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Supporting Information

Facile and Convenient Method of Deuterium Gas Generation through Pd/C-Catalyzed H_2–D_2 Exchange Reaction and Its Application in the Synthesis of Deuterium-Labelled Compounds

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General

The 10% Pd/C was purchased from Sigma-Aldrich Co. (product number; 20569-9, 50 g, Lot. 0581713C). The 10% Rh/C, 10% Ru/C, 10% Ir/C and 5% Au/C were obtained from N. E. Chemcat Co. The 5% Pt/C was purchased from Wako Pure Chemical Industries, Ltd. The Pd black was purchased from Kishida Chemical Co., Ltd. D_2O (>99.9 % D atom) was purchased from Cambridge Isotope Laboratories, Inc. or Spectra Gases, Inc. CD_3CN (99.8% D atom), CD_3OD (99.8% D atom) and CH_3OD (99% D atom) were purchased from Cambridge Isotope Laboratories, Inc. 20% DCl in D_2O (99.5% D atom) was purchased from Cambridge Isotope Laboratories, Inc. or Acros Organics. CH_3CO_2D (98% D atom) was purchased from Acros Organics. Dehydrated cyclohexane was purchased from Kanto Chemical Co., Inc. and they were used without purification. MeOH was distilled from sodium. All other reagents were purchased from commercial sources and used without further purification.

Analytical thin-layer chromatography (TLC) was carried out on pre-coated Silica Gel 60 F_{254} plates (Merck, Art 5715) and visualized with UV light and/or stain (10% phosphomolybdic acid in EtOH). Flash column chromatography was accomplished using a Silica Gel 60 (Merck; 230–400 mesh) or Silica Gel 60N (Kanto Chemical Co., Inc.; 63–210 μm, spherical, neutral). The ^1H, ^2H and ^{13}C NMR spectra were recorded by a JEOL AL 400 spectrometer or a JEOL EX 400 spectrometer (400 MHz for ^1H NMR; 60.7 MHz for ^2H NMR; 100 MHz for ^{13}C NMR). The chemical sifts (δ) are expressed in ppm and are internally referenced to tetramethylsilane or residual solvents (7.27 ppm/$CHCl_3$, 3.30 ppm/CH_3OH, 4.75 ppm/H_2O and 1.93 ppm/MeCN for ^2H NMR). Elemental analyses were performed using YANACO CHN CORDER MT-5 instrument. The EI and FAB mass spectra were taken by a JEOL JMS-SX102A instrument at the Mass Spectrometry Laboratory of the Gifu Pharmaceutical University. In Mass spectra data, M(d_2) indicates the molecular ion peak of deuterated product with two deuterium atoms, and M(d_0) indicates the peak of non-deuterated product. The Pressurized reactions were carried out using a 96 mL of pressure-resistant Hyper-glass cylinder (Taiatsu Techno® Co.). Unless otherwise noted, the deuterium content (D content) was determined by ^1H NMR on the basis of the integration of the aromatic protons.

Quantitative Analysis of DHO by ^{1}H NMR

[Figure 1]

• Reaction Conditions

Condition A: A suspension of 10% Pd/C (10.0 mg, 9.40 μmol) in D_2O (1.00 mL, 55.3 mmol) was stirred at room temperature in test tube under ordinary H_2 atmosphere [balloon (ca. 2 L, 82 mmol)] for 6, 12, 24 and 48 h.

Condition B: A suspension of 10% Pd/C (10.0 mg, 9.40 μmol) in D_2O (1.00 mL, 55.3 mmol) was stirred at room temperature in test tube under ordinary Ar atmosphere [balloon (ca. 2 L, 82 mmol)] for 6, 12, 24 and 48 h.

Condition C: D_2O (1.00 mL, 55.3 mmol) was stirred at room temperature in test tube under ordinary H_2 atmosphere [balloon (ca. 2 L, 82 mmol)] for 6, 12, 24 and 48 h.

• Method for the Determination of DHO Quantity by ^{1}H NMR

After the given time, the reaction mixture was filtered using a membrane filter (Millex®-LH, 0.45 μm). The filtrate (0.5 mL) was added to *p*-methoxybenzoic acid sodium salt (8.70 mg, 0.0500 mmol) as an internal standard, and measured by ^{1}H NMR. At 0 h, D_2O (0.50 mL) was added to *p*-methoxybenzoic acid sodium salt (8.70 mg, 0.0500 mmol) and measured by ^{1}H NMR.

• Results

D_2O (1.00 mL, 55.3 mmol)
10% Pd/C (10.0 mg, 9.40 μmol) ——rt——→ Filtrate (0.5 mL) + *p*-Methoxybenzoic acid sodium salt (8.70 mg, 0.0500 mmol) ————→ ^{1}H NMR
H_2 (balloon, ca. 2 L, 82 mmol)

Condition A

Time (h)	H_2O signal intensity	DHO (mmol)	D_2O (mmol)	DHO/D_2O+DHO (mol %)	D_2 (mL)
0	2.67	0.267	55.0	0.483	—
6	27.4	2.74	52.5	4.96	30.2
12	35.3	3.53	51.7	6.39	39.9
24	54.6	5.46	49.8	9.88	63.4
48	65.6	6.56	48.7	11.9	76.8

Condition B

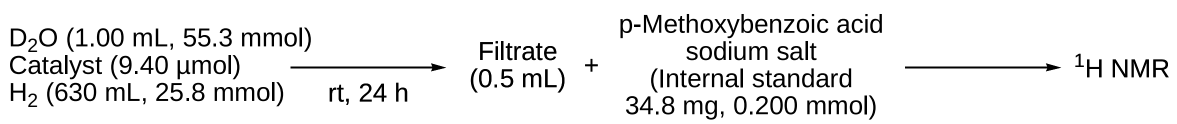
Time (h)	H_2O signal intensity	DHO (mmol)	D_2O (mmol)	DHO/D_2O+DHO (mol %)	D_2 (mL)
0	3.27	0.327	54.9	0.591	—
6	3.60	0.360	54.9	0.651	0.403
12	2.16	0.216	55.1	0.390	0
24	3.43	0.343	54.9	0.620	0.195
48	4.02	0.402	54.9	0.728	0.916

Condition C

Time (h)	H_2O signal intensity	DHO (mmol)	D_2O (mmol)	DHO/D_2O+DHO (mol %)	D_2 (mL)
0	2.32	0.232	55.0	0.419	—
6	2.63	0.263	55.0	0.475	0.379
12	2.69	0.269	55.0	0.487	0.452
24	3.27	0.327	54.9	0.592	1.17
48	4.37	0.437	54.8	0.790	2.50

[Figure 2]

A suspension of 10% Pd/C (10.0 mg), 10% Rh/C (9.70 mg), 10% Ru/C (9.50 mg), 10% Ir/C (18.1 mg), 5% Pt/C (36.7 mg), 10% Au/C (37.0 mg) or Pd black (1.00 mg) (each catalyst: 9.40 μmol) in D_2O (1.00 mL, 55.3 mmol) was stirred at room temperature in a hydrogen-charged sealed flask (630 mL, 25.8 mmol). After 24 h, the reaction mixture was filtered using a membrane filter (Millex®-LH, 0.45 μm). The filtrate (0.5 mL) was added to *p*-methoxybenzoic acid sodium salt (34.8 mg, 0.200 mmol) as an internal standard, and measured by ^1H NMR. At 0 h, D_2O (0.500 mL) was added to *p*-methoxybenzoic acid sodium salt (34.8 mg, 0.200 mmol) and measured by ^1H NMR.

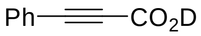


Catalyst	H_2O signal intensity	DHO (mmol)	D_2O (mmol)	DHO/D_2O+DHO (mol %)
Blank	1.77	0.708	54.6	1.28
10% Pd/C	56.0	22.4	32.9	40.6
10% Rh/C	72.8	29.1	26.1	52.7
10% Ru/C	28.5	11.4	43.9	20. 7
10% Ir/C	61.0	24.4	30.9	44.2
5% Pt/C	57.8	23.1	32.1	41.9

Time (h)	H_2O signal intensity	DHO (mmol)	D_2O (mmol)	DHO/D_2O+DHO (mol %)
Blank	1.83	0.73	54.5	1.32
5% Au/C	7.36	2.94	52.3	5.33
Pd black	6.85	2.74	52.5	4.96

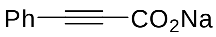
Synthesis of Substrates

Phenylpropiolic acid-d_1 (Table 3, entry 6)



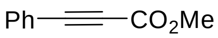
Phenylpropiolic acid (500 mg, 3.40 mmol) was added to D_2O (2.00 mL, 111 mmol) and the reaction mixture was stirred at room temperature for 30 min, and then concentrated in vacuo to give phenylpropiolic acid-d_1 in quantitative yield. Complete displace acidic proton to deuteron was confirmed by ^{1}H NNR spectrum using DMSO-d_6 as a solvent.

Phenylpropiolic acid sodium salt (Table 3, entry 7)



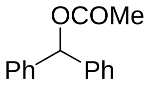
Phenylpropiolic acid (292 mg, 2.00 mmol) and sodium hydroxide (72.0 mg, 1.80 mmol) were added to H_2O (30 mL) and the reaction mixture was stirred at room temperature for 6 h, washed with Et_2O (2 × 10 mL), and then concentrated in vacuo to give phenylpropiolic acid sodium salt (259 mg, 88%).

Methyl phenylpropiolate (Table 3, entry 8)



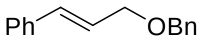
Phenylpropiolic acid (300 mg, 2.10 mmol) and concentrated H_2SO_4 (1 drop) were added to distilled MeOH (3 mL) and the reaction mixture was stirred at 90 °C for 10 h. Then, it was neutralized with saturated $NaHCO_3$ aqueous solution and the product was extracted with Et_2O (40 mL). The organic phases were washed with H_2O (2 × 30 mL) and brine (30 mL), dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by flash column chromatography (hexane : Et_2O = 5 : 1) to give methyl phenylpropiolate (248 mg, 73%) as a colorless oil. ^{1}H NMR spectrum was identical with that in the literature.[1]

Benzhydryl acetate (Table 4, entry 8)



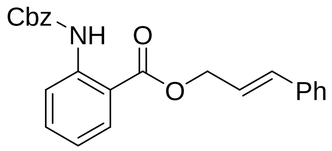
Benzhydrol (1.11 g, 6.00 mmol) and acetic anhydride (1.23 g, 12.0 mmol) were added to dehydrated pyridine (2 mL) and the reaction mixture was stirred at room temperature for 48 h. The mixture was partitioned between Et_2O (100 mL) and water (100 mL). The ethereal layer was washed with 1 N HCl (10 mL), water (100 mL) and brine (30 mL), dried over $MgSO_4$ and evaporated in vacuo. The residue was purified by flash silica gel column chromatography (hexane : Et_2O = 10 : 1) to give benzhydryl acetate (1.31 g, 96%) as a yellow oil. ^{1}H NMR spectrum was identical with that in the literature.[2]

Benzyl cinnamyl ether (Table 6, entry 7)



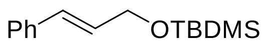
A suspension of cinnamyl alcohol (670 mg, 5.00 mmol) and NaH (60% W/W in mineral oil, 240 mg, 6.00 mmol) in DMF (10 mL) was stirred at 0 ˚C for 30 min. To the reaction mixture was added benzyl bromide (0.734 mL, 6.00 mmol) and the mixture was stirred at room temperature for 48 h and then concentrated in vacuo. The residue was partitioned between ethyl acetate (100 mL) and water (100 mL). The organic layer was washed with water (100 mL) and brine (50 mL), dried over $MgSO_4$ and evaporated in vacuo. The residue was purified by flash silica gel column chromatography (hexane : ethyl acetate = 10 : 1) to give benzyl cinnamyl ether (970 mg, 87%) as a colorless oil. ^{1}H and ^{13}C NMR spectra were identical with those in the literature.[3]

Cinnamyl *N*-(benzyloxycarbonyl)anthranilate (Table 6, entry 8)



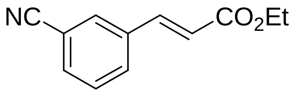
Prepared according to the literature.[4] To a solution of cinnamyl anthranilate (1.20 g, 5.00 mmol) and NaH (60% dispersion in mineral oil, 360 mg, 9.00 mmol) in THF (35 mL) was added benzyl chloroformate (1.54 g, 9.00 mmol). After the solution was stirred at room temperature for 71 h, the mixture was extracted with ethyl acetate (100 mL) and water (100 mL). The organic layer was washed with water (100 mL) and brine (50 mL), dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash silica gel column chromatography (hexane : ethyl acetate = 20 : 1) to give cinnamyl *N*-(benzyloxycarbonyl)anthranilate (1.87 g, 97%) as a colorless solid. ^{1}H NMR spectrum was identical with that in the literature.[4]

***tert*-Butyldimethylsilyl cinnamyl ether (Table 6, entry 9)**



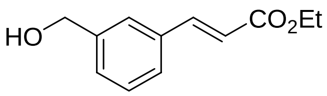
To a solution of cinnamyl alcohol (1.34 g, 10.0 mmol) and imidazol (1.45 g, 22.0 mmol) in DMF (20 mL) was added *tert*-butyldimethyl chlorosilane (1.44 g, 11.0 mmol). After the solution was stirred at room temperature for 21 h and then concentrated in vacuo. The residue was partitioned between ethyl acetate (100 mL) and water (100 mL). The organic layer was washed with water (100 mL) and brine (50 mL), dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash silica gel column chromatography (hexane : ethyl acetate = 30 : 1) to give *tert*-butyldimethylsilyl cinnamyl ether (622 mg, 25%) as a colorless oil. ^{1}H and ^{13}C NMR spectra were identical with those in the literature.[5]

Ethyl *trans*-3'-cyanocinnamate (Table 6, entry 11)



Prepared according to the literature.[6] To a solution of trioctylmethylammonium chloride (606 mg, 1.50 mmol), 10% Pd/C (266 mg, 250 μmol), Et_3N (759 mg, 7.50 mmol) and 3-iodobenzonitirile (1.15 g, 5.00 mmol) in isooctane (30 mL) and water (15 mL) was added ethyl acrylate (751 mg, 7.50 mmol). After the solution was stirred at 100 °C for 20 h, the mixture was extracted with ethyl acetate (100 mL) and water (100 mL). The organic layer was washed with water (100 mL) and brine (50 mL), dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash silica gel column chromatography (hexane : ethyl acetate = 5 : 1) to give ethyl *trans*-3'-cyanocinnamate (575 mg, 57%) as a colorless solid: ^{1}H NMR ($CDCl_3$) δ 1.35 (t, J = 7.1 Hz, 3H), 4.29 (q, J = 7.1 Hz, 2H), 6.49 (d, J = 16.4 Hz, 1H), 7.52 (t, J = 7.7 Hz, 1H), 7.64 (d, J = 16.4 Hz, 1H), 7.66 (dt, J = 7.7, 1.5 Hz, 1H), 7.74 (dt, J = 7.7, 1.5 Hz, 1H), 7.79 (t, J = 1.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 60.9, 113.4, 118.2, 121.1, 129.8, 131.3, 131.9, 133.2, 135.8, 141.8, 166.2; MS (EI) m/z (%): 201 (33) [M^+], 173 (22), 156 (100), 128 (34); HRMS (EI): m/z: calcd for $C_{12}H_{11}NO_2$: 201.0790, found 201.0795 [M^+]. Anal. calcd for $C_{12}H_{11}NO_2$: C 71.63, H 5.51; found C 71.49, H 5.54, mp. 66.0–67.0 °C.

Ethyl *trans*-3'-hydroxymethylcinnamate (Table 6, entry 12)



Prepared according to the literature.[6] To a solution of trioctylmethylammonium chloride (606 mg, 1.50 mmol), 10% Pd/C (266 mg, 250 μmol), Et_3N (759 mg, 7.50 mmol) and 3-iodobenzyl alcohol (1.17 g, 5.00 mmol) in isooctane (30 mL) and water (15 mL) was added ethyl acrylate (751 mg, 7.50 mmol). After the solution was stirred at 100 °C for 24 h, the mixture was extracted with ethyl acetate (100 mL) and water (100 mL). The organic layer was washed with water (100 mL) and brine (50 mL), dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash silica gel column chromatography (hexane : ethyl acetate = 5 : 1) to give ethyl *trans*-3'-hydroxymethylcinnamate (556 mg, 54%) as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.33 (t, J = 7.1 Hz, 3H), 4.25 (q, J = 7.1 Hz, 2H), 6.43 (d, J = 15.9 Hz, 1H), 7.35–7.37 (m, 2H), 7.41 (m, 1H), 7.51 (s, 1H), 7.65 (d, J = 15.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 60.5, 64.8, 118.5, 126.4, 127.3, 128.7, 129.1, 134.7, 141.7, 144.4, 167.1; MS (EI) m/z (%): 206 (100) [M^+], 161 (50), 143 (54), 131 (68), 115 (15), 103 (27), 77 (17); HRMS (EI): m/z: calcd for $C_{12}H_{14}O_3$: 206.0943, found 206. 0946 [M^+]. Anal. calcd for $C_{12}H_{14}O_3 \cdot 0.1H_2O$: C 69.28, H 6.88; found C 69.14, H 6,72.

Procedure for One-Pot Reductive Deuteration Using Pd/C–D_2O–H_2 System (Table 2, entry 1, Table 3 and Table 4, entries 6–9)

10% Pd/C (10 wt % of the substrate, Sigma-Aldrich Co.) and D_2O (3 mL, 166 mmol) was set in a 100 mL round-bottom flask (actual internal volume of the flask is 160 mL, 6.5 mmol). The system was sealed with a septum and filled with H_2 by five vacuum/H_2 cycles. The mixture was stirred at room temperature for 24 h and the substrate (0.5 mmol) (as a 0.5 mL MeOH or acetone solution in the case of solid substrate) was added. The mixture was stirred at room temperature (except for 50 °C, Table 4, entry 9) for the given time in the table, diluted with Et_2O (10 mL), and passed through a membrane filter (Millex®-LH, 0.45 μm). The filtrate was separated into two layers. The aqueous layer was extracted with Et_2O (2 × 15 mL) and the combined organic layers were washed with brine (30 mL), dried over $MgSO_4$, filtered, and concentrated in vacuo to give the analytically pure deuterated product.

The D content was determined by ^1H NMR on the basis of the integration of the ethyl protons (Table 3, entry 9).

(Table 4, entries 1–5)

10% Pd/C (10 wt % of the substrate, Sigma-Aldrich Co.) and D_2O (3 mL, 166 mmol) was set in a 100 mL round-bottom flask (actual internal volume of the flask is 160 mL, 6.5 mmol). The system was sealed with a septum and filled with H_2 by five vacuum/H_2 cycles. The mixture was stirred at room temperature for 24 h and the substrate (0.5 mmol) (as a 0.5 mL MeOH or acetone solution in the case of solid substrate) and Et_3N[7] (83.6 μL, 0.6 mmol) were added. The mixture was stirred at room temperature for the given time in the table, diluted with Et_2O (10 mL), and passed through a membrane filter (Millex®-LH, 0.45 μm). The filtrate was neutralized using a 5% $NaHSO_4$ aqueous solution and extracted with Et_2O (2 × 15 mL). The ethereal layers were washed with water (10 mL) and brine (30 ml), dried over $MgSO_4$, filtered, and concentrated in vacuo to give the analytically pure deuterated product.

The D content was determined by ^1H NMR of the corresponding methyl ester on the basis of the integration of the methyl protons (entries 1–4) or the corresponding acetate on the basis of the integration of the acetyl protons (entry 5).

(Table 4, entries 10, 11)

Followed by the Procedure for One-Pot Reductive Deuteration Using Pd/C–D_2O–H_2 System except for the addition of DCl (117 µL, 0.75 mmol) as a 20 wt % solution in D_2O together with substrate.

Procedure for One-Pot Reductive Deuteration of Aromatic Nuclei[8] Using Rh/C–D_2O–H_2 System (Scheme 1)

10% Rh/C (116.5 mg, 20 wt % of the substrate, N. E. Chemcat. Co., K type, dry) and D_2O (3 mL, 166 mmol) was set in a 100 mL round-bottom flask (actual internal volume of the flask is 160 mL, 6.5 mmol). The system was sealed with a septum and filled with H_2 by five vacuum/H_2 cycles. The mixture was stirred at room temperature for 24 h and 5-phenoxy-*n*-valeric acid (97.1 mg, 0.5 mmol) (as a 0.5 mL CH_3OD solution) was added. The mixture was stirred at 50 °C for 24 h. The resulting reaction mixture was diluted with Et_2O (10 mL) and passed through a membrane filter (Millex®-LH, 0.45 µm). The filtrate was separated into two layers. The aqueous layer was extracted with Et_2O (2 × 15 mL) and the combined organic layers were washed with brine (30 mL), dried over $MgSO_4$, filtered, and concentrated in vacuo to give the analytically pure 5-cyclohexyloxy-*n*-varelic acid-d_6 (**25-d**, 49.1 mg, 49%) as a colorless oil.

The D content was determined by ^1H NMR of the corresponding methyl ester on the basis of the integration of the methyl protons.

Capturing Method of D_2 Generated in situ (Table 5, entry 8 and Scheme 2)

A suspension of 10% Pd/C (50.0 mg, Sigma-Aldrich Co.) in D_2O (10 mL, 553 mmol) was stirred in a 96 mL pressure-resistant Hyper-glass cylinder (Taiatsu Techno® Co.) under 3 atm of H_2 pressure (internal H_2 gas is ca. 260 mL under atmospheric pressure) at room temperature for 48 h. The generated D_2 gas was captured in a rubber balloon (ca. 170 mL).

Application of Capturing D_2 to the Pd/C-Catalyzed Reductive Mono-*N*-Alkylation[9] Using Nitrile (Scheme 2)

A mixture of the nitrobenzene (61.6 mg, 0.5 mmol), 10% Pd/C (12.3 mg, 20 wt % of nitrobenzene, Sigma-Aldrich Co.) in CD_3CN (D content: 99.8%, 14.4 mmol, 0.75 mL) in a test tube was stirred under the captured D_2 (balloon, ca. 170 mL) at room temperature for 24 h. The reaction mixture was diluted with Et_2O (30 mL), and passed through a membrane filter (Millex®-LH, 0.45 μm). The filtrate was concentrated in vacuo. The residue was purified by flash silica gel column chromatography (hexane : Et_2O = 20 : 1) to give *N*-ethylaniline-d_5 (**26-d**, 48.1 mg, 79%) as a colorless oil.

Typical Procedure for Chemoselective One-Pot Reductive Deuteration of Olefin and Acetylene Using Pd/C–D_2O–H_2–Ph_2S System (Table 6)

10% Pd/C (10 wt % of the substrate, Sigma-Aldrich, Co.) and D_2O (3 mL, 166 mmol) was set in a 100 mL round-bottom flask (actual internal volume of the flask is 160 mL, 6.5 mmol). The system was sealed with a septum and filled with H_2 by five vacuum/H_2 cycles. The mixture was stirred at room temperature for 24 h and the substrate (0.5 mmol) (as a 0.5 mL MeOH or acetone solution in the case of solid substrate) and Ph_2S (0.93 mg, 0.005 mmol) were added. The mixture was stirred at room temperature for the given time in the table, diluted with ethyl acetate (10 mL), and passed through a membrane filter (Millex®-LH, 0.45 μm). The filtrate was separated into two layers. The aqueous layer was extracted with ethyl acetate (2 × 15 mL) and the combined organic layers were washed with and brine (30 mL), dried over $MgSO_4$, filtered, and concentrated in vacuo. The residue was purified by flash silica gel column chromatography (hexane/ethyl acetate) to give the analytically pure deuterated product.

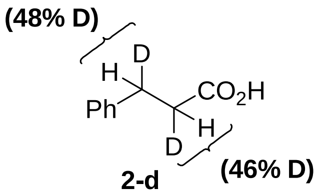
Pd/C-Catalyzed Chemoselective Hydrogenation in the Presence of Ph_2S[10]

Pd/C + Ph_2S

Reducible	Irreducible	
Olefin	ArCOR	$ArCH_2OH$
Acetylene	$-CO_2Bn$	ArCl
$R-N_3$	-*N*CBz	-*O*Bn
	ArCN	-OTBDMS

Characterization Data

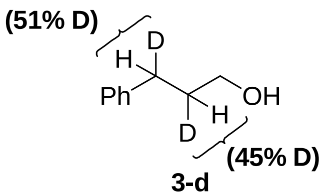
Dideuterocinnamic acid (3-Phenylpropionic acid-d_2) (2-d, Table 1, entry 2)



2-d

^{1}H NMR (CD_3OD) δ 2.47 (m, 1.07H), 2.79 (m, 1.01H), 7.05–7.18 (m, 5H); ^{2}H NMR (CH_3OH) δ 2.56 (br s), 2.87 (br s); MS (EI) m/z (%): 152 (48) $[M(d_2)]^+$, 151 (14) $[M(d_1)]^+$, 150 (2) $[M(d_0)]^+$.

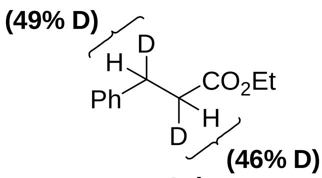
3-Phenyl-1-propanol-d_2 (Table 3, entry 1)



3-d

^{1}H NMR (CD_3OD) δ 1.80 (m, 1.10H), 2.64 (m, 0.987H), 3.55 (t, J = 5.9 Hz, 2H), 7.11–7.25 (m, 5H); MS (EI) m/z (%): 138 (24) $[M(d_2)]^+$, 137 (12) $[M(d_1)]^+$, 136 (5) $[M(d_0)]^+$, 119 (82), 92 (100).

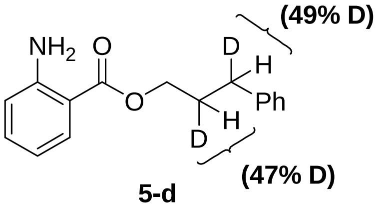
Ethyl 3-Phenylpropanoate-d_2 (Table 3, entry 2)



4-d

^{1}H NMR (CD_3OD) δ 1.09 (t, J = 7.1 Hz, 3H), 2.48 (m, 1.09H), 2.78 (m, 1.03H), 3.98 (q, J = 7.1 Hz, 2H), 7.04–7.17 (m, 5H); ^{2}H NMR (CH_3OH) δ 2.58 (br s), 2.86 (br s); MS (EI) m/z (%): 180 (43) $[M(d_2)]^+$, 179 (14) $[M(d_1)]^+$, 178 (3) $[M(d_0)]^+$, 107 (56), 106 (100), 105 (73), 92 (68).

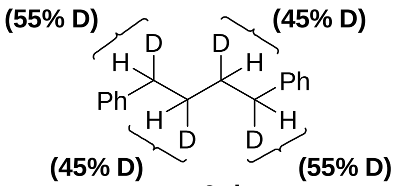
3-Phenylpropyl anthranilate-d_2 (Table 3, entry 3)



5-d

^{1}H NMR (CD_3OD) δ 2.02 (m, 1.06H), 2.73 (m, 1.03H), 4.22 (d, J = 6.3 Hz, 2H), 6.56 (m, 1H), 6.73 (d, J = 8.2 Hz, 1H), 7.13–7.27 (m, 6H), 7.76 (dd, J = 8.2, 1.4 Hz, 1H); ^{2}H NMR (CH_3OH) δ 2.02 (br s), 2.70 (br s); MS (EI) m/z (%): 257 (93) $[M(d_2)]^+$, 256 (30) $[M(d_1)]^+$, 255 (2) $[M(d_0)]^+$, 138 (84), 137 (100), 120 (63), 119 (87), 92 (66).

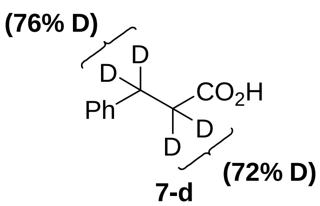
1,4-Diphenylbutane-d_4 (Table 3, entry 4)



6-d

^{1}H NMR (CD_3OD) δ 1.49 (m, 2.21H), 2.48 (m, 1.82H), 7.00–7.03 (m, 6H), 7.10–7.14 (m, 4H); ^{2}H NMR (CH_3OH) δ 1.56 (br s), 2.54 (br s); MS (EI) m/z (%): 214 (52) $[M(d_4)]^+$, 213 (33) $[M(d_3)]^+$, 212 (9) $[M(d_2)]^+$, 211 (1) $[M(d_1)]^+$, 210 (0) $[M(d_0)]^+$, 93 (72), 92 (100).

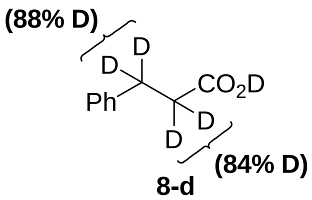
3-Phenylpropionic acid-d_4 (Table 3, entry 5)



7-d

^{1}H NMR (CD_3OD) δ 2.62 (m, 0.56H), 2.93 (m, 0.48H), 7.18–7.34 (m, 5H); MS (EI) m/z (%): 154 (35) $[M(d_4)]^+$, 153 (41) $[M(d_3)]^+$, 152 (21) $[M(d_2)]^+$, 151 (5) $[M(d_1)]^+$, 150 (1) $[M(d_0)]^+$, 109 (20), 108 (28), 107 (50), 106 (42), 105 (20), 93 (96), 92 (100), 91 (24).

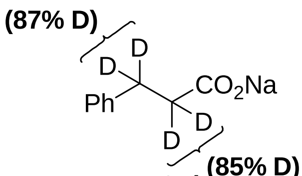
3-Phenylpropionic acid-d_5 (Table 3, entry 6)



8-d

^{1}H NMR (CD_3OD) δ 2.53 (m, 0.31H), 2.85 (m, 0.25H), 7.13–7.25 (m, 5H); MS (EI) m/z (%): 155 (8) $[M(d_5)]^+$, 154 (42) $[M(d_4)]^+$, 153 (26) $[M(d_3)]^+$, 152 (6) $[M(d_2)]^+$, 151 (4) $[M(d_1)]^+$, 150 (1) $[M(d_0)]^+$, 109 (22), 108 (33), 107 (46), 106 (25), 105 (10), 93 (100), 92 (58), 91 (10).

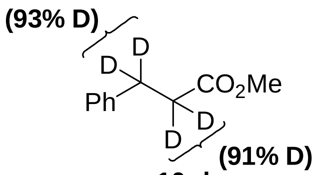
3-Phenylpropionic acid-d_4 sodium salt (Table 3, entry 7)



9-d

The obtained **9-d** was dissolved in water (0.5 mL) followed by the addition of few drops of 1 N HCl. The mixture was shaken with ethyl acetate (1 mL) and the organic layer was used as the mass sample. ^{1}H NMR (DMSO-d_6) δ 2.09 (m, 0.31H), 2.70 (m, 0.26H), 7.08–7.23 (m, 5H); MS (EI) m/z (%): 154 (14) $[M(d_4)]^+$, 153 (10) $[M(d_3)]^+$, 152 (2) $M(d_2)]^+$, 151 (1) $[M(d_1)]^+$, 150 (1) $[M(d_0)]^+$, 84 (90), 66 (100).

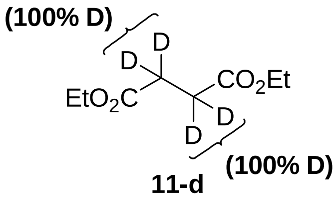
Methyl 3-phenylpropionate-d_4 (Table 3, entry 8)



10-d

^{1}H NMR (CD_3OD) δ 2.53 (m, 0.17H), 2.82 (m, 0.14H), 3.57 (s, 3H), 7.09–7.22 (m, 5H); MS (EI) m/z (%): 168 (58) $[M(d_4)]^+$, 167 (32) $[M(d_3)]^+$, 166 (73) $[M(d_2)]^+$, 165 (27) $[M(d_1)]^+$, 164 (6) $[M(d_0)]^+$, 109 (51), 108 (76), 107 (82), 106 (30), 105 (15), 93 (100), 92 (21).

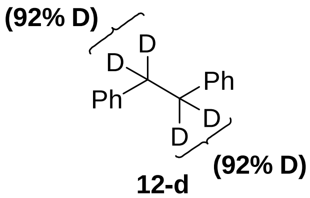
Diethyl succinate-d_4 (Table 3, entry 9)



11-d

^{1}H NMR (CD_3OD) δ 1.21 (t, J = 7.3 Hz, 6H), 4.20 (q, J = 7.3 Hz, 4H), (The proton peak corresponding to ethylene group, which was generated after deuterogenation, was not detected within the limits of ^{1}H NMR spectroscopy.); MS (EI) m/z (%): 178 (4) $[M(d_4)]^+$, 177 (29) $[M(d_3)]^+$, 176 (1) $[M(d_2)]^+$, 175 (1) $[M(d_1)]^+$, 174 (1) $[M(d_0)]^+$, 150 (89), 122 (81), 70 (100).

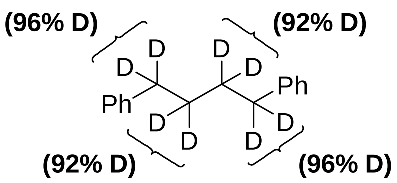
Diphenylethane-d_4 (Table 3, entry 10)



12-d

^{1}H NMR (CD_3OD) δ 2.83 (s, 0.32H), 7.19–7.23 (m, 10H); MS (EI) m/z (%): 186 (30) $[M(d_4)]^+$, 185 (11) $[M(d_3)]^+$, 184 (10) $[M(d_2)]^+$, 183 (3) $[M(d_1)]^+$, 182 (5) $[M(d_0)]^+$, 93 (100), 92 (19).

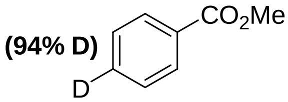
1,4-Diphenylbutane-d_8 (Table 3, entry 11)



13-d

^{1}H NMR (CD_3OD) δ 1.48 (s, 0.32H), 2.48 (s, 0.18H), 7.00–7.04 (m, 6H), 7.11–7.14 (m, 4H); ^{2}H NMR (CH_3OH) δ 1.44 (br s), 2.43 (br s); MS (EI) m/z (%): 218 (53) $[M(d_8)]^+$, 217 (31) $[M(d_7)]^+$, 216 (8) $[M(d_6)]^+$, 215 (2) $[M(d_5)]^+$, 214 (1) $[M(d_4)]^+$, 213 (0) $[M(d_3)]^+$, 212 (0) $[M(d_2)]^+$, 209 (0) $[M(d_1)]^+$, 208 (0) $[M(d_0)]^+$, 93 (100).

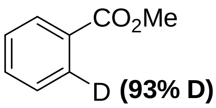
Methyl 4-deuterobenzoate (Table 4, entry 1)



14-d

^{1}H NMR (CD_3OD) δ 3.93 (s, 3H), 7.49–7.50 (m, 2H), 7.62 (m, 0.06H), 8.04–8.05(m, 2H); MS (EI) m/z (%): 137 (42) $[M(d_1)]^+$, 136 (9) $[M(d_0)]^+$, 106 (100), 105 (20), 78 (53).

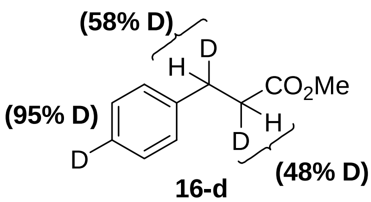
Methyl 2-deuterobenzoate (Table 4, entry 2)



15-d

^{1}H NMR (CD_3OD) δ 3.87 (s, 3H), 7.43–7.46 (m, 2H), 7.55–7.57 (1H, m), 7.99 (m, 1.14H); MS (EI) m/z (%): 137 (43) $[M(d_1)]^+$, 136 (12) $[M(d_0)]^+$, 106 (100), 105 (25), 78 (55).

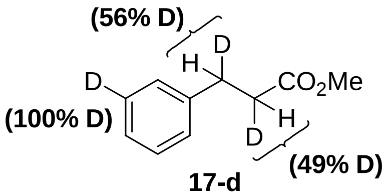
Methyl 4'-deutero-3-phenylpropionate-d_2 (Table 4, entry 3)



16-d

^{1}H NMR (CD_3OD) δ 2.55 (m, 1.05H), 2.84 (m, 0.84H), 3.58 (s, 3H), 7.11–7.22 (m, 4.05H); MS (EI) m/z (%): 167 (40) $[M(d_3)]^+$, 166 (12) $[M(d_2)]^+$, 165 (4) $[M(d_1)]^+$, 164 (1) $[M(d_0)]^+$, 108 (51), 107 (100), 106 (55), 93 (59).

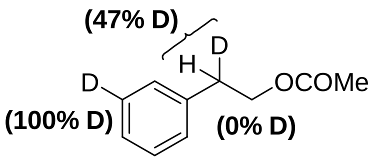
Methyl 3’-deutero-3-phenylpropionate-d_2 (Table 4, entry 4)



17-d

^{1}H NMR (CD_3OD) δ 2.51 (m, 1.02H), 2.80 (m, 0.90H), 3.54 (s, 3H), 7.06–7.20 (m, 3.96H); MS (EI) m/z (%): 167 (42) [M(d_3)]$^+$, 166 (14) [M(d_2)]$^+$, 165 (2) [M(d_1)]$^+$, 164 (1) [M(d_0)]$^+$, 108 (49), 107 (100), 106 (55), 93 (65).

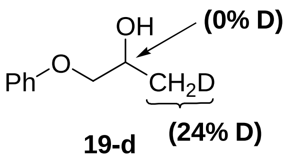
3-Deutero-2’-phenylethyl acetate-d_1 (Table 4, entry 5)



18-d

^{1}H NMR (CD_3OD) δ 2.04 (3H, s), 2.96 (m, 1.06H), 4.30 (m, 2H), 7.24–7.35 (m, 3.96H); MS (EI) m/z (%): 166 (2) [M(d_2)]$^+$, 165 (3) [M(d_1)]$^+$, 164 (1) [M(d_0)]$^+$, 107 (24), 106 (62), 105 (69), 43 (100).

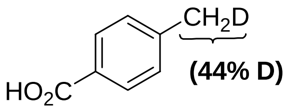
1-Phenoxymethyl ethanol-d_1 (Table 4, entry 6)



19-d

^{1}H NMR (CD_3OD) δ 1.31 (m, 2.28H), 3.90 (m, 2H), 4.14 (m, 1H), 6.94–6.99 (3H, m), 7.28–7.32 (2H, m); MS (EI) m/z (%): 153 (24) [M(d_1)]$^+$, 152 (6) [M(d_0)]$^+$, 108 (19), 94 (100), 77 (27).

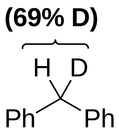
Toluic acid-d_1 (Table 4, entry 7)



20-d

^{1}H NMR (CD_3OD) δ 2.36 (s, 1.68H), 7.24–7.36 (2H, m), 7.88–7.91 (2H, m); MS (EI) m/z (%): 139 (48) $[M(d_3)]^+$, 138 (73) $[M(d_2)]^+$, 137 (71) $[M(d_1)]^+$, 136 (24) $[M(d_0)]^+$, 120 (74), 94 (48), 93 (77), 92 (100), 91 (48), 107 (100), 106 (55), 93 (59).

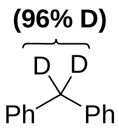
Diphenylmethane-d_1 (Table 4, entry 8)



21-d

^{1}H NMR (CD_3OD) δ 3.93 (s, 0.62H), 7.18–7.28 (m, 10H); MS (EI) m/z (%): 170 (66) $[M(d_2)]^+$, 169 (100) $[M(d_1)]^+$, 168 (72) $[M(d_0)]^+$.

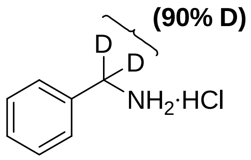
Diphenylmethane-d_2 (Table 4, entry 9)



22-d

^{1}H NMR (CD_3OD) δ 3.80 (s, 0.29H), 7.01–7.07 (m, 6H), 7.11–7.15 (m, 4H); ^{2}H NMR (CH_3OH) δ 3.88 (br s); MS (EI) m/z (%): 170 (100) $[M(d_2)]^+$, 169 (89) $[M(d_1)]^+$, 168 (37 $[M(d_0)]^+$, 93 (15).

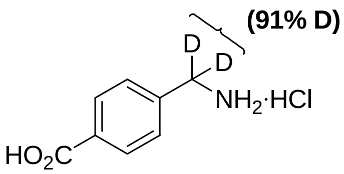
Benzylamine-d_2 hydrochloride (Table 4, entry 10)



23-d

^{1}H NMR (CD_3OD) δ 4.10 (s, 0.20H), 7.42–7.52 (m, 5H); ^{2}H NMR (CH_3OH) δ 4.15 (d, J = 7.7 Hz); MS (EI) m/z (%): 109 (56) $[M(d_2)–HCl]^+$, 108 (100) $[M(d_1)–HCl]^+$, 107 (89) $[M(d_0)–HCl]^+$, 93 (77).

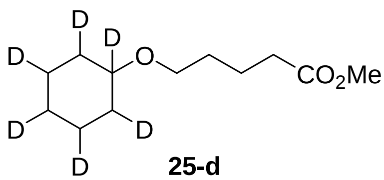
4-Aminomethylbenzoic acid-d_2 hydrochloride (Table 4, entry 11)



24-d

^{1}H NMR (D_2O) δ 4.10 (s, 0.19H), 7.39–7.43 (m, 2H), 7.88–7.93 (m, 2H); ^{2}H NMR (H_2O) δ 4.21 (br s); MS (EI) m/z (%): 153 (31) $[M(d_2)–HCl]^+$, 152 (45) $[M(d_1)–HCl]^+$, 151 (66) $[M(d_0)–HCl]^+$, 137 (47), 136 (24), 135 (61), 108 (100), 107 (88), 106 (63).

Methyl 5-(cyclohexyloxy)pentanate-d_6 (Scheme 1)

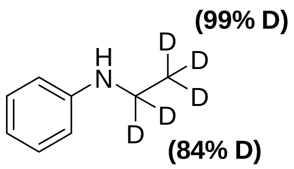


25-d

D content at cyclohexyl group: 48%
(Theoretical D content 55%)

^{1}H NMR (CD_3OD) δ 1.15–1.26 (m, 3.53H), 1.49 (m, 0.13H), 1.55–1.74 (m, 5.50H), 1.84–1.88 (m, 0.22H), 2.34 (t, J = 7.5 Hz, 2H), 3.18 (m, 0.09H), 3.44 (t, J = 6.3 Hz, 2H), 3.47–3.51 (m, 0.22H), 3.67 (s, 3H); ^{2}H NMR (CH_3OH) δ 1.19 (br s), 1.48 (br s), 1.67 (br s), 1.87 (br, s), 3.17 (br s), 3.42 (br s), 3.44 (br s); MS (NBA, FAB) m/z (%): 221 (9) $[M^+(d_6)+H]$, 220 (6) $[M^+(d_5)+H]$, 219 (7) $[M^+(d_4)+H]$, 218 (2) $[M^+(d_3)+H]$, 217 (3) $[M^+(d_2)+H]$, 216 (3) $[M^+(d_1)+H]$, 215 (3) $[M^+(d_0)+H]$.

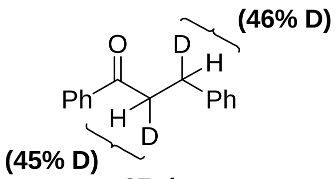
***N*-Ethylaniline-d_5 (Scheme 2)**



26-d

^{1}H NMR (CD_3OD) δ 1.15 (m, 0.03H), 3.06 (d, *J* = 7.2 Hz, 0.33H), 6.59–6.64 (m, 3H), 7.08 (m, 2H); ^{2}H NMR (CH_3OH) δ 1.15 (br s), 3.04 (br s); MS (EI) m/z (%): 126 (44) $[M(d_5)]^+$, 125 (36) $[M(d_4)]^+$, 124 (12) $[M(d_3)]^+$, 123 (17) $[M(d_2)]^+$, 122 (8) $[M(d_1)]^+$, 121 (13) $[M(d_0)]^+$, 108 (100), 107 (56).

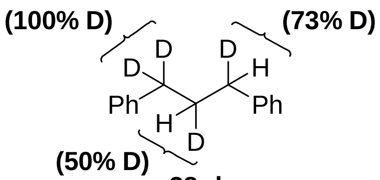
3-Phenylpropiophenone-d_2 (Table 6, entry 1)



27-d

^{1}H NMR (CD_3CN) δ 2.98 (m, 1.09H), 3.31 (m, 1.10H), 7.18 (m, 1H), 7.25–7.30 (m, 4H), 7.48 (m, 2H), 7.59 (tt, *J* = 7.4, 1.5 Hz, 1H), 7.96 (m, 2H); ^{2}H NMR (CH_3CN) δ 3.00 (br s), 3.34 (br s); MS (EI) m/z (%): 212 (52) $[M(d_2)]^+$, 211 (21) $[M(d_1)]^+$, 210 (3) $[M(d_0)]^+$, 105 (100), 77 (27).

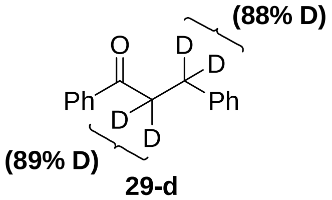
1,3-Diphenylpropane-d_4 (Table 6, entry 2)



28-d

^{1}H NMR (CD_3OD) δ 1.77 (m, 1.00H), 2.48 (m, 0.55H), 7.02–7.06 (m, 6H), 7.14 (m, 4H); ^{2}H NMR (CH_3OH) δ 1.84 (br s), 2.54 (br s); MS (EI) m/z (%): 200 (17) $[M(d_4)]^+$, 199 (6) $[M(d_3)]^+$, 198 (7) $[M(d_2)]^+$, 197 (33) $[M(d_1)]^+$, 196 (24) $[M(d_0)]^+$, 108 (100).

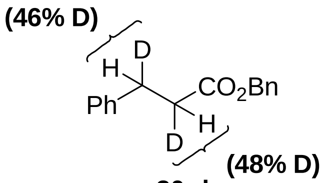
3-Phenylpropiophenone-d_4 (Table 6, entry 3)



29-d

^{1}H NMR (CD_3CN) δ 2.96 (m, 0.22H), 3.28 (m, 0.24H), 7.18 (m, 1H), 7.25–7.28 (m, 4H), 7.48 (m, 2H), 7.59 (m, 1H), 7.96 (m, 2H); ^{2}H NMR (CH_3CN) δ 2.97 (br s), 3.30 (br s); MS (EI) m/z (%): 214 (42) $[M(d_4)]^+$, 213 (26) $[M(d_3)]^+$, 212 (8) $[M(d_2)]^+$, 211 (1) $[M(d_1)]^+$, 210 (1) $[M(d_0)]^+$, 105 (100), 77 (26).

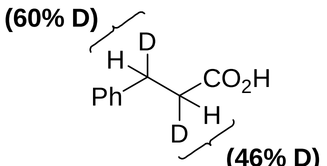
Benzyl 3-phenylpropionate-d_2 (Table 6, entry 4)



30-d

^{1}H NMR (CD_3OD) δ 2.54 (m, 1.05H), 2.79 (m, 1.08H), 4.97 (s, 2H), 7.03–7.23 (m, 10H); ^{2}H NMR (CH_3OH) δ 2.64 (br s), 2.87 (br s); MS (NBA, FAB) m/z (%): 243 (7) $[M(d_2)]^+$, 242 (4) $[M(d_1)]^+$, 241 (1) $[M(d_0)]^+$, 154 (100), 137 (54), 136 (64), 91 (38).

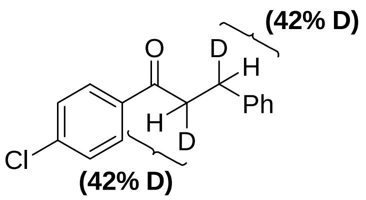
3-Phenylpropionic acid-d_2 (Table 6, entry 5)



2-d

^{1}H NMR (CD_3OD) δ 2.47 (m, 1.08H), 2.78 (m, 0.80H), 7.04–7.18 (m, 5H); ^{2}H NMR (CH_3OH) δ 2.52 (br s), 2.84 (br s); MS (EI) m/z (%): 152 (49) $[M(d_2)]^+$, 151 (13) $[M(d_1)]^+$, 150 (1) $[M(d_0)]^+$, 106 (53), 92 (100).

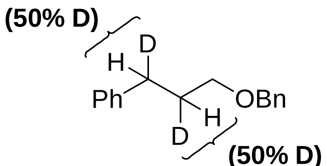
4’-Chloro-3-phenylpropiophenone-d_2 (Table 6, entry 6)



31-d

^{1}H NMR (CD_3CN) δ 2.89 (m, 1.17H), 3.21 (m, 1.17H), 7.09–7.12 (m, 4H), 7.41 (dt, J = 8.7, 1.9 Hz, 2H), 7.85 (dt, J = 8.7, 1.9 Hz, 2H); ^{2}H NMR (CH_3CN) δ 2.98 (br s), 3.31 (br s); MS (EI) m/z (%): 248 (12) 246 (40)[M(d_2)]$^+$, 247 (15) 245 (18) [M(d_1)]$^+$, 246 (40) 244 (3)[M(d_0)]$^+$, 141 (31), 139 (100).

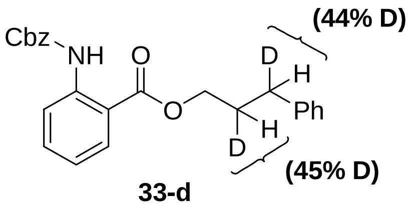
3-Phenylpropyl benzyl ether-d_2 (Table 6, entry 7)



32-d

^{1}H NMR (CD_3OD) δ 1.84 (m, 0.99H), 2.63 (m, 1.00H), 3.43 (d, J = 6.3 Hz, 2H), 4.44 (s, 2H), 7.10–7.32 (m, 10H); ^{2}H NMR (CH_3OH) 1.85 (br s), 2.64 (br s); MS (NBA, FAB) m/z (%): 229 (10) [M$^+$(d_2)+H], 228 (7) [M$^+$(d_1)+H], 227 (11) [M$^+$(d_0)+H], 136 (94), 91 (82).

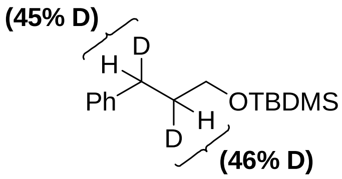
3-Phenylpropyl *N*-benzyloxycarbonyl anthranilate-d_2 (Table 6, entry 8)



33-d

^{1}H NMR (CD_3OD) δ 2.04 (m, 1.11H), 2.72 (m, 1.22H), 4.27 (d, J = 6.3 Hz, 2H), 5.17 (s, 2H), 7.04 (m, 1H), 7.11–7.41 (m, 10H), 7.51 (m, 1H), 7.92 (dd, J = 7.7, 1.4 Hz, 1H), 8.32 (d, J = 8.7 Hz, 1H); ^{2}H NMR (CH_3OH) δ 2.04 (br s), 2.72 (br s); MS (NBA, FAB) m/z (%): 392 (19) [M$^+$(d_2)+H], 391 (22) [M$^+$(d_1)+H], 390 (7) [M$^+$(d_0)+H], 106 (53), 348 (19), 136 (65), 91 (73).

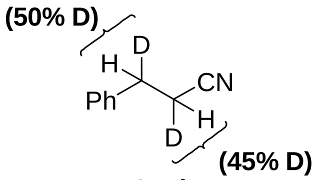
***tert*-Butyldimethylsilyl 3-phenylpropyl ether-d_2 (Table 6, entry 9)**



34-d

^{1}H NMR (CD_3OD) δ 0.00 (s, 6H), 0.86 (s, 9H), 1.74 (m, 1.08H), 2.59 (m, 1.11H), 3.57 (d, J = 6.3 Hz, 2H), 7.06–7.21 (m, 5H); ^{2}H NMR (CH_3OH) δ 1.76 (br s), 2.61 (br s); MS (EI) m/z (%): 195 (100) [M(d_2)–*t*-Bu]$^+$, 194 (35) [M(d_1)–*t*-Bu]$^+$, 193 (4) [M(d_0)–*t*-Bu]$^+$.

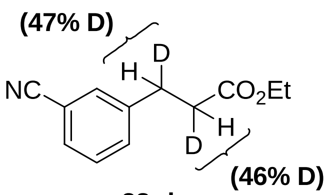
3-Phenylpropanenitrile-d_2 (Table7, entry 10)



35-d

^{1}H NMR (CD_3OD) δ 2.64 (m, 1.15H), 2.86 (m, 1.01), 7.21–7.32 (m, 5H); ^{2}H NMR (CH_3OH) δ 2.66 (br s), 2.86 (br s); MS (EI) m/z (%): 133 (21) [M(d_2)]$^+$, 132 (16) [M(d_1)]$^+$, 131 (2) [M(d_0)]$^+$, 92 (100).

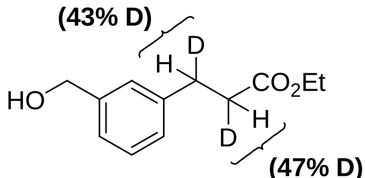
Ethyl 3'-cyano-3-phenylpropionate-d_2 (Table 6, entry 11)



38-d

^{1}H NMR (CD_3OD) δ 1.19 (t, J = 7.2 Hz, 3H), 2.63 (m, 1.09H), 2.95 (m, 1.06H), 4.08 (q, J = 7.2 Hz, 2H), 7.44 (m, 1H), 7.53–7.59 (m, 3H); ^{2}H NMR (CH_3OH) δ 2.62 (br s), 2.93 (br s); MS (EI) m/z (%): 205 (53) [M(d_2)]$^+$, 204 (14) [M(d_1)]$^+$, 203 (1) [M(d_0)]$^+$, 131 (100).

Ethyl 3'-hydroxymethyl-3-phenylpropionate-d_2 (Table 6, entry 12)



37-d

^{1}H NMR (CD_3OD) δ 1.19 (t, J = 7.1 Hz, 3H), 2.59 (m, 1.07H), 2.89 (m, 1.14H), 4.08 (q, J = 7.1 Hz, 2H), 4.56 (s, 2H), 7.09–7.25 (m, 4H); ^{2}H NMR (CH_3OH) δ 2.58 (br s), 2.88 (br s); MS (EI) m/z (%): 210 (3) $[M(d_2)]^+$, 209 (1) $[M(d_1)]^+$, 208 (2) $[M(d_0)]^+$, 149 (100), 148 (76).

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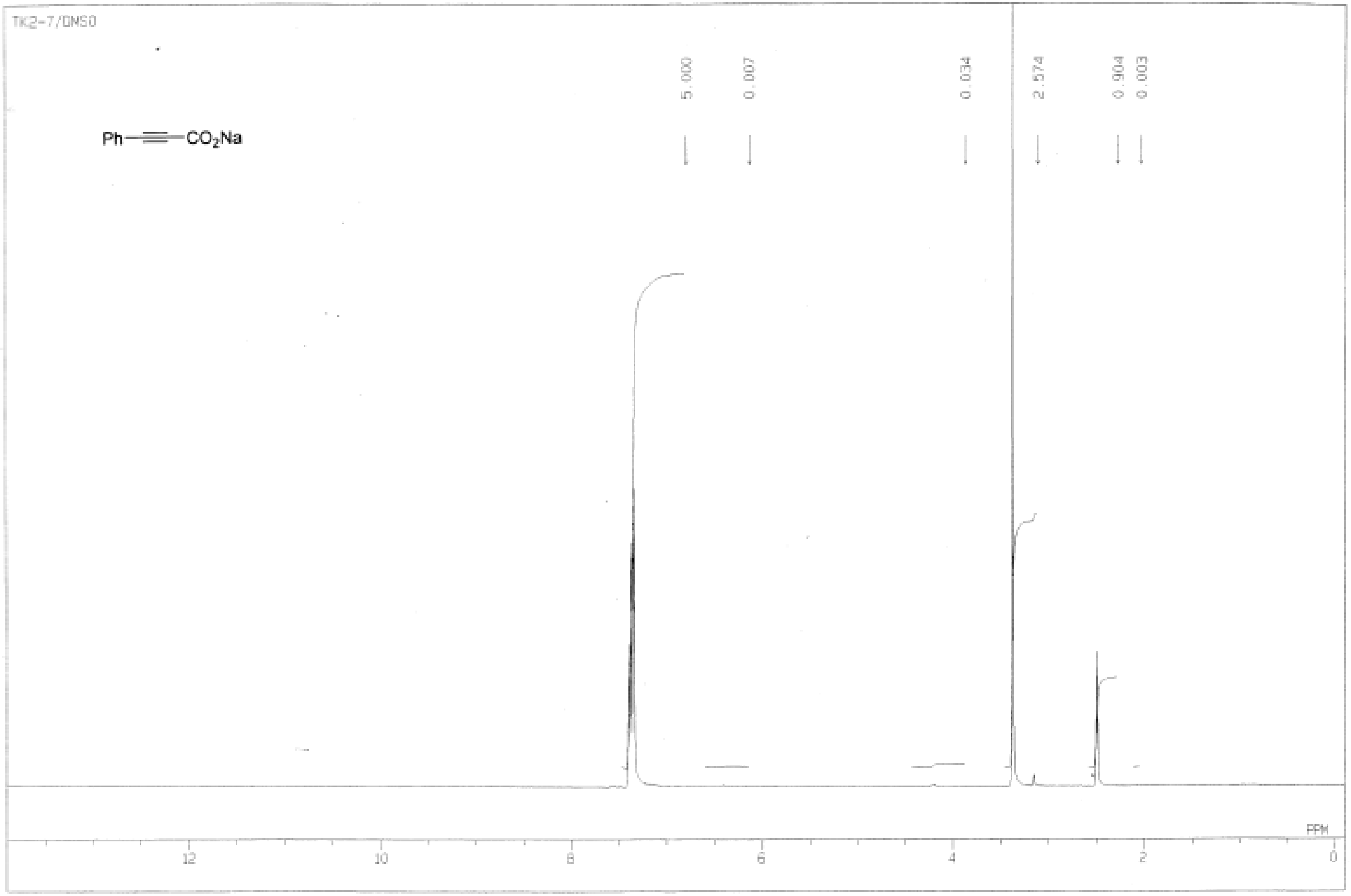
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Ph—≡—CO2Na
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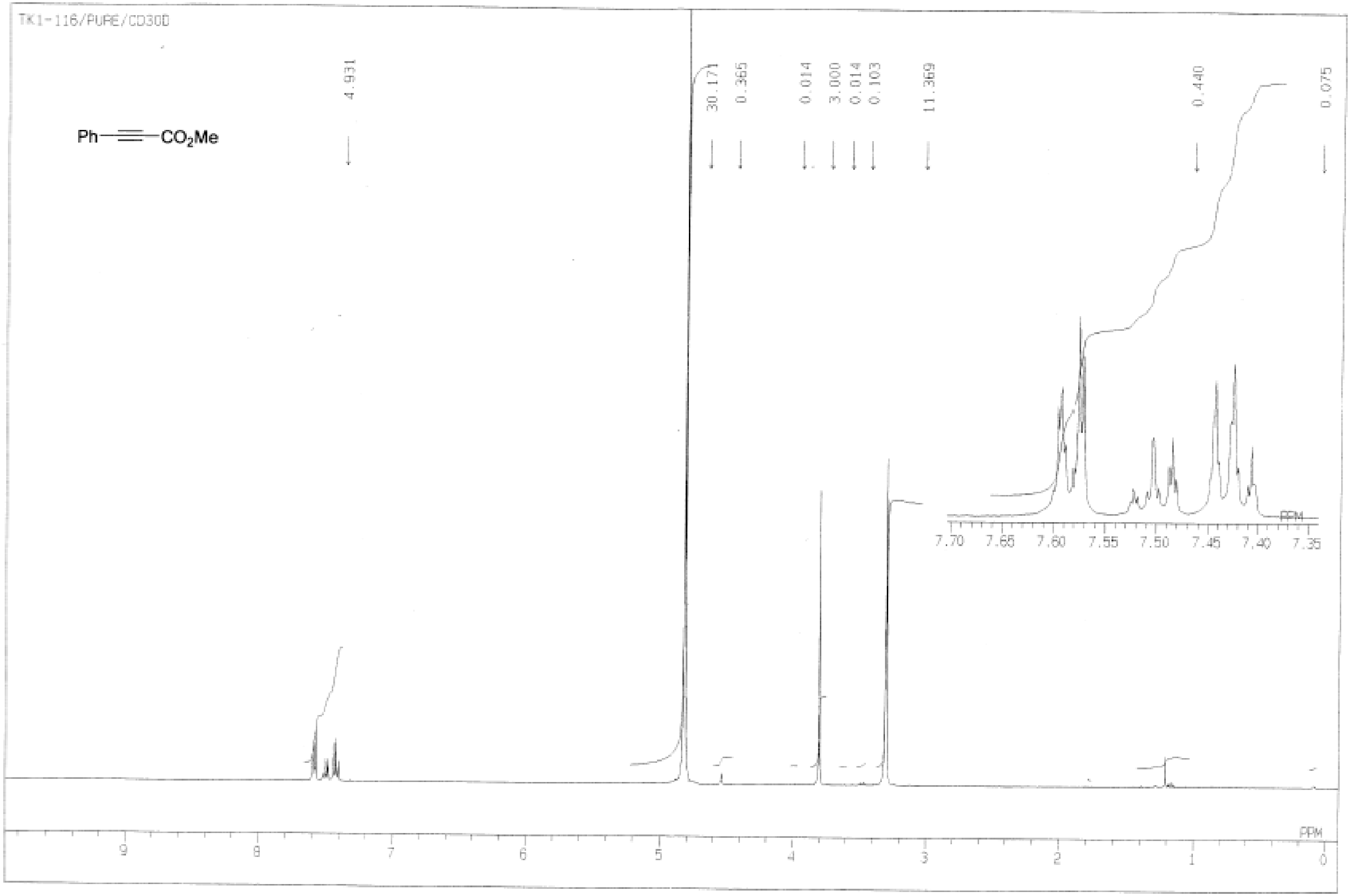
0.007
0.034
2.574
0.904
0.003
PPM
12
10
8
6
4
2
0


TK1-116/PURE/CD3OD
Ph—≡—CO2Me
4.931
30.171
0.365
0.014
3.000
0.014
0.103
11.369
0.440
0.075
7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35
PPM
9 8 7 6 5 4 3 2 1 0

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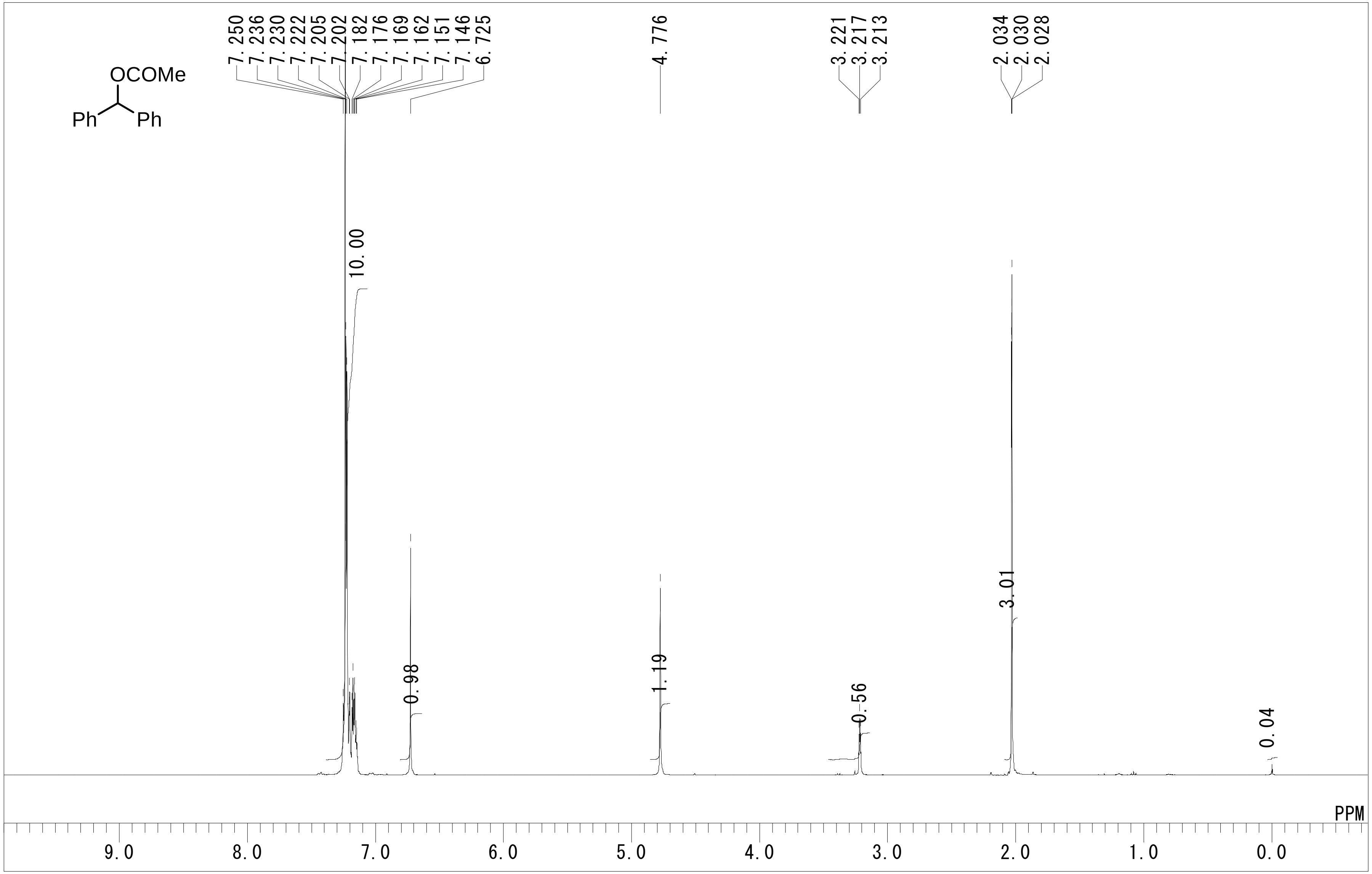
TK2-57



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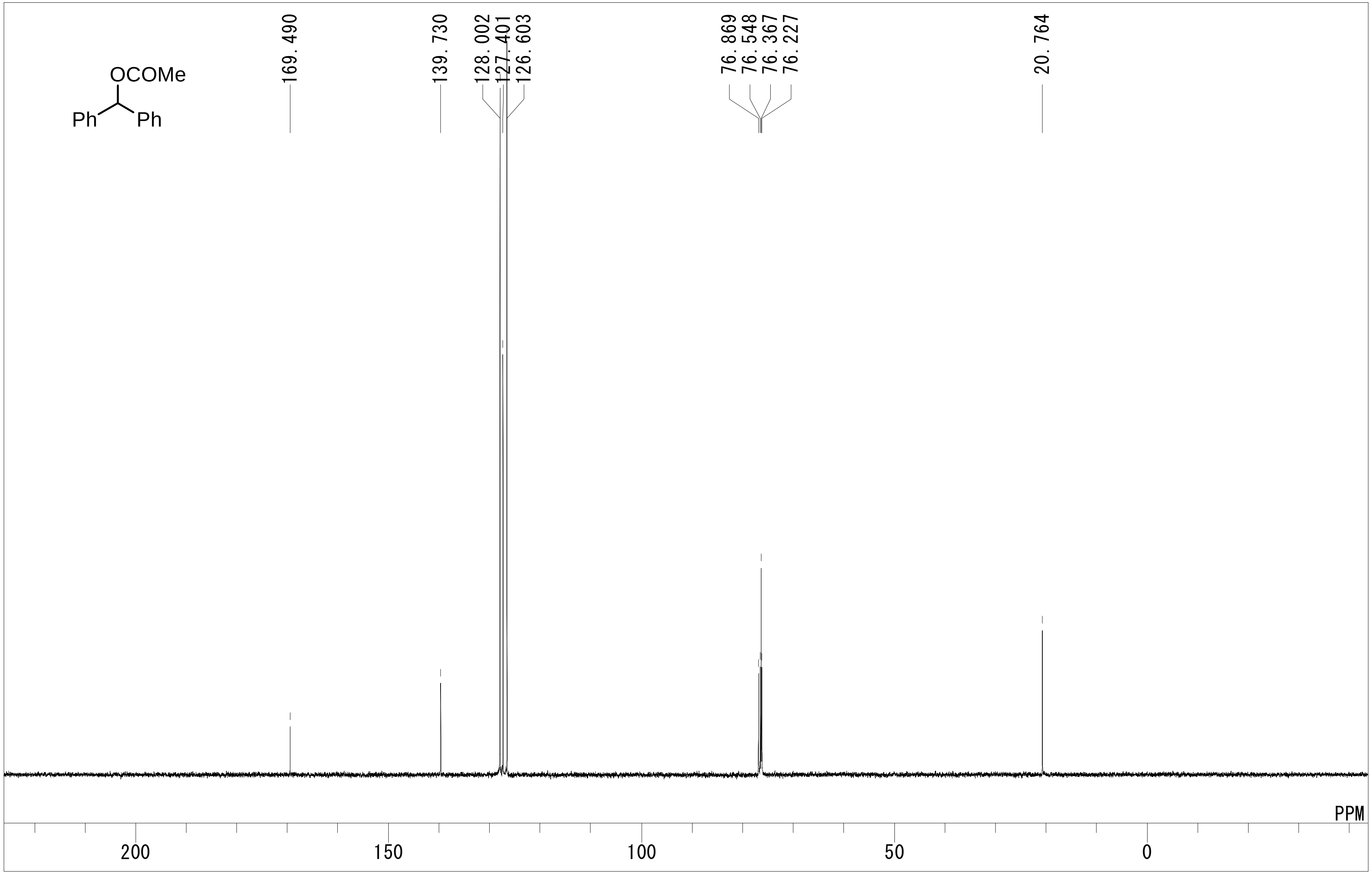
TK2-57



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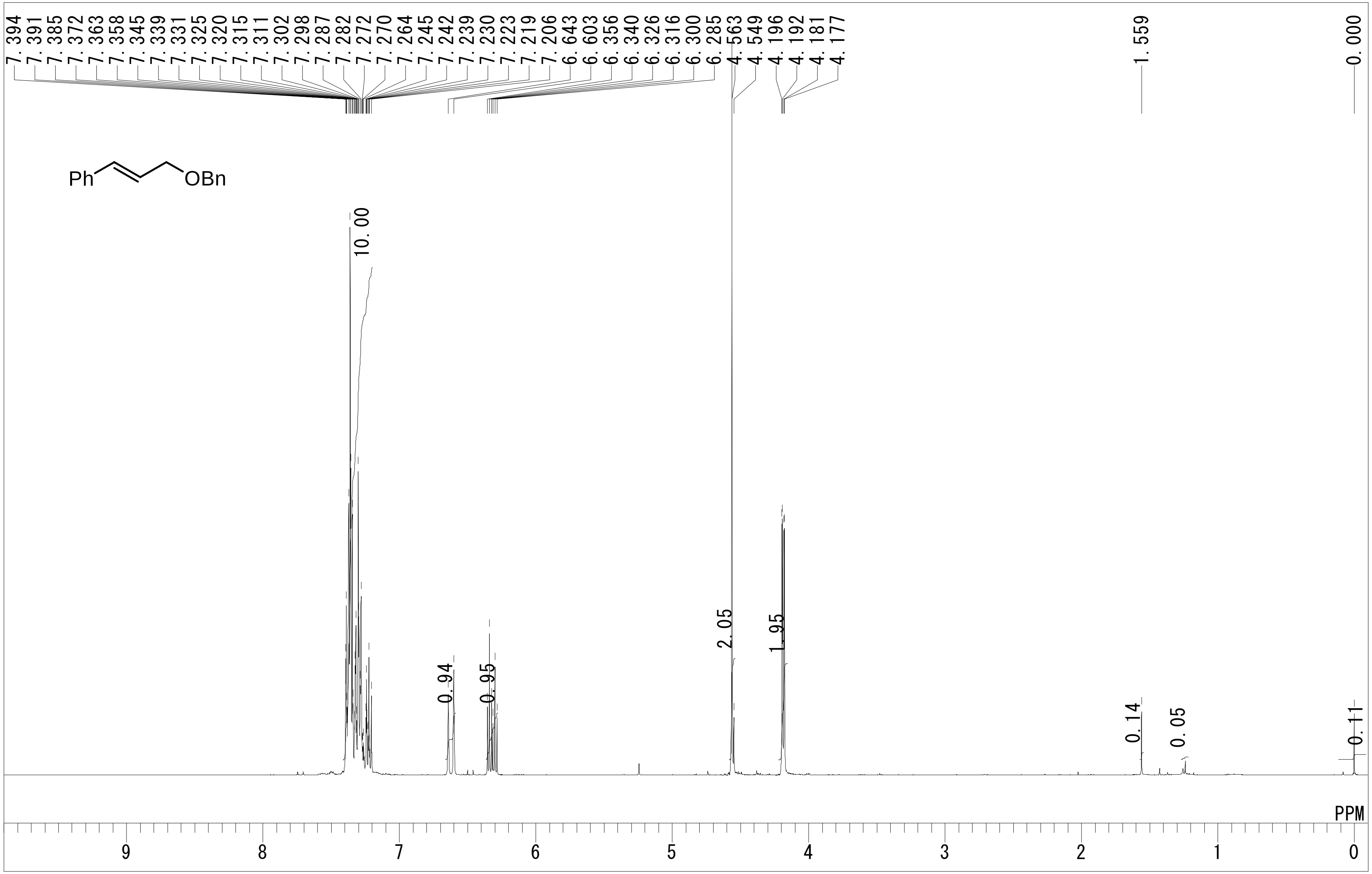
TMR36-1



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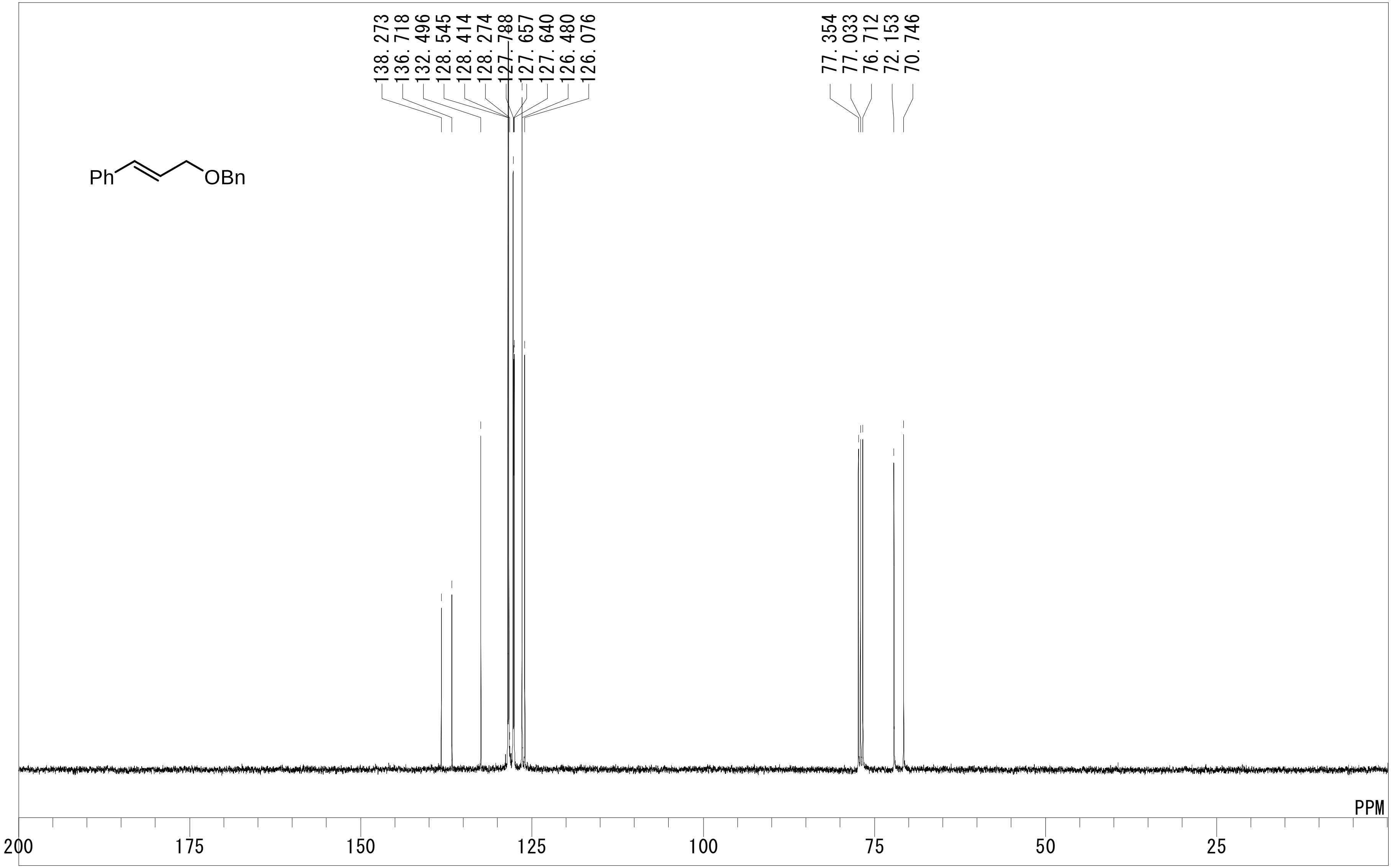

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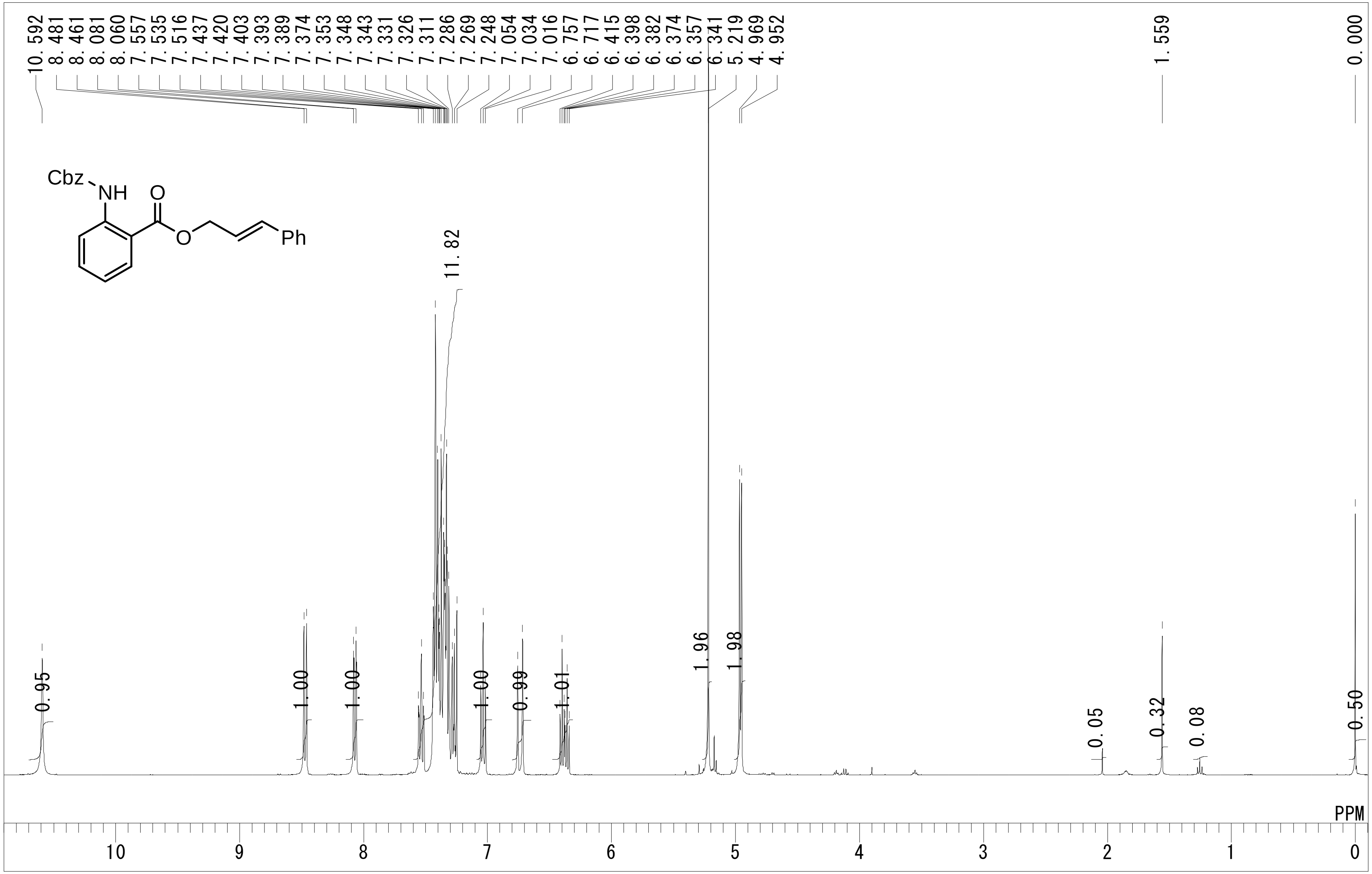

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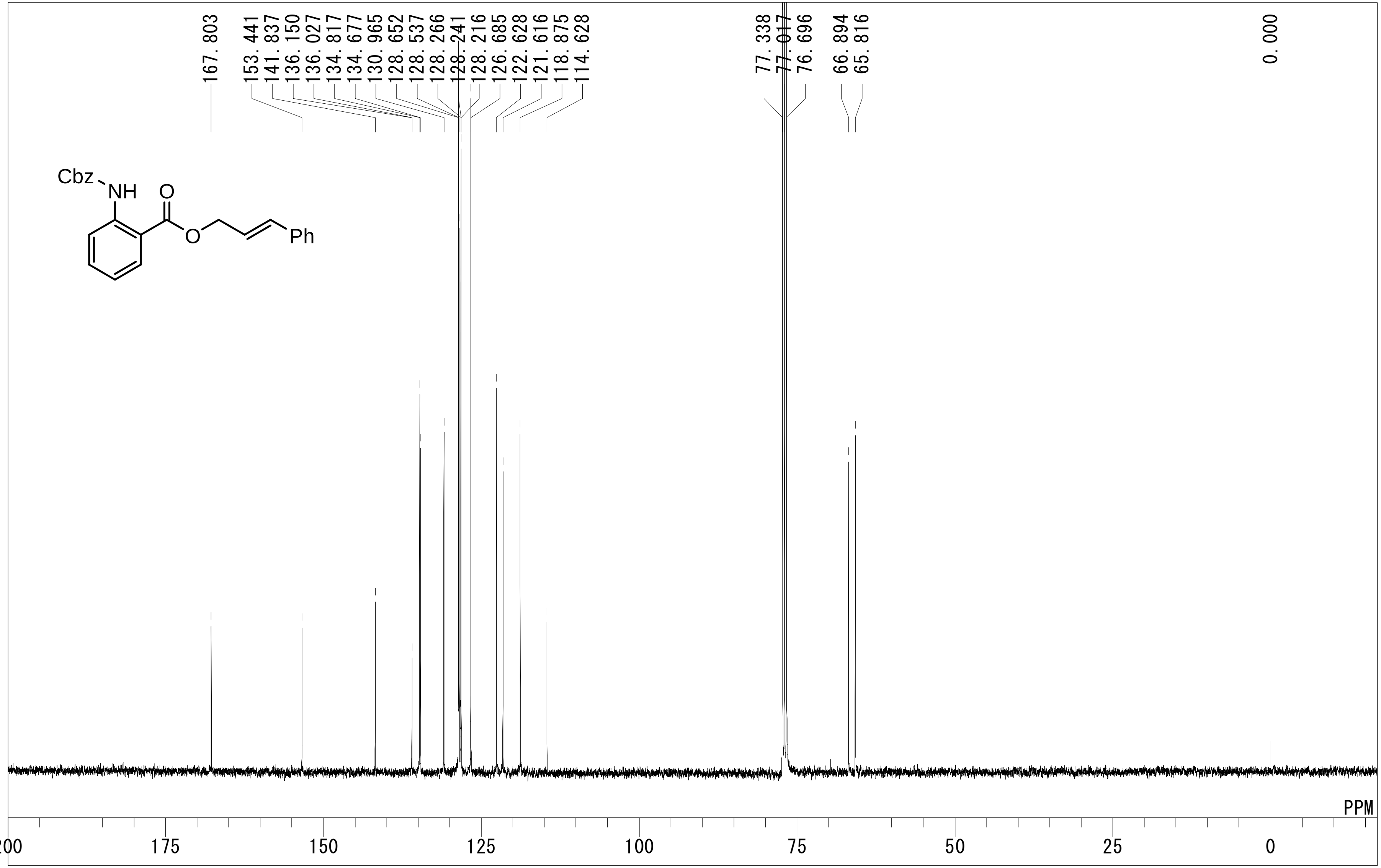

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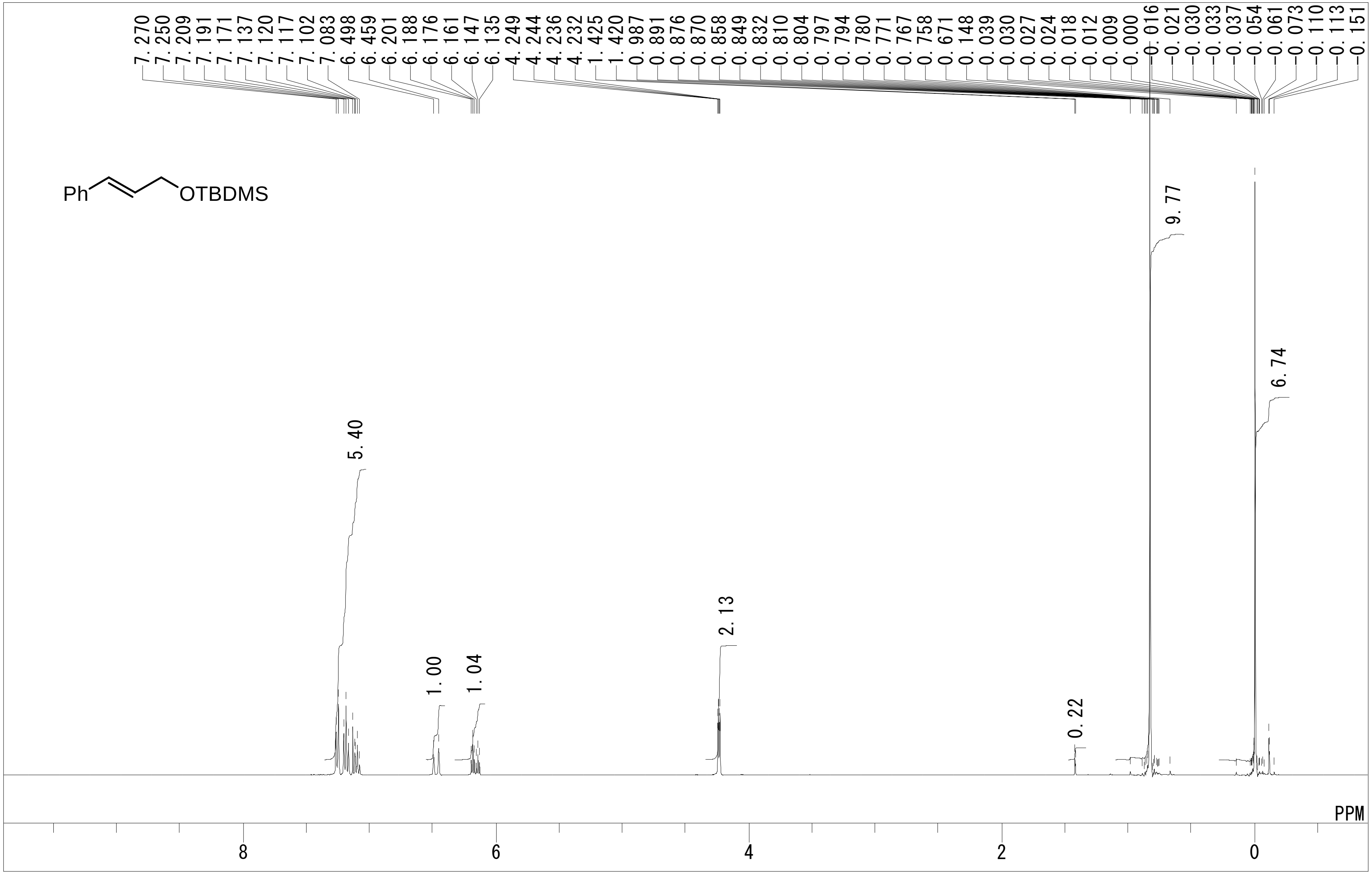

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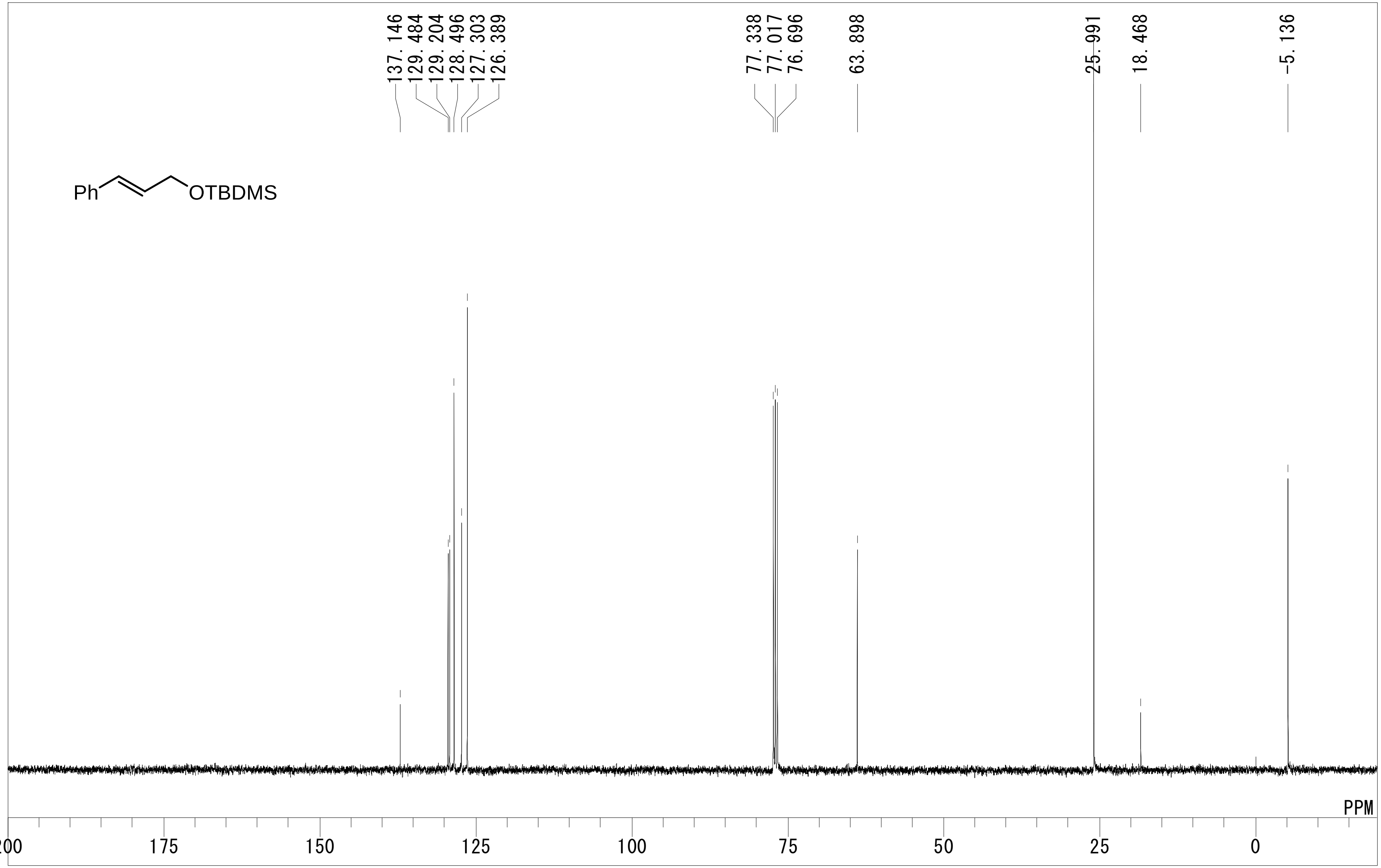
TK7-82-1



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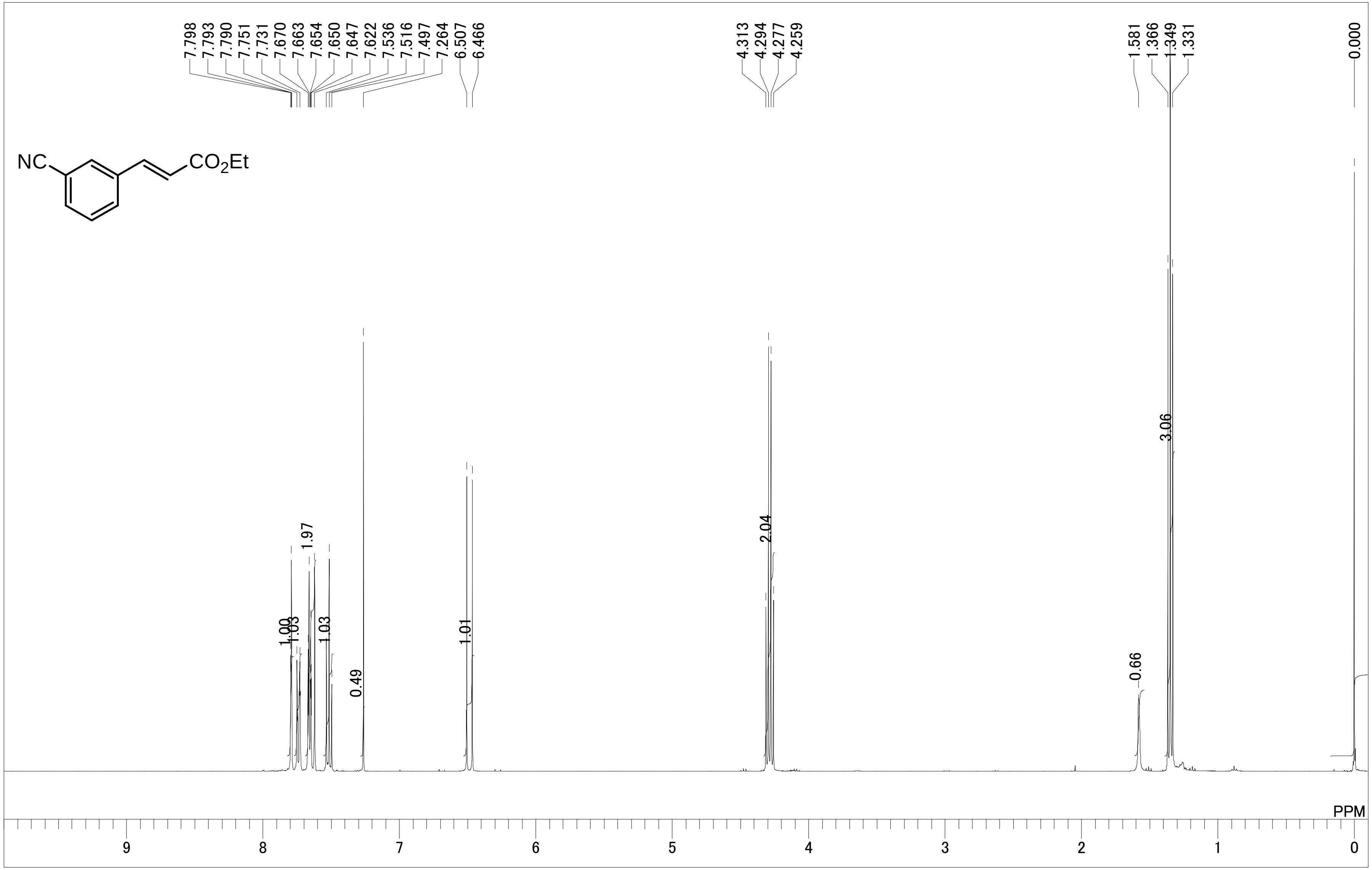

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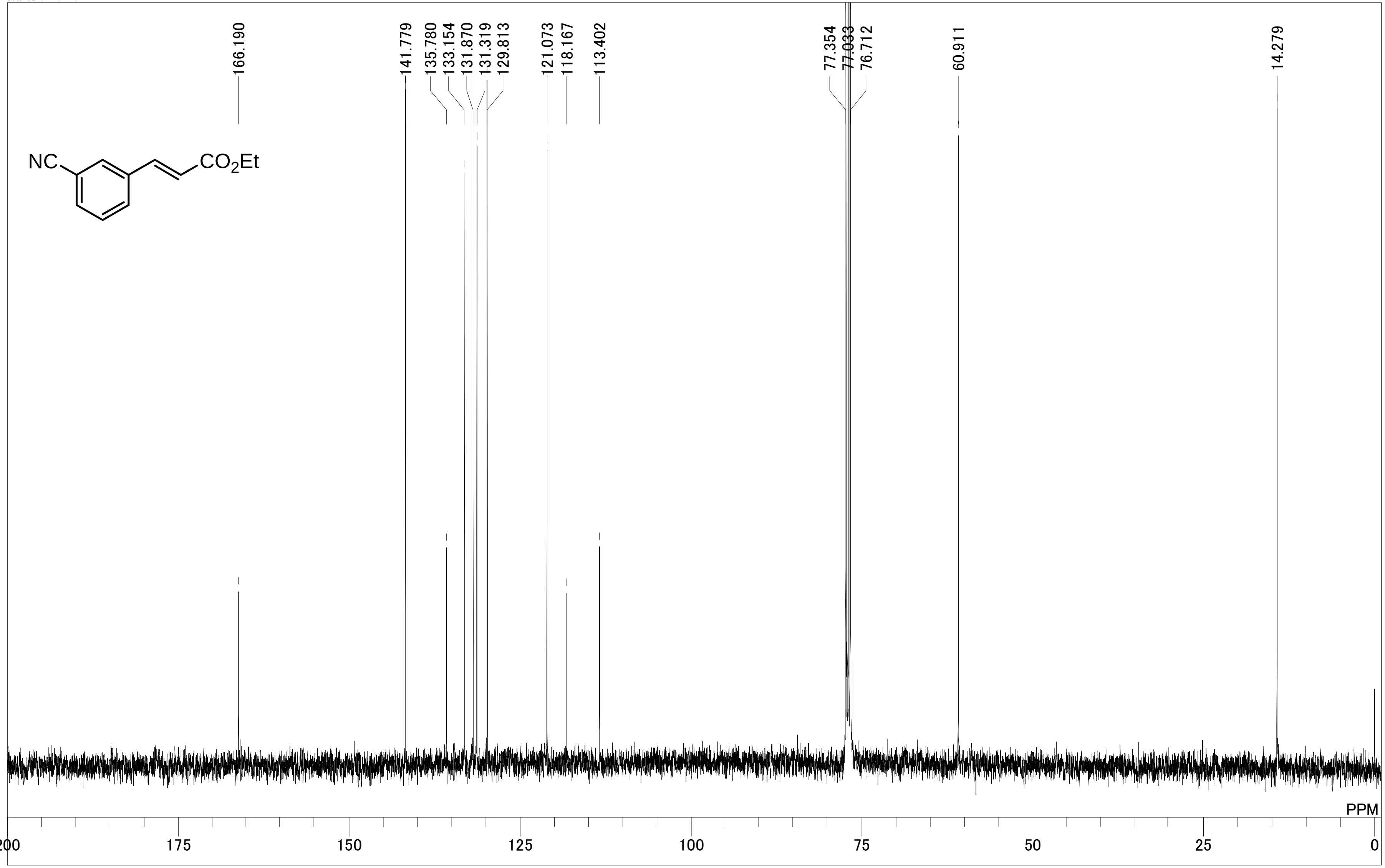

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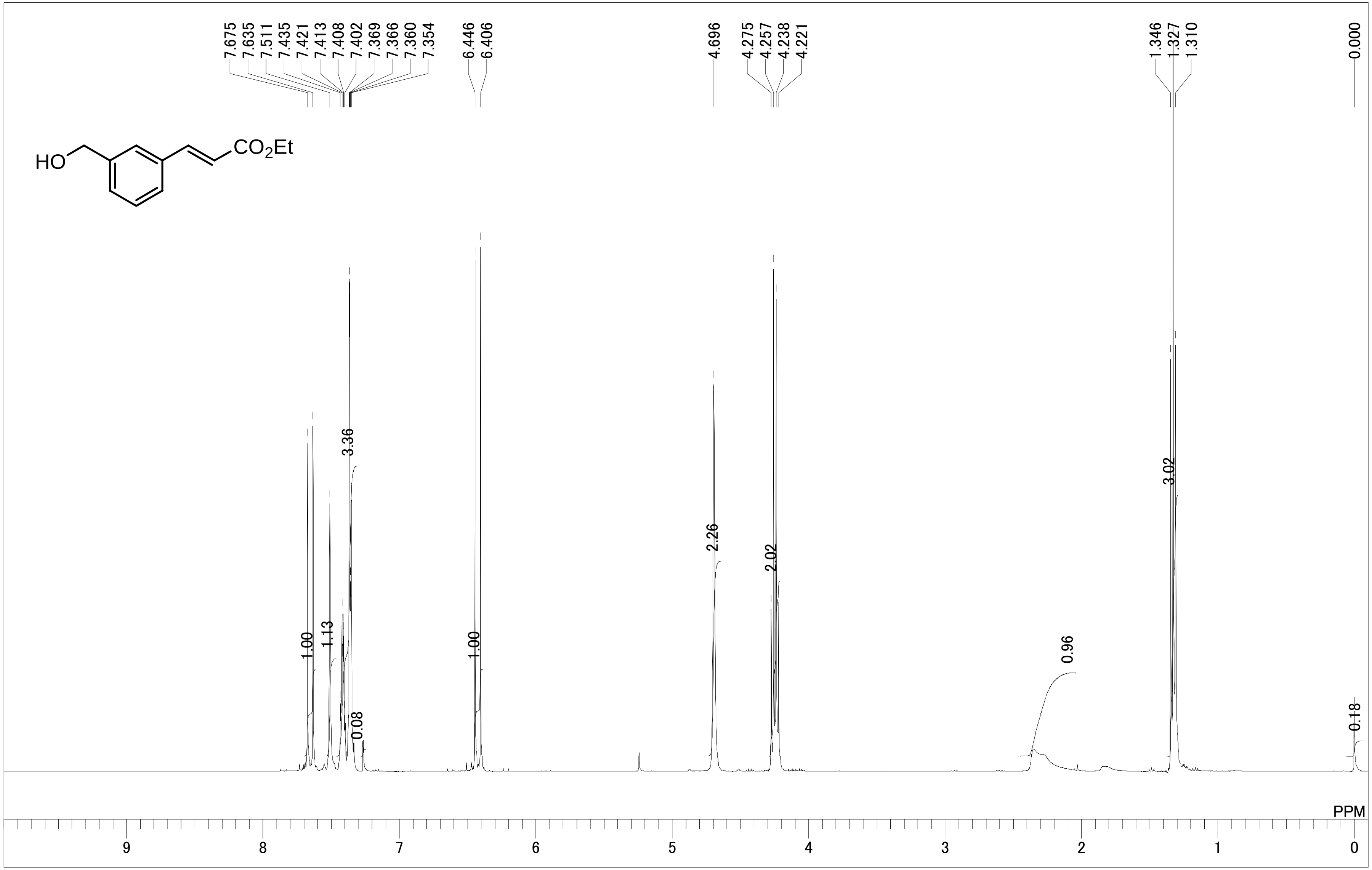
MAJ3-3



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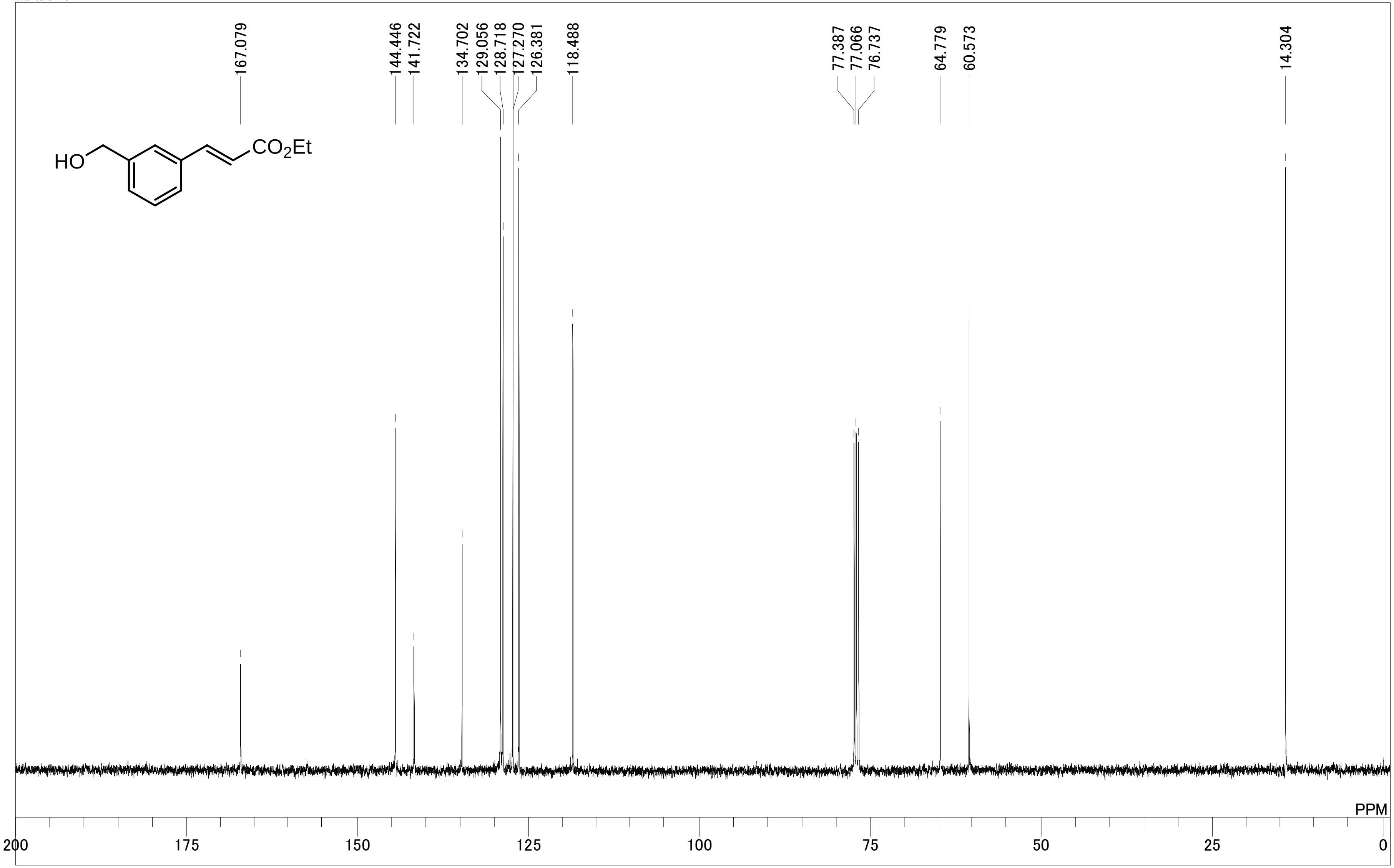
MAJ3-3



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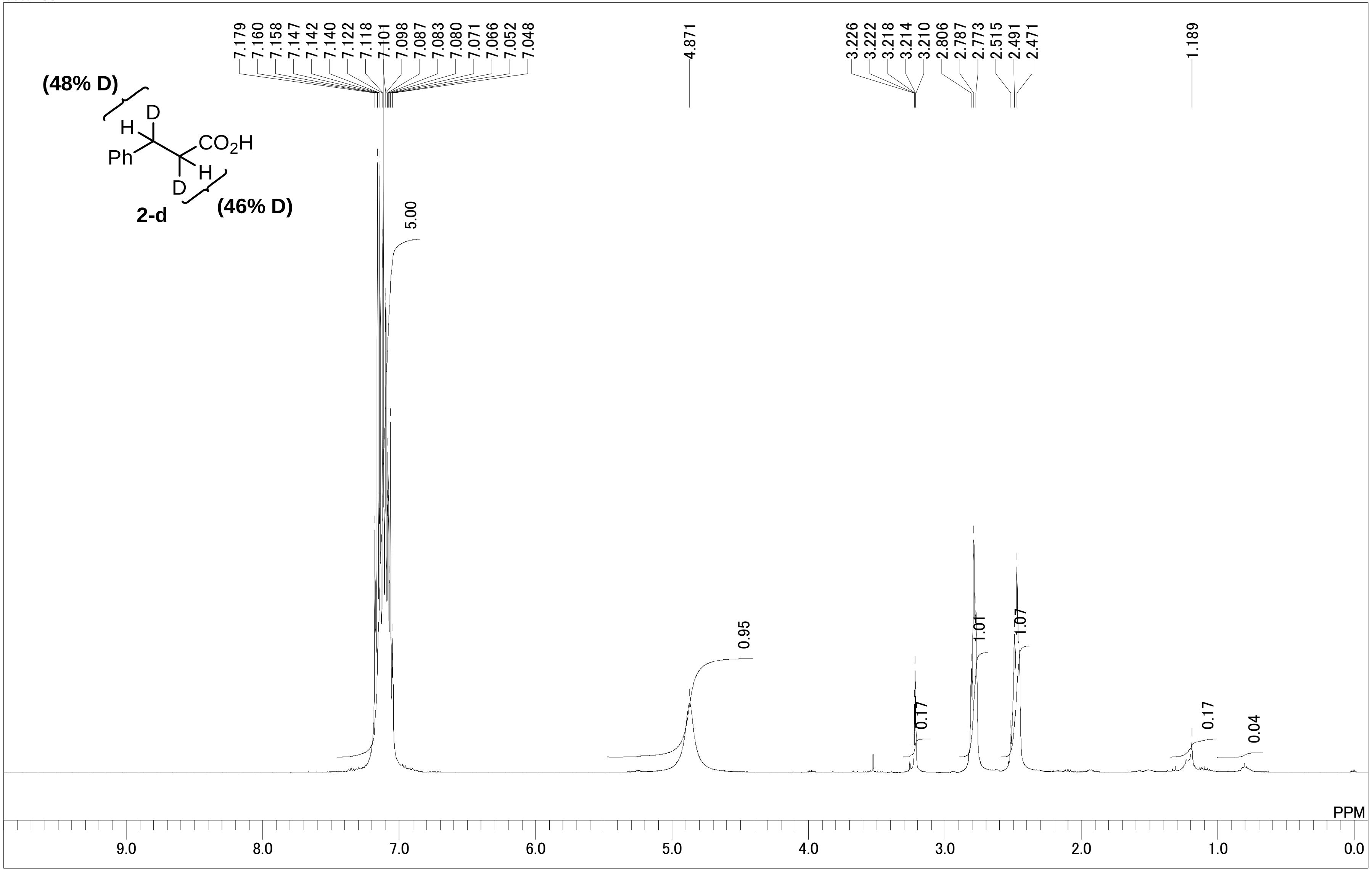
TK1-85



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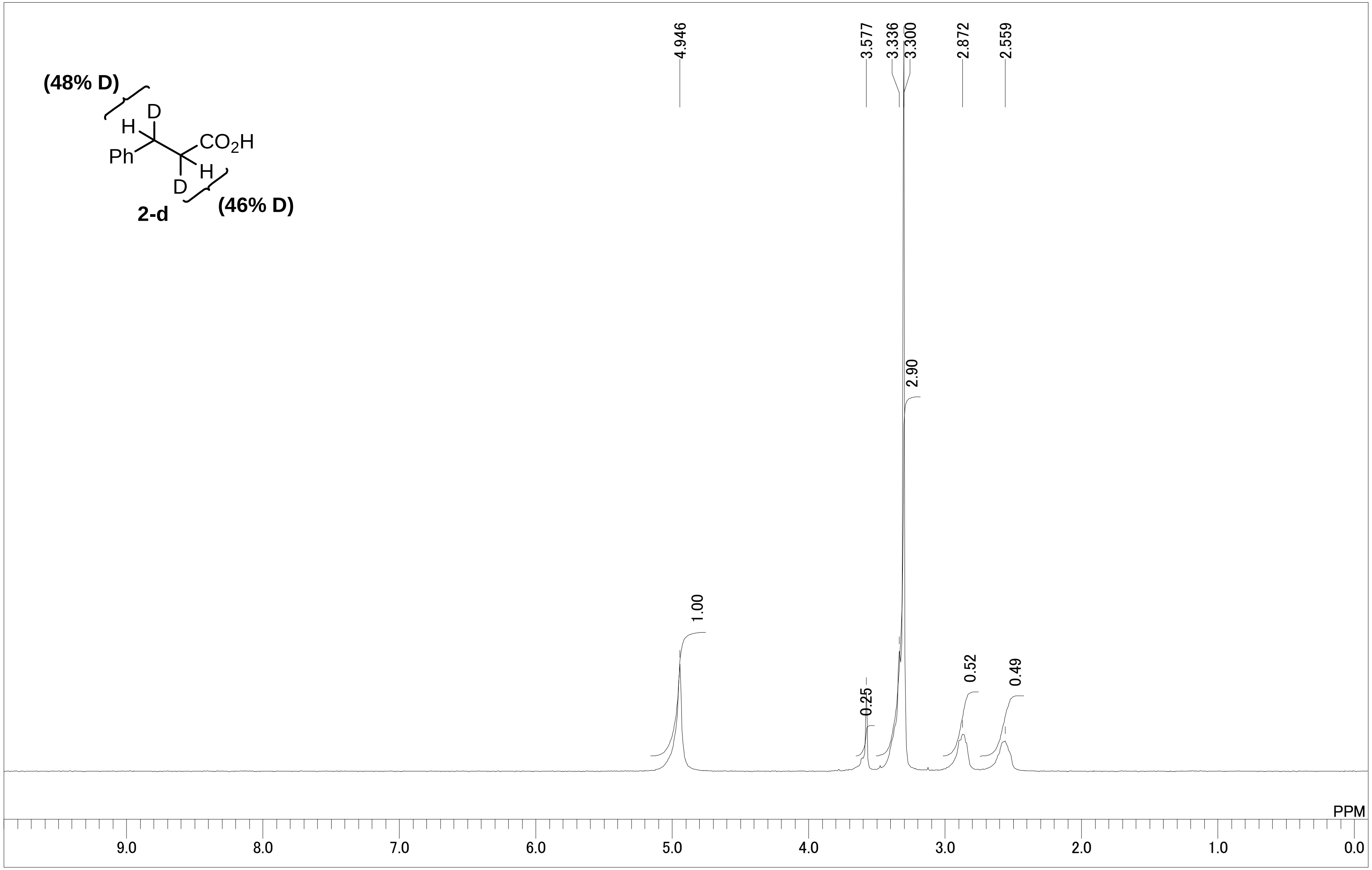
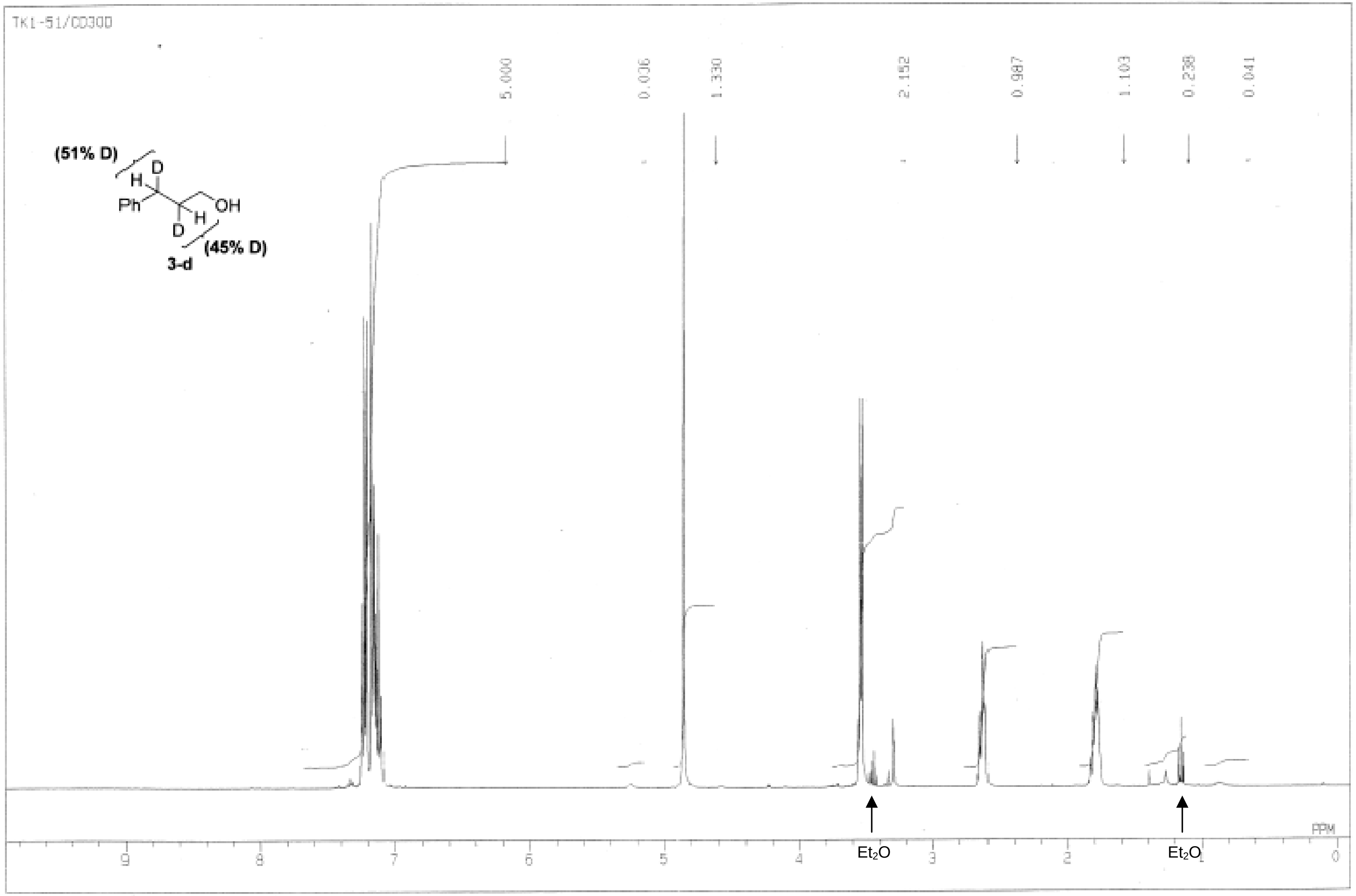
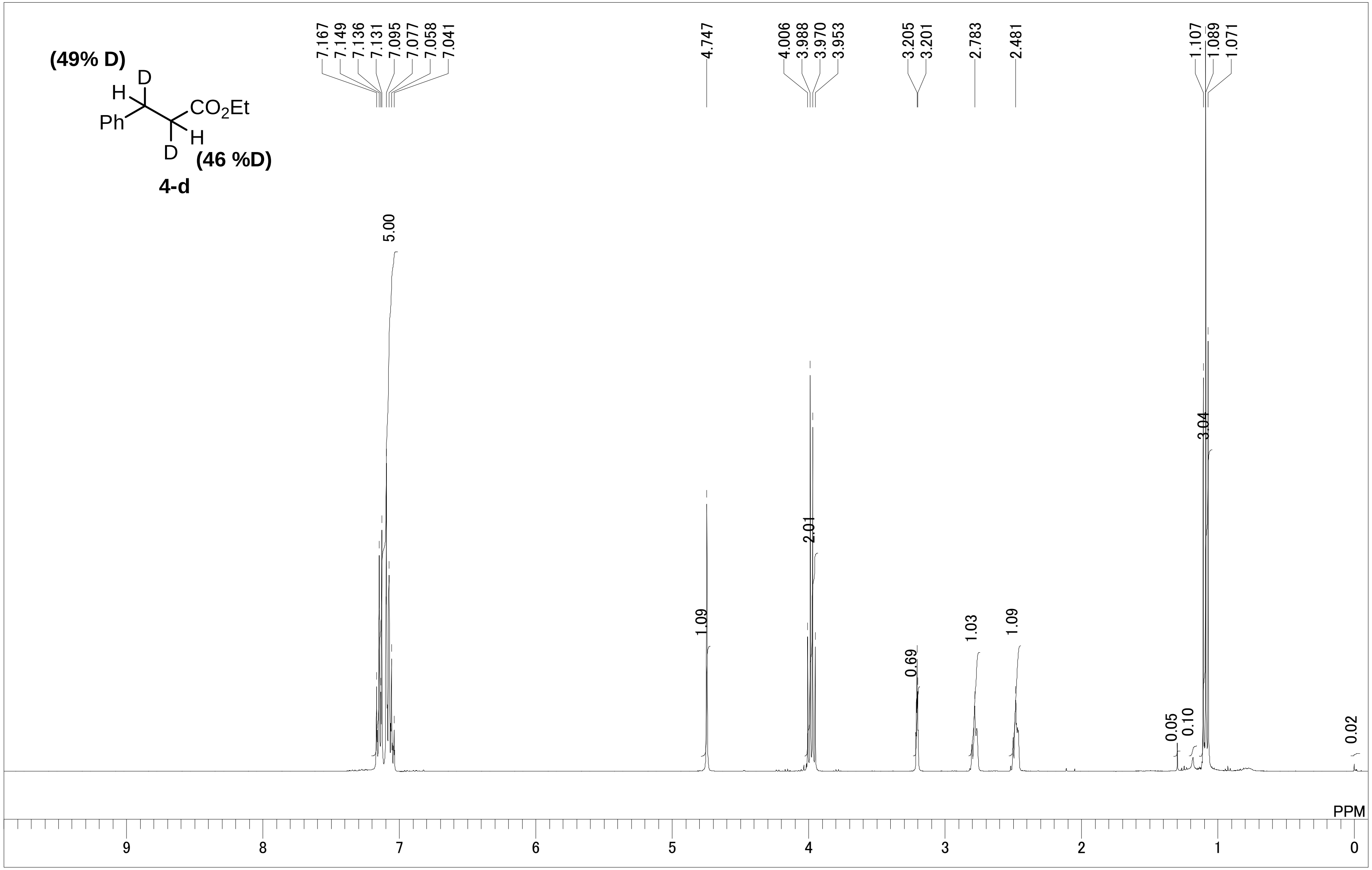


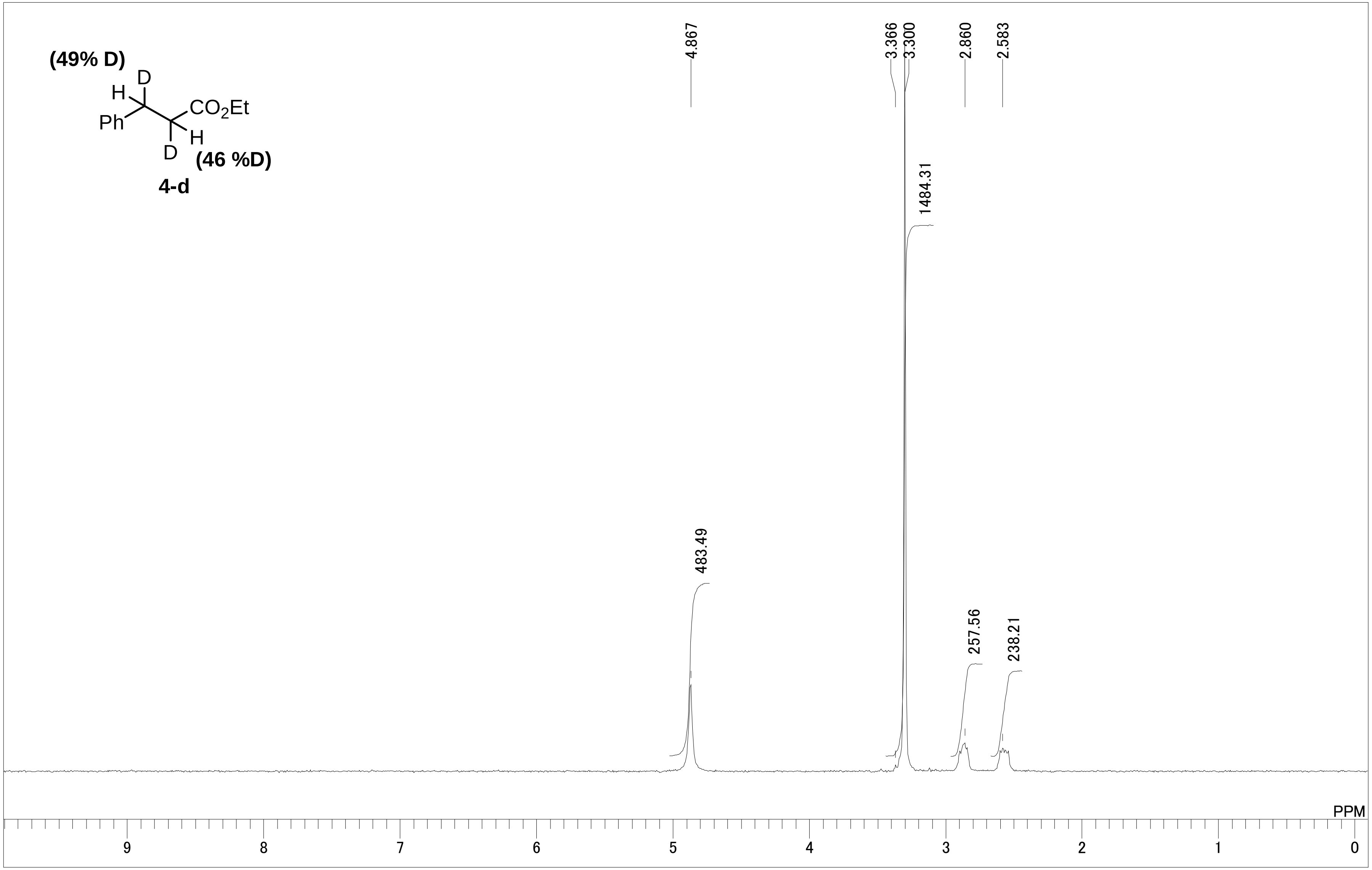


TK1-51/CD3OD
5.000
0.036
1.330
2.152
0.987
1.103
0.238
0.041
(51% D)
D
H
Ph
OH
D
H
(45% D)
3-d
Et2O
Et2O
PPM
9
8
7
6
5
4
3
2
1
0

F:¥NMR 2007-5月¥TK7-1301NON_E5_FT.als
TK7-130



F:¥2H NMR 6-1¥TK7-130-CH3OH-2H NMR-FT.als
TK7-130-CH3OH-2H NMR



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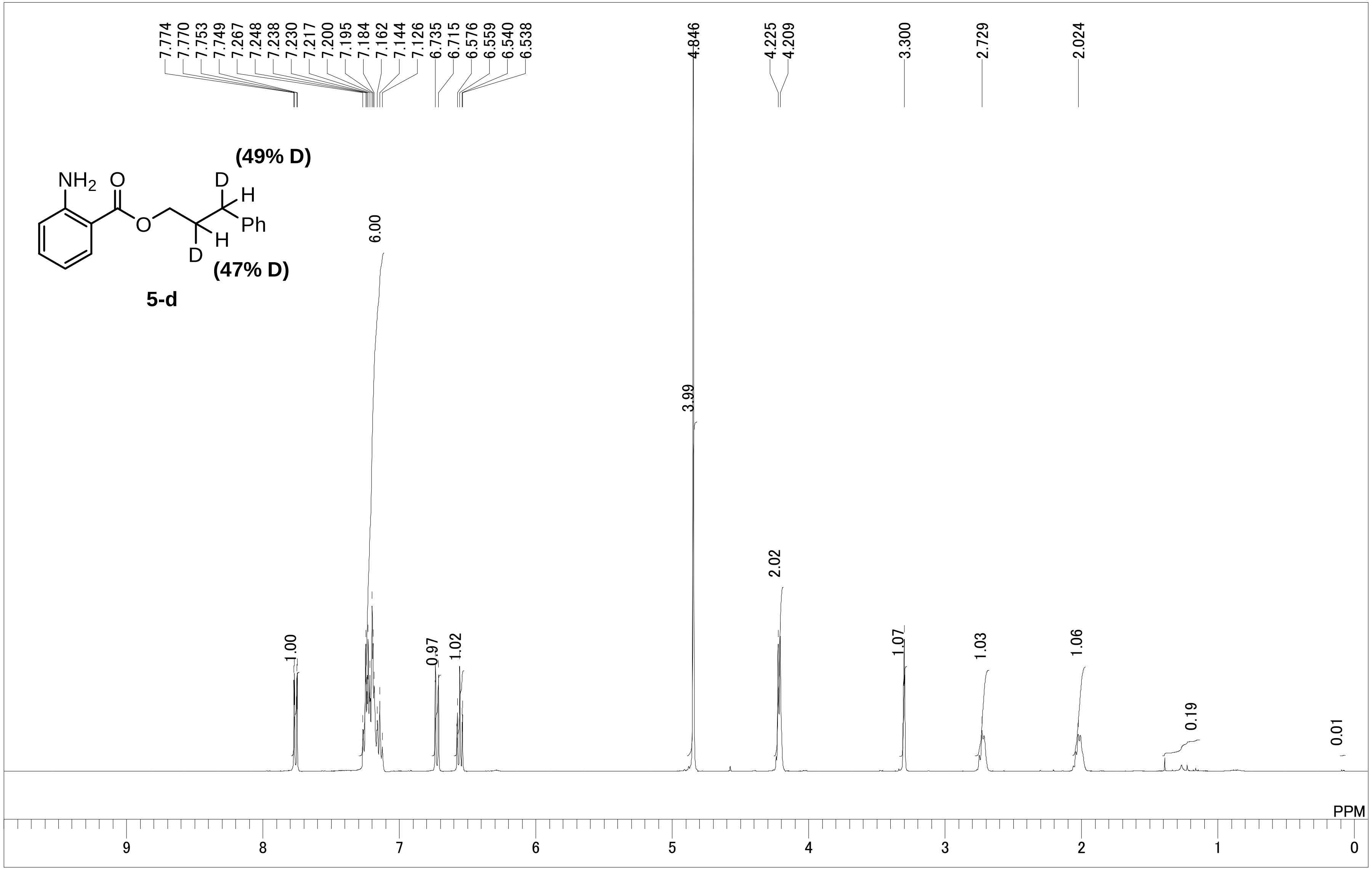
TK7-142-2



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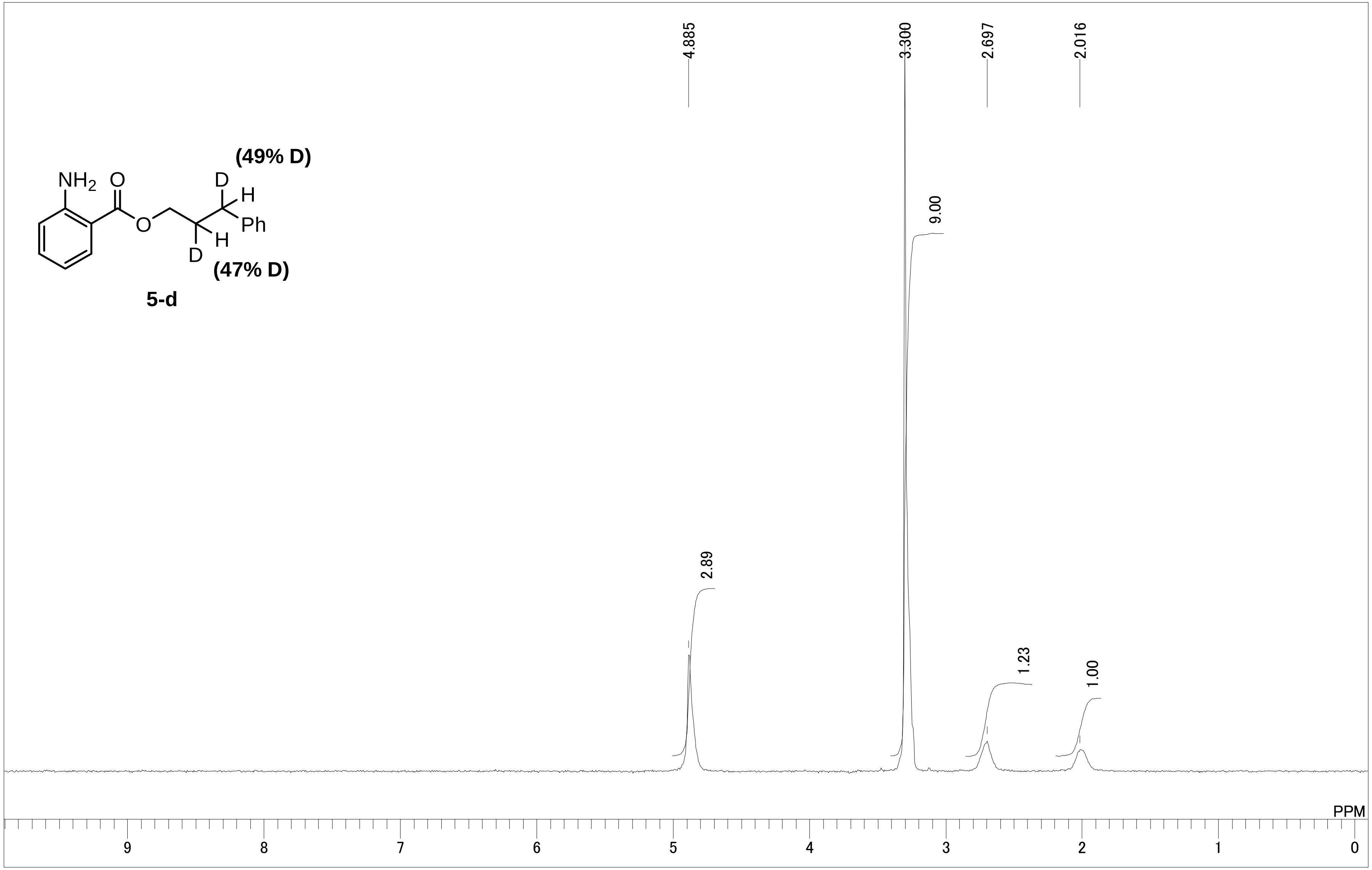

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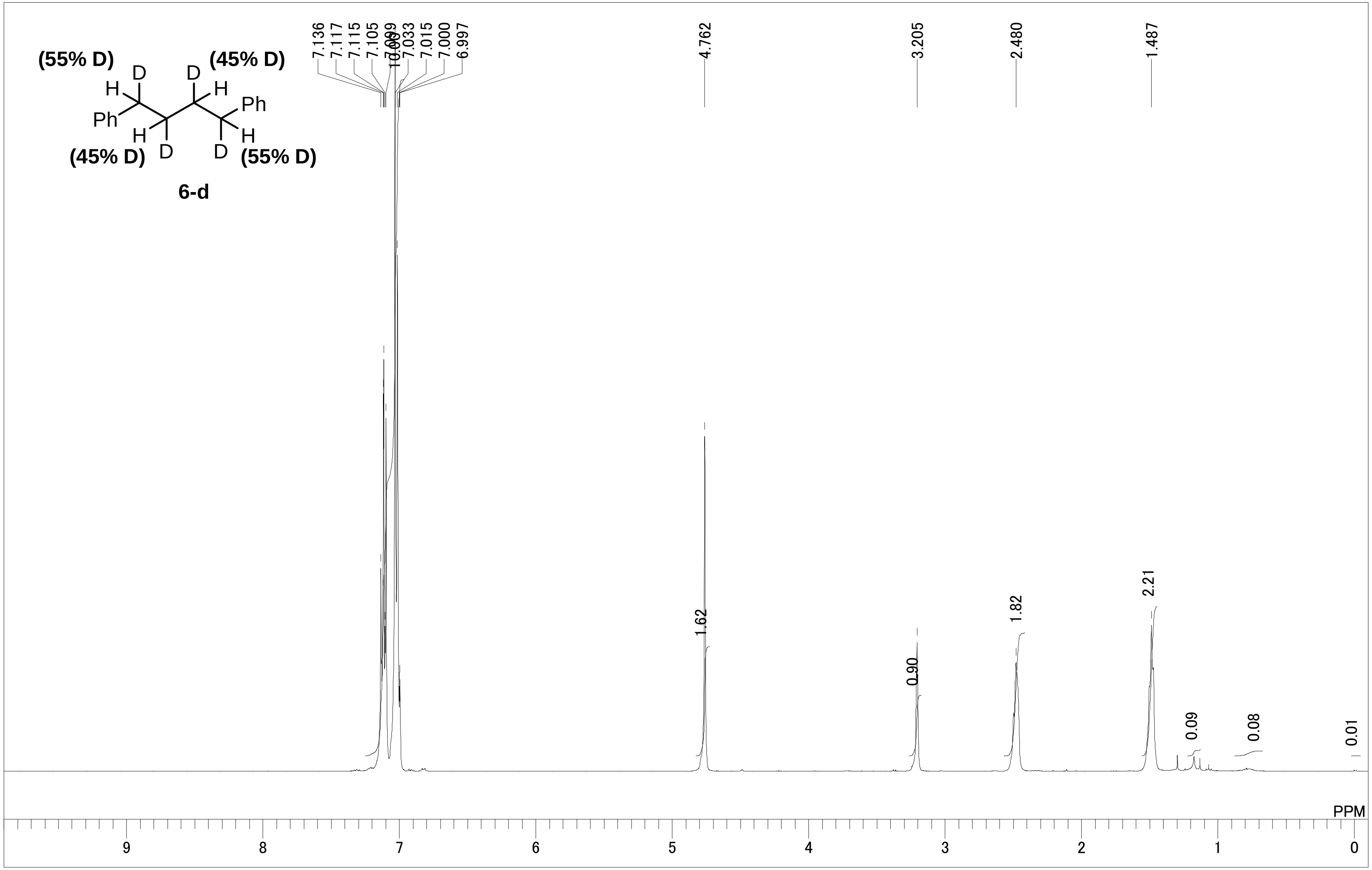
TK7-149



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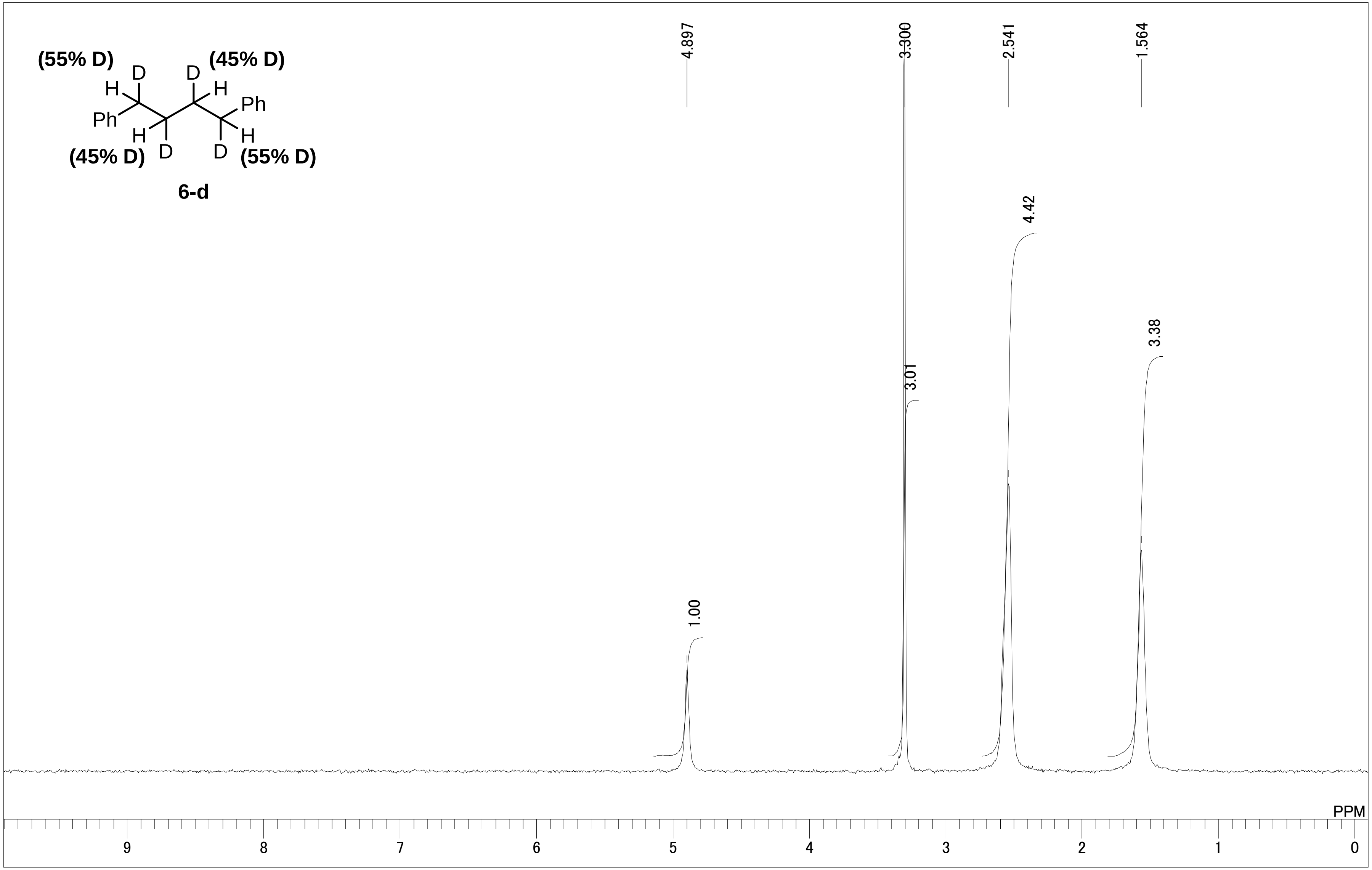
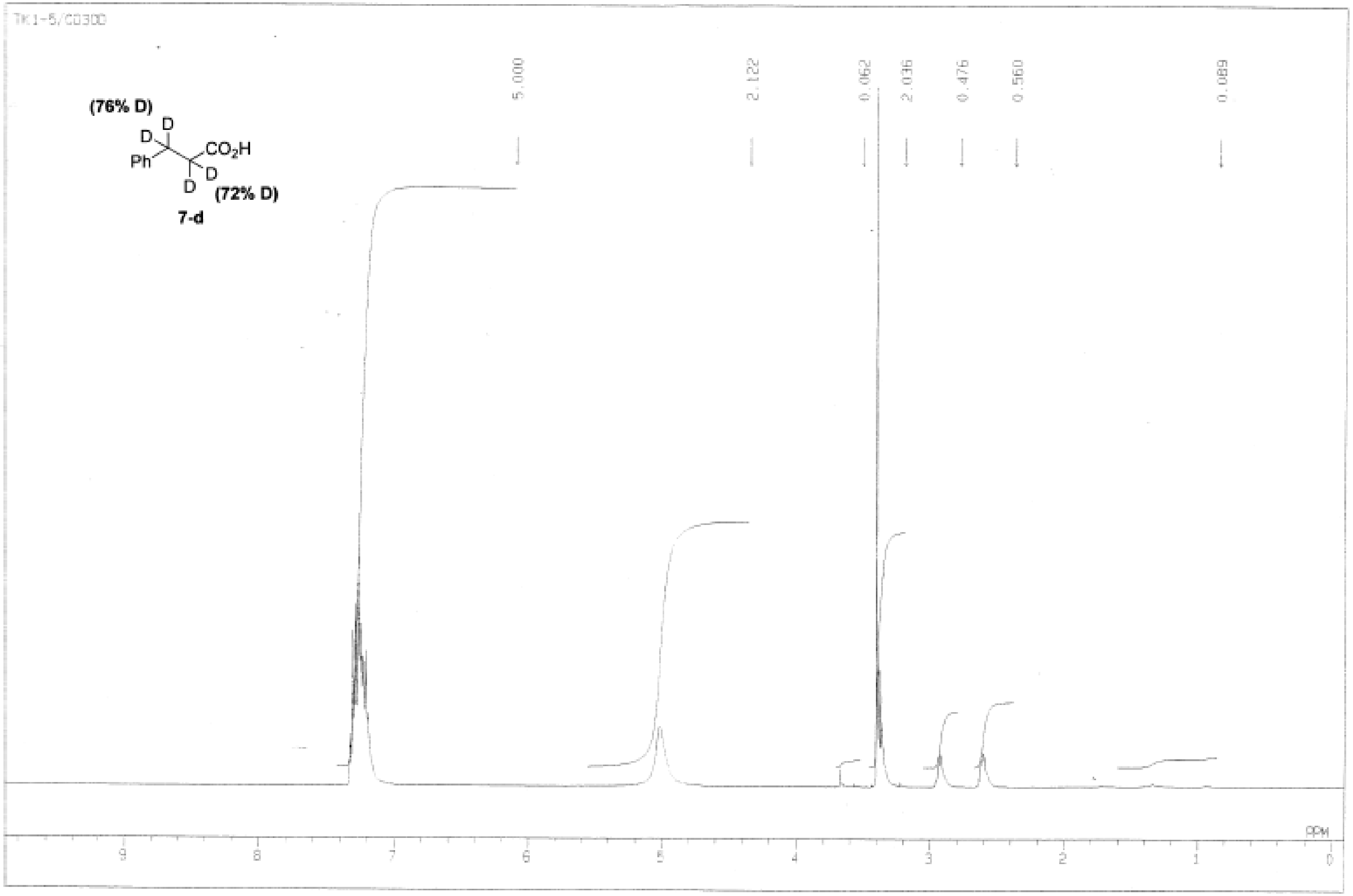
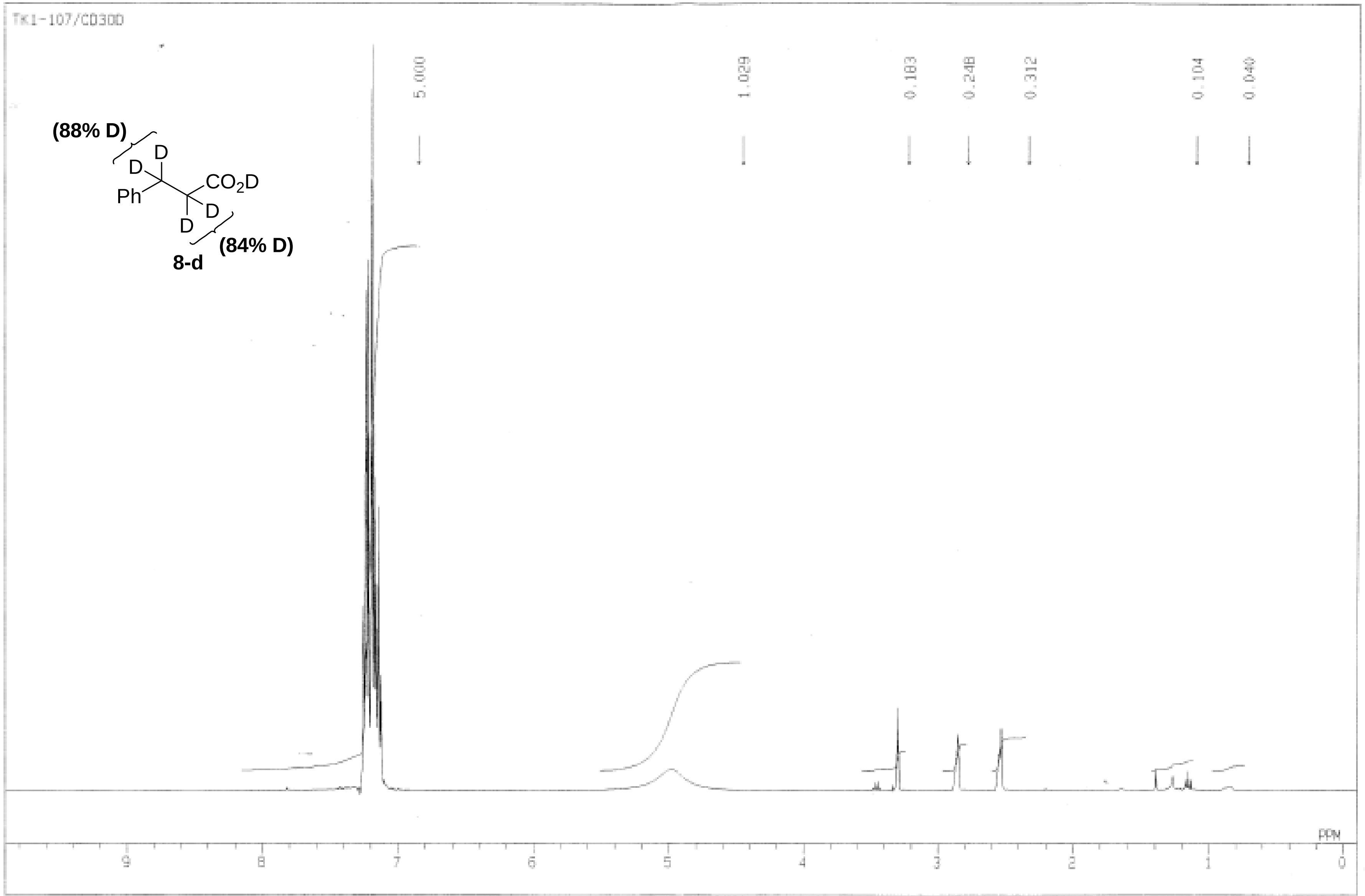
TK7-149-CH3OH-2H NMR

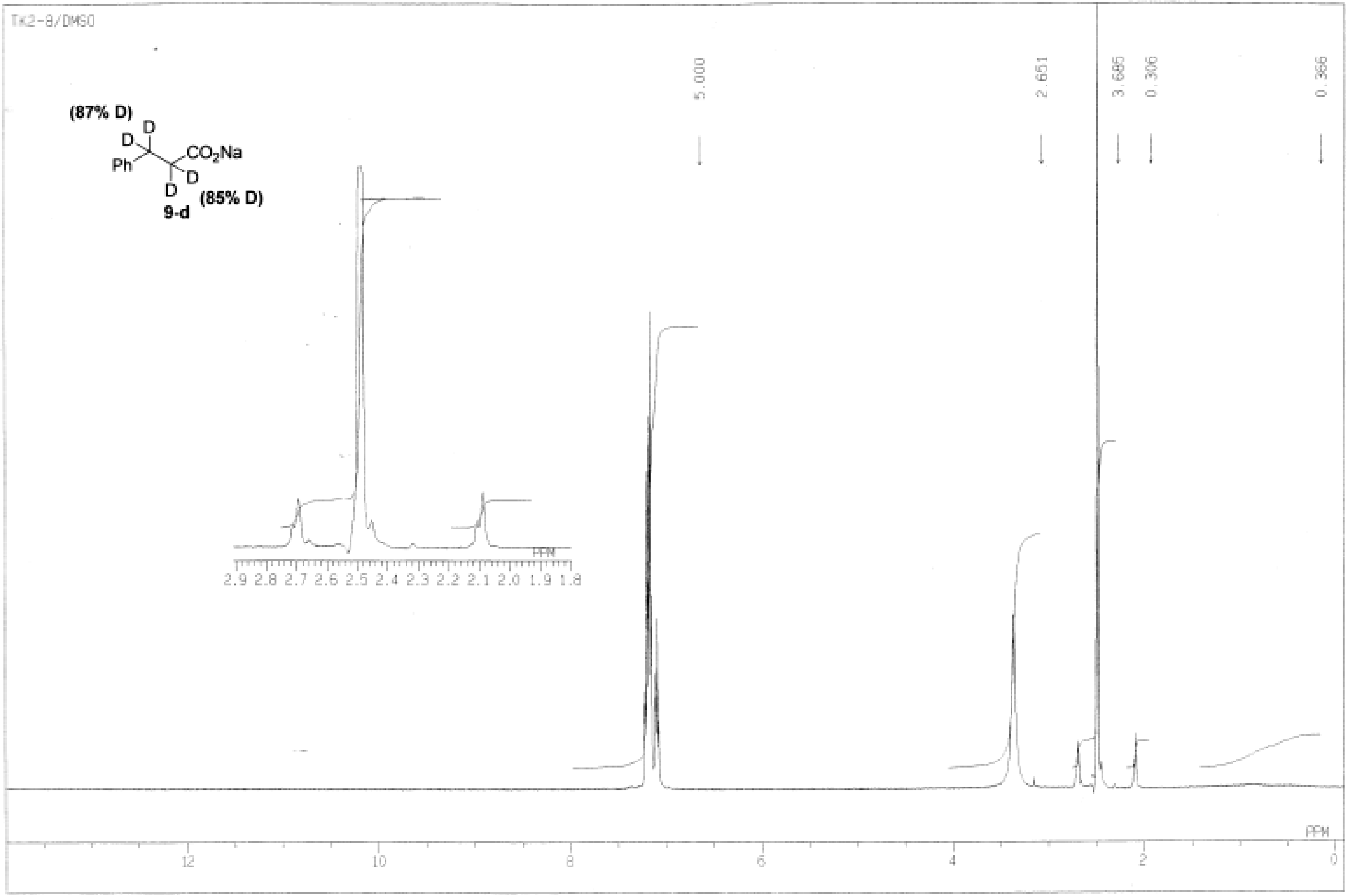




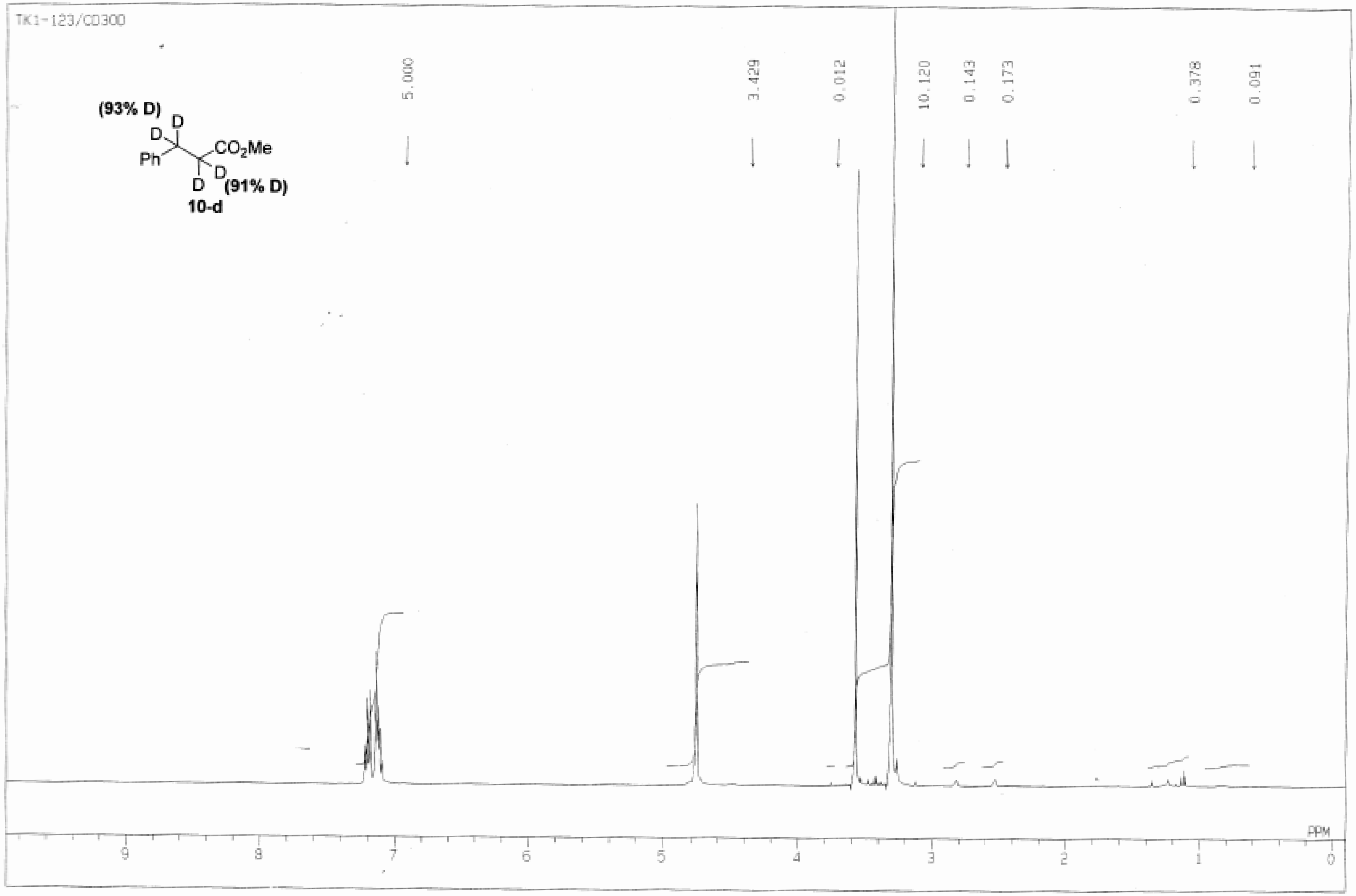
TK1-5/CD3OD
(76% D)
(72% D)
7-d
5.000
2.122
0.062
2.036
0.476
0.560
0.089
PPM
9
8
7
6
5
4
3
2
1
0



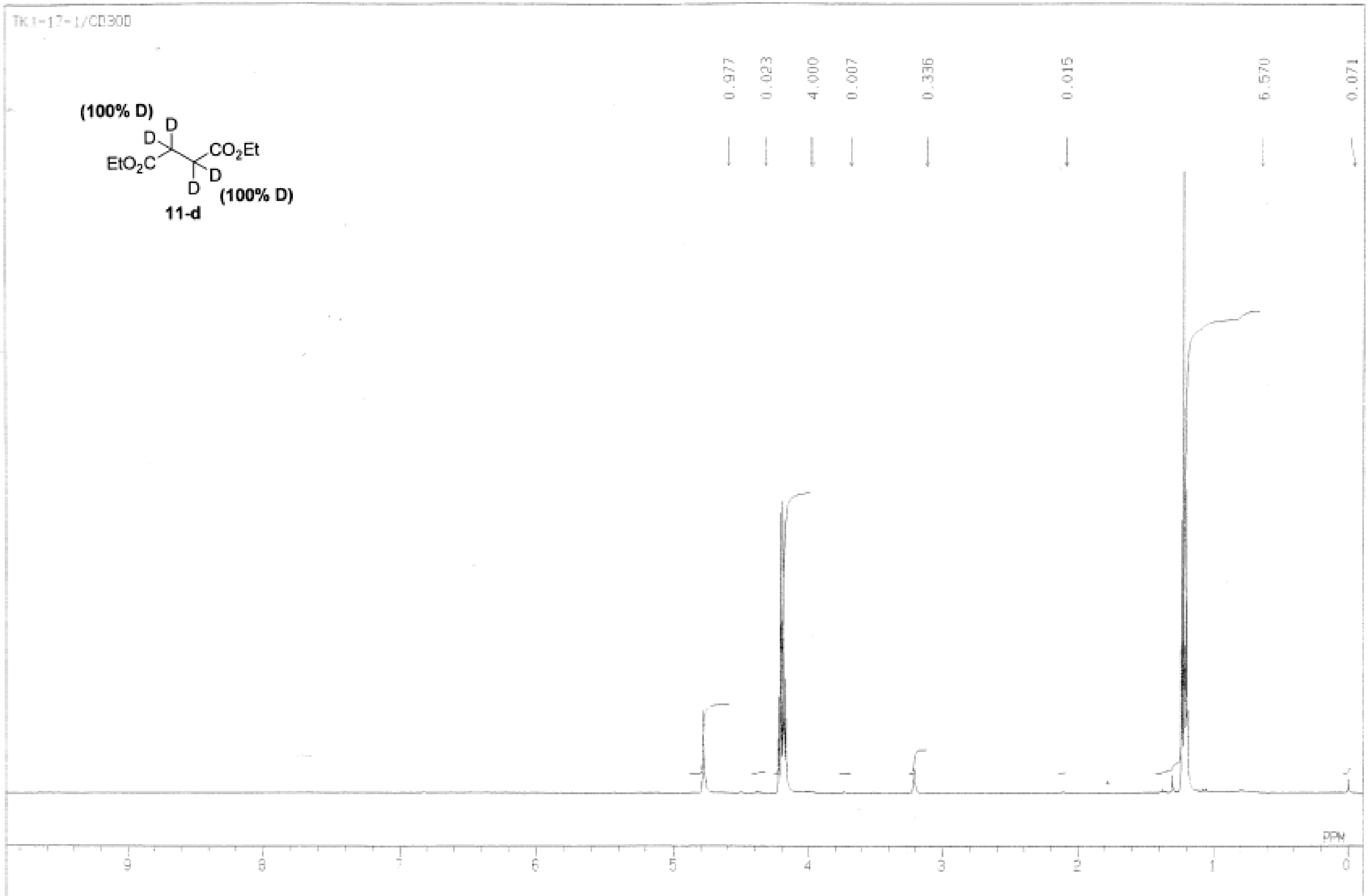
TK1-107/CD3OD
(88% D)
D
D
CO2D
Ph
D
D
(84% D)
8-d
5.000
1.029
0.183
0.248
0.312
0.104
0.040
PPM
9
8
7
6
5
4
3
2
1
0

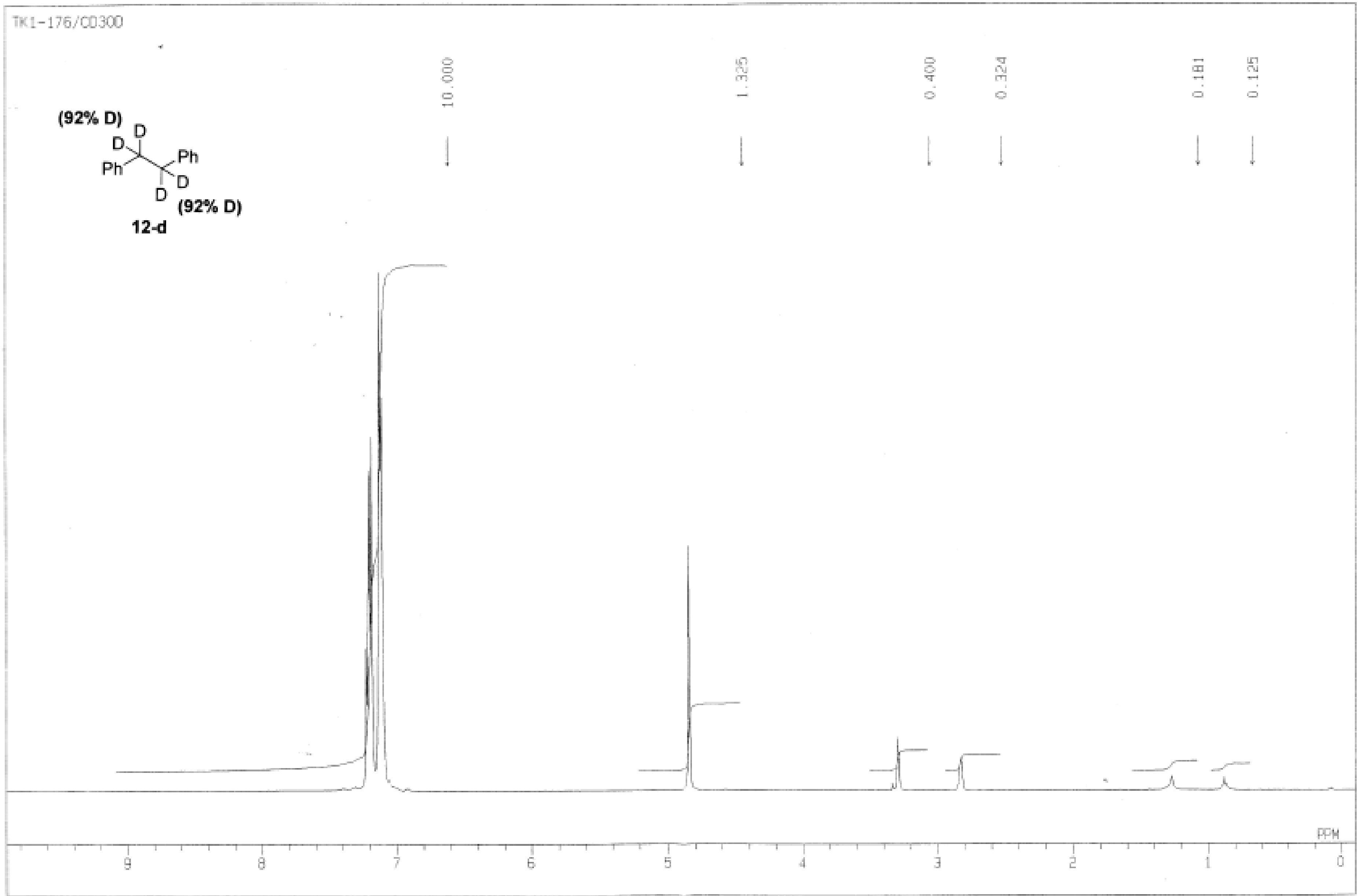


TK2-8/DMSO
(87% D)
D
D
CO2Na
Ph
D
D
(85% D)
9-d
5.000
2.651
3.685
0.306
0.366
PPM
2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8
PPM
12
10
8
6
4
2
0

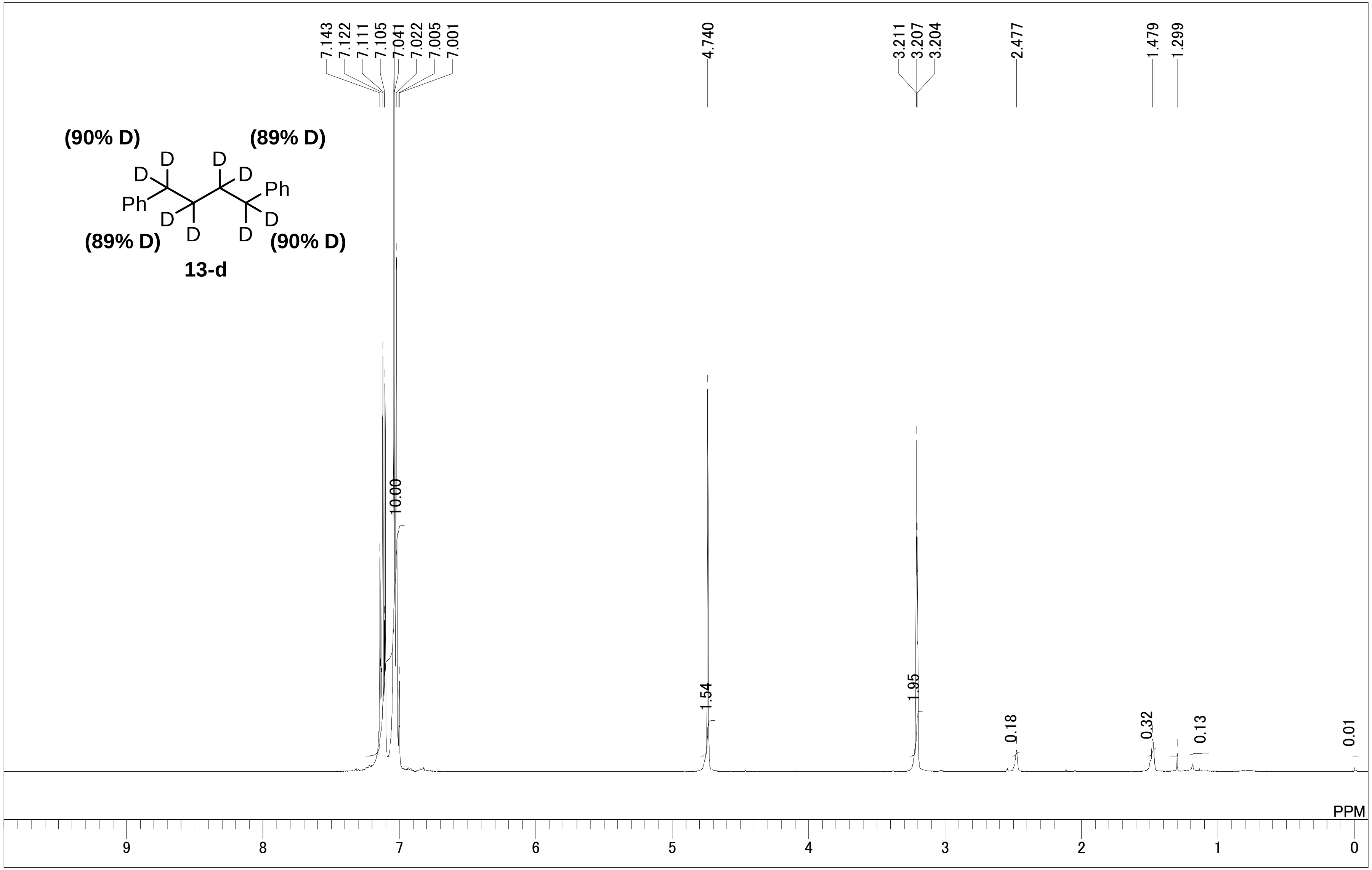


TK1-123/CD3OD
(93% D)
D
D
Ph
CO2Me
D
D
(91% D)
10-d
5.000
3.429
0.012
10.120
0.143
0.173
0.378
0.091
PPM
9
8
7
6
5
4
3
2
1
0

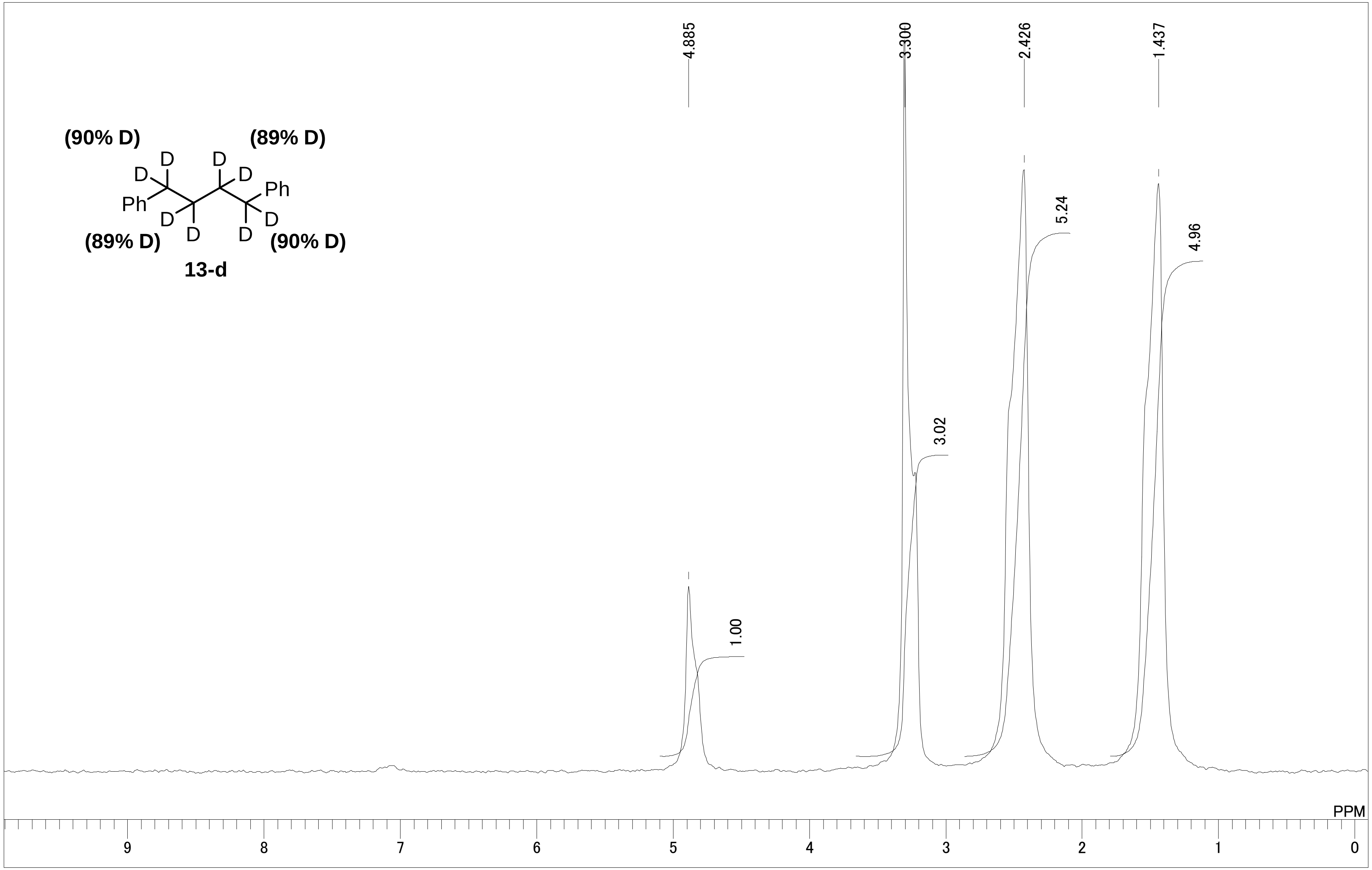

TK1-17-1/CD3OD
(100% D)
D
D
CO_2Et
EtO_2C
D
D
(100% D)
11-d
0.977
0.023
4.000
0.007
0.336
0.015
6.570
0.071
PPM
9
8
7
6
5
4
3
2
1
0

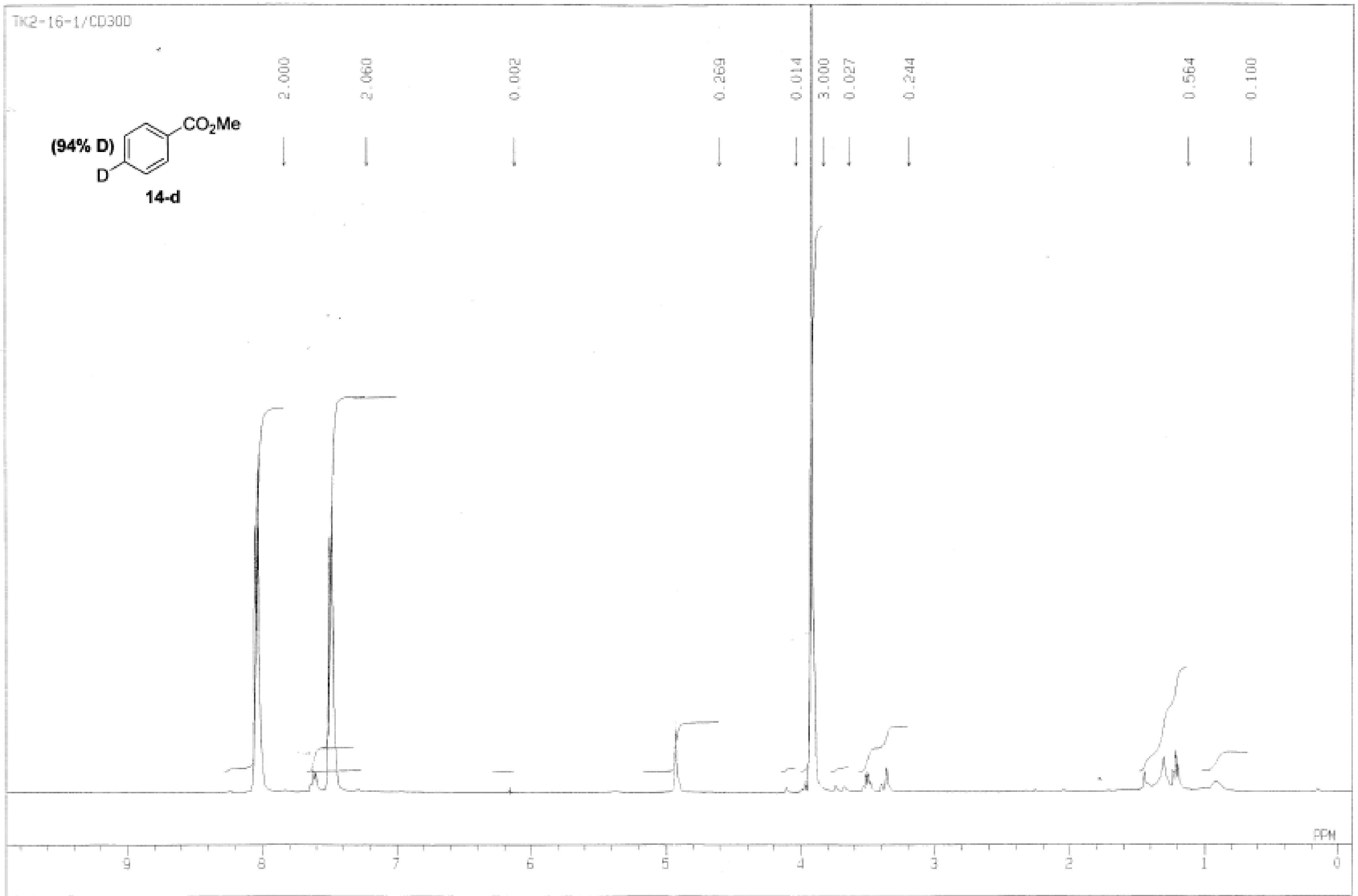

TK1-176/CD3OD
(92% D)
D
D
Ph
Ph
D
D
(92% D)
12-d
10.000
1.325
0.400
0.324
0.181
0.125
PPM
9
8
7
6
5
4
3
2
1
0

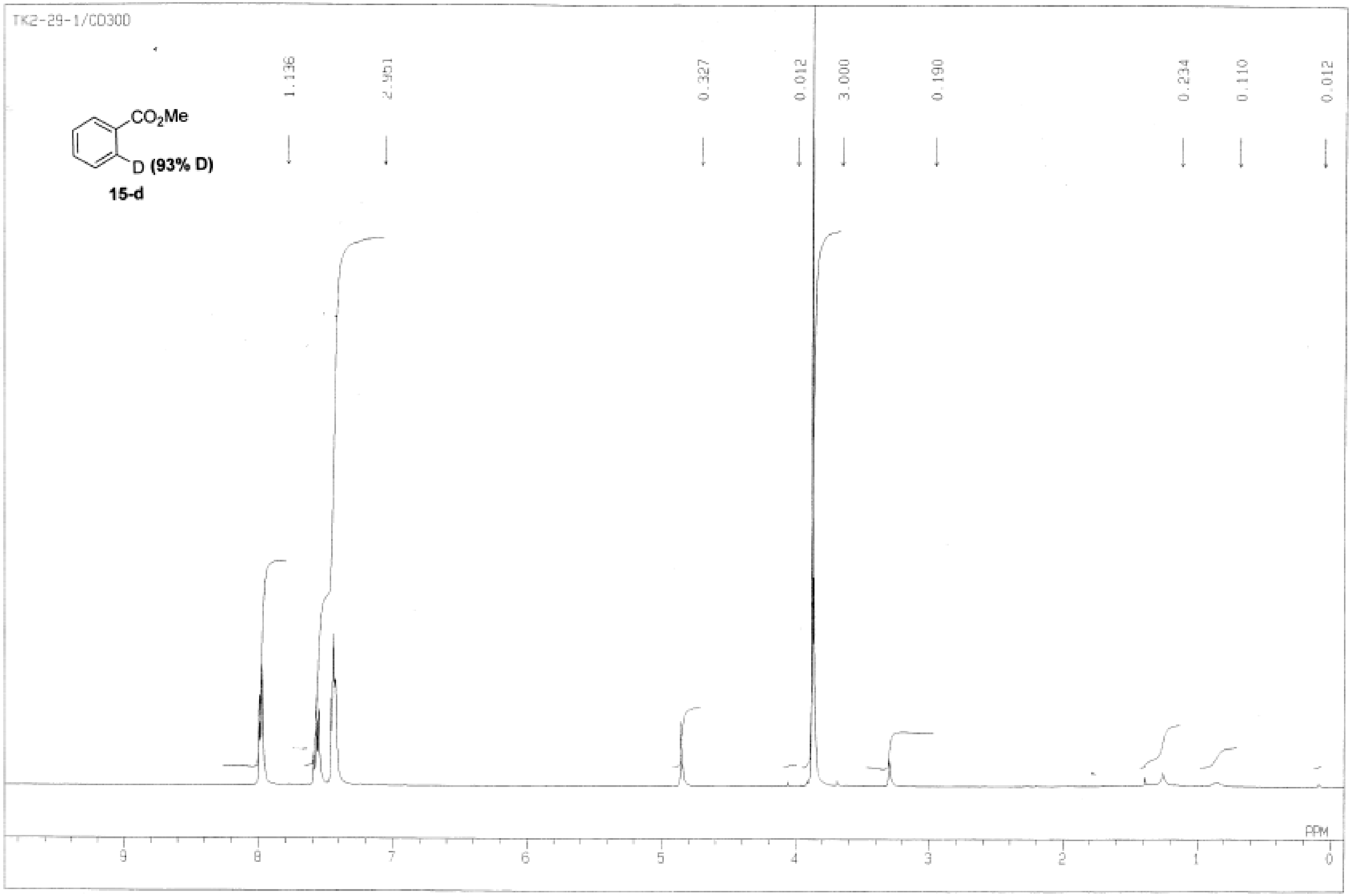
F:¥NMR 2007-5月¥TK7-1081NON_E2_FT.als
TK7-108



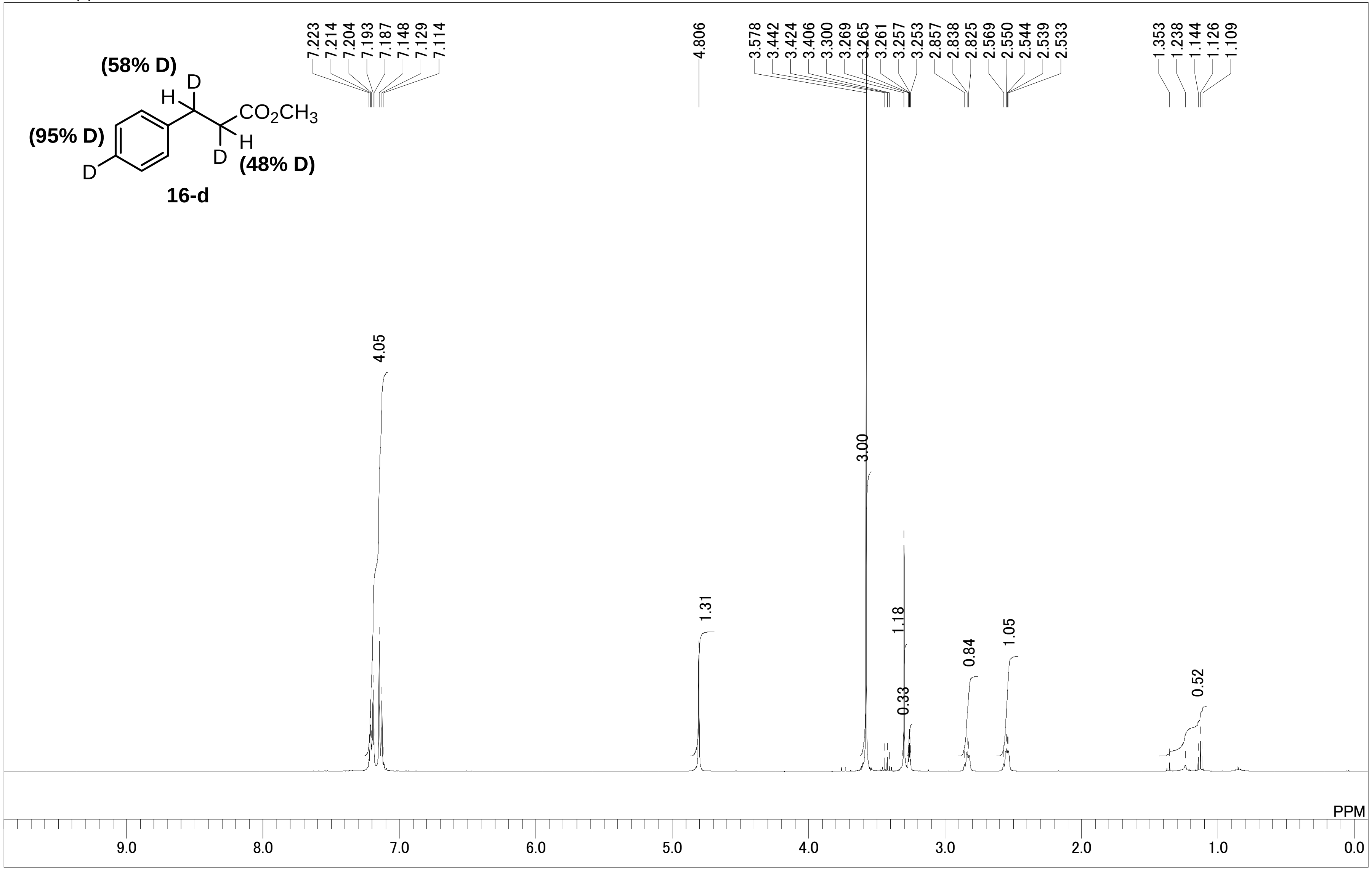
F:¥NMR 2007-5月¥2H NMR 5-28¥TK7-108-CH3OH-2H NMR-FT.als
TK7-108-CH3OH-2H NMR



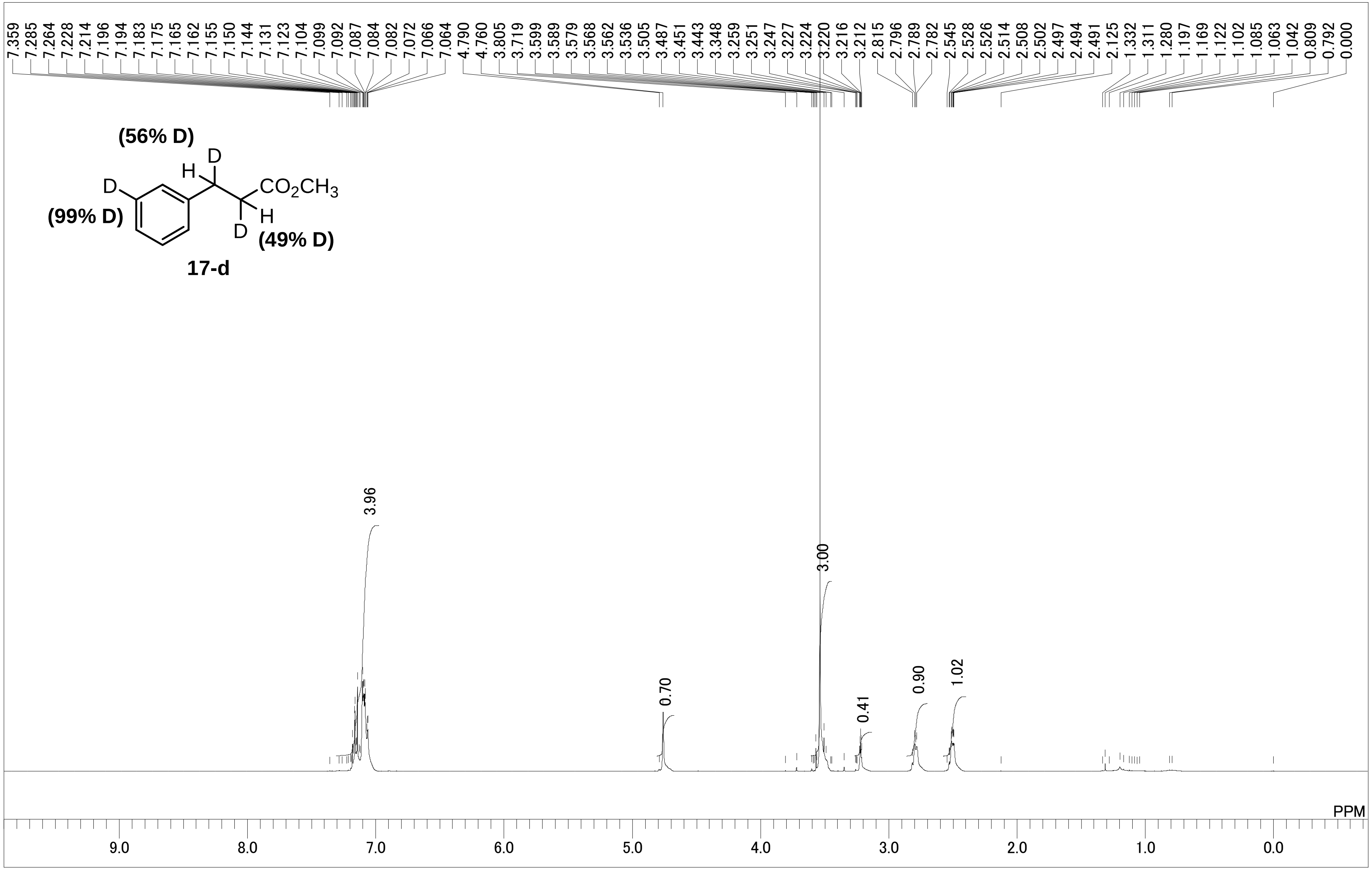

TK2-16-1/CD3OD
(94% D)
CO2Me
D
14-d
2.000
2.060
0.002
0.269
0.014
3.000
0.027
0.244
0.564
0.100
PPM
9
8
7
6
5
4
3
2
1
0

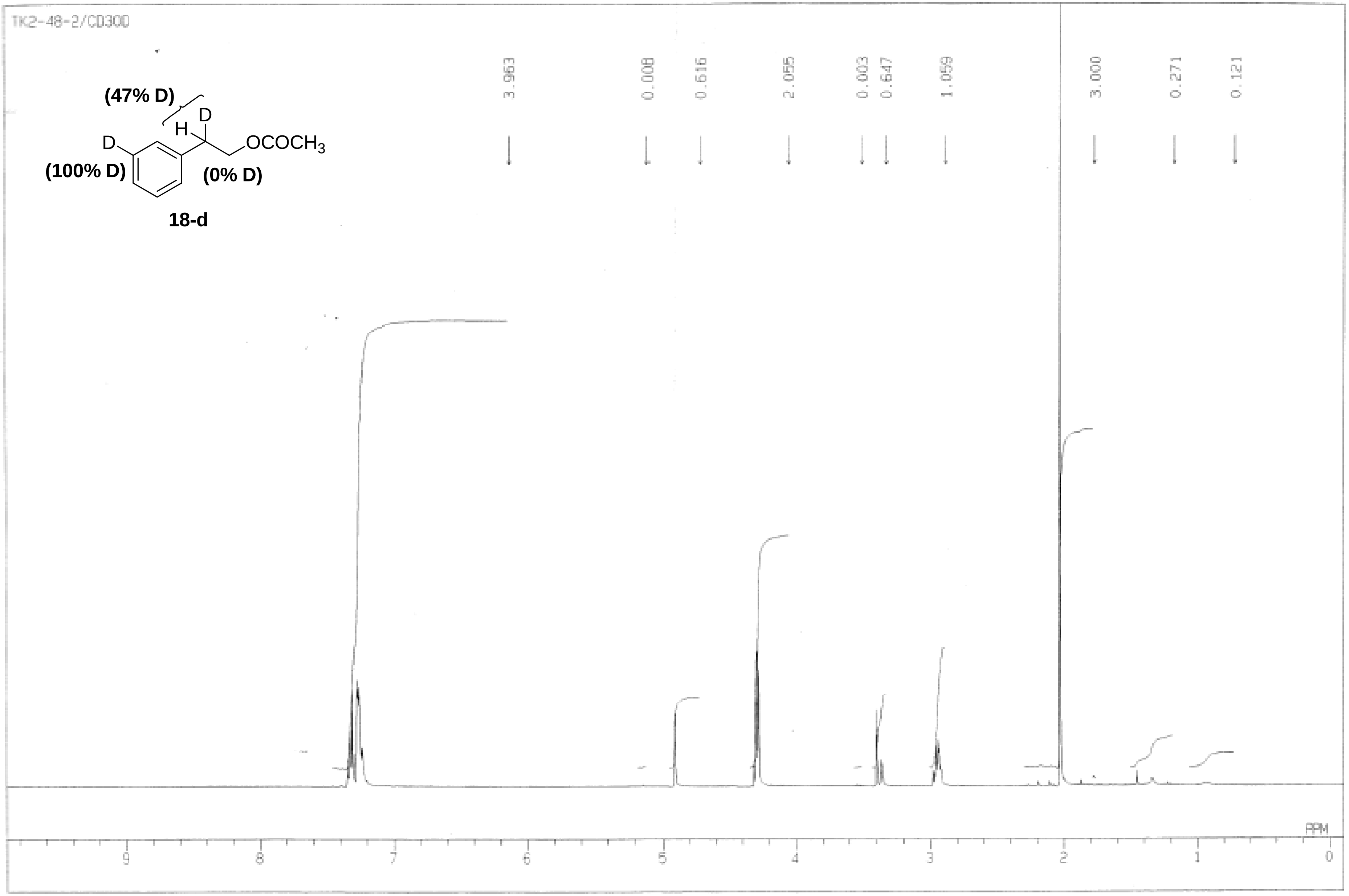

TK2-29-1/CD3OD
CO2Me
D (93% D)
15-d
1.136
2.951
0.327
0.012
3.000
0.190
0.234
0.110
0.012
PPM
9
8
7
6
5
4
3
2
1
0

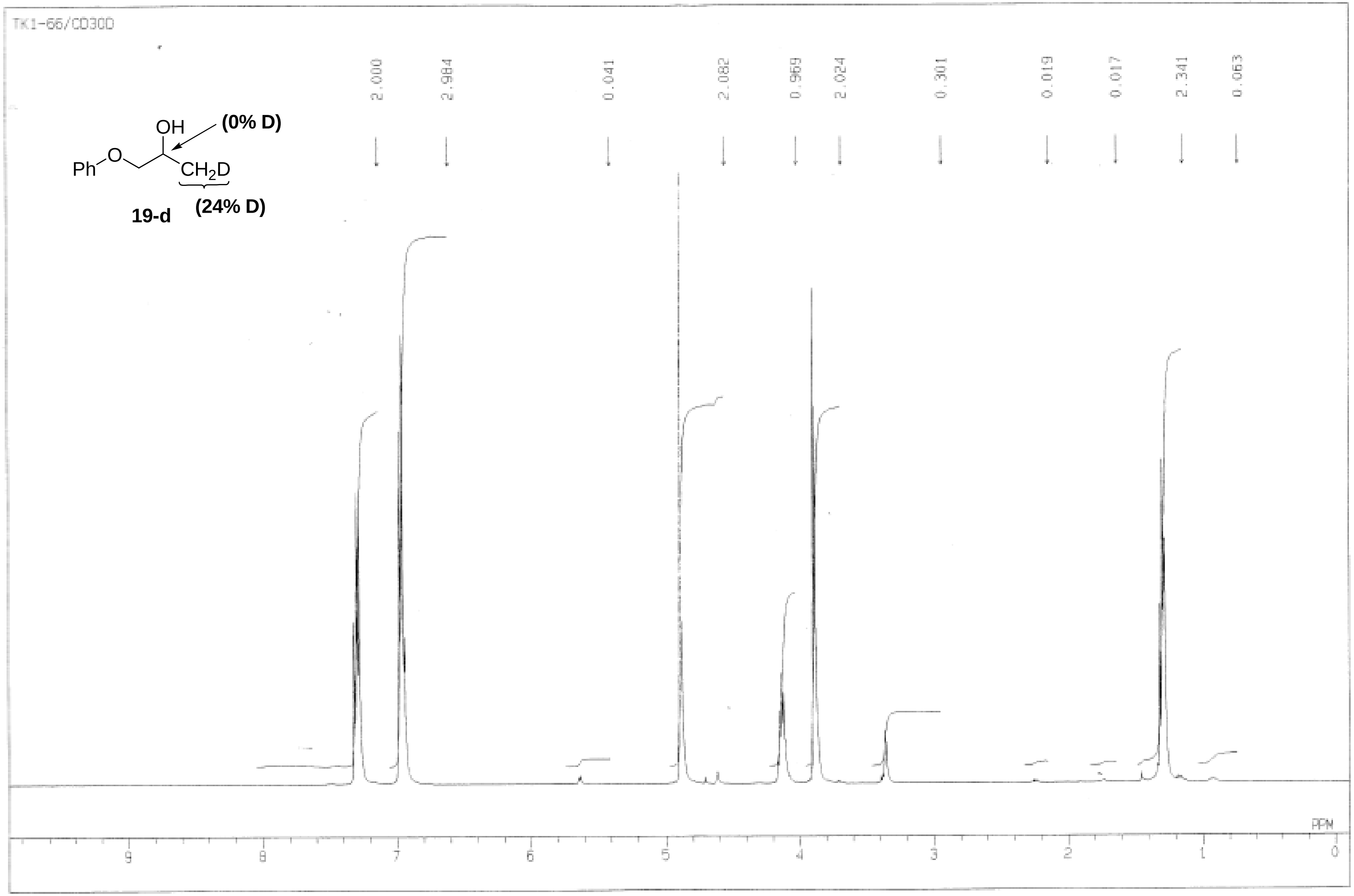
D:¥Data¥保存版 My Document¥NMR DATA¥NMR 2004-2005年¥NMR¥TK2-62-2(2)-FT.als
TK2-62-2(2)

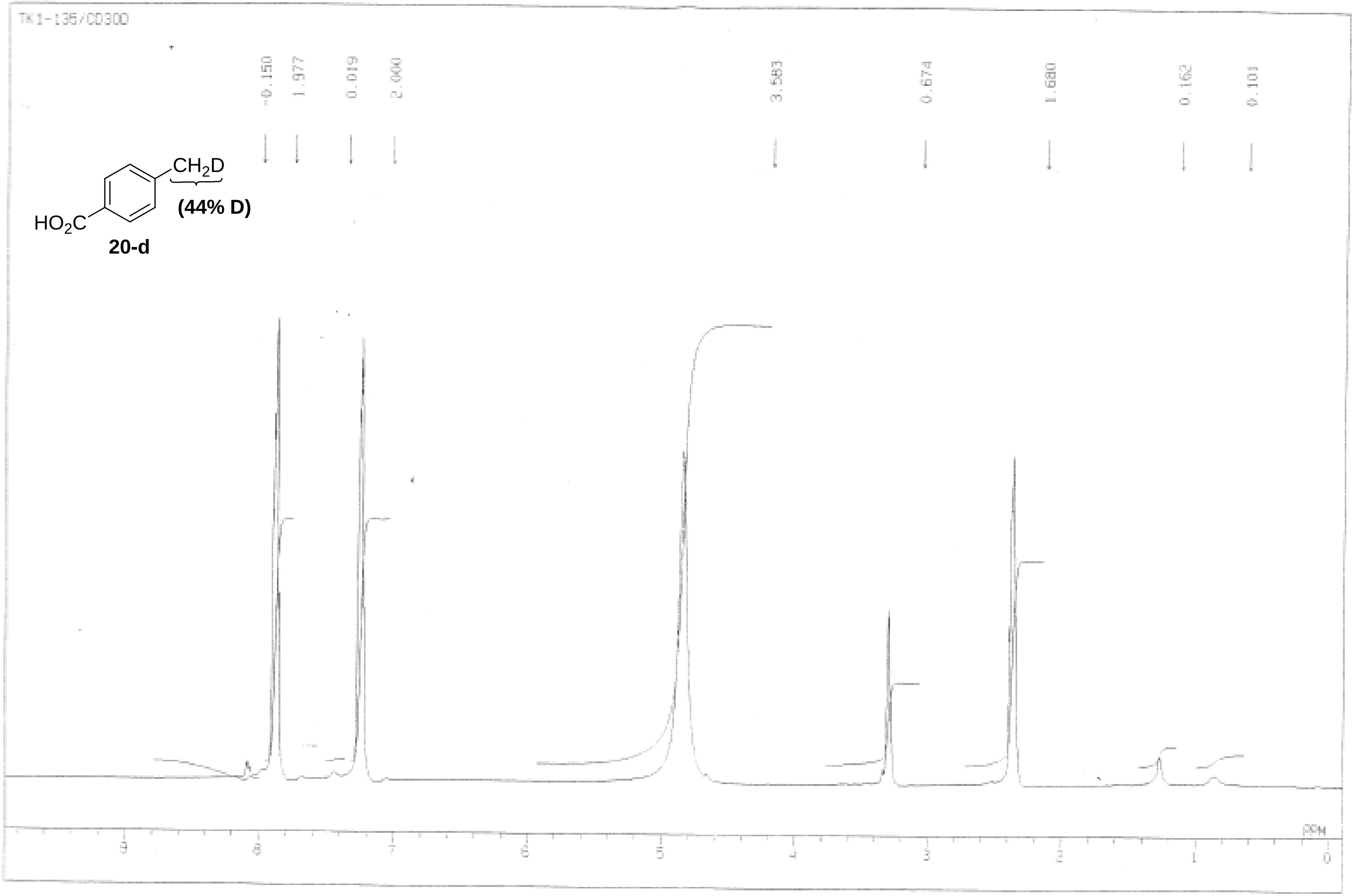


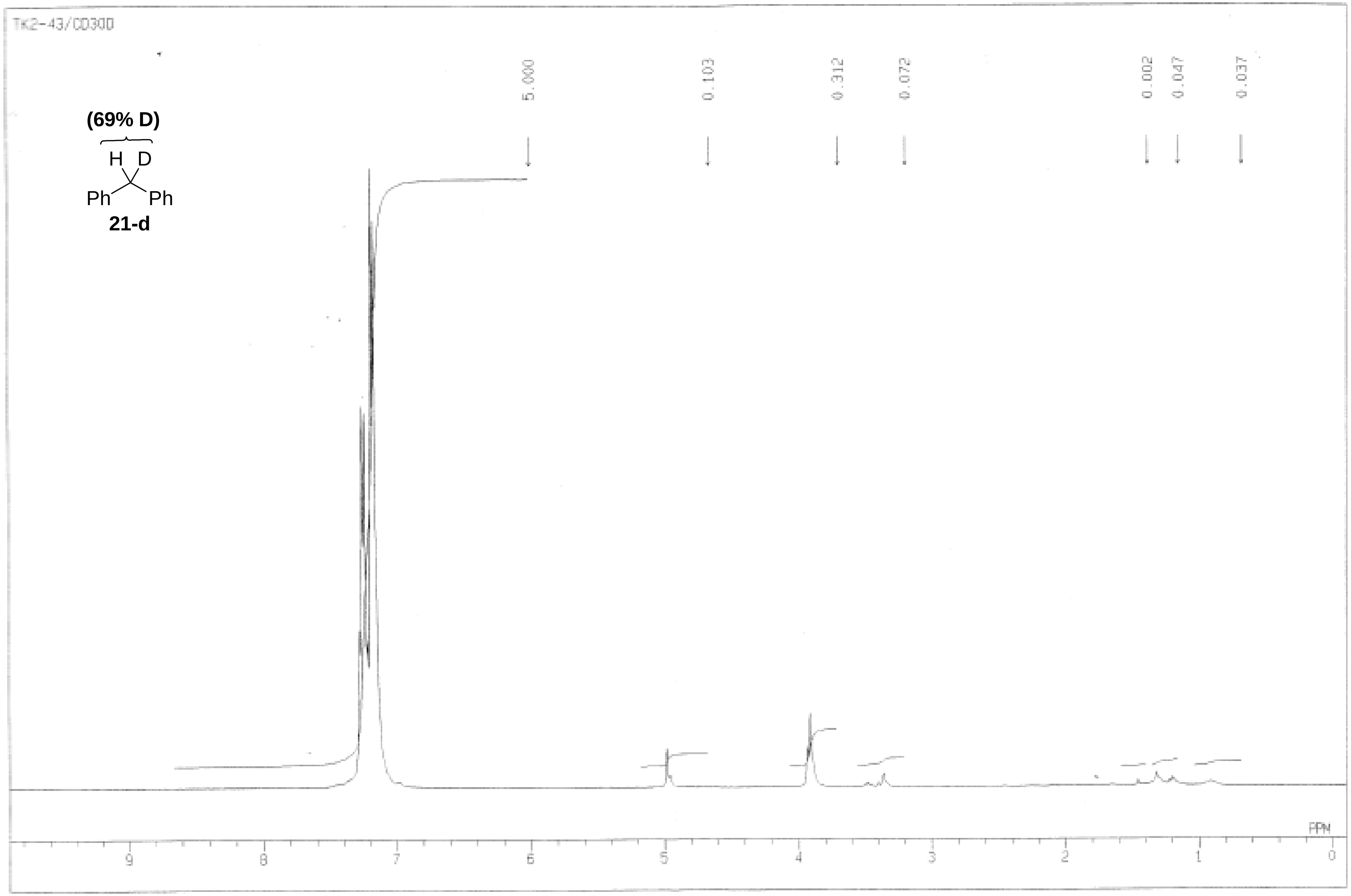
D:¥Data¥保存版 My Document¥NMR DATA¥NMR 2004-2005年¥NMR¥TK2-68-21NON_E2_FT.als
TK2-68-2



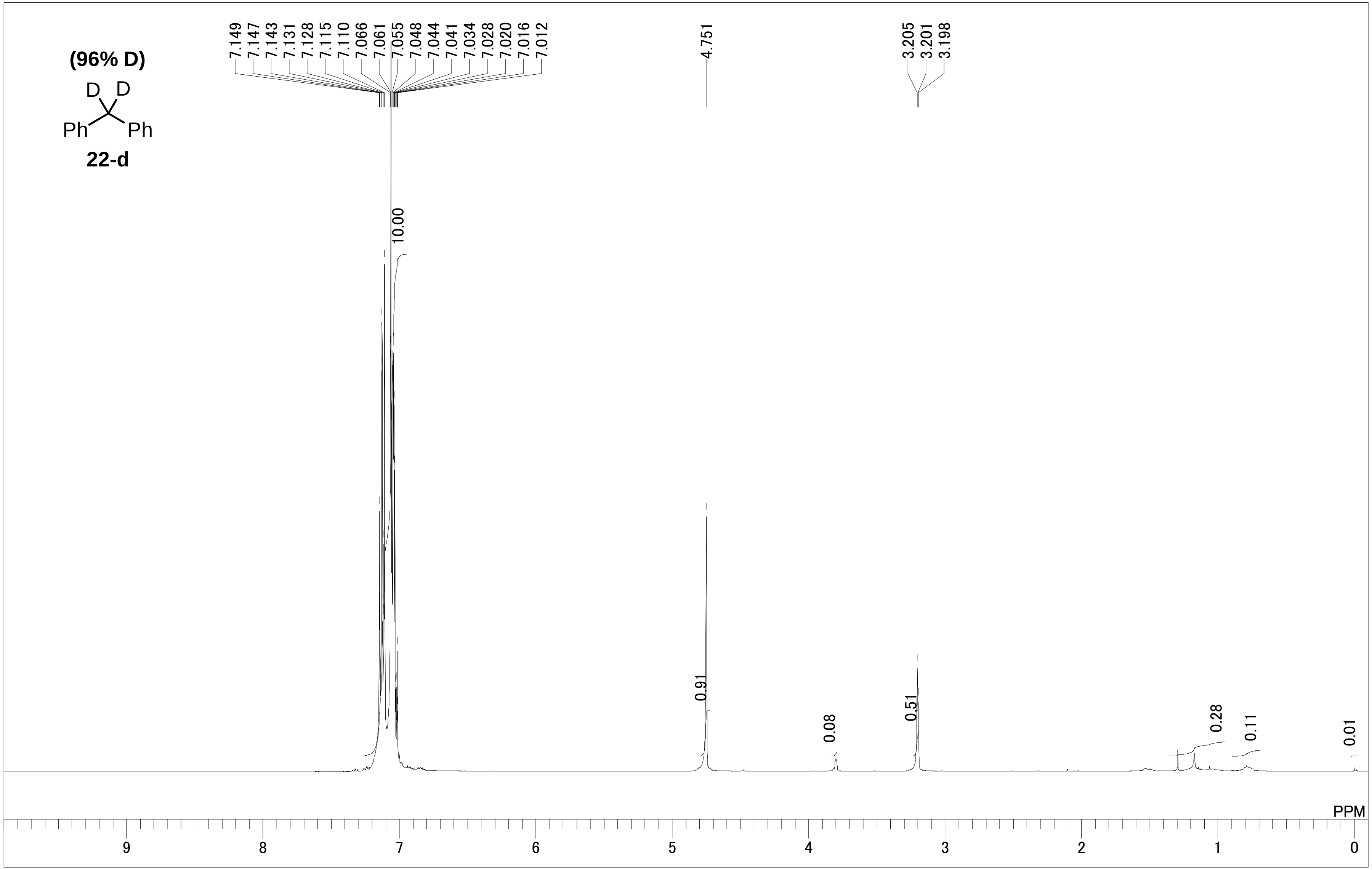

TK2-48-2/CD3OD
(47% D)
D
H
OCOCH3
D
(100% D)
(0% D)
18-d
3.963
0.008
0.616
2.055
0.003
0.647
1.059
3.000
0.271
0.121
PPM
9
8
7
6
5
4
3
2
1
0


TK1-66/CD3OD
2.000
2.984
0.041
2.082
0.969
2.024
0.301
0.019
0.017
2.341
0.063
OH
(0% D)
Ph
O
CH2D
19-d
(24% D)
PPM
9
8
7
6
5
4
3
2
1
0

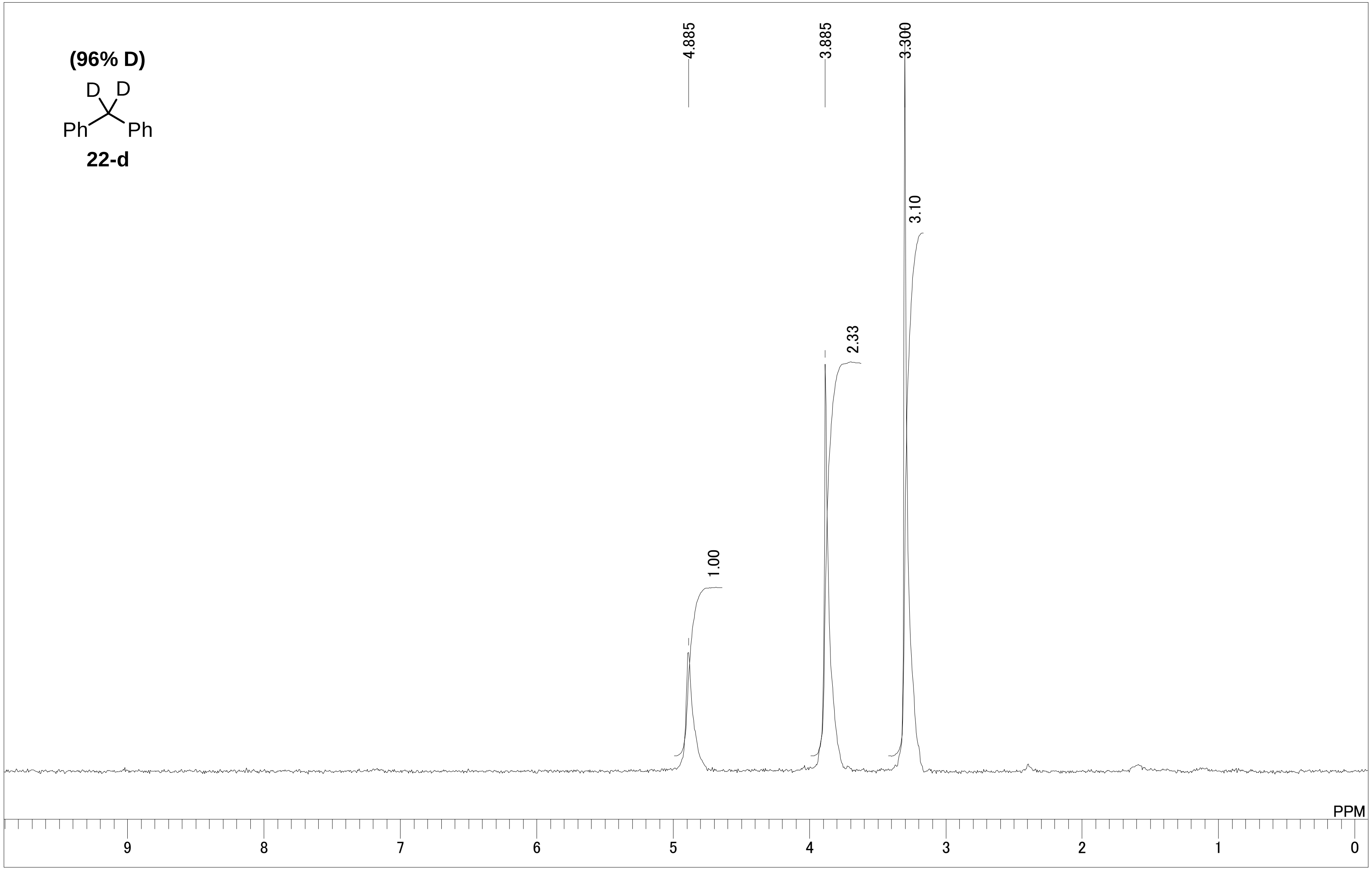

TK1-135/CD3OD
HO2C
CH2D
(44% D)
20-d
PPM


TK2-43/CD3OD
(69% D)
H D
Ph Ph
21-d
5.000
0.103
0.312
0.072
0.002
0.047
0.037
PPM
9
8
7
6
5
4
3
2
1
0

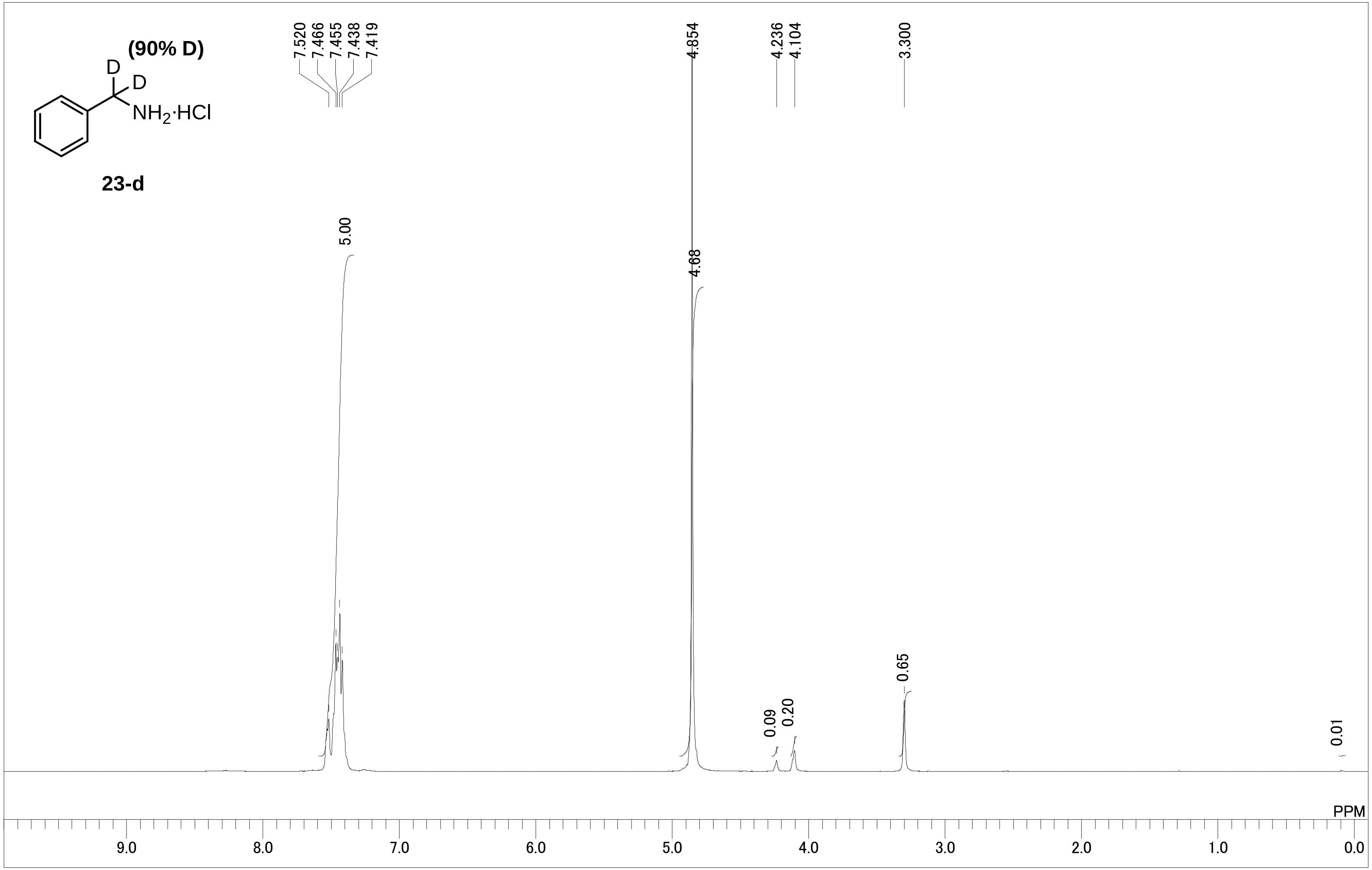
F:¥NMR 2007-5月¥TK7-1011NON_E5_FT.als
TK7-101



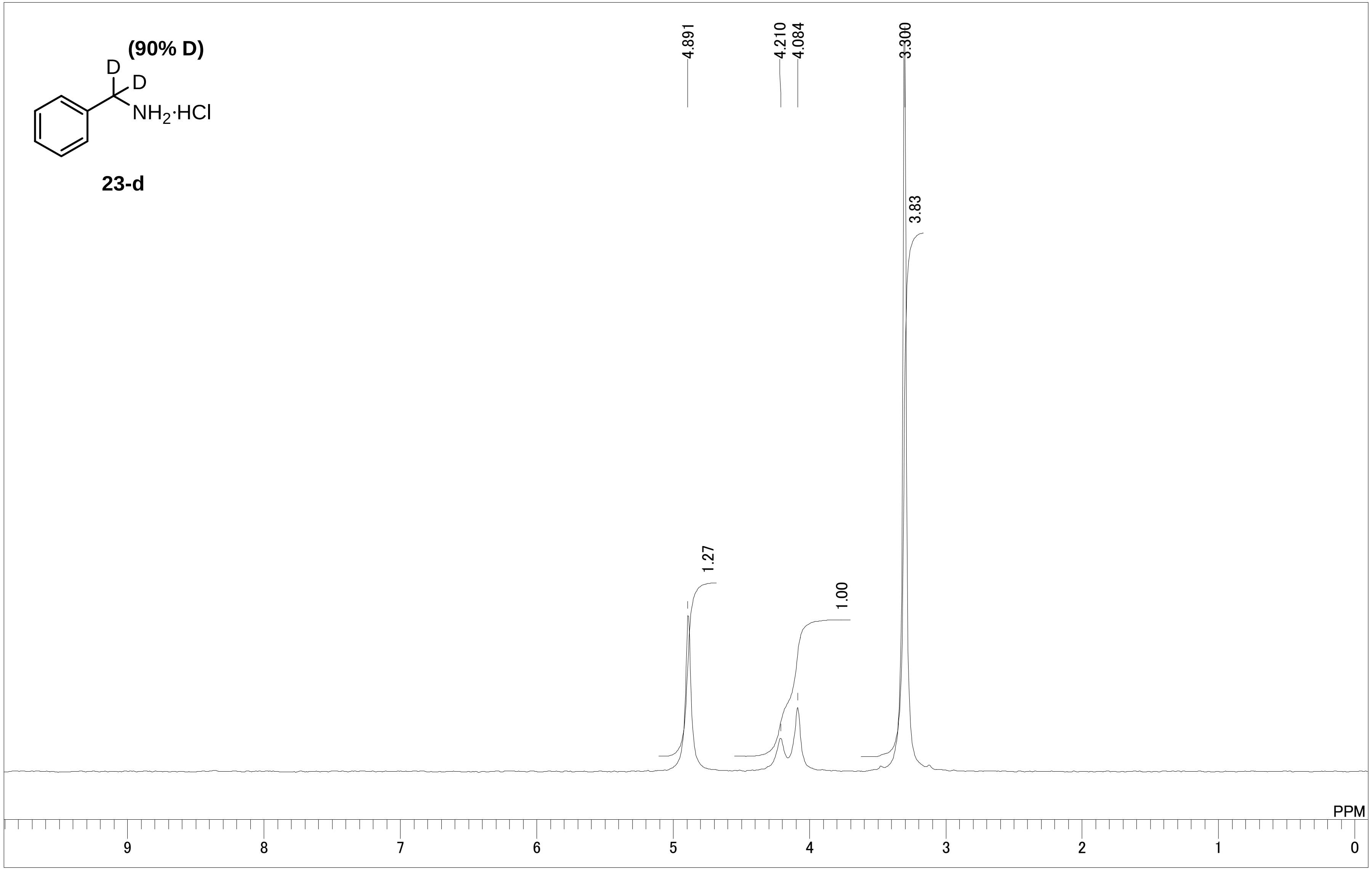
F:¥NMR 2007-5月¥2H NMR, 5-13¥TK7-101-2H NMR-CH3OH-FT.als
TK7-101-2H NMR-CH3OH



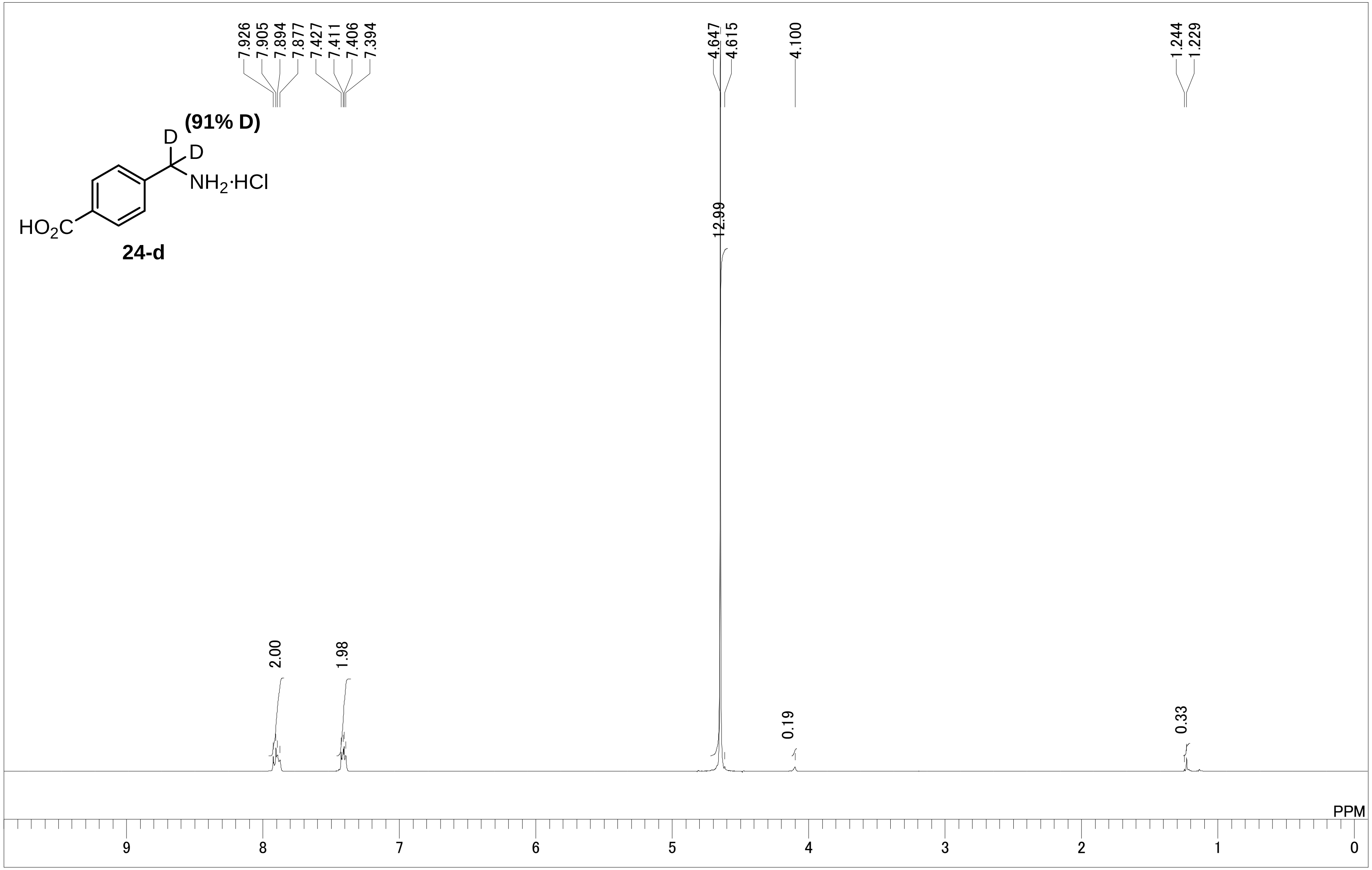
F:¥NMR 2007-5月¥TK7-102-FT.als
TK7-102



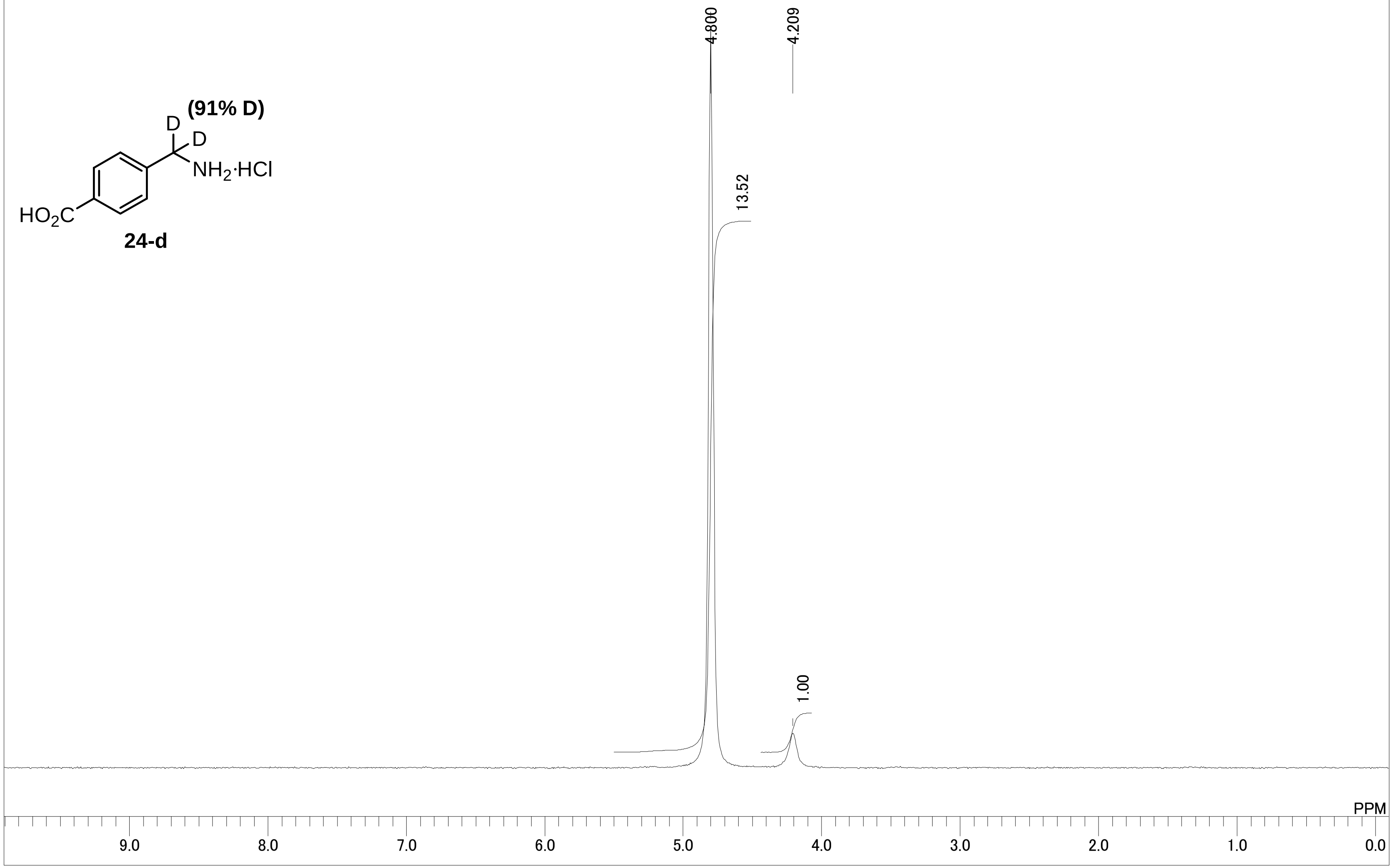
F:¥NMR 2007-5月¥2H NMR 5-28¥TK7-102-CH3OH-2H NMR-FT.als
TK7-102-CH3OH-2H NMR



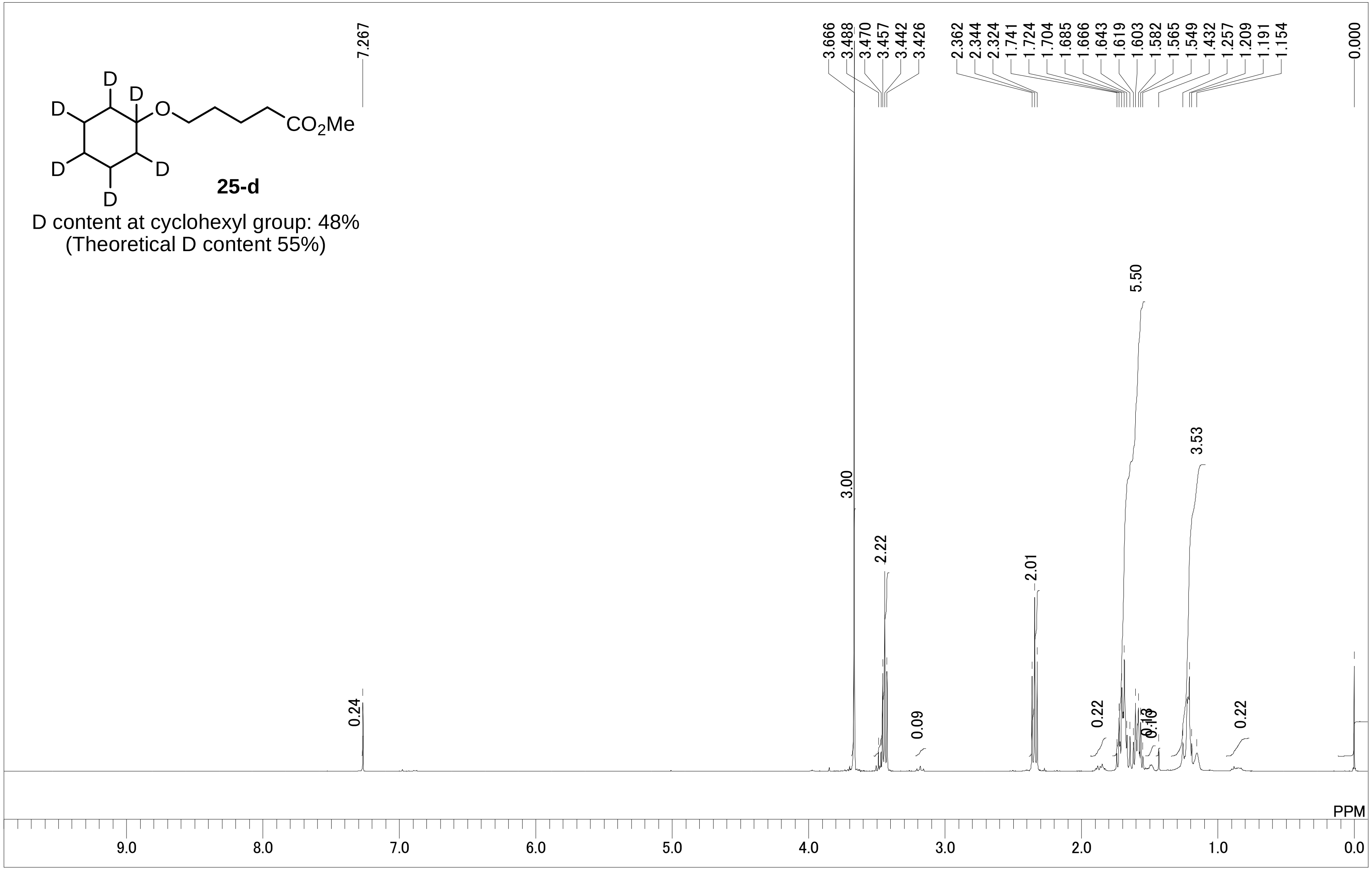
D:¥Data¥NMR 2007年¥NMR 2007-2月¥TK6-1961NON_E8_FT.als
TK6-196



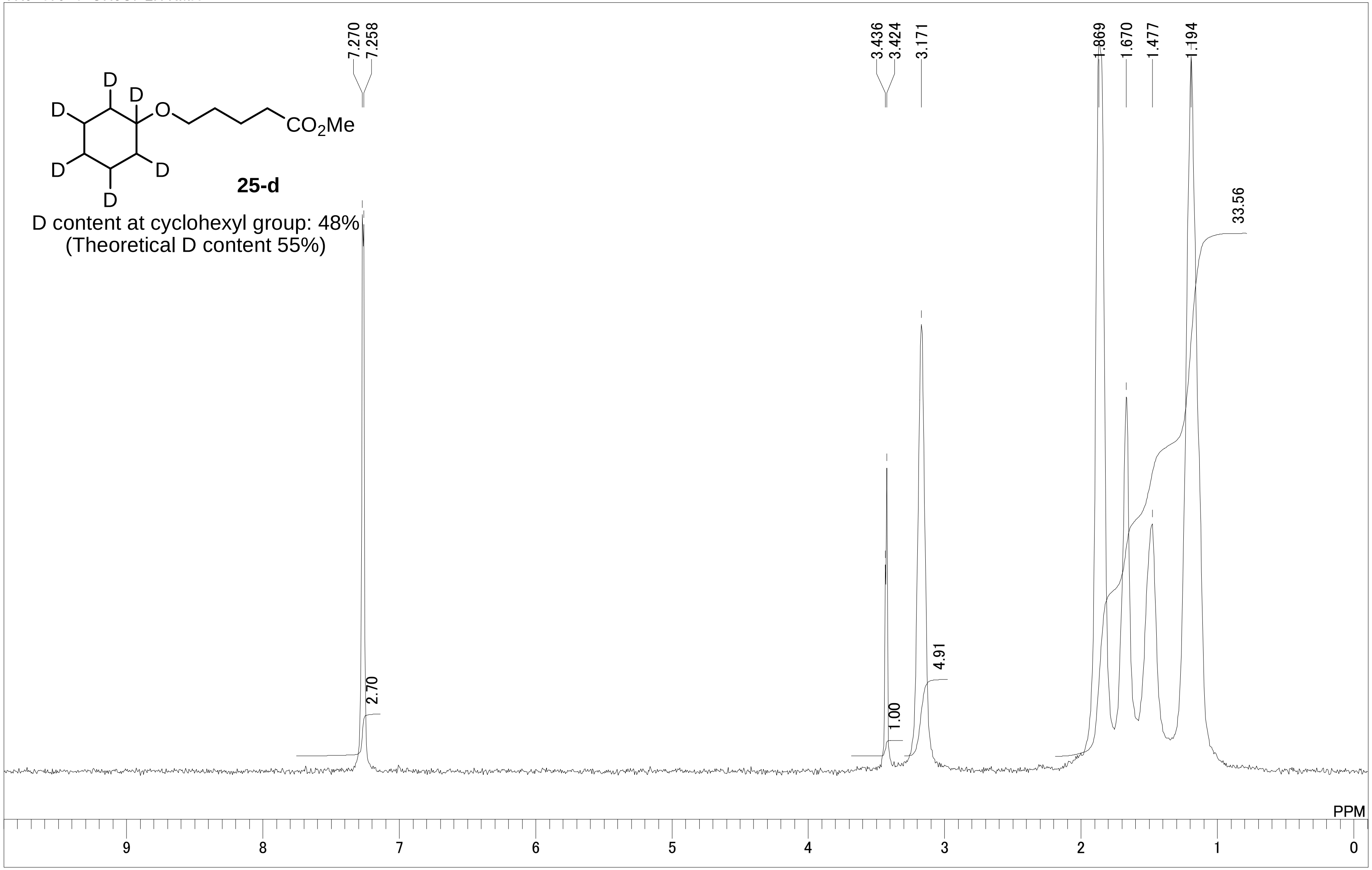
D:¥Data¥NMR 2007年¥NMR 2007-2月¥2H 2-21¥TK6-196-H2O-2H NMR-FT.als
TK6-196-H2O-2H NMR



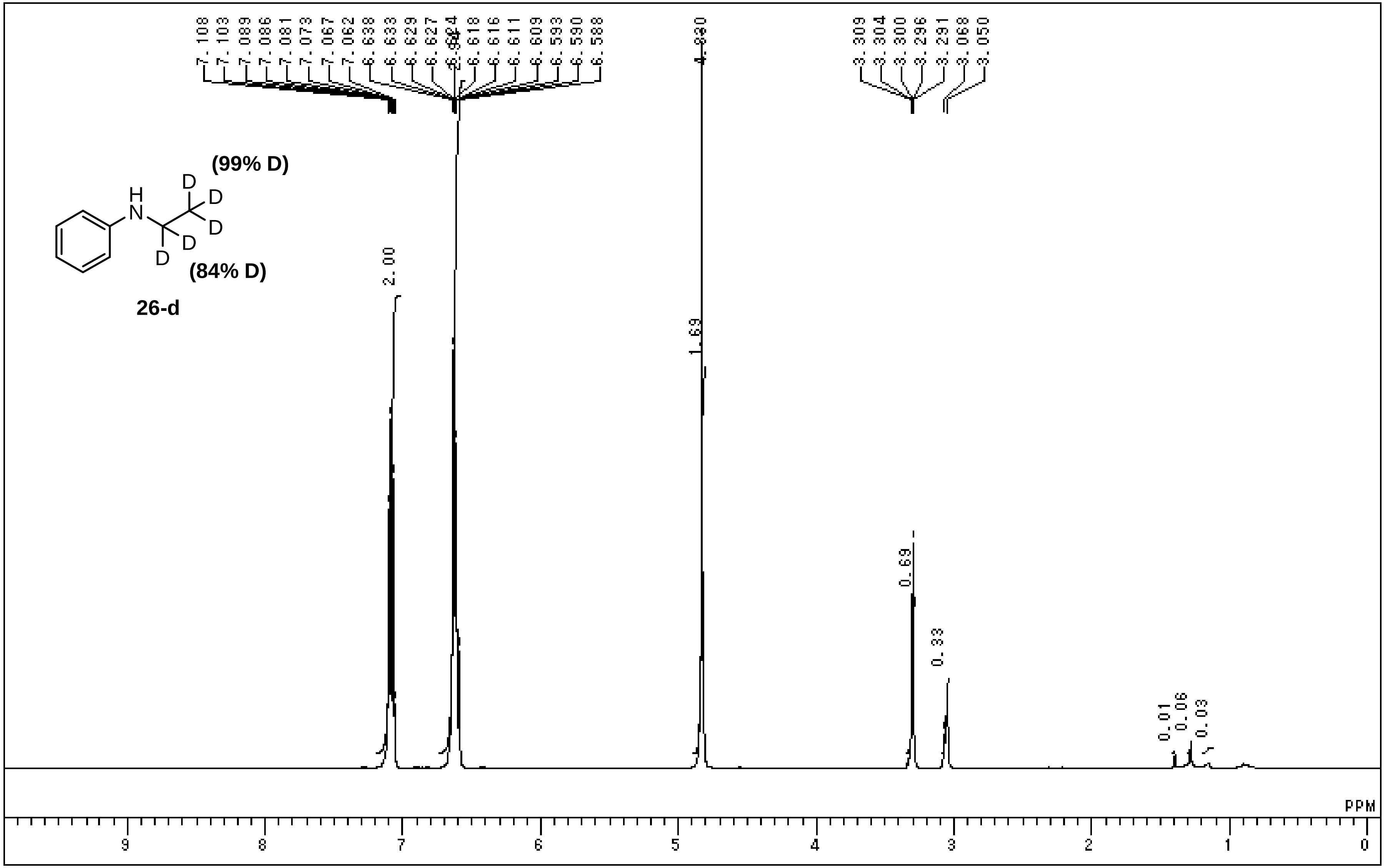
D:¥Data¥NMR 2007年¥NMR 2007-2月¥TK6-179-1-CDCl31NON_E1_FT.als
TK6-179-1-CDCl3



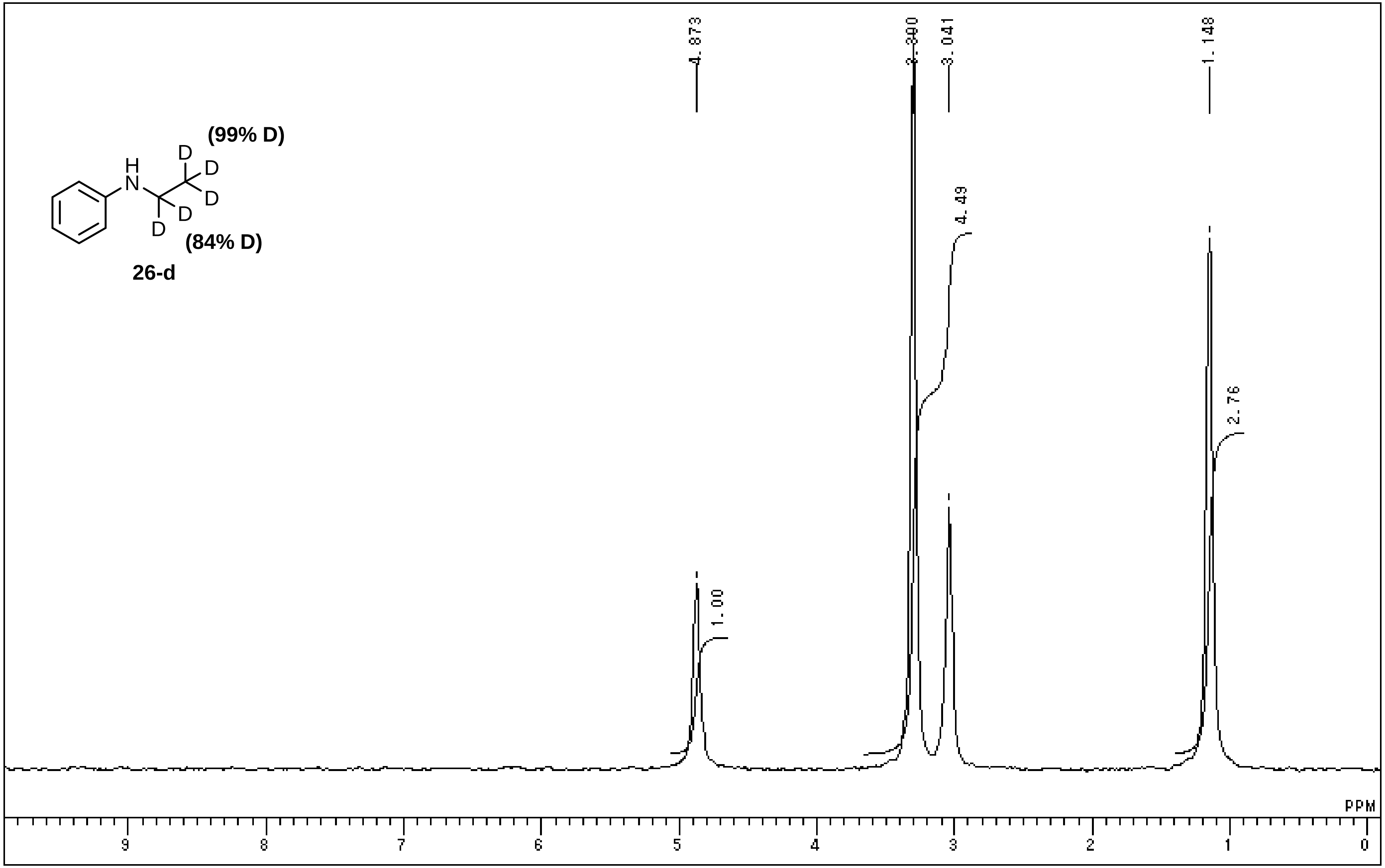
D:¥Data¥NMR 2007年¥NMR 2007-2月¥TK6-179-1-CH3Cl-2H NMR-FT.als
TK6-179-1-CH3Cl-2H NMR



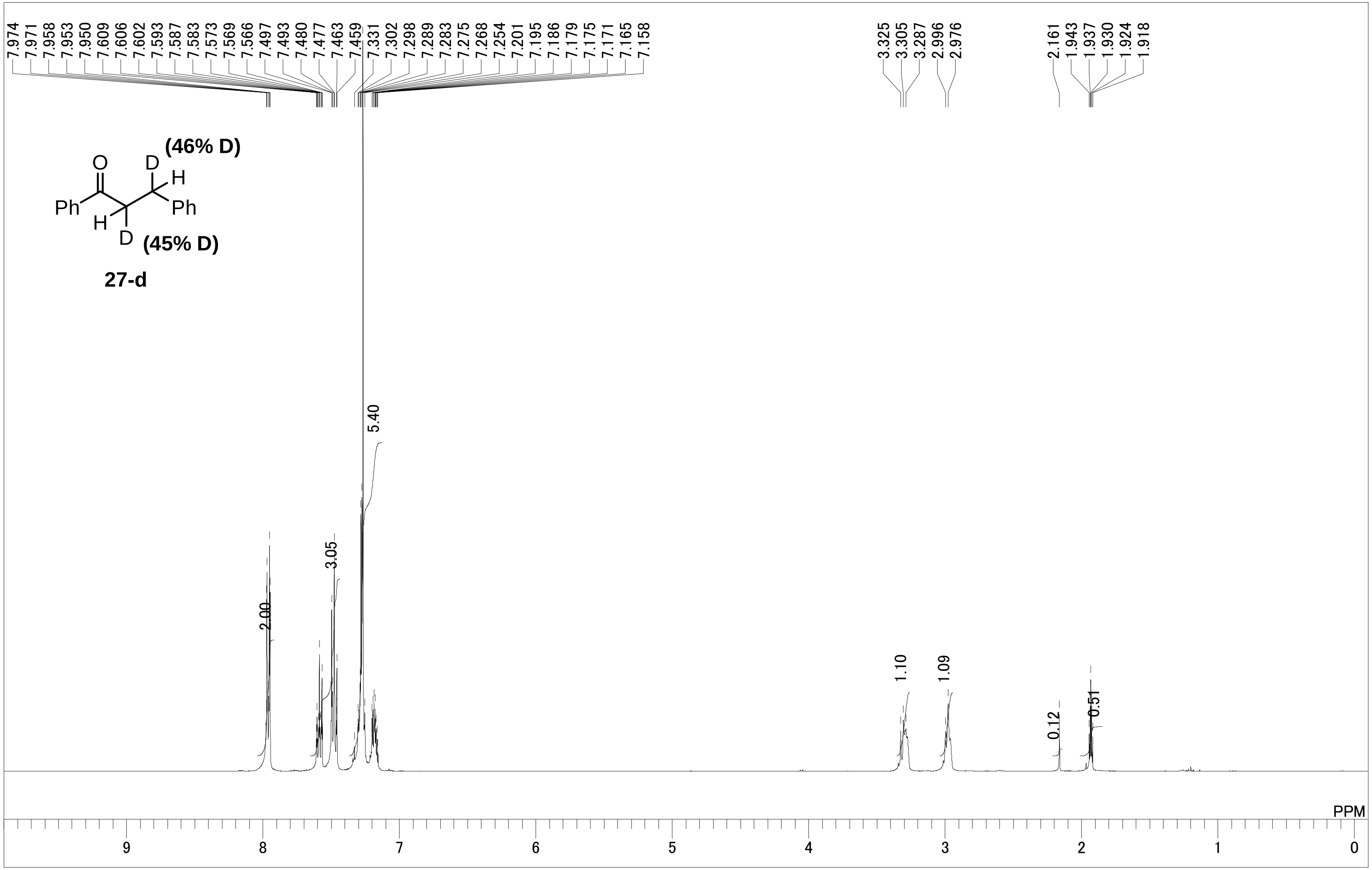
F:¥NMR 2007-9月¥TK8-121NON_E2_FT.als
TK8-12



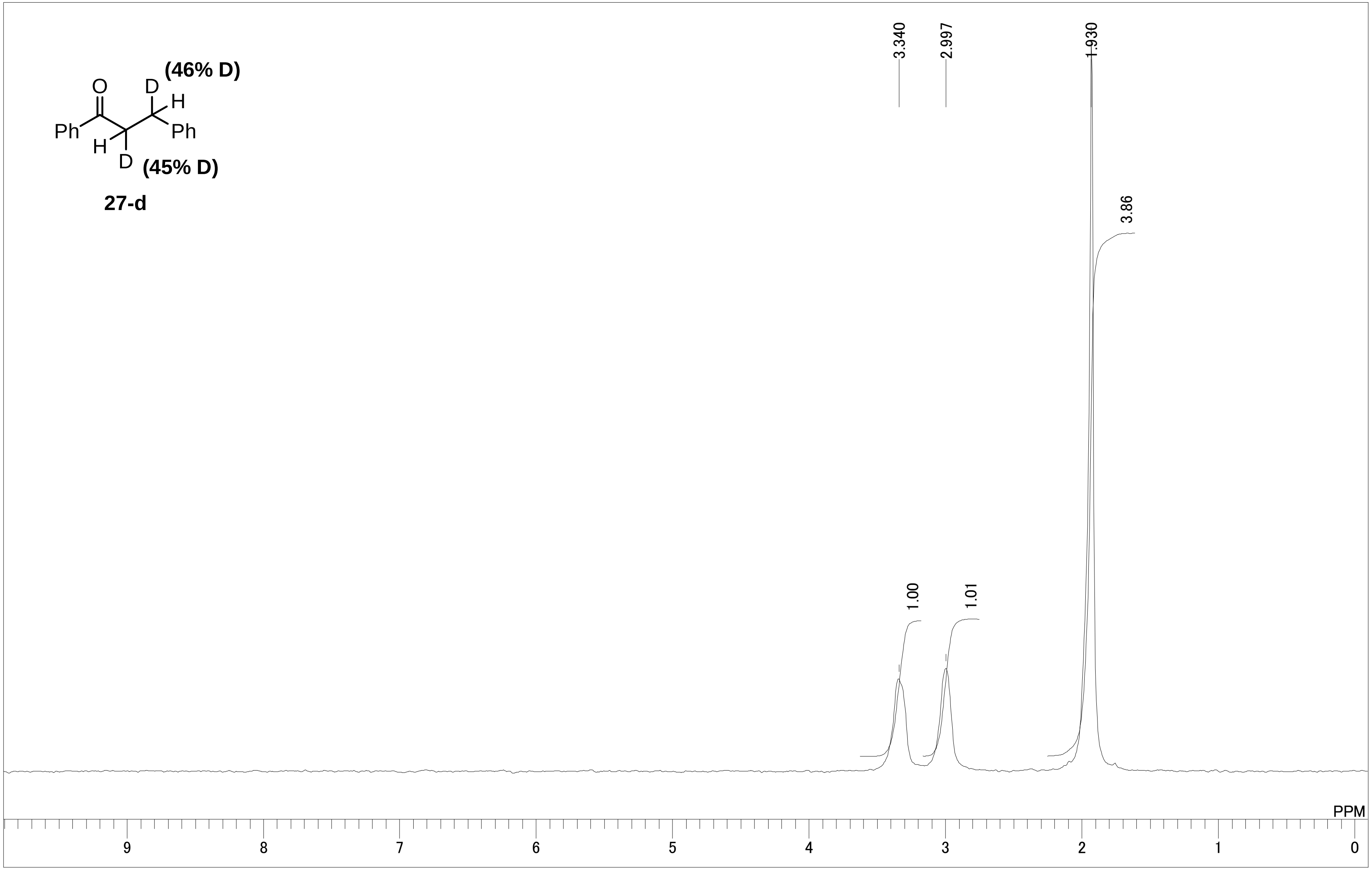
F:¥NMR 2007-9月¥TK8-12-2H NMR-CH3OH-FT.als
TK8-12-2H NMR-CH3OH

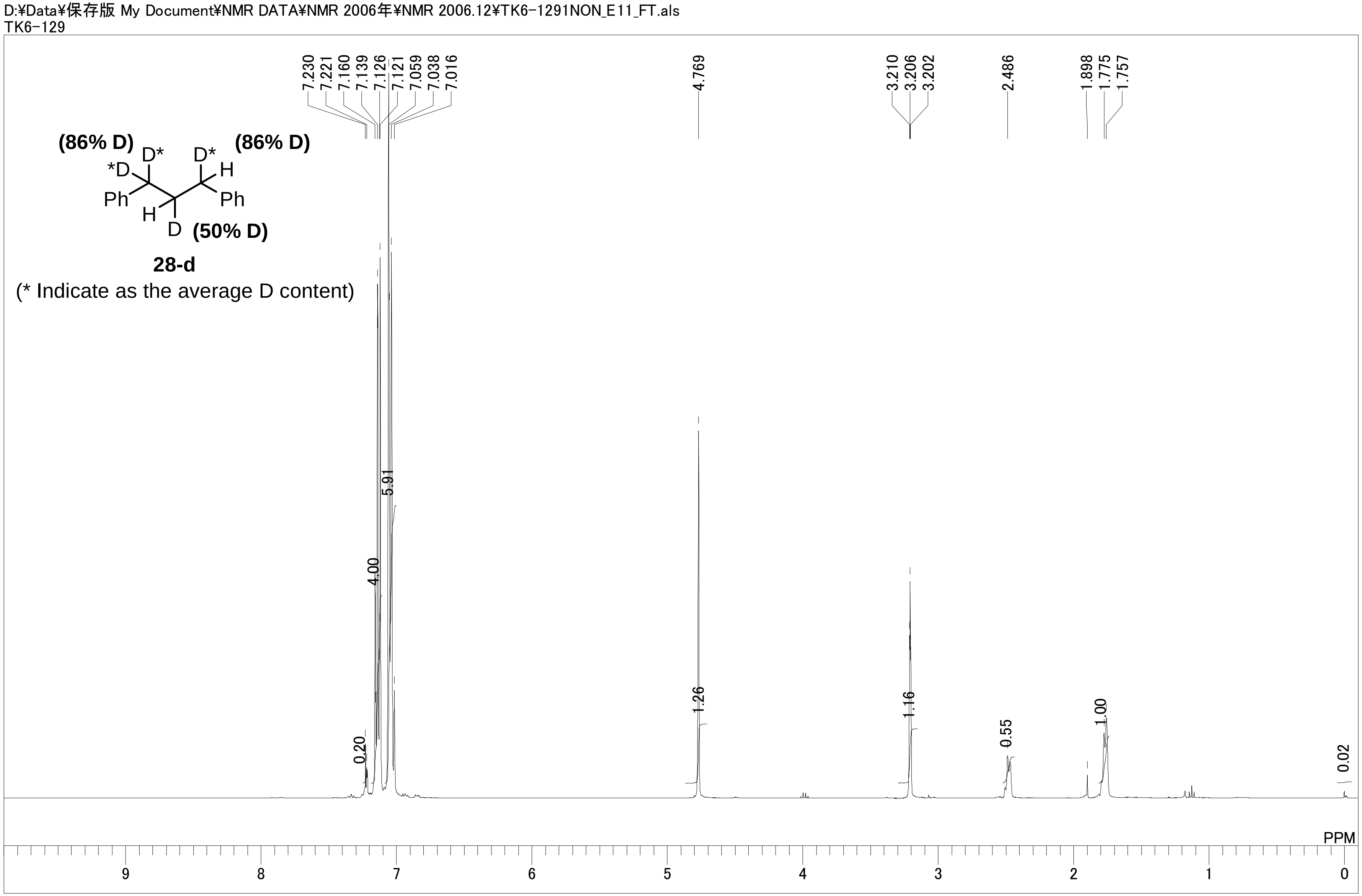


D:¥Data¥保存版 My Document¥NMR DATA¥NMR 2006年¥NMR 2006.12¥TK6-136-FT.als
TK6-136

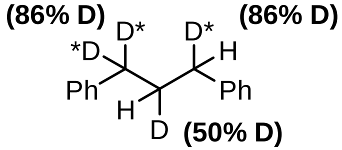


F:¥NMR 2007-5月¥2H NMR 5-31¥TK6-136-CH3CN-2H NMR-FT.als
TK6-136-CH3CN-2H NMR



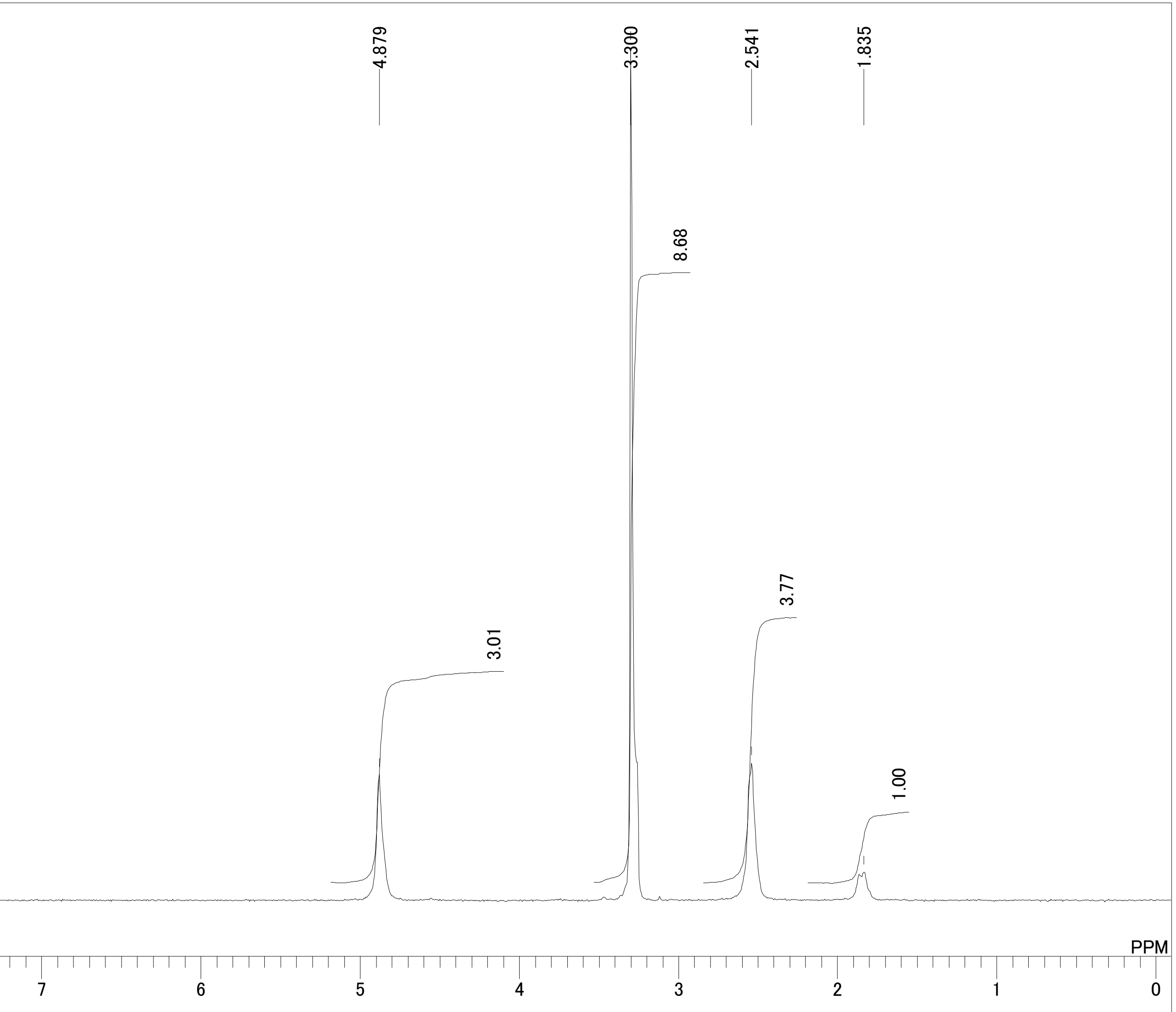

D:¥Data¥保存版 My Document¥NMR DATA¥NMR 2006年¥NMR 2006.12¥TK6-1291NON_E11_FT.als
TK6-129
7.230
7.221
7.160
7.139
7.126
7.121
7.059
7.038
7.016
4.769
3.210
3.206
3.202
2.486
1.898
1.775
1.757
(86% D)
D*
*D
D*
H
(86% D)
Ph
Ph
H
D
(50% D)
28-d
(* Indicate as the average D content)
5.91
4.00
0.20
1.26
1.16
0.55
1.00
0.02
PPM
9
8
7
6
5
4
3
2
1
0

D:¥Data¥NMR 2007年¥NMR 2007-1月¥TK6-129-2H NMR-FT.als
TK6-129-2H NMR

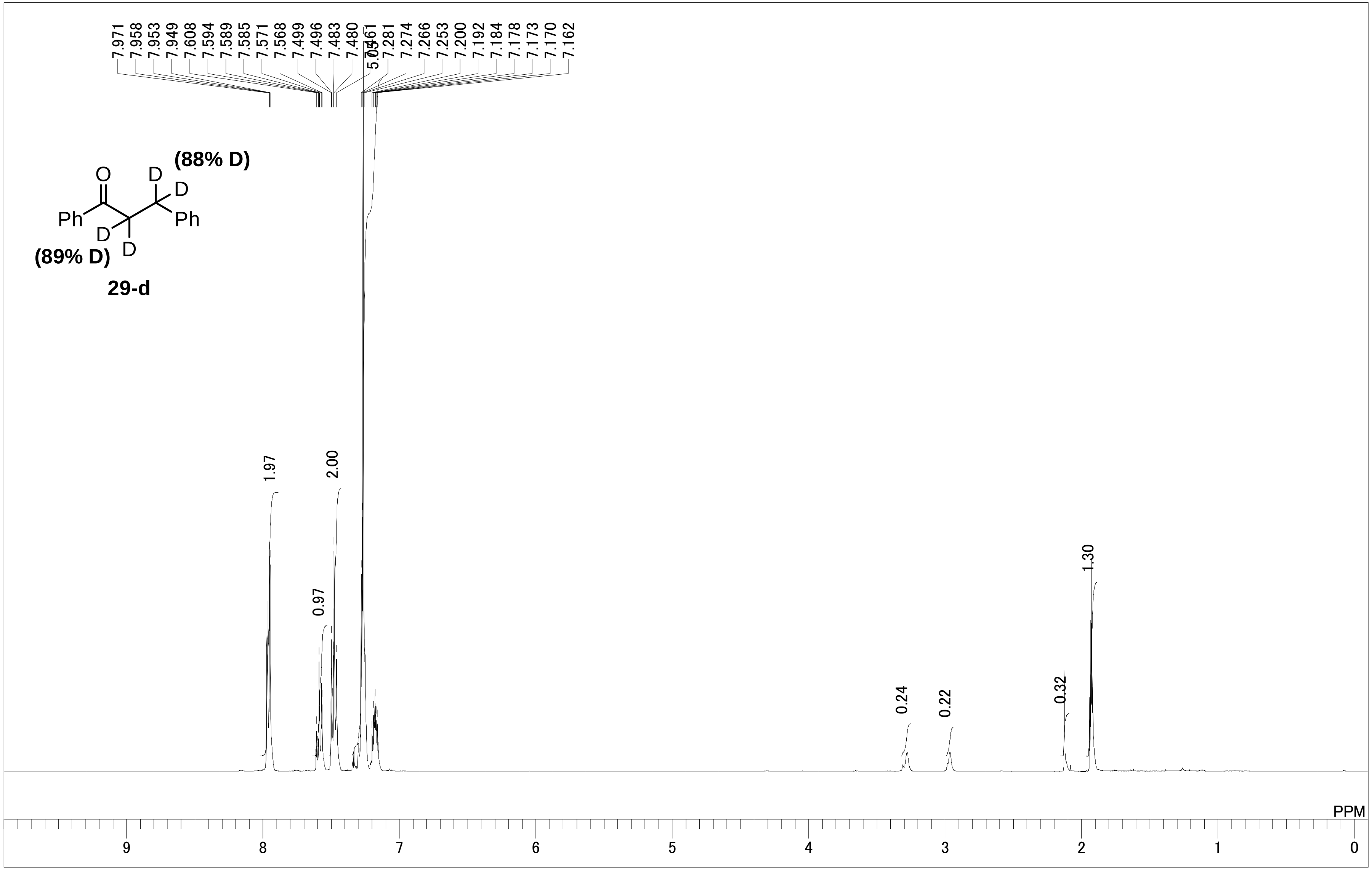


28-d

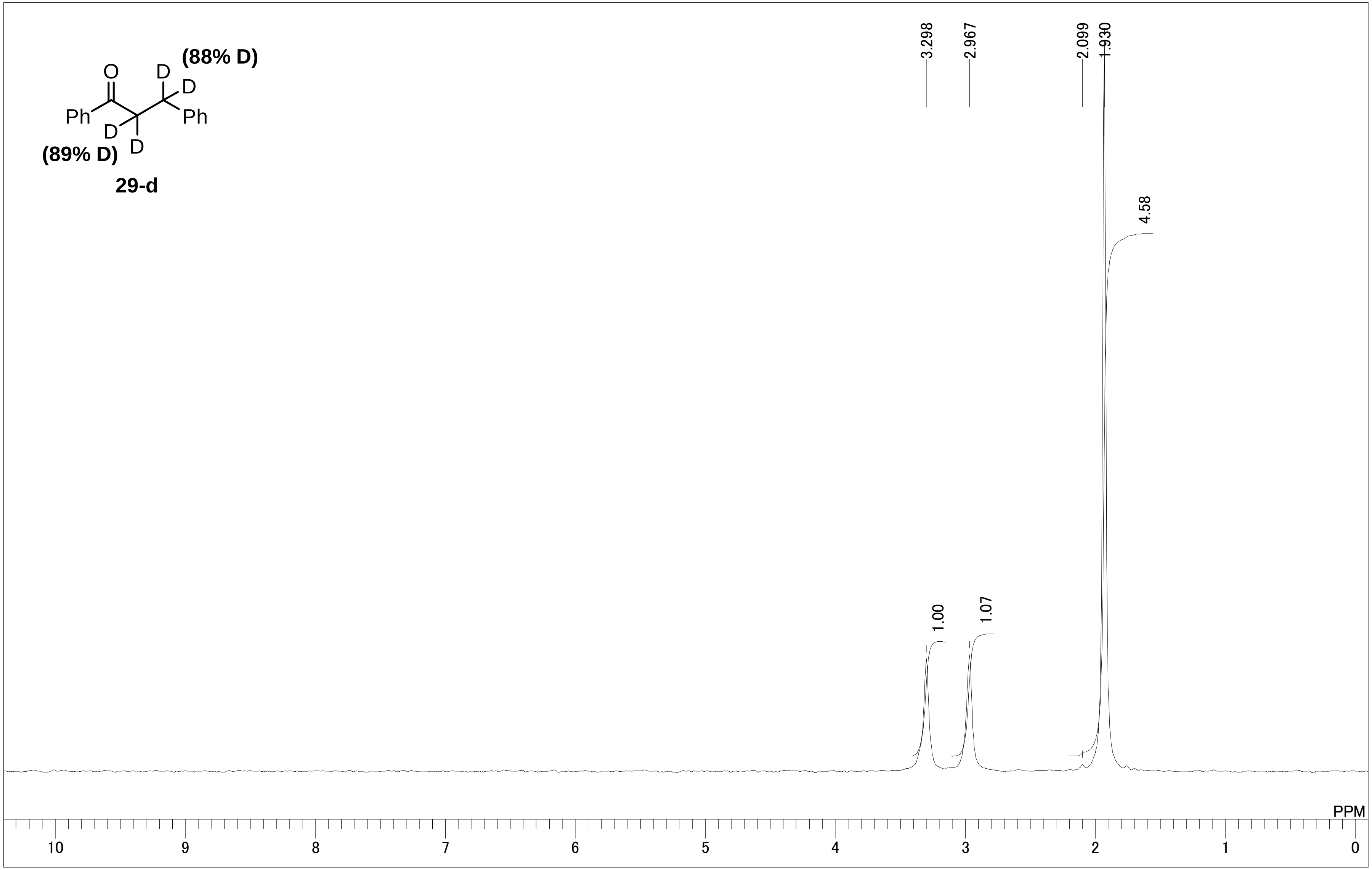
(* Indicate as the average D content)



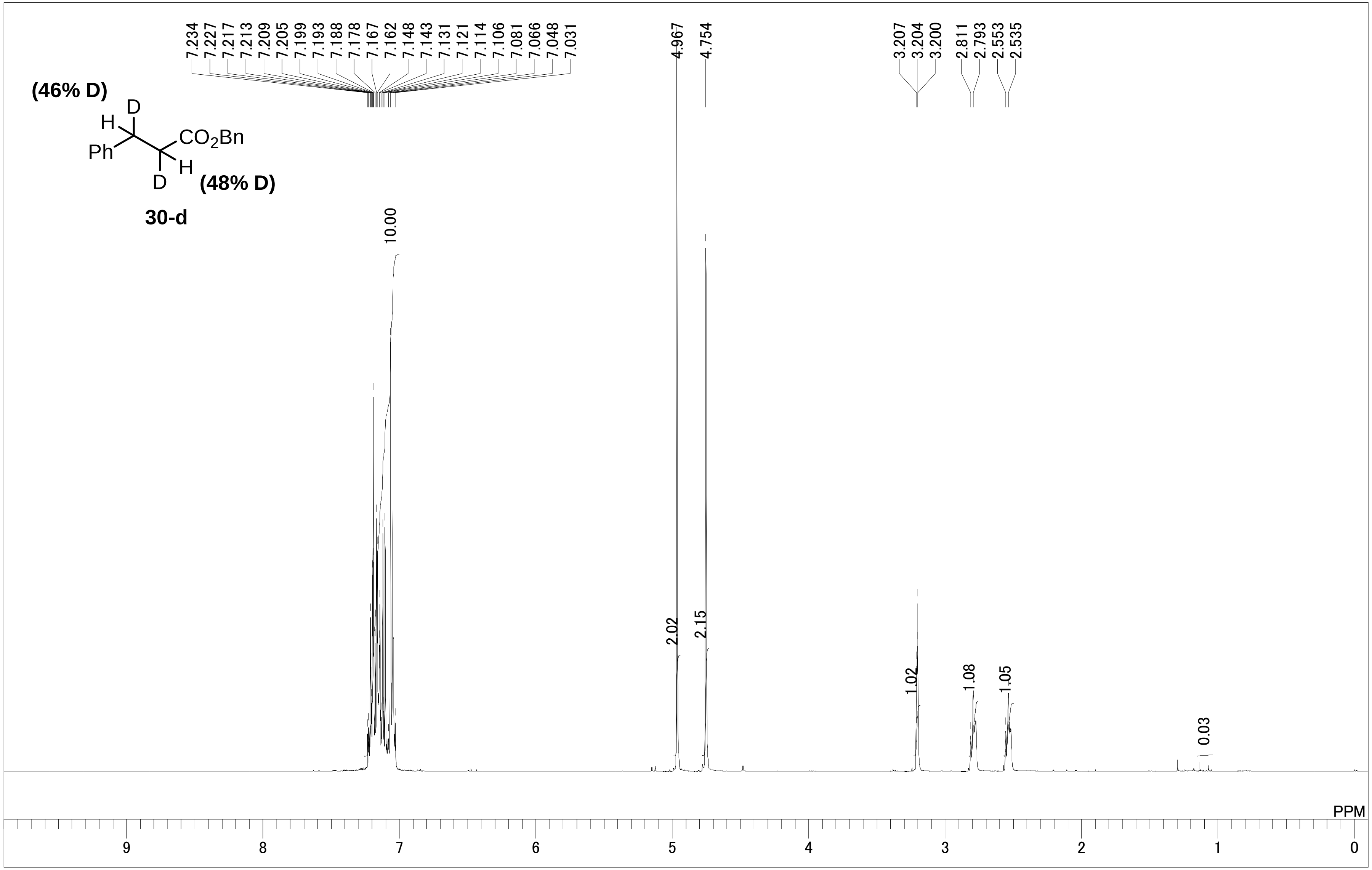
F:¥NMR 2007-5月¥TK7-114-11NON_E2_FT.als
TK7-114-1



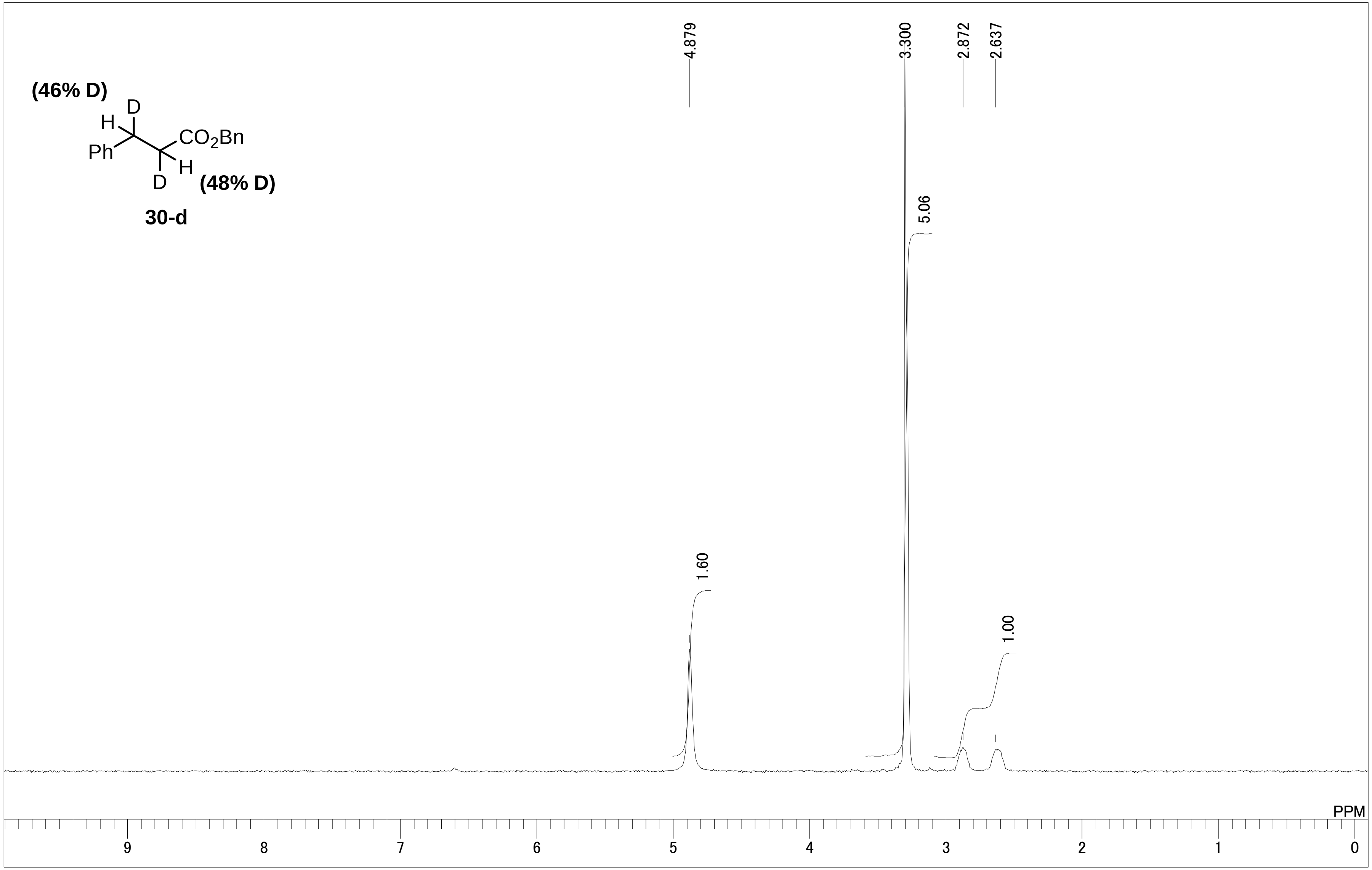
F:¥NMR 2007-5月¥2H NMR 5-31¥TK7-114-1-CH3CN-2H NMR-FT.als
TK7-114-1-CH3CN-2H NMR



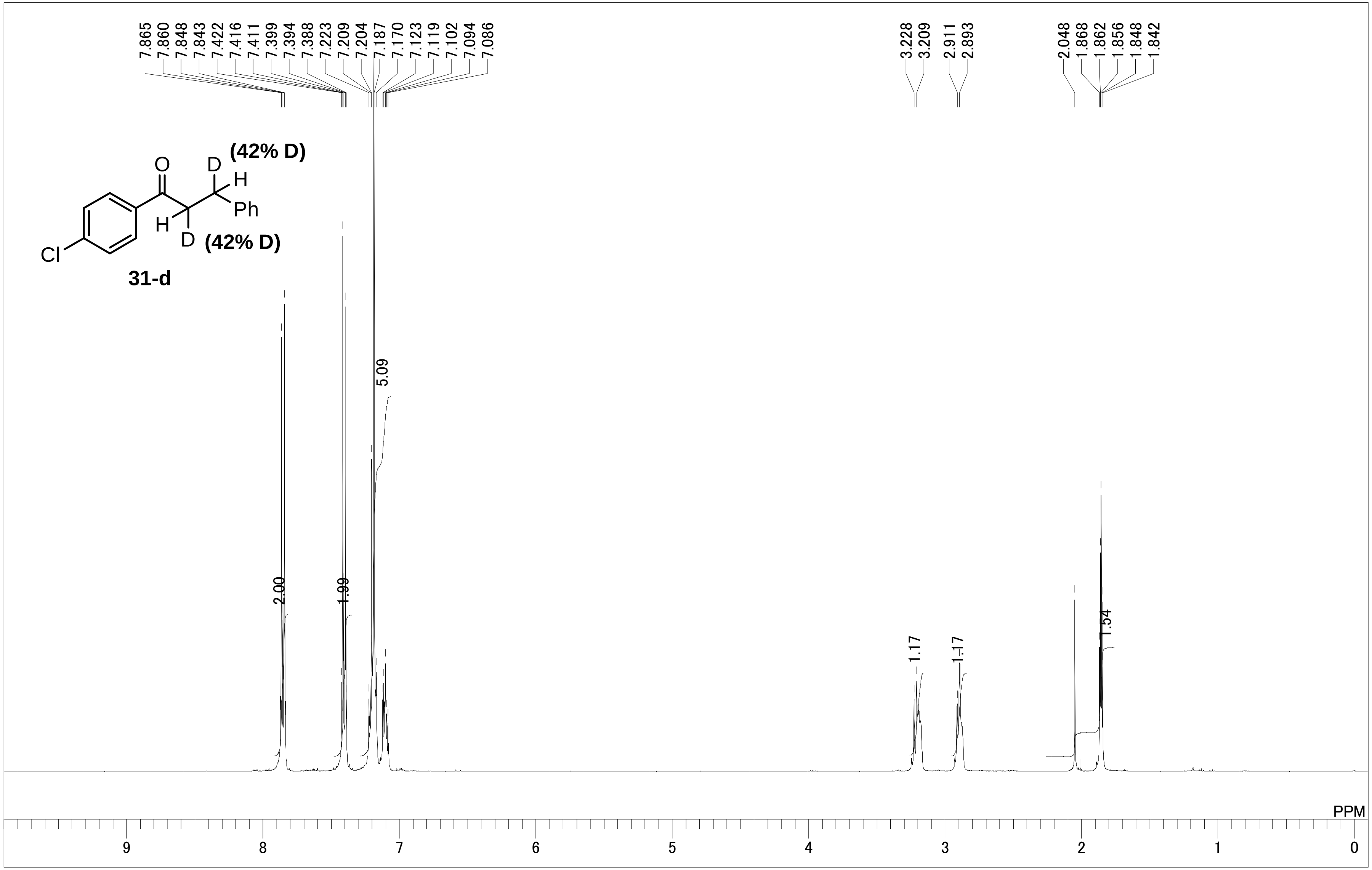
F:¥NMR 2007-6月¥TK7-151-21NON_E7_FT.als
TK7-151-2



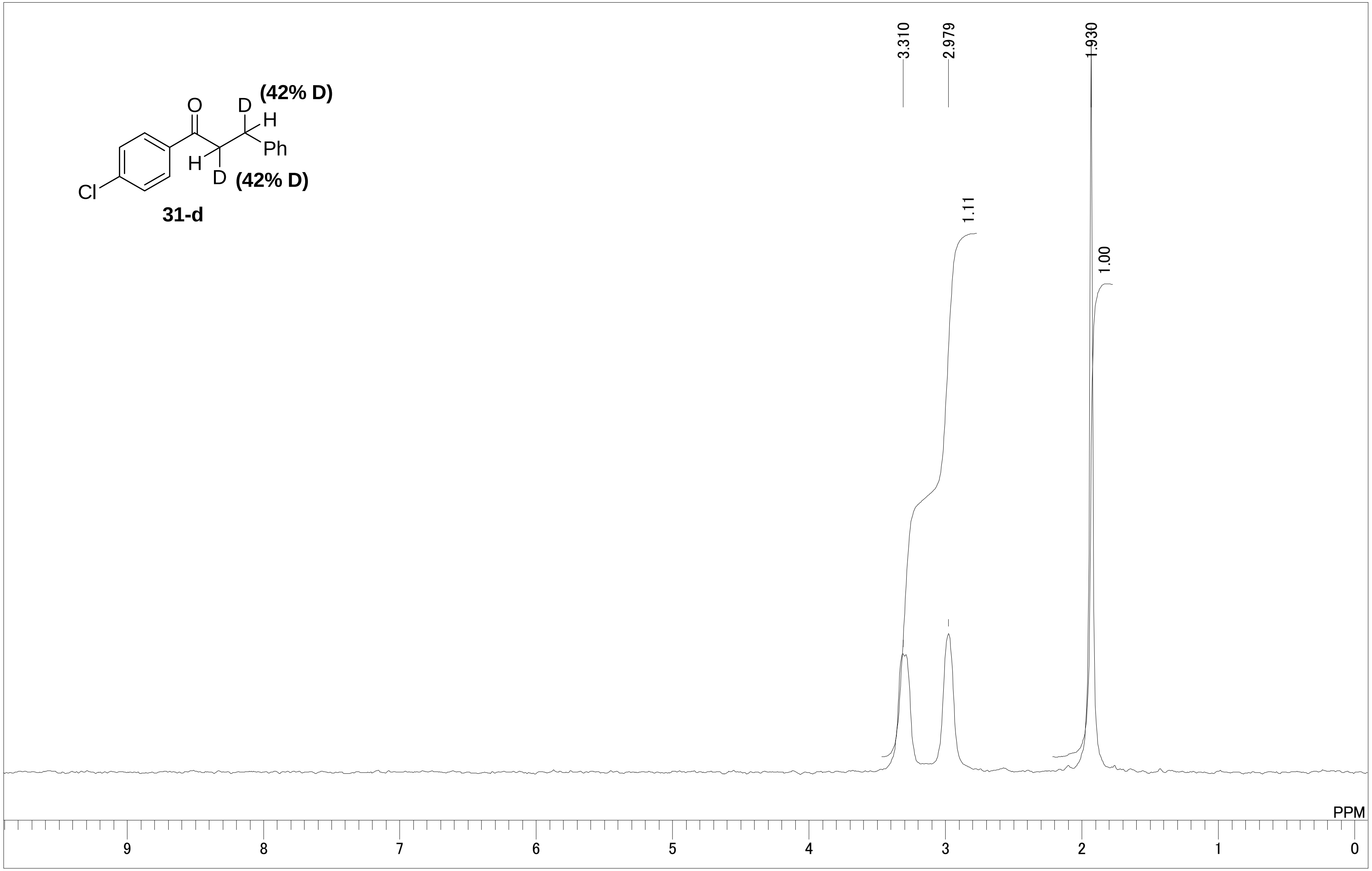
F:¥NMR 2007-6月¥2H NMR 6-10¥TK7-151-2-CH3OH-2H NMR-FT.als
TK7-151-2-CH3OH-2H NMR



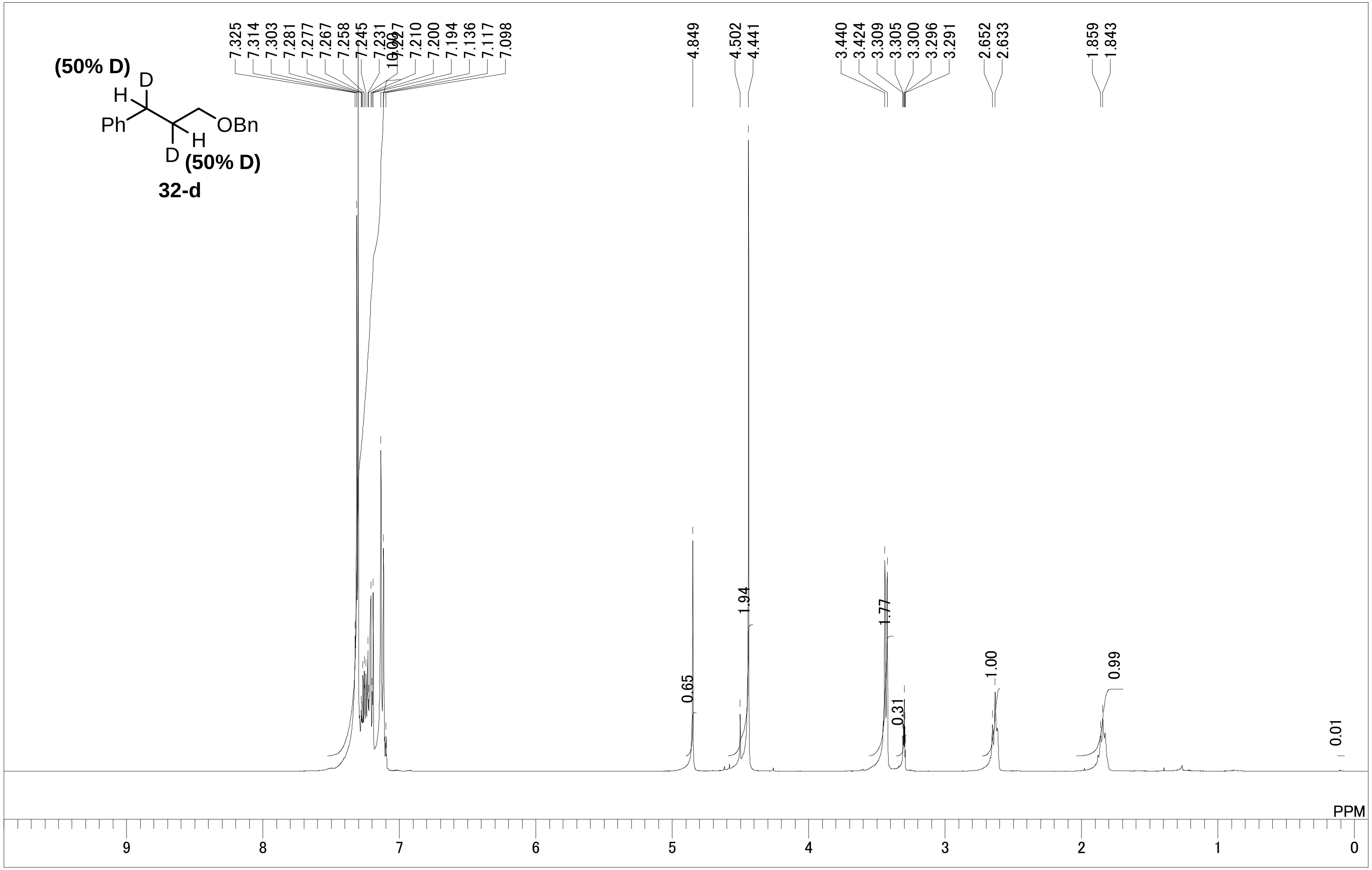
F:¥NMR 2007-5月¥TK7-1101NON_E1_FT.als
TK7-110



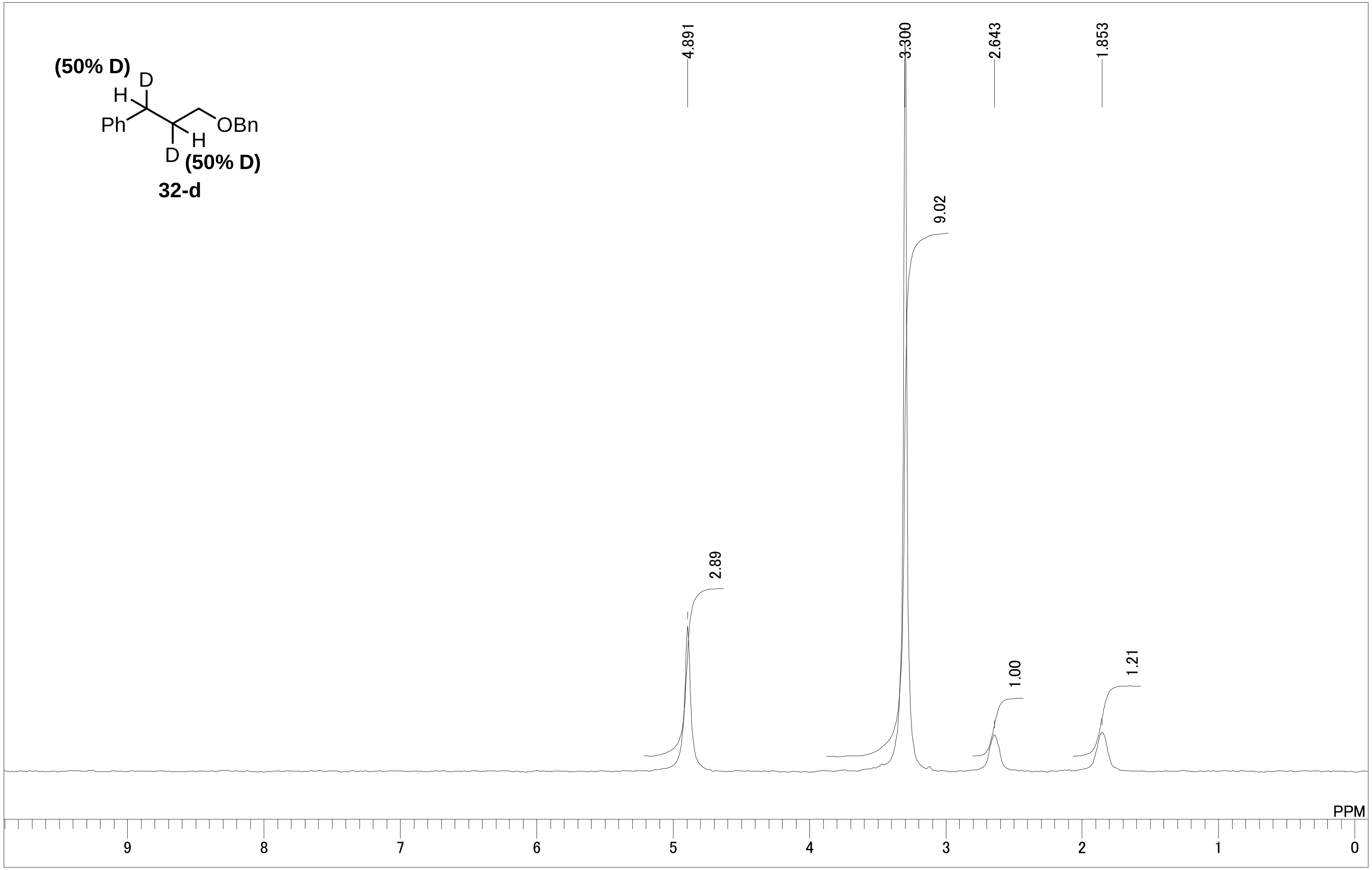
F:¥NMR 2007-5月¥2H NMR 5-31¥TK7-110-CH3CN-2H NMR-FT.als
TK7-110-CH3CN-2H NMR



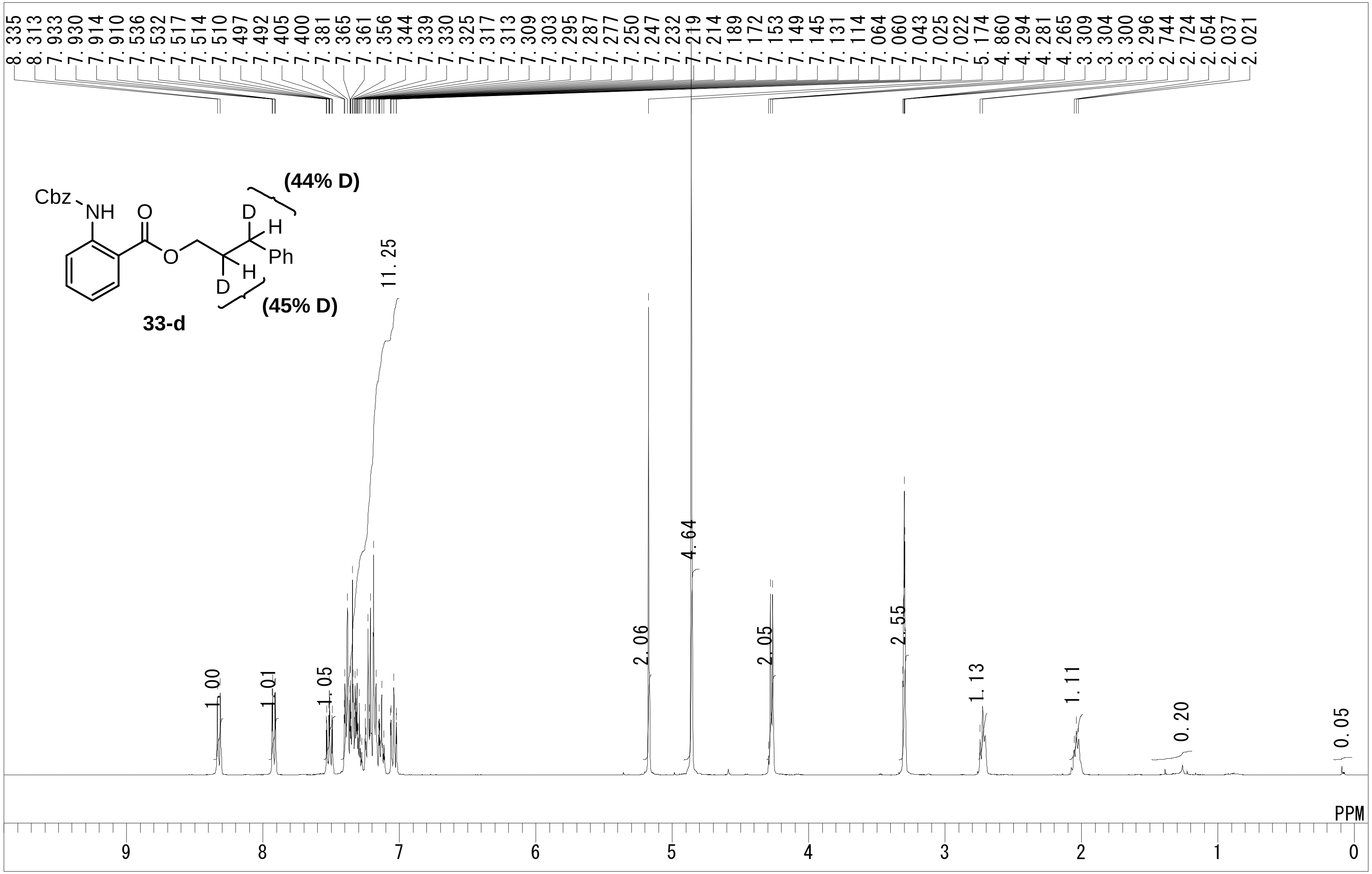
D:¥Data¥水本¥TMR NMR Data (重水素ガス論文用)¥TMR491NON_E5-FT.als
TMR49



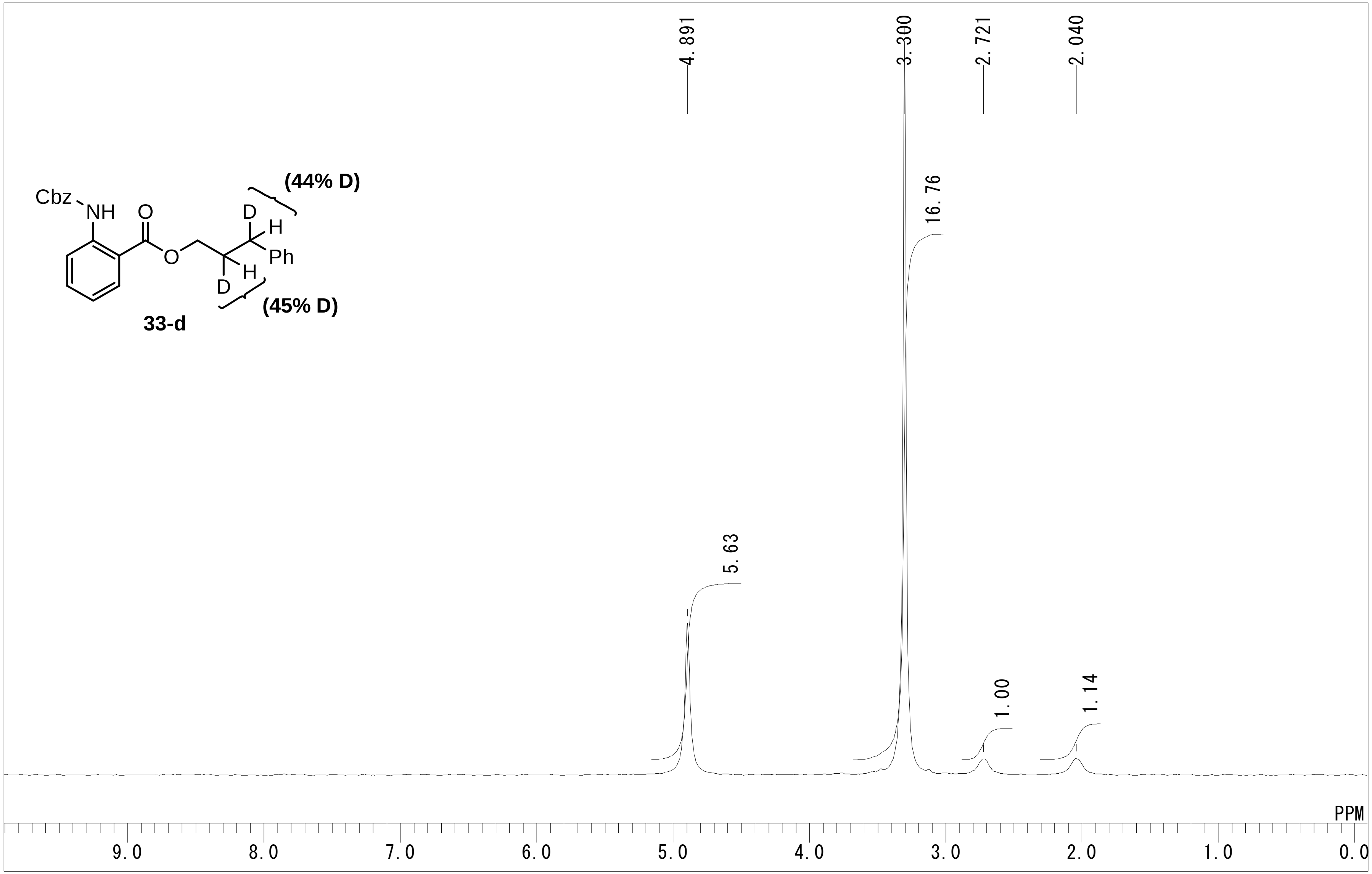
D:¥Data¥水本¥TMR NMR Data (重水素ガス論文用)¥TMR49-2H NMR-CH3OH-FT.als
TMR49-2H NMR-CH3OH



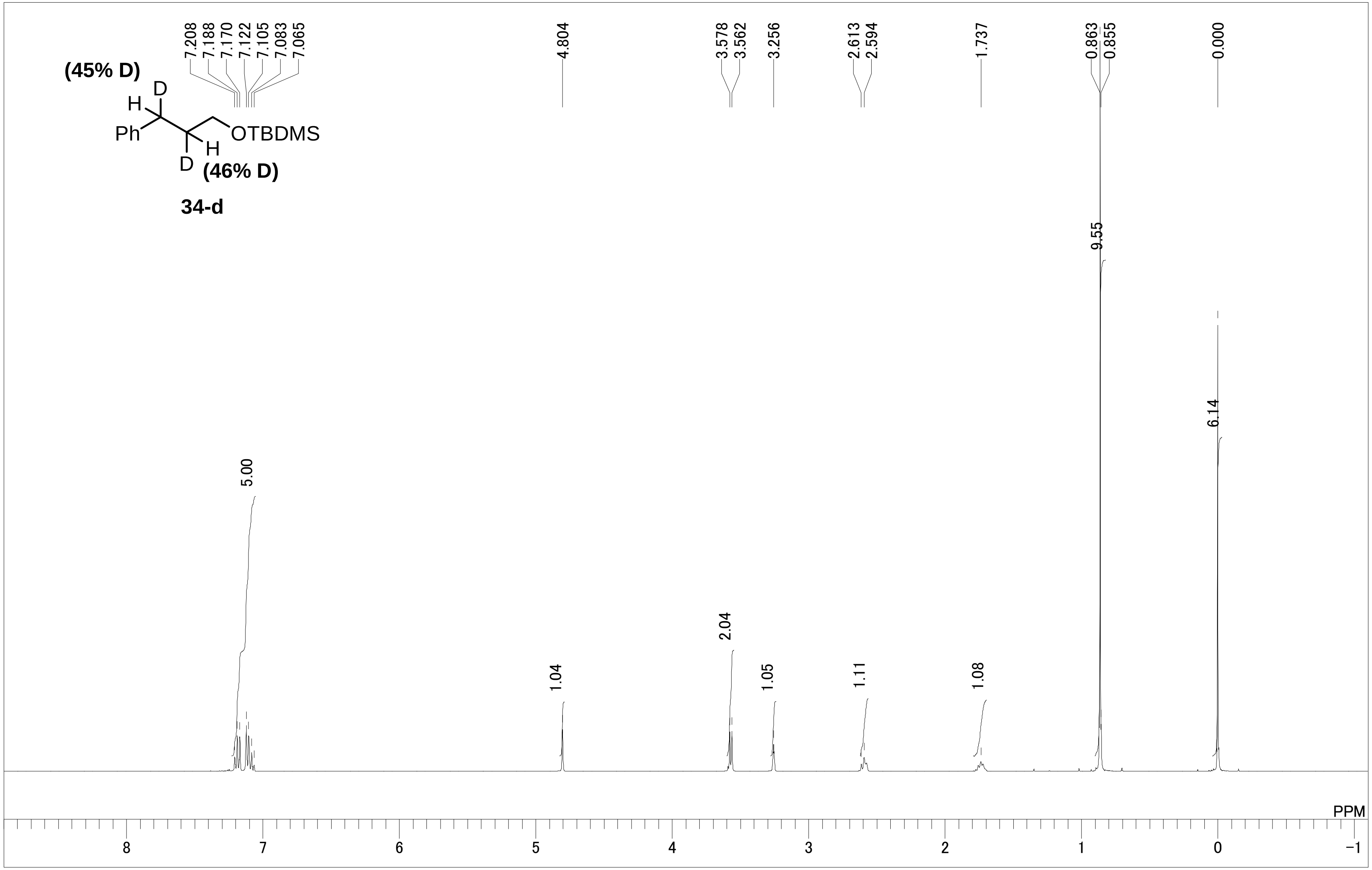
D:¥Data¥NMR 2007年¥NMR 2007-1月¥TK6-157-31NON_E4_FT.als
TK6-157-3



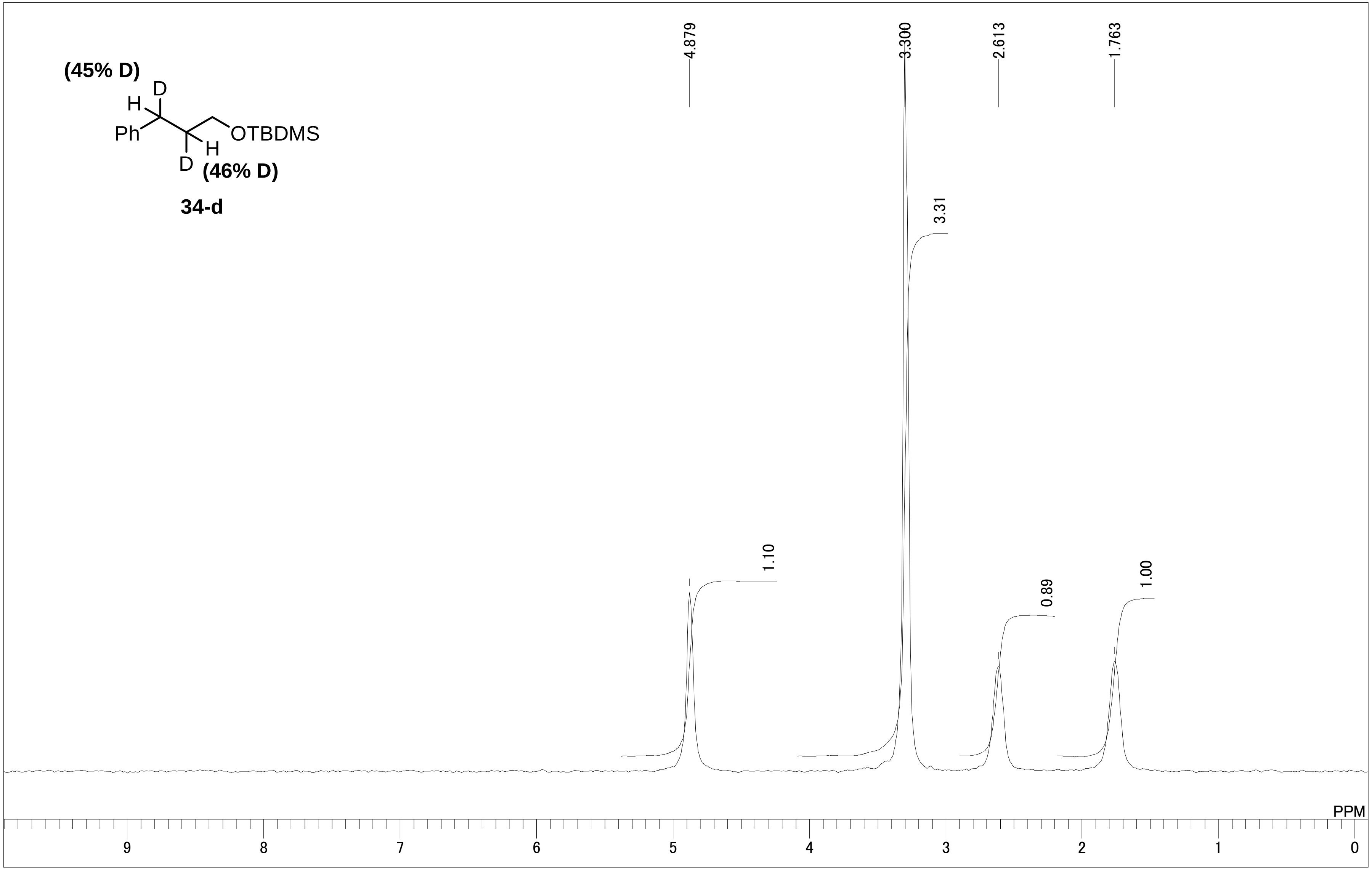
D:¥Data¥NMR 2007年¥NMR 2007-2月¥2-6-2H NMR¥TK6-157-3-CH3OH-2H NMR-FT.als
TK6-157-3-CH3OH-2H NMR



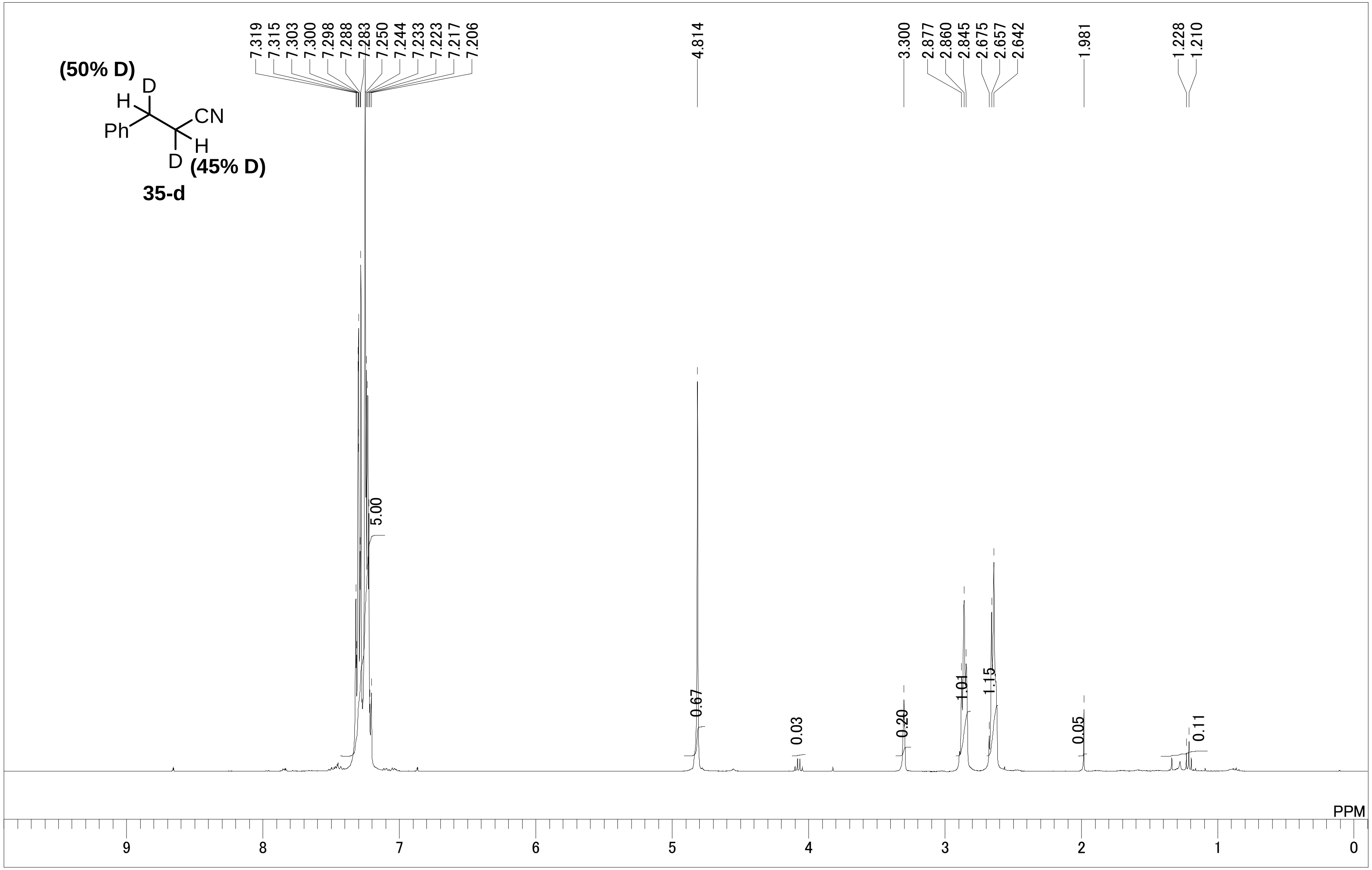
F:¥NMR 2007-5月¥TK7-1051NON_E3_FT.als
TK7-105



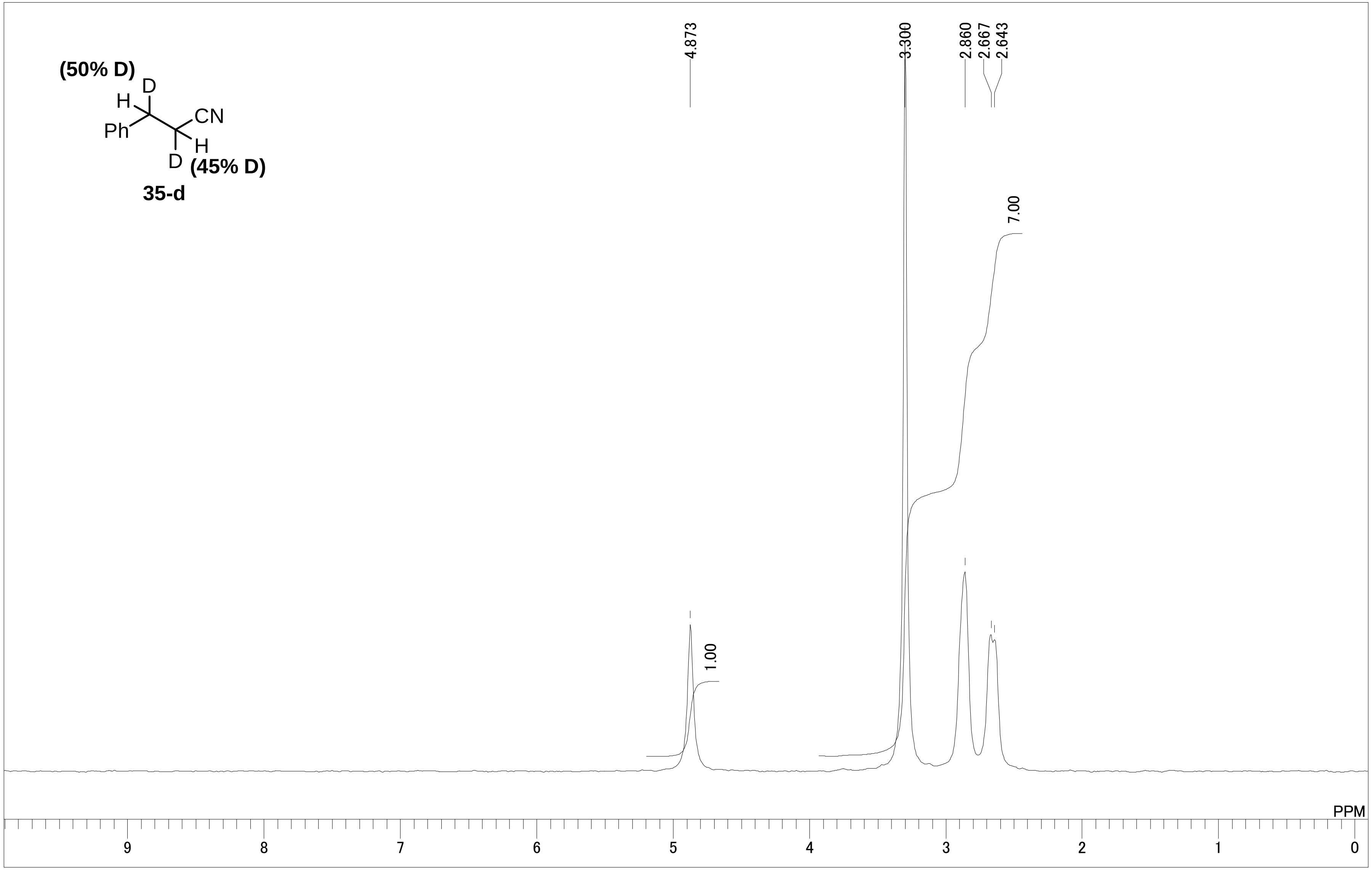
F:¥2H NMR 5-28¥TK7-105-CH3OH-2H NMR-FT.als
TK7-105-CH3OH-2H NMR



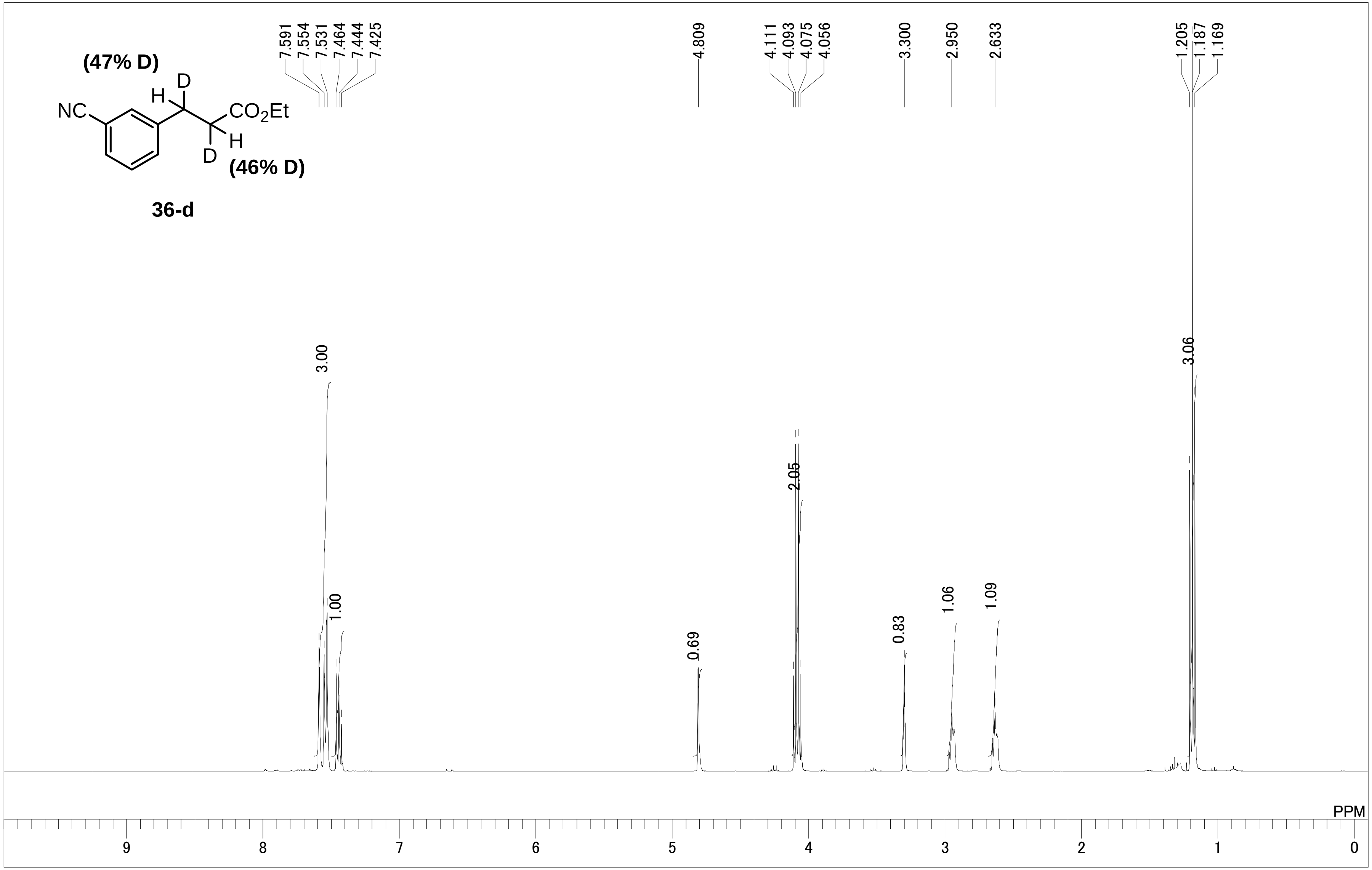
D:¥Data¥水本¥TMR NMR Data (重水素ガス論文用)¥TMR731NON_E9-FT.als
TMR73



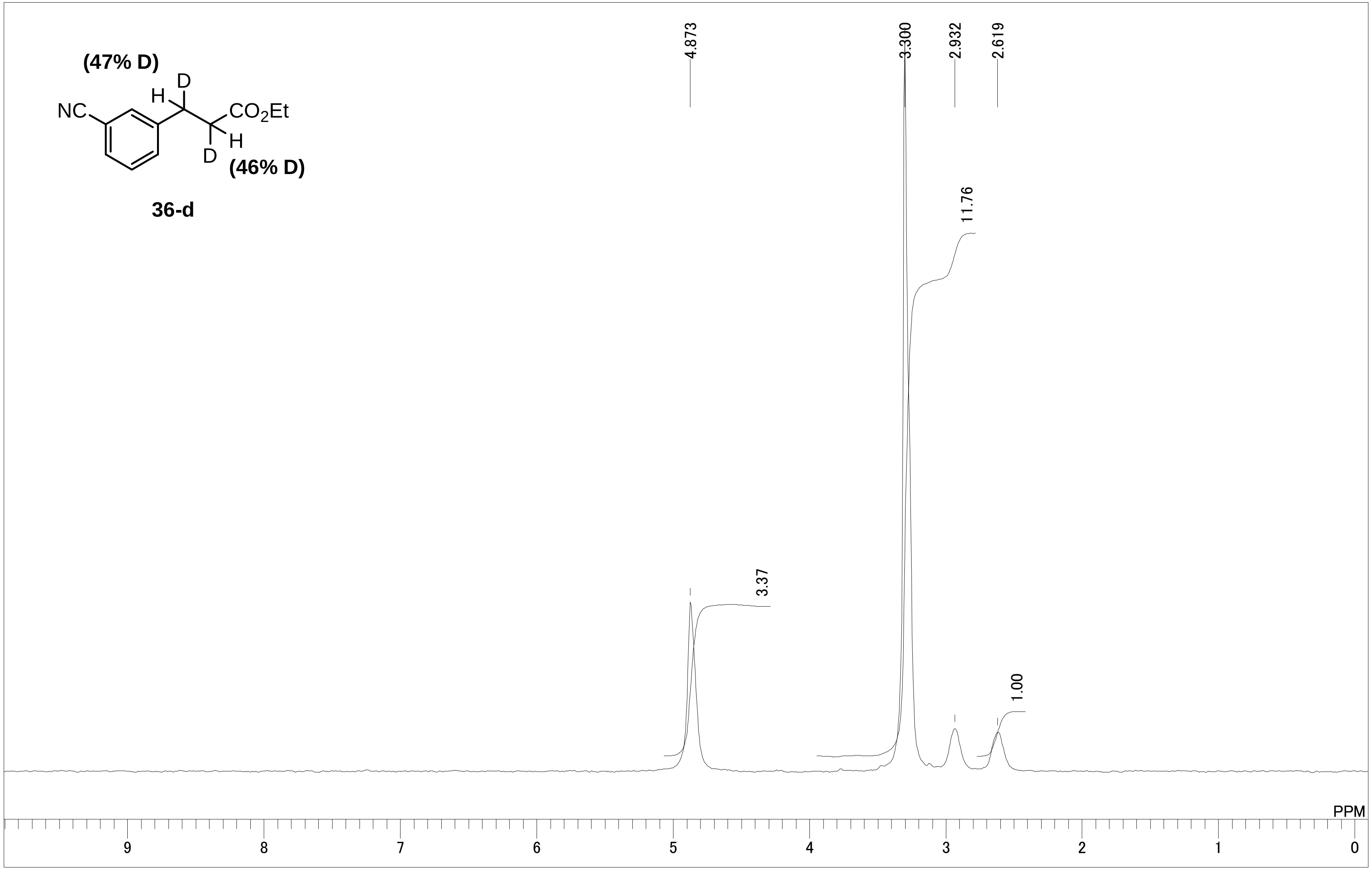
D:¥Data¥水本¥TMR NMR Data (重水素ガス論文用)¥TMR73-2H NMR-CH3OH-FT.als
TMR73-2H NMR-CH3OH



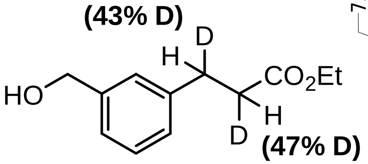
F:¥NMR 2007-5月¥TK7-1111NON_E1_FT.als
TK7-111



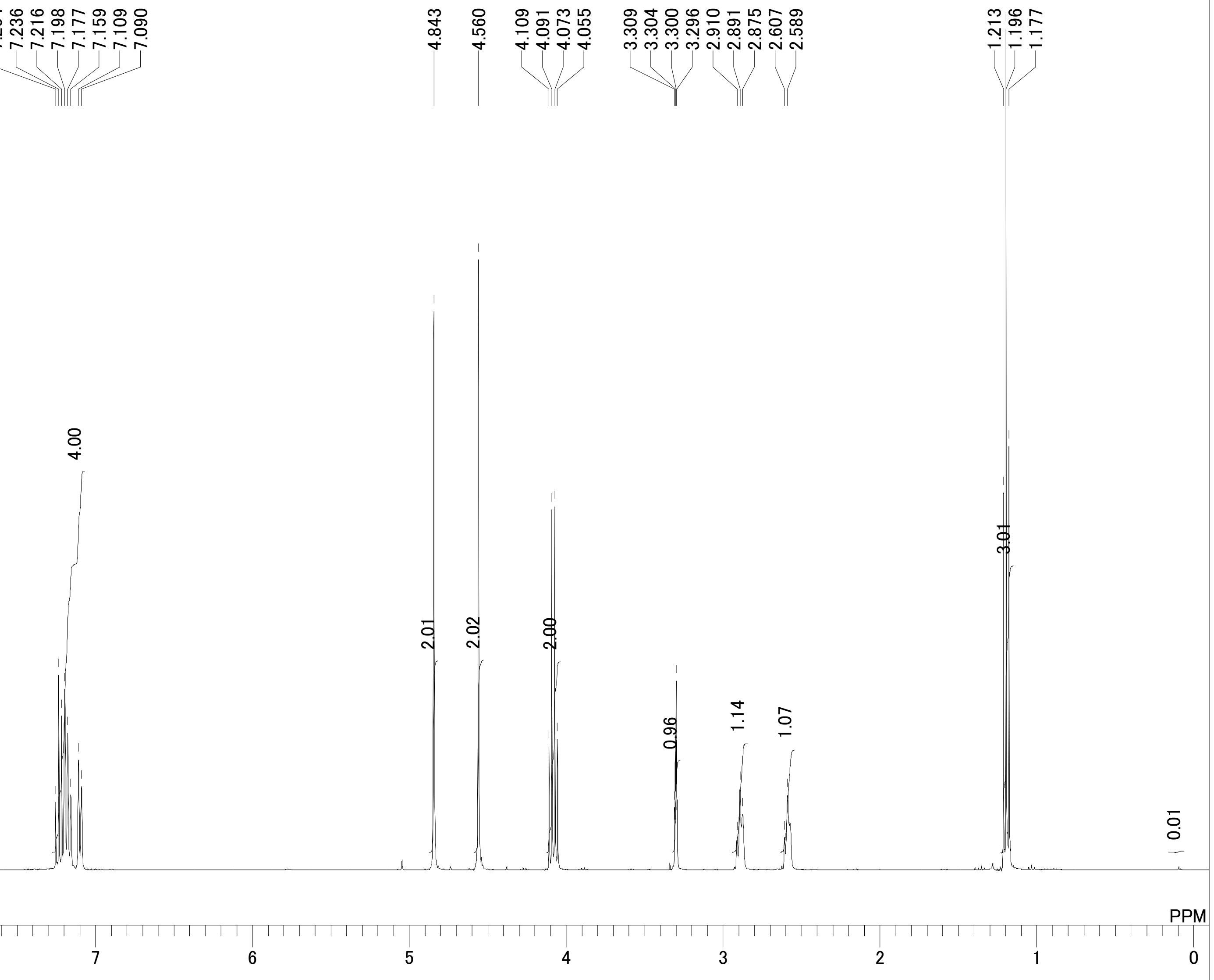
F:¥2H NMR 5-28¥TK7-111-CH3OH-2H NMR-FT.als
TK7-111-CH3OH-2H NMR



F:¥NMR 2007-5月¥TK7-1201NON_E3_FT.als
TK7-120



37-d



F:¥2H NMR 5-28¥TK7-120-CH3OH-2H NMR-FT.als
TK7-120-CH3OH-2H NMR