Supporting Information for:

Cubane Chirality via Substitution of "Hidden" Regular Tetrahedron

Nana Yoshino,[a] Yumi Kato,[a] Thibaud Mabit,[a,b] Yuuya Nagata,[c] Craig M. Williams,[d] Mei Harada,[e] Atsuya Muranaka,[f] Masanobu Uchiyama,[e,f] and Seijiro Matsubara*[a]

[a]*Department of Material Chemistry, Graduate School of Engineering, Kyoto University, Kyoto-daigaku Katsura, Nishikyo-ku, Kyoto 615-8510, Japan*

[b]*JSPS International Fellowship for Research*

[c]*Institute for Chemical Reaction Design and Discovery, Hokkaido University*

[d]*School of Chemistry and Molecular Biosciences, University of Queensland, Brisbane, 4072, Queensland, Australia*

[e]*Cluster for Pioneering Research (CPR), Advanced Elements Chemistry Laboratory, RIKEN, 2-1 Hirosawa, Wako-shi, Saitama 351-0198, Japan*

[f]*University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033, Japan*

Contents

Instrumentation and Chemicals

Nuclear magnetic resonance spectra were taken on Varian UNITY INOVA 500 (^{1}H, 500 MHz; ^{13}C, 125.7 MHz) spectrometer using tetramethylsilane for ^{1}H NMR as an internal standard (δ = 0 ppm), $CDCl_3$ for ^{13}C NMR as an internal standard (δ = 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, sept = septet, br = broad, m = multiplet), coupling constants (Hz), and integration. High-resolution mass spectra were obtained with a JEOL JMS-700 spectrometer for EI and with a Thermo Fisher SCIENTIFIC EXTRACTIVE spectrometer for ESI and APCI. Infrared (IR) spectra were determined on a SHIMADZU FTIR-8200PC spectrometer. Melting points were determined using a YANAKO MP-500D. High performance liquid chromatography (HPLC) was performed with a SHIMADZU Prominence. Electronic absorption spectra were collected on a JASCO V-630 spectrometer. CD spectra were recorded with a JASCO J-820 spectrodichrometer. A 1 mm quartz cell was used for these measurements. The magnitude of the CD signal is expressed in terms of molar circular dichroism $\Delta\varepsilon$ / M^{-1} cm^{-1}. TLC analyses were performed by means of Merck Kieselgel 60 F254 (0.25 mm) Plates. Visualization was accomplished with UV light (254 nm) and an aqueous anisaldehyde solution followed by heating. Flash column chromatography was carried out using Kanto Chemical silica gel (spherical, 40–100 μm). Unless otherwise noted, commercially available reagents were used without purification. Tetrahydrofuran, Dehydrated stabilizer free ―Super― was purchased from Kanto Chemical Co., stored under argon, and used as it is.

4-Deuteriocubane-*N*,*N*-diisopropylcarboxamide (**6**) was prepared by Iodine-Metal Exchange reaction[1] of 4-Iodocubane-*N*,*N*-diisopropylcarboxamide with *n*-Bu_4ZnLi_2 solution[2] followed by a reaction with D_2O. The characterization of the isolated compounds **3**, **5**, **8**, **12**, and **13** were also shown.

Experimental Procedure

Dibromination of 4-deuteriocubane-*N,N*-diisopropylcarboxamide 6: Preparation of 8. The site-selective bromination reported by Alexanian[3] was applied to **6**. A flame-dried 20 mL pyrex vial was charged with 4-deuteriocubane-*N,N*-diisopropylcarboxamide (**6**, 116 mg, 0.5 mmol), *N*-bromo-*N*-(*t*-butyl)-3,5-bis(trifluoromethyl)benzamide (**2,** 196 mg, 0.5 mmol), and anhydrous benzene (7.0 mL). The reaction vial was purged with argon for 10 minutes, and placed in a water bath held at 25 °C, followed by irradiated with visible light for 1 h. Aldrich® Micro Photochemical Reactor blue LED (ALDKIT001) was used as a light source. The pyrex vial was placed in the middle of a circle device (ALDKIT001) with a diameter of 11 cm. An additional one-molar equivalent of bromoamide (**2**, 196 mg, 0.5 mmol) in 3.0 mL benzene was added to the mixture and stirred for 1 h. The another one-molar equivalent of bromoamide (196 mg, 0.5 mmol) in 3.0 mL benzene was added to the mixture and stirred for 2 h. The resulting mixture was concentrated *in vacuo* and dissolved in pentanes. The resulting suspension was run through a plug of silica and concentrated *in vacuo*. Purification by silica gel chromatography (Hexane/AcOEt = 5/1 as an eluent) gave the corresponding product **8** in 72% yield (139 mg), along with **3** (3% yield) and **7** (16% yield). The monobromide **3** and **7** was obtained as a mixture, which cannot be separated by silica gel chromatography. Pure compound **3** was prepared in 28% yield from 4-(diisopropylcarbamoyl)cubane-1-carboxylic acid and *N*-bromophtalic imide by Fu's procedure for visible light-induced decarboxylative iodination.[4] The ration of the mono-bromides (**3** and **7**) was calculated by ^{1}H NMR of the crude product. The rati of **3**, **4**, and **5** in Scheme 1 was also determined in the same way.

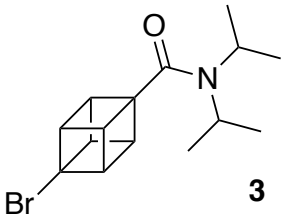
Preparation of chiral cubane (12a-12e).

The dianionic zincate was prepared according to the reported procedure by Uchiyama.[2] To a solution of anhydrous $ZnCl_2$ (Commercially available "anhydrous $ZnCl_2$" was dried in *vacuo* at 150 °C; 402 mg, 3.0 mmol) in anhydrous THF (18 ml), *n*-BuLi (1.57 M in hexane, 7.7 ml, 12.0 mmol) was added dropwise at –78 °C. The resulting mixture was stirred for 30 min at 0 °C. To a thus prepared pale yellow *n*-Bu_4ZnLi_2 THF solution, a solution of 3,5-Dibromo-*N,N*-diisopropylcubane-1-carboxamide-4-*d* (**8**, 390 mg, 1.0 mmol) in THF (5.0 mL) was added dropwise at –78 °C. The whole was stirred for 2h at 25 °C. To the resulting mixture, 4-bromobenzyl bromide (1.25g, 5.0 mmol) in THF (3.0 mL). The resulting mixture was stirred at 25 °C for 12 h. The mixture was quenched with sat. NH_4Claq, and extracted with ether. The organic layers were washed with sat. $NaHCO_3$ aq and brine. The obtained organic solution was dried over Na_2SO_4 and concentrated in *vacuo*. Purification by silica gel

chromatography (Hexane/AcOEt = 5/1 as an eluent) gave chiral cubane (**12a-12d**) as a racemic mixture.

Characterization Data

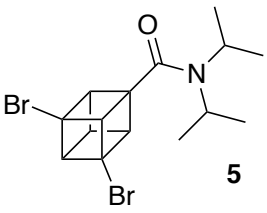
4-Bromo-*N,N*-diisopropylcubane-1-carboxamide-4-d (3)



3

A white solid (mp 122.5–124.5 ℃); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.28-4.24 (m, 3H),1H), 4.24-4.20 (m, 3H), 3.38 (sept, *J* = 7.0 Hz, 1H), 3.30 (sept, *J* = 7.0 Hz, 1H), 1.40 (d, *J* = 7.0 Hz, 6H), 1.18(d, *J* = 7.0 Hz, 6H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 169.8, 62.8, 59.8, 53.8, 48.4, 47.6, 45.9, 21.0, 20.4; HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{20}DBrNONa$ 332.0620; Found332.0625; IR (KBr): 2966, 1617, 1448, 1370, 1348 cm^{-1}..

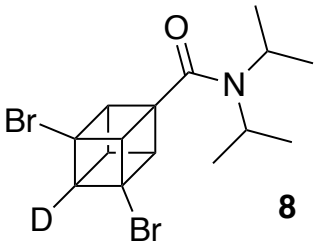
3,5-Dibromo-*N,N*-diisopropylcubane-1-carboxamide (5)



5

Yield (36%,70 mg). A white solid (mp 112–112.5 ℃); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.57 (m, 1H), 4.45 (ddd, *J* = 5.5, 3.0, 3.0 Hz, 2H), 4,41 (dddd, *J*= 5.5, 3.0, 3.0, 3.0 Hz, 1H), 4.14 (dddd, *J* = 5.5, 5.5, 5.5, 1.0 Hz, 1H), 3.53 (sept, *J* = 7.0 Hz, 1H), 3.32 (sept, *J* = 7.0 Hz, 1H), 1.41 (d, *J* = 7.0 Hz, 6H), 1.22 (d, *J* = 7.0 Hz, 6H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 166.9, 66.9, 64.2, 58.6, 55.4, 55.0, 48.9, 46.2, 40.4, 20.7, 20.4; HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{19}Br_2NONa$ 412.9711; Found 411.9708; IR (KBr): 2960, 1650, 1625, 1448cm^{-1}.

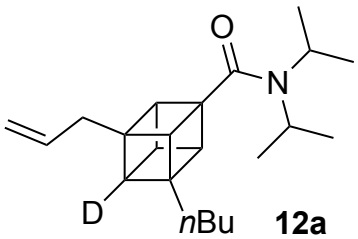
3,5-Dibromo-*N,N*-diisopropylcubane-1-carboxamide-4-d (8)



8

Yield (72%,139 mg). A white solid (mp 111–112 ℃); ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.56 (ddd, *J* = 3.0, 3.0, 1.0 Hz, 1H), 4.44 (dd, *J* = 5.5, 3.0 Hz, 2H), 4.13 (ddd, *J* = 5.5, 5.5, 1.0 Hz, 1H), 3.53 (sept, *J* = 7.0 Hz, 1H), 3.32 (sept, *J* = 7.0 Hz, 1H), 1.41 (d, *J* = 7.0 Hz, 6H), 1.22 (d, *J* = 7.0 Hz, 6H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 166.9, 66.9, 63.8 (t), 58.5, 55.4, 54.9, 48.8, 46.2, 40.3, 20.7, 20.4; HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{18}DBr_2NONa$ 412.9768; Found 412.9764; IR (KBr): 2260, 1653, 1628, 1457, 1437, 1374, 1346, 754.

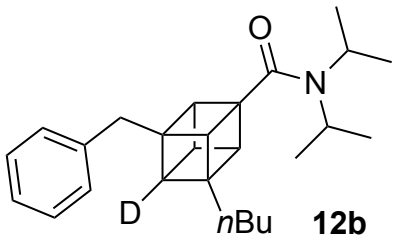
3-Allyl-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12a)



12a

Prepared following the general procedure using 0.1 mmol of **8**: Yield 67% (colorless oil, 22.0 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.72 (dddd, *J* = 17.0, 13.5, 9.5, 6.5 Hz, 1H), 5.07-5.00 (m, 2H), 3.94 (ddd, *J* = 5.0, 2.5, 2.5 Hz, 1H), 3.90 (ddd, *J* = 5.0, 2.5, 2.5 Hz, 1H), 3.68 (dd, *J* = 2.5, 2.5 Hz, 1H), 3.62 (dd, *J* = 5.0, 5.0 Hz, 1H), 3.58 (sept, *J* = 7.0 Hz, 1H), 3.27 (sept, *J* = 7.0 Hz, 1H), 2.37-2.28 (m, 2H), 1.56 (dt, *J* = 16.0, 6.5 Hz, 2H), 1.54 (dt, *J* = 16.0, 6.5 Hz, 2H), 1.40 (d, *J* = 6.5 Hz, 6H), 1.35-1.19 (m, 4H), 1.16 (d, *J* = 6.5 Hz, 6H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125.7 MHz, $CDCl_3$): δ 171.3, 133.6, 116.5, 52.7, 52.5, 51.8, 51.1, 50.9, 50.8, 48.0, 45.7, 37.3, 37.2, 32.2, 26.3, 22.9, 20.8, 20.6, 14.1 (The signal of D-substituted carbon in cubane skeleton wan not strong enough to be detected because of coupling with D); HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{22}H_{32}DNONa$ 351.2517; Found 351.2524; IR (KBr): 2963, 2925, 2224, 1627, 1441, 1369, 1340, 1217, 1046 cm^{-1}.

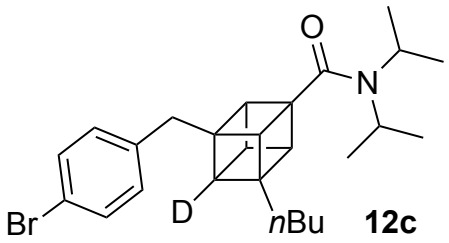
3-Benzyl-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12b)



12b

Prepared following the general procedure using 0.1 mmol of **8**: Yield 48% (colorless oil, 18.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28 (dd, *J* = 7.5, 7.0 Hz, 2H), 7.19 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.12 (d, *J* = 7.0 Hz, 2H), 3.99 (ddd, *J* = 5.0, 2.5, 2.5 Hz, 1H), 3.86 (ddd, *J* = 5.0, 2.5, 2.5 Hz, 1H), 3.71 (dd, *J* = 2.5, 2.5 Hz, 1H), 3.58 (dd, *J* = 5.0, 5.0 Hz, 1H), 3.58 (sept, *J* = 7.0 Hz, 1H), 3.27 (sept, *J* = 7.0 Hz, 1H), 2.90 (d, *J* = 19.0 Hz, 1H), 2.87 (d, *J* = 19.0 Hz, 1H), 1.48-1.30 (m, 2H), 1.41 (d, *J* = 6.5 Hz, 6H), 1.23-1.10 (m, 2H), 1.16 (d, *J* = 6.5 Hz, 6H), 0.93-0.83 (m, 2H), 0.79 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 171.3, 138.1, 128.9, 128.2, 125.9, 52.5, 52.2, 51.8, 51.7, 51.2, 51.1, 48.1, 45.7, 39.1, 36.9, 32.2, 25.9, 22.9, 20.8, 20.6, 14.1 (The signal of D-substituted carbon in cubane skeleton wan not strong enough to be detected because of coupling with D); HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{26}H_{34}DNO$ 401.2674; Found 401.2678; IR (KBr): 2960, 2922, 2224, 1625, 1436, 1368, 1340 cm^{-1}.

3-(4-Bromobenzyl)-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12c)

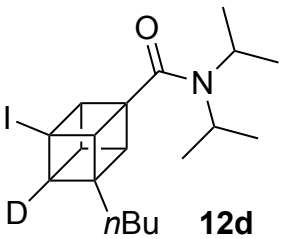


12c

Prepared following the general procedure using 0.1 mmol of **8**: Yield 53% (colorless oil, 24.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39(d, *J* = 8.1 Hz, 2H), 7.00 (d, *J* = 8.1 Hz, 2H), 3.97 (ddd, *J* = 5.1, 2.0, 2.0 Hz, 1H), 3.84 (ddd, *J* = 5.1, 2.0, 2.0 Hz, 1H), 3.69 (bs, 1H), 3.58 (dd, *J* = 5.1, 5.1 Hz, 1H), 3.56 (sept, *J* = 6.6 Hz, 1H), 3.27 (sept, *J* = 6.6 Hz, 1H), 2.83 (d, *J* = 15.0 Hz, 1H), 2.82 (d, *J* = 15.0 Hz, 1H), 1.43 – 1.36 (m, 2H), 1.41 (d, *J* = 6.6 Hz, 6H), 1.19 – 1.12 (m, 2H), 1.16 (d, *J* = 6.6 Hz, 6H), 0.89 – 0.79 (m, 2H), 0.81 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 171.1, 137.0, 131.3, 130.7, 119.8, 52.5, 52.2, 51.6, 51.5, 51.4, 50.9, 48.1, 45.8, 38.4, 36.9, 32.2, 25.9, 22.9, 20.8, 20.6, 14.1 (The signal of D-substituted carbon in cubane skeleton wan not strong enough to be detected because of coupling with D); HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{26}H_{33}DBrNONa$ 479.1779; Found 479.1781; IR (KBr): 2961, 2225, 1628, 1490, 1441, 1369, 1341 cm^{-1}.

The racemic micture was resolved by HPLC DAICEL CHIRALPAK ID *iso*-PrOH/Hexane: 5/95, flow rate: 1.0 mL/min. The sample was separated into two eluents (1st fraction: 8.15 min, $[\alpha]^{25}_D$ = –13.5 (*c* 9.2, CHCl3); 2nd fraction 9.01 min, $[\alpha]^{25}_D$ = +13.9 (*c* 7.3, CHCl3).

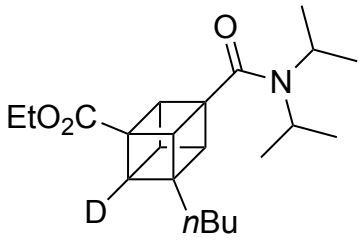
3-Iodo-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12d)



12d

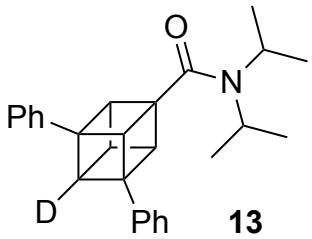
Prepared following the general procedure using 0.3 mmol of **8**: Yield 31% (colorless oil, 38.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.41 (ddd, *J* = 5.5, 2.0, 2.0 Hz, 1H), 4.17 (bs, 1H), 4.13 (ddd, *J* = 4.5, 2.0, 2.0 Hz, 1H), 3.96 (dd, *J* = 4.5, 5.5, Hz, 1H), 3.66 (sept, *J* = 7.0 Hz, 1H), 3.31 (sept, *J* = 7.0 Hz, 1H), 1.66 (m, 2H), 1.41 (d, *J* = 7.0 Hz, 6H), 1.37 – 1.24 (m, 4H), 1.21 (d, *J* = 7.0 Hz, 6H), 0.91 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 168.7, 61.1, 60.1, 57.9, 57.3, 57.0, 50.3, 48.5, 46.0, 41.7, 41.5, 31.5, 26.2, 22.6, 20.7, 20.5, 14.0 (The signal of D-substituted carbon in cubane skeleton wan not strong enough to be detected because of coupling with D); HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{27}DINO$ 437.1171; Found 437.1173; IR (KBr): 2964, 2926, 2239, 1628, 1437, 1369, 1340, 1255, 1215, 1045 cm^{-1}.

3-Carboxylateethyl-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12e)



Prepared following the general procedure using 0.3 mmol of **8**: Yield 40% (colorless oil, 43.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.37 (ddd, *J* = 5.1, 2.5, 2.5 Hz, 1H), 4.16 (q, *J* = 7.0 Hz, 2H), 4.11 (dd, *J* = 2.5, 2.5 Hz, 1H), 3.92 (ddd, *J* = 5.1, 2.5, 2.5 Hz, 1H), 3.77 (dd, *J* = 5.1, 5.1 Hz, 1H), 3.64 (sept, *J* = 6.5 Hz, 1H), 3.30 (sept, *J* = 7.0 Hz, 1H), 1.62 (t, *J* = 7.5 Hz , 2H), 1.41 (d, *J* = 6.8 Hz, 6H), 1.36 – 1.25 (m, 4H), 1.27 (t, *J* = 7.1 Hz, 3H), 1.19 (d, *J* = 6.6 Hz, 6H), 0.90 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 171.7, 169.9, 60.3, 53.7, 53.5, 53.1, 52.2, 49.9, 49.6, 48.3, 45.8, 38.3, 31.5, 26.0, 22.7, 20.8, 20.5, 14.3, 14.0 (The signal of D-substituted carbon in cubane skeleton wan not strong enough to be detected because of coupling with D); HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{22}H_{32}DNO_3Na$ 383.2415; Found 383.2417; IR (KBr): 2968, 2929, 2239, 1722, 1627, 1441, 1369, 1343, 1313, 1191 cm^{-1}

3,5-Diphenyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (13)



13

Prepared following the general procedure using 0.1 mmol of **1a**: Yield 48% (18.5 mg, decomposed at >250 °C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.46 – 7.20 (m, 10H), 4.49 (dd, *J* = 5.1, 2.4 Hz, 2H), 4.46 (dd, *J* = 2.4, 2.2 Hz, 1H), 3.94 (dd, *J* = 5.1, 5.4 Hz, 1H), 3.60 (sept, *J* = 6.6 Hz, 1H), 3.28 (sept, *J* = 6.8 Hz, 1H), 1.42 (d, *J* = 6.8 Hz, 6H), 1.09 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.2, 141.6, 128.6, 126.3, 125.0, 57.2, 53.3(t), 53.1, 48.4, 45.9, 37.2, 20.8, 20.6; HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{27}H_{28}NONa$ 407.2204; Found 407.2208; IR (KBr): 3744, 2928, 2231, 1700, 1625, 1437cm^{-1}

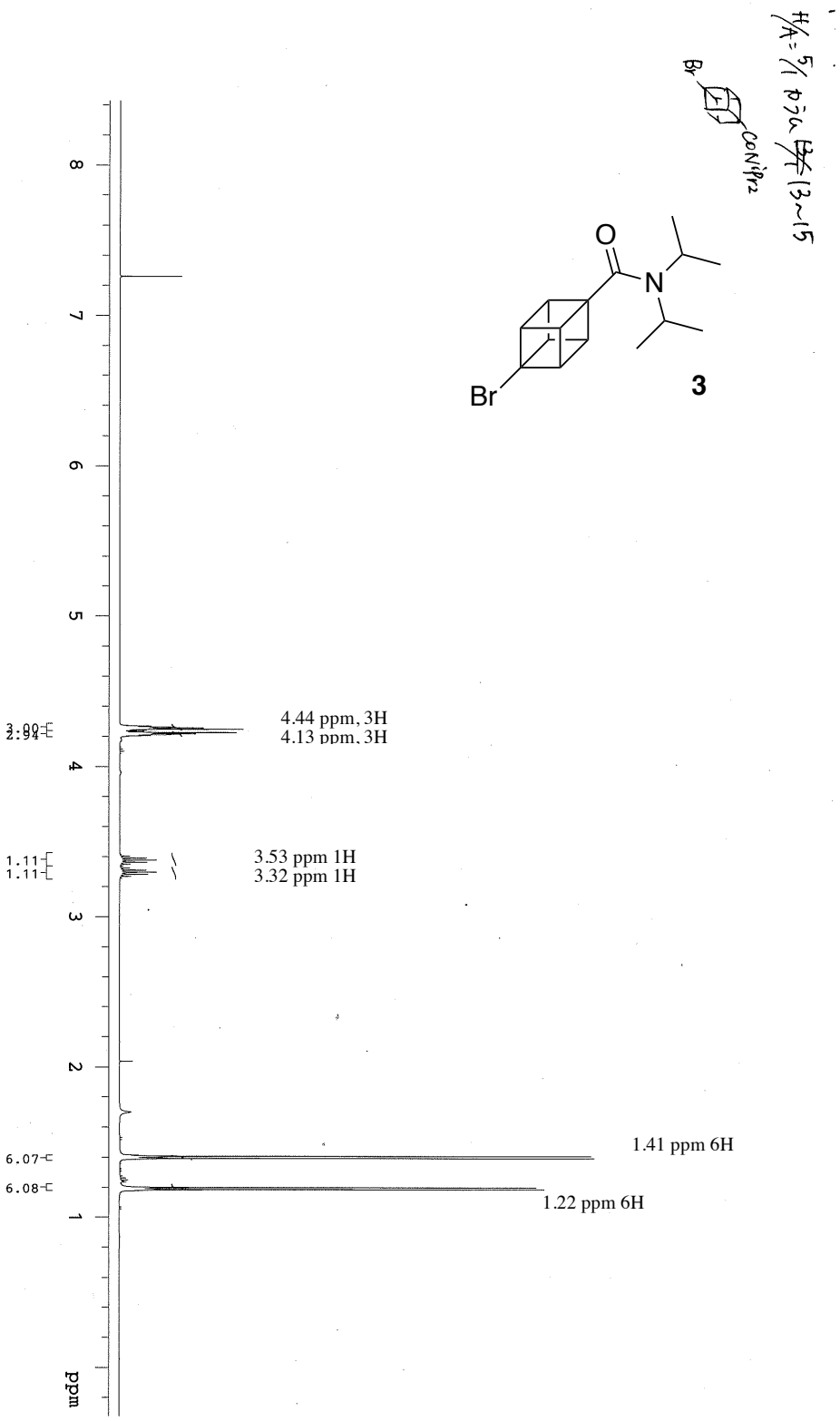
(1) Y. Kato, C. M. Williams, M. Uchiyama, S. Matsubara, *Org. Lett.* **2019**, *21,* 473-475.

(2) Uchiyama, M.; Furuyama, T.; Kobayashi, M.; Matsumoto, Y.; Tanaka, K. *J. Am. Chem. Soc.* **2006**, *128*, 8404-8405.

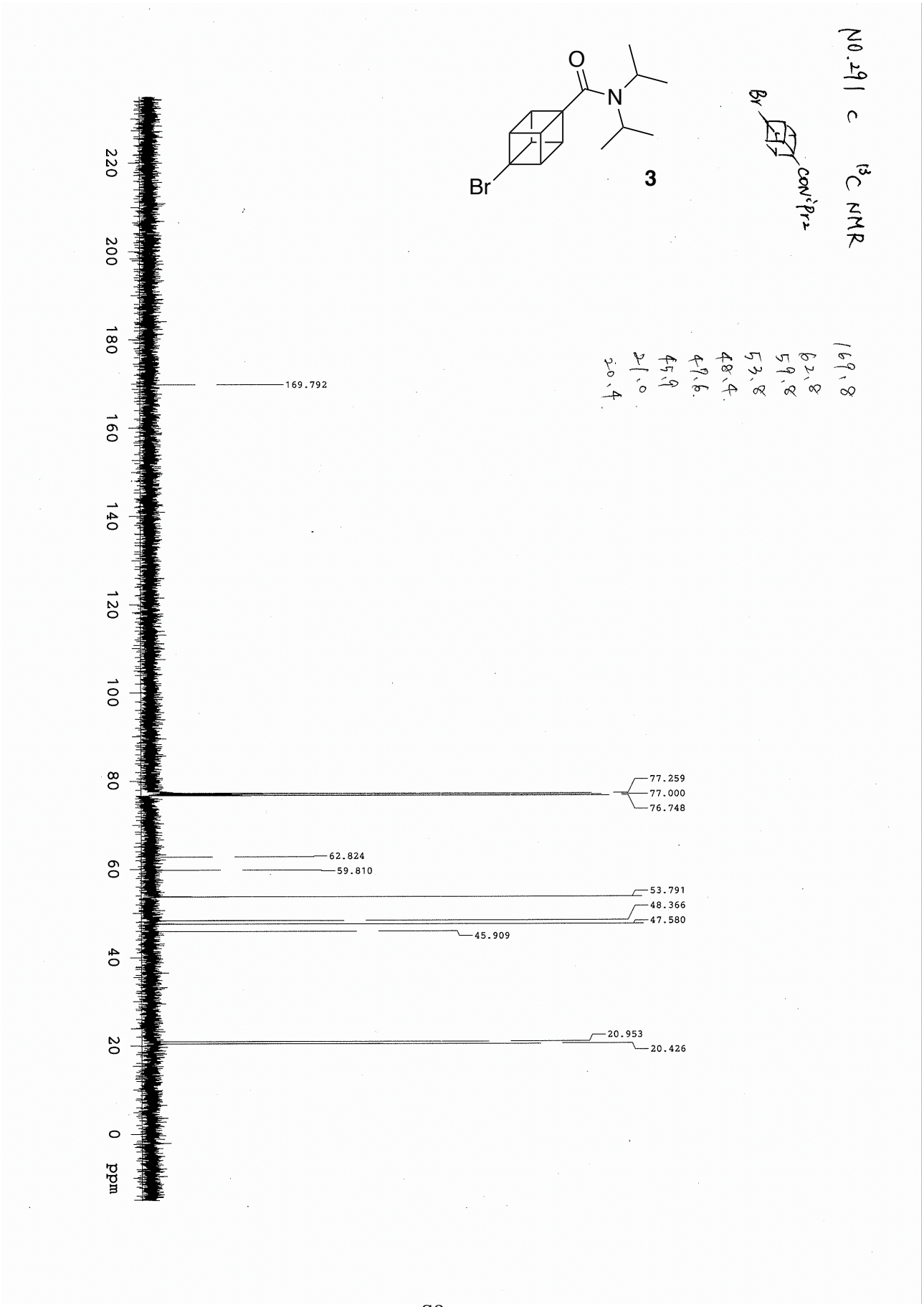
(3) Schmidt, V. A.; Quinn, R. K.; Brusoe, A. T.; Alexanian, E. J. *J. Am. Chem. Soc.* **2014**, *136*, 14389-14392.

(4) Candish, L.; Standley, E. A.; Gómez-Suárez, A.; Mukherjee, S.; Glorius, F. *Chem. Eur. J.* **2016**, *22*, 9971-9974.

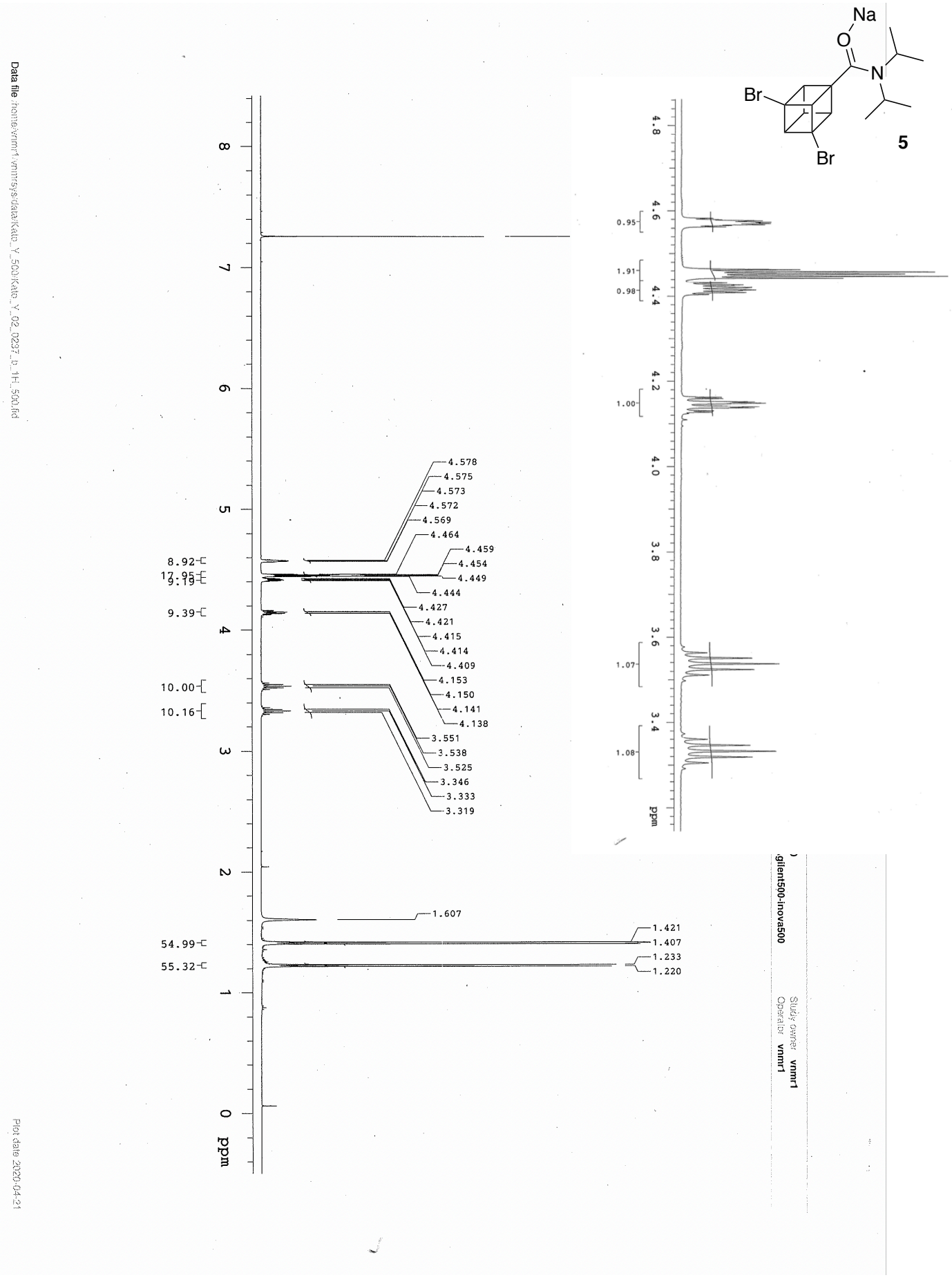
^{1}H NMR Spectra of 3 (500 MHz, $CDCl_3$)



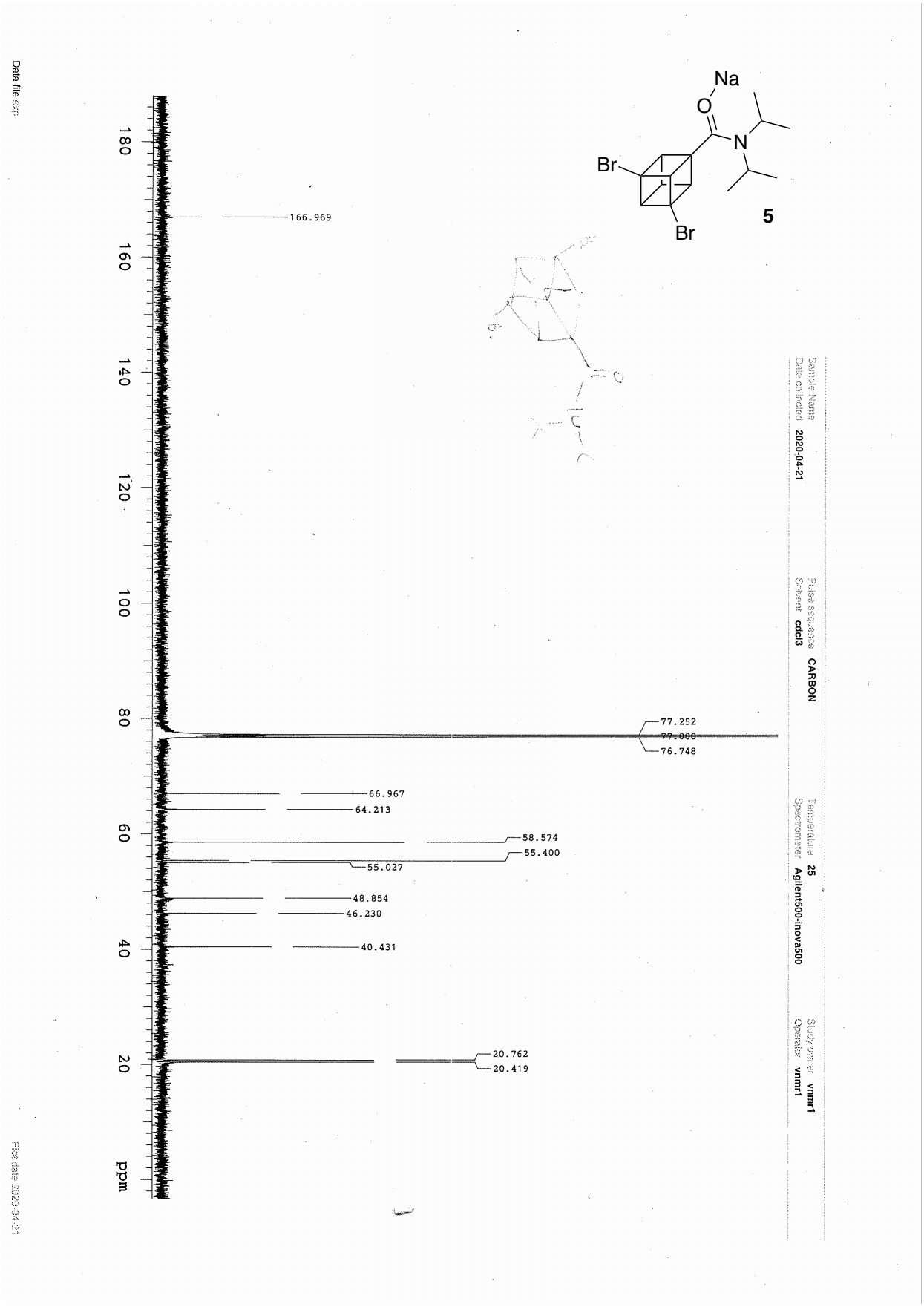
^{13}C NMR Spectra of 3 (125.7MHz, $CDCl_3$)



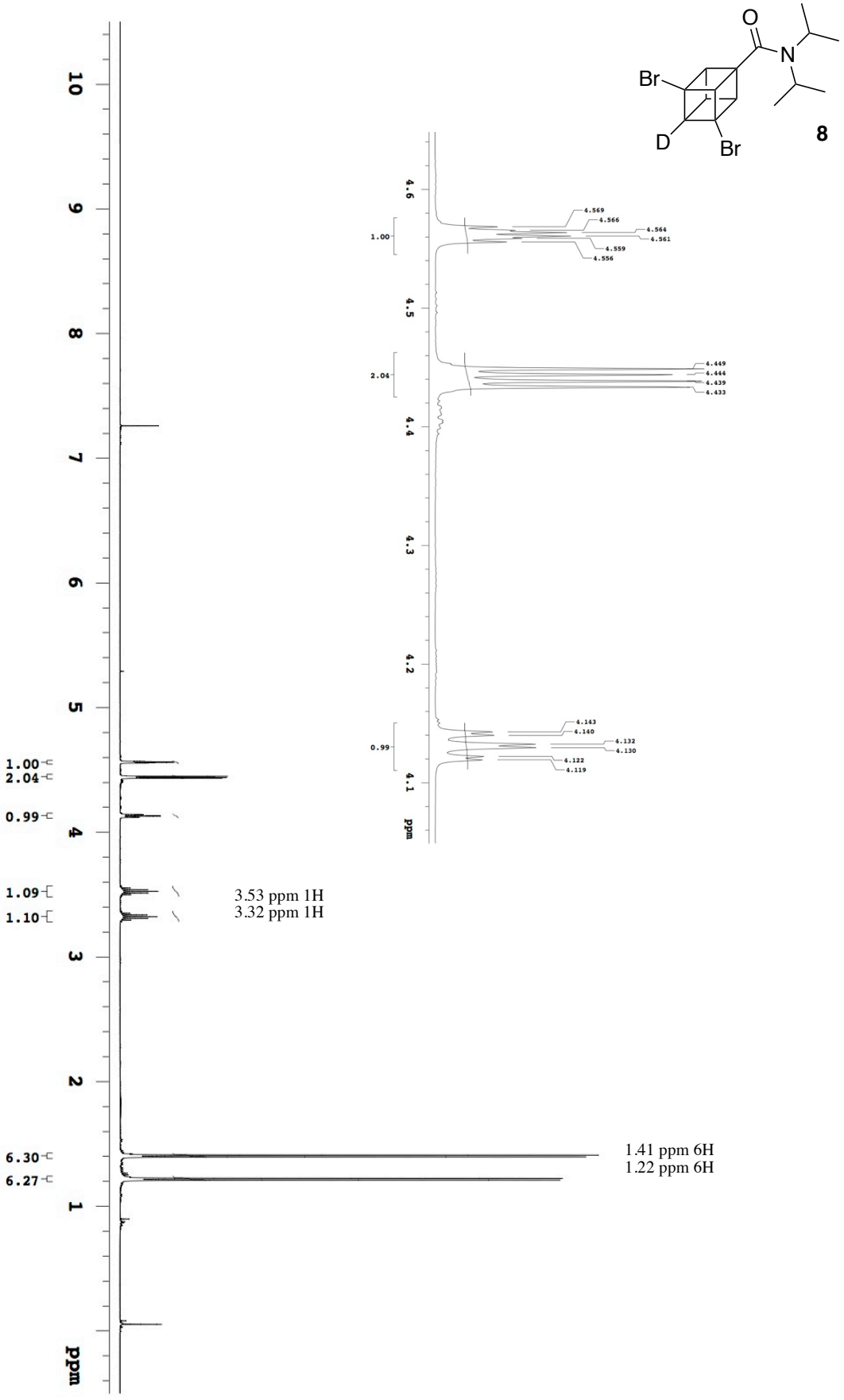
^{1}H NMR Spectra of 5 (500 MHz, $CDCl_3$)



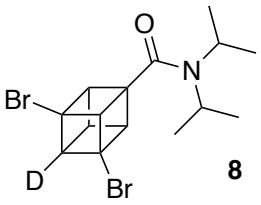
^{13}C NMR Spectra of 5 (125.7MHz, $CDCl_3$)



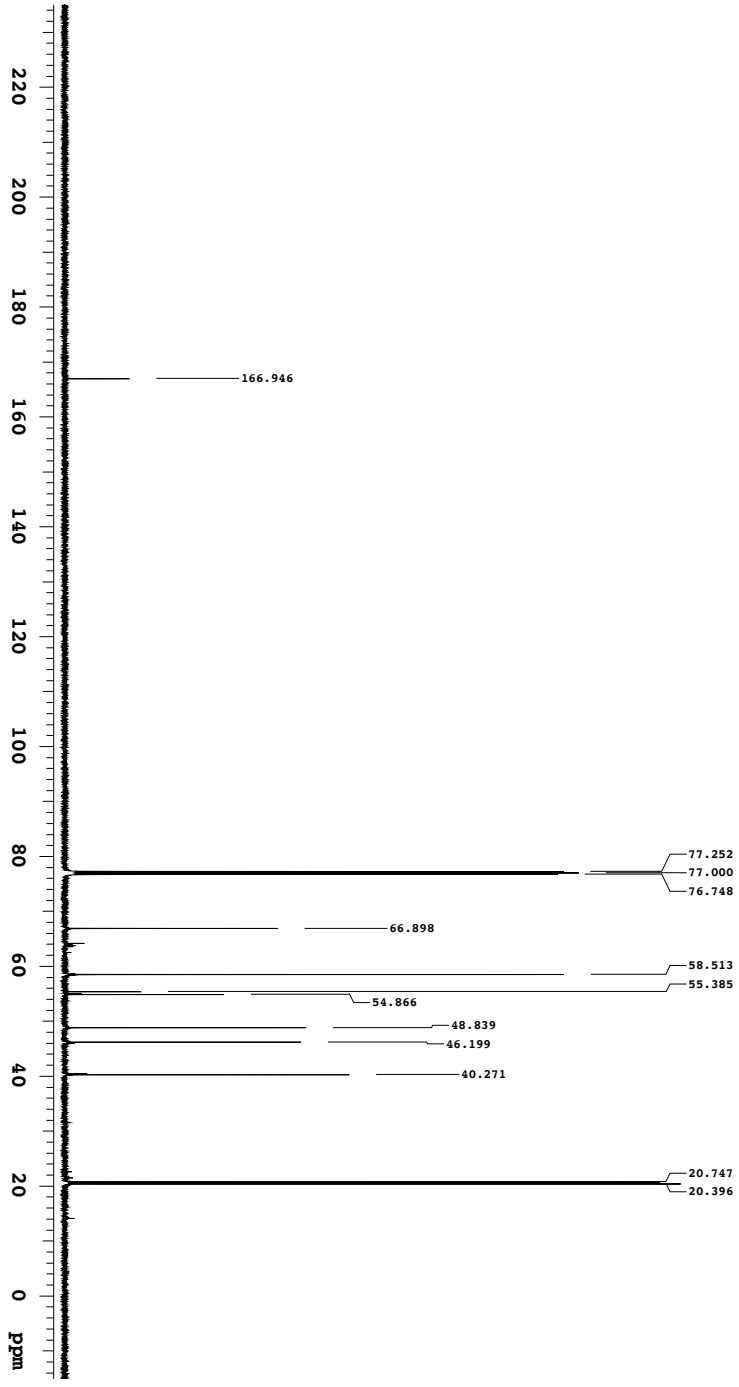
^{1}H NMR Spectra of 8 (500 MHz, $CDCl_3$)



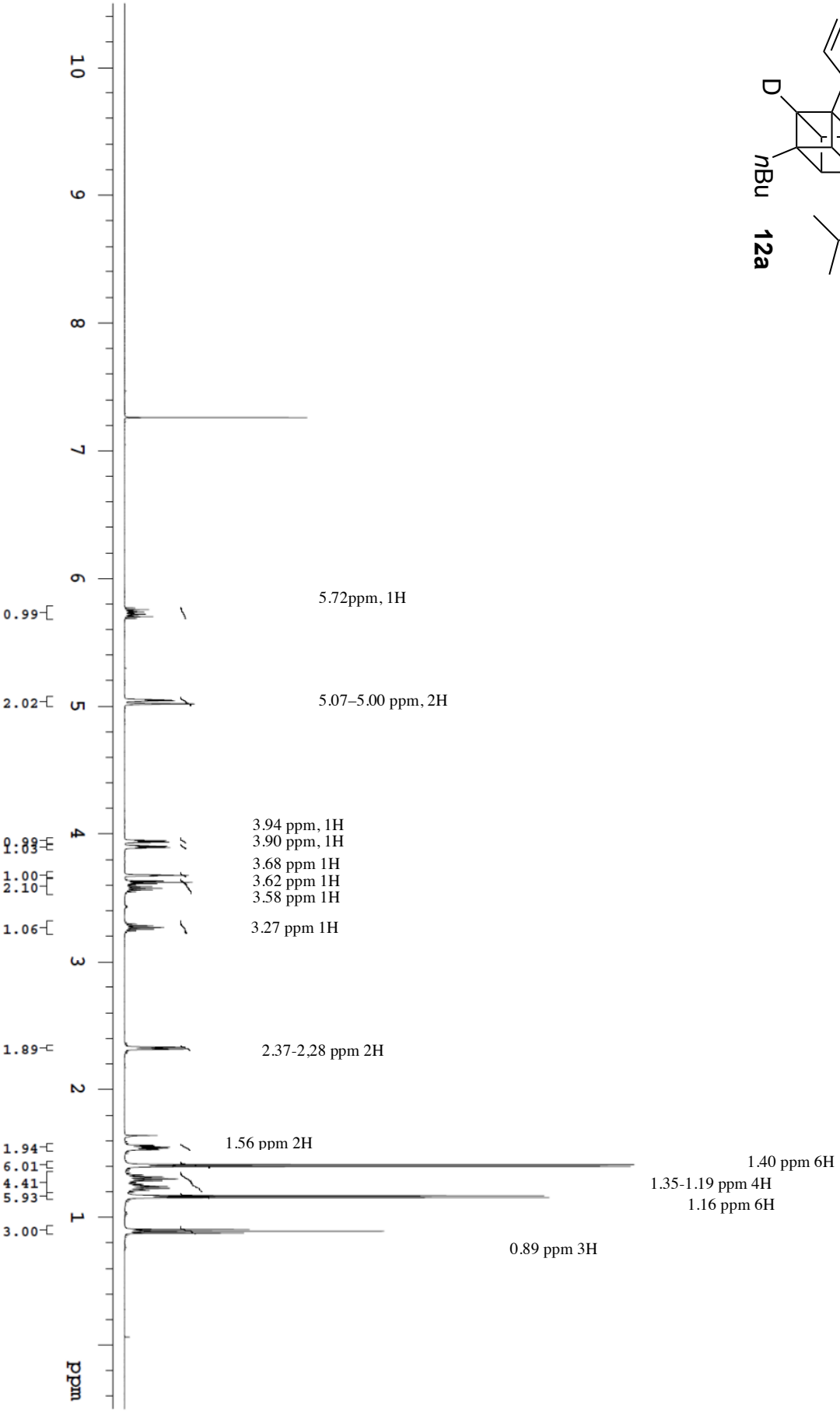
^{13}C NMR Spectra of 8 (125.7MHz, $CDCl_3$)



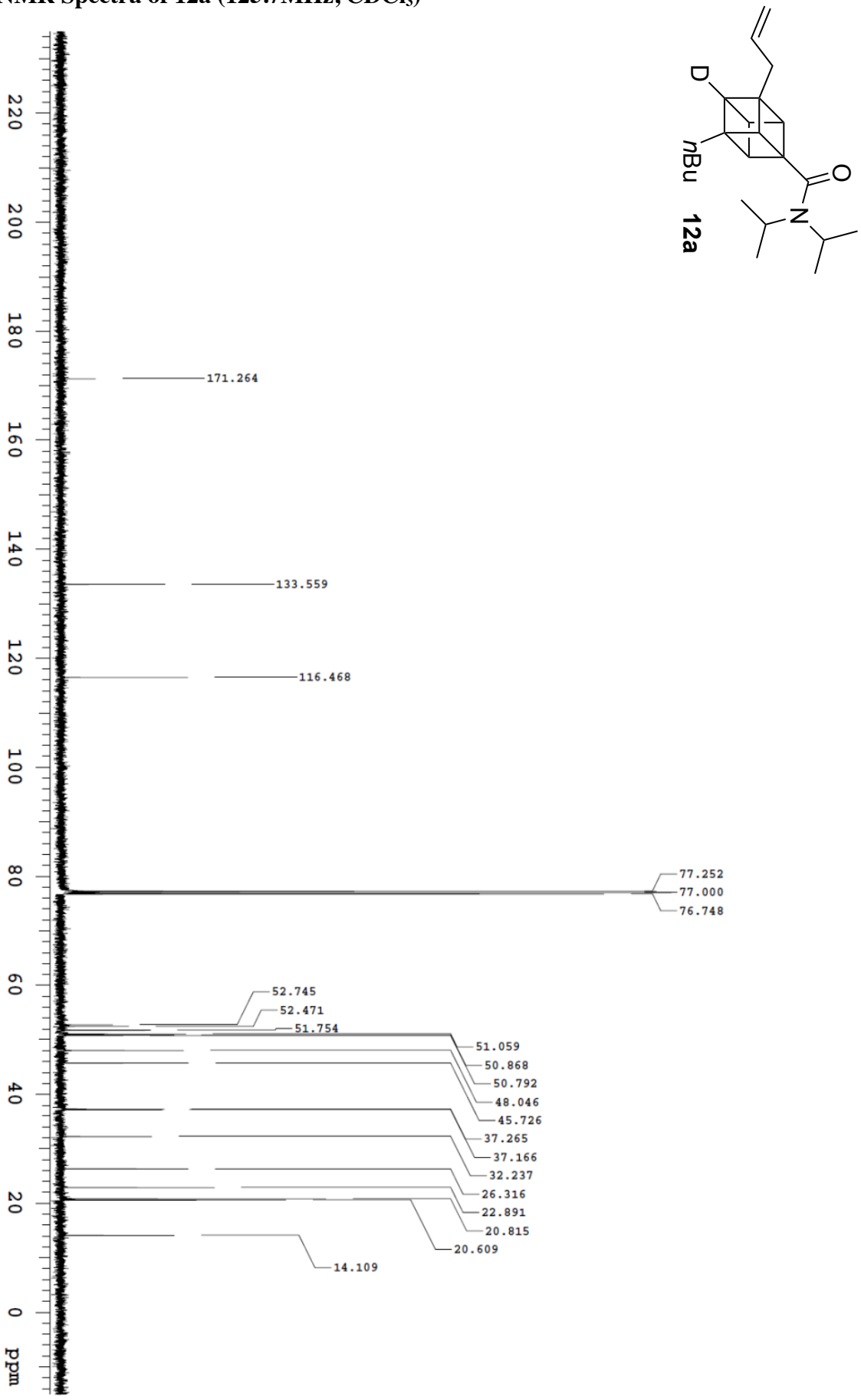
8



^{1}H NMR Spectra of 12a (500 MHz, $CDCl_3$)

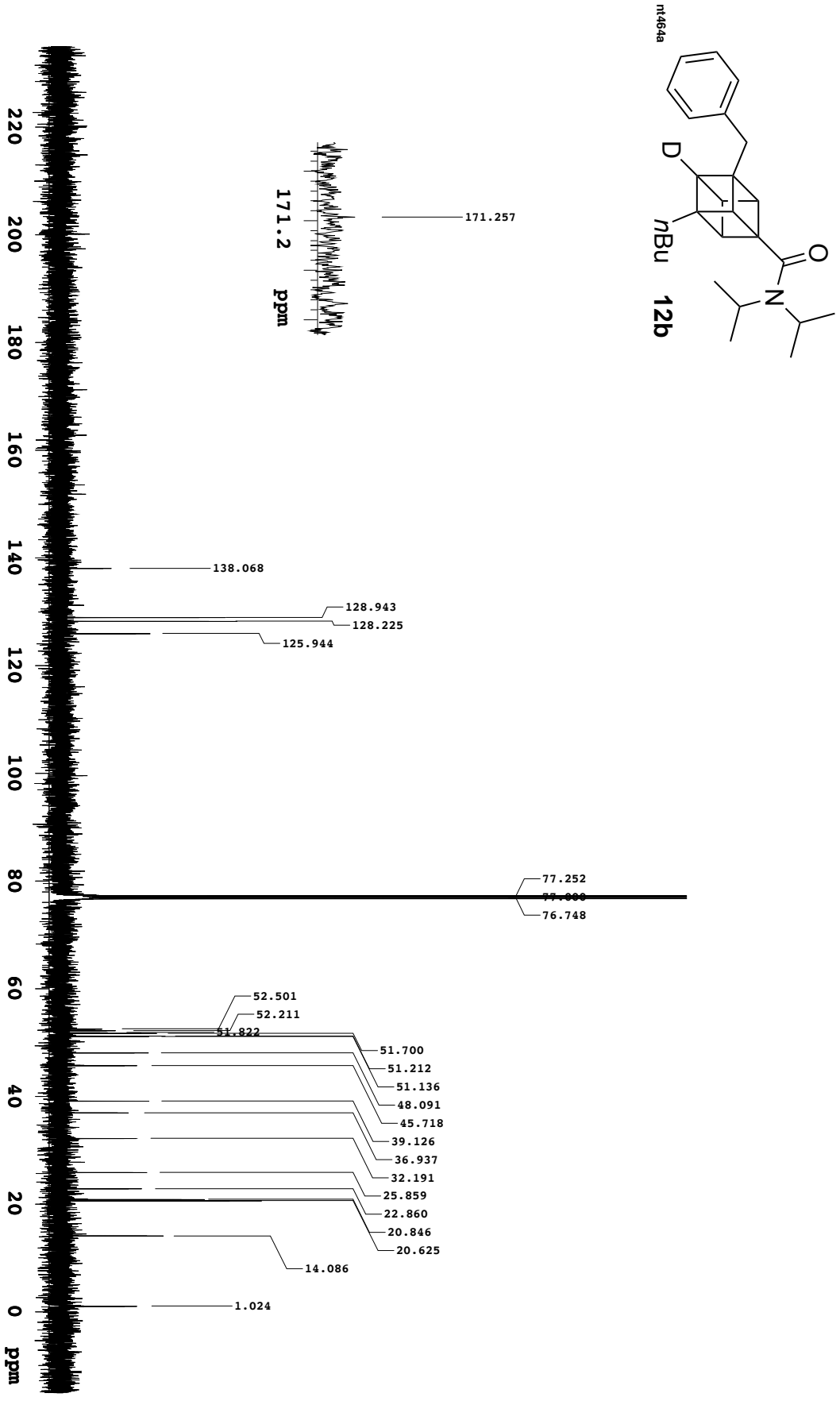


^{13}C NMR Spectra of 12a (125.7MHz, $CDCl_3$)

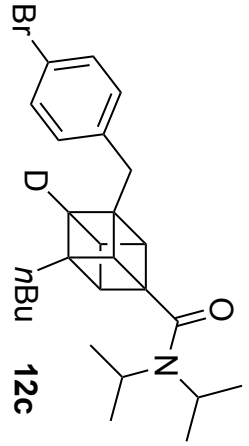


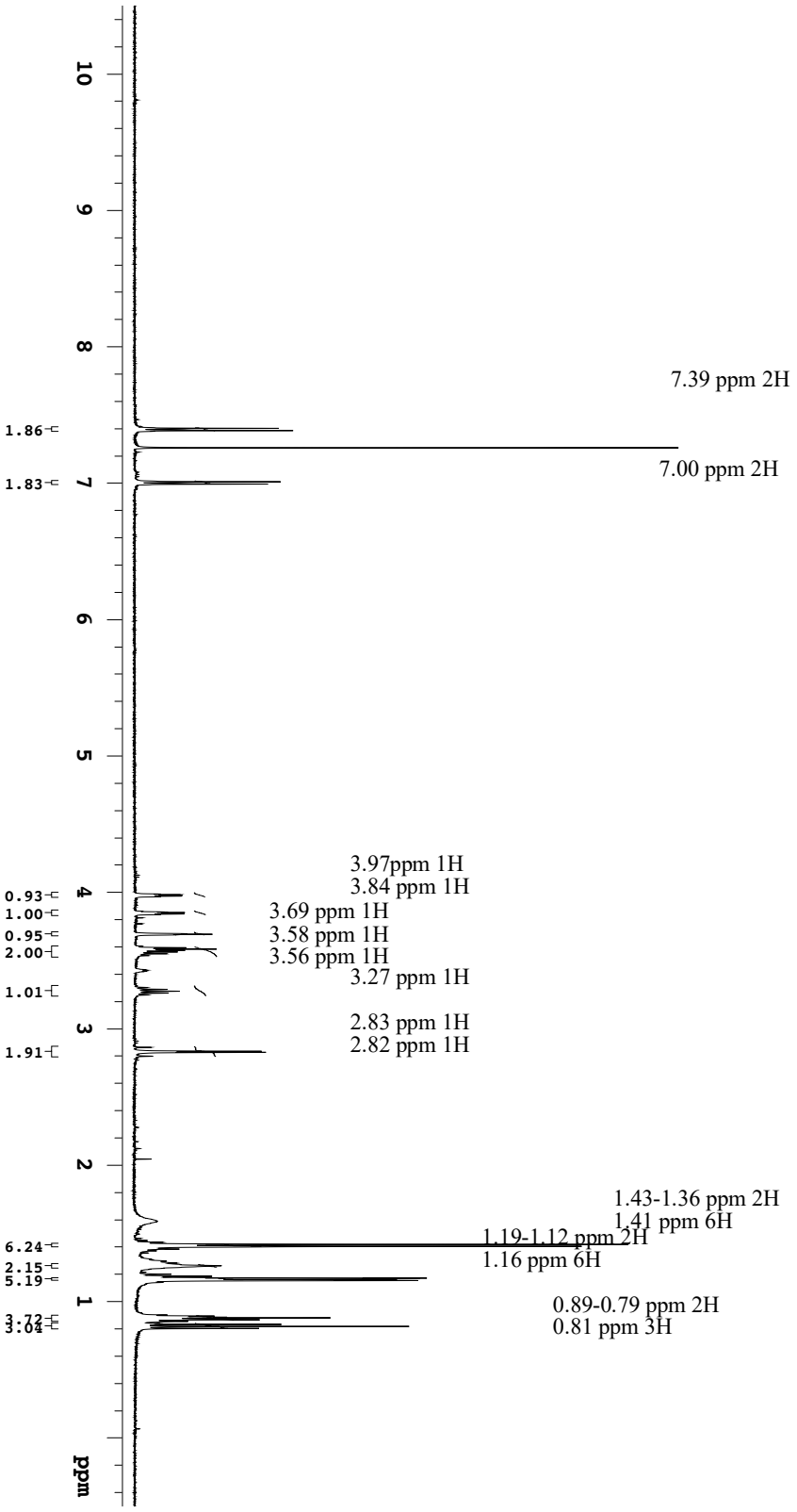
^{1}H NMR of 12b **(500 MHz, $CDCl_3$)**

^{13}C NMR Spectra of 12b (125.7MHz, $CDCl_3$)

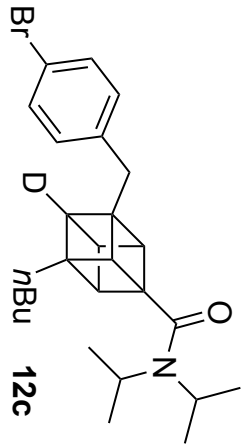


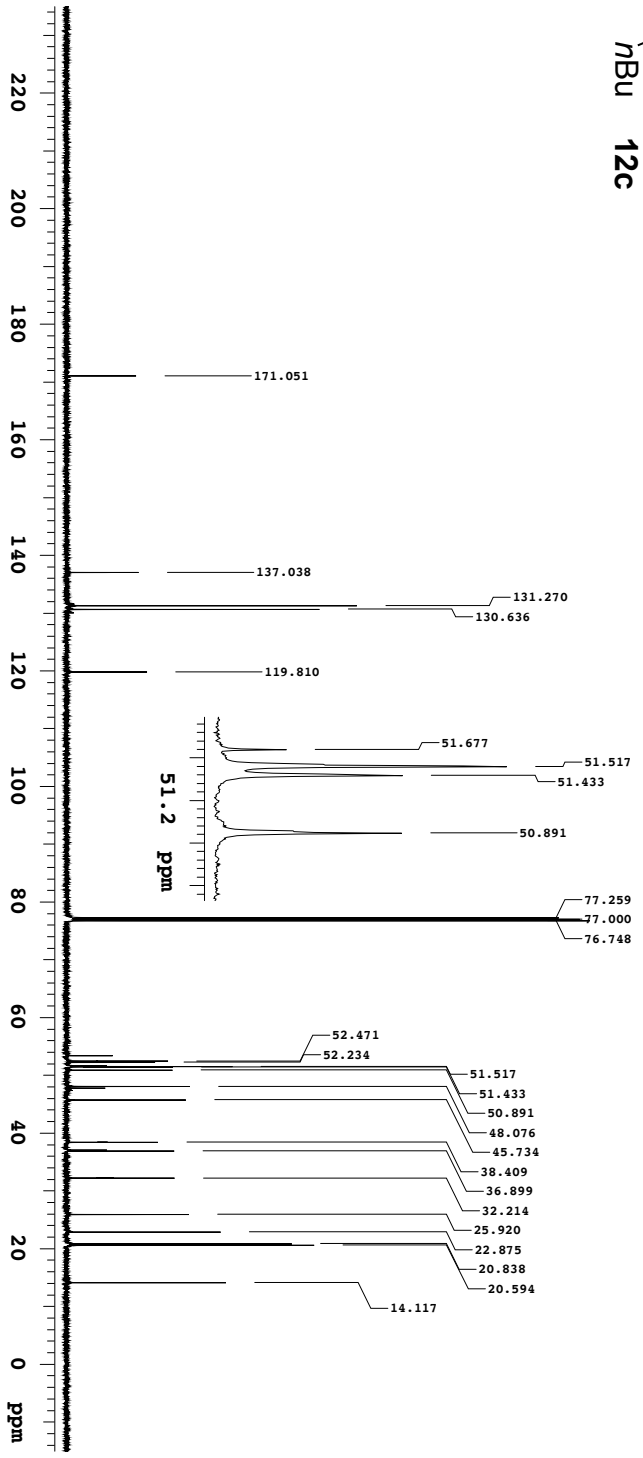
^{1}H NMR of 12c (500 MHz, $CDCl_3$)



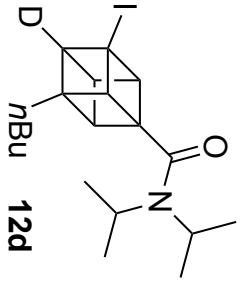


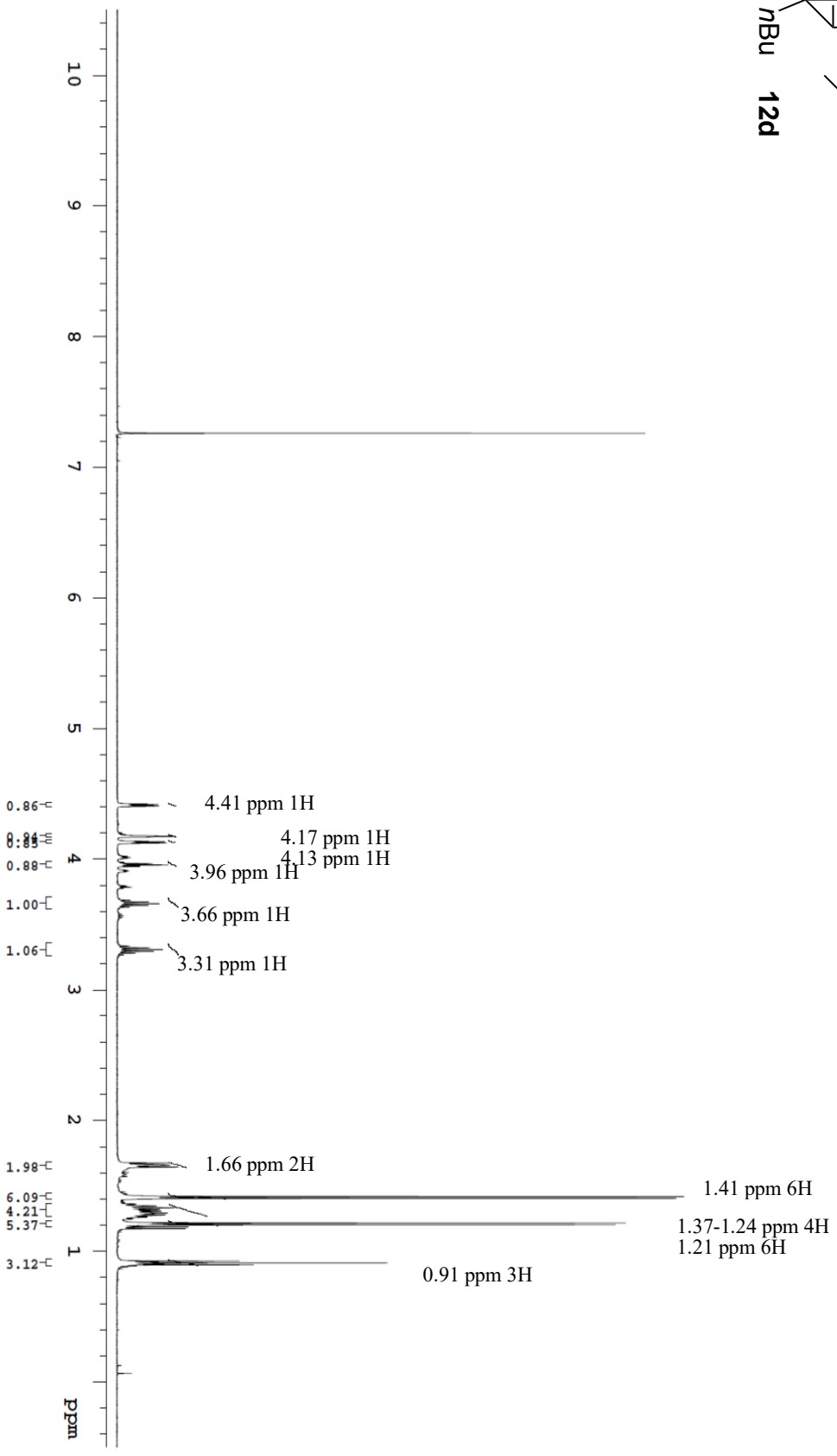
^{13}C NMR Spectra of 12c (125.7MHz, $CDCl_3$)



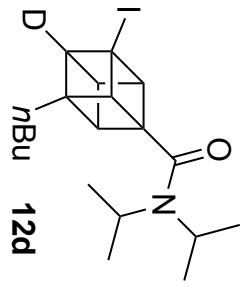


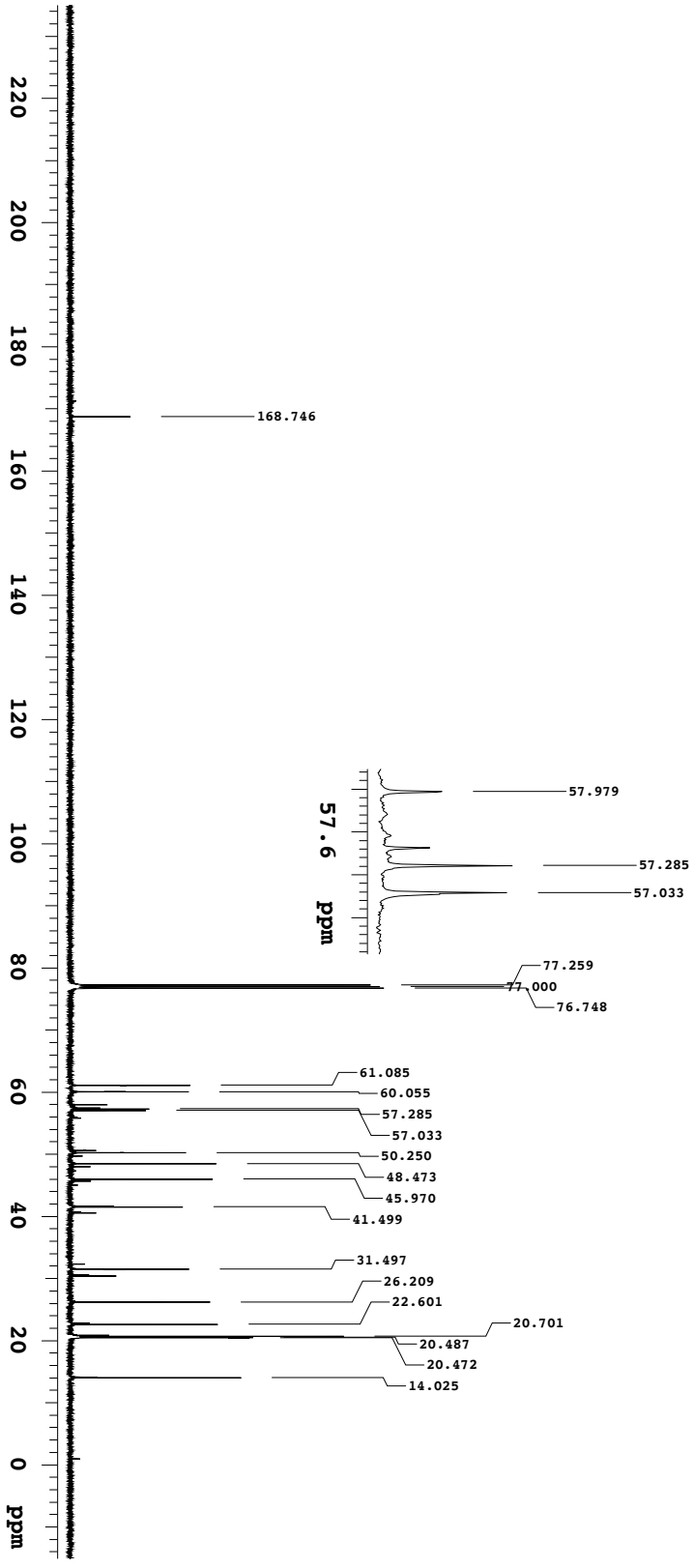
^{1}H NMR Spectra of 12d (500 MHz, $CDCl_3$)



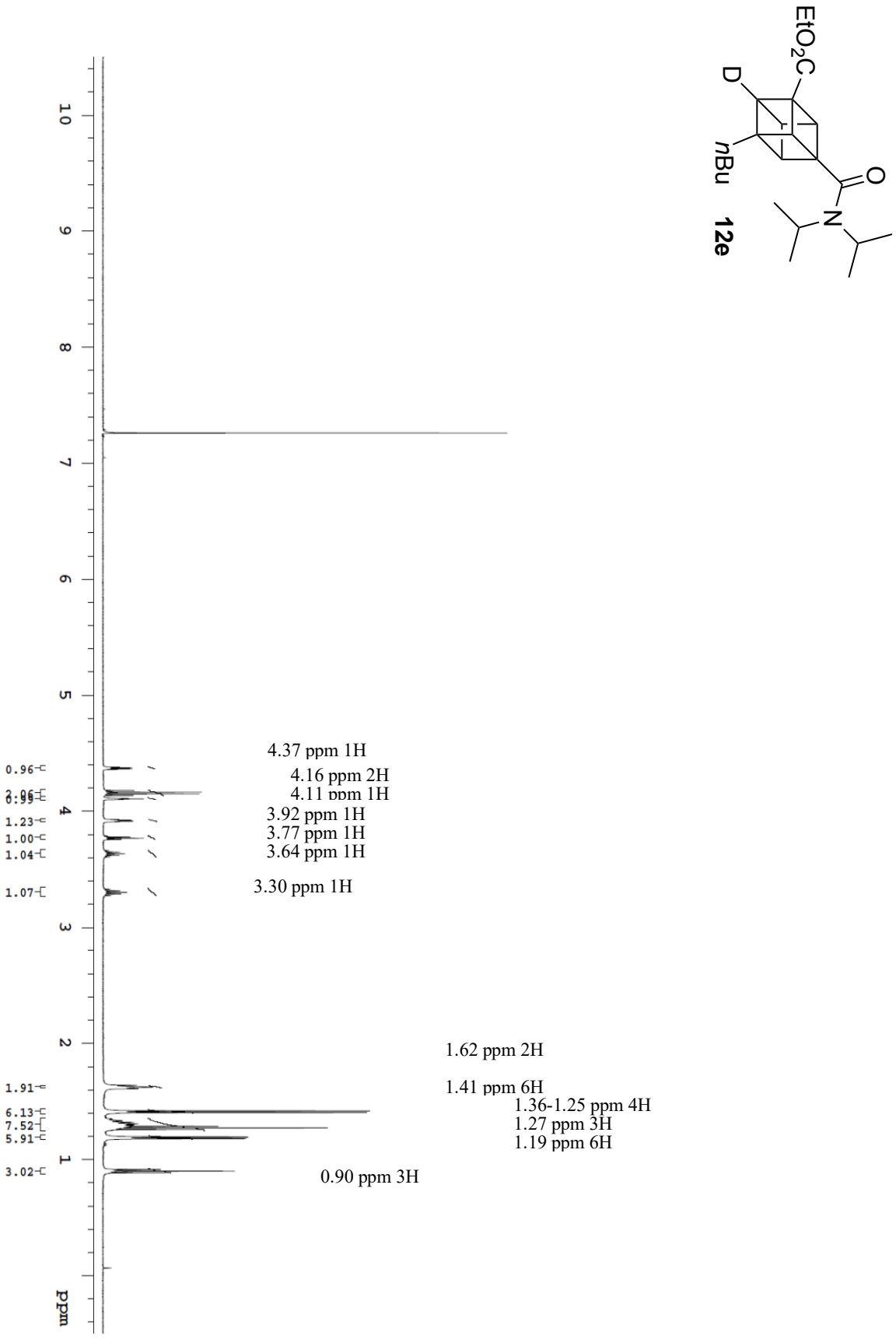


^{13}C NMR Spectra of 12d (125.7MHz, $CDCl_3$)

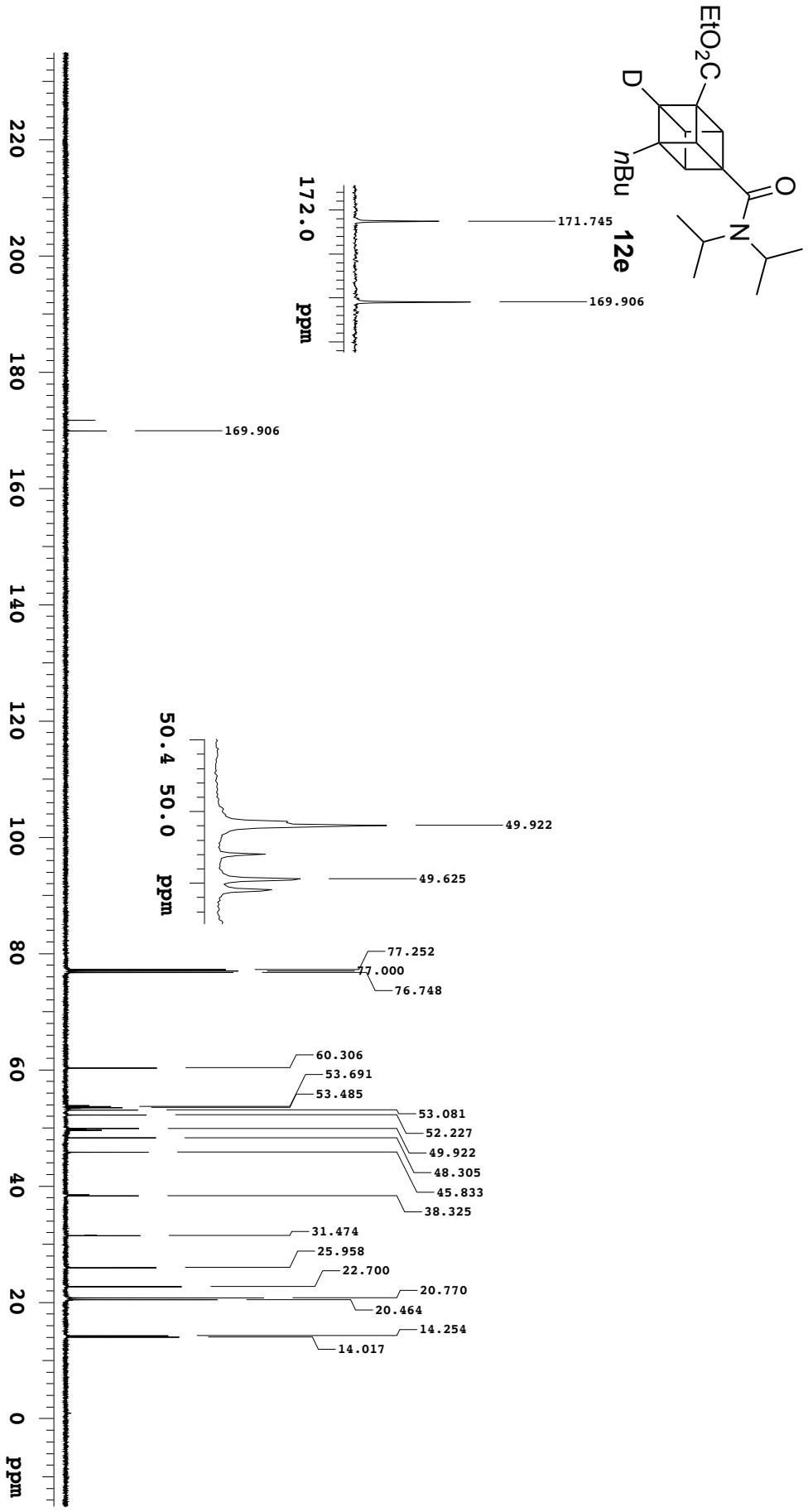




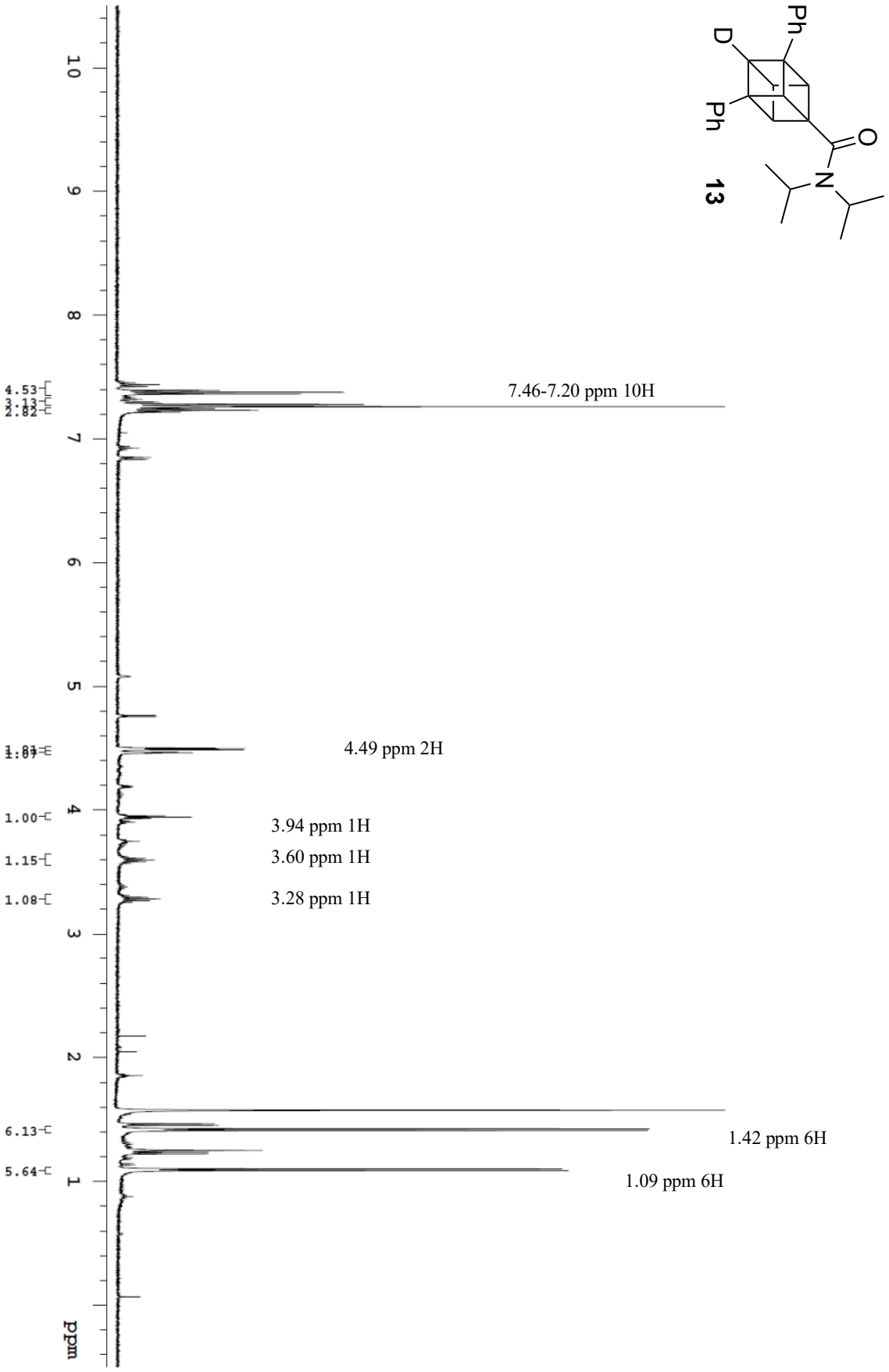
^{1}H NMR NMR Spectra of 12e **(500 MHz, $CDCl_3$)**



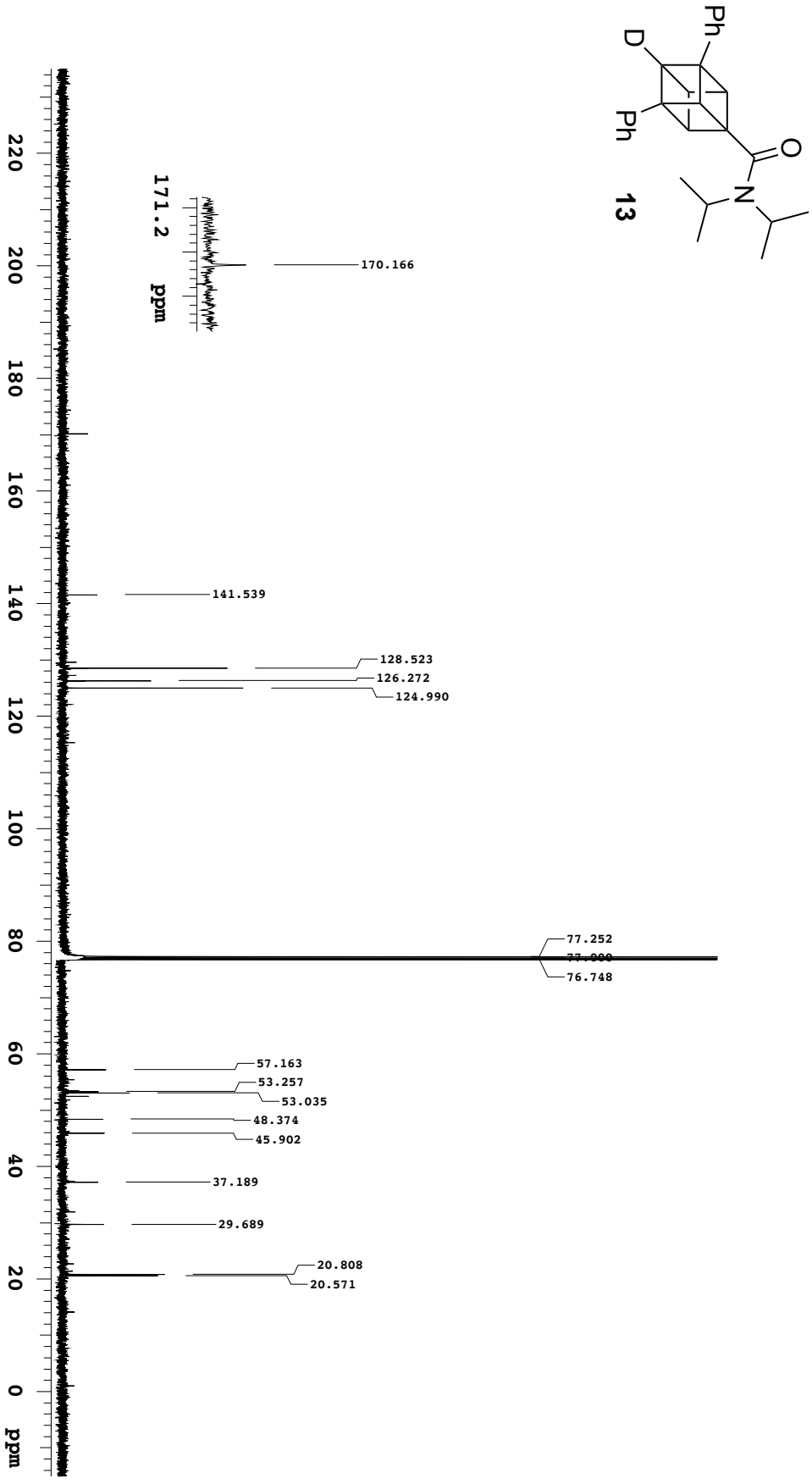
^{13}C NMR Spectra of 12e (125.7MHz, $CDCl_3$)



^{1}H NMR NMR Spectra of 13 **(500 MHz, $CDCl_3$)**



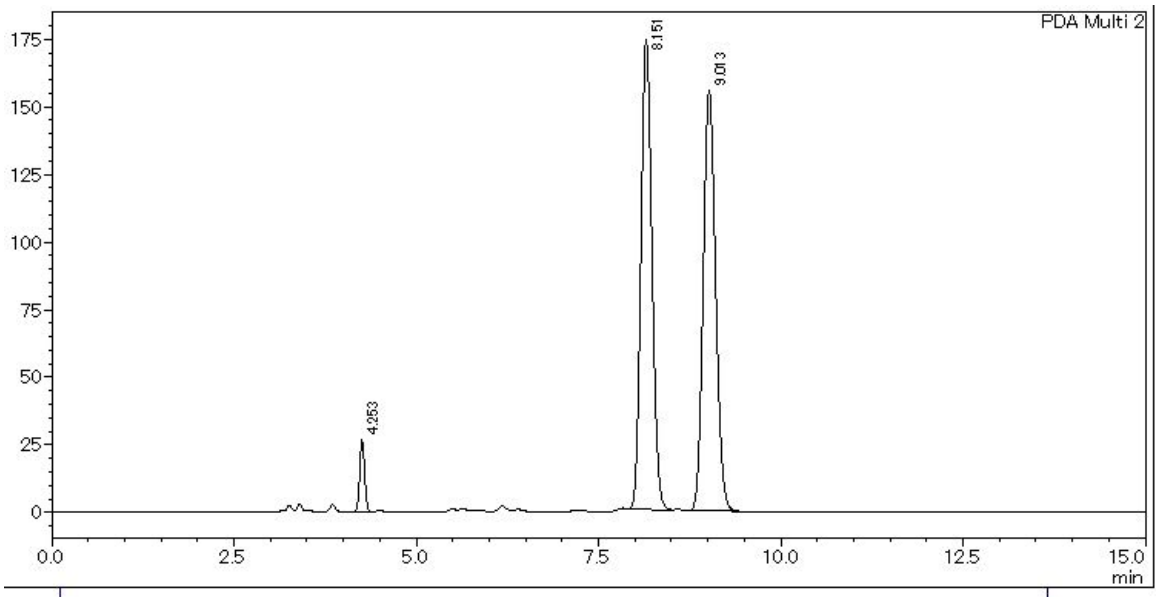
^{13}C NMR Spectra of 13 (125.7MHz, $CDCl_3$)



HPLC Analysis by SHIMADZU Prominence.

3-(4-Bromobenzyl)-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d*

12c (Racemate) DAICEL CHIRAL PAK ID *i*-PrOH/Hexane: 5/95 1.0 mL/min



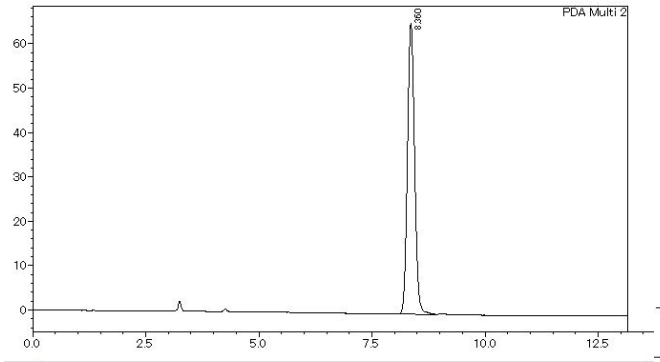
PDA Ch2 220nm 4nm

ピーク#	保持時間	面積	高さ	面積%	高さ%
1	4.253	124887	26582	3.256	7.462
2	8.151	1845700	174008	48.125	48.849
3	9.013	1864640	155626	48.619	43.689
合計		3835227	356216	100.000	100.000

Retention Time (min) | Area Integration | Hight | Area (%) | Hight (%)

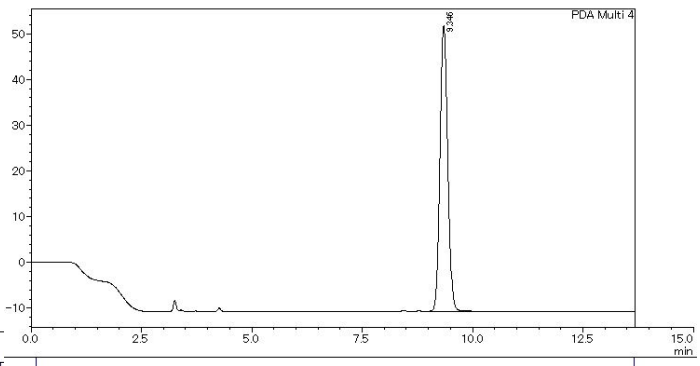
Sum

After Separation by HPLC



PDA Ch2 220nm 4nm

ピーク#	保持時間	面積	高さ	面積%	高さ%
1	8.360	725670	65600	100.000	100.000
合計		725670	65600	100.000	100.000

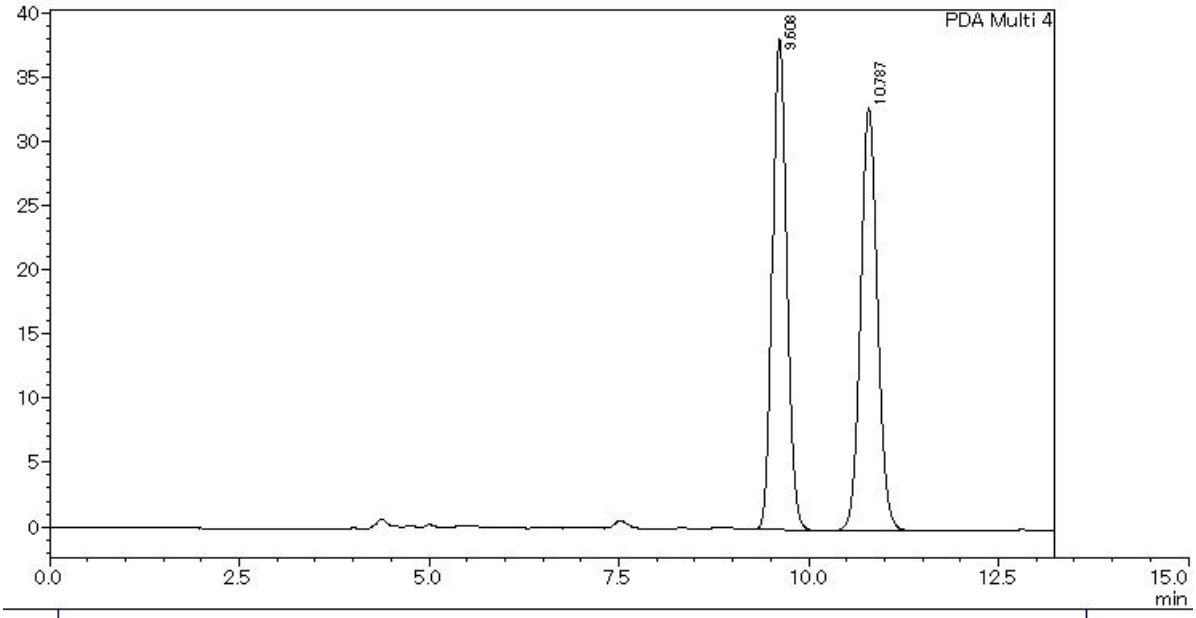


PDA Ch4 220nm 4nm

ピーク#	保持時間	面積	高さ	面積%	高さ%
1	9.346	763221	62515	100.000	100.000
合計		763221	62515	100.000	100.000

3-Benzyl-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12b)

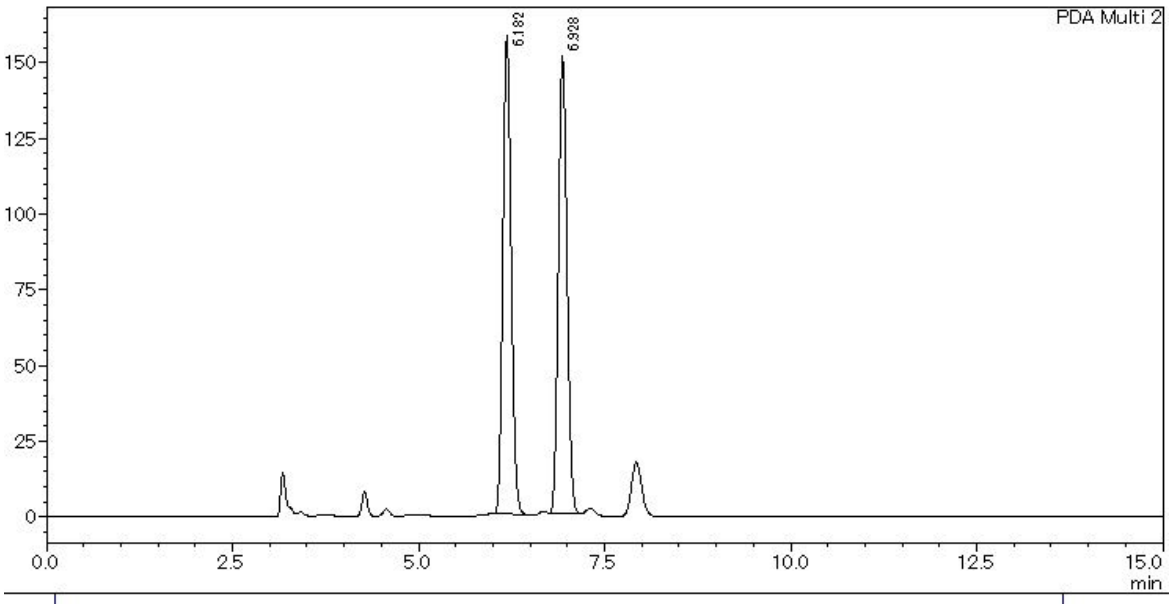
12b (Racemate) DAICEL CHIRAL PAK IC *i*-PrOH/Hexane: 1/99 1.0 mL/min



PDA Ch4 22	Retention Time (min)	Area Integration	Hight	Area (%)	Hight (%)
ピーク#	保持時間	面積	高さ	面積%	高さ%
1	9.608	498289	38277	49.266	53.781
2	10.787	513143	32896	50.734	46.219
Sum 計		1011433	71173	100.000	100.000

3-Iodo-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12d)

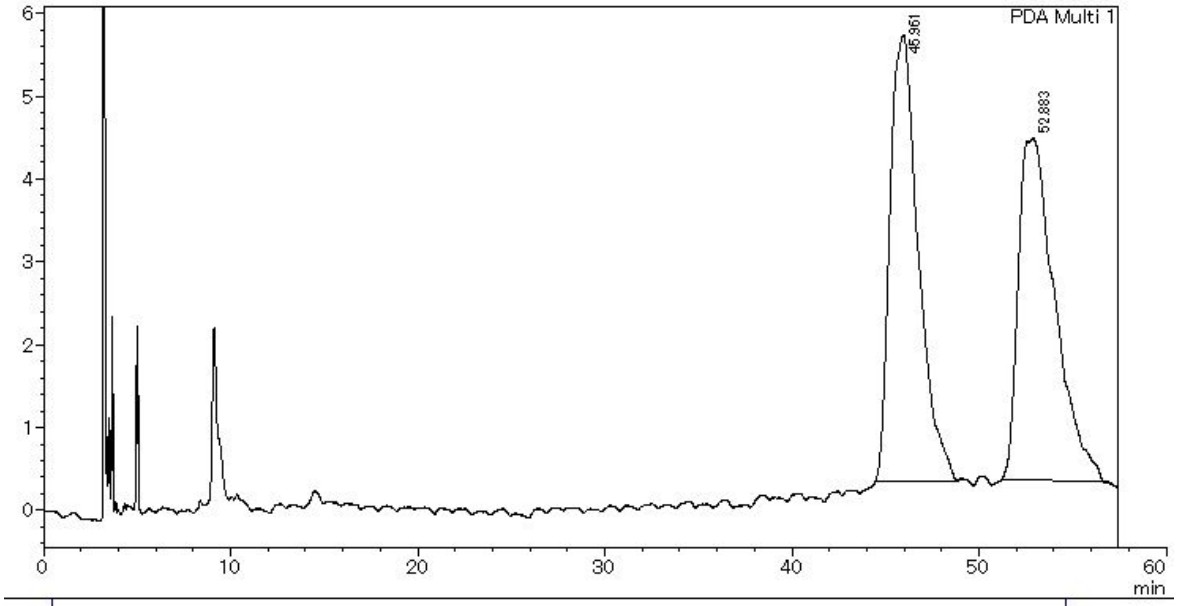
12d (Racemate) DAICEL CHIRAL PAK ID *i*-PrOH/Hexane: 5/95 1.0 mL/min



PDA Ch2 22	Retention Time (min)	Area Integration	Hight	Area (%)	Hight (%)
ピーク#	保持時間	面積	高さ	面積%	高さ%
1	6.182	1247817	158473	50.295	51.203
2	6.928	1233196	151027	49.705	48.797
Sum 計		2481012	309500	100.000	100.000

3-Carboxylateethyl-5-*n*-buthyl-*N*,*N*-diisopropylcubane-1-carboxamide-4-*d* (12e)

12e (Racemate) DAICEL CHIRAL PAK ID *i*-PrOH/Hexane: 1/99 1.0 mL/min



PDA Ch1 2

ピーク#	Retention Time (min)	Area Integration	Hight	Area (%)	Hight (%)
1	45.961	569116	5390	50.429	56.696
2	52.883	559436	4117	49.571	43.304
Sum		1128552	9507	100.000	100.000