

## *Supporting Information*

### **Catalytic Asymmetric Ring-Opening of *meso*-Aziridines with Malonates Under Heterodinuclear Rare Earth Metal Schiff Base Catalysis**

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#### **Experimental Section**

**General:** Infrared (IR) spectra were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. NMR spectra were recorded on JEOL JNM-LA500 and JNM-ECX500 spectrometers, operating at 500 MHz for <sup>1</sup>H NMR and 125.65 MHz for <sup>13</sup>C NMR. Chemical shifts in CDCl<sub>3</sub> were reported in the scale relative to CHCl<sub>3</sub> (7.26 ppm for <sup>1</sup>H NMR) and CDCl<sub>3</sub> (77.0 ppm for <sup>13</sup>C NMR) as an internal reference, respectively. ESI mass spectra were measured on a Waters ZQ4000 spectrometer (for LRMS), and JEOL JMS-T100LC AccuTOF spectrometer (for HRMS). X-ray crystallographic analysis was performed on a Rigaku R-Axis RAPID II imaging plate area detector with graphite-monochromated Cu-K $\alpha$  radiation. Optical rotation was recorded using a 1 mL cell with a 0.5 dm path length on a JASCO polarimeter P-1010. The enantiomeric excess (ee) was determined by HPLC analysis (JASCO HPLC systems; pump: PU-2080; detector: UV-2075, measured at 254 nm; column: DAICEL CHIRALPAK IB or IC). Column chromatography was performed with silica gel Merck 60 (230-400 mesh ASTM). Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl. Dry Toluene was

purchased from Kanto and dried over activated MS 4Å before use. Reactions were carried out using flame-dried glassware in dry solvents under an argon atmosphere unless otherwise stated. Manolates **3** were purchased from Tokyo Chemical Industry Co., Ltd. (TCI) and purified by distillation before use. Aziridines **2** were prepared by following the literature procedure.<sup>[S1]</sup>

## References

[S1] Fukuta, Y.; Mita, T.; Fukuda, N.; Kanai, M.; Shibasaki, M. *J. Am. Chem. Soc.* **2006**, *128*, 6312.

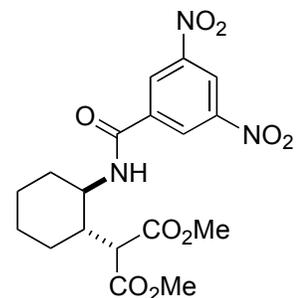
## Preparation of La(O-*i*Pr)<sub>3</sub>/Yb(OTf)<sub>3</sub>/Schiff Base **1** Complex and General Procedure for Catalytic Asymmetric Ring-Opening of *meso*-Aziridines with Malonates:

To a solution of Schiff base **1** (11.1 mg, 0.02 mmol) in THF (0.2 mL) in a test tube at room temperature was added La(O-*i*Pr)<sub>3</sub> (0.2 M THF solution, 0.1 mL, 0.02 mmol). The mixture was stirred at room temperature for 0.5 h to afford yellow suspension. THF was, then, removed under reduced pressure. To the test tube were added Yb(OTf)<sub>3</sub> (12.4 mg, 0.02 mmol) and THF (0.2 mL), and the mixture was stirred at room temperature for 0.5 h to afford the La/Yb/**1** catalyst in THF. THF was, then, removed under reduced pressure. After drying the residue under reduced pressure (ca. 2 mmHg) for 1 h at room temperature, toluene (0.4 mL) was added. To the resulting red suspension were added malonate **3a** (35 μL, 0.30 mmol, 1.5 equiv) and aziridine **2a** (58.3 mg, 0.20 mmol, 1.0 equiv), and the mixture was stirred for 4 h at 40 °C. The reaction was quenched by adding a suspension of silica gel (ca. 150 mg) in EtOAc, and the mixture was filtered through a short silica gel pad. After evaporation under reduced pressure, the residue was purified by silica gel flash column chromatography (hexane: AcOEt = 10:1 to 4:1) to afford **4aa**.

## Dimethyl

### 2-((1*S*,2*R*)-2-(3,5-dinitrobenzamido)cyclohexyl)malonate (**4aa**):

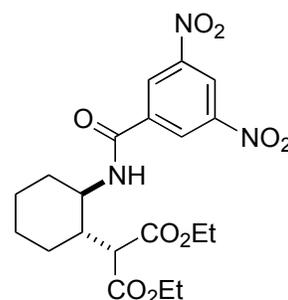
colorless solid; IR (KBr)  $\nu$  3100, 2937, 2857, 1741, 1650, 1542, 1433, 1344 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.22-1.34 (m, 2 H), 1.37-1.45 (m, 1 H), 1.60-1.68 (m, 1 H), 1.75-1.80 (m, 3 H),



2.22-2.32 (m, 2 H), 3.50 (d,  $J = 3.1$  Hz, 1 H), 3.63 (s, 3 H), 3.72-3.79 (m, 1H), 3.82 (s, 3 H), 7.70 (d,  $J = 7.6$  Hz, 1 H), 8.96 (d,  $J = 2.2$  Hz, 2 H), 9.10 (t,  $J = 2.2$  Hz, 1 H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  24.6, 25.8, 31.6, 33.7, 41.2, 52.8, 52.9, 53.1, 55.9, 120.6, 127.3, 138.4, 148.6, 162.0, 169.1, 171.8; LRMS (ESI):  $m/z$  446  $[\text{M}+\text{Na}]^+$ ; HRMS (ESI):  $m/z$  calculated for  $\text{C}_{18}\text{H}_{21}\text{N}_3\text{NaO}_9^+ [\text{M}+\text{Na}]^+$ : 446.1170, found: 466.1169; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R = 18.6$  min (major) and 24.1 min (minor);  $[\alpha]_D^{23.7} = +28.0$  ( $c = 0.90$ ,  $\text{CHCl}_3$ ).

**Diethyl 2-((1*S*,2*R*)-2-(3,5-dinitrobenzamido)cyclohexyl)malonate (4ab):** colorless solid;

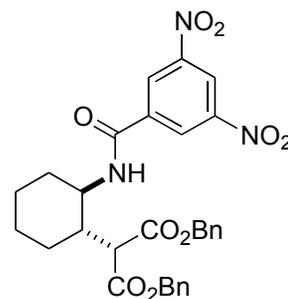
IR (KBr)  $\nu$  3116, 3089, 2991, 2938, 2856, 1744, 1646, 1558, 1345, 1247, 1191  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.14 (t,  $J = 7.2$  Hz, 3 H), 1.20-1.43 (m, 6 H), 1.67-1.82 (m, 4 H), 2.25-2.31 (m, 2 H), 3.46 (d,  $J = 2.8$  Hz, 1 H), 3.70-3.76 (m, 1 H), 4.07-4.14 (m, 2 H), 4.25-4.36 (m, 2H), 7.92 (d,  $J = 7.0$  Hz, 1 H), 8.98 (d,  $J = 1.9$  Hz, 2 H), 9.11 (t,  $J = 1.9$  Hz, 1 H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  13.9, 14.0, 24.6, 25.9, 32.1, 33.6, 41.0, 52.9, 56.5, 62.0, 62.4, 120.6,



127.3, 138.5, 148.6, 161.8, 168.6, 171.9; LRMS (ESI):  $m/z$  474  $[\text{M}+\text{Na}]^+$ ; HRMS (ESI):  $m/z$  calculated for  $\text{C}_{20}\text{H}_{25}\text{N}_3\text{NaO}_9^+ [\text{M}+\text{Na}]^+$ : 474.1483, found: 474.1481; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R = 15.0$  min (major) and 21.7 min (minor);  $[\alpha]_D^{23.7} = +25.6$  ( $c = 1.00$ ,  $\text{CHCl}_3$ ).

**Dibenzyl 2-((1*S*,2*R*)-2-(3,5-dinitrobenzamido)cyclohexyl)malonate (4ac):** colorless

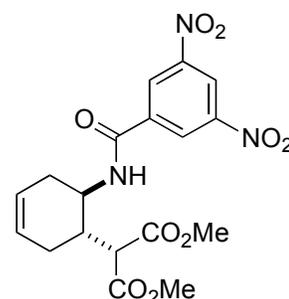
solid; IR (KBr)  $\nu$  3097, 2938, 2858, 1735, 1646, 1542, 1343, 1188  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.20-1.43 (m, 3 H), 1.66-1.81 (m, 4 H), 2.27-2.36 (m, 2 H), 3.62 (d,  $J = 2.5$  Hz, 1 H), 3.76-3.83 (m, 1 H), 4.93 (d,  $J = 12.2$  Hz, 1 H), 5.07 (d,  $J = 12.2$  Hz, 1 H), 5.27 (d,  $J = 12.7$  Hz, 1 H), 5.30 (d,  $J = 12.7$  Hz, 1 H), 7.01-7.10 (m, 5 H), 7.30-7.34 (m, 5 H), 7.77 (d,  $J = 7.4$  Hz, 1 H), 8.78 (d,  $J = 2.2$  Hz, 2 H), 8.98 (t,  $J = 2.2$  Hz, 1 H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125



MHz)  $\delta$  24.5, 29.8, 31.9, 33.6, 41.1, 52.8, 56.5, 67.7, 68.1, 120.4, 127.1, 128.0, 128.2, 128.3, 128.5, 134.4, 134.7, 138.0, 148.2, 161.6, 167.8, 171.5; LRMS (ESI):  $m/z$  598  $[M+Na]^+$ ; HRMS (ESI):  $m/z$  calculated for  $C_{30}H_{29}N_3NaO_9^+$   $[M+Na]^+$ : 598.1796, found: 598.1786; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  = 12.8 min (major) and 22.8 min (minor);  $[\alpha]_D^{23.7} = -1.6$  ( $c = 1.00$ ,  $CHCl_3$ ).

**Dimethyl 2-((1*S*,6*R*)-6-(3,5-dinitrobenzamido)cyclohex-3-enyl)malonate (4ba):**

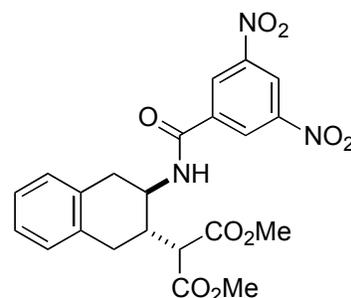
colorless solid; IR (KBr)  $\nu$  3102, 3031, 2953, 2915, 2844, 1734, 1649, 1535, 1346, 1162  $cm^{-1}$ ;  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  2.04-2.10 (m, 1 H), 2.22-2.26 (m, 1 H), 2.48-2.54 (m, 1 H), 2.58-2.69 (m, 2 H), 3.57 (d,  $J = 3.1$  Hz, 1 H), 3.64 (s, 3 H), 3.86 (s, 3 H), 4.10-4.17 (m, 1 H), 5.62-5.67 (m, 2 H), 7.90 (d,  $J = 8.0$  Hz, 1 H), 8.97 (d,  $J = 2.2$  Hz, 2 H), 9.11 (t,  $J = 2.2$  Hz, 1 H);  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz)  $\delta$  31.2, 33.2, 37.4, 49.1, 52.9, 53.3, 54.6, 120.7,



124.8, 125.5, 127.4, 138.2, 148.6, 162.2, 169.0, 172.1; LRMS (ESI):  $m/z$  444  $[M+Na]^+$ ; HRMS (ESI):  $m/z$  calculated for  $C_{18}H_{19}N_3NaO_9^+$   $[M+Na]^+$ : 444.1014, found: 444.1006; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  = 17.0 min (major) and 29.5 min (minor);  $[\alpha]_D^{23.7} = -2.1$  ( $c = 1.03$   $CHCl_3$ ).

**Dimethyl 2-((2*S*,3*R*)-3-(3,5-dinitrobenzamido)-1,2,3,4-tetrahydronaphthalen-2-yl)malonate (4ca):**

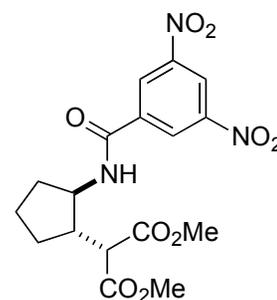
colorless solid; IR (KBr)  $\nu$  3094, 2954, 2844, 1737, 1651, 1541, 1435, 1346, 1275  $cm^{-1}$ ;  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  2.75-2.86 (m, 2 H), 2.97 (dd,  $J = 4.9$ , 16.2 Hz, 1 H), 3.27 (dd,  $J = 12.4$ , 16.2 Hz, 1 H), 3.38 (dd,  $J = 5.2$ , 16.5 Hz, 1 H), 3.67 (s, 3 H), 3.71 (d,  $J = 3.1$  Hz, 1 H), 3.90 (s, 3 H), 4.29-4.36 (m, 1 H), 7.04-7.26 (m, 4 H), 8.07 (d,  $J = 7.7$  Hz, 1 H), 9.01 (d,  $J = 1.9$  Hz, 2 H), 9.11 (t,  $J = 1.9$  Hz, 1 H);  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz)  $\delta$  34.8, 36.7, 38.0, 49.5, 52.9, 53.4, 54.5, 120.8, 126.3,



126.4, 127.4, 128.3, 128.7, 133.5, 133.9, 138.0, 148.6, 162.4, 168.8, 172.0; LRMS (ESI):  $m/z$  494  $[M+Na]^+$ ; HRMS (ESI):  $m/z$  calculated for  $C_{22}H_{21}N_3NaO_9^+ [M+Na]^+$ : 494.1170, found: 494.1163; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  = 25.7 min (major) and 70.8 min (minor);  $[\alpha]_D^{23.7} = -23.7$  ( $c = 1.03$  CHCl<sub>3</sub>).

**Dimethyl 2-((1*S*,2*R*)-2-(3,5-dinitrobenzamido)cyclopentyl)malonate (4da):** colorless solid; IR (KBr)  $\nu$  2105, 2956, 2873, 1745, 1646, 1541, 1434, 1344,

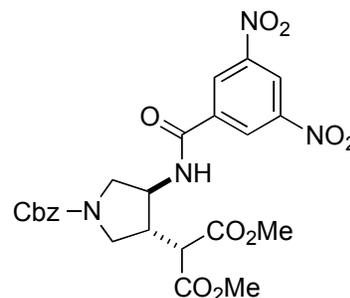
1155  $cm^{-1}$ ;  $^1H$  NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.50-1.64 (m, 2 H), 1.70-1.79 (m, 2 H), 1.90-1.96 (m, 1 H), 2.35-2.41 (m, 1H), 2.61-2.68 (m, 1 H), 3.55 (d,  $J = 8.3$  Hz, 1 H), 3.65 (s, 3 H), 3.75 (s, 3 H), 4.01-4.08 (m, 1H), 7.58 (d,  $J = 6.4$  Hz, 1 H), 8.97 (d,  $J = 2.2$  Hz, 2 H), 9.09 (t,  $J = 2.2$  Hz, 1 H);  $^{13}C$  NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  21.4, 28.5, 32.8, 43.9, 52.7, 52.8, 54.6, 56.1, 120.8, 127.3, 138.1,



148.6, 162.6, 169.5, 169.5; LRMS (ESI):  $m/z$  432  $[M+Na]^+$ ; HRMS (ESI):  $m/z$  calculated for  $C_{17}H_{19}N_3NaO_9^+ [M+Na]^+$ : 432.1014, found: 432.1019; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  = 18.0 min (major) and 40.2 min (minor);  $[\alpha]_D^{23.7} = -32.2$  ( $c = 1.00$ , CHCl<sub>3</sub>).

**Dimethyl 2-((3*R*,4*S*)-1-(benzyloxycarbonyl)-4-(3,5-dinitrobenzamido)pyrrolidin-3-yl)malonate (4ea):** colorless oil; IR (neat)  $\nu$  3093, 2955, 1735,

1671, 1542, 1434, 1345  $cm^{-1}$ ;  $^1H$  NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  3.00-3.05, (m, 1 H), 3.29-3.39 (m, 2 H), 3.63 (d,  $J = 7.6$  Hz, 1 H), 3.72 (s, 3 H), 3.76 (s, 3 H), 3.79-3.85 (m, 1H), 4.01-4.19 (m, 1 H), 4.33-4.46 (m, 1 H), 5.02-5.11 (m, 2 H), 7.23-7.27 (m, 5 H), 9.01 (brs, 2 H), 9.07-9.09, (m, 1 H);  $^{13}C$  NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  41.2, 42.6, 47.7, 47.8, 50.8,

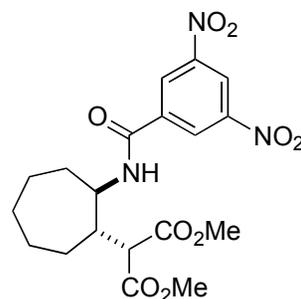


50.9, 52.5, 53.2, 53.3, 53.4, 67.3, 121.1, 127.4, 127.5, 127.8, 128.1, 128.5, 136.2, 137.2, 148.6, 154.6, 162.8, 168.6; LRMS (ESI):  $m/z$  567  $[M+Na]^+$ ; HRMS (ESI):  $m/z$  calculated for  $C_{24}H_{24}N_4NaO_{11}^+ [M+Na]^+$ : 567.1334, found: 567.1340; HPLC (chiral column: DAICEL

CHIRALPAK IB; solvent: hexane/2-propanol = 1/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R = 44.1$  min (minor) and 55.1 min (major);  $[\alpha]_D^{23.7} = -16.6$  ( $c = 1.25$ ,  $\text{CHCl}_3$ ).

**Dimethyl 2-((1*S*,2*R*)-2-(3,5-dinitrobenzamido)cycloheptyl)malonate (4fa):** colorless

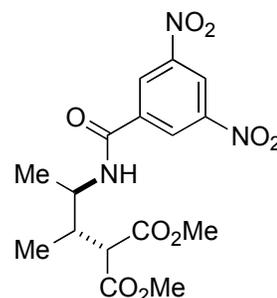
solid; IR (KBr)  $\nu$  3102, 2952, 2859, 1747, 1646, 1543, 1428, 1343, 1202, 1150  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.54-1.89 (m, 9 H), 2.01-2.04 (m, 1 H), 2.39-2.44 (m, 1 H), 3.60 (d,  $J = 3.4$  Hz, 1 H), 3.63 (s, 3 H), 3.784 (s, 3 H), 4.07-4.13 (m, 1H), 7.58 (d,  $J = 8.0$  Hz, 1 H), 8.97 (d,  $J = 1.8$  Hz, 2 H), 9.14 (t,  $J = 1.8$  Hz, 1 H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  23.8, 26.7, 27.0, 32.3, 34.9, 44.2, 52.7, 53.2, 53.7, 56.8, 120.8, 127.4, 138.3, 148.6, 161.8,



169.3, 171.9; LRMS (ESI):  $m/z$  460  $[\text{M}+\text{Na}]^+$ ; HRMS (ESI):  $m/z$  calculated for  $\text{C}_{19}\text{H}_{23}\text{N}_3\text{NaO}_9^+$   $[\text{M}+\text{Na}]^+$ : 460.1327, found: 460.1335; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R = 16.0$  min (major) and 26.7 min (minor);  $[\alpha]_D^{23.7} = +29.8$  ( $c = 0.55$ ,  $\text{CHCl}_3$ ).

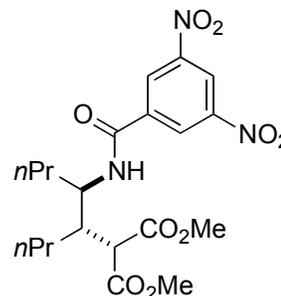
**Dimethyl 2-((2*S*,3*R*)-3-(3,5-dinitrobenzamido)butan-2-yl)malonate (4ga):** colorless

solid; IR (KBr)  $\nu$  3140, 3094, 2954, 1731, 1665, 1629, 1542, 1347, 1267  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.18 (d,  $J = 7.4$  Hz, 3 H), 1.30 (d,  $J = 6.7$  Hz, 3 H), 2.43-2.49 (m, 1 H), 3.61 (d,  $J = 4.0$  Hz, 1 H), 3.67 (s, 3 H), 3.81 (s, 3 H), 4.10-4.17 (m, 1H), 7.90 (d,  $J = 8.2$  Hz, 1 H), 8.98 (d,  $J = 2.2$  Hz, 2 H), 9.09 (t,  $J = 2.2$  Hz, 1 H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  16.3, 19.3, 37.9, 49.5, 52.7, 53.0, 54.3, 120.7, 127.3, 138.1, 148.5, 162.0, 169.2, 171.4; LRMS (ESI):  $m/z$



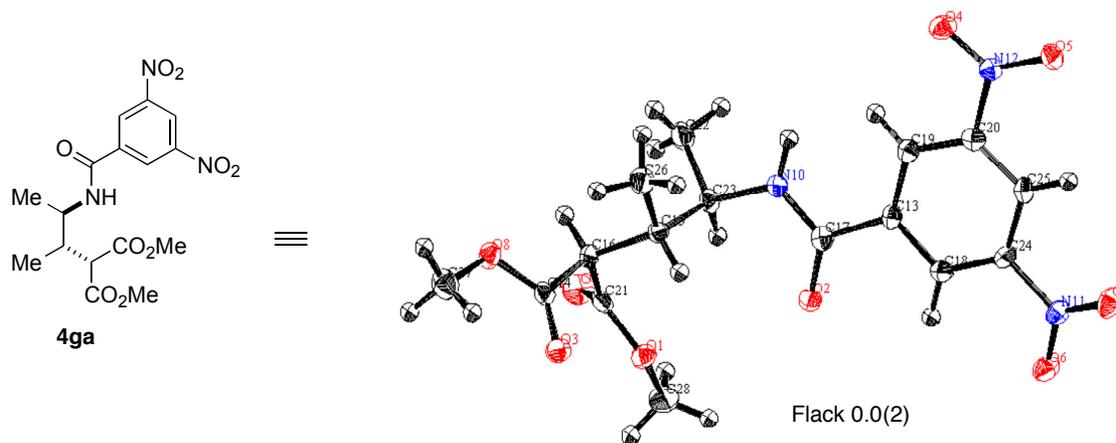
420  $[\text{M}+\text{Na}]^+$ ; HRMS (ESI):  $m/z$  calculated for  $\text{C}_{16}\text{H}_{19}\text{N}_3\text{NaO}_9^+$   $[\text{M}+\text{Na}]^+$ : 420.1014, found: 420.1018 HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R = 18.5$  min (major) and 43.7 min (minor);  $[\alpha]_D^{23.7} = +8.7$  ( $c = 1.04$ ,  $\text{CHCl}_3$ ).

**Dimethyl 2-((4*S*,5*R*)-5-(3,5-dinitrobenzamido)octan-4-yl)malonate (4ha):** colorless solid; IR (KBr)  $\nu$  3110, 2958, 2873, 1740, 1655, 1547, 1456, 1350, 1199, 1150  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.89-0.94 (m, 6 H), 1.34-1.47 (m, 5 H), 1.58-1.62 (m, 3 H), 2.32-2.34 (m, 1 H), 3.71 (s, 3 H), 3.75 (d,  $J = 2.5$  Hz, 1 H), 3.85 (s, 3 H), 4.30-4.35 (m, 1H), 8.03 (d,  $J = 9.2$  Hz, 1 H), 9.03 (d,  $J = 2.2$  Hz, 2 H), 9.12 (t,  $J = 2.2$  Hz, 1 H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  13.8, 13.9, 19.2, 20.4, 32.1, 36.4, 41.3, 50.7, 51.5, 52.8, 53.3, 120.7, 127.3, 138.2, 148.6, 162.1, 169.6, 172.4; LRMS (ESI):  $m/z$  476  $[\text{M}+\text{Na}]^+$ ; HRMS (ESI):  $m/z$  calculated for  $\text{C}_{20}\text{H}_{27}\text{N}_3\text{NaO}_9^+$   $[\text{M}+\text{Na}]^+$ : 476.1640, found: 476.1646; HPLC (chiral column: DAICEL CHIRALPAK IB; solvent: hexane/2-propanol = 2/1; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R = 10.2$  min (major) and 15.8 min (minor);  $[\alpha]_D^{23.7} = +21.9$  ( $c = 1.04$ ,  $\text{CHCl}_3$ ).



#### Determination of Relative and Absolute Configurations:

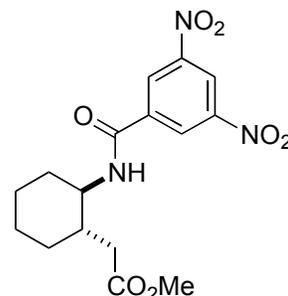
The relative and absolute configuration of **4ga** was determined by X-ray crystallographic analysis. Flack parameter was 0.0(2). CIF file of **4ga** is available as Supporting Information. Those of others were assigned by analogy.



### Transformation of Ring-opening Adduct (Scheme 1):

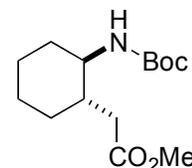
#### Methyl 2-((1*S*,2*R*)-2-(3,5-dinitrobenzamido)cyclohexyl)acetate (**5aa**):

To a solution **4aa** (169.3 mg, 0.40 mmol) in DMSO (0.8 mL) in a test tube were added H<sub>2</sub>O (8  $\mu$ L, 0.44 mmol, 1.1 equiv) and LiCl (35.6 mg, 0.84 mmol, 2.1 equiv), and the reaction mixture was stirred for 5 h at 130° C. After cooling down to rt, the reaction mixture was diluted with water, and extracted with EtOAc (x 3). The combined organic layers were washed with 1 M HCl aqueous solution, saturated NaHCO<sub>3</sub> aqueous solution, and brine, dried

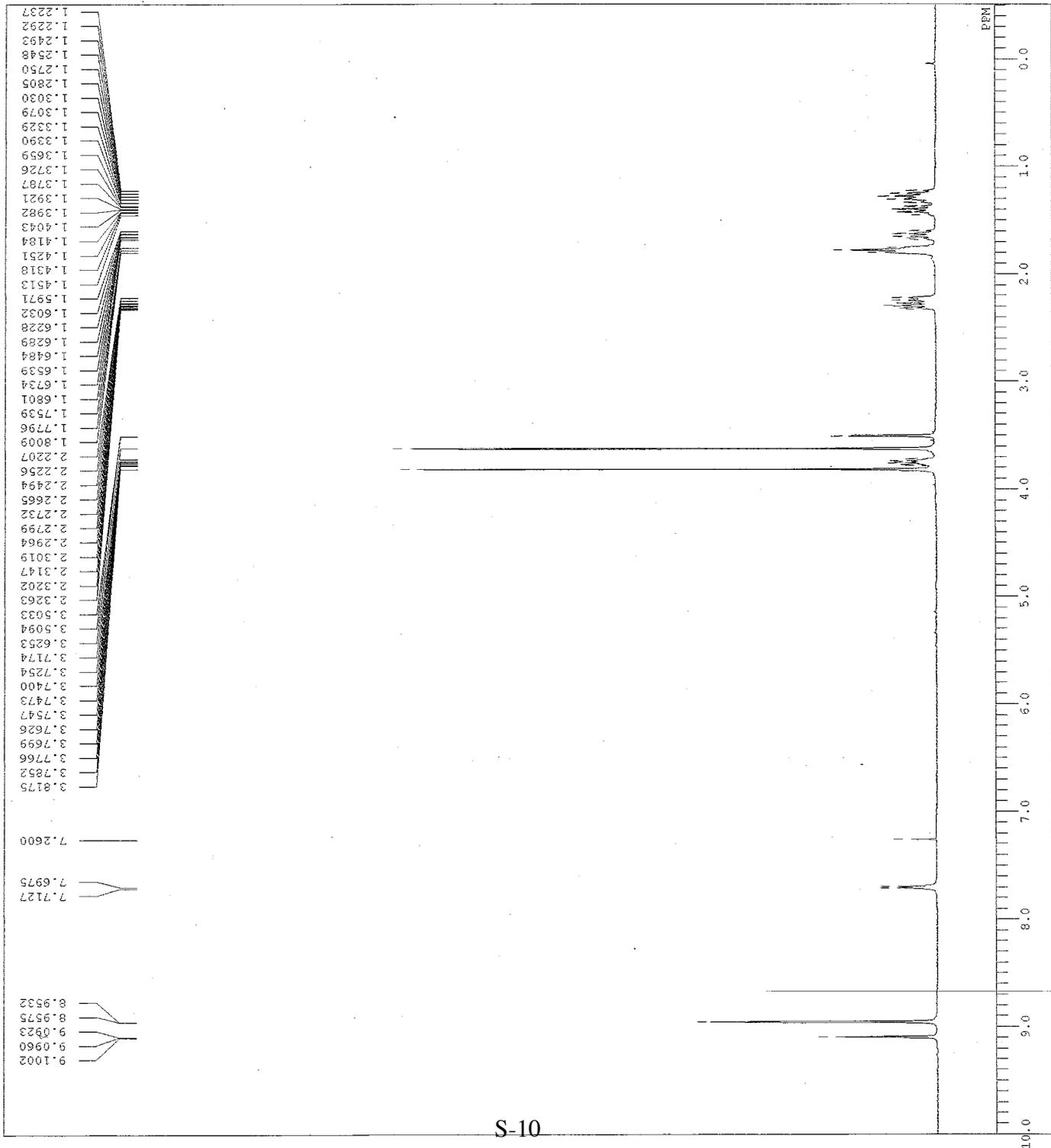


over Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The residue was purified by silica gel flash column chromatography (hexane: AcOEt = 10:1 to 4:1) to afford **5aa** (124.1 mg, 85% yield) as a colorless solid; IR (KBr)  $\nu$  3109, 2933, 2855, 1734, 1646, 1541, 1344 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.20-1.46 (m, 4 H), 1.77-1.84 (m, 3 H), 1.98-2.04 (m, 1 H), 2.25-2.28 (m, 1 H), 2.35 (dd, *J* = 3.2, 18.0 Hz, 1 H), 2.49 (dd, *J* = 8.3, 18.0 Hz, 1 H), 3.65-3.72 (m, 1H), 3.69 (s, 3H), 7.36 (d, *J* = 7.5 Hz, 1 H), 8.99 (d, *J* = 2.3 Hz, 2 H), 9.14 (t, *J* = 2.3 Hz, 1 H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  24.9, 25.6, 33.0, 33.2, 38.5, 39.2, 52.1, 55.9, 120.8, 127.2, 138.2, 148.6, 162.0, 175.7; LRMS (ESI): *m/z* 388 [M+Na]<sup>+</sup>; HRMS (ESI): *m/z* calculated for C<sub>16</sub>H<sub>19</sub>N<sub>3</sub>NaO<sub>7</sub><sup>+</sup> [M+Na]<sup>+</sup>: 388.1115, found: 388.1125; [ $\alpha$ ]<sub>D</sub><sup>23.7</sup> = -3.6 (*c* = 0.83, CHCl<sub>3</sub>).

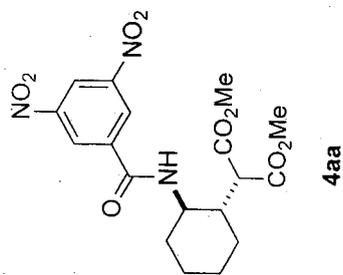
**Methyl 2-((1*S*,2*R*)-2-(tert-butoxycarbonylamino)cyclohexyl)acetate (**6aa**):** To a solution of **5aa** (109.6 mg, 0.30 mmol) in THF (0.6 mL) were added Boc<sub>2</sub>O (589.3 mg, 2.70 mmol, 9 equiv), Et<sub>3</sub>N (46  $\mu$ L, 0.33 mmol, 1.1 equiv) and DMAP (7.3 mg, 0.06 mmol, 0.2 equiv), and the mixture stirred at rt for 24 h. The volatile material was removed under reduced poressure and the residue was purified by silica gel flash column chromatography (hexane: AcOEt = 15:1) to afford *N*-Boc protected intermediate as a colorless oil. The intermediate was dissolved in MeOH (1.5 ml), and NaOMe (17.8 mg, 0.33 mmol, 1.1 equiv) was added at rt. The resulting mixture was stirred for 1 h at rt. The reaction was quenched with citric acid (230 mg, 1.20 mmol, 4 equiv) and then the volatile material was removed under reduced poressure. The



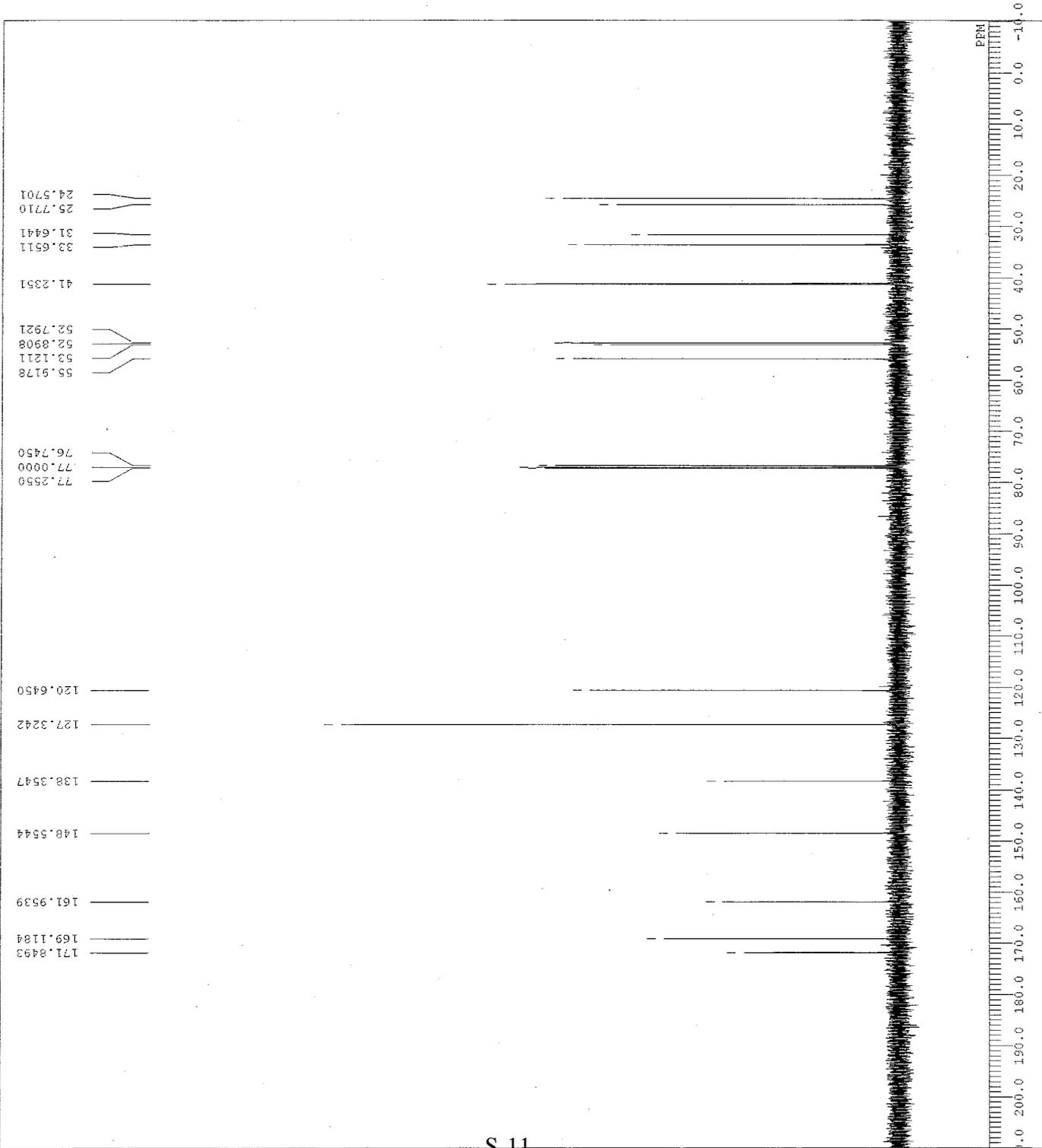
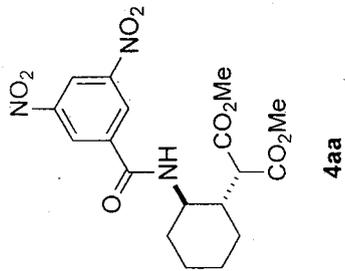
residue was taken up in H<sub>2</sub>O (10 mL), and the organic material was extracted with AcOEt (3 × 10 mL). The combined organic layers were dried (MgSO<sub>4</sub>), filtered, and evaporated. The residue was purified by silica gel flash column chromatography (hexane:CH<sub>2</sub>Cl<sub>2</sub>:Et<sub>2</sub>O = 15:1:1 to 10:1:1) to afford **6aa** (77.4 mg, 95% yield in 2 steps) as a colorless solid IR (KBr)  $\nu$  2979, 2936, 2857, 1736, 1682, 1520 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.01-1.31 (m, 4 H), 1.39 (s, 9 H), 1.62-1.71 (m, 3 H), 1.75-1.79 (m, 1 H), 1.92-1.95 (m, 1 H), 2.07 (dd,  $J$  = 7.6, 15.7 Hz, 1 H), 2.51 (dd,  $J$  = 5.5, 15.7 Hz, 1 H), 3.14-3.21 (m, 1H), 3.63 (s, 3 H), 4.40 (d,  $J$  = 9.2 Hz, 1 H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  25.3, 25.4, 28.3, 32.2, 34.2, 38.4, 40.6, 51.5, 54.1, 79.0, 155.4, 174.2; LRMS (ESI):  $m/z$  294 [M+Na]<sup>+</sup>; HRMS (ESI):  $m/z$  calculated for C<sub>14</sub>H<sub>25</sub>N<sub>1</sub>NaO<sub>4</sub><sup>+</sup> [M+Na]<sup>+</sup>: 294.1676, found: 294.1688; [ $\alpha$ ]<sub>D</sub><sup>23.7</sup> = -3.1 ( $c$  = 1.08, CHCl<sub>3</sub>).



DFILE xul092-cHex-1H.als  
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 EXMOD non  
 OBFRQ 500.00 MHz  
 OBSEF 160.00 KHz  
 OBFIN 2160.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 ACQTM 5  
 SCANS 3.2768 sec  
 PD 3.7232 sec  
 PFI 5.90 usec  
 IRNUC 1H  
 CTENP 27.5 c  
 SLVNT CDCL3  
 EXREF 7.26 ppm  
 BF 0.12 Hz  
 RGAIN 15

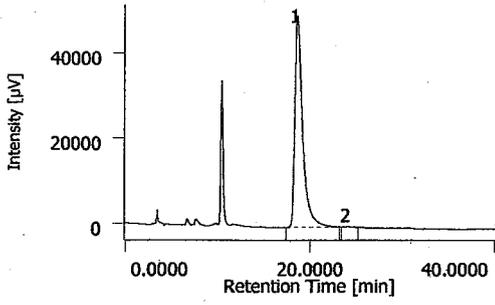


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 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 113  
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 PD 2.0333 sec  
 PWL 5.12 usec  
 IRNUC 1H 28.9 c  
 CTEMP  
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 EXREF 0.12 Hz  
 RF  
 RGAIN 30



xu101218-2 xu1113  
クロマト

chiral 4aa



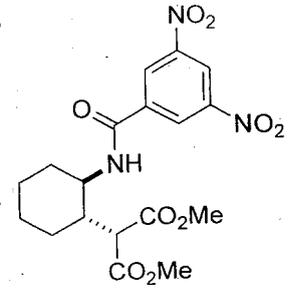
ピーク情報

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area %

99.5% ee

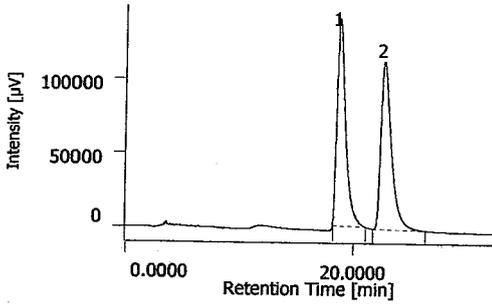
測定日 2010/12/18 14:44:07  
コントロールメソッド 1ml\_254nm



4aa

xu101008 xu1011  
クロマト

rac-4aa



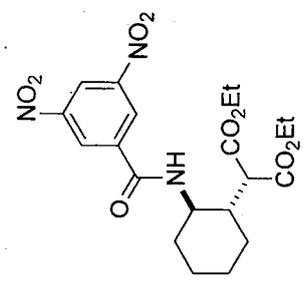
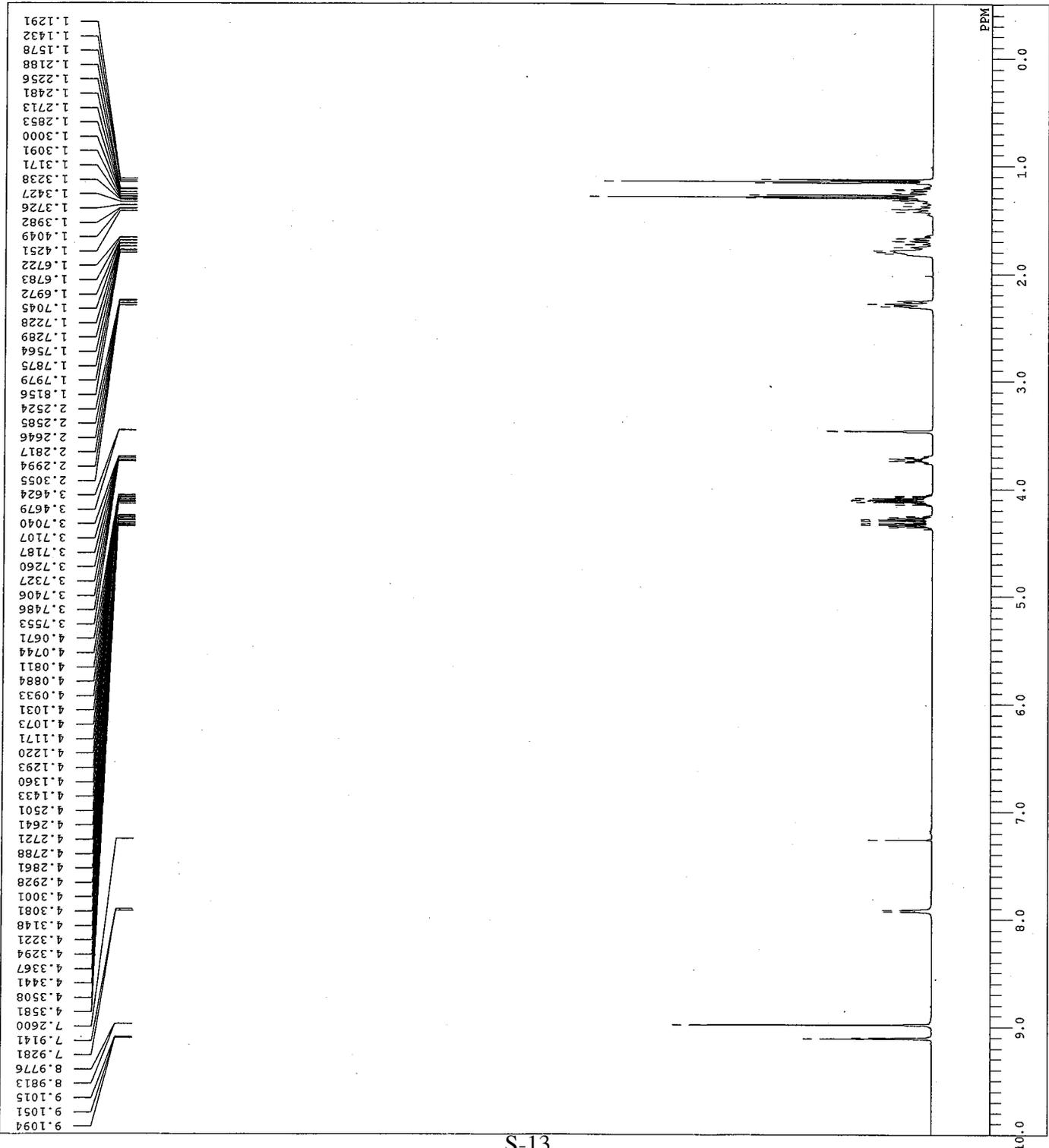
ピーク情報

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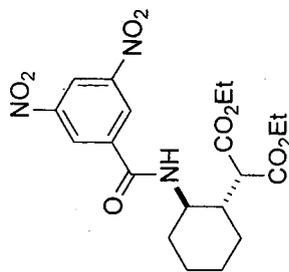
area %

測定日 2010/10/08 20:25:42  
コントロールメソッド 1ml\_254nm

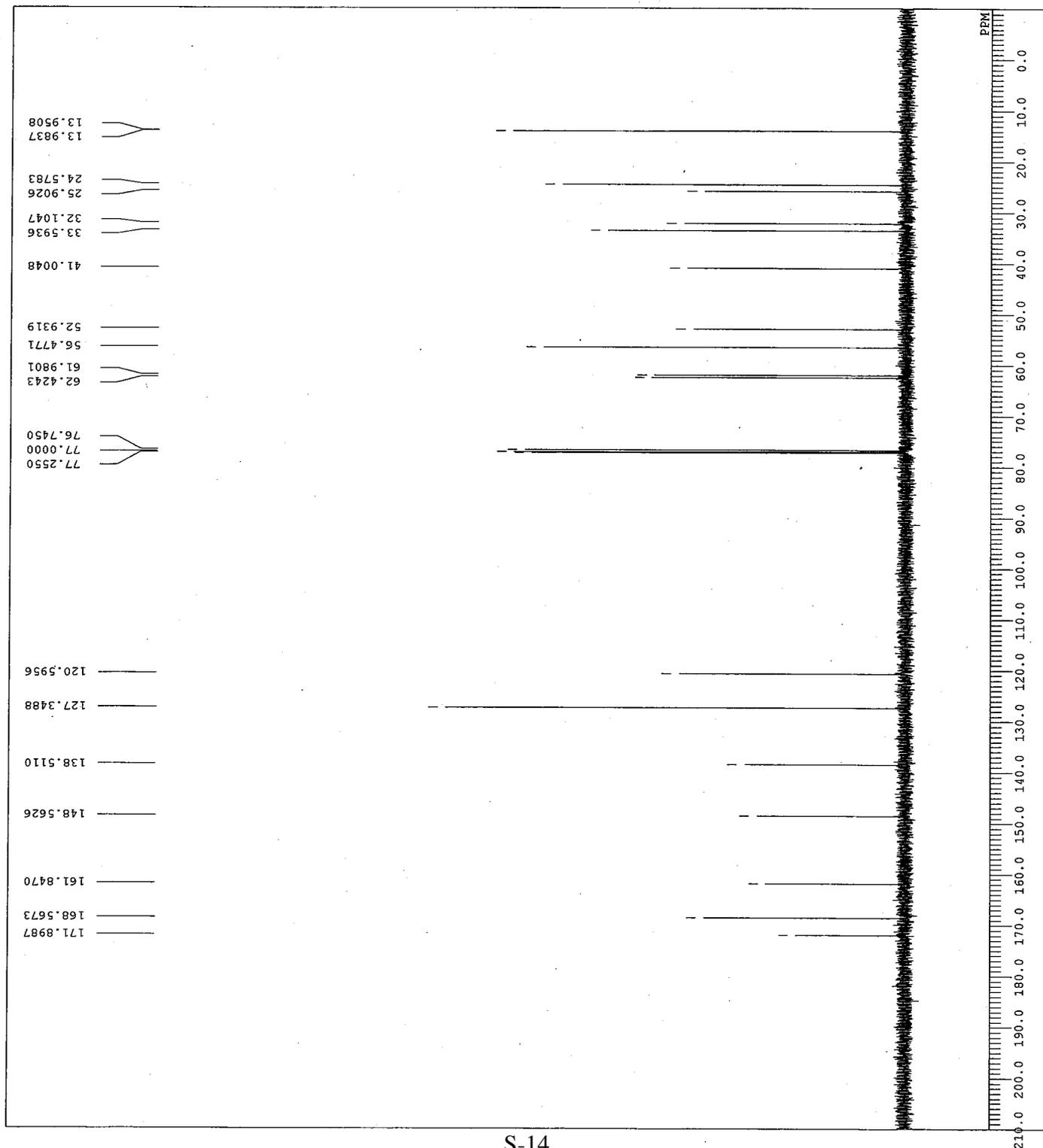
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DATIM Sat Jan 22 22:18:54 2011  
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EXMOD non  
OBFRO 500.00 MHz  
OBFET 160.00 KHz  
OBFIN 2160.00 Hz  
POINT 32768  
FREQU 10000.00 Hz  
SCANS 8  
ACQTM 3.2768 sec  
PD 3.7232 sec  
FW1 5.90 usec  
IRNUC 1H  
CTEMP 27.0 c  
SLVNT CDCL3  
EXREF 7.26 ppm  
BF 0.12 Hz  
RGAIN 16



DTITLE XU1130-OEt-13C.als  
 COMNT 1130-OEt-13C  
 DAYTM Sat Jan 22 22:29:03 2011  
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 EXMOD bcm  
 OBFREQ 125.65 MHz  
 OBSET 120.00 KHz  
 OBFIN 7958.00 Hz  
 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 191  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
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 CTEMP 28.9 C  
 SIVNT CDCL3  
 EXREF 77.00 ppm  
 BF 0.12 Hz  
 RGAIN 30

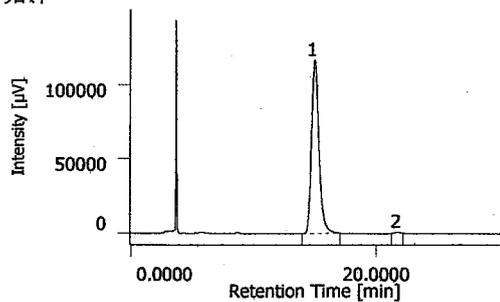


4ab



xu110122-OEt-IC xu1130-Et  
クロマト

chiral 4ab



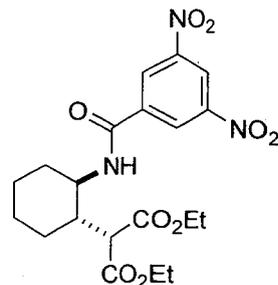
ピーク情報

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1	Unknown	14.958	4828176	99.674
2	Unknown	21.742	15796	0.326

area%

99.34% ee

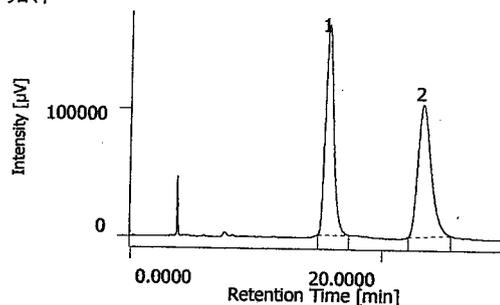
測定日 2011/01/22 20:03:33  
コントロールメソッド 1ml\_254nm



4ab

xu110113-1117-EtMalonate3 xu1117-IC  
クロマト

rac 4ab

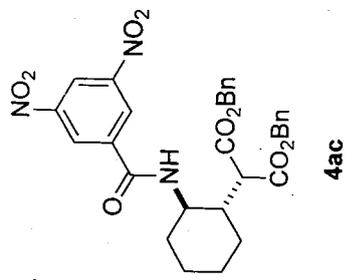
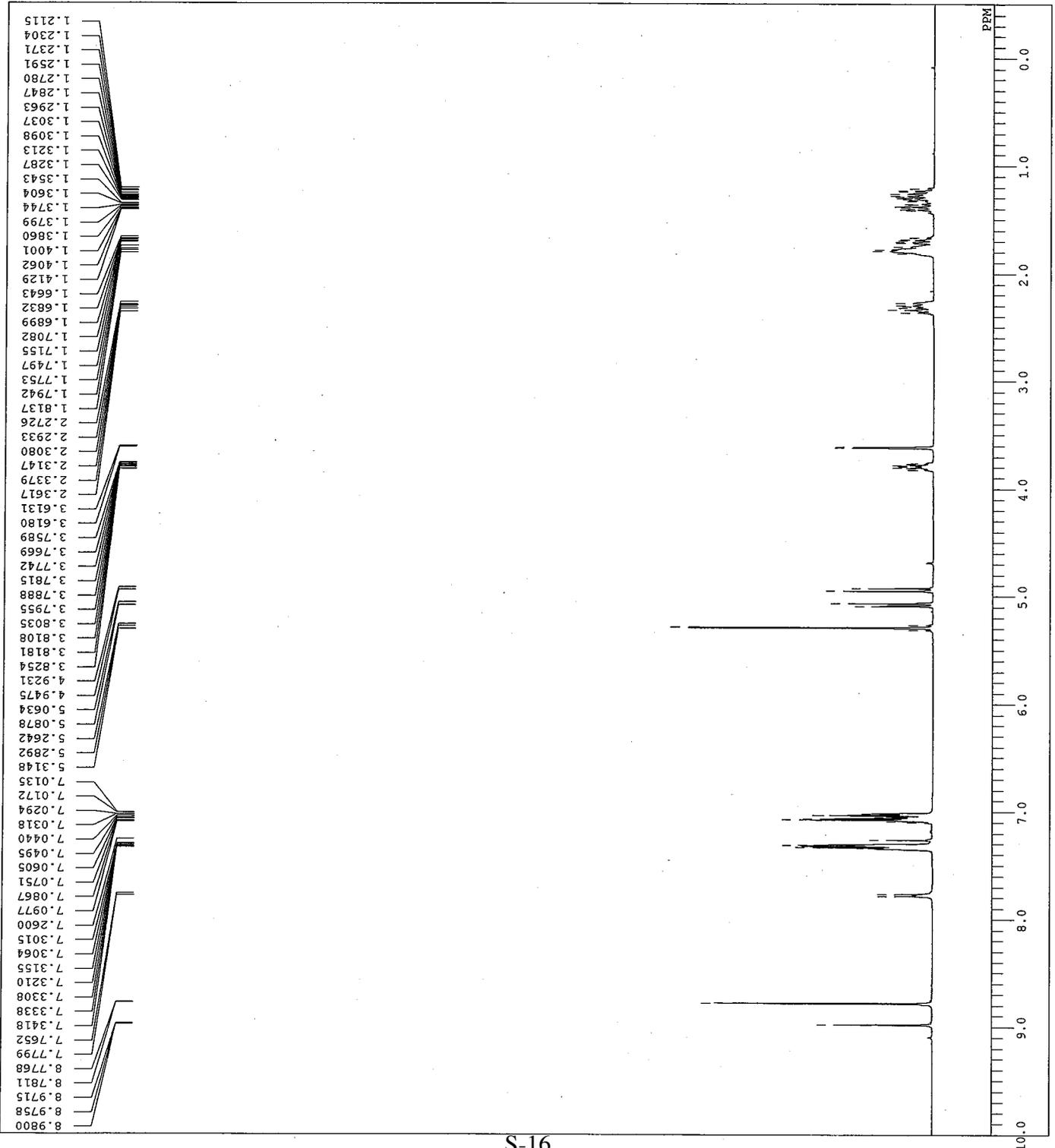


ピーク情報

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1	Unknown	15.667	7371412	49.846
2	Unknown	23.100	7417036	50.154

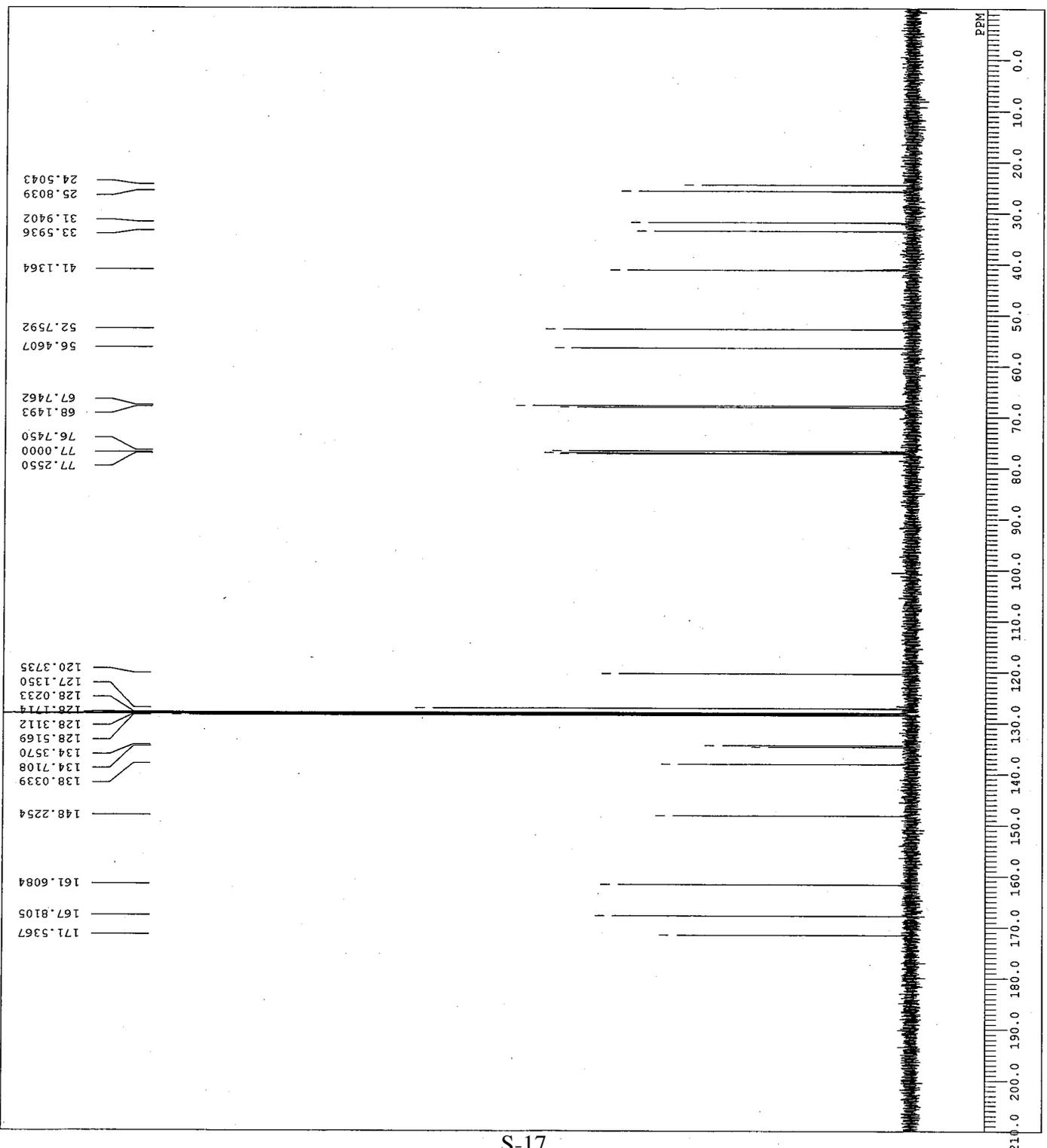
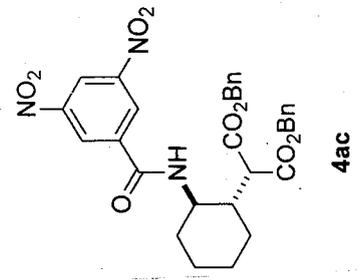
area%

測定日 2011/01/13 22:04:14  
コントロールメソッド 1ml\_254nm



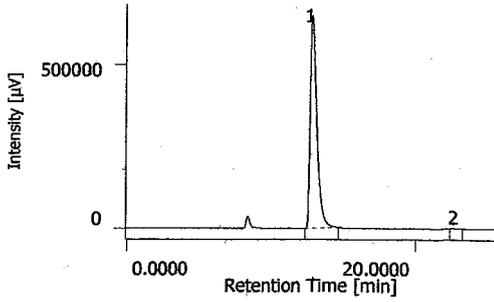
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 EXMOD non  
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 OBFIN 2160.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 SCANS 8  
 ACQTM 3.2768 sec  
 PD 3.7232 sec  
 PW 5.90 usec  
 IRRUC 1H  
 CTEMP 28.1 c  
 SLVNT CDCL3  
 EXREF 7.26 ppm  
 BF 0.12 Hz  
 RGAIN 14

DEFILE xu1131-OBn-13C.a1s  
 COMNT 1131-OBn-13C  
 DATIM Sat Jan 22 22:40:05 2011  
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 EXMOD bcm  
 OBFRQ 125.65 MHz  
 OBSET 120.00 KHz  
 OBFIN 7958.00 Hz  
 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 120  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 PW1 5.12 usec  
 IRNUC 1H  
 CTMP 28.7 c  
 CDCL3  
 SLVNT  
 EXREF 77.00 ppm  
 BF 0.12 Hz  
 RGAIN 30



xu110122-OBn xu1131-Bn  
クロマト

Chiral 4ac



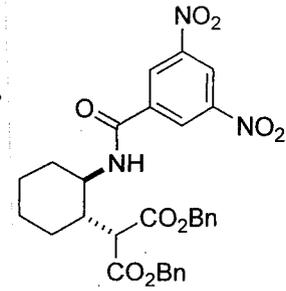
ピーク情報

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1	Unknown	12.842	20347208	99.856
2	Unknown	22.842	29303	0.144

area%

99.7% ee

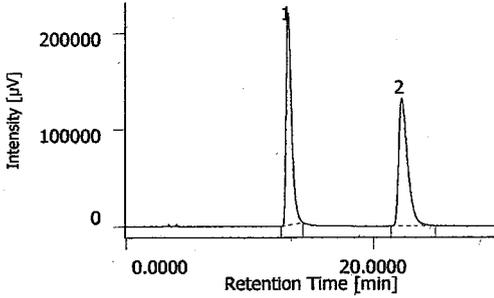
測定日 2011/01/22 13:09:02  
コントロールメソッド 1ml\_254nm



4ac

xu110122-OBn xu1118-Bn-R  
クロマト

rac 4ac



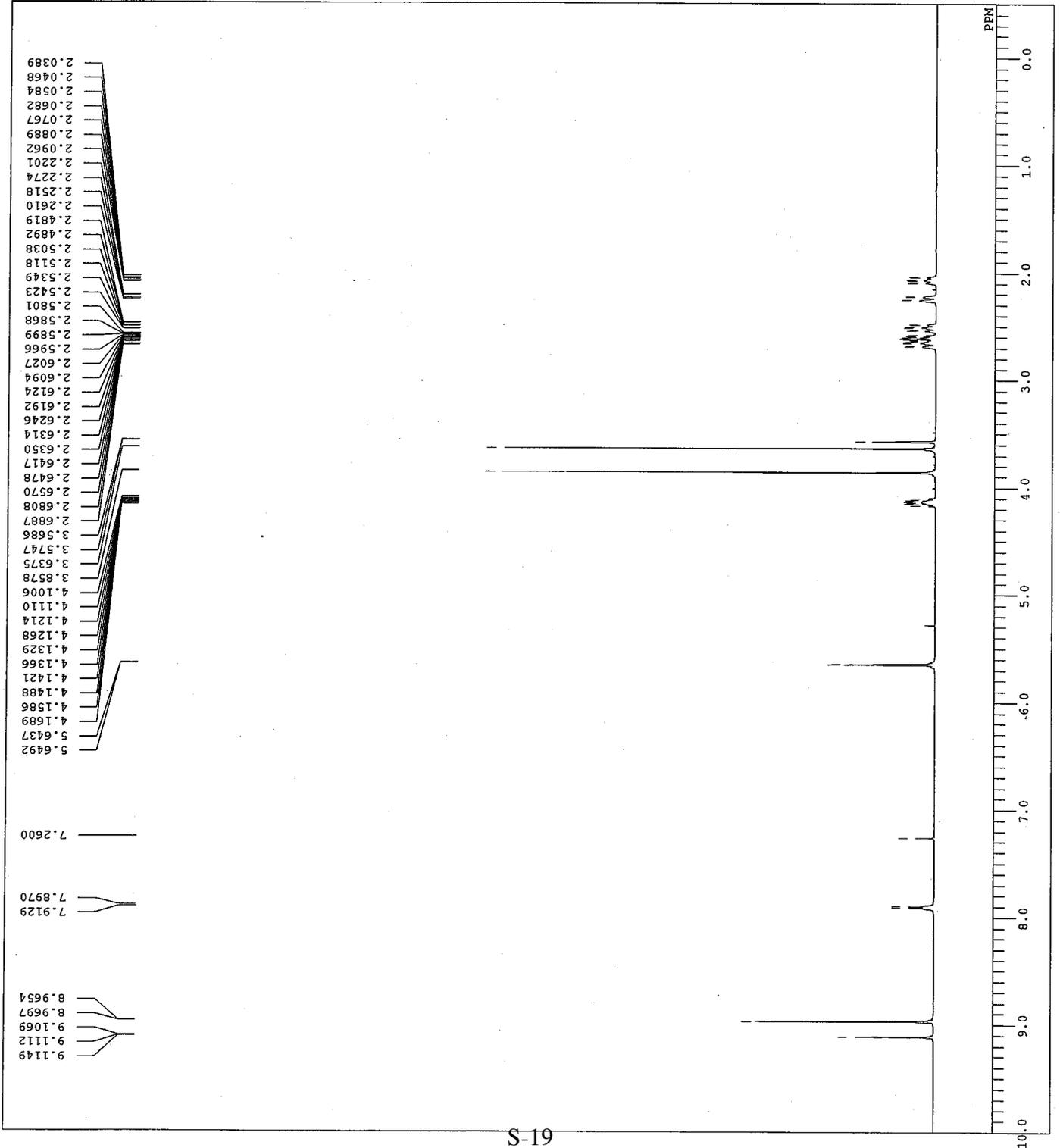
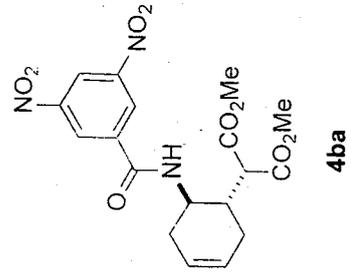
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1	Unknown	13.167	6701192	50.361
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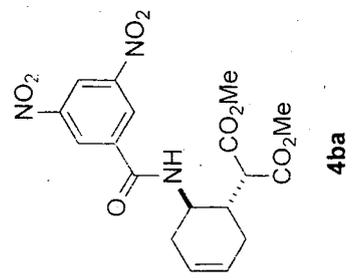
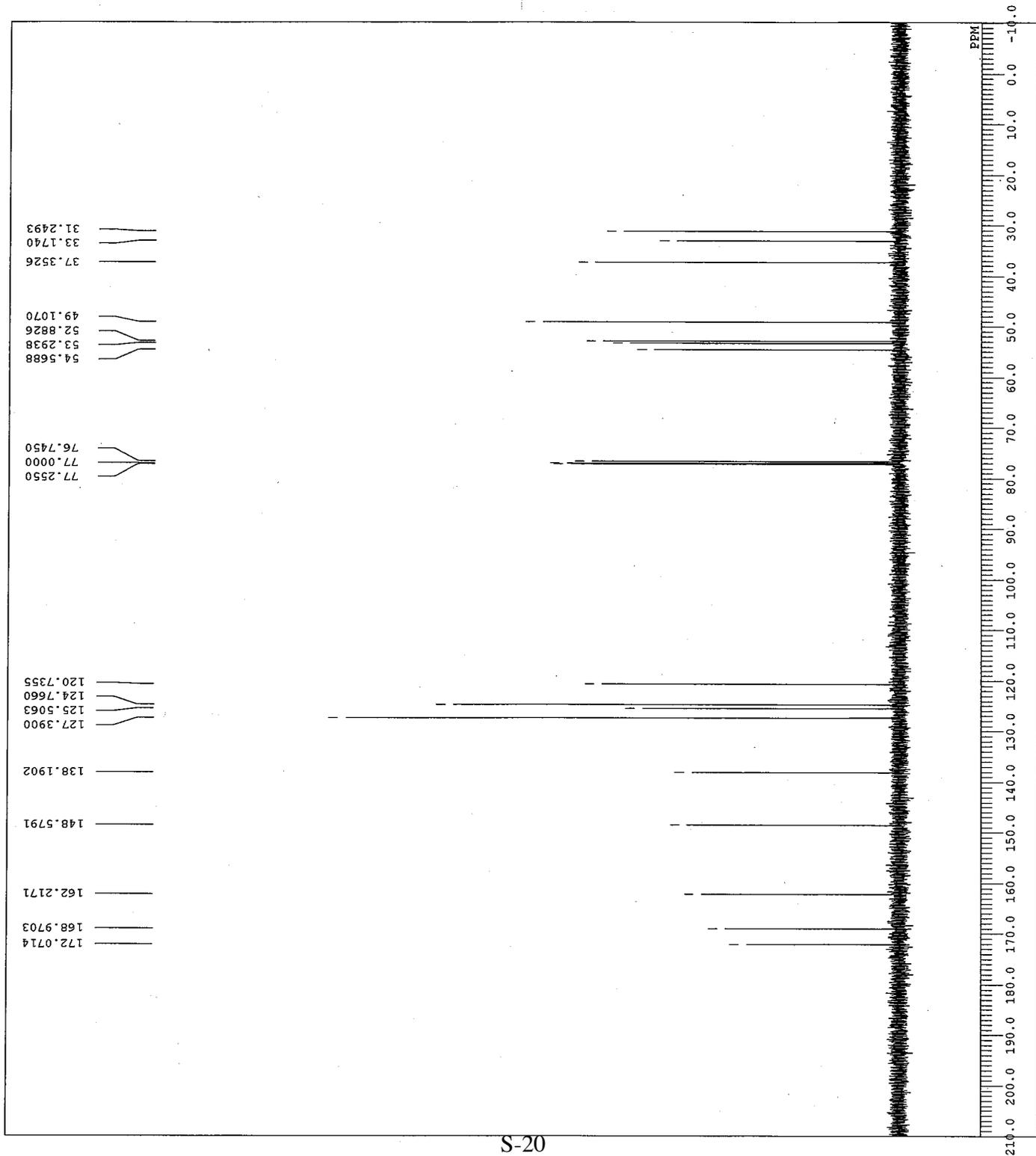
area%

測定日 2011/01/22 13:35:13  
コントロールメソッド 1ml\_254nm

DFILF xul126-cHexene-1H.als  
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 EXMOD non  
 OBFRO 500.00 MHz  
 OBSET 160.00 KHz  
 OBEIN 2160.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 SCANS 6  
 ACQTM 3.2768 sec  
 PD 3.7232 sec  
 PW1 5.90 usec  
 IRNUC 1H  
 CTEMP 27.5 c  
 SLVNT CDCl3  
 EXREF 7.26 ppm  
 BF 0.12 Hz  
 RGAIN 15

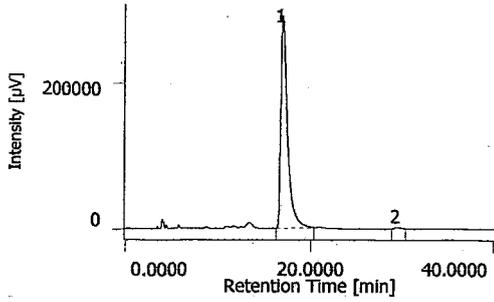


DFILE xul126-cHexene-13C.als  
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 FREQU 33898.30 Hz  
 SCANS 121  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 FWI 5.12 usec  
 IRNUC 1H  
 CTEMP 28.9 c  
 SLVNT CDCL3  
 EXREF 77.00 ppm  
 BF 0.12 Hz  
 RGAIN 30



xu110114 xu1126-cHexene  
クロマト

chiral 4ba



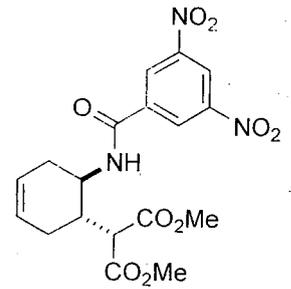
ピーク情報

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2	Unknown	29.450	81033	0.557

area%

99.100

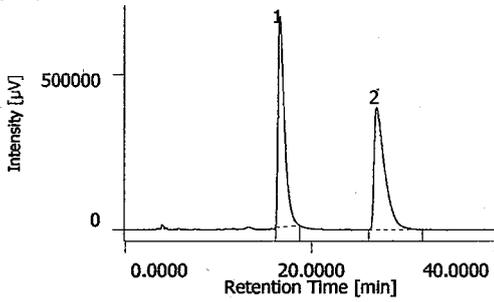
測定日 2011/01/14 12:19:17  
コントロールメソッド 1ml\_254nm



4ba

xu110114 xu1103-cHexene-R  
クロマト

rac 4ba



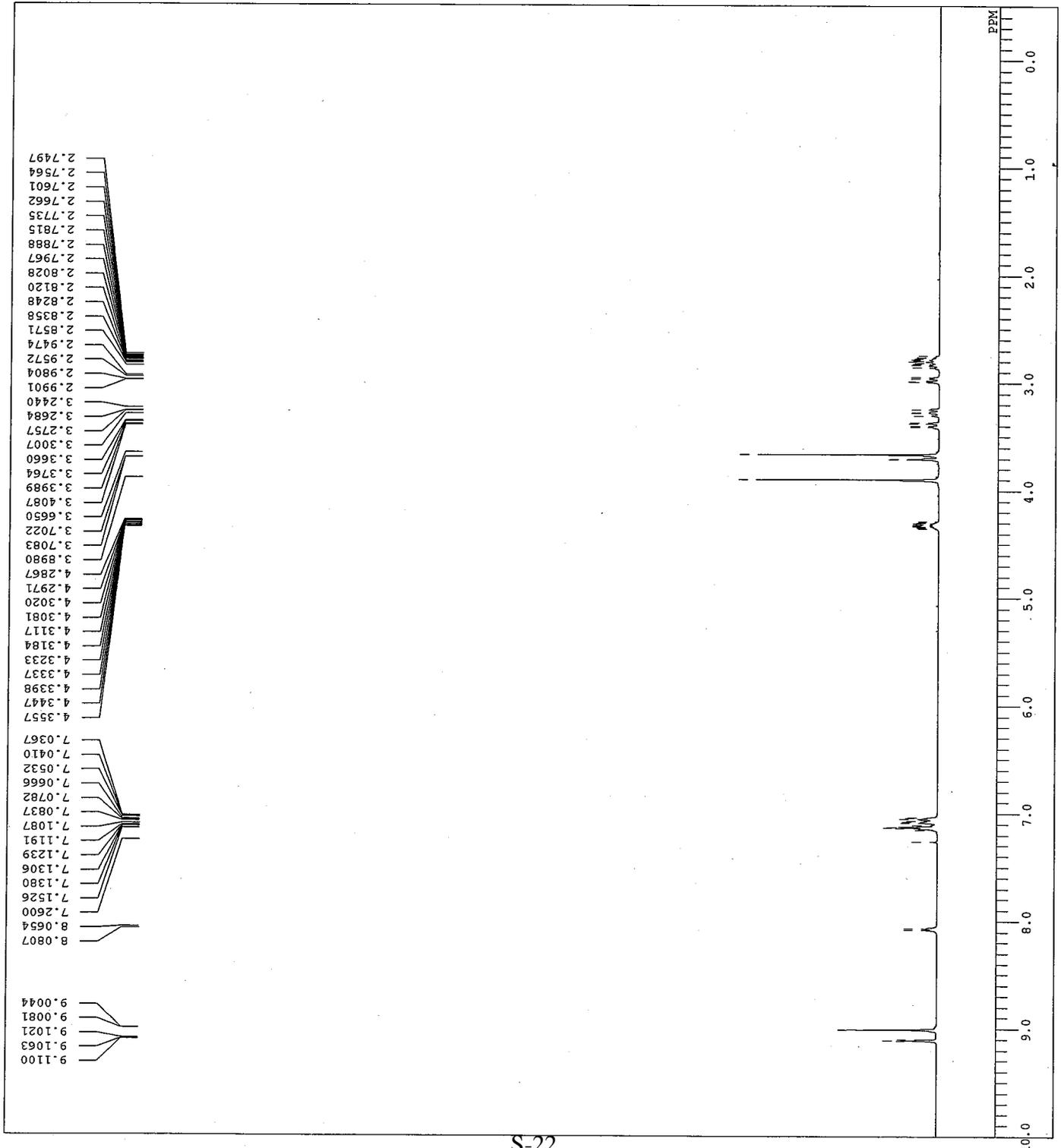
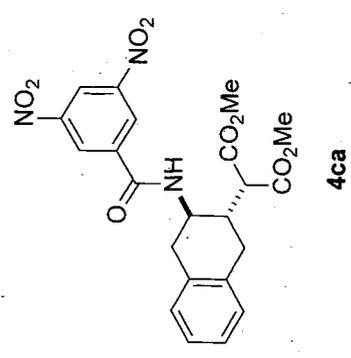
ピーク情報

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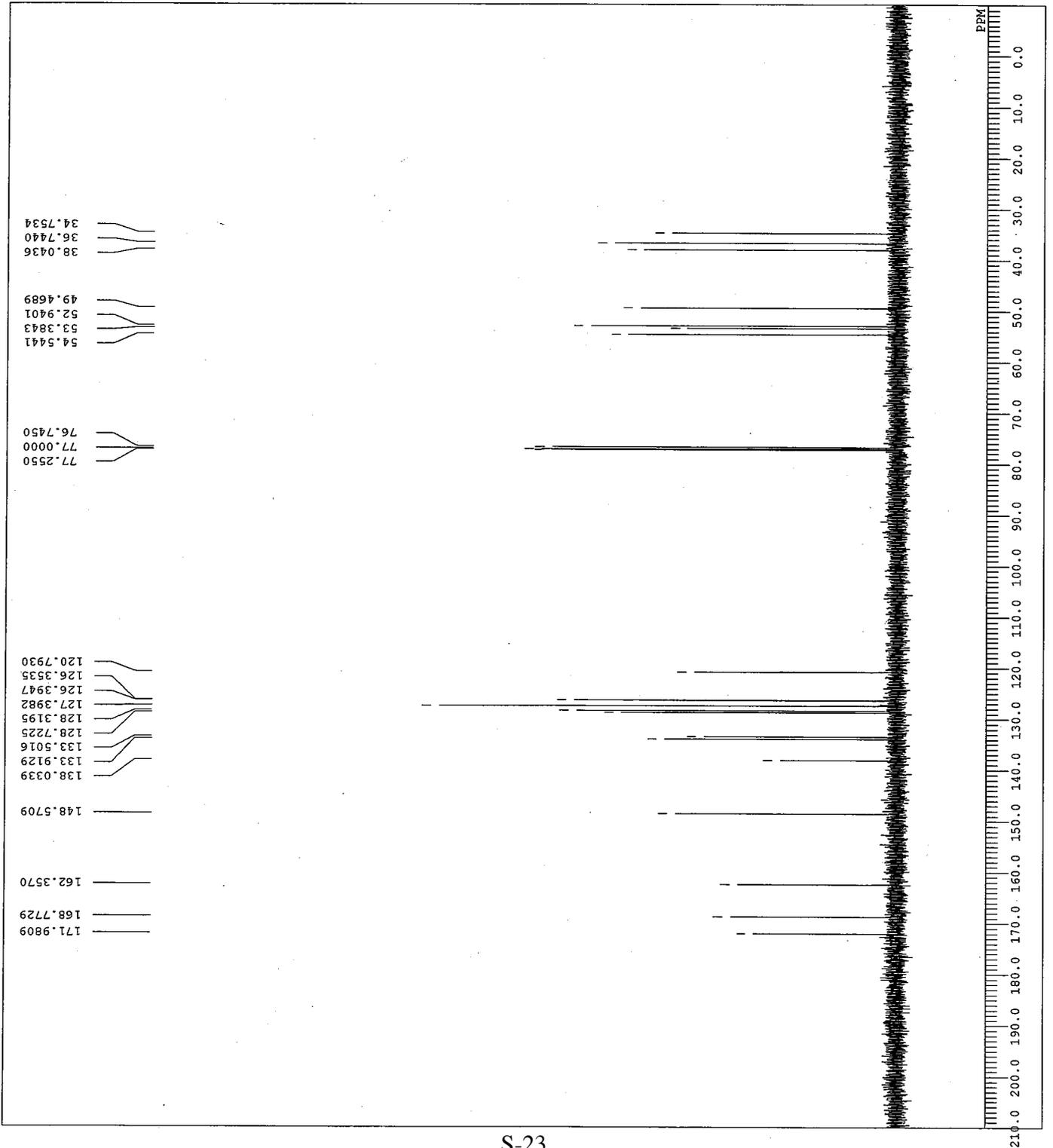
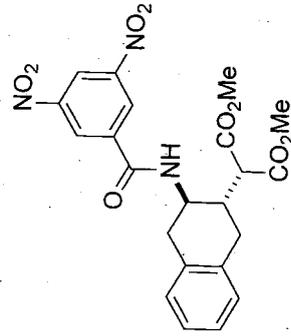
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測定日 2011/01/14 11:37:17  
コントロールメソッド 1ml\_254nm

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 EXMOD non  
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 OBSET 160.00 KHz  
 OBFLN 2160.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 SCANS 5  
 ACQTM 3.2768 sec  
 PD 3.7232 sec  
 PWL 5.90 usec  
 IRNUC 1H  
 CTEMP 27.9 c  
 SLVNT CDCL3  
 EXREF 7.26 ppm  
 BF 1.00 Hz  
 RGAIN 14

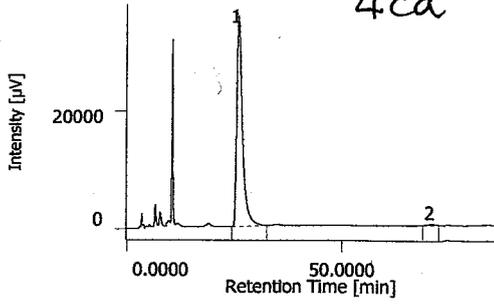


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 EXMOD bcm  
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 OBSET 120.00 KHz  
 OBFIN 7958.00 Hz  
 POINT 32768  
 FREOU 33898.30 Hz  
 SCANS 102  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 PWL 5.12 usec  
 IRNUC 1H  
 CTEMP 29.0 C  
 SIVNT CDCL3  
 EXREF 77.00 ppm  
 BF 1.00 Hz  
 RGAIN 31



xu10127 xu1122  
クロマト

chiral  
4ca



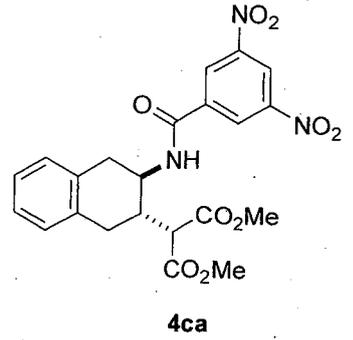
ピーク情報

area%

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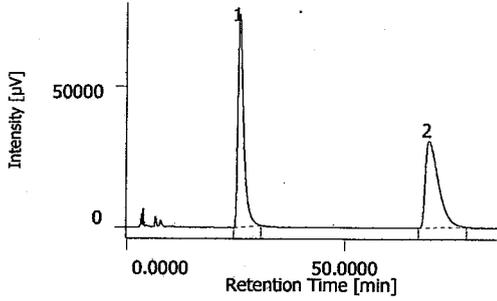
99.99%

測定日 2010/12/27 15:20:29  
コントロールメソッド 1ml\_254nm



xu10124 xu1111-R  
クロマト

IB 2/1  
rac 4ca



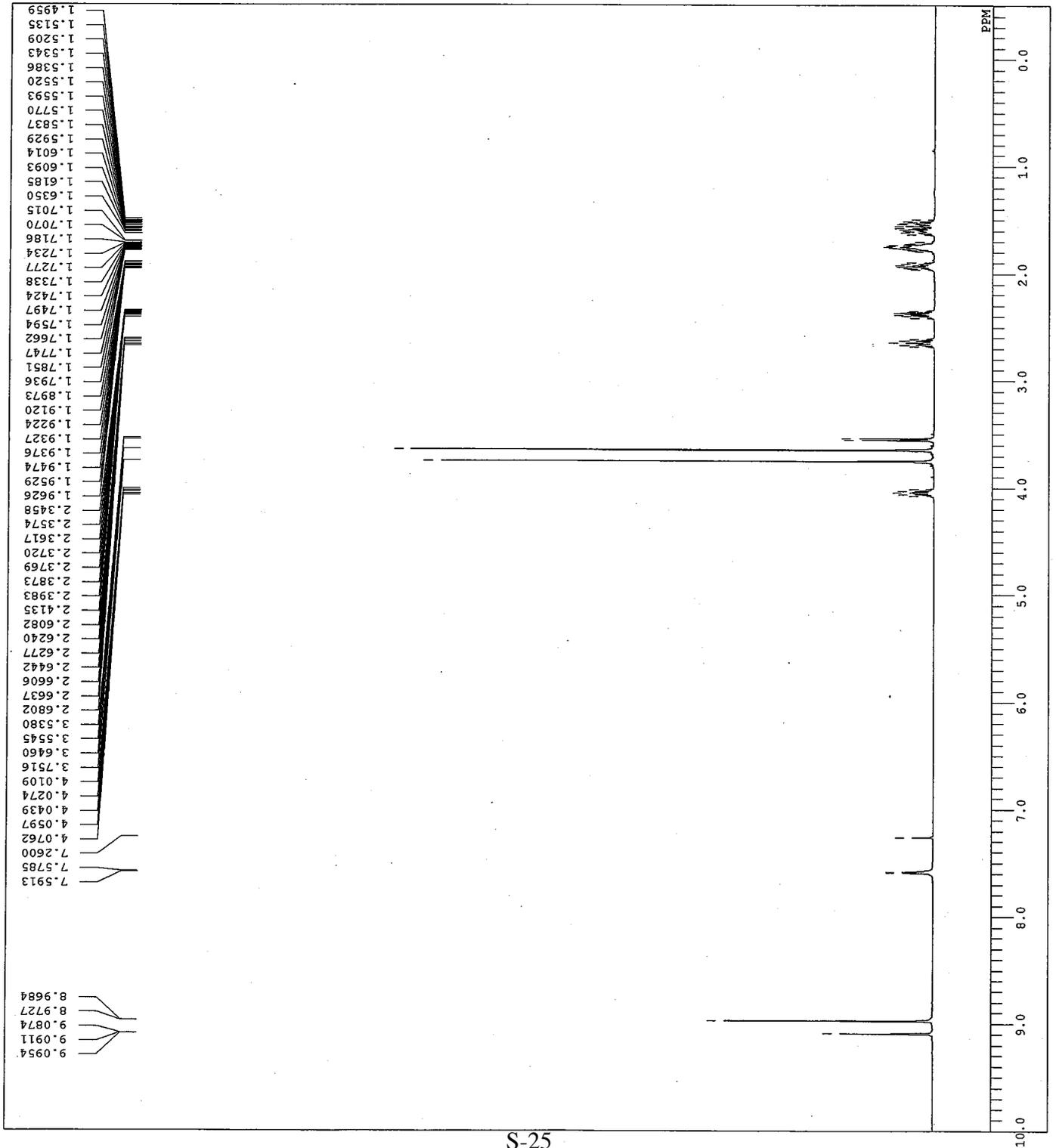
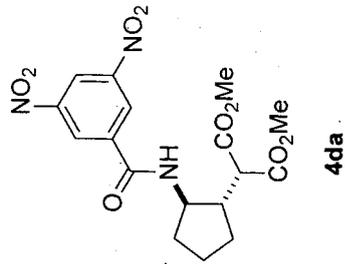
ピーク情報

area%

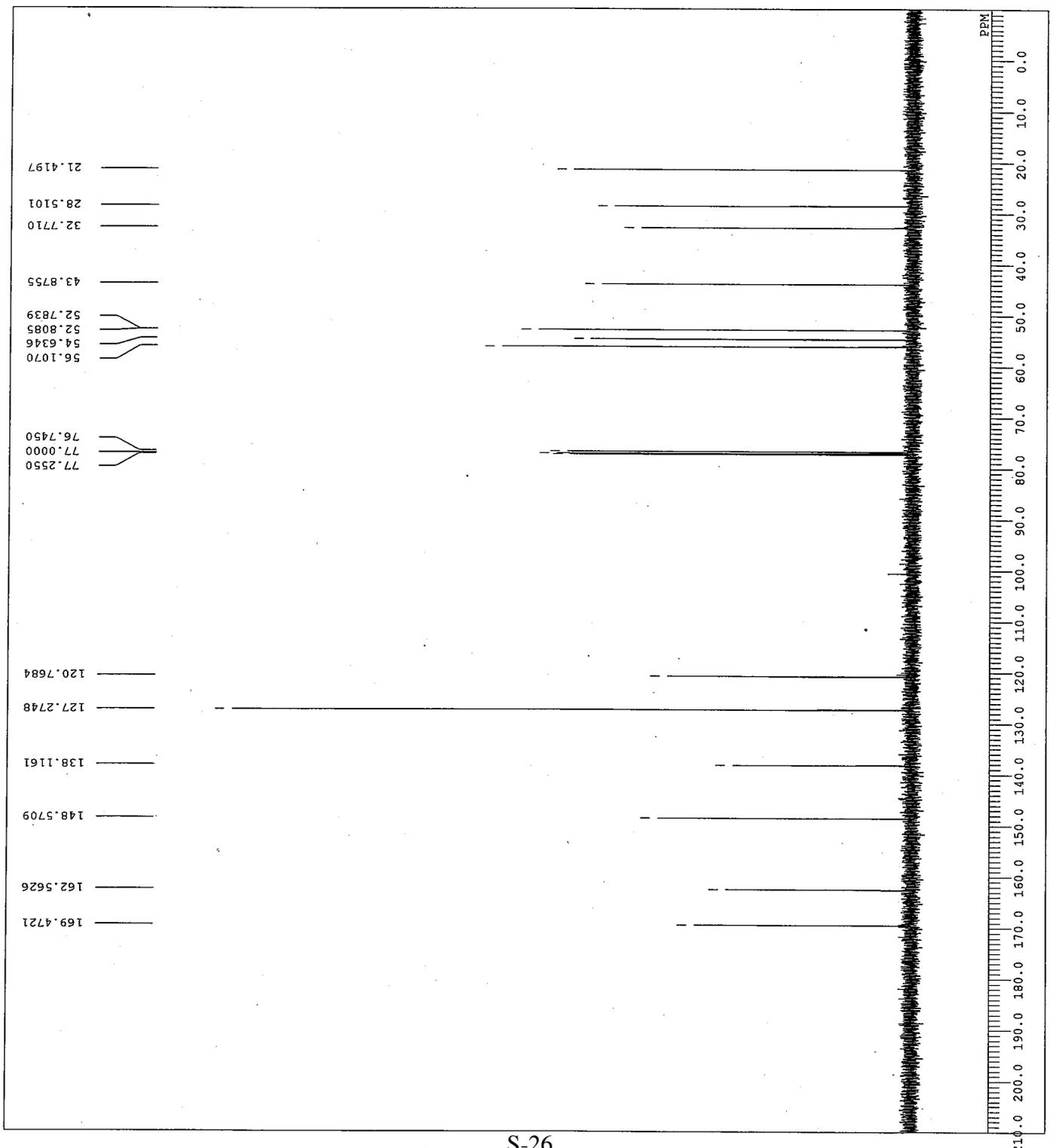
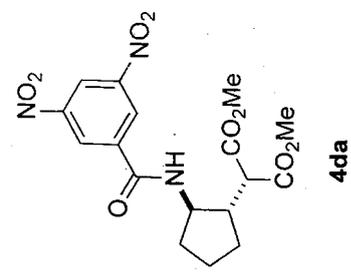
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1	Unknown	25.575	6660324	50.921
2	Unknown	69.092	6419281	49.079

測定日 2010/12/24 15:30:58  
コントロールメソッド 1ml\_254nm

DFILE cul105-Pen-1H.als  
 COMNT 1105-Pen-1H  
 DATIM Fri Jan 28 21:23:05 2011  
 OBNUC 1H  
 EXMOD non  
 OBFRQ 500.00 MHz  
 OBSET 160.00 KHz  
 OBFIN 2160.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 SCANS 6  
 ACQIM 3.2768 sec  
 PD 3.7232 sec  
 PW1 5.90 usec  
 IRNUC 1H  
 CTMP 26.8 c  
 SLVNT CDCL3  
 EXREF 7.26 ppm  
 BF 1.00 Hz  
 RGAIN 15

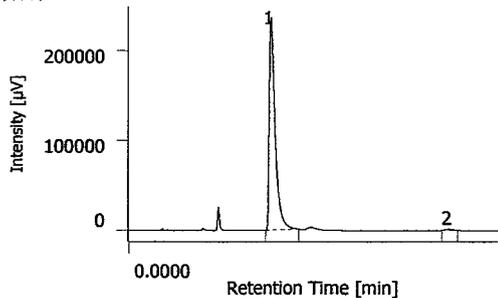


DFILE xul105-pen-13c.als  
 COMNT 1105-Pen-13C  
 DATIM Fri Jan 28 21:31:34 2011  
 OBNUC 13C  
 EXMOD bcm  
 OBFRQ 125.65 MHz  
 OBSET 120.00 KHz  
 OBFIN 7958.00 Hz  
 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 158  
 ACOPTM 0.9667 sec  
 PD 2.0333 sec  
 PWL 5.12 usec  
 IRNUC 1H  
 CTMPC CDCL3  
 SLVNT 28.4 C  
 EXREF 77.00 ppm  
 BF 0.12 Hz  
 RGAIN 30



chiral 4da

xu110127-4 xu1105  
クロマト

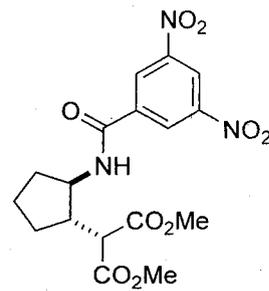


ピーク情報 *area%*

#	ピーク名	tR [min]	面積 [μV·sec]	面積%
1	Unknown	17.958	12060186	99.418
2	Unknown	40.175	70596	0.582

99.418

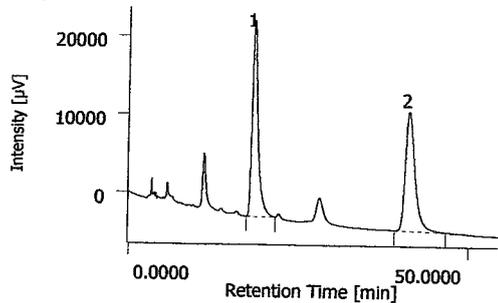
測定日 2011/01/27 18:09:42  
コントロールメソッド 1ml\_254nm



4da

rac 4da

xu101117 xu1044-Rac  
クロマト



ピーク情報 *area%*

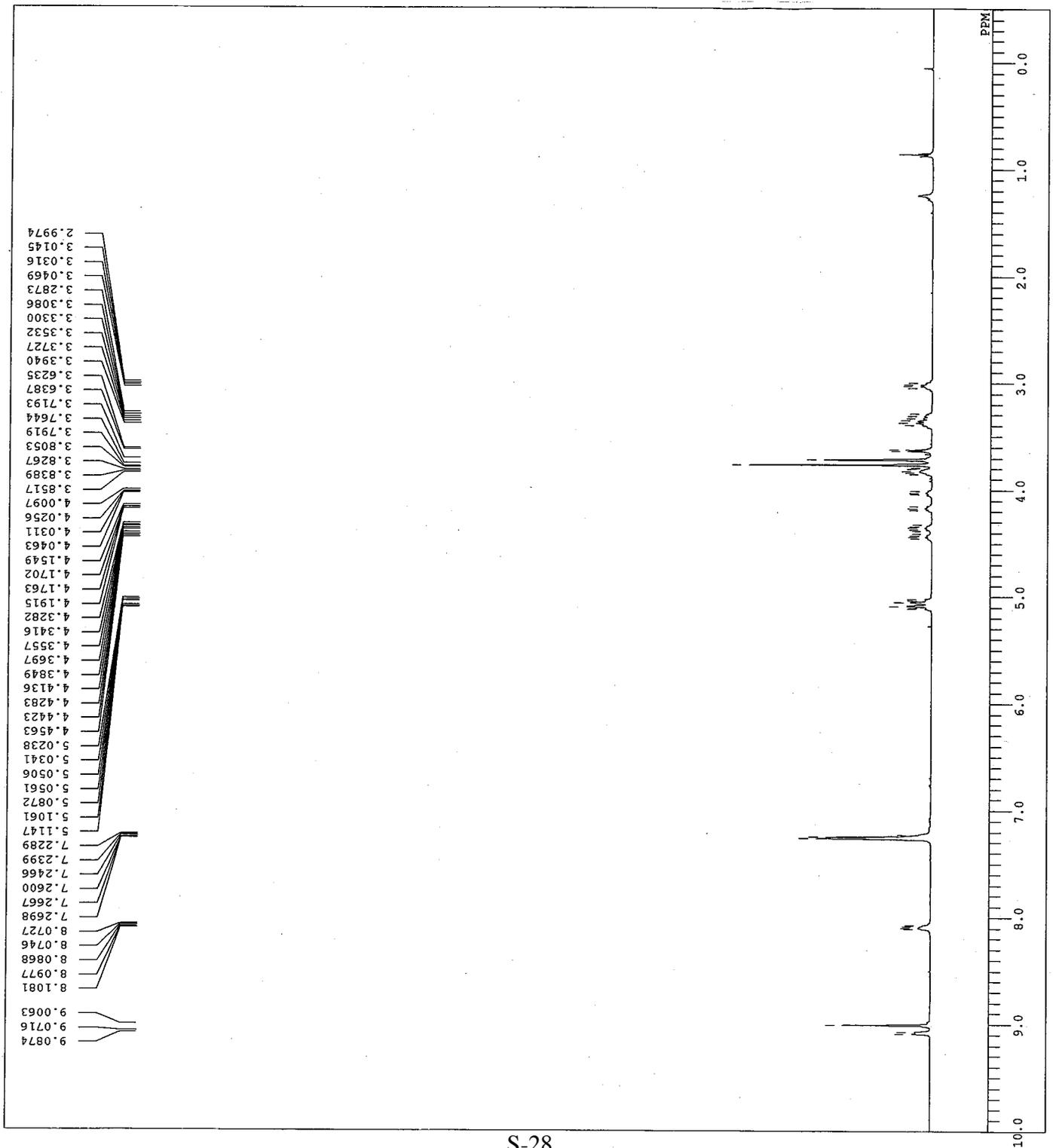
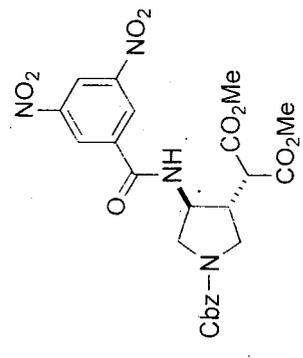
#	ピーク名	tR [min]	面積 [μV·sec]	面積%
1	Unknown	18.358	1507707	50.611
2	Unknown	41.067	1471324	49.389

測定日 2010/11/17 15:56:48  
コントロールメソッド 1ml\_254nm

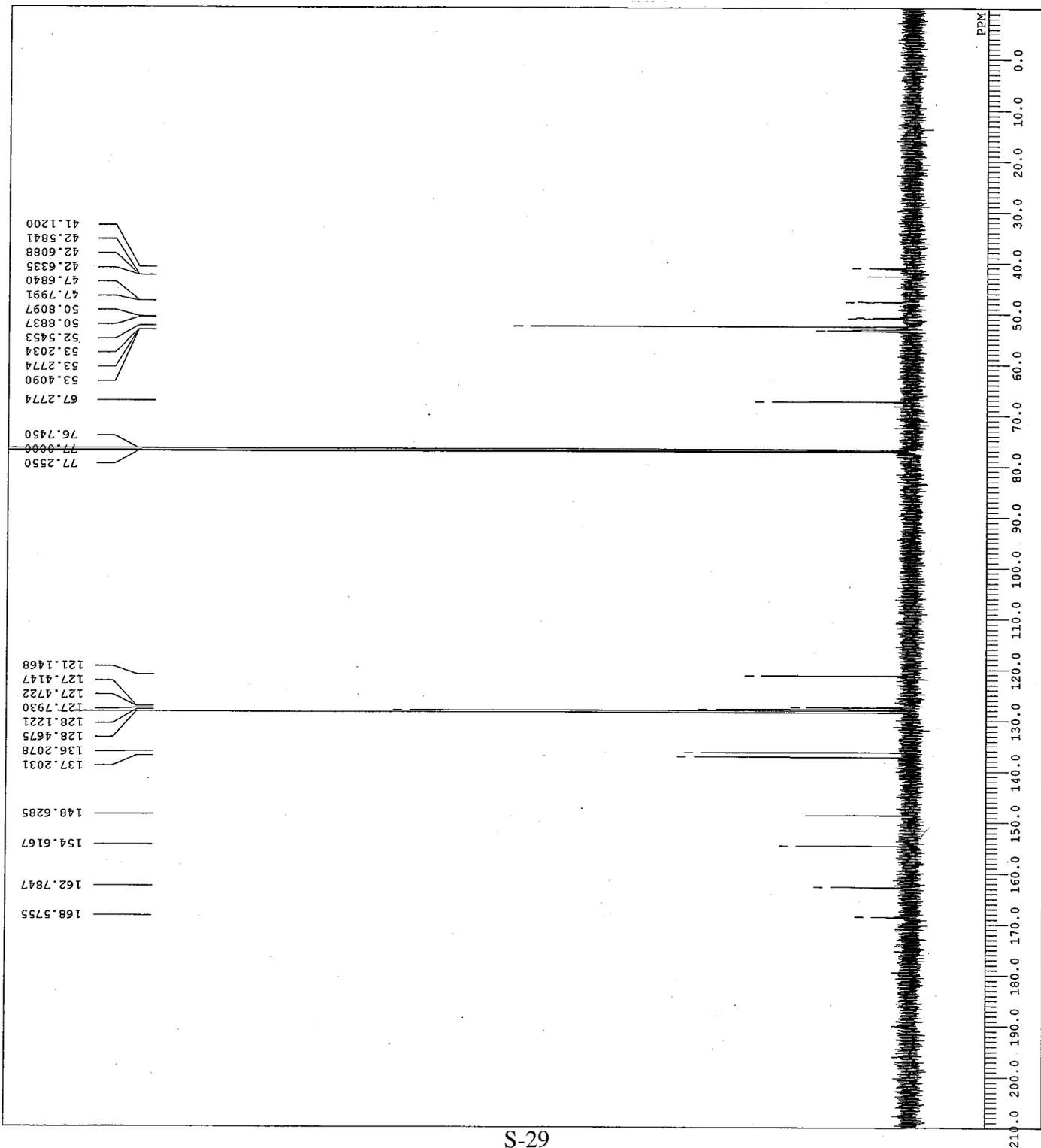
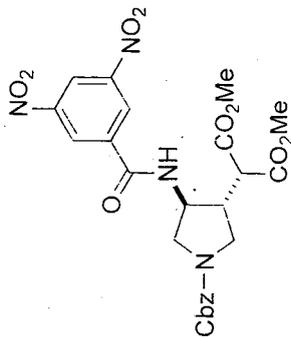
```

DFILE Xul134-Cbz-1H.als
COMNT 1134-Cbz-1H
DATIM  Fri Jan 21 22:49:41 2011
ORNUC  1H
EXMOD  non
OBFRQ  500.00 MHz
OBSEY  160.00 KHz
OBFIN  2160.00 Hz
POINT  32768
FREQU  10000.00 Hz
SCANS  6
ACQTM  3.2768 sec
PD      3.7232 sec
PWL     5.90 usec
IRNUC  1H
CTEMP  27.1 c
SLVNT  CDCL3
EXREF  7.26 ppm
BF      0.12 Hz
RGAIN  16

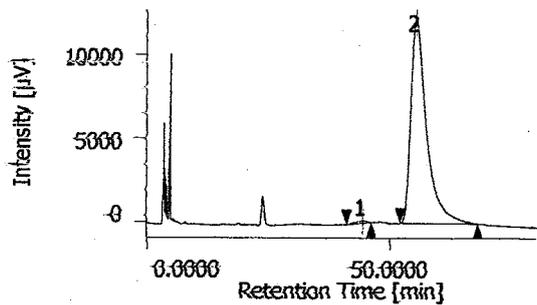
```



DFILE xul134-Cbz-13C-2.als  
 COMMT 1134-Cbz-13C-2  
 DATIM Sun Jan 23 00:23:52 2011  
 OBNUC 13C  
 EXMOD bcm  
 OBFRQ 125.65 MHz  
 OBSST 120.00 KHz  
 OBFIN 7956.00 Hz  
 POINT 32768  
 FREQU 33896.30 Hz  
 SCANS 759  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 PWL 5.12 usec  
 IH 29.7 C  
 CDCL3 77.00 ppm  
 EXREF 0.12 Hz  
 BF 30  
 RGAIN



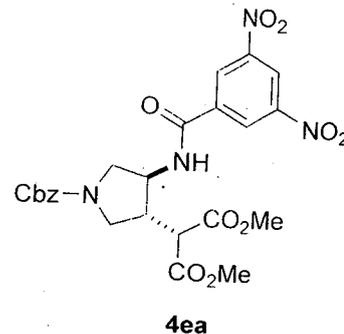
chiral 4ea



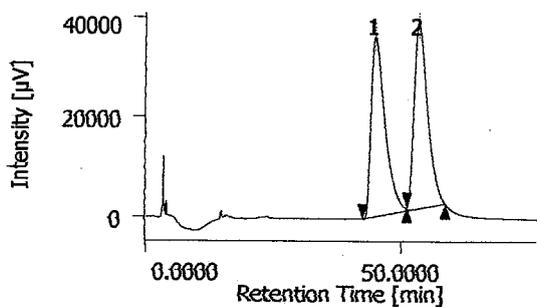
ピーク情報 area%

#	tR [min]	面積 [μV·sec]	高さ [μV]	面積%
1	44.067	24927	164	0.900
2	55.142	2746216	12050	99.100

98% ee



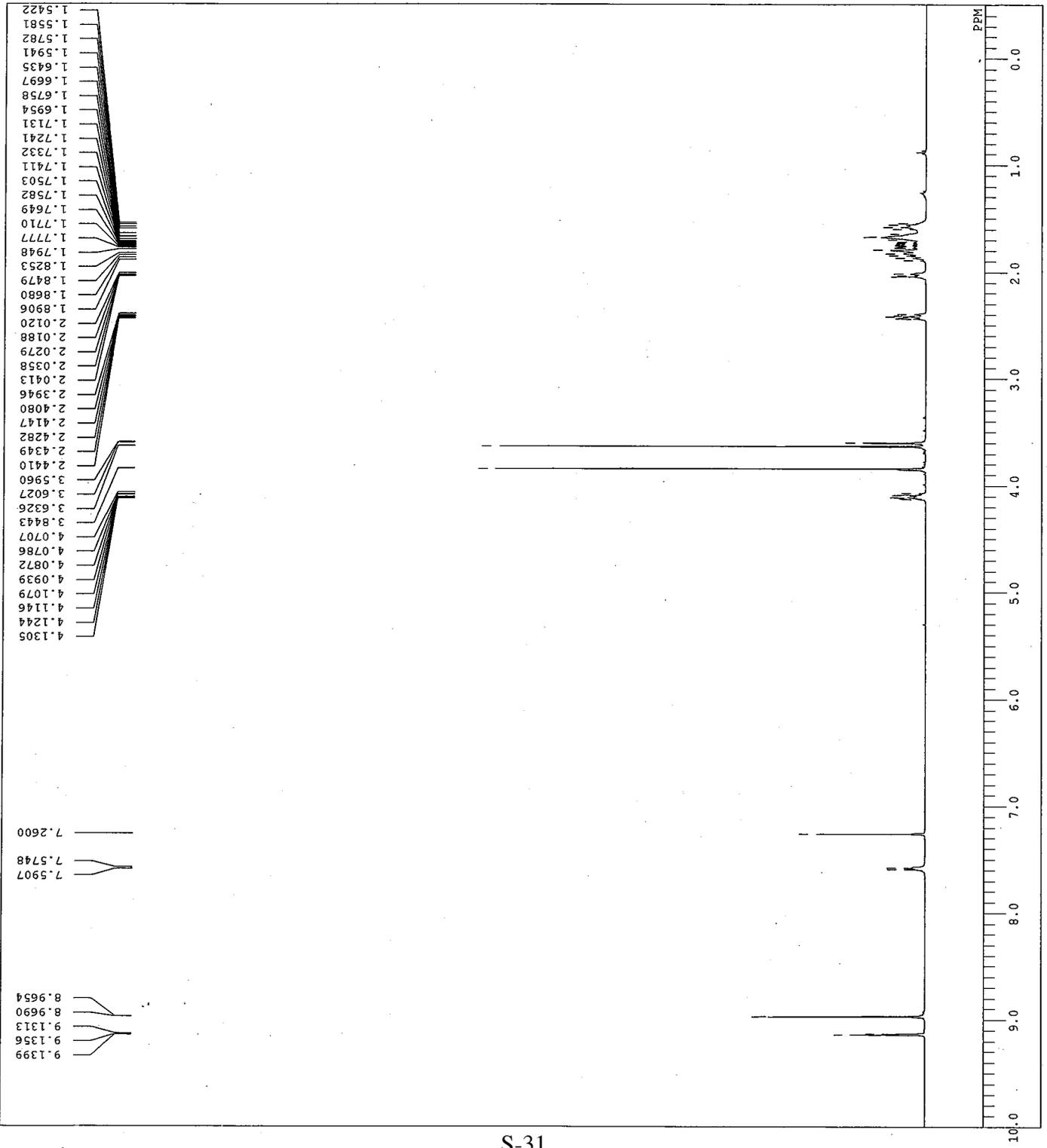
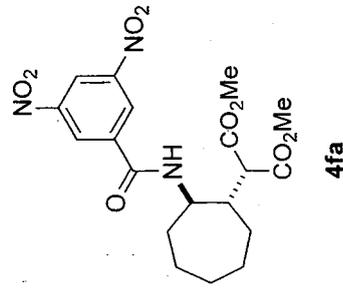
rac 4ea



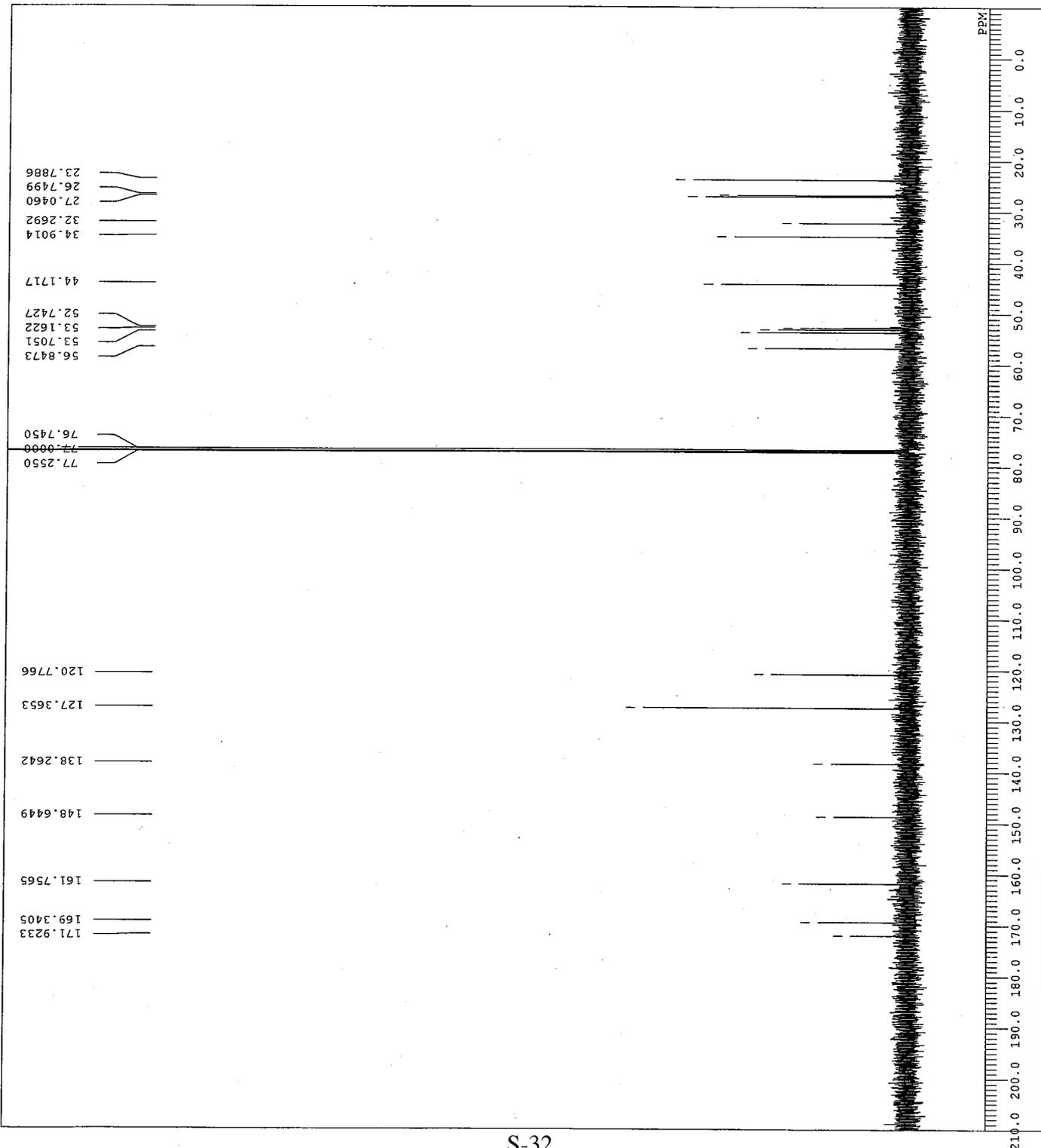
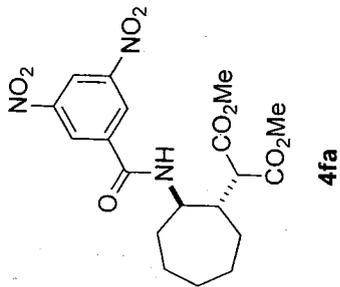
ピーク情報 area%

#	tR [min]	面積 [μV·sec]	高さ [μV]	面積%
1	44.567	6794144	35943	49.783
2	53.183	6853339	36922	50.217

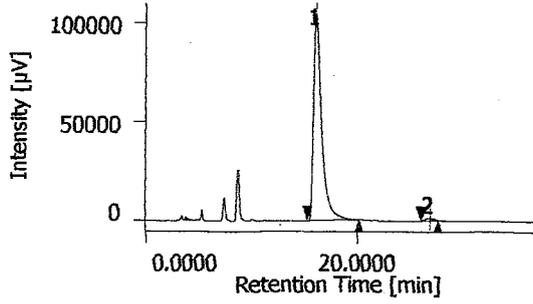
DFLE xul125-Hep-1H.als  
 CONNT 1125-Hep-1H  
 DATIM Mon Jan 24 22:00:16 2011  
 OBNUC 1H  
 EXMOD non  
 OBFRO 500.00 MHz  
 OBSET 160.00 KHz  
 OBFET 2160.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 SCANS 6  
 ACQTM 3.2768 sec  
 PD 3.7232 sec  
 FWL 5.90 usec  
 IRNUC 1H  
 CTEMP 26.8 c  
 SLVNT CDCL3  
 EXREF 7.26 ppm  
 BF 0.12 Hz  
 RGAIN 21



DFILF xu1125-Hep-13C.als  
 COMNT 1125-Hep-13C  
 DATIM Mon Jan 24 22:19:33 2011  
 OBNUC 13C  
 EXMOD bcm  
 OBFRO 125.65 MHz  
 ORSET 120.00 KHz  
 OBFIN 7958.00 Hz  
 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 373  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 PWT 5.112 usec  
 IRNUC 1H  
 CTEMP 29.1 c  
 SILVNT CDCL3  
 EXREF 77.00 ppm  
 BF 0.12 Hz  
 RGAIN 30



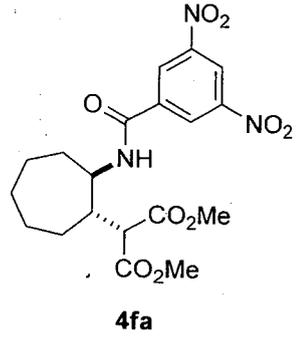
chiral 4fa



ピーク情報 area-f.

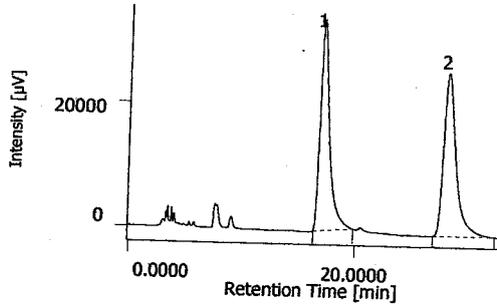
#	tR [min]	面積 [μV·sec]	高さ [μV]	面積%
1	15.950	5512966	104189	98.780
2	26.692	68101	1325	1.220

98/100



xu110121-Hep xu1119-Hep-R  
クロマト

rac 4fa

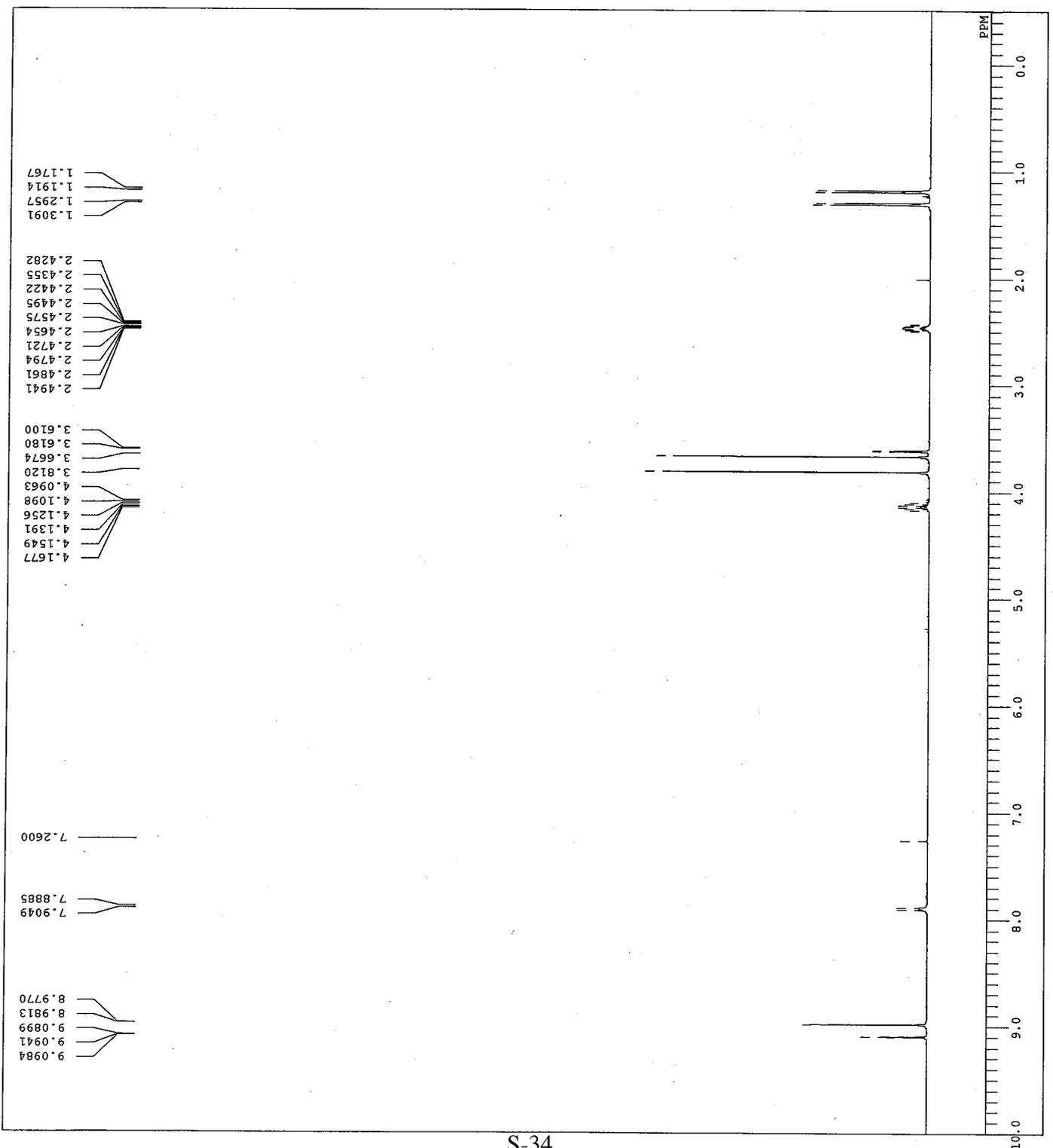
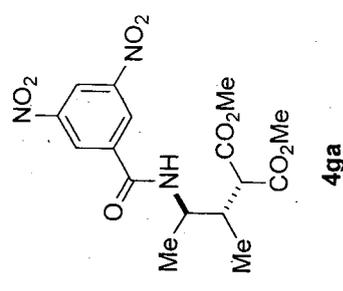


ピーク情報 area-f.

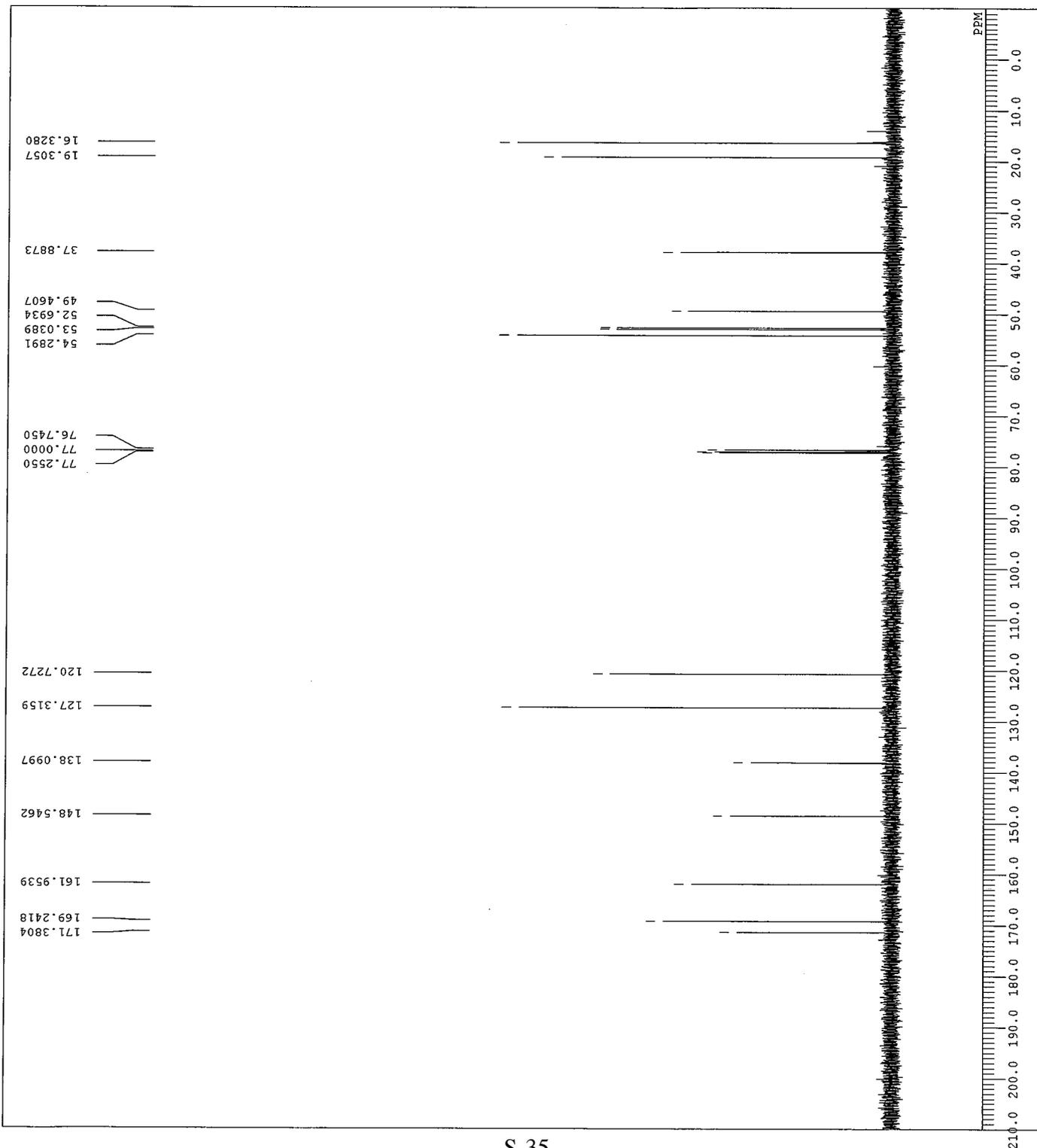
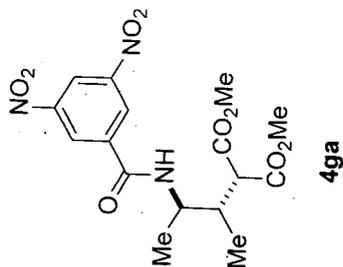
#	ピーク名	tR [min]	面積 [μV·sec]	面積%
1	Unknown	16.967	1958215	50.057
2	Unknown	27.967	1953792	49.943

測定日 2011/01/21 17:07:05  
コントロールメソッド 1ml\_254nm

DEFILE xu1110-Me-H1.als  
 COMMT 1110-Me-1H  
 DATIM Thu Jan 13 15:43:58 2011  
 OBNUC 1H  
 non  
 EXMOD 500.00 MHz  
 OBFRQ 160.00 KHz  
 OBSET 2160.00 Hz  
 OBFIN 32768  
 POINT 10000.00 Hz  
 FREQU 9  
 SCANS 3.2768 sec  
 ACOJM 3.7232 sec  
 PD 5.90 usec  
 FWI 1H  
 IRNUC 27.9 c  
 CTEMP 7.26 ppm  
 SILVNT CDCL3  
 EXREF 0.12 Hz  
 BF 13  
 RGAIN



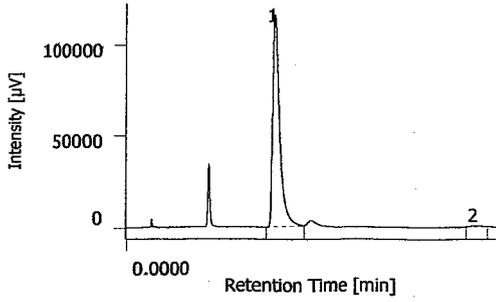
DFIL1 xul110-Me-Cl13.als  
 COMNT 1110-Me-Cl13  
 DATIM Sat Dec 18 12:10:06 2010  
 OBNUC 13C  
 EXMOD bcm  
 OBFREQ 125.65 MHz  
 OBSET 120.00 KHz  
 OBFIN 7958.00 Hz  
 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 46  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 FWI 5.12 usec  
 IRNUC 1H  
 CTEMP 27.9 c  
 SLVNT CDCL3  
 EXREF 77.00 ppm  
 BF 0.12 Hz  
 RGAIN 30



xu101217-3 xu1110  
クロマト

chiral 4ga

area%

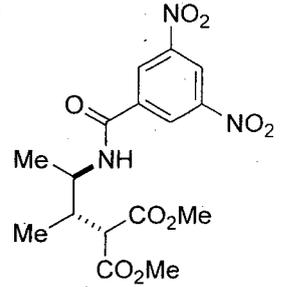


ピーク情報

#	ピーク名	tR [min]	面積 [μV·sec]	面積%
1	Unknown	18.533	6763640	99.321
2	Unknown	43.742	46253	0.679

99.00%

測定日 2010/12/17 17:35:40  
コントロールメソッド 1ml\_254nm

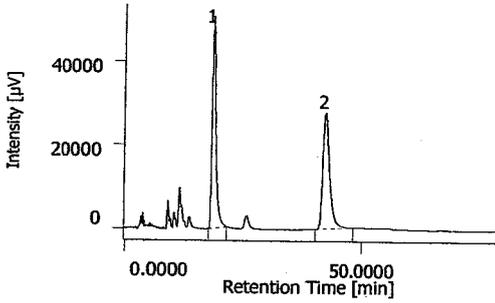


4ga

xu101217 xu1104-Me-R  
クロマト

rac 4ga

area%

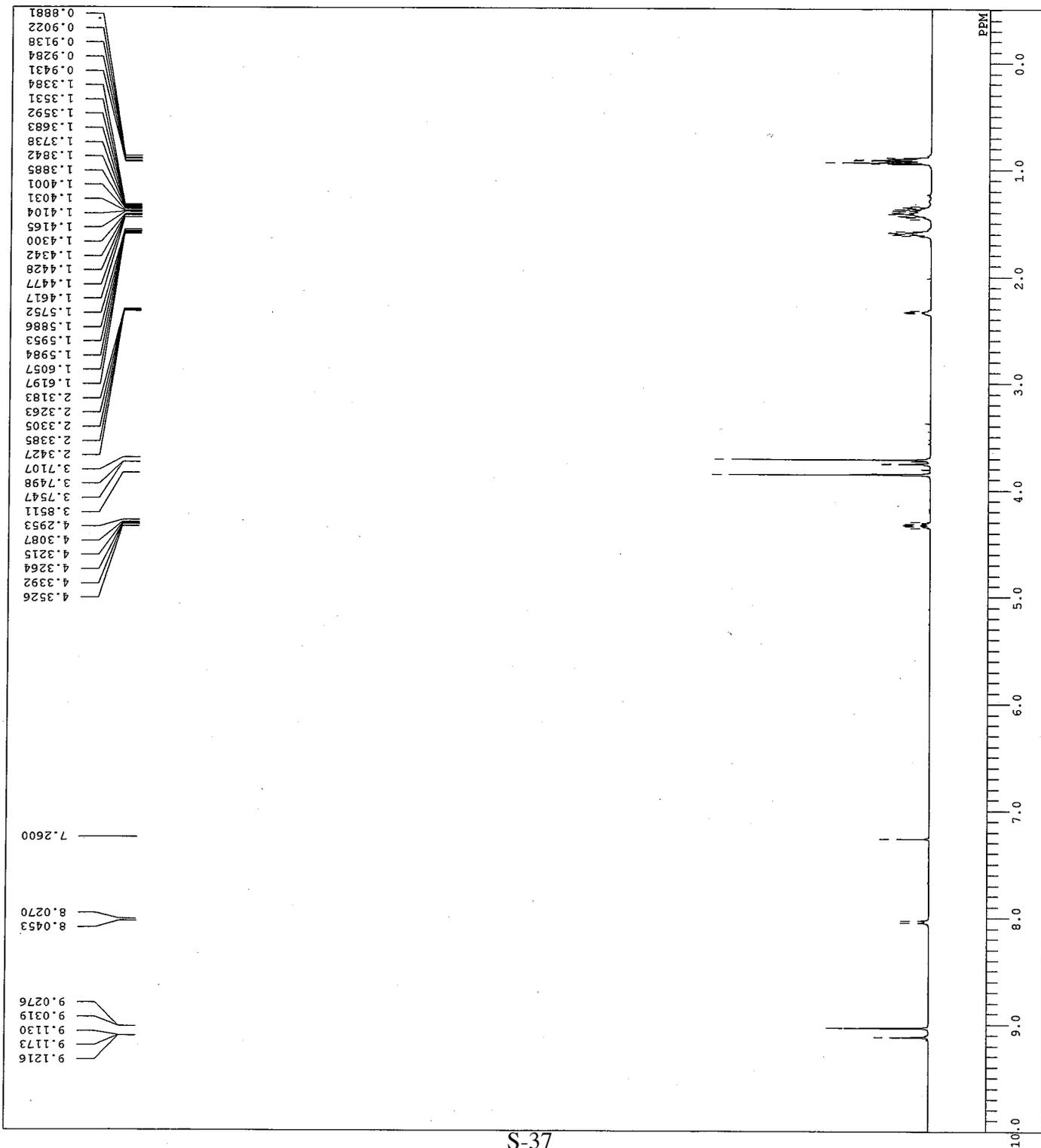
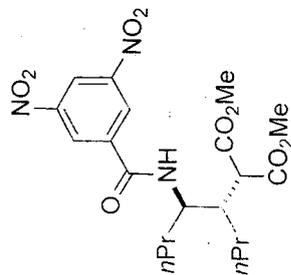


ピーク情報

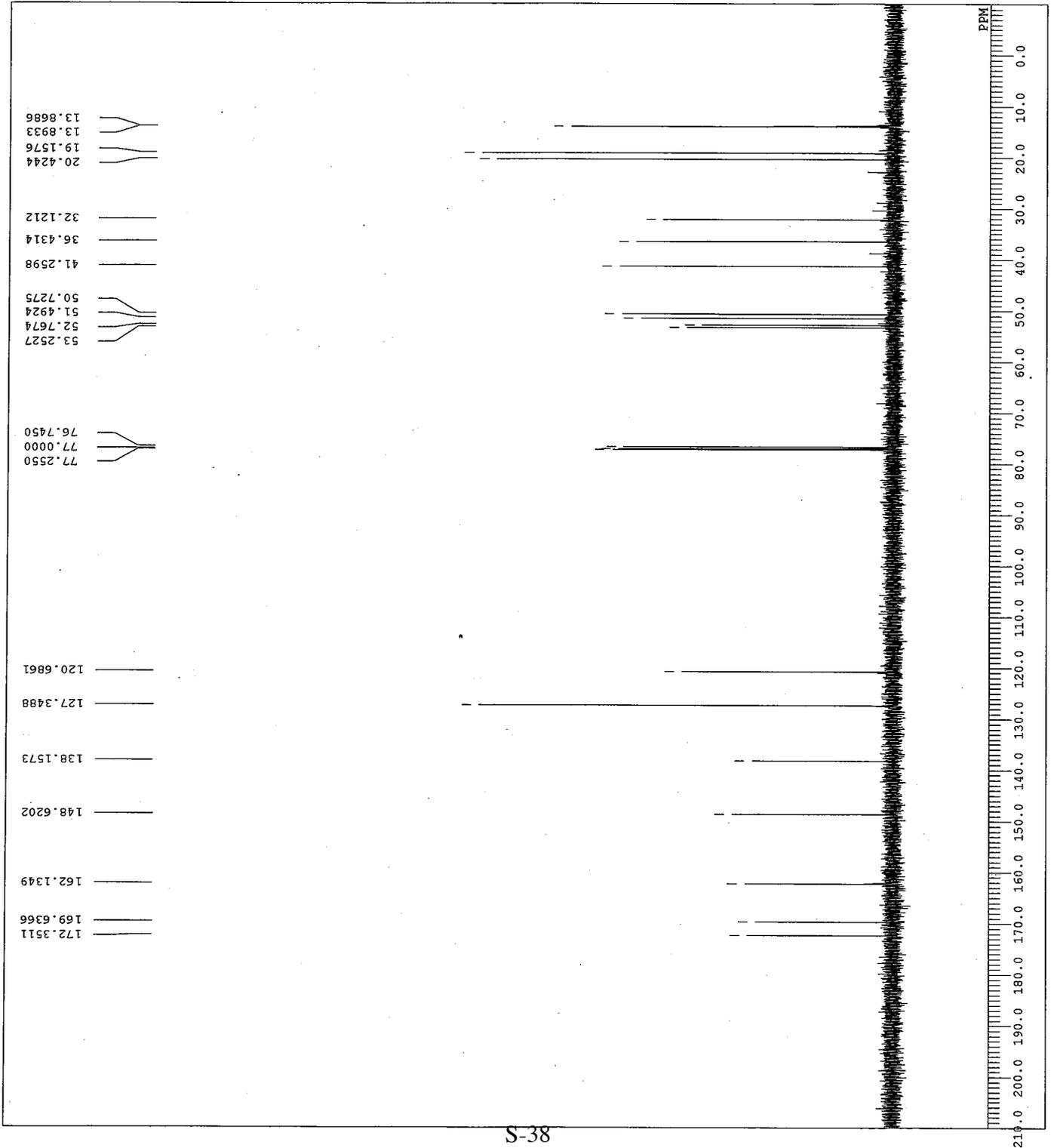
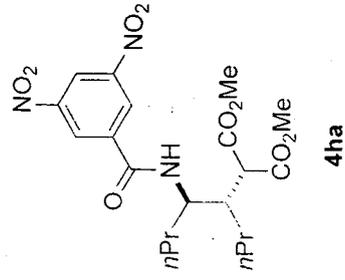
#	ピーク名	tR [min]	面積 [μV·sec]	面積%
1	Unknown	18.367	2852905	50.843
2	Unknown	41.850	2758333	49.157

測定日 2010/12/17 13:24:44  
コントロールメソッド 1ml\_254nm

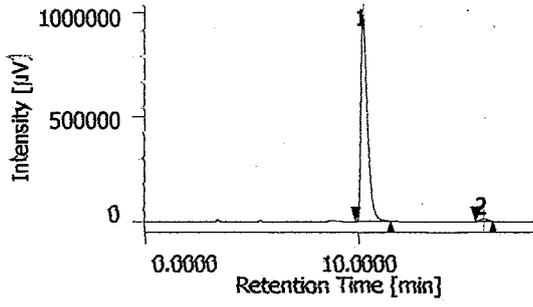
DFILE xul123-nPr-1H.als  
 COMNT 1123-nbu-1H  
 DATIM Tue Dec 28 17:13:07 2010  
 OBNUC 1H  
 EXMOD non  
 OBFRQ 500.00 MHz  
 OBSEF 160.00 KHz  
 OBFIN 2160.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 SCANS 5  
 ACQTM 3.2768 sec  
 PD 3.7232 sec  
 PW1 5.90 usec  
 1H 27.7 c  
 CDCL3 7.26 ppm  
 EXREF 1.00 Hz  
 BF 14  
 RGAIN



DFILE xul123-nBu-13C.als  
 COMNT 1123-Hep-13C  
 DATIM Tue Dec 28 17:18:46 2010  
 OBNUC 13C  
 EXMOD btm  
 OBFRO 125.65 MHz  
 OBSET 120.00 KHz  
 OBEIN 7958.00 Hz  
 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 80  
 ACQTM 0.9657 sec  
 PD 2.0533 sec  
 FWI 5.12 usec  
 IRNUC 1H  
 CTEMP 28.9 c  
 SLVNT CDCL3  
 EXREF 77.00 ppm  
 BF 1.00 Hz  
 RGAIN 31



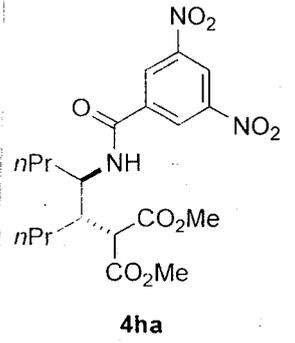
chiral 4ha



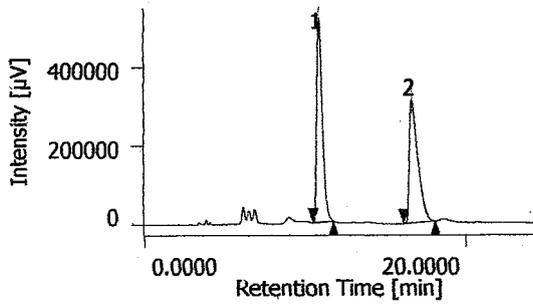
ピーク情報 area%

#	tR [min]	面積 [μV·sec]	高さ [μV]	面積%
1	10.150	21173729	983285	98.513
2	15.817	319575	13412	1.487

97.02%



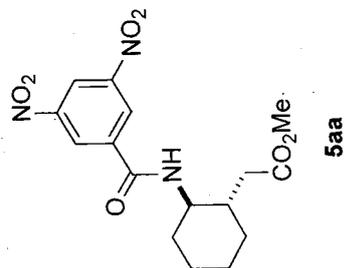
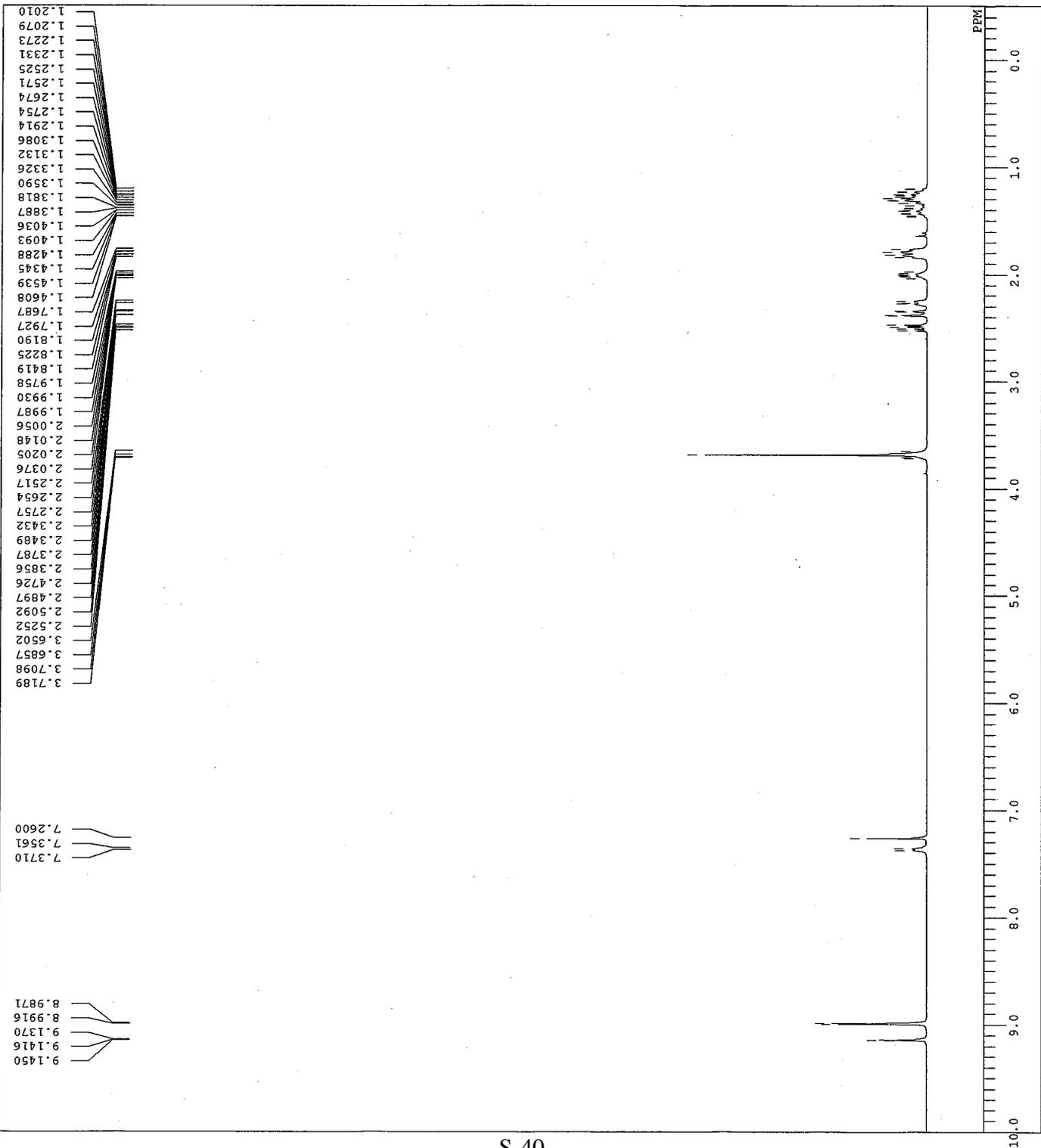
rac 4ha



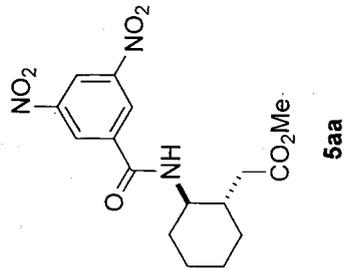
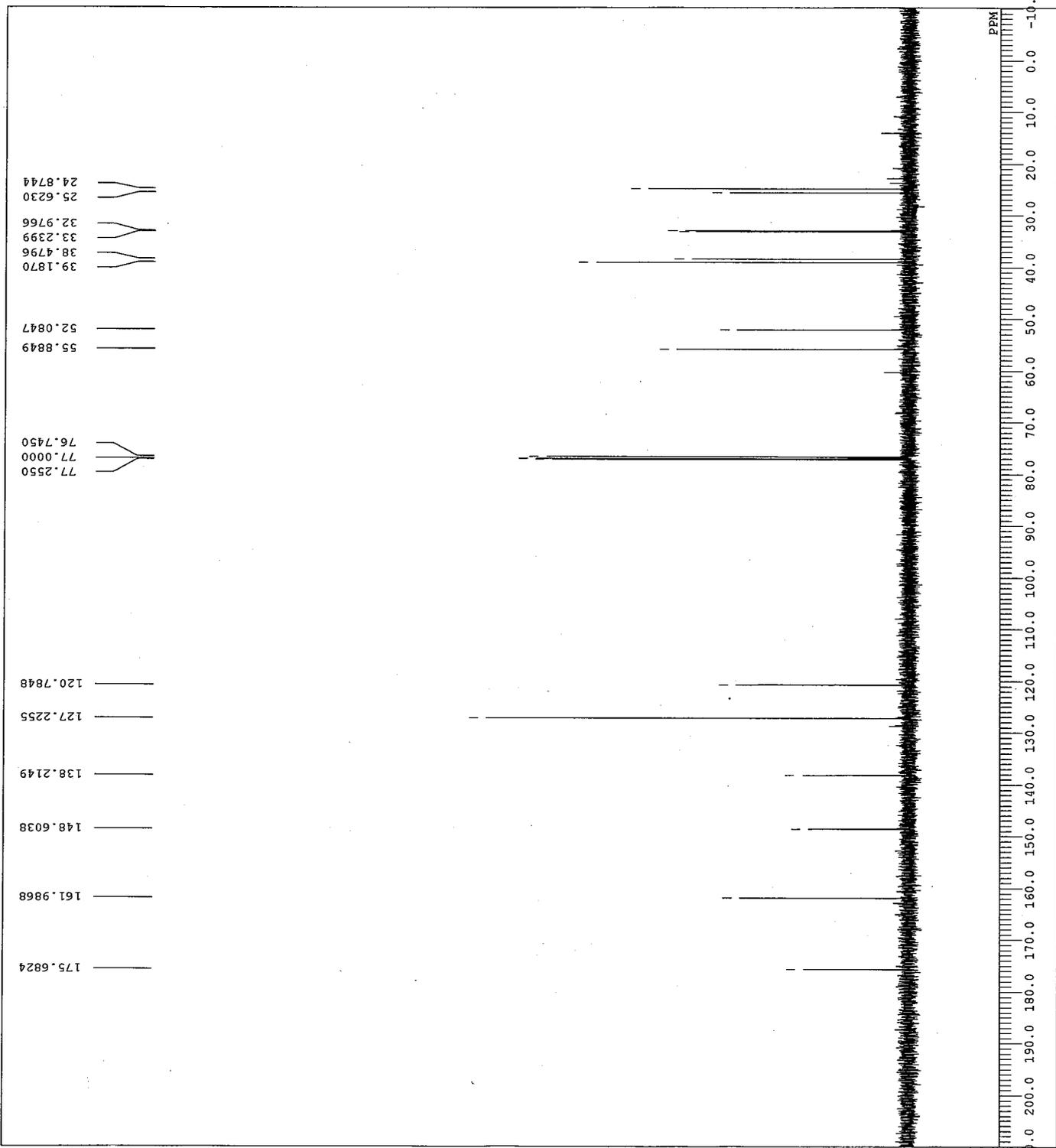
ピーク情報 area%

#	tR [min]	面積 [μV·sec]	高さ [μV]	面積%
1	10.742	11840777	516882	50.610
2	16.567	11555282	310661	49.390

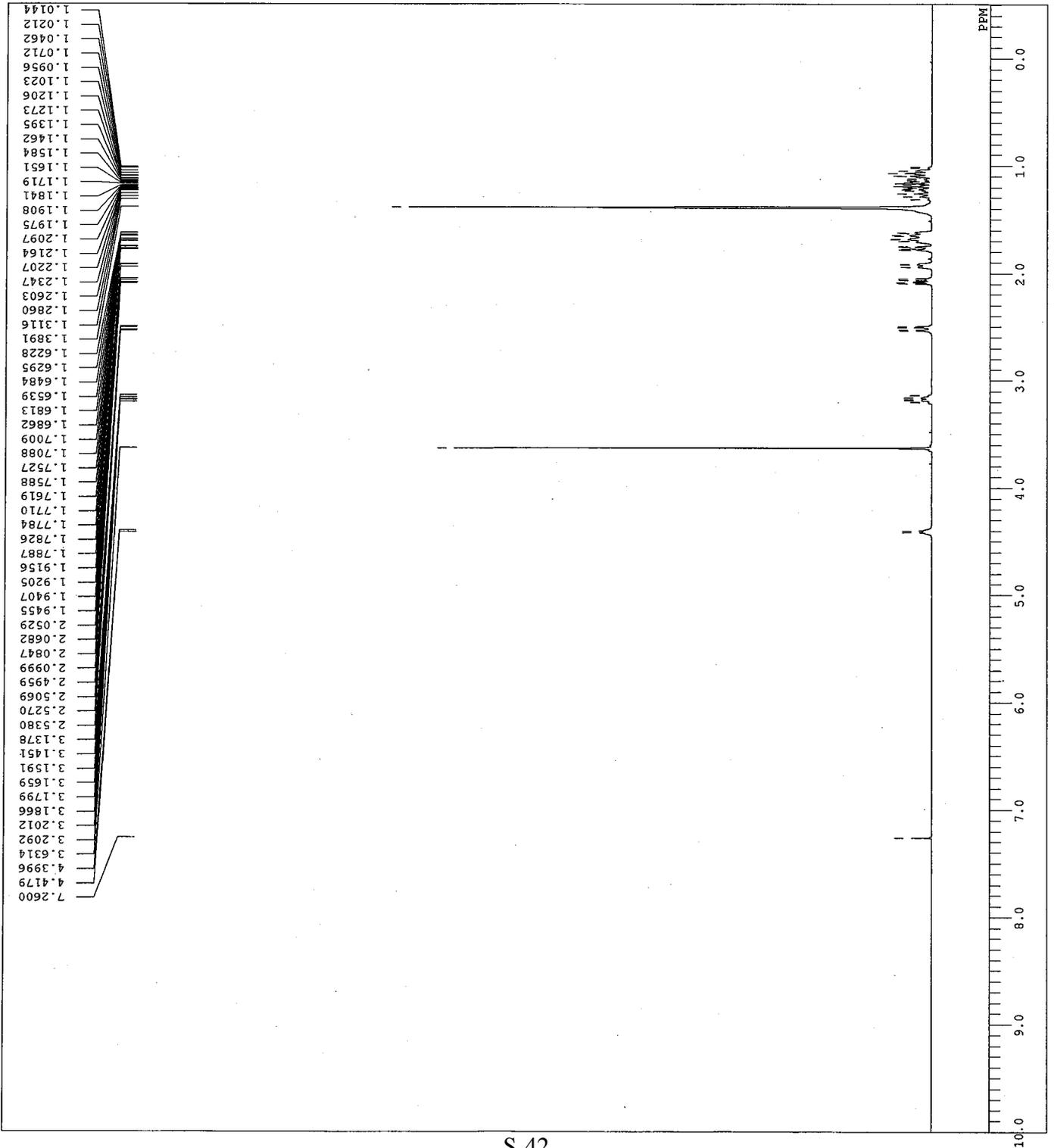
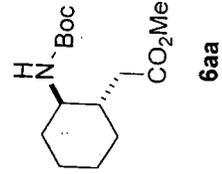
DFILE xul148-DeCarb-1H.als  
 COMMT 1148-DeCarb-1H  
 DATIM 26-01-2011 22:41:19  
 OBNUC 1H  
 single\_pulse.ex2  
 EXMOD 490.15 MHz  
 OBFRQ 9.16 KHz  
 OBSET 7.60 Hz  
 OBFIN 16384  
 POINT 9191.18 Hz  
 FREQU 15  
 SCANS 15  
 ACOPTM 1.7826 sec  
 PD 5.0000 sec  
 PWL 8.55 usec  
 IRNUC 1H  
 CTEMP 22.2 C  
 SLVNT CDCL3  
 EXREF 7.26 ppm  
 BF 1.00 Hz  
 RGAIN 40



DFILE xul148-DeCarb-13C.als  
 COMNT 1148-DeCarb-13C  
 DATIM Tue Jan 25 17:32:25 2011  
 OBNUC 13C  
 EXMOD bcm  
 OBFRO 125.65 MHz  
 OBSET 120.00 KHz  
 OBFIN 7956.00 Hz  
 POINT 32768  
 FREQU 33896.30 Hz  
 SCANS 159  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 PW1 5.12 usec  
 IRNUC 1H  
 CTEMP 28.9 c  
 SLVNT CDCL3  
 EXREF 77.00 ppm  
 BF 0.12 Hz  
 RGAIN 31



DFILE xul159-1H.a1s  
 COMNT 1159-1H  
 DATIM Thu Feb 3 15:31:56 2011  
 OBNUC 1H  
 EXMOD non  
 OBFREQ 500.00 MHz  
 OBSET 160.00 KHz  
 OBFIN 2150.00 Hz  
 POINT 32768  
 FREQU 10000.00 Hz  
 SCANS 8  
 ACQTM 3.2768 sec  
 PD 3.7232 sec  
 PWI 5.90 usec  
 IRRUC 1H  
 CTEMP 26.3 C  
 SLVNT CDCL3  
 EXREF 7.26 ppm  
 BF 1.00 Hz  
 RGAIN 12



DFILE xul159-13c.als  
 COMNT 1159-13C  
 DATIM Thu Feb 3 15:37:45 2011  
 OBNUC 13C  
 EXMOD bcm  
 OBFRQ 125.65 MHz  
 OBSET 120.00 KHz  
 OBFIN 7956.00 Hz  
 POINT 32768  
 FREQU 33898.30 Hz  
 SCANS 102  
 ACQTM 0.9667 sec  
 PD 2.0333 sec  
 PWI 5.12 usec  
 IIRNUC 1H  
 CTEMP 27.9 C  
 CDCL3  
 SLVNT  
 EXREF 77.00 ppm  
 BF 1.00 Hz  
 RGAIN 30

