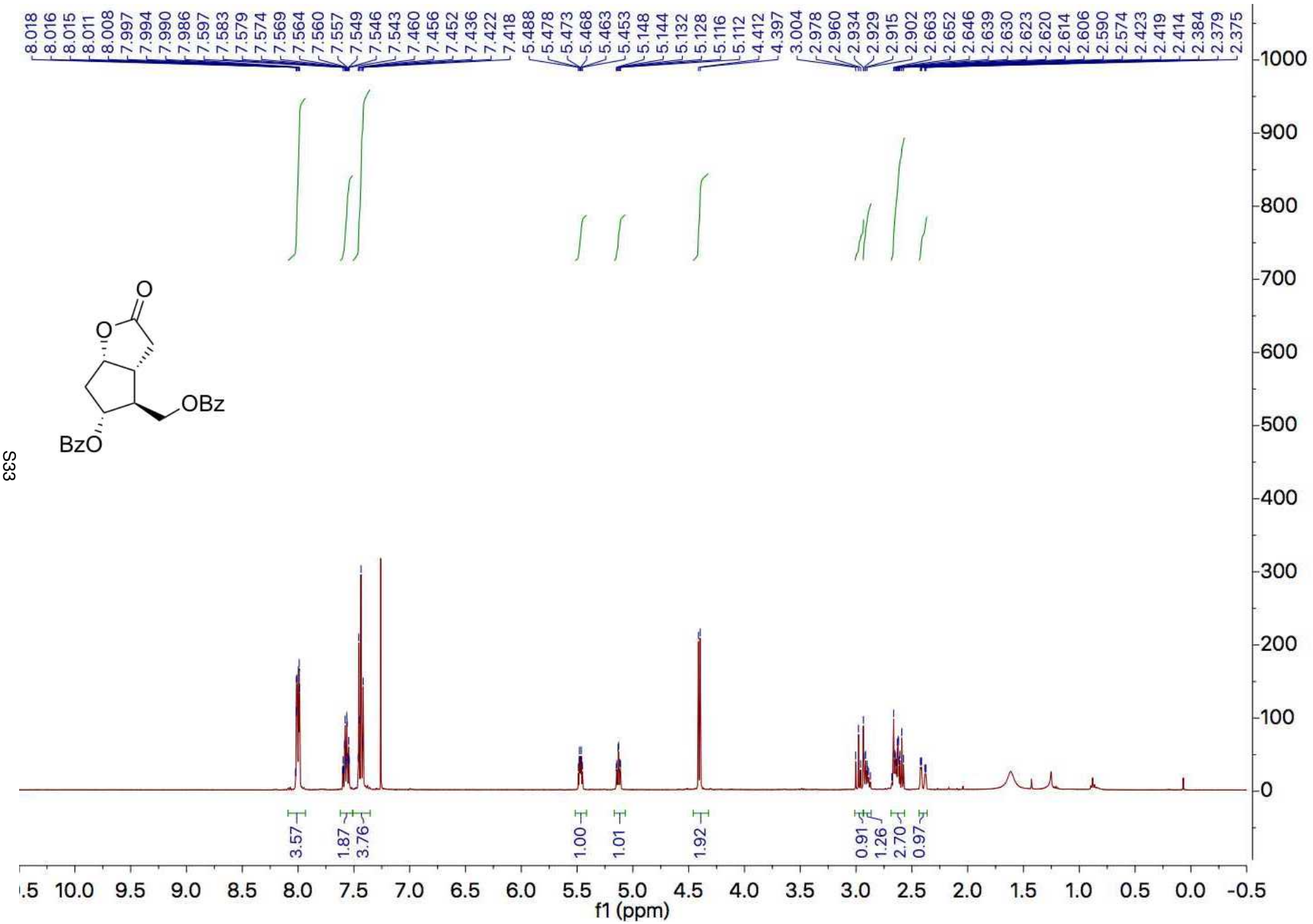

176.98
83.44
75.49
63.59
55.08
40.68
39.44
38.29
35.23
O
O
OH
HO
f1 (ppm)

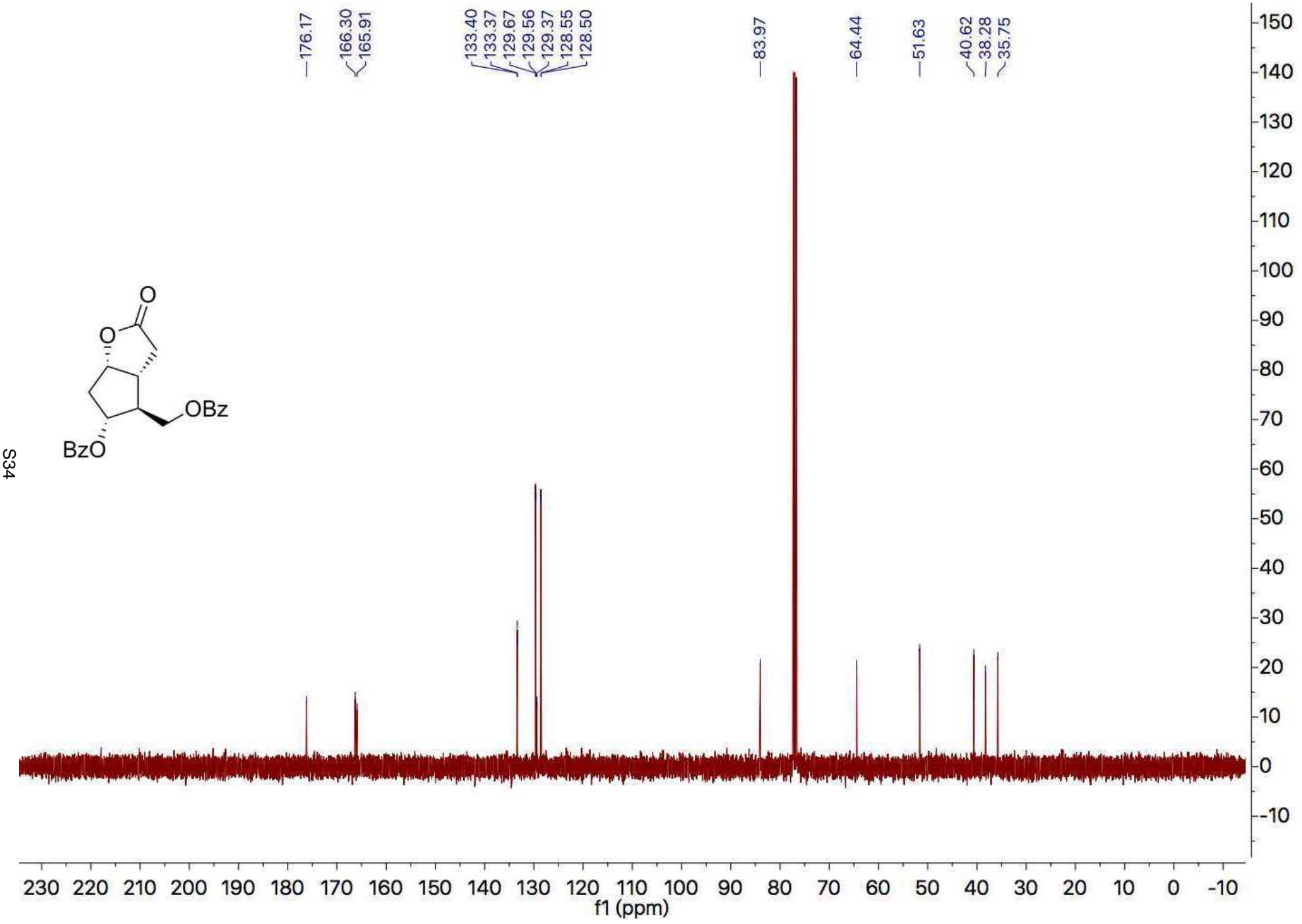


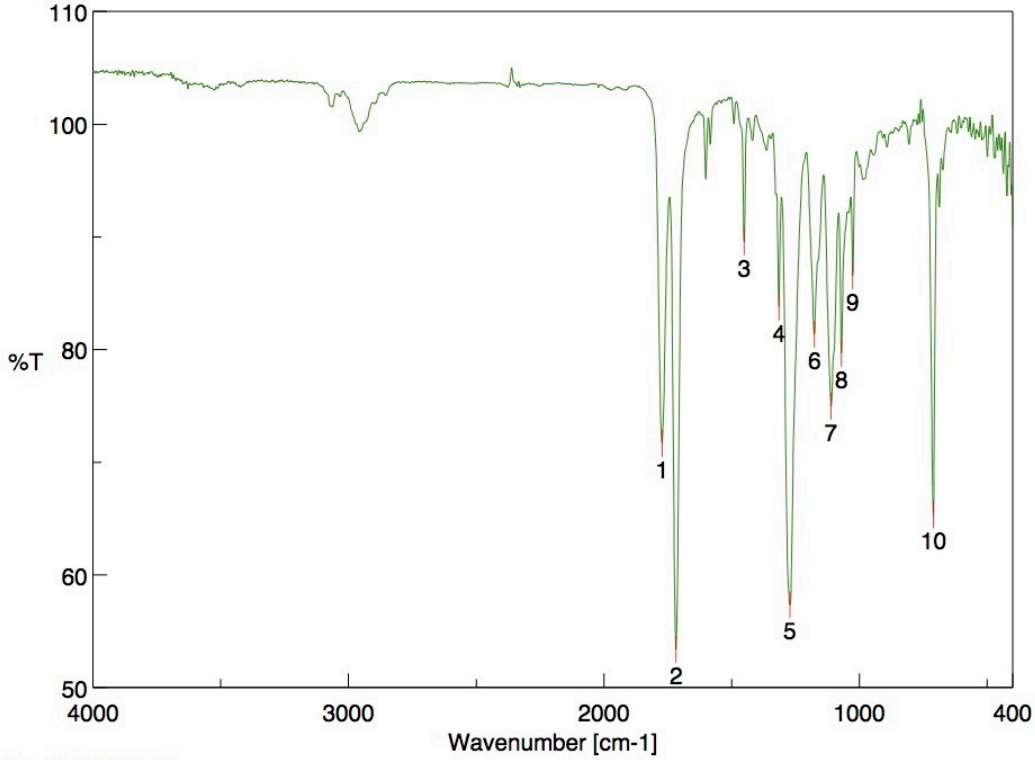
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	3419.17	31.0254	2	2925.48	74.9757
3	1759.73	62.5587	4	1634.38	60.4869
5	1336.43	75.6638	6	1071.26	76.6164
7	668.214	69.8556			



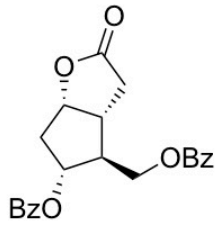

8.018
8.016
8.015
8.011
8.008
7.997
7.994
7.990
7.986
7.597
7.583
7.579
7.574
7.569
7.564
7.560
7.557
7.549
7.546
7.543
7.460
7.456
7.452
7.436
7.422
7.418
5.488
5.478
5.473
5.468
5.463
5.453
5.148
5.144
5.132
5.128
5.116
5.112
4.412
4.397
3.004
2.978
2.960
2.934
2.929
2.915
2.902
2.663
2.652
2.646
2.639
2.630
2.623
2.620
2.614
2.606
2.590
2.574
2.423
2.419
2.414
2.384
2.379
2.375
O
O
OBz
BzO
3.57
1.87
3.76
1.00
1.01
1.92
0.91
1.26
2.70
0.97
f1 (ppm)


176.17
166.30
165.91
133.40
133.37
129.67
129.56
129.37
128.55
128.50
83.97
64.44
51.63
40.62
38.28
35.75
O
O
OBz
BzO
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



[ピーク検出結果]

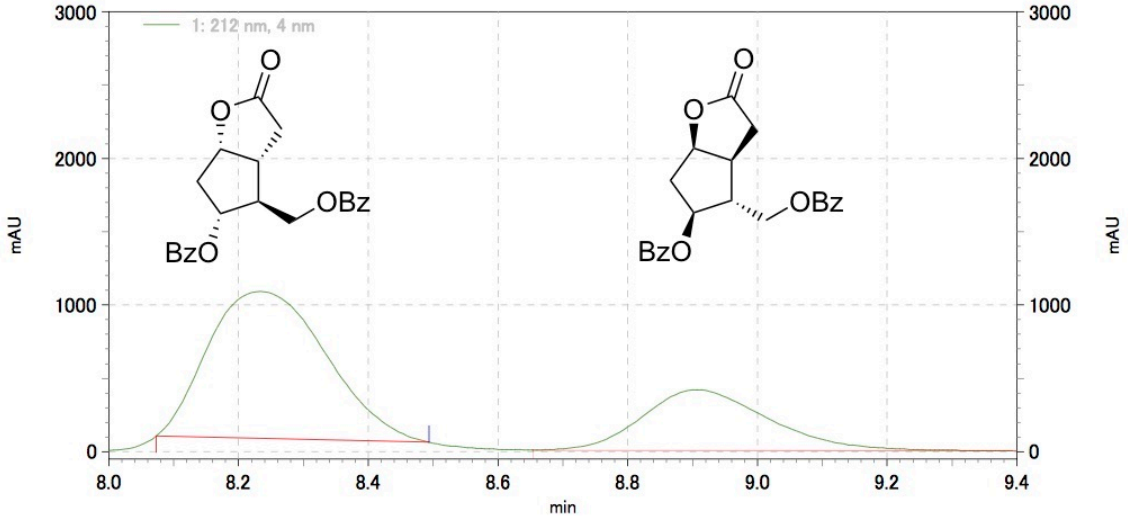
No.	位置	強度	No.	位置	強度
1	1772.26	71.6605	2	1718.26	53.3946
3	1451.17	89.5658	4	1315.21	83.7841
5	1271.82	57.3493	6	1176.36	81.3353
7	1110.8	74.9736	8	1070.3	79.667
9	1025.94	86.5182	10	710.64	65.3257



面積%レポート

ﾃﾞｰﾀﾌｧｲﾙ名 : C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012129-IF-45vs55-corey-lactone-racemi.dat
ﾒｿｯﾄﾞﾌｧｲﾙ名: C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥200vs1 0.5ml.met
ﾕｰｻﾞｰ名: System
分析日時: 2019/07/30 13:57:10
印刷日時: 2019/07/30 15:26:03

p



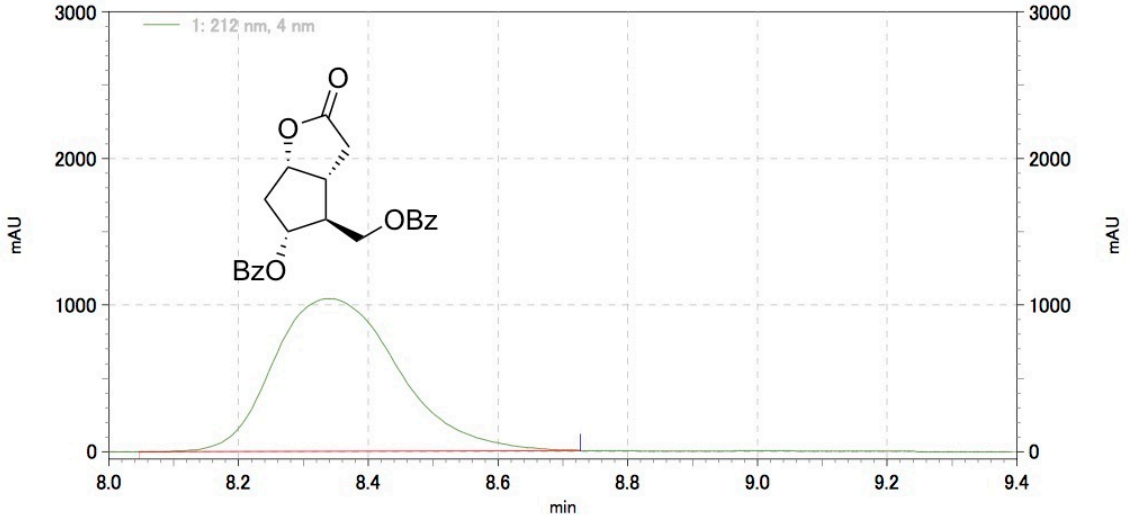
1: 212 nm, 4 nm結果

Pk #	名前	保持時間	面積	面積%	ﾍﾞｰｽﾗｲﾝｺｰﾄﾞ
1		8.23	50691322	69.690	MM
2		8.91	22047087	30.310	MM
ﾄｰﾀﾙ			72738409	100.000	

面積%レポート

ﾃﾞｰﾀﾌｧｲﾙ名 : C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012129-IF-45vs55-corey-lactone-chiral-Re.dat
ﾒｿｯﾄﾞﾌｧｲﾙ名: C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥200vs1 0.5ml.met
ﾕｰｻﾞｰ名: System
分析日時: 2019/07/30 14:47:34
印刷日時: 2019/07/30 15:28:02

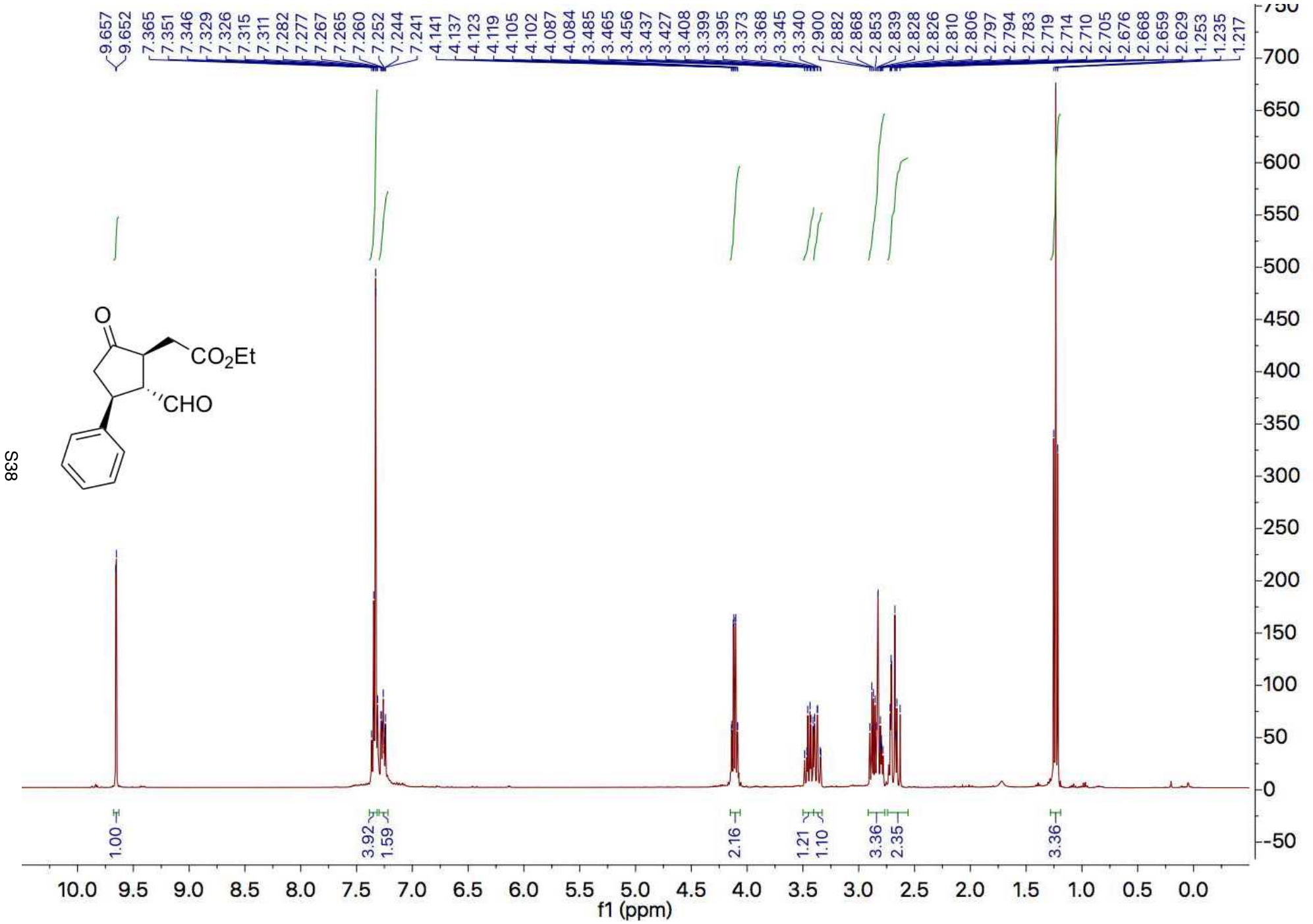
p

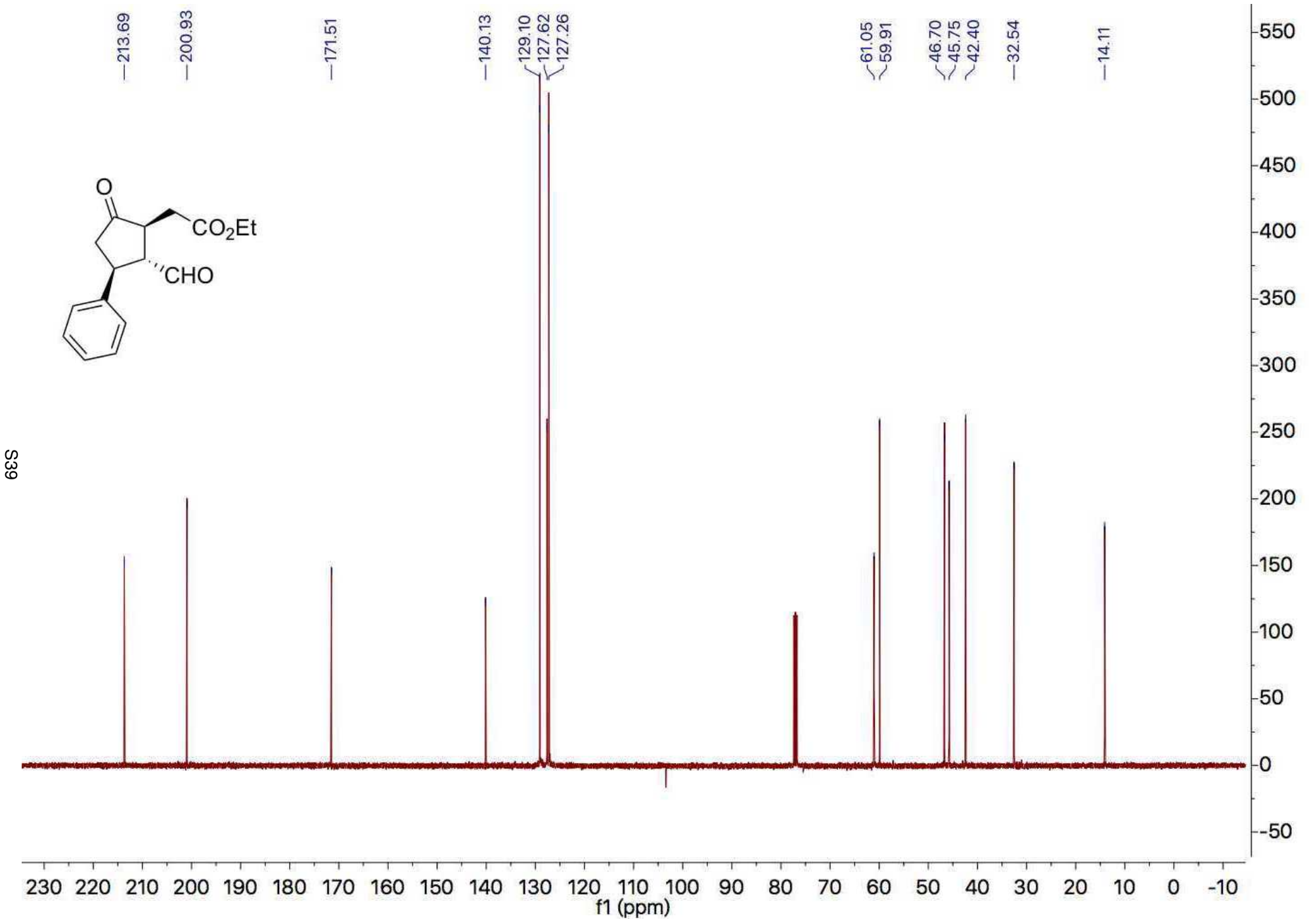


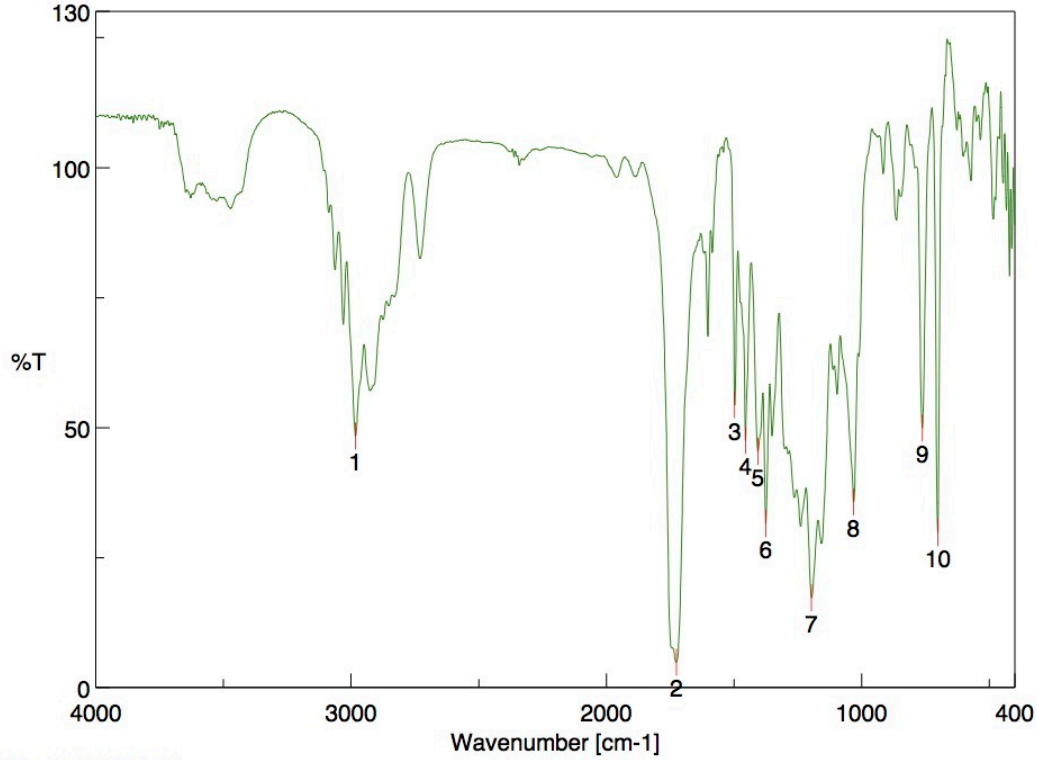
1: 212 nm, 4 nm結果

Pk #	名前	保持時間	面積	面積%	ﾍﾞｰｽﾗｲﾝｺｰﾄﾞ
1		8.34	55337534	100.000	MM

ﾄｰﾀﾙ			55337534	100.000	

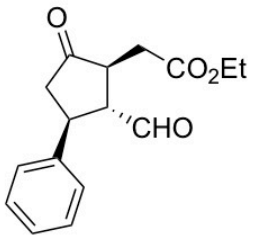

9.657
9.652
7.365
7.351
7.346
7.329
7.326
7.315
7.311
7.282
7.277
7.267
7.265
7.260
7.252
7.244
7.241
4.141
4.137
4.123
4.119
4.105
4.102
4.087
4.084
3.485
3.465
3.456
3.437
3.427
3.408
3.399
3.395
3.373
3.368
3.345
3.340
2.900
2.882
2.868
2.853
2.839
2.828
2.826
2.810
2.806
2.797
2.794
2.783
2.719
2.714
2.710
2.705
2.676
2.668
2.659
2.629
1.253
1.235
1.217
O
CO2Et
CHO
1.00
3.92
1.59
2.16
1.21
1.10
3.36
2.35
3.36
f1 (ppm)

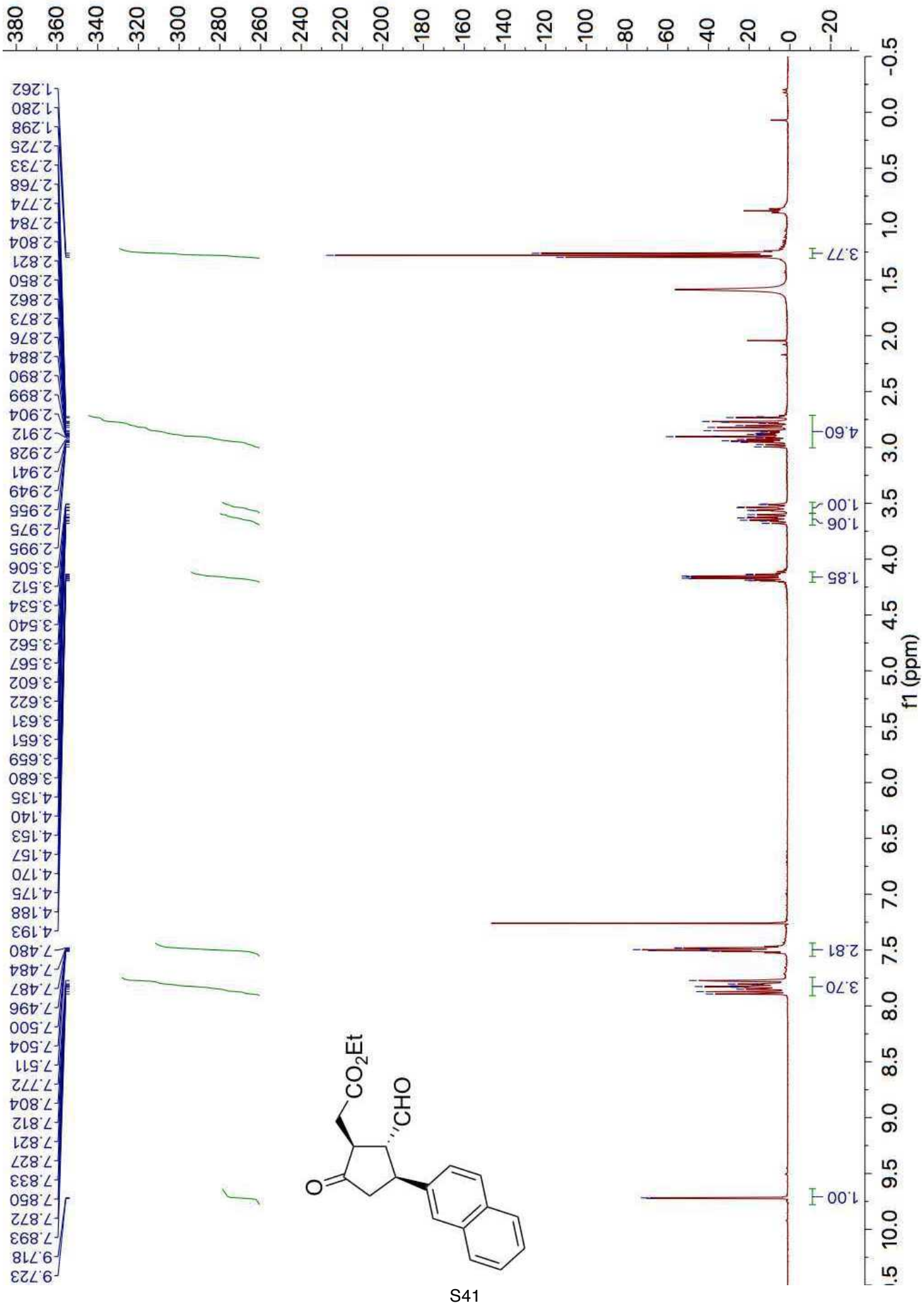

213.69
200.93
171.51
140.13
129.10
127.62
127.26
61.05
59.91
46.70
45.75
42.40
32.54
14.11
O
CO2Et
CHO
550
500
450
400
350
300
250
200
150
100
50
0
-50
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

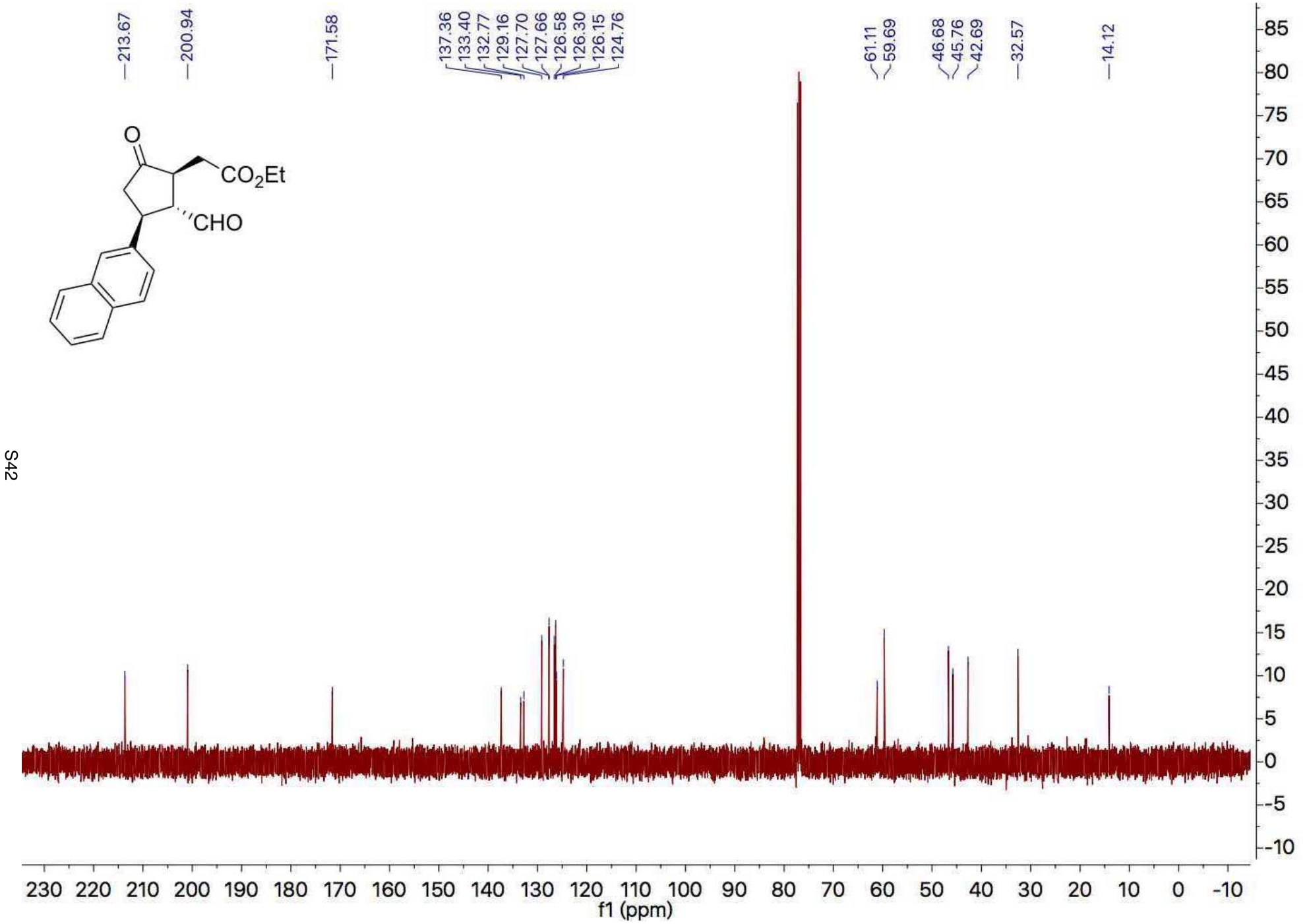


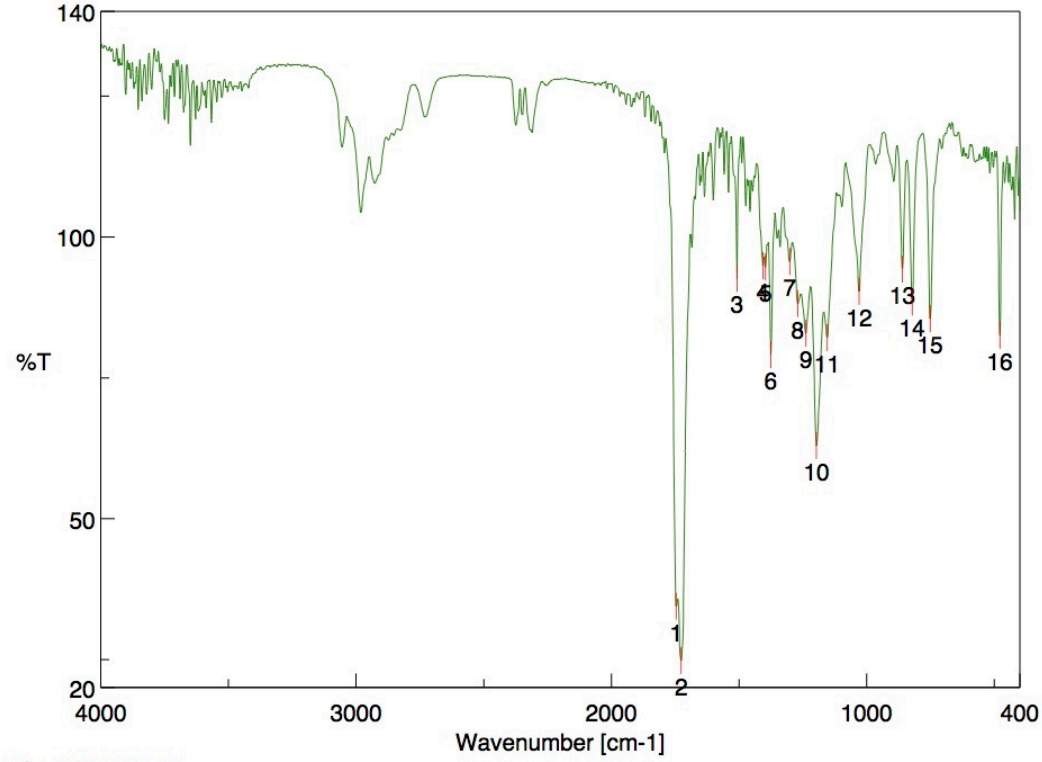
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	2982.37	48.3757	2	1725.98	4.84746
3	1497.45	54.3441	4	1455.03	47.4782
5	1405.85	45.3626	6	1375.96	31.4635
7	1196.61	17.2628	8	1031.73	35.6824
9	762.709	49.8992	10	701.962	29.7549



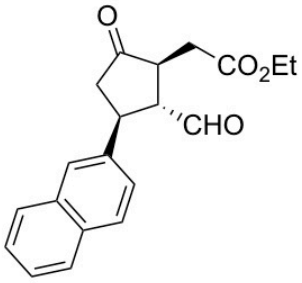

CO2Et
CHO
O
f1 (ppm)

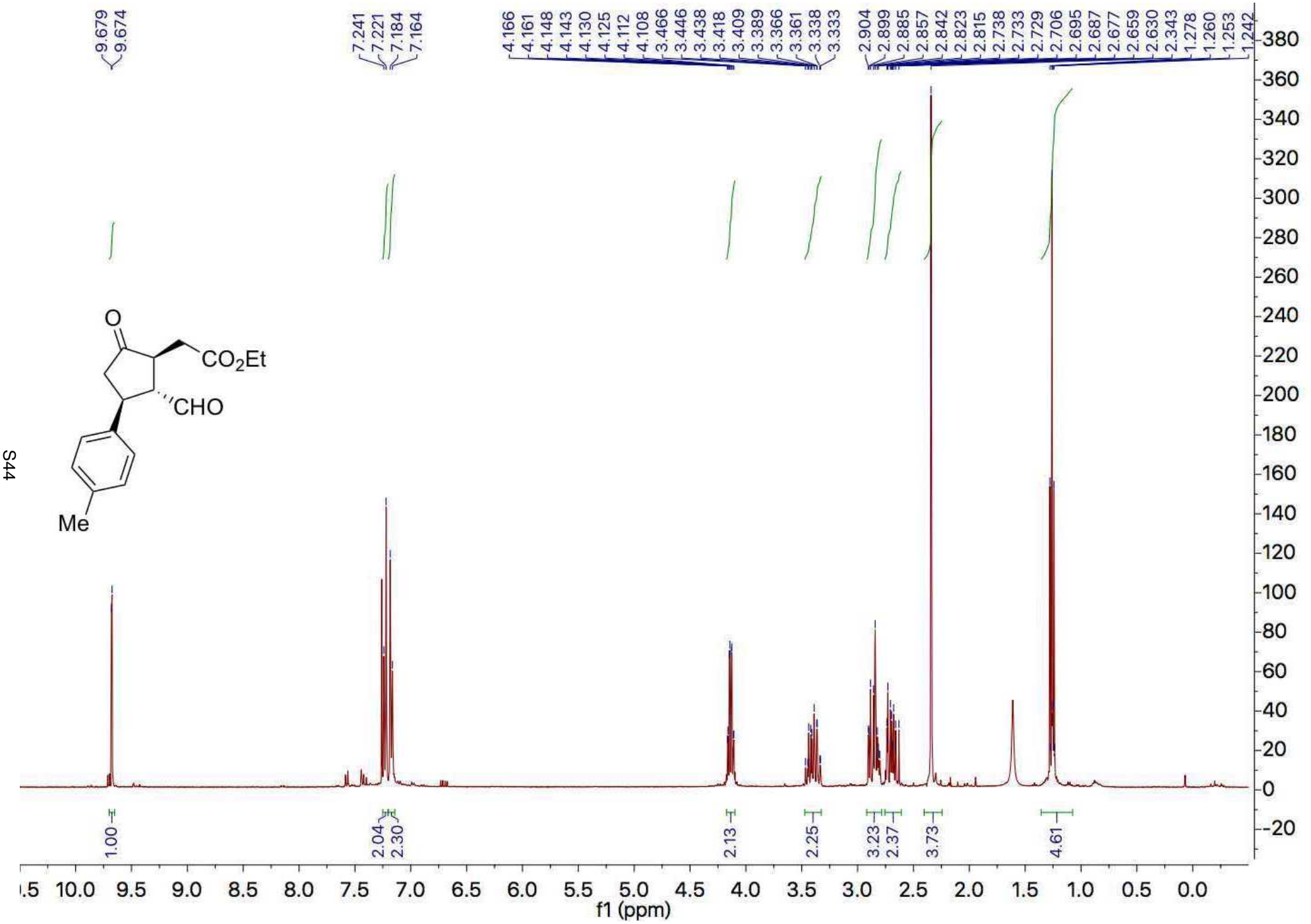

213.67
200.94
171.58
137.36
133.40
132.77
129.16
127.70
127.66
126.58
126.30
126.15
124.76
61.11
59.69
46.68
45.76
42.69
32.57
14.12
O
CO2Et
CHO
f1 (ppm)

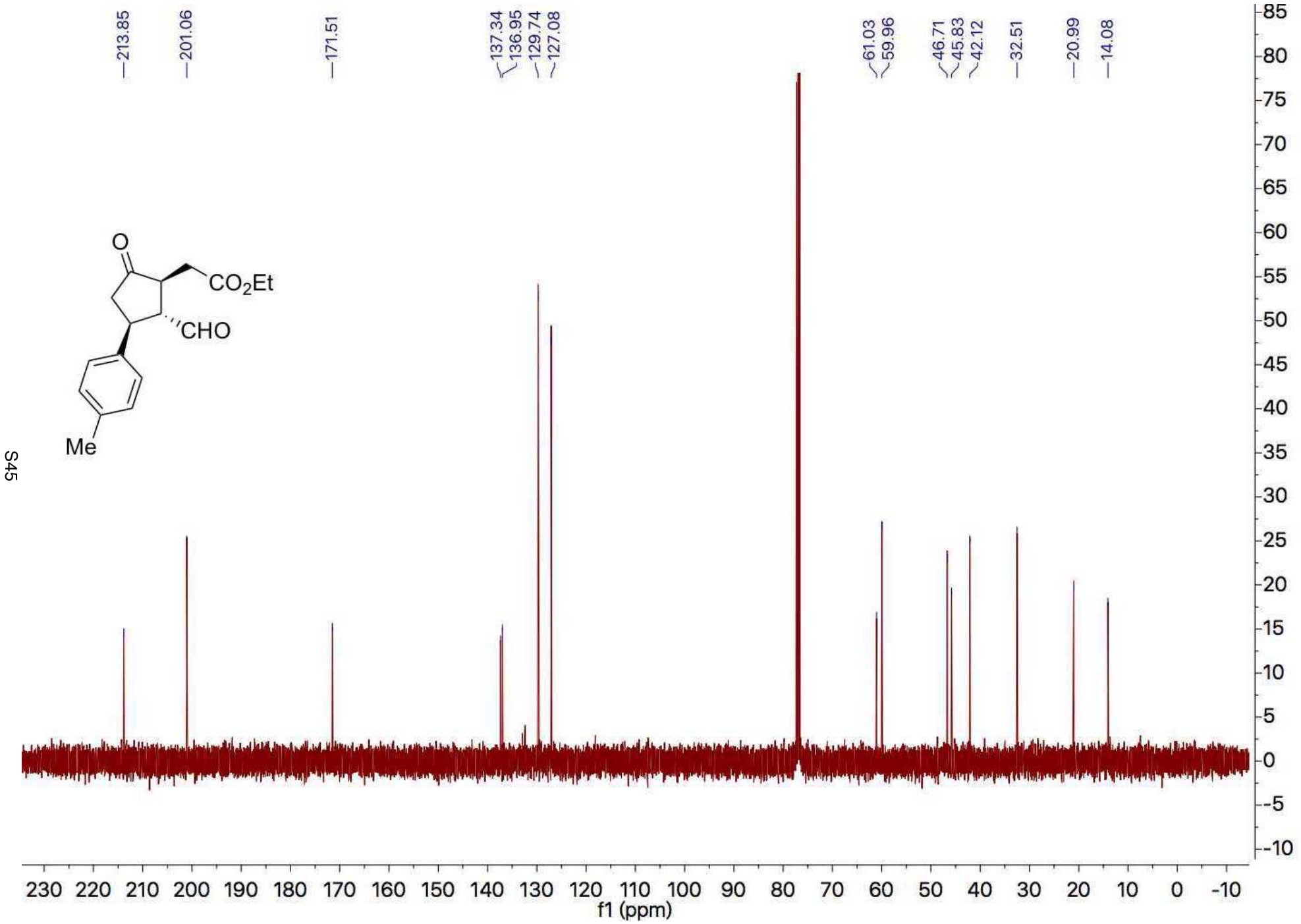


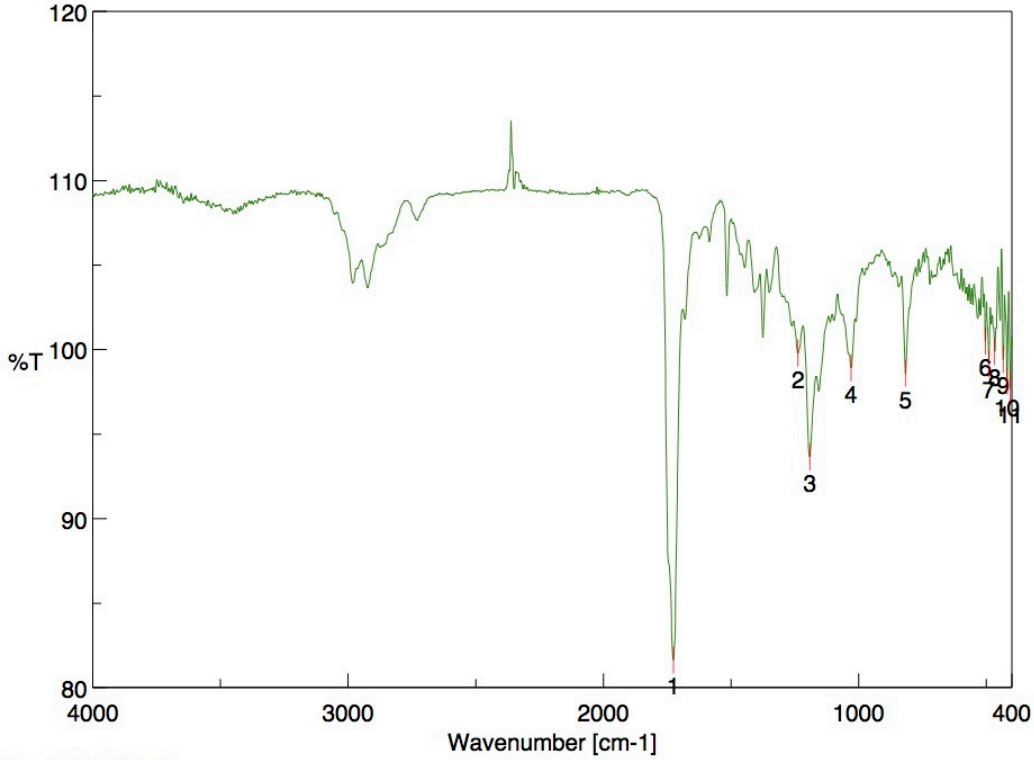
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1746.23	34.4241	2	1726.94	24.7776
3	1508.06	92.6066	4	1405.85	94.8681
5	1396.21	94.6292	6	1375	79.0962
7	1301.72	95.6301	8	1269.9	88.1492
9	1238.08	82.8522	10	1196.61	62.9595
11	1154.19	82.1396	12	1029.8	90.3213
13	860.096	94.2804	14	821.527	88.3949
15	751.138	85.4714	16	478.26	82.5147



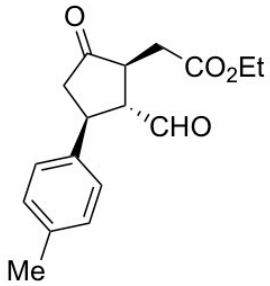

9.679
9.674
7.241
7.221
7.184
7.164
4.166
4.161
4.148
4.143
4.130
4.125
4.112
4.108
3.466
3.446
3.438
3.418
3.409
3.389
3.366
3.361
3.338
3.333
2.904
2.899
2.885
2.857
2.842
2.823
2.815
2.738
2.733
2.729
2.706
2.695
2.687
2.677
2.659
2.630
2.343
1.278
1.260
1.253
1.242
O
CO2Et
CHO
Me
1.00
2.04
2.30
2.13
2.25
3.23
2.37
3.73
4.61
f1 (ppm)

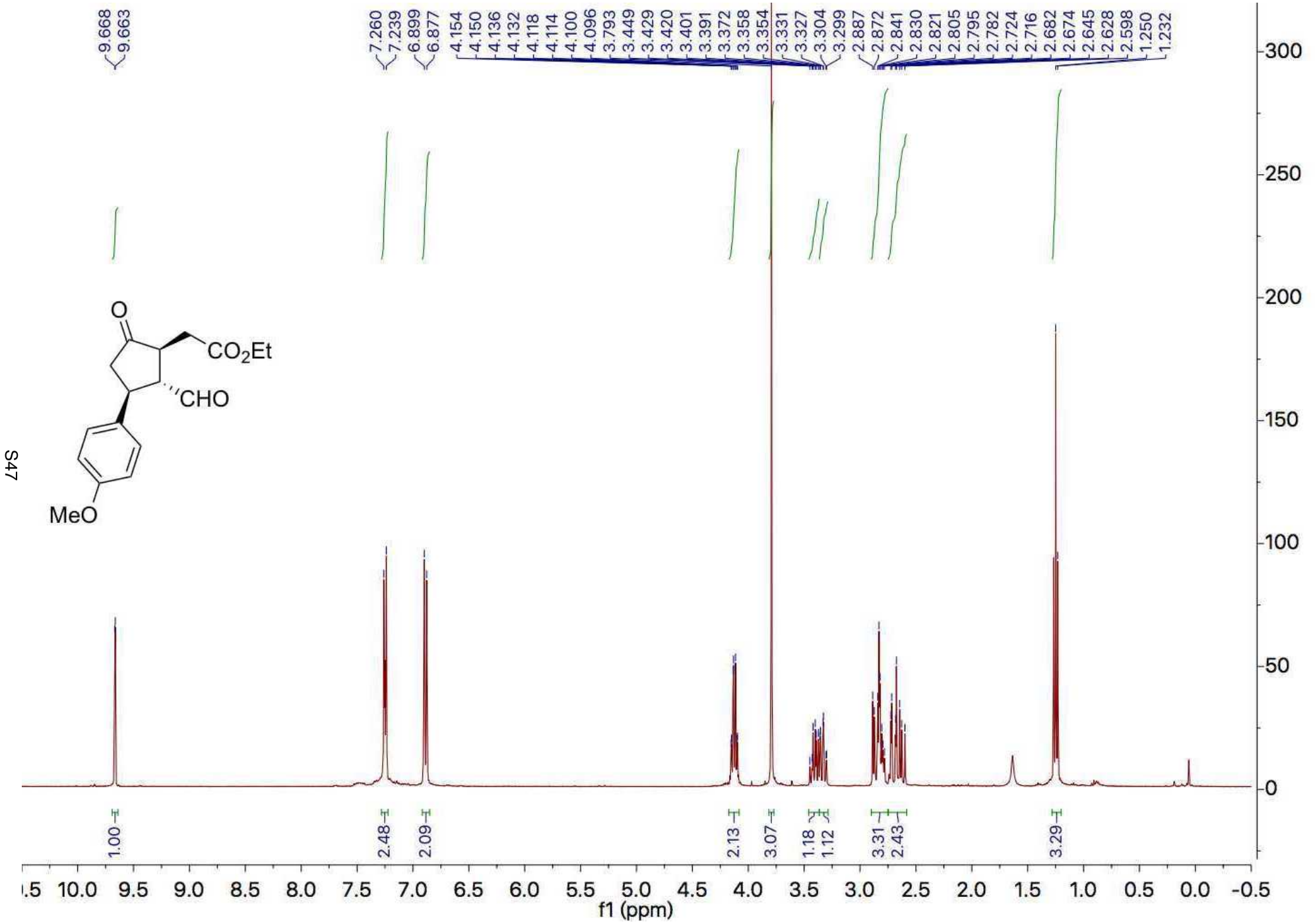

213.85
201.06
171.51
137.34
136.95
129.74
127.08
61.03
59.96
46.71
45.83
42.12
32.51
20.99
14.08
O
CO2Et
CHO
Me
f1 (ppm)

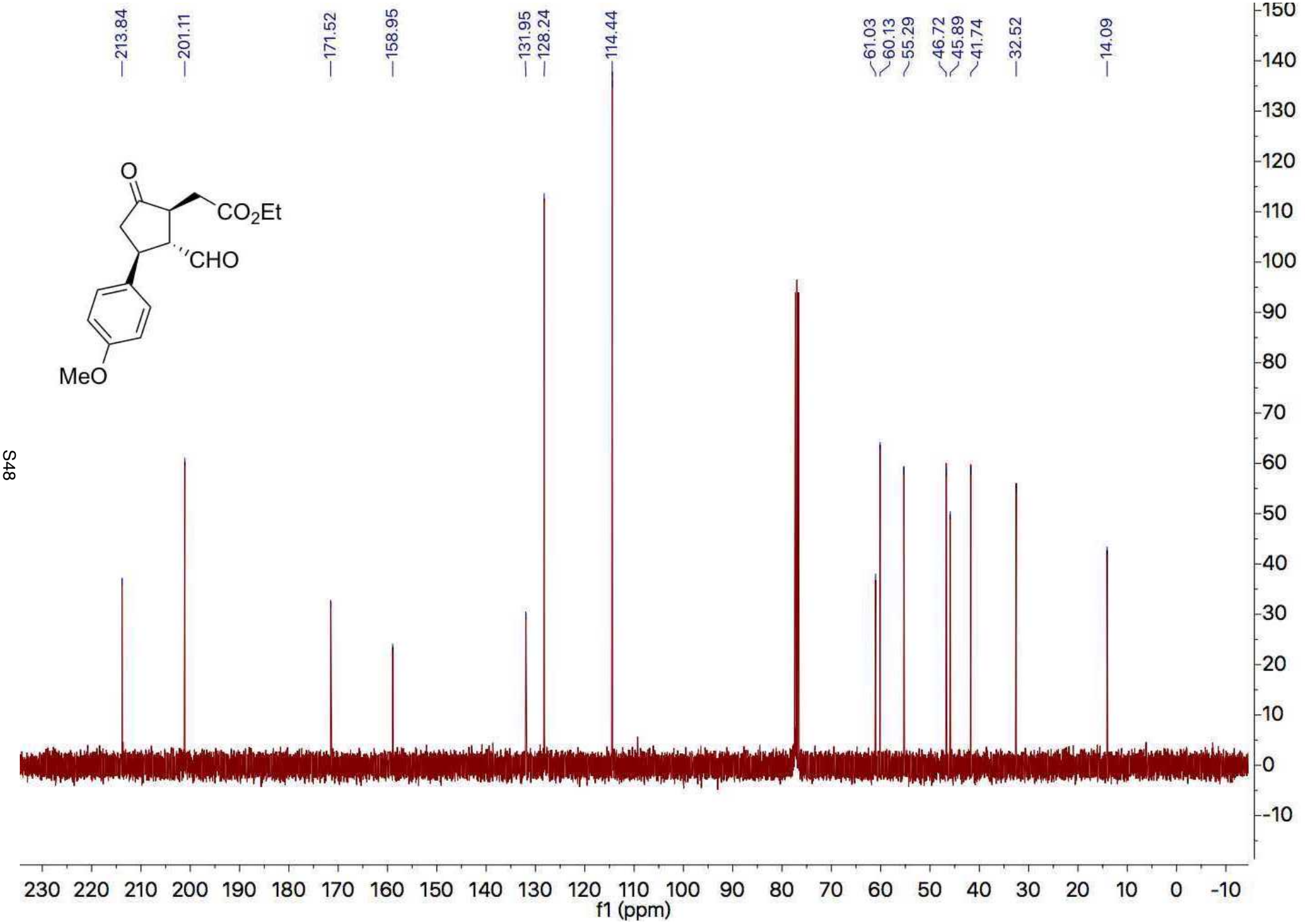


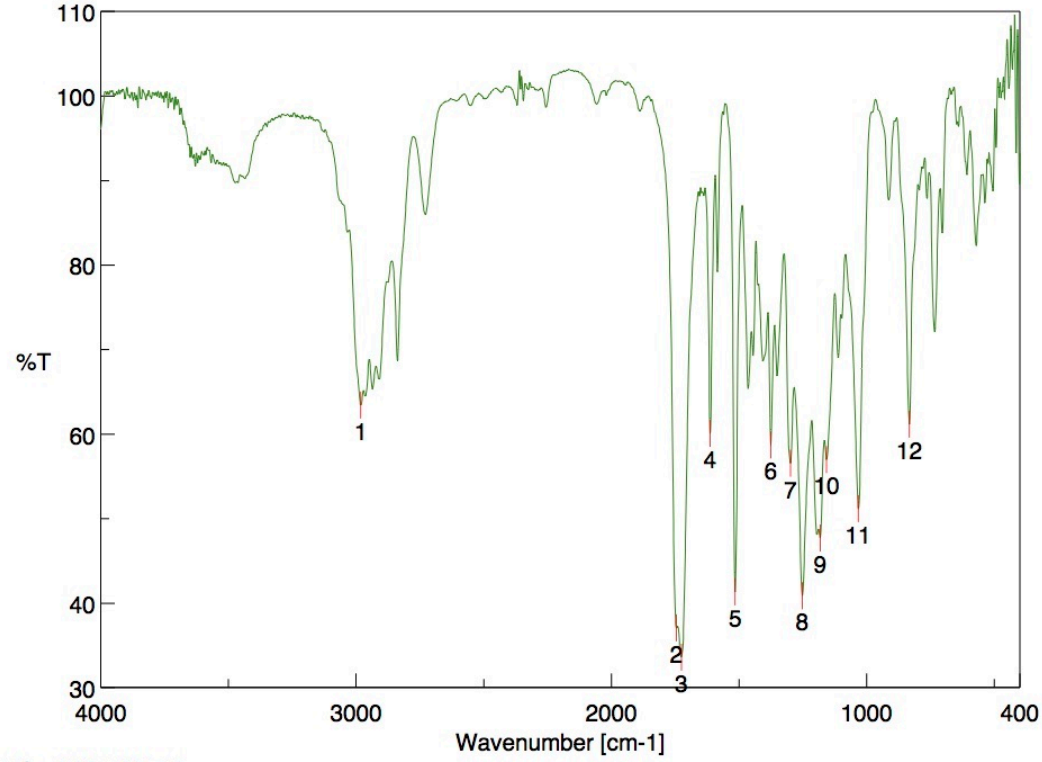
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1725.98	81.6257	2	1238.08	99.7774
3	1191.79	93.6438	4	1029.8	98.9271
5	816.706	98.592	6	503.33	100.488
7	489.831	99.1663	8	466.689	99.8755
9	433.905	99.4049	10	418.477	98.1335
11	405.942	97.6823			



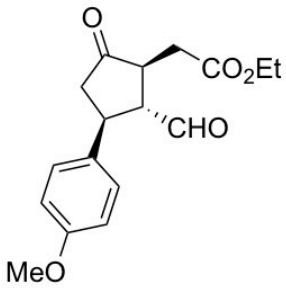

9.668
9.663
7.260
7.239
6.899
6.877
4.154
4.150
4.136
4.132
4.118
4.114
4.100
4.096
3.793
3.449
3.429
3.420
3.401
3.391
3.372
3.358
3.354
3.331
3.327
3.304
3.299
2.887
2.872
2.841
2.830
2.821
2.805
2.795
2.782
2.724
2.716
2.682
2.674
2.645
2.628
2.598
1.250
1.232
O
CO2Et
CHO
MeO
1.00
2.48
2.09
2.13
3.07
1.18
1.12
3.31
2.43
3.29
f1 (ppm)

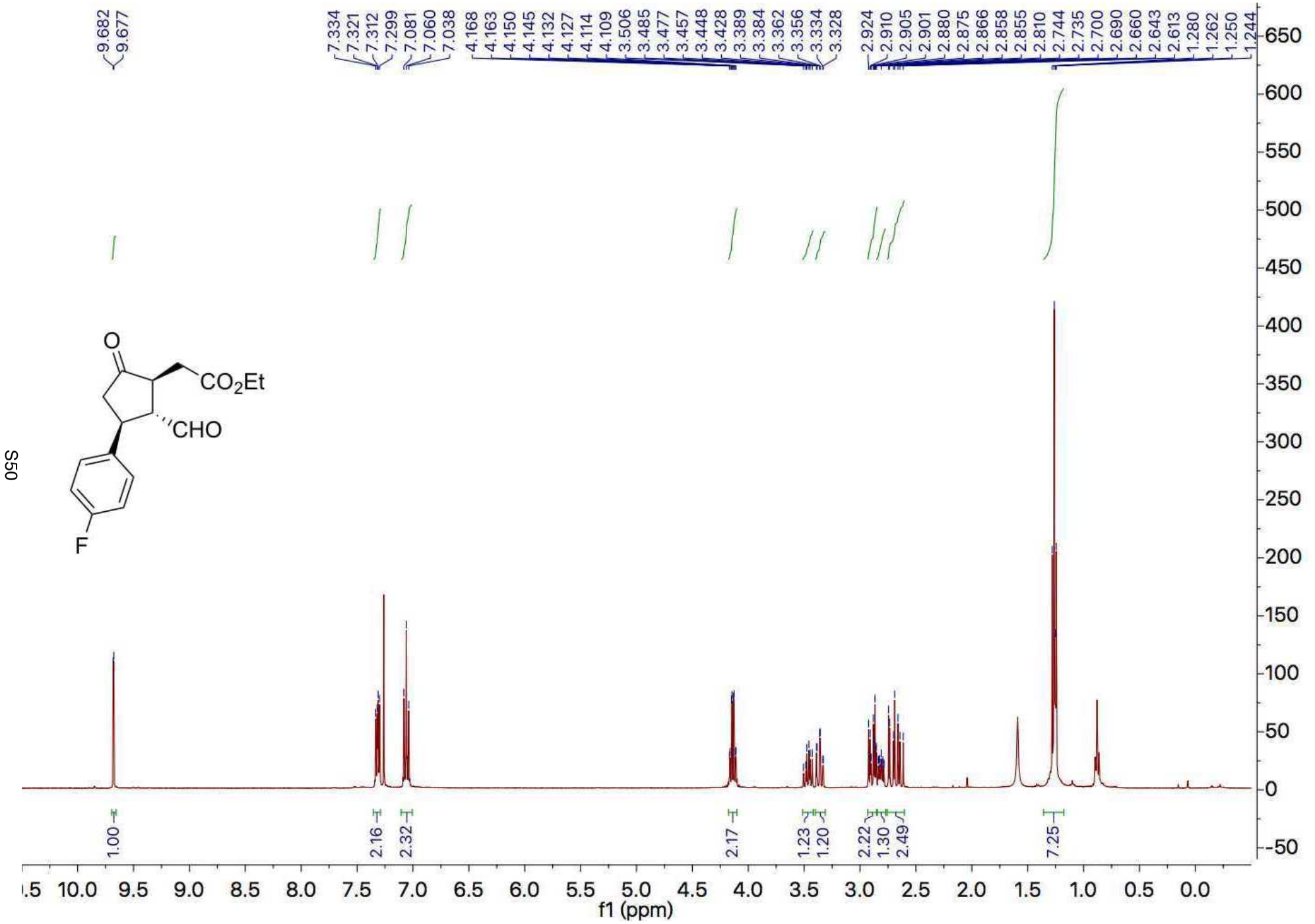

213.84
201.11
171.52
158.95
131.95
128.24
114.44
61.03
60.13
55.29
46.72
45.89
41.74
32.52
14.09
O
CO2Et
CHO
MeO
f1 (ppm)

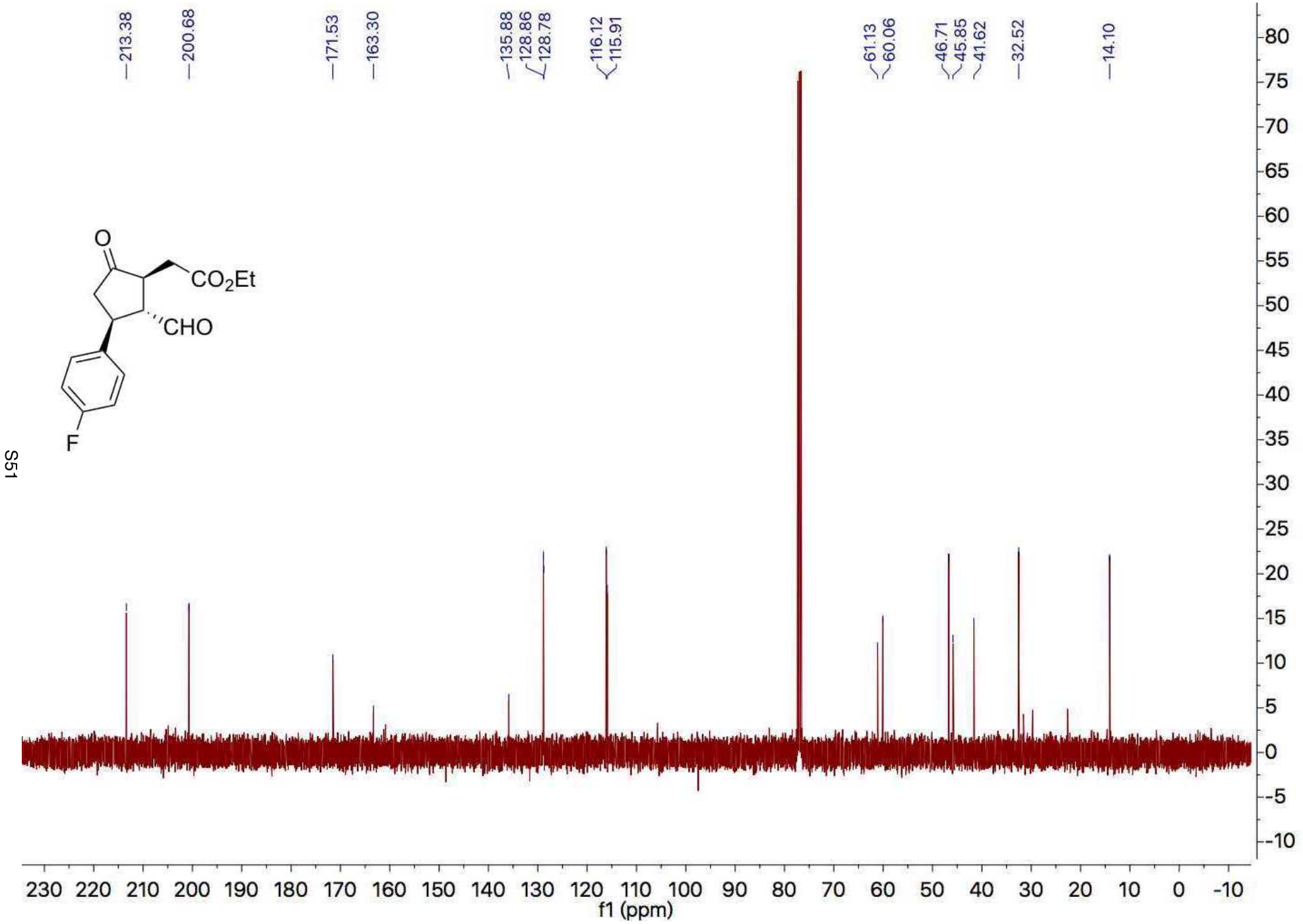


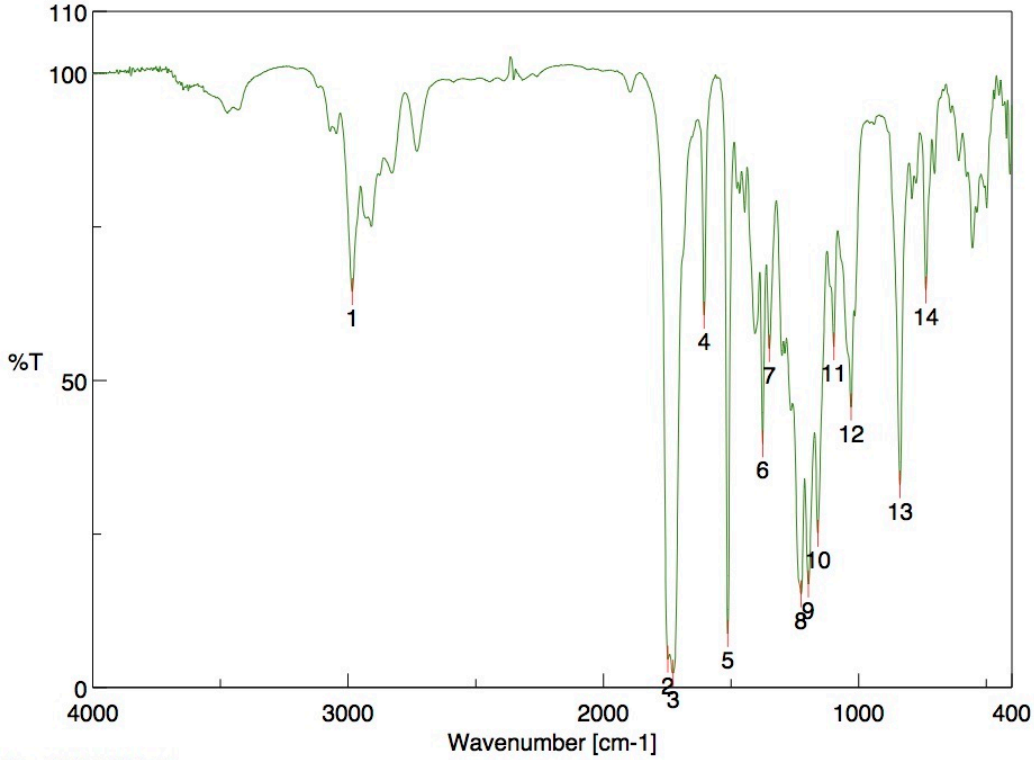
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	2981.41	63.4164	2	1745.26	37.0564
3	1725.98	33.5864	4	1613.16	60.0739
5	1515.78	41.3058	6	1375.96	58.6875
7	1297.86	56.534	8	1251.58	40.861
9	1182.15	47.6655	10	1157.08	56.9727
11	1032.69	51.18	12	832.133	61.1534



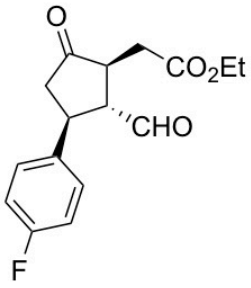

9.682
9.677
7.334
7.321
7.312
7.299
7.081
7.060
7.038
4.168
4.163
4.150
4.145
4.132
4.127
4.114
4.109
3.506
3.485
3.477
3.457
3.448
3.428
3.389
3.384
3.362
3.356
3.334
3.328
2.924
2.910
2.905
2.901
2.880
2.875
2.866
2.858
2.855
2.810
2.744
2.735
2.700
2.690
2.660
2.643
2.613
1.280
1.262
1.250
1.244
O
CO2Et
CHO
F
1.00
2.16
2.32
2.17
1.23
1.20
2.22
1.30
2.49
7.25
f1 (ppm)

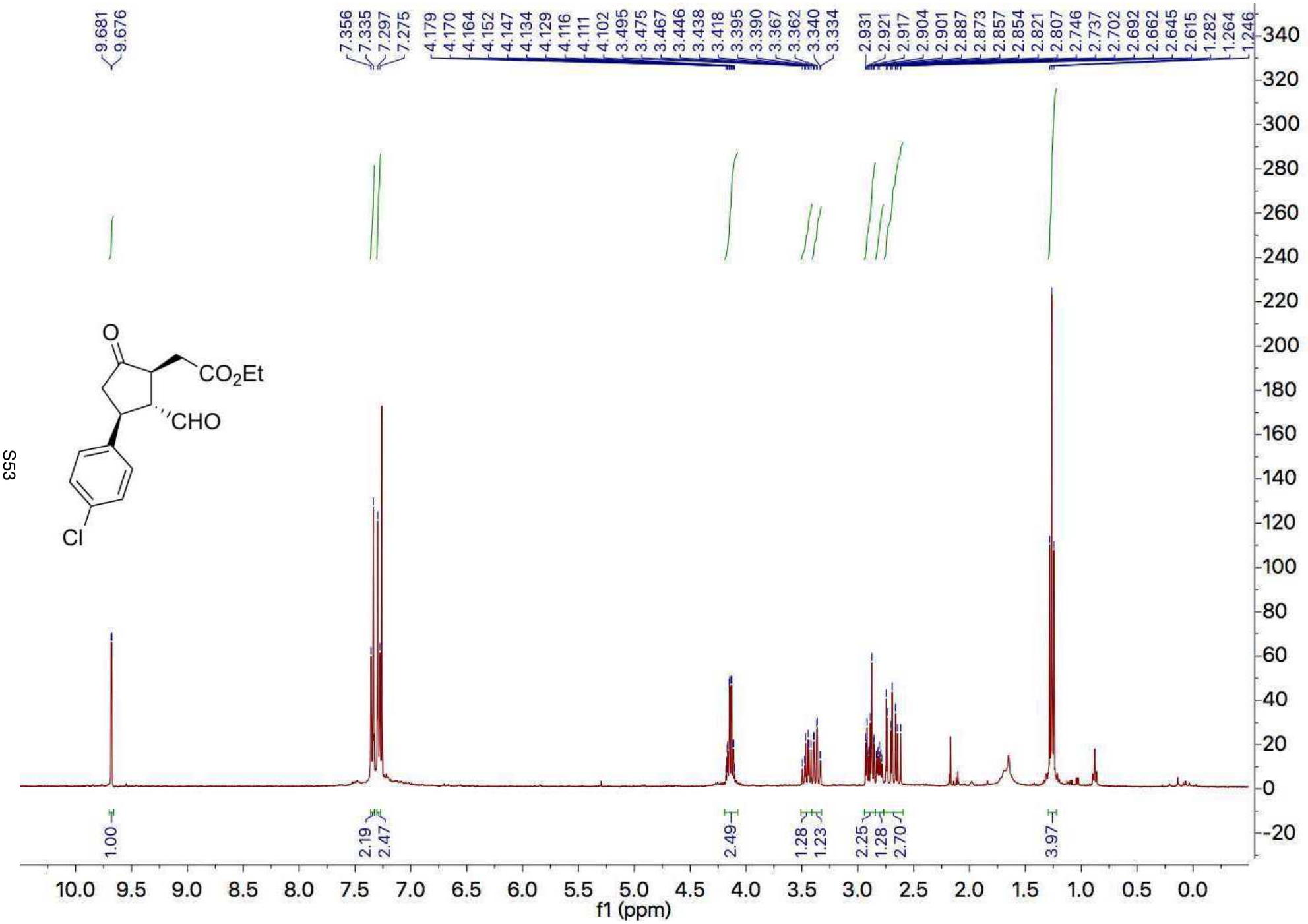

213.38
200.68
171.53
163.30
135.88
128.86
128.78
116.12
115.91
61.13
60.06
46.71
45.85
41.62
32.52
14.10
O
CO2Et
CHO
F
f1 (ppm)

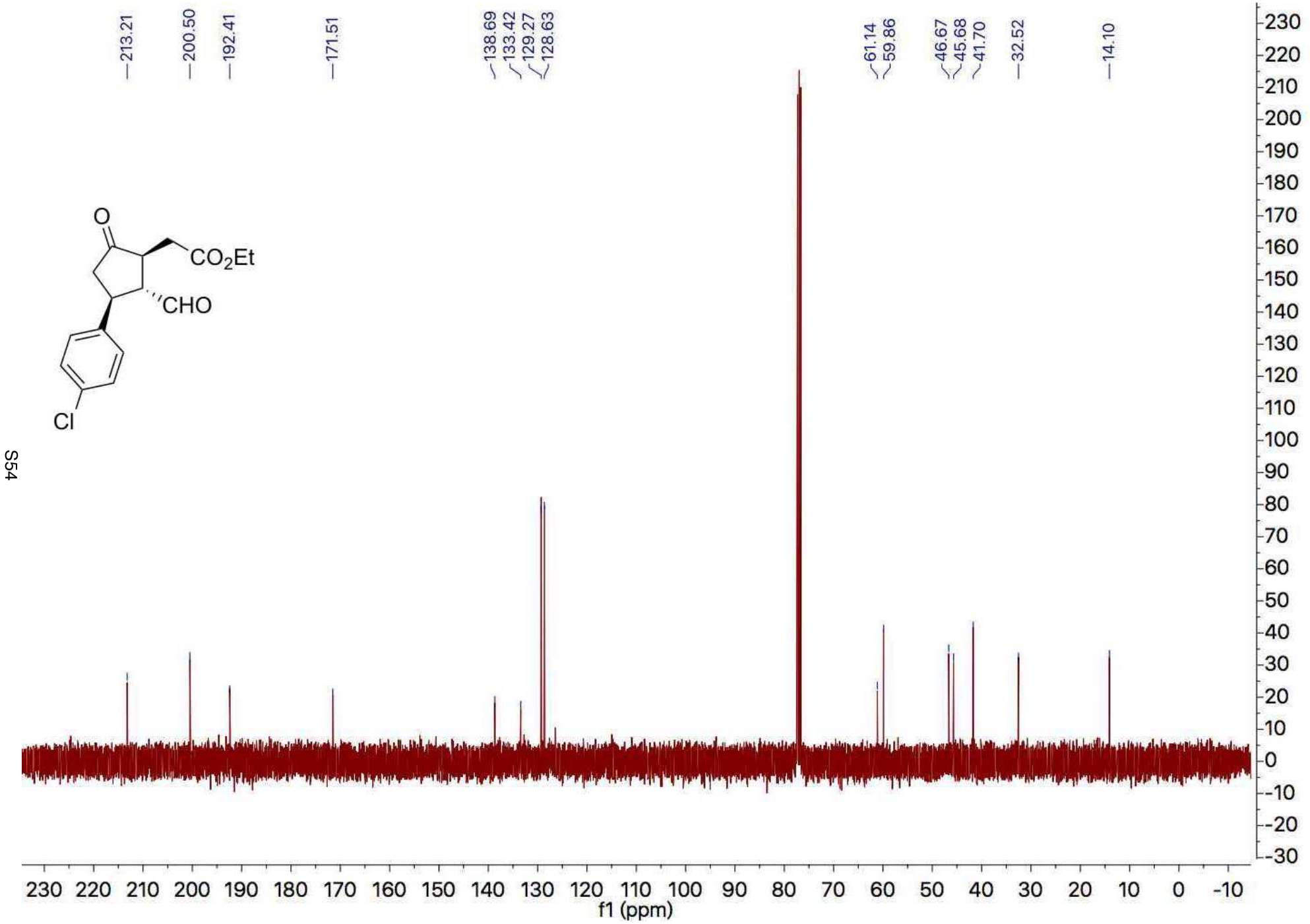


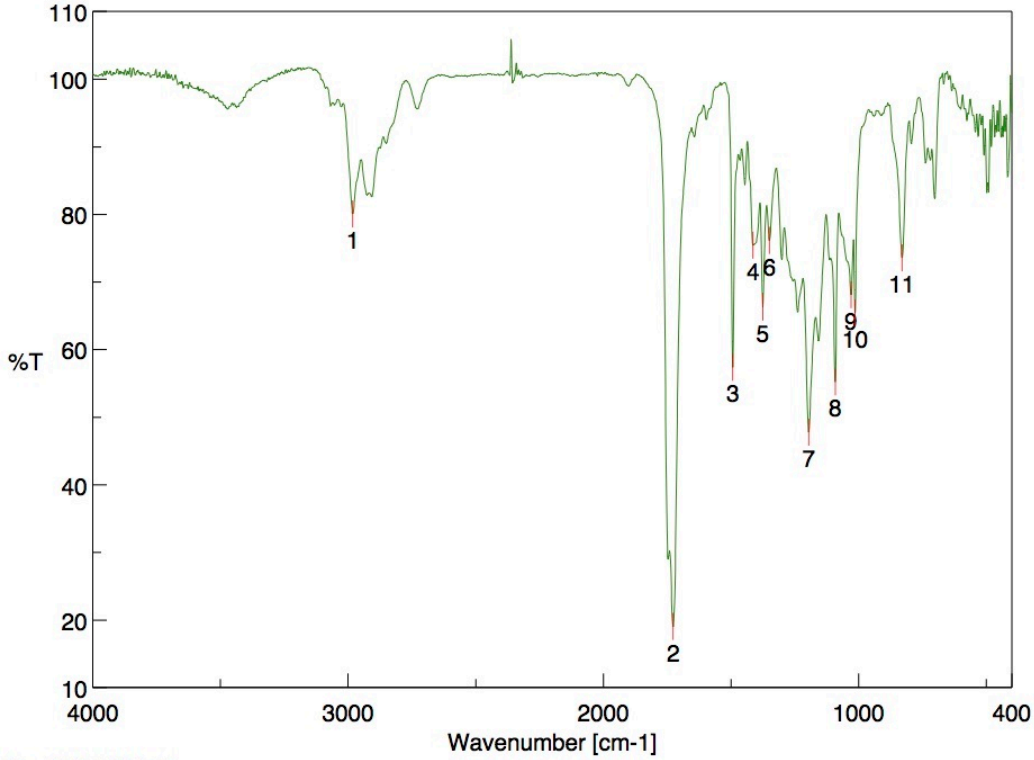
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	2983.34	64.404	2	1747.19	4.64413
3	1726.94	2.35719	4	1605.45	60.5417
5	1512.88	8.79937	6	1375.96	39.6431
7	1349.93	55.1463	8	1225.54	15.2353
9	1196.61	16.8314	10	1159.97	25.0998
11	1097.3	55.4379	12	1029.8	45.6373
13	837.919	33.0156	14	736.674	64.6894



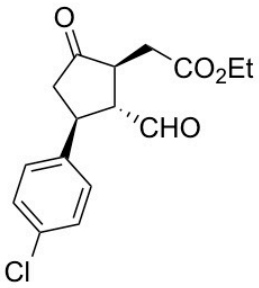

9.681
9.676
7.356
7.335
7.297
7.275
4.179
4.170
4.164
4.152
4.147
4.134
4.129
4.116
4.111
4.102
3.495
3.475
3.467
3.446
3.438
3.418
3.395
3.390
3.367
3.362
3.340
3.334
2.931
2.921
2.917
2.904
2.901
2.887
2.873
2.857
2.854
2.821
2.807
2.746
2.737
2.702
2.692
2.662
2.645
2.615
1.282
1.264
1.246
O
CO2Et
CHO
Cl
1.00
2.19
2.47
2.49
1.28
1.23
2.25
1.28
2.70
3.97
f1 (ppm)

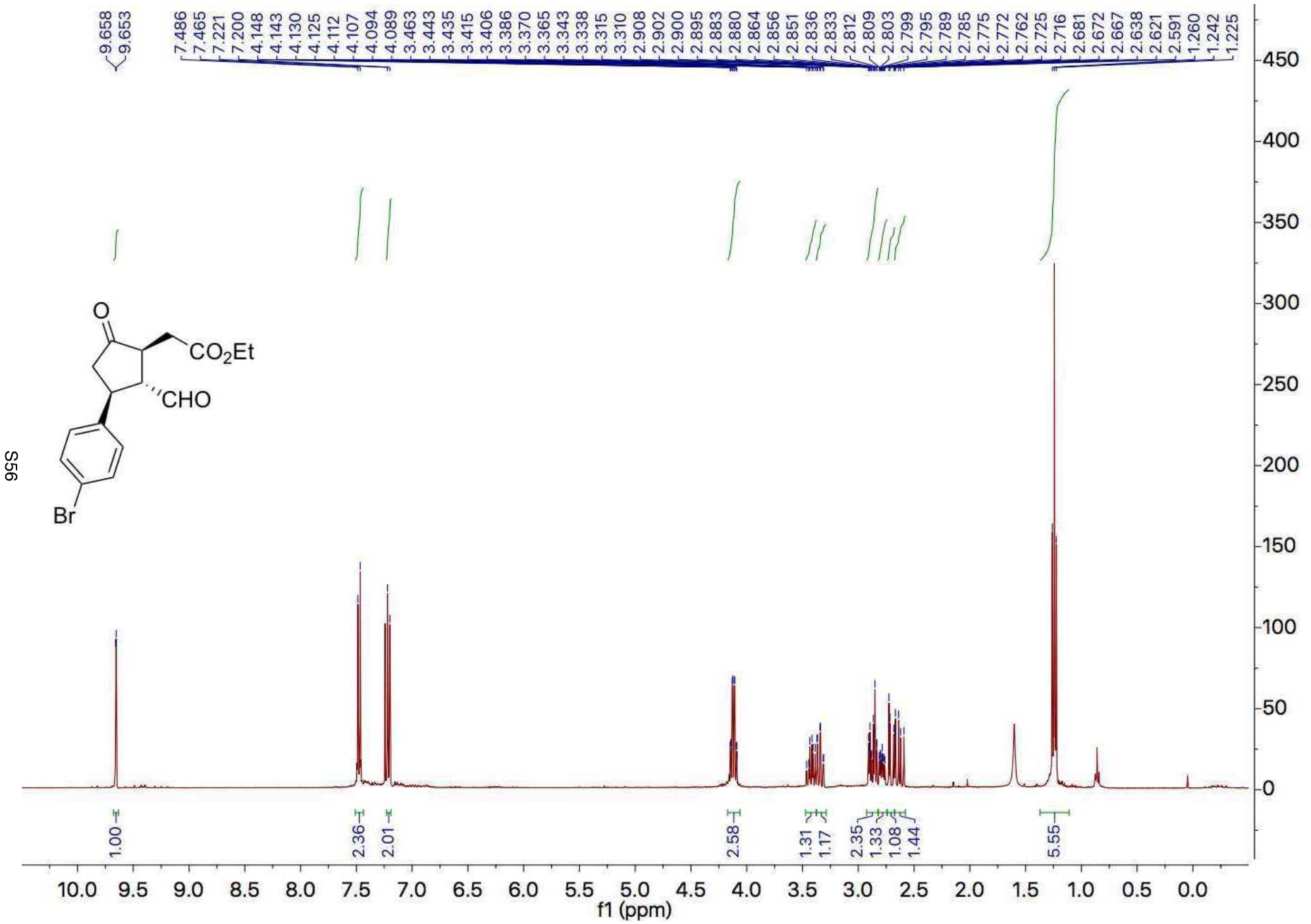

213.21
200.50
192.41
171.51
138.69
133.42
129.27
128.63
61.14
59.86
46.67
45.68
41.70
32.52
14.10
O
CO2Et
CHO
Cl
f1 (ppm)

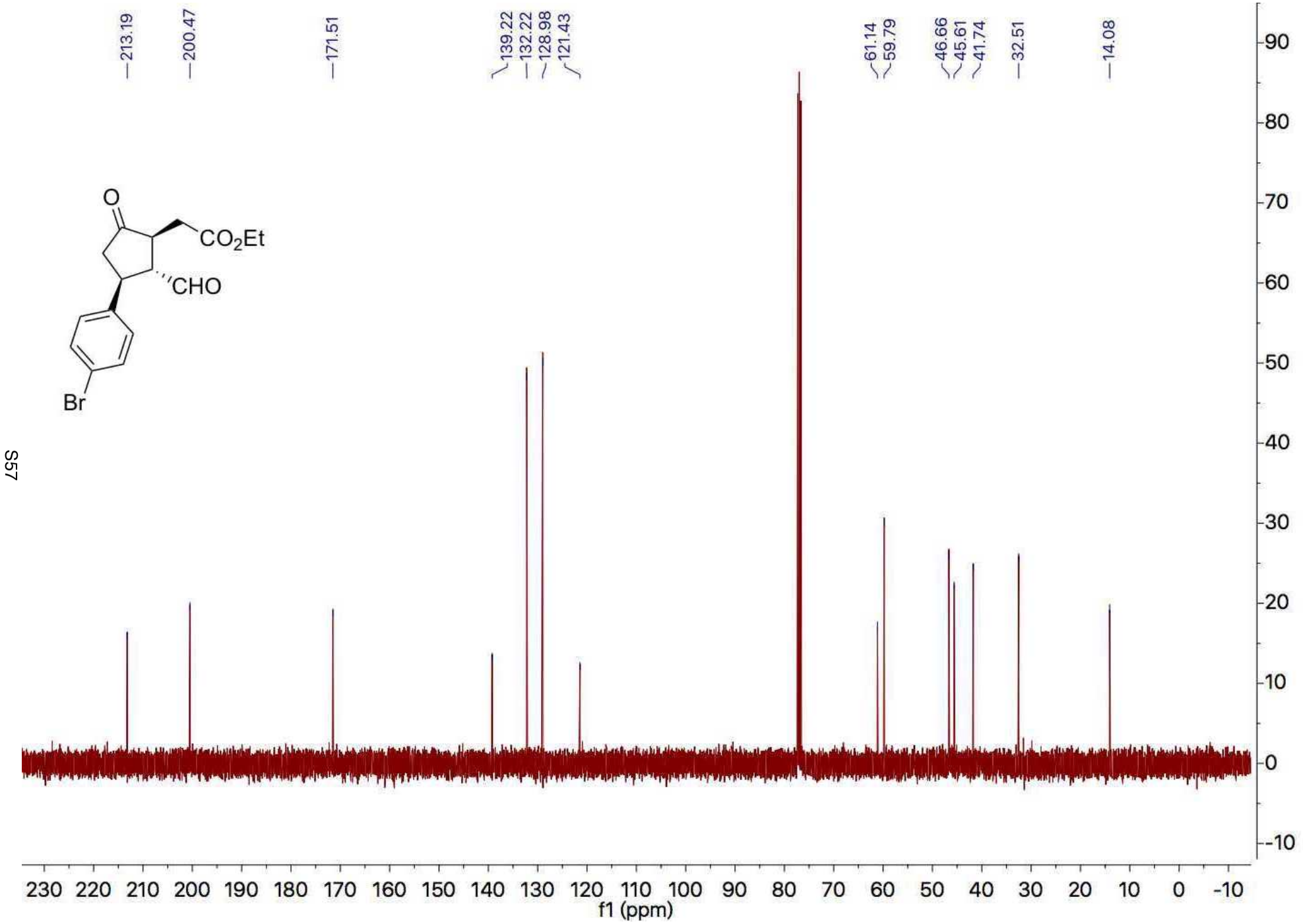


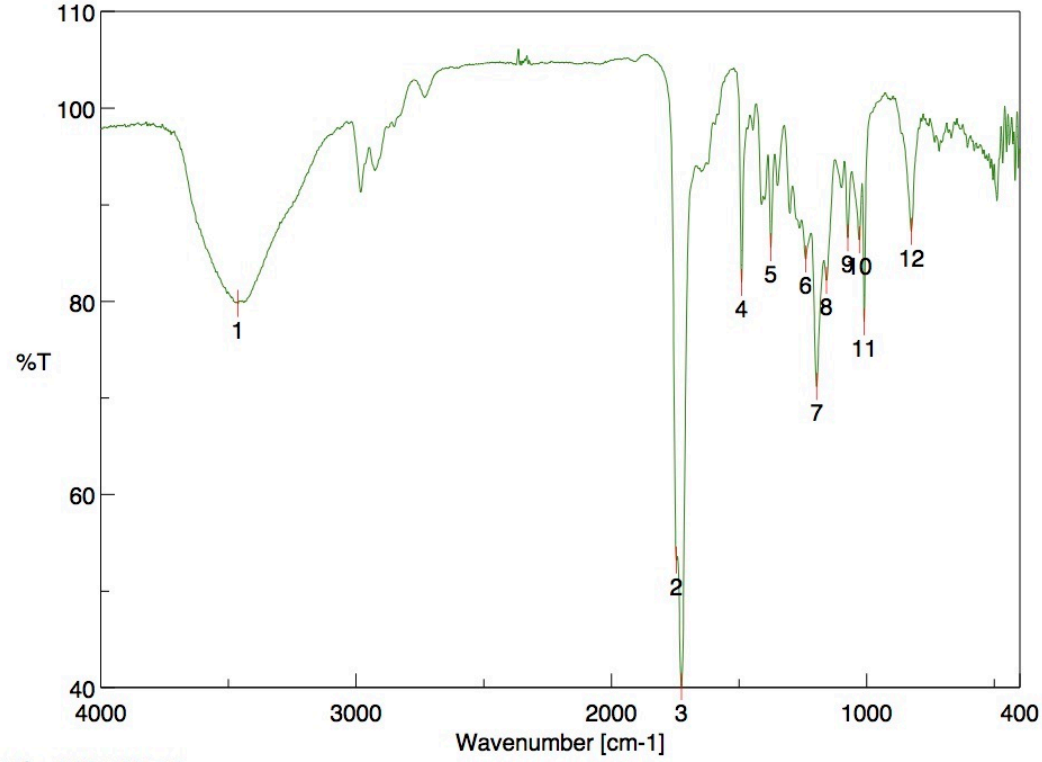
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	2981.41	80.0191	2	1726.94	19.0345
3	1493.6	57.3374	4	1413.57	75.4141
5	1375.96	66.2668	6	1349.93	76.1357
7	1195.65	47.7478	8	1091.51	55.2062
9	1029.8	68.0826	10	1014.37	65.4094
11	830.205	73.5434			



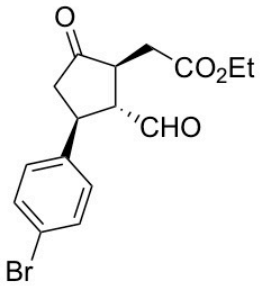

9.658
9.653
7.486
7.465
7.221
7.200
4.148
4.143
4.130
4.125
4.112
4.107
4.094
4.089
3.463
3.443
3.435
3.415
3.406
3.386
3.370
3.365
3.343
3.338
3.315
3.310
2.908
2.902
2.900
2.895
2.883
2.880
2.864
2.856
2.851
2.836
2.833
2.812
2.809
2.803
2.799
2.795
2.789
2.785
2.775
2.772
2.762
2.725
2.716
2.681
2.672
2.667
2.638
2.621
2.591
1.260
1.242
1.225
O
CO2Et
CHO
Br
1.00
2.36
2.01
2.58
1.31
1.17
2.35
1.33
1.08
1.44
5.55
f1 (ppm)

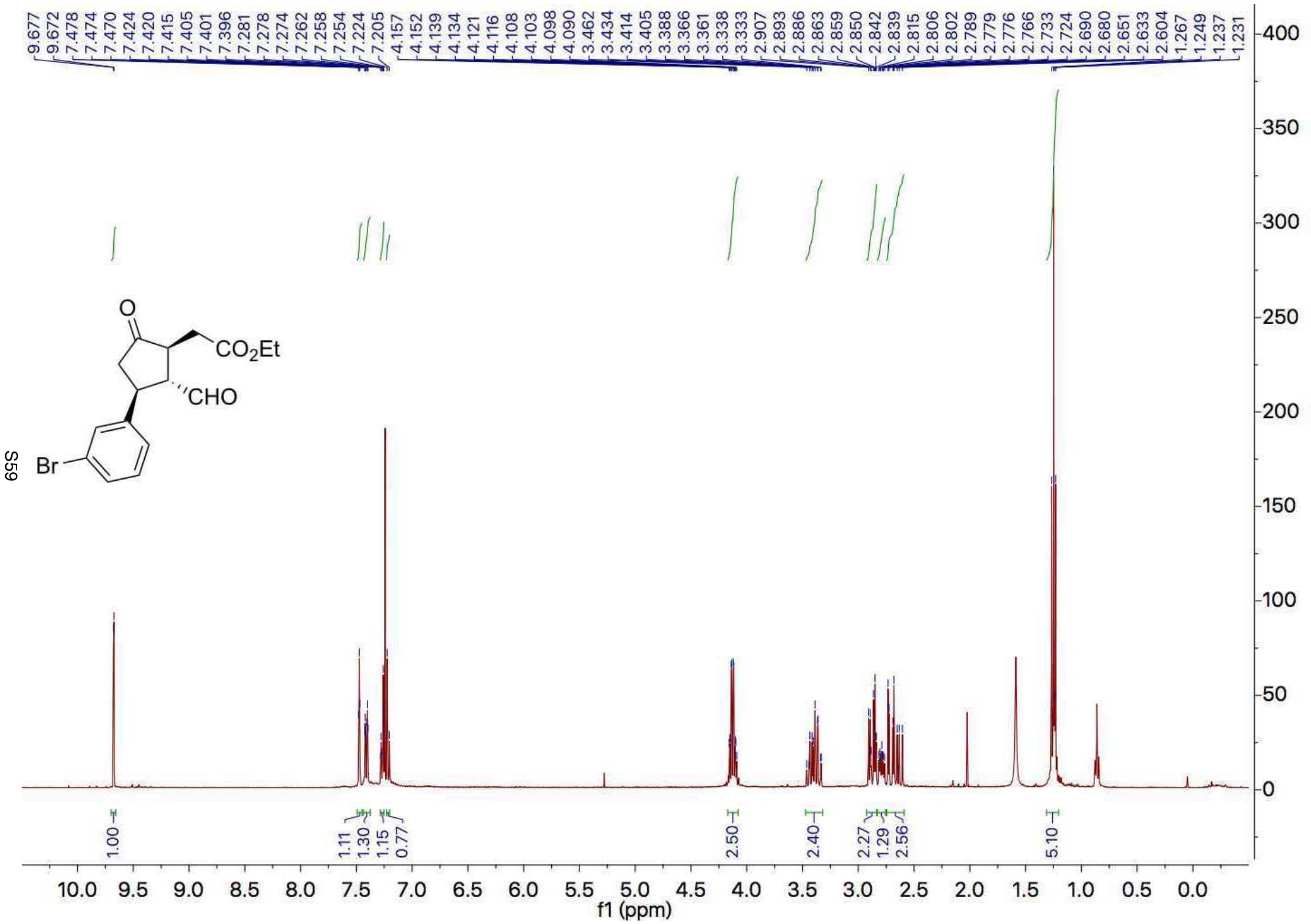

213.19
200.47
171.51
139.22
132.22
128.98
121.43
61.14
59.79
46.66
45.61
41.74
32.51
14.08
O
CO2Et
CHO
Br
f1 (ppm)

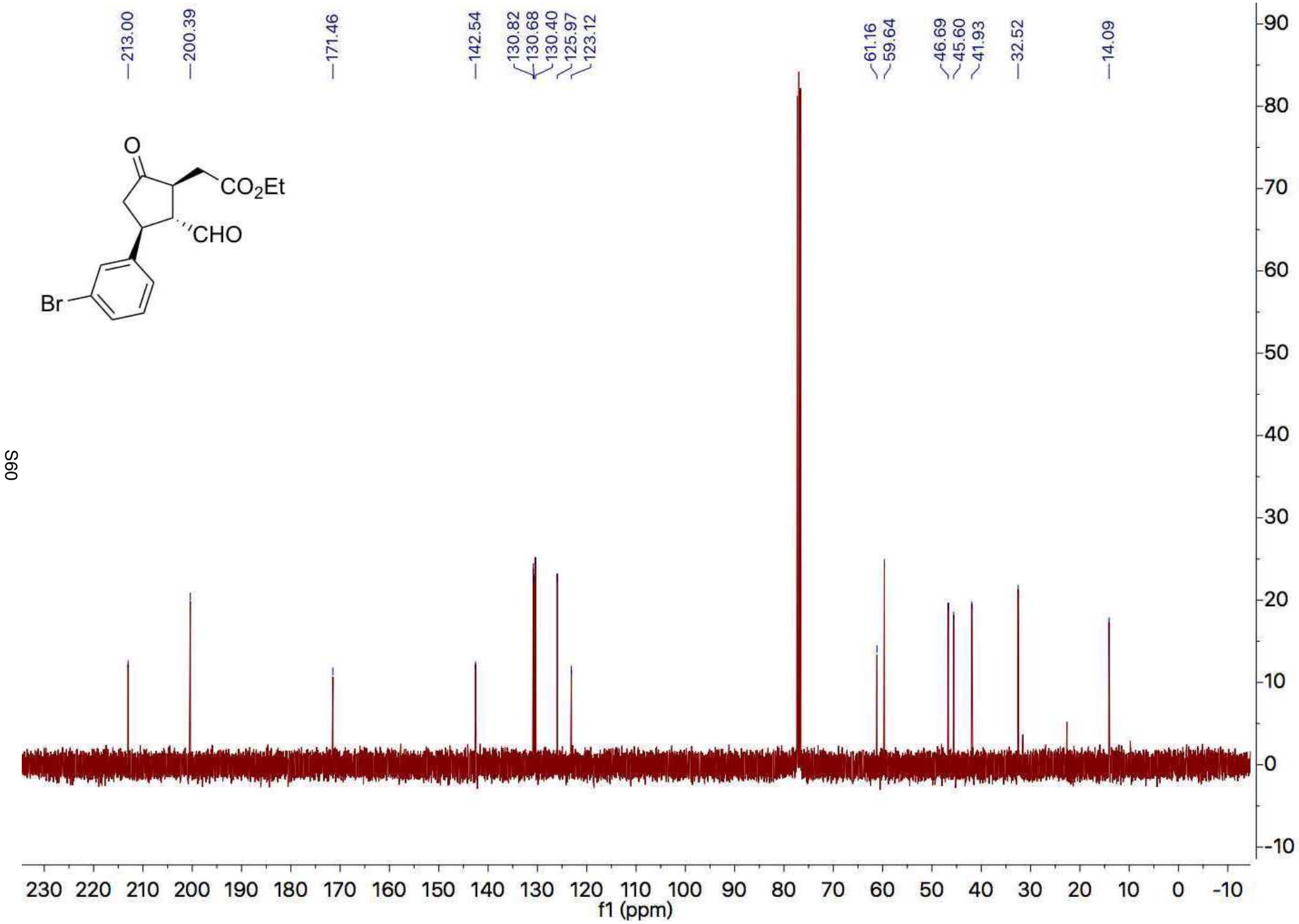


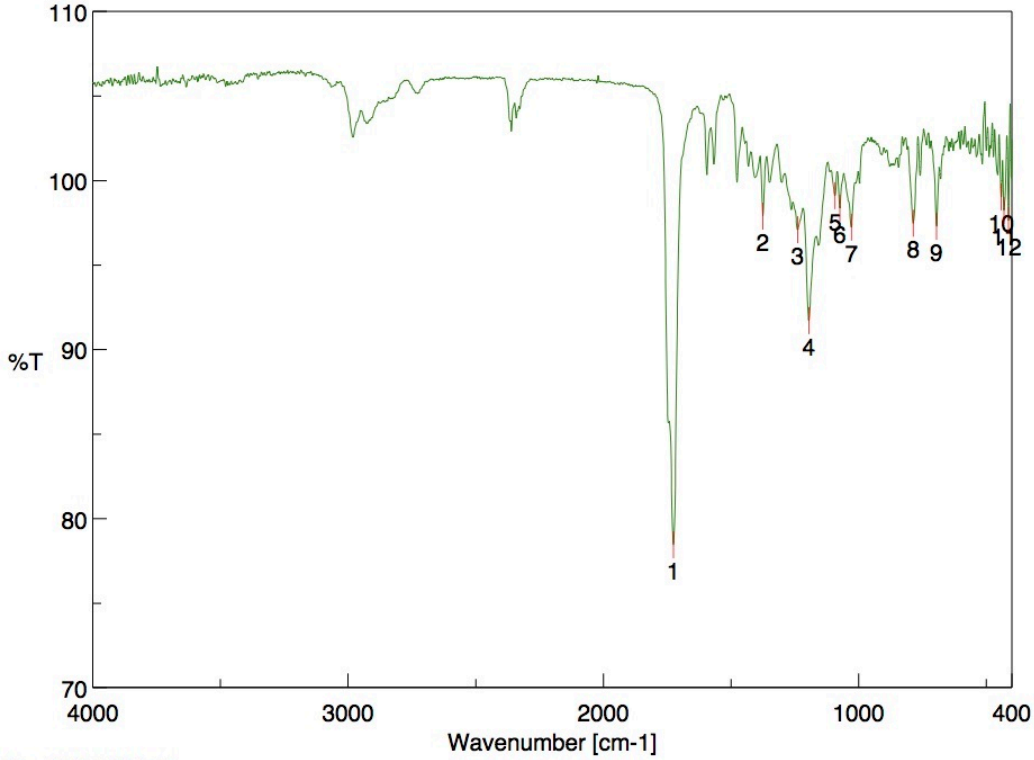
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	3462.56	79.7666	2	1745.26	53.2028
3	1725.98	40.0745	4	1489.74	81.944
5	1375	85.5186	6	1239.04	84.3536
7	1195.65	71.1607	8	1157.08	82.1592
9	1074.16	86.5611	10	1028.84	86.3433
11	1009.55	77.8566	12	825.384	87.2308



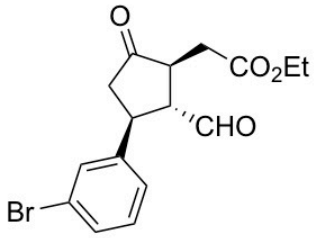

9.677
9.672
7.478
7.474
7.470
7.424
7.420
7.415
7.405
7.401
7.396
7.281
7.278
7.274
7.262
7.258
7.254
7.224
7.205
4.157
4.152
4.139
4.134
4.121
4.116
4.108
4.103
4.098
4.090
3.462
3.434
3.414
3.405
3.388
3.366
3.361
3.338
3.333
2.907
2.893
2.886
2.863
2.859
2.850
2.842
2.839
2.815
2.806
2.802
2.789
2.779
2.776
2.766
2.733
2.724
2.690
2.680
2.651
2.633
2.604
1.267
1.249
1.237
1.231
O
CO2Et
CHO
Br
1.00
1.11
1.30
1.15
0.77
2.50
2.40
2.27
1.29
2.56
5.10
f1 (ppm)

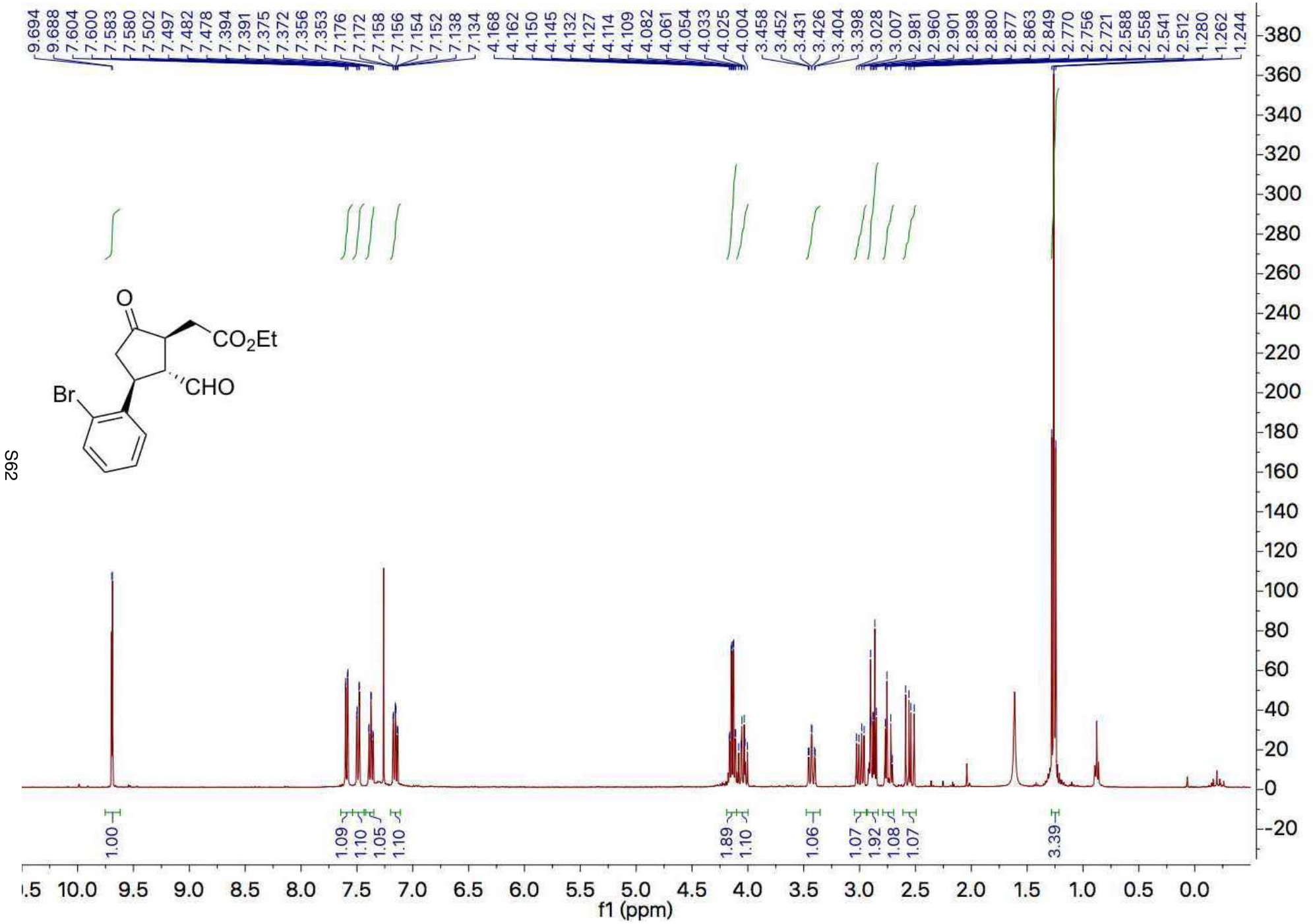

213.00
200.39
171.46
142.54
130.82
130.68
130.40
125.97
123.12
61.16
59.64
46.69
45.60
41.93
32.52
14.09
O
CO2Et
CHO
Br
f1 (ppm)

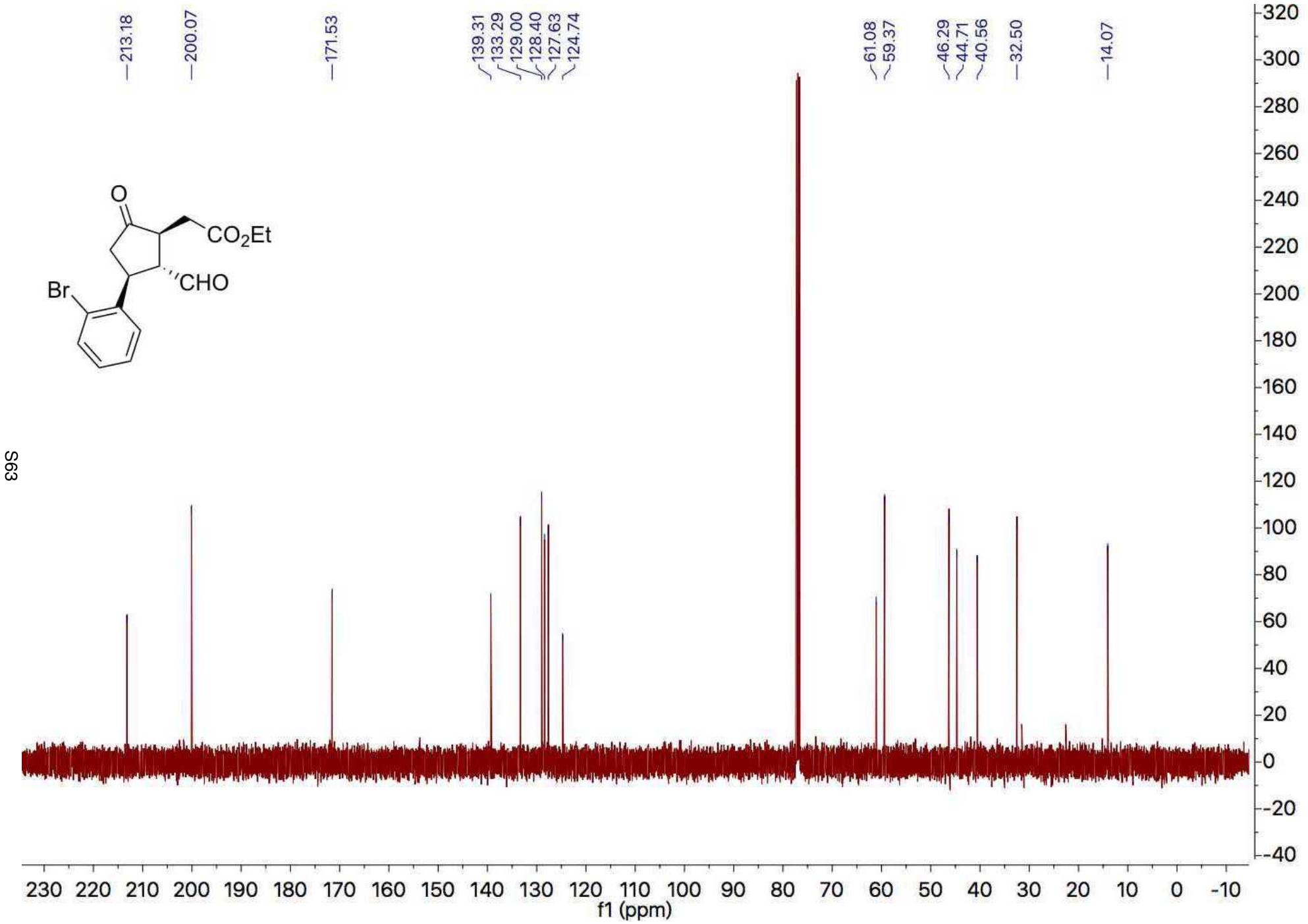


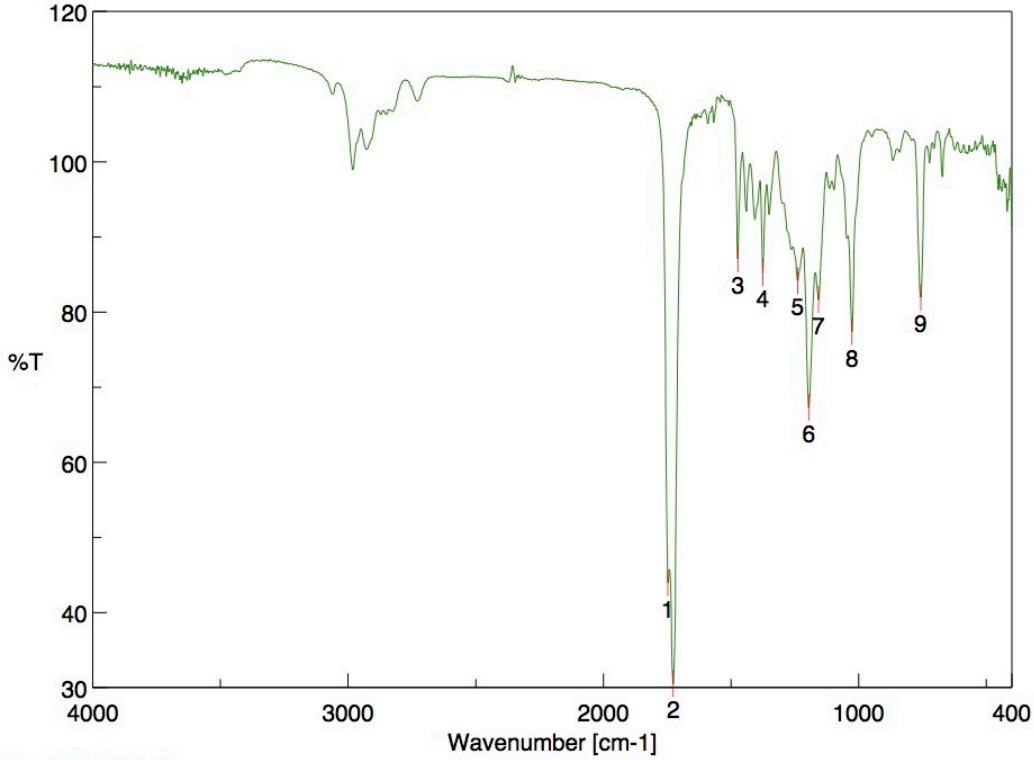
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1725.98	78.4429	2	1375	97.8827
3	1239.04	97.0783	4	1194.69	91.704
5	1092.48	99.09	6	1074.16	98.3608
7	1028.84	97.2314	8	786.815	97.4586
9	695.212	97.2897	10	441.619	99.0363
11	431.012	98.2104	12	412.692	97.6382



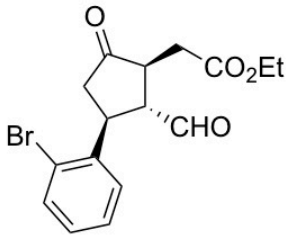

9.694 9.688 7.604 7.600 7.583 7.580 7.502 7.497 7.482 7.478 7.394 7.391 7.375 7.372 7.356 7.353 7.176 7.172 7.158 7.156 7.154 7.152 7.138 7.134 4.168 4.162 4.150 4.145 4.132 4.127 4.114 4.109 4.082 4.061 4.054 4.033 4.025 4.004 3.458 3.452 3.431 3.426 3.404 3.398 3.028 3.007 2.981 2.960 2.901 2.898 2.880 2.877 2.863 2.849 2.770 2.756 2.721 2.588 2.558 2.541 2.512 1.280 1.262 1.244
O
CO2Et
CHO
Br
1.00
1.09
1.10
1.05
1.10
1.89
1.10
1.06
1.07
1.92
1.08
1.07
3.39
f1 (ppm)

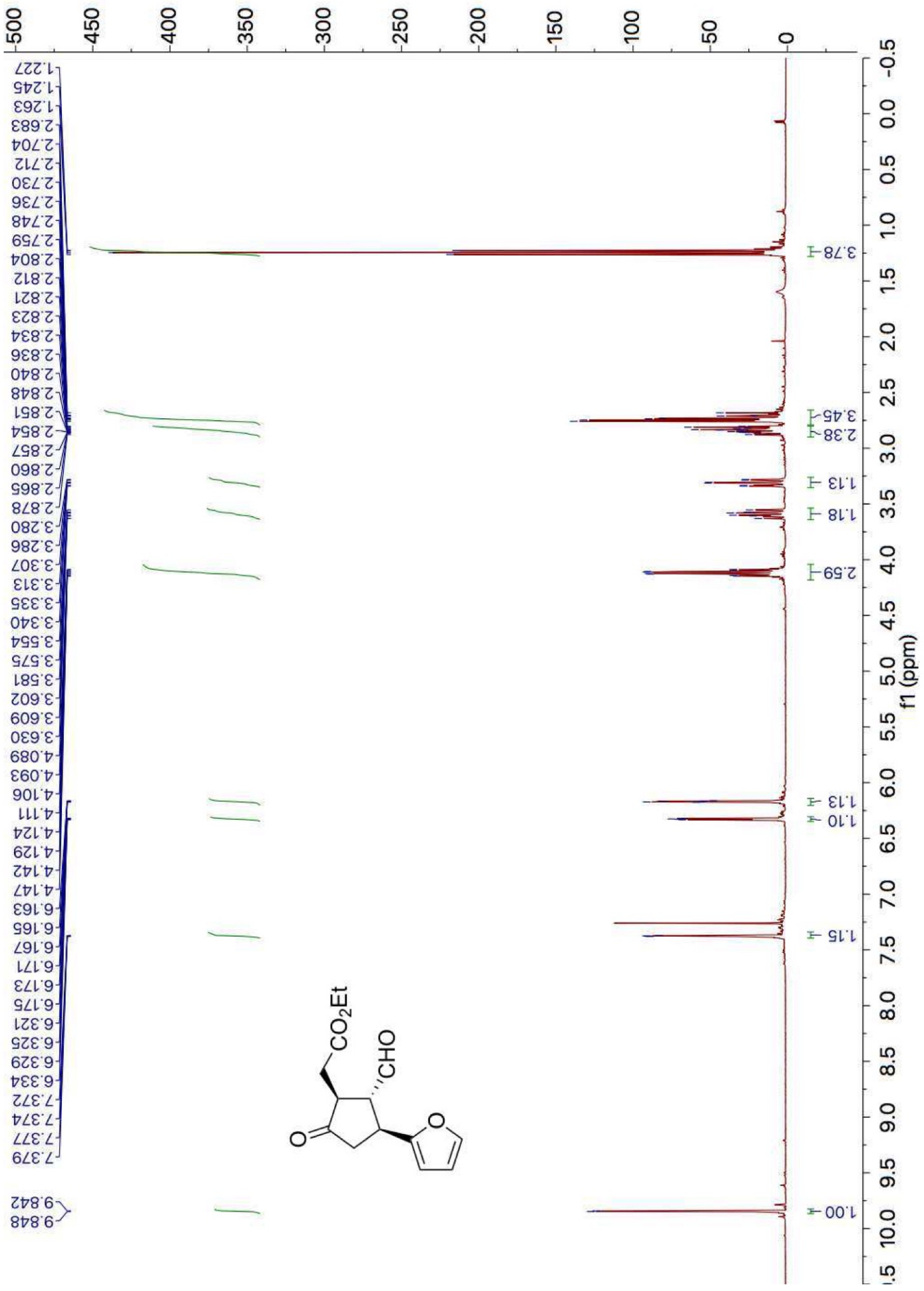

213.18
200.07
171.53
139.31
133.29
129.00
128.40
127.63
124.74
61.08
59.37
46.29
44.71
40.56
32.50
14.07
O
CO2Et
CHO
Br
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

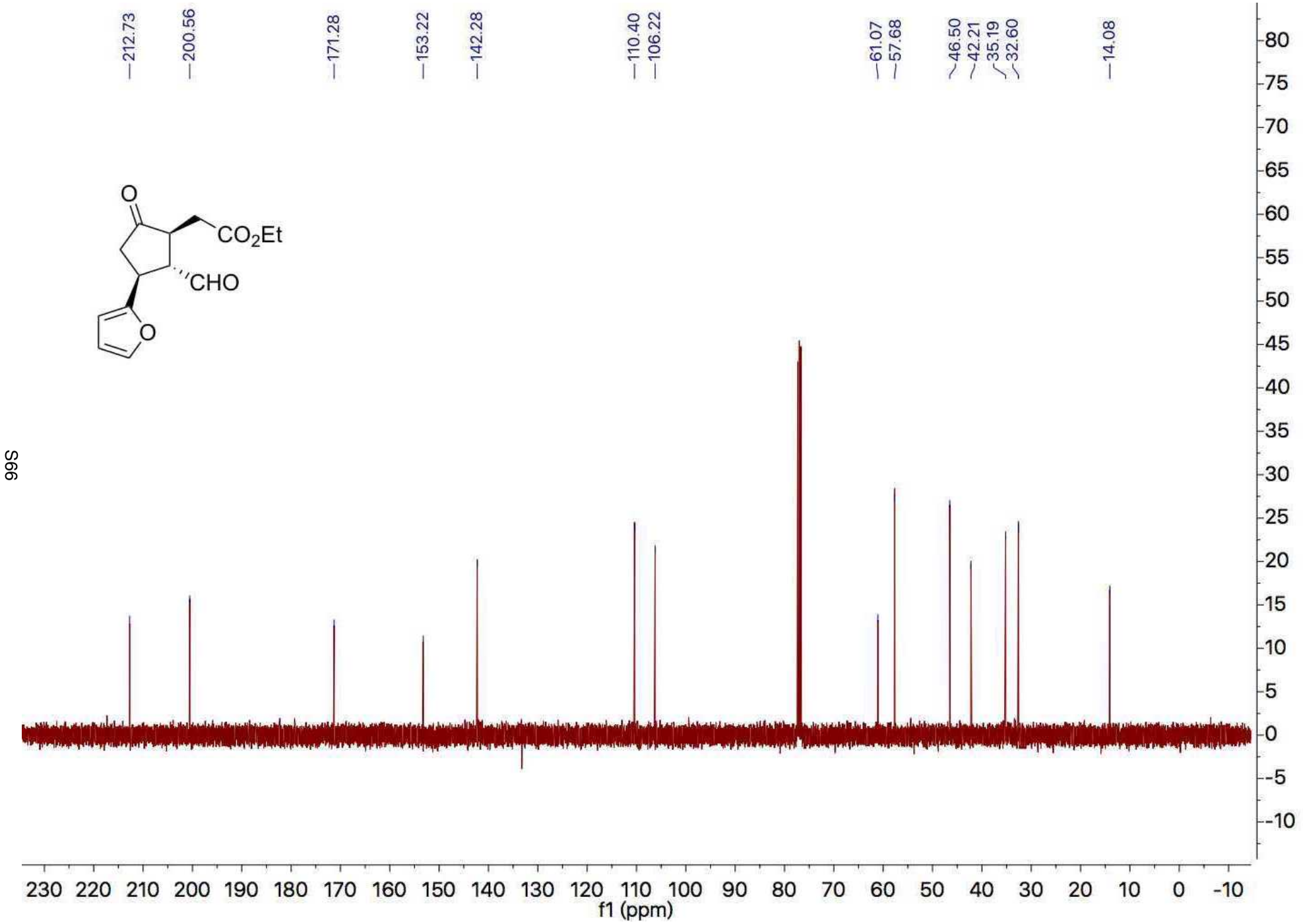


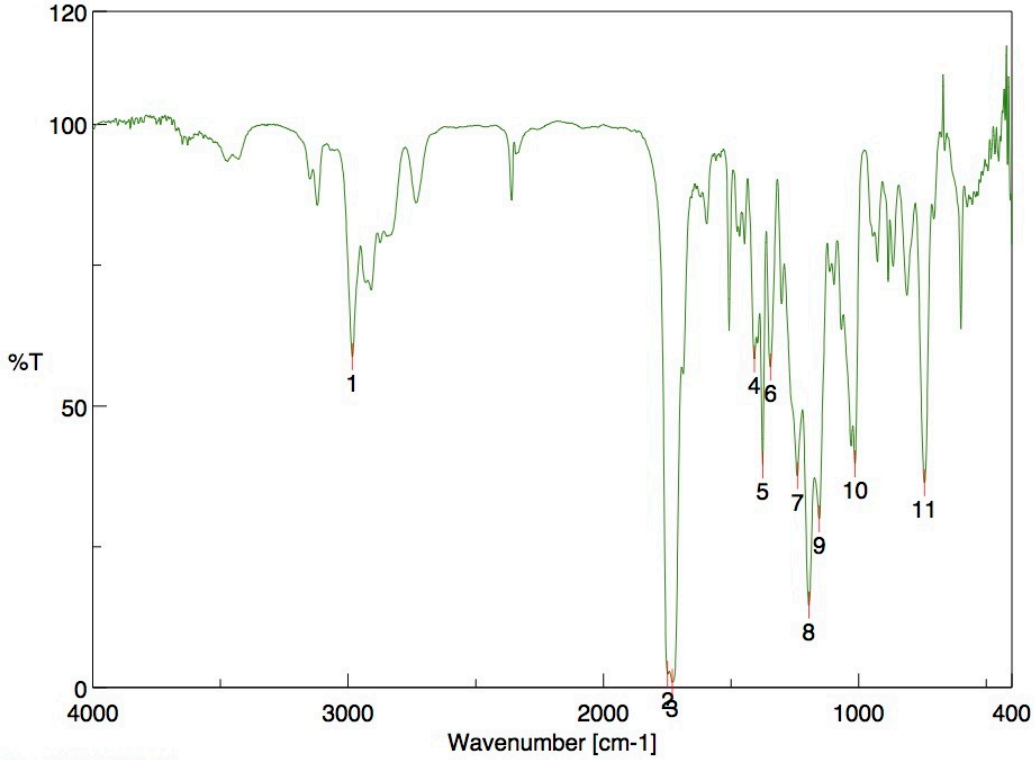
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1747.19	43.9832	2	1726.94	30.5284
3	1473.35	87.0751	4	1375	85.1956
5	1239.04	84.1402	6	1195.65	67.3053
7	1157.08	81.663	8	1025.94	77.3717
9	756.923	81.9575			



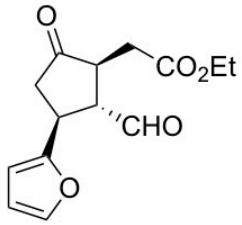

CO2Et
CHO
O
O
f1 (ppm)

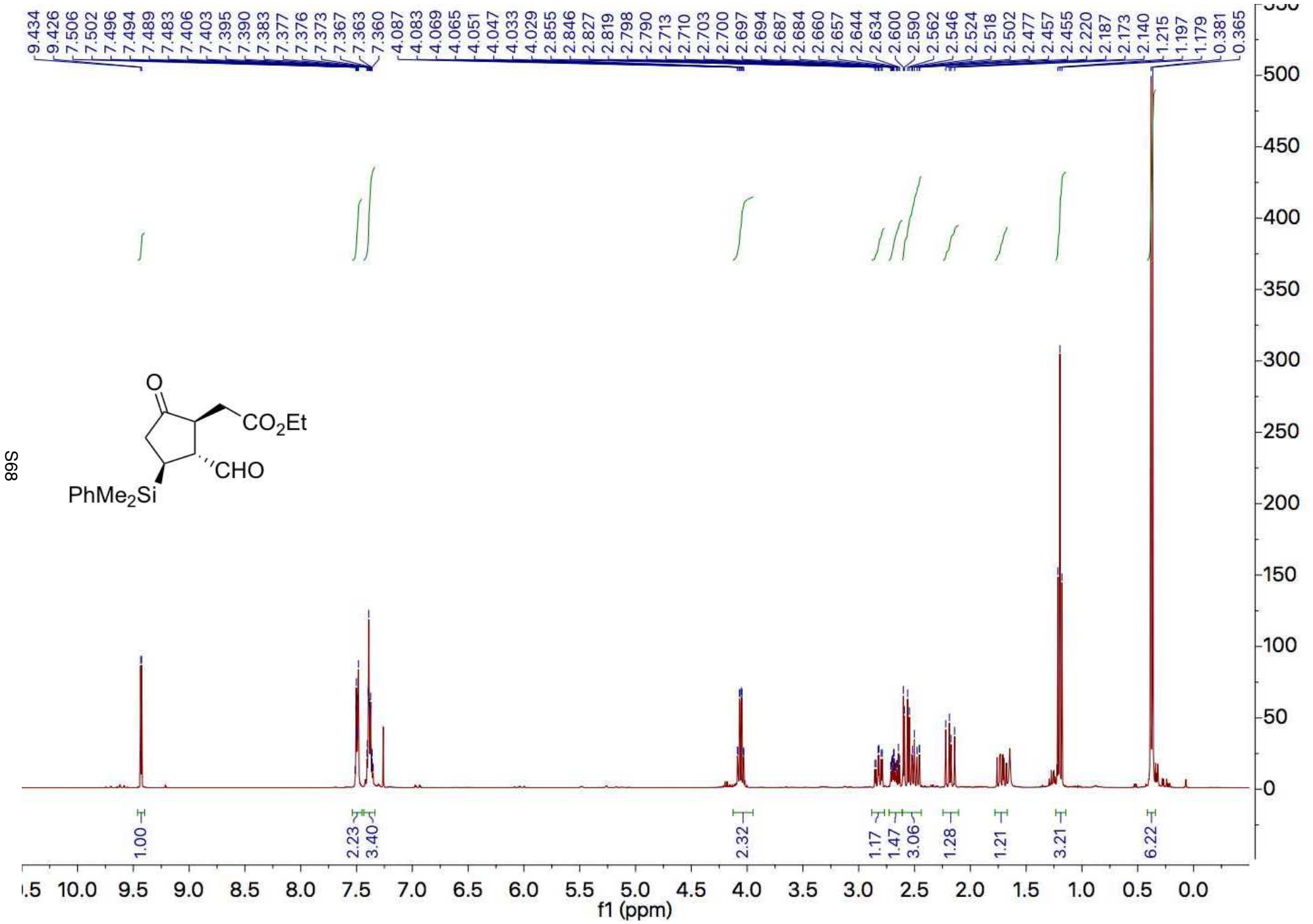

212.73
200.56
171.28
153.22
142.28
110.40
106.22
61.07
57.68
46.50
42.21
35.19
32.60
14.08
O
CO2Et
CHO
O
f1 (ppm)

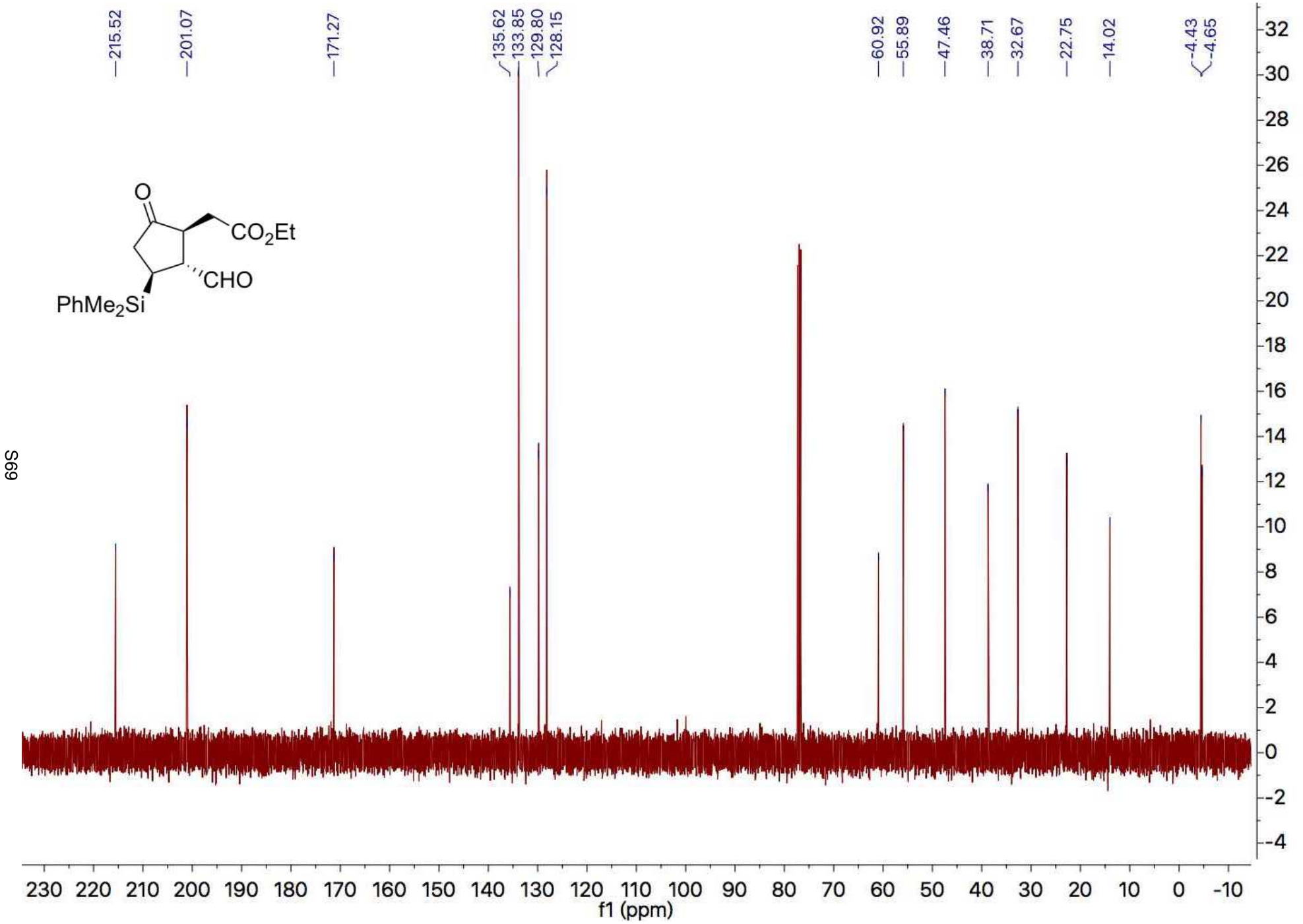


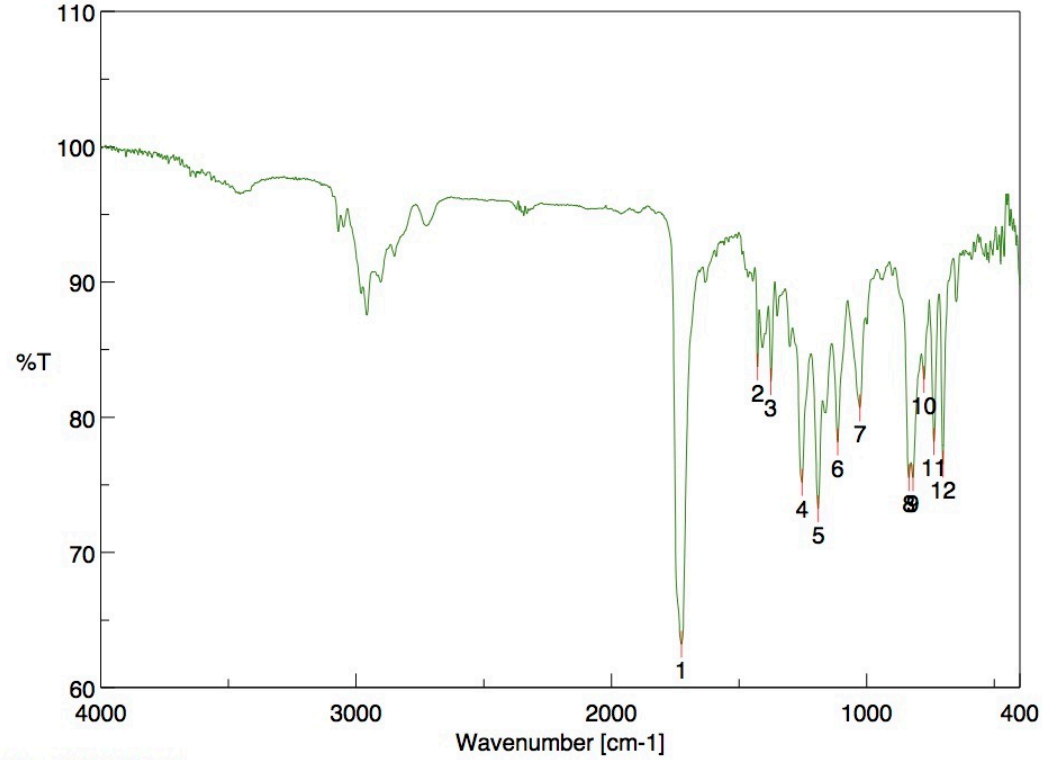
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	2983.34	58.7025	2	1748.16	2.36068
3	1729.83	0.916432	4	1408.75	58.3171
5	1375.96	39.5173	6	1346.07	56.8658
7	1240	37.5829	8	1194.69	14.6206
9	1154.19	29.9445	10	1014.37	39.7018
11	742.46	36.3558			



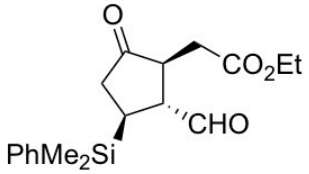

9.434
9.426
7.506
7.502
7.496
7.494
7.489
7.483
7.406
7.403
7.395
7.390
7.383
7.377
7.376
7.373
7.367
7.363
7.360
4.087
4.083
4.069
4.065
4.051
4.047
4.033
4.029
2.855
2.846
2.827
2.819
2.798
2.790
2.713
2.710
2.703
2.700
2.697
2.694
2.687
2.684
2.660
2.657
2.644
2.634
2.600
2.590
2.562
2.546
2.524
2.518
2.502
2.477
2.457
2.455
2.220
2.187
2.173
2.140
1.215
1.197
1.179
0.381
0.365
O
CO2Et
CHO
PhMe2Si
1.00
2.23
3.40
2.32
1.17
1.47
3.06
1.28
1.21
3.21
6.22
f1 (ppm)

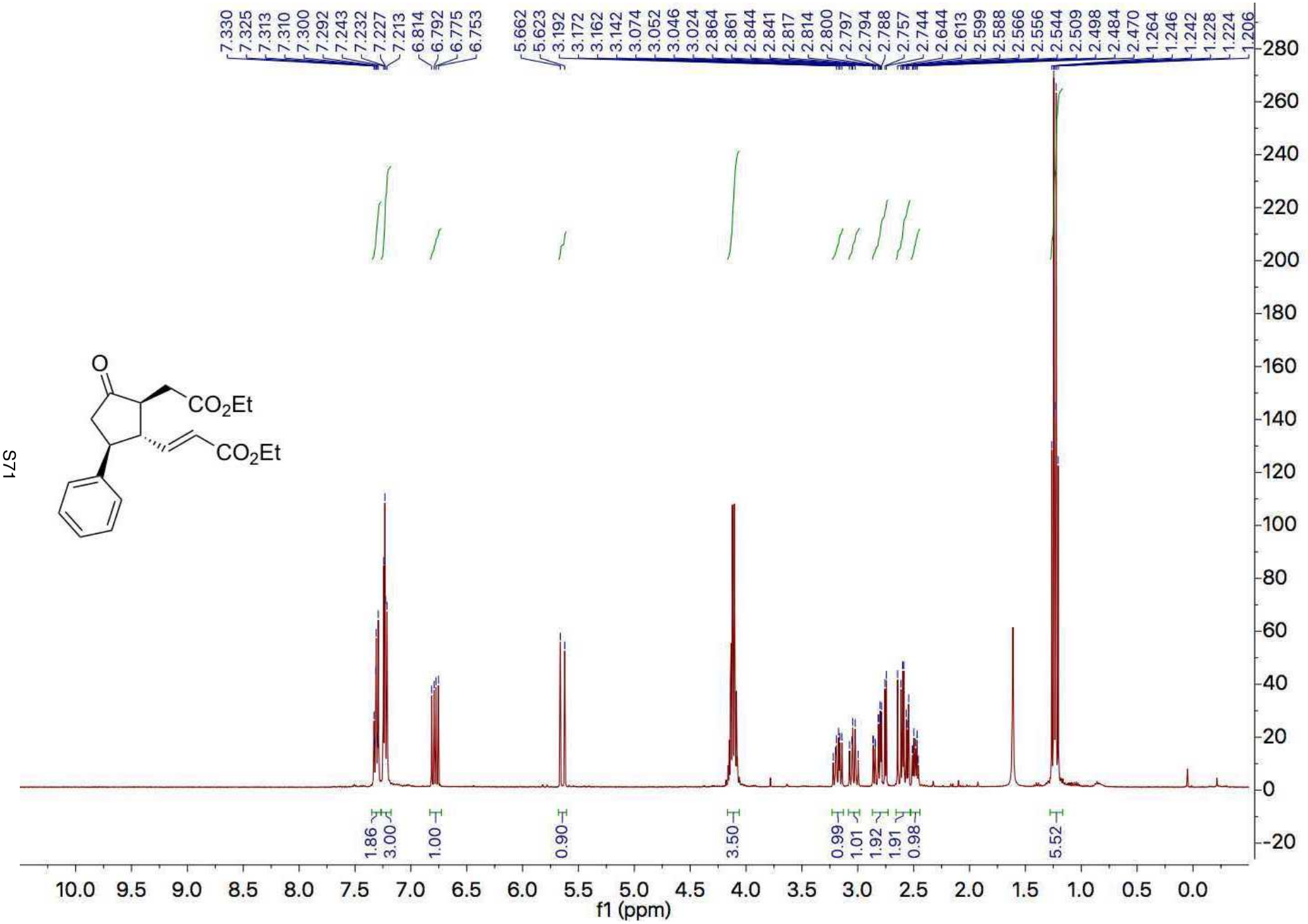

215.52
201.07
171.27
135.62
133.85
129.80
128.15
60.92
55.89
47.46
38.71
32.67
22.75
14.02
4.43
4.65
O
CO2Et
CHO
PhMe2Si
f1 (ppm)

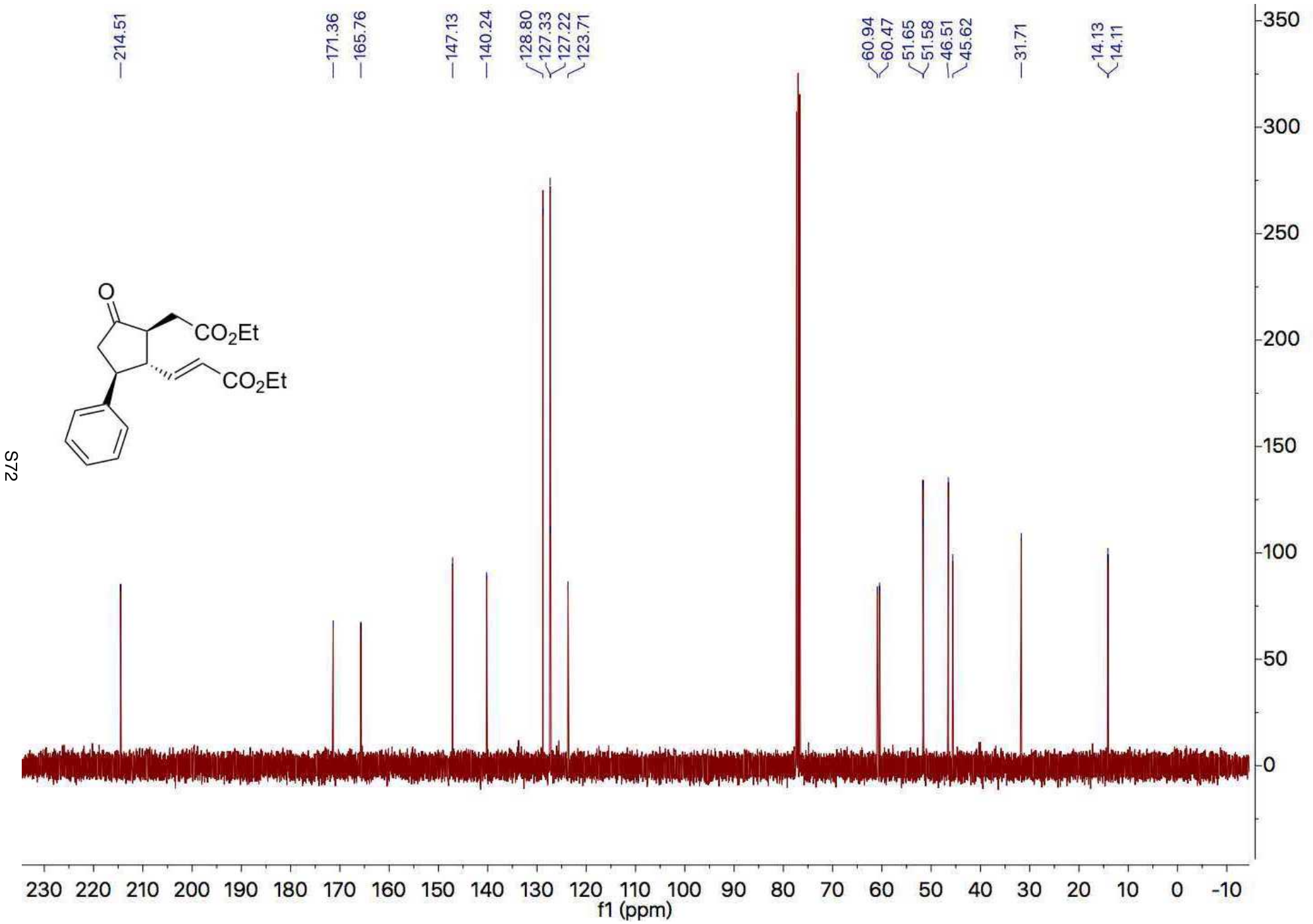


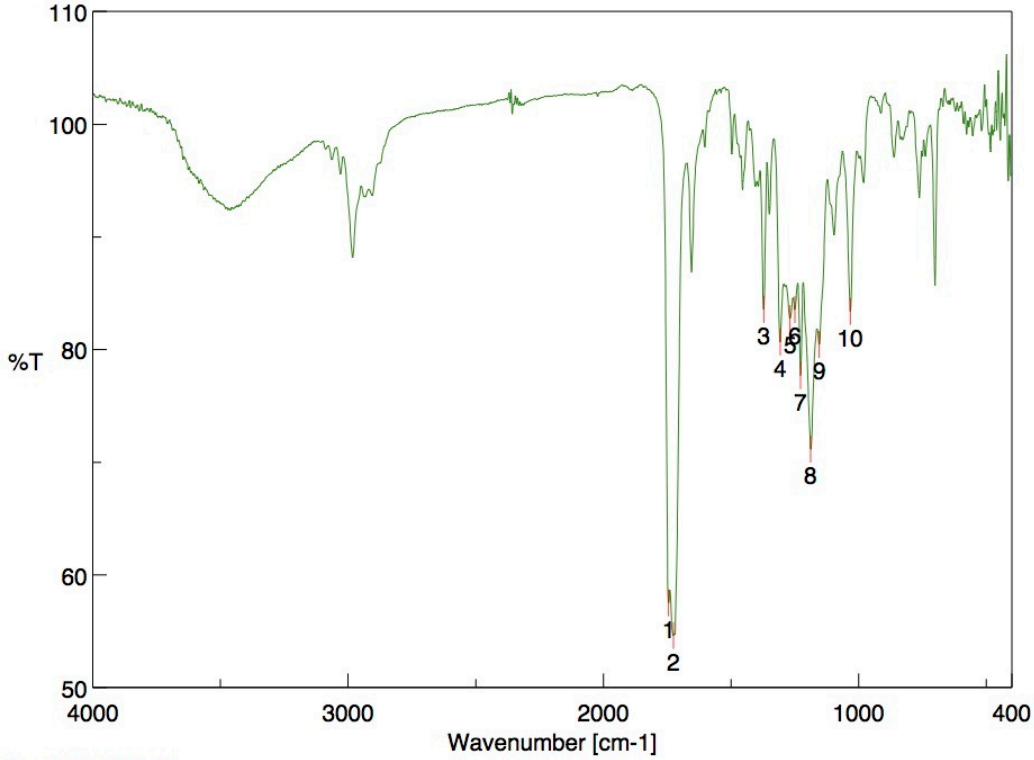
[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1725.01	63.2039	2	1427.07	83.7189
3	1375	82.6205	4	1253.5	75.1643
5	1189.86	73.2347	6	1113.69	78.17
7	1026.91	80.6785	8	835.026	75.5319
9	818.634	75.5323	10	775.244	82.79
11	736.674	78.1806	12	700.998	76.5157



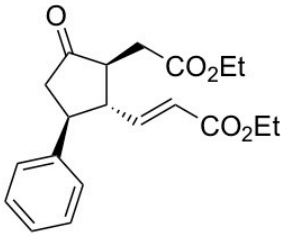

7.330
7.325
7.313
7.310
7.300
7.292
7.243
7.232
7.227
7.213
6.814
6.792
6.775
6.753
5.662
5.623
3.192
3.172
3.162
3.142
3.074
3.052
3.046
3.024
2.864
2.861
2.844
2.841
2.817
2.814
2.800
2.797
2.794
2.788
2.757
2.744
2.644
2.613
2.599
2.588
2.566
2.556
2.544
2.509
2.498
2.484
2.470
1.264
1.246
1.242
1.228
1.224
1.206
O
CO2Et
CO2Et
1.86
3.00
1.00
0.90
3.50
0.99
1.01
1.92
1.91
0.98
5.52
f1 (ppm)

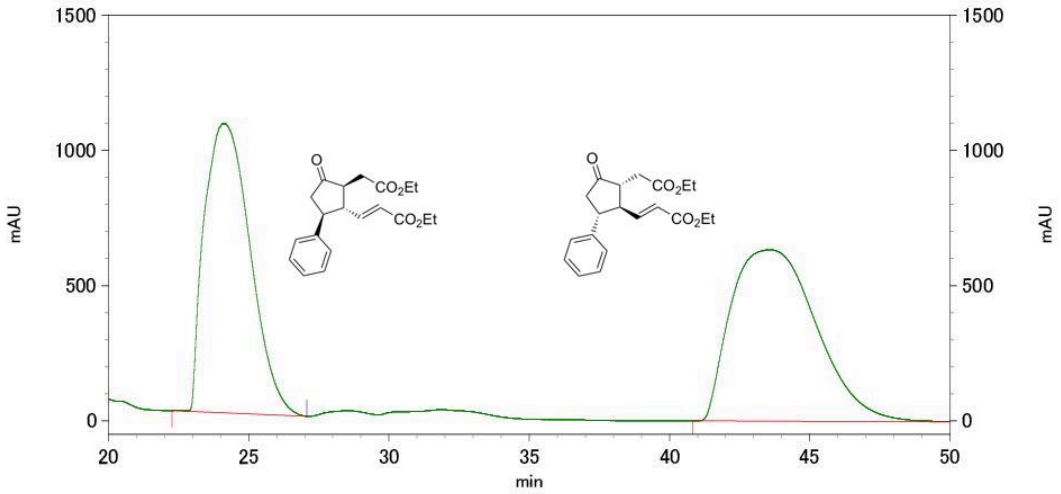

214.51
171.36
165.76
147.13
140.24
128.80
127.33
127.22
123.71
60.94
60.47
51.65
51.58
46.51
45.62
31.71
14.13
14.11
O
CO2Et
CO2Et
350
300
250
200
150
100
50
0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

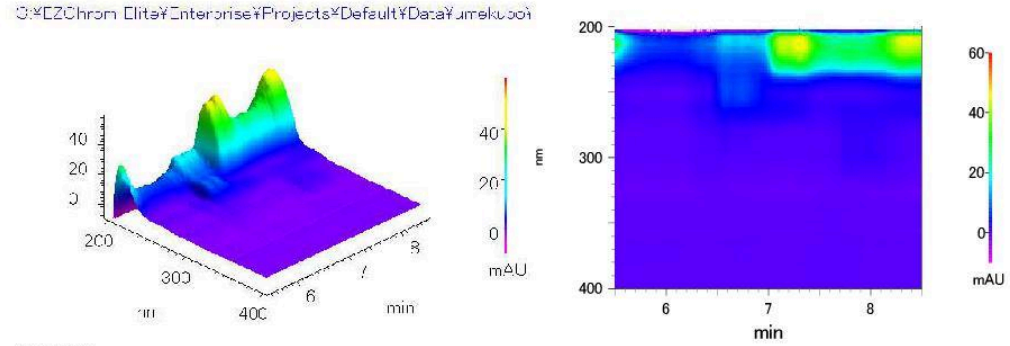


[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1744.3	57.5116	2	1725.98	54.6298
3	1372.1	83.5534	4	1307.5	80.6506
5	1268.93	82.7282	6	1249.65	83.5252
7	1227.47	77.6921	8	1187.94	71.1588
9	1154.19	80.4319	10	1032.69	83.3771



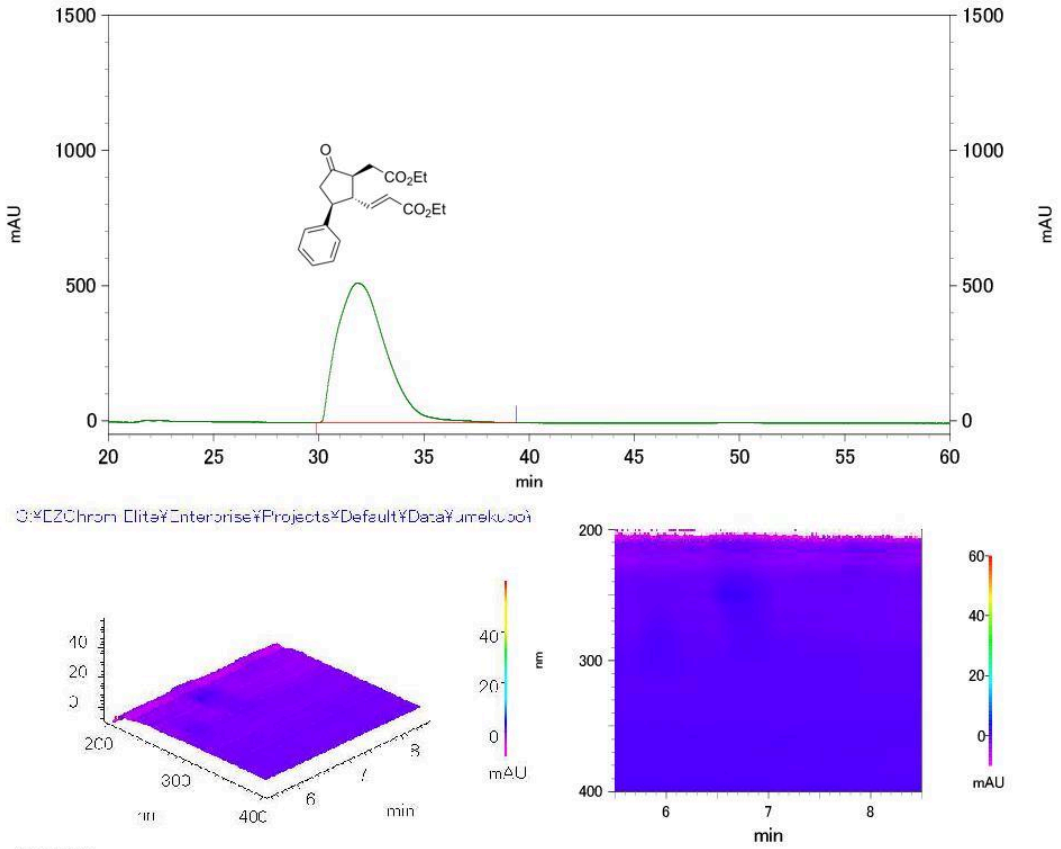




C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012107-ID-10vs1-racemi-Ph.dat

7: 214 nm, 4 nm結果

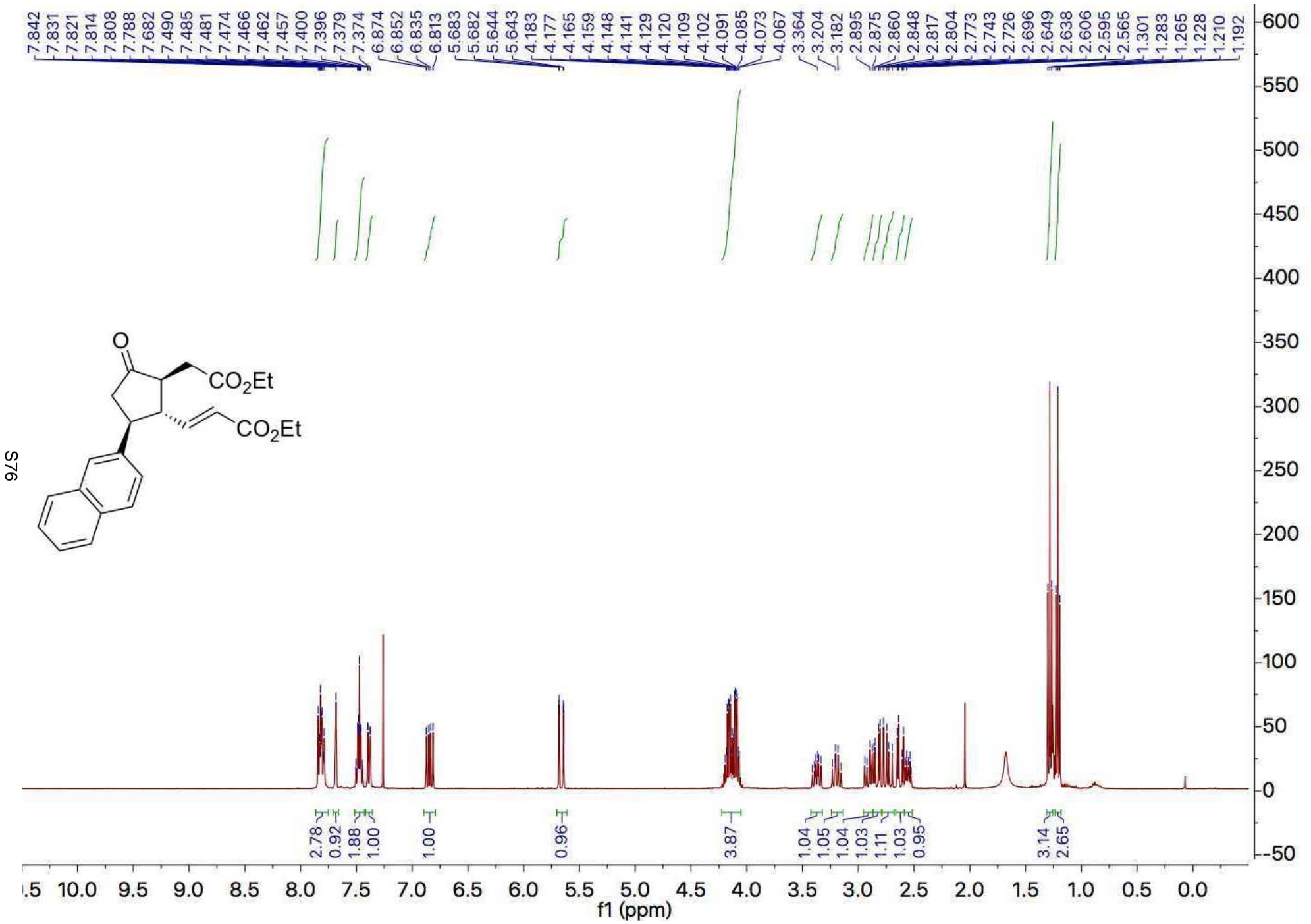
Pk #	Retention Time	Area	Area%
1	24.160	484960312	47.513
2	43.513	535729889	52.487
トータル		1020690201	100.000

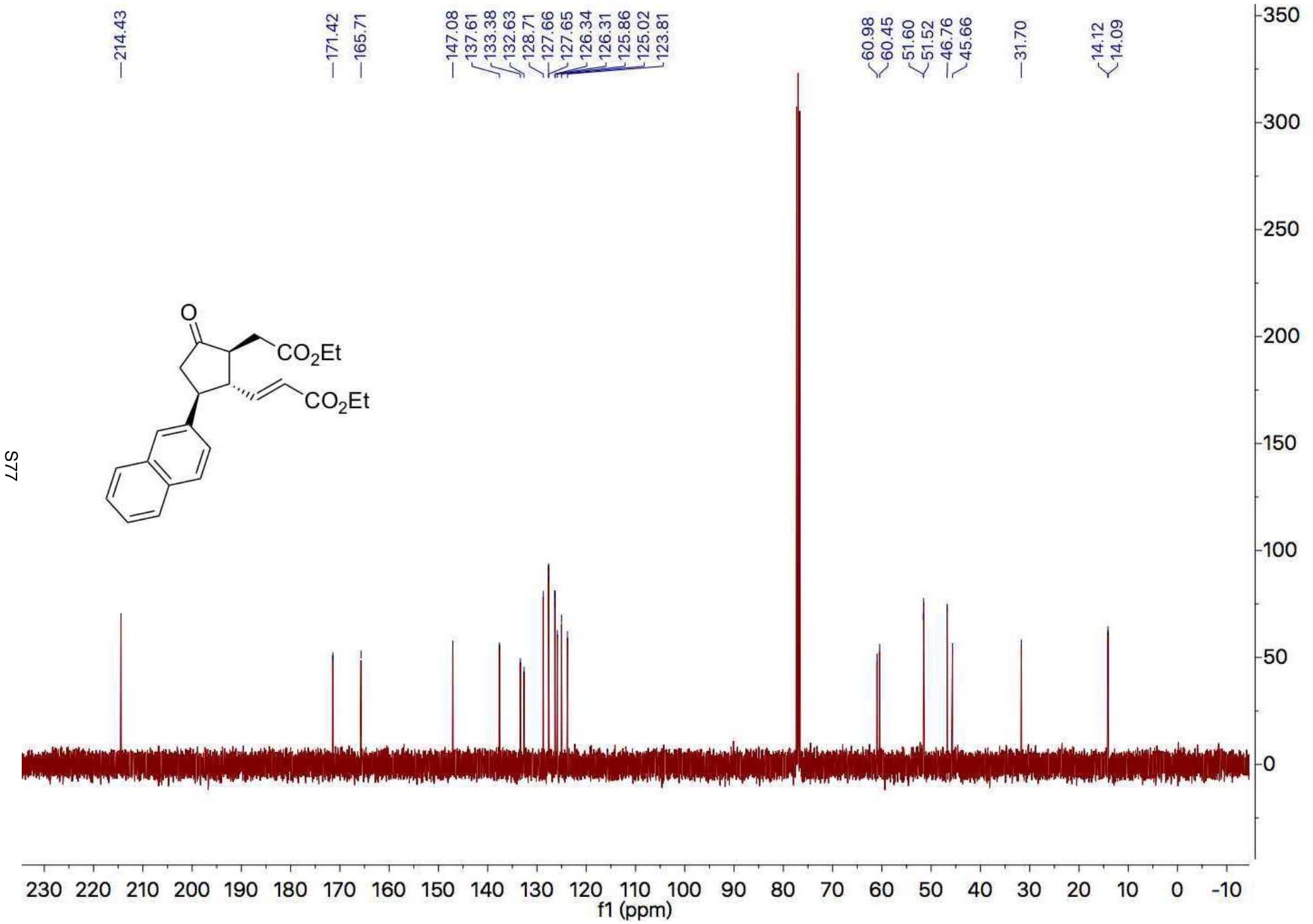


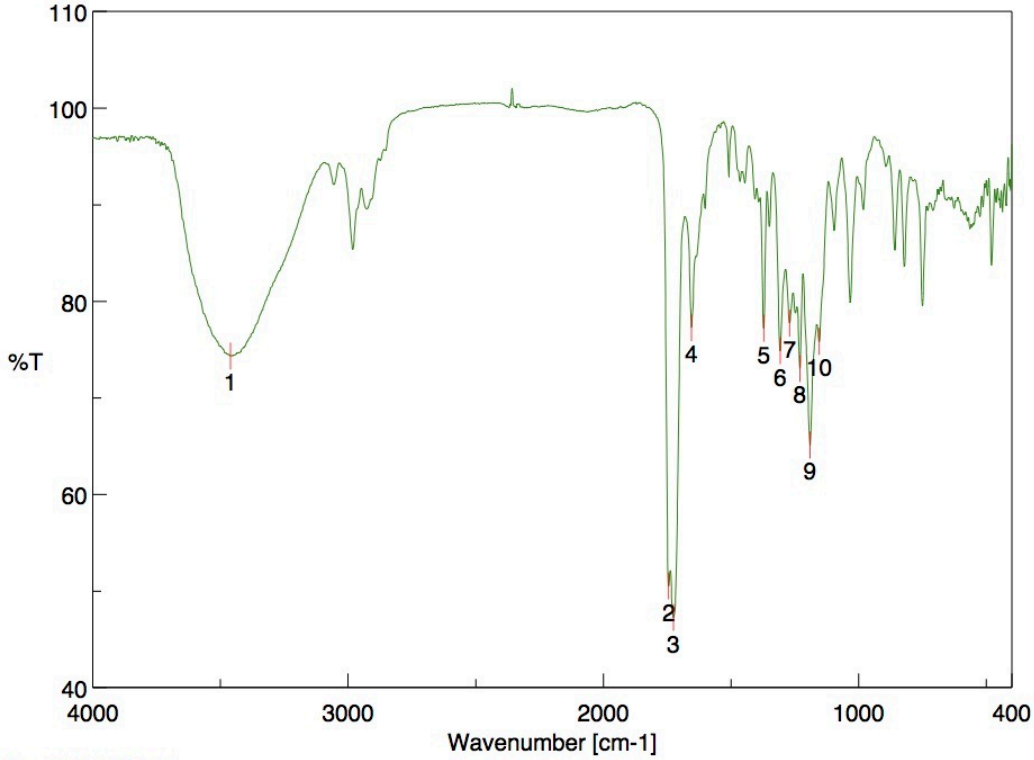
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012107-ID-10vs1-chira-Ph.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	31.820	328852081	100.000
トータル		328852081	100.000

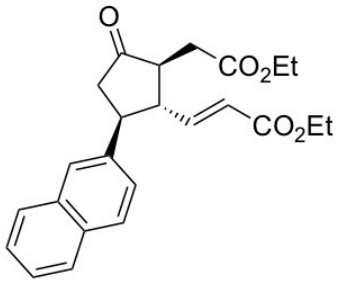

7.842
7.831
7.821
7.814
7.808
7.788
7.682
7.490
7.485
7.481
7.474
7.466
7.462
7.457
7.400
7.396
7.379
7.374
6.874
6.852
6.835
6.813
5.683
5.682
5.644
5.643
4.183
4.177
4.165
4.159
4.148
4.141
4.129
4.120
4.109
4.102
4.091
4.085
4.073
4.067
3.364
3.204
3.182
2.895
2.875
2.860
2.848
2.817
2.804
2.773
2.743
2.726
2.696
2.649
2.638
2.606
2.595
2.565
1.301
1.283
1.265
1.228
1.210
1.192
O
CO2Et
CO2Et
2.78
0.92
1.88
1.00
1.00
0.96
3.87
1.04
1.05
1.04
1.03
1.11
1.03
0.95
3.14
2.65
f1 (ppm)

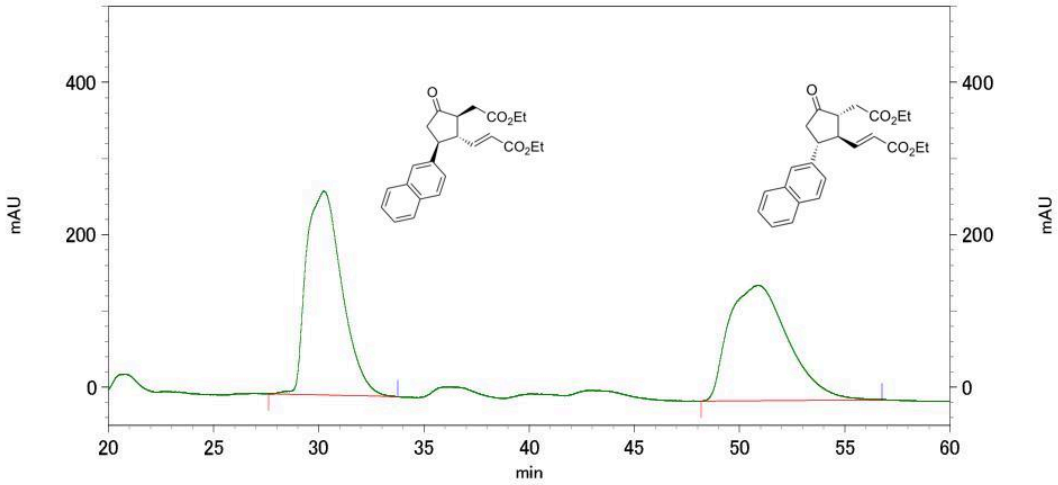

214.43
171.42
165.71
147.08
137.61
133.38
132.63
128.71
127.66
127.65
126.34
126.31
125.86
125.02
123.81
60.98
60.45
51.60
51.52
46.76
45.66
31.70
14.12
14.09
O
CO2Et
CO2Et
350
300
250
200
150
100
50
0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

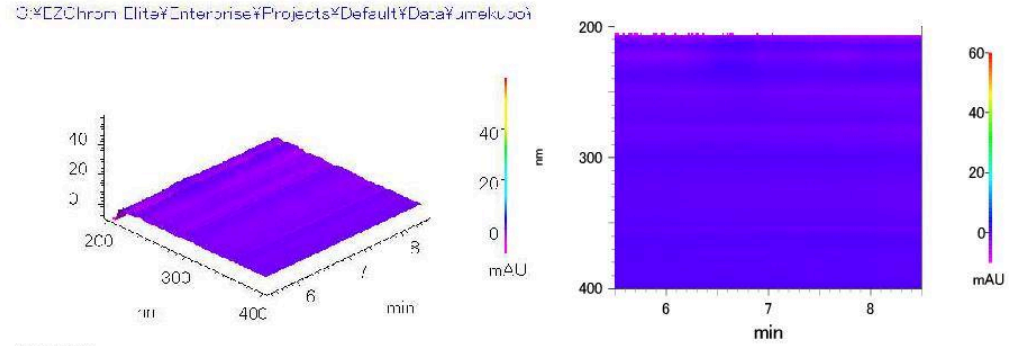


[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	3460.63	74.3165	2	1744.3	50.5229
3	1725.01	47.2506	4	1654.62	77.2699
5	1372.1	77.2071	6	1307.5	74.862
7	1270.86	77.7381	8	1230.36	73.0228
9	1190.83	65.1136	10	1154.19	75.8348



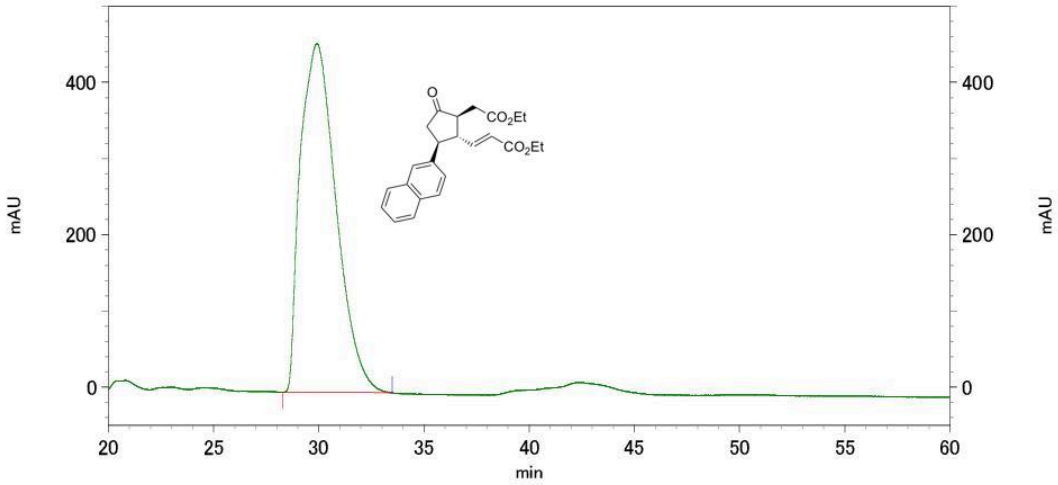


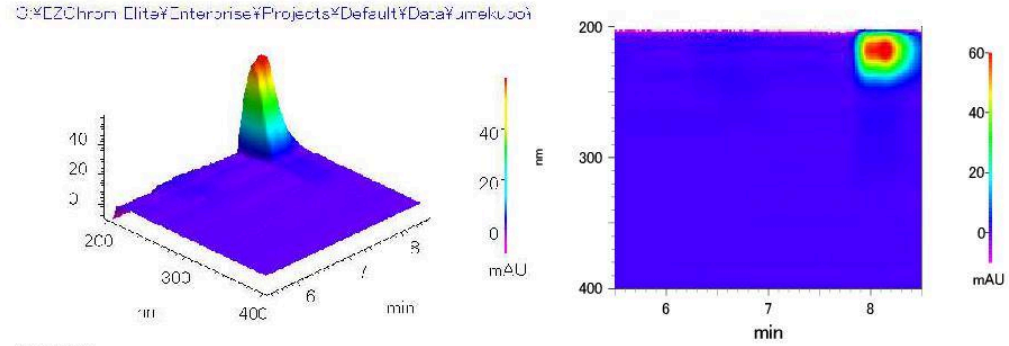


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012121-ID-10vs1-racemi-nap.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	30.240	126214769	51.612
2	50.873	118332620	48.388
トータル		244547389	100.000

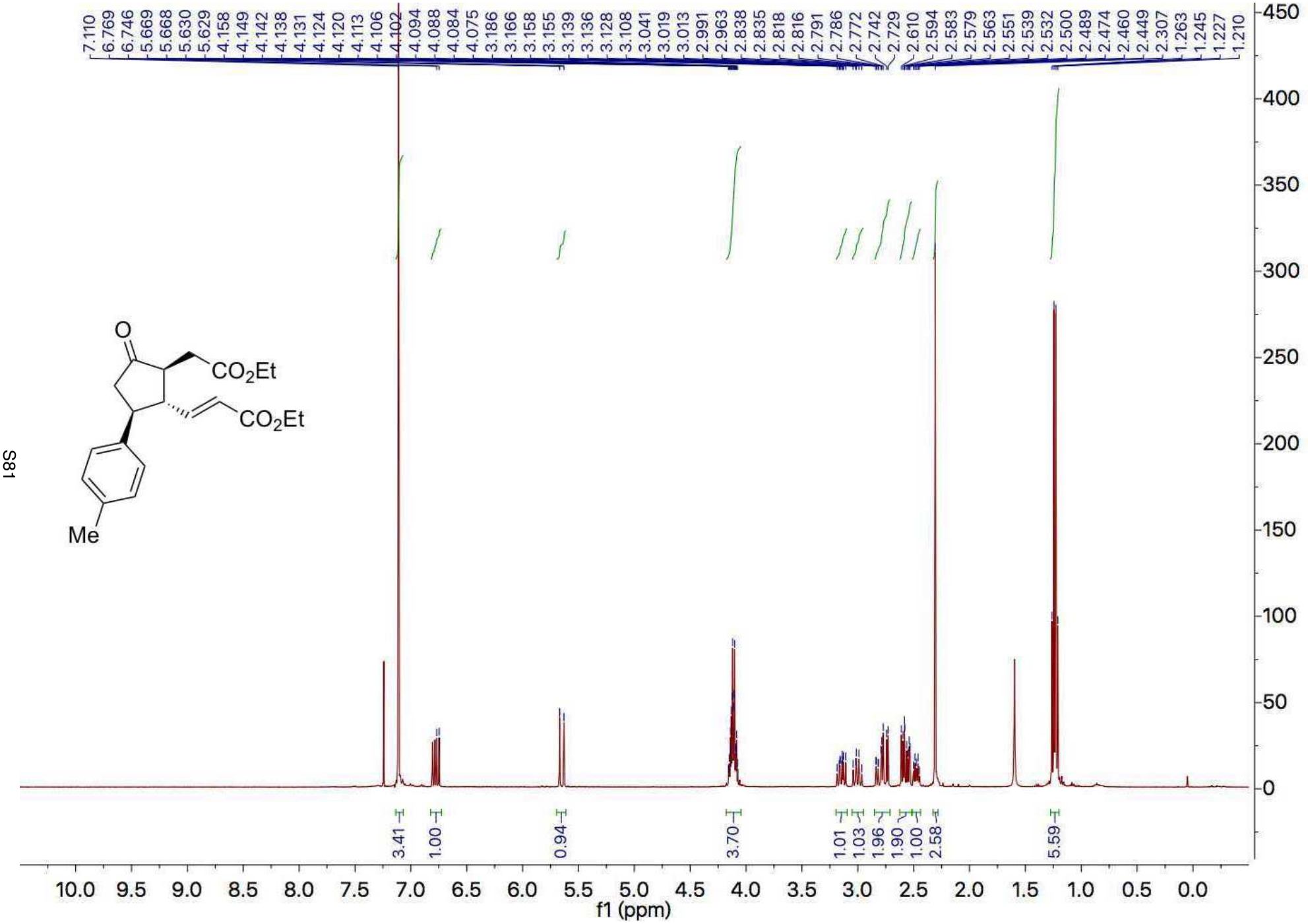


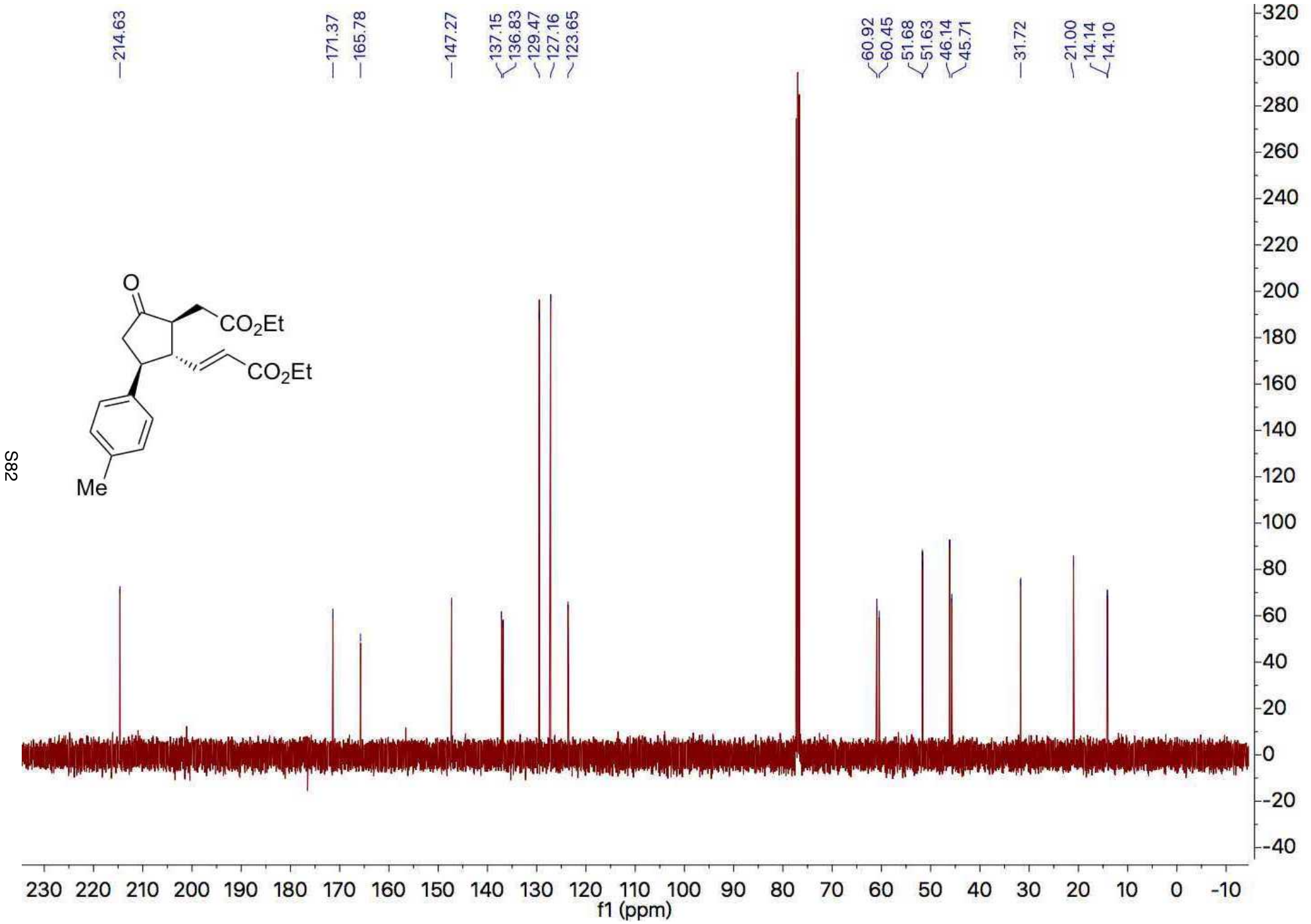


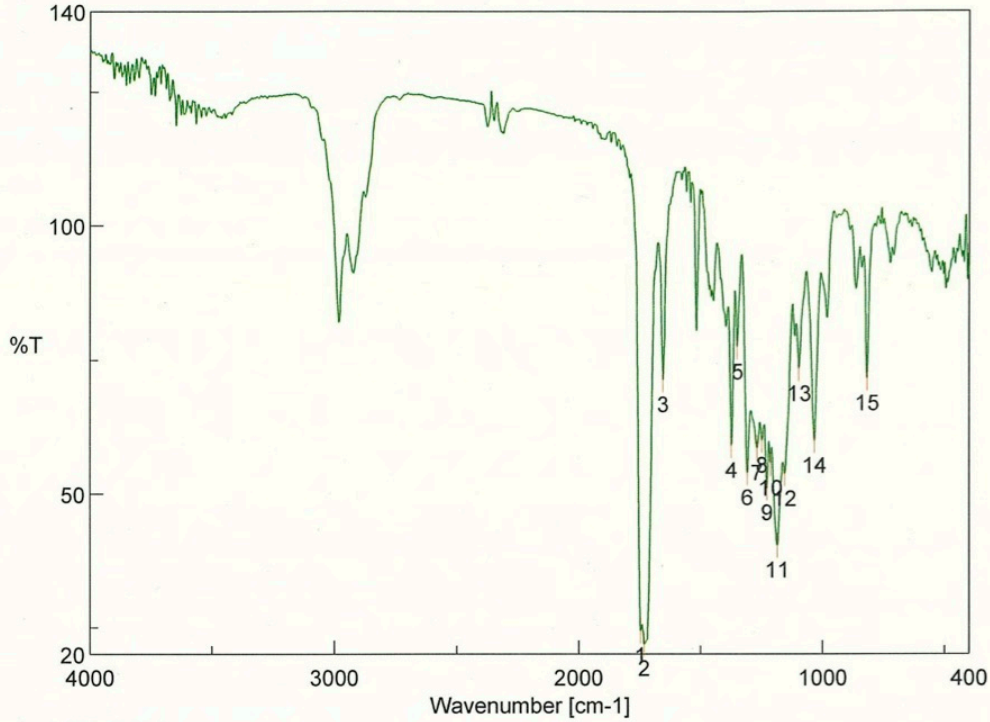
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012121-ID-10vs1-chiral-nap.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	29.907	216507416	100.000
トータル		216507416	100.000

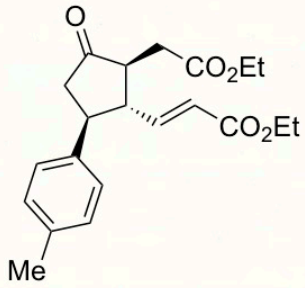

7.110
6.769
6.746
5.669
5.668
5.630
5.629
4.158
4.149
4.142
4.138
4.131
4.124
4.120
4.113
4.106
4.102
4.094
4.088
4.084
4.075
3.186
3.166
3.158
3.155
3.139
3.136
3.128
3.108
3.041
3.019
3.013
2.991
2.963
2.838
2.835
2.818
2.816
2.791
2.786
2.772
2.742
2.729
2.610
2.594
2.583
2.579
2.563
2.551
2.539
2.532
2.500
2.489
2.474
2.460
2.449
2.307
1.263
1.245
1.227
1.210
O
CO2Et
CO2Et
Me
3.41
1.00
0.94
3.70
1.01
1.03
1.96
1.90
1.00
2.58
5.59
f1 (ppm)

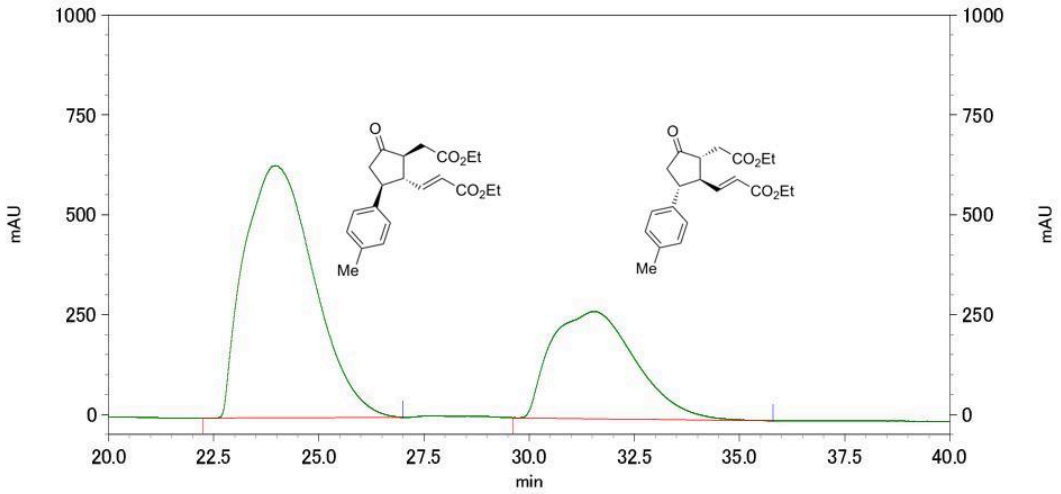

214.63
171.37
165.78
147.27
137.15
136.83
129.47
127.16
123.65
60.92
60.45
51.68
51.63
46.14
45.71
31.72
21.00
14.14
14.10
O
CO2Et
CO2Et
Me
f1 (ppm)

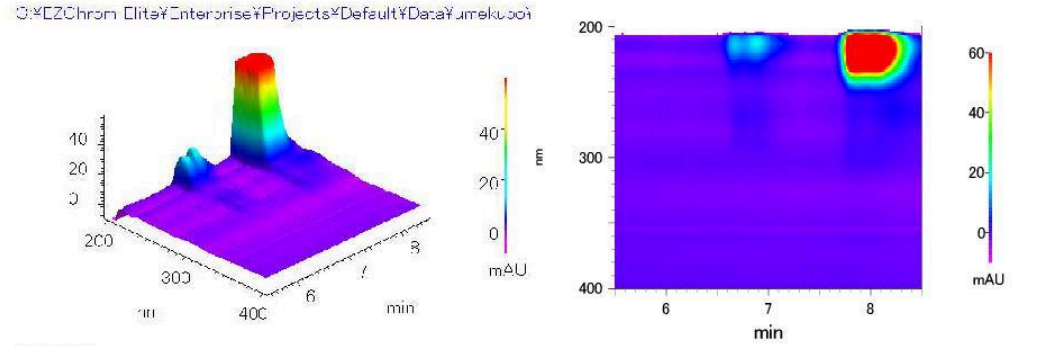


[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1746.23	24.3822	2	1730.8	21.9545
3	1654.62	71.2961	4	1373.07	58.9965
5	1348.96	77.4473	6	1308.46	53.8523
7	1268.93	58.5529	8	1248.68	59.9357
9	1228.43	51.0331	10	1213.97	55.633
11	1185.04	40.3121	12	1155.15	53.6336
13	1096.33	73.3191	14	1033.66	59.9092
15	817.67	71.5321			



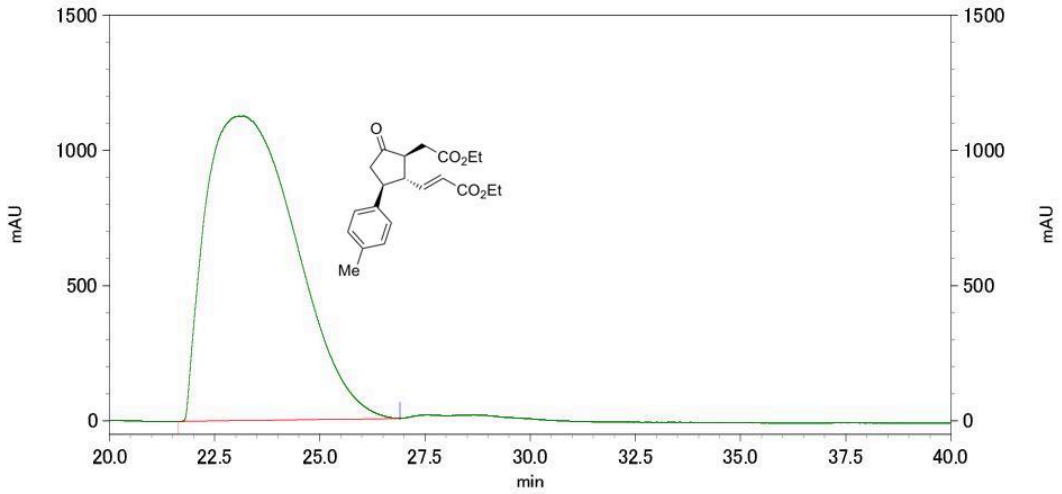


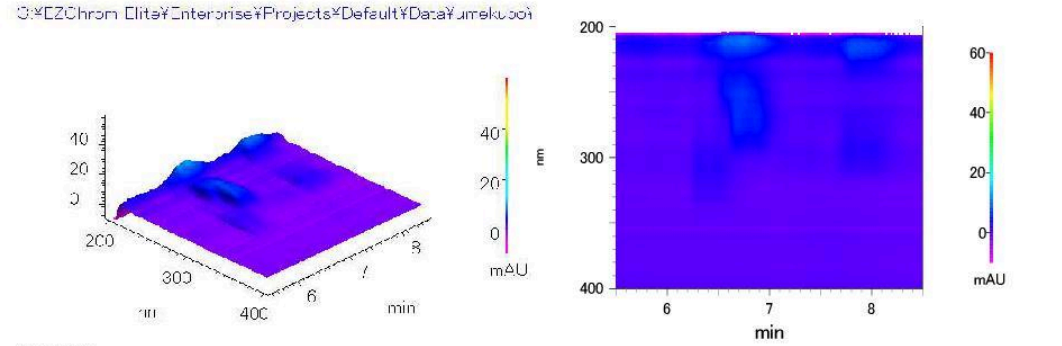


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012118-ID-10vs1-racemi-tol.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	23.960	290309264	66.053
2	31.547	149198877	33.947
トータル		439508141	100.000

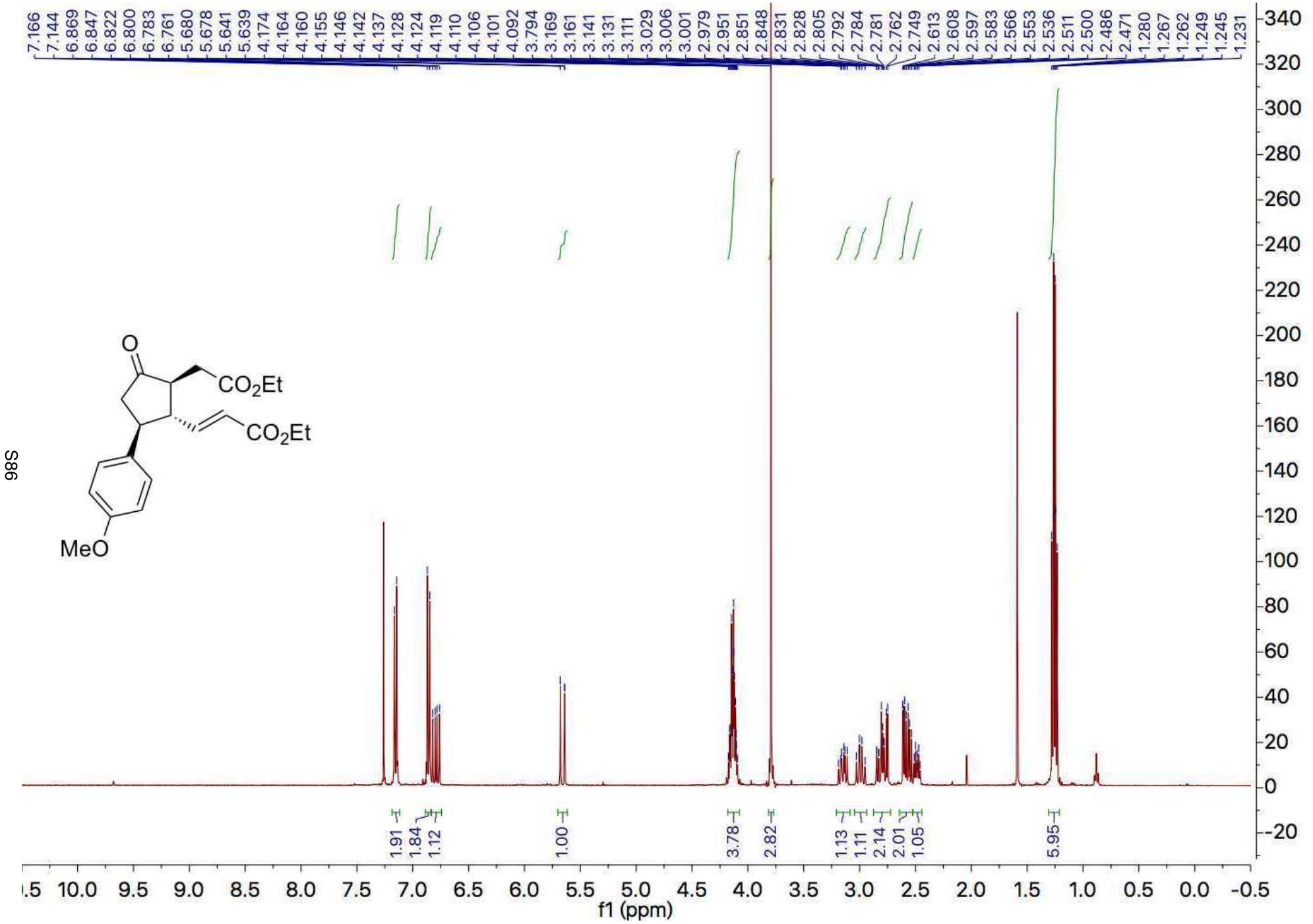


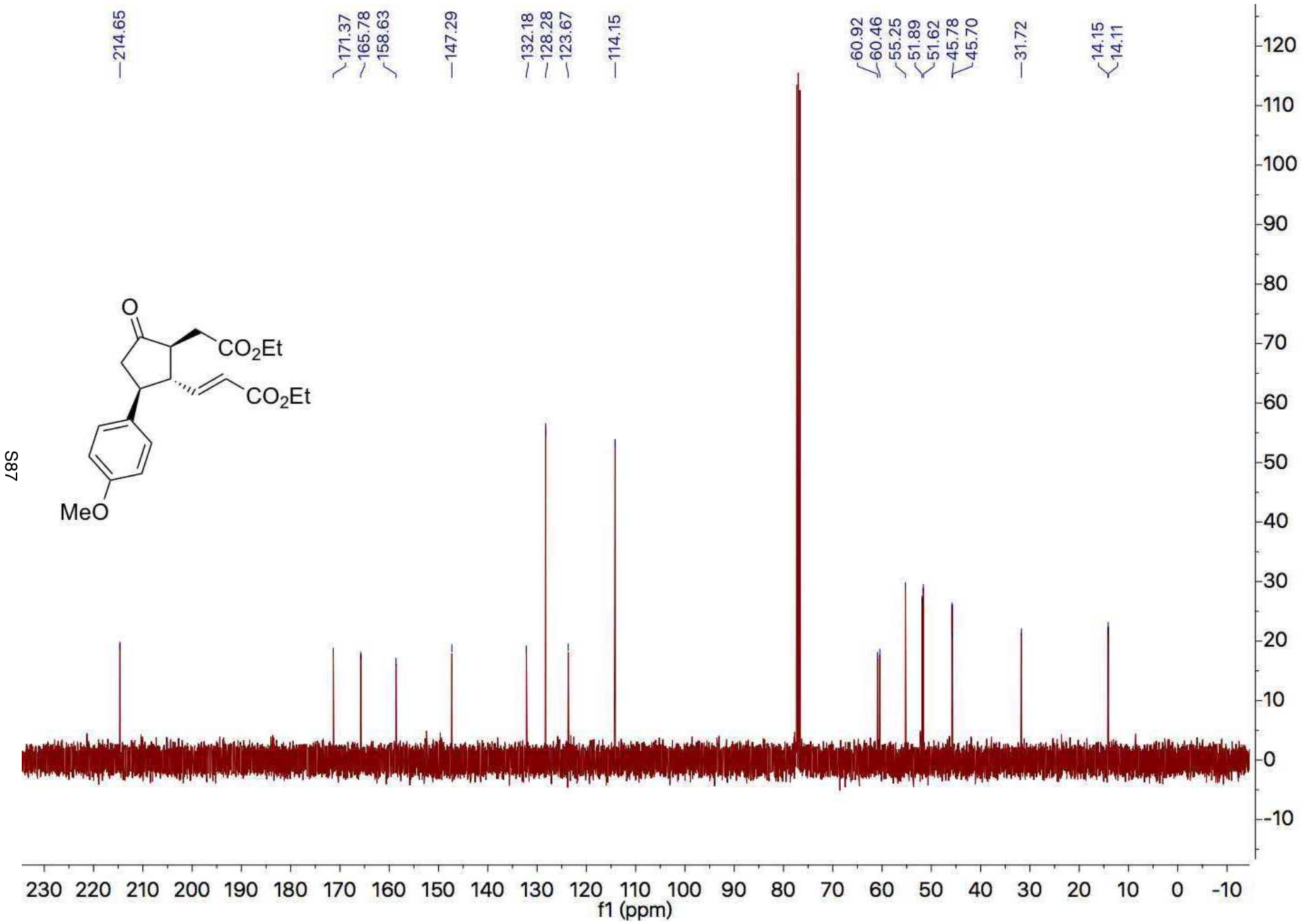


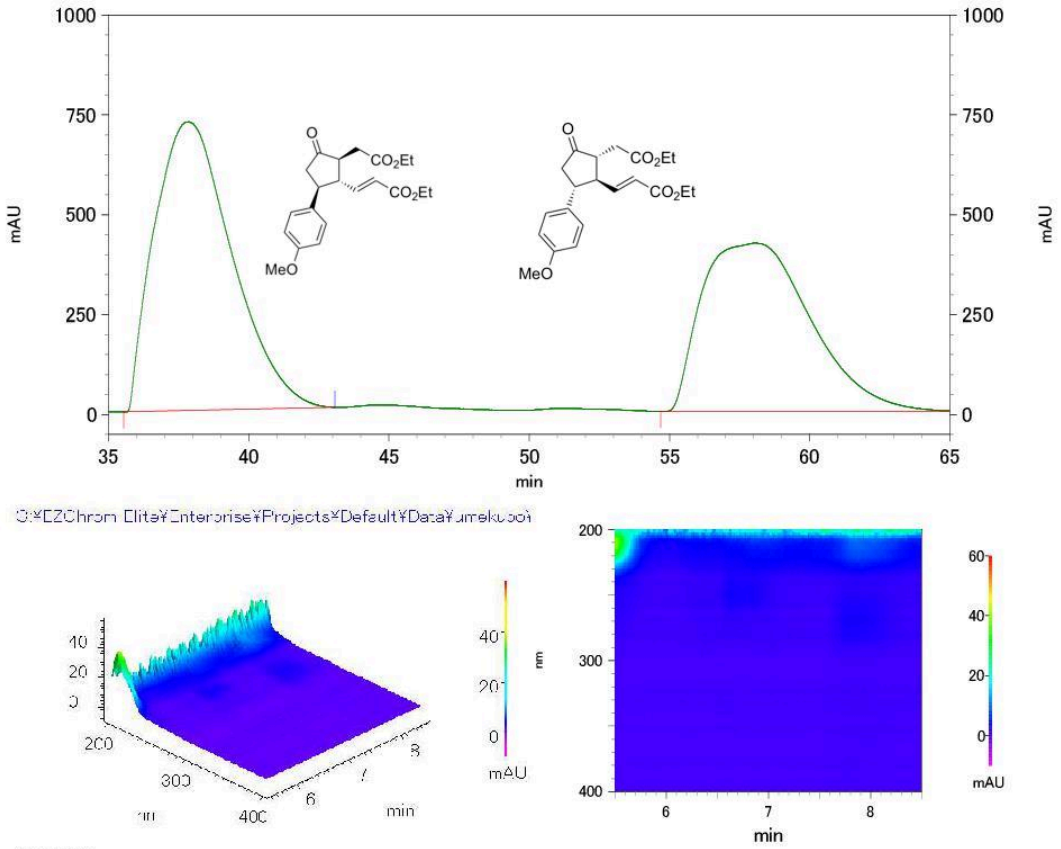
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012118-ID-10vs1-chiral-tol.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	23.087	673205833	100.000
トータル		673205833	100.000


7.166
7.144
6.869
6.847
6.822
6.800
6.783
6.761
5.680
5.678
5.641
5.639
4.174
4.164
4.160
4.155
4.146
4.142
4.137
4.128
4.124
4.119
4.110
4.106
4.101
4.092
3.794
3.169
3.161
3.141
3.131
3.111
3.029
3.006
3.001
2.979
2.951
2.851
2.848
2.831
2.828
2.805
2.792
2.784
2.781
2.762
2.749
2.613
2.608
2.597
2.583
2.566
2.553
2.536
2.511
2.500
2.486
2.471
1.280
1.267
1.262
1.249
1.245
1.231
O
CO2Et
CO2Et
MeO
1.91
1.84
1.12
1.00
3.78
2.82
1.13
1.11
2.14
2.01
1.05
5.95
f1 (ppm)

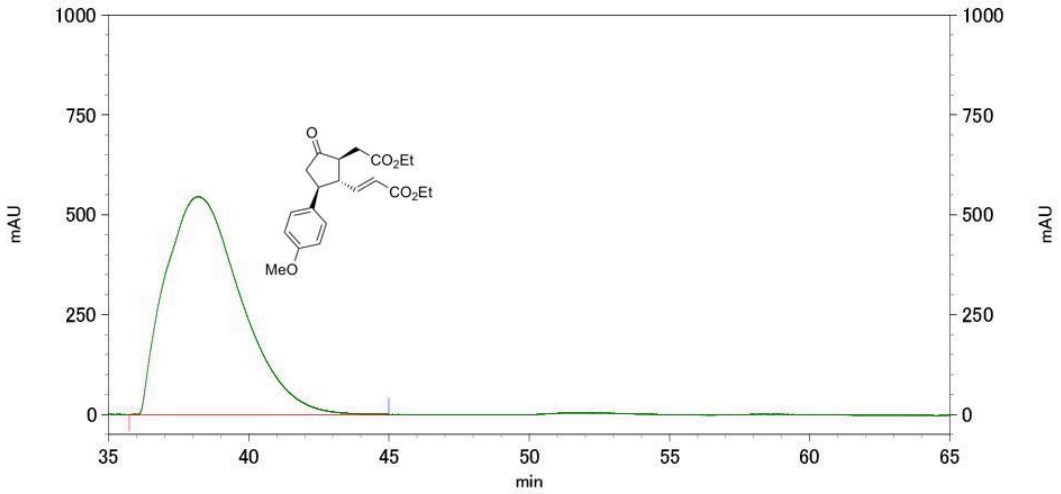

214.65
171.37
165.78
158.63
147.29
132.18
128.28
123.67
114.15
60.92
60.46
55.25
51.89
51.62
45.78
45.70
31.72
14.15
14.11
O
CO2Et
CO2Et
MeO
f1 (ppm)

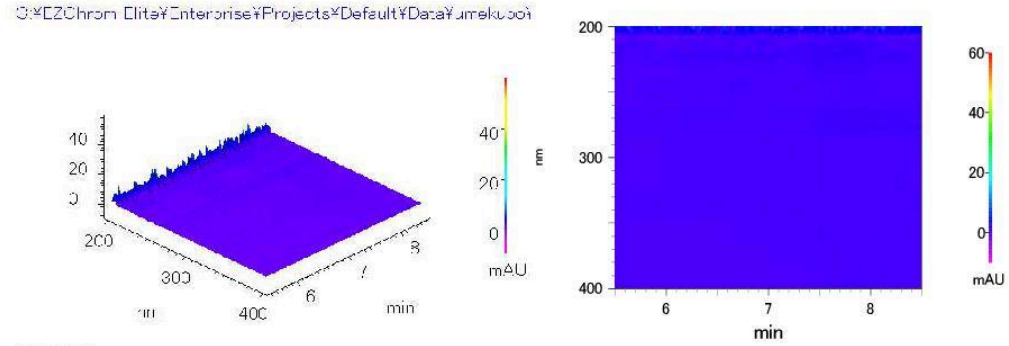


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012128-ID-10vs1-racemi-p-OMe.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	37.860	555274838	55.035
2	58.100	453677450	44.965
トータル		1008952288	100.000

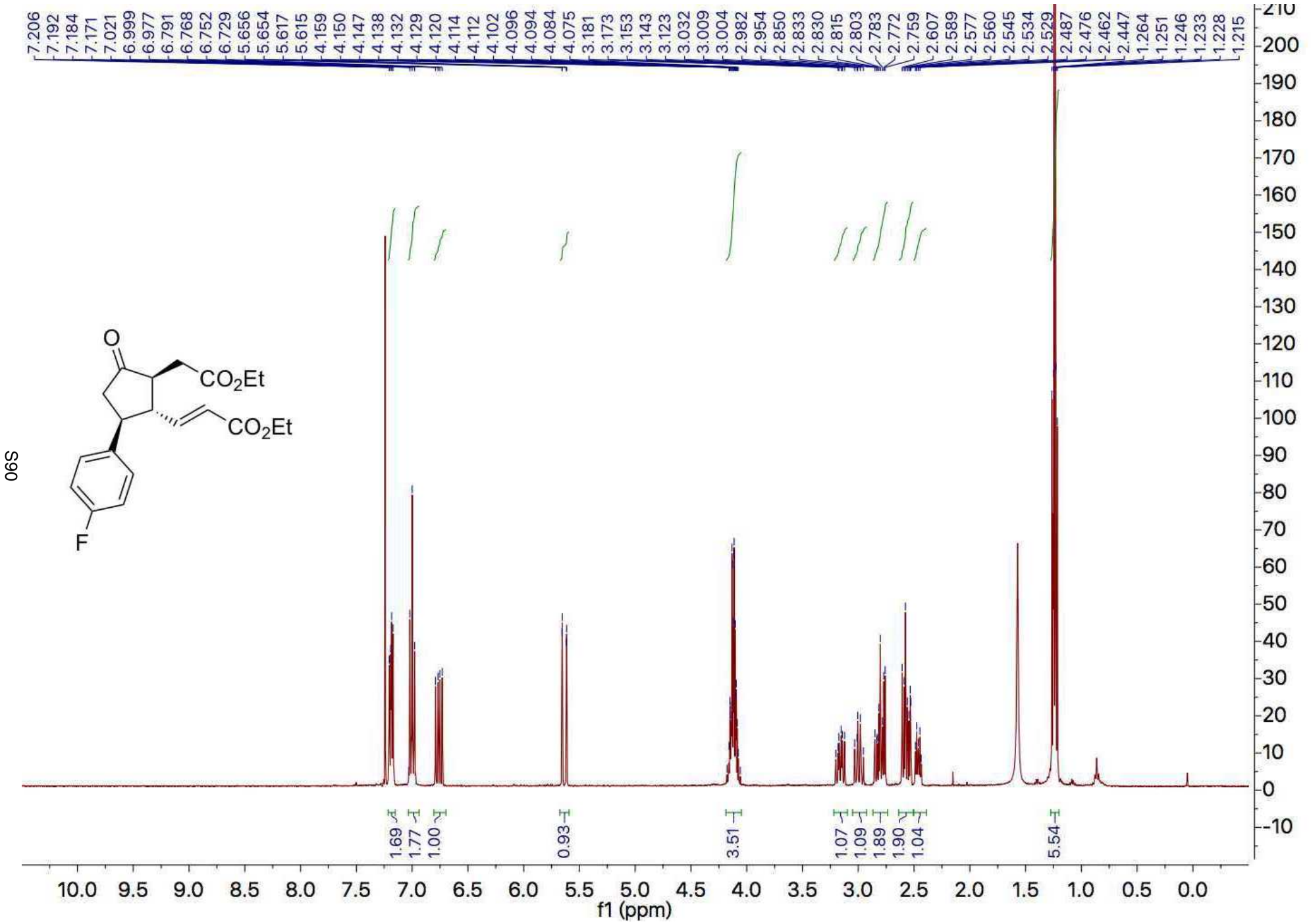


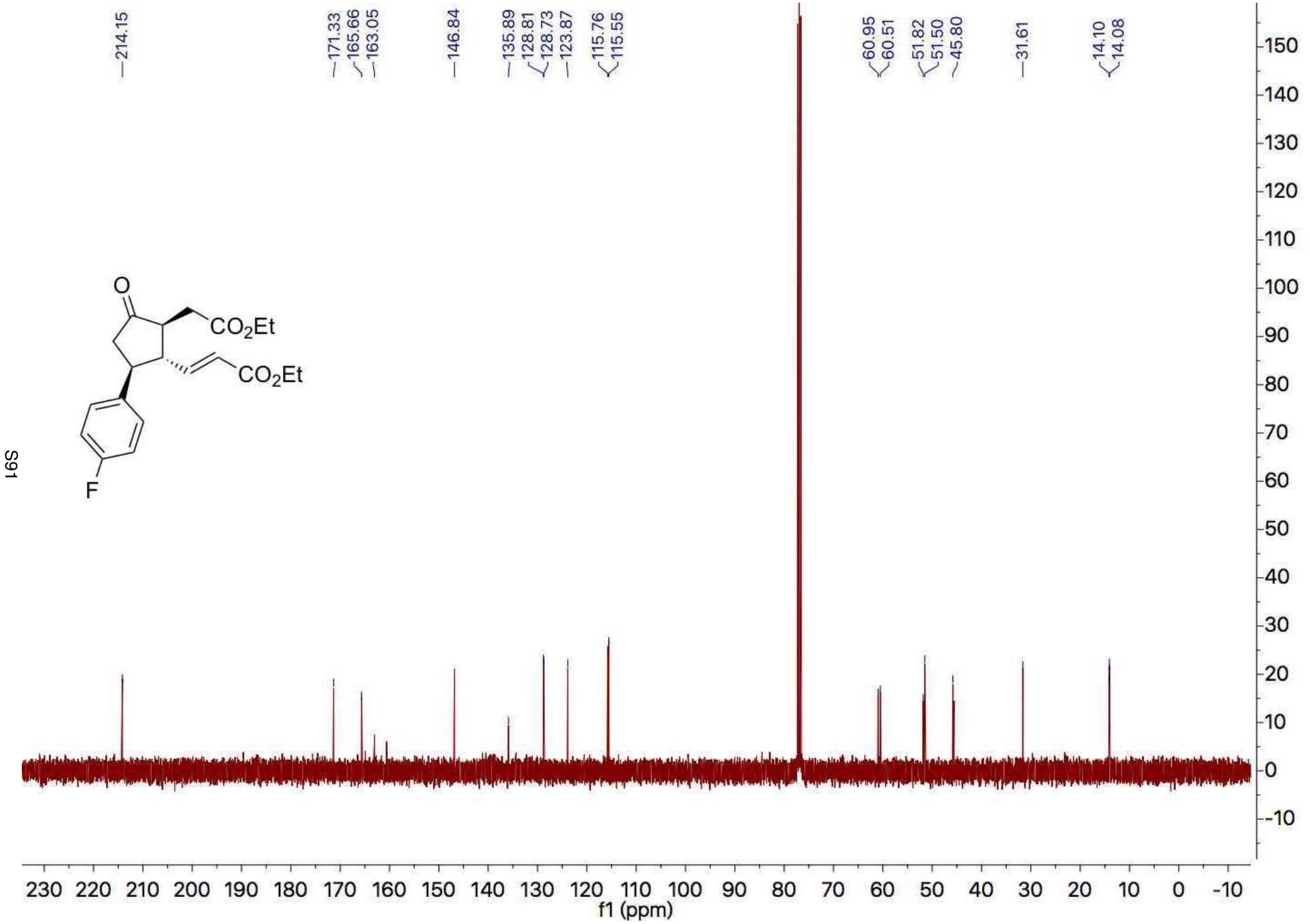


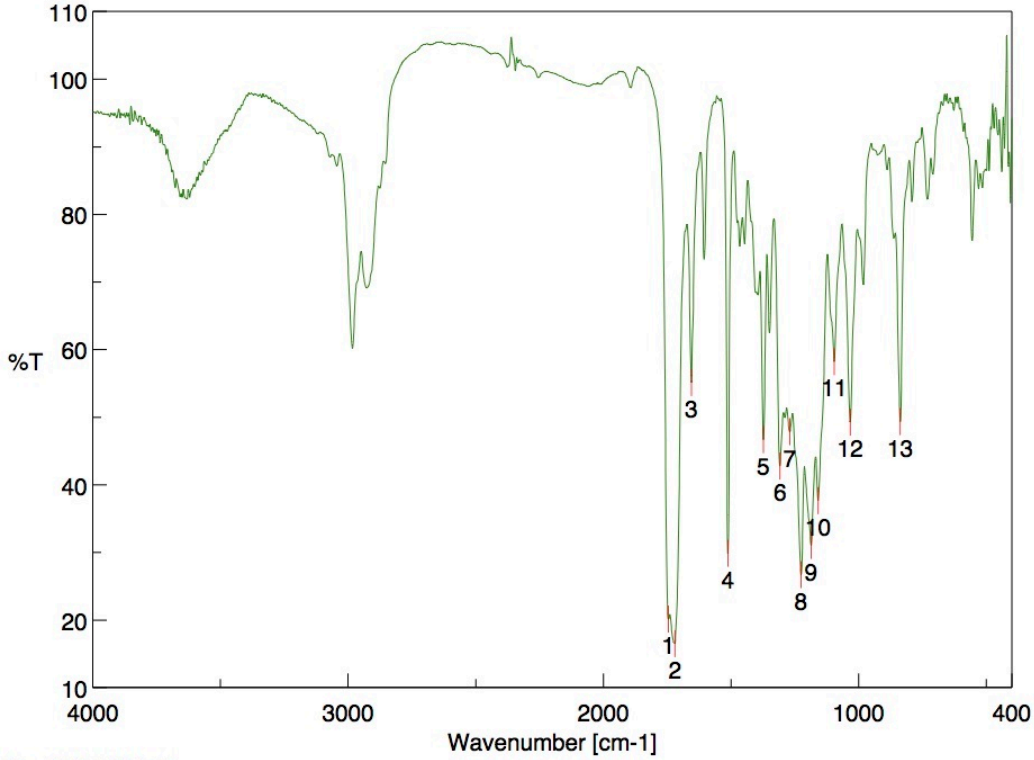
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012128-ID-10vs1-chiral-p-OMe.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	38.207	408356578	100.000
トータル		408356578	100.000

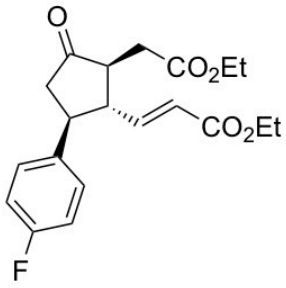

7.206 7.192 7.184 7.171 7.021 6.999 6.977 6.791 6.768 6.752 6.729 6.656 5.654 5.617 5.615 4.159 4.150 4.147 4.138 4.132 4.129 4.120 4.114 4.112 4.102 4.096 4.094 4.084 4.075 3.181 3.173 3.153 3.143 3.123 3.032 3.009 3.004 2.982 2.954 2.850 2.833 2.830 2.815 2.803 2.783 2.772 2.759 2.607 2.589 2.577 2.560 2.545 2.534 2.529 2.487 2.476 2.462 2.447 1.264 1.251 1.246 1.233 1.228 1.215
O
CO2Et
CO2Et
F
1.69 1.77 1.00 0.93 3.51 1.07 1.09 1.89 1.90 1.04 5.54
f1 (ppm)

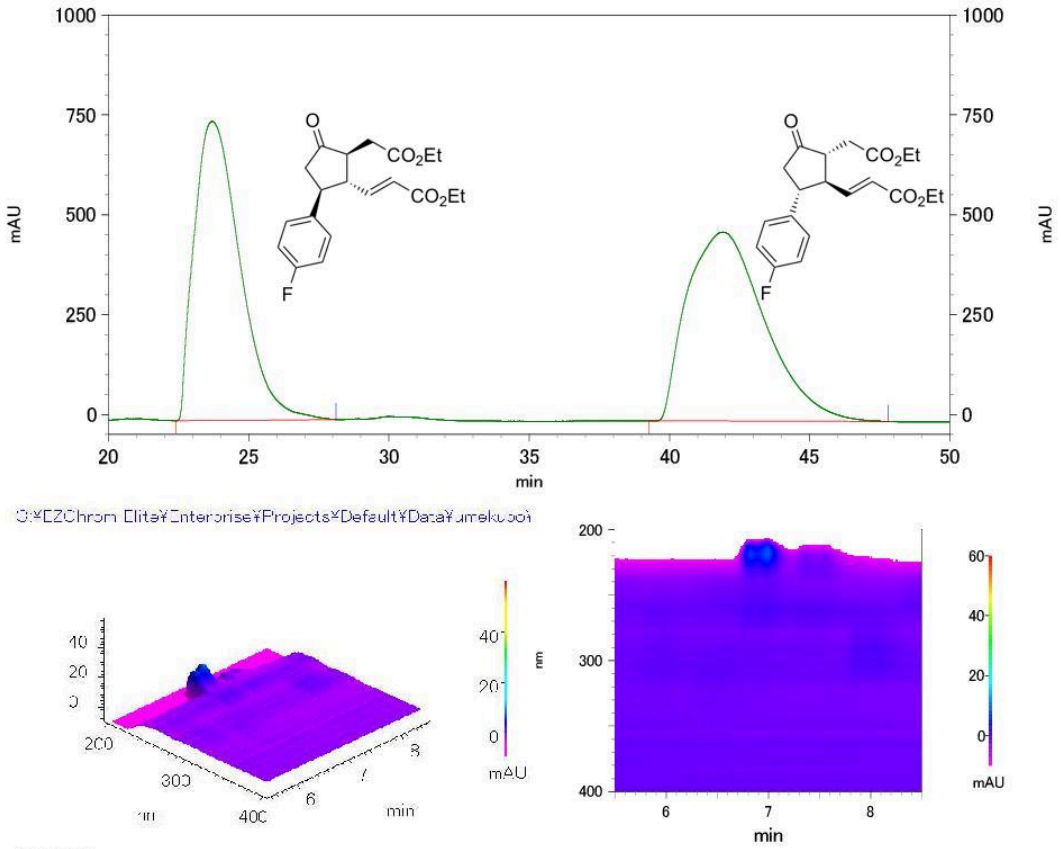

214.15
171.33
165.66
163.05
146.84
135.89
128.81
128.73
123.87
115.76
115.55
60.95
60.51
51.82
51.50
45.80
31.61
14.10
14.08
O
CO2Et
CO2Et
F
f1 (ppm)



[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1745.26	20.116	2	1720.19	16.4958
3	1654.62	55.1218	4	1511.92	29.8406
5	1373.07	46.6583	6	1308.46	42.7673
7	1269.9	47.8079	8	1225.54	26.667
9	1186.01	31.0044	10	1159.01	37.6156
11	1095.37	58.2072	12	1032.69	49.26
13	836.955	49.3131			

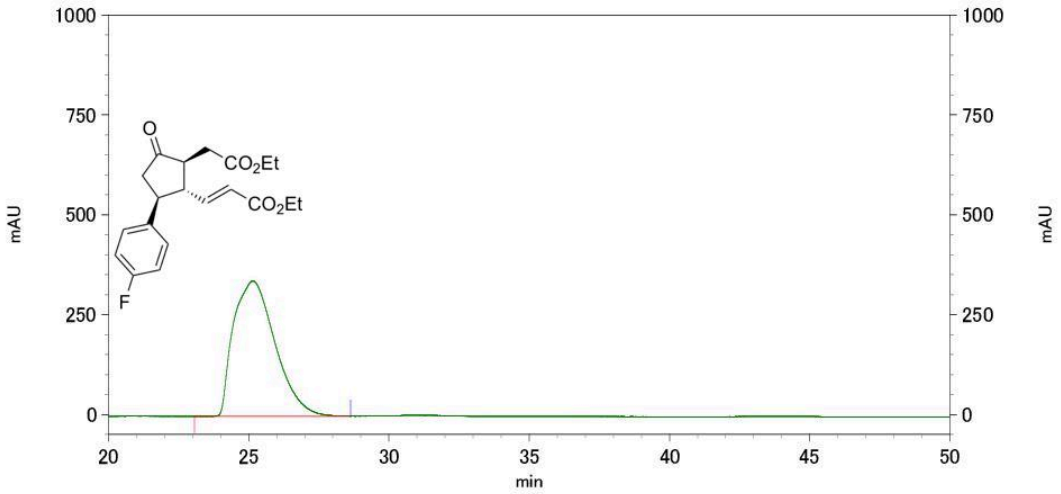


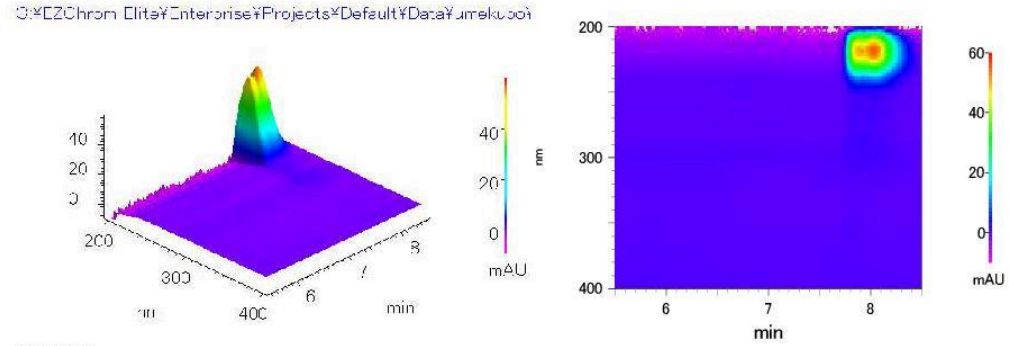


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012125-ID-10vs1-racemi-F.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	23.727	339259155	48.199
2	41.900	364616622	51.801
トータル		703875777	100.000

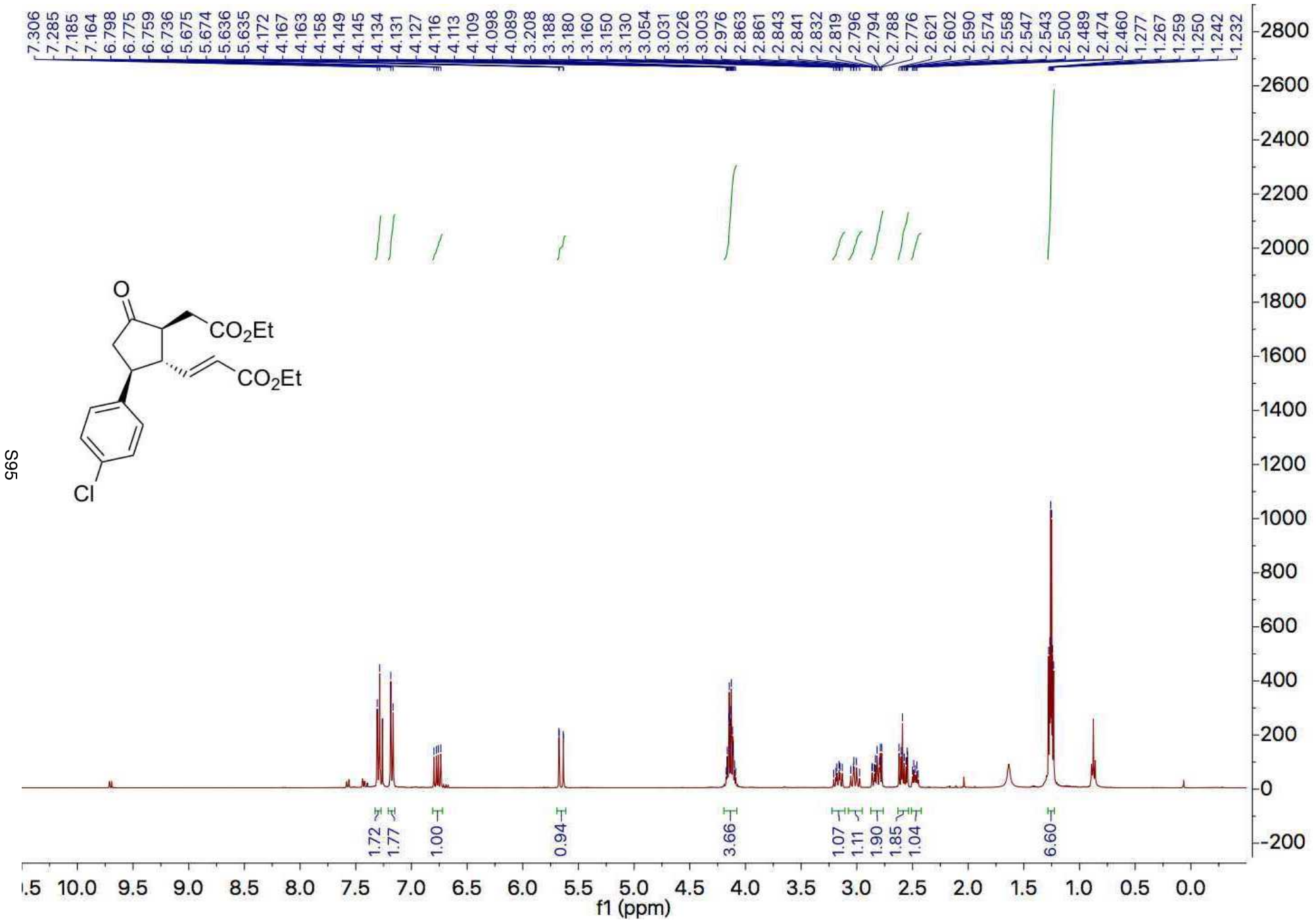


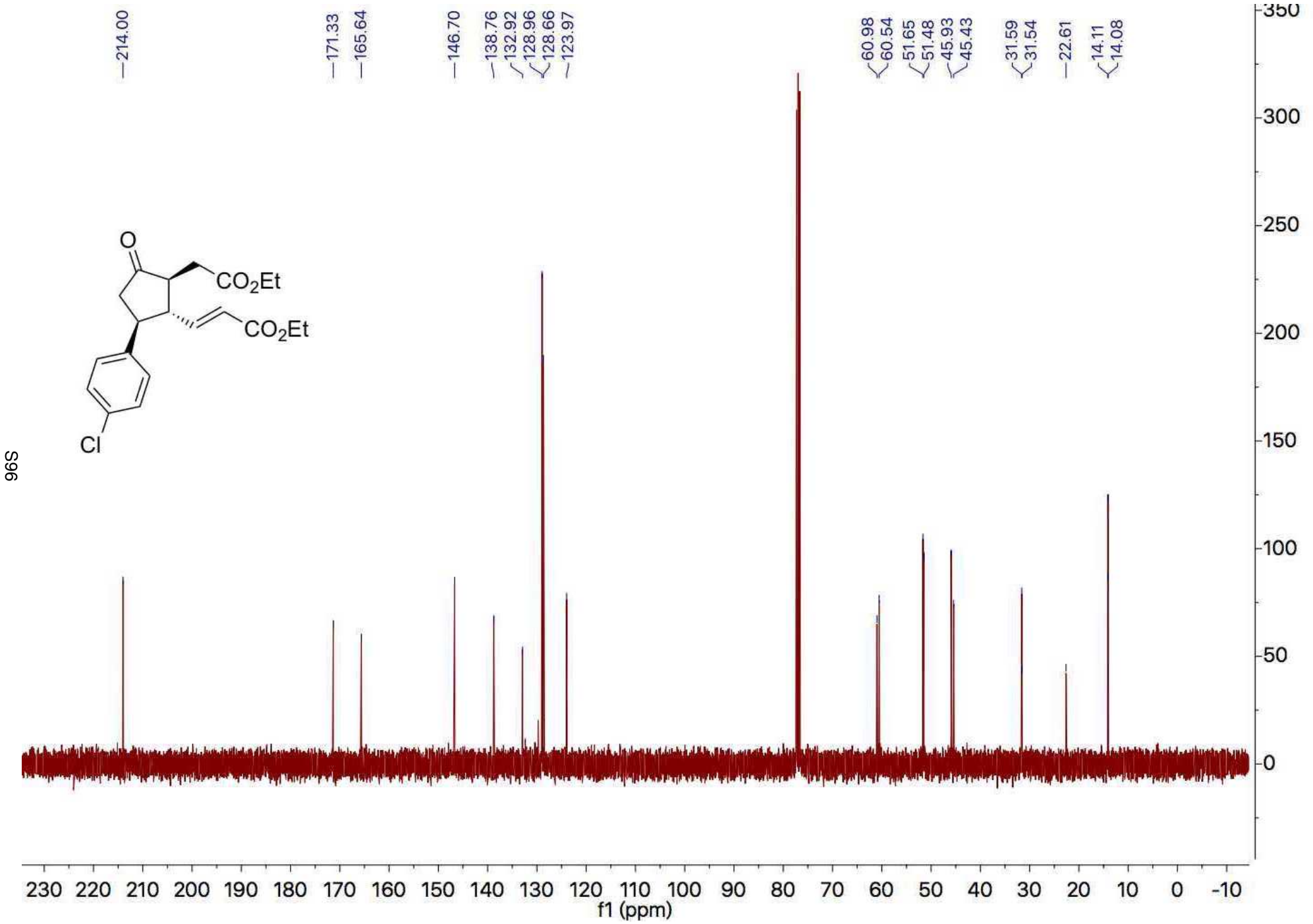


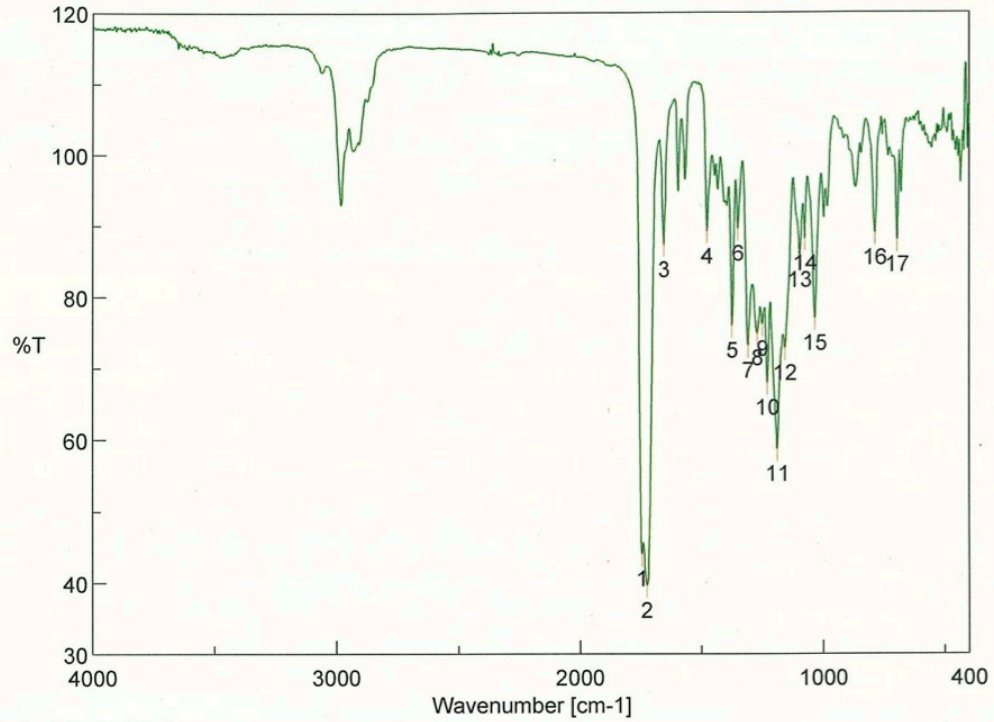
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012125-ID-10vs1-chiral-F.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	25.147	139516117	100.000
トータル		139516117	100.000

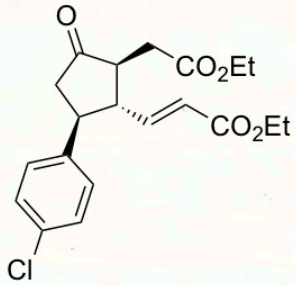

7.306
7.285
7.185
7.164
6.798
6.775
6.759
6.736
5.675
5.674
5.636
5.635
4.172
4.167
4.163
4.158
4.149
4.145
4.134
4.131
4.127
4.116
4.113
4.109
4.098
4.089
3.208
3.188
3.180
3.160
3.150
3.130
3.054
3.031
3.026
3.003
2.976
2.863
2.861
2.843
2.841
2.832
2.819
2.796
2.794
2.788
2.776
2.621
2.602
2.590
2.574
2.558
2.547
2.543
2.500
2.489
2.474
2.460
1.277
1.267
1.259
1.250
1.242
1.232
O
CO2Et
CO2Et
Cl
1.72
1.77
1.00
0.94
3.66
1.07
1.11
1.90
1.85
1.04
6.60
f1 (ppm)

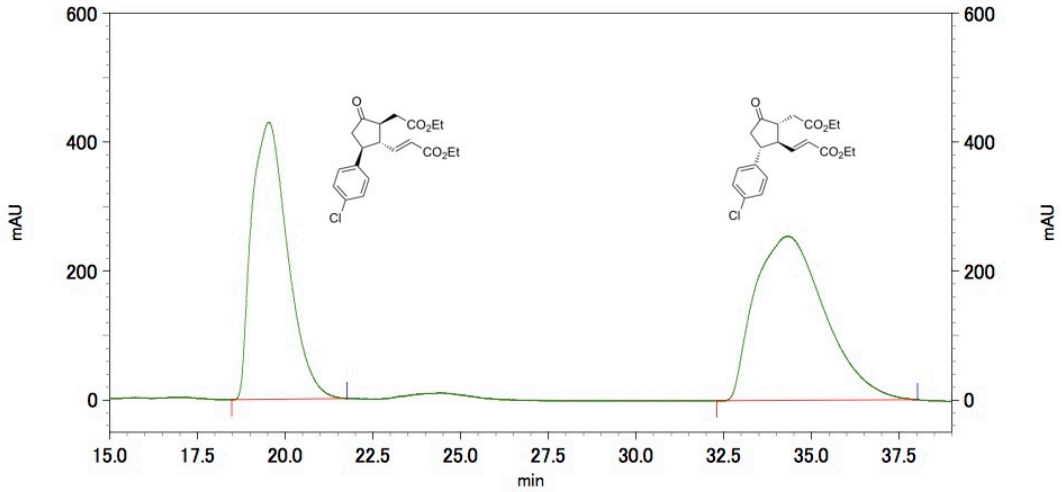

214.00
171.33
165.64
146.70
138.76
132.92
128.96
128.66
123.97
60.98
60.54
51.65
51.48
45.93
45.43
31.59
31.54
22.61
14.11
14.08
O
CO2Et
CO2Et
Cl
350
300
250
200
150
100
50
0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

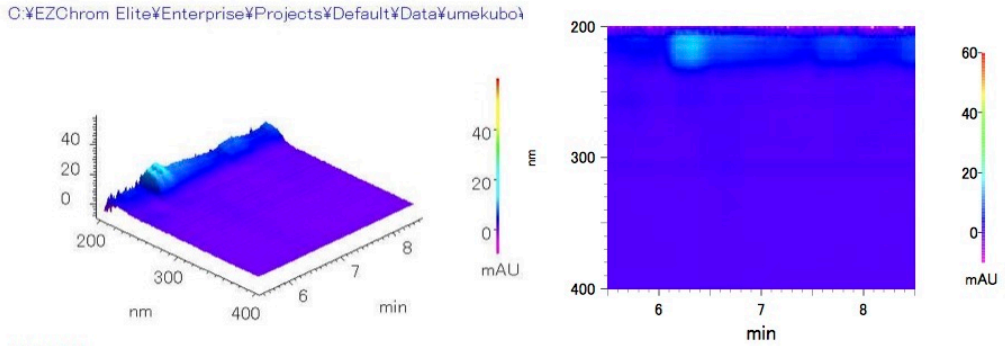


[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1745.26	44.021	2	1724.05	39.5863
3	1654.62	87.3789	4	1476.24	89.2813
5	1373.07	76.0543	6	1348.96	89.6907
7	1307.5	73.2737	8	1269.9	74.9866
9	1248.68	76.2808	10	1227.47	67.9993
11	1186.97	58.7508	12	1155.15	72.938
13	1095.37	85.9745	14	1074.16	88.3156
15	1032.69	77.1008	16	786.815	89.0651
17	695.212	88.0761			



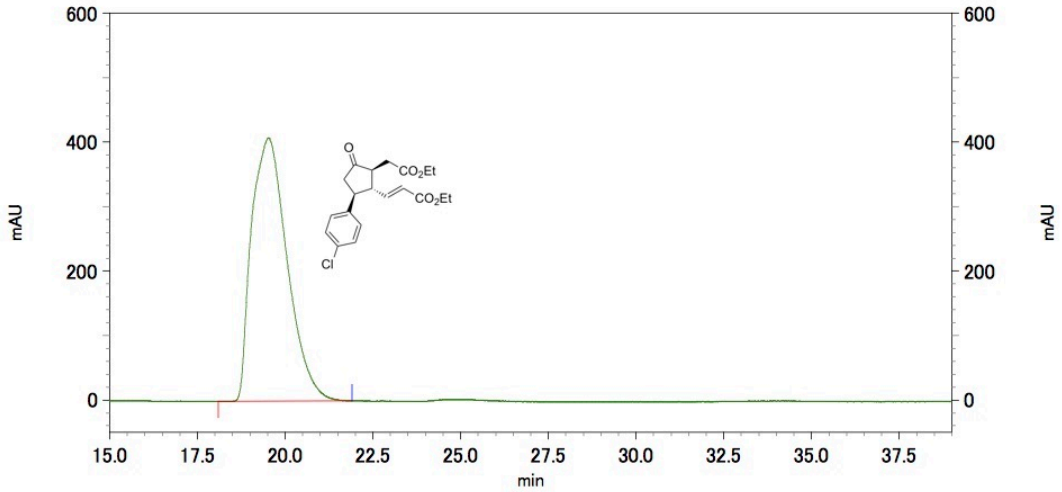


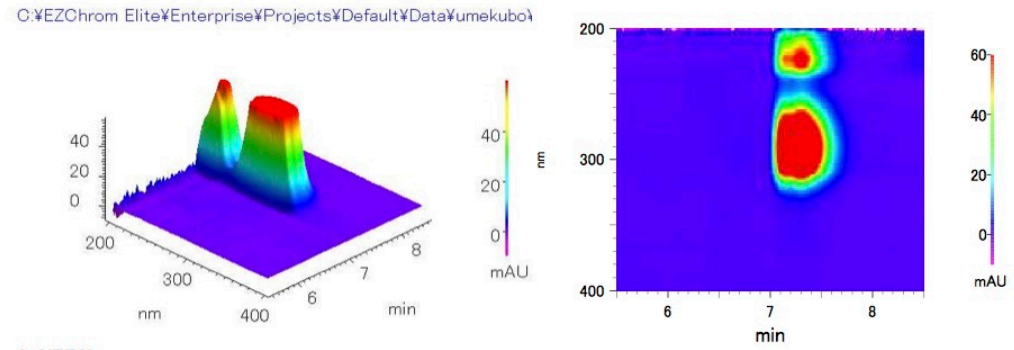


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012113-ID-10vs1-racemi-p-Cl.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	19.520	122140246	46.341
2	34.333	141429029	53.659
トータル		263569275	100.000

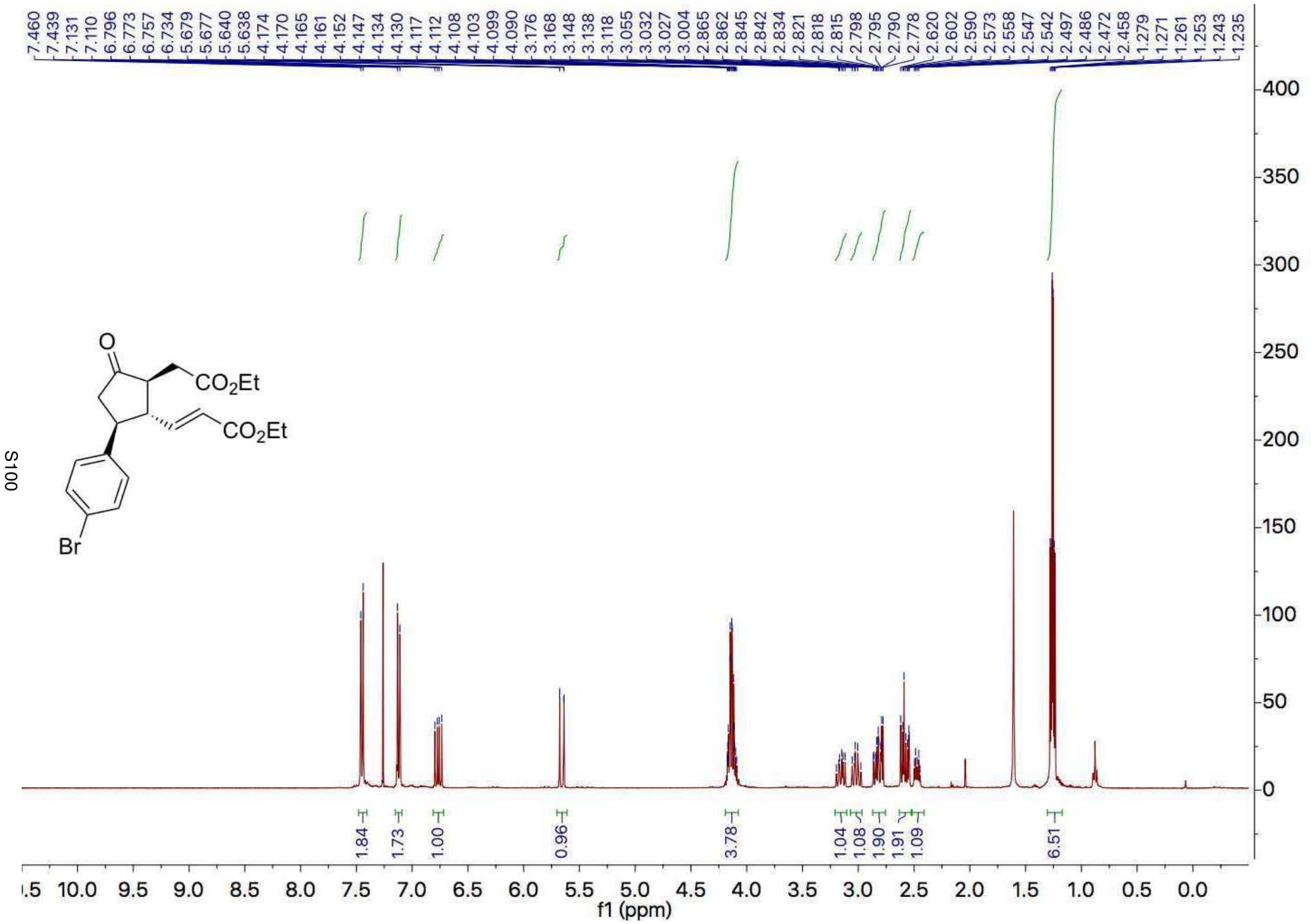


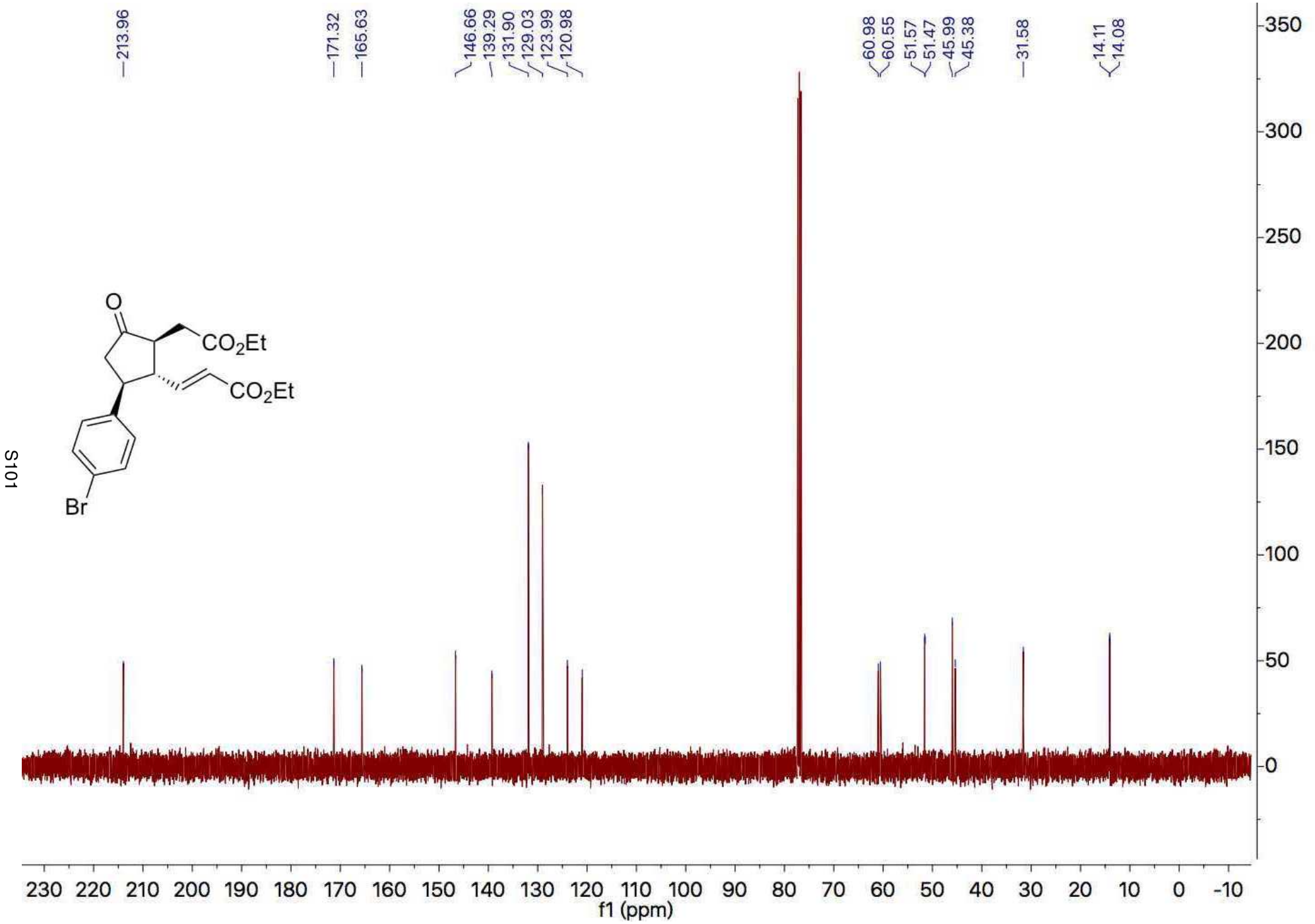


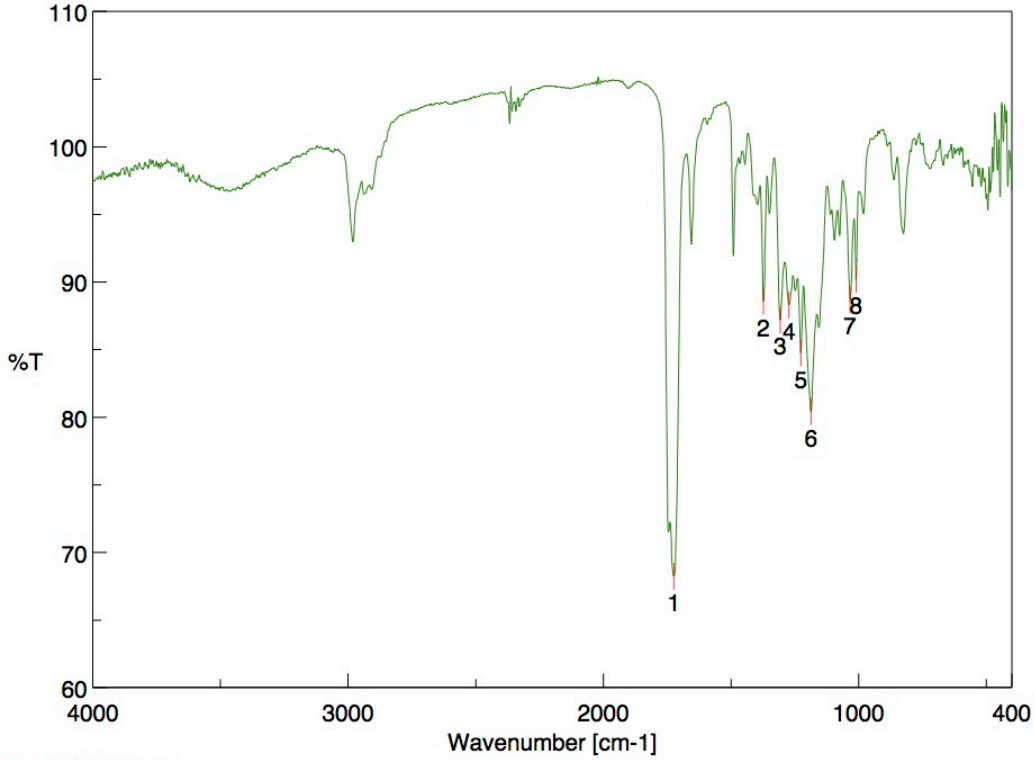
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012113-ID-10vs1-chiral-p-Cl.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	19.513	115317984	100.000
トータル		115317984	100.000

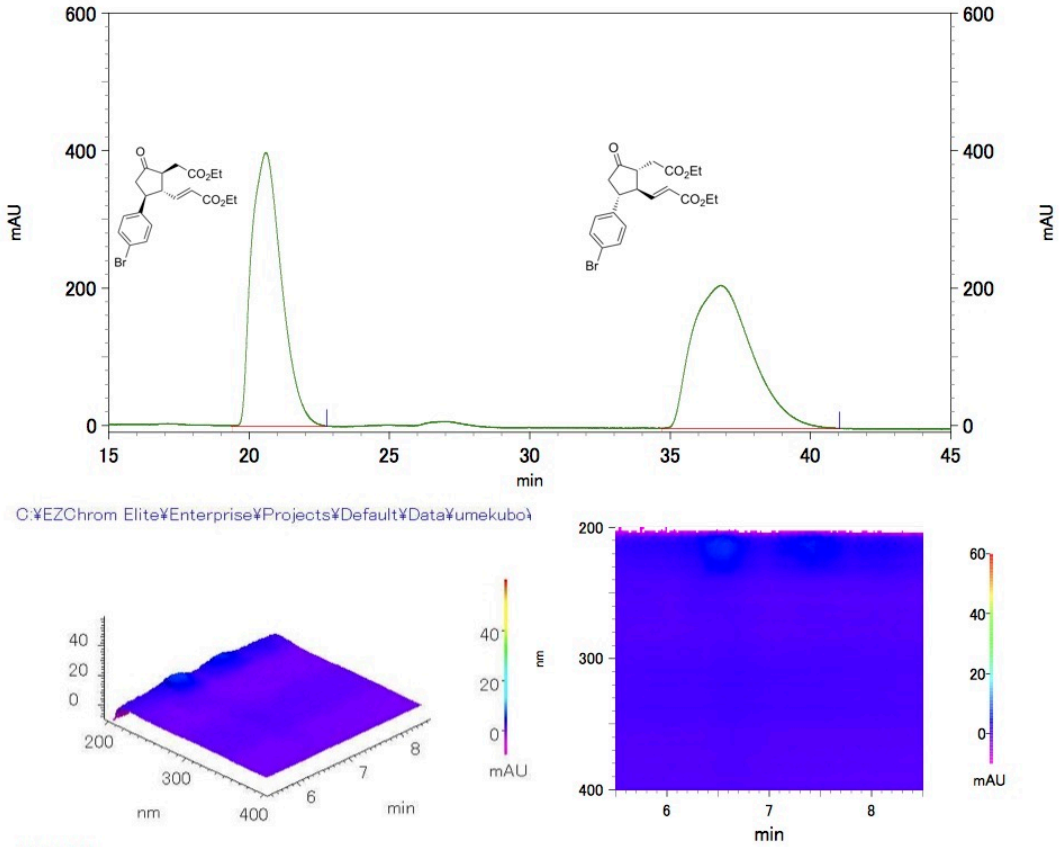

7.460
7.439
7.131
7.110
6.796
6.773
6.757
6.734
5.679
5.677
5.640
5.638
4.174
4.170
4.165
4.161
4.152
4.147
4.134
4.130
4.117
4.112
4.108
4.103
4.099
4.090
3.176
3.168
3.148
3.138
3.118
3.055
3.032
3.027
3.004
2.865
2.862
2.845
2.842
2.834
2.821
2.818
2.815
2.798
2.795
2.790
2.778
2.620
2.602
2.590
2.573
2.558
2.547
2.542
2.497
2.486
2.472
2.458
1.279
1.271
1.261
1.253
1.243
1.235
O
CO2Et
CO2Et
Br
1.84
1.73
1.00
0.96
3.78
1.04
1.08
1.90
1.91
1.09
6.51
f1 (ppm)


213.96
171.32
165.63
146.66
139.29
131.90
129.03
123.99
120.98
60.98
60.55
51.57
51.47
45.99
45.38
31.58
14.11
14.08
O
CO2Et
CO2Et
Br
350
300
250
200
150
100
50
0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



[ピーク検出結果]

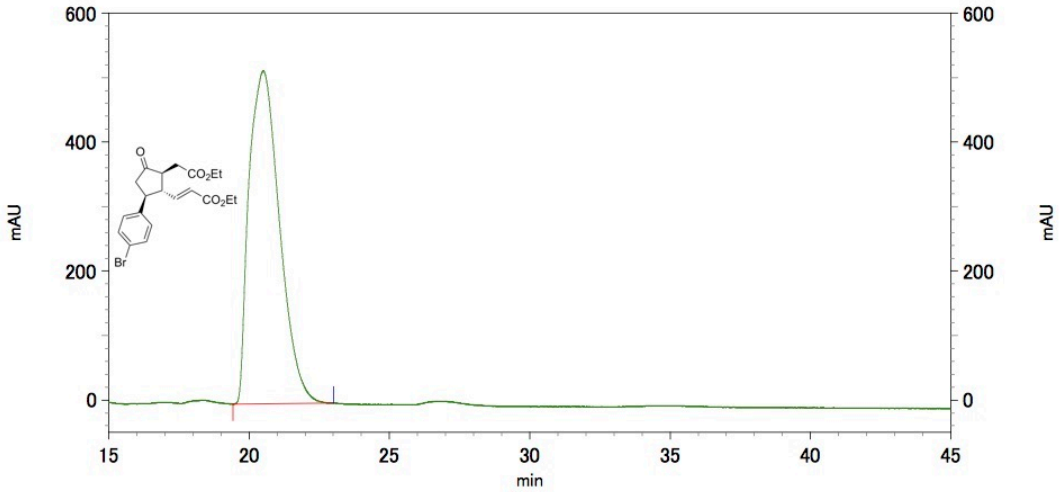
No.	位置	強度	No.	位置	強度
1	1724.05	68.2264	2	1373.07	88.5786
3	1307.5	87.1663	4	1272.79	88.2998
5	1226.5	84.7389	6	1186.97	80.4137
7	1033.66	88.6646	8	1009.55	90.2009

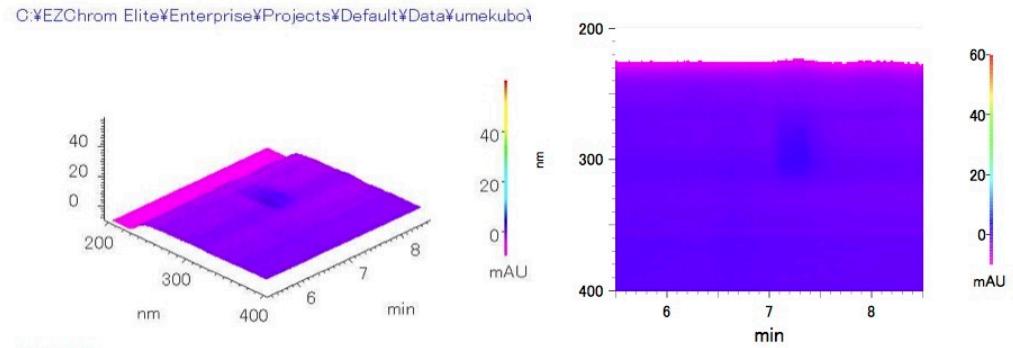


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012114-ID-10vs1-racemi-p-Br.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	20.593	117237849	48.836
2	36.820	122828496	51.164
トータル		240066345	100.000

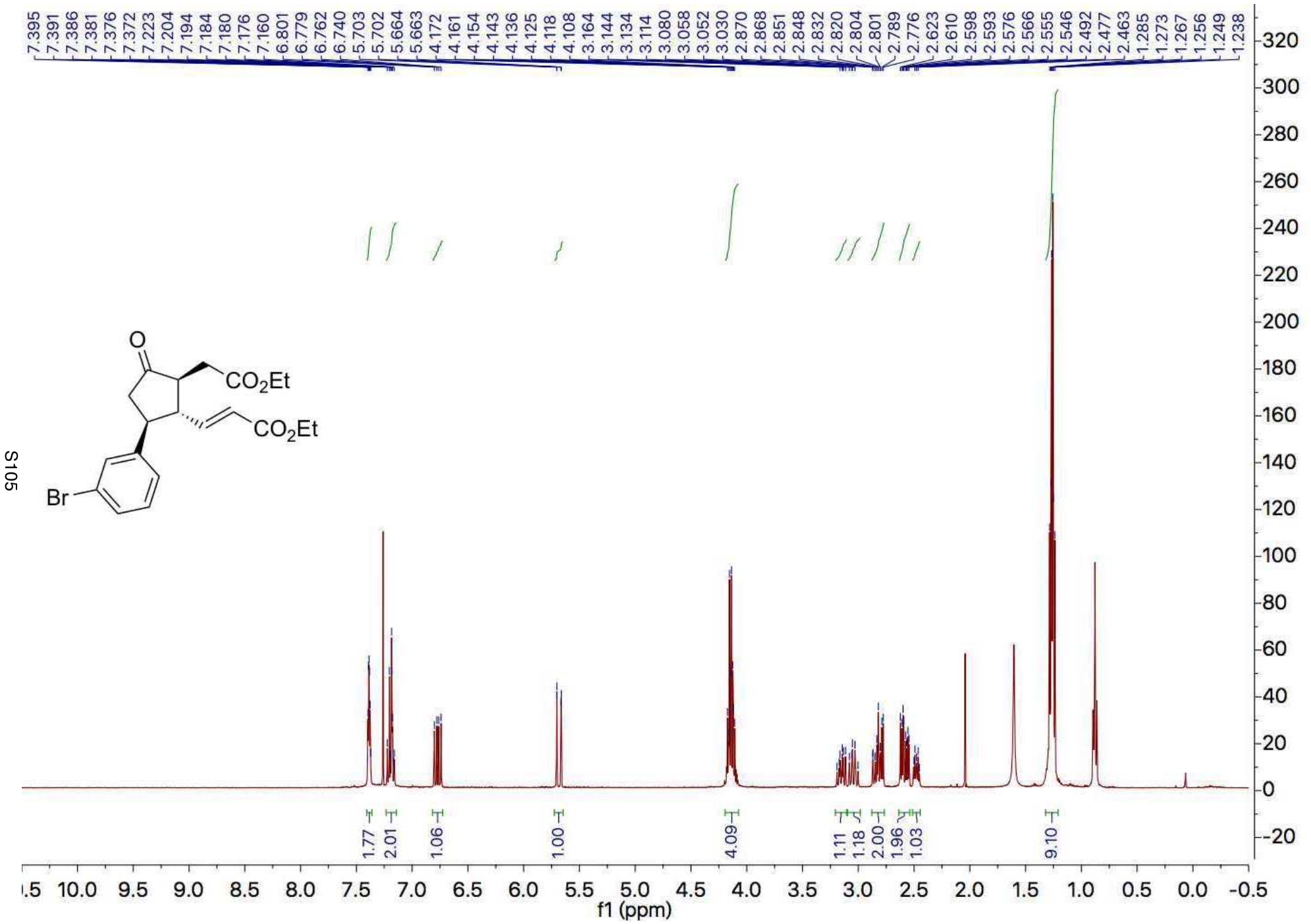


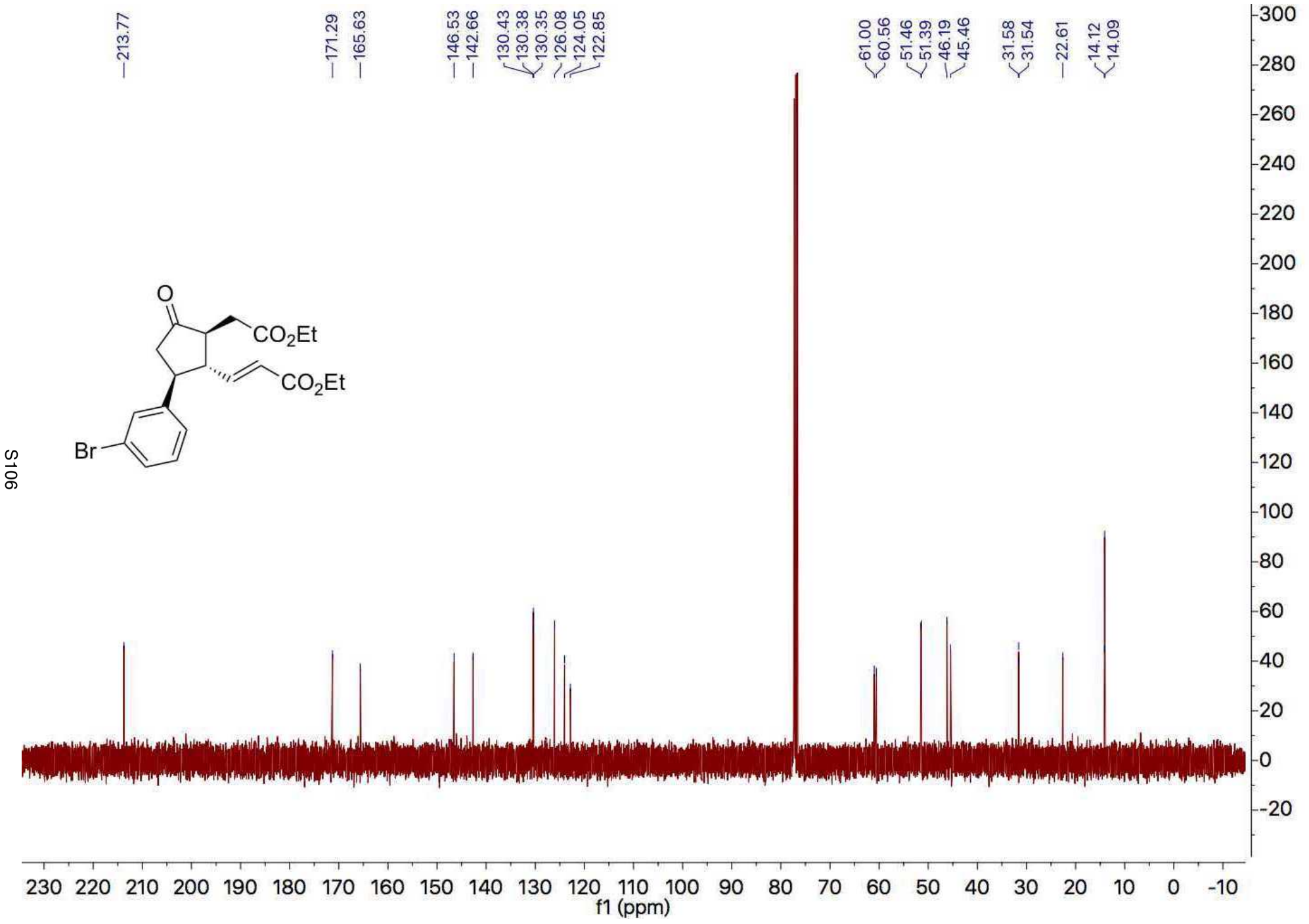


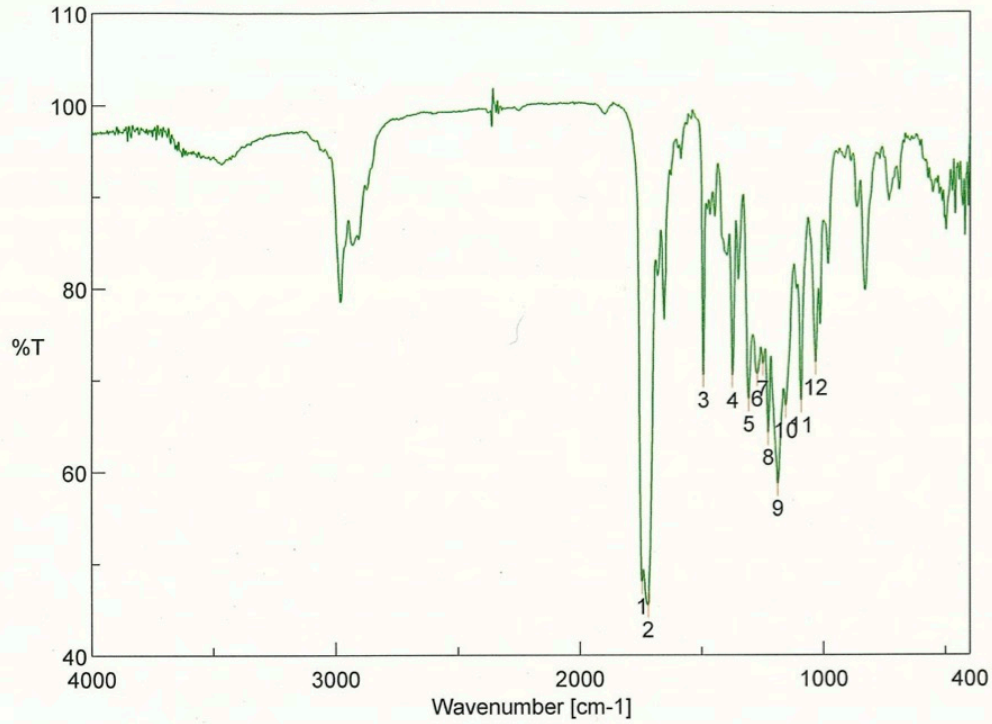
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012114-ID-10vs1-chiral-p-Br.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	20.520	155865829	100.000
トータル		155865829	100.000

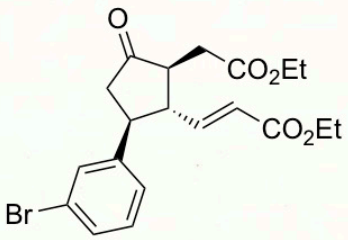

7.395 7.391 7.386 7.381 7.376 7.372 7.223 7.204 7.194 7.184 7.180 7.176 7.160 6.801 6.779 6.762 6.740 5.703 5.702 5.664 5.663 4.172 4.161 4.154 4.143 4.136 4.125 4.118 4.108 3.164 3.144 3.134 3.114 3.080 3.058 3.052 3.030 2.870 2.868 2.851 2.848 2.832 2.820 2.804 2.801 2.789 2.776 2.623 2.610 2.598 2.593 2.576 2.566 2.555 2.546 2.492 2.477 2.463 1.285 1.273 1.267 1.256 1.249 1.238
O
CO2Et
CO2Et
Br
1.77 2.01 1.06 1.00 4.09 1.11 1.18 2.00 1.96 1.03 9.10
f1 (ppm)

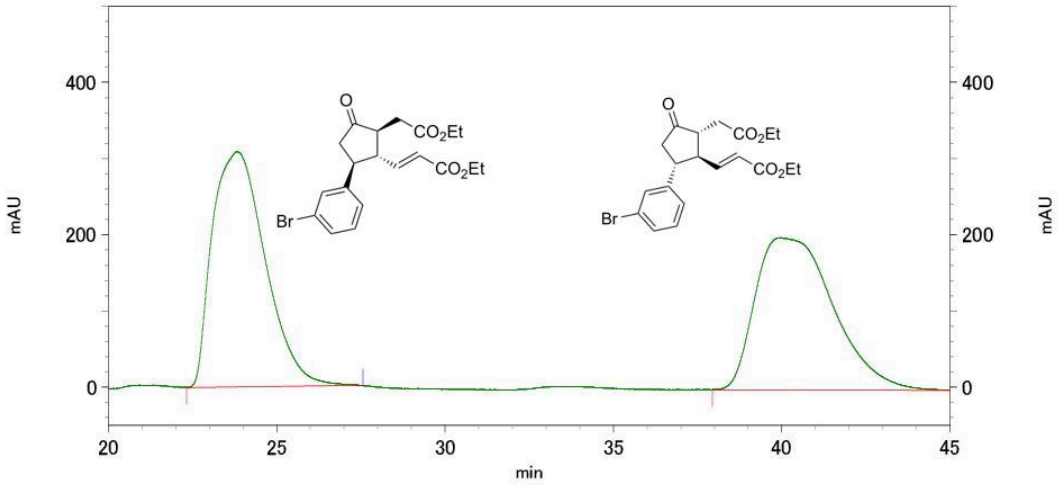

213.77
171.29
165.63
146.53
142.66
130.43
130.38
130.35
126.08
124.05
122.85
61.00
60.56
51.46
51.39
46.19
45.46
31.58
31.54
22.61
14.12
14.09
O
CO2Et
CO2Et
Br
f1 (ppm)

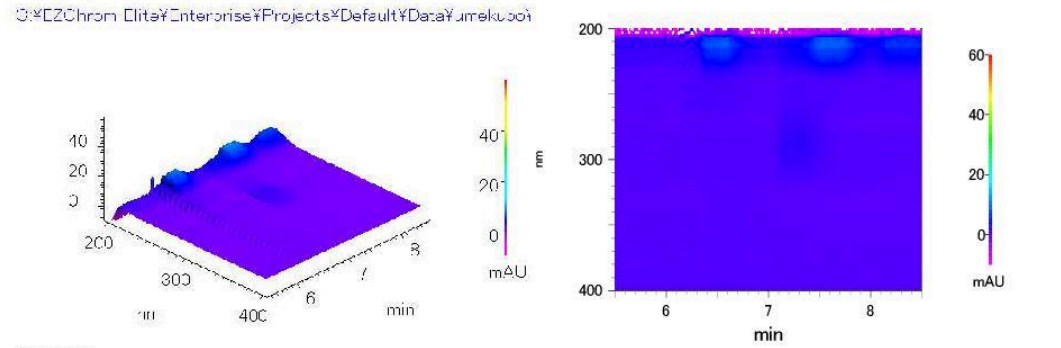


[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1745.26	48.0446	2	1720.19	45.4924
3	1493.6	70.5329	4	1373.07	70.4659
5	1307.5	67.9065	6	1271.82	70.657
7	1248.68	71.741	8	1226.5	64.2344
9	1186.97	58.7	10	1155.15	67.191
11	1091.51	67.7709	12	1032.69	71.8502



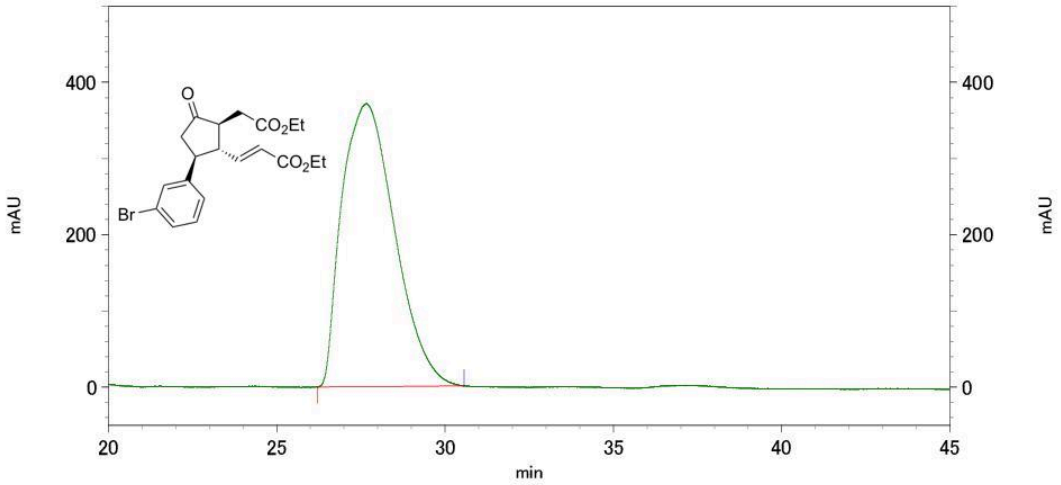


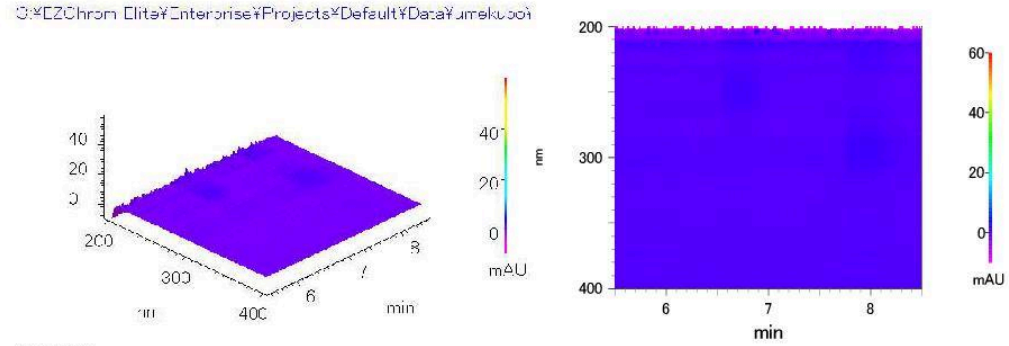


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012117-ID-10vs1-racemi-m-Br.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	23.833	134839518	51.241
2	39.967	128308089	48.759
トータル		263147607	100.000

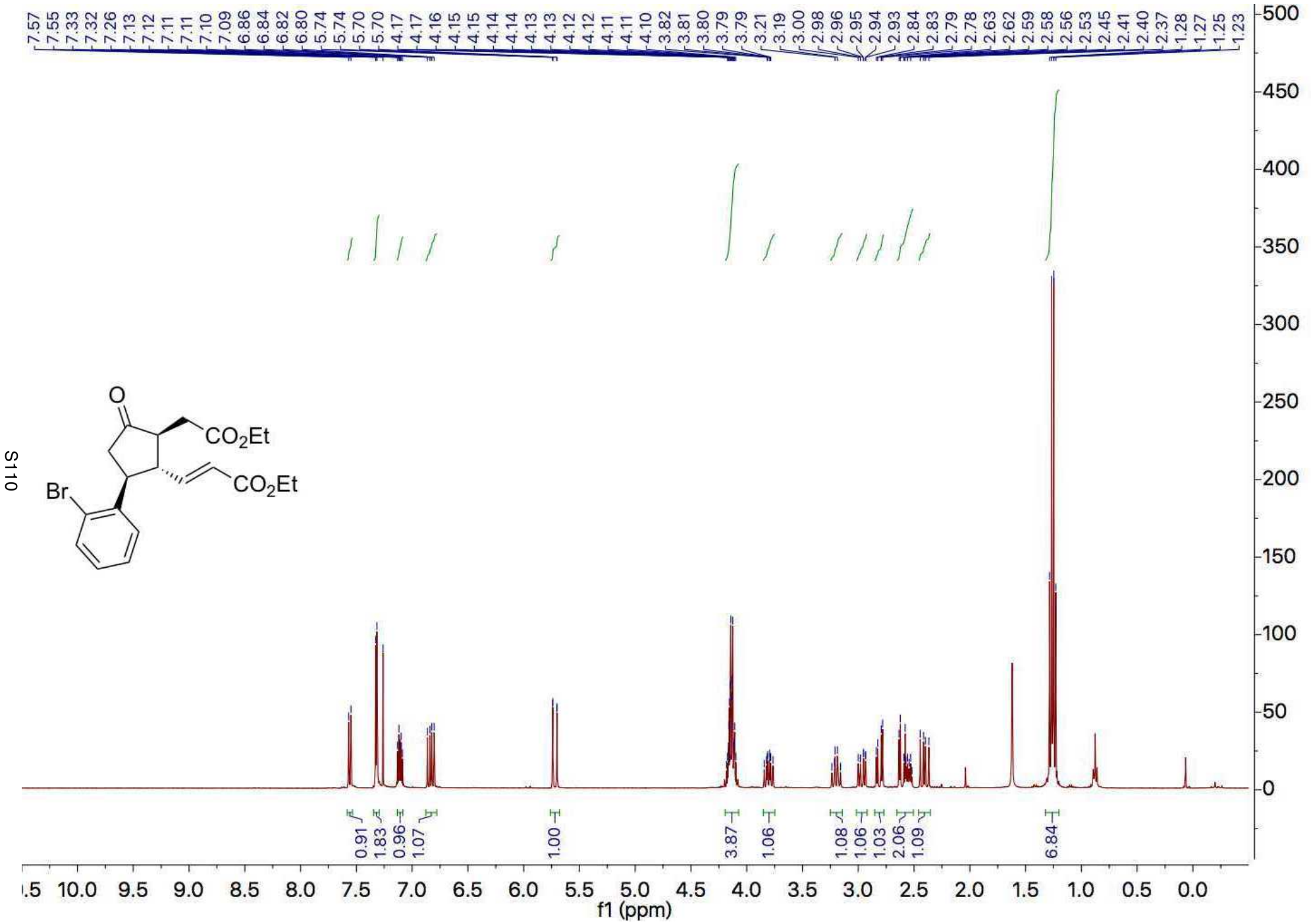


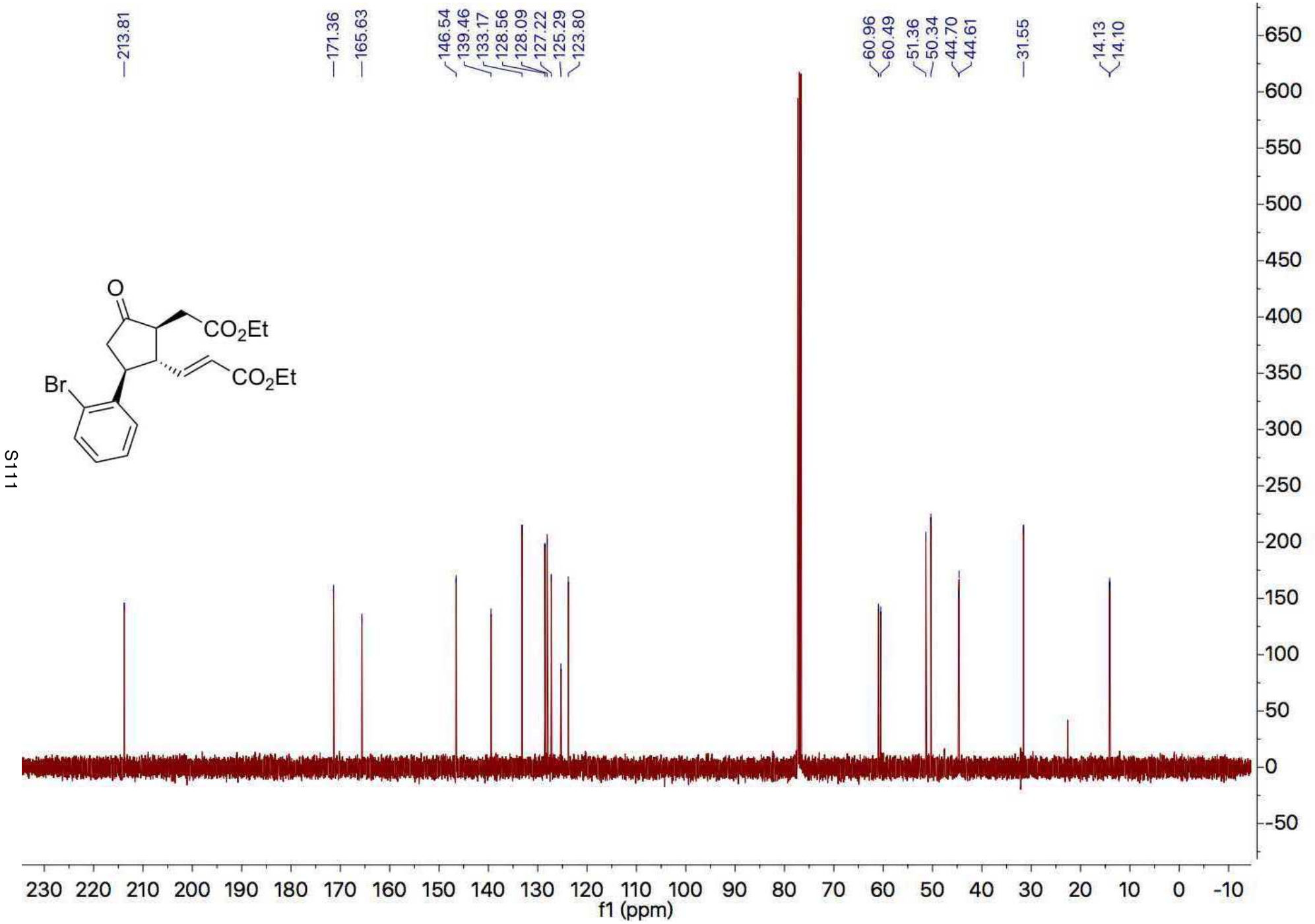


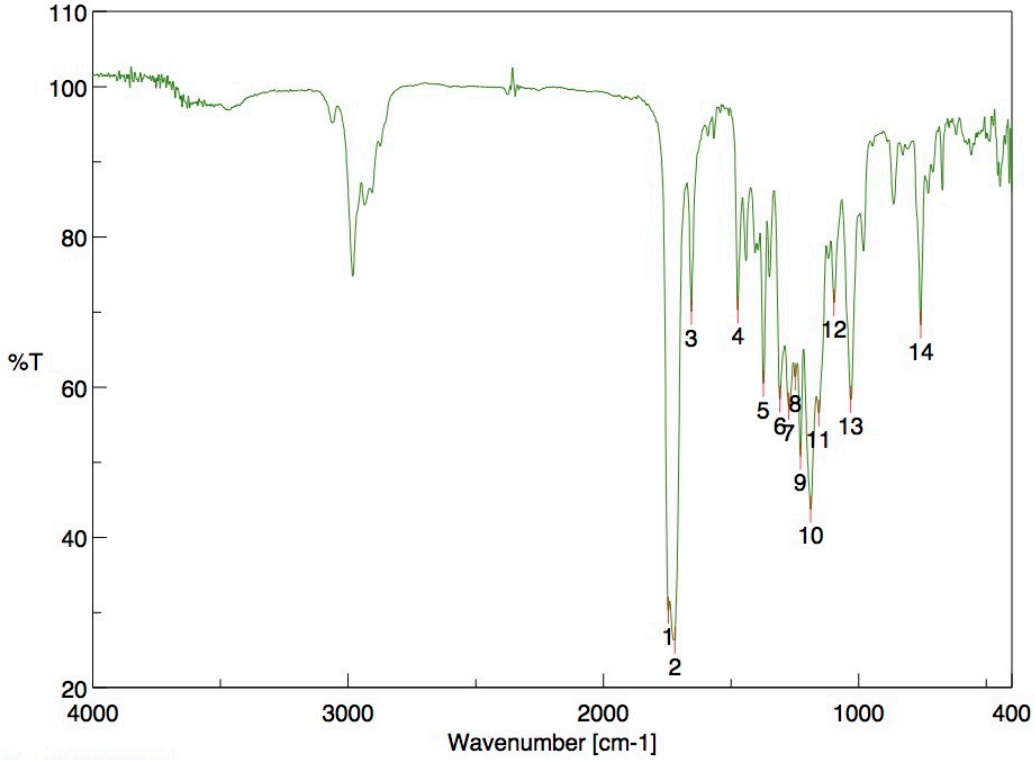
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012117-ID-10vs1-chira-m-Br.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	27.673	161867944	100.000
トータル		161867944	100.000

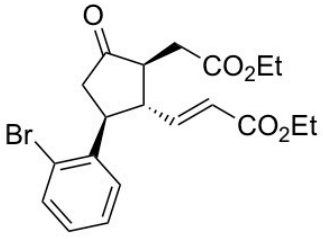

7.57 7.55 7.33 7.32 7.26 7.13 7.12 7.11 7.11 7.10 7.09 6.86 6.84 6.82 6.80 5.74 5.74 5.70 5.70 4.17 4.17 4.16 4.15 4.15 4.14 4.14 4.13 4.13 4.12 4.12 4.11 4.11 4.10 3.82 3.81 3.80 3.79 3.79 3.21 3.19 3.00 2.98 2.96 2.95 2.94 2.93 2.84 2.83 2.79 2.78 2.63 2.62 2.59 2.58 2.56 2.53 2.45 2.41 2.40 2.37 1.28 1.27 1.25 1.23
O
CO2Et
CO2Et
Br
0.91 1.83 0.96 1.07 1.00 3.87 1.06 1.08 1.06 1.03 2.06 1.09 6.84
f1 (ppm)

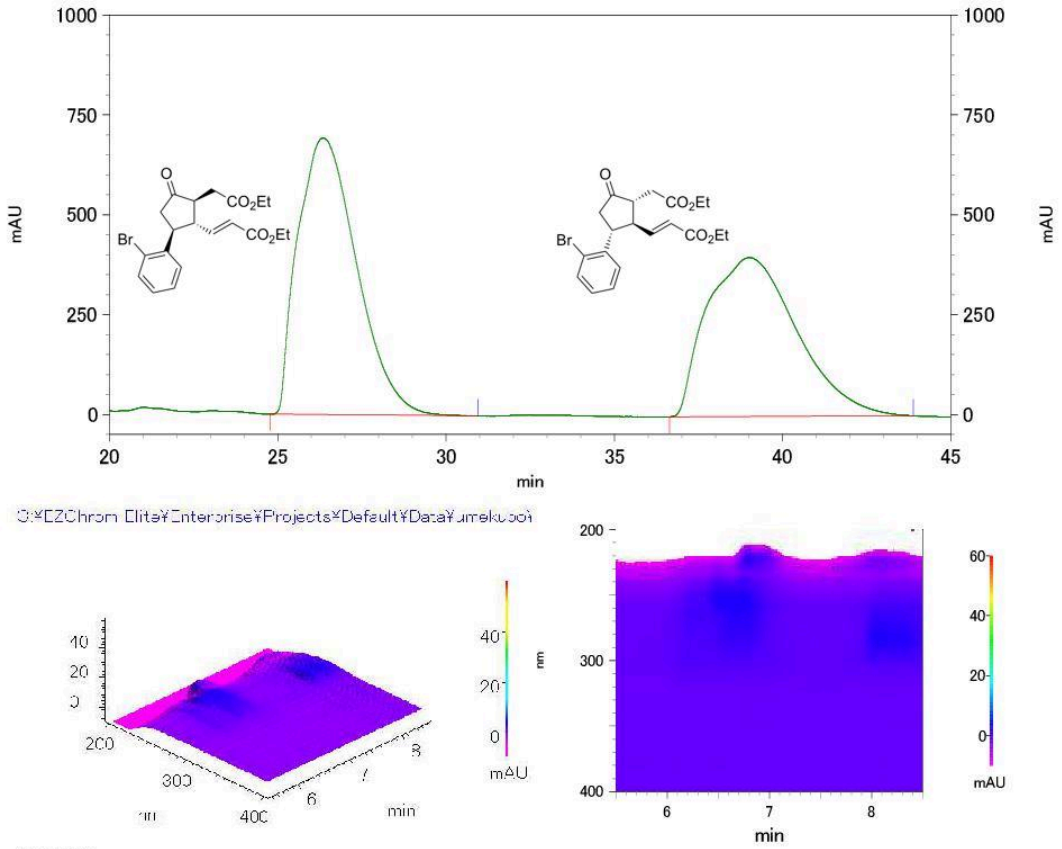

213.81
171.36
165.63
146.54
139.46
133.17
128.56
128.09
127.22
125.29
123.80
60.96
60.49
51.36
50.34
44.70
44.61
31.55
14.13
14.10
O
CO2Et
CO2Et
Br
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	1745.26	30.2447	2	1720.19	26.2998
3	1654.62	70.0776	4	1473.35	70.2689
5	1373.07	60.4904	6	1308.46	58.4004
7	1273.75	57.4637	8	1248.68	61.3539
9	1228.43	50.7923	10	1187.94	43.7277
11	1156.12	56.5423	12	1096.33	71.1836
13	1030.77	58.3615	14	756.923	68.2829

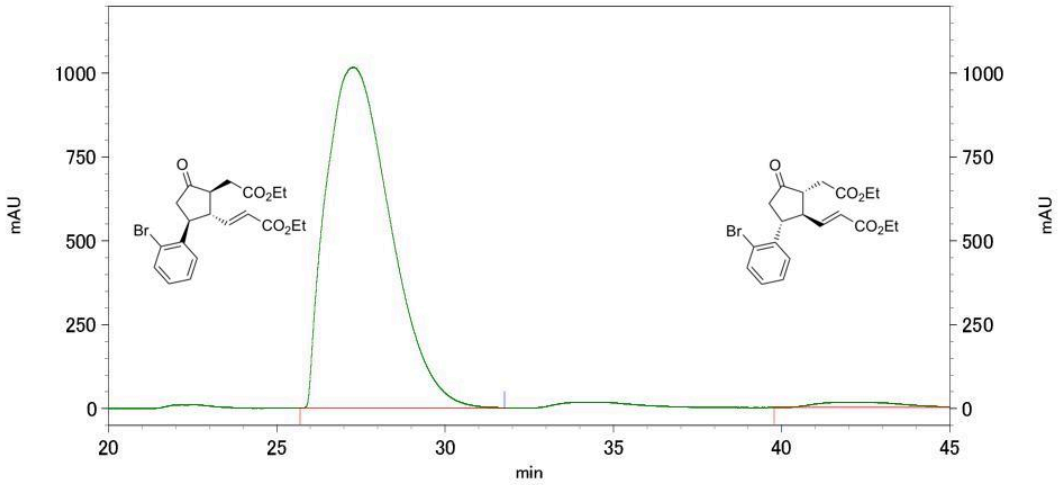


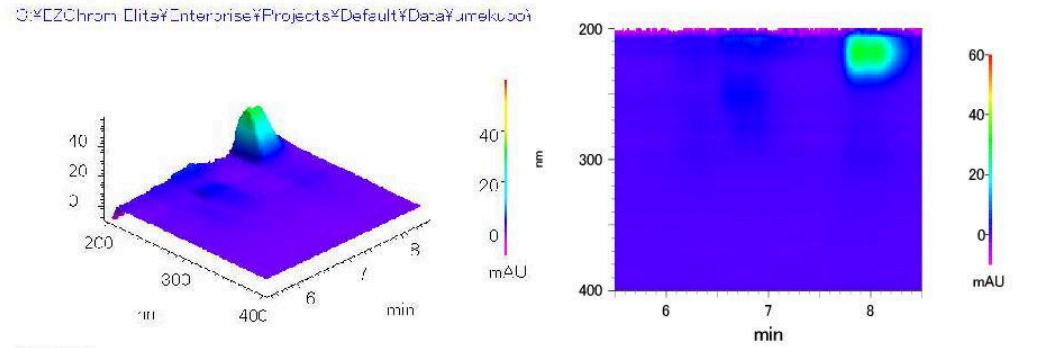


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012126-ID-10vs1-racemi-o-Br.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	26.347	335966008	53.185
2	39.000	295723474	46.815
トータル		631689482	100.000

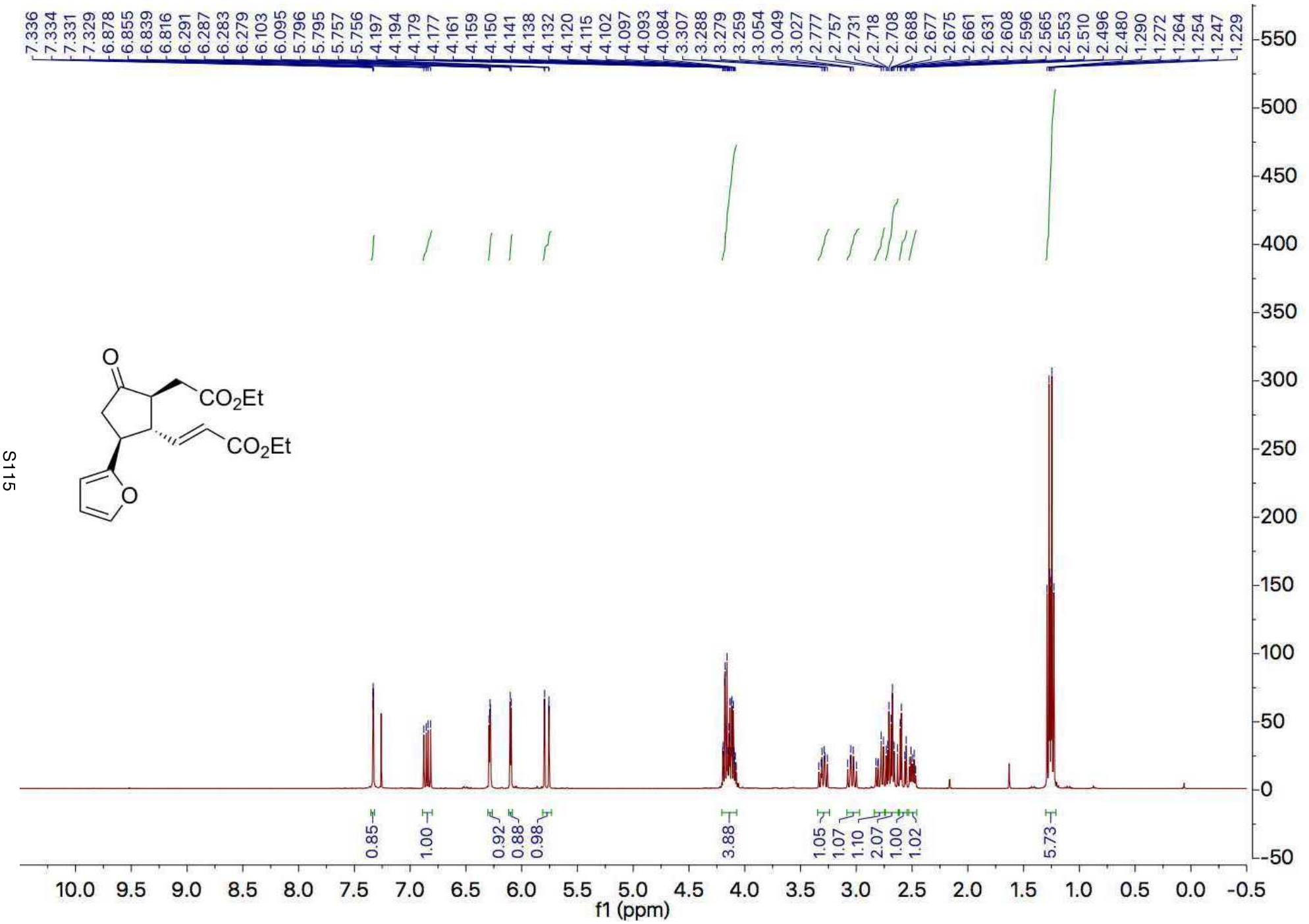


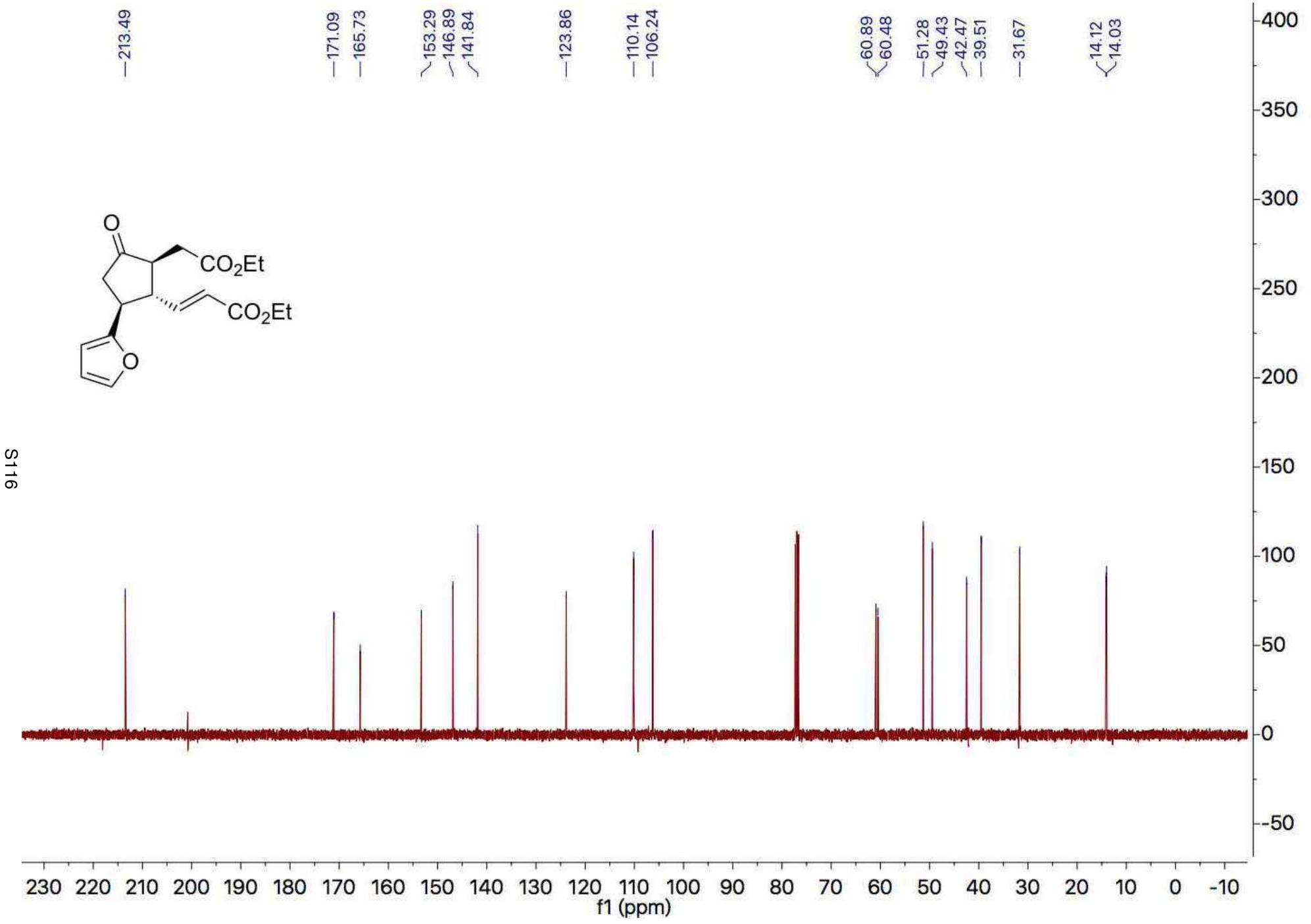


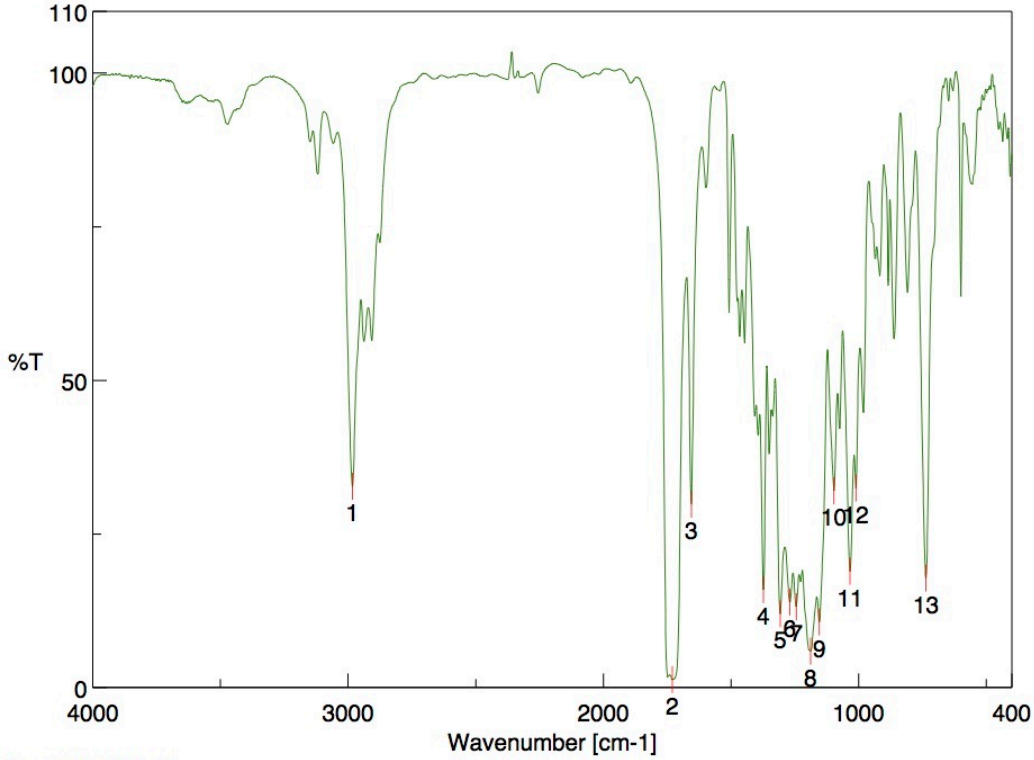
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012126-ID-10vs1-chiral-o-Br.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	27.307	537581014	97.912
2	41.760	11461911	2.088
トータル		549042925	100.000

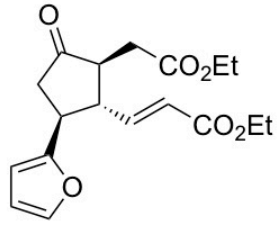

7.336
7.334
7.331
7.329
6.878
6.855
6.839
6.816
6.291
6.287
6.283
6.279
6.103
6.095
5.796
5.795
5.757
5.756
4.197
4.194
4.179
4.177
4.161
4.159
4.150
4.141
4.138
4.132
4.120
4.115
4.102
4.097
4.093
4.084
3.307
3.288
3.279
3.259
3.054
3.049
3.027
2.777
2.757
2.731
2.718
2.708
2.688
2.677
2.675
2.661
2.631
2.608
2.596
2.565
2.553
2.510
2.496
2.480
1.290
1.272
1.264
1.254
1.247
1.229
O
CO2Et
CO2Et
O
0.85
1.00
0.92
0.88
0.98
3.88
1.05
1.07
1.10
2.07
1.00
1.02
5.73
f1 (ppm)

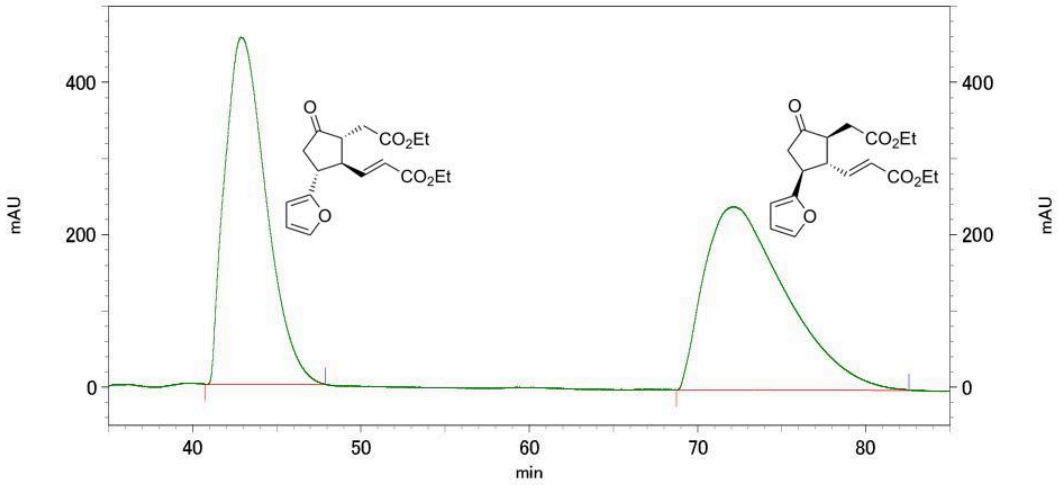

213.49
171.09
165.73
153.29
146.89
141.84
123.86
110.14
106.24
60.89
60.48
51.28
49.43
42.47
39.51
31.67
14.12
14.03
O
CO2Et
CO2Et
O
400
350
300
250
200
150
100
50
0
-50
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

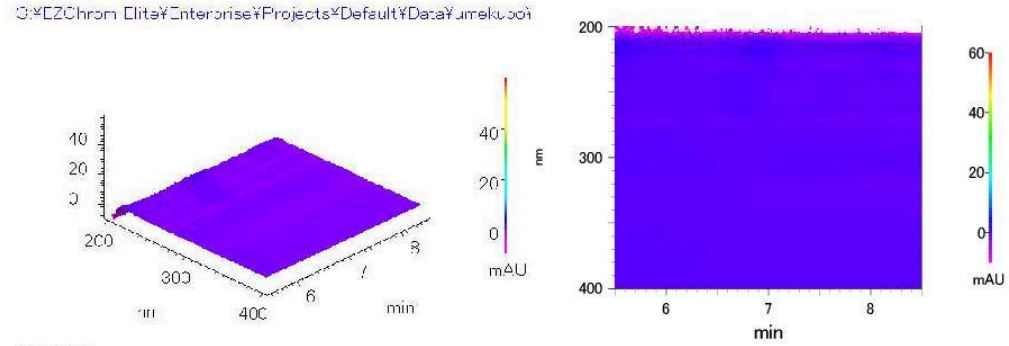


[ピーク検出結果]

No.	位置	強度	No.	位置	強度
1	2982.37	32.7167	2	1729.83	1.28262
3	1655.59	29.8213	4	1373.07	15.92
5	1307.5	11.9922	6	1269.9	13.9356
7	1244.83	13.1422	8	1187.94	5.90006
9	1154.19	10.6812	10	1096.33	32.02
11	1033.66	18.9269	12	1010.52	32.463
13	736.674	17.8141			



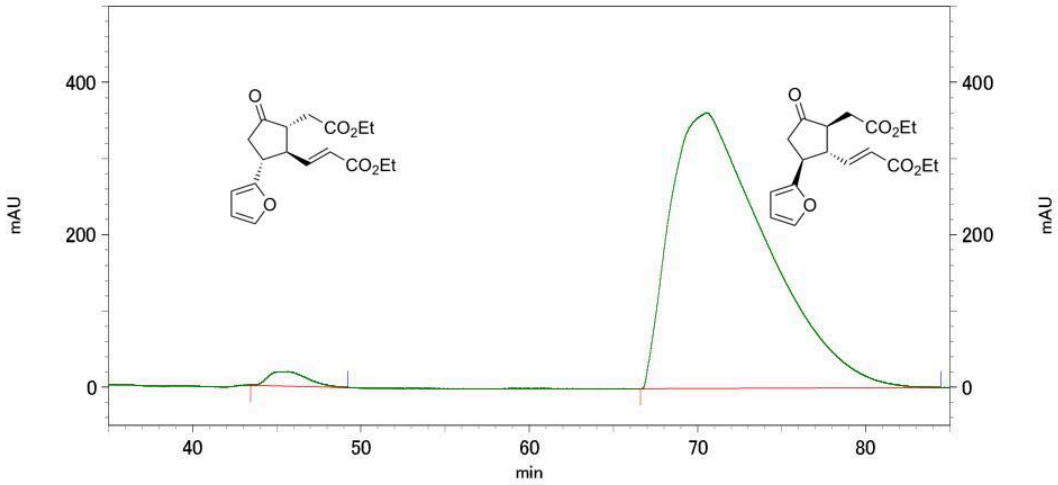


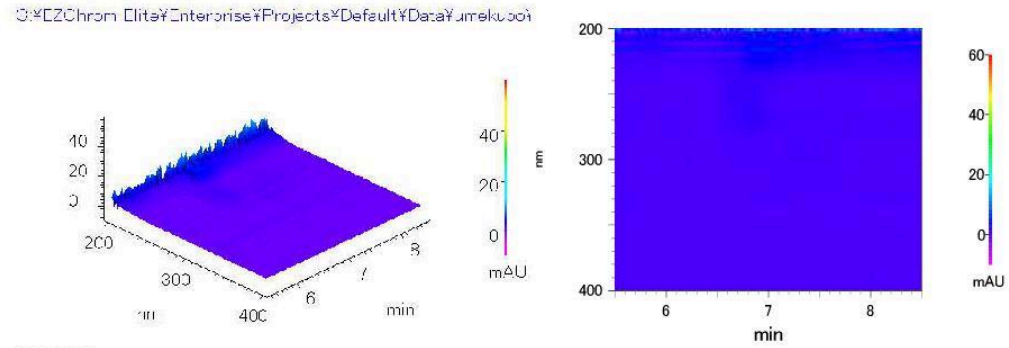


C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012122-ID-10vs1-racemi-furyl.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	42.880	309719063	48.775
2	72.173	325272914	51.225
トータル		634991977	100.000

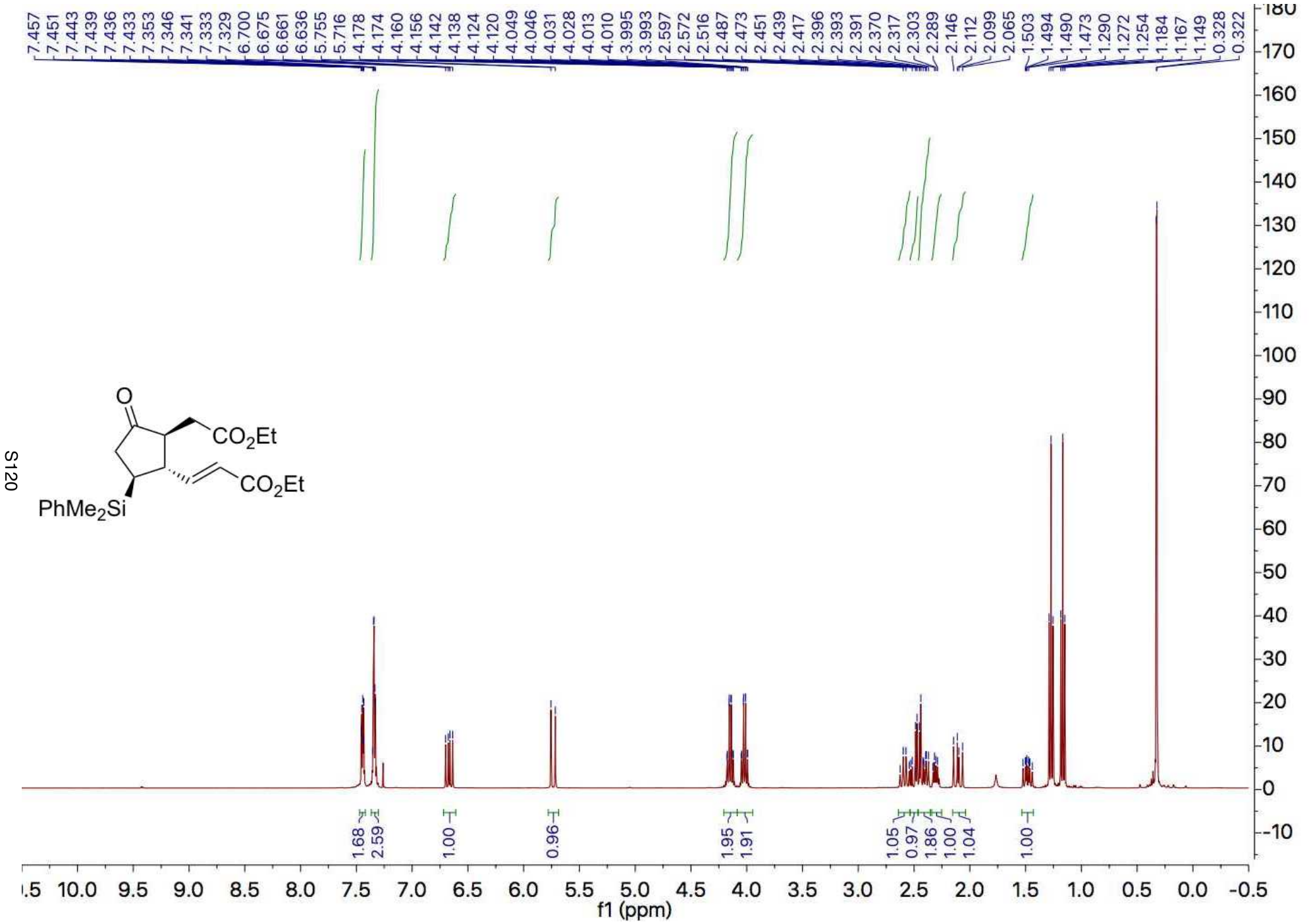


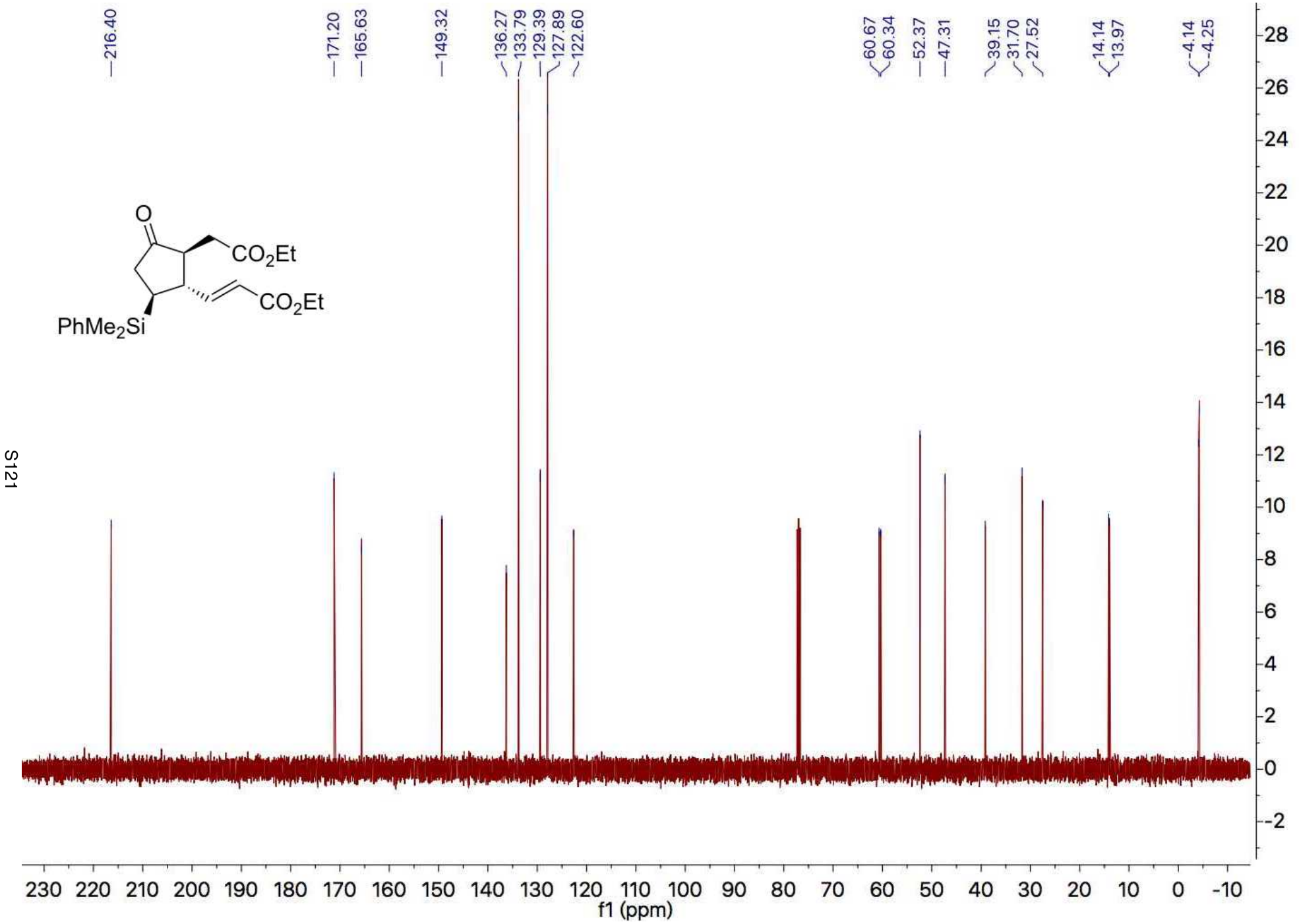


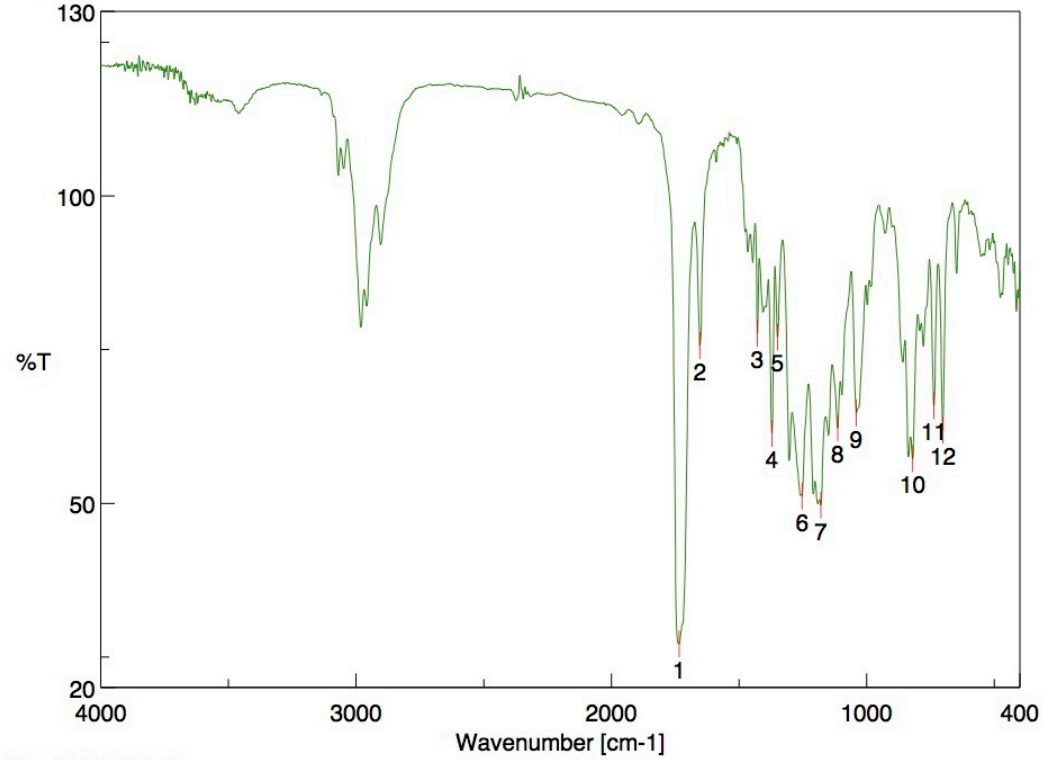
C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Data¥umekubo¥NU012122-ID-10vs1-chiral-furyl.dat

7: 214 nm, 4 nm結果

Pk #	Retention Time	Area	Area%
1	45.653	12058131	2.055
2	70.507	574745789	97.945
トータル		586803920	100.000

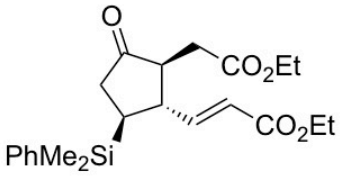

7.457
7.451
7.443
7.439
7.436
7.433
7.353
7.346
7.341
7.333
7.329
6.700
6.675
6.661
6.636
5.755
5.716
4.178
4.174
4.160
4.156
4.142
4.138
4.124
4.120
4.049
4.046
4.031
4.028
4.013
4.010
3.995
3.993
2.597
2.572
2.516
2.487
2.473
2.451
2.439
2.417
2.396
2.393
2.391
2.370
2.317
2.303
2.289
2.146
2.112
2.099
2.065
1.503
1.494
1.490
1.473
1.290
1.272
1.254
1.184
1.167
1.149
0.328
0.322
O
CO2Et
CO2Et
PhMe2Si
1.68
2.59
1.00
0.96
1.95
1.91
1.05
0.97
1.86
1.00
1.04
1.00
f1 (ppm)

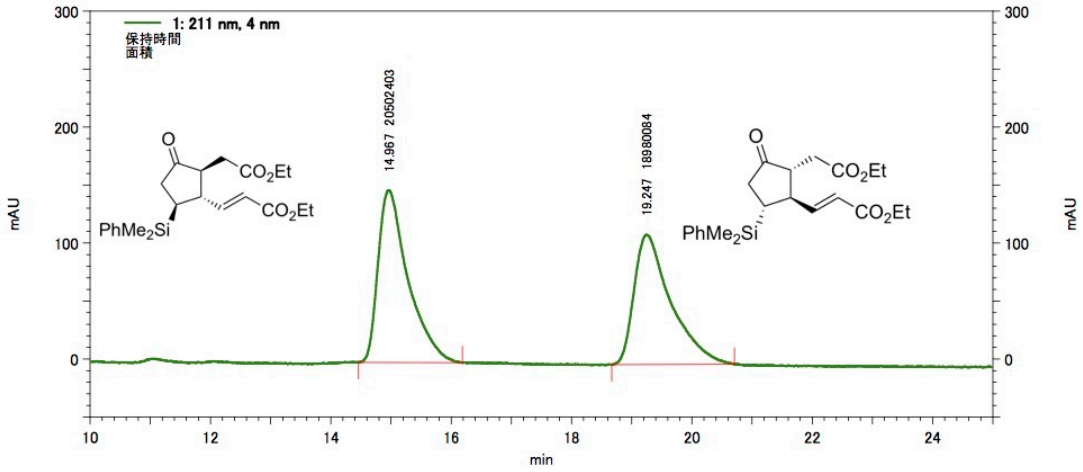

216.40
171.20
165.63
149.32
136.27
133.79
129.39
127.89
122.60
60.67
60.34
52.37
47.31
39.15
31.70
27.52
14.14
13.97
-4.14
-4.25
O
CO2Et
CO2Et
PhMe2Si
f1 (ppm)



[ピーク検出結果]

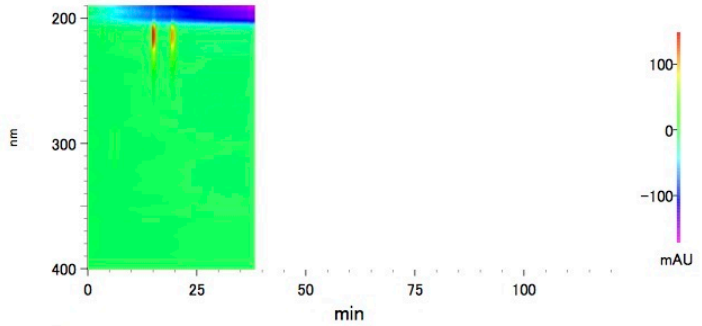
No.	位置	強度	No.	位置	強度
1	1734.66	27.0777	2	1653.66	75.5988
3	1428.03	77.5679	4	1371.14	61.3371
5	1348.96	77.033	6	1253.5	51.1937
7	1179.26	49.713	8	1113.69	62.1894
9	1040.41	64.6873	10	819.598	57.2593
11	736.674	65.8815	12	701.962	61.863



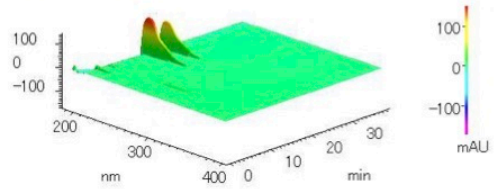


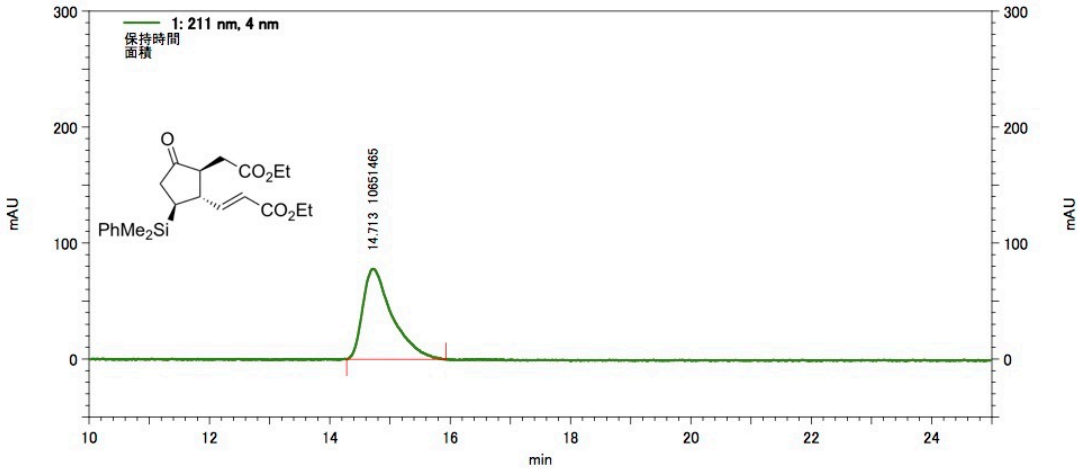
1: 211 nm, 4 nm結果

保持時間	面積	面積%	高さ	高さ%
14.967	20502403	51.928	593623	57.022
19.247	18980084	48.072	447421	42.978
トータル	39482487	100.000	1041044	100.000



D:¥R[[Ng-SI¥Wittig-Me2PhSi-¥NU012101-ID-10vs1-racemi.dat

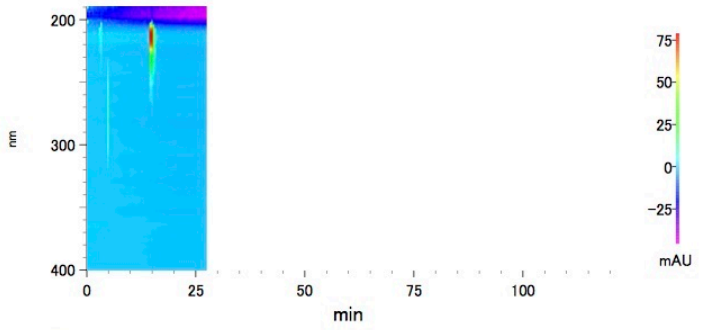




1: 211 nm, 4 nm結果

保持時間	面積	面積%	高さ	高さ%
14.713	10651465	100.000	311774	100.000

トータル	10651465	100.000	311774	100.000



D:¥R[[Ng-SI¥Wittig-Me2PhSi-¥NU012099-ID-10vs1-chiral.dat