Eur. J. Org. Chem. **2008** · © WILEY-VCH Verlag GmbH & Co. KGaA, 69451 Weinheim, 2008 · ISSN 1434–193X

SUPPORTING INFORMATION

Title: Chirality Transfer from Epoxide to Carbanion: Base-Induced Alkylation of *O*-Carbamoyl Cyanohydrins of β-Silyl-α,β-epoxy Aldehyde

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Ref. No.: O200800300

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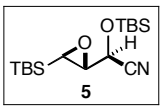
General. Melting points (uncorrected) were determined by using a Büchi Melting Point B-545. Optical rotations were measured on a Horiba SEPA-200 polarimetera or Jasco DIP-1000 digital polarimeter. Infrared spectra (IR) were recorded on a Perkin Elmer PARAGON 1000 spectrometer or Horiba FT-710 spectrometer. ^{1}H NMR spectra were taken on a JEOL Delta-500 (500 MHz) or a Varian Gemini 2000 (300 MHz) spectrometer in $CDCl_3$ unless otherwise indicated with reference to $CHCl_3$ (δ 7.26). ^{13}C NMR spectra were measured with a JEOL Delta-500 (125 MHz) or a JEOL JNM-LA400 (100 MHz) spectrometer in $CDCl_3$ unless otherwise indicated with reference to the $CDCl_3$ triplet (δ 77.2). Resonance patterns were described as s = singlet, d = doublet, t = triplet, q = quartet, sep = septet, m = multiplet, and br = broad. The assignment of ^{1}H and ^{13}C NMR spectra is based on H-H decoupling and HMQC experiments. Low- and high-resolution mass spectra (EI-MS, FAB-MS) were obtained with a JEOL JMS-SX102 spectrometer. APCI-LC/MS was obtained with a Finnigan MAT SSQ7000C spectrometer (eluent 10 mM $AcONH_4$/MeOH). Analytical high performance liquid chromatography (HPLC) was performed a Waters 2695 separation module. Semi-preparative HPLC was performed a Waters 600E system controller. Medium pressures liquid chromatography (MPLC) was carried out with a JASCO PU-980 pump system by using prepacked columns (Kusano Kagaku, silica gel 10 μm, 22 mm x 300 mm, or silica gel 5 μm, 22 mm x 150 mm). For routine chromatography, the following adsorbents were used: Fuji-Davison silica gel BW-200 (150-325 mesh) for column chromatography; Merck precoated silica gel 60 F-254 plates for analytical thin layer chromatography. All moisture sensitive reactions were performed under a positive pressure of nitrogen. Anhydrous $MgSO_4$ was used for drying all organic solvent extracts in workup, and the removal of the solvents was performed with a rotary evaporator. Dry solvents and reagents were obtained by using standard procedures.

(2*S*,3*R*,4*R*)-2-(*tert*-Butyldimethylsiloxy)-4-(*tert*-butyldimethylsilyl)-3,4-epoxybutyronitrile (5).

To a solution of (2*R*,3*R*)-3-(*tert*-butyldimethylsilyl)oxirane-2-carbaldehyde (7)[1] (5.00 g, 26.8 mmol) in CH_2Cl_2 (54 mL) was added zinc iodide (428 mg, 1.34 mmol) and *tert*-butyldimethylsilyl cyanide (4.93 g, 34.9 mmol). After stirring at room temperature for 17 h, the reaction mixture was concentrated. The residue was diluted with hexane-Et_2O (1:1, 100 mL) and filtrated through a pad of Celite with hexane-Et_2O (1:1, 100 mL). The filtrate was combined and concentrated. The residual oil was subjected to column chromatography (silica gel 800 g, elution with hexane:Et_2O = 19:1 to 9:1) to give 5 (3.32 g, 38%) and *epi*-5 (2.24 g, 25%).

5: colorless plates, mp 45-47 °C (hexane). R_f = 0.56 (hexane:Et_2O = 9:1). $[\alpha]^{25}_D$ +21.9 (*c* 0.210, $CHCl_3$). IR (KBr) 1253 cm^{-1}. ^{1}H NMR δ -0.04 and 0.05



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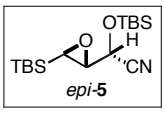
(each 3H, s, Si*Me*$_2$), 0.17 and 0.19 (each 3H, s, Si*Me*$_2$), 0.93 (9H, s, Si*Bu*^{t}), 0.97 (9H, s, Si*Bu*^{t}), 2.31 (1H, d, *J* = 3.4 Hz, H-4), 3.11 (1H, dd, *J* = 5.9, 3.4 Hz, H-3), 4.23 (1H, d, *J* = 5.9 Hz, H-2). ^{13}C NMR δ -8.6 and -7.9 (Si*Me*$_2$), -5.1 and -5.0 (OSi*Me*$_2$), 16.7 (Si*C*Me$_3$), 18.3 (OSi*C*Me$_3$), 25.7 (SiC*Me*$_3$), 26.6 (OSiC*Me*$_3$), 47.1 (C-4), 56.7 (C-3), 66.2 (C-2), 117.2 (C-1). APCI-LC/MS *m*/*z* 345 ($M^+ + NH_4$). Anal Calcd for $C_{16}H_{33}NO_2Si_2$: C, 58.66; H, 10.15; N, 4.28. Found: C, 58.37; H, 10.13; N, 4.37.

***epi*-5**: colorless plates, mp 41-43 °C (hexane). R_f = 0.47 (hexane:Et_2O = 9:1). $[\alpha]^{25}_D$ +5.82 (*c* 0.206, $CHCl_3$). IR (KBr) 1257 cm^{-1}. ^{1}H NMR δ -0.03 and



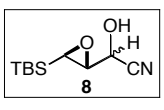
epi-**5**

0.03 (each 3H, s, Si*Me*$_2$), 0.14 and 0.19 (each 3H, s, Si*Me*$_2$), 0.91 (9H, s, Si*Bu*^{t}), 0.97 (9H, s, Si*Bu*^{t}), 2.37 (1H, d, *J* = 3.2 Hz, H-4), 3.09 (1H, dd, *J* = 4.4, 3.2 Hz, H-3), 4.39 (1H, d, *J* = 4.4 Hz, H-2). ^{13}C NMR δ -8.4 and -8.1 (Si*Me*$_2$), -5.1 and -5.1 (OSi*Me*$_2$), 16.8 (*C*Me$_3$), 18.3 (OSi*C*Me$_3$), 25.6 (SiC*Me*$_3$), 26.6 (OSiC*Me*$_3$), 48.0 (C-4), 55.9 (C-3), 64.1 (C-2), 117.7 (C-1). APCI-LC/MS *m*/*z* 345 ($M^+ + NH_4$). Anal Calcd for $C_{16}H_{33}NO_2Si_2$: C, 58.66; H, 10.15; N, 4.28. Found: C, 58.35; H, 10.29; N, 4.19.

(2*S*,3*R*,4*R*)-4-(*tert*-Butyldimethylsilyl)-4-chloro-2-(dimethylcarbamoyloxy)-3-hydroxybutyronitrile (10a)
and (2*R*,3*R*,4*R*)-4-(*tert*-butyldimethylsilyl)-4-chloro-2-(dimethylcarbamoyloxy)-3-hydroxybutyronitrile (10b).

To a cooled (0 °C) solution of **7** (39.6 g, 213 mmol) in CH_2Cl_2 was added trimethylsilyl cyanide (34.0 mL, 255 mmol), tetrabutylphosphonium bromide (14.5 g, 42.7 mmol) and potassium cyanide (2.78 g, 42.7 mmol). After the cooling bath was removed, the reaction mixture was stirred for 3 h at room temperature, and then fluorosilicic acid solution (33%, 95.0 mL) was added. The mixture was stirred for 17 h at room temperature, and the bulk of CH_2Cl_2 was removed under reduced pressure. The residue was diluted with water (1000 mL), and extracted with Et_2O (700 mL x 2). The combined organic phases were washed with water (500 mL) and saturated brine (500 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 1000 g, elution with hexane:Et_2O = 2:1 to 1:1) to give 4-(*tert*-butyldimethylsilyl)-2-hydroxy-3,4-epoxybutyronitrile (**8**) (42.6 g, 94%) as an inseparable 1:1 mixture of two diastereomers.

8: a colorless viscous oil. R_f = 0.26 (hexane:Et_2O = 1:1). IR (neat) 3427, 1252, 1067 cm^{-1}. ^{1}H NMR δ -0.01, 0.05, and 0.05 (s, Si*Me*$_2$), 0.97 and 0.98

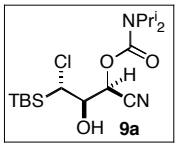


8

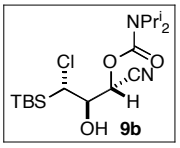
(each 9H, s, Si*Bu*^{t}), 2.44 and 2.58 (each 1H, d, *J* = 3.5 Hz, H-4), 2.71 (1H, br, O*H*), 3.21 and 3.22 (each 1H, d, *J* = 3.5, 3.3 Hz, H-3), 4.52 and 4.76 (each 1H, d, *J* = 3.3 Hz, H-2). APCI-LC/MS *m*/*z* 236 ($M^+ + NH_4$ + MeOH - HCN), 186 (M^+ - HCN).

(1) Le Bideau, F.; Gilloir, F.; Nilsson, Y.; Aubert, C.; Malacria, M. *Tetrahedron* **1996**, *52*, 7487-7510.

A solution of **8** (1.01 g, 4.73 mmol) and diisopropylcarbamoyl chloride (775 mg, 4.74 mmol) in pyridine (0.900 mL, 11.1 mmol) was heated at 90 °C for 8 h. The reaction mixture was diluted with water (40 mL), and then extracted with AcOEt (40 mL x 2). The combined organic phases were washed with 10% aqueous $CuSO_4$ solution (40 mL), and saturated brine (40 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 100 g, elution with hexane:Et_2O = 4:1) to give **9a** (639 mg, 36%) and **9b** (551 mg, 31%).

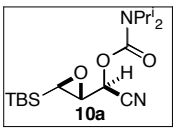


9a: a colorless viscous oil. R_f = 0.45 (hexane:Et_2O = 1:1). $[\alpha]^{25}_D$ +18.5 (*c* 0.216, $CHCl_3$). IR (neat) 3442, 1682, 1440, 1309, 1062 cm^{-1}. ^1H NMR δ 0.19 (6H, s, Si*Me*$_2$), 0.98 (9H, s, Si*Bu*^{t}), 1.26 (12H, d, *J* = 7.0 Hz, NCH*Me*$_2$), 3.20 (1H, d, *J* = 7.7 Hz, O*H*), 3.37 (1H, d, *J* = 9.5 Hz, H-4), 3.86 and 4.02 (each 1H, br, NC*H*Me$_2$), 4.26 (1H, ddd, *J* = 9.5, 7.7, 2.9 Hz, H-3), 5.76 (1H, d, *J* = 2.9 Hz, H-2). ^{13}C NMR δ -6.0 and -5.2 (Si*Me*$_2$), 17.7 (Si*C*Me$_3$), 20.6 and 21.6 (NCH*Me*$_2$), 27.4 (SiC*Me*$_3$), 45.8 (C-4), 46.6 and 47.6 (N*C*HMe$_2$), 64.2 (C-2), 74.8 (C-3), 116.5 (C-1), 153.4 (O*C*ONPr$^i_2$). APCI-LC/MS *m/z* 394/396 ($M^+ + NH_4$). Anal Calcd for $C_{17}H_{33}ClN_2O_3Si$ C, 54.16; H, 8.82; Cl, 9.40; N, 7.43. C, 54.45; H, 8.85; Cl, 9.28; N, 7.27.



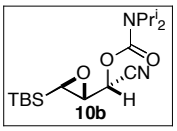
9b: colorless needles, mp 94-95 °C (hexane-AcOEt). R_f = 0.51 (hexane:Et_2O = 1:1). $[\alpha]^{25}_D$ -0.92 (*c* 0.216, $CHCl_3$). IR (KBr) 3495, 1713, 1435, 1331, 1071 cm^{-1}. ^1H NMR δ 0.20 and 0.20 (each 3H, s, Si*Me*$_2$), 1.00 (9H, s, Si*Bu*^{t}), 1.26 (12H, d, *J* = 7.0 Hz, NCH*Me*$_2$), 2.87 (1H, d, *J* = 5.5 Hz, O*H*), 3.43 (1H, d, *J* = 9.5 Hz, H-4), 3.87 and 3.99 (each 1H, br, NC*H*Me$_2$), 4.19 (1H, ddd, *J* = 9.5, 5.5, 3.0 Hz, H-3), 5.89 (1H, d, *J* = 3.0 Hz, H-2). ^{13}C NMR δ -6.3 and –5.5 (Si*Me*$_2$), 17.6 (Si*C*Me$_3$), 20.3 and 21.5 (NCH*Me*$_2$), 27.3 (SiC*Me*$_3$), 46.2 (C-4), 46.2 and 47.4 (N*C*HMe$_2$), 66.3 (C-2), 74.4 (C-3), 114.9 (C-1), 152.7 (O*C*ONPr$^i_2$). APCI-LC/MS *m/z* 394/396 (M+NH_4). Anal Calcd for $C_{17}H_{33}ClN_2O_3Si$: C, 54.16; H, 8.82; Cl, 9.40; N, 7.43. Found: C, 54.27; H, 8.86; Cl, 9.33; N, 7.40.

(2*S*,3*R*,4*R*)-4-(*tert*-Butyldimethylsilyl)-2-(diisopropylcarbamoyloxy)-3,4-epoxybutyronitrile (10a)



To a cooled (0°C) solution of **9a** (896 mg, 2.38 mmol) in CH_2Cl_2 (24 mL) was added DBU (1.07 mL, 7.15 mmol). After stirring at the same temperature for 1.5 h, the reaction mixture was poured into water (100 mL), and extracted with Et_2O (100 mL x 2). The combined organic phases were washed with saturated brine (100 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 100 g, elution with hexane:Et_2O = 6:1) to give **10a** (424 mg, 51%). colorless plates, mp 49-50 °C (hexane). R_f = 0.38 (hexane:Et_2O = 4:1). $[\alpha]^{25}_D$ +23.8 (*c* 0.244, $CHCl_3$). IR (neat) 1709, 1291, 1068 cm^{-1}. ^1H NMR δ -0.01 and 0.06 (each 3H, s, $SiMe_2$), 0.98 (9H, s, Si*Bu*^{t}), 1.24 (12H, d, *J* = 7.0 Hz, NCH*Me*$_2$), 2.42 (1H, d, *J* = 3.5 Hz, H-4), 3.25 (1H, dd, *J* = 5.7, 3.5 Hz, H-3), 3.84 and 4.01 (each 1H, br, NC*H*Me$_2$), 5.36 (1H, d, *J* = 5.7 Hz, H-2). ^{13}C NMR δ -8.6 and -8.2 (Si*Me*$_2$), 16.6 (Si*C*Me$_3$), 20.4 and 21.4 (NCH*Me*$_2$), 26.4 (SiC*Me*$_3$), 46.2 and 47.4 (N*C*HMe$_2$), 47.4 (C-4), 53.3 (C-3), 64.4 (C-2), 114.8 (C-1), 152.6 (O*C*ONMe$_2$). APCI-LC/MS *m/z* 341 (M^+ + H). Anal Calcd for $C_{17}H_{32}N_2O_3Si$: C, 59.96; H, 9.47; N, 8.23. Found: C, 59.72; H, 9.37; N, 8.10.

(2*R*,3*R*,4*R*)-4-(*tert*-Butyldimethylsilyl)-2-(diisopropylcarbamoyloxy)-3,4-epoxybutyronitrile (10b)



To a cooled (0°C) solution of **9b** (683 mg, 1.81 mmol) in CH_2Cl_2 (18 mL) was added DBU (0.815 mL, 5.45 mmol). After stirring at the same temperature for 1 h, the reaction mixture was poured into water (100 mL), and the mixture was extracted with Et_2O (100 mL x 2). The combined organic phases were washed with saturated brine (100 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 100 g, elution with hexane:Et_2O = 6:1) to give **10b** (337 mg, 55%). colorless prisms, mp 69-70 °C (hexane). R_f = 0.38 (hexane:Et_2O = 4:1). $[\alpha]^{25}_D$ +37.2 (*c* 0.274, $CHCl_3$). IR (KBr) 1719, 1320, 1161, 1143 cm^{-1}. ^1H NMR δ -0.03 and 0.05 (each 3H, s, Si*Me*$_2$), 0.96 (9H, s, Si*Bu*^{t}), 1.25 (12H, d, *J* = 7.0 Hz, NCH*Me*$_2$), 2.48 (1H, d, *J* = 3.3 Hz, H-4), 3.18 (1H, dd, *J* = 4.2, 3.3 Hz, H-3), 3.87 and 3.99 (each 1H, br, NC*H*Me$_2$), 5.57 (1H, d, *J* = 4.2 Hz, H-2). ^{13}C NMR δ -8.4 and -8.1 (Si*Me*$_2$), 16.8 (Si*C*Me$_3$), 20.5 and 21.5 (NCH*Me*$_2$), 26.6 (SiC*Me*$_3$), 46.4 and 47.5 (N*C*HMe$_2$), 48.5 (C-4), 53.6 (C-3), 64.2 (C-2), 115.0 (C-1), 152.7 (O*C*ONMe$_2$). APCI-LC/MS *m/z* 358 (M+NH_4). Anal Calcd for $C_{17}H_{32}N_2O_3Si$: C, 59.96; H, 9.47; N, 8.23. Found: C, 59.97; H, 9.32; N, 8.47.

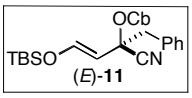
(*E*)-2-Benzyl-4-(*tert*-butyldimethylsilyloxy)-2-(diisopropylcarbamoyloxy)-3-butenonitrile ((*E*)-11)

and (*Z*)-2-Benzyl-4-(*tert*-butyldimethylsilyloxy)-2-(diisopropylcarbamoyloxy)-3-butenonitrile ((*Z*)-11)

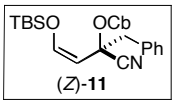
To a cooled (-80 °C) solution of **10a** (100 mg, 0.294 mmol) and benzyl bromide (0.175 mL, 1.47 mmol) in Et_2O (2.4 mL) was added dropwise a solution of lithium diisopropylamide (0.4 M in Et_2O, 0.808 mL, 0.323 mmol) over 2 min. After being stirred at the same temperature for 1 min, the reaction mixture was quenched with saturated ammonium chloride solution (5 mL), and then extracted with Et_2O (10 mL x 3). The combined organic phases were washed with water (10 mL) and saturated brine (10 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica gel 15 g, elution with hexane:Et_2O = 5:1) to give (*Z*)-**11** (13.4 mg). chiral HPLC: Daicel CHIRALPAK® AD-H (4.6 mm x 250 mm), hexane:Pr^{i}OH = 97:3, flow rate 0.70 mL/min, $t_{R(S\text{-isomer})}$ = 7.0 min, $t_{R(R\text{-isomer})}$ = 10.5 min (major, 39% ee). The other two fractions in the chromatography consisted of the mixture of (*E*,Z)-**11** and (*E*,Z)-**12** in different ratios: (*E*)-**11**:(*Z*)-**11**:(*E*)-**12**:(*Z*)-**12** = 0.13:0.07:0.07:1.0 (43.5 mg) and (*E*)-**11**:(*Z*)-**11**:(*E*)-**12**:(*Z*)-**12** = 0.11:1.0:0.14:1.12 (23.4 mg). Chromatographic separation should be finished as fast as possible (within a few minutes), otherwise a facile 3,3-sigmatropic shift of the carbamoyl group of (*E*,Z)-**11** occurs on the silica gel, making the isolation of (*E*)-**11** difficult.

To a cooled (0 °C) solution of a mixture of (*E*)-**11**, (*Z*)-**11**, (*E*)-**12** and (*Z*)-**12** (43.8 mg) in THF (1.5 mL) was added lithium aluminum hydride (25 mg). After stirring for 10 min, cooling bath was removed. The mixture was stirred for 1 h at room temperature, and then sat. Na_2SO_4 (a few drops) was added carefully. The mixture was filtered through a pad of Celite with Et_2O (20 mL). The filtrates were combined and concentrated. The residual solid was subjected to column chromatography (silica gel 1 g, hexane:AcOEt = 4:1) to give (*E*)-**13** (5.5 mg) chiral HPLC: Daicel CHIRALPAK® AD-H (4.6 mm x 250 mm), hexane:Pr^{i}OH = 97:3, flow rate 0.50 mL/min, $t_{R(S\text{-isomer})}$ = 16.6 min, $t_{R(R\text{-isomer})}$ = 18.1 min (major, 40% ee).

Since the purification of (*E*)- and (*Z*)-**12** from the mixture was found difficult, they were prepared by quenching with AcOH from **10a** and **10b**, respectively.

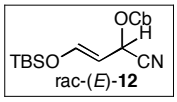


(*E*)-**11**: a colorless clear oil. R_f = 0.40 (hexane:Et_2O = 4:1) IR (neat) 1709, 1661, 1319, 1206 cm^{-1}. ^{1}H NMR δ 0.13 and 0.13 (each 3H, s, Si*Me*$_2$), 0.88 (9H, s, Si*Bu*^{t}), 1.05-1.30 (6H, m, NCH*Me*$_2$), 3.37 and 3.44 (each 1H, d, *J* = 13.5 Hz, C*H*$_2$Ph), 3.83 (2H, m, NC*H*Me$_2$), 5.15 (1H, d, *J* = 12.1 Hz, H-3), 6.84 (1H, d, *J* = 12.1 Hz, H-4), 7.26-7.32 (5H, m, CH$_2$*Ph*). ^{13}C NMR δ -5.3 and -5.3 (Si*M*e$_2$), 18.3 (Si*C*Me$_3$), 20.5 and 21.2 (NCH*Me*$_2$), 25.5 (SiC*Me*$_3$), 45.9 (*C*H$_2$Ph), 45.9 and 46.5 (N*C*HMe$_2$), 73.6 (C-2), 108.1 (C-3), 118.0 (C-1), 127.5, 128.2, 131.1, and 133.4 (CH$_2$*Ph*), 147.6 (C-4), 152.4 (N*C*OPr$^i_2$). APCI-LC/MS *m*/*z* 448 (M$^+$ + NH$_4$). Anal Calcd for $C_{24}H_{38}N_2O_3Si$: C, 66.94; H, 8.89; N, 6.50. Found: C, 66.85; H, 9.04; N, 6.55.



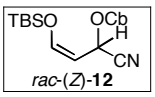
(*Z*)-**11**: colorless prisms, mp 99-100 °C (hexane). R_f = 0.28 (hexane:Et_2O = 4:1). IR (KBr) 1706, 1658, 1331, 1257, 1150, 1083, 1053 cm^{-1}. ^{1}H NMR δ 0.21 and 0.23 (each 3H, s, Si*Me*$_2$), 0.99 (9H, s, Si*Bu*^{t}), 1.00 and 1.22 (each 6H, br, NCH*Me*$_2$), 3.59 (2H, s, *CH*$_2$Ph), 3.69 and 3.92 (each 1H, br, NC*H*Me$_2$), 4.65 (1H, d, *J* = 6.1 Hz, H-3), 6.37 (1H, d, *J*= 6.1 Hz, H-4), 7.25-7.34 (5H, m, C*H*$_2$Ph). ^{13}C NMR δ –5.4 and -5.2 (Si*M*e$_2$), 18.1 (Si*C*Me$_3$), 20.5 and 21.0 (NCH*Me*$_2$), 25.6 (SiC*Me*$_3$), 43.3 (*C*H$_2$Ph), 46.0 and 46.3 (N*C*HMe$_2$), 72.0 (C-2), 107.2 (C-3), 118.3 (C-1), 127.3, 128.1, 131.0, and 134.3 (CH$_2$*Ph*), 141.8 (C-4), 152.4 (N*C*OPr$^i_2$). APCI-LC/MS *m*/*z* 431 (M$^+$ + H). Anal Calcd for $C_{24}H_{38}N_2O_3Si$: C, 66.94; H, 8.89; N, 6.50. Found: C, 67.02; H, 8.93; N, 6.59.

(±)-(*E*)-4-(*tert*-butyldimethylsilyloxy)-1-(*N*,*N*-diisopropylcarbamoyloxy)-3-butenonitrile (*rac*-(*E*)-12)



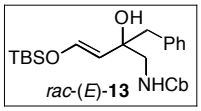
To a solution of (±)-**10a** (170 mg, 0.499 mmol) in Et_2O (1.7 mL) was added dropwise a solution of lithium diisopropylamide prepared from diisopropylamine (77 μL, 0.549 mmol) and *n*-butyl lithium (1.60 M in hexane, 0.340 mL, 0.544 mmol) in Et_2O (0.13 mL) over 2 min at -80 °C. The solution was stirred at the same temperature for 5 min before addition of AcOH in Et_2O (1.0 M, 0.550 mL, 0.550 mmol). After being stirred at the same temperature for 10 min, the reaction mixture was poured into saturated ammonium chloride solution (20 mL), and then extracted with Et_2O (15 mL x 3). The combined organic phases were washed with water (20 mL) and saturated brine (20 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica gel 40 g, elution with hexane:Et_2O = 10:1) to give a mixture of *rac*-(*E*)-**12** and *rac*-(*Z*)-**12** (6.7:1, 103 mg, 61%). This was purified again by column chromatography (silica gel 40 g, elution with hexane:Et_2O = 12:1). a colorless oil. R_f = 0.21 (hexane:Et_2O = 8:1). IR (neat) 1705, 1660, 1207, 1044 cm^{-1}. ^{1}H NMR δ 0.19 (6H, s, Si*Me*$_2$), 0.92 (9H, s, Si*Bu*^{t}), 1.21 and 1.22 (each 6H, d, *J* = 6.8 Hz, NCH*Me*$_2$), 3.77 and 4.00 (each 1H, br, NC*H*Me$_2$), 5.18 (1H, dd, *J* = 11.9, 8.6 Hz, H-3), 5.83 (1H, dd, *J*= 8.6, 0.7 Hz, H-2), 6.81 (1H, dd, *J* = 11.9, 0.7 Hz, H-4). ^{13}C NMR δ -5.3 and -5.3 (Si*M*e$_2$), 18.3 (Si*C*Me$_3$), 20.3 and 20.4 (NCH*Me*$_2$), 25.5 (SiC*Me*$_3$), 45.8 and 46.2 (N*C*HMe$_2$), 59.7 (C-2), 103.5 (C-3), 117.1 (C-1), 149.3 (C-4), 153.2 (N*C*OPr$^i_2$). APCI-LC/MS *m*/*z* 341 (M$^+$ + H). Anal Calcd for $C_{17}H_{32}N_2O_3Si$: C, 59.96; H, 9.47; N, 8.23. Found: C, 60.06; H, 9.19; N, 8.17.

***rac*-(*Z*)-4-(*tert*-butyldimethylsilyloxy)-1-(*N*,*N*-diisopropylcarbamoyloxy)-3-butenonitrile (*rac*-(*Z*)-12)**



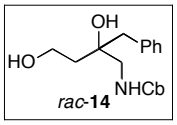
To a solution of (±)-**10b** (170 mg, 0.499 mmol) in Et_2O (1.7 mL) was added dropwise a solution of lithium diisopropylamide prepared from diisopropylamine (77 μL, 0.549 mmol) and *n*-butyl lithium (1.60 M in hexane, 0.340 mL, 0.544 mmol) in Et_2O (0.13 mL) over 2 min at -80 °C. The solution was stirred at the same temperature for 5 min before addition of AcOH in Et_2O (1.0 M, 0.550 mL, 0.550 mmol). After being stirred at the same temperature for 10 min, the reaction mixture was poured into saturated ammonium chloride solution (20 mL), and then extracted with Et_2O (15 mL x 3). The combined organic phases were washed with water (20 mL) and saturated brine (20 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica gel 40 g, elution with hexane:Et_2O = 10:1) to give a mixture of *rac*-(*E*)-**12** and *rac*-(*Z*)-**12** (1:4.5, 111 mg, 65%). This was purified again by column chromatography (silica gel 40 g, elution with hexane:Et_2O = 12:1). a colorless oil. R_f = 0.26 (hexane:Et_2O = 8:1). IR (neat) 1705, 1660, 1435, 1127, 1044 cm^{-1}. ^{1}H NMR δ 0.18 and 0.19 (each 3H, s, Si*Me*$_2$), 0.93 (9H, s, Si*Bu*^{t}), 1.21 and 1.22 (each 6H, d, *J* = 6.8 Hz, NCH*Me*$_2$), 3.75 and 4.03 (each 1H, br, NC*H*Me$_2$), 4.81 (1H, dd, *J* = 8.7, 5.5 Hz, H-3), 6.24 (1H, dd, *J*= 8.6, 1.0 Hz, H-2), 6.48 (1H, dd, *J* = 5.5, 1.0 Hz, H-4). ^{13}C NMR δ -5.5 and -5.4 (Si*M*e$_2$), 18.2 (Si*C*Me$_3$), 20.4 and 20.5 (NCH*Me*$_2$), 25.4 (SiC*Me*$_3$), 45.8 and 46.9 (N*C*HMe$_2$), 55.5 (C-2), 101.6 (C-3), 117.6 (C-1), 145.5 (C-4), 153.3 (N*C*OPr$^i_2$). APCI-LC/MS *m*/*z* 341 (M$^+$ + H). Anal Calcd for $C_{17}H_{32}N_2O_3Si$: C, 59.96; H, 9.47; N, 8.23. Found: C, 60.12; H, 9.56; N, 8.16.

***rac*-(*E*)-2-Benzyl-4-(*tert*-butyldimethylsilyloxy)-1-(*N*,*N*-diisopropylureido)-3-buten-2-ol (*rac*-(*E*)-13)**



To a cooled (0 °C) solution of *rac*-(*E*)-**11** (380 mg, 0.882 mmol) in THF (5.0 mL) was added lithium aluminum hydride (167 mg, 4.40 mmol) in several portions. After stirring for 10 min, cooling bath was removed. The mixture was stirred for 1 h at room temperature, then water (0.170 mL) and 10% aqueous NaOH solution (0.255 mL) were added. The mixture was filtered through a pad of Celite with Et_2O (50 mL). The filtrates were combined and concentrated. The residual solid was subjected to column chromatography (silica gel 33 g, hexane:AcOEt = 4:1 to 1:1) to give *rac*-(*E*)-**13** (199 mg, 52%). colorless needles, mp 119-120 °C (Pr^i_2O). R_f = 0.59 (hexane:AcOEt = 1:1). IR (KBr) 3470, 3294, 1669, 1610, 1172 cm^{-1}. ^1H NMR δ 007 and 0.07 (each 3H, s, Si*Me*$_2$), 0.86 (9H, s, Si*Bu*^{t}), 1.22 and 1.23 (each 3H, d, *J* = 6.9 Hz, NCH*Me*$_2$), 2.81 (2H, s, C*H*$_2$Ph), 3.10, 3.51 (each 1H, dd, *J* = 14.1, 7.0 Hz, H-1), 3.83 (2H, sep, *J* = 6.9 Hz, NC*H*Me$_2$), 4.00 (1H, s, O*H*), 4.54 (1H, m, N*H*), 4.94 (1H, d, *J* = 11.7 Hz, H-3), 6.41 (1H, d, *J* = 11.7 Hz, H-4), 7.16-7.29 (5H, m, CH$_2$*Ph*). ^{13}C NMR δ -5.3 and -5.2 (Si*Me*$_2$), 18.3 (Si*C*Me$_3$), 21.2 and 21.5 (NCH*Me*$_2$), 25.7 (SiC*Me*$_3$), 45.4 (N*C*HMe$_2$), 46.9 (*C*H$_2$Ph), 50.0 (C-1), 74.8 (C-2), 114.4 (C-3), 126.4, 128.0, 130.0, and 137.0 (CH$_2$*Ph*), 142.8 (C-4), 158.7 (NH*C*ONPr$^i_2$). APCI-LC/MS *m*/*z* 417 (M^+ + H - H_2O). Anal Calcd for $C_{24}H_{42}N_2O_3Si$: C, 66.31; H, 9.74; N, 6.44. Found: C, 66.15; H, 9.75; N, 6.53.

***rac*-3-Benzyl-4-(*N*,*N*-diisopropylureido)-1,3-butandiol (*rac*-14)**

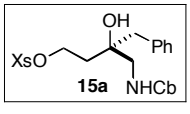


To a solution of *rac*-(*E*)-**13** (10 mg, 23.0 μmol) in THF (1.0 mL) was added TBAF (1.0 M in THF, 25 μL, 25.0 mmol) at 0 °C. After being stirred for 30 min at same temperature, EtOH (0.5 mL) and sodium borohydride (1.0 mg, 26.4 μmol) were added. The reaction mixture was stirred for 1 h at same temperature, and then poured into water (10 mL). The mixture was extracted with AcOEt (15 mL x 2), and the combined organic phases were washed with water (10 mL) and saturated brine (10 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 12 g, elution with hexane:AcOEt = 1:1 to AcOEt only) to give *rac*-**14** (7 mg, 94%). colorless prisms, mp 92-93 °C (hexane:AcOEt). R_f = 0.08 (hexane:AcOEt = 1:1). IR (KBr) 3390, 1628, 1513, 1041 cm^{-1}. ^1H NMR δ 1.23 (12H, d, *J* = 7.0 Hz, NCH*Me*$_2$), 1.63-1.80 (2H, m, H-2), 2.85 and 2.91 (each 1H, d, *J* = 13.7 Hz, C*H*$_2$Ph), 3.25 (1H, dd, *J* = 14.5, 5.9 Hz, H-4), 3.36 (1H, m, O*H*-1), 3.36 (1H, dd, *J* = 14.5, 5.7 Hz, H-4), 3.82 (2H, sep, *J* = 7.0 Hz, NC*H*Me$_2$), 3.80-3.90 (1H, m, H-1), 3.95-4.05 (1H, m, H-1), 4.70 (1H, m, N*H*), 5.08 (1H, s, O*H*-3), 7.15-7.30 (5H, m, CH$_2$*Ph*). ^{13}C NMR δ 21.3 (NCH*Me*$_2$), 38.2 (C-2), 44.8 (*C*H$_2$Ph), 45.5 (N*C*HMe$_2$), 49.2 (C-4), 59.4 (C-1), 75.7 (C-3), 126.5, 128.3, 130.5, and 137.4 (CH$_2$*Ph*), 158.9 (NH*C*ONPr$^i_2$). APCI-LC/MS *m*/*z* 323 (M^+ + H). Anal Calcd for $C_{18}H_{30}N_2O_3$: C, 67.05; H, 9.38; N, 8.69. Found: C, 66.88; H, 9.28; N, 8.73. chiral HPLC: Daicel CHIRALPAK® AD-H (4.6 mm x 150 mm), hexane:Pr^{i}OH = 95:5, flow rate 1.0 mL/min, $t_{R(R\text{-isomer})}$ = 11.2 min, $t_{R(S\text{-isomer})}$ = 12.6 min

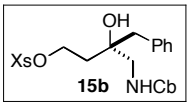
(1*S*,2*R*,4*R*)-*N*-(2-Carboxy-4,5-dichlorobenzoyl)-10,2-camphorsultam ester of (3*R*)-(+)-3-Benzyl-4-(*N*,*N*-diisopropylureido)-1,3-butandiol (15a) and (1*S*,2*R*,4*R*)-*N*-(2-Carboxy-4,5-dichlorobenzoyl)-10,2-camphorsultam ester of (3*S*)-(-)-3-Benzyl-4-(*N*,*N*-diisopropylureido)-1,3-butandiol (15b)

A solution of *rac*-(*E*)-**14** (30 mg, 93.0 μmol), (1*S*,2*R*,4*R*)-*N*-(2-carboxy-4,5-dichlorobenzoyl)-10,2-camphorsultam (48 mg, 0.111 mmol), and 4-dimethylaminopyridine (14 mg, 0.115 mmol) in CH_2Cl_2 (5.0 mL) was added *N*-ethyl-*N*'-(3-dimethylaminopropyl) carbodiimide hydrochloride (36 mg, 0.188 mmol) at room temperature. After being stirred at room temperature for 17 h, the reaction mixture was poured into water (30 mL), and the mixture was extracted with AcOEt (20 mL x 2). The combined organic phases were washed with saturated brine (20 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 33 g, elution with hexane:AcOEt = 7:3 to 1:1) to give a mixture of **15a** and **15b** (58 mg, 85%), which was separated by preparative chiral HPLC (Daicel CHIRALPAK® IA, semi-prep column (20 mm x 250 mm), hexane:AcOEt = 70:30, flow rate 8.0 mL/min).

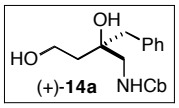
15a: colorless amorphous powder. R_f = 0.36 (hexane:AcOEt = 1:1). $[\alpha]^{25}_D$ -85.1 (*c* 0.202, $CHCl_3$). IR (KBr) 3435, 1726, 1687, 1615, 1520, 1334, 1298 cm^{-1}. ^1H NMR δ 0.97 and 1.20 (each 3H, s, H-8', H-9'), 1.22 and 1.23 (each 6H, d, *J* = 6.9 Hz, NCH*Me*$_2$), 1.35-1.50 (2H, m), 1.86-1.97 (5H, m), 2.07-2.21 and 2.38-2.51 (each 1H, m, H-3), 2.82 (2H, s, H-10'), 3.32 (2H, d, *J* = 5.9 Hz, H-1), 3.37 and 3.43 (each 1H, d, *J* = 13.8 Hz, C*H*$_2$Ph), 3.79 (2H, sep, *J* = 6.9 Hz, NC*H*Me$_2$), 4.04 (1H, br, H-2'), 4.43-4.58 (2H, m, H-4), 4.66 (1H, br, N*H*), 7.19-7.35 (5H, CH$_2$*Ph*), 7.51 and 8.02 (each 1H, s, $Cl_2C_6H_2$). ^{13}C NMR δ 20.2, 20.8, 21.5, 26.6, 33.3, 36.6, 37.8, 45.0, 45.1, 45.8, 48.0, 48.7, 49.1, 53.2, 62.9, 65.9, 74.1, 126.7, 128.5, 129.0, 130.7, 131.2, 131.6, 134.99, 135.03, 136.9, 137.3, 159.0, 163.8, 165.4. APCI-LC/MS *m*/*z* 736/738 (M^+ + H). The diastereomeric excess (100% de) was determined by chiral HPLC: Daicel CHIRALPAK® IA (4.6 mm x 150 mm), hexane:AcOEt = 70:30, flow rate 0.50 mL/min, $t_{R(R\text{-isomer})}$ = 17.1 min.



15b: colorless amorphous powder. R_f = 0.36 (hexane:AcOEt = 1:1). $[\alpha]^{25}_D$ -69.9 (*c* 0.206, $CHCl_3$). IR (KBr) 3435, 1726, 1686, 1616, 1521, 1333, 1298 cm^{-1}. ^1H NMR δ 0.98 and 1.20 (each 3H, s, H-8', H-9'), 1.20 (12H, d, *J* = 6.9 Hz, NCH*Me*$_2$), 1.36-1.49 (2H, m), 1.84-1.97 (5H, m), 2.08-2.23 and 2.40-2.52 (each 1H, m, H-3), 2.79 and 2.84 (each 1H, d, J = 13.5 Hz, H-10'), 3.32 (2H, dd, *J* = 5.6, 2.8 Hz, H-1), 3.38 and 3.43 (each 1H, d, *J* = 14.1 Hz, C*H*$_2$Ph), 3.79 (2H, sep, *J* = 6.9 Hz, NC*H*Me$_2$), 4.05 (1H, br, 2'), 4.39-4.60 (2H, m, H-4), 7.19-7.34 (5H, CH$_2$*Ph*), 7.52 and 8.02 (each 1H, s, $Cl_2C_6H_2$). ^{13}C NMR δ 20.2, 21.0, 21.5, 26.6, 33.3, 36.5, 37.8, 44.99, 45.04, 45.8, 48.0, 48.7, 49.2, 53.3, 63.0, 66.0, 74.2, 126.7, 128.5, 128.8, 129.1, 130.7, 131.3, 131.5, 135.0, 136.9, 137.4, 159.0, 163.8, 165.4. APCI-LC/MS *m/z* 736/738 (M^+ + H). The diastereomeric excess (99% de) was determined by chiral HPLC: Daicel CHIRALPAK® IA (4.6 mm x 150 mm), hexane:AcOEt = 70:30, flow rate 0.50 mL/min, $t_{R(S\text{-isomer})}$ = 22.4 min.

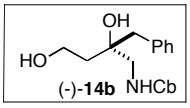


(3*R*)-(+)-3-Benzyl-4-(*N*,*N*-diisopropylureido)-1,3-butandiol ((+)-14a)



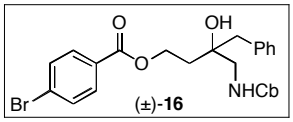
A suspension of **15a** (236 mg, 0.320 mmol) and K_2CO_3 (221 mg, 1.60 mmol) in MeOH (3.0 mL) was stirred at room temperature for 17 h. The reaction mixture was poured into water (3.0 mL), and the mixture was extracted with AcOEt (10 mL x 2). The combined organic phases were washed with saturated brine (10 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 33 g, elution with hexane:AcOEt = 1:1 to AcOEt only) to give (+)-**14a** (54 mg, 52%). colorless prisms, mp 90-91 °C (hexane-AcOEt). $[\alpha]^{25}_D$ +7.61 (*c* 0.210, $CHCl_3$). The enantiomeric excess (100% ee), determined by chiral HPLC: Daicel CHIRALPAK® AD-H (4.6 mm x 150 mm), hexane:Pr^{i}OH = 95:5, flow rate 1.0 mL/min, $t_{R(R\text{-isomer})}$ = 11.2 min.

(3*S*)-(-)-3-Benzyl-4-(*N*,*N*-diisopropylureido)-1,3-butandiol ((-)-14b)



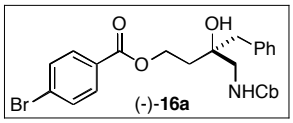
A suspension of **15b** (191 mg, 0.259 mmol) and K_2CO_3 (179 mg, 1.30 mmol) in MeOH (2.5 mL) was stirred at room temperature for 17 h. The reaction mixture was poured into water (3.0 mL), and the mixture was extracted with AcOEt (10 mL x 2). The combined organic phases were washed with saturated brine (10 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel 33 g, elution with hexane:AcOEt = 1:1 to AcOEt only) to give (-)-**14b** (49 mg, 59%). colorless prisms, mp 88-90 °C (hexane-AcOEt). $[\alpha]^{25}_D$ -7.54 (*c* 0.212, $CHCl_3$). The enantiomeric excess (99.2% ee) was determined by chiral HPLC: Daicel CHIRALPAK® AD-H (4.6 mm x 150 mm), hexane:Pr^{i}OH = 95:5, flow rate 1.0 mL/min, $t_{R(S\text{-isomer})}$ = 12.6 min.

(±)-2-Benzyl-4-(4'-bromobenzoyloxy)-1-(*N*,*N*-diisopropylureido)-2-butanol (*rac*-16)



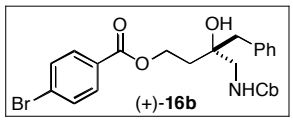
To a solution of (±)-**14** (100 mg, 0.310 mmol) and 4-bromobenzoyl chloride (82 mg, 0.374 mmol) in CH_2Cl_2 (2.0 mL) was added triethylamine (55 μL, 0.395 mmol), and 4-dimethylaminopyridine (4 mg, 32.7 μmol) at 0 °C. After the cooling bath was removed, the reaction mixture was stirred at room temperature for 1 h. The reaction mixture was subjected to column chromatography (silica gel 33 g, elution with hexane:AcOEt = 4:1 to 1:1) to give *rac*-**16** (143 mg, 91%). colorless prisms, mp 112-114 °C (hexane-AcOEt). R_f = 0.51 (hexane:AcOEt = 1:1). IR (KBr) 3361, 1718, 1602, 1534, 1270 cm^{-1}. ^1H NMR δ 1.22 and 1.23 (each 6H, d, *J* = 6.8 Hz, NCH*Me*$_2$), 1.93 (2H, t, *J* = 7.1 Hz, H-3), 2.85 (2H, s, C*H*$_2$Ph), 3.33 and 3.40 (each 1H, dd, *J* = 14.5, 6.0 Hz, H-1), 3.80 (2H, sep, *J* = 6.8 Hz, NC*H*Me$_2$), 4.54 and 4.54 (each 1H, t, *J* = 7.1 Hz, H-4), 4.67 (1H, br t, *J* = 6.0 Hz, N*H*), 4.69 (1H, br, O*H*), 7.18-7.34 (5H, m, CH$_2$*Ph*), 7.57 and 7.87 (each 2H, dt, *J* = 8.9, 2.2 Hz, 4-Br$C_6H_4CO_2$). ^{13}C NMR δ 21.5 and 21.5 (NCH*Me*$_2$), 37.0 (C-3), 45.2 (N*C*HMe$_2$), 45.8 (*C*H$_2$Ph), 49.2 (C-1), 62.1 (C-4), 74.1 (C-2), 126.8, 128.3, 128.5, 129.4, 130.7, 131.3, 131.9, and 137.3 (*Ar*), 158.9 (NH*C*ONPr$^i_2$), 166.2 (*C*O$_2$Ar). APCI-LC/MS *m/z* 505/507 (M^+ + H). Anal Calcd for $C_{25}H_{33}N_2O_4$: C, 59.41; H, 6.58; Br, 15.81; N, 5.54. Found: C, 59.34; H, 6.56; Br, 15.79; N, 5.57.

(2*R*)-(-)-2-Benzyl-4-(4'-bromobenzoyloxy)-1-(*N*,*N*-diisopropylureido)-2-butanol ((-)-16a)



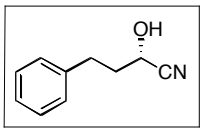
To a cooled (0 °C) solution of (+)-**14a** (44 mg, 0.136 mmol) and 4-bromobenzoyl chloride (33 mg, 0.150 mmol) in CH_2Cl_2 (1.0 mL) was added triethylamine (21 μL, 0.151 mmol) and 4-dimethylaminopyridine (2 mg, 16.4 μmol). After the cooling bath was removed, the reaction mixture was stirred at room temperature for 1 h. After removal of the solvent, the residue was subjected to column chromatography (silica gel 12 g, elution with hexane:AcOEt = 4:1 to 1:1) to give (-)-**16a** (57 mg, 83%). a colorless clear oil. $[\alpha]^{25}_D$ -15.4 (*c* 0.764, $CHCl_3$).

(2*S*)-(+)-2-Benzyl-4-(4'-bromobenzoyloxy)-1-(*N*,*N*-diisopropylureido)-2-butanol ((+)-16b)



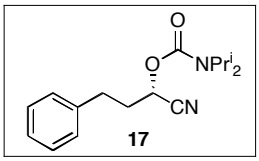
To a solution of (-)-**15b** (32 mg, 99.2 mmol) and 4-bromobenzoyl chloride (24 mg, 0.109 mmol) in CH_2Cl_2 (1.0 mL) was added triethylamine (15 μL, 0.108 mmol), and 4-dimethylaminopyridine (1 mg, 8.19 μmol) at 0 °C. After the cooling bath was removed, the reaction mixture was stirred at room temperature for 1 h. The reaction mixture was subjected to column chromatography (silica gel 12 g, elution with hexane:AcOEt = 4:1 to 1:1) to give (+)-**16b** (46 mg, 92%). colorless prisms, mp 77-79 °C (Pr$^i_2$O). $[\alpha]^{25}_D$ +16.4 (*c* 0.232, $CHCl_3$).

(*S*)-2-hydroxy-4-phenylbutanenitrile



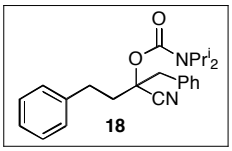
To a solution of 1-cyano-3-phenylpropyl ethanoate (4.88g, 24.0 mmol) in 10% THF-H_2O (30 mL) was added Amano lipase PS (0.51g). After stirring for 2 hr at 50 °C, AcOEt (30 mL) and sat. brine (30mL) were added. The mixture was filtered through a pad of Celite, extracted with AcOEt (25 mL X 2), dried and concentrated. The residual oil was subjected to column chromatography (silica gel. 120g, elution with hexane: Et_2O = 4:1) to give the title compound (1.57 g, 41%, 98% ee) and starting material (2.83 g, 58%). Chiral HPLC: Daicel Chiralcel® OD-H (4.6 mm x 250 mm), *n*-hexane/Pr^{i}OH = 100:1, flow rate 1.0 mL/min, t_R = 6.5 and 8.2 min (major) for the *O*-TBS-cyanohydrin.

(*S*)-1-cyano-3-phenylpropyl diisopropylcarbamate (17)



To a solution of triphosgene (359 mg, 1.21 mmol) in Et_2O (10 mL) was added triethylamine (467 μL, 3.35 mmol) at room temperature. After stirring for 30 min, (*S*)-2-hydroxy-4-phenylbutanenitrile (97% ee, 540 mg, 3.35 mmol) in Et_2O (5 mL) was added. After stirring for 30 min, diisopropylamine (471 μL, 3.35 mmol) and triethylamine (467 μL, 3.35 mmol) were added successively. After stirring for 2 hr, the mixture was filtered through a pad of Celite and concentrated. The residual oil was subjected to column chromatography (silica gel. 6g, elution with hexane: Et_2O = 6:1 to 4:1) to give **17** (335 mg, 35%). colorless plates, mp 30.5-31.0 °C (hexane). R_f = 0.23 (hexane:Et_2O = 4:1). IR (KBr) 1712 cm^{-1}. $[\alpha]^{21}_D$ -20.4 (*c* 1.20, $CHCl_3$). ^{1}H NMR δ 1.24 (12H, app d, *J* = 6.9 Hz, NC*Me*$_2$), 2.22-2.28 (2H, m, H-2), 2.81-2.90 (2H, m, Hz H-3), 3.77 (1H, br, N*C*Me$_2$), 4.04 (1H, br, N*C*Me$_2$), 5.39 (1H, t, *J* = 6.9 Hz, H-1), 7.19-7.26 (3H, m, Ph), 7.29-7.33 (2H, m, Ph). ^{13}C NMR δ 20.6 and 21.7 (NC*Me*$_2$), 31.2, 34.6 (C-3, C-4), 46.0 and 47.4 (N*C*Me$_2$), 61.4 (C-1), 117.7 (CN), 126.8, 128.5, 128.9, 139.6 (Ph), 153.3 (N*C*O^{i}Pr$_2$). HRMS calcd for $C_{17}H_{24}N_2O_2$ (M^+ + 1) 289.1916, found 289.1889.

2-cyano-1,4-diphenylbutan-2-yl diisopropylcarbamate (18)



To a cooled (-80 °C) solution of **17** (50 mg, 0.173 mmol) and benzyl bromide (103 μL, 0.865 mmol) in Et_2O (1.44 mL) was added dropwise a solution of lithium diisopropylamide (1.0 M in Et_2O, 0.190 mL, 0.190 mmol) over 15 sec. After being stirred at the same temperature for 60 min, the reaction mixture was poured into saturated ammonium chloride solution (3 mL), and then extracted with Et_2O (10 mL x 2). The combined organic phases were washed with saturated brine (10 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica gel 13 g, elution with hexane:Et_2O = 7:1 to 5:1) to give **18** (55.4 mg, 84%, 5% ee) Chiral HPLC: Chiralpak® IC (4.6 mm x 250 mm), hexane:*i*-PrOH = 20:1, flow rate 1mL/min, t_R = 13.6 min and 15.7 min (major). colorless plates, mp 63.5-64.0 °C (hexane). R_f = 0.28 (hexane:Et_2O = 4:1). IR (KBr) 1704 cm^{-1}. ^{1}H NMR δ 1.05 (3H, app. d, *J* = 6.2 Hz, NC*Me*$_2$), 1.11 (3H, app. d, *J* = 6.2 Hz, NC*Me*$_2$), 1.29 (6H, br, NC*Me*$_2$), 2.17-2.26 (1H, m, H-3), 2.32-2.39 (1H, m, H-3), 2.86-2.96 (2H, m, Hz H-4), 3.45 (1H, d, 13.7 Hz), 3.56 (1H, d, 13.7 Hz), 3.71 (1H, br, N*C*Me$_2$), 3.98 (1H, br, N*C*Me$_2$), 7.18-7.35 (10H, m, Ph).^{13}C NMR δ 20.6 and 21.4 (NC*Me*$_2$), 30.8 (C-4), 39.4 (C-3), 42.8 (C-1), 46.3 and 46.8 (N*C*Me$_2$), 75.7 (C-2), 119.2 (CN), 126.6, 127.8, 128.6, 128.7, 128.8, 130.9, 133.7, 140.4 (Ph), 152.6 (N*C*O^{i}Pr$_2$). HRMS calcd for $C_{24}H_{30}N_2O_2$(M^+ + 1), 379.2386, found 379.2381. Anal Calcd for $C_{24}H_{30}N_2O_2$, C, 76.16; H, 7.99; N, 7.40. found C, 76.30; H, 8.22; N, 7.46.

Crystal Data of *epi*-5

Empirical Formula	$C_{16}H_{33}NO_2Si_2$
Formula Weight	327.61
Crystal Color, Habit	clear, prism
Crystal Dimensions	0.43 x 0.18 x 0.10 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2θ range)	0 (0.0 - 0.0°)
Lattice Parameters	a = 6.968(1) Å
	b = 11.050(2) Å
	c = 27.581(6) Å
	V = 2123.7(8) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.025 g/cm^3
F_{000}	720.00
μ(MoKa)	1.71 cm^{-1}
R factor	0.0520

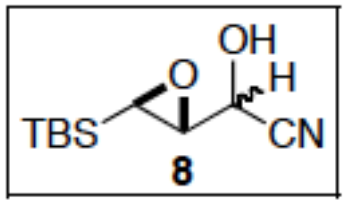
Crystal Data of 9b

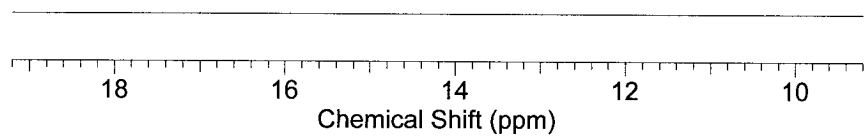
Empirical Formula	$C_{17}H_{33}N_2O_3SiCl$
Formula Weight	377.00
Crystal Color, Habit	clear, prism
Crystal Dimensions	0.45 x 0.36 x 0.11 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	240 (0.0 - 26.0°)
Lattice Parameters	a = 6.416(2) Å b = 15.975(3) Å c = 10.627(3) Å β = 97.883(3)° V = 1078.9(5) Å^3
Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.160 g/cm^3
F_{000}	408.00
μ(MoKa)	2.48 cm^{-1}
R factor	0.0397

Crystal Data of (+)-16

Empirical formula	$C_{25}H_{33}BrN_2O_4$
Formula weight	505.44
Temperature	150 K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P1
Unit cell dimensions	a = 9.118(2) Å α = 92.341(5)°.
	b = 10.660(3) Å β = 93.643(5)°.
	c = 13.666(3) Å γ = 111.726(4)°.
Volume	1228.5(5) $Å^3$
Z value	2
Density (calculated)	1.366 Mg/m^3
Absorption coefficient	1.706 mm^{-1}
F(000)	528
Crystal size	0.43 x 0.20 x 0.10 mm^3
Theta range for data collection	2.06 to 27.54°.
Index ranges	-11<=h<=10, -13<=k<=13, -14<=l<=17
Reflections collected	7358
Independent reflections	6287 [R(int) = 0.0494]
Completeness to theta = 27.54°	93.7 %
Absorption correction	Empirical
Max. and min. transmission	0.8479 and 0.5275
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6287 / 3 / 560
Goodness-of-fit on F^2	0.945
Final R indices [I>2sigma(I)]	R1 = 0.0668, wR2 = 0.1509
R indices (all data)	R1 = 0.1636, wR2 = 0.1927
Absolute structure parameter	-0.029(16)
Largest diff. peak and hole	0.904 and -0.996 e.$Å^{-3}$

300M NMR (Gemini 2000)





Comment	AT20050218005 E1109020A CDCl3	**Date**	Feb 18 2005
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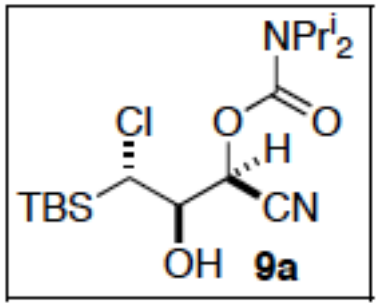
Frequency (MHz)	300.04	**Number of Transients**	80
Sweep Width (Hz)	5999.00	**Pulse Sequence**	s2pul

S12

Operator :N.Fukui

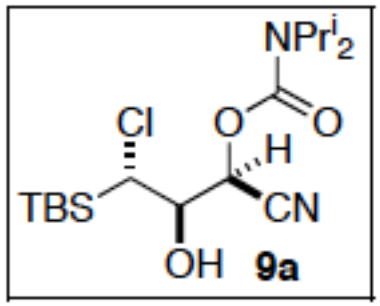
300M NMR (Gemini 2000)



Comment	AT20051114007 E1133198B CDCl3	**Date**	Nov 14 2005
Nucleus	1H	**Solvent**	CHLOROFORM-D
Frequency (MHz)	300.04	**Number of Transients**	96
Sweep Width (Hz)	5999.00	**Pulse Sequence**	s2pul

S13

Operator : N.Fukui



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77.4594
77.2000
76.9482
64.2431
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X : parts per Million : 13C

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Revision Date	= 24-NOV-2005 07:14
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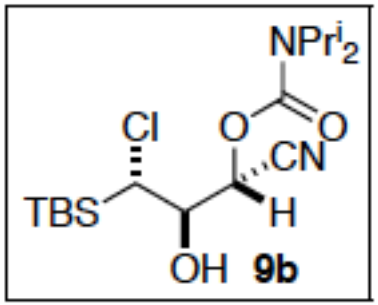
300M NMR (Gemini 2000)



Comment	AT20050623022 E1133106A CDCl3	**Date**	Jun 24 2005
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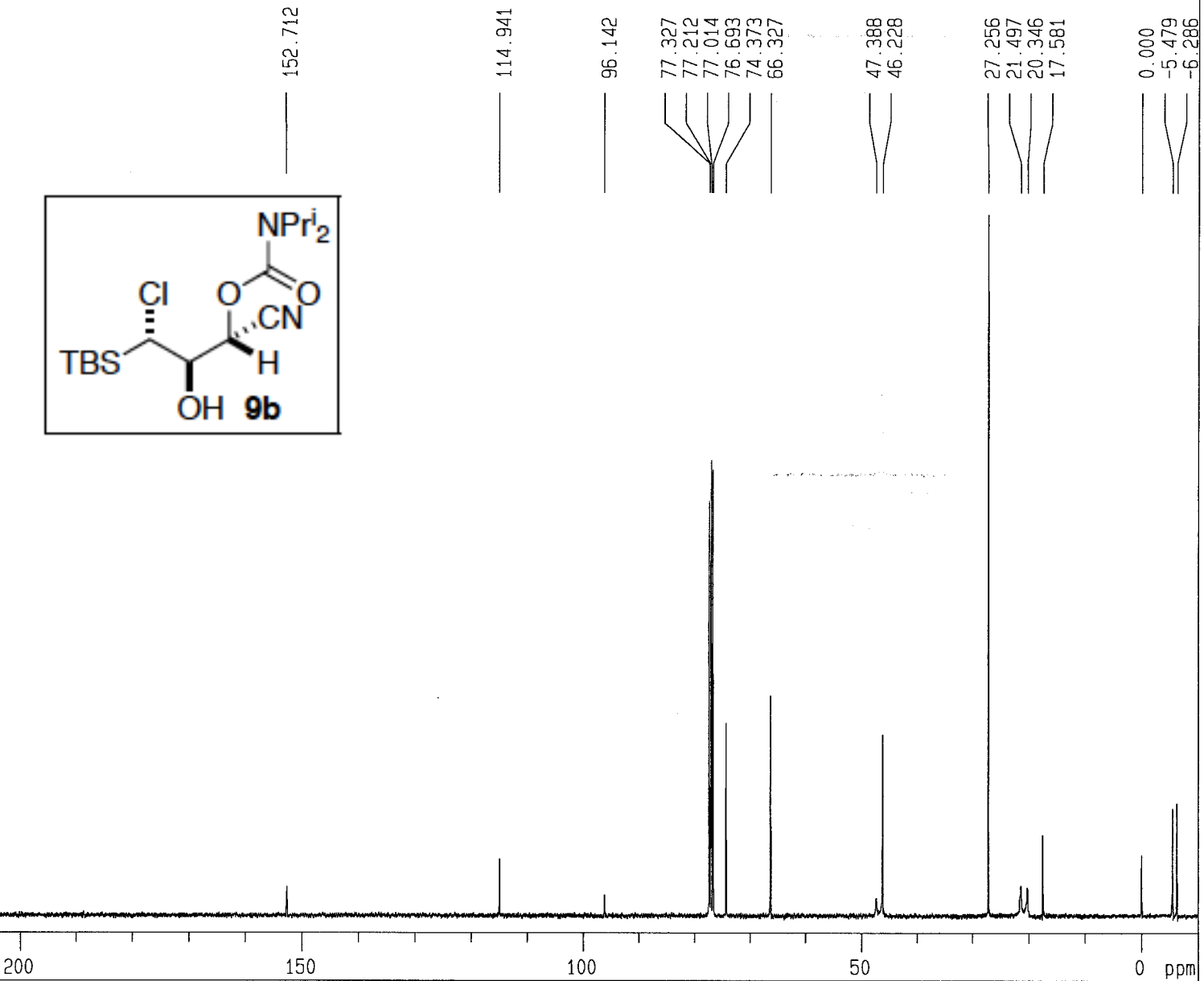
Frequency (MHz)	300.04	**Number of Transients**	128
Sweep Width (Hz)	5999.00	**Pulse Sequence**	s2pul

Operator :N.Fukui

MT20051007002_E1133106A//JNM-LA400//N.Fukui



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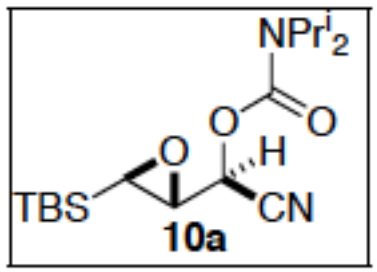

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1.02

1.02

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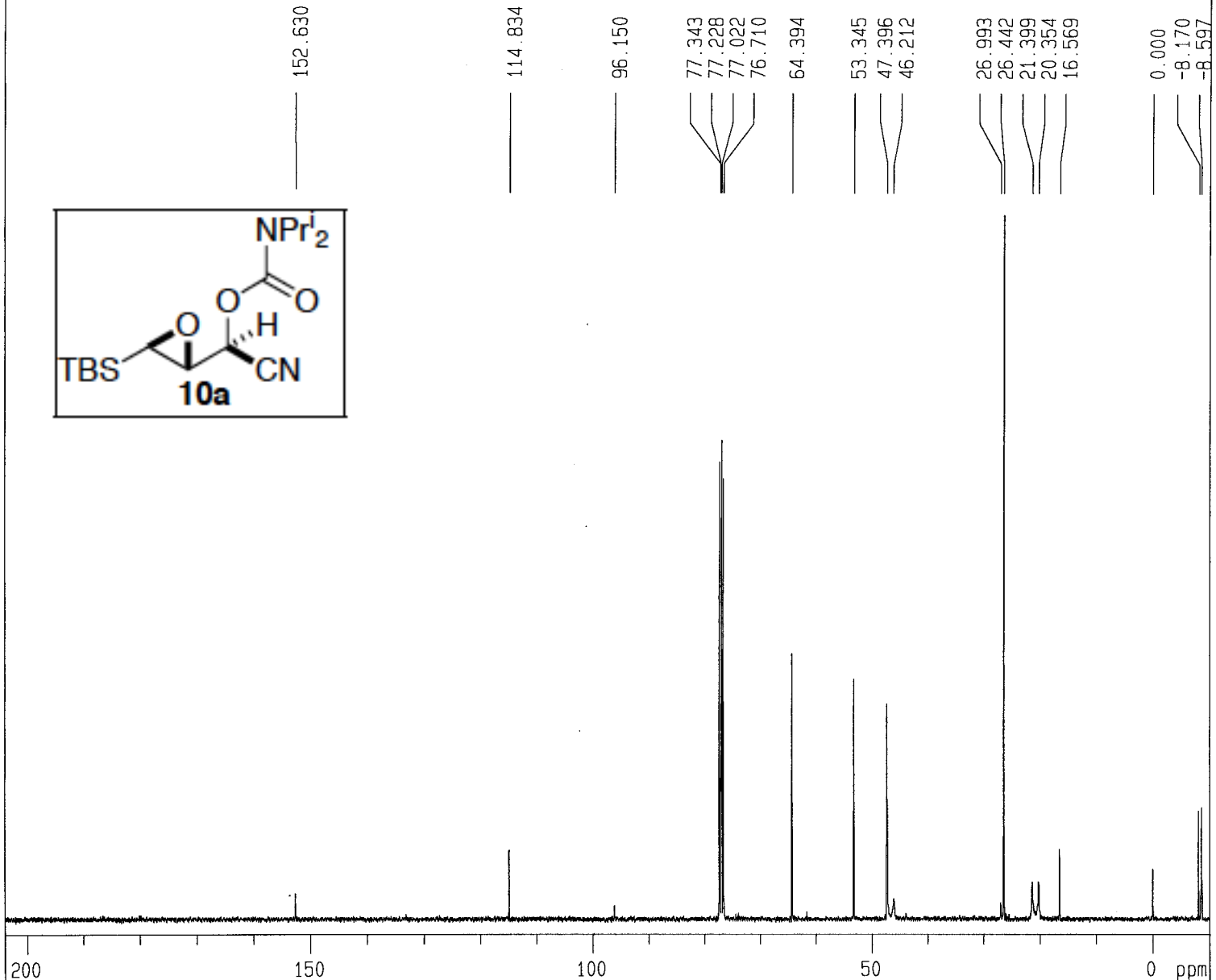
MT20051006002_E1133093A//JNM-LA400//N.Fukui



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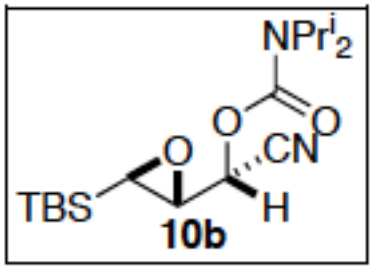
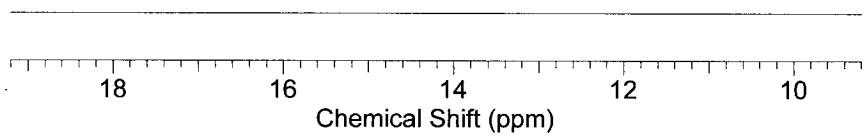
300M NMR (Gemini 2000)





Comment	AT20050202009 E1109005A CDCl3	**Date**	Feb 2 2005
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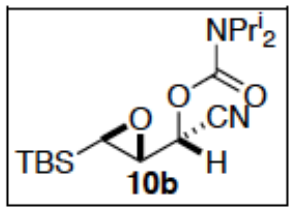
Frequency (MHz)	300.04	**Number of Transients**	64



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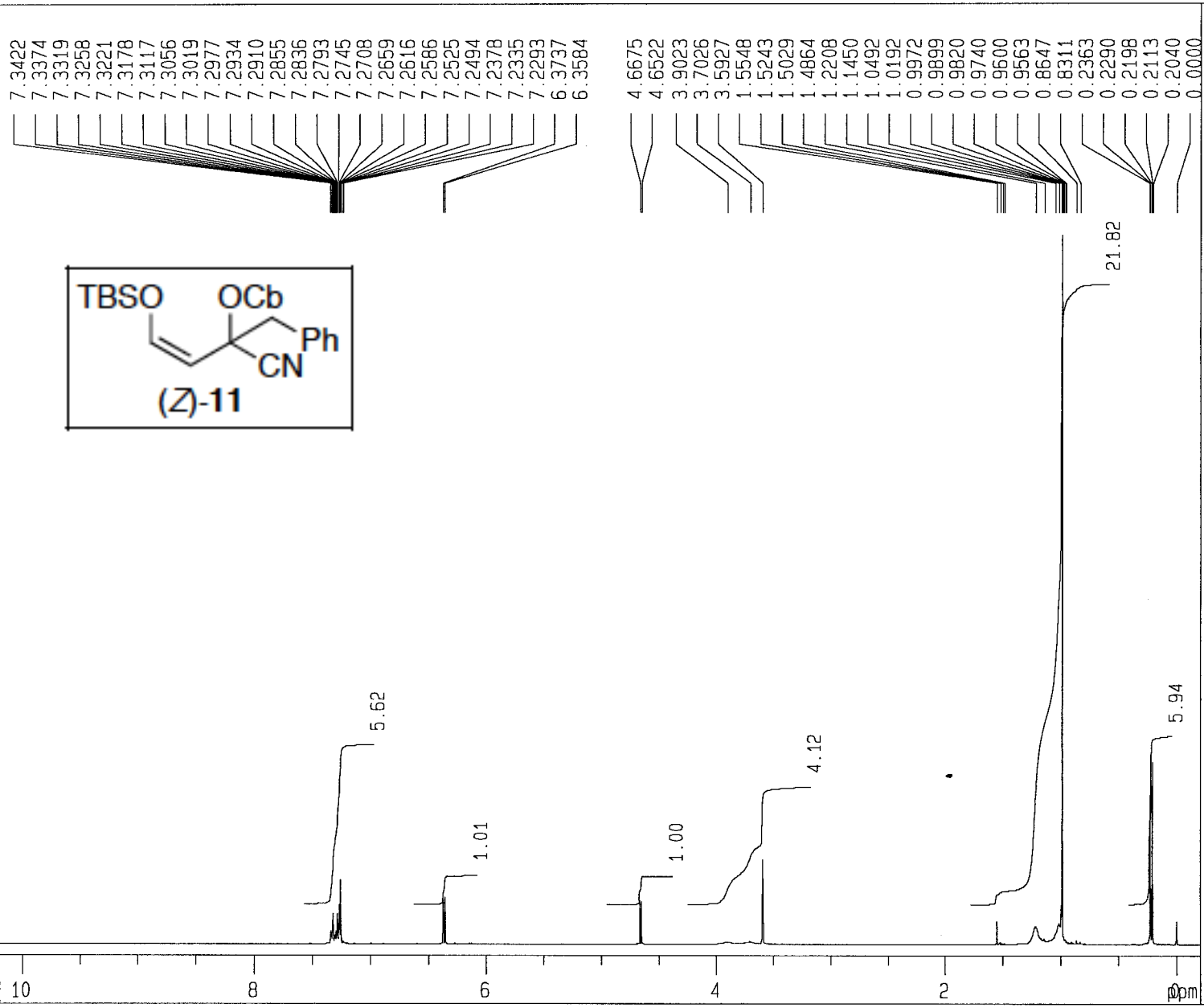
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 3-NOV-2005 09:21
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 0.165[ms]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH

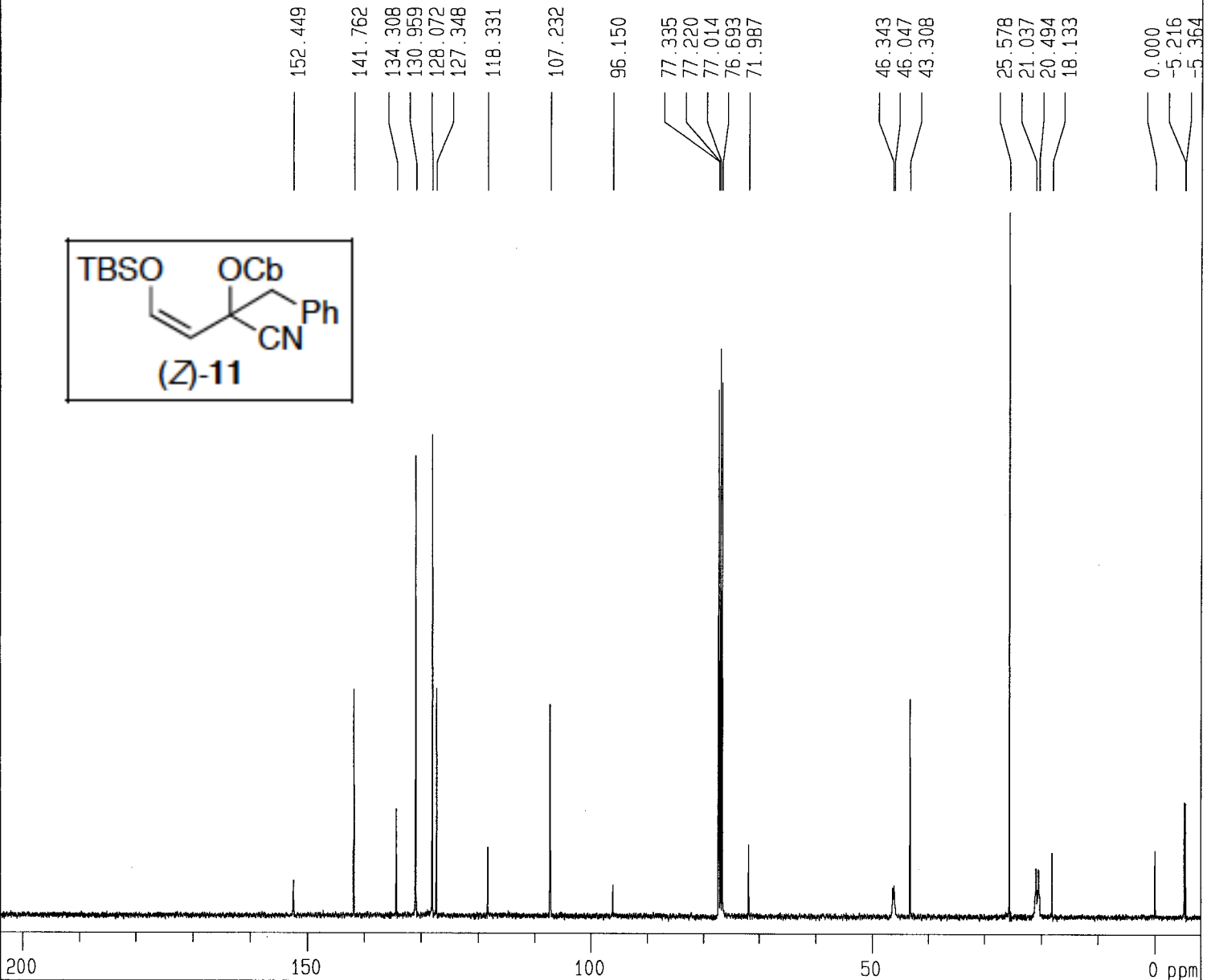
MT20051114001_E1133153A//JNM-LA400//N.Fukui



Date : Mon Nov 14 13:54:23 2005

FileName	:	MT20051114001.nmdata
Comment	:	MT20051114001_E1133153A
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	8000.0 Hz
FILTR	:	7000 Hz
DELAY	:	28.6 usec
DEADT	:	39.5 usec
INTVL	:	125.0 usec
TIMES	:	16 times
DUMMY	:	4 times
PD	:	5.0000 sec
ACQTM	:	2048.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.75 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	136000.00 Hz
RGAIN	:	17
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	30.0 C

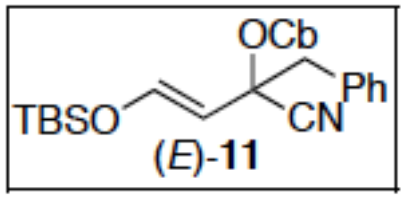
MT20051114001_E1133153A//JNM-LA400//N.Fukui



Date : Mon Nov 14 19:04:11 2005

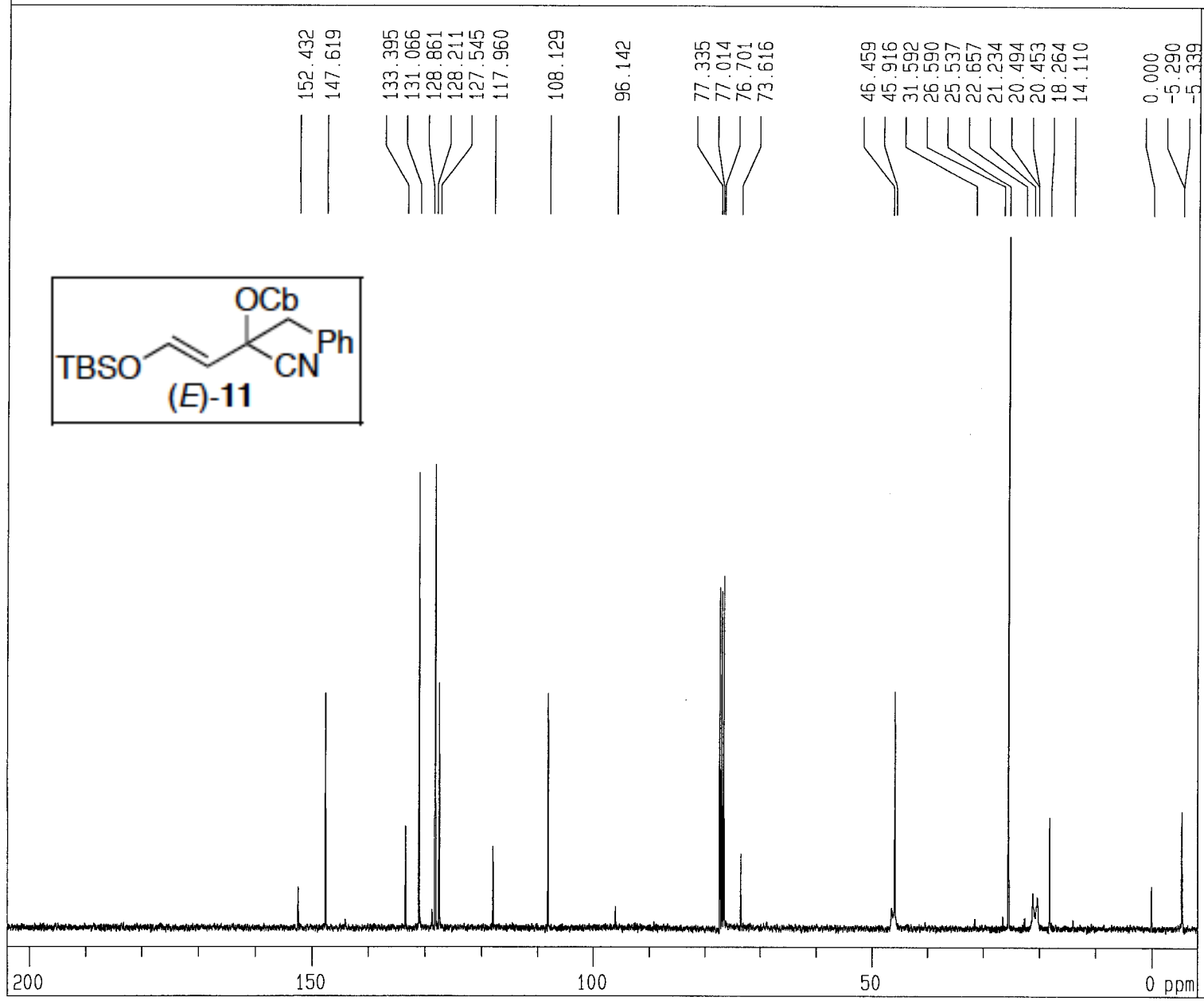
FileName	:	MT20051114001c2.nmdata
Comment	:	MT20051114001_E1133153A
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	27100.3 Hz
FILTR	:	13550 Hz
DELAY	:	14.8 usec
DEADT	:	19.8 usec
INTVL	:	36.9 usec
TIMES	:	10000 times
DUMMY	:	4 times
PD	:	1.0000 sec
ACQTM	:	604.5696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.83 Hz
PW1	:	4.65 usec
OBNUC	:	13C
OBFRQ	:	100.40 MHz
OBSET	:	137500.00 Hz
RGAIN	:	26
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRRPW	:	55.0 usec
IRRNS	:	0
SCANS	:	10000 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	30.0 C

300M NMR (Gemini 2000)



Comment	AT20050316011 E1109032A CDCl3	**Date**	Mar 16 2005
Nucleus	1H	**Solvent**	CHLOROFORM-D
Frequency (MHz)	300.04	**Number of Transients**	160
Sweep Width (Hz)	5999.00	**Pulse Sequence**	s2pul

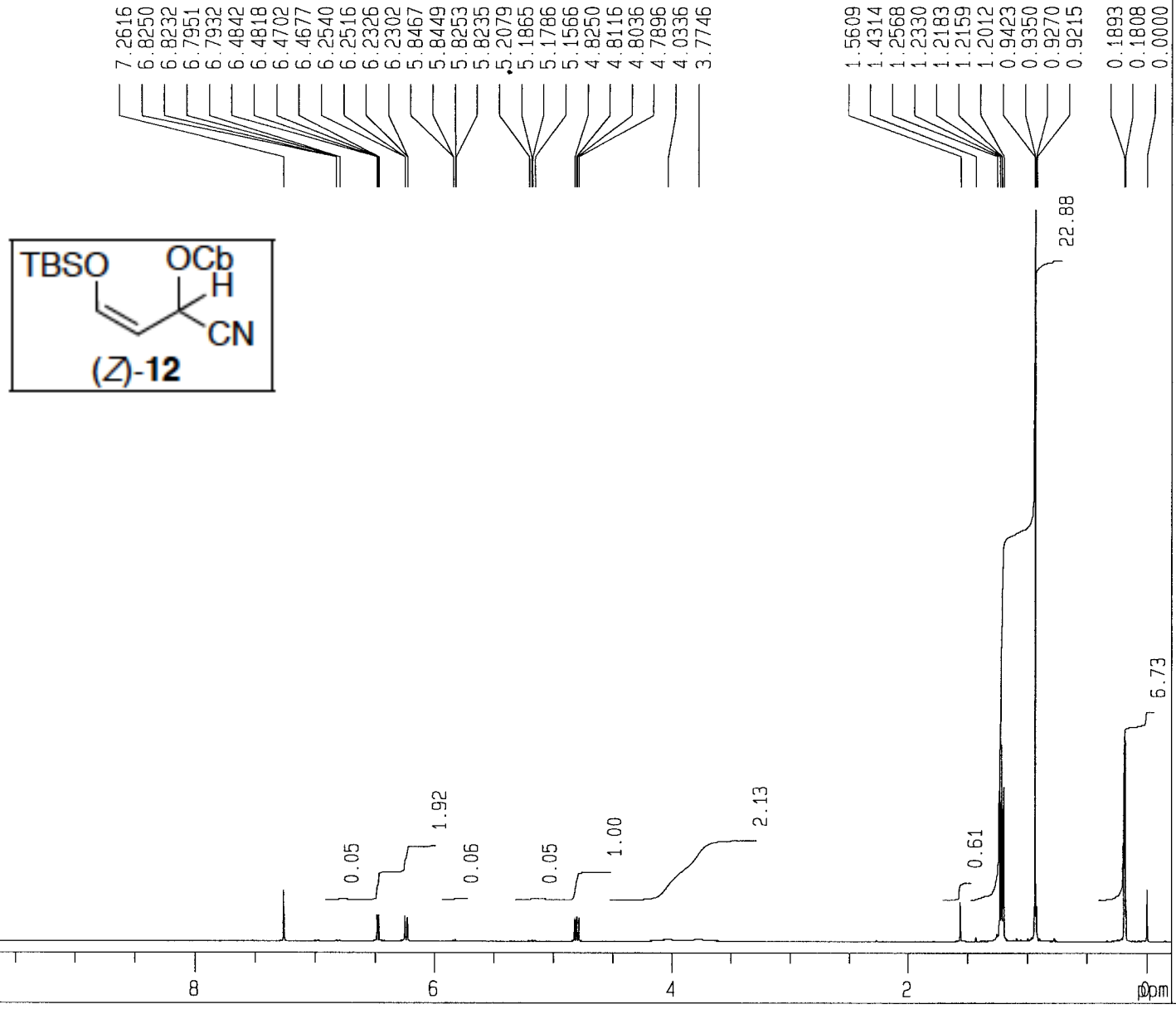
MT20051128001_E1232032A//JNM-LA400//N.Fukui



Date : Mon Nov 28 15:32:05 2005

FileName	:	MT20051128001c2.nmdata
Comment	:	MT20051128001_E1232032A
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	27100.3 Hz
FILTR	:	13550 Hz
DELAY	:	14.8 usec
DEADT	:	19.8 usec
INTVL	:	36.9 usec
TIMES	:	3000 times
DUMMY	:	4 times
PD	:	1.0000 sec
ACQTM	:	604.5696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.83 Hz
PW1	:	4.65 usec
OBNUC	:	13C
OBFRQ	:	100.40 MHz
OBSET	:	137500.00 Hz
RGAIN	:	26
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRRPW	:	55.0 usec
IRRNS	:	0
SCANS	:	3000 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	30.0 C

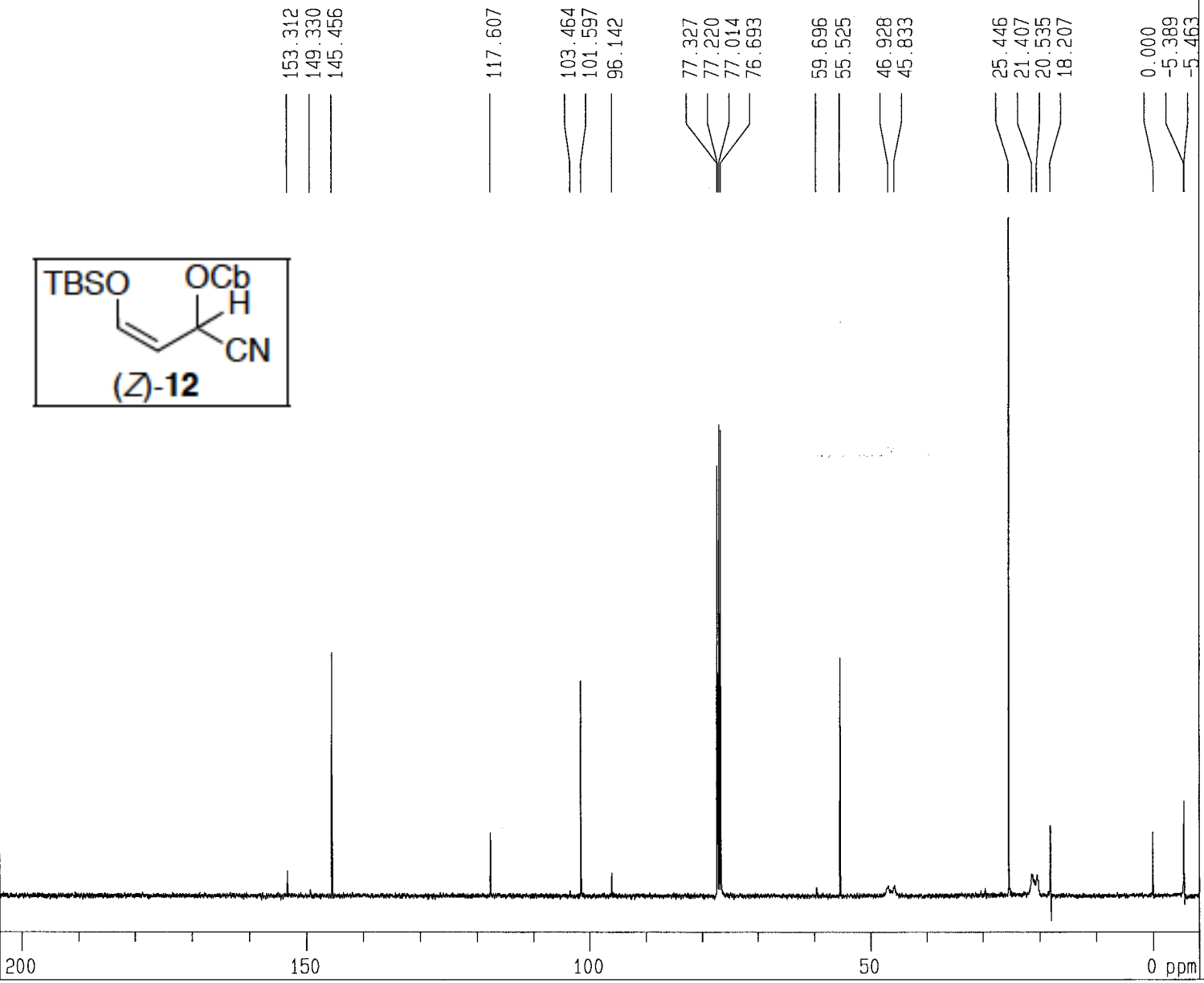
MT20051121001_E1232007A//JNM-LA400//N.Fukui



Date : Mon Nov 21 19:48:06 2005

FileName	:	MT20051121001.nmdata
Comment	:	MT20051121001_E1232007A
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	8000.0 Hz
FILTR	:	7000 Hz
DELAY	:	28.6 usec
DEADT	:	39.5 usec
INTVL	:	125.0 usec
TIMES	:	16 times
DUMMY	:	4 times
PD	:	5.0000 sec
ACQTM	:	2048.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.75 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	136000.00 Hz
RGAIN	:	16
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	30.0 C

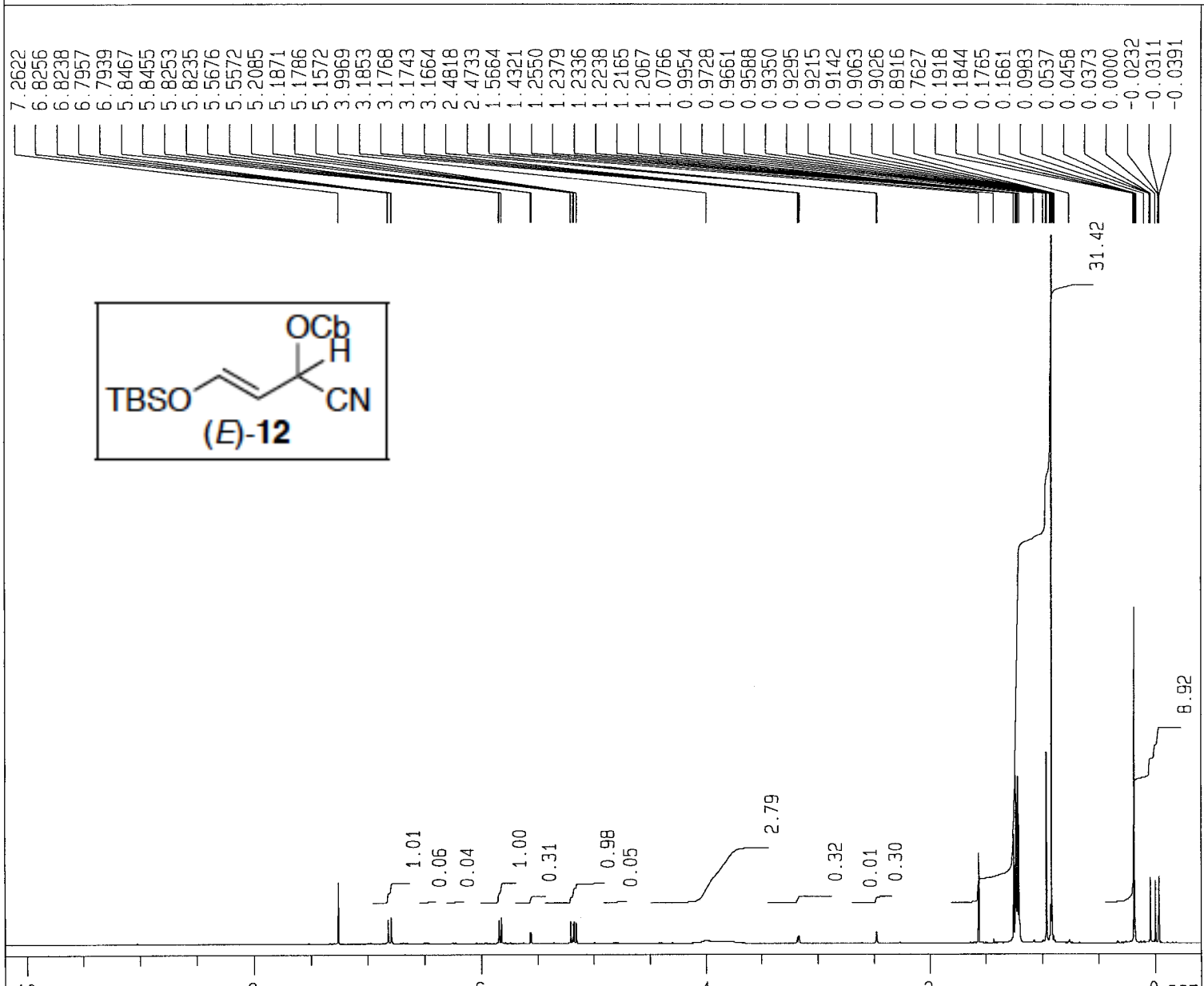
MT20051121001_E1232007A//JNM-LA400//N.Fukui



Date : Tue Nov 22 00:57:20 2005

FileName	:	MT20051121001c2.nmdata
Comment	:	MT20051121001_E1232007A
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	27100.3 Hz
FILTR	:	13550 Hz
DELAY	:	14.8 usec
DEADT	:	19.8 usec
INTVL	:	36.9 usec
TIMES	:	10000 times
DUMMY	:	4 times
PD	:	1.0000 sec
ACQTM	:	604.5696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.83 Hz
PW1	:	4.65 usec
OBNUC	:	13C
OBFRQ	:	100.40 MHz
OBSET	:	137500.00 Hz
RGAIN	:	26
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRRPW	:	55.0 usec
IRRNS	:	0
SCANS	:	10000 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	30.0 C

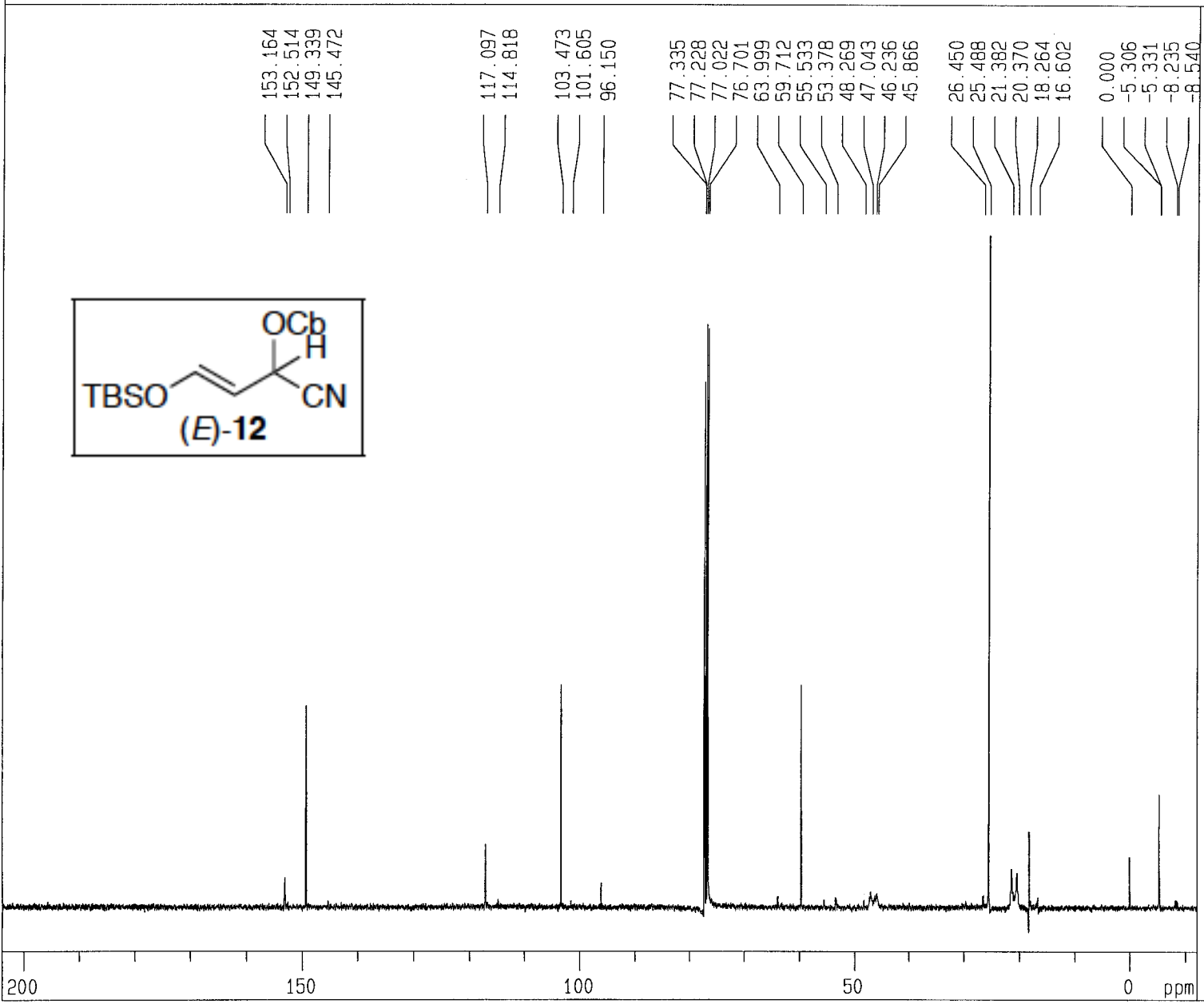
MT20051124001_E1232005A//JNM-LA400//N.Fukui



Date : Thu Nov 24 16:41:00 2005

FileName	:	MT20051124001.nmdata
Comment	:	MT20051124001_E1232005A
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	8000.0 Hz
FILTR	:	7000 Hz
DELAY	:	28.6 usec
DEADT	:	39.5 usec
INTVL	:	125.0 usec
TIMES	:	16 times
DUMMY	:	4 times
PD	:	5.0000 sec
ACQTM	:	2048.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.75 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	136000.00 Hz
RGAIN	:	16
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	30.0 C

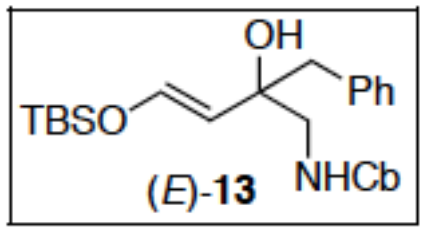
MT20051124001_E1232005A//JNM-LA400//N.Fukui



Date : Thu Nov 24 22:11:25 2005

FileName	:	MT20051124001c2.nmdata
Comment	:	MT20051124001_E1232005A
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	27100.3 Hz
FILTR	:	13550 Hz
DELAY	:	14.8 usec
DEADT	:	19.8 usec
INTVL	:	36.9 usec
TIMES	:	10000 times
DUMMY	:	4 times
PD	:	1.0000 sec
ACQTM	:	604.5696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.83 Hz
PW1	:	4.65 usec
OBNUC	:	13C
OBFRQ	:	100.40 MHz
OBSET	:	137500.00 Hz
RGAIN	:	26
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRRPW	:	55.0 usec
IRRNS	:	0
SCANS	:	10000 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	30.0 C

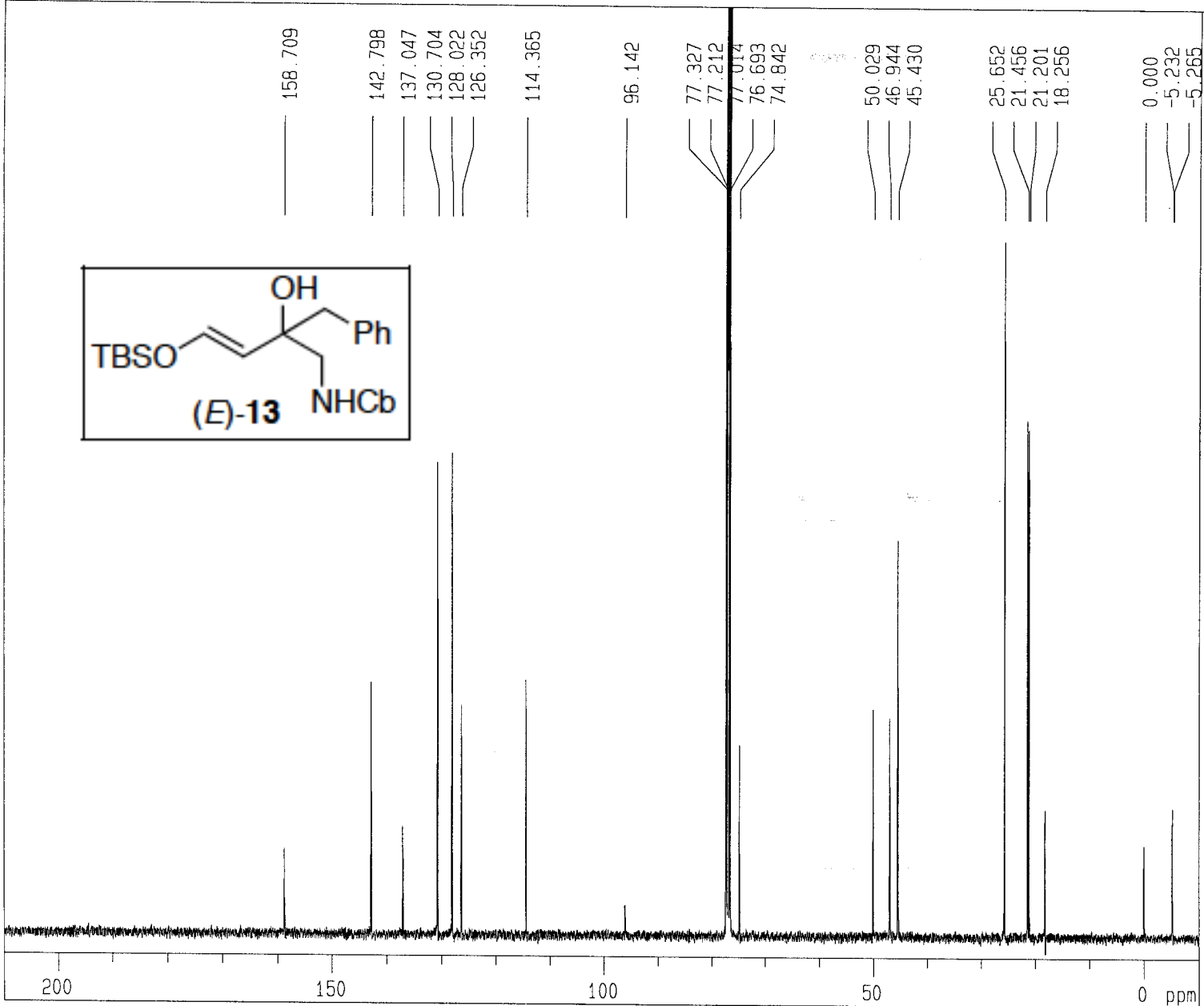
MT20050531001_E1133002A//JNM-LA400//N.Fukui



Date : Tue May 31 16:41:50 2005

FileName	:	MT20050531001.nmdata
Comment	:	MT20050531001_E1133002A
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	8000.0 Hz
FILTR	:	7000 Hz
DELAY	:	28.6 usec
DEADT	:	39.5 usec
INTVL	:	125.0 usec
TIMES	:	32 times
DUMMY	:	4 times
PD	:	5.0000 sec
ACQTM	:	2048.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.75 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	136000.00 Hz
RGAIN	:	18
SCANS	:	32 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	30.0 C

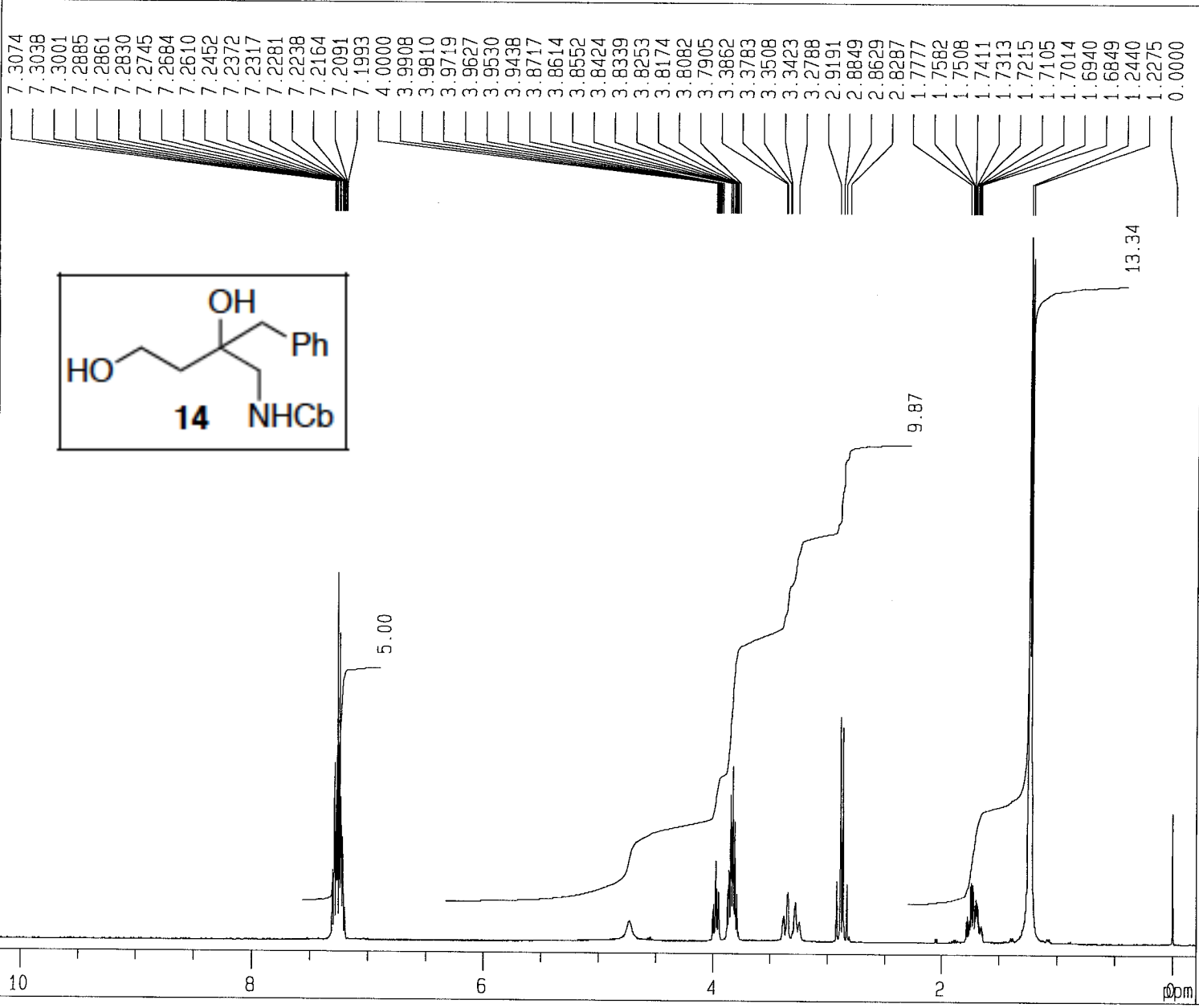
MT20050531001_E1133002A//JNM-LA400//N.Fukui



Date : Wed Jun 1 17:12:11 2005

FileName	:	MT20050531001c3.nmdata
Comment	:	MT20050531001_E1133002A
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	27100.3 Hz
FILTR	:	13550 Hz
DELAY	:	14.8 usec
DEADT	:	19.8 usec
INTVL	:	36.9 usec
TIMES	:	10000 times
DUMMY	:	4 times
PD	:	2.0000 sec
ACQTM	:	604.5696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.83 Hz
PW1	:	4.65 usec
OBNUC	:	13C
OBFRQ	:	100.40 MHz
OBSET	:	137500.00 Hz
RGAIN	:	26
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRRPW	:	55.0 usec
IRRNS	:	0
SCANS	:	10000 times
SLVNT	:	DMSO
SPINNING	:	13 Hz
TEMP	:	30.0 C

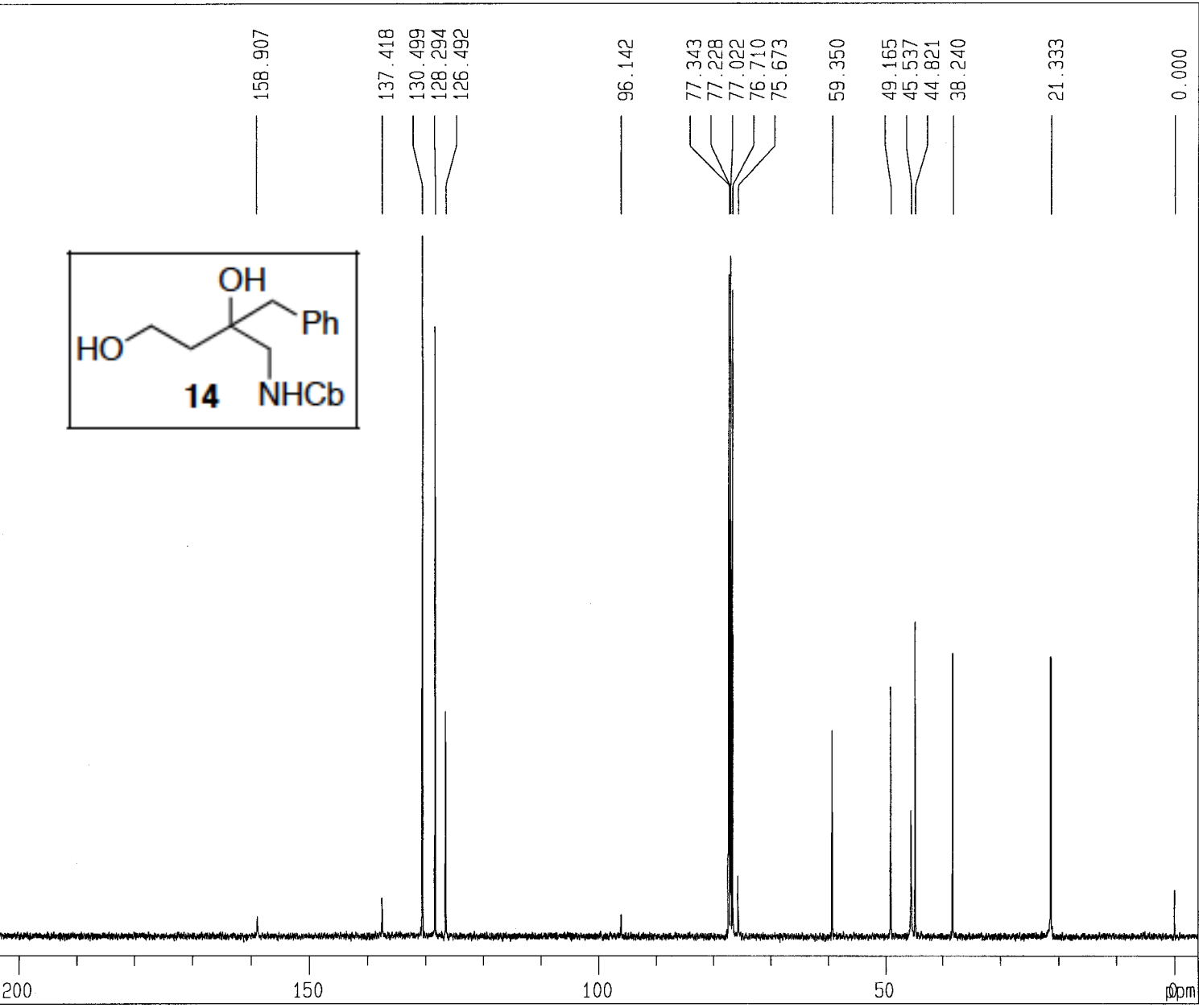
MT20051020001_E1133114C//JNM-LA400//N.Fukui



Date : Thu Oct 20 17:33:52 2005

FileName	:	MT20051020001.nmdata
Comment	:	MT20051020001_E1133114C
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	8000.0 Hz
FILTR	:	7000 Hz
DELAY	:	28.6 usec
DEADT	:	39.5 usec
INTVL	:	125.0 usec
TIMES	:	16 times
DUMMY	:	4 times
PD	:	5.0000 sec
ACQTM	:	2048.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.75 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	136000.00 Hz
RGAIN	:	16
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	10 Hz
TEMP	:	30.1 C

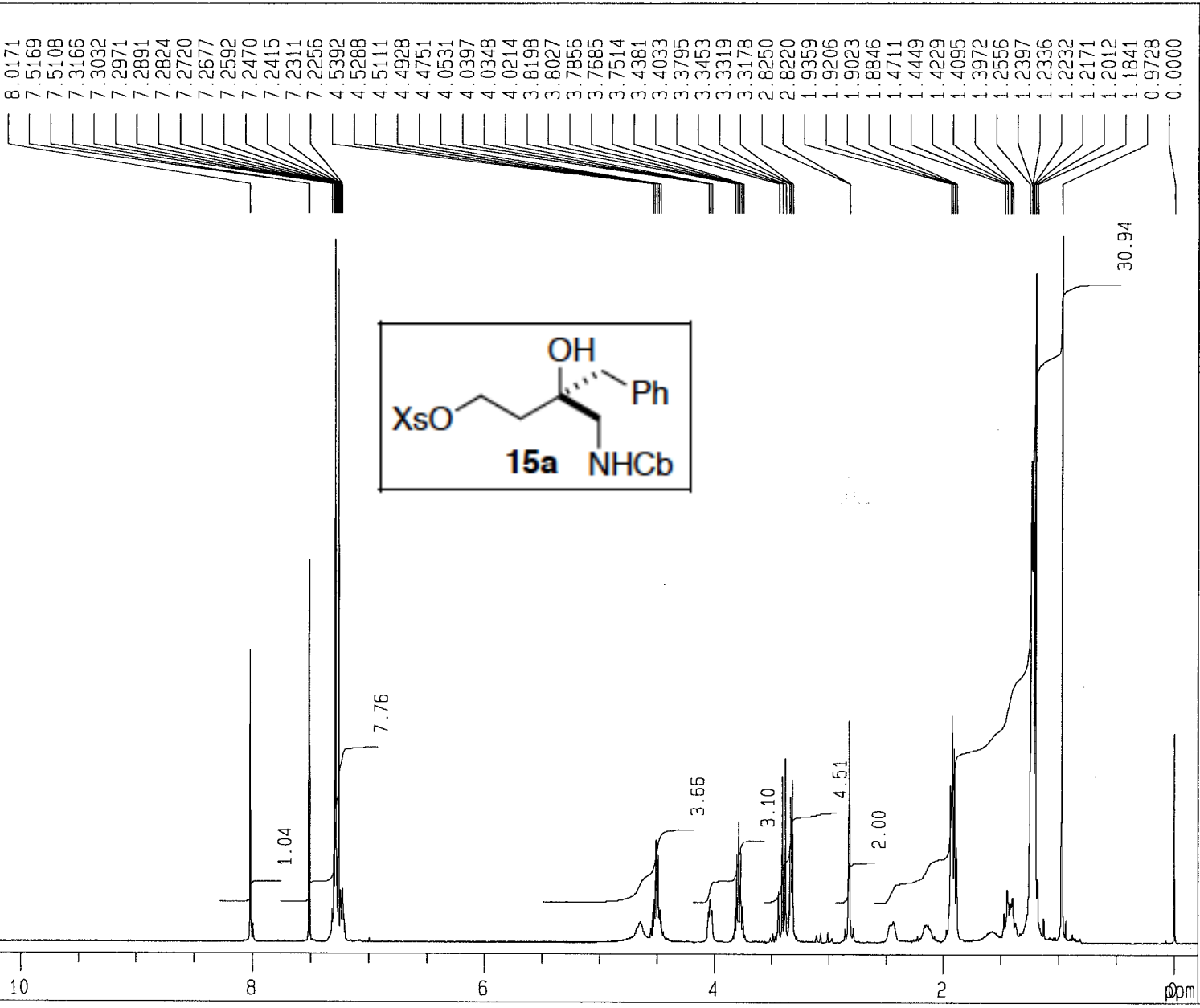
MT20051020001_E1133114C//JNM-LA400//N.Fukui



Date : Thu Oct 20 23:09:50 2005

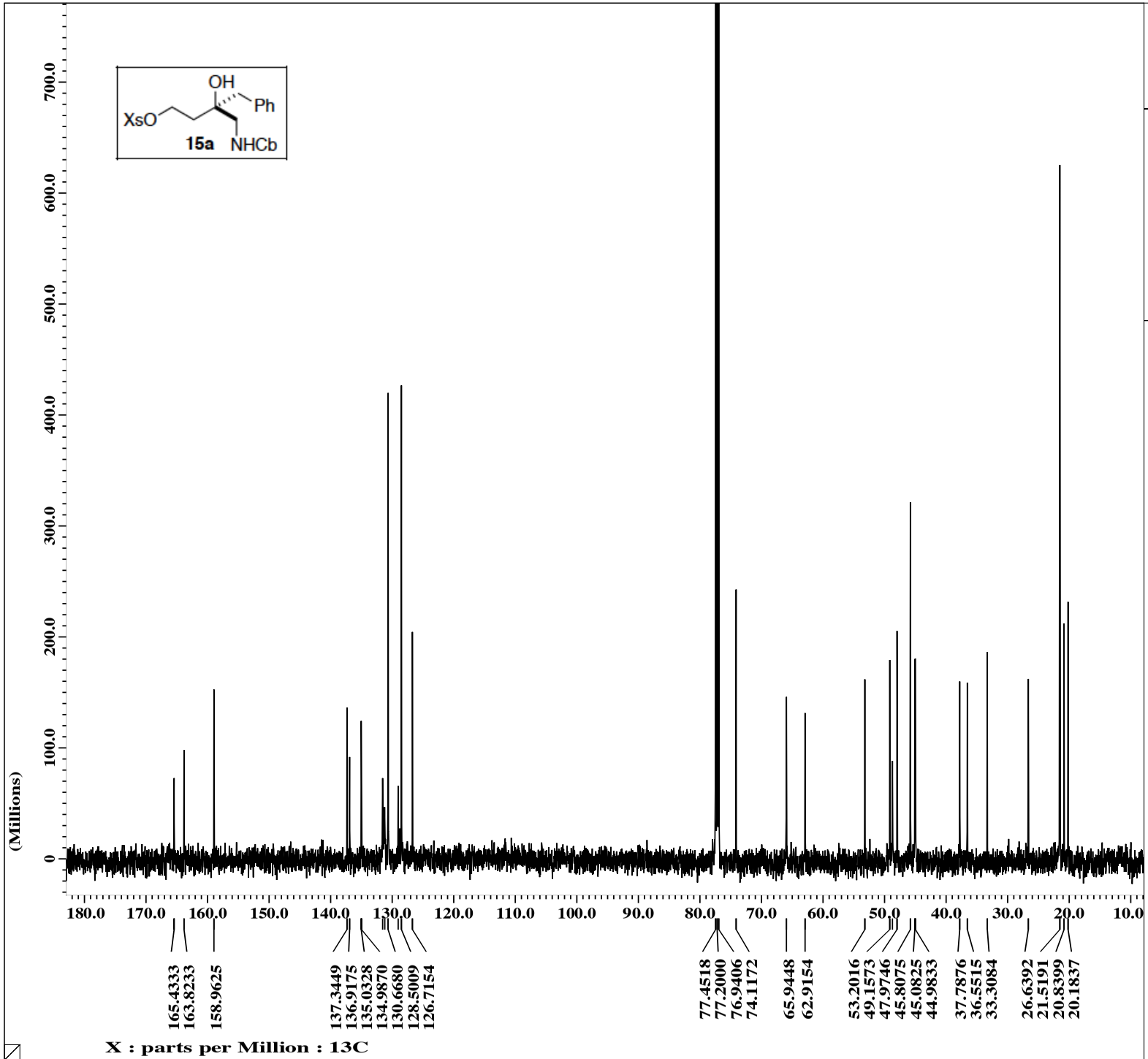
FileName	:	MT20051020001c2.nmdata
Comment	:	MT20051020001_E1133114C
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	27100.3 Hz
FILTR	:	13550 Hz
DELAY	:	14.8 usec
DEADT	:	19.8 usec
INTVL	:	36.9 usec
TIMES	:	10000 times
DUMMY	:	4 times
PD	:	1.0000 sec
ACQTM	:	604.5696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.83 Hz
PW1	:	4.65 usec
OBNUC	:	13C
OBFRQ	:	100.40 MHz
OBSET	:	137500.00 Hz
RGAIN	:	26
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRRPW	:	55.0 usec
IRRNS	:	0
SCANS	:	10000 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	30.0 C

MT20051024001_E1133117A//JNM-LA400//N.Fukui



Date : Mon Oct 24 15:02:18 2005

FileName	:	MT20051024001.nmdata
Comment	:	MT20051024001_E1133117A
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	8000.0 Hz
FILTR	:	7000 Hz
DELAY	:	28.6 usec
DEADT	:	39.5 usec
INTVL	:	125.0 usec
TIMES	:	32 times
DUMMY	:	4 times
PD	:	5.0000 sec
ACQTM	:	2048.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.75 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	136000.00 Hz
RGAIN	:	18
SCANS	:	32 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	30.0 C



JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2[Hz]
fft : 1 : TRUE
machinephase
ppm
```

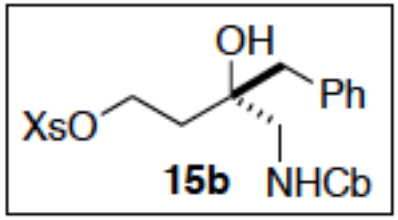
```
---- ACQUISITION PARAMETERS ----
File Name               = E1133117A_CNMR.5
Author                  =
Sample ID               = 1
Content                 = E1133117A_CNMR
Creation Date           = 15-NOV-2005 00:42

Revision Date           = 15-NOV-2005 07:06
Spec Site               = ECP500

Spec Type               = DELTA_NMR
Data Format             = 1D COMPLEX
Dimensions              = X
Dim Title               = 13C
Dim Size                = 32768
Dim Units               = [ppm]
Scans                   = 1000
Mod_return              = 1
X_points                = 32768
X_prescans              = 4
X_domain                = 13C
X_offset                = 100[ppm]
X_freq                  = 125.76529768[MHz]
X_sweep                 = 31.44654088[kHz]
X_resolution            = 0.95967227[Hz]
Irr_domain              = 1H
Irr_offset              = 5[ppm]
Irr_freq                = 500.15991521[MHz]
X_acq_duration          = 1.0420224[s]
Digital_filter          = FALSE
Filter_factor           = 1
Delay_of_start          = 1[s]
Actual_start_time       = 14-NOV-2005 21:37
Acq_delay               = 27.9[us]
X90                     = 23[us]
Irr90                   = 7.4[us]
Tri90                   = 10[us]
Qua90                   = 10[us]
X90_hi                  = 23[us]
Irr90_hi                = 6[us]
Tri90_hi                = 10[us]
Qua90_hi                = 10[us]
X90_lo                  = 0.165[ms]
Irr90_lo                = 25[us]
Tri90_lo                = 10[us]
Qua90_lo                = 10[us]
Spin_lock_90            = 1[us]
Spin_lock_attn          = 29[dB]
Deut_grad_shim_90       = 1[us]
Deut_grad_shim_attn     = 10[dB]
Adc_card                = 16/1MHz/20
Field_strength          = 11.7473579[T]
Filter_mode             = BUTTERWORTH
```

MT20051025001_E1133117B//JNM-LA400//N.Fukui

8.0225 8.0085 7.5261 7.5145 7.3172 7.3074 7.3032 7.2983 7.2964 7.2873 7.2818 7.2702 7.2659 7.2629 7.2616 7.2592 7.2470 7.2421 7.2329 7.2256 7.2171 4.5612 4.5496 4.5331 4.4555 4.0494 4.0452 3.8272 3.8101 3.7930 3.7759 3.7588 3.4119 3.4046 3.3966 3.3624 3.3398 3.3264 3.3130 2.8214 2.8128 1.9579 1.9401 1.9255 1.9059 1.8895 1.8724 1.4479 1.4186 1.4015 1.2397 1.2256 1.2061 0.9783 0.0000

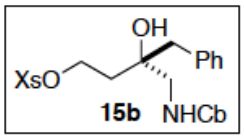


32.31
8.15
1.10
3.61
3.24
4.66
2.00

10 8 6 4 2 0 ppm

Date : Tue Oct 25 17:27:55 2005

FileName	:	MT20051025001.nmdata
Comment	:	MT20051025001_E1133117B
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	16384 points
FREQU	:	8000.0 Hz
FILTR	:	7000 Hz
DELAY	:	28.6 usec
DEADT	:	39.5 usec
INTVL	:	125.0 usec
TIMES	:	32 times
DUMMY	:	4 times
PD	:	5.0000 sec
ACQTM	:	2048.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.75 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	136000.00 Hz
RGAIN	:	20
SCANS	:	32 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	30.0 C



(Millions)

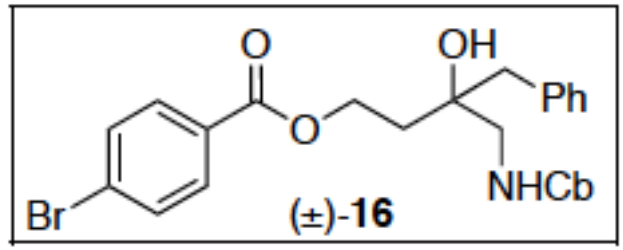
X : parts per Million : 13C

---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2[Hz]
fft : 1 : TRUE
machinephase
dc_correct
ppm
peak_pick : 0[Hz] : 50[Hz] : Both

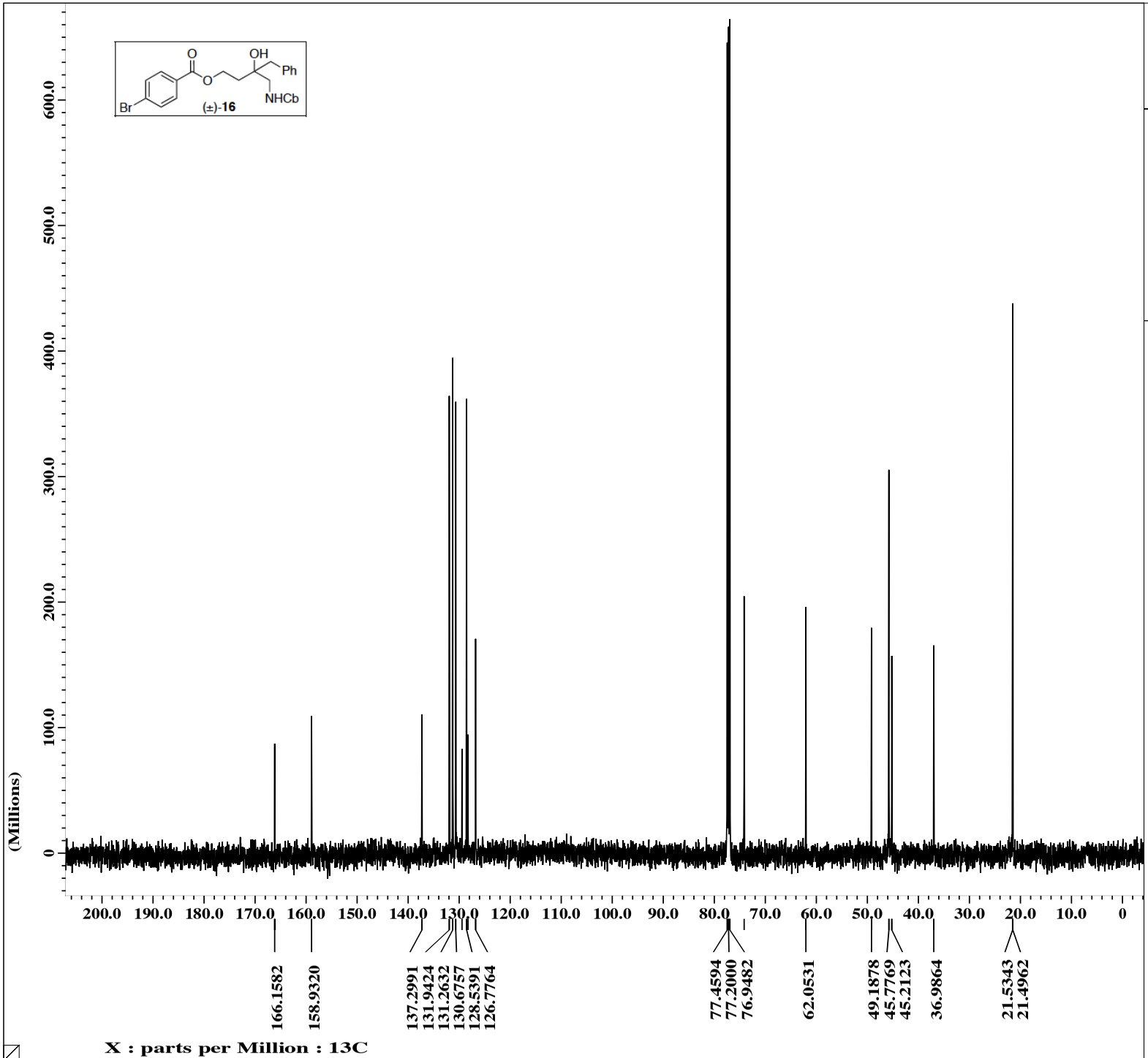
---- ACQUISITION PARAMETERS ----

File Name	= E1133117B.12
Author	=
Sample ID	= 1
Content	= E1133117B
Creation Date	= 9-NOV-2005 00:48
Revision Date	= 9-NOV-2005 09:08
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 1000
Mod_return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_sweep	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
Irr_domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.0420224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 8-NOV-2005 21:43
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 0.165[ms]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH

300M NMR (Gemini 2000)



Comment	AT20050715008 E1133126A CDCl3	**Date**	Jul 15 2005
Nucleus	1H	**Solvent**	CHLOROFORM-D
Frequency (MHz)	300.04	**Number of Transients**	160
Sweep Width (Hz)	5999.00	**Pulse Sequence**	s2pul



---- PROCESSING PARAMETERS ----
dc_balance
sexp : 2[Hz]
fft : 1 : TRUE
machinephase
ppm

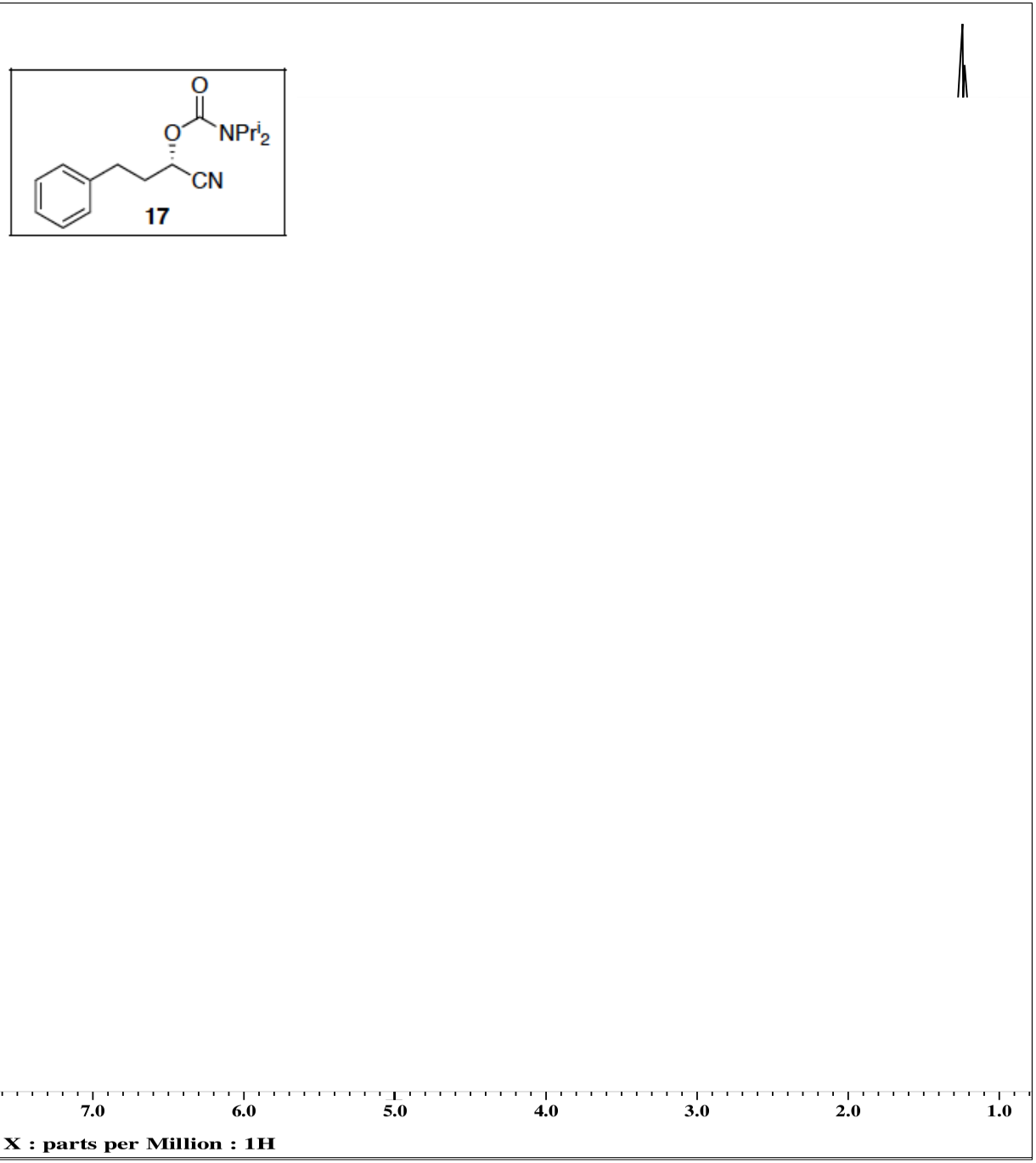
---- ACQUISITION PARAMETERS ----

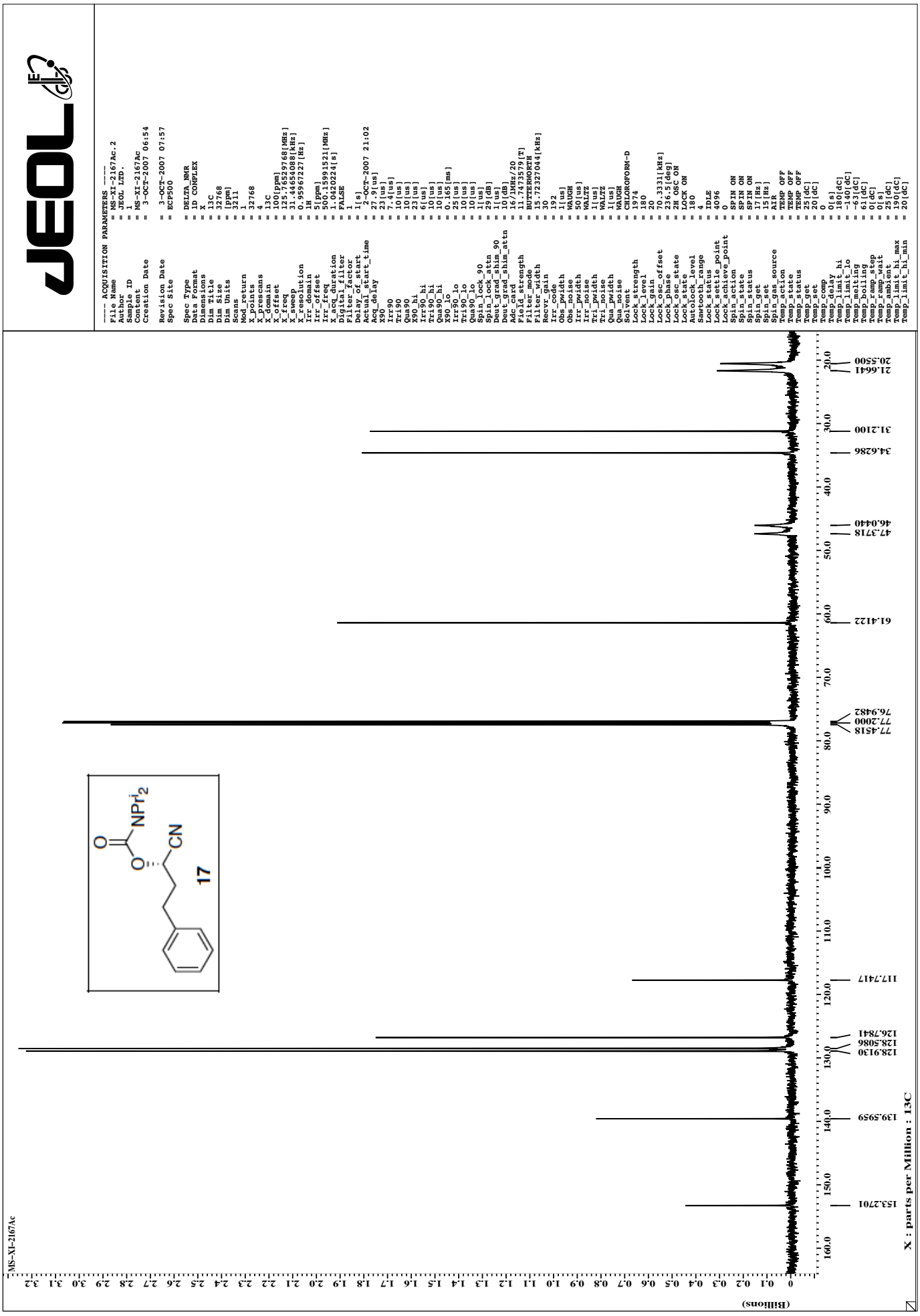
File Name	= E1133126A_CNMR.3
Author	=
Sample ID	= 1
Content	= E1133126A_CNMR
Creation Date	= 5-NOV-2005 22:51
Revision Date	= 5-NOV-2005 22:24
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 711
Mod_return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_sweep	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
Irr_domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.0420224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 5-NOV-2005 22:51
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 0.165[ms]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH

```
---- ACQUISITION PARAMETERS ----
File Name             = MS-XI-2167A.3
Author                =
Sample ID             = 1
Content               = MA-XI-22167A
Creation Date         =  5-OCT-2007 13:34

Revision Date         =  5-OCT-2007 14:57
Spec Site             = ECP500

Spec Type             = DELTA_NMR
Data Format           = 1D COMPLEX
Dimensions            = X
Dim Title             = 1H
Dim Size              = 32768
Dim Units             = [ppm]
Scans                 = 8
Mod_return            = 1
X_points              = 32768
X_prescans            = 0
X_domain              = 1H
X_offset              = 5[ppm]
X_freq                = 500.15991521[MHz]
X_sweep               = 7.50750751[kHz]
X_resolution          = 0.22911095[Hz]
X_acq_duration        = 4.3646976[s]
Digital_filter        = FALSE
Filter_factor         = 1
Delay_of_start        = 1[s]
Actual_start_time     =  5-OCT-2007 13:32
Acq_delay             = 0.1314[ms]
X90                   = 7.4[us]
Irr90                 = 7.4[us]
Tri90                 = 10[us]
Qua90                 = 10[us]
X90_hi                = 6[us]
Irr90_hi              = 6[us]
Tri90_hi              = 10[us]
Qua90_hi              = 10[us]
X90_lo                = 25[us]
Irr90_lo              = 25[us]
Tri90_lo              = 10[us]
Qua90_lo              = 10[us]
Spin_lock_90          = 1[us]
Spin_lock_attn        = 29[dB]
Deut_grad_shim_90     = 1[us]
Deut_grad_shim_attn   = 10[dB]
Adc_card              = 16/1MHz/20
Field_strength        = 11.7473579[T]
Filter_mode           = BUTTERWORTH
Filter_width          = 3.75375375[kHz]
Recvr_gain            = 12
Irr_code              = 192
Obs_pwidth            = 1[us]
Obs_noise             = WAUGH
Irr_pwidth            = 0.165[ms]
Irr_noise             = WALTZ
Tri_pwidth            = 1[us]
Tri_noise             = WALTZ
Qua_pwidth            = 1[us]
Qua_noise             = WAUGH
Solvent               = CHLOROFORM-D
Lock_strength         = 1037
Lock_level            = 180
Lock_gain             = 17
Lock_osc_offset       = 70.3331[kHz]
Lock_phase            = 236.5[deg]
Lock_osc_state        = 2H OSC ON
Lock_state            = LOCK ON
Autolock_level        = 180
```

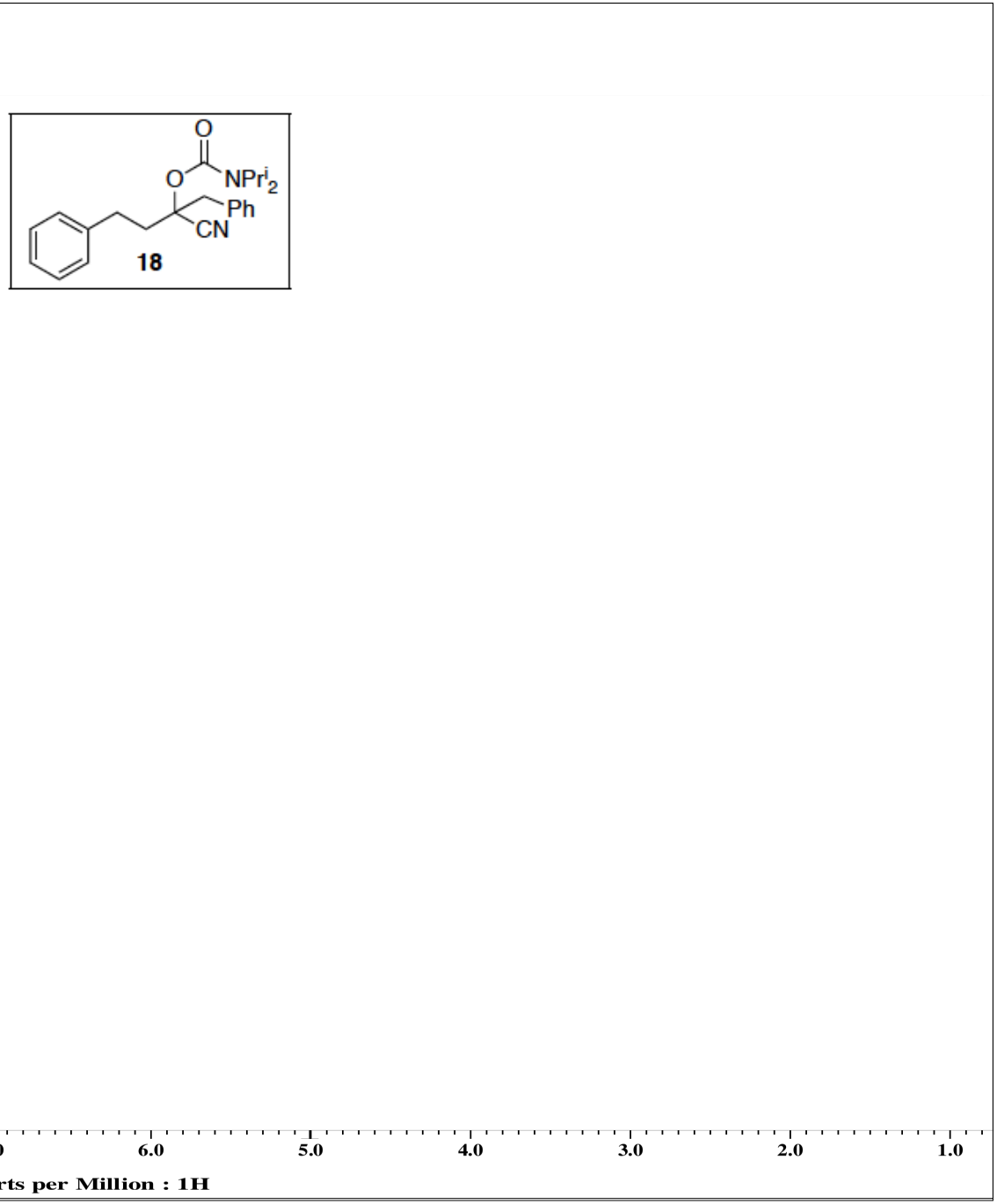


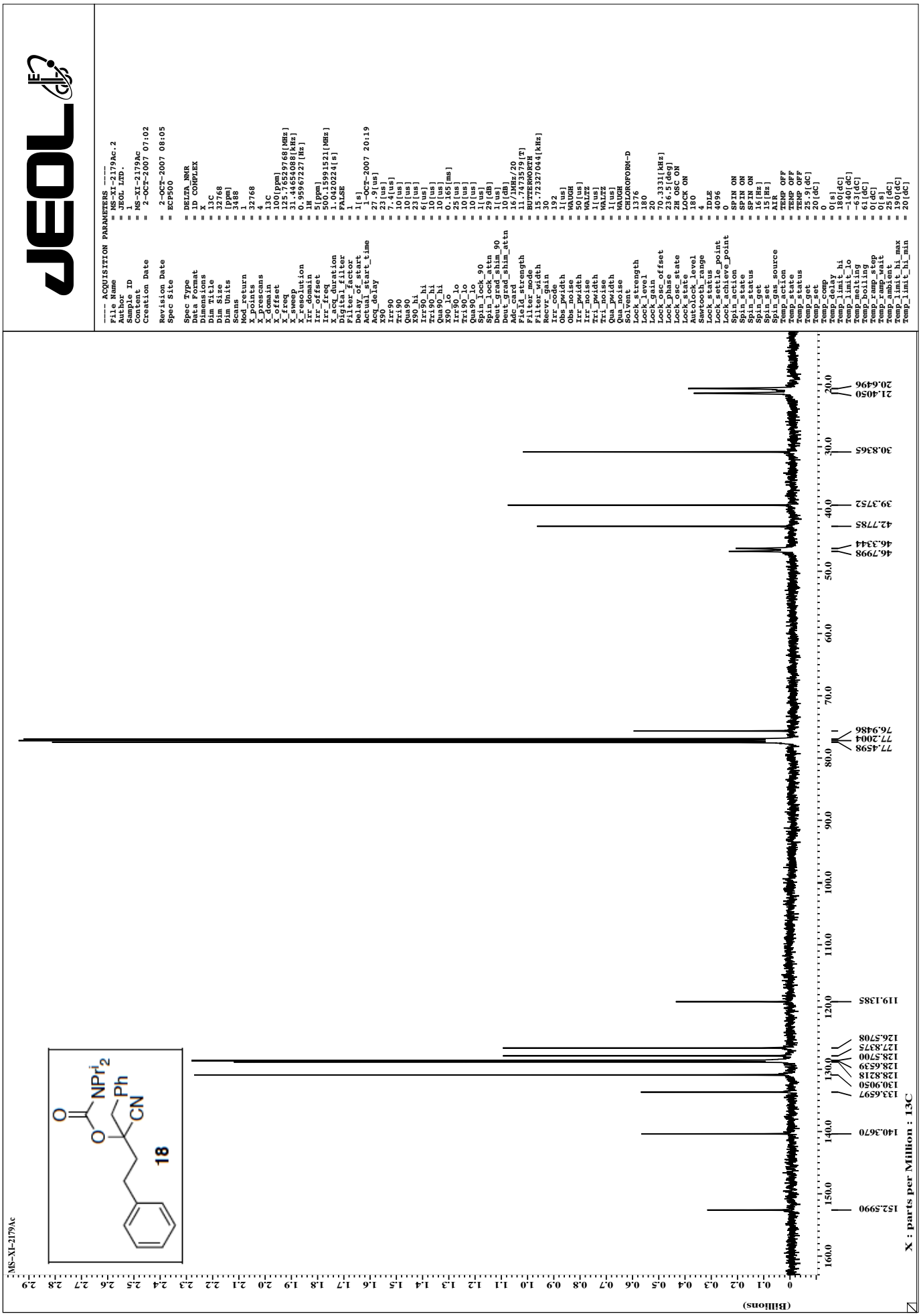

JEOL
--- ACQUISITION PARAMETERS ---
File Name = MS-XI-2167Ac.2
Author = JEOL LTD.
Sample ID = _
Content = MS-XI-2167Ac
Creation Date = 3-OCT-2007 06:54
Revision Date = 3-OCT-2007 07:57
Spec Site = ECP500
Spec Type = DELTA NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Scans = 3211
Mod_return = 32768
X_points = 4
X_prescans = 13C
X_domain = 100[ppm]
X_offset = 125.76529768[MHz]
X_freq = 31.44654088[kHz]
X_sweep = 0.95967227[Hz]
X_resolution = 1H
Irr_domain = 1.[ppm]
Irr_offset = 500.15991521[MHz]
Irr_freq = 1.04220224[s]
X_acq_duration = FALSE
Digital_filter = 1
Filter_factor = 1[s]
Delay_of_start = 2-OCT-2007 21:02
Actual_start_time = 23.9[us]
X90 = 7.4[us]
Irr90 = 10[us]
Tri90 = 10[us]
Qua90 = 23[us]
X90_hi = 6[us]
Irr90_hi = 10[us]
Tri90_hi = 10[us]
Qua90_hi = 165[ms]
X90_lo = 25[us]
Irr90_lo = 10[us]
Tri90_lo = 10[us]
Qua90_lo = 1[us]
Spin_lock_90 = 29[dB]
Spin_lock_attn = 1[us]
Deut_grad_shim_90 = 10[dB]
Deut_grad_shim_attn = 16/1WHz/20
Adc_coef = 11.74735579[T]
Field_strength = BUTTERWORTH
Filter_mode = 15.72327044[kHz]
Filter_width = 30
Recvr_gain = 192
Irr_code = 1[us]
Obs_pwidth = WALTZ
Obs_noise = 50[us]
Irr_pwidth = WALTZ
Irr_noise = 1[us]
Tri_pwidth = WALTZ
Tri_noise = 1[us]
Qua_pwidth = WALTZ
Qua_noise = CHLOROFORM-D
Solvent = 1.74
Lock_strength = 180
Lock_level = 20
Lock_gain = 70.3331[kHz]
Lock_osc_offset = 236.5[deg]
Lock_phase = 2H OSC ON
Lock_osc_state = LOCK ON
Lock_state = 180
AutoLock_level = 4
Search_range = IDLE
Lock_status = 4096
Lock_settle_point = 0
Lock_achieve_point = SPIN ON
Spin_action = SPIN ON
Spin_status = SPIN ON
Spin_get = 15[Hz]
Spin_set = 15[Hz]
Spin_gas_source = AIR
Temp_action = TEMP OFF
Temp_state = TEMP OFF
Temp_status = TEMP OFF
Temp_get = 25[dC]
Temp_set = 20[dC]
Temp_comp = 0
Temp_delay = 0[s]
Temp_limit_hi = 180[dC]
Temp_limit_lo = -140[dC]
Temp_melting = -63[dC]
Temp_boiling = 61[dC]
Temp_ramp_step = 0[dC]
Temp_ramp_wait = 0[s]
Temp_limit_hi_max = 190[dC]
Temp_limit_hi_min = 20[dC]
MS-XI-2167Ac
(Billions)
X : parts per Million : 13C
153.2701
139.5959
128.9130
128.5086
126.7841
117.7417
77.4518
77.2000
76.9482
61.4122
47.3718
46.0440
34.6286
31.2100
21.6641
20.5500
O
NPri2
O
CN
17

```
---- ACQUISITION PARAMETERS ----
File Name             = MS-XI-2179A.4
Author                =
Sample ID             = 1
Content               = MA-XI-2179A
Creation Date         =  5-OCT-2007 13:46

Revision Date         =  5-OCT-2007 14:55
Spec Site             = ECP500

Spec Type             = DELTA_NMR
Data Format           = 1D COMPLEX
Dimensions            = X
Dim Title             = 1H
Dim Size              = 32768
Dim Units             = [ppm]
Scans                 = 16
Mod_return            = 1
X_points              = 32768
X_prescans            = 0
X_domain              = 1H
X_offset              = 5[ppm]
X_freq                = 500.15991521[MHz]
X_sweep               = 7.50750751[kHz]
X_resolution          = 0.22911095[Hz]
X_acq_duration        = 4.3646976[s]
Digital_filter        = FALSE
Filter_factor         = 1
Delay_of_start        = 1[s]
Actual_start_time     =  5-OCT-2007 13:43
Acq_delay             = 0.1314[ms]
X90                   = 7.4[us]
Irr90                 = 7.4[us]
Tri90                 = 10[us]
Qua90                 = 10[us]
X90_hi                = 6[us]
Irr90_hi              = 6[us]
Tri90_hi              = 10[us]
Qua90_hi              = 10[us]
X90_lo                = 25[us]
Irr90_lo              = 25[us]
Tri90_lo              = 10[us]
Qua90_lo              = 10[us]
Spin_lock_90          = 1[us]
Spin_lock_attn        = 29[dB]
Deut_grad_shim_90     = 1[us]
Deut_grad_shim_attn   = 10[dB]
Adc_card              = 16/1MHz/20
Field_strength        = 11.7473579[T]
Filter_mode           = BUTTERWORTH
Filter_width          = 3.75375375[kHz]
Recvr_gain            = 13
Irr_code              = 192
Obs_pwidth            = 1[us]
Obs_noise             = WAUGH
Irr_pwidth            = 0.165[ms]
Irr_noise             = WALTZ
Tri_pwidth            = 1[us]
Tri_noise             = WALTZ
Qua_pwidth            = 1[us]
Qua_noise             = WAUGH
Solvent               = CHLOROFORM-D
Lock_strength         = 1019.0
Lock_level            = 180
Lock_gain             = 17
Lock_osc_offset       = 70.3331[kHz]
Lock_phase            = 236.5[deg]
Lock_osc_state        = 2H OSC ON
Lock_state            = LOCK ON
Autolock_level        = 180
```




JEOL
--- ACQUISITION PARAMETERS ---
File Name = MS-XI-2179Ac.2
Author = JEOL LTD.
Sample ID = MS-XI-2179Ac
Content = MS-XI-2179Ac
Creation Date = 2-OCT-2007 07:02
Revision Date = 2-OCT-2007 08:05
Spec Site = ECP500
Spec Type = DELTA NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Scans = 3488
Mod_return = 32768
X_points = 4
X_prescans = 13C
X_domain = 100[ppm]
X_offset = 125.76529768[MHz]
X_freq = 31.44654088[kHz]
X_sweep = 0.95967227[Hz]
X_resolution = 1H
Irr_domain = 1.[ppm]
Irr_freq = 500.15991521[MHz]
X_acq_duration = 1.04202224[s]
Digital_filter = FALSE
Filter_factor = 1
Delay_of_start = 1[s]
Actual_start_time = 1-OCT-2007 20:19
Acq_delay = 23.9[us]
X90 = 7.4[us]
Irr90 = 10[us]
Tri90 = 10[us]
Qua90 = 10[us]
X90_hi = 23[us]
Irr90_hi = 6[us]
Tri90_hi = 10[us]
Qua90_hi = 10[us]
X90_lo = 165[ms]
Irr90_lo = 25[us]
Tri90_lo = 10[us]
Qua90_lo = 10[us]
Spin_lock_90 = 1[us]
Spin_lock_attn = 29[dB]
Dec_grad_shim_90 = 1[us]
Dec_grad_shim_attn = 16[dB]
Adc_coef = 16/1MHz/20
Field_strength = 11.74735579[T]
Filter_mode = BUTTERWORTH
Filter_width = 15.72327044[kHz]
Recvr_gain = 30
Irr_code = 192
Irr_width = 1[us]
Obs_pwidth = WALTZ
Irr_pwidth = 50[us]
Irr_noise = WALTZ
Tri_pwidth = 1[us]
Tri_noise = WALTZ
Qua_pwidth = 1[us]
Qua_noise = WALTZ
Solvent = CHLOROFORM-D
Lock_strength = 136
Lock_level = 180
Lock_gain = 20
Lock_osc_offset = 70.3331[kHz]
Lock_phase = 236.5[deg]
Lock_osc_state = 2H OSC ON
Lock_state = LOCK ON
Autolock_level = 180
Autolock_range = 4
Lock_status = IDLE
Lock_settle_point = 4096
Lock_achieve_point = 0
Spin_action = SPIN ON
Spin_status = SPIN ON
Spin_set = 15[Hz]
Spin_get = 15[Hz]
Spin_gas_source = AIR
Temp_action = TEMP OFF
Temp_state = TEMP OFF
Temp_status = TEMP OFF
Temp_get = 25.9[dC]
Temp_set = 20[dC]
Temp_comp = 0
Temp_delay = 0[s]
Temp_limit_hi = 180[dC]
Temp_limit_lo = -140[dC]
Temp_melting = -63[dC]
Temp_boiling = 61[dC]
Temp_ramp_step = 0[dC]
Temp_ramp_wait = 0[s]
Temp_limit_hi_max = 190[dC]
Temp_limit_hi_min = 20[dC]
MS-XI-2179Ac
O
O
NPr$^i_2$
Ph
CN
18
(Billions)
X : parts per Million : 13C
152.5990
140.3670
133.6597
130.9050
128.8218
128.6539
128.5700
127.8375
126.5708
119.1385
77.4598
77.2004
76.9486
46.7998
46.3344
42.7785
39.3752
30.8365
21.4050
20.6496

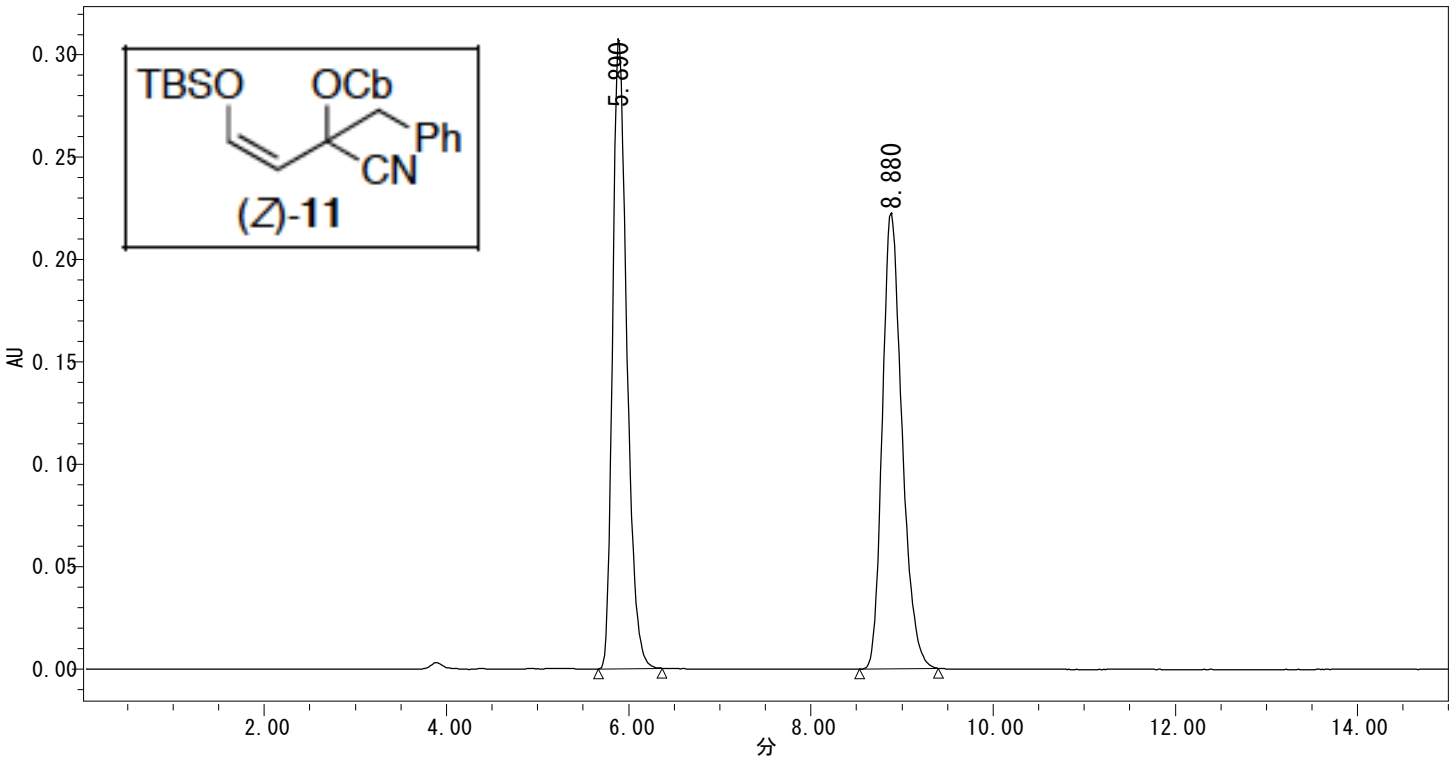
分析担当者 ohno　　プロジェクト名 2005_01_03B

HPLC 測定結果

サンプル名： KT20050216003
E1109015A
バイアル： 3
注入＃： 1
注入量： 2.00 μl
分析時間： 15.00 分

分析担当者： ohno
分析日： 2005/02/16 11:40:54
取り込みメソッドセット :chiral_N_03
解析日： 2005/02/16 12:56:21
チャンネル名： W2996 210.0nm-1.2
解析チャンネルの説明： W2996 PDA 210.0 nm at 1.2

機器名／資産No. Waters 2695 ／ 1517310　　ｶﾗﾑ名 CHIRALPAK AD-H (4.6 x 150mm)
移動相 Hexane/2-PrOH (97/3)
流量 0.500 ml/mi　　ｶﾗﾑ温度 25℃　　Sample 1mg/ml Hexane



	成分名	保持時間 (分)	面積 (μV秒)	%面積	高さ (μV)
1		5.890	3348079	50.009	309318
2		8.880	3346846	49.991	222878

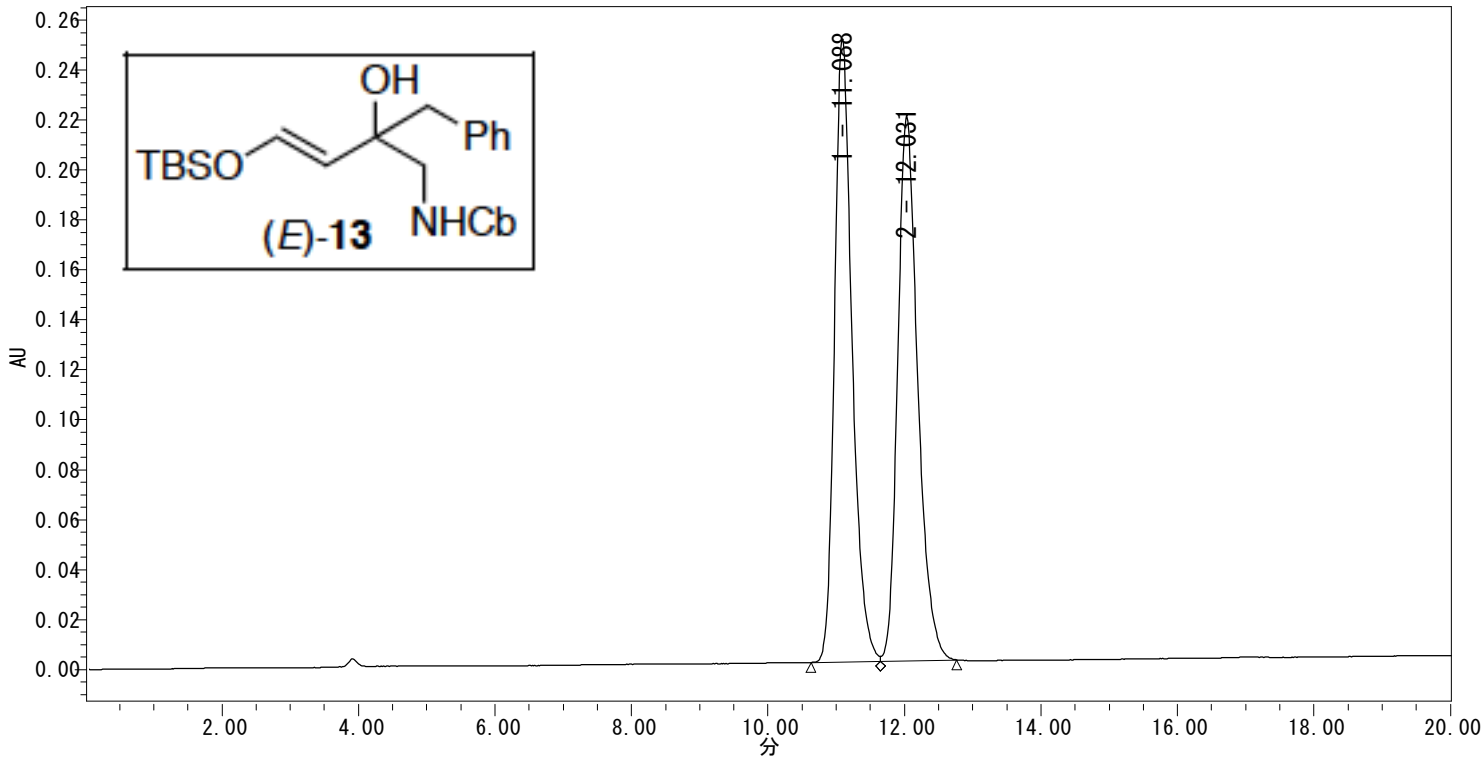
分析担当者 ohno　　プロジェクト名 2005_04_06B

HPLC 測定結果

サンプル名： KT20050401001
E1133002A
バイアル： 12
注入＃： 1
注入量： 2.00 μl
分析時間： 20.00 分

分析担当者： ohno
分析日： 2005/04/01 14:51:51
取り込みメソッドセット :chiral_N_11
解析日： 2005/04/01 15:12:40
チャンネル名： 波長 Ch 1
解析チャンネルの説明： PDA 210.0 nm

機器名／資産No. Waters 2695 ／ 1517310　　ｶﾗﾑ名 CHIRALPAK AD-H (4.6 x 150mm)
移動相 Hexane/2-PrOH (97/3)
流量 0.500 ml/mi　　ｶﾗﾑ温度 25℃　　Sample 1mg/ml Hexane



	成分名	保持時間 (分)	面積 (μV秒)	%面積	高さ (μV)
1	1	11.088	4477697	50.10C	249998
2	2	12.031	4459735	49.90C	218536

SIC 480II FOR WINDOWS TEST REPORT

ファイル名: MSXI2238C.CHR
試料注入,日付:12-28-2007　時間:11:53:18

Analysis method:　　Instrument:
Column :AD-H25cm
Column Temperature (-C) :
Detector :UV
Internal Standard :
Mobile Phase :
 Flow rate(ml/Min.) :0.7　　Attenuation :
Solvent A :hexane97
　　B :IPA3
　　C :
　　D :

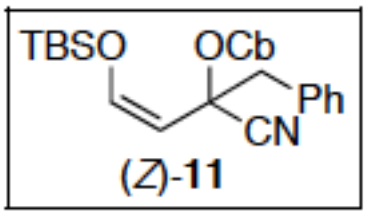
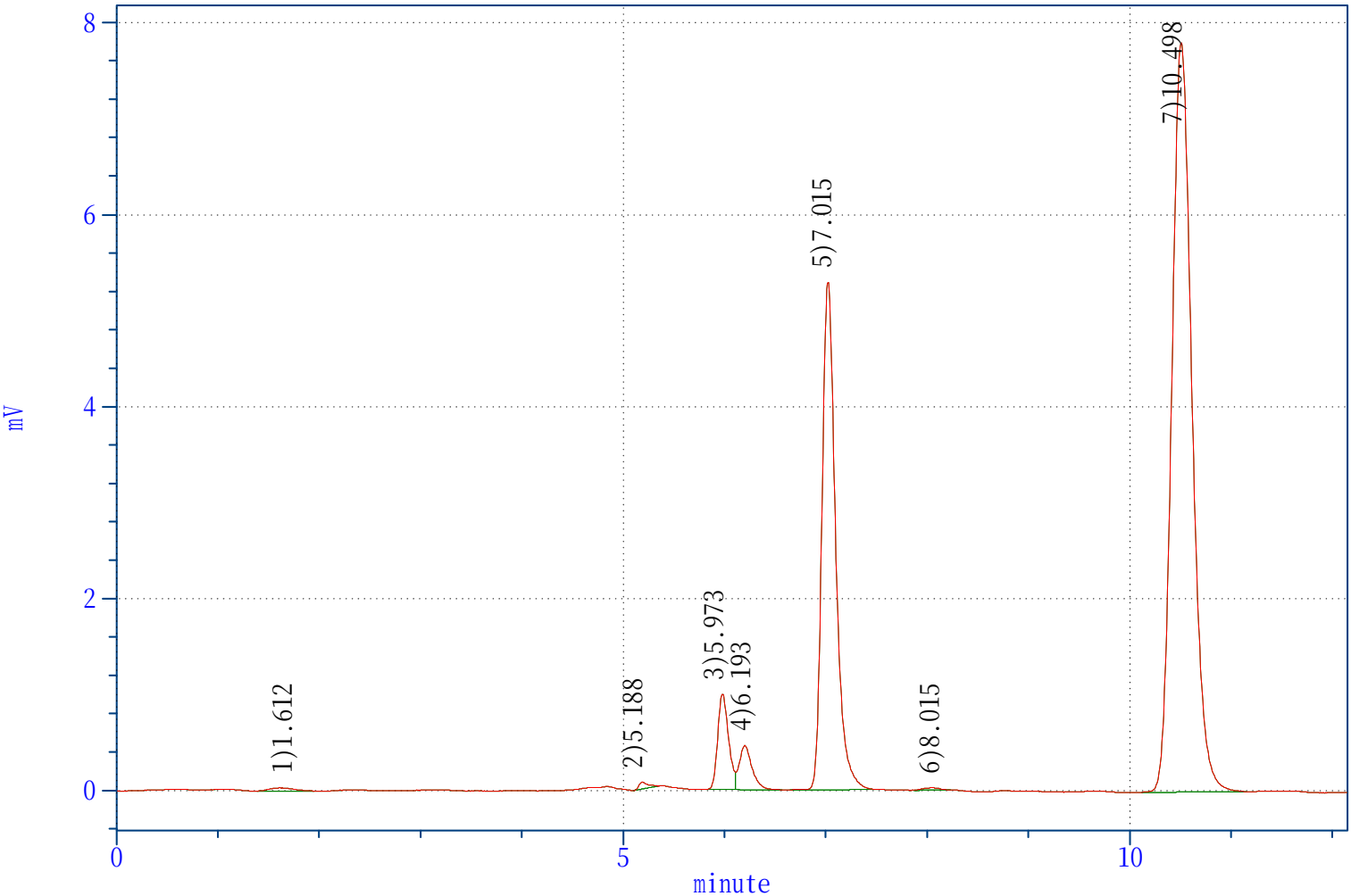


Table 2
from **10a** (Et_2O, 1 min)

Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:

データ処理パラメータ
アップスロープ= 5,ダウンスロープ= 5,最小ピーク幅= 10.00 sec , ベース感度= 5 points
計算範囲= 0.000 to 2000.000 min.,最小ピーク面積= 200 uv*sec , ベースライン補正=0

No.	R.T. (分)	面積 (uv*sec)	面積%	高さ (uv)	高さ%	濃度	濃度%	濃度単位	処理マーク	理論段数	ピーク名
1	1.612	565	0.3336	35	0.2378	0.0000	0.0000		PP	200	
2	5.188	430	0.2539	68	0.4641	0.0000	0.0000		PP	22784	
3	5.973	7338	4.3330	993	6.7653	0.0000	0.0000		PV	0	
4	6.193	4096	2.4186	461	3.1425	0.0000	0.0000		VP	0	
5	7.015	49195	29.0491	5289	36.0301	0.0000	0.0000		PP	14422	
6	8.015	293	0.1730	26	0.1777	0.0000	0.0000		PP	9911	
7	10.498	107434	63.4387	7807	53.1825	0.0000	0.0000		PP	13820	
	合計	169351		14679		0.0000	0.0000				



SIC 480II FOR WINDOWS TEST REPORT

ファイル名: MS-XI-2239B2.CHR
試料注入,日付:12-28-2007　時間:10:56:29

Analysis method:　　Instrument:
Column :AD-H25cm
Column Temperature (-C) :
Detector :UV
Internal Standard :
Mobile Phase :
 Flow rate(ml/Min.) :0.5　　Attenuation :
Solvent A :hexane97
　　B :IPA3
　　C :
　　D :

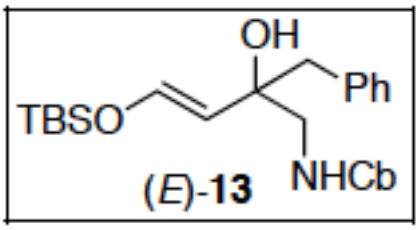
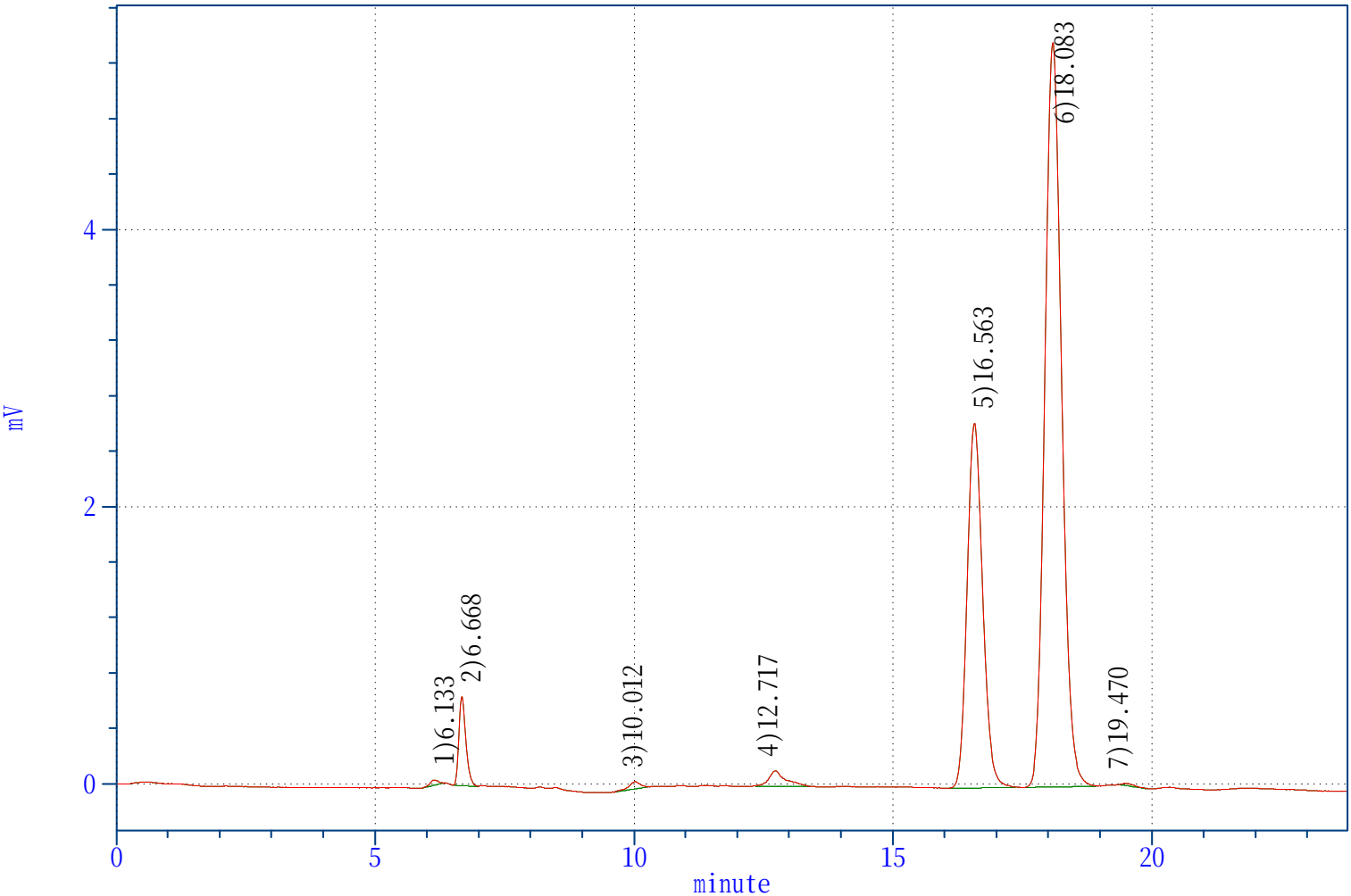


Table 2
from**10a** (Et_2O, 1 min)

Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:

データ処理パラメータ
アップスロープ= 5,ダウンスロープ= 5,最小ピーク幅= 10.00 sec , ベース感度= 5 points
計算範囲= 0.000 to 2000.000 min.,最小ピーク面積= 200 uv*sec , ベースライン補正=0

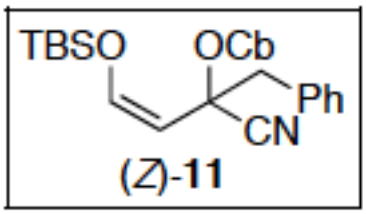
No.	R.T. (分)	面積 (uv*sec)	面積%	高さ (uv)	高さ%	濃度	濃度%	濃度単位	処理マーク	理論段数	ピーク名
1	6.133	416	0.2257	38	0.4313	0.0000	0.0000		PP	7436	
2	6.668	5942	3.2234	639	7.2175	0.0000	0.0000		PP	12296	
3	10.012	800	0.4340	52	0.5924	0.0000	0.0000		PP	10808	
4	12.717	2564	1.3909	112	1.2628	0.0000	0.0000		PP	10917	
5	16.563	54348	29.4825	2632	29.7148	0.0000	0.0000		PP	15146	
6	18.083	120014	65.1047	5370	60.6228	0.0000	0.0000		PP	15275	
7	19.470	256	0.1389	14	0.1583	0.0000	0.0000		PP	24867	
	合計	184340		8858		0.0000	0.0000				



SIC 480II FOR WINDOWS TEST REPORT

ファイル名: MSXI2242CIA.CHR
試料注入,日付:12-29-2007　時間:13:00:25

Analysis method:　　Instrument:
Column :IA25cm
Column Temperature (-C) :
Detector :UV
Internal Standard :
Mobile Phase :
 Flow rate(ml/Min.) :1.0　　Attenuation :
Solvent A :hexane97
　　B :IPA3
　　C :
　　D :



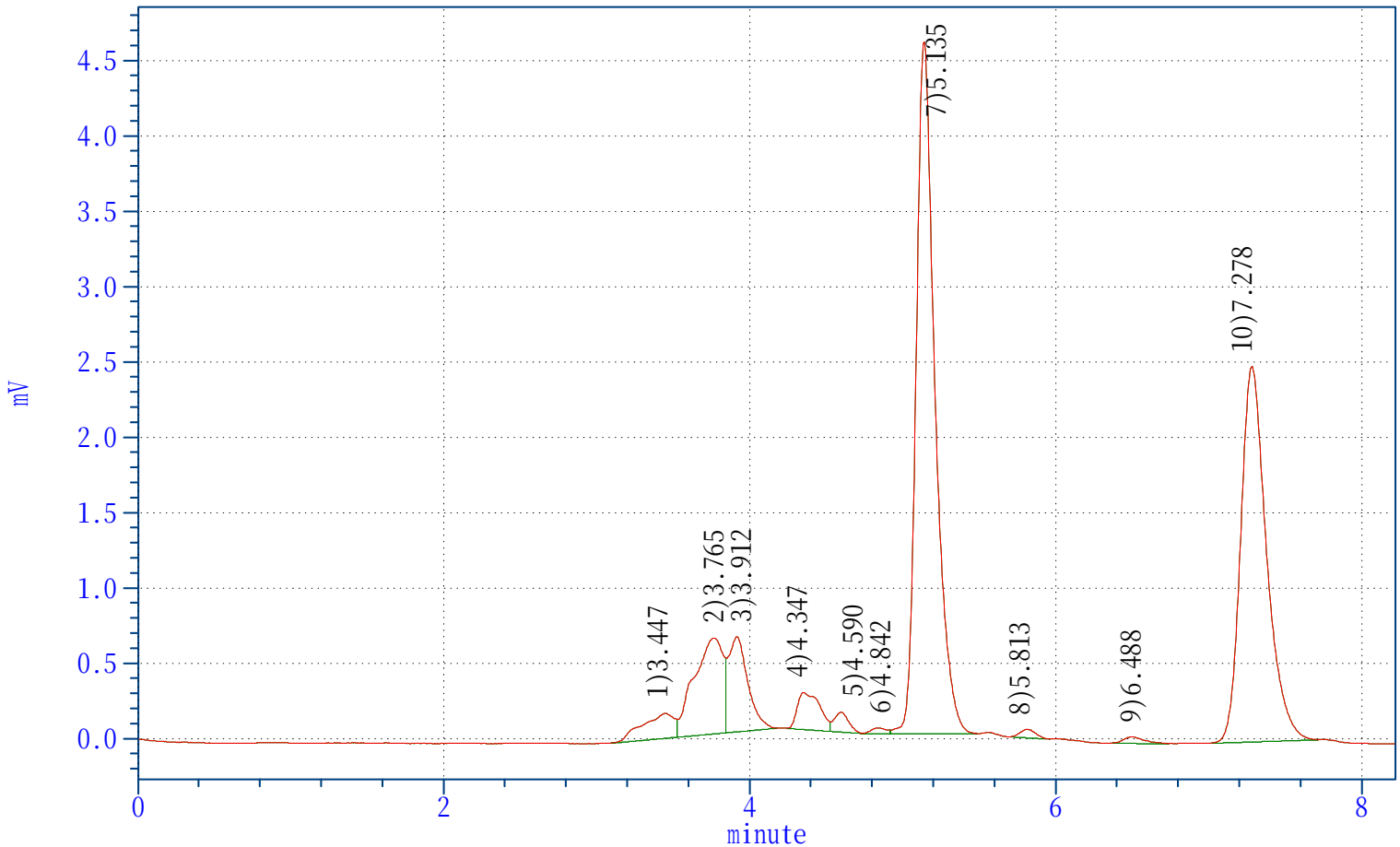
(*Z*)-**11**

Table 2
from **10b** (toluene, 60 min)

Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:

データ処理パラメータ
アップスロープ= 5,ダウンスロープ= 5,最小ピーク幅= 10.00 sec , ベース感度= 5 points
計算範囲= 0.000 to 2000.000 min.,最小ピーク面積= 200 uv*sec , ベースライン補正=0

No.	R.T. (分)	面積 (uv*sec)	面積%	高さ (uv)	高さ%	濃度	濃度%	濃度単位	処理マーク	理論段数	ピーク名
1	3.447	2425	2.7221	165	1.8242	0.0000	0.0000		PV	0	
2	3.765	8349	9.3721	637	7.0546	0.0000	0.0000		VV	0	
3	3.912	5623	6.3120	631	6.9908	0.0000	0.0000		VP	0	
4	4.347	2518	2.8265	245	2.7177	0.0000	0.0000		PV	0	
5	4.590	868	0.9744	133	1.4688	0.0000	0.0000		VP	0	
6	4.842	248	0.2784	37	0.4155	0.0000	0.0000		PV	0	
7	5.135	39053	43.8384	4588	50.8359	0.0000	0.0000		VP	0	
8	5.813	401	0.4501	57	0.6334	0.0000	0.0000		PP	14684	
9	6.488	417	0.4681	43	0.4802	0.0000	0.0000		PP	11090	
10	7.278	29182	32.7578	2489	27.5789	0.0000	0.0000		PP	9687	
	合計	89084		9025		0.0000	0.0000				



SIC 480II FOR WINDOWS TEST REPORT

ファイル名: MSXI2243B2.CHR
試料注入,日付:12-29-2007　時間:12:29:45

Analysis method:　　Instrument:
Column :AD-H25cm
Column Temperature (-C) :
Detector :UV
Internal Standard :
Mobile Phase :
 Flow rate(ml/Min.) :0.7　　Attenuation :
Solvent A :hexane97
　　B :IPA3
　　C :
　　D :

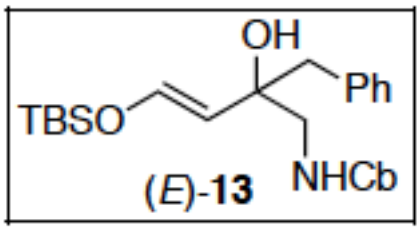
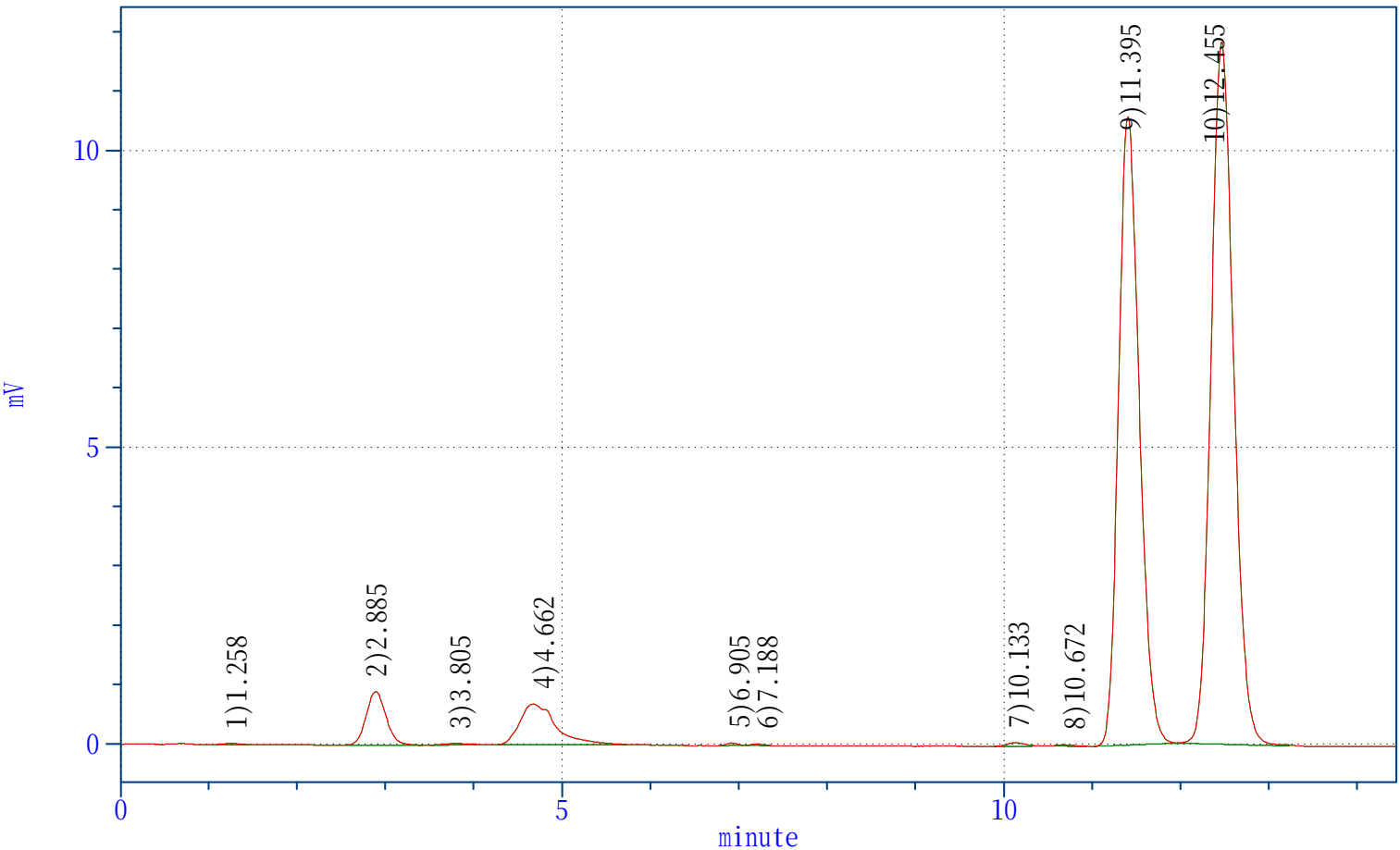


Table 2
from**10b** (toluene, 60 min)

Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:

データ処理パラメータ
アップスロープ= 5,ダウンスロープ= 5,最小ピーク幅= 10.00 sec , ベース感度= 5 points
計算範囲= 0.000 to 2000.000 min.,最小ピーク面積= 200 uv*sec , ベースライン補正=0

No.	R.T. (分)	面積 (uv*sec)	面積%	高さ (uv)	高さ%	濃度	濃度%	濃度単位	処理マーク	理論段数	ピーク名
1	1.258	245	0.0585	20	0.0826	0.0000	0.0000		PP	197	
2	2.885	14037	3.3492	901	3.7271	0.0000	0.0000		PP	804	
3	3.805	394	0.0940	20	0.0835	0.0000	0.0000		PP	679	
4	4.662	19302	4.6054	684	2.8290	0.0000	0.0000		PP	752	
5	6.905	347	0.0828	43	0.1761	0.0000	0.0000		PP	16383	
6	7.188	234	0.0558	26	0.1082	0.0000	0.0000		PP	13347	
7	10.133	871	0.2078	60	0.2501	0.0000	0.0000		PP	11448	
8	10.672	294	0.0701	23	0.0947	0.0000	0.0000		PP	14049	
9	11.395	173282	41.3443	10583	43.7545	0.0000	0.0000		PP	11157	
10	12.455	210113	50.1321	11826	48.8943	0.0000	0.0000		PB	0	
	合計	419119		24186		0.0000	0.0000				



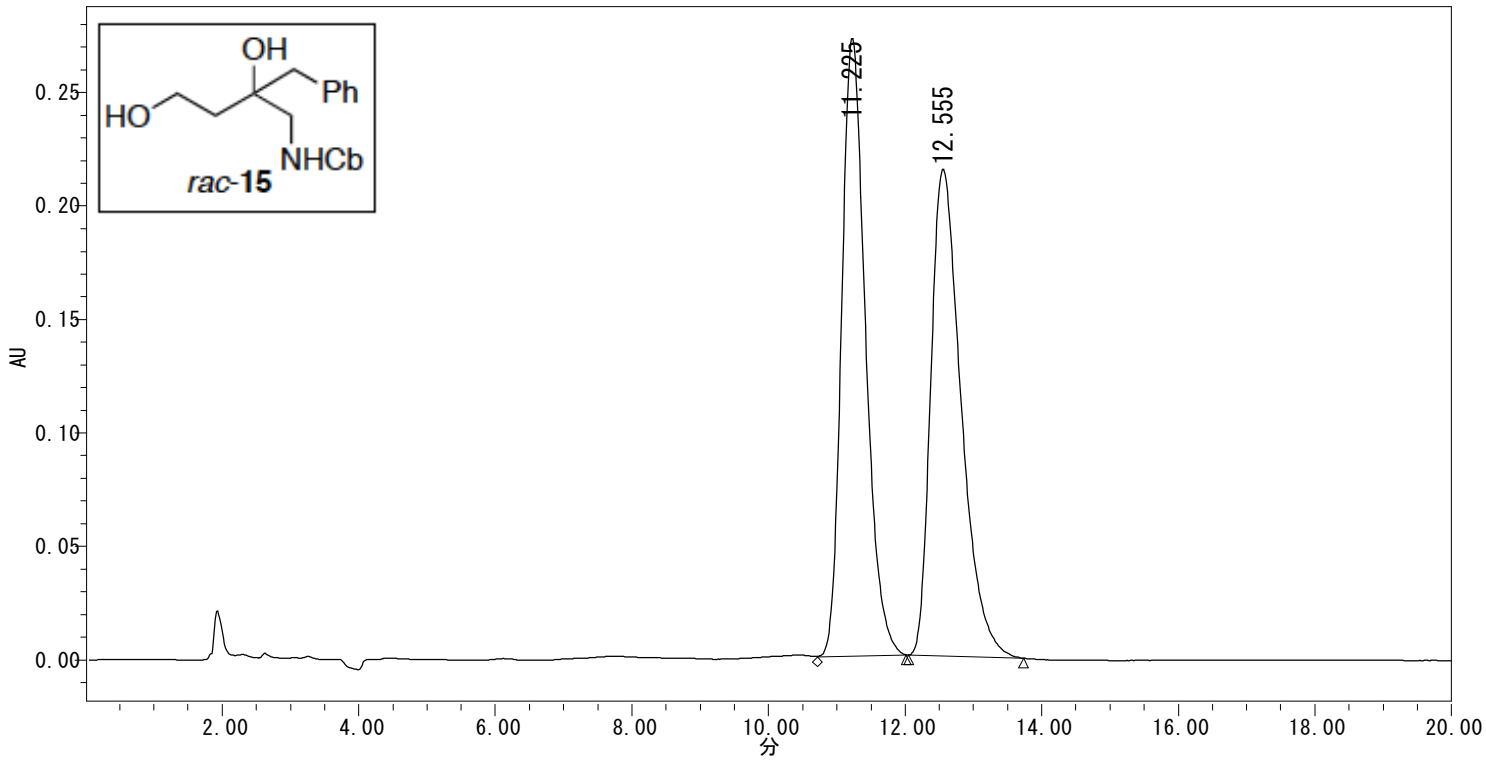
分析担当者 ohno　　プロジェクト名 2005_04_06B

HPLC 測定結果

サンプル名： KT20050414001
E1133020A
バイアル： 1
注入＃： 1
注入量： 5.00 μl
分析時間： 20.00 分

分析担当者： ohno
分析日： 2005/04/15 9:52:16
取り込みメソッドセット :HP_95_5
解析日： 2005/04/15 11:07:02
チャンネル名： 波長 Ch 1
解析チャンネルの説明： PDA 210.0 nm

機器名／資産No. Waters 2695 ／ 1517310　　ｶﾗﾑ名 CHIRALPAK AD-H (4.6 x 150mm)
移動相 Hexane/2-PrOH (95/5)
流量 1.000 ml/mi　　ｶﾗﾑ温度 25℃　　Sample 1mg/ml EtOH



	成分名	保持時間 (分)	面積 (μV秒)	%面積	高さ (μV)
1		11.225	6610097	49.959	272620
2		12.555	6620889	50.041	214693

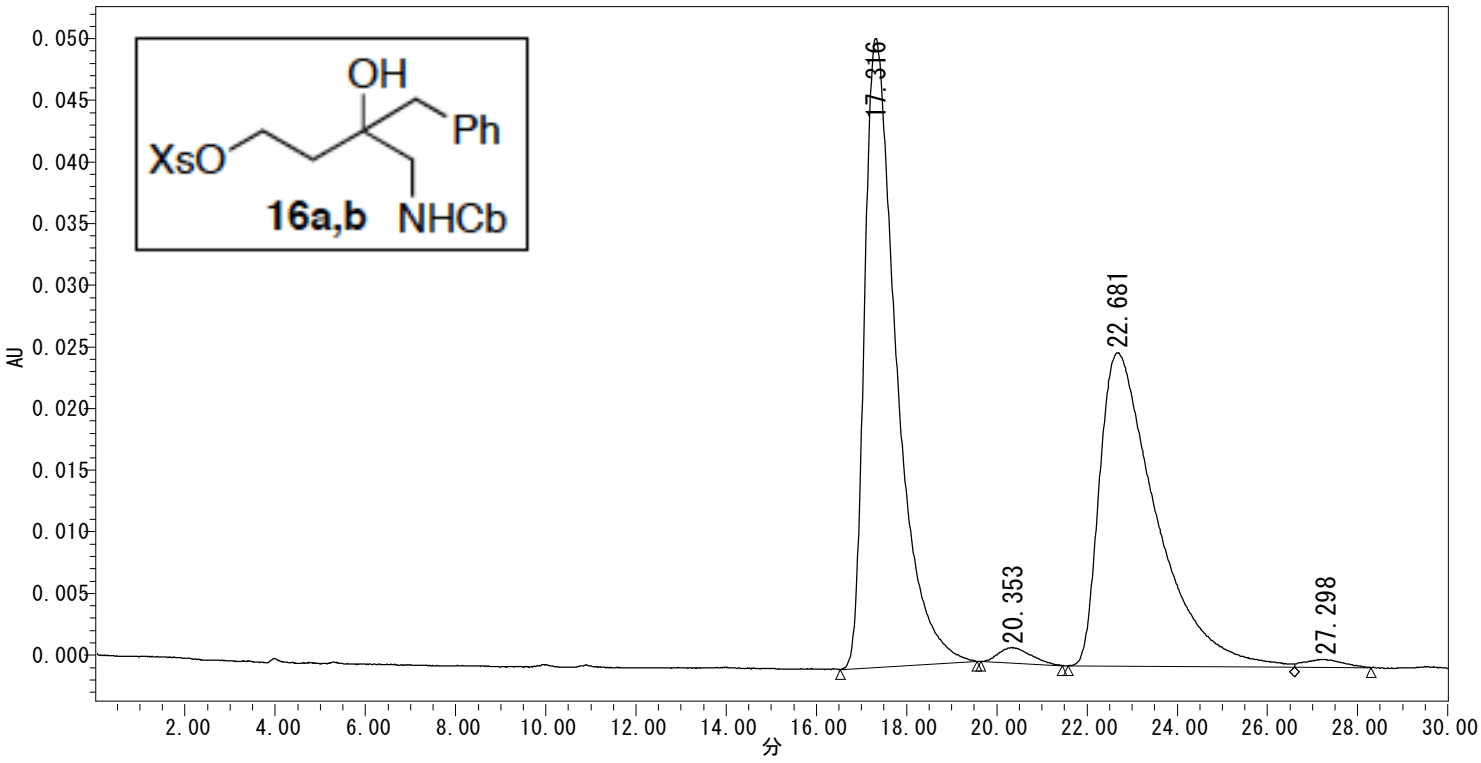
分析担当者 ohno　　プロジェクト名 2005_07_09B

HPLC 測定結果

サンプル名： KT20050706001
E1133116A
バイアル： 1
注入＃： 1
注入量： 10.00 μl
分析時間： 30.00 分

分析担当者： ohno
分析日： 2005/07/06 11:13:10
取り込みメソッドセット :HX05_70_30
解析日： 2005/07/06 11:44:37
チャンネル名： 波長 Ch 1
解析チャンネルの説明： PDA 260.0 nm

機器名／資産No. Waters 2695 ／ 1517310　　ｶﾗﾑ名 CHIRALPAK IA (4.6 x 150mm)
移動相 Hexane/Ethyl Acetate (70/30)
流量 0.500 ml/mi　　ｶﾗﾑ温度 25℃　　Sample 1mg/ml 移動相



	成分名	保持時間 (分)	面積 (μV秒)	%面積	高さ (μV)
1		17.316	2511915	51.998	51013
2		20.353	63539	1.315	1268
3		22.681	2217420	45.902	25394
4		27.298	37877	0.784	626

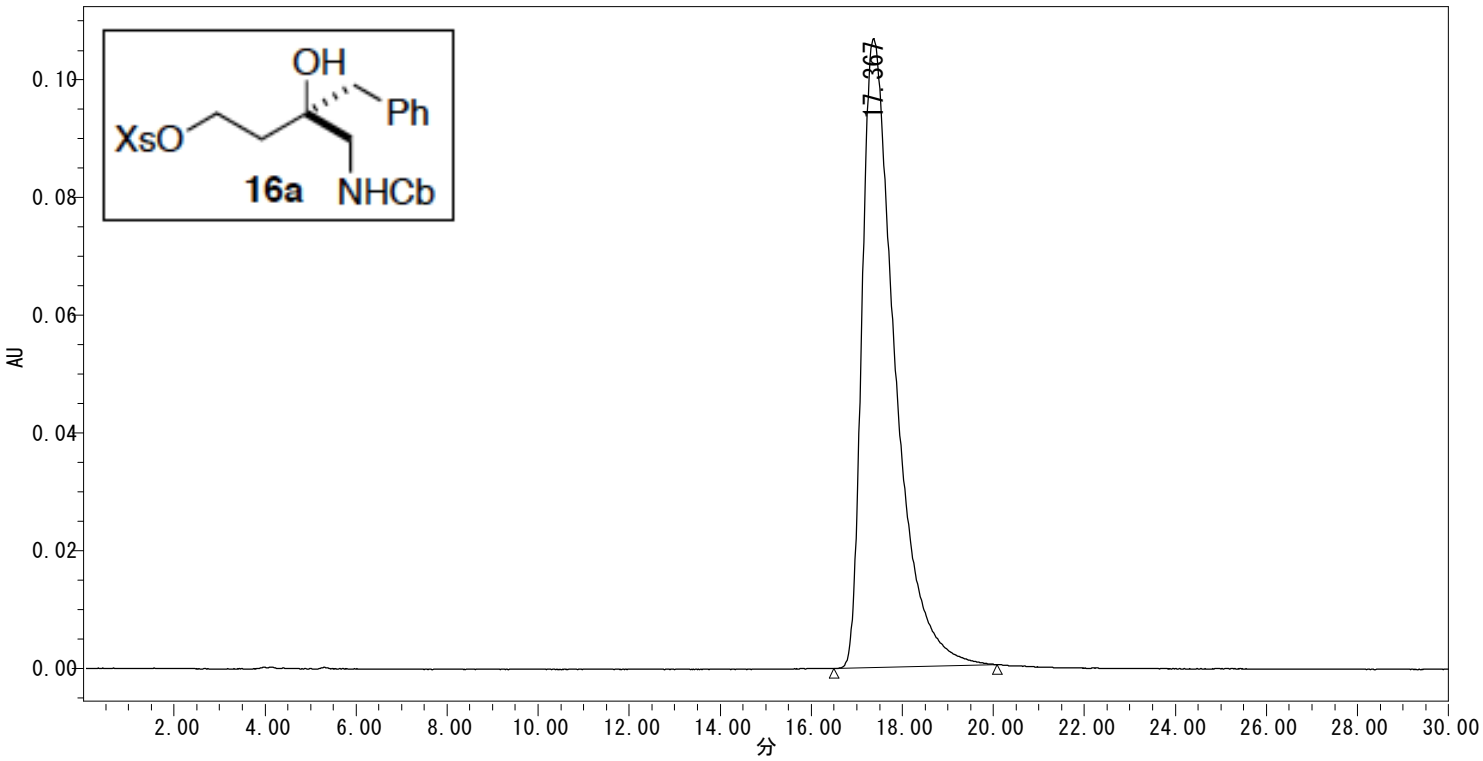
分析担当者 ohno　　プロジェクト名 2005_07_09B

HPLC 測定結果

サンプル名： KT20050715001
E1133117A
バイアル： 1
注入＃： 1
注入量： 10.00 μl
分析時間： 30.00 分

分析担当者： ohno
分析日： 2005/07/15 11:05:57
取り込みメソッドセット :HX05_70_30
解析日： 2005/07/15 11:46:29
チャンネル名： 波長 Ch 1
解析チャンネルの説明： PDA 260.0 nm

機器名／資産No. Waters 2695 ／ 1517310　　ｶﾗﾑ名 CHIRALPAK IA (4.6 x 150mm)
移動相 Hexane/Ethyl Acetate (70/30)
流量 0.500 ml/mi　　ｶﾗﾑ温度 25℃　　Sample 1mg/ml 移動相



	成分名	保持時間 (分)	面積 (μV秒)	%面積	高さ (μV)
1		17.367	5365821	100.000	106931

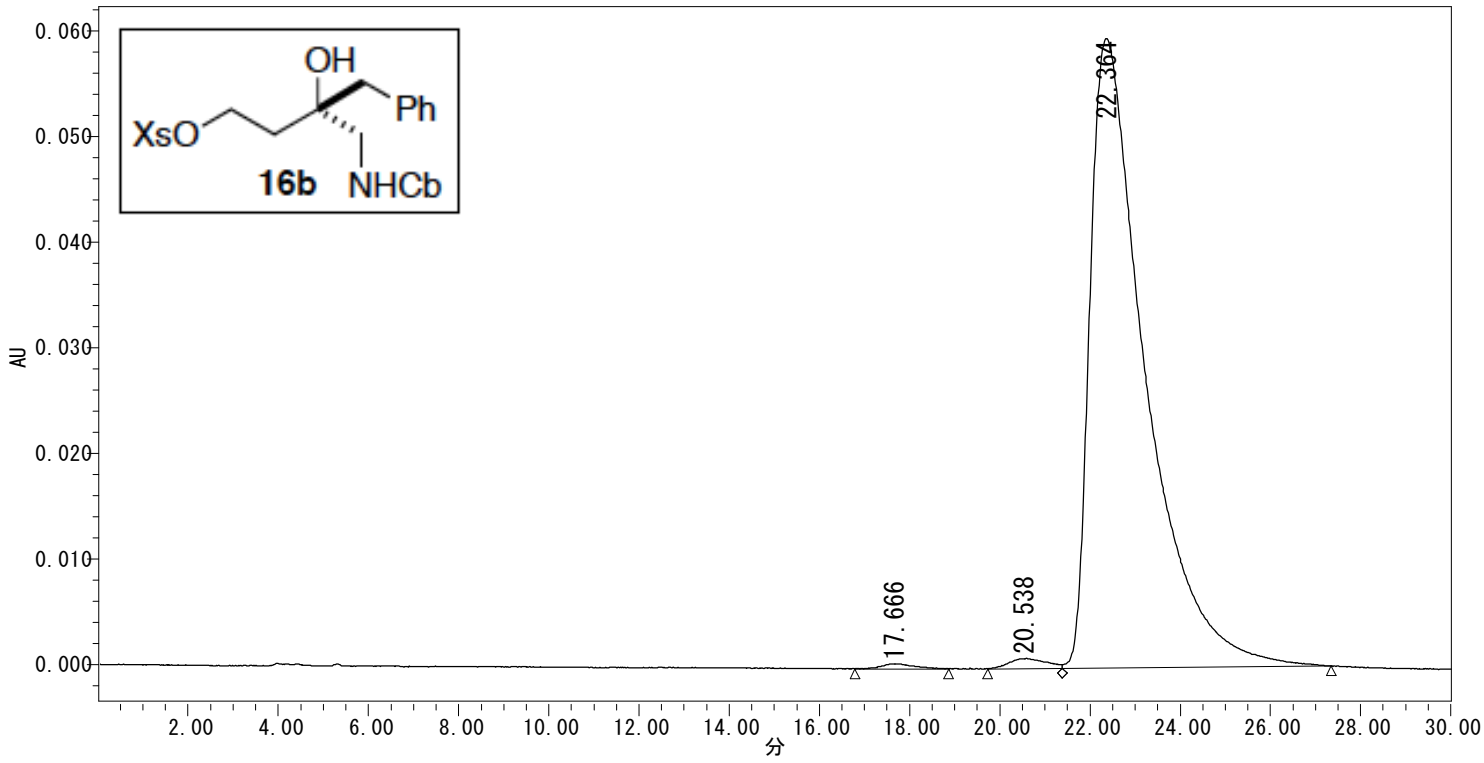
分析担当者 ohno　　プロジェクト名 2005_07_09B

HPLC 測定結果

サンプル名： KT20050715002
E1133117B
バイアル： 2
注入＃： 1
注入量： 10.00 μl
分析時間： 30.00 分

分析担当者： ohno
分析日： 2005/07/15 11:37:32
取り込みメソッドセット：HX05_70_30
解析日： 2005/07/15 12:38:51
チャンネル名： 波長 Ch 1
解析チャンネルの説明： PDA 260.0 nm

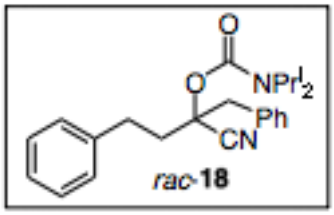
機器名／資産No. Waters 2695 ／ 1517310　　ｶﾗﾑ名 CHIRALPAK IA (4.6 x 150mm)
移動相 Hexane/Ethyl Acetate (70/30)
流量 0.500 ml/mi　　ｶﾗﾑ温度 25℃　　Sample 1mg/ml 移動相



	成分名	保持時間 (分)	面積 (μV秒)	%面積	高さ (μV)
1		17.666	24822	0.483	476
2		20.538	54722	1.065	941
3		22.364	5061078	98.453	59568

SIC 480II FOR WINDOWS TEST REPORT

ファイル名: A200801161300.CHR YRS-III-582G
試料注入,日付:01-16-2008　時間:13:00:13

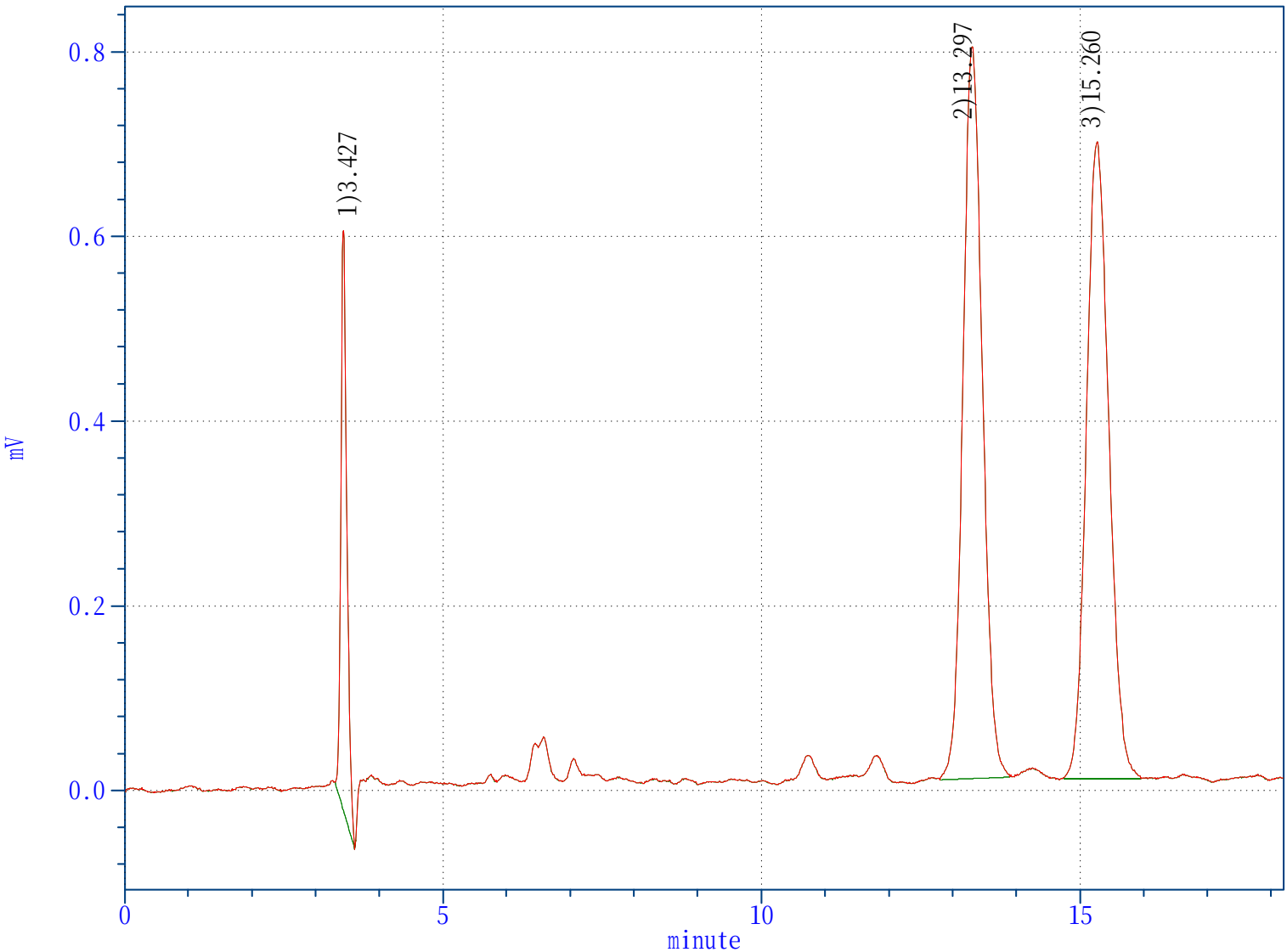


Analysis method:　　Instrument:
Column :IC
Column Temperature (-C) :
Detector :UV
Internal Standard :
Mobile Phase :
Flow rate(ml/Min.) :1.0　　Attenuation :
Solvent A :hexane20
B :iPA1
C :
D :

Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:

データ処理パラメータ
アップスロープ= 5,ダウンスロープ= 5,最小ピーク幅= 10.00 sec , ベース感度= 5 points
計算範囲= 0.000 to 2000.000 min.,最小ピーク面積= 1000 uv*sec , ベースライン補正=0

No.	R.T. (分)	面積 (uv*sec)	面積%	高さ (uv)	高さ%	濃度	濃度%	濃度単位	処理マーク	理論段数	ピーク名
1	3.427	4069	10.7708	628	29.7391	0.0000	0.0000		PP	6842	
2	13.297	16821	44.5259	792	37.5571	0.0000	0.0000		PP	9097	
3	15.260	16888	44.7033	690	32.7038	0.0000	0.0000		PP	8885	
	合計	37778		2110		0.0000	0.0000				



SIC 480II FOR WINDOWS TEST REPORT

ファイル名: MSXI2215Bfr3-2.CHR
試料注入,日付:12-10-2007　時間:12:33:48

Analysis method:　　Instrument:
Column :IC
Column Temperature (-C) :
Detector :UV
Internal Standard :
Mobile Phase :
 Flow rate(ml/Min.) :1mL/min　　Attenuation :
Solvent A :hexane20
　　B :iPA1
　　C :
　　D :

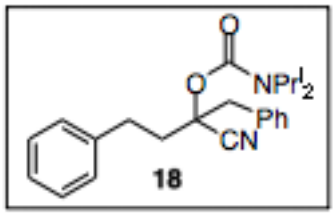
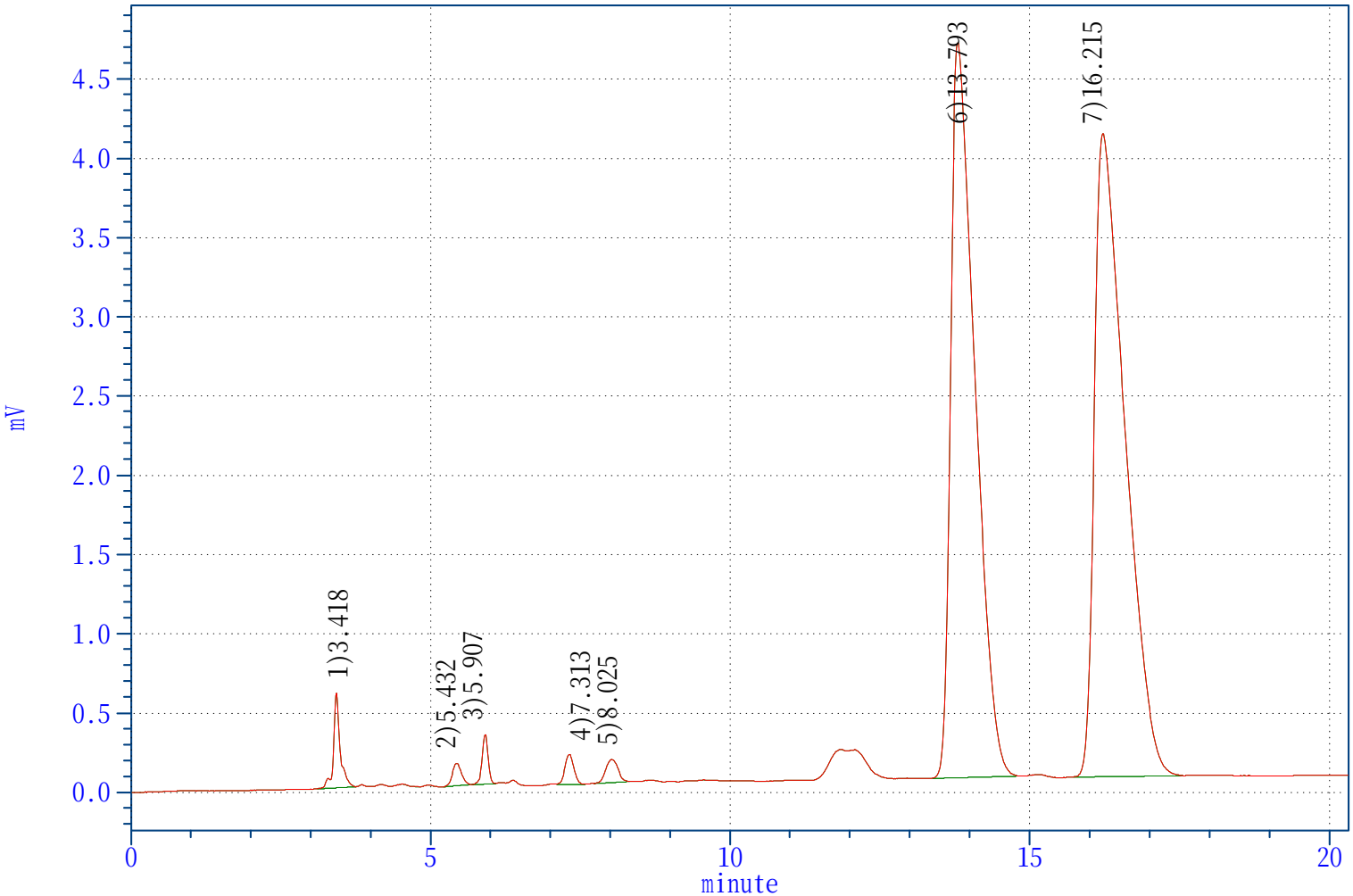


Table 3 (Et_2O, 1 min)

Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:

データ処理パラメータ
アップスロープ= 5,ダウンスロープ= 5,最小ピーク幅= 10.00 sec , ベース感度= 5 points
計算範囲= 0.000 to 2000.000 min.,最小ピーク面積= 1000 uv*sec , ベースライン補正=0

No.	R.T. (分)	面積 (uv*sec)	面積%	高さ (uv)	高さ%	濃度	濃度%	濃度単位	処理マーク	理論段数	ピーク名
1	3.418	4605	1.6048	598	5.9331	0.0000	0.0000		PP	7936	
2	5.432	1506	0.5248	141	1.4017	0.0000	0.0000		PP	5919	
3	5.907	2184	0.7611	313	3.1035	0.0000	0.0000		PP	17818	
4	7.313	1949	0.6792	188	1.8627	0.0000	0.0000		PP	11230	
5	8.025	2060	0.7179	145	1.4422	0.0000	0.0000		PP	6970	
6	13.793	130663	45.5351	4637	46.0050	0.0000	0.0000		PP	5429	
7	16.215	143983	50.1770	4057	40.2518	0.0000	0.0000		PP	4716	
	合計	286950		10079		0.0000	0.0000				



SIC 480II FOR WINDOWS TEST REPORT

ファイル名: A200801221411.CHR YRS-III-590E
試料注入,日付:01-22-2008　時間:14:11:23

S55

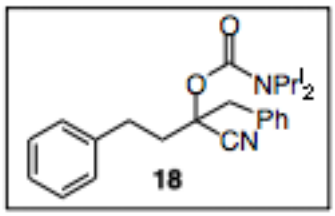


Table 3 (toluene, 1 min)

Analysis method:　　Instrument:
Column :IC
Column Temperature (-C) :
Detector :UV
Internal Standard :
Mobile Phase :
Flow rate(ml/Min.) :1.0　　Attenuation :
Solvent A :hexane20
B :iPA1
C :
D :

Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:
Time :	Flow :	(%A):	(%B):	(%C):	(%D):	Curve:

データ処理パラメータ
アップスロープ= 5,ダウンスロープ= 5,最小ピーク幅= 10.00 sec , ベース感度= 5 points
計算範囲= 0.000 to 2000.000 min.,最小ピーク面積= 1000 uv*sec , ベースライン補正=0

No.	R.T. (分)	面積 (uv*sec)	面積%	高さ (uv)	高さ%	濃度	濃度%	濃度単位	処理マーク	理論段数	ピーク名
1	3.413	11713	19.3607	1905	47.3393	0.0000	0.0000		VV	0	
2	13.347	25850	42.7280	1203	29.8770	0.0000	0.0000		PP	8930	
3	15.377	20610	34.0667	824	20.4799	0.0000	0.0000		PP	8595	
4	19.300	2326	3.8447	93	2.3038	0.0000	0.0000		PP	14057	
	合計	60499		4025		0.0000	0.0000