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Direct enantioseparation of axially chiral 1,1'-biaryl-2,2'-diols using amidine-based resolving agents

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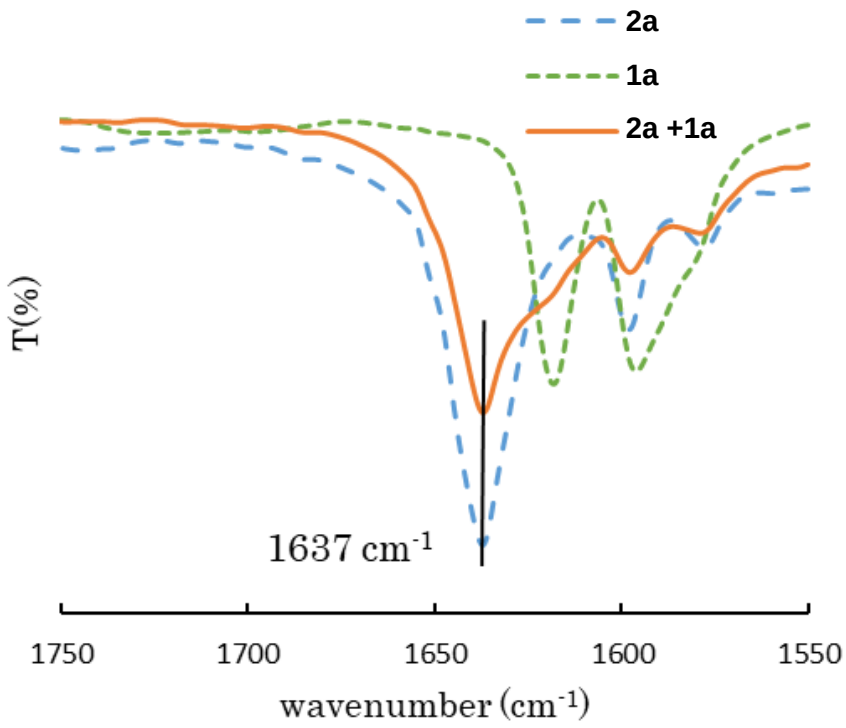
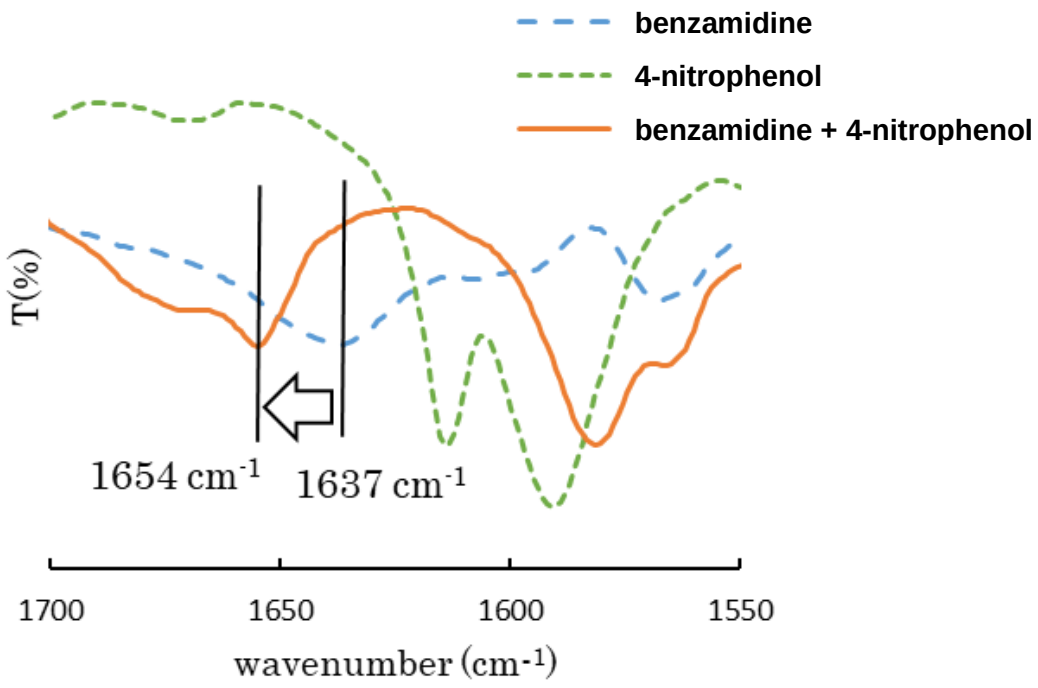


Figure S1. IR spectra of the chiral amidine (**2a**), phenol (**1a**) and their equimolar mixture.



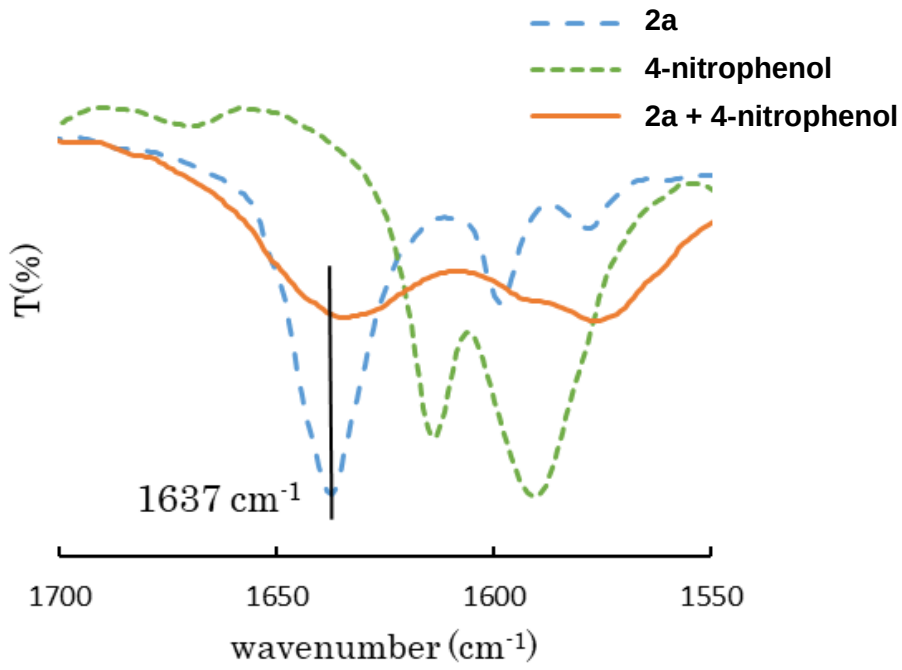


Figure S2. IR spectra of a) benzamidine, 4-nitrophenol and their equimolar mixture and b) chiral amidine (**2a**), 4-nitrophenol and their equimolar mixture.

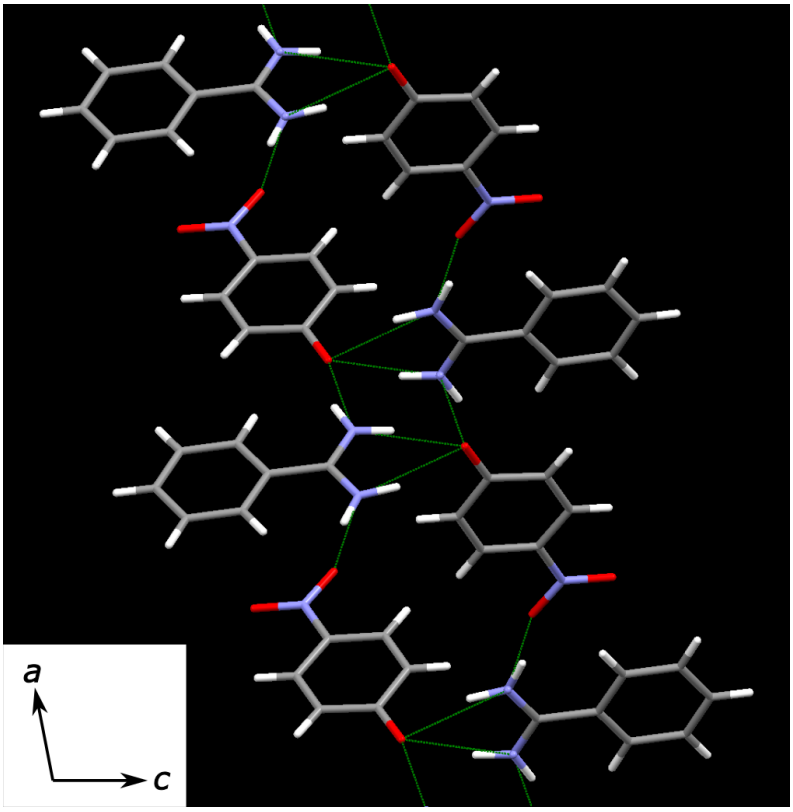


Figure S3. Crystal structure of the salt of benzamidine and 4-nitrophenol viewed from the *b* axis. Oxygen and nitrogen atoms are represented by red and blue. The dotted lines show hydrogen bonds.

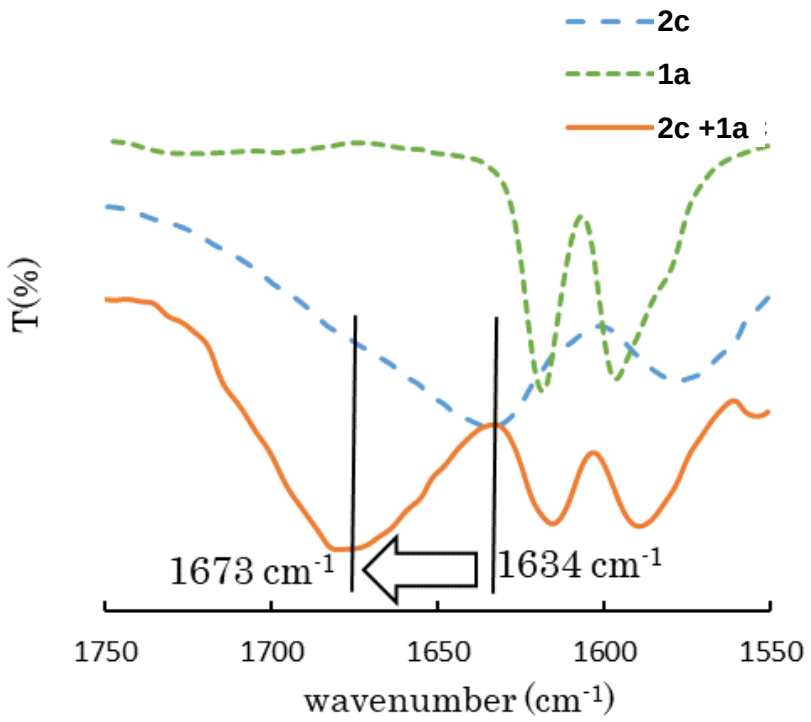


Figure S4. IR spectra of the chiral amidine (**2c**), 1,1'-binaphthyl-2,2'-diol (**1a**) and their equimolar mixture.

Table S1. Summary of crystallographic data reported in this study.

	2a ▪ 2(*S*)-1a	**benzamidine ▪ 4-nitrophenol**	**2c ▪ (*R*)-1a**
empirical formula	$C_{63}H_{52}N_2O_4$	$C_{13}H_{13}N_3O_3$	$C_{40}H_{44}N_2O_2$
formula weight	901.06	259.26	584.77
temperature (K)	150	150	150
crystal size (mm)	0.13 × 0.12 × 0.04	0.23 × 0.07 × 0.03	0.21 × 0.16 × 0.08
crystal system	orthorhombic	monoclinic	orthorhombic
space group	$P2_12_12_1$	$P2_1$	$P2_12_12_1$
a (Å)	10.0606(15)	9.4244(13)	10.6751(15)
b (Å)	20.736(3)	5.0509(7)	12.6375(17)
c (Å)	22.810(3)	13.2982(18)	23.647(3)
α (°)	90	90	90
β (°)	90	100.928(2)	90
γ (°)	90	90	90
V (Å^3)	4758.6(12)	621.54(15)	3190.1(8)
Z	4	2	4
Dc (g/cm^3)	1.258	1.385	1.218
μ ($Mo_{K\alpha}$) (mm^{-1})	0.078	0.101	0.074
$\theta_{min/max}$ (°)	1.327/24.997	1.560/24.987	1.722/27.478
*R*1 [$F_0 > 2\sigma(F_0)$]	0.0419	0.0518	0.0416
*wR*2 (all F_0^2)	0.0754	0.1366	0.0901
GOF	0.794	1.037	0.950
measured reflns	22966	2944	18184
independent reflns	8364	1955	7201
observed reflns	5316	1891	5929
reflns used	8364	1955	7201
parameters	644	188	421
CCDC number	2057487	2057488	2057489

Experimental details

General and Materials

All the ^{1}H and ^{13}C NMR spectra were measured using 300, 400, or 500 MHz spectrometers. IR spectra were reported in reciprocal centimeters. Melting points are uncorrected. Optical rotation values were measured with a polarimeter. All commercially available reagents and solvents were purchased and used as received unless noted. Dry THF was freshly distilled from sodium under a nitrogen atmosphere. Dry CH_2Cl_2 and dry CCl_4 were distilled after drying over $CaCl_2$ and stored with Molecular Sieves 4A under a nitrogen atmosphere. Dry triethylamine was distilled after drying over KOH and stored with KOH under a nitrogen atmosphere. Dry toluene was distilled from sodium under a nitrogen atmosphere and stored with sodium under a nitrogen atmosphere. Dry EtOH was distilled from sodium under a nitrogen atmosphere and stored with Molecular Sieves 4A under a nitrogen atmosphere. The enantiomeric excess of the compounds was determined by chiral HPLC analysis (Daicel Chiralcel OD-3 column 4.6 × 250 mm or Chiralpak AS-3 column 2.1 × 250 mm) with UV detection at 254 nm.

Synthesis and characterization

(*S*)-*N*-(1-phenylethyl)benzamide (4).[1] To a vigorously stirred mixture of (*S*)-1-phenylethylamine (**3**) (2.81 g, 23.2 mmol) and NaOH (1.29 g, 32.2 mmol) in H_2O (25 mL) was added dropwise benzoylchloride (3.62 g, 25.8 mmol) over 10 min at 0 °C. After the suspension was stirred for 2 h at room temperature, the white precipitate was filtered, washed several times with H_2O and then dried in vacuo. The desired product **4** (4.24 g, 18.8 mmol, 81%) was obtained as a white solid. Mp: 118.5-120.3 °C. $[\alpha]_D^{17}$ = +7.2 ° (c 0.251, MeOH). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.84-7.70 (m, 2H), 7.58-7.22 (m, 8H), 6.42-6.18 (br, 1H), 5.44-5.26 (m, 1H), 1.62 (d, *J* = 6.9 Hz, 3H). IR (KBr): ν (cm^{-1}) 3451, 3331, 1634, 1523, 1490, 1319, 758, 700.

(*S*,*S*)-*N*,*N*'-bis(1-phenylethyl)benzamidine (2a).[1] A solution of **4** (0.903 g, 4.01 mmol) and 2,6-lutidine (0.647 g, 6.04 mmol) in dry CH_2Cl_2 (10 mL) was cooled to 0 °C. Oxalyl chloride (0.543 g, 4.28 mmol) diluted with dry CH_2Cl_2 (5 mL) was slowly added to the solution over 30 min. Stirring was continued at 0 °C for 30 min, and the solution was allowed to warm to room temperature and stirred for 30 min. **3** (0.490 g, 4.04 mmol) diluted with dry CH_2Cl_2 (5 mL) was slowly added to the solution over 30 min at room temperature. The reaction mixture was refluxed for 24 h, and then concentrated under reduced pressure. The residue was dissolved in AcOEt (20 mL) and extracted with 1 N HCl aq. (10 mL × 15). The aqueous phase was basified with 6 N NaOH aq. and extracted with $CHCl_3$ (5 mL × 5). The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was suspended in hexane, and the resulting solid

was collected by filtration. The crude product (0.530 g) was recrystallized from EtOH (0.8 mL) and the desired product **2a** (0.441 g, 1.34 mmol, 33%) was obtained as colorless crystals. Mp: 124.3-125.0 °C. $[\alpha]_D^{24}$ = −48.5 ° (c 1.00, $CHCl_3$). ^{1}H NMR (300 MHz, DMSO-d_6): δ (ppm) 7.52-6.72 (m, 15H), 6.70-6.56 (m, 1H), 5.26-5.02 (m, 1H), 4.12-3.96 (m, 1H), 1.49 (d, *J* = 7.2 Hz, 3H), 1.17 (d, *J* = 6.3 Hz, 3H). IR (KBr): ν (cm^{-1}) 3060, 2955, 2877, 1637, 1599, 1494, 1484, 1451, 1361, 1349, 1309, 1268, 1142, 1090, 766, 699.

(*S*)-2-(6-methoxy-2-naphthyl)propionamide (6).[2] Oxalyl chloride (3.0 mL) and DMF (3 drops) were added to (*S*)-2-(6-methoxy-2-naphthyl)propanoic acid (**5**) (2.32 g, 10.1 mmol) under a nitrogen atmosphere at 0 °C, and the resulting solution was refluxed for 2 h. After the excess of oxalyl chloride was distilled off, dry toluene (20 mL) and 28% NH_3 aq. (4 mL) were added at 0 °C. After the resulting suspension was stirred at room temperature for 1 h, the white precipitate formed was filtered, washed several times with H_2O and then dried in vacuo. The desired product **6** (2.21 g, 9.63 mmol, 96%, >99% ee) was obtained as a white solid, which was used for next step without further purification. Mp: 177.0-179.0 °C. $[\alpha]_D^{22}$ = +33.3 ° (c 0.195, MeOH). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.80-7.66 (m, 3H), 7.44-7.34 (m, 1H), 7.20-7.08 (m, 2H), 3.92 (s, 3H), 3.74 (q, *J* = 7.2 Hz, 1H), 1.61 (d, *J* = 7.2 Hz, 3H). IR (KBr): ν (cm^{-1}) 3348, 3195, 2983, 2898, 1660, 1606, 1505, 1486, 1461, 1403, 1309, 1267, 1228, 1217, 1173, 1114, 1027, 927, 894, 854, 814. HPLC analysis (Daicel Chiralcel OD-3, hexane/2-propanol=80:20, 1.0 mL/min, 254 nm UV detector; t_r(*S*) = 10.3 min, t_r(*R*) = 16.4 min).

(*S*)-2-(6-methoxy-2-naphthyl)propionitrile (7).[3] To a stirred solution of **6** (0.300 g, 1.31 mmol) in dry CH_2Cl_2 (15 mL) were added PPh_3 (0.420 g, 1.60 mmol), dry triethylamine (0.161 g, 1.59 mmol) and dry CCl_4 (0.246 g, 1.60 mmol) under a nitrogen atmosphere. The solution was refluxed for 20 h. After the reaction was quenched with H_2O, the organic layer was separated, washed with 1 N HCl aq., dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product (0.875 g) was purified by silica gel column chromatography (eluent: $CHCl_3$). The desired product **7** (0.244 g, 1.15 mmol, 88%, >99% ee) was obtained as a pale yellow solid. Mp: 98.7-100.0 °C. $[\alpha]_D^{17}$ = −28.9 ° (c 1.00, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.84-7.68 (m, 3H), 7.46-7.34 (m, 1H), 7.22-7.08 (m, 2H), 4.04 (q, *J* = 7.2 Hz, 1H), 3.93 (s, 3H), 1.72 (d, *J* = 7.2 Hz, 3H). IR (KBr): ν (cm^{-1}) 3065, 3020, 2992, 2963, 2942, 2905, 2840, 2240, 1916, 1777, 1712, 1632, 1604, 1506, 1483, 1448, 1419, 1393, 1376, 1356, 1260, 1214, 1188, 1165, 1085, 1024, 960, 927, 891, 856. HPLC analysis (Daicel Chiralpak AS-3, hexane/2-propanol=99.5:0.5, 0.3 mL/min, 254 nm UV detector; t_r(*S*) = 34.6 min, t_r(*R*) = 41.2 min).

(*S*)-*N*-hydroxy-2-(6-methoxy-2-naphthyl)propionamidine (8). To a stirred solution of **7** (1.14 g, 5.40 mmol) in dry EtOH (3 mL) and dry DMF (3 mL) were added H_2NOH•HCl (1.13 g, 16.3 mmol) and dry triethylamine (1.65 g, 16.3 mmol), and then the solution was refluxed under a nitrogen

atmosphere for 10 h. After cooling to room temperature, the solution was concentrated under reduced pressure. The residue was dissolved in AcOEt (40 mL) and washed with H_2O / *sat*. NaCl aq. = 1/1. The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product (0.893 g) was purified by silica gel column chromatography (eluent: $CHCl_3$/MeOH = 30/1, v/v). The desired product **8** (0.272 g, 1.11 mmol, 21%, 51% ee) was obtained as a white solid. Mp: 142.0-143.5 ℃ (21% ee). $[\alpha]_D^{18}$ = −10.5 ° (c 0.506, MeOH) (21% ee). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.80-7.64 (m, 3H), 7.46-7.36 (m, 1H), 7.20-7.04 (m, 2H), 4.50-4.26 (br, 2H), 3.92 (s, 3H), 3.76 (q, *J* = 7.2 Hz, 1H), 1.58 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 157.7, 156.6, 136.9, 133.8, 129.2, 128.9, 127.5, 126.2, 125.6, 119.1, 105.7, 55.3, 41.8, 18.0. IR (KBr): ν (cm^{-1}) 3479, 3372, 3268, 3055, 2976, 2936,1663, 1637, 1607, 1586, 1505, 1486, 1460, 1449, 1418, 1390, 1266, 1231, 1216, 1192, 1174, 1160, 1124, 1078, 1026, 921, 889, 849. HPLC analysis (Daicel Chiralcel OD-3, hexane/2-propanol=80:20, 1.0 mL/min, 254 nm UV detector; t_r(*S*) = 10.5 min, t_r(*R*) = 13.1 min).

(*S*)-*N*-acetoxy-2-(6-methoxy-2-naphthyl)propionamidine (9). To a stirred solution of **8** (0.267 g, 1.10 mmol) in THF (5 mL) were added pyridine (0.111 g, 1.40 mmol) and acetic anhydride (0.135 g, 1.32 mmol) at 0 ℃, and then the solution was stirred at room temperature for 1 h. After the solvent was distilled off, the residue was dissolved in $CHCl_3$ (50 mL), and washed with 1 N HCl aq., *sat*. $NaHCO_3$ aq. and *sat*. NaCl aq. The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The desired product **9** (0.301 g, 1.05 mmol, 96%, 51% ee) was obtained as a white solid, which was used for next step without further purification. Mp: 105.0-108.0 ℃. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.80-7.68 (m, 3H), 7.50-7.38 (m, 1H), 7.22-7.08 (m, 2H), 4.66-4.42 (br, 2H), 4.04-3.84 (m, 4H), 2.19 (s, 3H), 1.67 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 168.8, 160.3, 157.9, 135.4, 133.9, 129.2, 128.9, 127.6, 126.2, 125.5, 119.3, 105.7, 55.4, 41.3, 19.9, 17.7. IR (KBr): ν (cm^{-1}) 3449, 3332, 3196, 3060, 2962, 2936, 2841, 1744, 1627, 1505, 1486, 1464, 1439, 1418, 1392, 1371, 1266, 1227, 1173, 1119, 1027, 1010, 957, 927, 885, 855, 814. HPLC analysis (Daicel Chiralcel OD-3, hexane/2-propanol=80:20, 1.0 mL/min, 254 nm UV detector; t_r(*S*) = 13.8 min, t_r(*R*) = 30.7 min).

2-(6-methoxy-2-naphthyl)propionamidine (2b). A suspension of **9** (0.720 g, 2.51 mmol) and 10% Pd-C (0.173 g) in EtOH (30 mL) was stirred under a hydrogen atmosphere at room temperature for 2 h. The solid was filtered off and the filtrate was concentrated under reduced pressure. The residue (0.752 g) was recrystallized from MeOH (8.5 mL) to give the acetate salt of the product as a solid. The separated filtrate was concentrated under reduced pressure and the residue was recrystallized from EtOH / H_2O (4 mL / 0.5 mL). The combined solid was dissolved in $CHCl_3$ (30 mL) and 1 N NaOH aq. (20 mL) was added. The aqueous layer was separated and extracted with $CHCl_3$ (15 mL × 5). The combined organic layer was washed with *sat*. NaCl aq., dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The desired product **2b** (0.597 g, 2.07 mmol, 83%,

racemic) was obtained as a white solid. Mp: 125.0-129.0 °C. $[\alpha]_D^{26}$ = 0 ° (c 1.00, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.80-7.62 (m, 3H), 7.40-7.30 (m, 1H), 7.22-7.04 (m, 2H), 3.92 (s, 3H), 3.73 (q, *J* = 7.2 Hz, 1H), 1.58 (d, *J* = 7.2 Hz, 3H). IR (KBr): ν (cm^{-1}) 3321, 3163, 2966, 1684, 1635, 1606, 1505, 1485, 1455, 1436, 1392, 1264, 1216, 1163, 1030, 927, 891, 853.

dehydroabietyl amide (11).[4] To a stirred solution of dehydroabietic acid (**10**) (2.01 g, 6.09 mmol) in dry CH_2Cl_2 (3 mL) were added DMF (3 drops) and oxalyl chloride (1.5 mL) under a nitrogen atmosphere at 0 °C, and the solution was refluxed for 2 h. After the volatile components were distilled off, dry toluene (10 mL) and 28% NH_3 aq. (3 mL) were added at 0 °C. The resulting suspension was stirred at room temperature for 2 h, and then was added to AcOEt (100 mL) and H_2O (50 mL). The organic layer was separated and washed with *sat*. $NaHCO_3$ aq. and *sat*. NaCl aq. The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The desired product **11** (1.95 g, 6.50 mmol, 97%) was obtained as a pale yellow solid, which was used for next step without further purification. Mp: 153.0-156.0 °C. $[\alpha]_D^{25}$ = +41.1 ° (c 1.00, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.22-7.12 (m, 1H), 7.04-6.96 (m, 1H), 6.90-6.82 (m, 1H), 5.90-5.60 (br, 1H), 5.50-5.20 (br, 1H), 2.96-2.85 (m, 2H), 2.85-2.73 (m, 1H), 2.39-2.25 (m, 1H), 2.16-2.04 (m, 1H), 1.90-1.40 (m, 7H), 1.29 (s, 3H), 1.26-1.14 (m, 9H). IR (KBr): ν (cm^{-1}) 3428, 3328, 2927, 2867, 1629, 1575, 1498, 1456, 1383, 1362, 1085, 1036, 905, 883, 822.

dehydroabietyl cyanide (12).[5] To a stirred solution of **11** (5.49 g, 18.3 mmol) in dry CH_2Cl_2 (100 mL) were added PPh_3 (7.20 g, 27.4 mmol), dry triethylamine (2.78 g, 27.4 mmol) and dry CCl_4 (4.23 g, 27.5 mmol) under a nitrogen atmosphere. The solution was refluxed for 17 h. After the reaction was quenched with H_2O, the organic layer was separated, washed with 1 N HCl aq., dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product (14.8 g) was purified by silica gel column chromatography (eluent: hexane/$CHCl_3$ = 1/1, v/v). The desired product **12** (4.77 g, 16.9 mmol, 93%) was obtained as a white solid. Mp: 77.0-80.0 °C. $[\alpha]_D^{26}$ = +37.0 ° (c 1.00, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.20-7.10 (m, 1H), 7.06-6.92 (m, 1H), 6.92-6.86 (m, 1H), 3.10-2.92 (m, 2H), 2.92-2.70 (m, 1H), 2.40-2.24 (m, 1H), 2.14-1.66 (m, 8H), 1.42 (s, 3H), 1.22 (d, *J* = 6.9 Hz, 6H), 1.18 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 146.2, 145.4, 134.2, 127.0, 126.6, 124.2, 124.0, 46.8, 37.5, 37.4, 37.3, 37.2, 33.5, 29.8, 25.2, 23.9, 21.7, 18.9, 17.7. IR (KBr): ν (cm^{-1}) 3010, 2930, 2930, 2863, 2225, 1612, 1496, 1458, 1419, 1383, 889, 819. MS (MALDI-TOF) *m/z* calcd for $C_{20}H_{27}N+Na^+$: 304.204 $[M+Na]^+$; found: 304.245.

***N*-hydroxy-dehydroabietyl amidine (13).** To a stirred solution of **12** (4.76 g, 16.9 mmol) in dry EtOH (35 mL) were added H_2NOH•HCl (5.89 g, 84.8 mmol) and dry triethylamine (8.58 g, 84.8 mmol), and then the solution was refluxed under a nitrogen atmosphere for 28 h. After cooling to room temperature, the solution was concentrated under reduced pressure. The residue was dissolved in AcOEt (100 mL) and washed with H_2O and *sat*. NaCl aq. The organic layer was dried over

anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product (5.76 g) was purified by silica gel column chromatography (eluent: hexane/AcOEt = 2/1, v/v). The desired product **13** (1.66 g, 5.29 mmol, 31%) was obtained as a white solid. Mp: 89.7-91.7 °C. $[\alpha]_D^{24}$ = +86.2 ° (c 1.00, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.22-7.12 (m, 1H), 7.04-6.94 (m, 1H), 6.94-6.84 (m, 1H), 4.80-4.48 (br, 2H), 2.98-2.74 (m, 3H), 2.45-2.28 (m, 1H), 2.00-1.56 (m, 8H), 1.34-1.18 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 160.0, 147.2, 145.9, 134.8, 127.0, 124.0, 123.9, 46.3, 42.8, 38.3, 37.5, 37.4, 33.5, 30.0, 25.5, 24.0, 20.1, 18.7, 16.0. IR (KBr): ν (cm^{-1}) 3500, 3399, 3255, 2930, 1651, 1575, 1497, 1457, 1382, 1362, 1230, 1197, 1173, 1140, 1074, 923, 822. MS (MALDI-TOF) *m/z* calcd for $C_{20}H_{30}N_2O+H^+$: 315.244 $[M+H]^+$; found: 315.242.

***N*-acetoxy-dehydroabietyl amidine (14).** To a stirred solution of **13** (1.64 g, 5.21 mmol) in THF (44 mL) were added pyridine (0.503 g, 6.36 mmol) and acetic anhydride (0.638 g, 6.25 mmol) at 0 °C, and then the solution was stirred at room temperature for 1 h. After the solvent was distilled off, the residue was dissolved in $CHCl_3$ (50 mL), and washed with 1 N HCl aq., *sat*. $NaHCO_3$ aq. and *sat*. NaCl aq. The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The desired product **14** (1.69 g, 4.73 mmol, 91%) was obtained as a white solid, which was used for next step without further purification. Mp: 125.8-127.8 °C. $[\alpha]_D^{24}$ = +66.6 ° (c 1.00, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.24-7.12 (m, 1H), 7.06-6.98 (m, 1H), 6.92-6.86 (m, 1H), 4.86-4.64 (br, 2H), 2.98-2.74 (m, 3H), 2.44-2.28 (m, 1H), 2.20 (s, 3H), 1.98-1.62 (m, 8H), 1.31 (s, 3H), 1.26 (s, 3H), 1.22 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 169.7, 163.5, 147.0, 145.9, 134.6, 127.0, 124.0, 124.0, 46.4, 43.5, 38.1, 37.9, 37.4, 33.5, 30.0, 25.5, 24.0, 20.4, 20.2, 18.6, 16.0. IR (KBr): ν (cm^{-1}) 3501, 3378, 2957, 2869, 1743, 1626, 1582, 1497, 1459, 1384, 1364, 1231, 1008, 939, 882, 822. MS (MALDI-TOF) *m/z* calcd for $C_{22}H_{32}N_2O_2+Na^+$: 379.236 $[M+Na]^+$; found: 379.217.

dehydroabietyl amidine (2c). A suspension of **14** (1.68 g, 4.72 mmol) and 10% Pd-C (0.614 g) in EtOH (60 mL) was stirred under a hydrogen atmosphere at room temperature for 1 day. Pd-C was filtered off and the filtrate was concentrated under reduced pressure. The residue was suspended in hexane, and the resulting solid was collected by filtration. The residue (1.44 g) was dissolved in $CHCl_3$ (30 mL) and *sat*. $NaHCO_3$ aq. (70 mL) was added. The aqueous layer was extracted with $CHCl_3$ (15 mL × 3). The combined organic layer was washed with *sat*. NaCl aq., dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The desired product **2c** (1.22 g, 4.09 mmol, 87%) was obtained as a white solid. Mp: 76.7-79.7 °C. $[\alpha]_D^{25}$ = +54.7 ° (c 1.00, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.24-7.12 (m, 1H), 7.08-6.96 (m, 1H), 6.94-6.84 (m, 1H), 3.00-2.76 (m, 3H), 2.44-2.30 (m, 1H), 1.92-1.38 (m, 8H), 1.27, (s, 3H), 1.25 (s, 3H), 1.22 (d, *J* = 7.2, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 174.8, 147.0, 146.0, 134.5, 127.0, 124.1, 124.0, 46.8, 45.6, 38.2, 38.1, 37.3, 33.5, 29.9, 25.3, 24.0, 20.5, 19.0, 16.8. IR (KBr): ν (cm^{-1}) 3345, 2958, 2869, 1634, 1577, 1497, 1459, 1382, 1363, 1201, 1171, 822. MS (MALDI-TOF) *m/z* calcd for

$C_{20}H_{30}N_2+H^+$: 299.249 $[M+H]^+$; found: 299.241.

(*rac*)-6,6'-dibromo-1,1'-bi-2-naphthol (1b).[6] *Rac*-1,1'-bi-2-naphthol (**1a**) (1.00 g, 3.49 mmol) was dissolved in dry CH_2Cl_2 (40 mL) under a nitrogen atmosphere. After the mixture was cooled to -10 °C, bromine (1.51 g, 9.45 mmol) diluted with CH_2Cl_2 (4 mL) was added dropwise over 30 min and the solution was stirred for an additional 2.5 h. After the solution was gradually warmed to room temperature and stirred for another 1 h, the reaction was quenched with *sat*. Na_2SO_3 aq. (25 mL). The aqueous phase was extracted with $CHCl_3$ (15 mL × 3) and the combined organic phase was washed with *sat*. NaCl aq. dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The crude product (1.59 g) was recrystallized from toluene/heptane (7 mL/5 mL) and the desired product **1b** (1.11 g, 2.51 mmol, 72%) was obtained as colorless needles. Mp: 206.0-207.0 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.06 (d, *J* = 1.8 Hz, 2H), 7.90 (d, *J* = 9.0 Hz, 2H), 7.46-7.32 (m, 4H), 6.97 (d, *J* = 9.0 Hz, 2H), 5.00 (s, 2H). IR (KBr): ν (cm^{-1}) 3451, 2952, 1612, 1586, 1502, 1466, 1407, 1382, 1350, 1319, 1268, 1216, 1162, 1145, 1125, 1066, 951, 930, 876, 811.

(*rac*)-6,6'-dimethyl-2,2'-bisphenol (1c).[7] To a solution of 4,6-di-*tert*-butyl-4-methylphenol (1.01 g, 4.60 mmol) in CH_2Cl_2 (9 mL) were added CuCl (45.5 mg, 0.460 mmol) and *N*,*N*,*N*',*N*'-tetramethylethylenediamine (80.1 mg, 0.689 mmol). The suspension was stirred under air at room temperature for 8 h. After addition of H_2O (20 mL) to the reaction mixture, the whole was extracted with $CHCl_3$ (10 mL × 3). The organic phase was washed with *sat*. NaCl aq. and dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product (1.19 g) was purified by silica gel column chromatography (eluent: hexane/$CHCl_3$ = 1/1, v/v) to afford *rac*-3,3',5,5'-tetra-*tert*-butyl-6,6'-dimethyl-2,2'-bisphenol (0.594 g, 1.35 mmol, 59%) as a white solid. Mp: 244.0-245.3 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.39 (s, 2H), 4.80 (s, 2H), 2.00 (s, 6H), 1.42 (s, 18H), 1.40 (s, 18H). IR (KBr): ν (cm^{-1}) 3504, 2991, 2959, 2909, 2871, 1560, 1470, 1414, 1395, 1362, 1332, 1280, 1254, 1233, 1196, 1167, 1116, 1033, 927.

To a solution of (*rac*)-3,3',5,5'-tetra-*tert*-butyl-6,6'-dimethyl-2,2'-bisphenol (0.303 g, 0.691 mmol) in dry toluene (5 mL) was added $AlCl_3$ (39.2 mg, 0.294 mmol) in small portions at 0 °C under a nitrogen atmosphere. The suspension was stirred at 50 °C for 18 h, and $AlCl_3$ (99.3 mg, 0.745 mmol) was added to the suspension at 0 °C. The suspension was stirred at 50 °C for 3 h, and $AlCl_3$ (58.8 mg, 0.441 mmol) was added to the suspension at 0 °C. The suspension was stirred at 50 °C for 3 h, and $AlCl_3$ (58.4 mg, 0.438 mmol) was added to the suspension at 0 °C. After the suspension was stirred at 50 °C for 13 h, the suspension was cooled to 0 °C and carefully quenched by addition of H_2O (14 mL) and 3 N HCl aq. (56 mL). The organic phase was separated and the aqueous phase was extracted with Et_2O (10 mL × 3). The combined organic phase was extracted with 3 N NaOH aq. (10 mL × 3). The aqueous phase was acidified with 6 N HCl aq. and extracted with Et_2O (10 mL × 3). The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced

pressure. The desired product **1c** (0.145 g, 0.677 mmol, 98%) was obtained as a white solid. Mp: 159.7-162.7 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.32-7.18 (m, 2H), 7.00-6.86 (m, 4H), 4.66 (s, 2H), 2.01 (s, 6H). IR (KBr): ν (cm^{-1}) 3464, 3413, 3034, 2973, 2915, 1605, 1575, 1465, 1378, 1335, 1281, 1261, 1180, 1090, 1025, 1006, 947, 883.

(*rac*)-1-[hydroxy(phenyl)methyl]-2-naphthol (1d).[8] To a stirred suspension of Mg turnings (0.304 g, 12.5 mmol) in dry THF (4 mL) under a nitrogen atmosphere was added dropwise a solution of bromobenzene (1.96 g, 12.5 mmol) in dry THF (9 mL) over 1.5 h at room temperature. After formation of the Grignard reagent has started, the suspension was stirred at room temperature for 30 min, and then refluxed for 1 h. After cooling with an ice bath, 2-hydroxy-1-naphthaldehyde (0.861 g, 5.00 mmol), which was dissolved in dry THF (5.5 mL), was added dropwise to the mixture over 30 min and the suspension was stirred for 2 h at room temperature. The reaction was quenched with *sat*. NH_4Cl aq. (5 mL) and H_2O (15 mL) and the whole was extracted with $CHCl_3$ (10 mL × 3). The organic phase was washed with *sat*. NaCl aq., dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (eluent: hexane/AcOEt = 4/1, v/v). The desired product **1d** (1.23 g, 4.91 mmol, 98%) was obtained as a white solid. Mp: 118.5-120.3 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 9.21 (s, 1H), 7.82-7.62 (m, 3H), 7.50-7.12 (m, 8H), 6.82 (d, *J* = 2.6 Hz, 1H), 2.92 (d, *J* = 2.6 Hz, 1H). IR (KBr): ν (cm^{-1}) 3363, 3029, 1625, 1602, 1521, 1469, 1455, 1411, 1326, 1265, 1226, 1154, 1065, 1011, 939, 830.

Enantiomer separation of phenols (**1**) with chiral amidines (**2**)

Equimolar amounts of **2** and *rac*-**1** (0.250 mmol) were dissolved in MeOH or $CHCl_3$, and the solution was concentrated under reduced pressure to give a salt. An appropriate solvent was added to the salt with heating until a homogeneous solution was formed. The solution was gradually cooled to room temperature and left at the temperature for several days to induce crystallization. The salt **1**·**2** was collected by filtration and dried in vacuo at room temperature. The yield was calculated based on a half amount of *rac*-**1** initially used. A part of the salt was dissolved in ethyl acetate, and the organic phase was washed with 1 N HCl aq. to remove **2**. The organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by silica gel preparative TLC to give **2**. The enantiomeric excess of **2** was determined by chiral HPLC analysis. **1a** (Column: Chiralcel OD-3, Eluent: 2-propanol/ hexane = 1/9, Flow rate: 0.5 mL / min, Detection: 254 nm, Retention time: t_r(*S*) = 29.0 min, t_r(*R*) = 30.7 min). **1b** (Column: Chiralcel OD-3, Eluent: 2-propanol/ hexane = 1/9, Flow rate: 1.0 mL / min, Detection: 254 nm, Retention time: $t_r(1^{st})$ = 15.6 min, $t_r(2^{nd})$ = 35.0 min). **1c** (Column: Chiralcel OD-3, Eluent: 2-propanol/ hexane = 1/9, Flow rate: 0.5 mL / min, Detection: 254 nm, Retention time: t_r(*S*) = 17.6 min, t_r(*R*) = 32.1 min).

X-ray crystallographic analysis

Single crystals suitable for X-ray diffraction analysis were prepared by slow evaporation of the saturated solutions of the salt. X-ray crystallographic data were collected on a Bruker Smart APEX II diffractometer with graphite monochromated Mo Kα radiation. Data collections were carried out at 150 K. The structures were solved by a direct method (SIR 2014) and refined by SHELXL-2013 or SHELXL-2018 programs.[9] Crystallographic information files have been deposited with the Cambridge Structural Database.

References

1) ref 12b in the manuscript.

2) refs 23 in the manuscript.

3) refs 24 in the manuscript.

4) ref 25 in the manuscript.

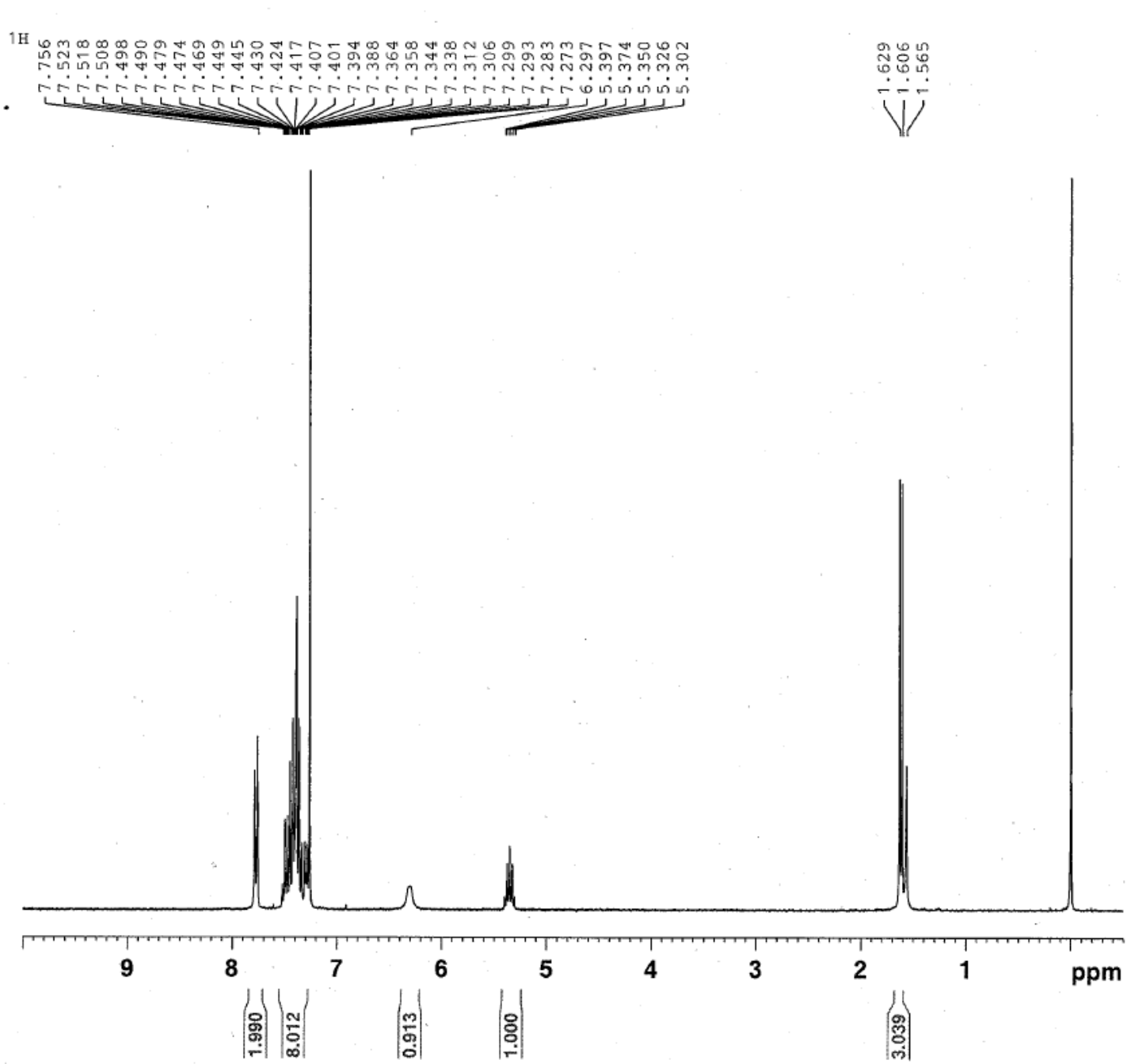
5) ref 26 in the manuscript.

6) ref 27 in the manuscript.

7) refs 28 in the manuscript.

8) ref 29 in the manuscript.

9) ref 30 in the manuscript.



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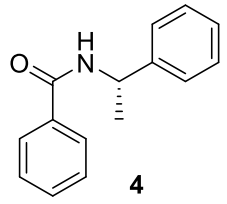

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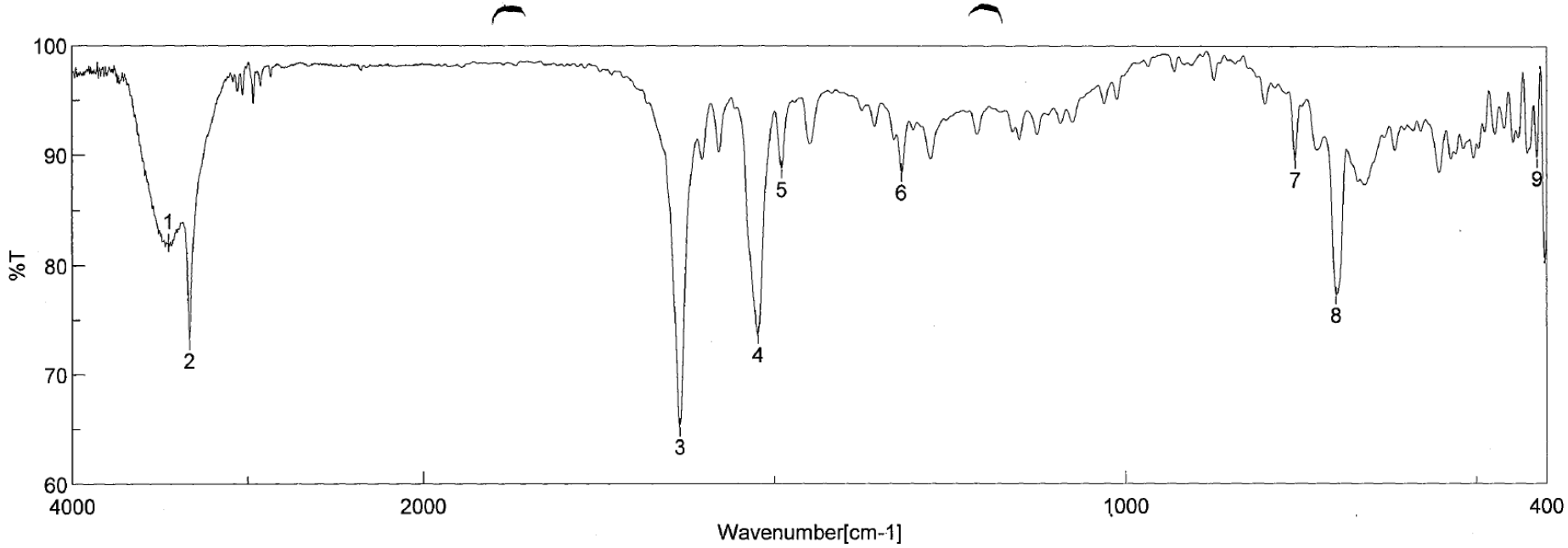

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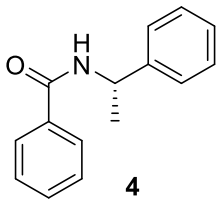

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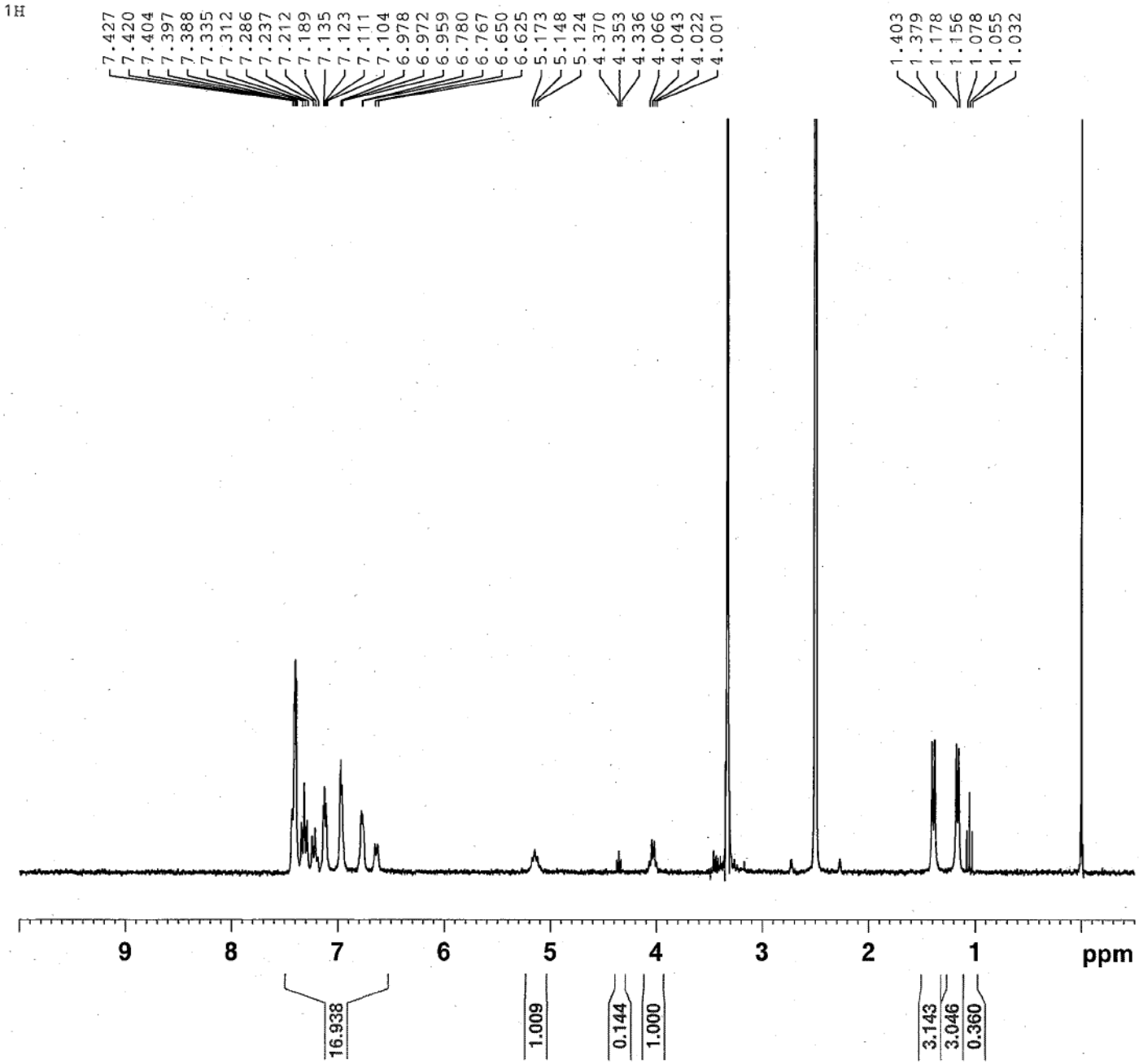

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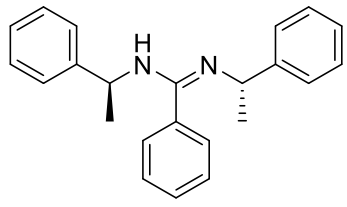

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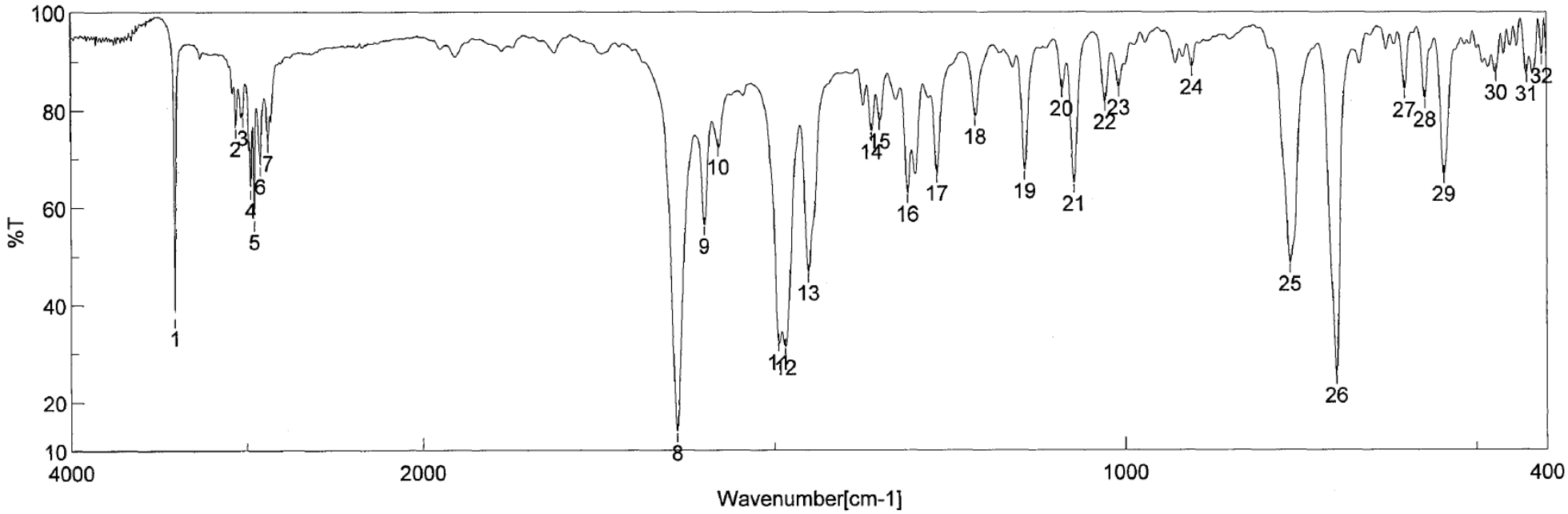

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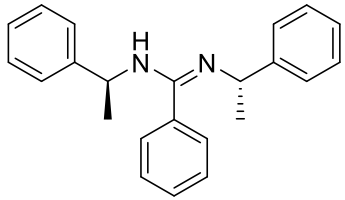

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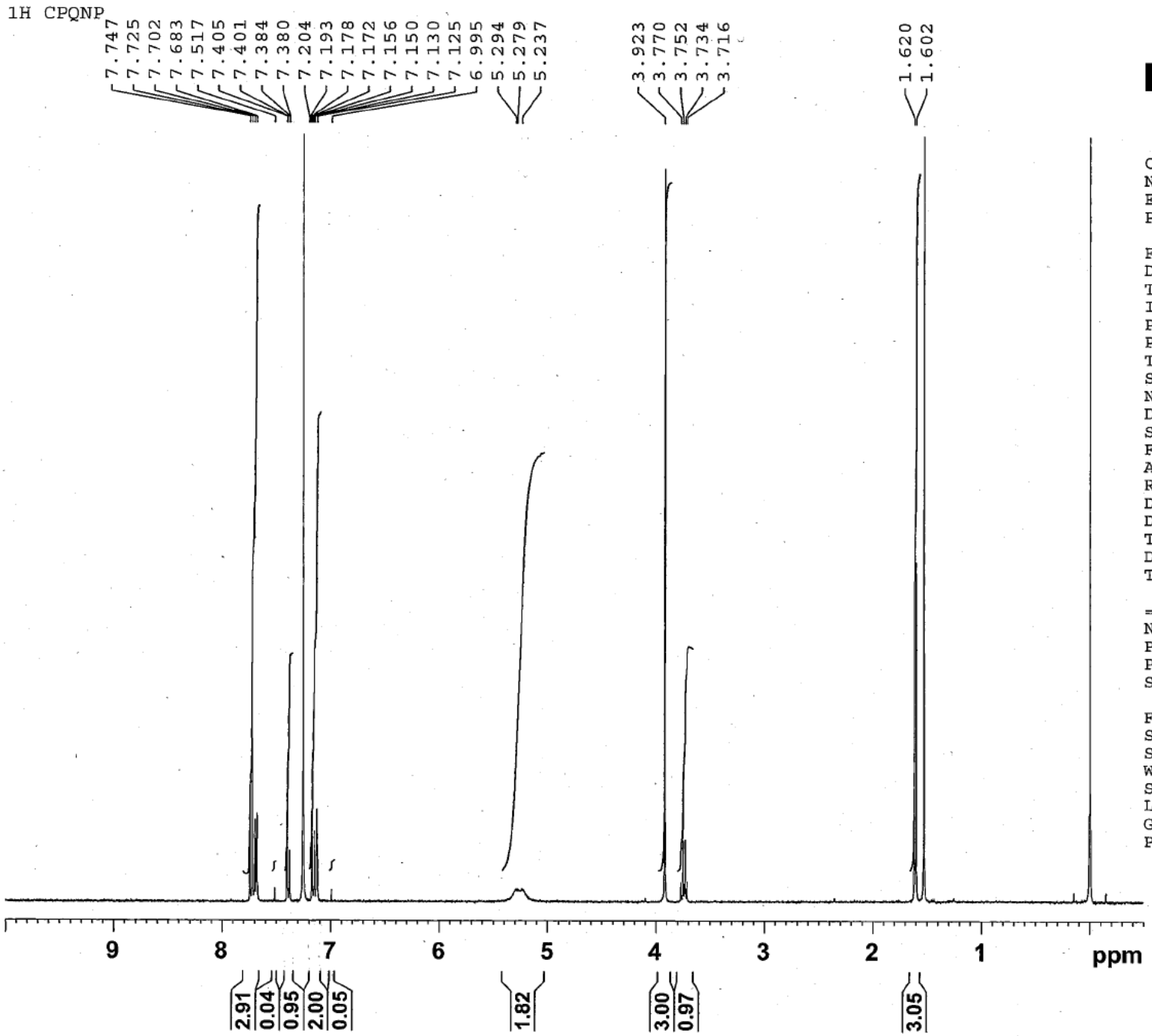


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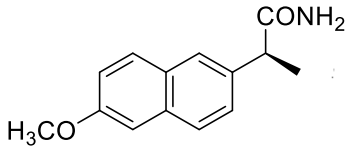

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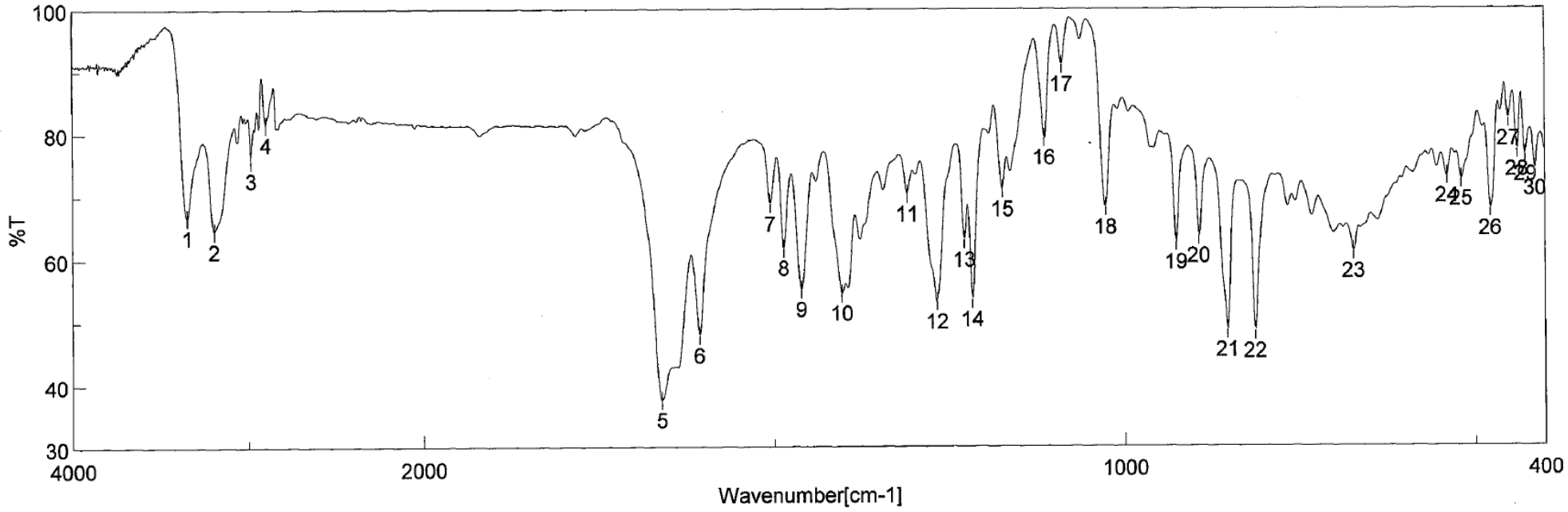

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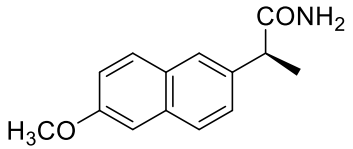

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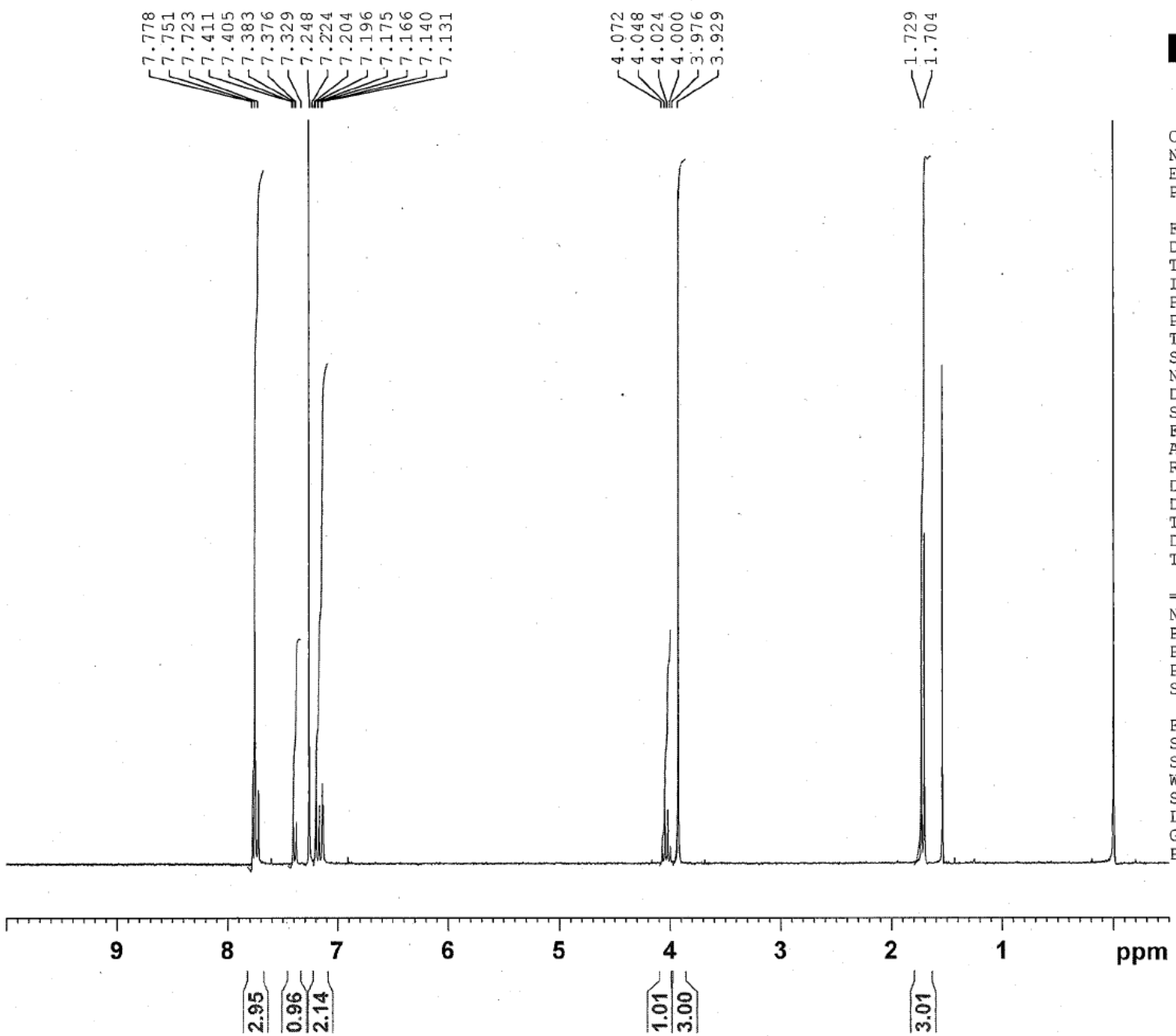

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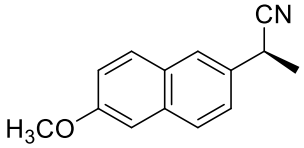
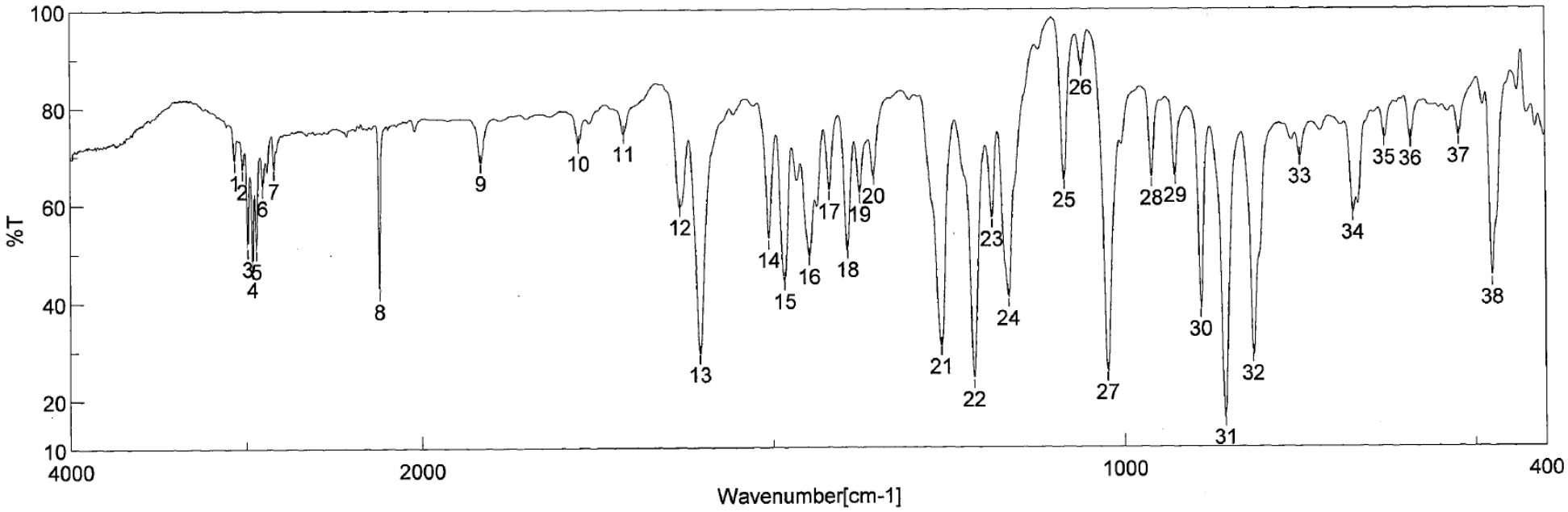
Time 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



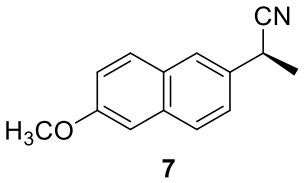
7



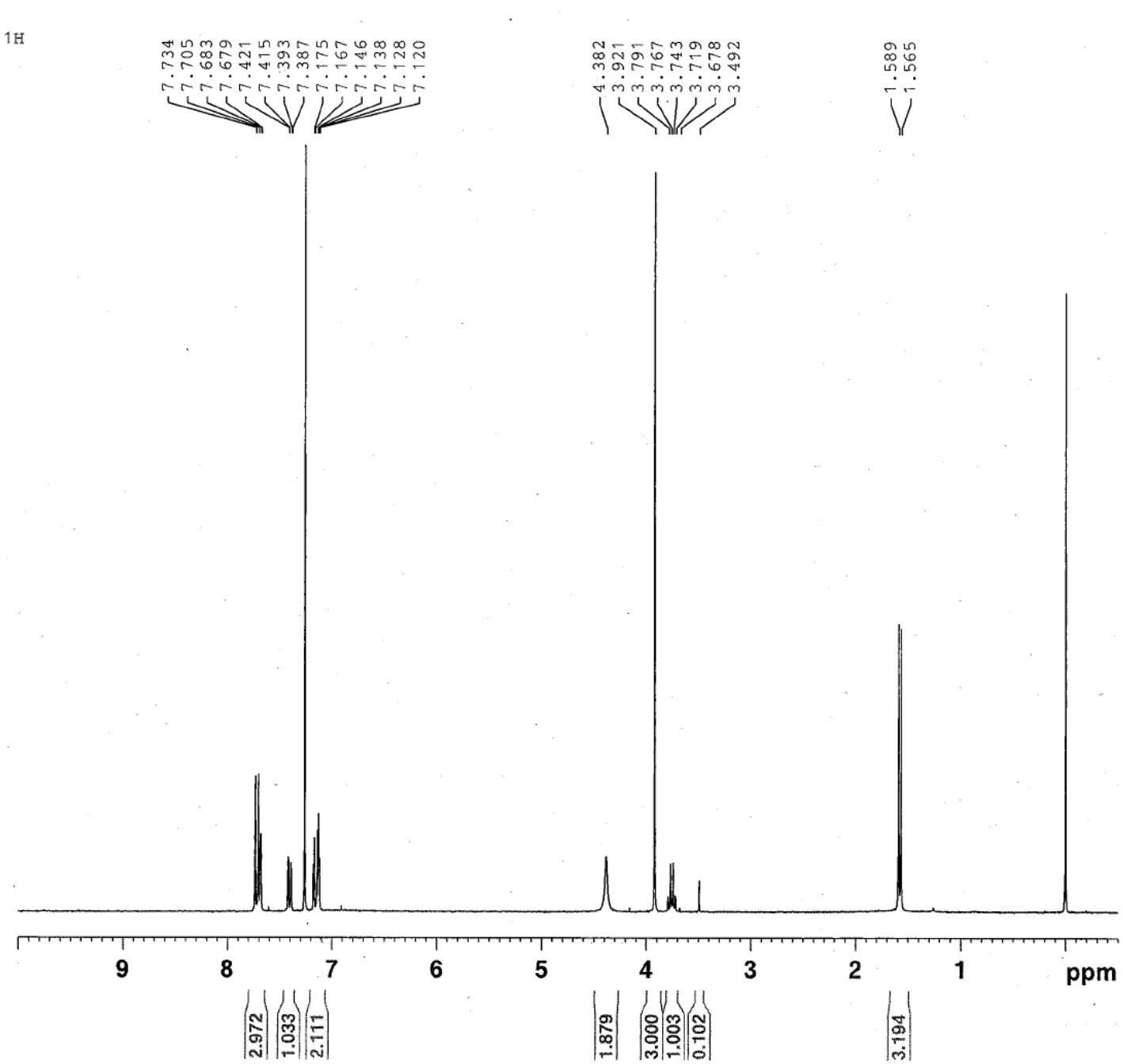
積算回数	16
ゼロフィリング	ON
ゲイン	8
測定日時	2017/12/23 12:07
測定者	takase
ファイル名	Memory#2
サンプル名	naproxene nitrile
コメント	

分解	4 cm-1
アポダイゼーション	Cosine
スキャンスピード	2 mm/sec
更新日時	2017/12/23 12:08

1:	3065.30,	68.69	2:	3019.98,	66.91	3:	2992.02,	51.16	4:	2963.09,	46.98
5:	2941.88,	50.67	6:	2905.24,	63.49	7:	2840.63,	67.05	8:	2239.91,	42.36
9:	1915.93,	68.52	10:	1777.08,	72.54	11:	1712.48,	74.52	12:	1632.45,	59.39
13:	1604.48,	29.18	14:	1506.13,	52.65	15:	1482.99,	44.21	16:	1448.28,	49.27
17:	1419.35,	62.59	18:	1393.32,	50.67	19:	1375.96,	61.97	20:	1355.71,	65.75
21:	1260.25,	30.81	22:	1213.97,	23.95	23:	1187.94,	56.92	24:	1164.79,	40.68
25:	1084.76,	64.80	26:	1059.69,	88.07	27:	1024.02,	25.39	28:	960.38,	65.22
29:	926.63,	65.72	30:	890.95,	38.36	31:	856.24,	16.01	32:	816.71,	29.20
33:	750.17,	69.58	34:	674.96,	58.15	35:	629.64,	73.51	36:	592.04,	73.25
37:	523.58,	73.80	38:	476.33,	44.80						

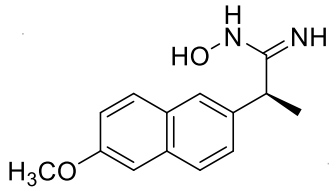


7



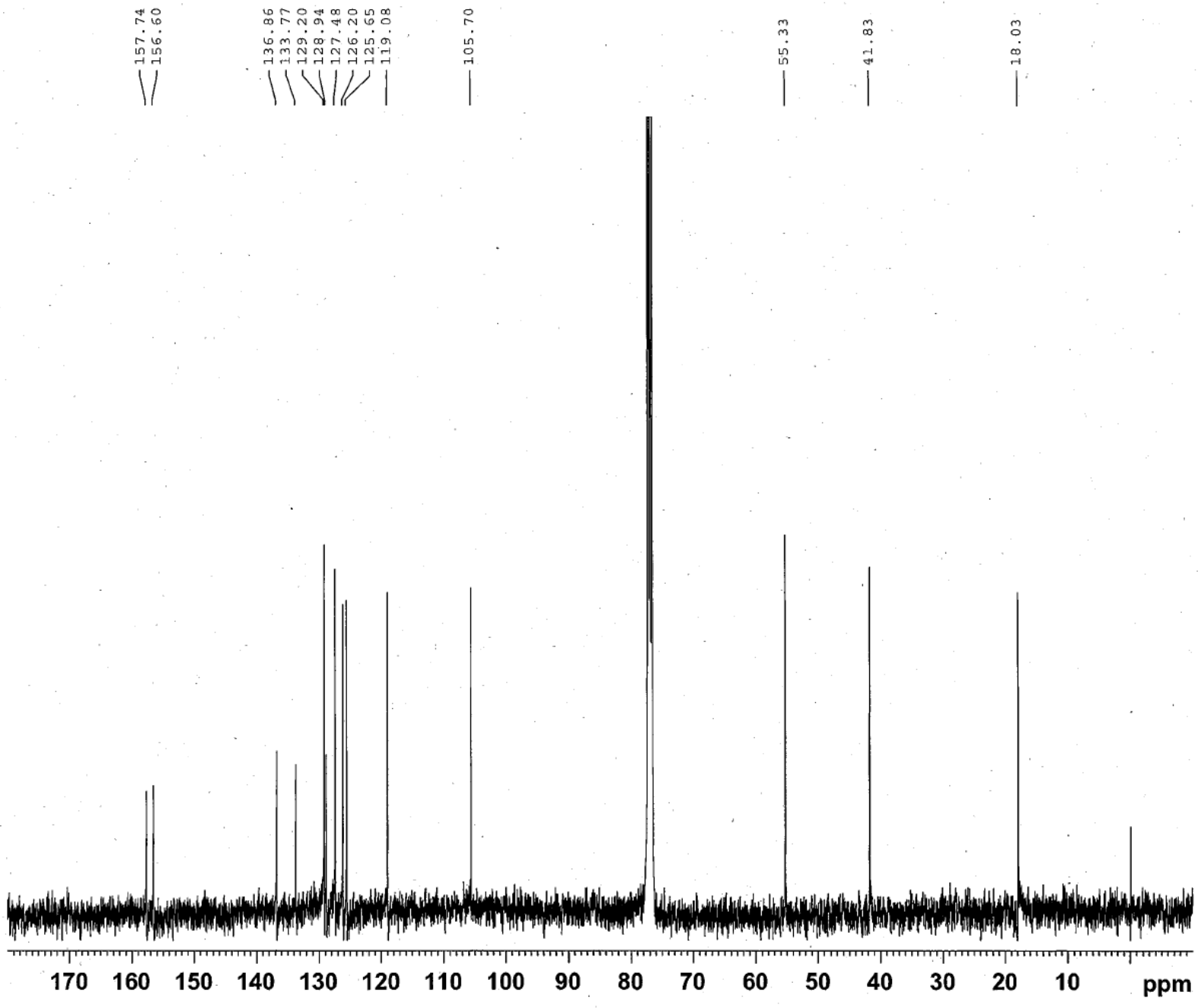
BRUKER

NAME	A17mc219tf	
EXPNO	18010903	
PROCNO	1	
Date_	20180109	
Time	18.05	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	6188.119	Hz
FIDRES	0.094423	Hz
AQ	5.2953587	sec
RG	203	
DW	80.800	usec
DE	6.50	usec
TE	296.8	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	15.00	usec
PL1	1.20	dB
PL1W	8.19348145	W
SFO1	300.1318534	MHz
SI	32768	
SF	300.1300061	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



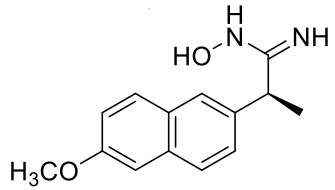
8

13C with dec. CPQNP

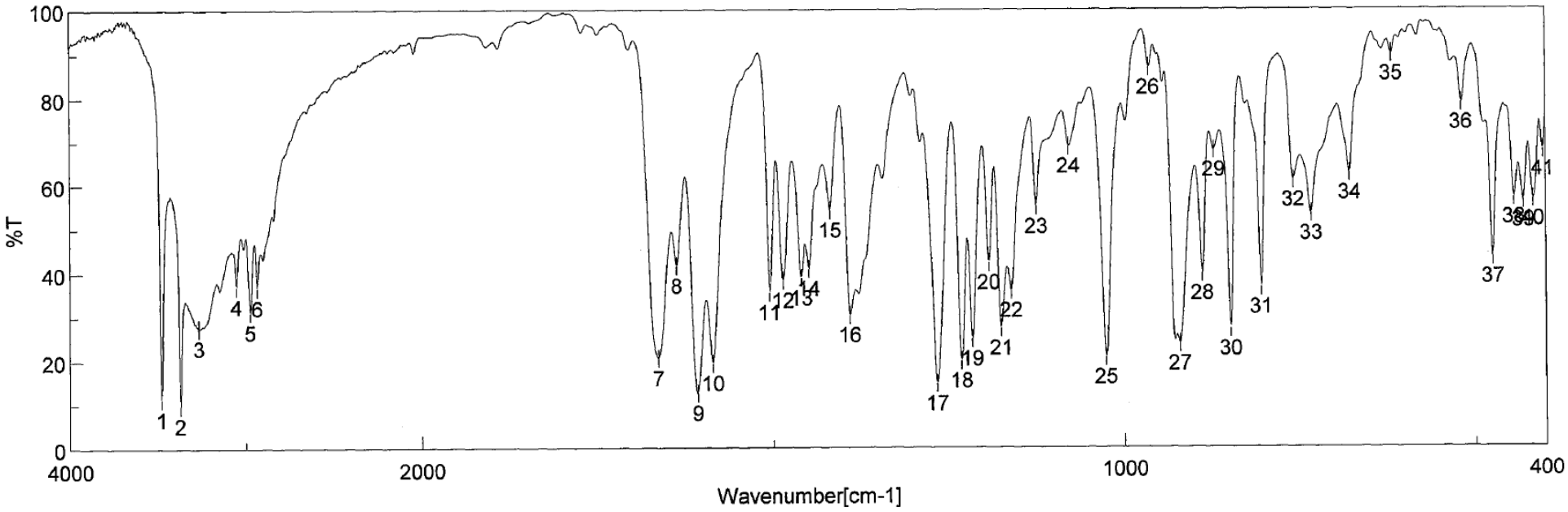


BRUKER

```
Current Data Parameters
NAME                  A17mc219tf
EXPNO                   19012401
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20190124
Time                       12.47
INSTRUM                    spect
PROBHD   5 mm CPQNP 1H/
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           256
DS                             2
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                        110.67
DW                        16.800 usec
DE                        18.00 usec
TE                        300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                        12.00 usec
PLW1                15.50000000 W
SFO1               100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                     90.00 usec
PLW2                 5.19999981 W
PLW12                0.14444000 W
PLW13                0.11700000 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                 100.6127690 MHz
WDW                           EM
SSB              0
LB                         2.00 Hz
GB               0
PC                         1.40
```

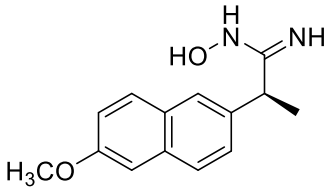


8

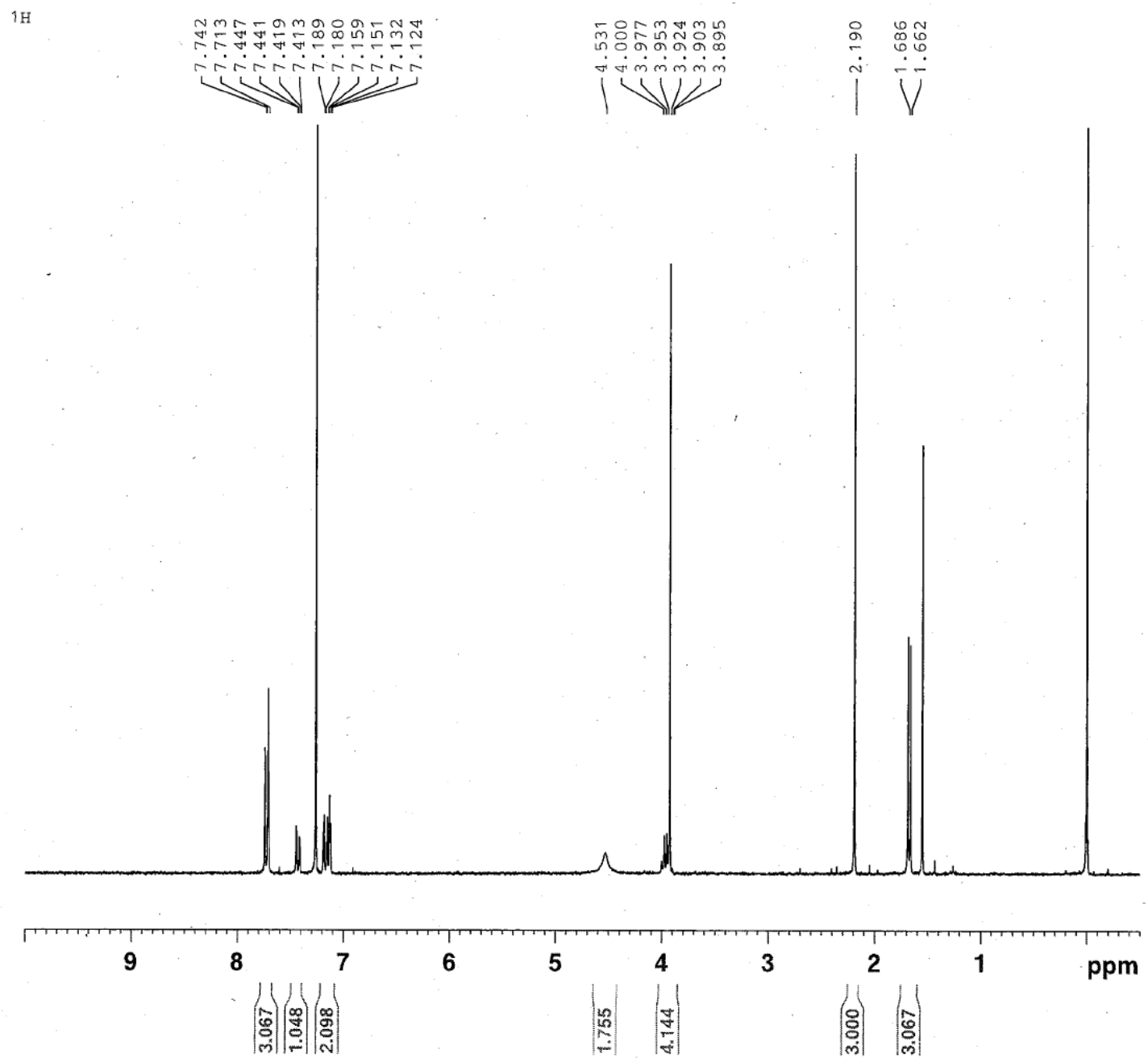


積算回数	16	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2018/01/11 10:33	更新日時	2018/01/11 10:34
測定者	takase		
ファイル名	Memory#2		
サンプル名	N-OH naproxen amidine		
コメント			

1:	3478.95,	11.40	2:	3371.92,	9.84	3:	3267.79,	27.59	4:	3054.69,	37.06
5:	2975.62,	31.22	6:	2936.09,	36.78	7:	1663.30,	20.52	8:	1637.27,	41.71
9:	1607.38,	12.53	10:	1586.16,	19.40	11:	1505.17,	35.56	12:	1485.88,	38.36
13:	1459.85,	38.90	14:	1449.24,	41.05	15:	1418.39,	54.32	16:	1390.42,	30.44
17:	1266.04,	14.70	18:	1231.33,	19.90	19:	1215.90,	24.97	20:	1191.79,	42.42
21:	1174.44,	27.40	22:	1159.97,	36.03	23:	1124.30,	55.19	24:	1078.01,	69.02
25:	1025.94,	20.72	26:	964.23,	87.04	27:	920.84,	23.82	28:	889.02,	39.96
29:	871.67,	68.13	30:	848.53,	27.14	31:	805.13,	36.74	32:	758.85,	61.20
33:	733.78,	53.54	34:	678.82,	62.78	35:	619.04,	90.08	36:	519.72,	78.93
37:	475.37,	43.58	38:	445.48,	57.09	39:	431.98,	56.44	40:	418.48,	56.70

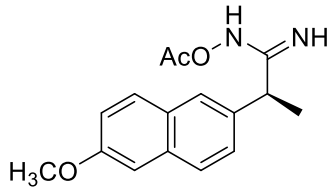


8



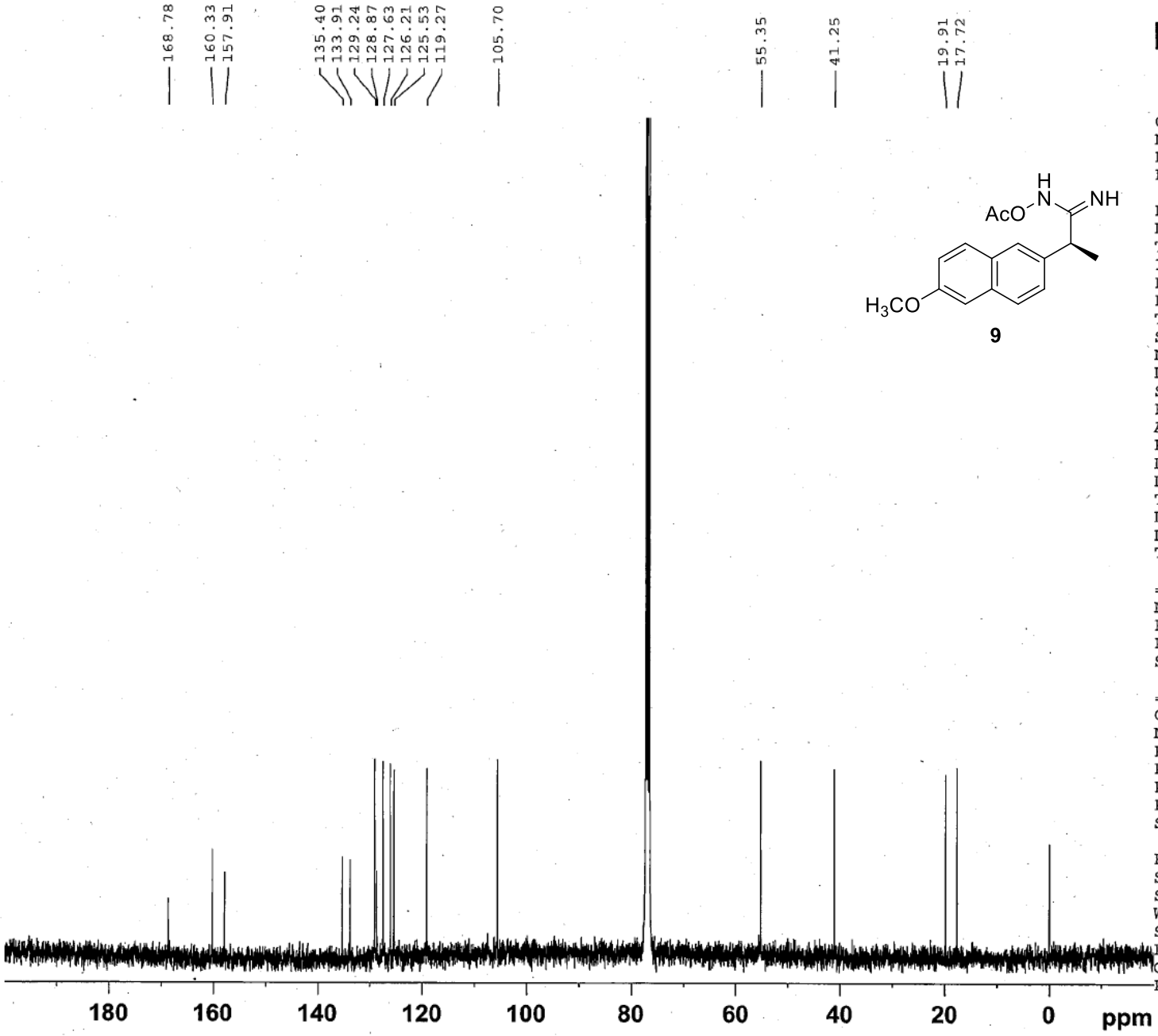
BRUKER

NAME	A17mc219tf
EXPNO	18022701
PROCNO	1
Date_	20180227
Time	15.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	6188.119 Hz
FIDRES	0.094423 Hz
AQ	5.2953587 sec
RG	203
DW	80.800 usec
DE	6.50 usec
TE	296.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	1.20 dB
PL1W	8.19348145 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300057 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



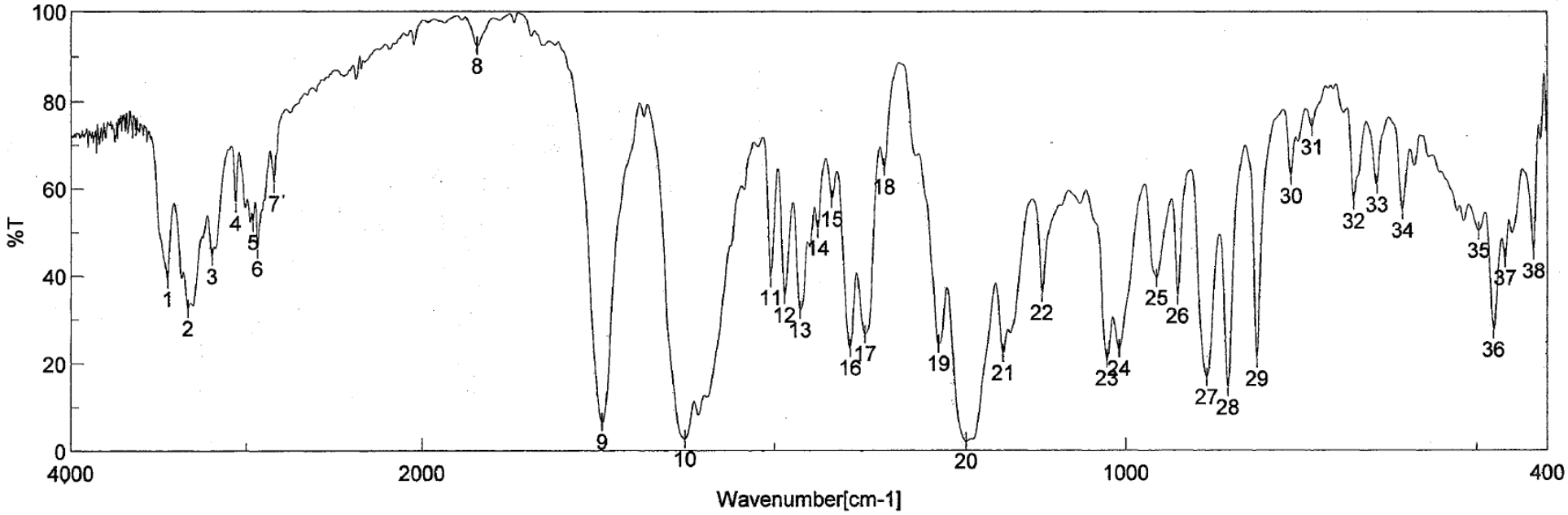
9

13C with dec. CPQNP



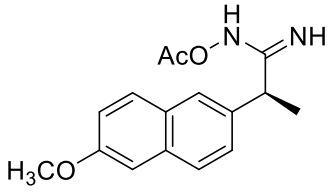
9

```
Current Data Parameters
NAME                 A17mc219tf
EXPNO                  19020101
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20190201
Time                      13.09
INSTRUM                   spect
PROBHD     5 mm CPQNP 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         186
DS                           2
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                       126.99
DW                       16.800 usec
DE                       18.00 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                       12.00 usec
PLW1                15.50000000 W
SFO1                100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PLW2                 5.19999981 W
PLW12                0.14444000 W
PLW13                0.11700000 W
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                          32768
SF                 100.6127682 MHz
WDW                            EM
SSB                 0
LB                       2.00 Hz
GB                  0
PC                       1.40
```

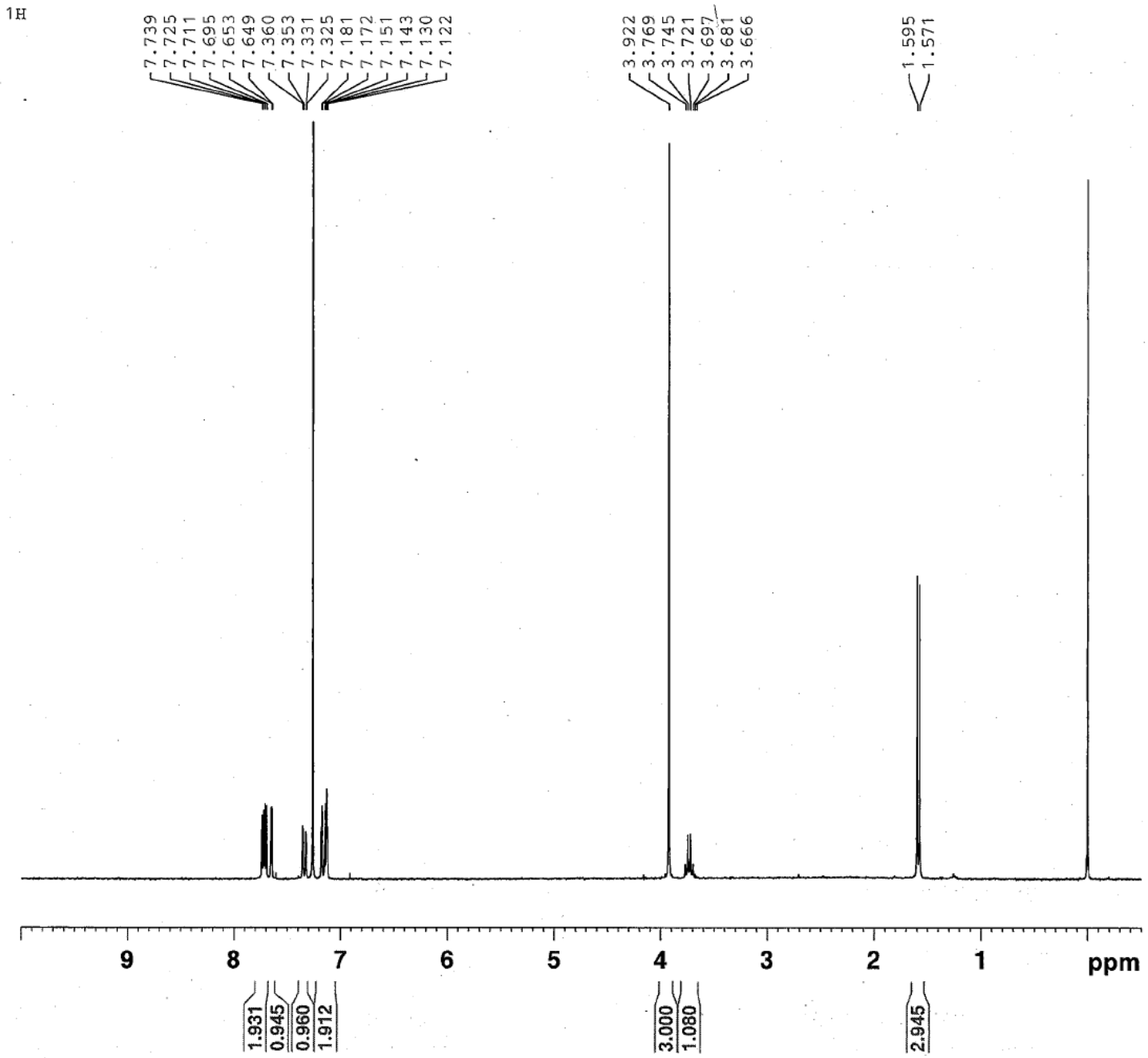


積算回数	8	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2019/01/26 14:14	更新日時	2019/01/26 14:15
測定者	takase		
ファイル名	Memory#2		
サンプル名	88, (S)-N-AcO-2-(6-methoxy-2-naphthyl)propionamidine		
コメント			

1:	3449.06,	39.29	2:	3332.39,	32.57	3:	3196.43,	44.47	4:	3060.48,	56.69
5:	2962.13,	52.31	6:	2936.09,	46.22	7:	2841.60,	61.19	8:	1921.72,	92.47
9:	1744.30,	6.59	10:	1626.66,	2.69	11:	1505.17,	39.61	12:	1485.88,	35.63
13:	1463.71,	32.22	14:	1438.64,	50.95	15:	1418.39,	57.71	16:	1392.35,	23.48
17:	1371.14,	26.73	18:	1344.14,	65.00	19:	1266.04,	24.50	20:	1226.51,	2.12
21:	1173.47,	22.32	22:	1118.51,	36.18	23:	1026.91,	21.13	24:	1009.55,	23.39
25:	956.52,	39.60	26:	926.63,	35.48	27:	885.17,	16.85	28:	855.28,	14.44
29:	813.81,	21.00	30:	765.60,	62.91	31:	735.71,	74.10	32:	675.93,	57.24
33:	643.14,	60.92	34:	606.50,	55.16	35:	497.54,	50.48	36:	476.33,	27.67
37:	459.94,	44.15	38:	419.44,	45.77						

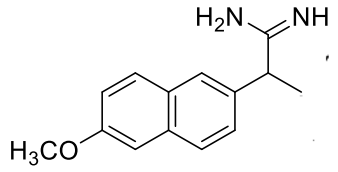


9

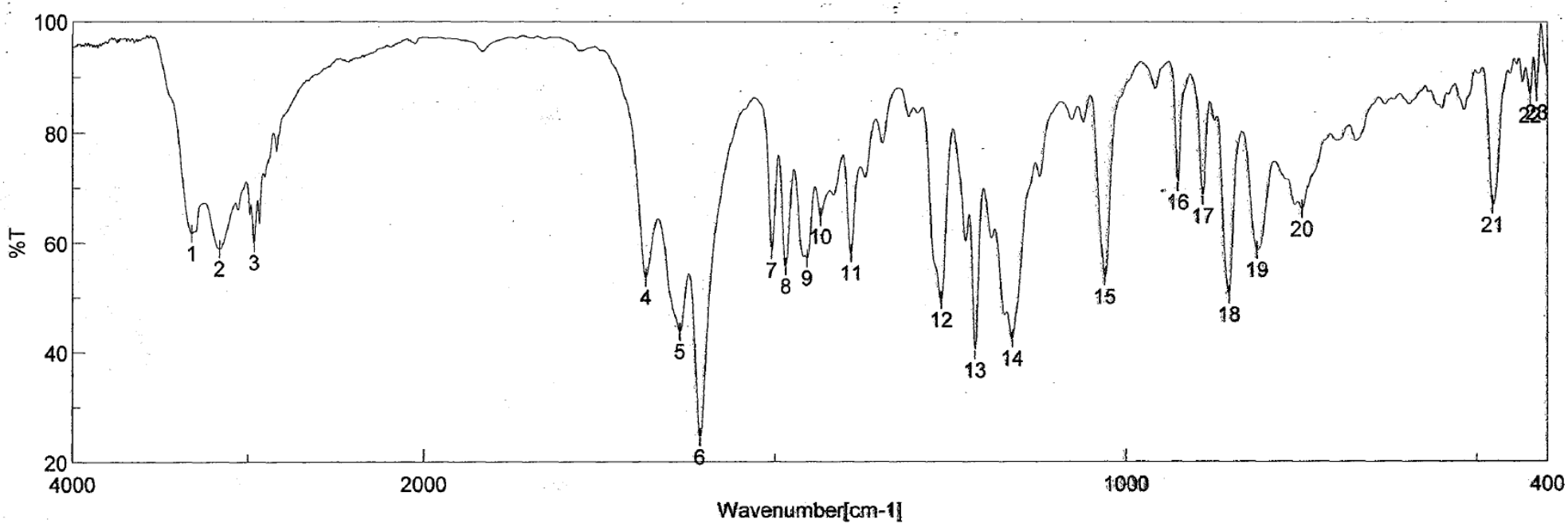


BRUKER

NAME	A17mc219tf	
EXPNO	18062701	
PROCNO	1	
Date_	20180627	
Time	10.31	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	6188.119	Hz
FIDRES	0.094423	Hz
AQ	5.2953587	sec
RG	203	
DW	80.800	usec
DE	6.50	usec
TE	296.6	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	15.00	usec
PL1	1.20	dB
PL1W	8.19348145	W
SFO1	300.1318534	MHz
SI	32768	
SF	300.1300058	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

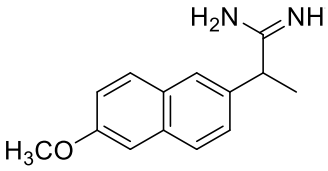


2b

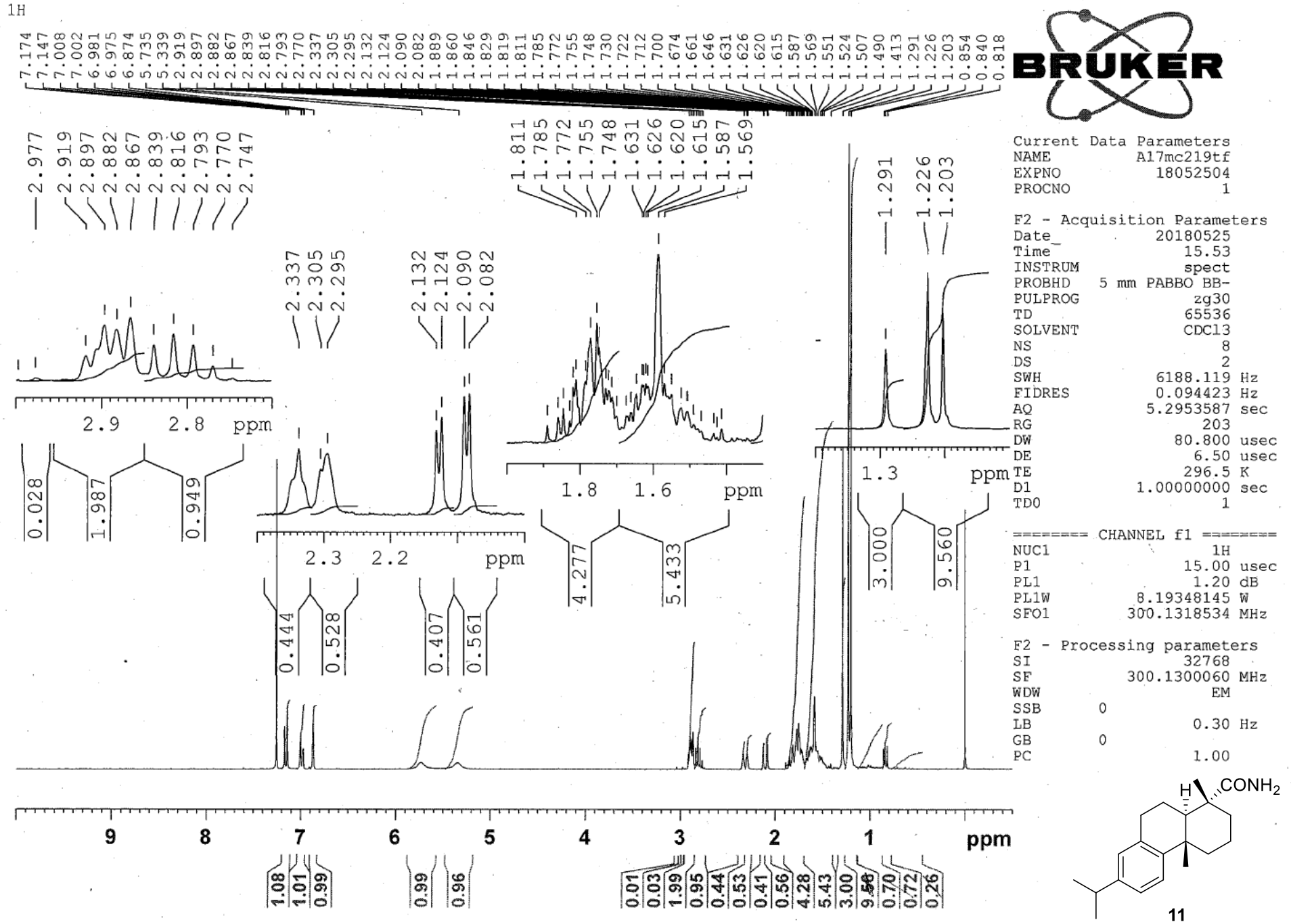


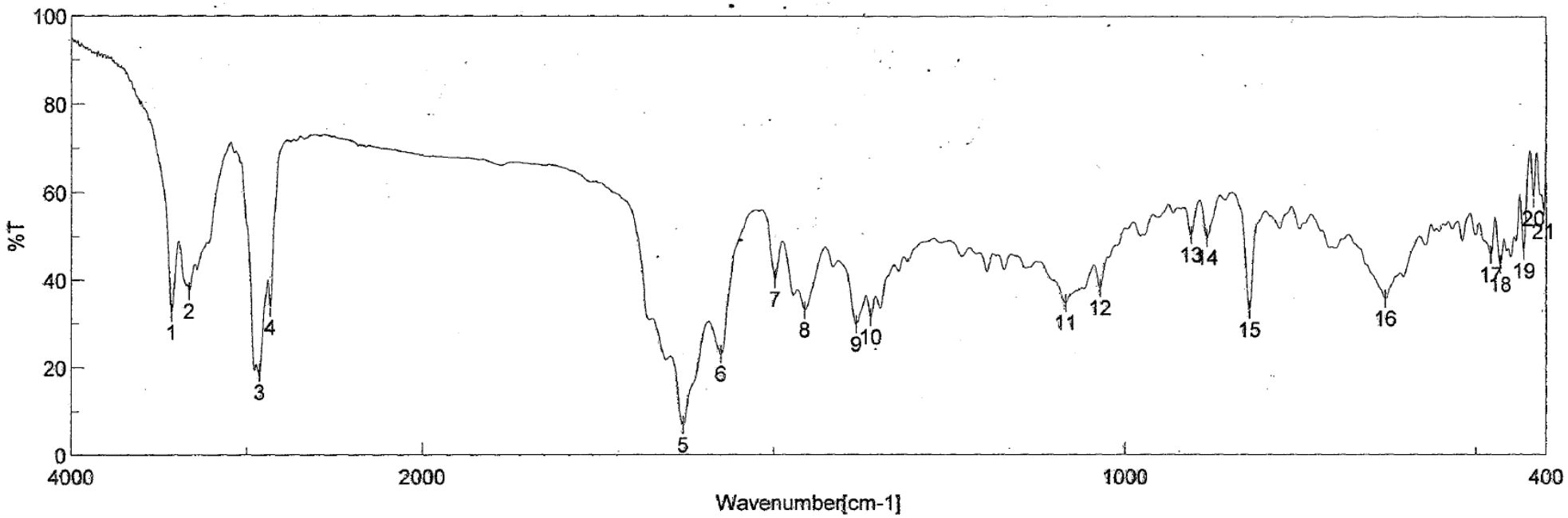
積算回数	16	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	8	スキャンスピード	2 mm/sec
測定日時	2018/03/02 16:17	更新日時	2018/03/02 16:18
測定者	takase		
ファイル名	Memory#2		
サンプル名	S-naproxene amidine		
コメント			

1:	3320.82,	61.77	2:	3162.69,	58.97	3:	2965.98,	60.08	4:	1683.55,	53.69
5:	1635.34,	43.79	6:	1606.41,	24.50	7:	1505.17,	58.68	8:	1484.92,	55.76
9:	1455.03,	57.26	10:	1435.74,	64.91	11:	1392.35,	58.17	12:	1264.11,	49.56
13:	1215.90,	40.32	14:	1162.87,	42.54	15:	1030.77,	53.83	16:	926.63,	71.15
17:	890.95,	68.67	18:	853.35,	50.61	19:	813.81,	58.81	20:	750.17,	66.30
21:	478.26,	67.08	22:	425.23,	86.87	23:	415.58,	87.36			



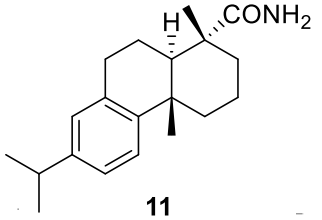
2b


1H
BRUKER
Current Data Parameters
NAME A17mc219tf
EXPNO 18052504
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180525
Time 15.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
CONH2
H
11

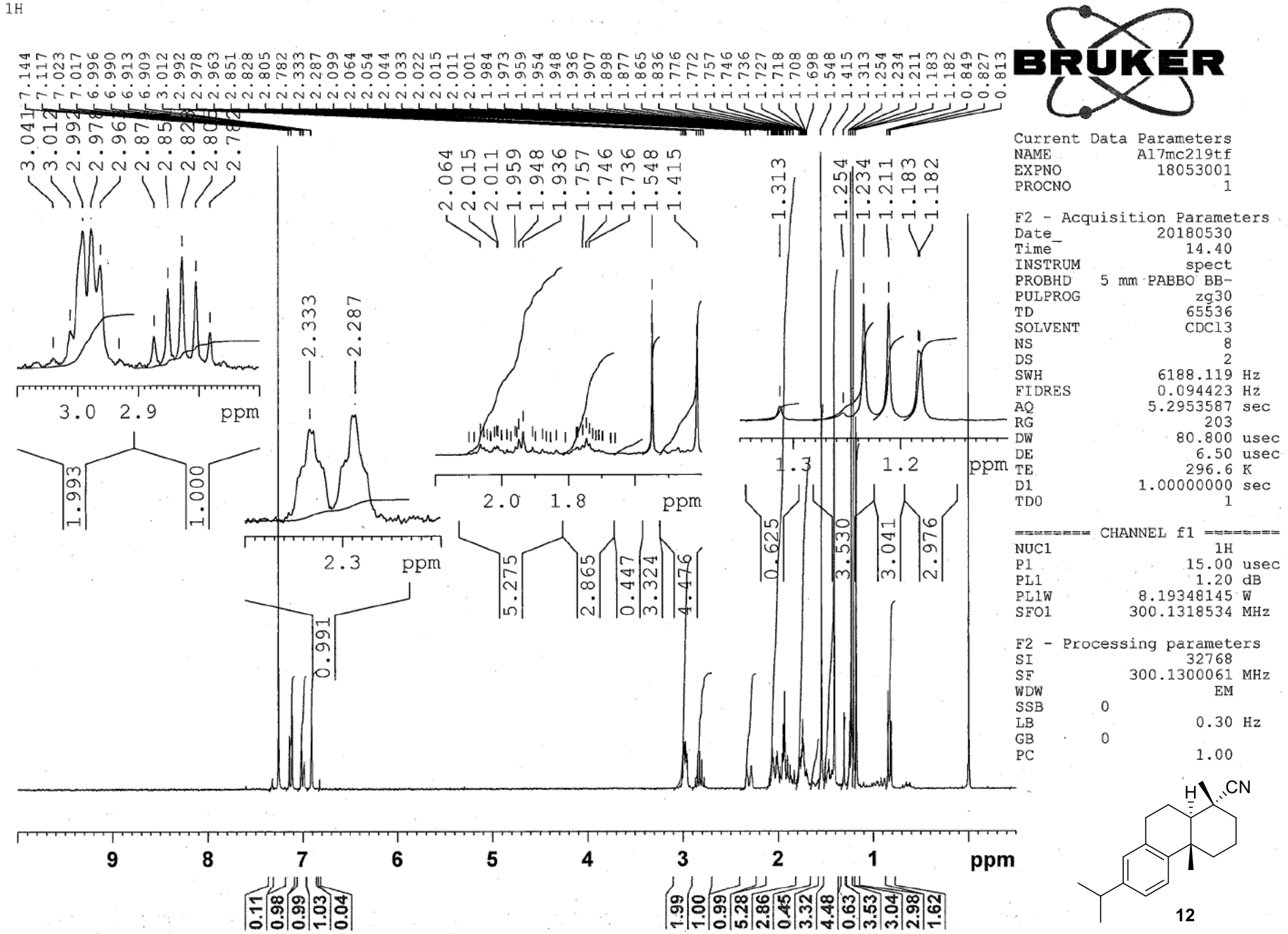


積算回数	4	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2018/05/30 15:34	更新日時	2018/05/30 15:36
測定者	takase		
ファイル名	Memory#2		
サンプル名	dehydroabietyl amide		
コメント			

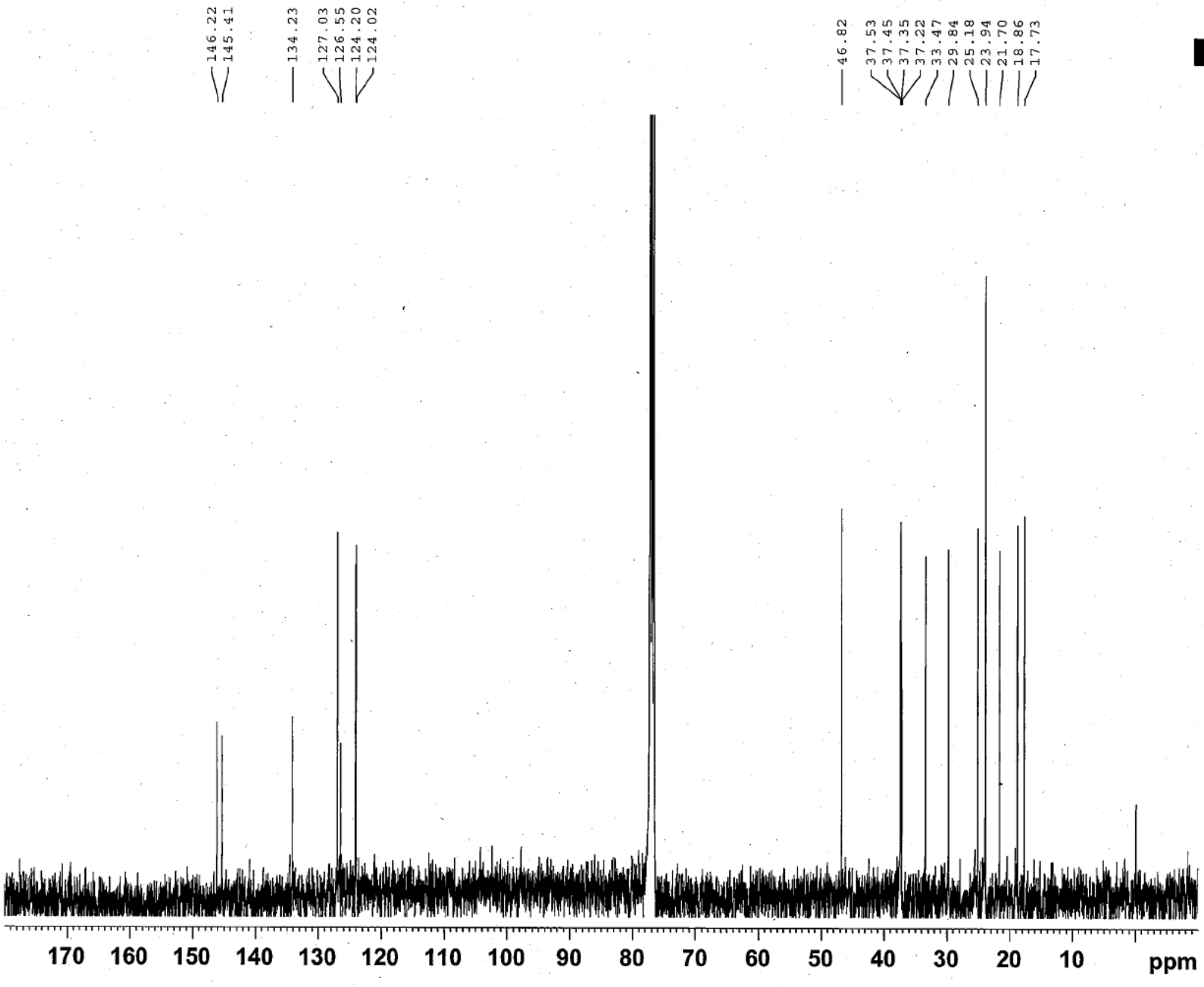
1:	3427.85,	32.57	2:	3327.57,	37.42	3:	2927.41,	18.90	4:	2866.67,	33.75
5:	1628.59,	6.87	6:	1574.59,	23.09	7:	1498.42,	40.22	8:	1455.99,	33.12
9:	1382.71,	29.90	10:	1362.46,	31.52	11:	1084.76,	34.84	12:	1035.59,	38.23
13:	905.42,	50.49	14:	883.24,	49.87	15:	822.49,	33.23	16:	629.64,	35.83
17:	479.22,	45.96	18:	465.72,	43.75	19:	431.98,	46.85	20:	418.48,	58.79
21:	404.01,	55.86									



11

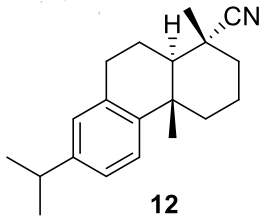

1H
BRUKER
Current Data Parameters
NAME A17mc219tf
EXPNO 18053001
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180530
Time 14.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
12

13C with dec. CPQNP

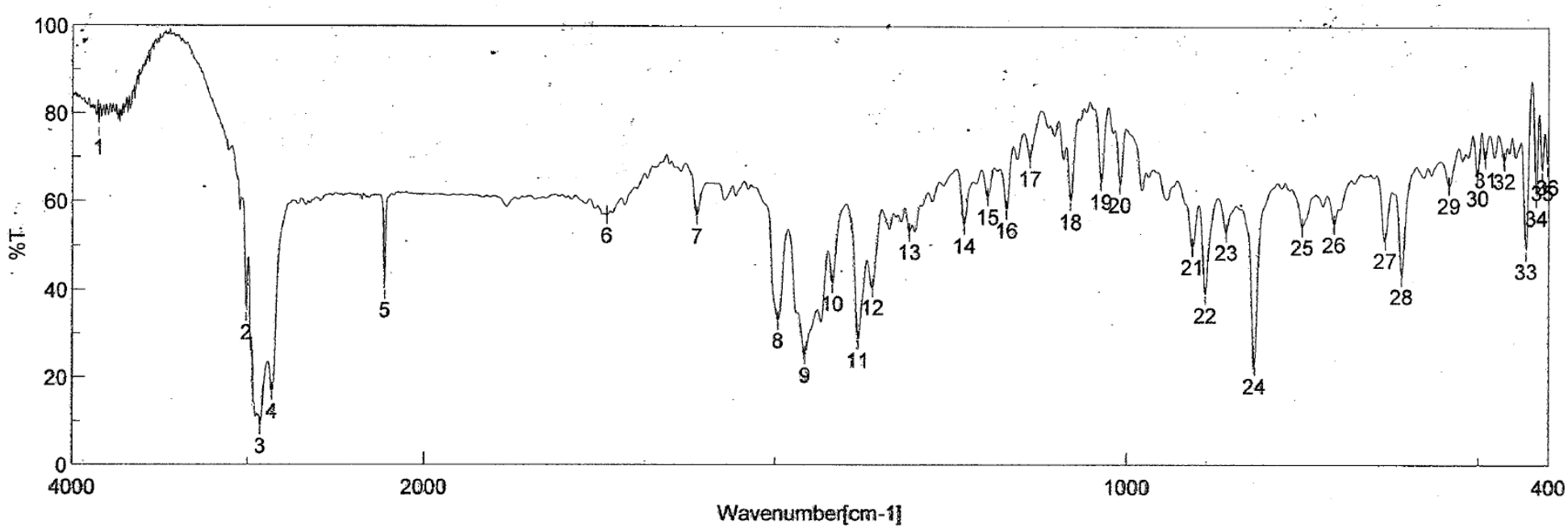


BRUKER

```
Current Data Parameters
NAME                 A17mc219tf
EXPNO                  19010701
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20190107
Time                      13.23
INSTRUM                   spect
PROBHD     5 mm CPQNP 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          64
DS                           2
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                       126.99
DW                       16.800 usec
DE                       18.00 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                       12.00 usec
PLW1                15.50000000 W
SFO1               100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PLW2                 5.19999981 W
PLW12                0.14444000 W
PLW13                0.11700000 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                          32768
SF                 100.6127690 MHz
WDW                            EM
SSB                 0
LB                           2.00 Hz
GB                  0
PC                           1.40
```

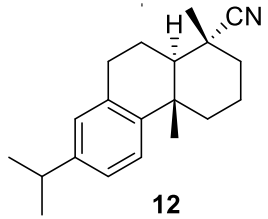


12

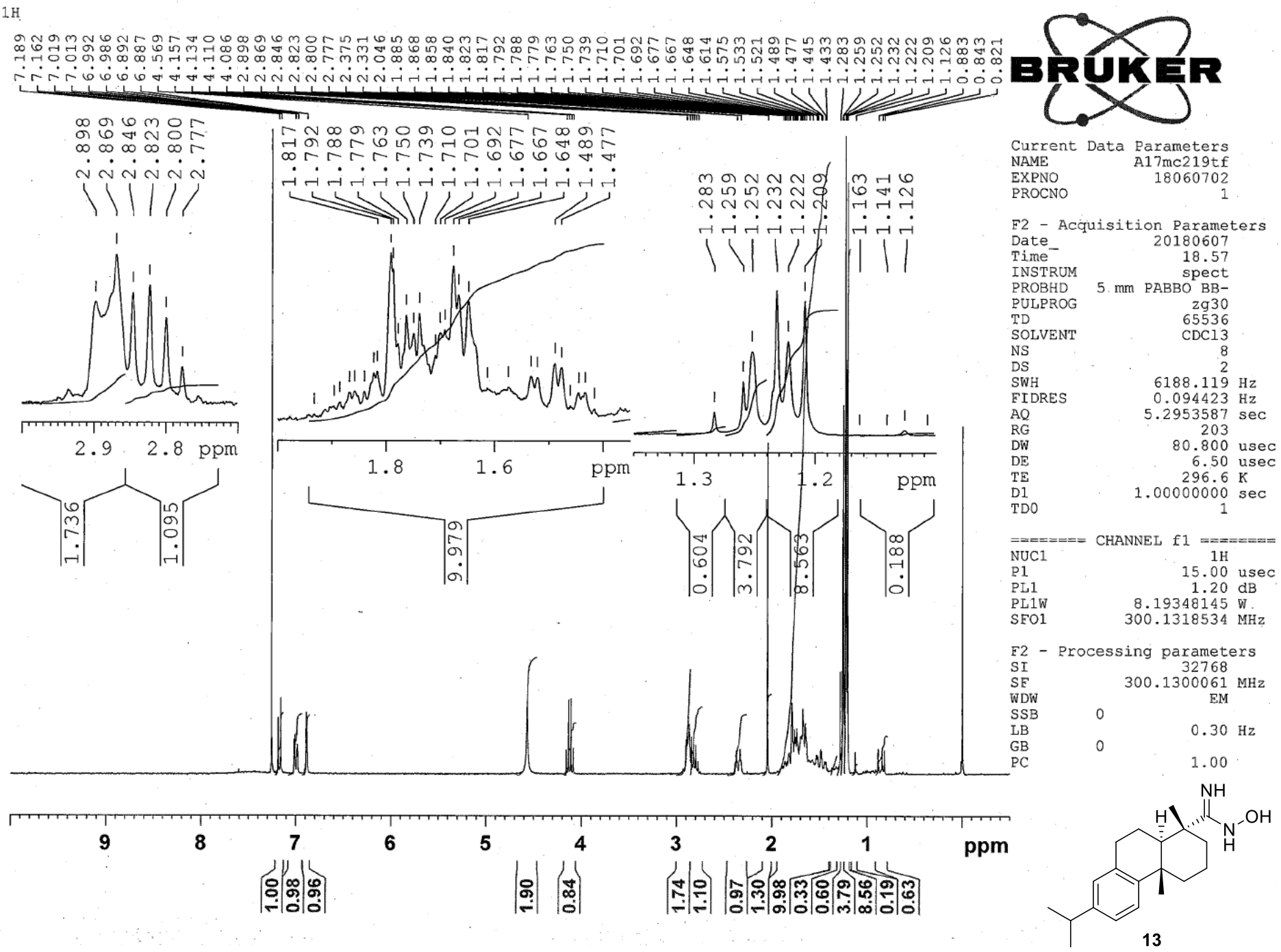


積算回数	4	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2018/05/30 15:52	更新日時	2018/05/30 15:53
測定者	takase		
ファイル名	Memory#2		
サンプル名	dehydroabietyl cyanide		
コメント			

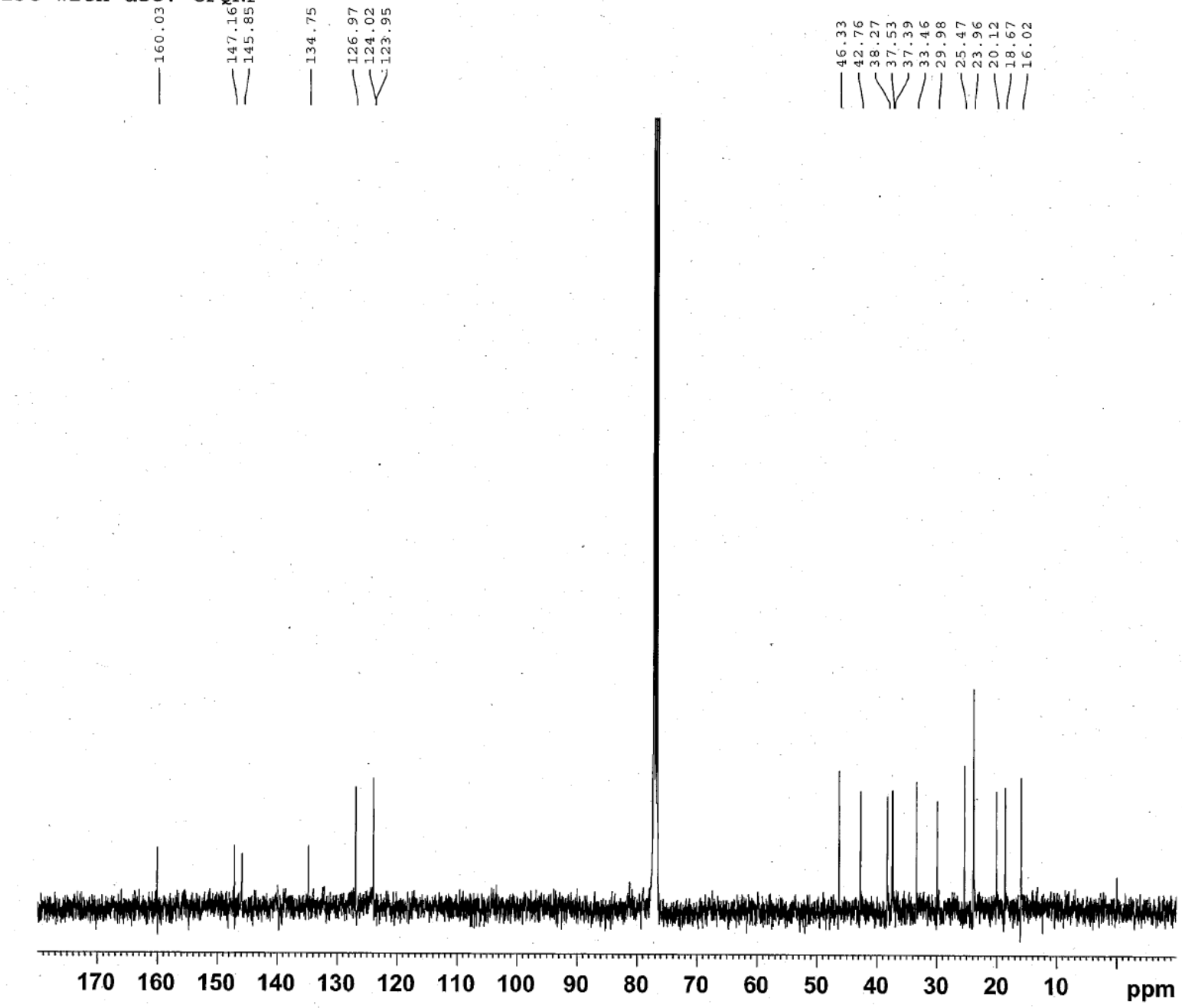
1:	3852.11,	76.68	2:	3010.34,	34.85	3:	2930.31,	8.90	4:	2862.81,	16.85
5:	2225.45,	39.91	6:	1740.44,	57.05	7:	1612.20,	56.90	8:	1496.49,	33.05
9:	1457.92,	25.15	10:	1419.35,	41.52	11:	1382.71,	28.58	12:	1362.46,	40.65
13:	1310.39,	53.31	14:	1232.29,	54.80	15:	1199.51,	61.21	16:	1172.51,	58.29
17:	1138.76,	69.87	18:	1080.91,	60.19	19:	1037.52,	64.63	20:	1010.52,	63.93
21:	907.34,	49.97	22:	889.02,	38.90	23:	859.13,	53.28	24:	818.63,	22.68
25:	751.14,	54.49	26:	705.82,	55.16	27:	633.50,	51.46	28:	610.36,	43.25
29:	542.86,	63.87	30:	502.37,	65.65	31:	490.79,	69.82	32:	463.80,	69.45
33:	432.94,	49.12	34:	418.48,	61.19	35:	409.80,	66.92	36:	402.09,	68.15



12

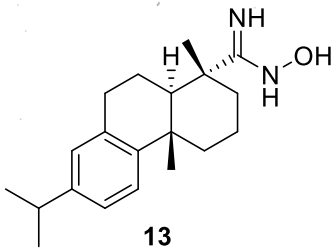

1H
BRUKER
Current Data Parameters
NAME A17mc219tf
EXPNO 18060702
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180607
Time 18.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
13

13C with dec. CPQNP

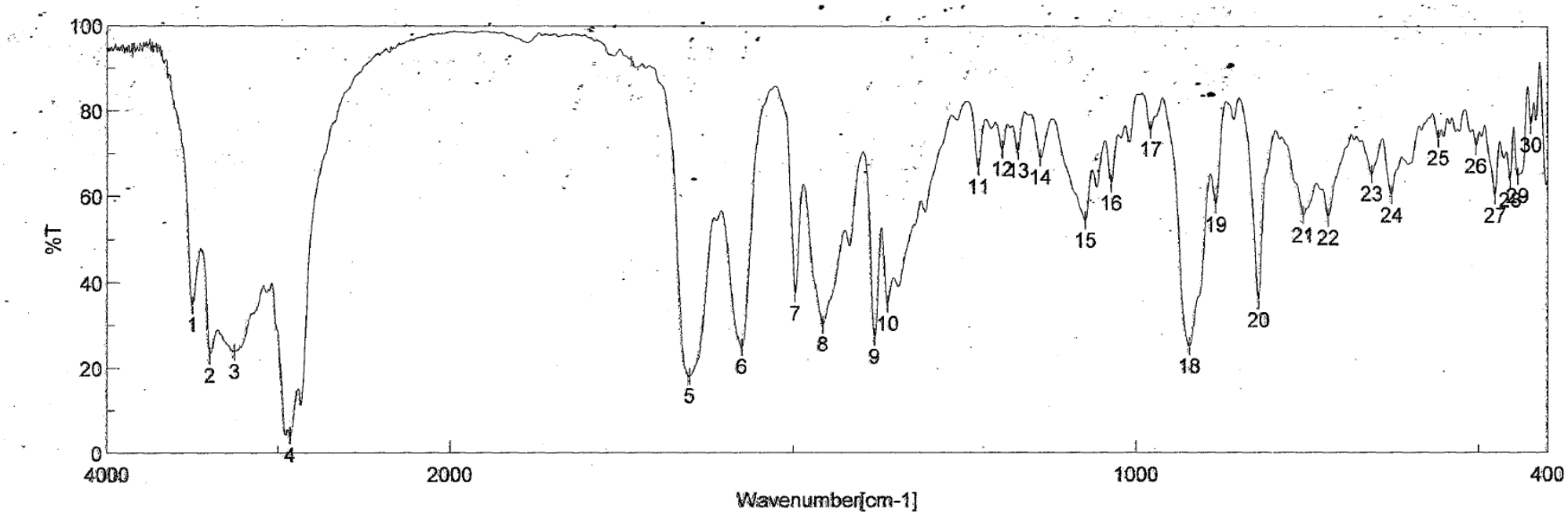


BRUKER

```
Current Data Parameters
NAME          A17mc219tf
EXPNO           19010801
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190109
Time               10.06
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                   91.75
DW                  16.800 usec
DE                  18.00 usec
TE                  300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.00 usec
PLW1          15.50000000 W
SFO1         100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2               90.00 usec
PLW2           5.19999981 W
PLW12          0.14444000 W
PLW13          0.11700000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.40
```

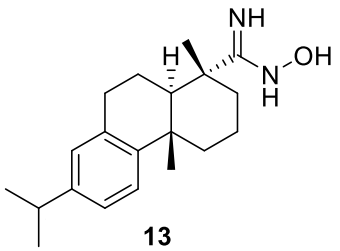


13

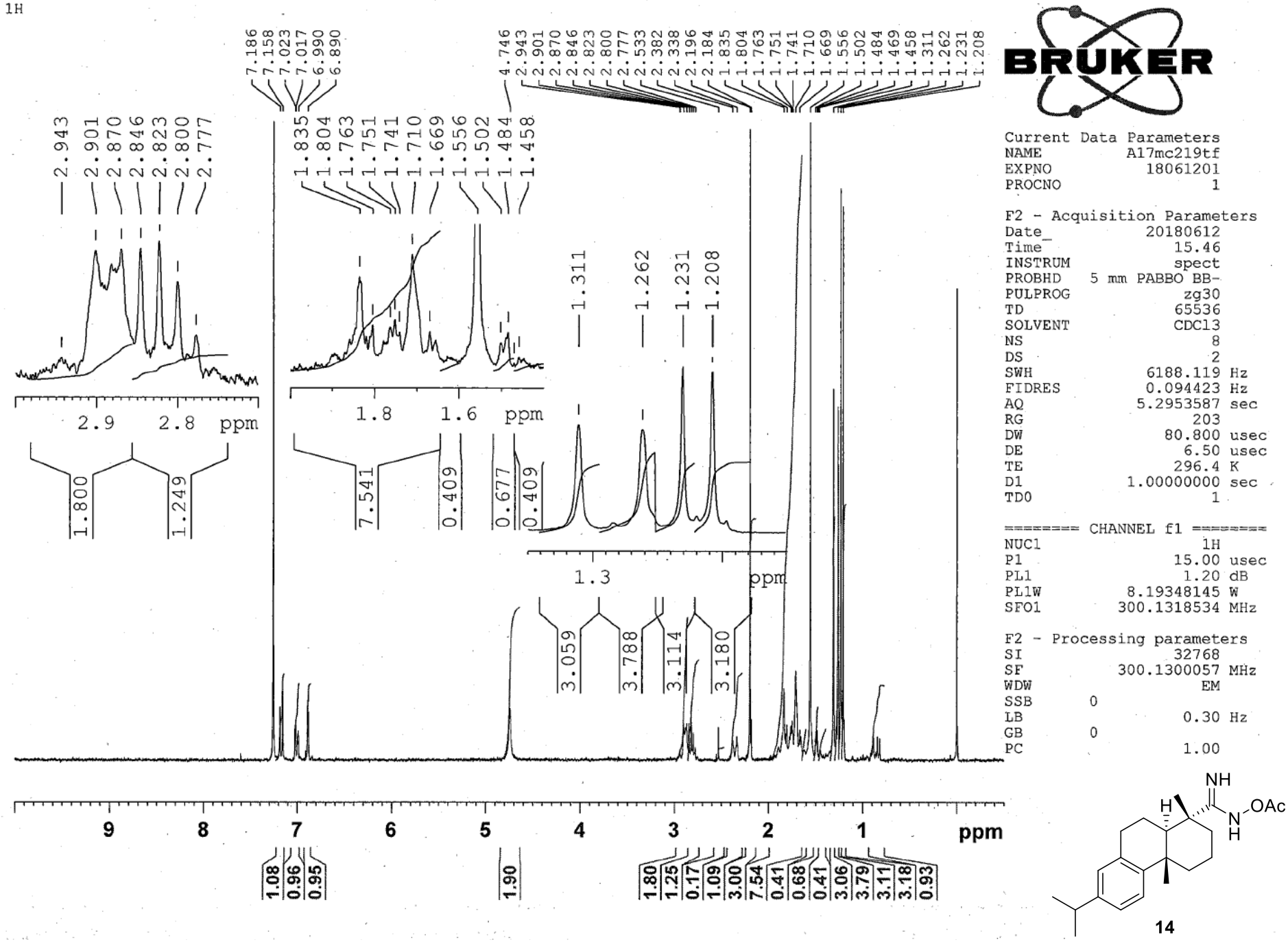


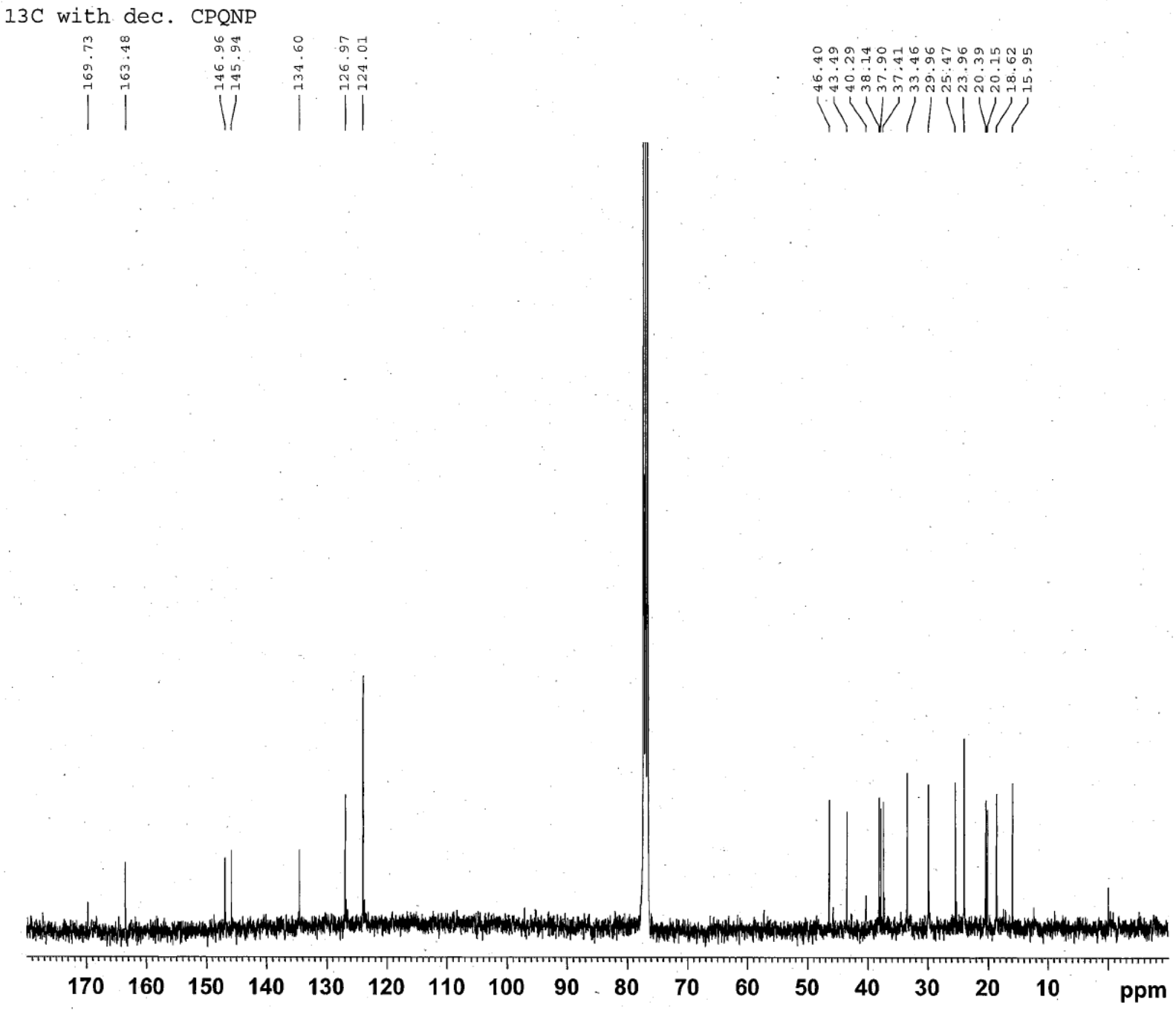
積算回数	4	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2018/06/08 13:40	更新日時	2018/06/08 13:40
測定者	takase		
ファイル名	Memory#2		
サンプル名	N-OH-dehydroabietyl amidine		
コメント			

1:	3500.17,	34.77	2:	3398.92,	22.89	3:	3255.25,	23.82	4:	2930.31,	4.13
5:	1650.77,	18.04	6:	1574.59,	24.96	7:	1497.45,	37.42	8:	1456.96,	30.18
9:	1381.75,	27.36	10:	1362.46,	35.04	11:	1230.36,	66.88	12:	1195.65,	70.98
13:	1172.51,	70.62	14:	1139.72,	69.07	15:	1074.16,	54.54	16:	1036.55,	63.03
17:	978.70,	75.61	18:	922.77,	25.09	19:	884.20,	58.13	20:	821.53,	35.89
21:	756.92,	55.72	22:	720.28,	55.16	23:	656.64,	65.21	24:	627.72,	60.30
25:	559.26,	73.60	26:	504.29,	71.75	27:	476.33,	60.22	28:	454.15,	63.76
29:	443.55,	64.76	30:	424.26,	76.69						

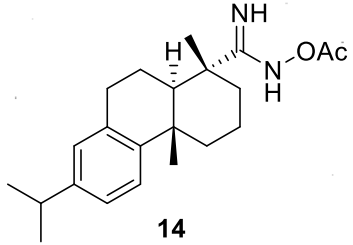


13

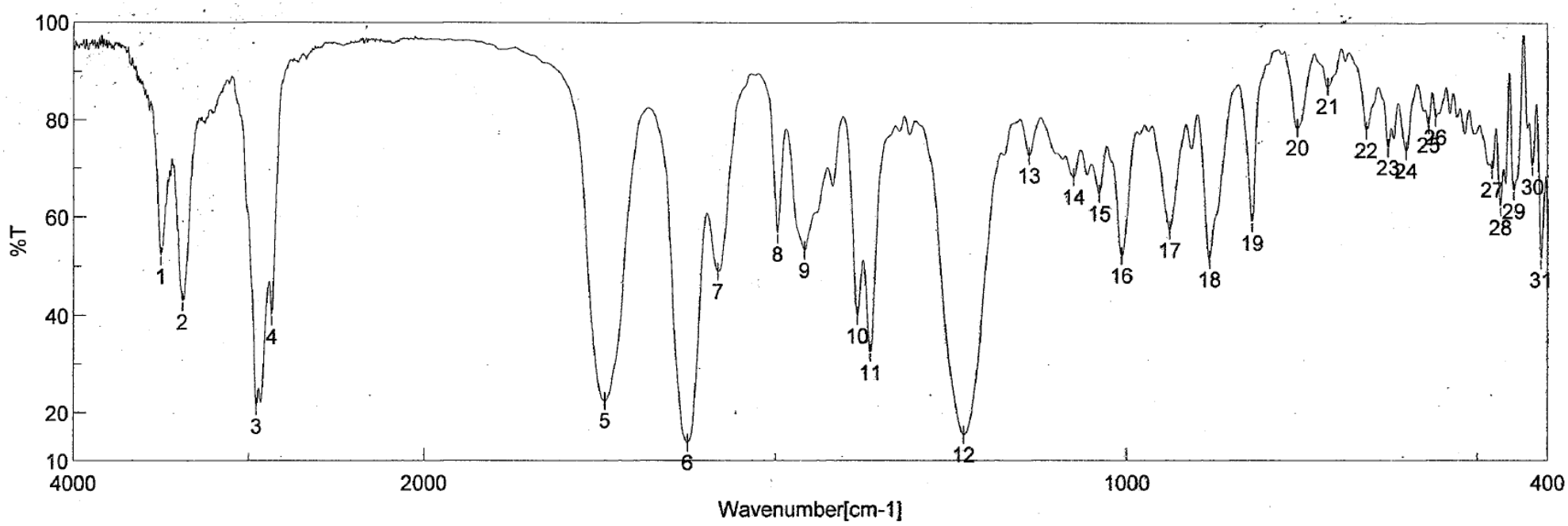

1H
BRUKER
Current Data Parameters
NAME A17mc219tf
EXPNO 18061201
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180612
Time 15.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
NH
H
N
OAc
H
14




```
Current Data Parameters
NAME                  Al7mc219tf
EXPNO                   19011001
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20190110
Time                      12.36
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          256
DS                            2
SWH                   29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010548 sec
RG                        126.99
DW                        16.800 usec
DE                        18.00 usec
TE                        300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                        12.00 usec
PLW1                 15.50000000 W
SFO1                100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     90.00 usec
PLW2                  5.19999981 W
PLW12                 0.14444000 W
PLW13                 0.11700000 W
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                           32768
SF                  100.6127690 MHz
WDW                             EM
SSB                0
LB                            2.00 Hz
GB                 0
PC                            1.40
```

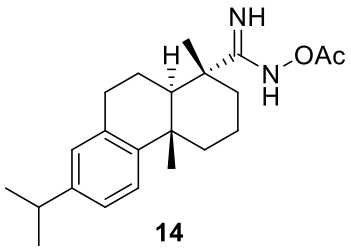


14

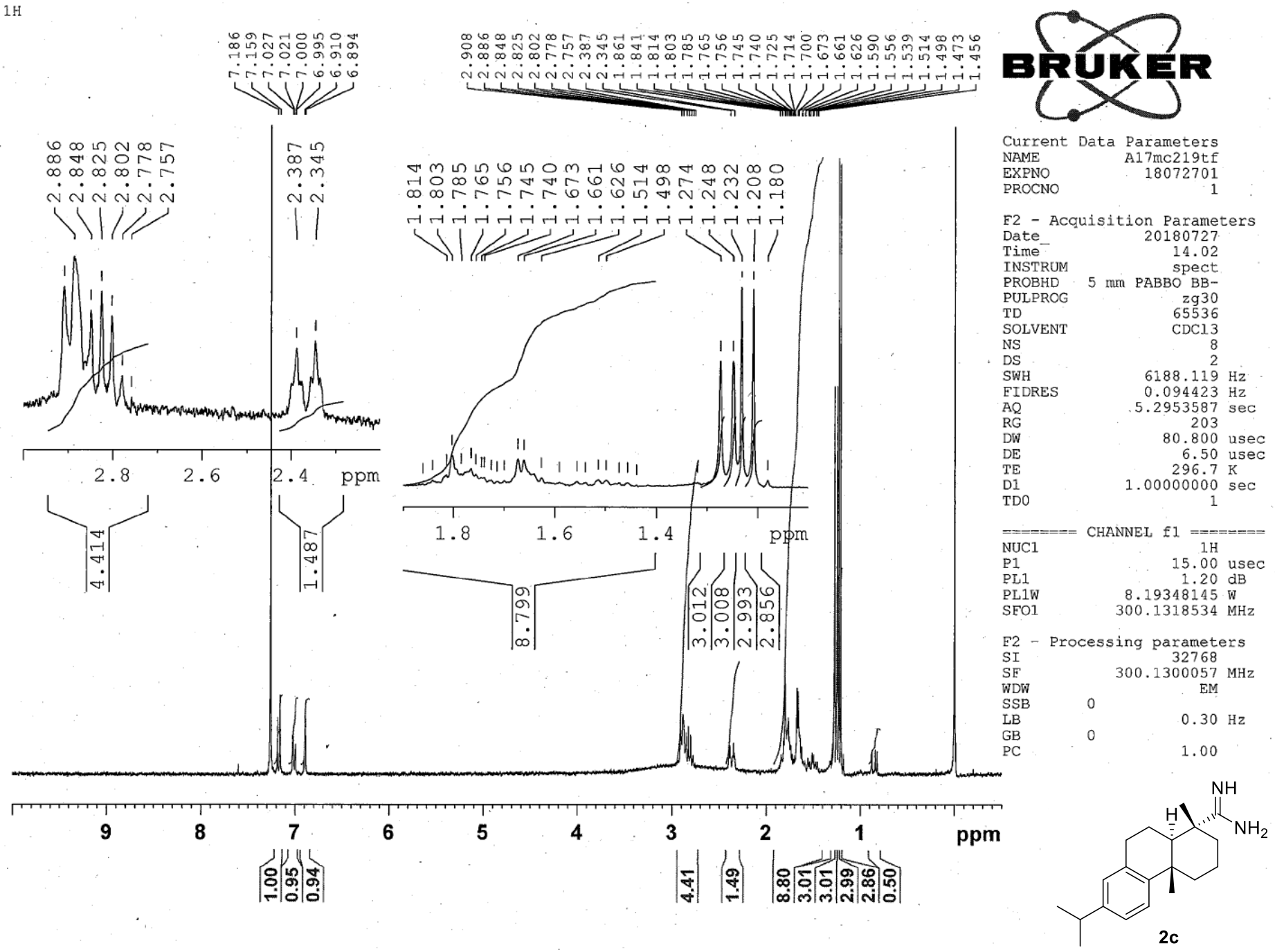


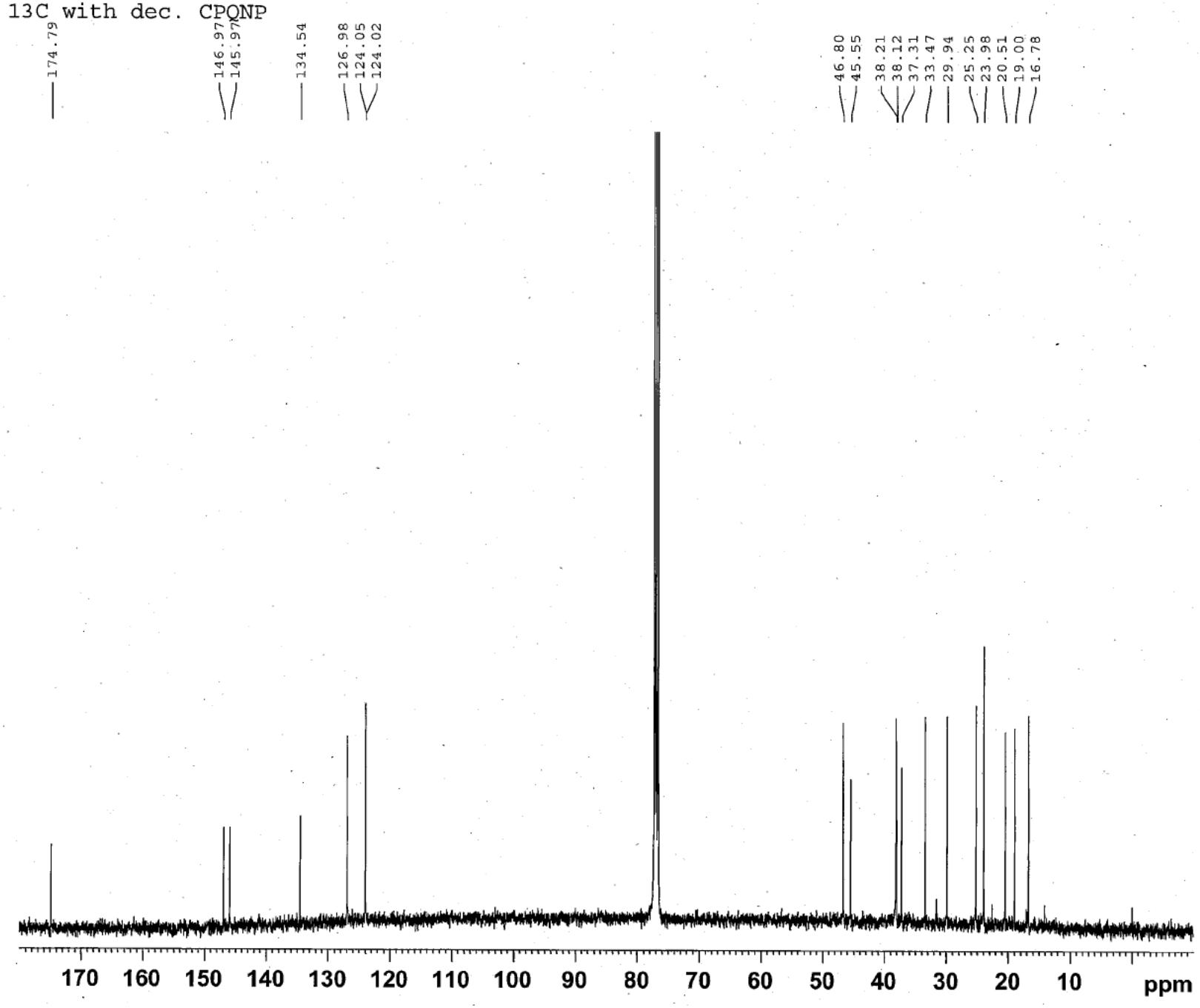
積算回数	4	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2018/06/12 17:45	更新日時	2018/06/12 17:46
測定者	takase		
ファイル名	Memory#2		
サンプル名	N-AcO-dehydroabietyl amidine		
コメント			

1:	3501.13,	52.07	2:	3377.71,	42.70	3:	2957.30,	21.25	4:	2868.59,	39.70
5:	1743.33,	22.37	6:	1625.70,	13.72	7:	1582.31,	49.04	8:	1497.45,	56.66
9:	1458.89,	53.37	10:	1383.68,	39.86	11:	1364.39,	32.31	12:	1231.33,	15.32
13:	1138.76,	72.66	14:	1076.08,	68.29	15:	1039.44,	64.88	16:	1007.62,	52.32
17:	939.16,	57.53	18:	882.27,	51.53	19:	821.53,	59.00	20:	756.92,	78.48
21:	713.53,	87.06	22:	658.57,	77.93	23:	627.72,	74.39	24:	601.68,	73.72
25:	569.86,	79.12	26:	560.22,	80.70	27:	479.22,	69.90	28:	467.65,	62.21
29:	448.37,	65.47	30:	421.37,	70.48	31:	409.80,	51.49			



14


1H
7.186
7.159
7.027
7.021
7.000
6.995
6.910
6.894
2.908
2.886
2.848
2.825
2.802
2.778
2.757
2.387
2.345
1.861
1.841
1.814
1.803
1.785
1.765
1.756
1.745
1.740
1.725
1.714
1.700
1.673
1.661
1.626
1.590
1.556
1.539
1.514
1.498
1.473
1.456
2.886
2.848
2.825
2.802
2.778
2.757
2.387
2.345
1.814
1.803
1.785
1.765
1.756
1.745
1.740
1.673
1.661
1.626
1.514
1.498
1.274
1.248
1.232
1.208
1.180
2.8
2.6
2.4
ppm
1.8
1.6
1.4
ppm
4.414
1.487
8.799
3.012
3.008
2.993
2.856
9
8
7
6
5
4
3
2
1
ppm
1.00
0.95
0.94
4.41
1.49
8.80
3.01
3.01
2.99
2.86
0.50
BRUKER
Current Data Parameters
NAME A17mc219tf
EXPNO 18072701
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180727
Time 14.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH
H
NH2
2c



BRUKER

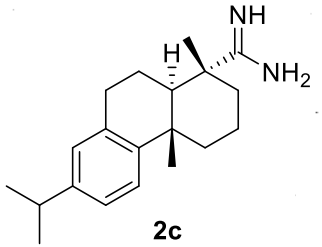
Current Data Parameters
NAME A17mc219tf
EXPNO 19011002
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190110
Time 12.52
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 126.99
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

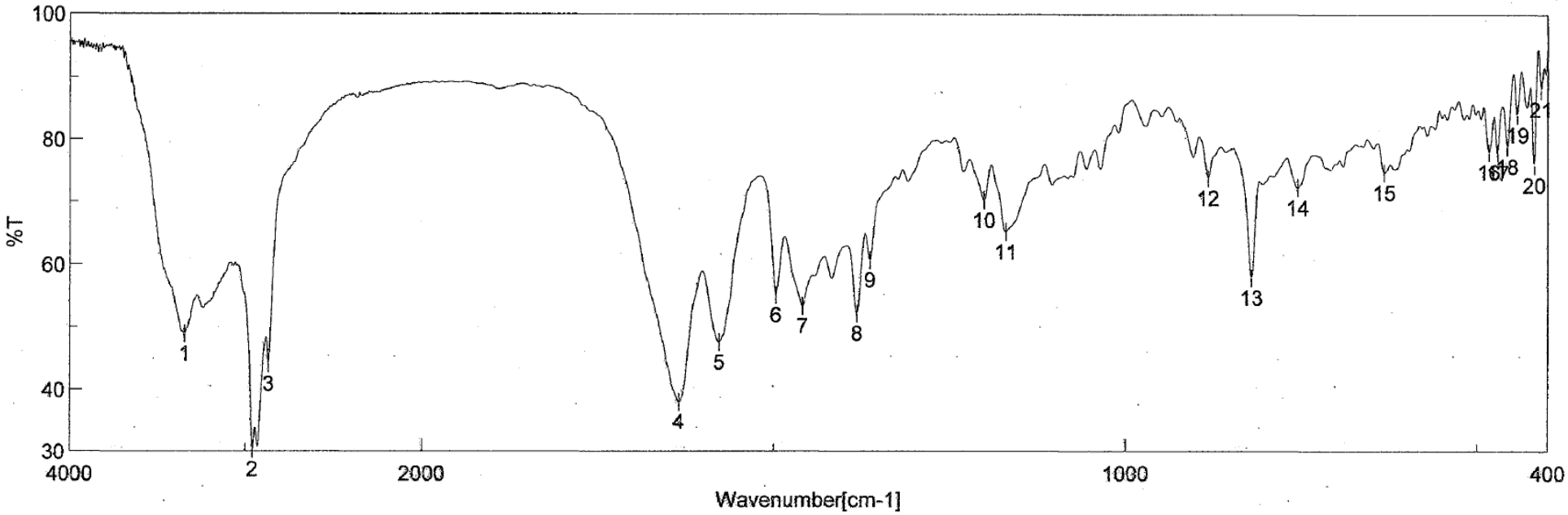
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PLW1 15.50000000 W
SFO1 100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 5.19999981 W
PLW12 0.14444000 W
PLW13 0.11700000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127681 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

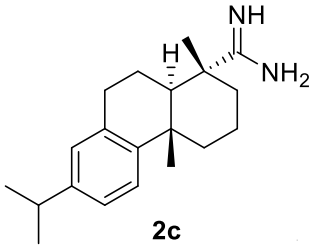


2c

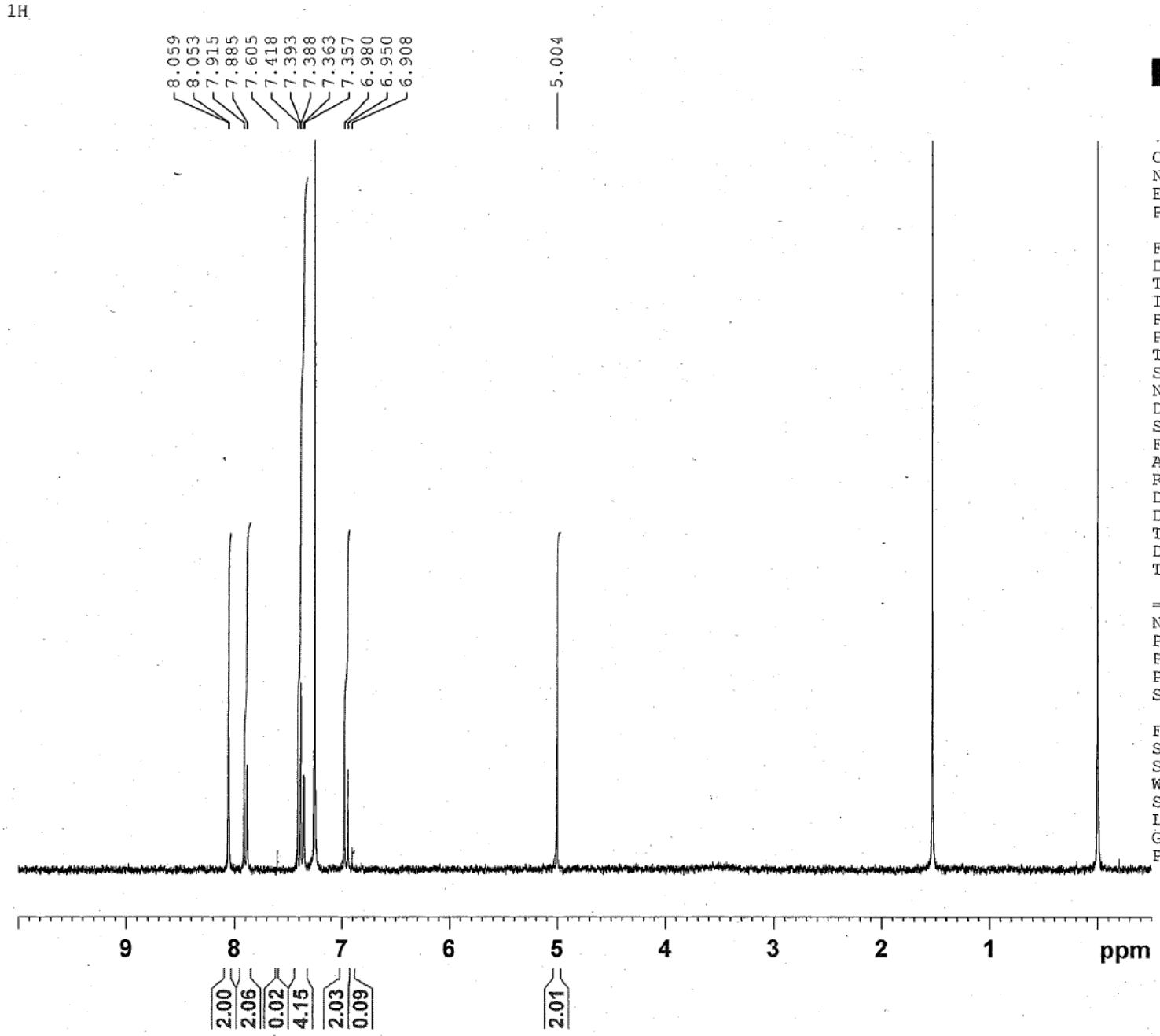


積算回数	16	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2018/07/25 15:11	更新日時	2018/07/25 15:11
測定者	takase		
ファイル名	Memory#2		
サンプル名	101, Dehydroabietyl amidine		
コメント			

1:	3344.93,	48.89	2:	2958.27,	30.33	3:	2868.59,	44.06	4:	1634.38,	37.97
5:	1577.49,	47.56	6:	1496.49,	55.13	7:	1458.89,	53.39	8:	1381.75,	52.19
9:	1363.43,	60.76	10:	1201.43,	70.32	11:	1170.58,	65.31	12:	883.24,	73.85
13:	821.53,	57.97	14:	755.96,	72.32	15:	632.54,	74.70	16:	484.05,	78.04
17:	471.51,	77.95	18:	458.01,	78.90	19:	443.55,	84.02	20:	419.44,	75.91
21:	409.80,	88.12									



2c



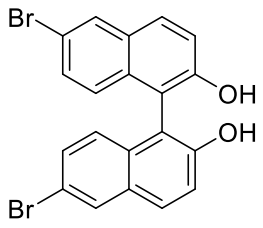
BRUKER

Current Data Parameters
NAME A17mc219tf
EXPNO 18092002
PROCNO 1

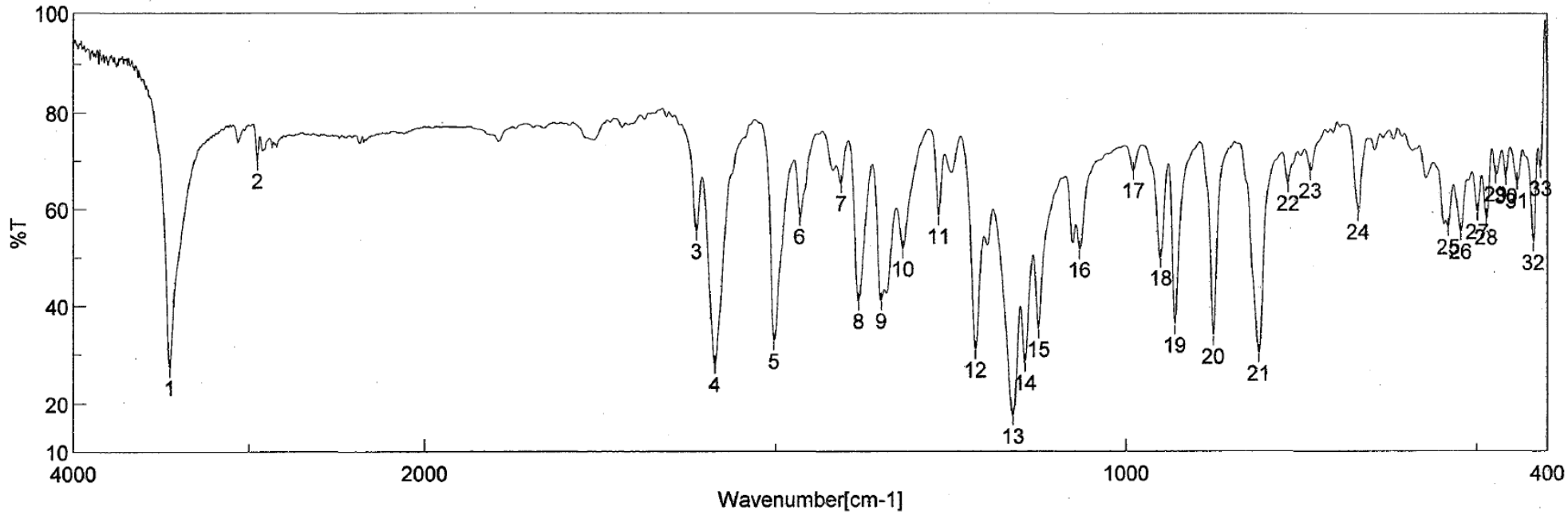
F2 - Acquisition Parameters
Date_ 20180920
Time 10.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

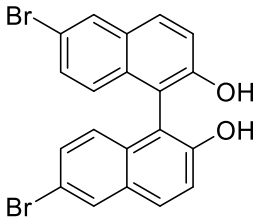


1b

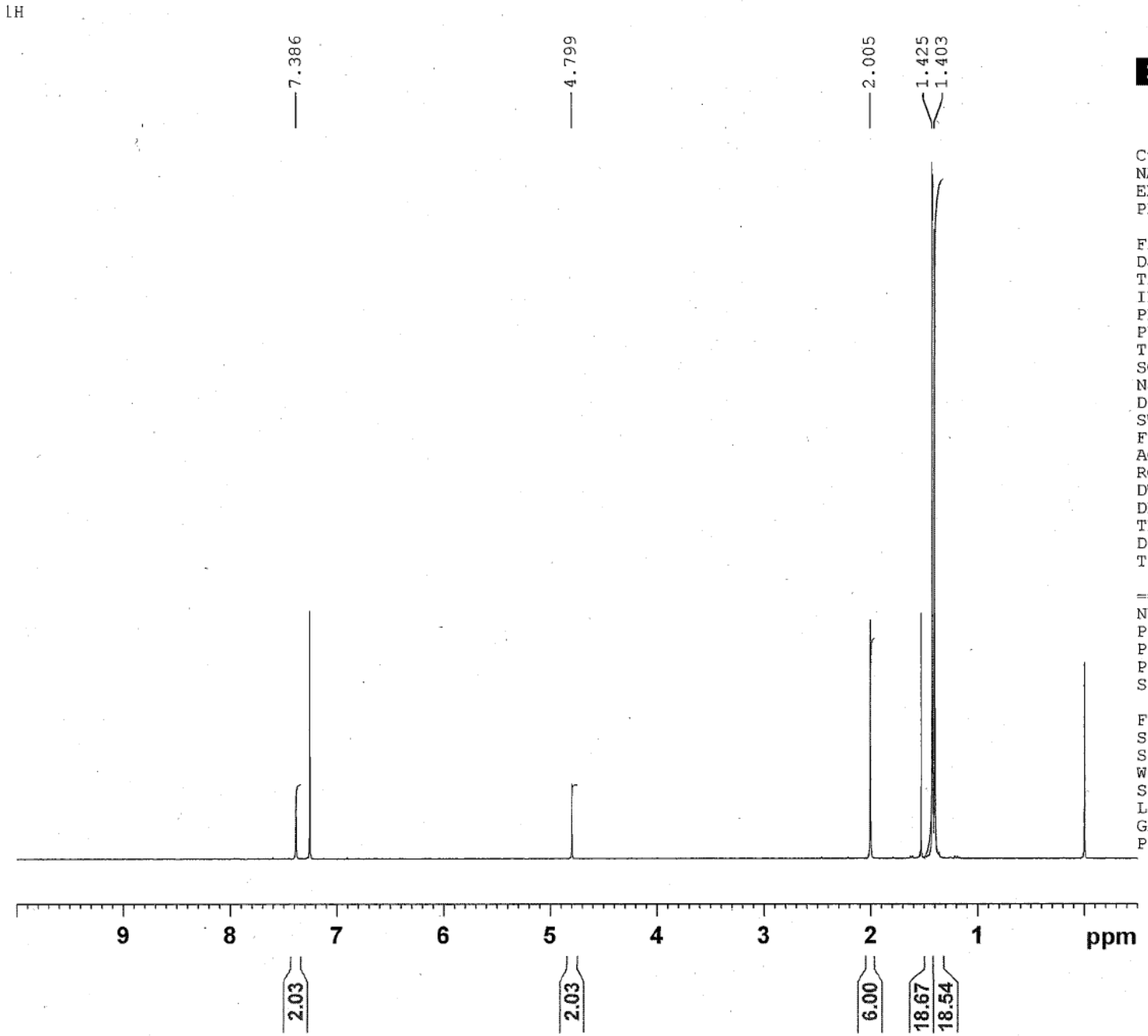


積算回数	8	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2019/01/26 12:52	更新日時	2019/01/26 12:52
測定者	takase		
ファイル名	Memory#2		
サンプル名	113, 6.6'-dibromo-1,1'-bi-2-naphthol		
コメント			

1:	3450.99,	27.31	2:	2952.48,	69.93	3:	1612.20,	55.39	4:	1586.16,	27.97
5:	1502.28,	32.70	6:	1465.63,	58.48	7:	1406.82,	65.23	8:	1381.75,	40.93
9:	1349.93,	40.96	10:	1319.07,	51.74	11:	1267.97,	58.55	12:	1215.90,	30.94
13:	1161.90,	17.26	14:	1144.55,	28.30	15:	1125.26,	35.17	16:	1066.44,	51.57
17:	989.30,	67.94	18:	950.73,	49.86	19:	930.49,	36.02	20:	875.52,	33.71
21:	810.92,	30.26	22:	769.46,	65.25	23:	737.64,	67.89	24:	670.14,	59.40
25:	540.93,	56.33	26:	523.58,	55.37	27:	499.47,	59.45	28:	486.94,	58.33
29:	473.44,	67.14	30:	458.98,	66.77	31:	443.55,	65.77	32:	420.41,	53.07
33:	409.80,	68.30									



1b

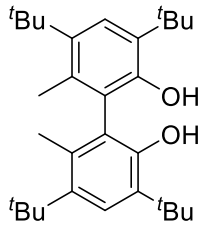


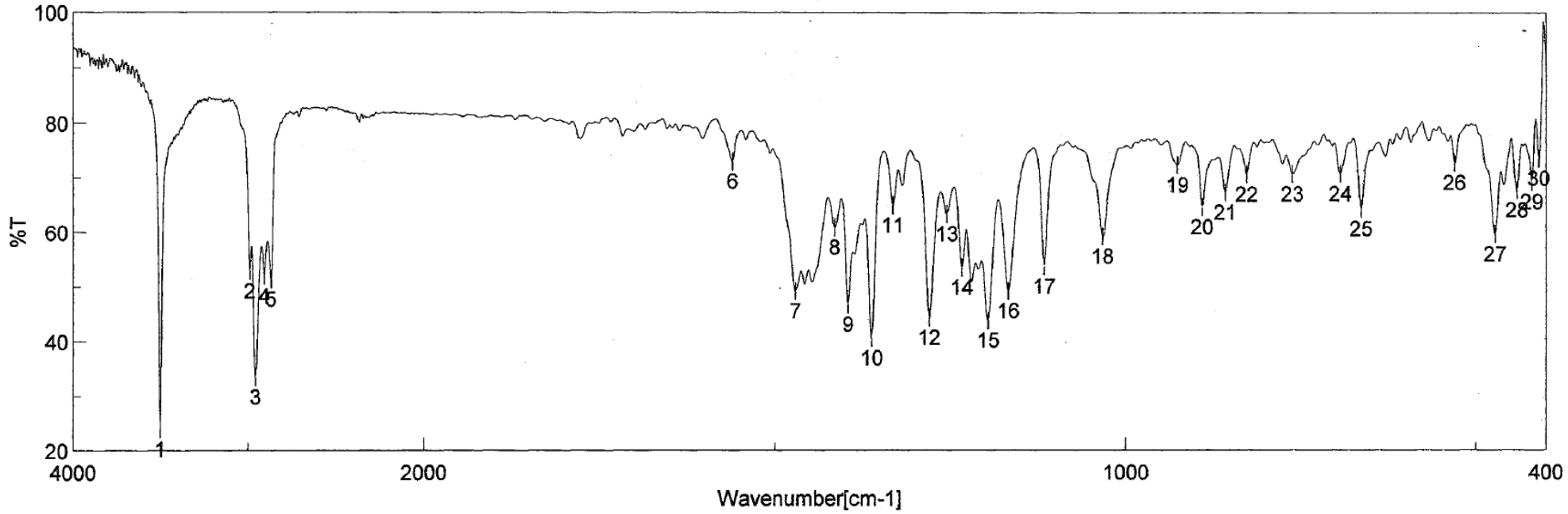
Current Data Parameters
NAME A17mc219tf
EXPNO 18100301
PROCNO 1

F2 - Acquisition Parameters
Date_ 20181003
Time 11.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 203
DW 80.800 usec
DE 6.50 usec
TE 299.5 K
D1 1.00000000 sec
TD0 1

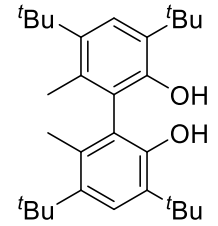
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 1.20 dB
PL1W 8.19348145 W
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

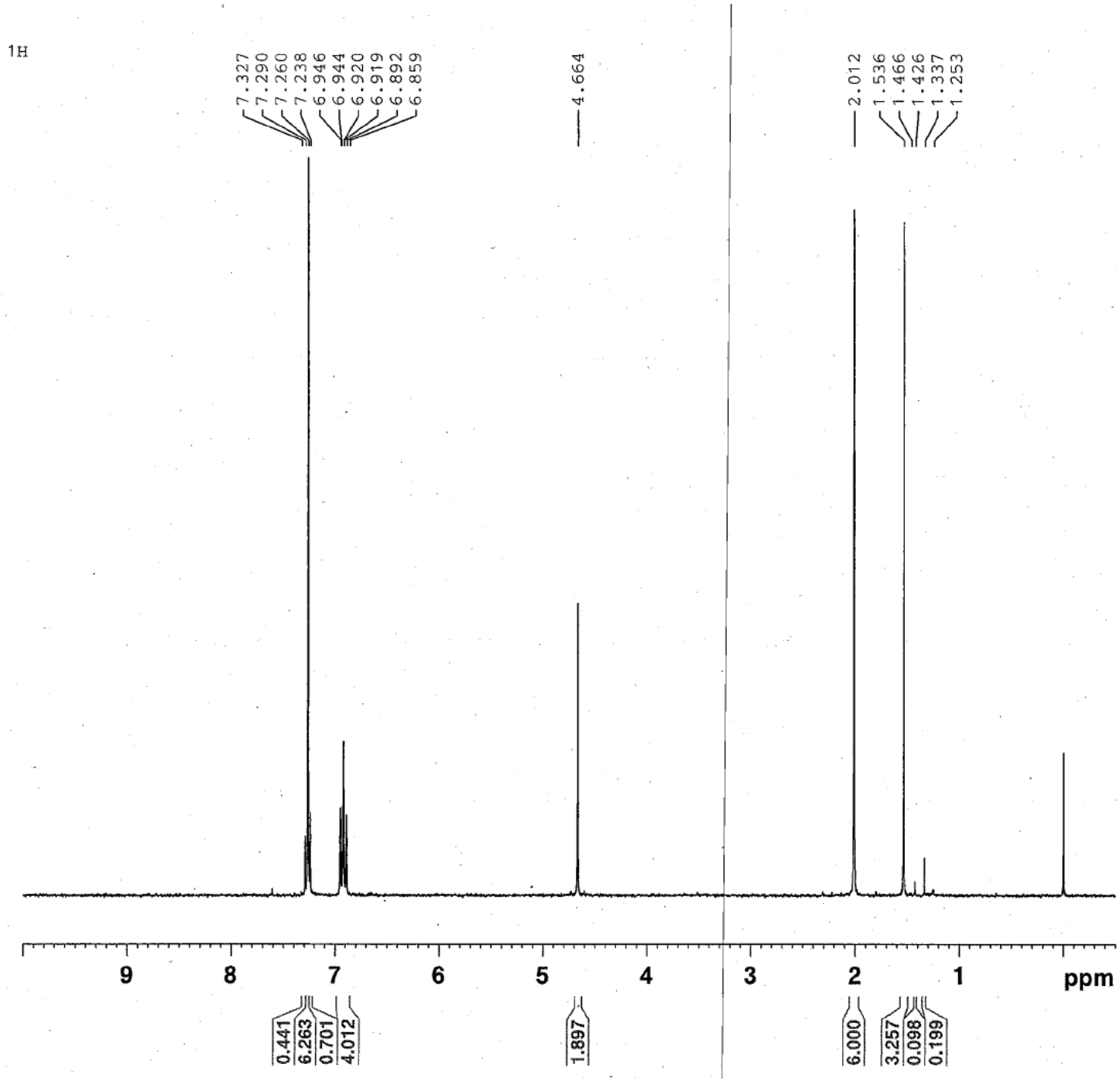




積算回数	8	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2019/01/26 13:11	更新日時	2019/01/26 13:11
測定者	takase		
ファイル名	Memory#2		
サンプル名	116, 3,3',5,5'-tetra-tert-Bu-6,6'-dimethyl-2,2'-bisphenol		
コメント			

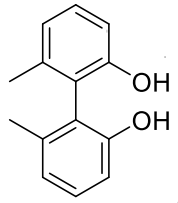


1:	3504.02,	23.95	2:	2991.05,	52.91	3:	2959.23,	33.50	4:	2909.09,	52.00
5:	2870.52,	51.45	6:	1560.13,	73.03	7:	1470.46,	49.24	8:	1414.53,	60.94
9:	1395.25,	46.80	10:	1361.50,	40.61	11:	1331.61,	65.04	12:	1279.54,	44.36
13:	1254.47,	63.40	14:	1233.25,	53.55	15:	1195.65,	43.85	16:	1166.72,	49.29
17:	1115.62,	53.81	18:	1032.69,	59.31	19:	926.63,	72.41	20:	890.95,	64.71
21:	858.17,	67.41	22:	827.31,	70.71	23:	761.74,	70.77	24:	693.28,	70.77
25:	663.39,	64.37	26:	530.33,	72.83	27:	472.47,	59.75	28:	441.62,	67.87
29:	420.41,	69.35	30:	410.76,	73.62						

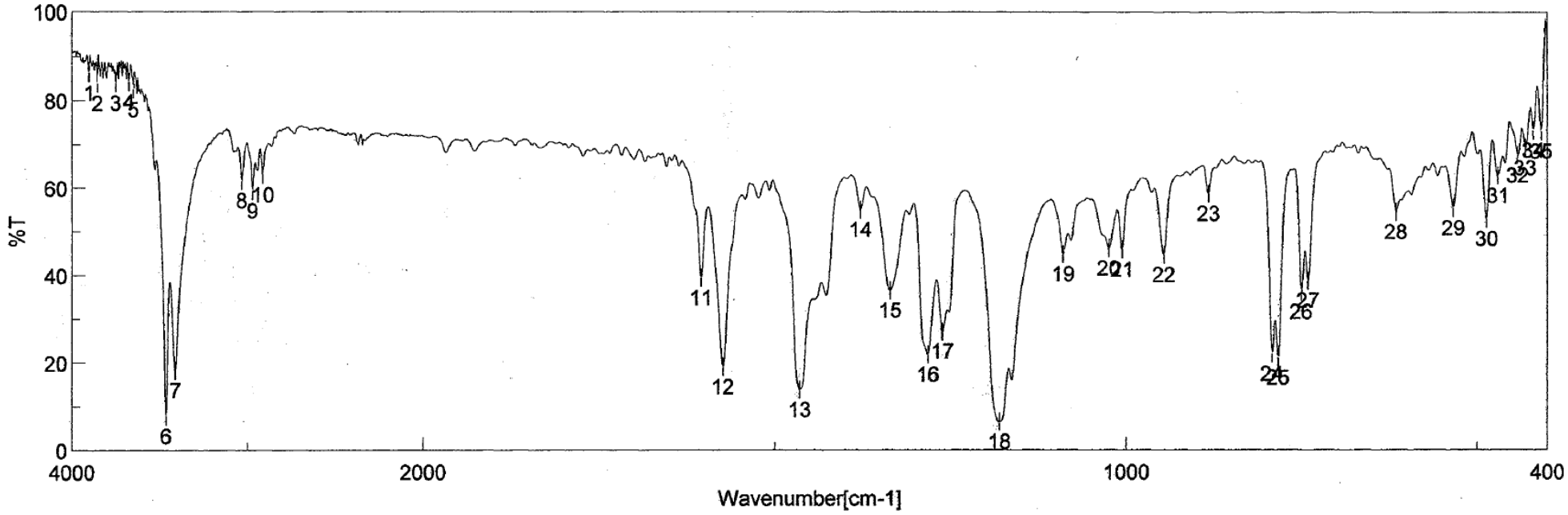


BRUKER

NAME	A17mc219tf
EXPNO	18101201
PROCNO	1
Date_	20181012
Time	13.38
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	6188.119 Hz
FIDRES	0.094423 Hz
AQ	5.2953587 sec
RG	203
DW	80.800 usec
DE	6.50 usec
TE	297.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	1.20 dB
PL1W	8.19348145 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

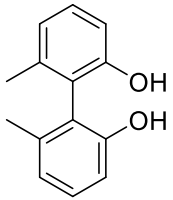


1c

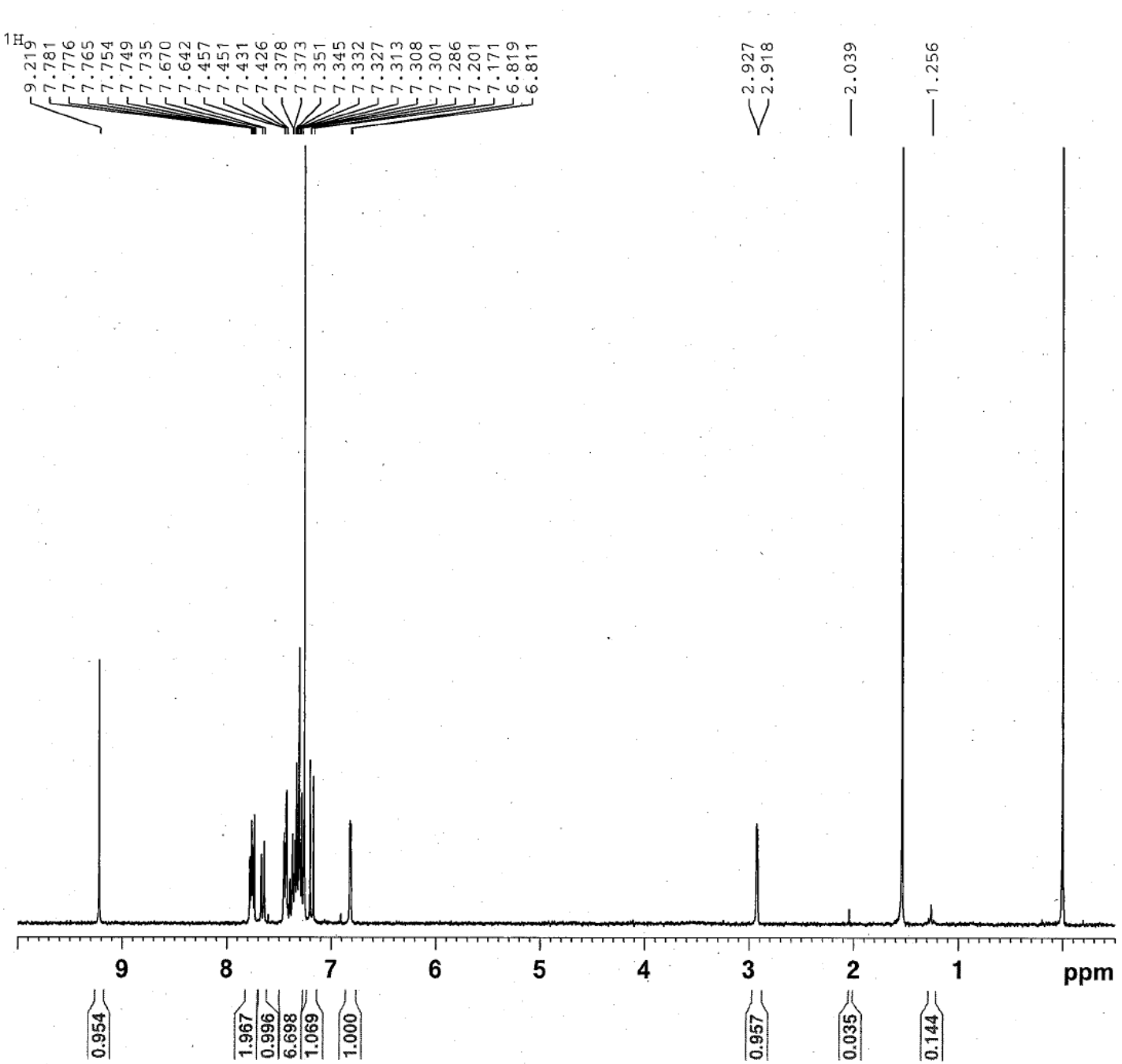


積算回数	8	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2019/01/26 13:29	更新日時	2019/01/26 13:29
測定者	takase		
ファイル名	Memory#2		
サンプル名	120, 6,6'-dimethyl-2,2'-bisphenol		
コメント			

1:	3904.18,	86.25	2:	3855.01,	83.84	3:	3752.80,	83.90	4:	3677.59,	84.21
5:	3650.59,	82.50	6:	3464.49,	7.77	7:	3413.39,	18.29	8:	3034.44,	61.71
9:	2972.73,	59.36	10:	2914.88,	63.04	11:	1605.45,	39.46	12:	1574.59,	19.06
13:	1464.67,	13.81	14:	1377.89,	55.16	15:	1335.46,	36.63	16:	1281.47,	21.89
17:	1261.22,	27.16	18:	1180.22,	6.51	19:	1089.58,	44.96	20:	1024.98,	46.32
21:	1005.70,	46.00	22:	946.88,	44.81	23:	883.24,	58.86	24:	792.60,	22.07
25:	783.92,	21.16	26:	751.14,	36.28	27:	741.50,	38.90	28:	616.15,	54.69
29:	534.19,	55.62	30:	486.94,	53.09	31:	470.55,	62.99	32:	442.58,	67.61
33:	431.98,	69.32	34:	420.41,	73.30	35:	408.83,	73.07			

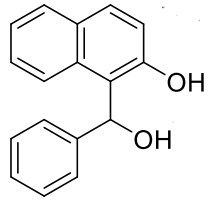


1c

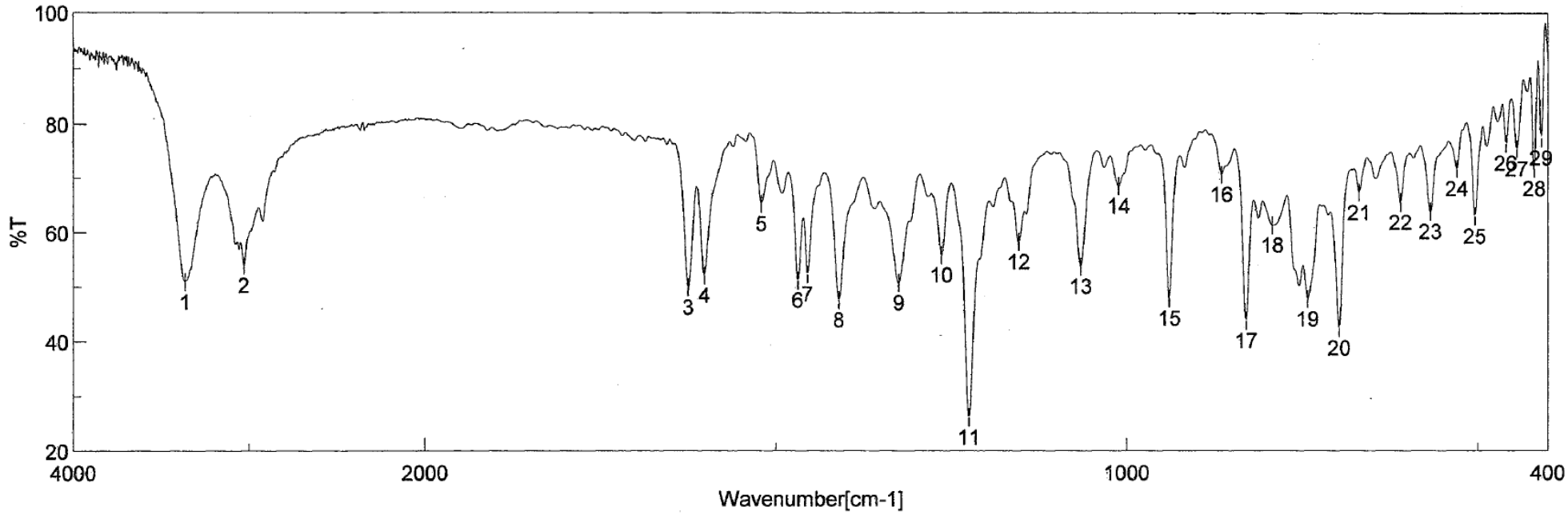


BRUKER

NAME	A17mc219tf
EXPNO	18091801
PROCNO	1
Date_	20180918
Time	16.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	6188.119 Hz
FIDRES	0.094423 Hz
AQ	5.2953587 sec
RG	203
DW	80.800 usec
DE	6.50 usec
TE	301.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	1.20 dB
PL1W	8.19348145 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300068 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

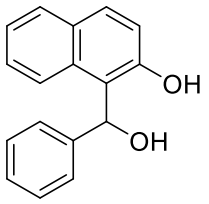


15

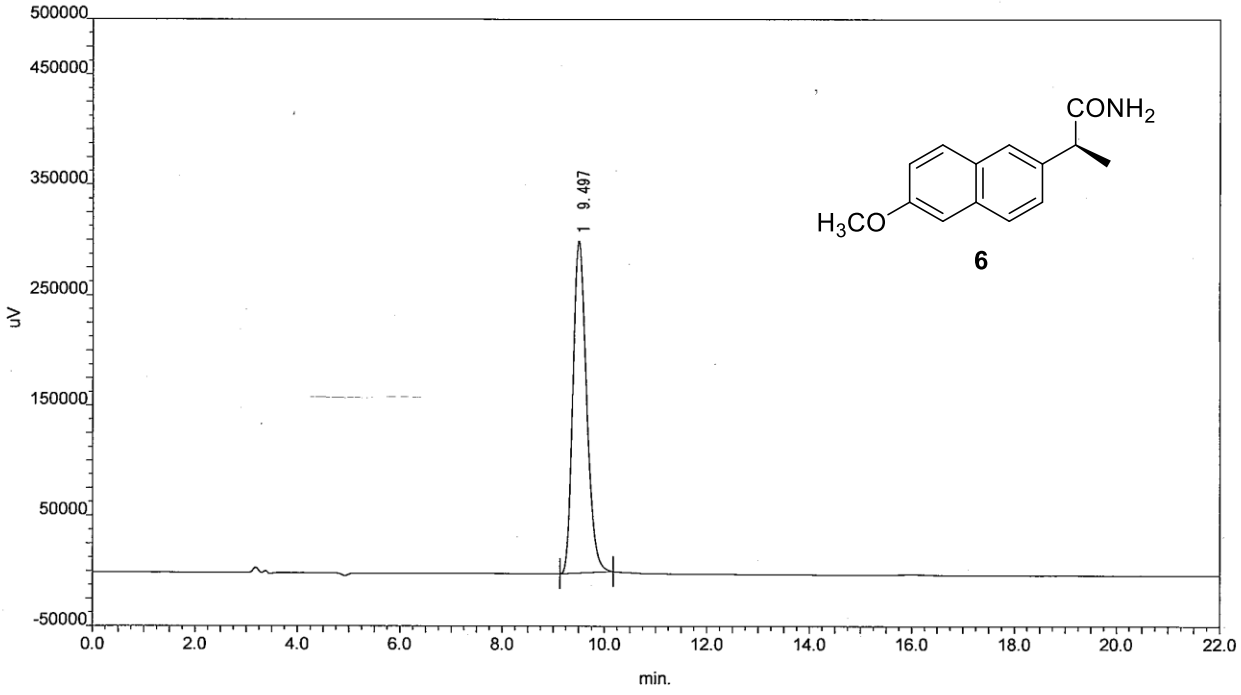


積算回数	8	分解	4 cm-1
ゼロフィリング	ON	アポダイゼーション	Cosine
ゲイン	16	スキャンスピード	2 mm/sec
測定日時	2019/01/26 12:38	更新日時	2019/01/26 12:38
測定者	takase		
ファイル名	Memory#2		
サンプル名	111, 1-[hydroxy(phenyl)methyl]-2-naphthol		
コメント			

1:	3363.25,	50.92	2:	3028.66,	54.00	3:	1624.73,	50.01	4:	1601.59,	52.19
5:	1520.60,	65.37	6:	1468.53,	51.29	7:	1455.03,	52.40	8:	1410.67,	47.68
9:	1325.82,	50.82	10:	1265.07,	55.94	11:	1225.54,	26.19	12:	1154.19,	58.40
13:	1065.48,	53.84	14:	1011.48,	68.62	15:	939.16,	48.01	16:	864.92,	70.71
17:	830.21,	43.85	18:	792.60,	61.45	19:	742.46,	47.87	20:	697.14,	42.51
21:	669.18,	67.39	22:	610.36,	65.62	23:	567.93,	63.95	24:	530.33,	71.78
25:	504.29,	63.07	26:	459.94,	76.45	27:	444.51,	75.43	28:	419.44,	71.79
29:	409.80,	77.44									



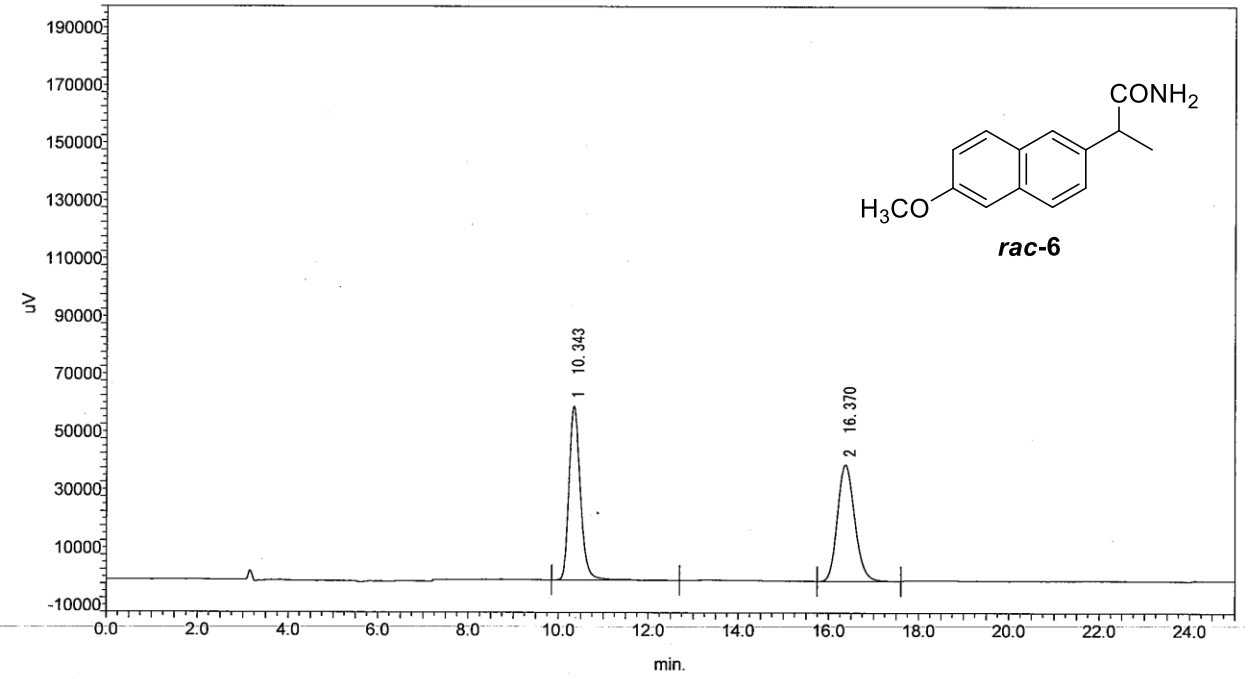
15



6

解析結果

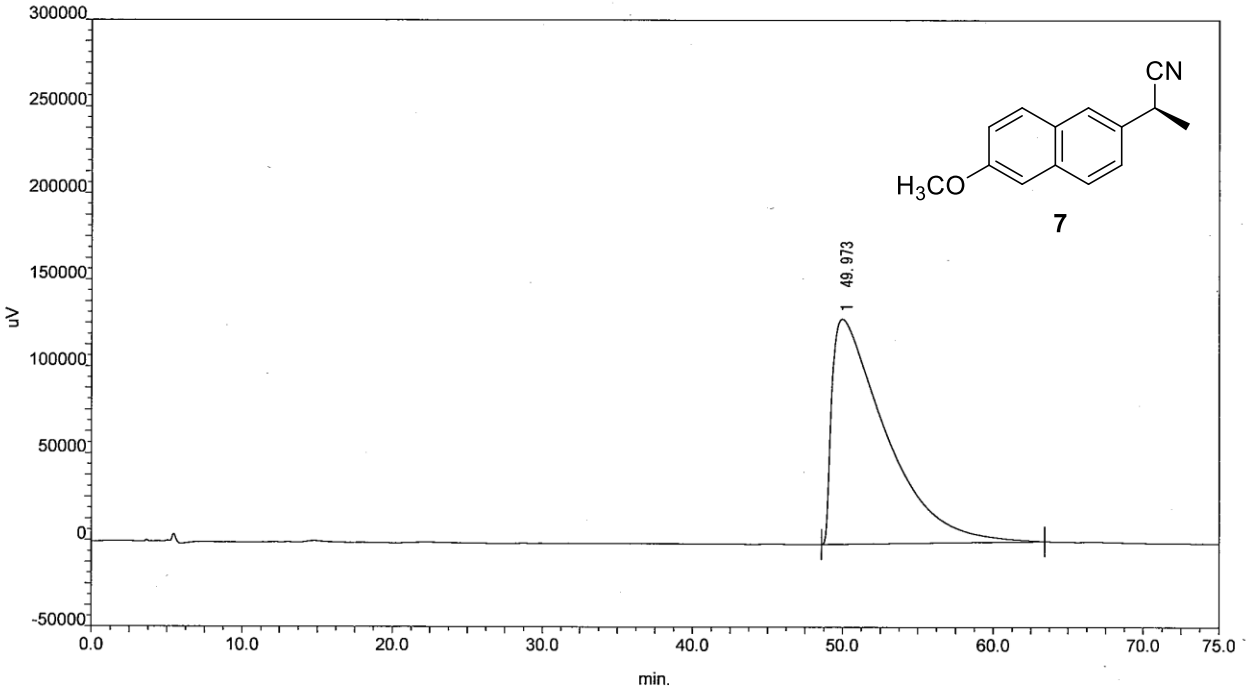
No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	9.50		5548002.100	100.0000	300720	5827.0	1.278	------
			5548002.100	100.0000	300720			



***rac*-6**

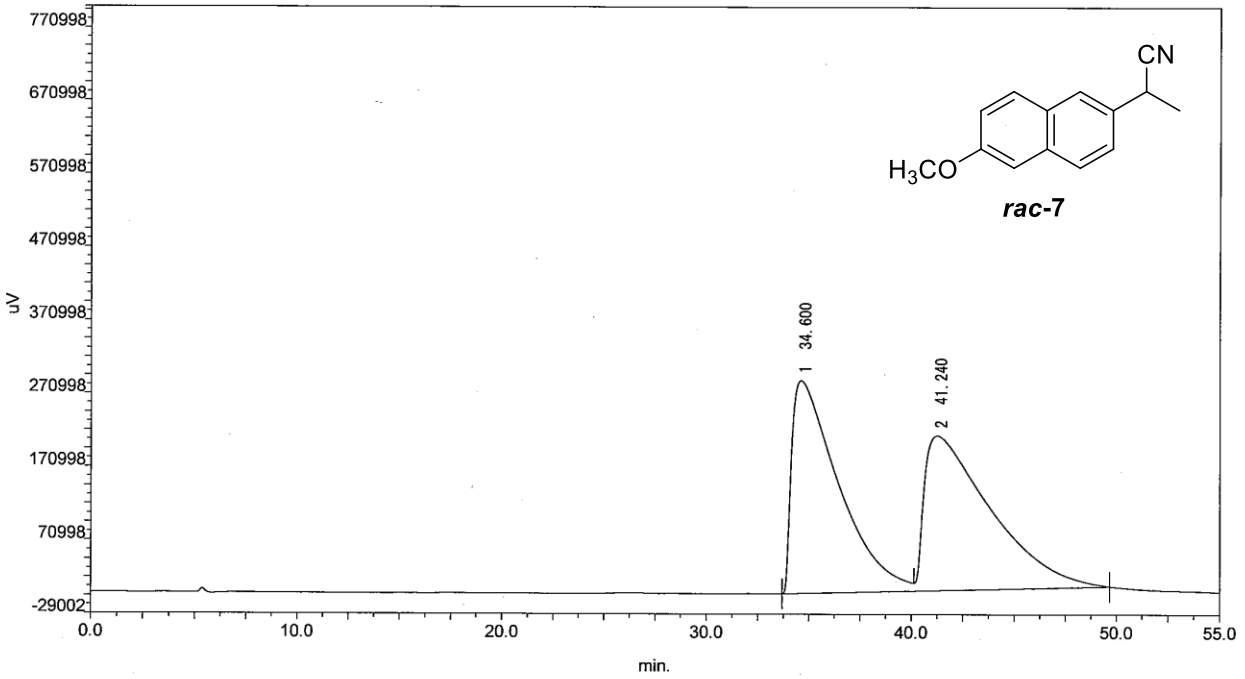
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	10.34		1066726.100	49.6836	59930	7825.4	1.274	10.166
2	16.37		1080312.500	50.3164	40211	8317.4	1.189	------
			2147038.600	100.0000	100141			



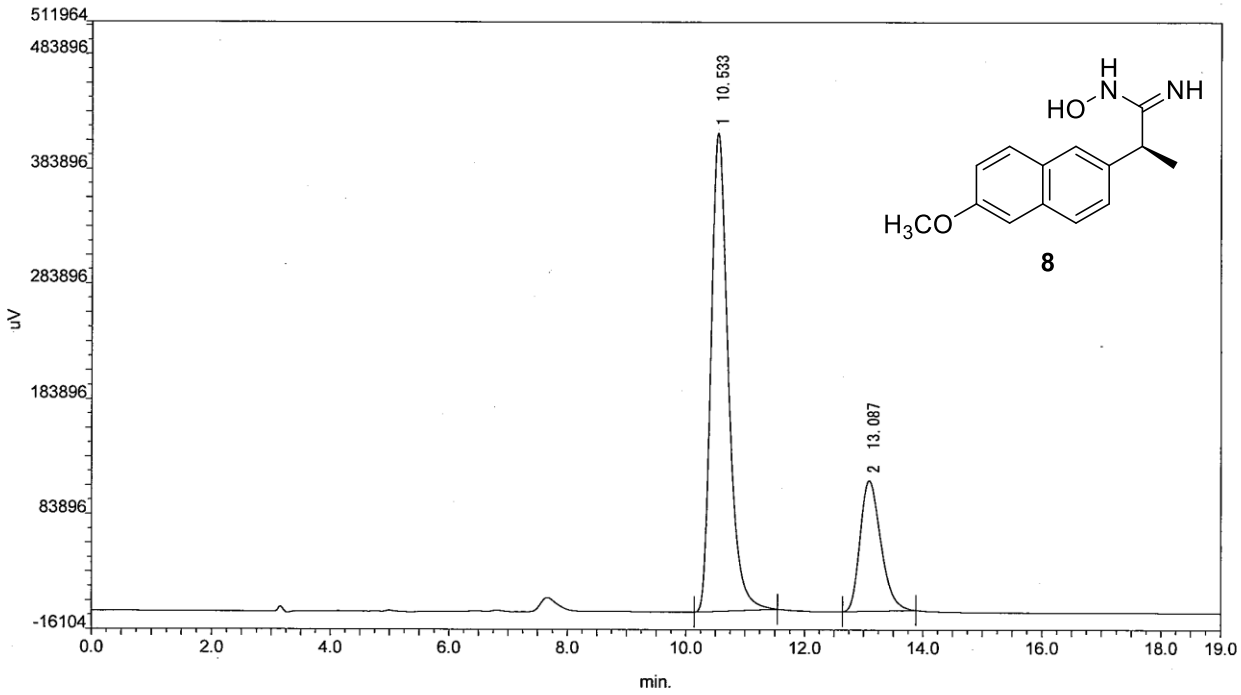
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	49.97		32233938.000	100.0000	129420	821.5	4.172	------
			32233938.000	100.0000	129420			



解析結果

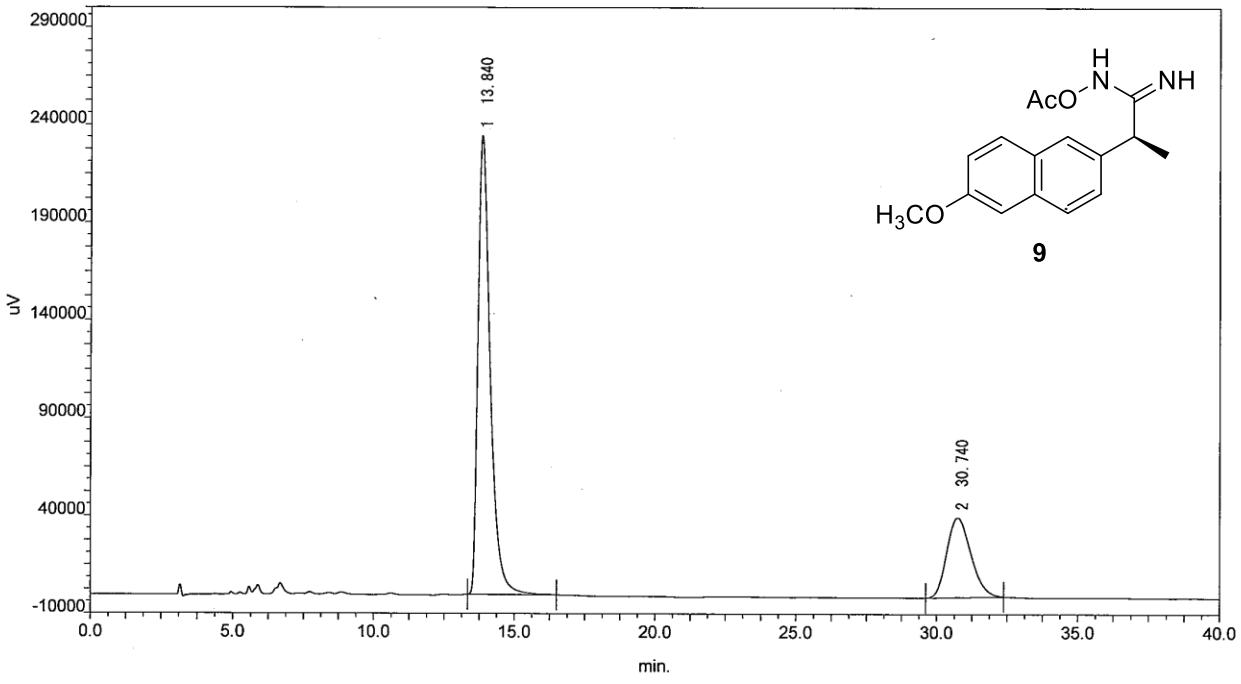
No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	34.60		46248486.110	49.4895	289587	1061.7	3.970	1.298
2	41.24		47202542.290	50.5105	209930	760.9	------	------
			93451028.400	100.0000	499517			



8

解析結果

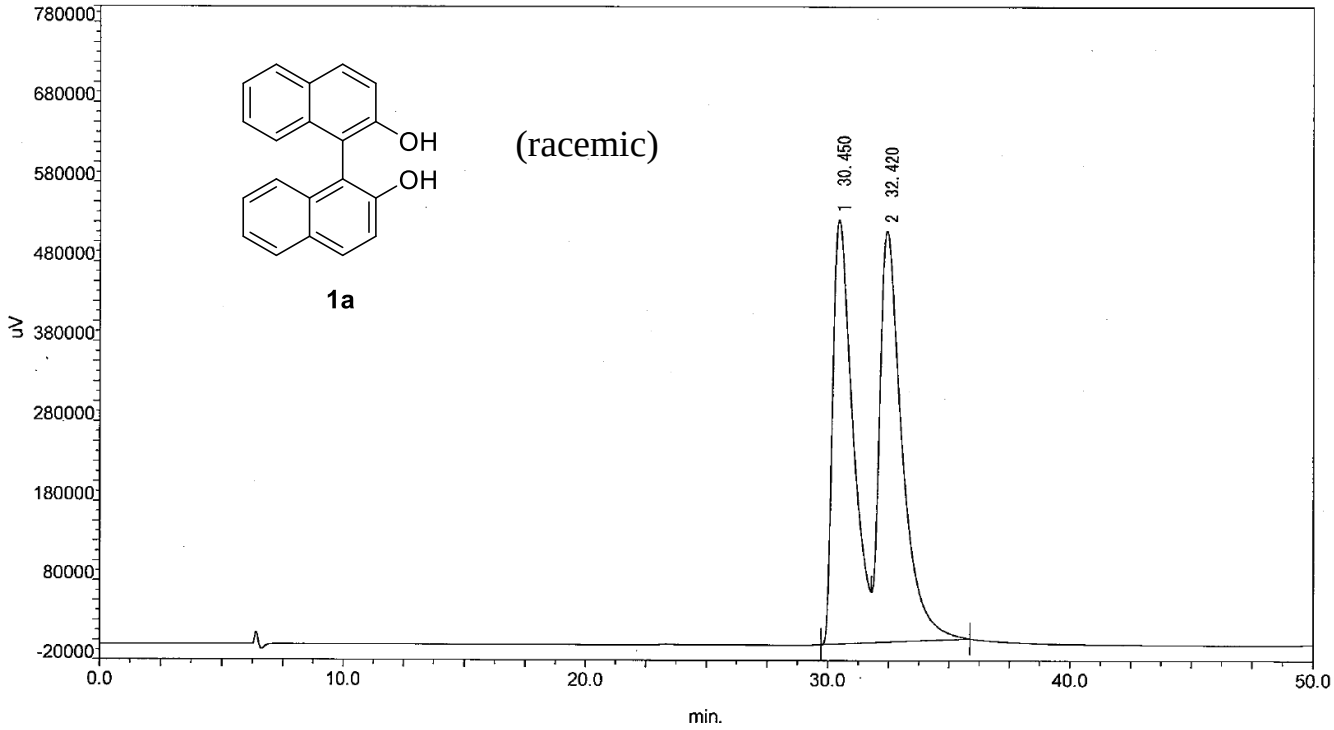
No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	10.53		8446919.800	75.3784	415545	5864.6	1.422	4.229
2	13.09		2759095.500	24.6216	113326	6340.4	1.308	------
			11206015.300	100.0000	528871			



9

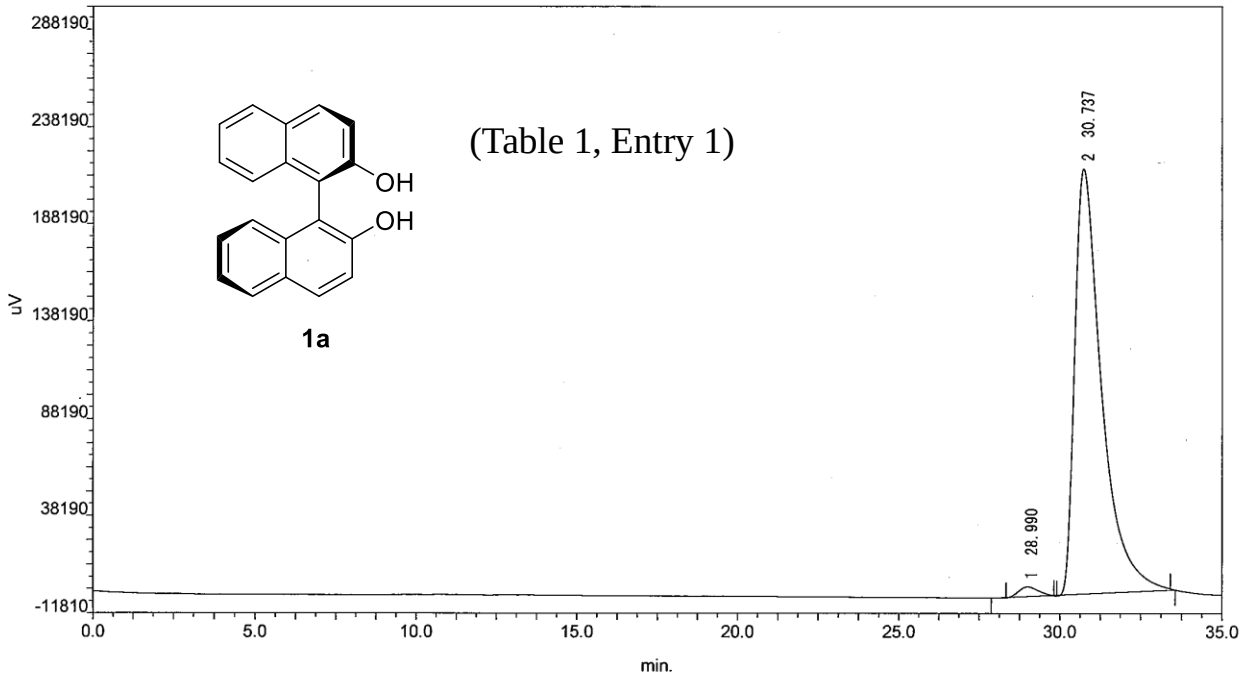
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	13.84		7175027.600	74.6401	234585	4458.9	1.731	13.879
2	30.74		2437800.100	25.3599	40839	5859.8	1.231	------
			9612827.700	100.0000	275424			



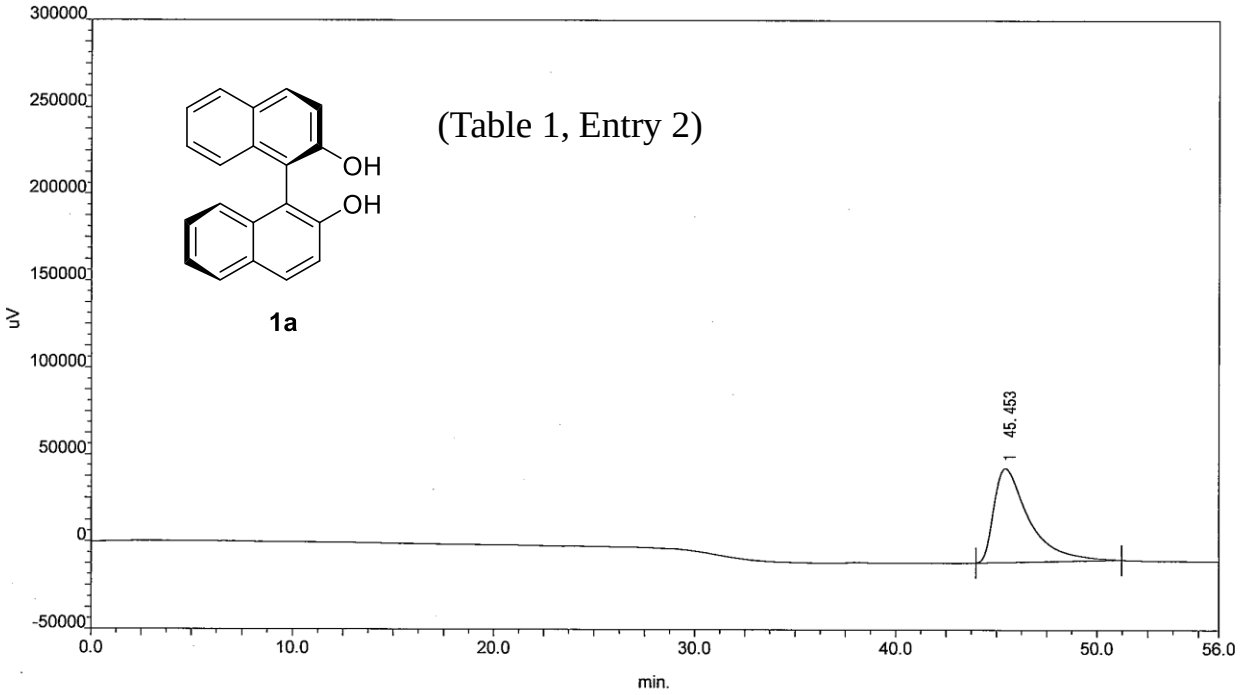
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	30.45		30234001.634	47.3348	531894	6491.1	------	1.214
2	32.42		33638722.366	52.6652	515685	5587.3	------	------
			63872724.000	100.0000	1047579			



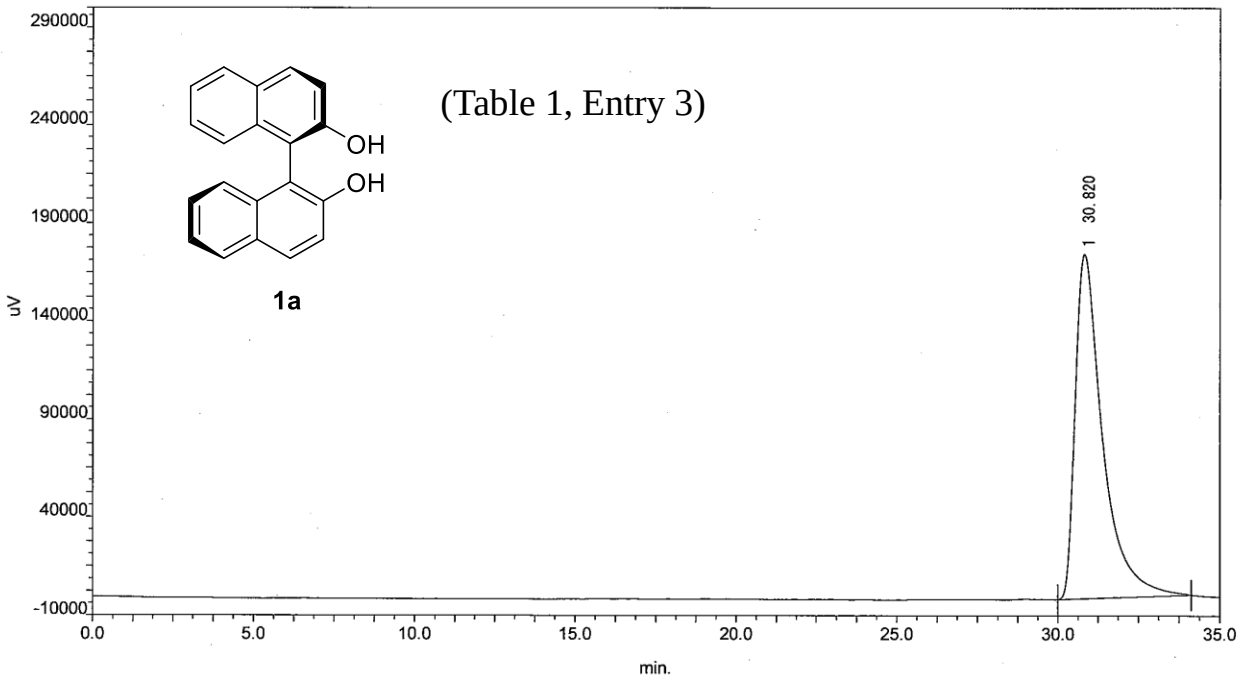
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	28.99		201816.556	1.5725	4997	11654.2	1.212	1.338
2	30.74		12632613.611	98.4275	218594	6398.6	2.002	------
			12834430.167	100.0000	223591			



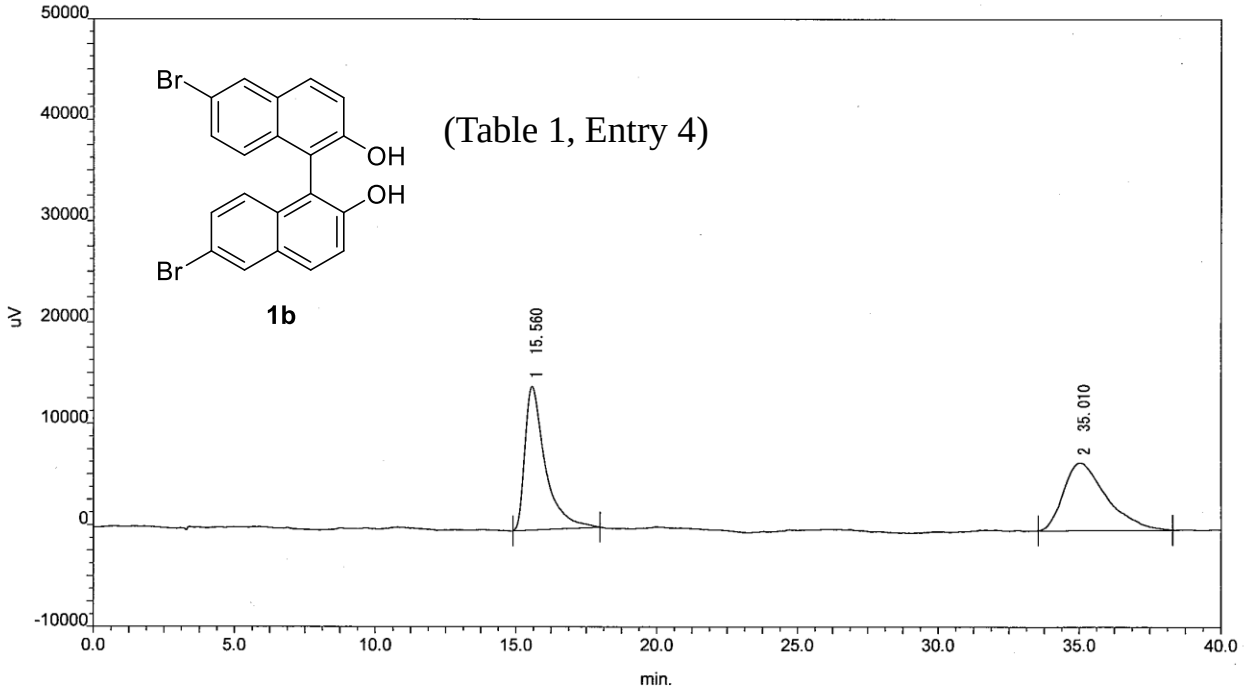
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	45.45		6542971.600	100.0000	53890	2780.0	2.138	------
			6542971.600	100.0000	53890			



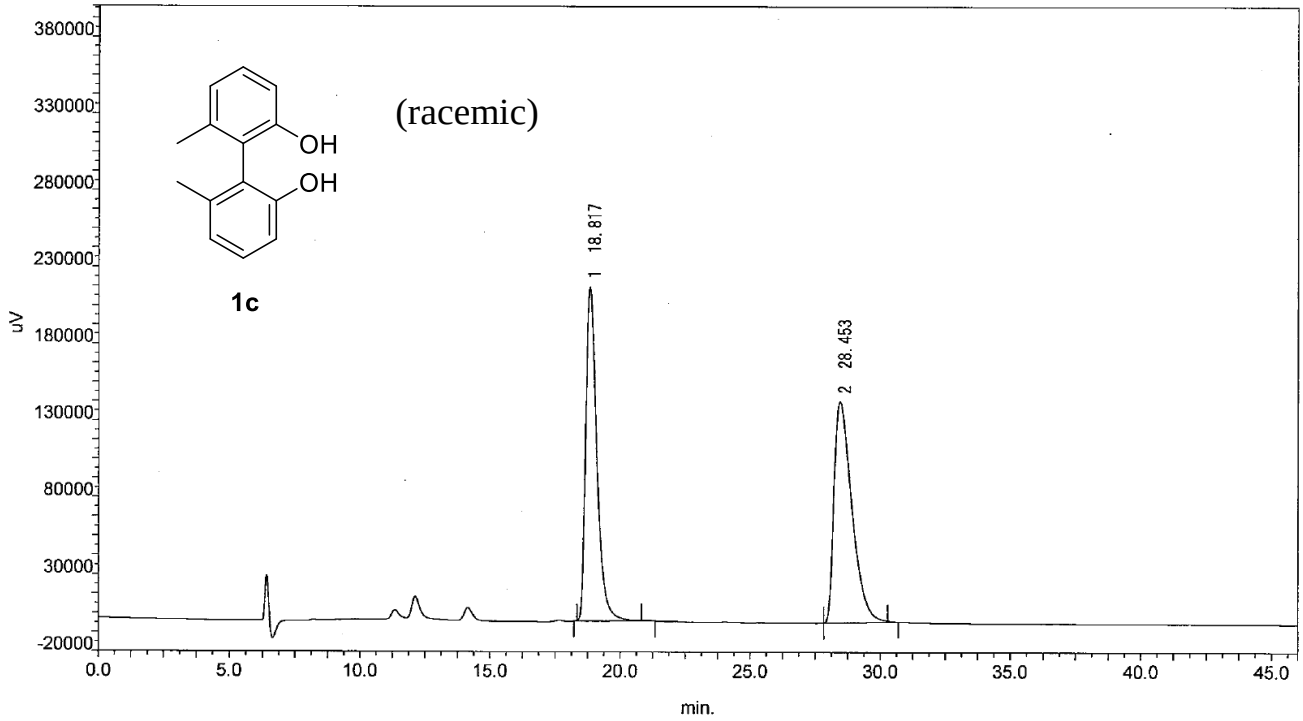
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	30.82		10442350.600	100.0000	175721	5574.5	2.029	------
			10442350.600	100.0000	175721			



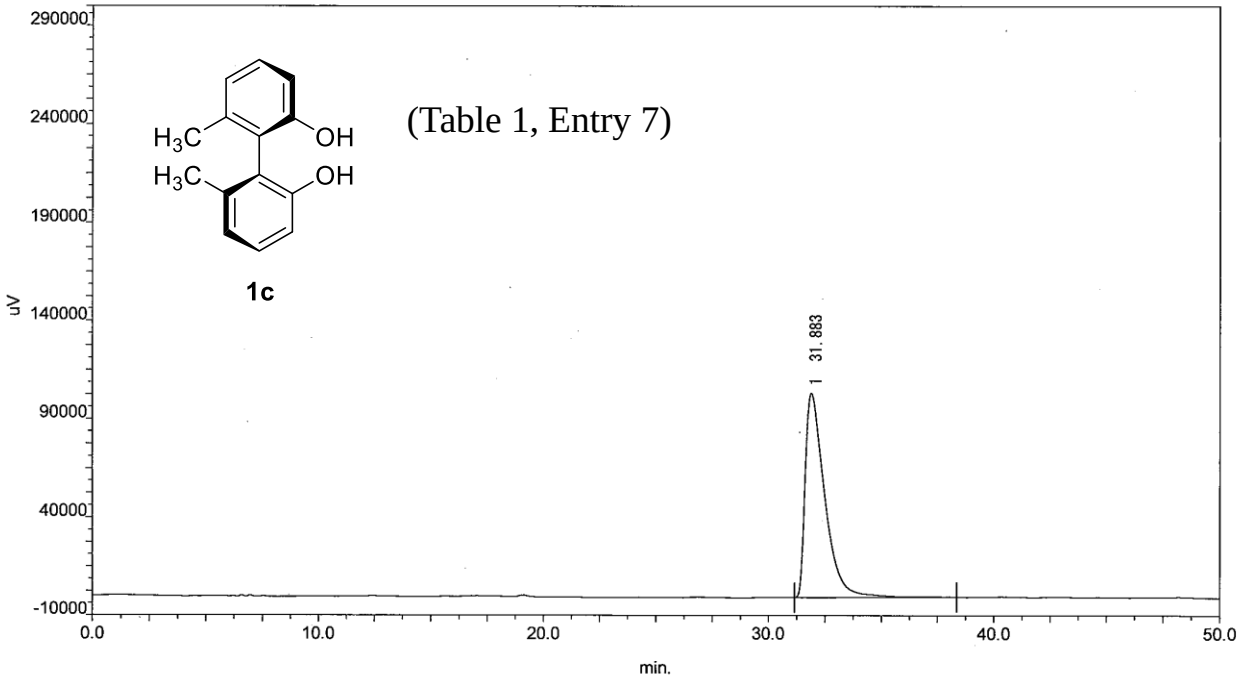
解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	15.56		713262.600	49.9847	14169	1854.8	2.001	8.582
2	35.01		713700.000	50.0153	6686	2057.3	1.538	------
			1426962.600	100.0000	20855			



解析結果

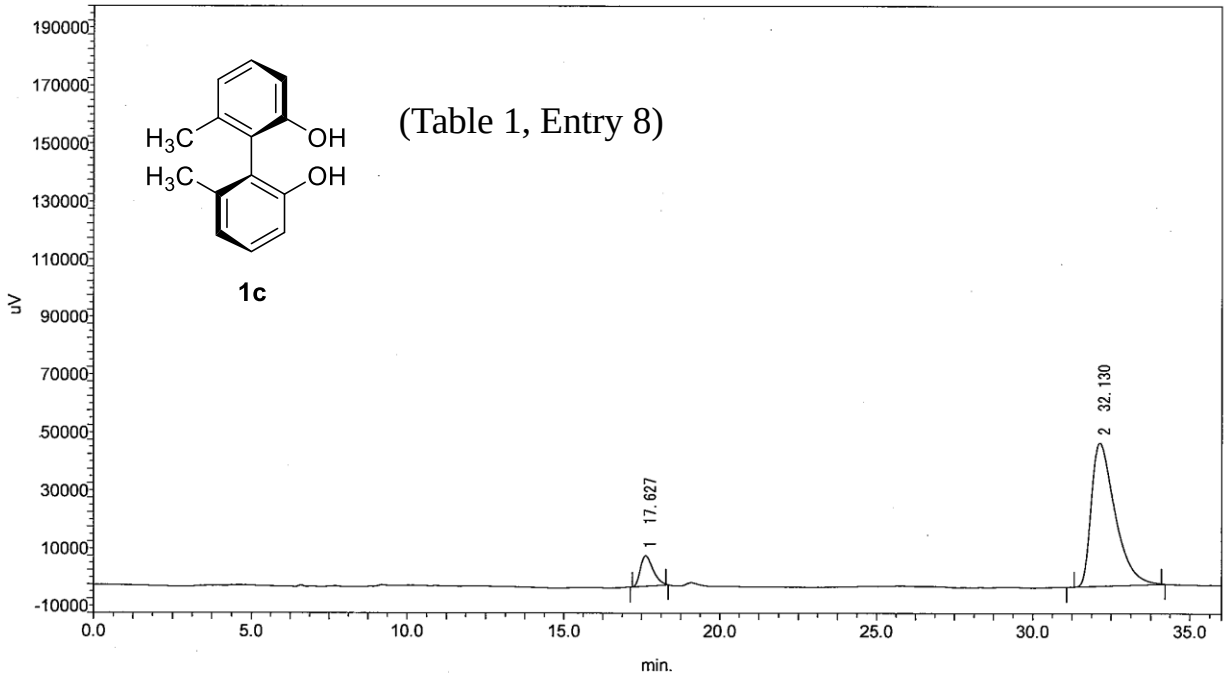
No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	18.82		6497751.898	48.8439	217322	8752.9	1.608	9.240
2	28.45		6805350.731	51.1561	143365	7890.3	1.819	------
			13303102.629	100.0000	360687			



(Table 1, Entry 7)

解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	31.88		6315474.000	100.0000	104173	6236.8	2.121	------
			6315474.000	100.0000	104173			



(Table 1, Entry 8)

解析結果

No.	Rt(min)	ピーク名	面積	面積(%)	高さ	NTP	対称性	分離度
1	17.63		300989.840	10.2862	10625	8127.8	1.399	13.009
2	32.13		2625161.932	89.7138	49523	7881.2	1.720	------
			2926151.771	100.0000	60148			