Supporting Information:

γ-Halo-Enones: A Method for their Synthesis from Arylacyl Halides and their Application to the Preparation of Five Membered Ring Heterocycles

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Table of Contents:

I. X-Ray Crystallographic Analyses. **Page** S2-S12

II. Figure 1. Crystal structure of γ-halo-enones **1b** and **1g** **Page** S13

III. Table 1. Screening of Brønsted Acids

for the γ-Bromo-Enone (1e) Forming Reaction of 4e. **Page** S13

IV. Copies of ^{1}H and ^{13}C NMR spectra. **Page** S14-S128

I. X-Ray Crystallographic Analyses

Table 1. Crystal data and structure refinement for **1b**.

Identification code	hyjii1330202	
Empirical formula	C11 H9 Cl3 O	
Formula weight	263.53	
Temperature	250(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 10.463(3) Å	α= 90°.
	b = 12.044(4) Å	β= 118.36(2)°.
	c = 10.740(4) Å	γ = 90°.
Volume	1191.1(7) $Å^3$	
Z	4	
Density (calculated)	1.470 Mg/m^3	
Absorption coefficient	0.738 mm^{-1}	
F(000)	536	
Crystal size	0.10 x 0.04 x 0.04 mm^3	
Theta range for data collection	2.24 to 28.28°.	
Index ranges	-13<=h<=13, -13<=k<=16, -8<=l<=13	
Reflections collected	7145	
Independent reflections	2867 [R(int) = 0.0272]	
Completeness to theta = 28.28°	97.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9711 and 0.9298	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2867 / 0 / 137	
Goodness-of-fit on F^2	0.890	
Final R indices [I>2sigma(I)]	R1 = 0.0397, wR2 = 0.1241	
R indices (all data)	R1 = 0.0753, wR2 = 0.1543	
Largest diff. peak and hole	0.261 and -0.253 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for HYJII1330202. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	2734(1)	341(1)	6773(1)	70(1)
Cl(3)	-1478(1)	-564(1)	7179(1)	74(1)
Cl(2)	5689(1)	-2694(1)	10760(1)	78(1)
C(1)	2989(3)	-372(2)	8285(2)	48(1)
C(6)	2131(2)	-125(2)	8911(2)	48(1)
C(9)	-95(3)	2452(2)	8830(3)	57(1)
C(7)	928(3)	709(2)	8347(2)	48(1)
C(8)	1007(2)	1580(2)	9141(2)	51(1)
C(3)	4317(3)	-1713(2)	10042(3)	56(1)
C(2)	4079(3)	-1150(2)	8836(3)	55(1)
C(11)	-361(3)	448(2)	6938(2)	57(1)
C(5)	2420(3)	-706(2)	10130(3)	60(1)
O(1)	-1202(2)	2535(2)	7720(2)	88(1)
C(4)	3494(3)	-1502(2)	10704(3)	62(1)
C(10)	231(4)	3253(3)	10002(4)	100(1)

Table 3. Bond lengths [Å] and angles [°] for HYJII1330202.

Cl(1)-C(1)	1.742(2)
Cl(3)-C(11)	1.789(3)
Cl(2)-C(3)	1.732(3)
C(1)-C(2)	1.374(3)
C(1)-C(6)	1.385(3)
C(6)-C(5)	1.386(3)
C(6)-C(7)	1.496(3)
C(9)-O(1)	1.209(3)
C(9)-C(8)	1.476(3)
C(9)-C(10)	1.491(4)
C(7)-C(8)	1.330(3)
C(7)-C(11)	1.506(3)
C(3)-C(2)	1.375(4)
C(3)-C(4)	1.377(4)
C(5)-C(4)	1.379(4)
C(2)-C(1)-C(6)	122.3(2)
C(2)-C(1)-Cl(1)	117.75(19)
C(6)-C(1)-Cl(1)	119.96(19)
C(1)-C(6)-C(5)	116.7(2)
C(1)-C(6)-C(7)	124.2(2)
C(5)-C(6)-C(7)	119.1(2)
O(1)-C(9)-C(8)	124.2(2)
O(1)-C(9)-C(10)	120.4(3)
C(8)-C(9)-C(10)	115.4(2)
C(8)-C(7)-C(6)	119.1(2)
C(8)-C(7)-C(11)	124.2(2)
C(6)-C(7)-C(11)	116.5(2)
C(7)-C(8)-C(9)	127.7(2)
C(2)-C(3)-C(4)	121.2(2)
C(2)-C(3)-Cl(2)	119.8(2)
C(4)-C(3)-Cl(2)	119.0(2)
C(1)-C(2)-C(3)	118.9(2)
C(7)-C(11)-Cl(3)	109.36(17)
C(4)-C(5)-C(6)	122.6(2)

C(3)-C(4)-C(5)	118.3(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for HYJII1330202. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	69(1)	96(1)	49(1)	4(1)	32(1)	-7(1)
Cl(3)	63(1)	83(1)	74(1)	-16(1)	33(1)	-22(1)
Cl(2)	69(1)	60(1)	101(1)	1(1)	37(1)	14(1)
C(1)	49(1)	56(1)	40(1)	-8(1)	20(1)	-8(1)
C(6)	47(1)	53(1)	42(1)	-5(1)	20(1)	-5(1)
C(9)	51(1)	61(2)	57(2)	4(1)	24(1)	3(1)
C(7)	42(1)	60(1)	39(1)	3(1)	17(1)	-5(1)
C(8)	42(1)	59(1)	46(1)	-1(1)	16(1)	0(1)
C(3)	55(1)	49(1)	59(2)	-7(1)	24(1)	-1(1)
C(2)	50(1)	61(2)	55(1)	-17(1)	26(1)	-8(1)
C(11)	51(1)	66(2)	45(1)	0(1)	15(1)	-4(1)
C(5)	68(2)	66(2)	55(1)	7(1)	38(1)	11(1)
O(1)	67(1)	113(2)	62(1)	-2(1)	12(1)	35(1)
C(4)	68(2)	65(2)	59(2)	10(1)	34(1)	8(1)
C(10)	82(2)	85(2)	96(2)	-29(2)	13(2)	23(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for HYJII1330202.

	x	y	z	U(eq)
H(8)	1859	1648	9995	61
H(2)	4646	-1294	8401	66
H(11A)	-33	162	6294	68
H(11B)	-918	1118	6531	68
H(5)	1868	-554	10580	71
H(4)	3657	-1886	11518	75
H(10A)	-536	3791	9701	150
H(10B)	1132	3623	10247	150
H(10C)	306	2860	10811	150

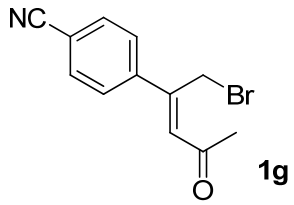


Table 1. Crystal data and structure refinement for **1g**.

Identification code	hyj1380201	
Empirical formula	C12 H10 Br N O	
Formula weight	264.12	
Temperature	250(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.3861(14) Å	α= 95.574(12)°.
	b = 8.3872(14) Å	β= 100.912(12)°.
	c = 9.0080(16) Å	γ = 112.227(11)°.
Volume	565.89(17) $Å^3$	
Z	2	
Density (calculated)	1.550 Mg/m^3	
Absorption coefficient	3.604 mm^{-1}	
F(000)	264	
Crystal size	0.28 x 0.25 x 0.23 mm^3	
Theta range for data collection	2.34 to 28.28°.	
Index ranges	-11<=h<=11, -11<=k<=11, -12<=l<=11	
Reflections collected	7697	
Independent reflections	2793 [R(int) = 0.0299]	
Completeness to theta = 28.28°	99.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.4912 and 0.4319	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2793 / 0 / 137	
Goodness-of-fit on F^2	1.007	
Final R indices [I>2sigma(I)]	R1 = 0.0341, wR2 = 0.0769	
R indices (all data)	R1 = 0.0551, wR2 = 0.0846	
Largest diff. peak and hole	0.382 and -0.505 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for hyj1380201. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	5845(1)	7566(1)	8321(1)	56(1)
O(1)	3245(3)	8696(2)	3890(2)	55(1)
N(1)	468(3)	-1840(3)	8519(3)	50(1)
C(12)	3079(5)	6798(4)	1689(3)	59(1)
C(11)	3006(3)	7228(3)	3325(3)	41(1)
C(10)	2577(3)	5742(3)	4166(3)	40(1)
C(8)	2911(3)	5819(3)	5692(3)	34(1)
C(5)	2371(3)	4168(3)	6317(3)	33(1)
C(4)	2528(3)	2689(3)	5609(3)	38(1)
C(3)	2019(3)	1155(3)	6176(3)	39(1)
C(2)	1342(3)	1056(3)	7473(3)	36(1)
C(1)	837(3)	-549(3)	8068(3)	40(1)
C(6)	1689(3)	4040(3)	7623(3)	40(1)
C(7)	1171(3)	2506(3)	8194(3)	40(1)
C(9)	3805(3)	7515(3)	6828(3)	40(1)

Table 3. Bond lengths [Å] and angles [°] for hyj1380201.

Br(1)-C(9)	1.949(2)
O(1)-C(11)	1.212(3)
N(1)-C(1)	1.146(3)
C(12)-C(11)	1.501(4)
C(12)-H(1A)	0.9600
C(12)-H(1B)	0.9600
C(12)-H(1C)	0.9600
C(11)-C(10)	1.484(4)
C(10)-C(8)	1.340(3)
C(10)-H(3)	0.9300
C(8)-C(5)	1.487(3)
C(8)-C(9)	1.501(3)
C(5)-C(6)	1.398(3)
C(5)-C(4)	1.402(3)
C(4)-C(3)	1.376(3)
C(4)-H(6)	0.9300
C(3)-C(2)	1.389(3)
C(3)-H(7)	0.9300
C(2)-C(7)	1.391(3)
C(2)-C(1)	1.440(4)
C(6)-C(7)	1.377(3)
C(6)-H(10)	0.9300
C(7)-H(11)	0.9300
C(9)-H(12A)	0.9700
C(9)-H(12B)	0.9700
C(11)-C(12)-H(1A)	109.5
C(11)-C(12)-H(1B)	109.5
H(1A)-C(12)-H(1B)	109.5
C(11)-C(12)-H(1C)	109.5
H(1A)-C(12)-H(1C)	109.5
H(1B)-C(12)-H(1C)	109.5
O(1)-C(11)-C(10)	123.1(2)
O(1)-C(11)-C(12)	121.1(2)
C(10)-C(11)-C(12)	115.8(2)

C(8)-C(10)-C(11)	127.7(2)
C(8)-C(10)-H(3)	116.2
C(11)-C(10)-H(3)	116.2
C(10)-C(8)-C(5)	119.6(2)
C(10)-C(8)-C(9)	122.9(2)
C(5)-C(8)-C(9)	117.4(2)
C(6)-C(5)-C(4)	117.9(2)
C(6)-C(5)-C(8)	121.2(2)
C(4)-C(5)-C(8)	120.8(2)
C(3)-C(4)-C(5)	121.1(2)
C(3)-C(4)-H(6)	119.5
C(5)-C(4)-H(6)	119.5
C(4)-C(3)-C(2)	120.0(2)
C(4)-C(3)-H(7)	120.0
C(2)-C(3)-H(7)	120.0
C(3)-C(2)-C(7)	119.9(2)
C(3)-C(2)-C(1)	119.5(2)
C(7)-C(2)-C(1)	120.6(2)
N(1)-C(1)-C(2)	178.0(3)
C(7)-C(6)-C(5)	121.3(2)
C(7)-C(6)-H(10)	119.4
C(5)-C(6)-H(10)	119.4
C(6)-C(7)-C(2)	119.7(2)
C(6)-C(7)-H(11)	120.1
C(2)-C(7)-H(11)	120.1
C(8)-C(9)-Br(1)	111.56(16)
C(8)-C(9)-H(12A)	109.3
Br(1)-C(9)-H(12A)	109.3
C(8)-C(9)-H(12B)	109.3
Br(1)-C(9)-H(12B)	109.3
H(12A)-C(9)-H(12B)	108.0

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for hyj1380201. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	51(1)	60(1)	45(1)	2(1)	-4(1)	19(1)
O(1)	78(1)	42(1)	46(1)	11(1)	14(1)	24(1)
N(1)	58(1)	46(1)	49(1)	15(1)	20(1)	20(1)
C(12)	84(2)	63(2)	42(2)	19(1)	22(2)	36(2)
C(11)	42(1)	44(1)	37(1)	10(1)	7(1)	17(1)
C(10)	43(1)	34(1)	37(1)	8(1)	7(1)	11(1)
C(8)	33(1)	36(1)	34(1)	6(1)	10(1)	15(1)
C(5)	30(1)	37(1)	31(1)	6(1)	6(1)	13(1)
C(4)	44(1)	43(1)	33(1)	9(1)	16(1)	19(1)
C(3)	46(1)	36(1)	38(1)	7(1)	12(1)	20(1)
C(2)	34(1)	40(1)	34(1)	10(1)	9(1)	14(1)
C(1)	38(1)	44(2)	39(1)	9(1)	12(1)	17(1)
C(6)	48(1)	42(1)	36(1)	6(1)	15(1)	23(1)
C(7)	45(1)	49(2)	35(1)	13(1)	19(1)	22(1)
C(9)	46(1)	39(1)	37(1)	7(1)	9(1)	20(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for hyj1380201.

	x	y	z	U(eq)
H(1A)	3317	7827	1235	89
H(1B)	1960	5896	1119	89
H(1C)	4006	6395	1667	89
H(3)	2009	4626	3562	48
H(6)	2983	2745	4741	46
H(7)	2129	185	5691	46
H(10)	1582	5008	8115	48
H(11)	710	2441	9058	48
H(12A)	2959	7669	7365	48
H(12B)	4193	8479	6282	48

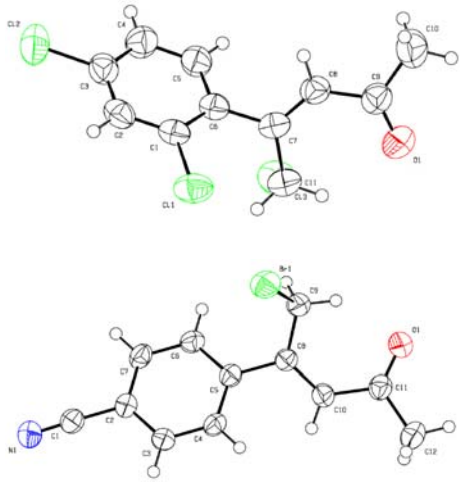
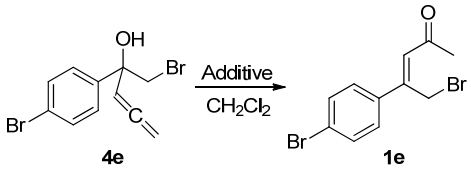


Figure 1. Crystal structure of γ-halo-enones **1b** and **1g.**

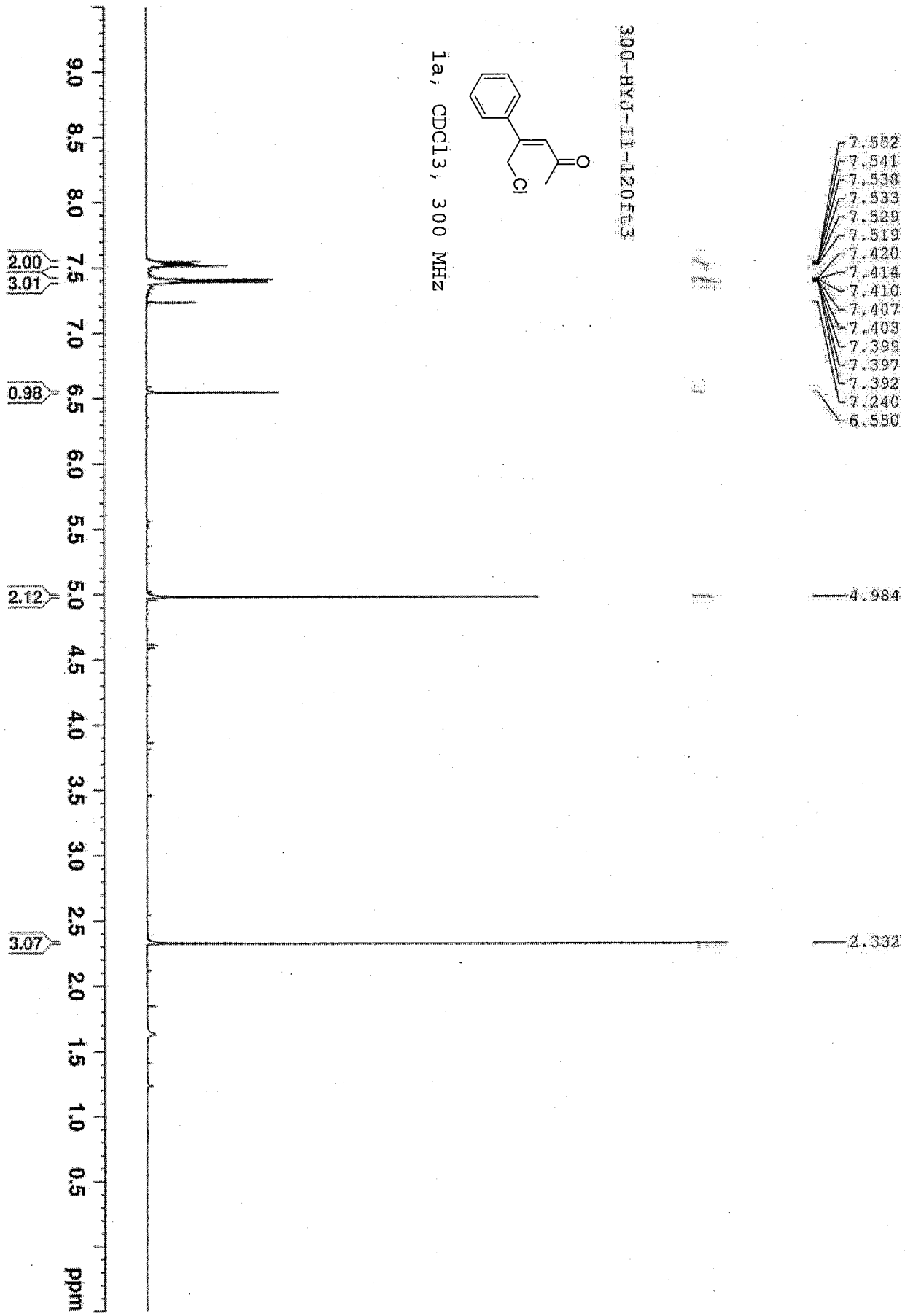
Table 1. Screening of Brønsted Acids for the γ-Bromo-Enone (1e) Forming Reaction of 4e.[a]



4e **1e**

entry	additive	**1e**, yield (%)
1[b]	HOAc	0
2[c]	HOAc	0
3	H_3PO_4	0
4	HCl	0
5	H_2SO_4	70
6	CF_3SO_3H	48

[a]Condition: **4e** (0.8 mmol) and additive (0.05 mL) in CH_2Cl_2 (5 mL) at rt for 2 h. Ratio determined by NMR spectroscopy. [b]Condition: **4e** (0.8 mmol) and HOAc (0.05 mL) in CH_2Cl_2 (5 mL) were heated to reflux for 2 h. [c]Condition: **4e** (0.8 mmol) in HOAc (2 mL) at 50 ^{o}C for 2 h.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1a, CDCl3, 300 MHz
Cl
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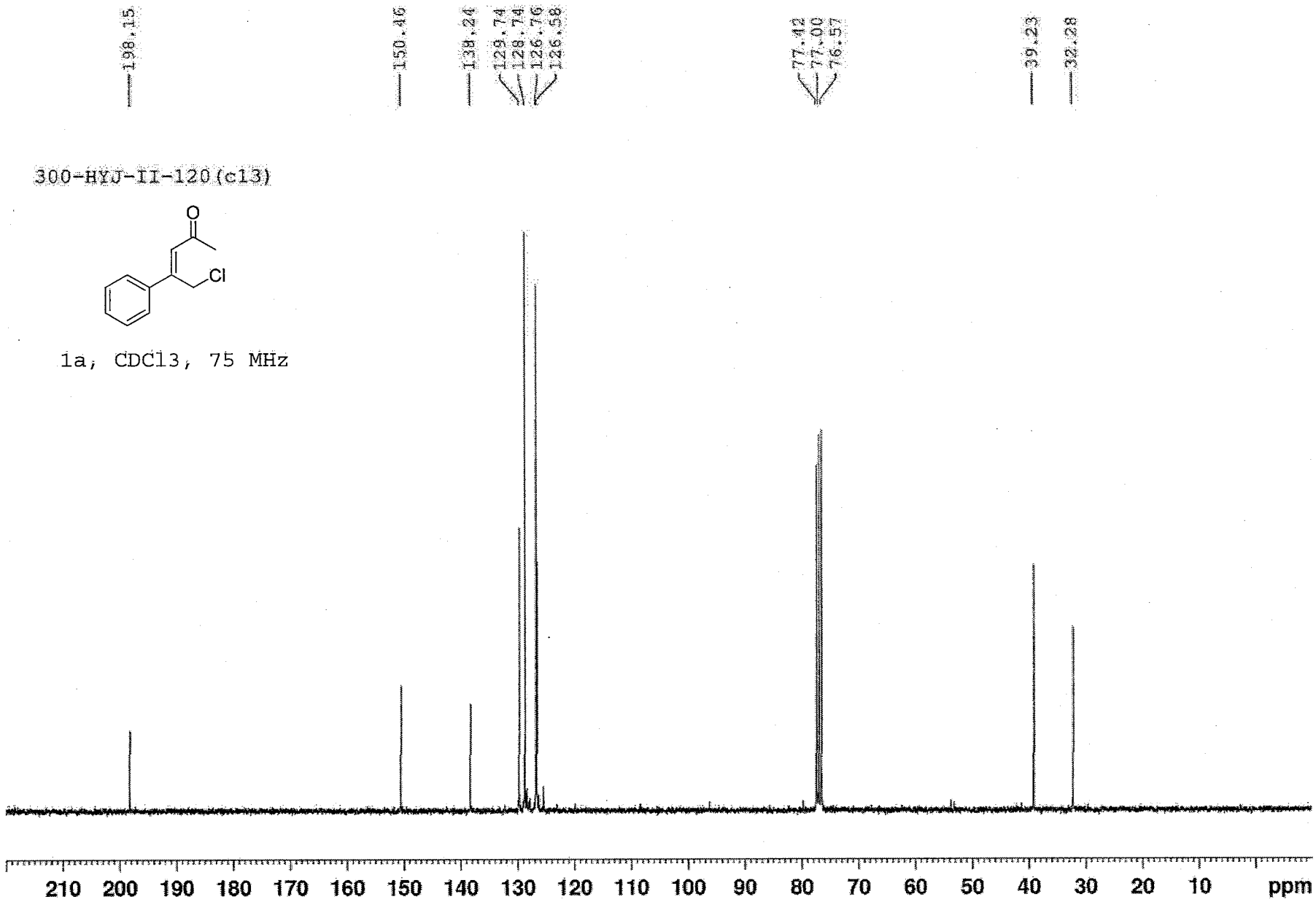
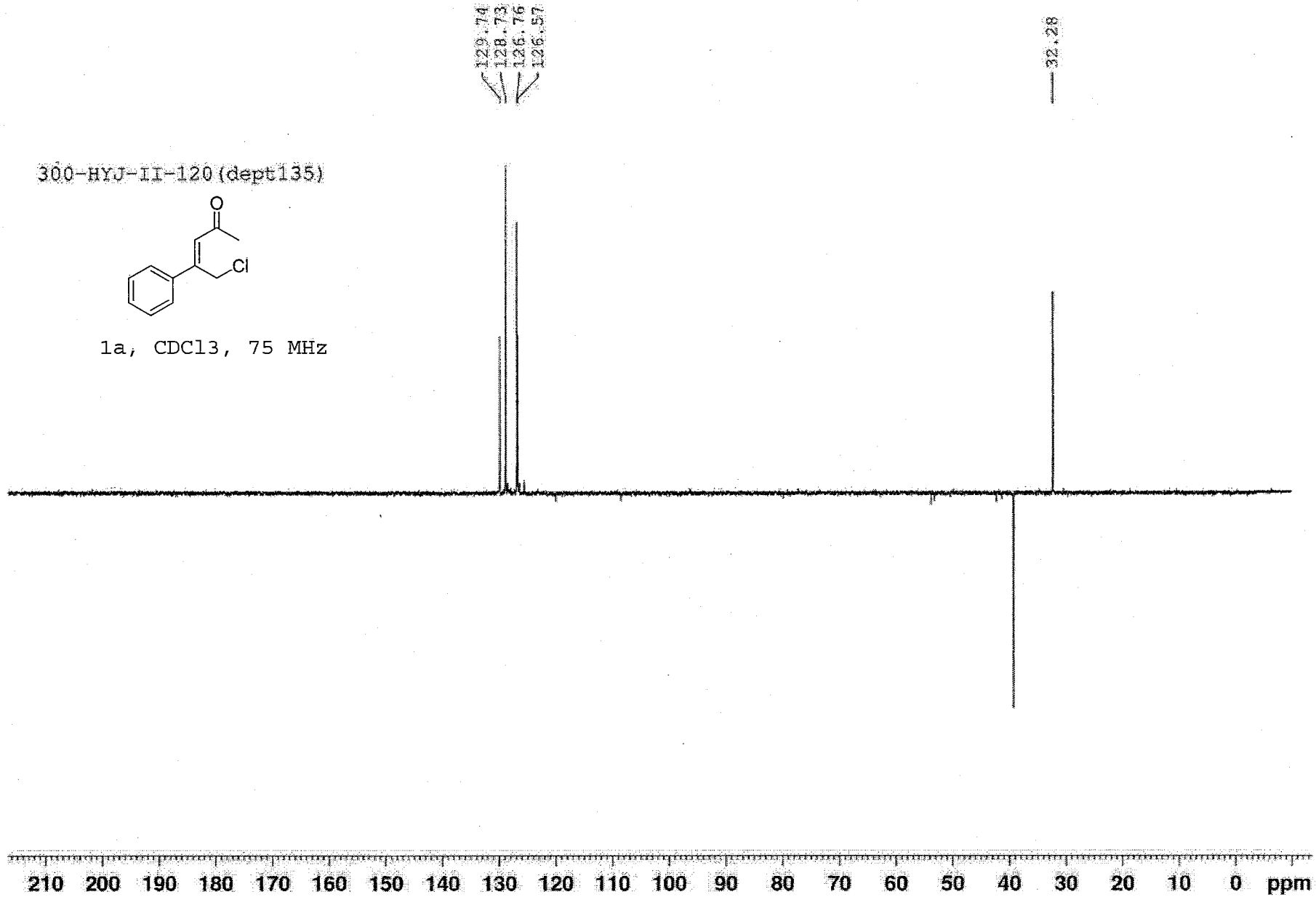
7.541
7.538
7.533
7.529
7.519
7.420
7.414
7.410
7.407
7.403
7.399
7.397
7.392
7.240
6.550
4.984
2.332
2.00
3.01
0.98
2.12
3.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


198.15
150.46
138.24
129.74
128.74
126.76
126.58
77.42
77.00
76.57
39.23
32.28
300-HYJ-II-120(c13)
O
Cl
1a, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


300-HYJ-II-120(dept135)
O
Cl
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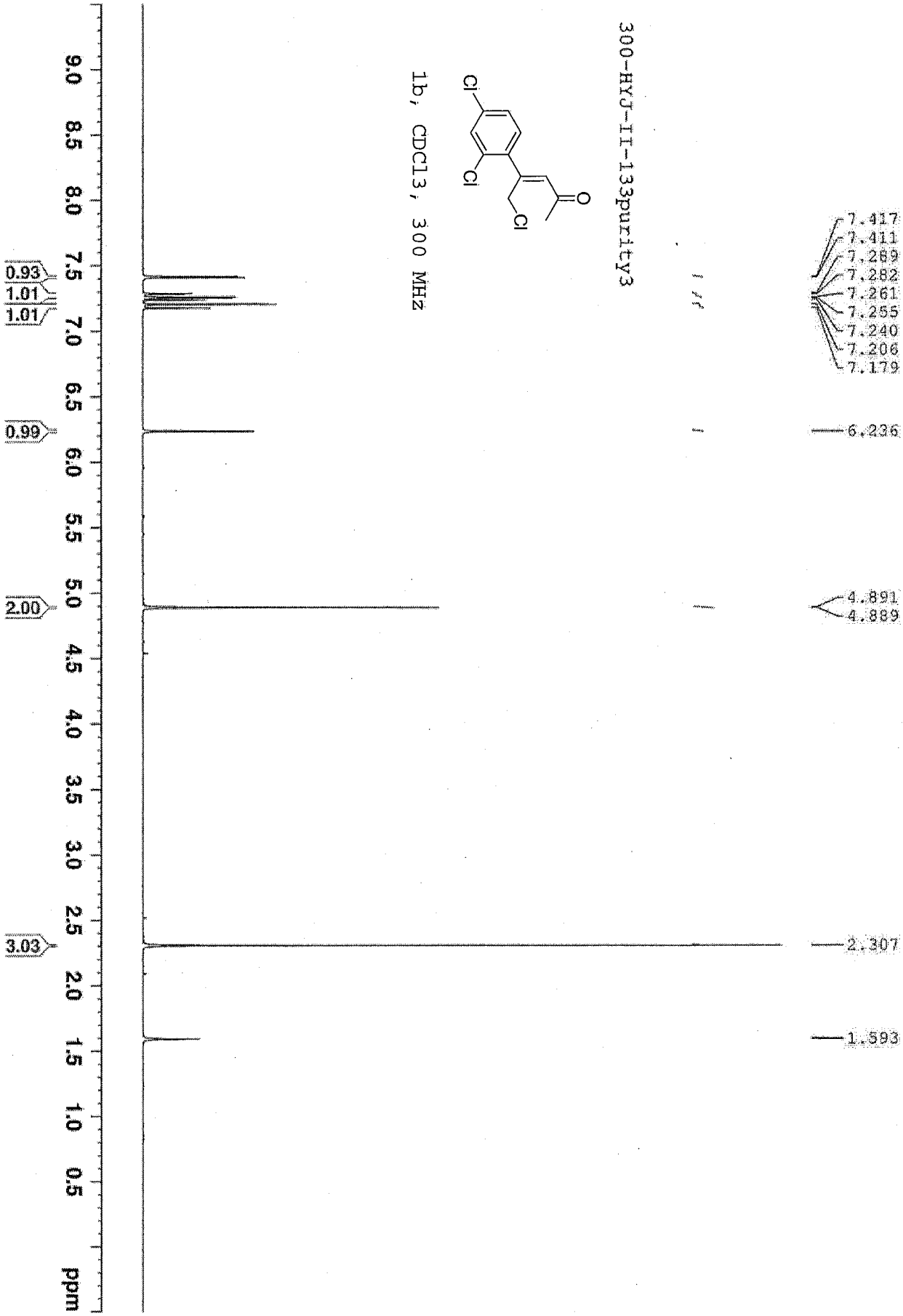
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Cl
Cl
O
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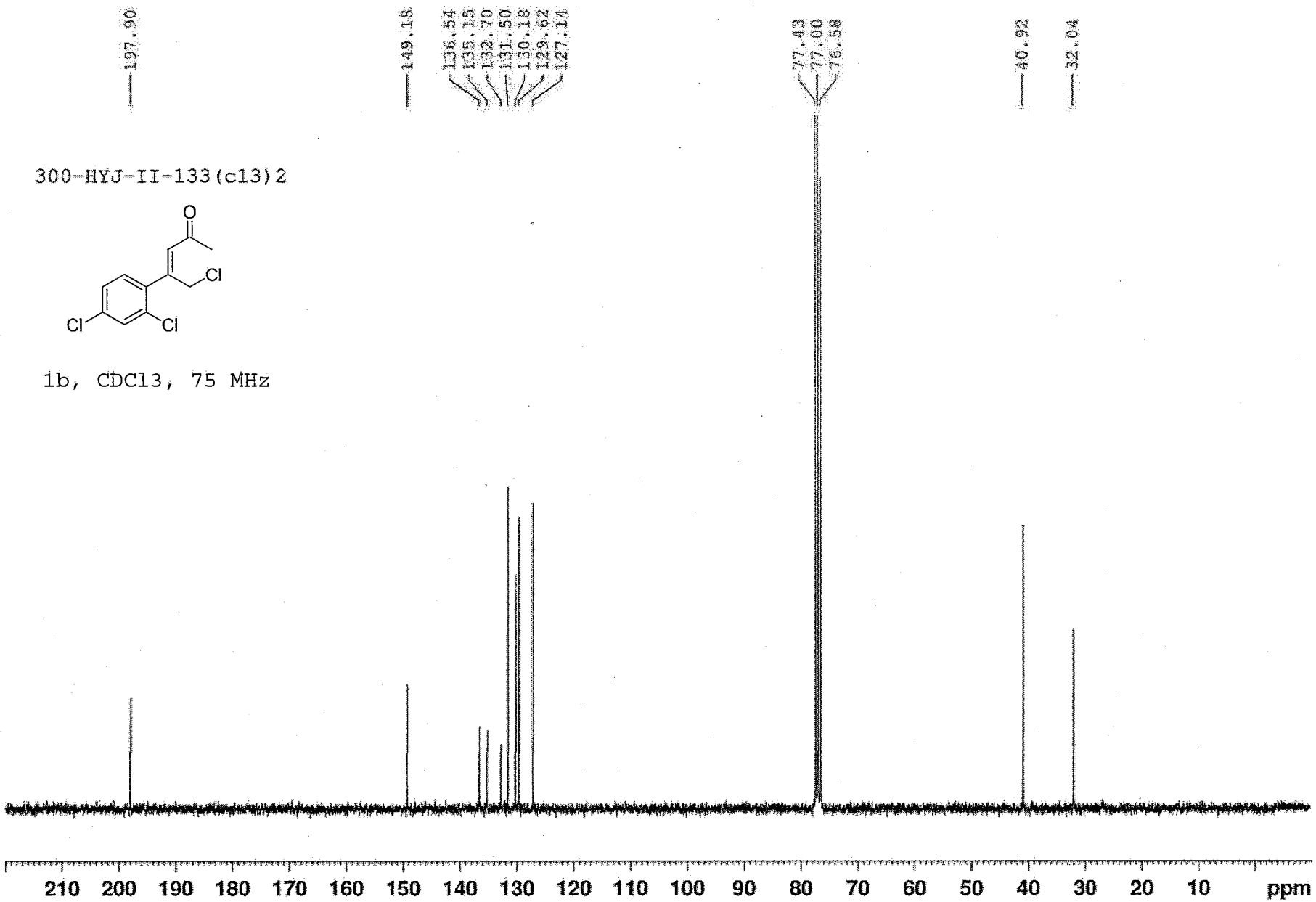
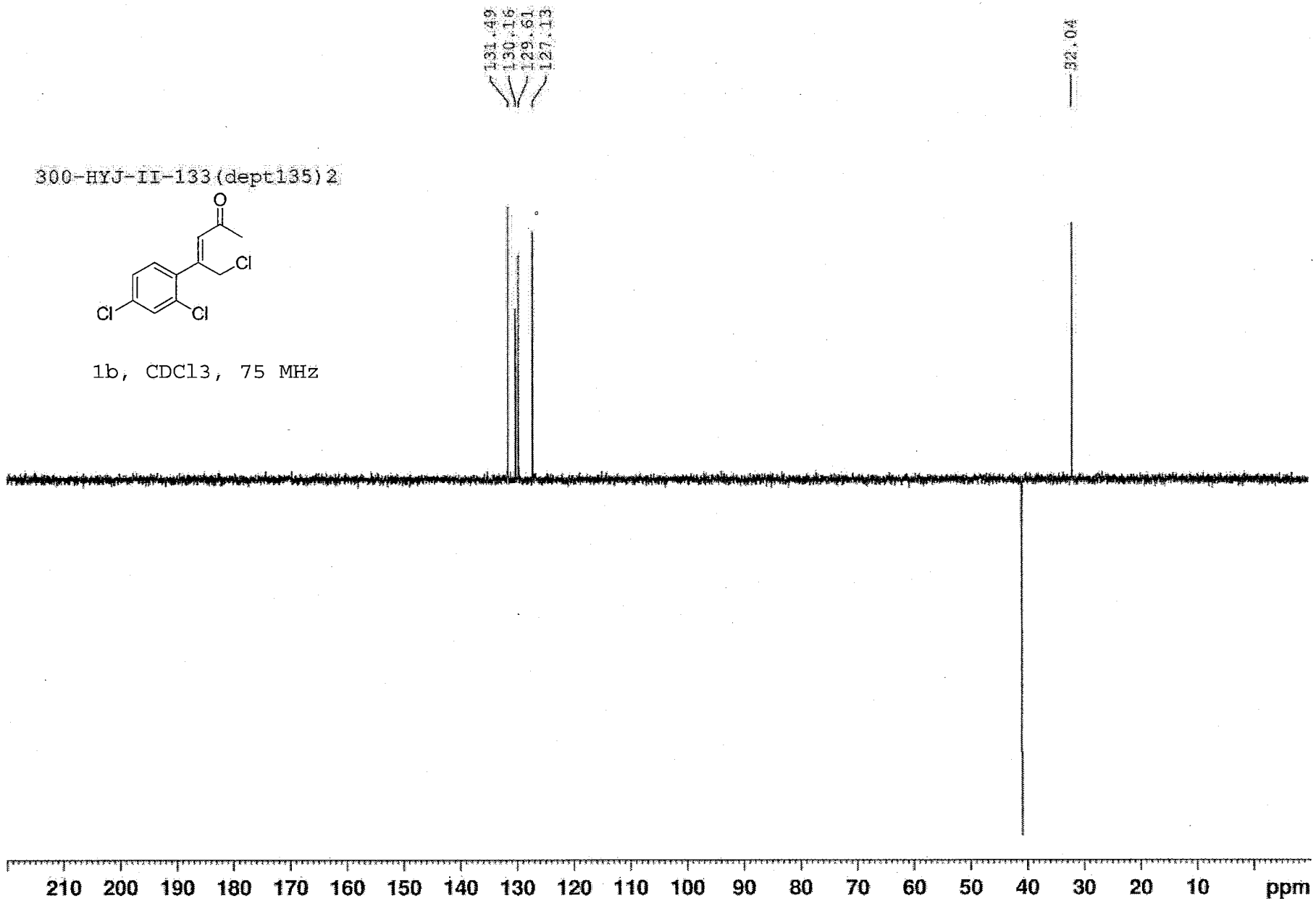
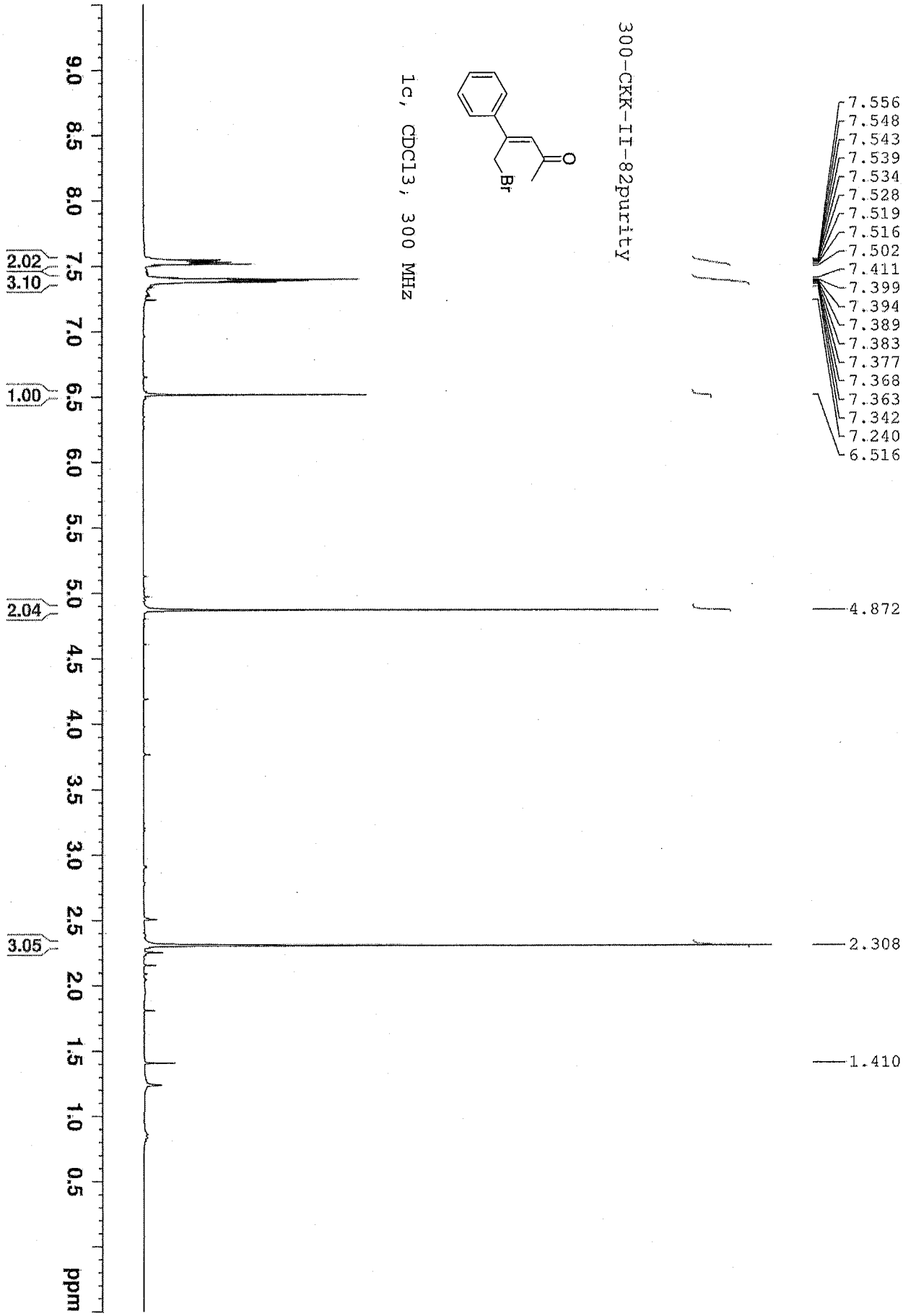
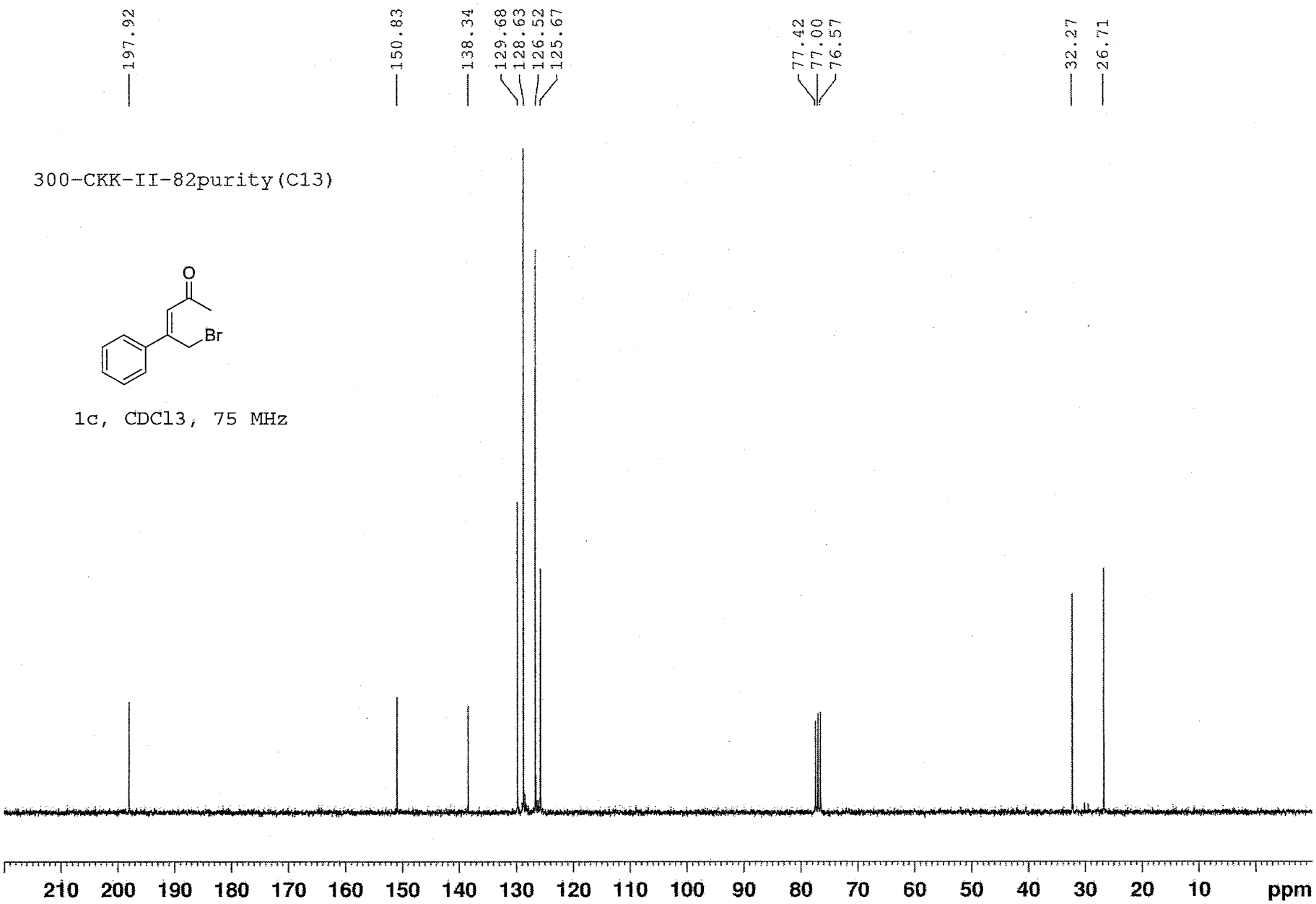
7.282
7.261
7.255
7.240
7.206
7.179
6.236
4.891
4.889
2.307
1.593
0.93
1.01
1.01
0.99
2.00
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


197.90
149.18
136.54
135.15
132.70
131.50
130.18
129.62
127.14
77.43
77.00
76.58
40.92
32.04
300-HYJ-II-133(c13)2
O
Cl
Cl
Cl
1b, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

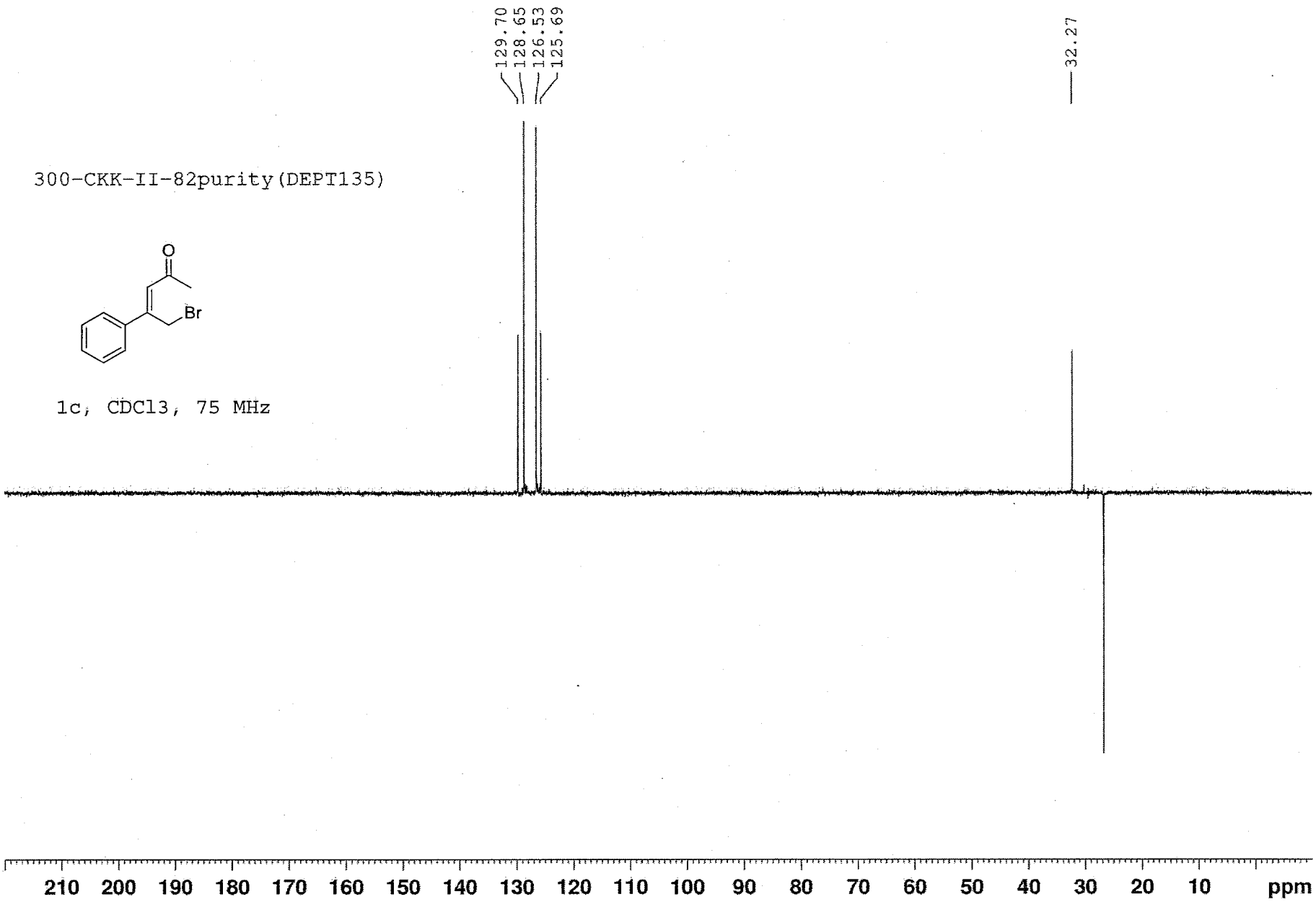

300-HYJ-II-133(dept135)2
O
Cl
Cl
Cl
1b, CDCl3, 75 MHz
131.49
130.16
129.61
127.13
32.04
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

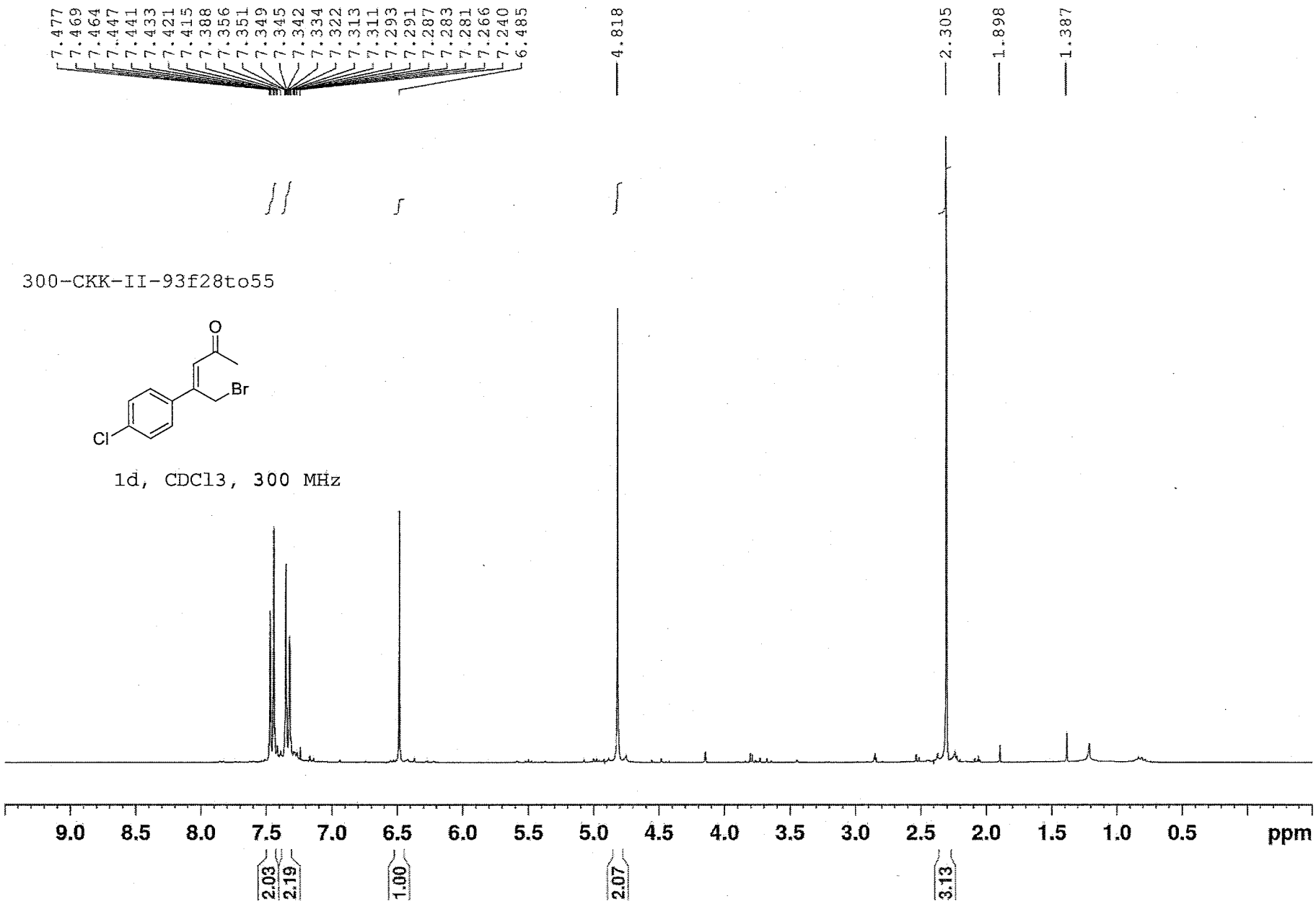

300-CKK-II-82purity
1c, CDCl3, 300 MHz
Br
O
7.556
7.548
7.543
7.539
7.534
7.528
7.519
7.516
7.502
7.411
7.399
7.394
7.389
7.383
7.377
7.368
7.363
7.342
7.240
6.516
4.872
2.308
1.410
2.02
3.10
1.00
2.04
3.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

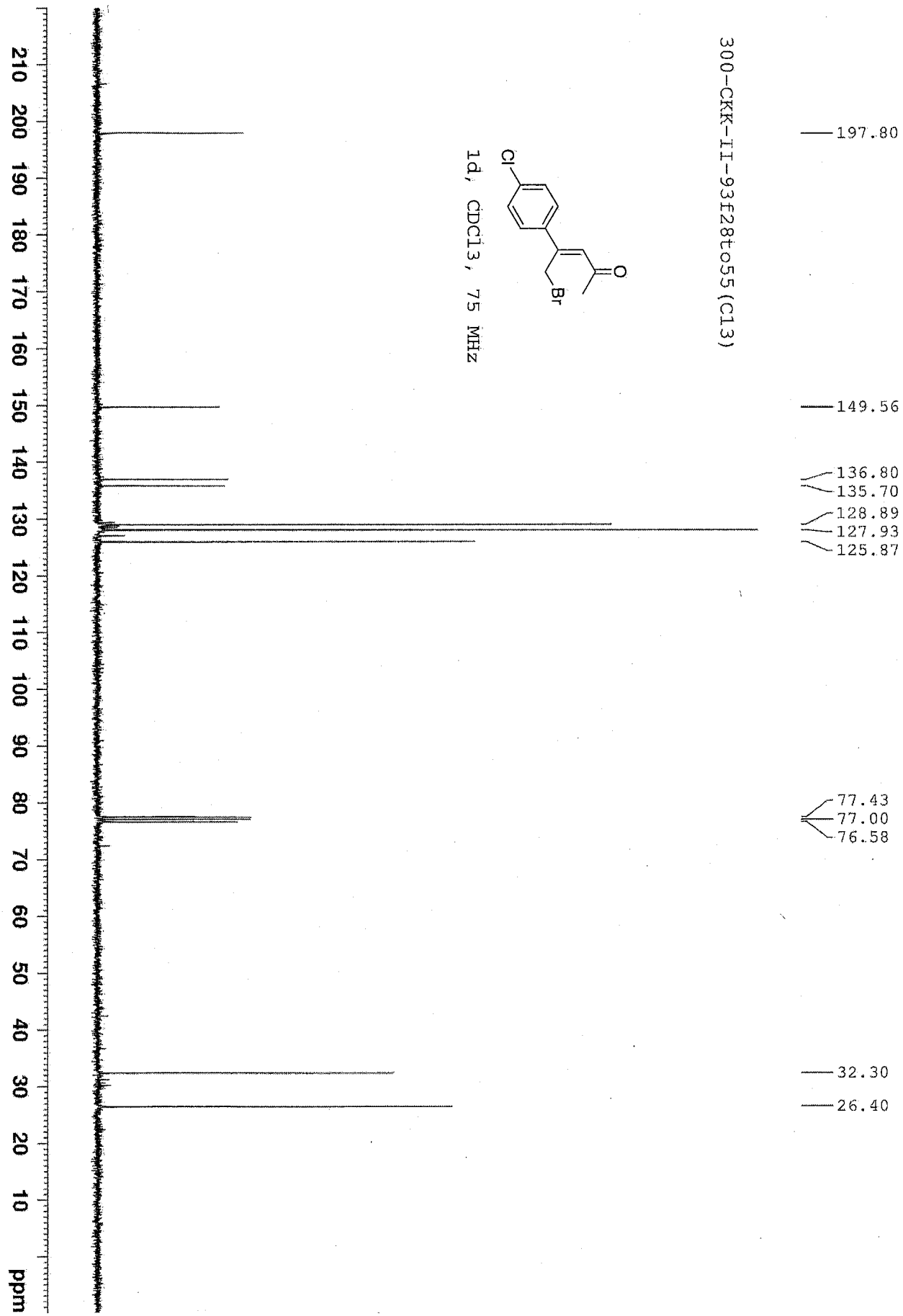

197.92
150.83
138.34
129.68
128.63
126.52
125.67
77.42
77.00
76.57
32.27
26.71
300-CKK-II-82purity(C13)
O
Br
1c, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

300-CKK-II-82purity(DEPT135)

1c, CDCl3, 75 MHz

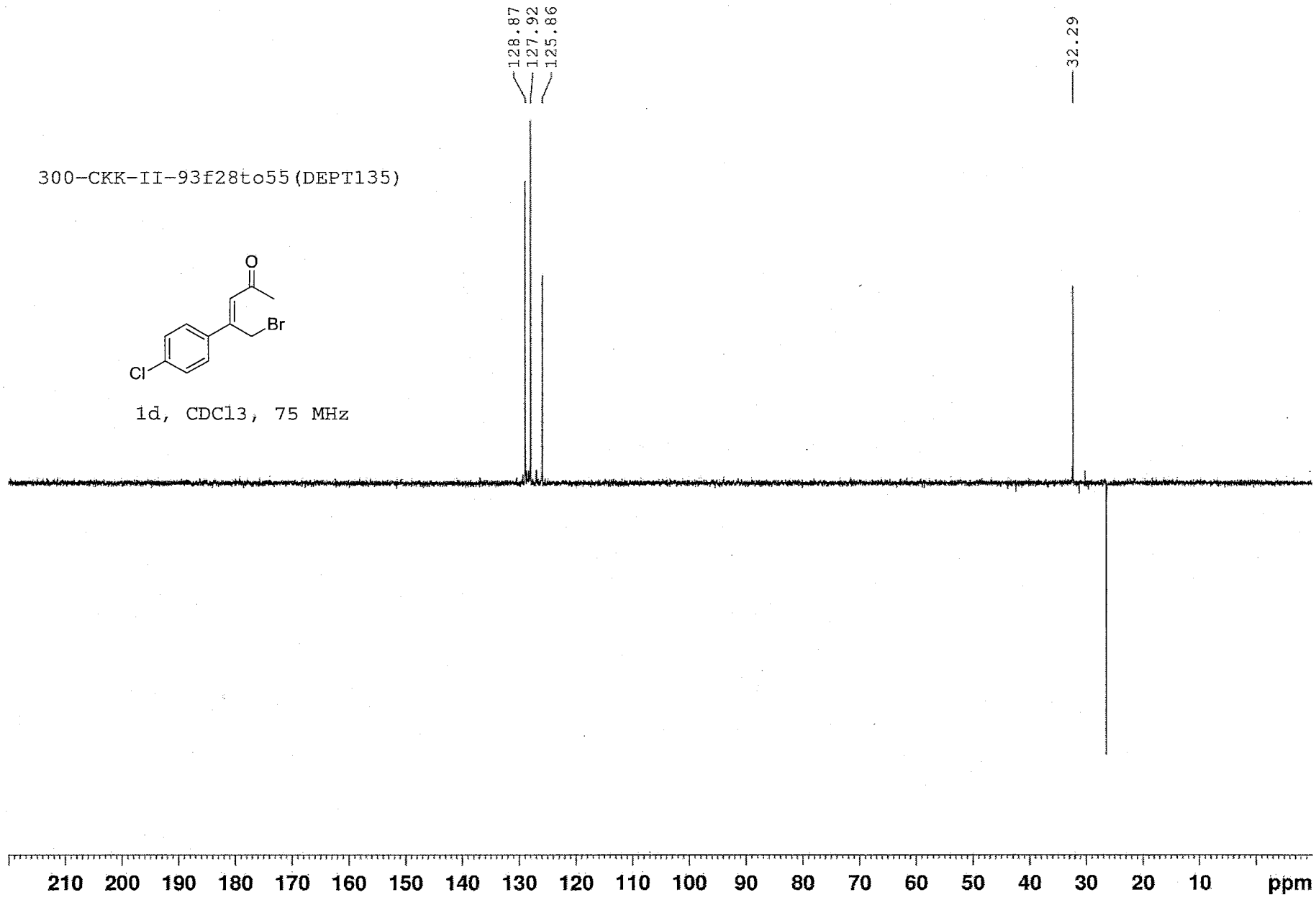


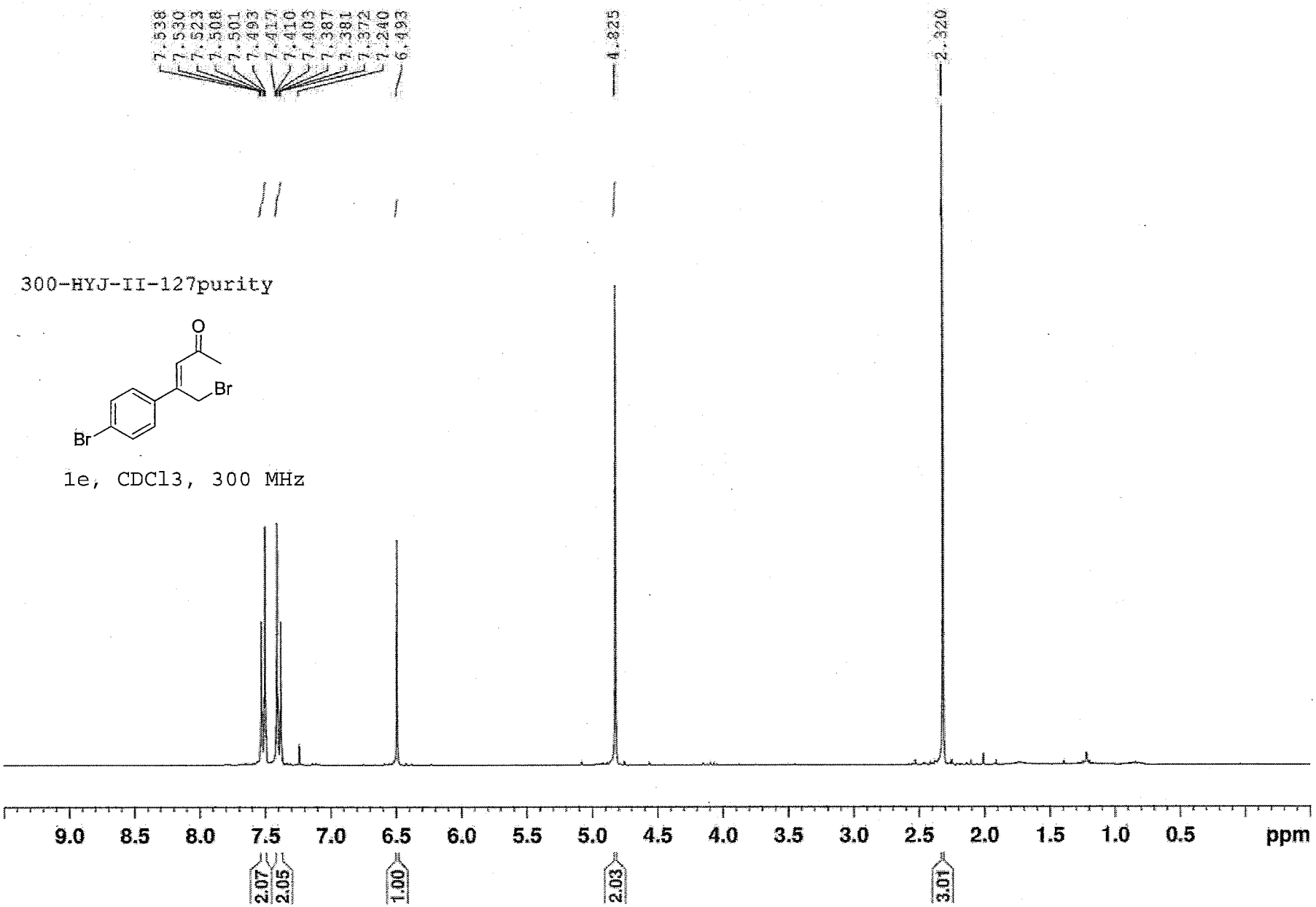

7.477
7.469
7.464
7.447
7.441
7.433
7.421
7.415
7.388
7.356
7.351
7.349
7.345
7.342
7.334
7.322
7.313
7.311
7.293
7.291
7.287
7.283
7.281
7.266
7.240
6.485
4.818
2.305
1.898
1.387
300-CKK-II-93f28to55
O
Br
Cl
1d, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.03
2.19
1.00
2.07
3.13

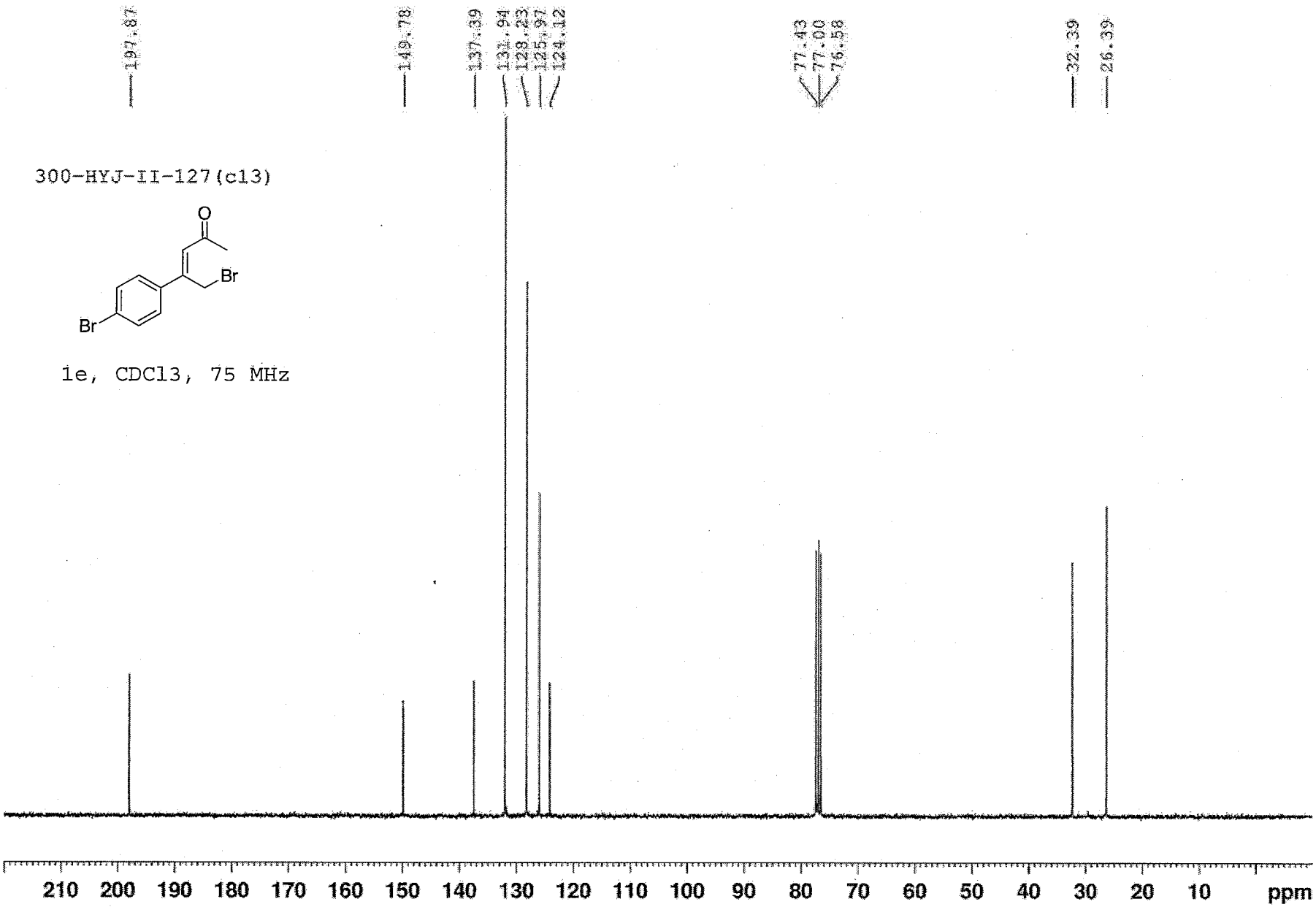

300-CKK-II-93f28to55(C13)
1d, CDCl3, 75 MHz
Cl
O
Br
197.80
149.56
136.80
135.70
128.89
127.93
125.87
77.43
77.00
76.58
32.30
26.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

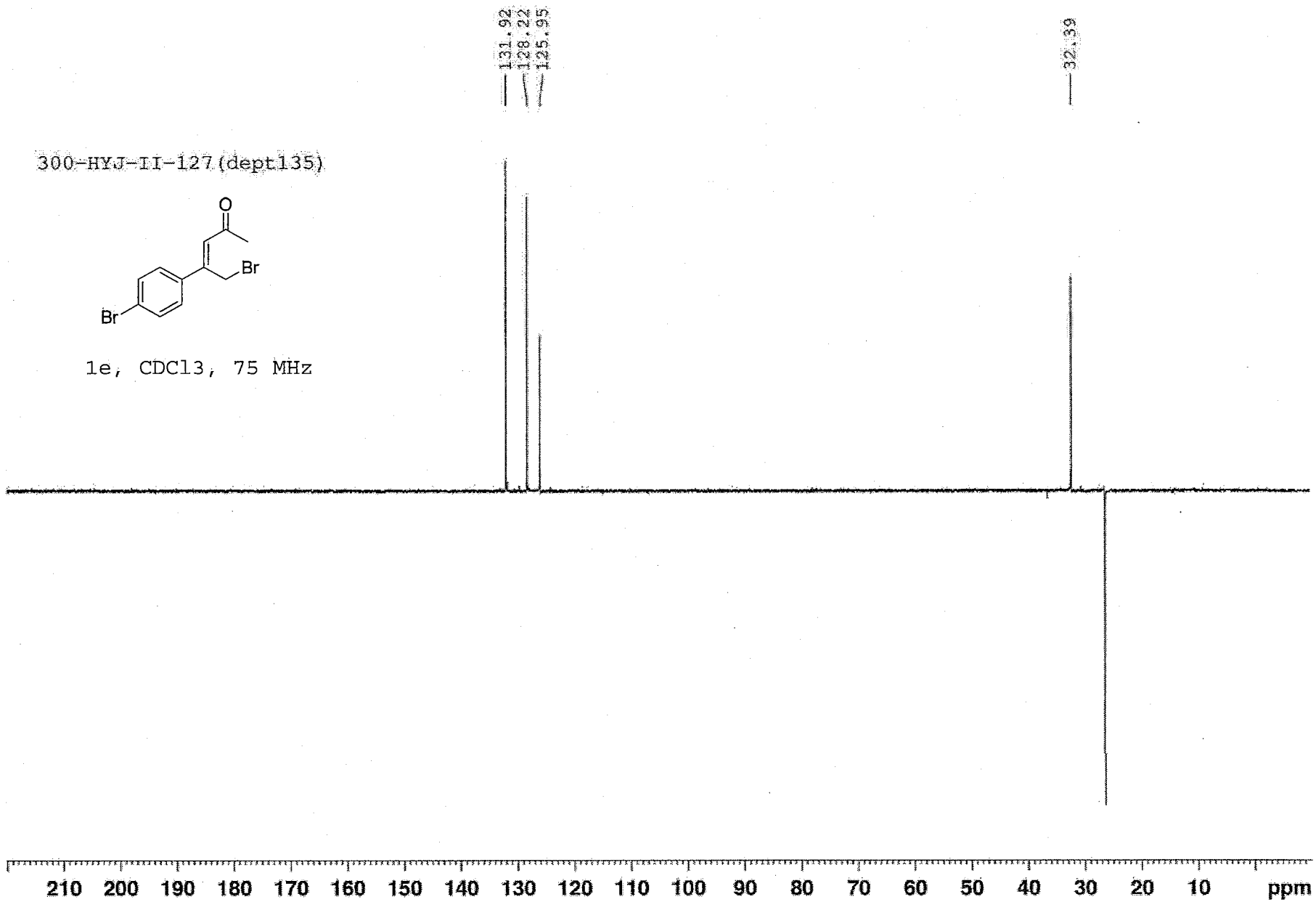
300-CKK-II-93f28to55(DEPT135)

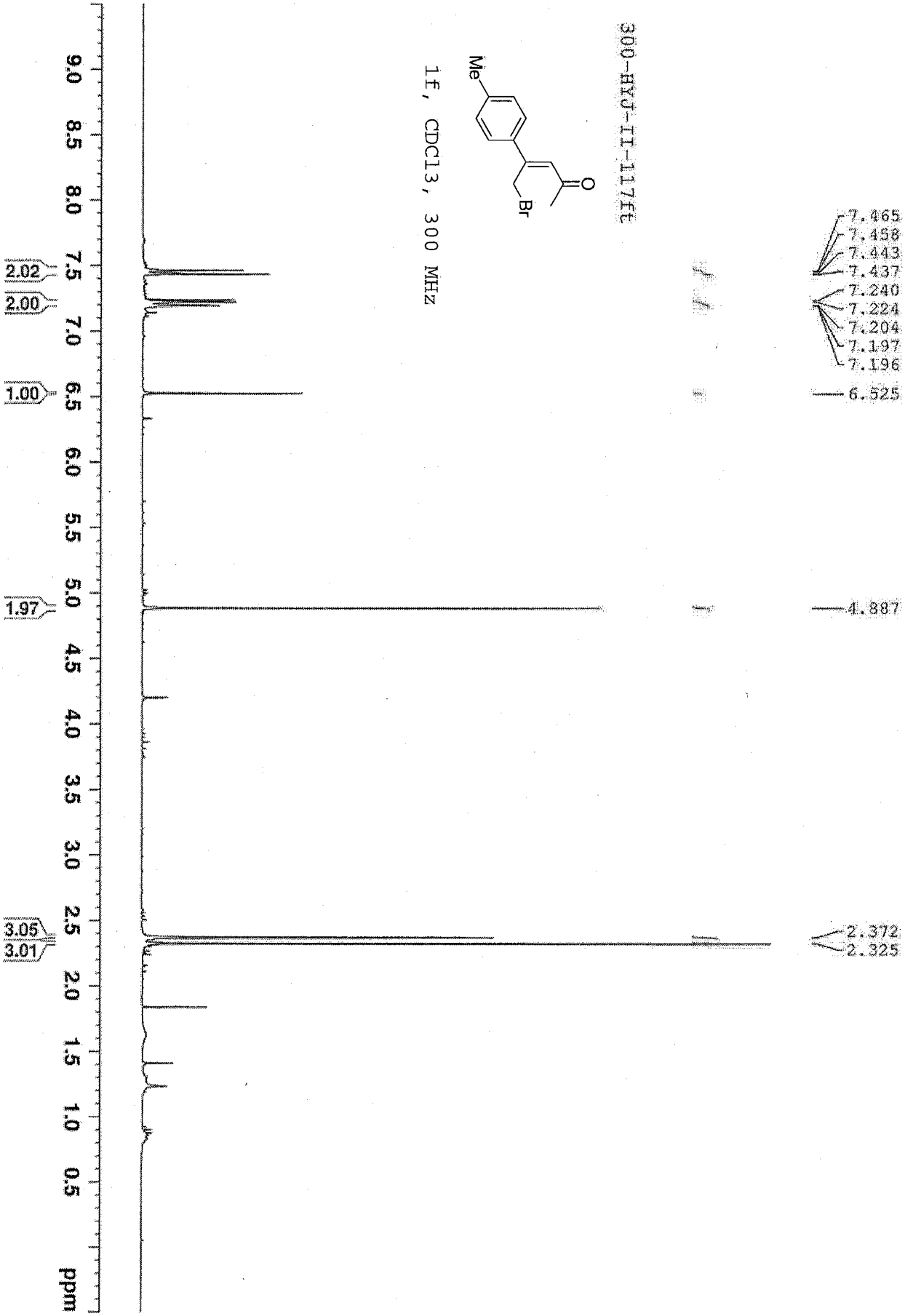
1d, CDCl3, 75 MHz

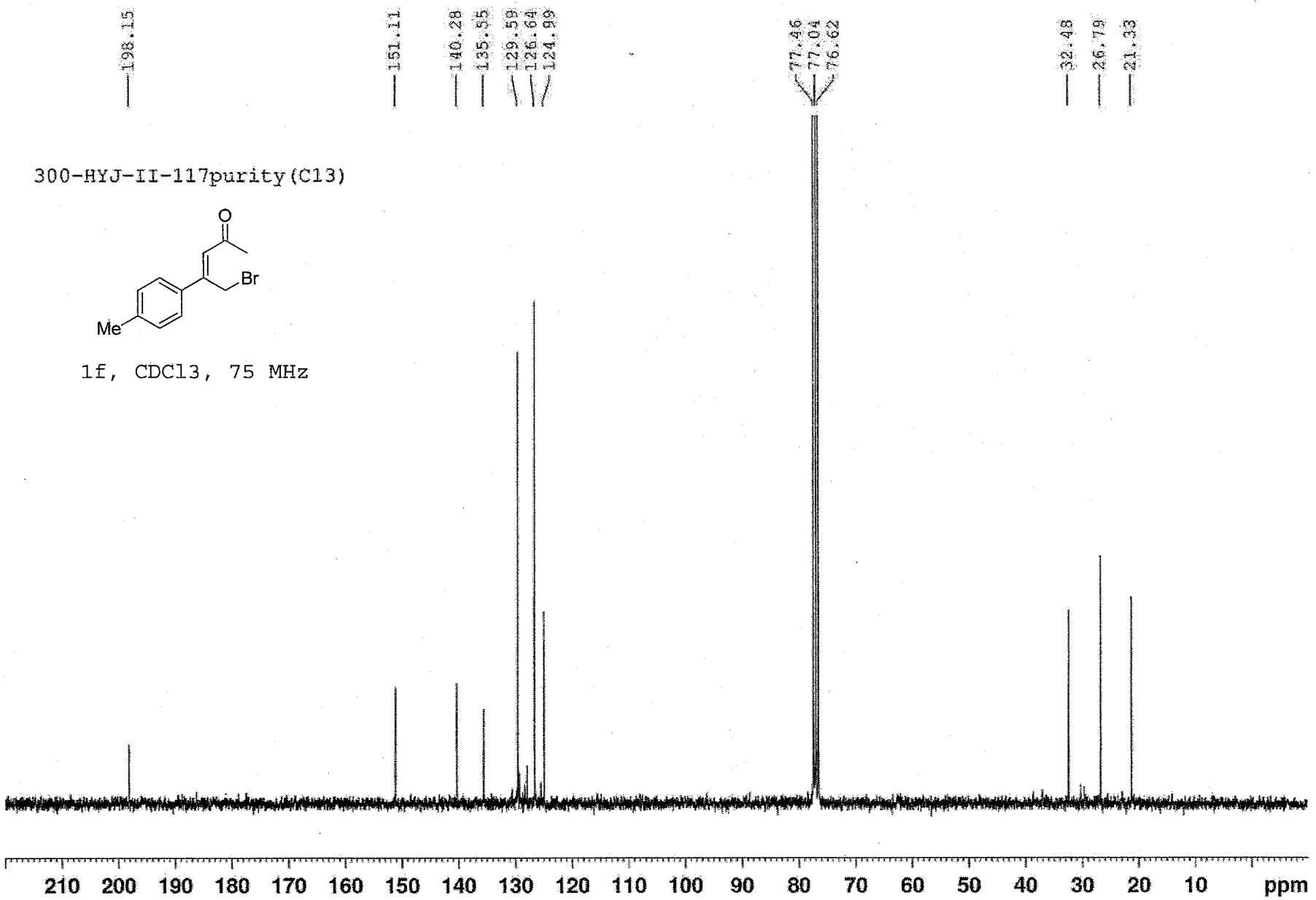



7.538
7.530
7.523
7.508
7.501
7.493
7.417
7.410
7.403
7.387
7.381
7.372
7.240
6.493
4.925
2.320
300-HYJ-II-127purity
O
Br
Br
1e, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.07
2.05
1.00
2.03
3.01


197.87
149.78
137.39
131.94
128.23
125.97
124.12
77.43
77.00
76.58
32.39
26.39
300-HYJ-II-127(c13)
O
Br
Br
1e, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

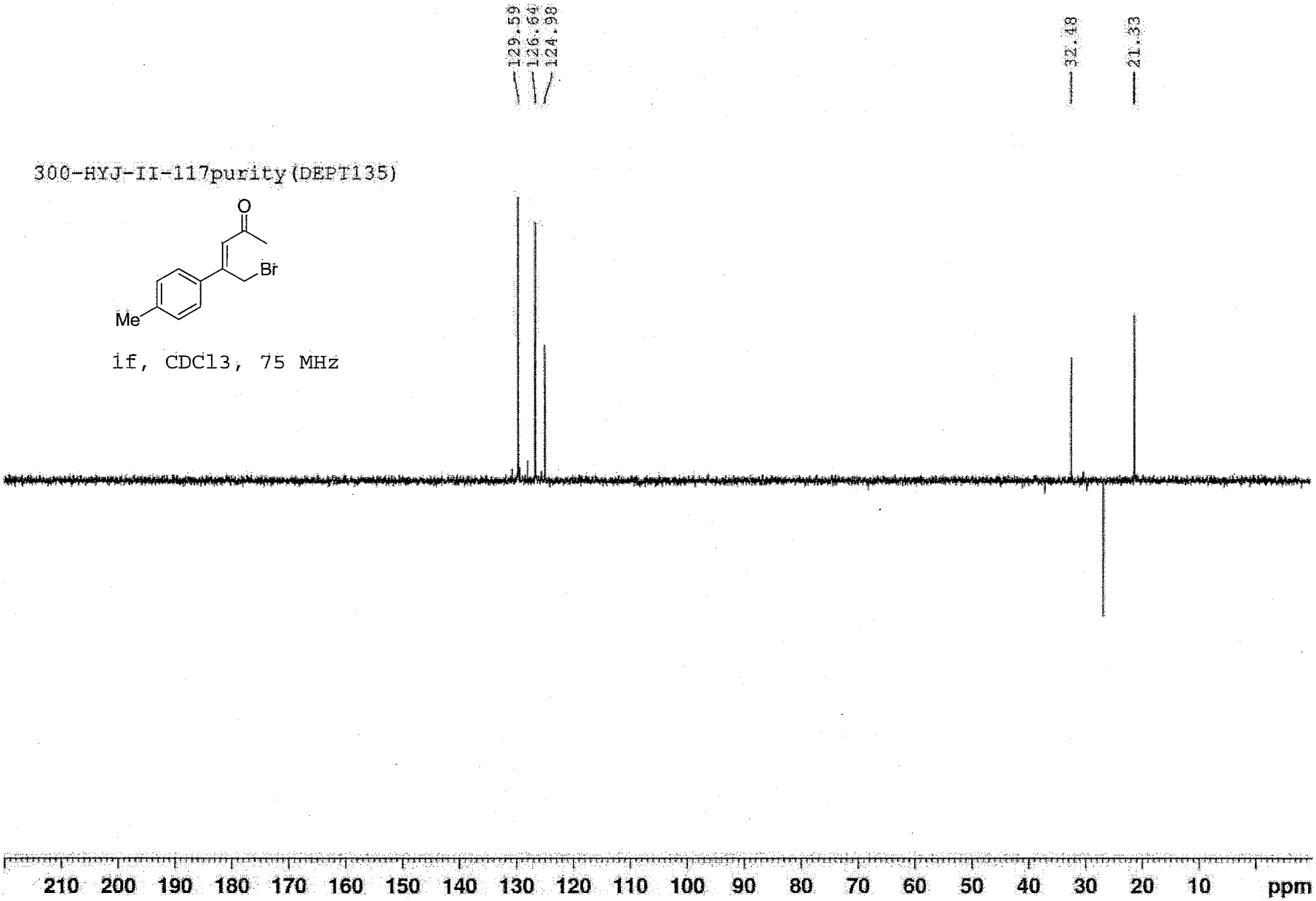

131.92
128.22
125.95
32.39
300-HYJ-II-127(dept135)
O
Br
Br
1e, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

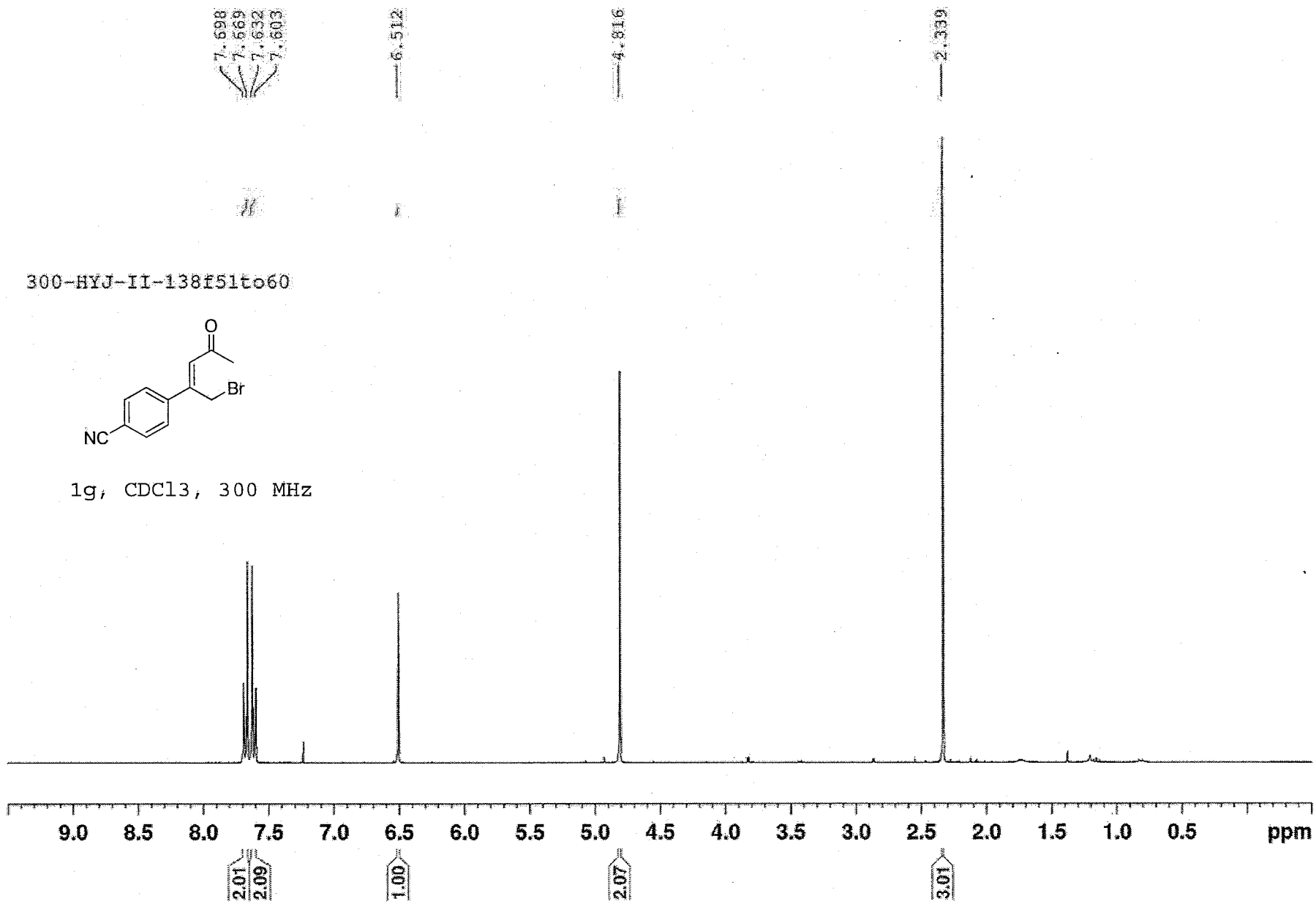

300-HYJ-II-117fE
1f, CDCl3, 300 MHz
Me
O
Br
7.465
7.458
7.443
7.437
7.240
7.224
7.204
7.197
7.196
6.525
4.887
2.372
2.325
2.02
2.00
1.00
1.97
3.05
3.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

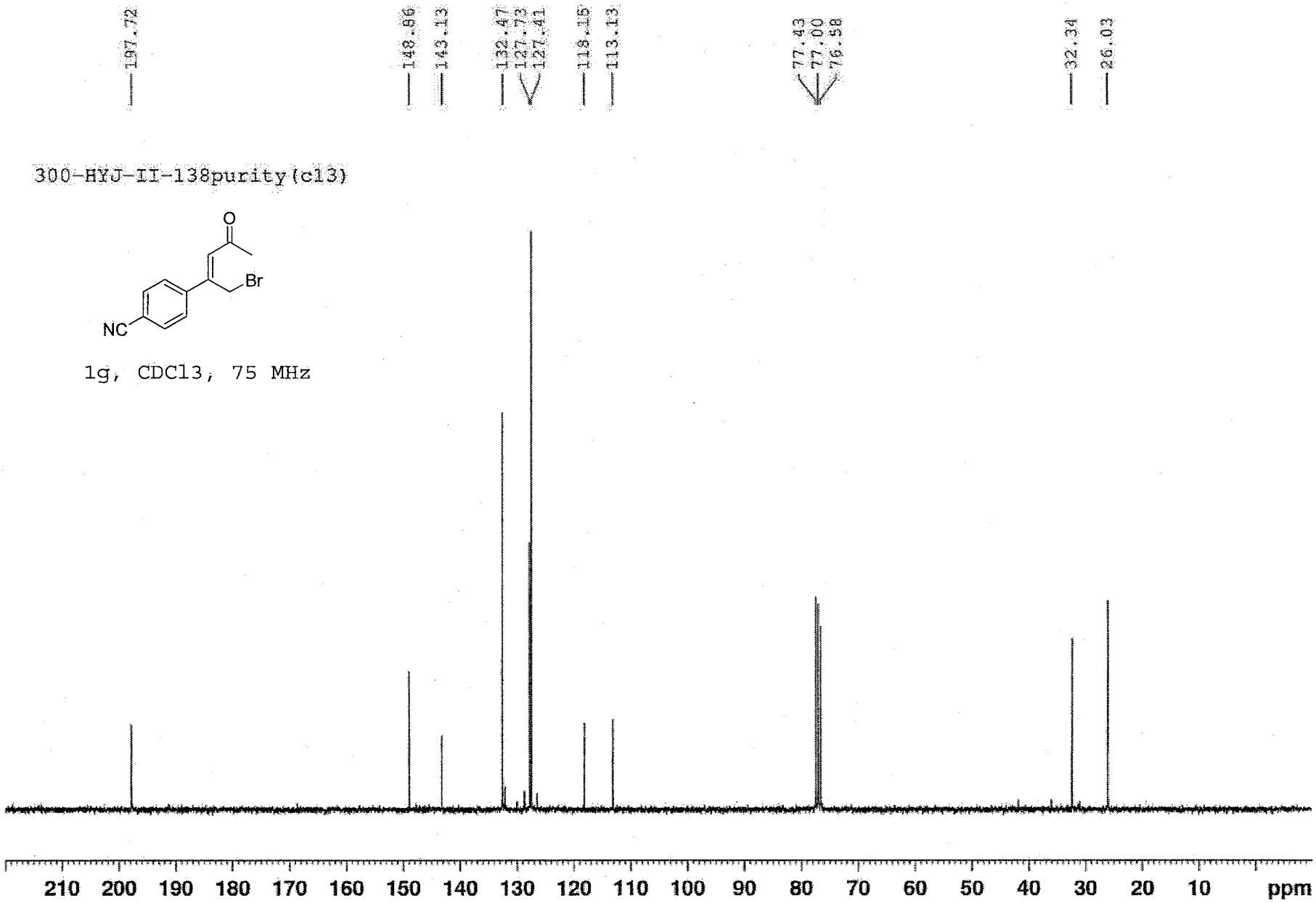

198.15
151.11
140.28
135.55
129.59
126.64
124.99
77.46
77.04
76.62
32.48
26.79
21.33
300-HYJ-II-117purity(C13)
O
Br
Me
1f, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

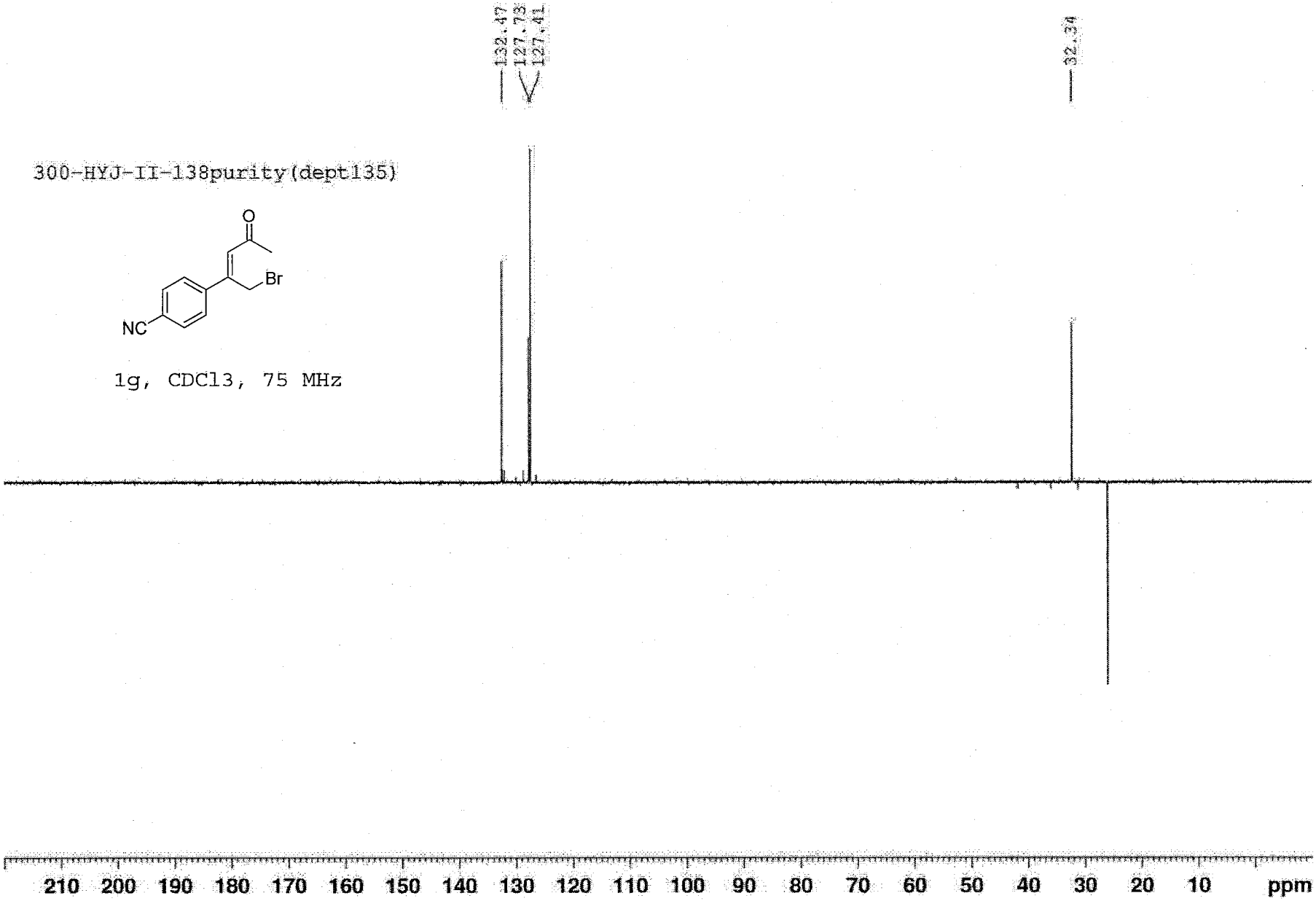
300-HYJ-II-117purity(DEPT135)

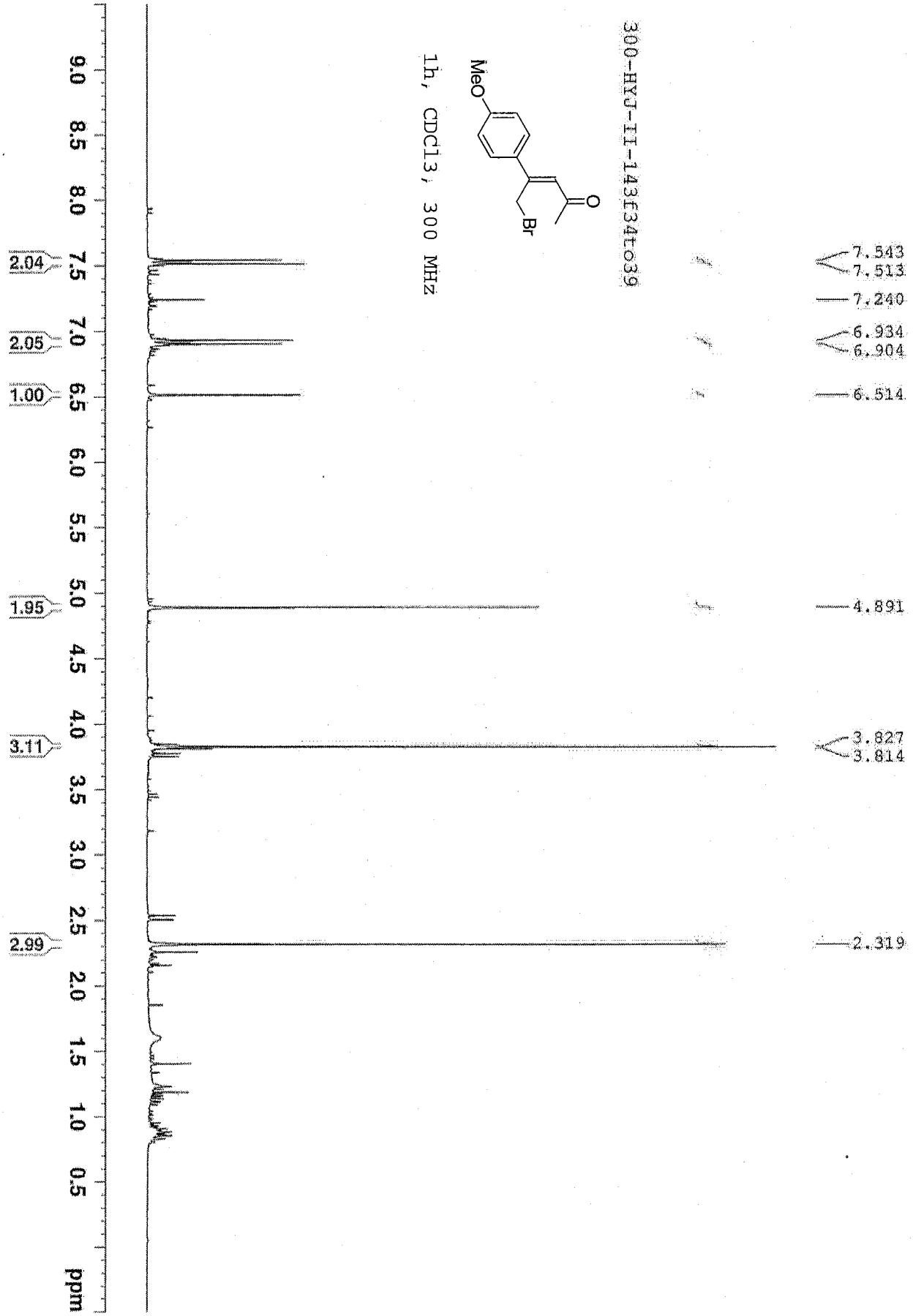
1f, CDCl3, 75 MHz

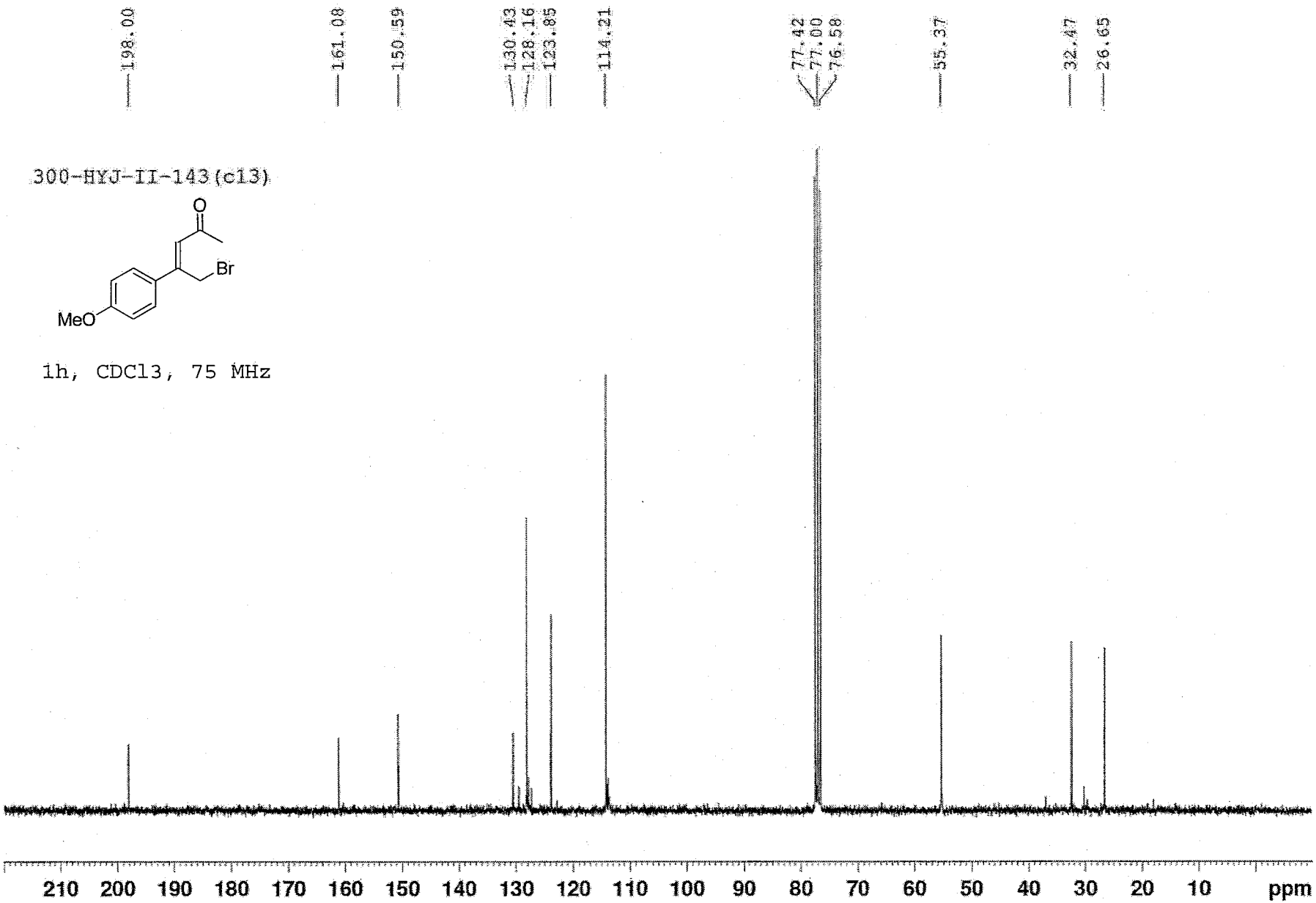



7.698
7.669
7.632
7.603
6.512
4.816
2.339
300-HYJ-II-138f51to60
O
Br
NC
1g, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.01
2.09
1.00
2.07
3.01


197.72
148.86
143.13
132.47
127.73
127.41
118.15
113.13
77.43
77.00
76.58
32.34
26.03
300-HYJ-II-138purity(c13)
O
Br
NC
1g, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

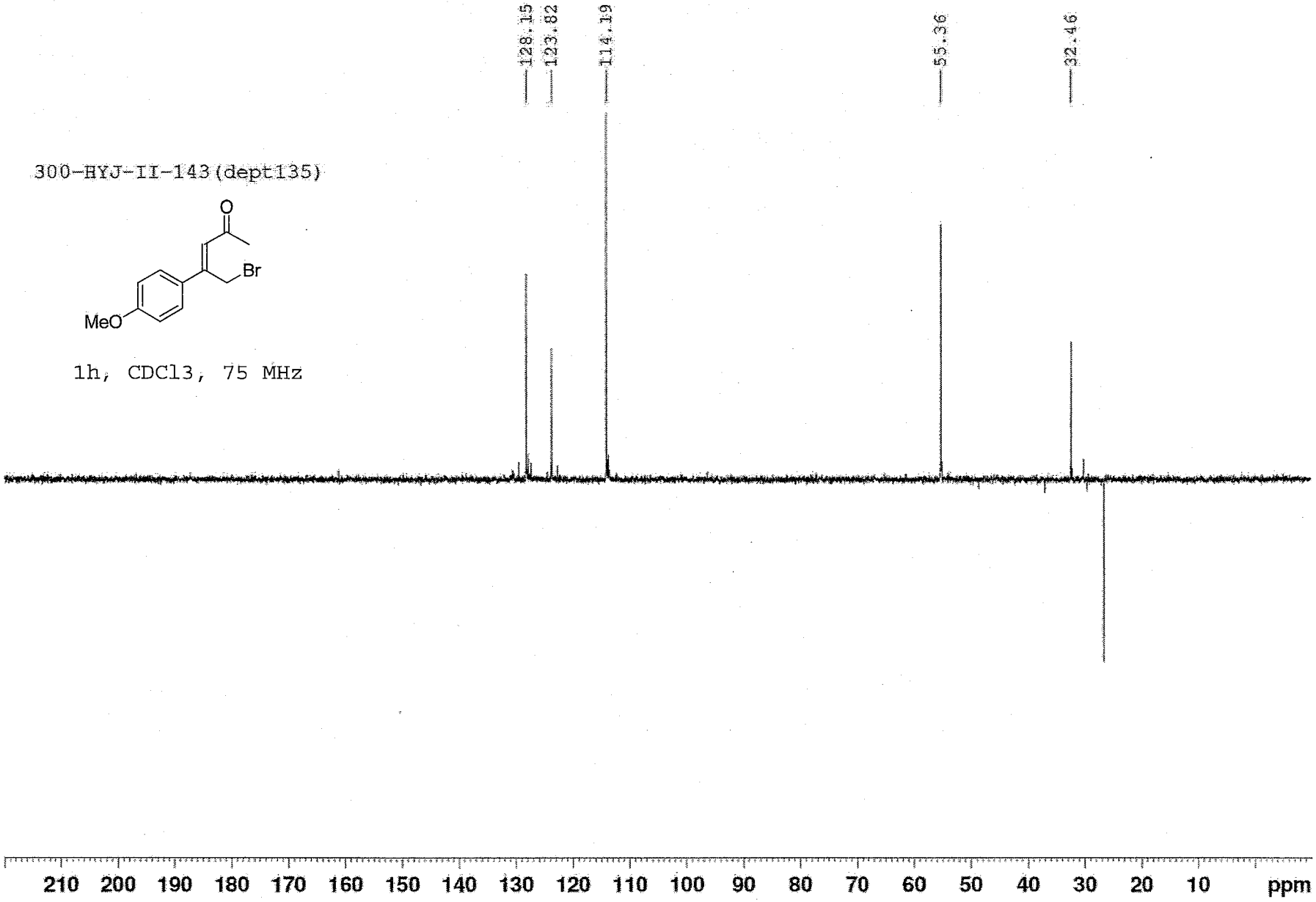

132.47
127.73
127.41
32.34
300-HYJ-II-138purity(dept135)
O
Br
NC
1g, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

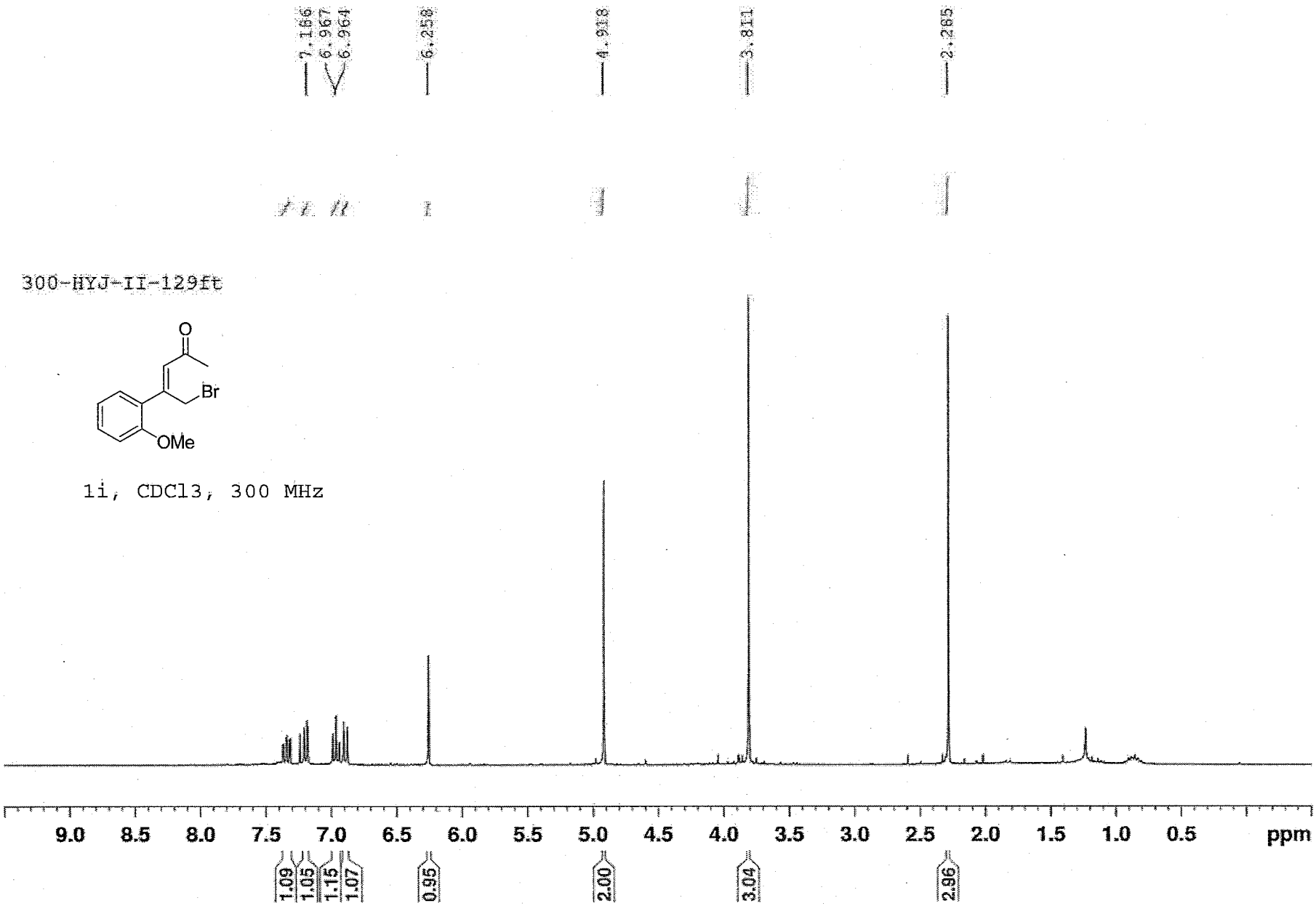

300-HYJ-II-143f34to39
1h, CDCl3, 300 MHz
MeO
Br
O
7.543
7.513
7.240
6.934
6.904
6.514
4.891
3.827
3.814
2.319
2.04
2.05
1.00
1.95
3.11
2.99
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


198.00
161.08
150.59
130.43
128.16
123.85
114.21
77.42
77.00
76.58
55.37
32.47
26.65
300-HYJ-II-143(c13)
O
Br
MeO
1h, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

300-HYJ-II-143(dept135)

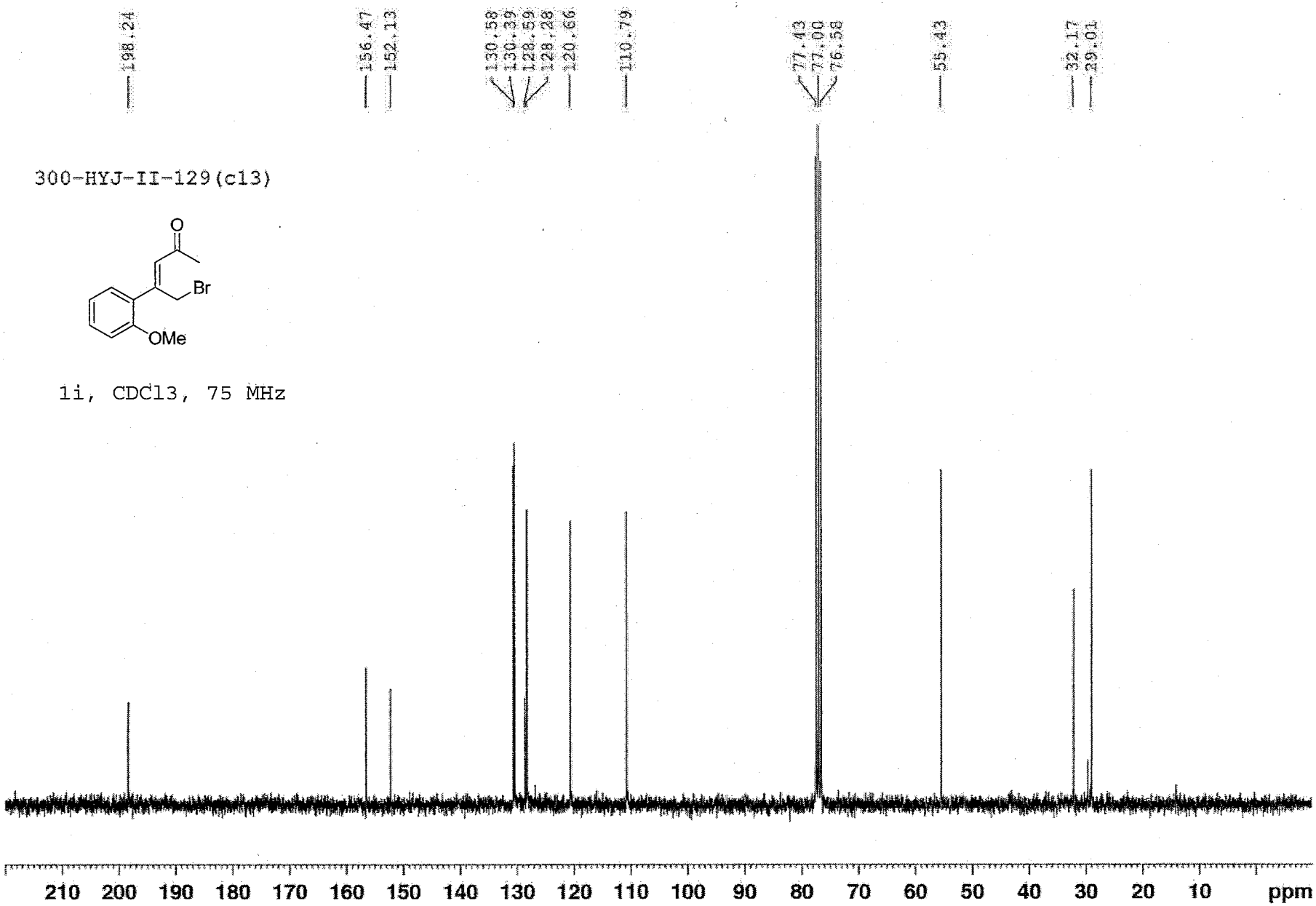
1h, CDCl3, 75 MHz

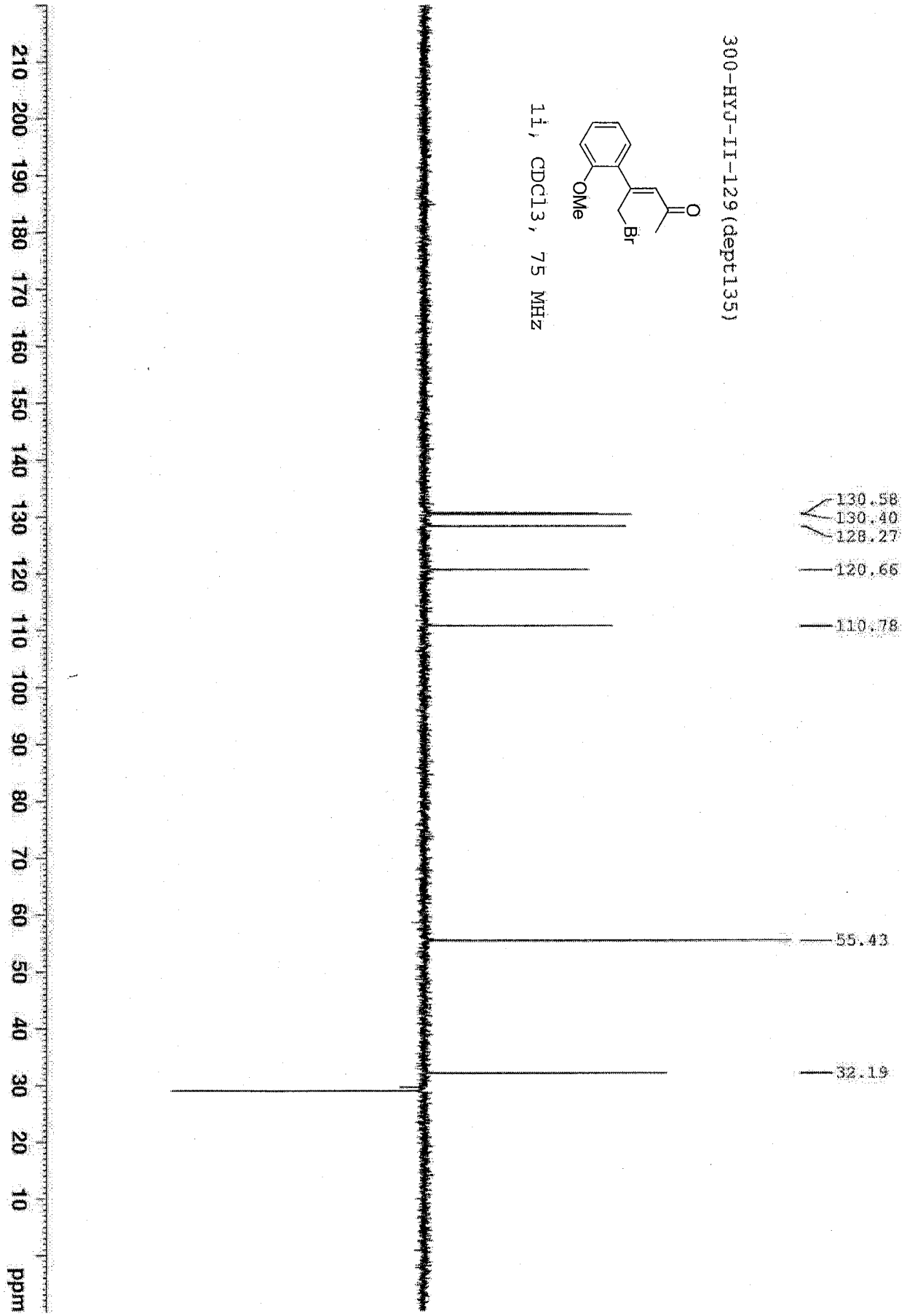


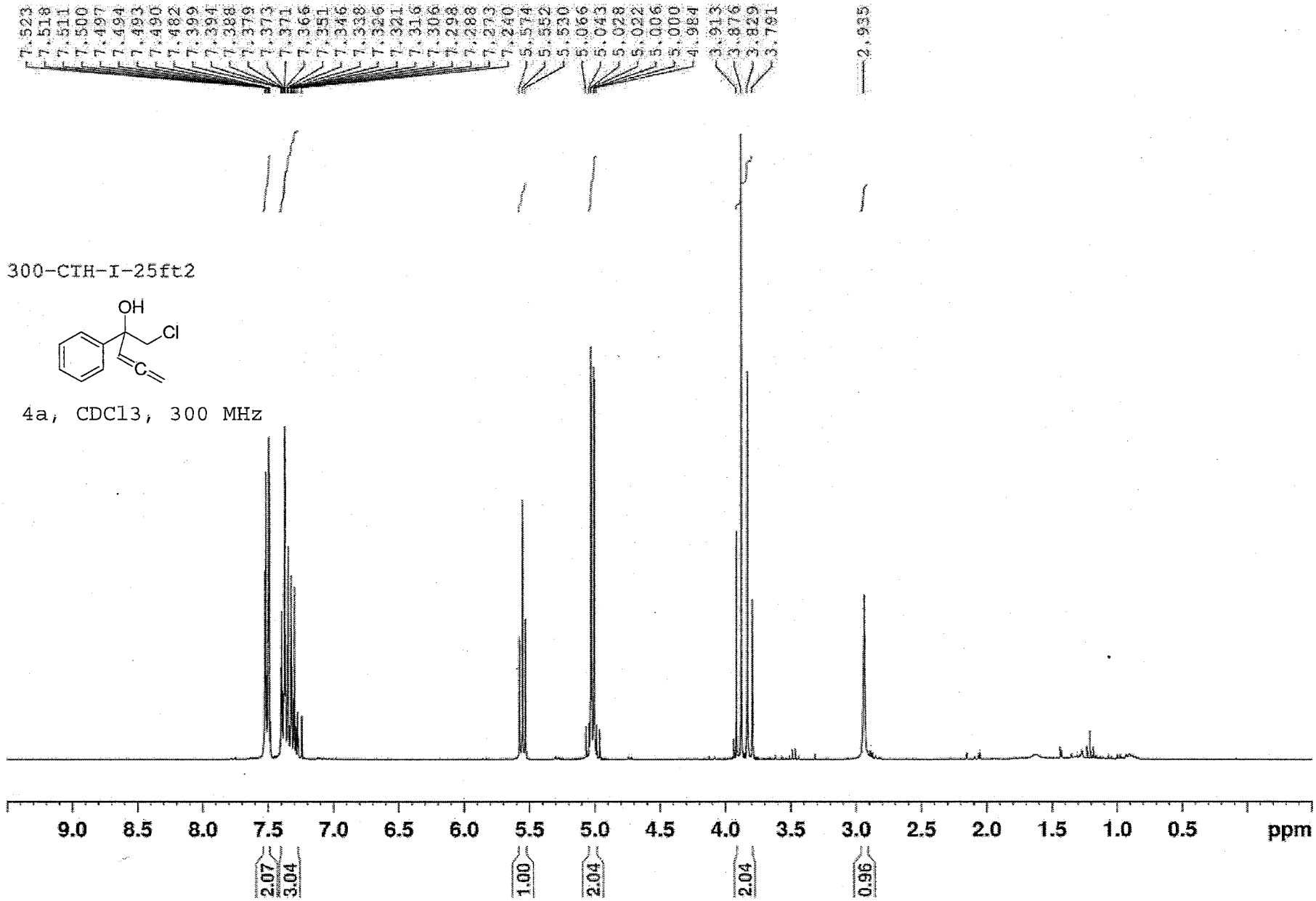

7.166
6.967
6.964
6.258
4.918
3.811
2.265
300-HYJ-II-129ft
O
Br
OMe
1i, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.09
1.05
1.15
1.07
0.95
2.00
3.04
2.96

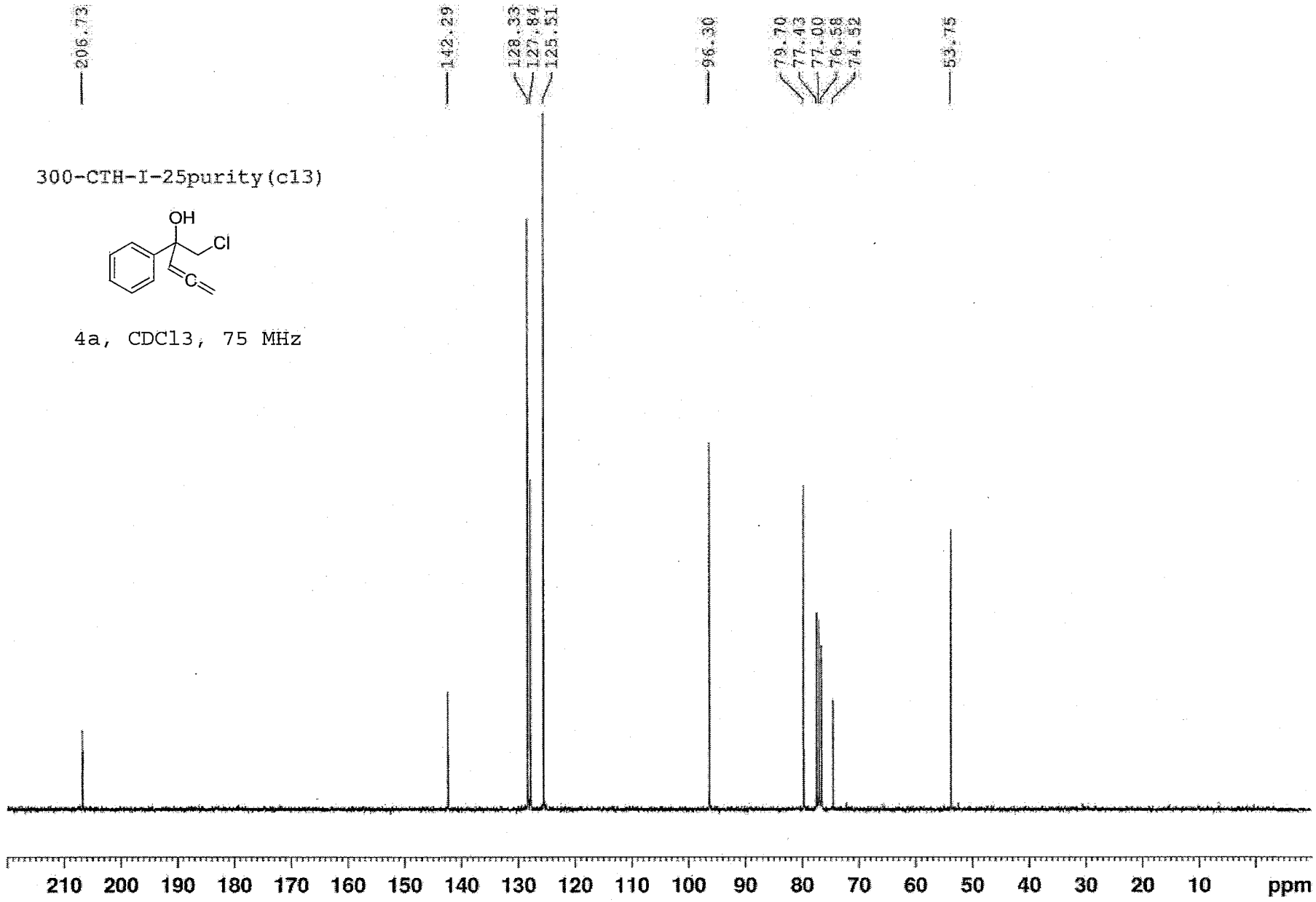
300-HYJ-II-129(c13)

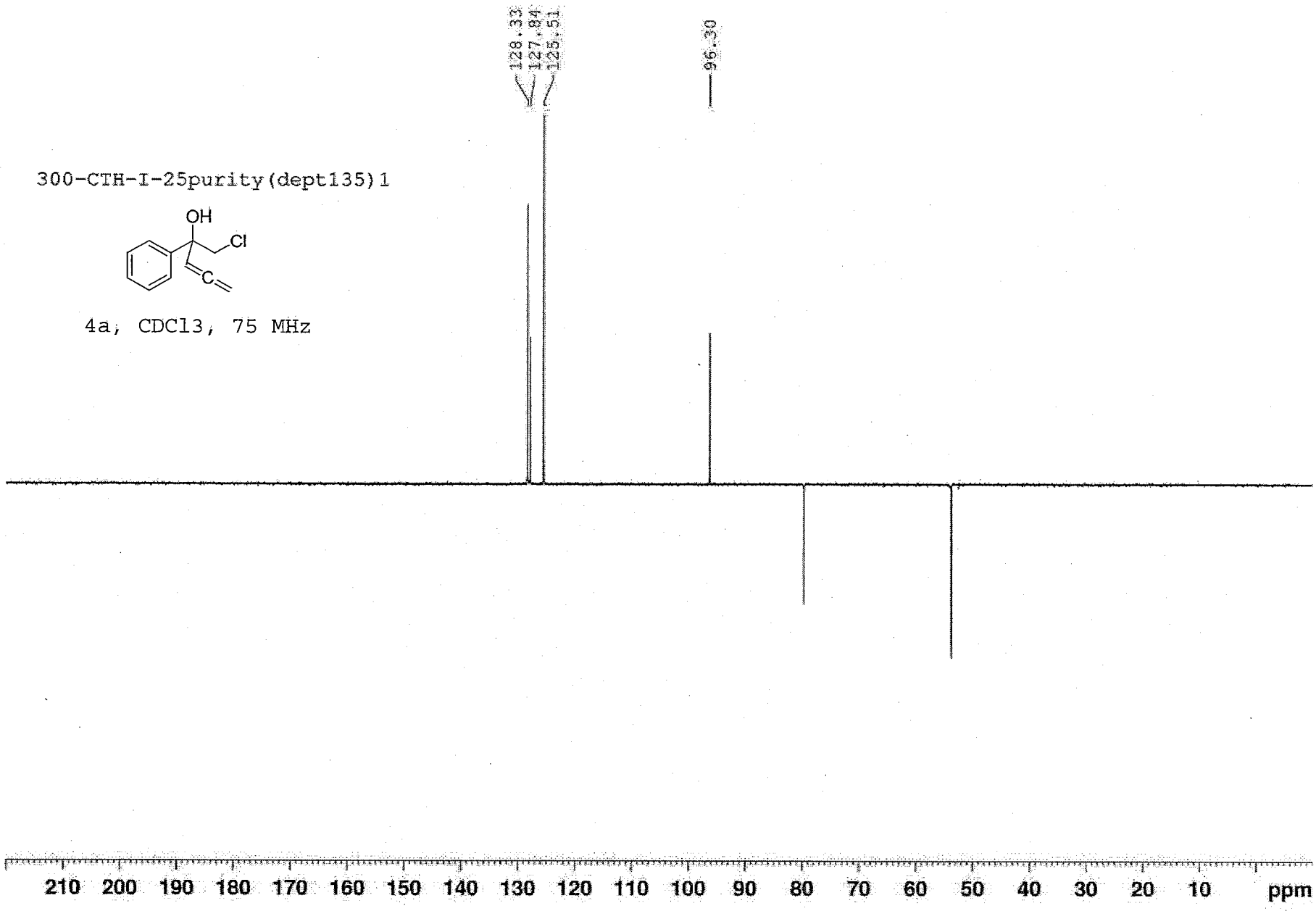
1i, CDCl3, 75 MHz

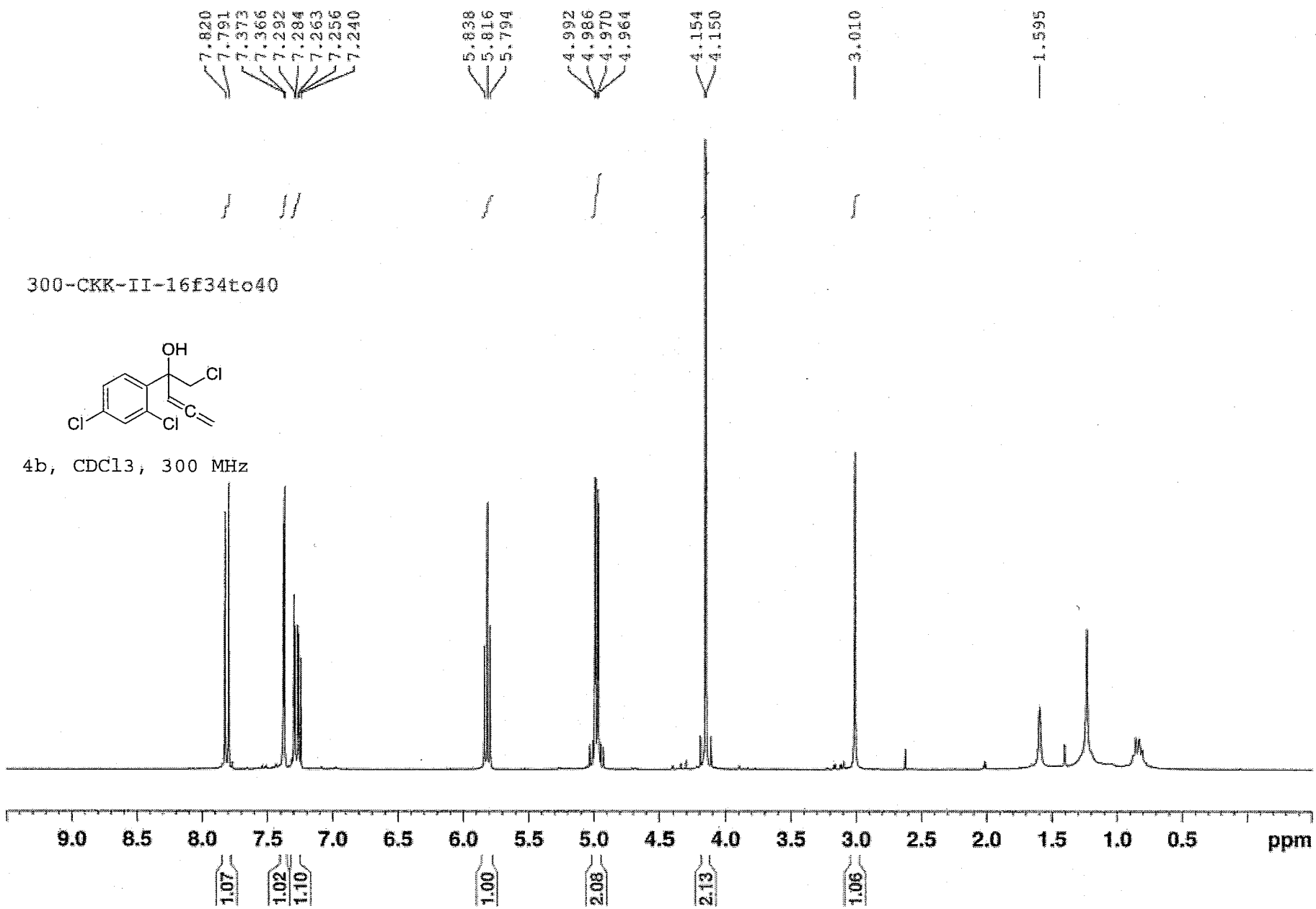


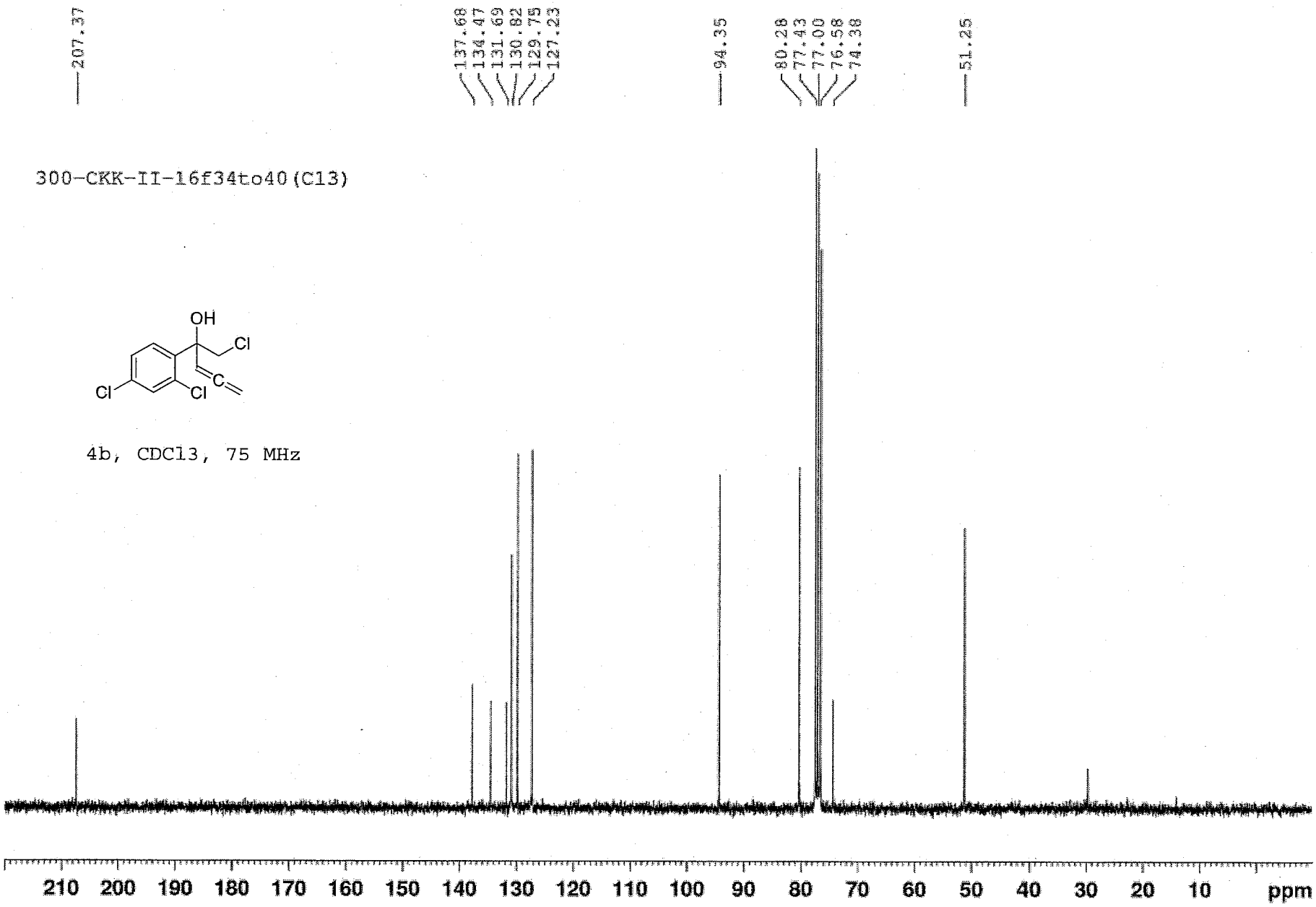

300-HYJ-II-129(dept135)
1i, CDCl3, 75 MHz
OMe
Br
O
130.58
130.40
128.27
120.66
110.78
55.43
32.19
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

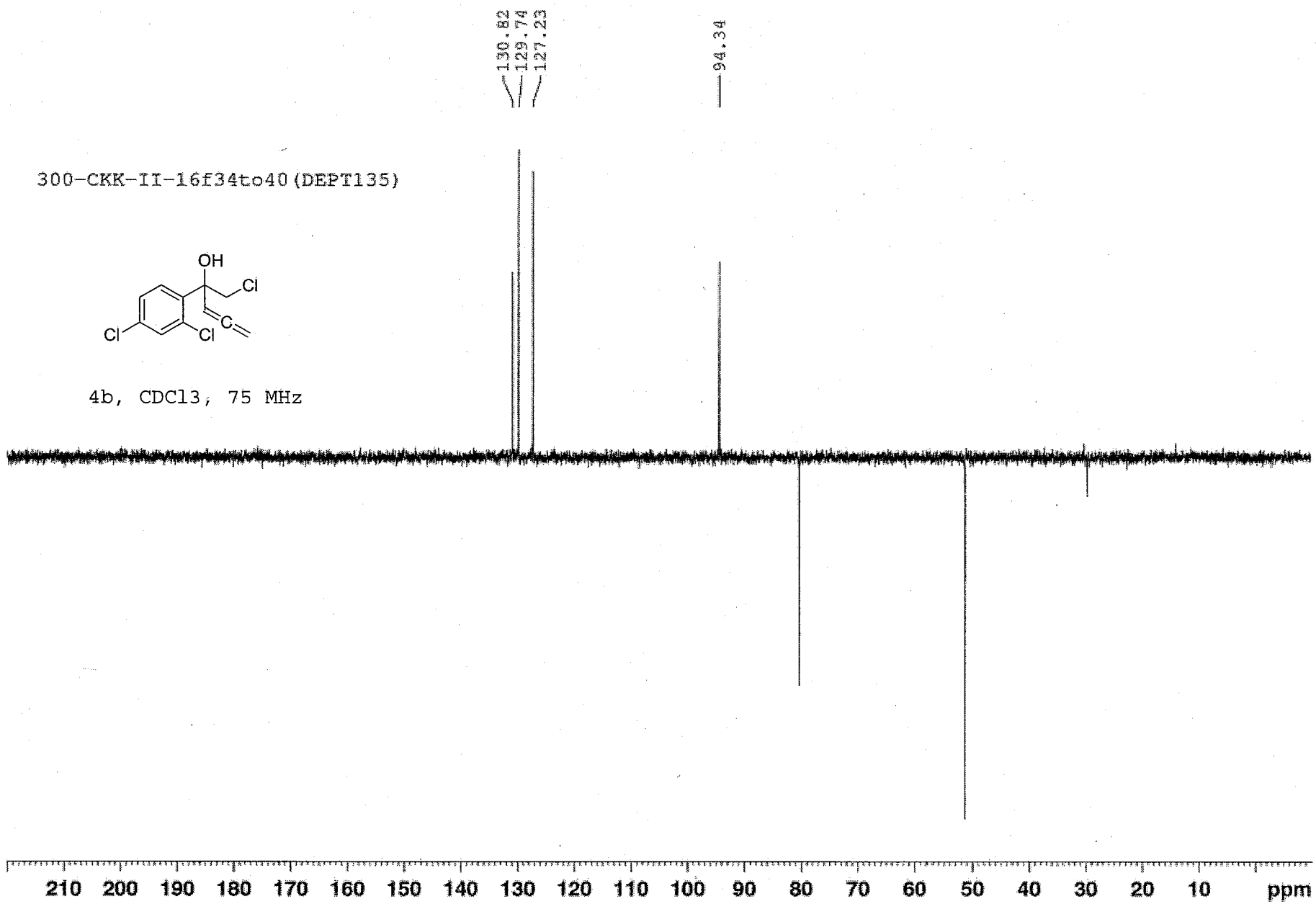

7.523
7.518
7.511
7.500
7.497
7.494
7.493
7.490
7.482
7.399
7.394
7.388
7.379
7.373
7.371
7.366
7.351
7.346
7.338
7.326
7.321
7.316
7.306
7.298
7.288
7.273
7.240
5.574
5.552
5.530
5.066
5.043
5.028
5.022
5.006
5.000
4.984
3.913
3.876
3.829
3.791
2.935
300-CTH-I-25ft2
OH
Cl
C
4a, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.07
3.04
1.00
2.04
2.04
0.96

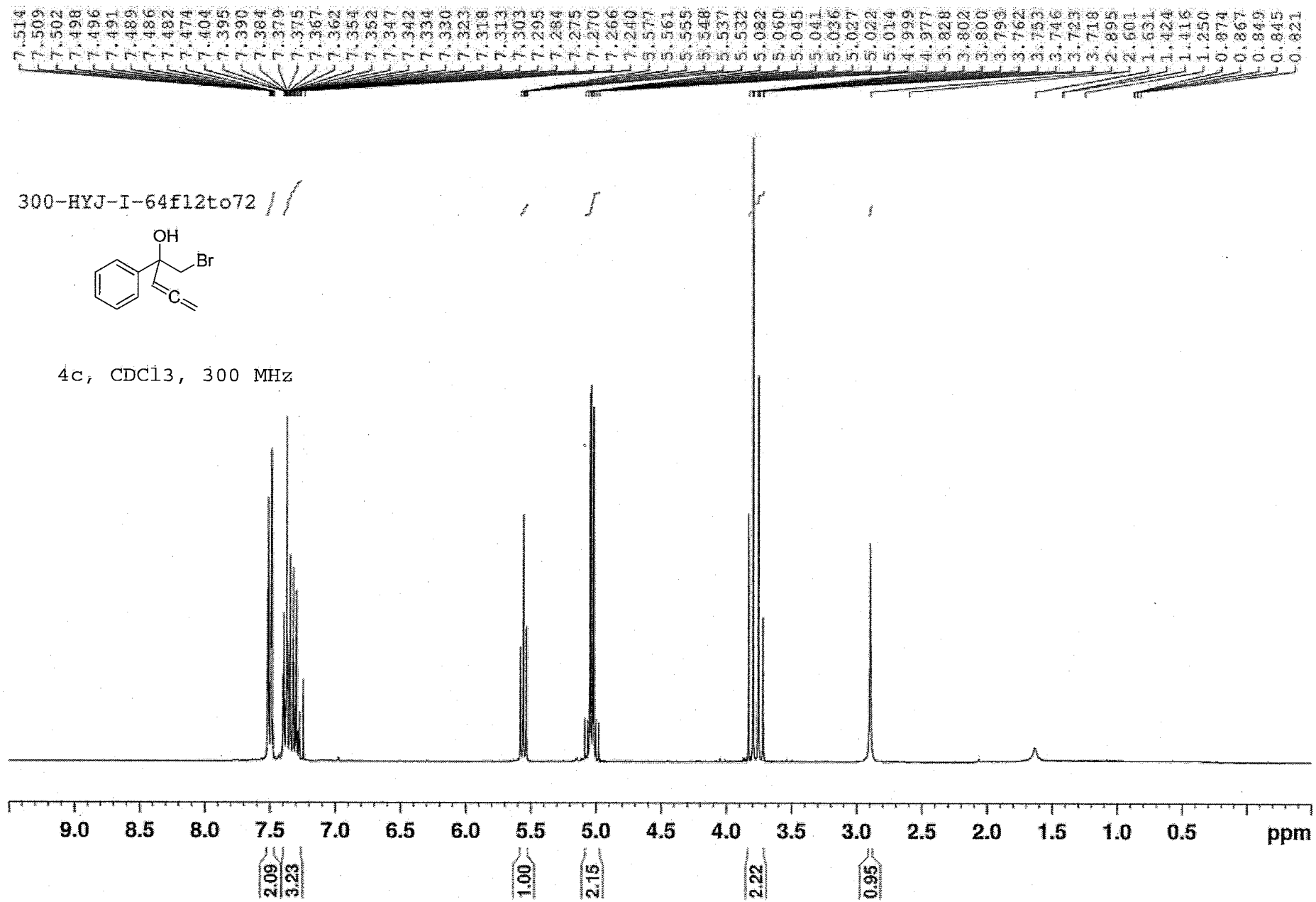

206.73
142.29
128.33
127.84
125.51
96.30
79.70
77.43
77.00
76.58
74.52
53.75
300-CTH-I-25purity(c13)
OH
Cl
C
4a, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

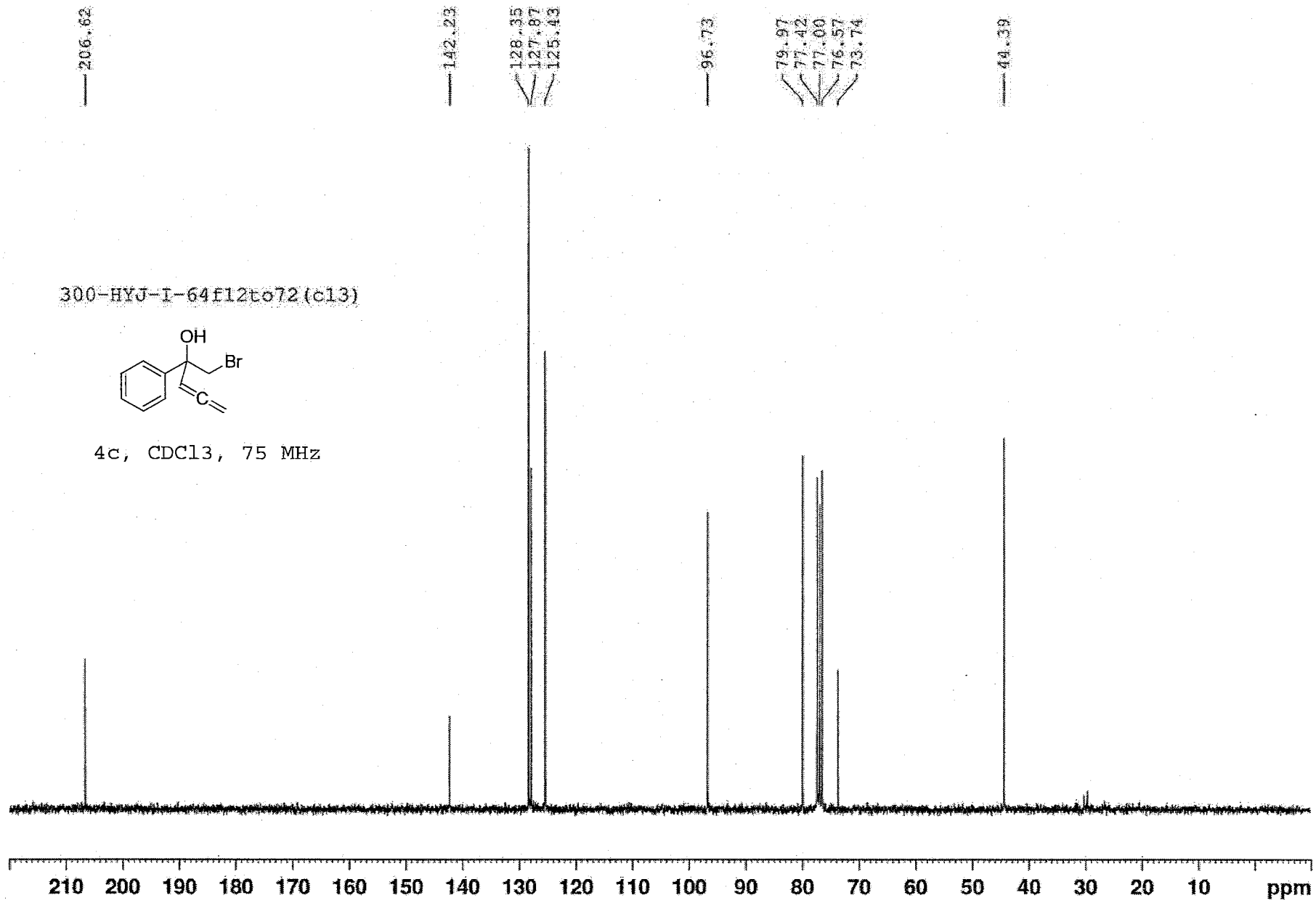

300-CTH-I-25purity(dept135)1
OH
Cl
C
4a, CDCl3, 75 MHz
128.33
127.84
125.51
96.30
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

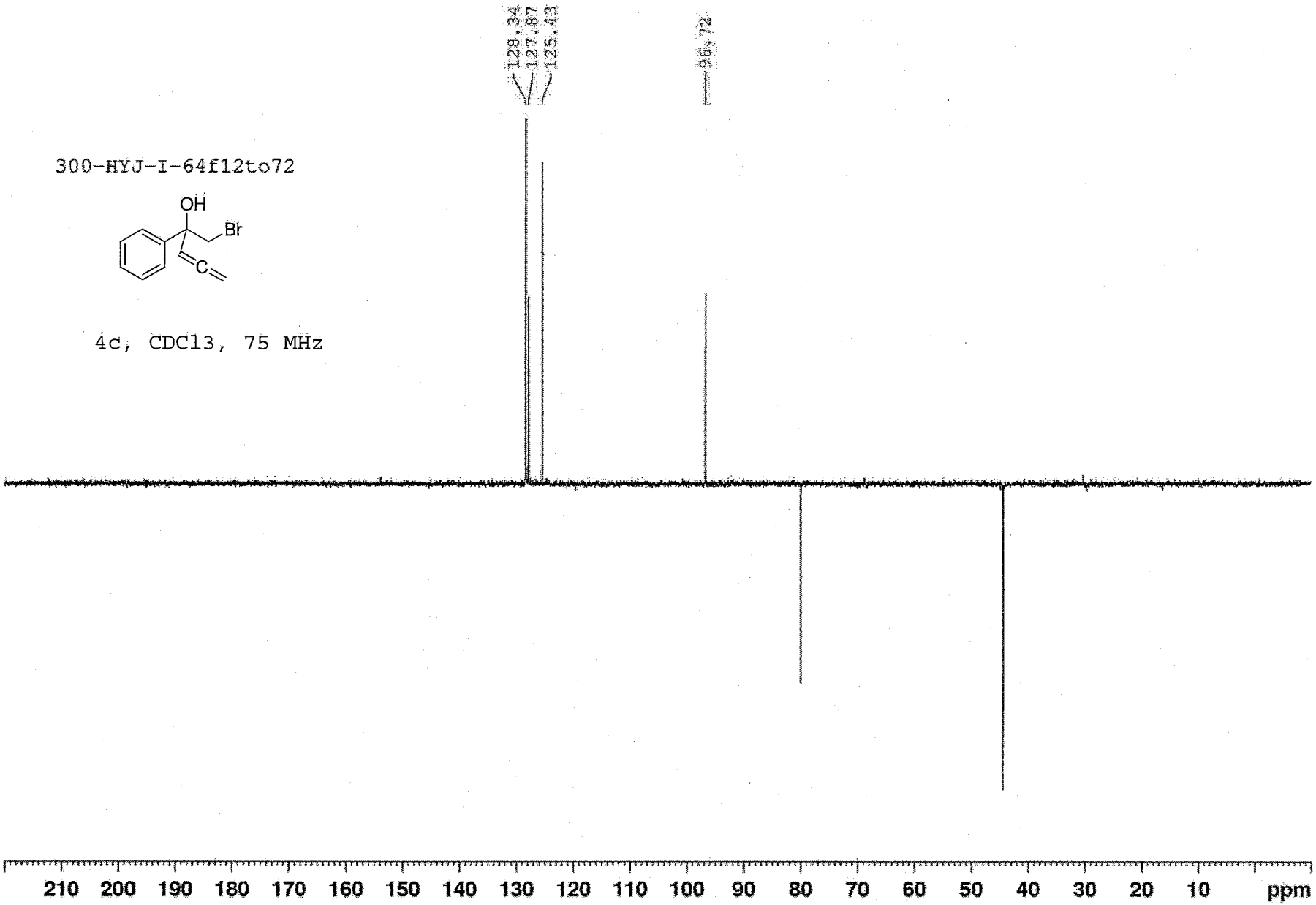

7.820
7.791
7.373
7.366
7.292
7.284
7.263
7.256
7.240
5.838
5.816
5.794
4.992
4.986
4.970
4.964
4.154
4.150
3.010
1.595
300-CKK-II-16f34to40
OH
Cl
Cl
Cl
C
4b, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.07
1.02
1.10
1.00
2.08
2.13
1.06

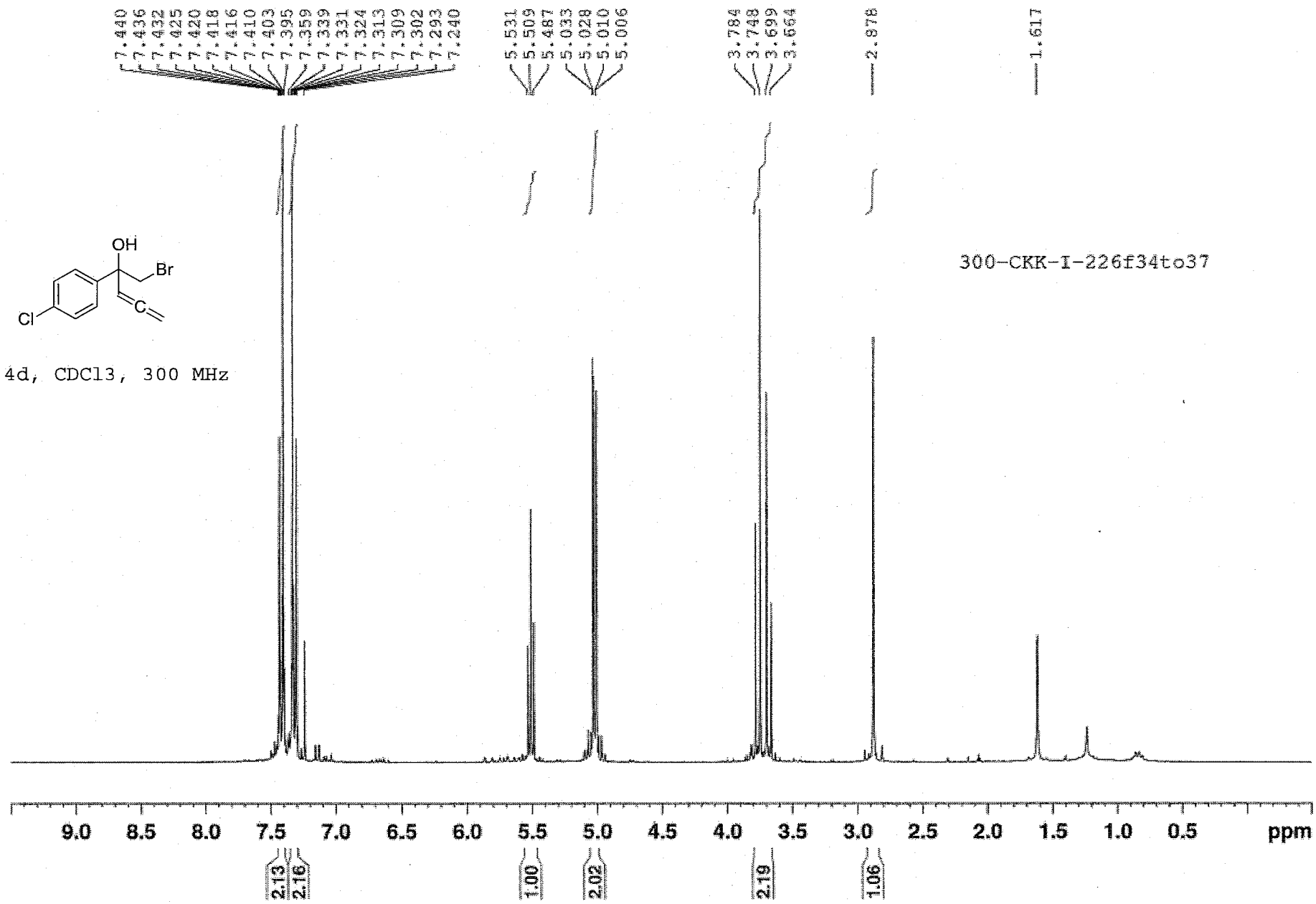

207.37
137.68
134.47
131.69
130.82
129.75
127.23
94.35
80.28
77.43
77.00
76.58
74.38
51.25
300-CKK-II-16f34to40(C13)
OH
Cl
Cl
Cl
C
4b, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

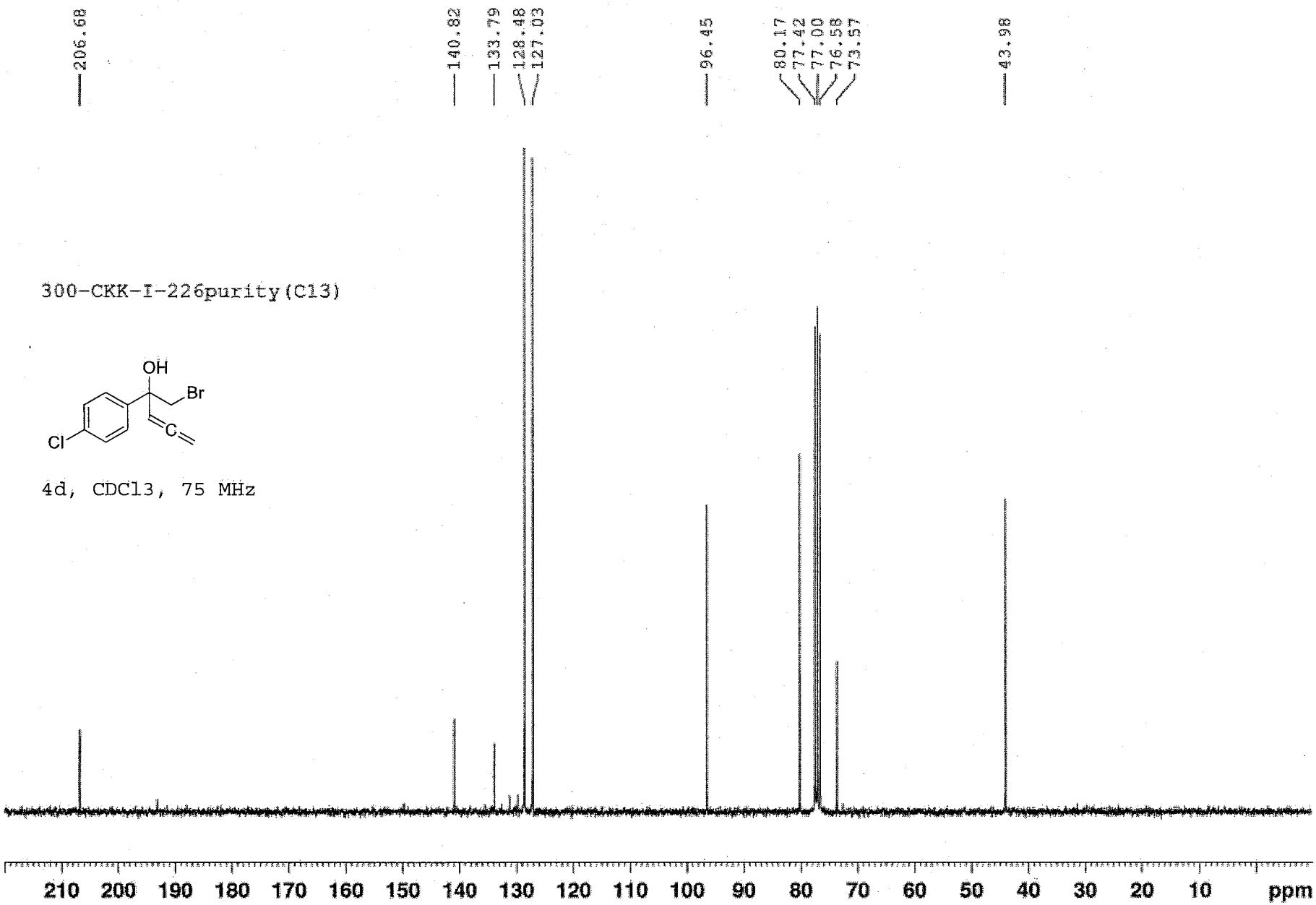

300-CKK-II-16f34to40(DEPT135)
OH
Cl
Cl
Cl
C
4b, CDCl3, 75 MHz
130.82
129.74
127.23
94.34
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.514
7.509
7.502
7.498
7.496
7.491
7.489
7.486
7.482
7.474
7.404
7.395
7.390
7.384
7.379
7.375
7.367
7.362
7.354
7.352
7.347
7.342
7.334
7.330
7.323
7.318
7.313
7.303
7.295
7.284
7.275
7.270
7.266
7.240
5.577
5.561
5.555
5.548
5.537
5.532
5.082
5.060
5.045
5.041
5.036
5.027
5.022
5.014
4.999
4.977
3.828
3.802
3.800
3.793
3.762
3.753
3.746
3.723
3.718
2.895
2.601
1.631
1.424
1.416
1.250
0.874
0.867
0.849
0.845
0.821
300-HYJ-I-64f12to72
OH
Br
C
4c, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.09
3.23
1.00
2.15
2.22
0.95


206.62
142.23
128.35
127.87
125.43
96.73
79.97
77.42
77.00
76.57
73.74
44.39
300-HYJ-I-64f12to72(c13)
OH
Br
C
4c, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

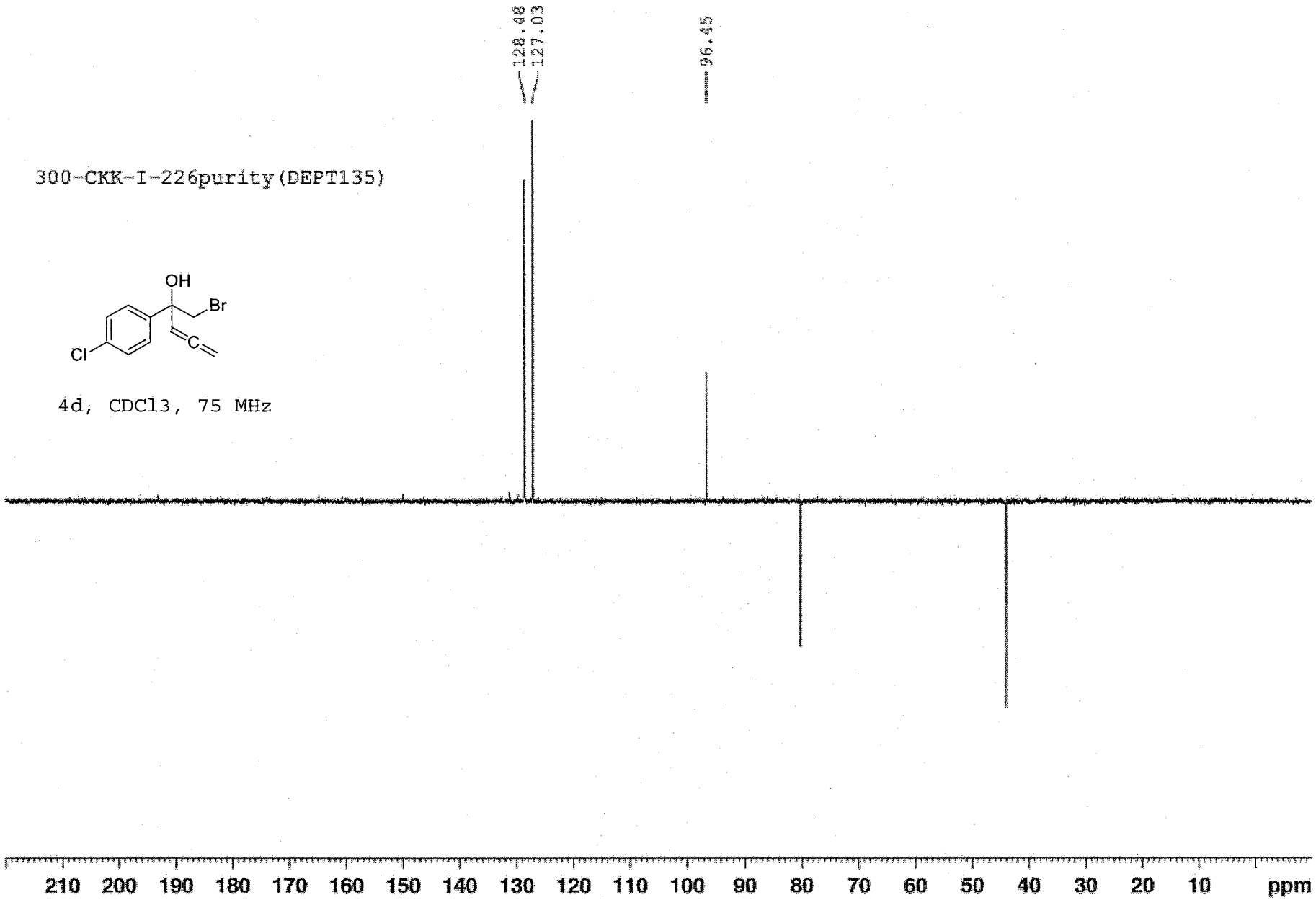

300-HYJ-I-64f12to72
OH
Br
C
4c, CDCl3, 75 MHz
128.34
127.87
125.43
96.72
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

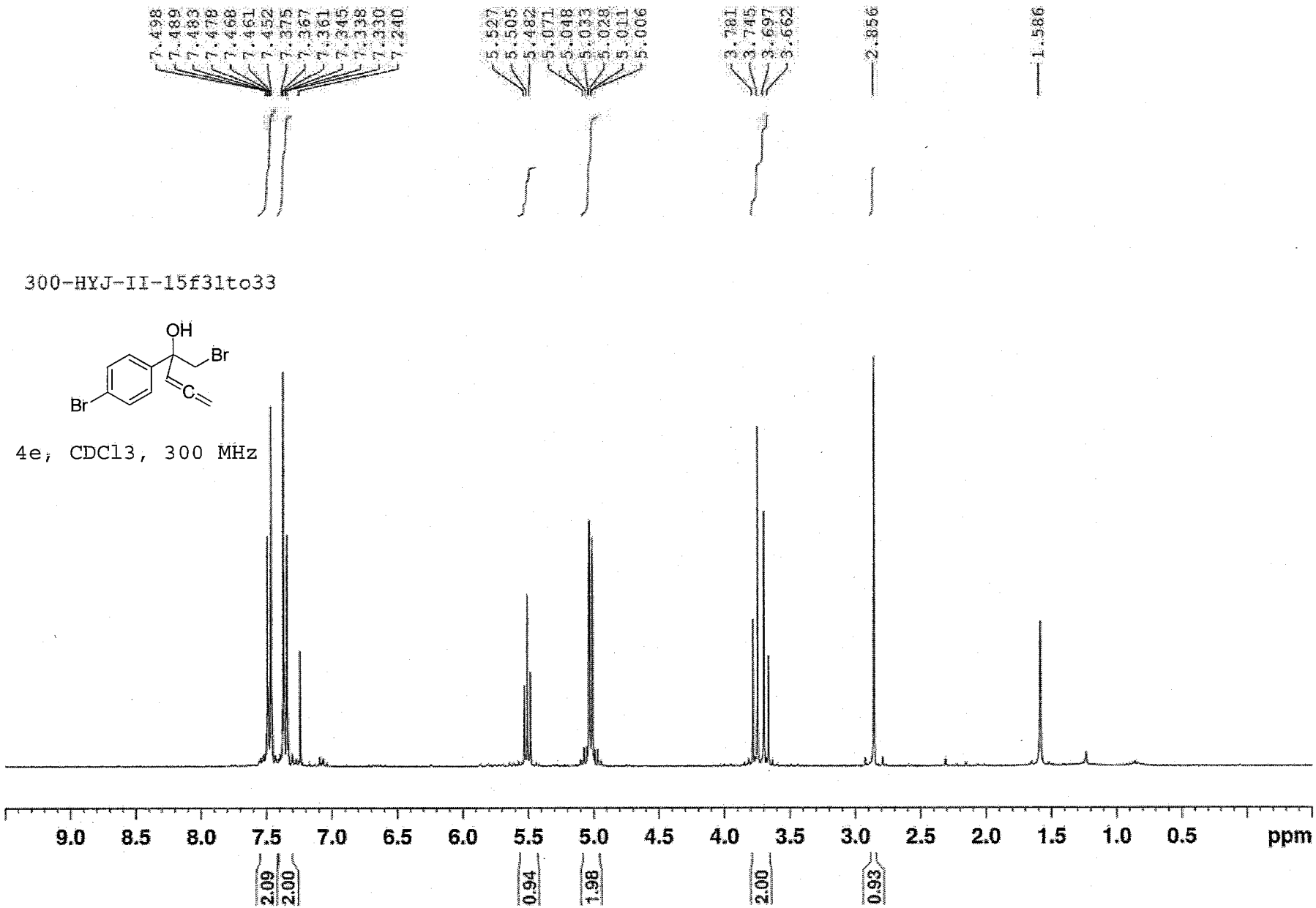

7.440
7.436
7.432
7.425
7.420
7.418
7.416
7.410
7.403
7.395
7.359
7.339
7.331
7.324
7.313
7.309
7.302
7.293
7.240
5.531
5.509
5.487
5.033
5.028
5.010
5.006
3.784
3.748
3.699
3.664
2.878
1.617
OH
Br
Cl
C
4d, CDCl3, 300 MHz
300-CKK-I-226f34to37
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.13
2.16
1.00
2.02
2.19
1.06

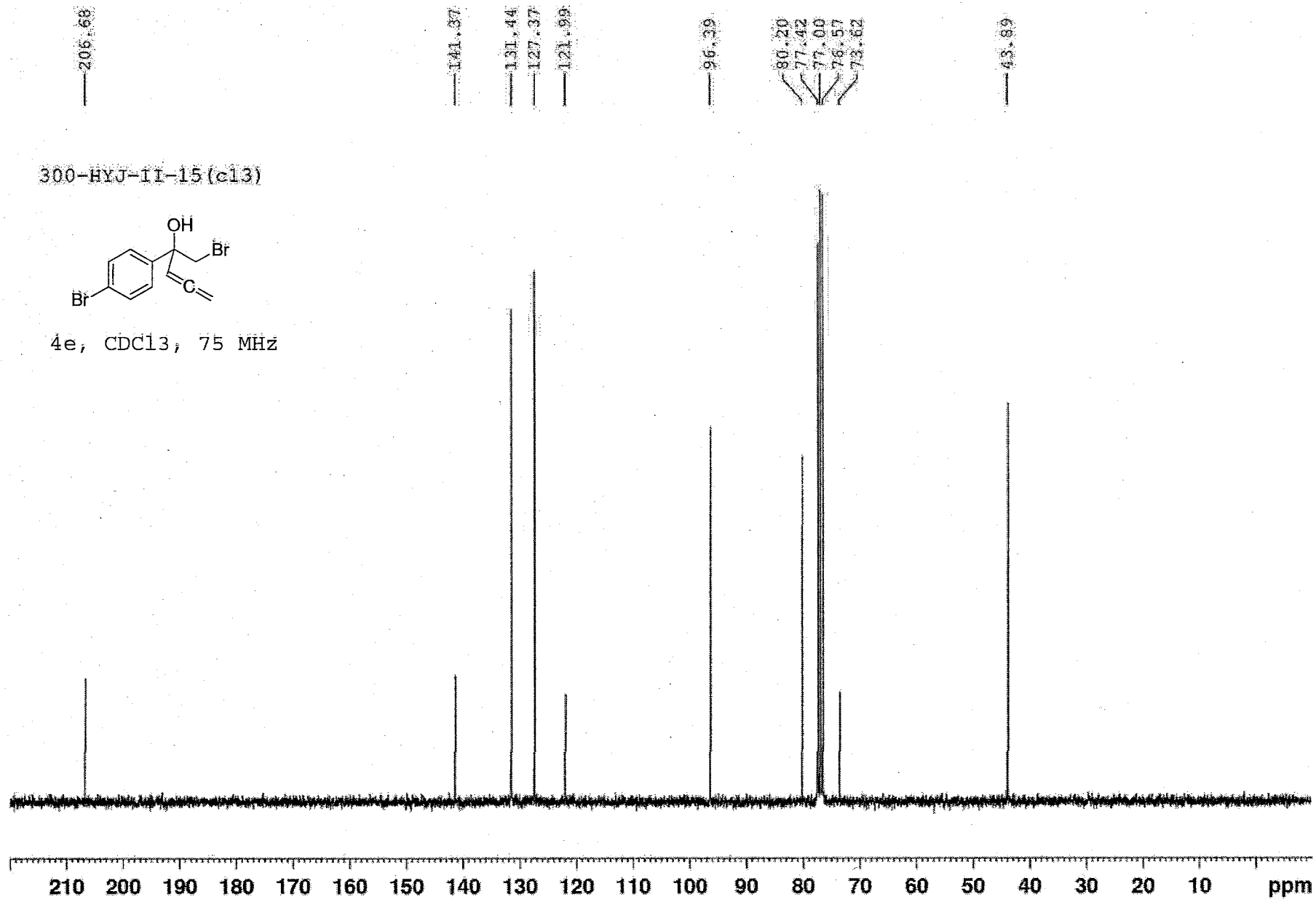

206.68
140.82
133.79
128.48
127.03
96.45
80.17
77.42
77.00
76.58
73.57
43.98
300-CKK-I-226purity(C13)
OH
Br
Cl
C
4d, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

300-CKK-I-226purity(DEPT135)

4d, CDCl3, 75 MHz

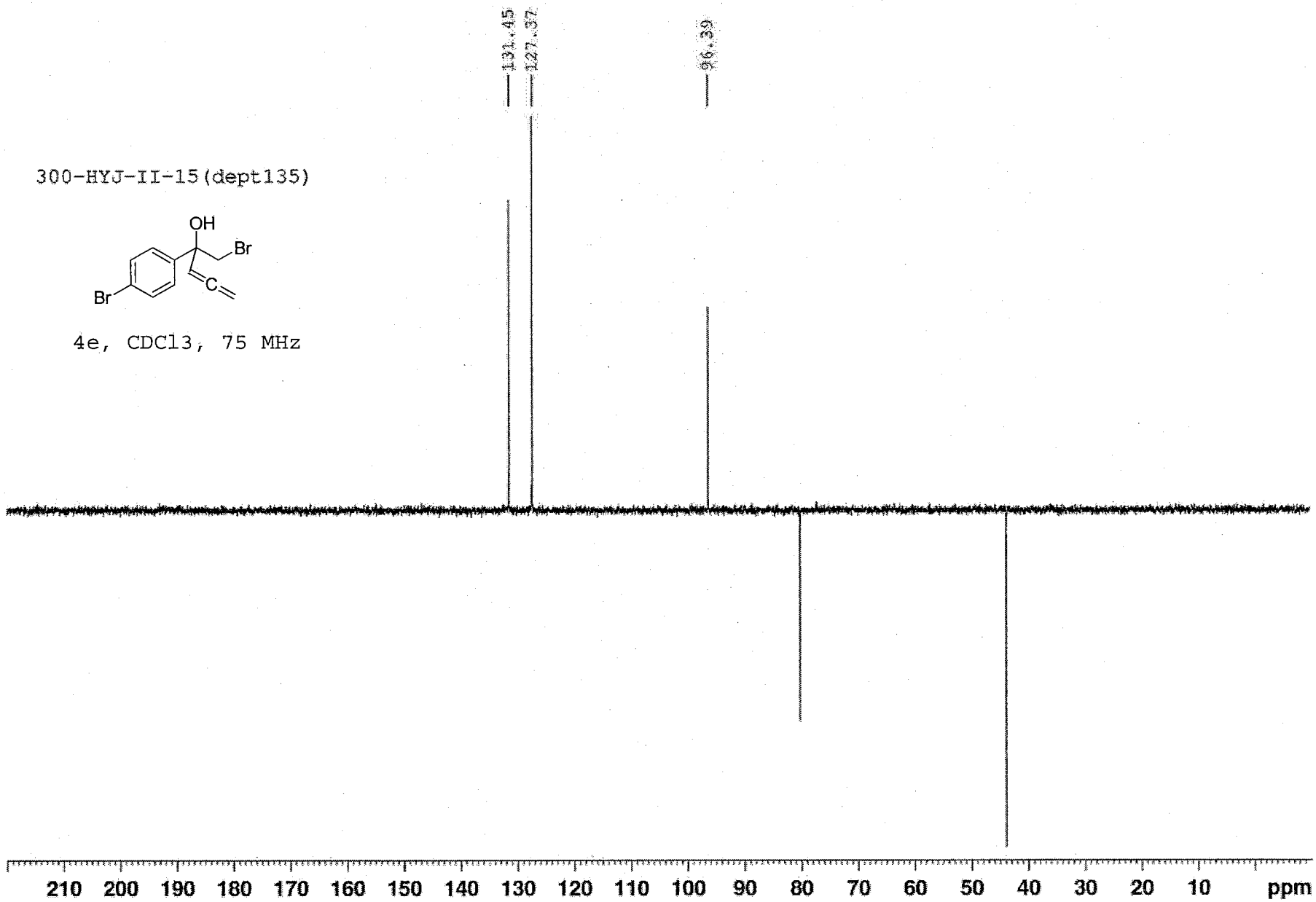


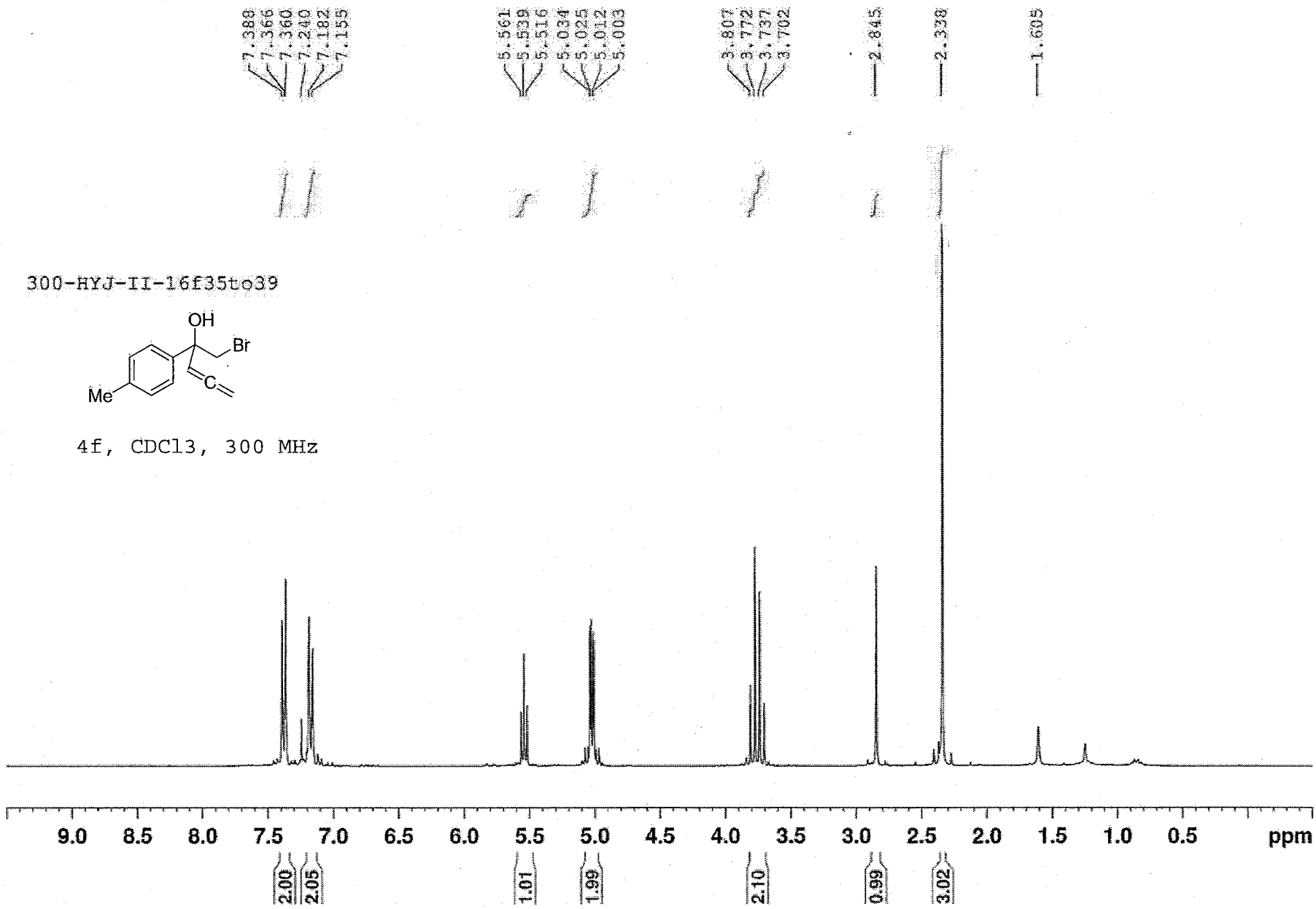

7.498
7.489
7.483
7.478
7.468
7.461
7.452
7.375
7.367
7.361
7.345
7.338
7.330
7.240
5.527
5.505
5.482
5.071
5.048
5.033
5.028
5.011
5.006
3.781
3.745
3.697
3.662
2.856
1.586
300-HYJ-II-15f31to33
OH
Br
Br
C
4e, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.09
2.00
0.94
1.98
2.00
0.93

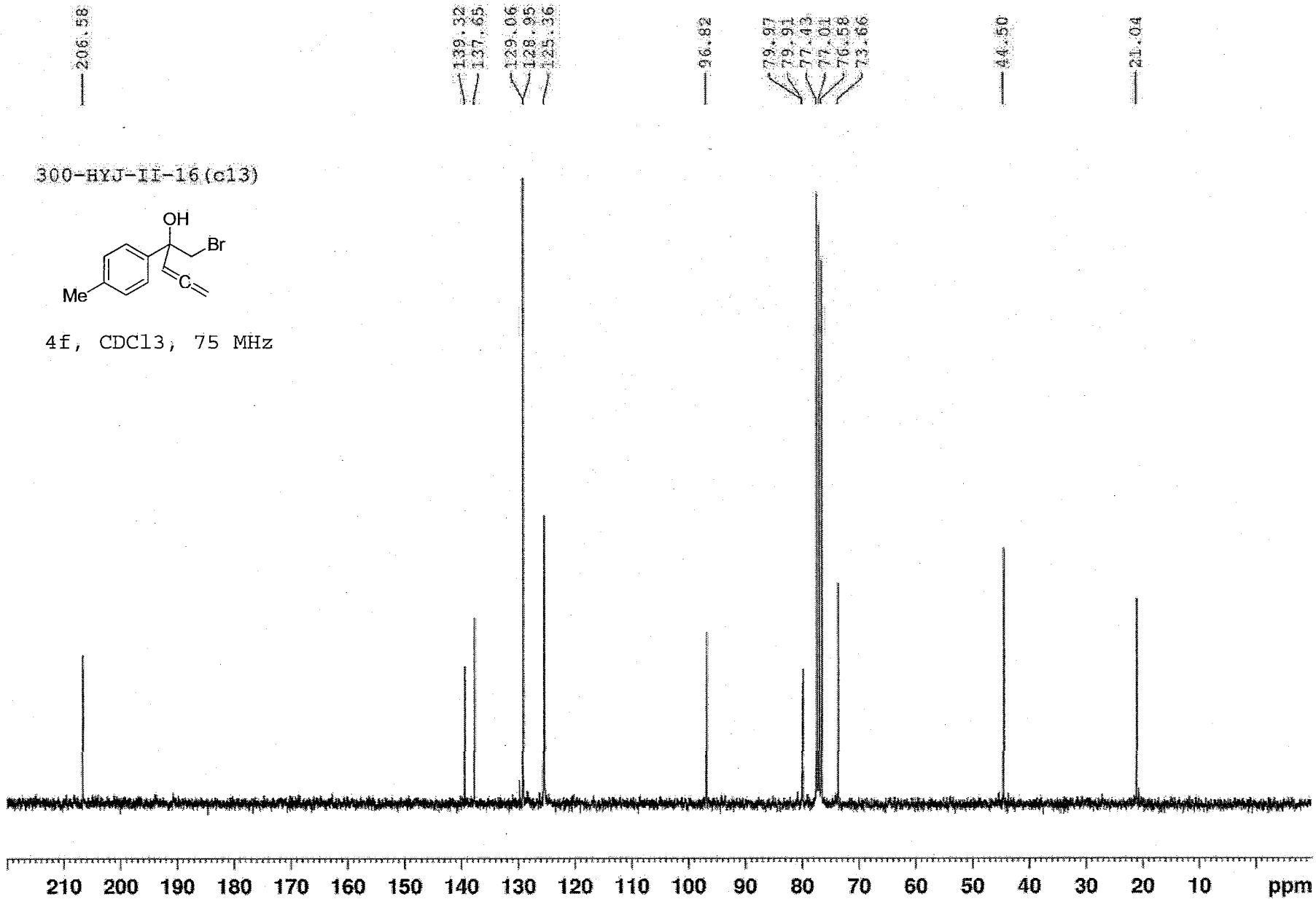

206.68
141.37
131.44
127.37
121.99
96.39
80.20
77.42
77.00
76.57
73.62
43.89
300-HYJ-II-15(c13)
OH
Br
Br
C
4e, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

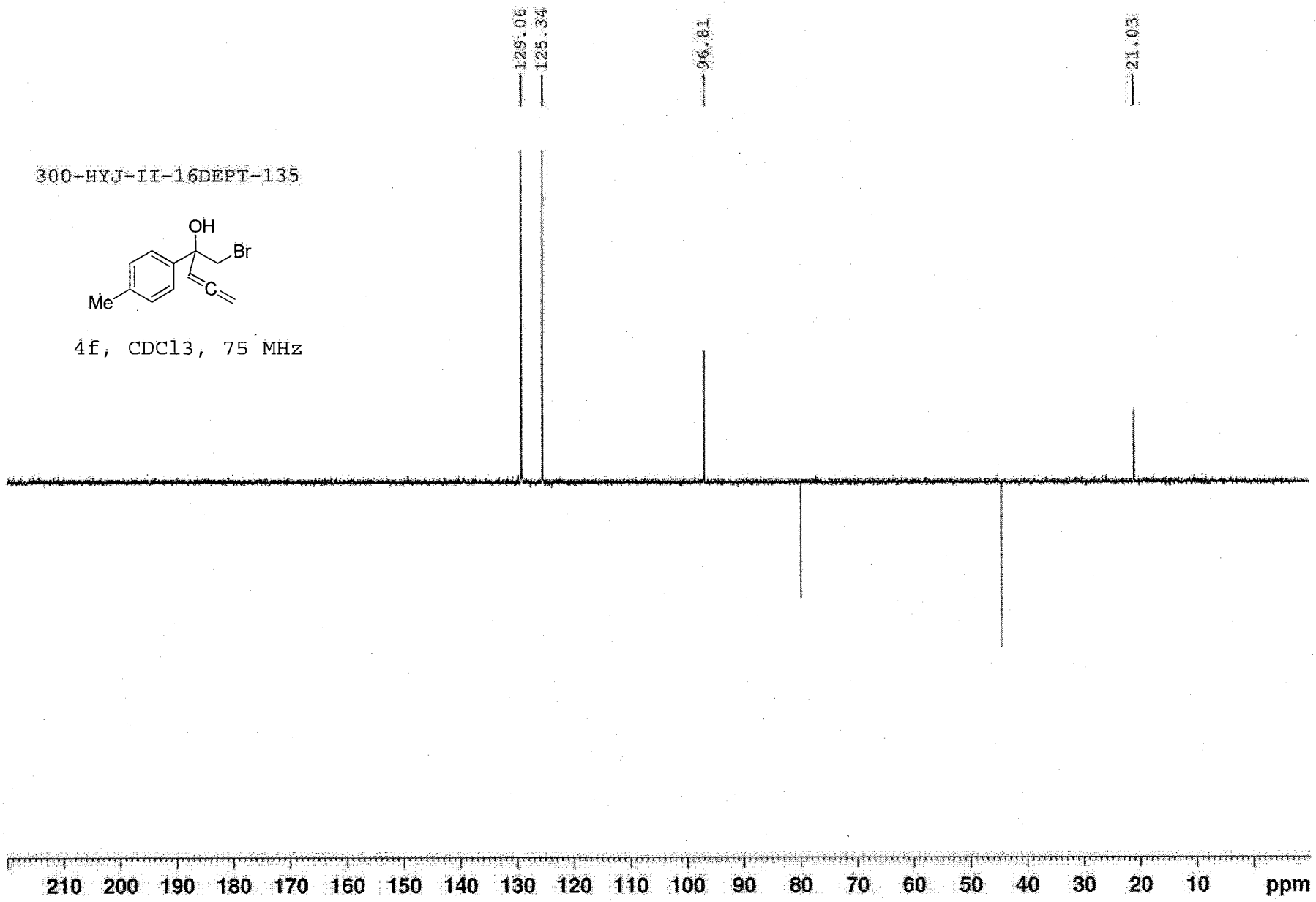
300-HYJ-II-15(dept135)

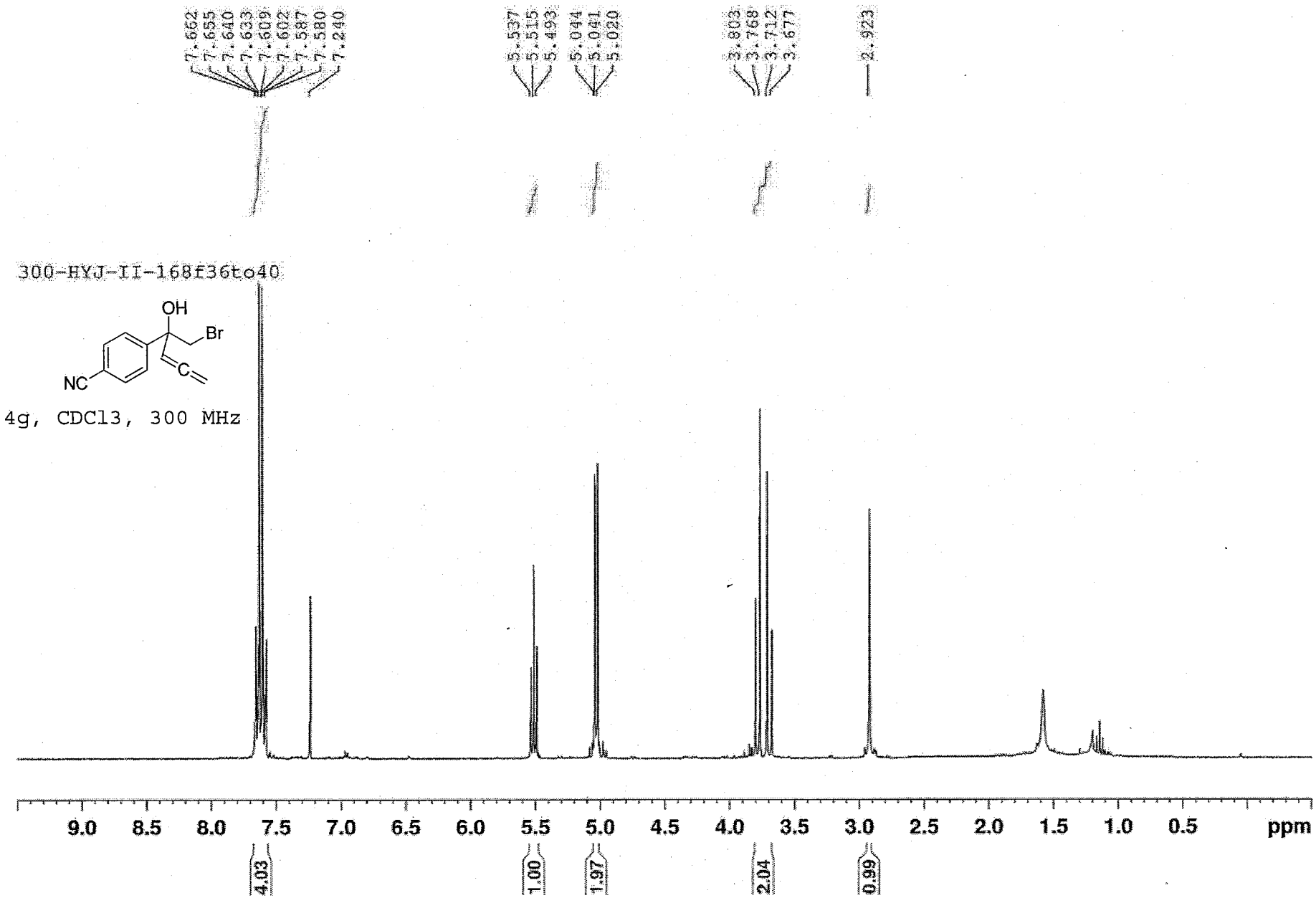
4e, CDCl3, 75 MHz

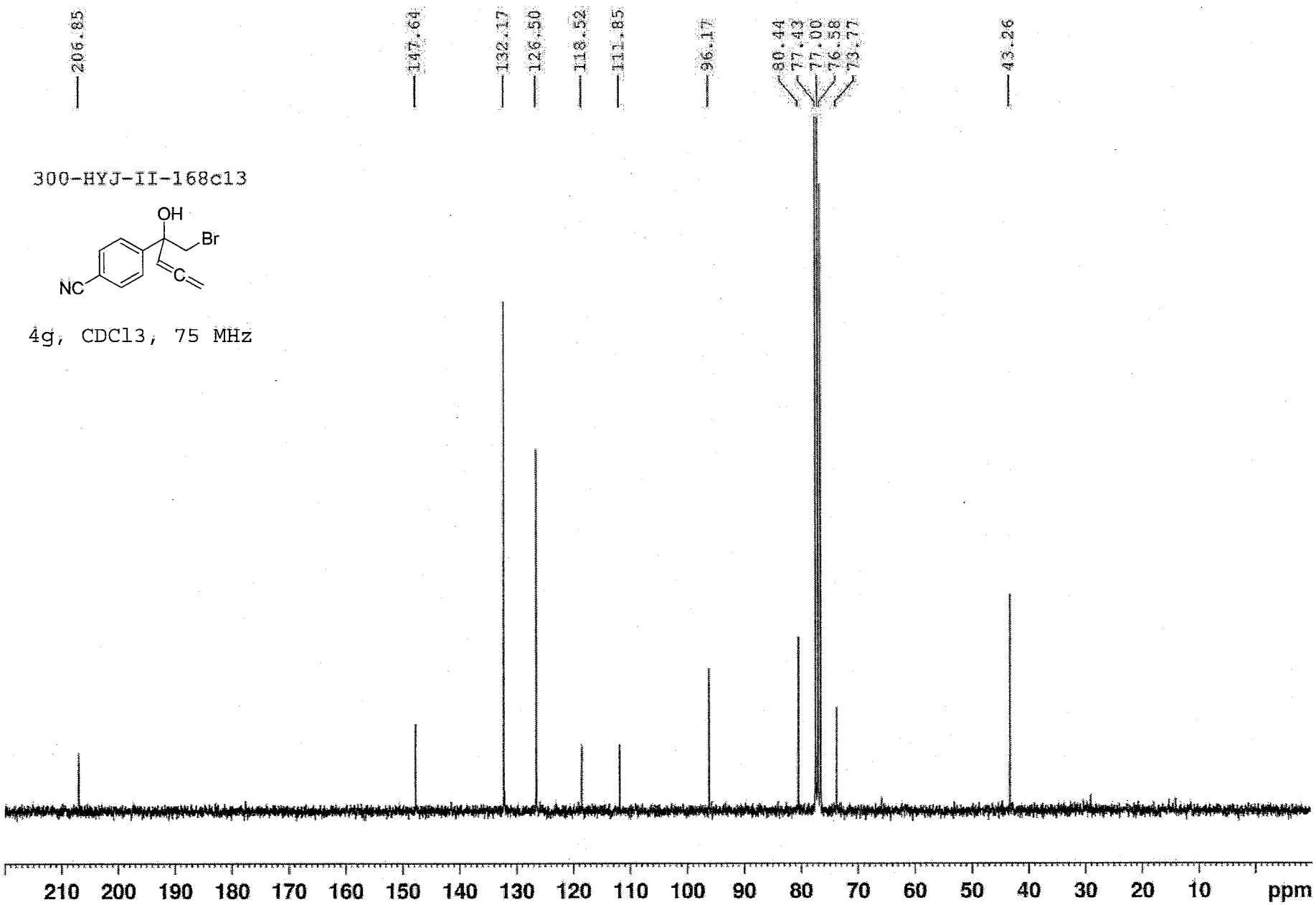


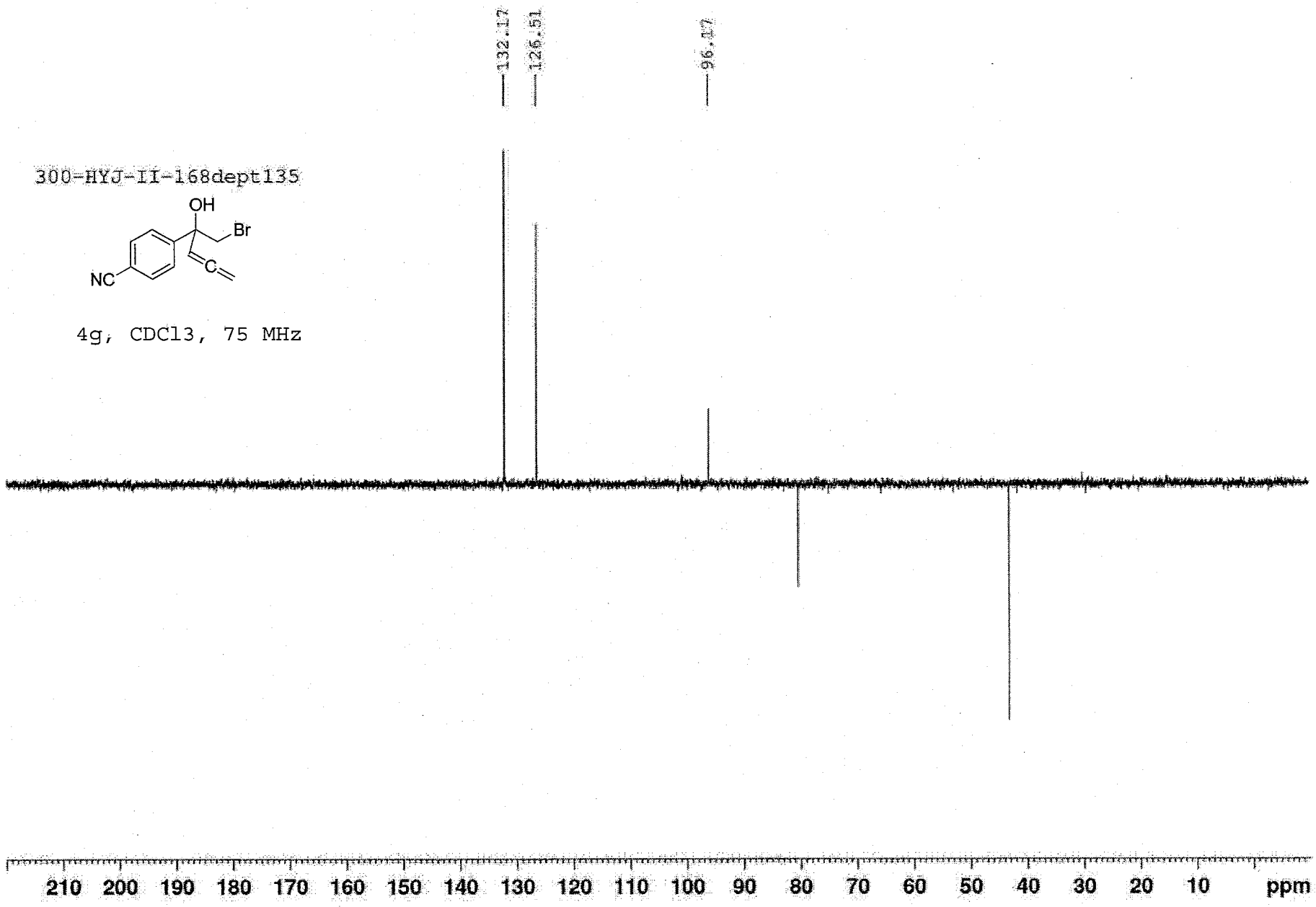

7.388
7.366
7.360
7.240
7.182
7.155
5.561
5.539
5.516
5.034
5.025
5.012
5.003
3.807
3.772
3.737
3.702
2.845
2.338
1.605
300-HYJ-II-16f35to39
OH
Br
Me
C
4f, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.00
2.05
1.01
1.99
2.10
0.99
3.02

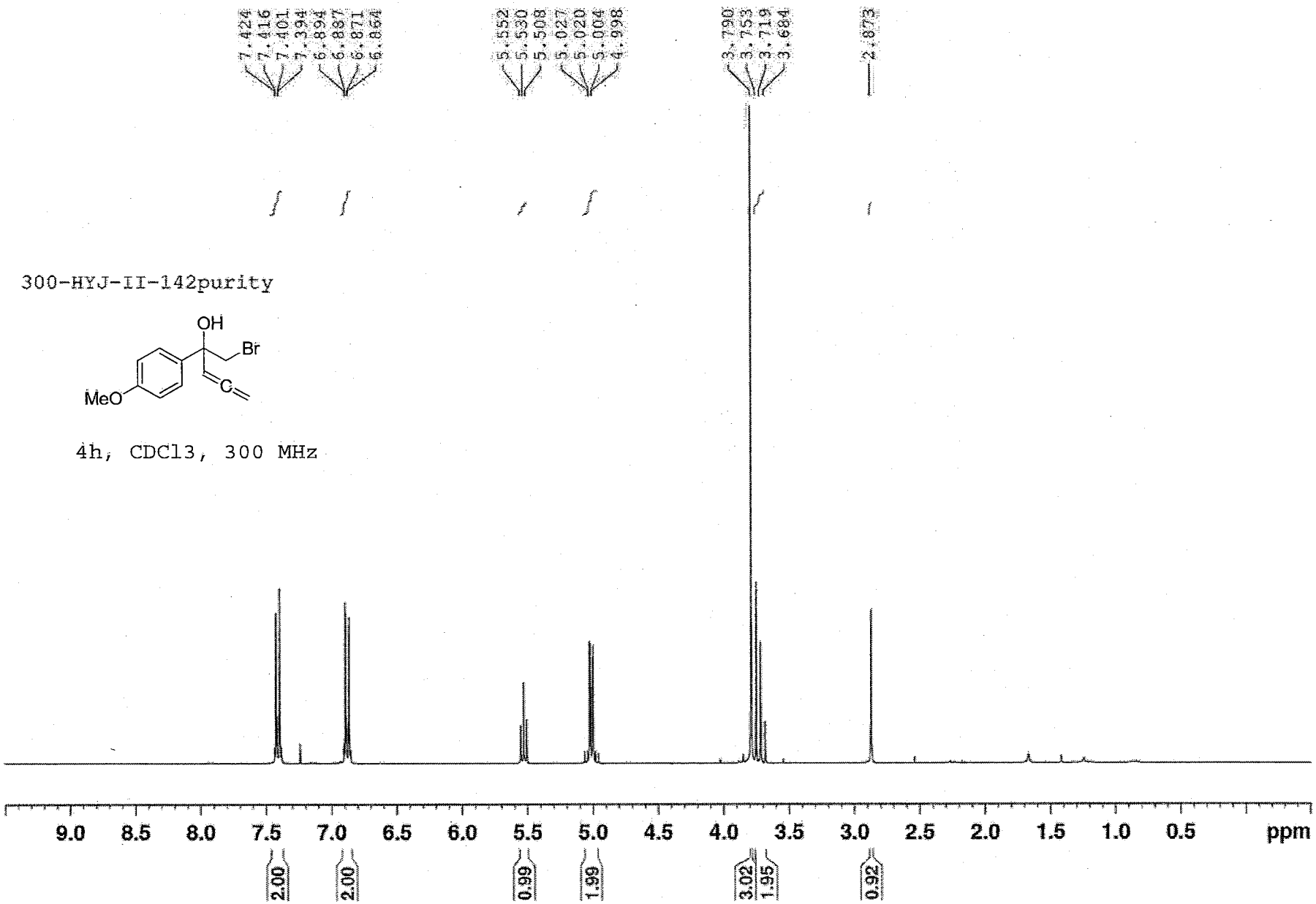

206.58
139.32
137.65
129.06
128.95
125.36
96.82
79.97
79.91
77.43
77.01
76.58
73.66
44.50
21.04
300-HYJ-II-16(c13)
OH
Br
Me
C
4f, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

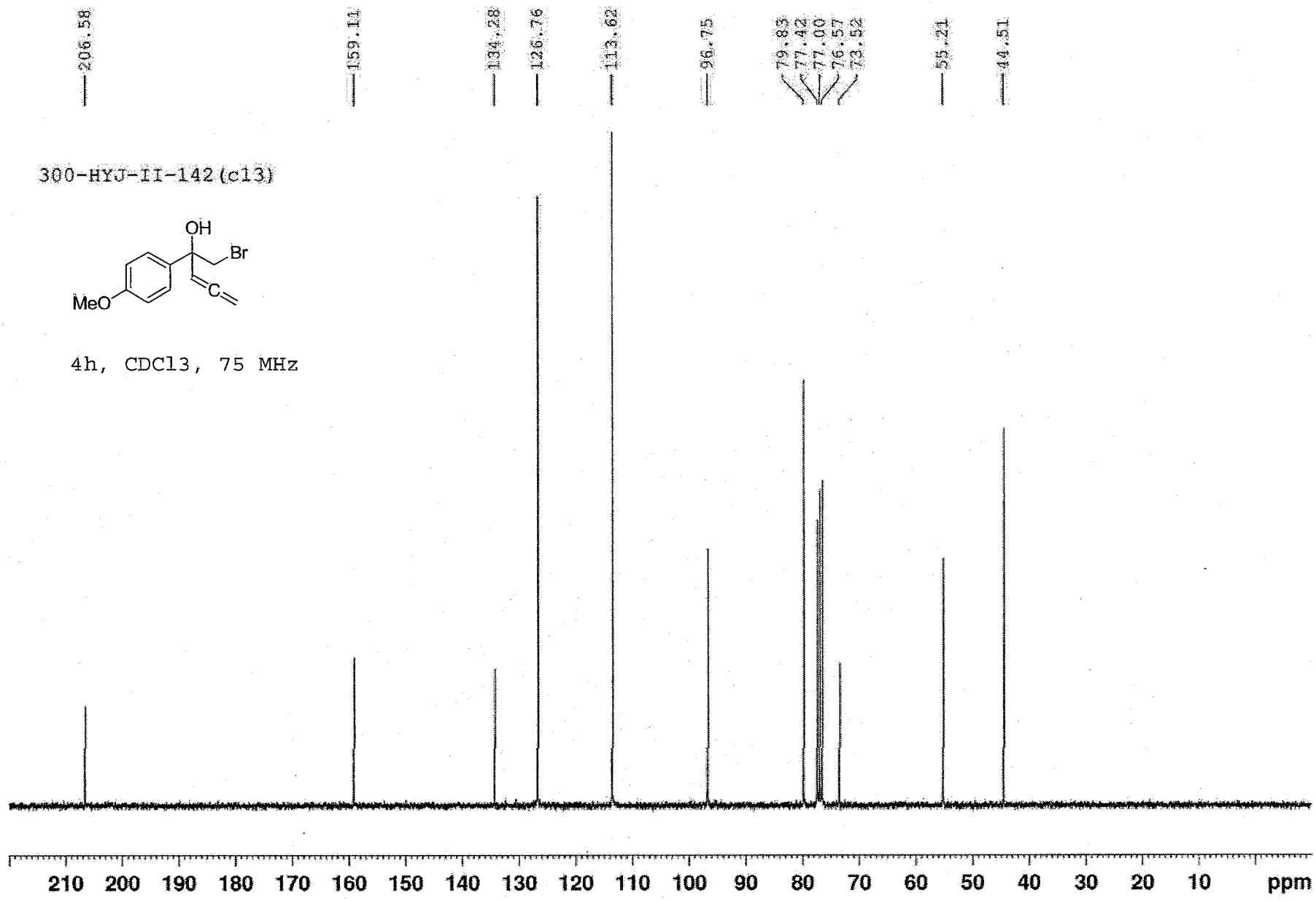

300-HYJ-II-16DEPT-135
OH
Br
C
Me
4f, CDCl3, 75 MHz
129.06
125.34
96.81
21.03
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

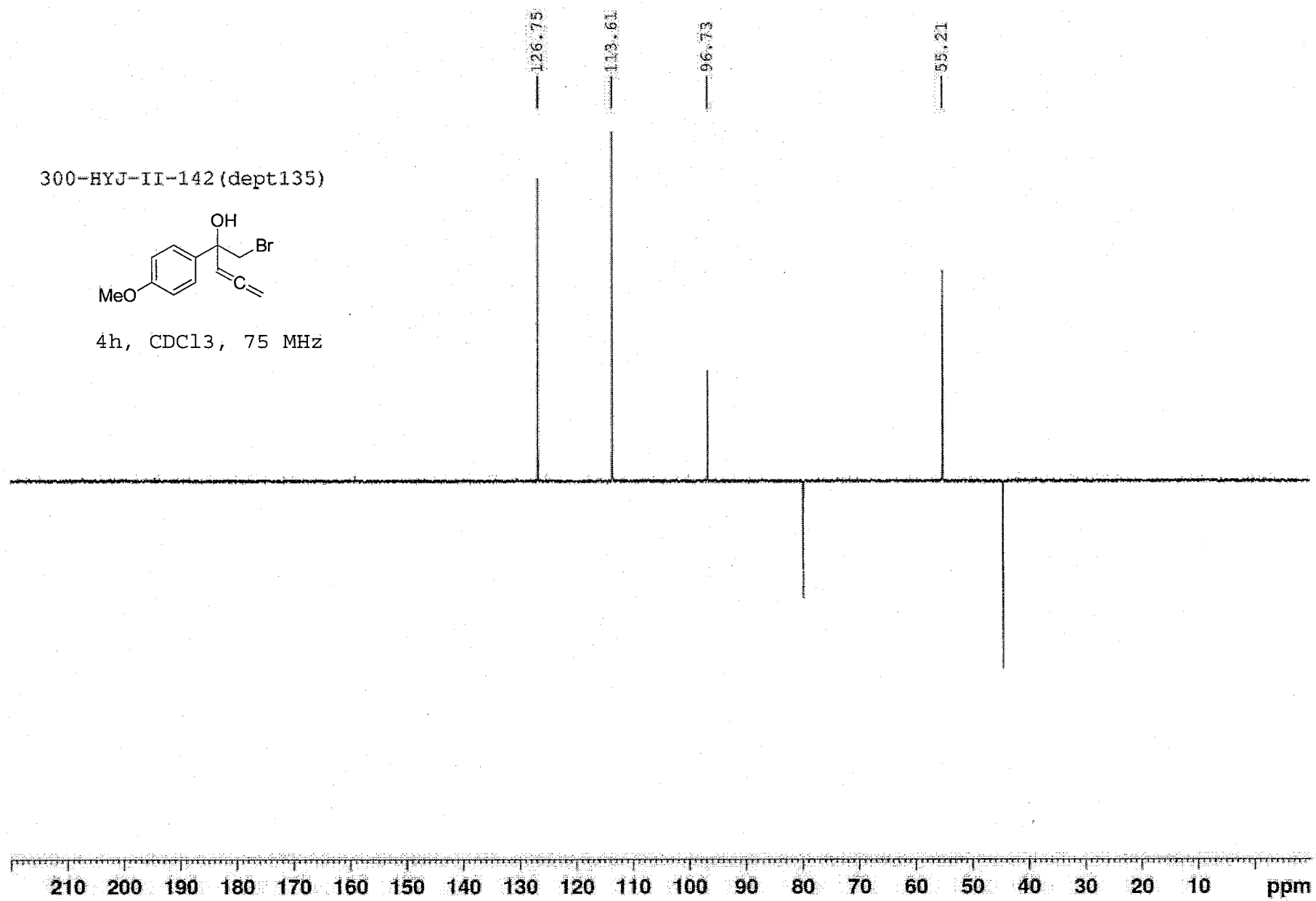

7.662
7.655
7.640
7.633
7.609
7.602
7.587
7.580
7.240
5.537
5.515
5.493
5.044
5.041
5.020
3.803
3.768
3.712
3.677
2.923
300-HYJ-II-168f36to40
OH
Br
NC
C
4g, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.03
1.00
1.97
2.04
0.99

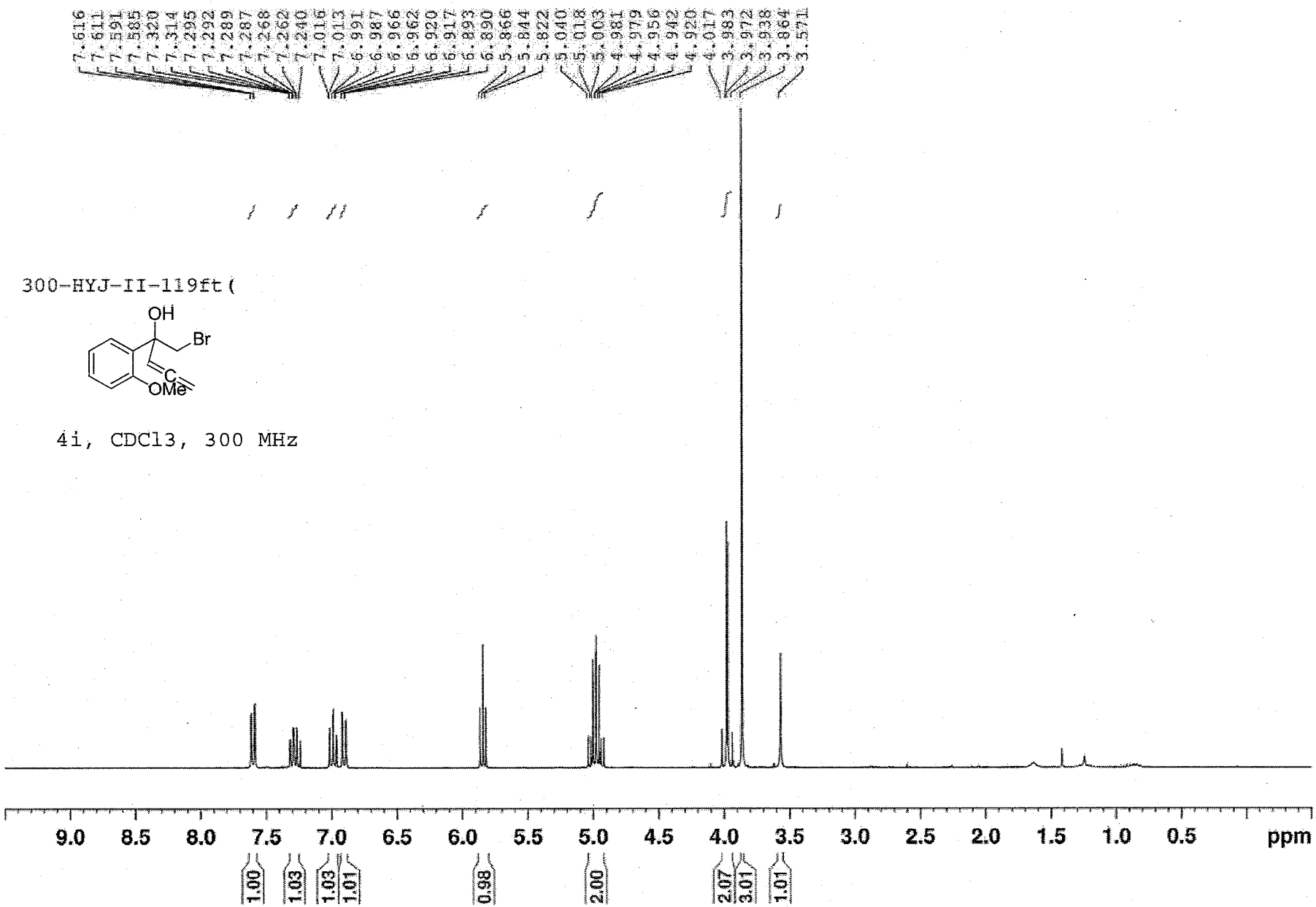

206.85
147.64
132.17
126.50
118.52
111.85
96.17
80.44
77.43
77.00
76.58
73.77
43.26
300-HYJ-II-168c13
OH
Br
NC
C
4g, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

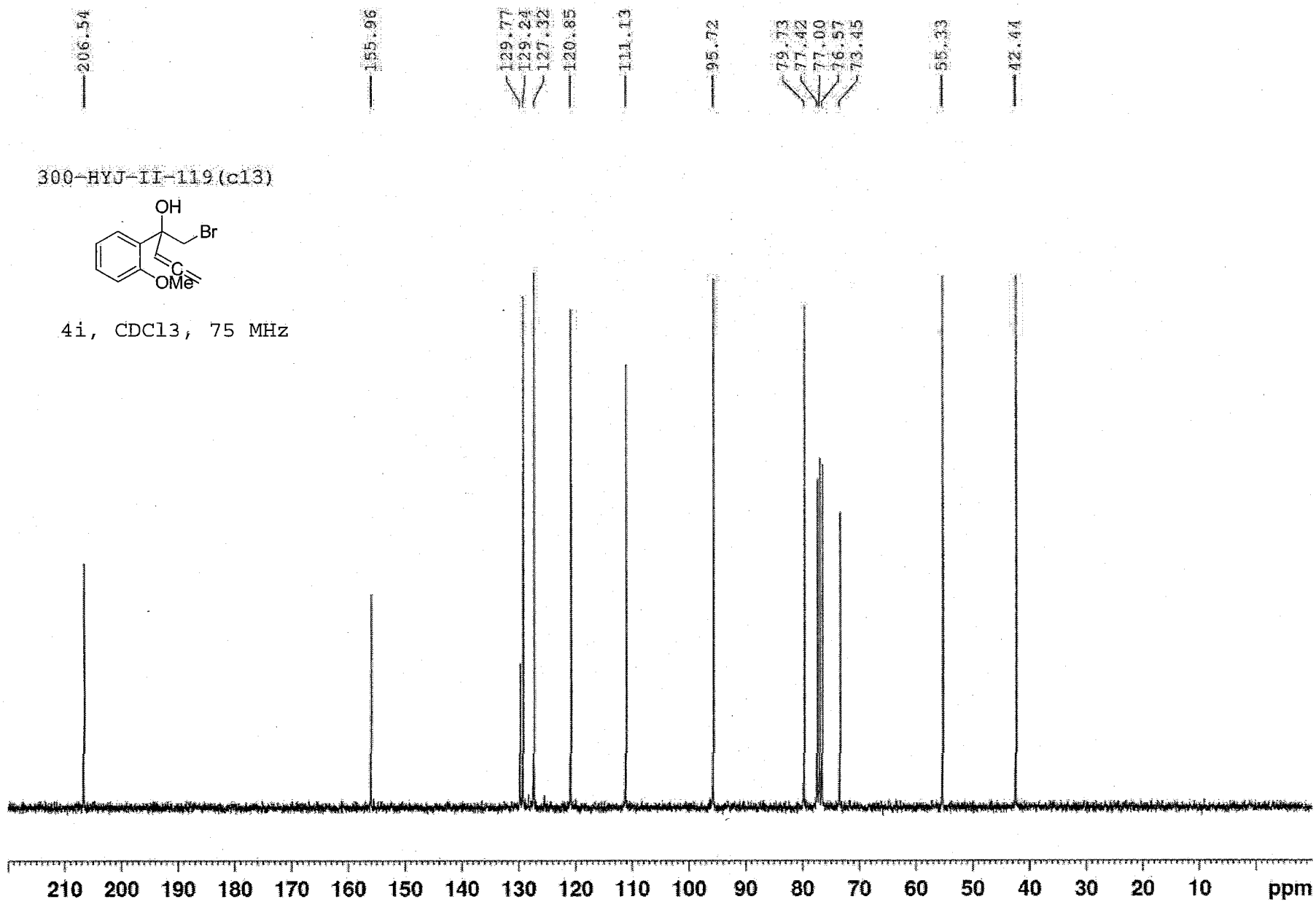

132.17
126.51
96.17
300-HYJ-II-168dept135
OH
Br
NC
C
4g, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.424
7.416
7.401
7.394
6.894
6.887
6.871
6.864
5.552
5.530
5.508
5.027
5.020
5.004
4.998
3.790
3.753
3.719
3.684
2.873
300-HYJ-II-142purity
OH
Br
MeO
C
4h, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.00
2.00
0.99
1.99
3.02
1.95
0.92


206.58
159.11
134.28
126.76
113.62
96.75
79.83
77.42
77.00
76.57
73.52
55.21
44.51
300-HYJ-II-142(c13)
OH
Br
MeO
C
4h, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

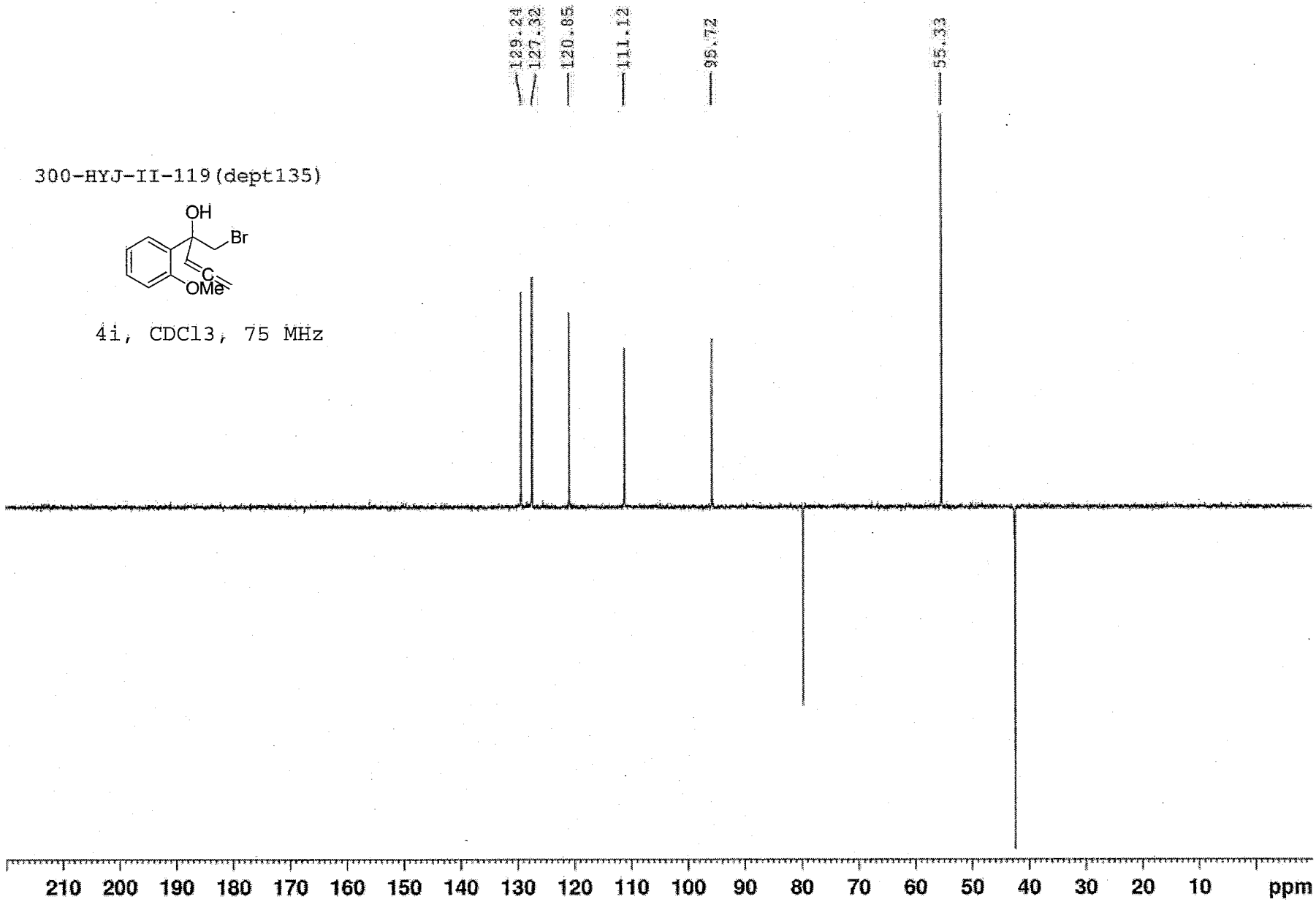

300-HYJ-II-142(dept135)
OH
Br
MeO
C
4h, CDCl3, 75 MHz
126.75
113.61
96.73
55.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

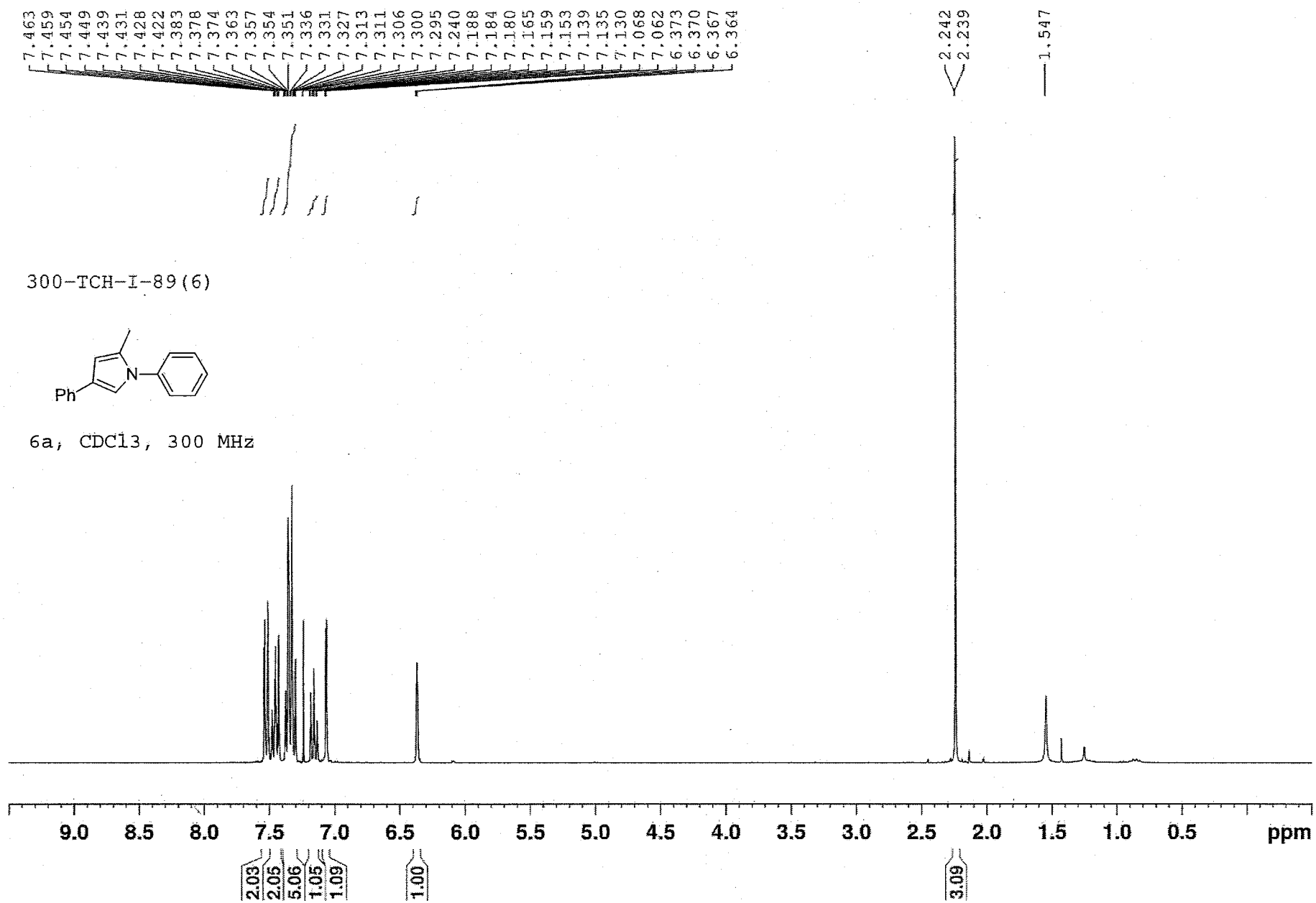

7.616
7.611
7.591
7.585
7.320
7.314
7.295
7.292
7.289
7.287
7.268
7.262
7.240
7.016
7.013
6.991
6.987
6.966
6.962
6.920
6.917
6.893
6.890
5.866
5.844
5.822
5.040
5.018
5.003
4.981
4.979
4.956
4.942
4.920
4.017
3.983
3.972
3.938
3.864
3.571
300-HYJ-II-119ft(
OH
Br
C
OMe
4i, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.03
1.03
1.01
0.98
2.00
2.07
3.01
1.01


206.54
155.96
129.77
129.24
127.32
120.85
111.13
95.72
79.73
77.42
77.00
76.57
73.45
55.33
42.44
300-HYJ-II-119(c13)
OH
Br
C
OMe
4i, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

300-HYJ-II-119(dept135)

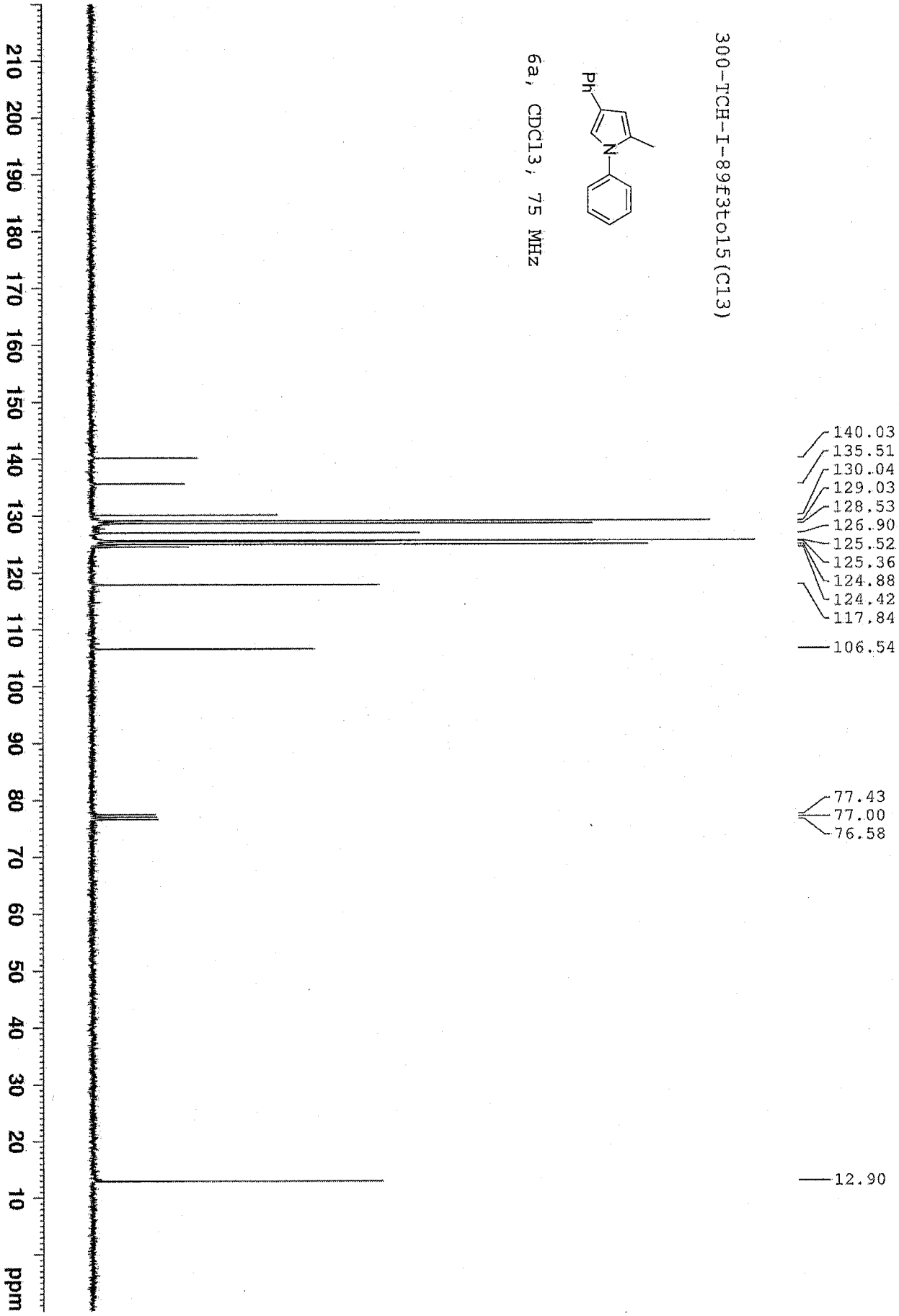
4i, CDCl3, 75 MHz




7.463
7.459
7.454
7.449
7.439
7.431
7.428
7.422
7.383
7.378
7.374
7.363
7.357
7.354
7.351
7.336
7.331
7.327
7.313
7.311
7.306
7.300
7.295
7.240
7.188
7.184
7.180
7.165
7.159
7.153
7.139
7.135
7.130
7.068
7.062
6.373
6.370
6.367
6.364
2.242
2.239
1.547
300-TCH-I-89(6)
Ph
N
6a, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.03
2.05
5.06
1.05
1.09
1.00
3.09

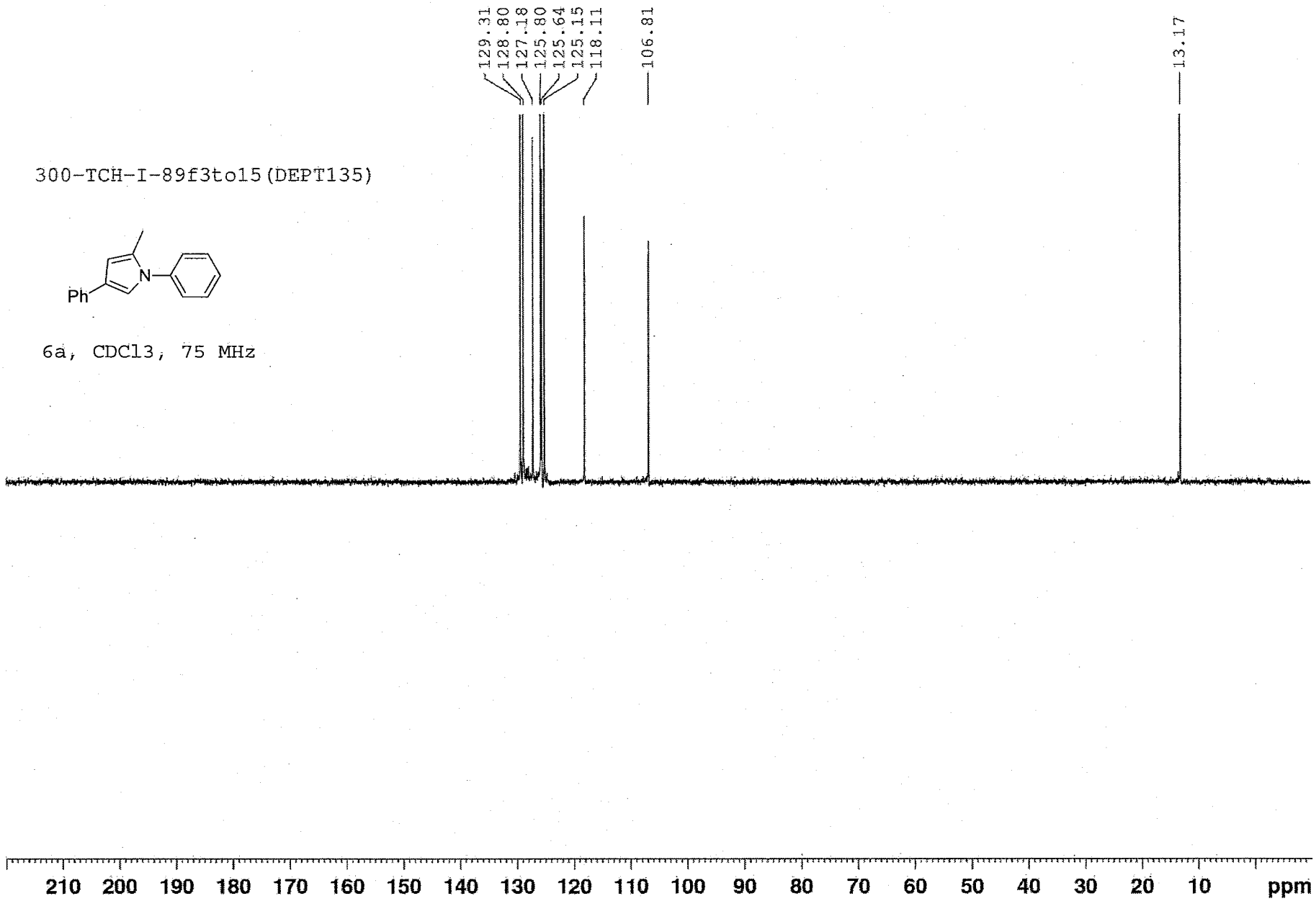
300-TCH-I-89f3to15(C13)

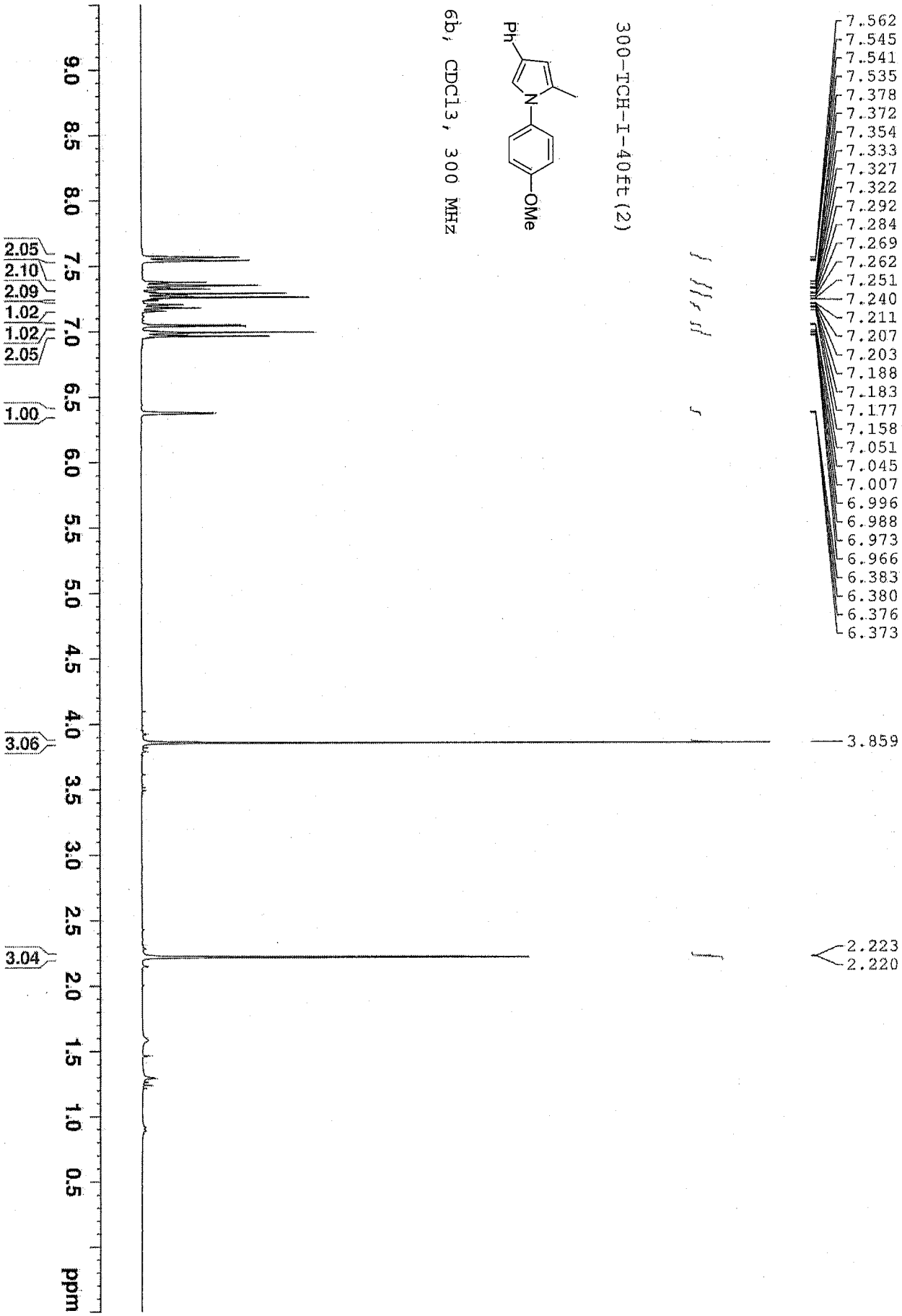
6a, CDCl3, 75 MHz

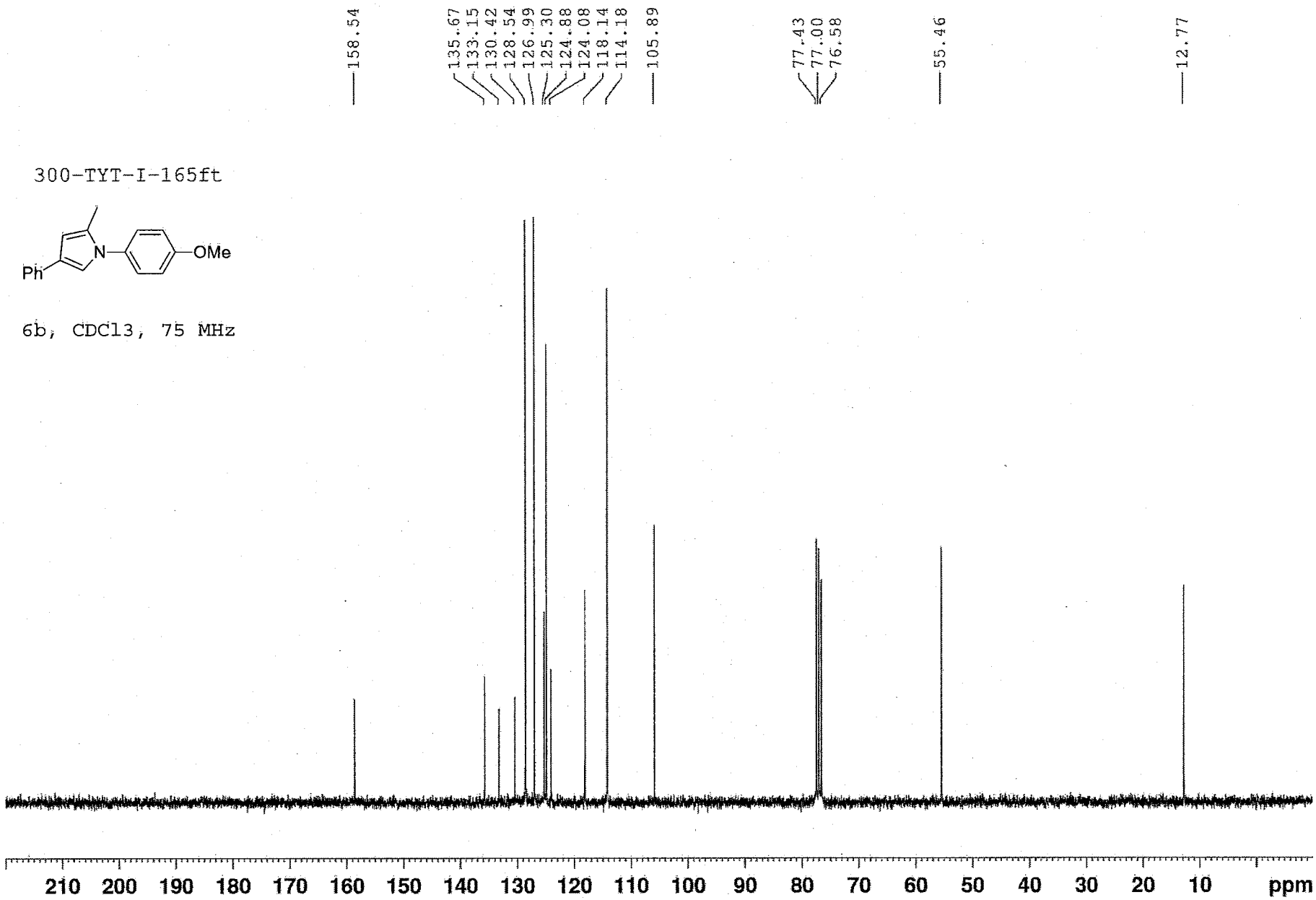


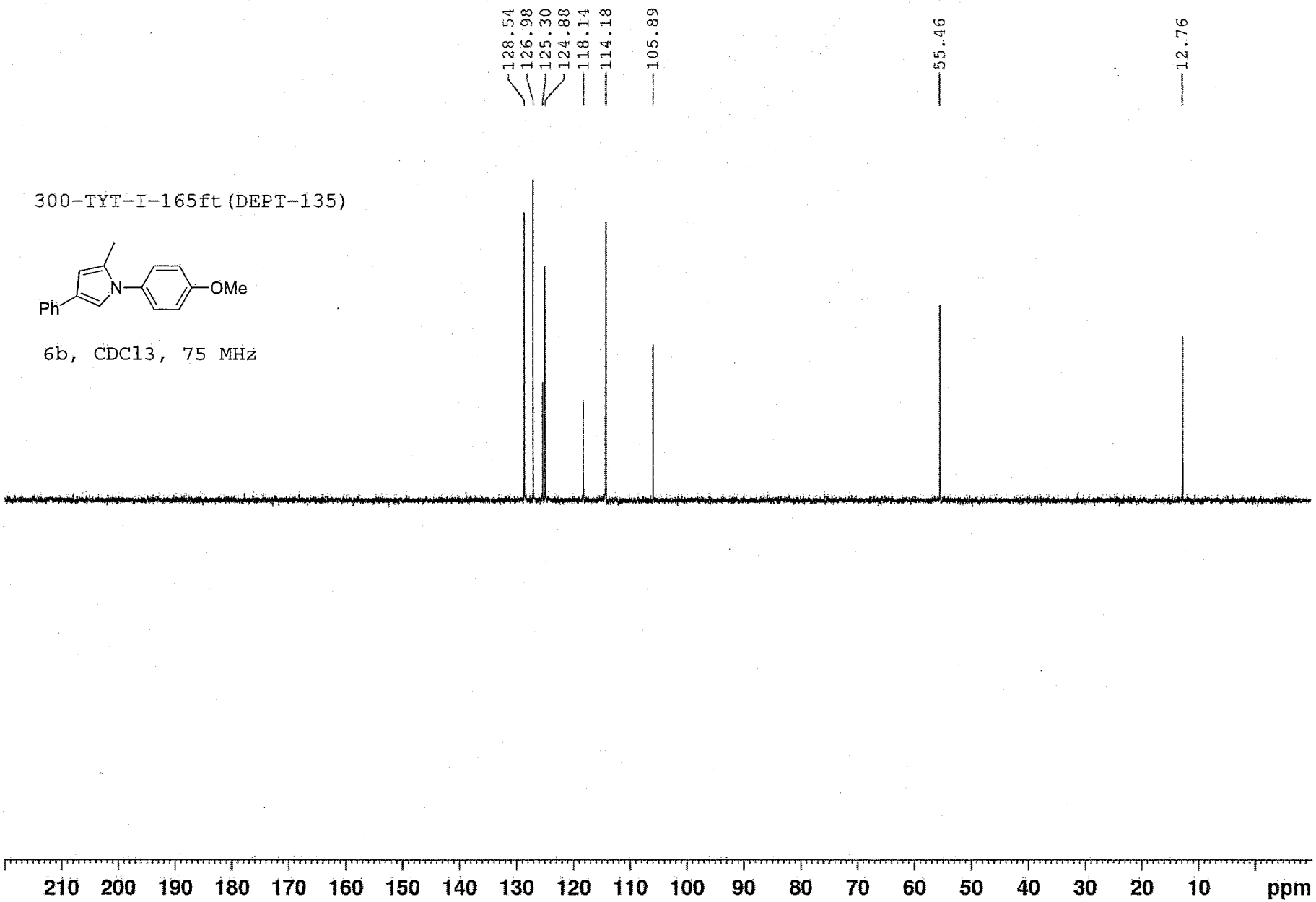
300-TCH-I-89f3to15(DEPT135)

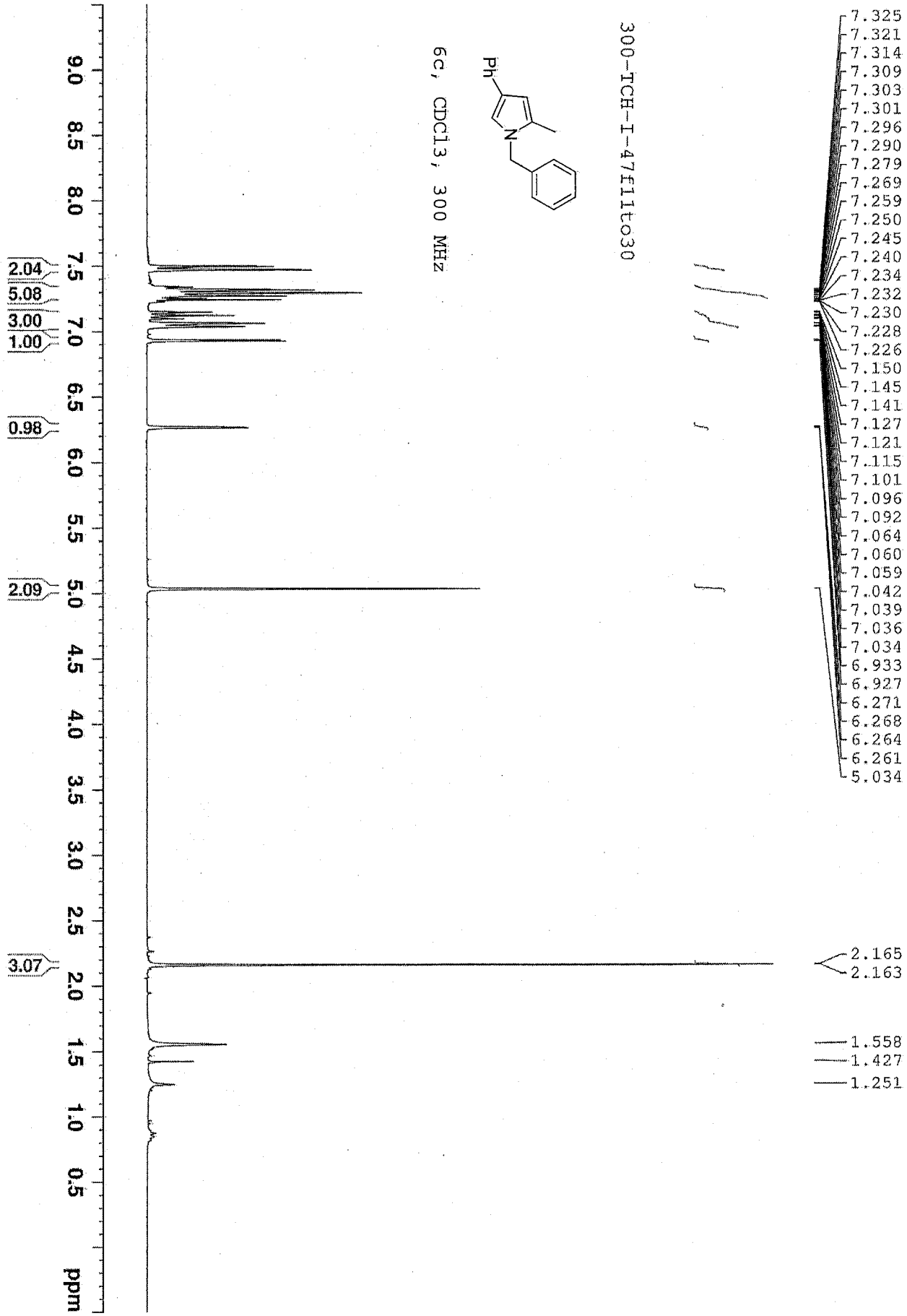
6a, CDCl3, 75 MHz

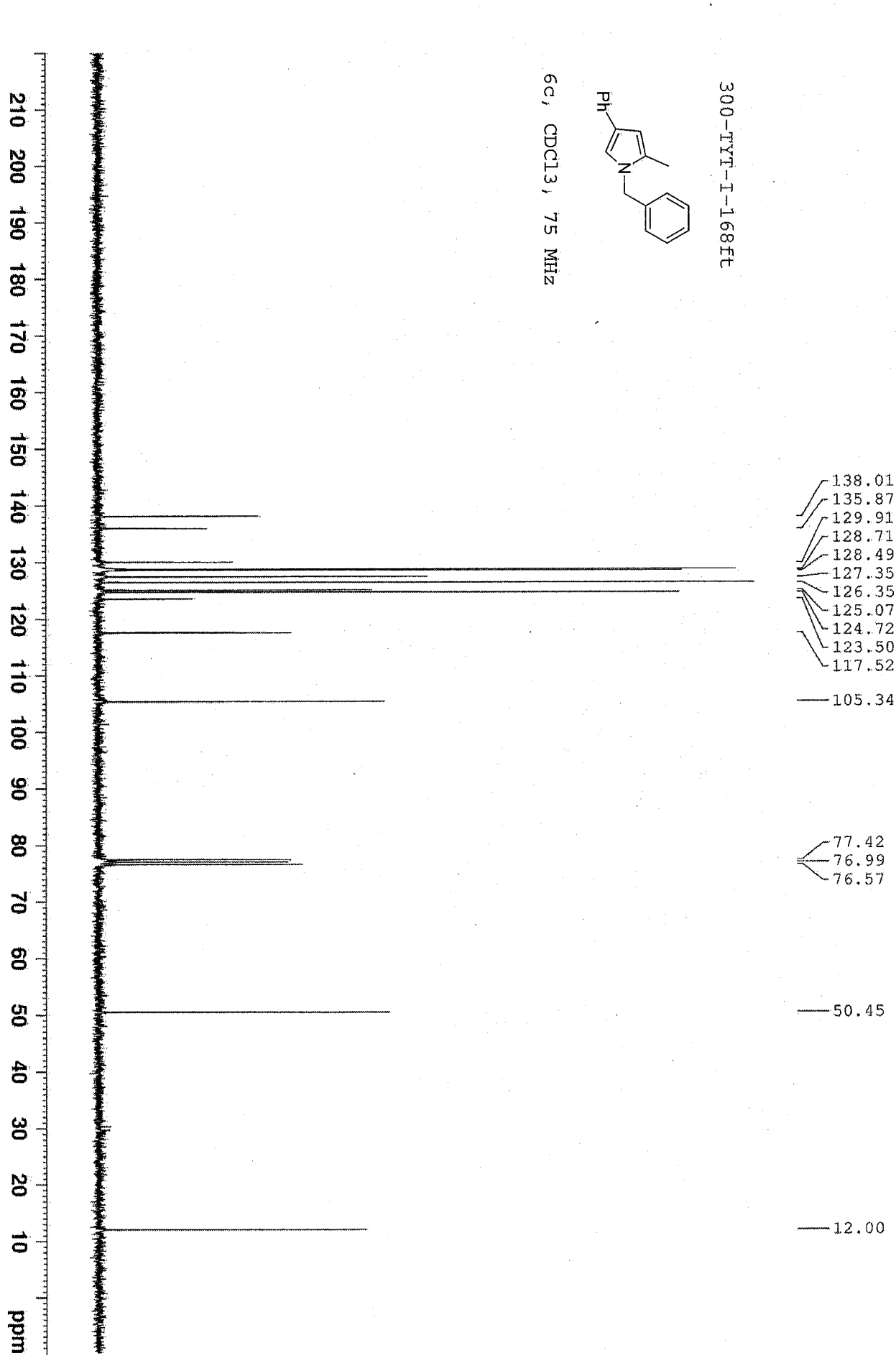


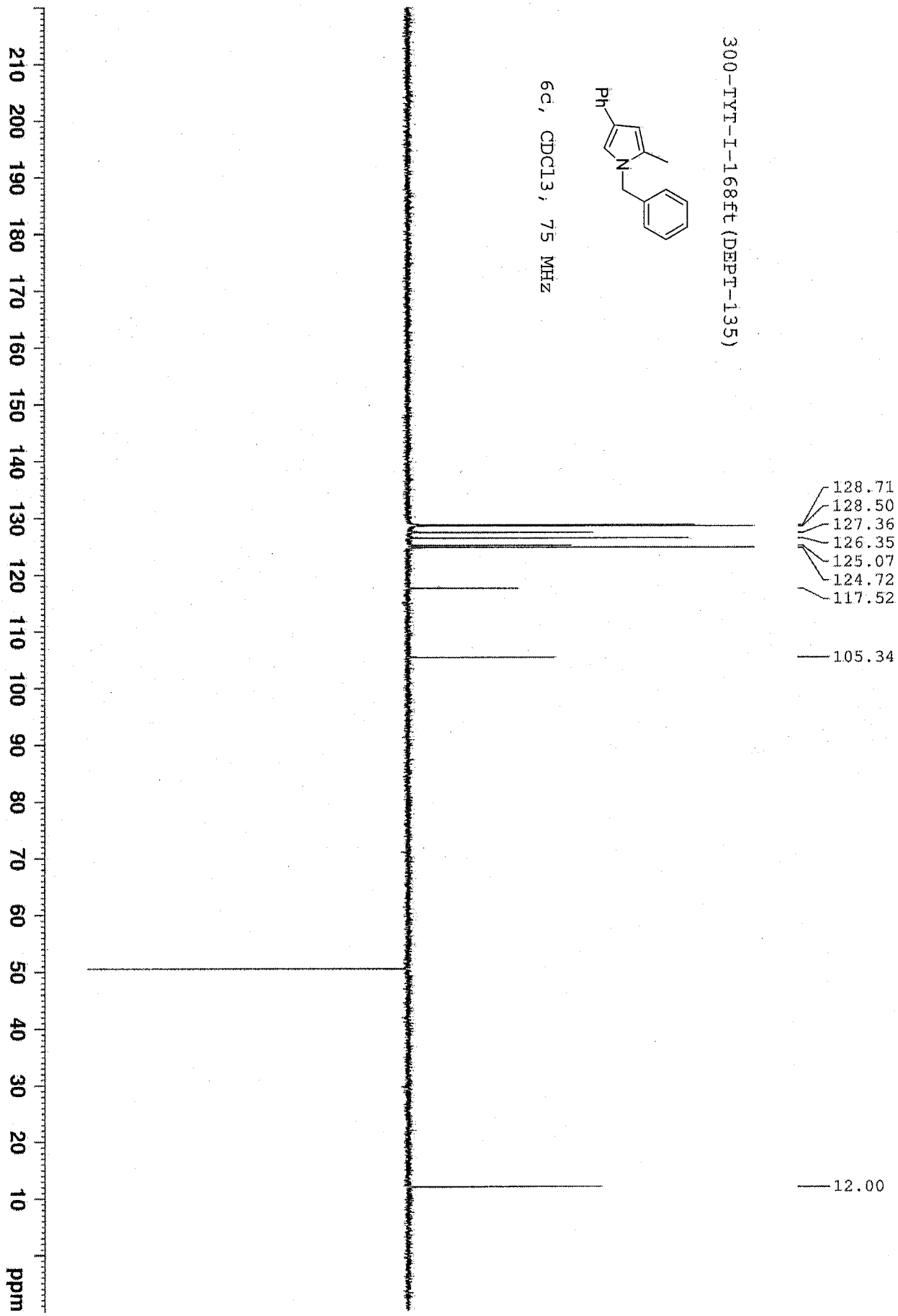

300-TCH-I-40ft(2)
6b, CDCl3, 300 MHz
Ph
N
OMe
7.562
7.545
7.541
7.535
7.378
7.372
7.354
7.333
7.327
7.322
7.292
7.284
7.269
7.262
7.251
7.240
7.211
7.207
7.203
7.188
7.183
7.177
7.158
7.051
7.045
7.007
6.996
6.988
6.973
6.966
6.383
6.380
6.376
6.373
3.859
2.223
2.220
2.05
2.10
2.09
1.02
1.02
2.05
1.00
3.06
3.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

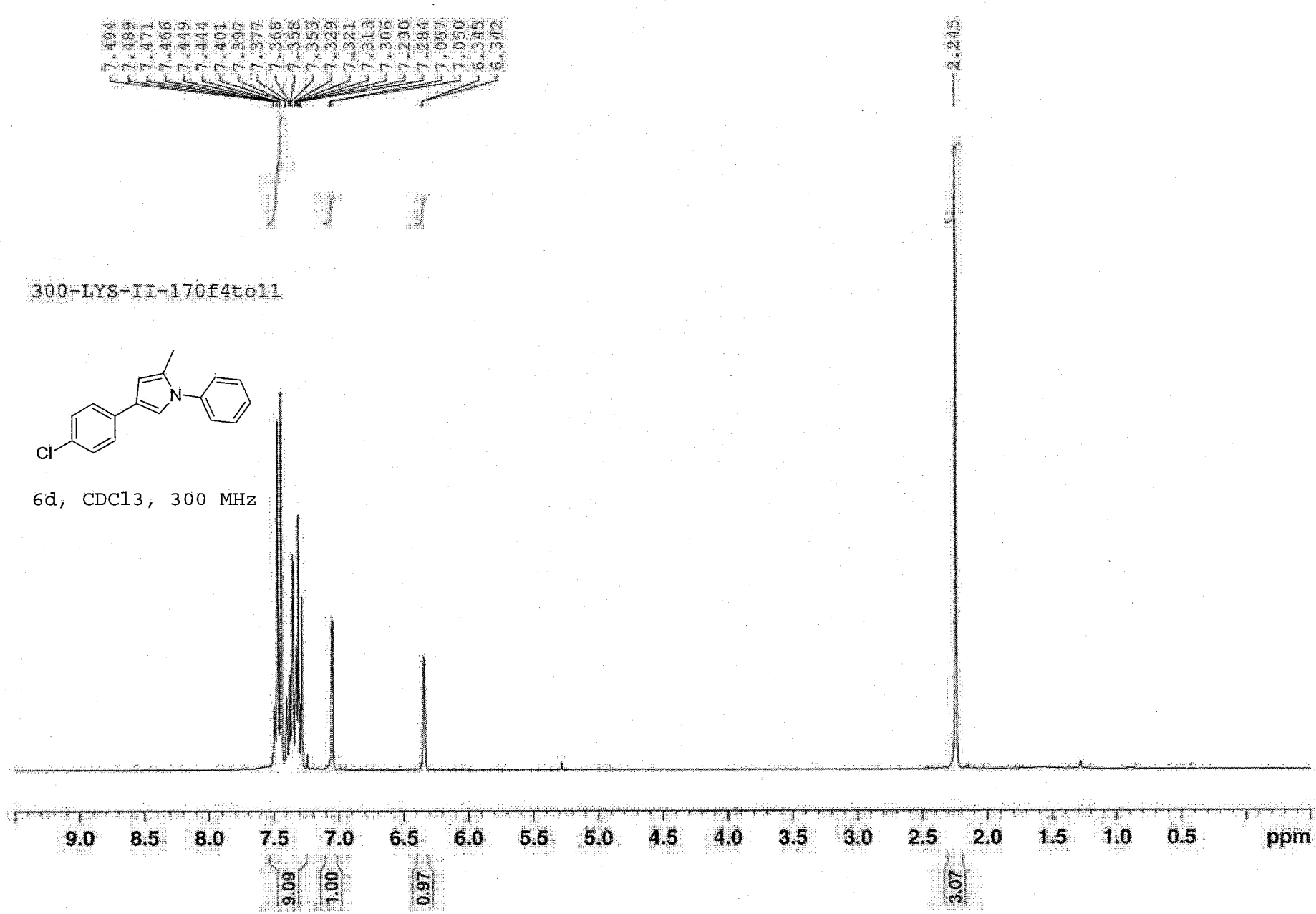

158.54
135.67
133.15
130.42
128.54
126.99
125.30
124.88
124.08
118.14
114.18
105.89
77.43
77.00
76.58
55.46
12.77
300-TYT-I-165ft
N
OMe
Ph
6b, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

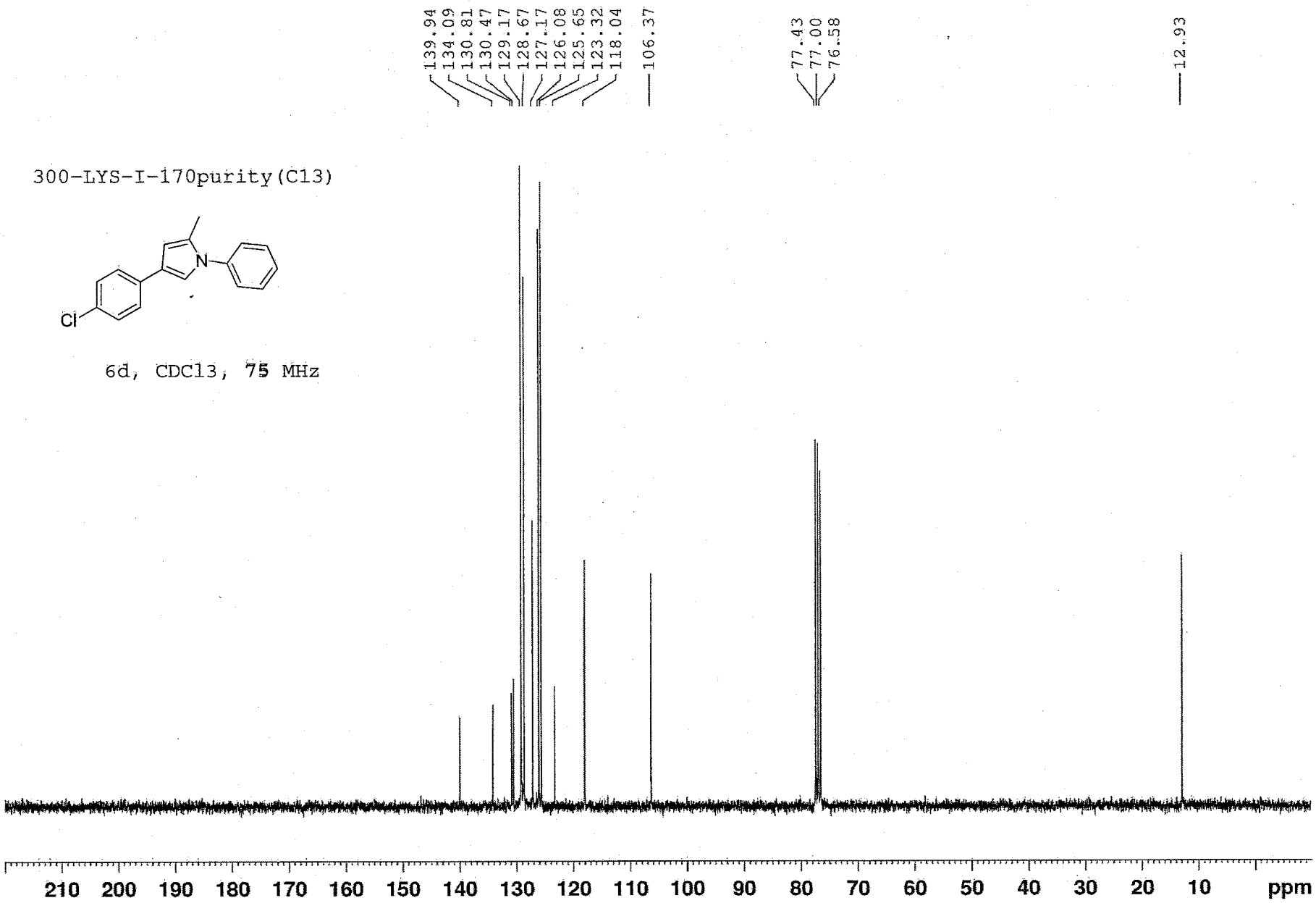

128.54
126.98
125.30
124.88
118.14
114.18
105.89
55.46
12.76
300-TYT-I-165ft(DEPT-135)
N
OMe
Ph
6b, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

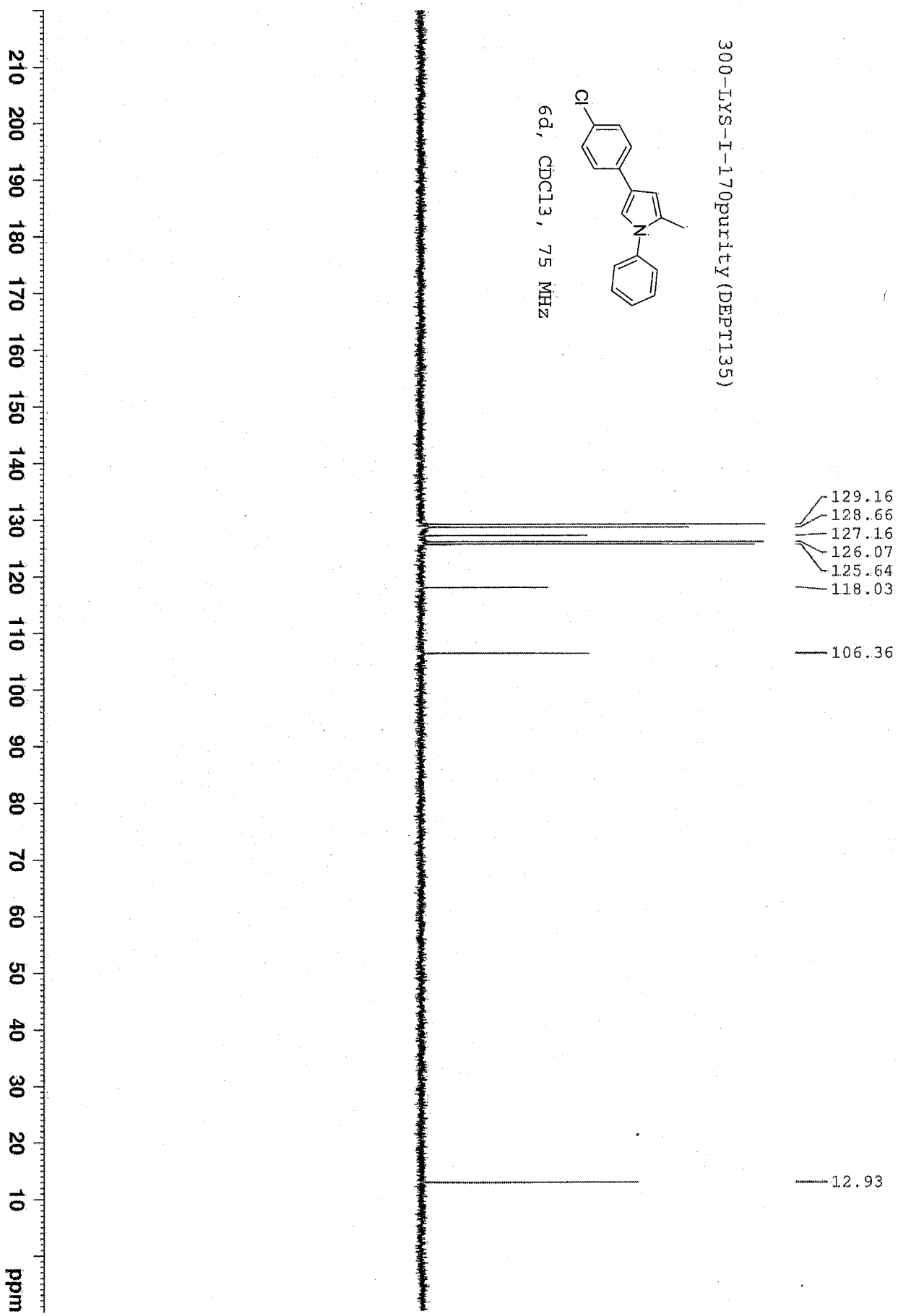

300-TCH-I-47f11to30
6c, CDCl3, 300 MHz
Ph
N
7.325
7.321
7.314
7.309
7.303
7.301
7.296
7.290
7.279
7.269
7.259
7.250
7.245
7.240
7.234
7.232
7.230
7.228
7.226
7.150
7.145
7.141
7.127
7.121
7.115
7.101
7.096
7.092
7.064
7.060
7.059
7.042
7.039
7.036
7.034
6.933
6.927
6.271
6.268
6.264
6.261
5.034
2.165
2.163
1.558
1.427
1.251
2.04
5.08
3.00
1.00
0.98
2.09
3.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

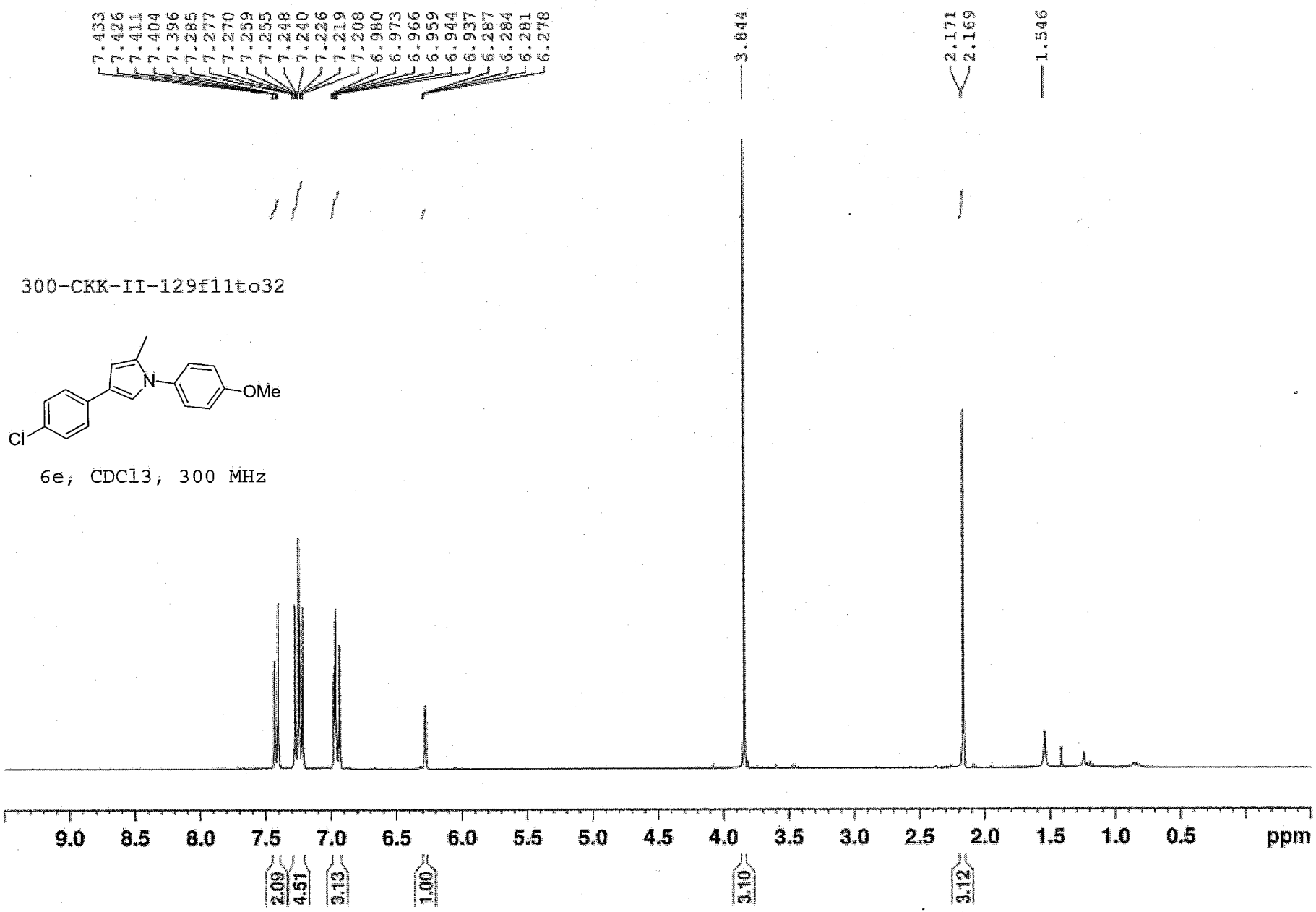

300-TYT-I-168ft
6c, CDCl3, 75 MHz
Ph
N
138.01
135.87
129.91
128.71
128.49
127.35
126.35
125.07
124.72
123.50
117.52
105.34
77.42
76.99
76.57
50.45
12.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

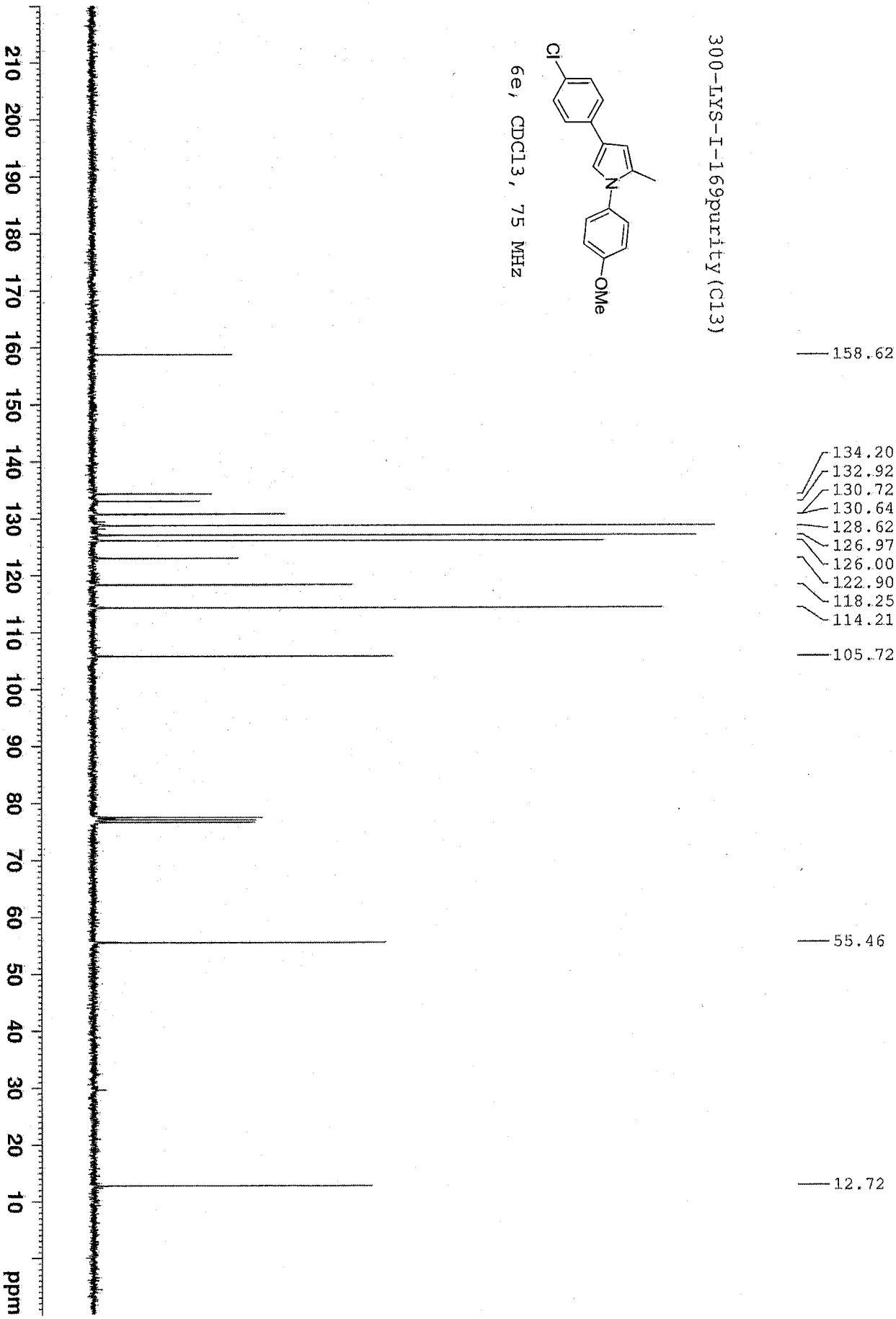

300-TYT-I-168ft(DEPT-135)
6c, CDCl3, 75 MHz
Ph
N
128.71
128.50
127.36
126.35
125.07
124.72
117.52
105.34
12.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.494
7.489
7.471
7.466
7.449
7.444
7.401
7.397
7.377
7.368
7.358
7.353
7.329
7.321
7.313
7.306
7.290
7.234
7.057
7.050
6.345
6.342
2.245
300-LYS-II-170f4to11
N
Cl
6d, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
9.09
1.00
0.97
3.07


139.94
134.09
130.81
130.47
129.17
128.67
127.17
126.08
125.65
123.32
118.04
106.37
77.43
77.00
76.58
12.93
300-LYS-I-170purity(C13)
N
Cl
6d, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

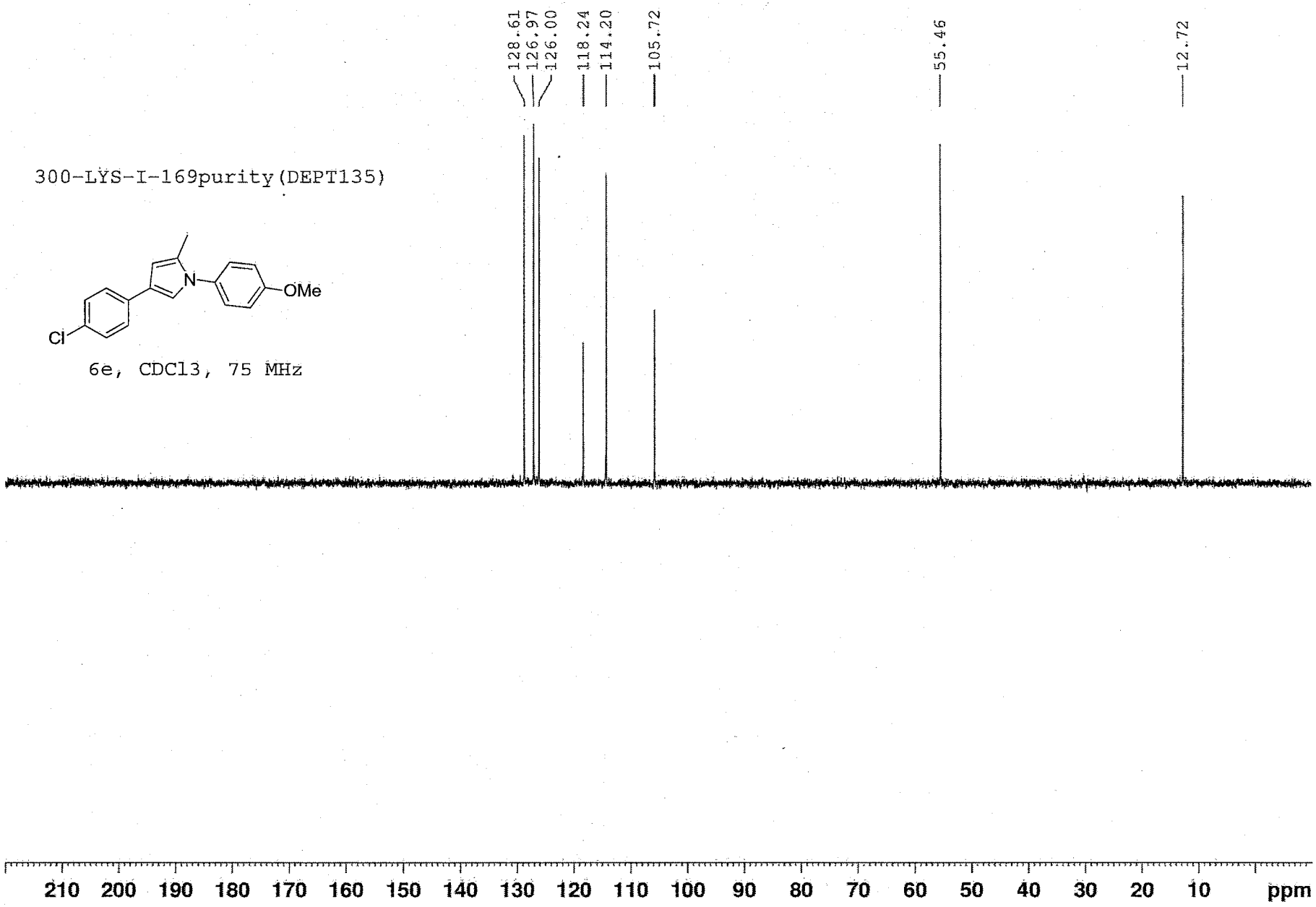

300-LYS-I-170purity(DEPT135)
6d, CDCl3, 75 MHz
Cl
N
129.16
128.66
127.16
126.07
125.64
118.03
106.36
12.93
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

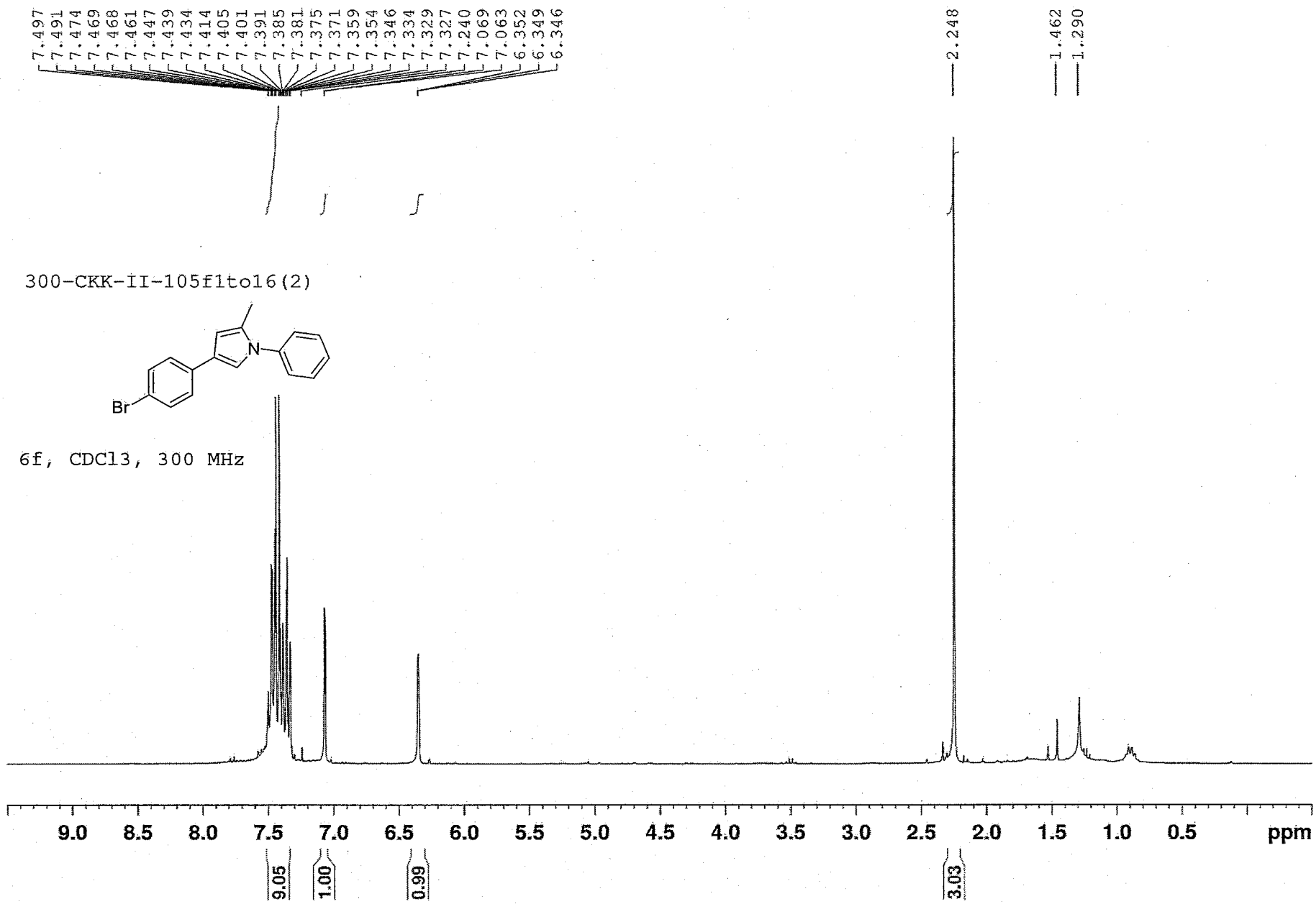

7.433
7.426
7.411
7.404
7.396
7.285
7.277
7.270
7.259
7.255
7.248
7.240
7.226
7.219
7.208
6.980
6.973
6.966
6.959
6.944
6.937
6.287
6.284
6.281
6.278
3.844
2.171
2.169
1.546
300-CKK-II-129f11to32
N
OMe
Cl
6e, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.09
4.51
3.13
1.00
3.10
3.12

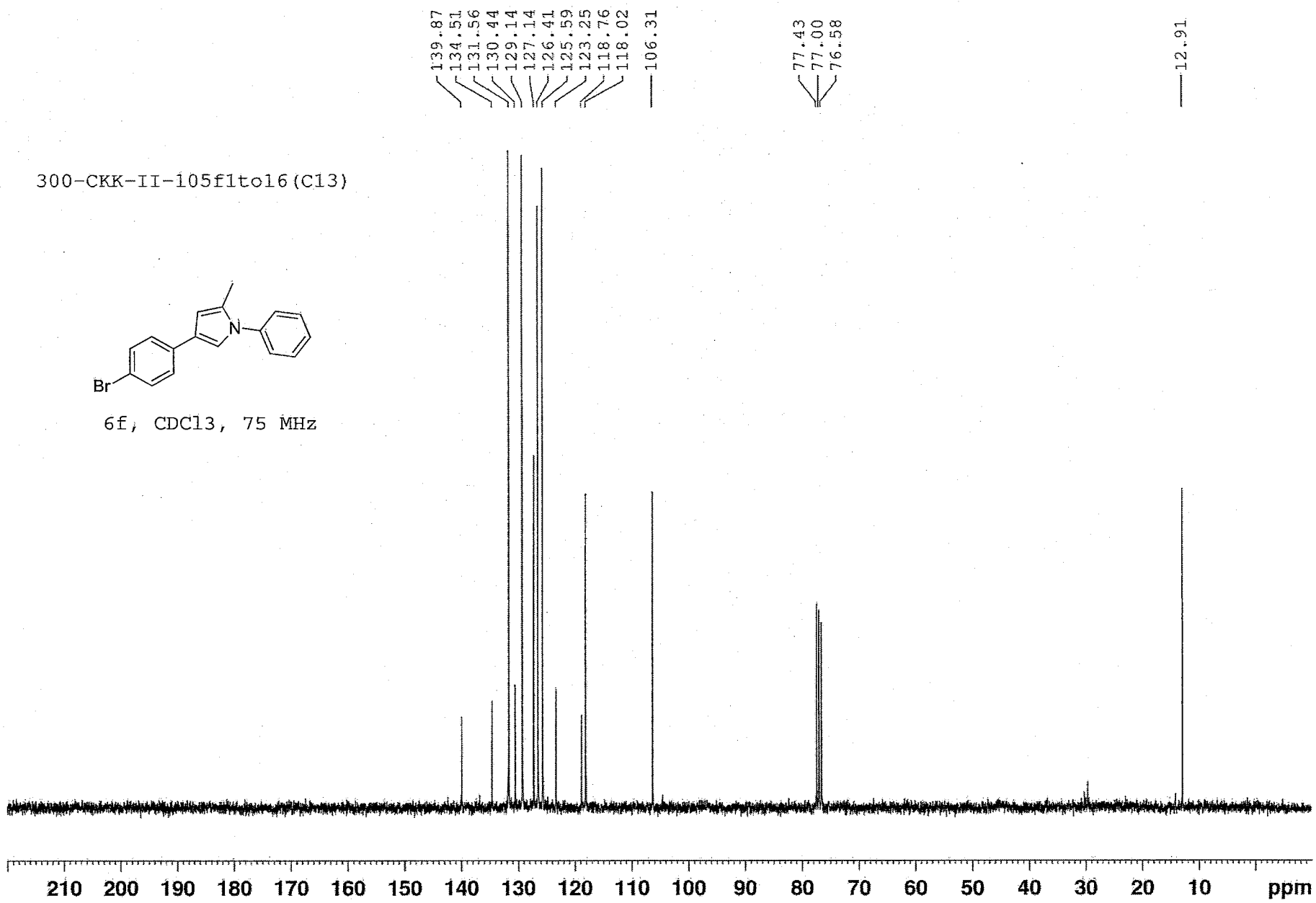

300-LYS-I-169purity(C13)
6e, CDCl3, 75 MHz
Cl
N
OMe
158.62
134.20
132.92
130.72
130.64
128.62
126.97
126.00
122.90
118.25
114.21
105.72
55.46
12.72
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

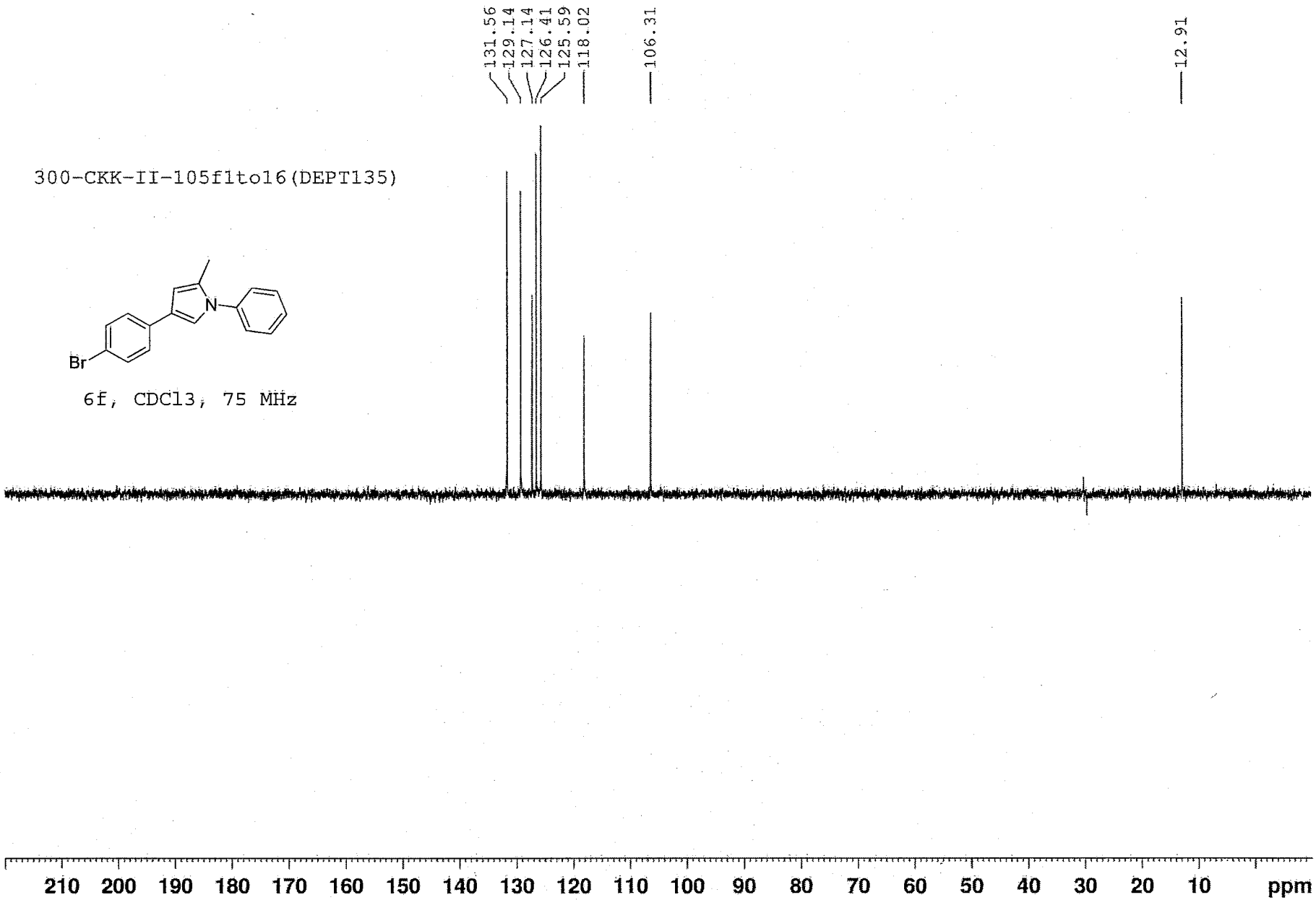
300-LYS-I-169purity(DEPT135)

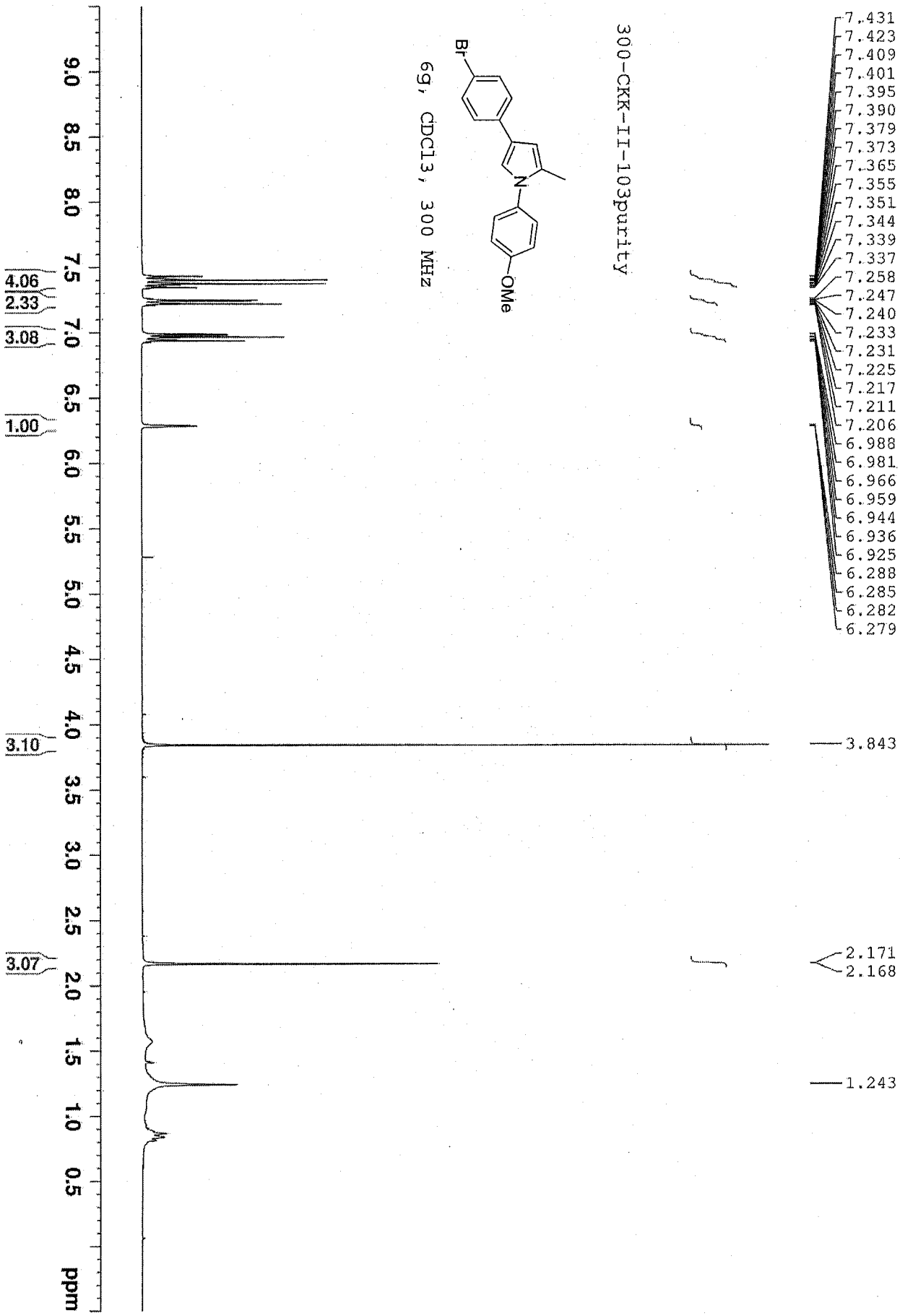
6e, CDCl3, 75 MHz

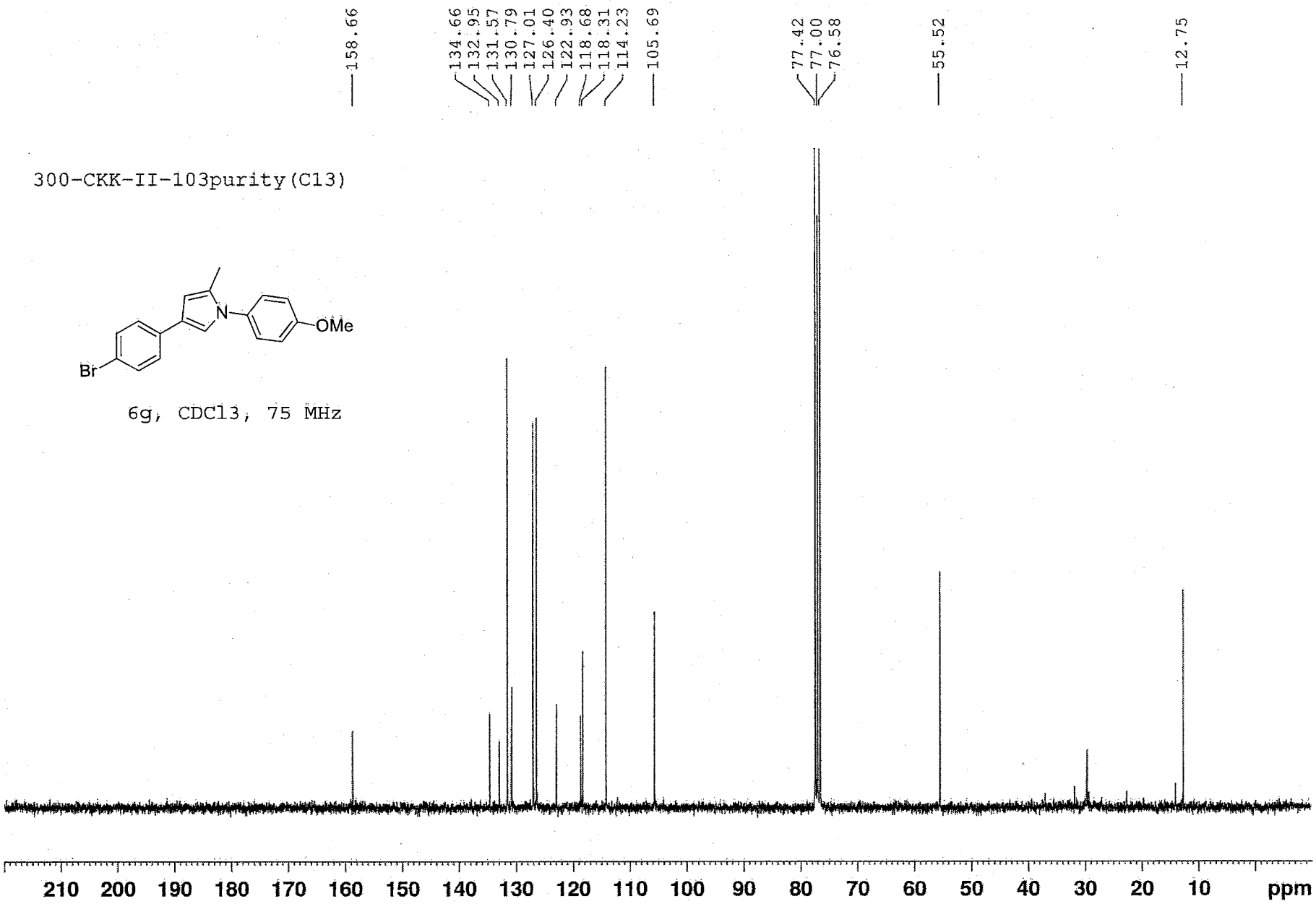


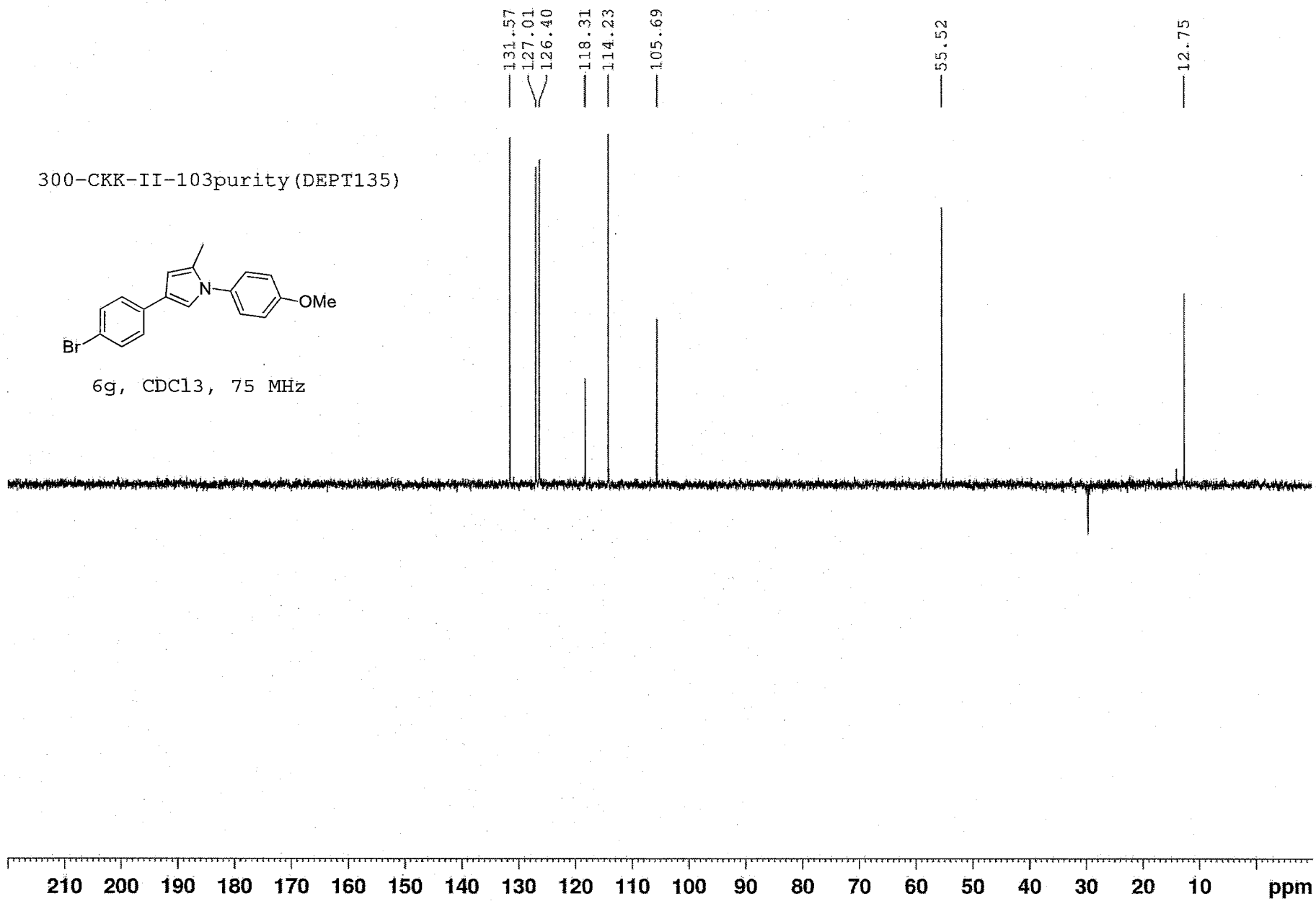

7.497
7.491
7.474
7.469
7.468
7.461
7.447
7.439
7.434
7.414
7.405
7.401
7.391
7.385
7.381
7.375
7.371
7.359
7.354
7.346
7.334
7.329
7.327
7.240
7.069
7.063
6.352
6.349
6.346
2.248
1.462
1.290
300-CKK-II-105f1to16(2)
N
Br
6f, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
9.05
1.00
0.99
3.03

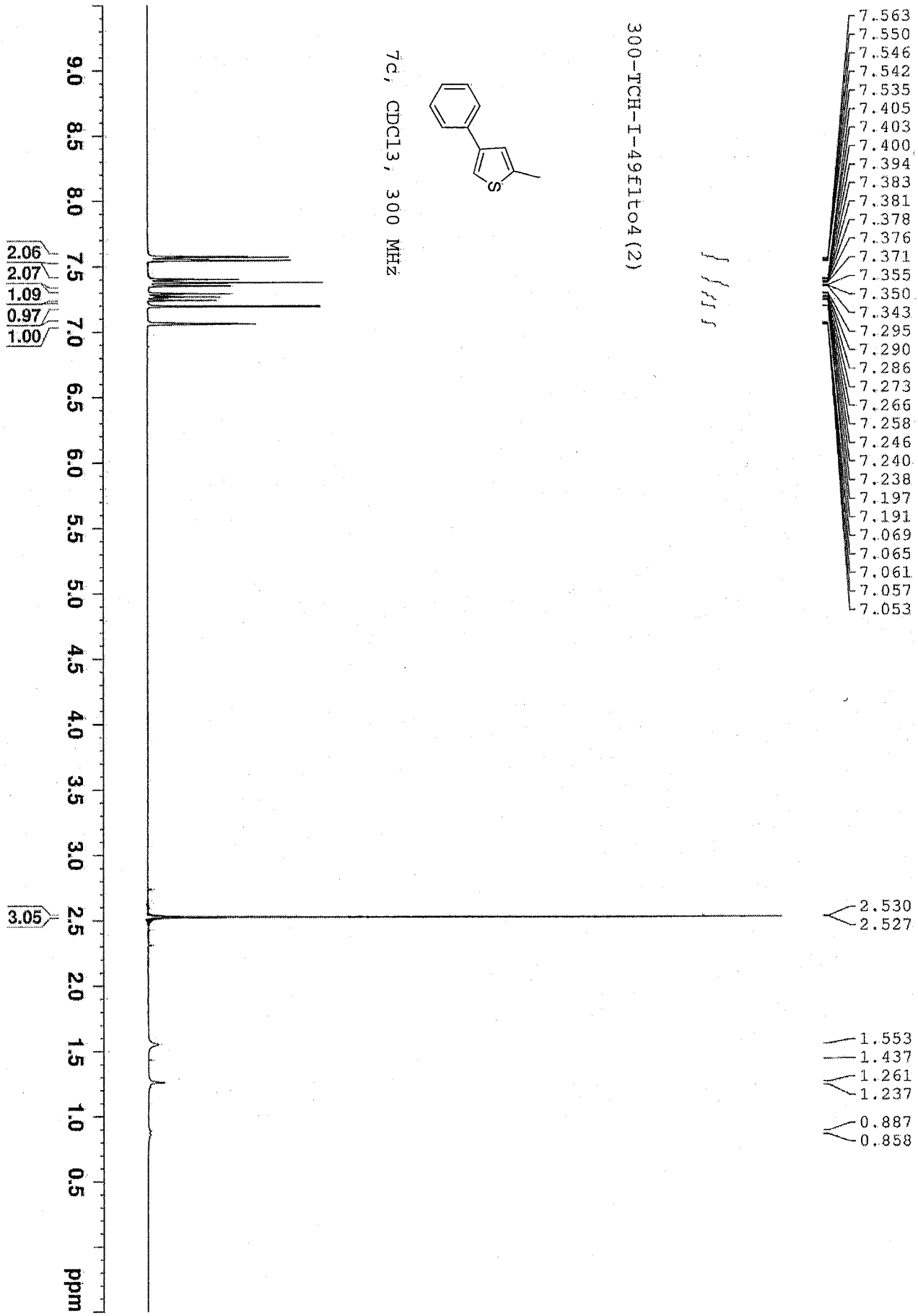

139.87
134.51
131.56
130.44
129.14
127.14
126.41
125.59
123.25
118.76
118.02
106.31
77.43
77.00
76.58
12.91
300-CKK-II-105f1to16(C13)
Br
N
6f, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

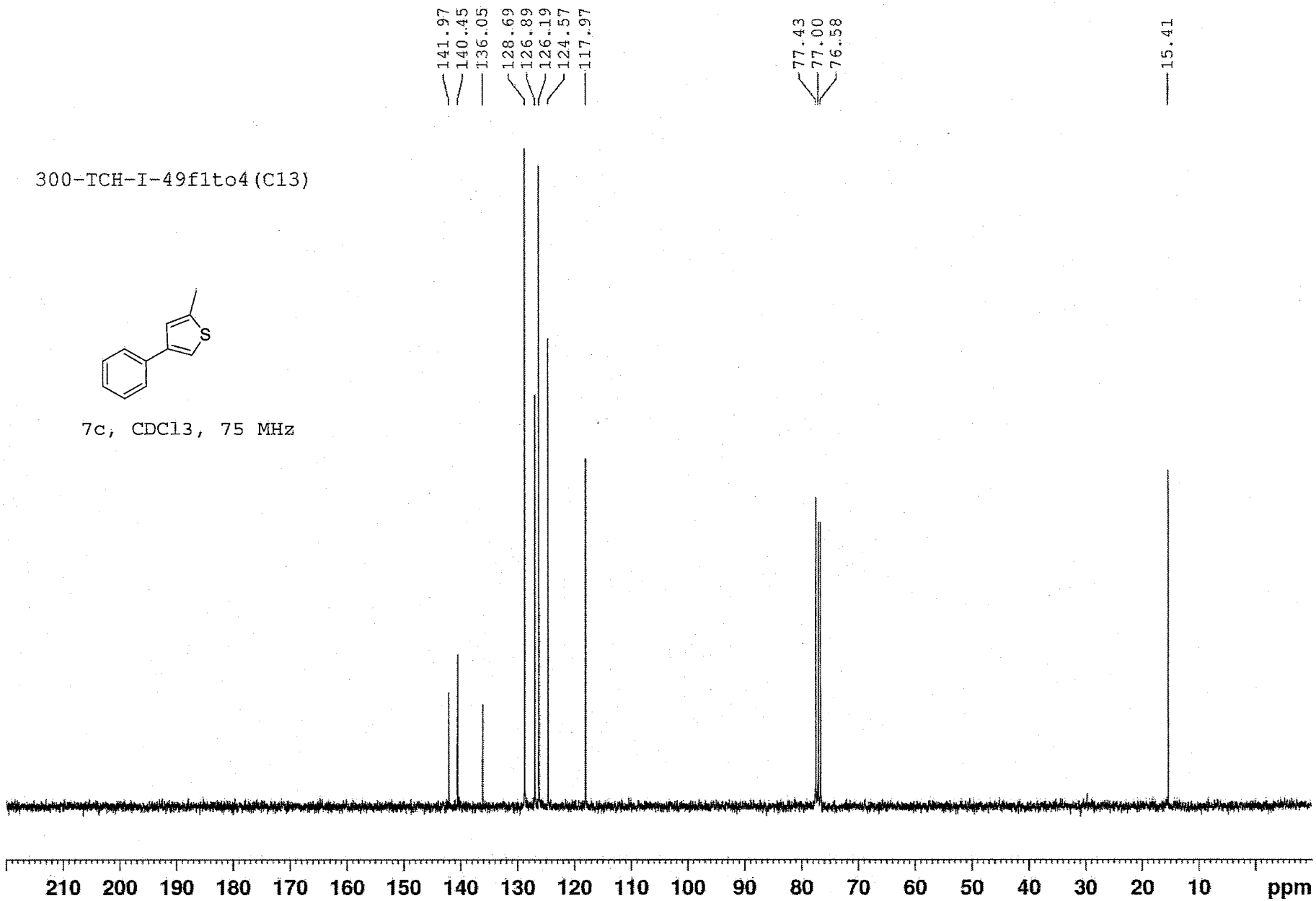

131.56
129.14
127.14
126.41
125.59
118.02
106.31
12.91
300-CKK-II-105f1to16(DEPT135)
N
Br
6f, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


300-CKK-II-103purity
6g, CDCl3, 300 MHz
Br
N
OMe
7.431
7.423
7.409
7.401
7.395
7.390
7.379
7.373
7.365
7.355
7.351
7.344
7.339
7.337
7.258
7.247
7.240
7.233
7.231
7.225
7.217
7.211
7.206
6.988
6.981
6.966
6.959
6.944
6.936
6.925
6.288
6.285
6.282
6.279
3.843
2.171
2.168
1.243
4.06
2.33
3.08
1.00
3.10
3.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


158.66
134.66
132.95
131.57
130.79
127.01
126.40
122.93
118.68
118.31
114.23
105.69
77.42
77.00
76.58
55.52
12.75
300-CKK-II-103purity(C13)
N
OMe
Br
6g, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

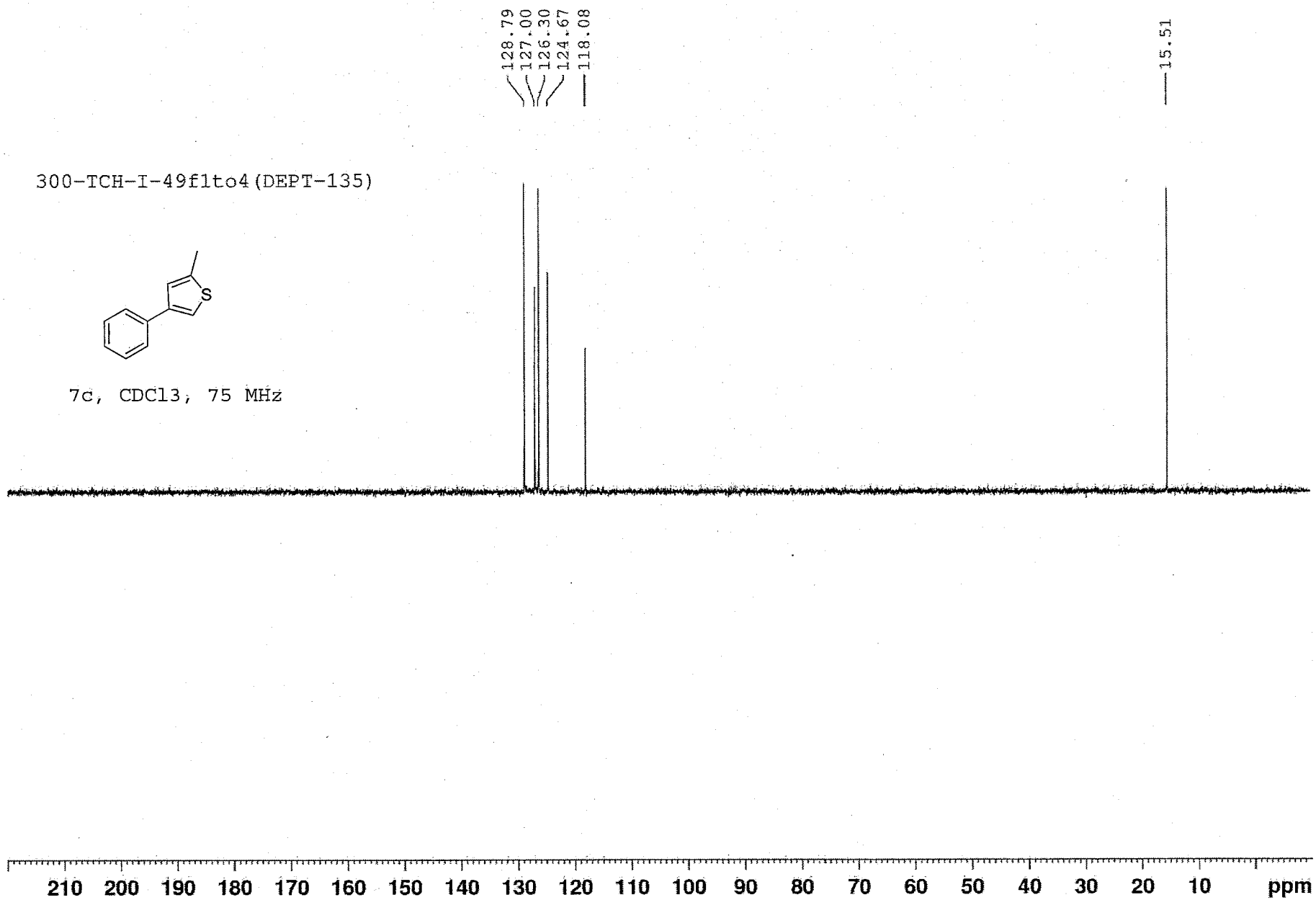

131.57
127.01
126.40
118.31
114.23
105.69
55.52
12.75
300-CKK-II-103purity(DEPT135)
N
OMe
Br
6g, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

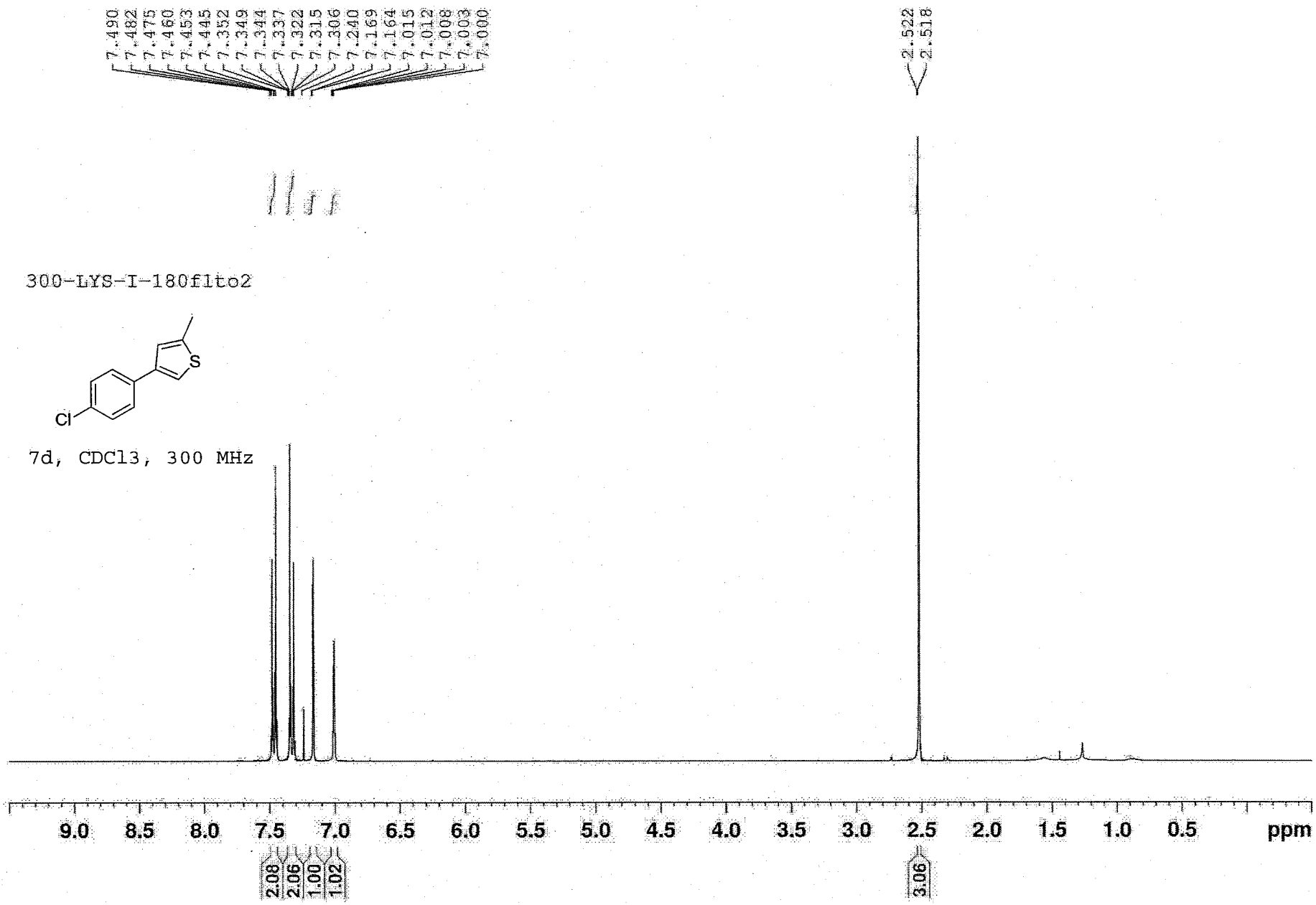

300-TCH-I-49f1to4(2)
7c, CDCl3, 300 MHz
7.563
7.550
7.546
7.542
7.535
7.405
7.403
7.400
7.394
7.383
7.381
7.378
7.376
7.371
7.355
7.350
7.343
7.295
7.290
7.286
7.273
7.266
7.258
7.246
7.240
7.238
7.197
7.191
7.069
7.065
7.061
7.057
7.053
2.530
2.527
1.553
1.437
1.261
1.237
0.887
0.858
2.06
2.07
1.09
0.97
1.00
3.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

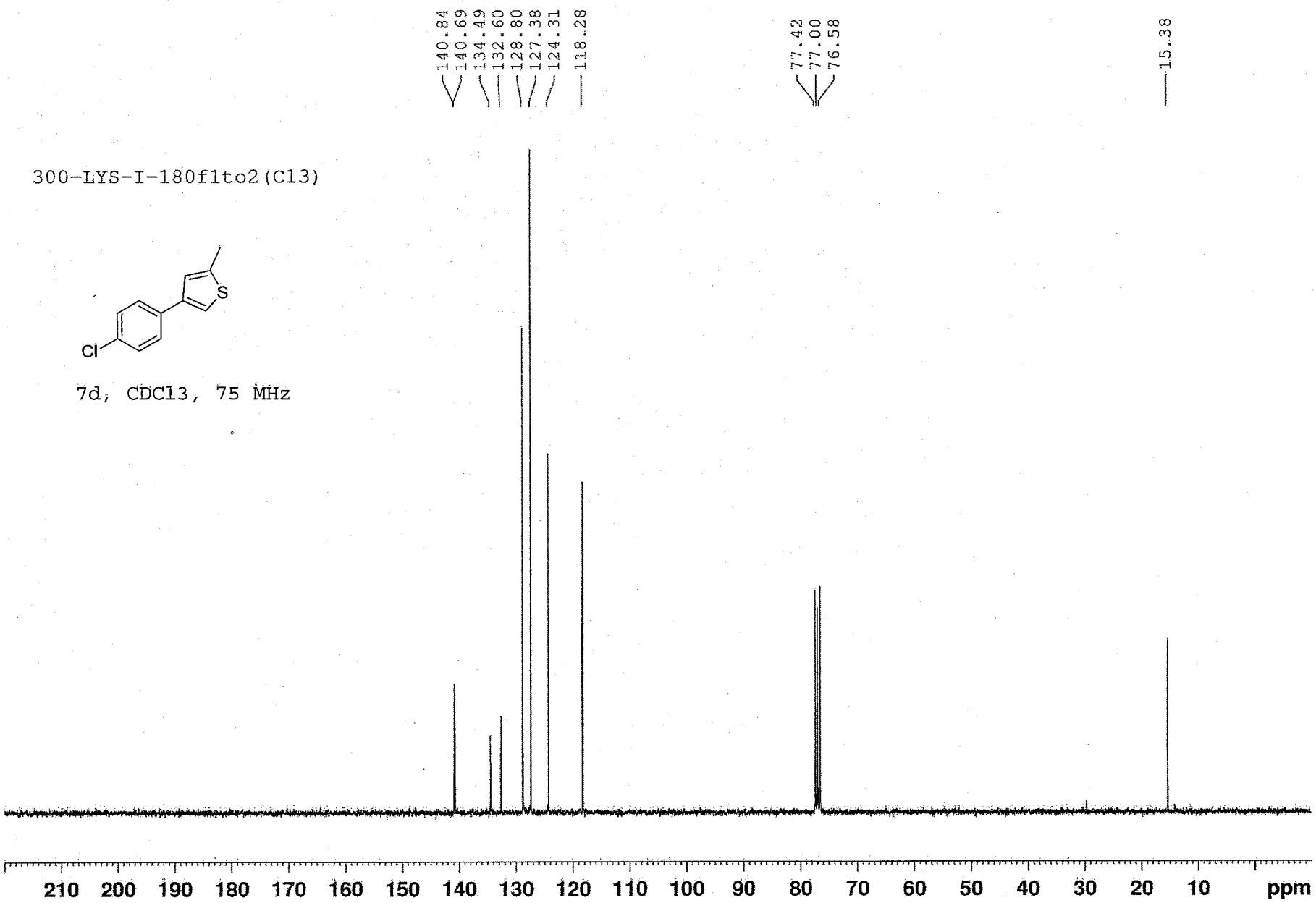

300-TCH-I-49f1to4(C13)
7c, CDCl3, 75 MHz
S
141.97
140.45
136.05
128.69
126.89
126.19
124.57
117.97
77.43
77.00
76.58
15.41
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

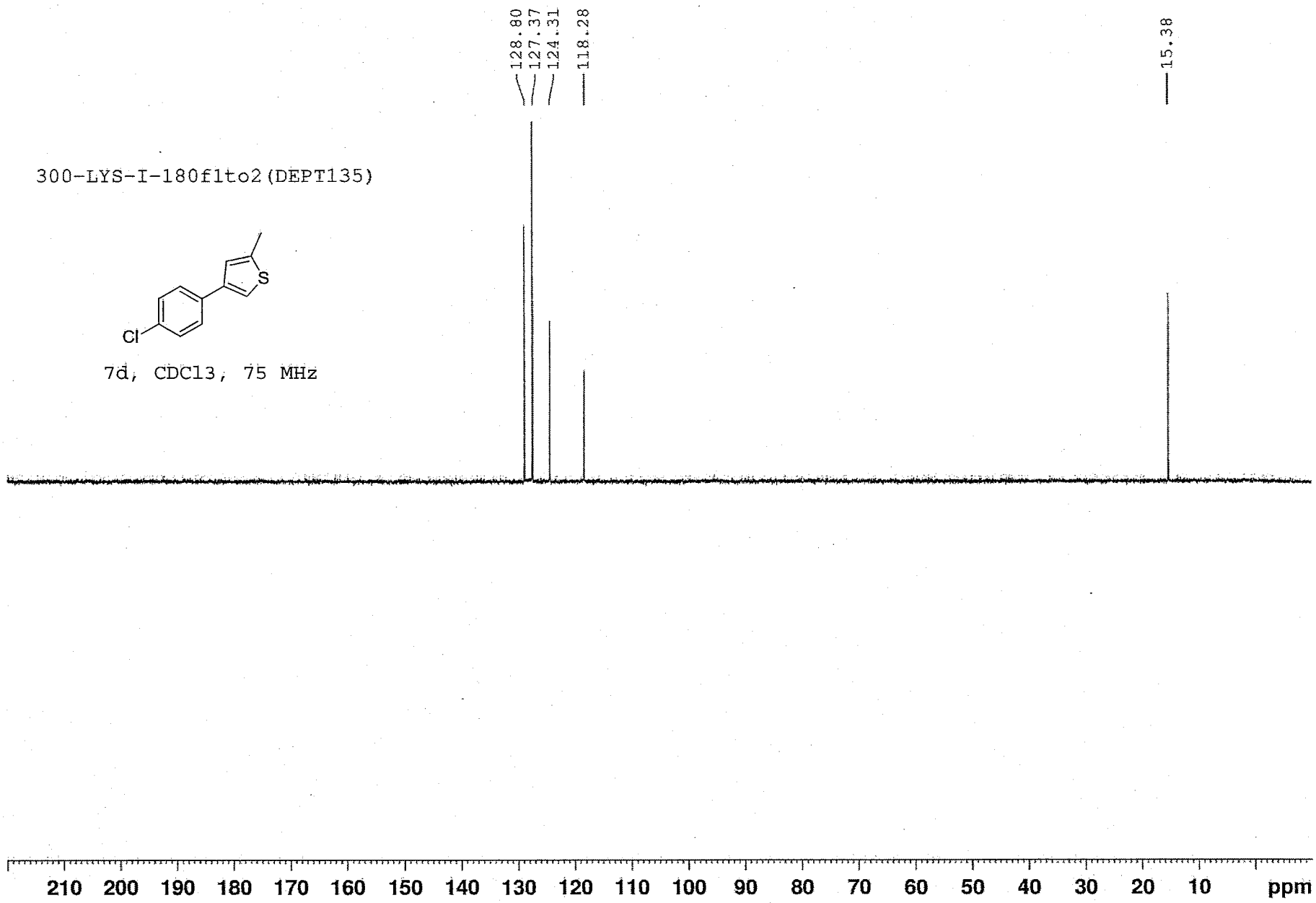
300-TCH-I-49f1to4(DEPT-135)

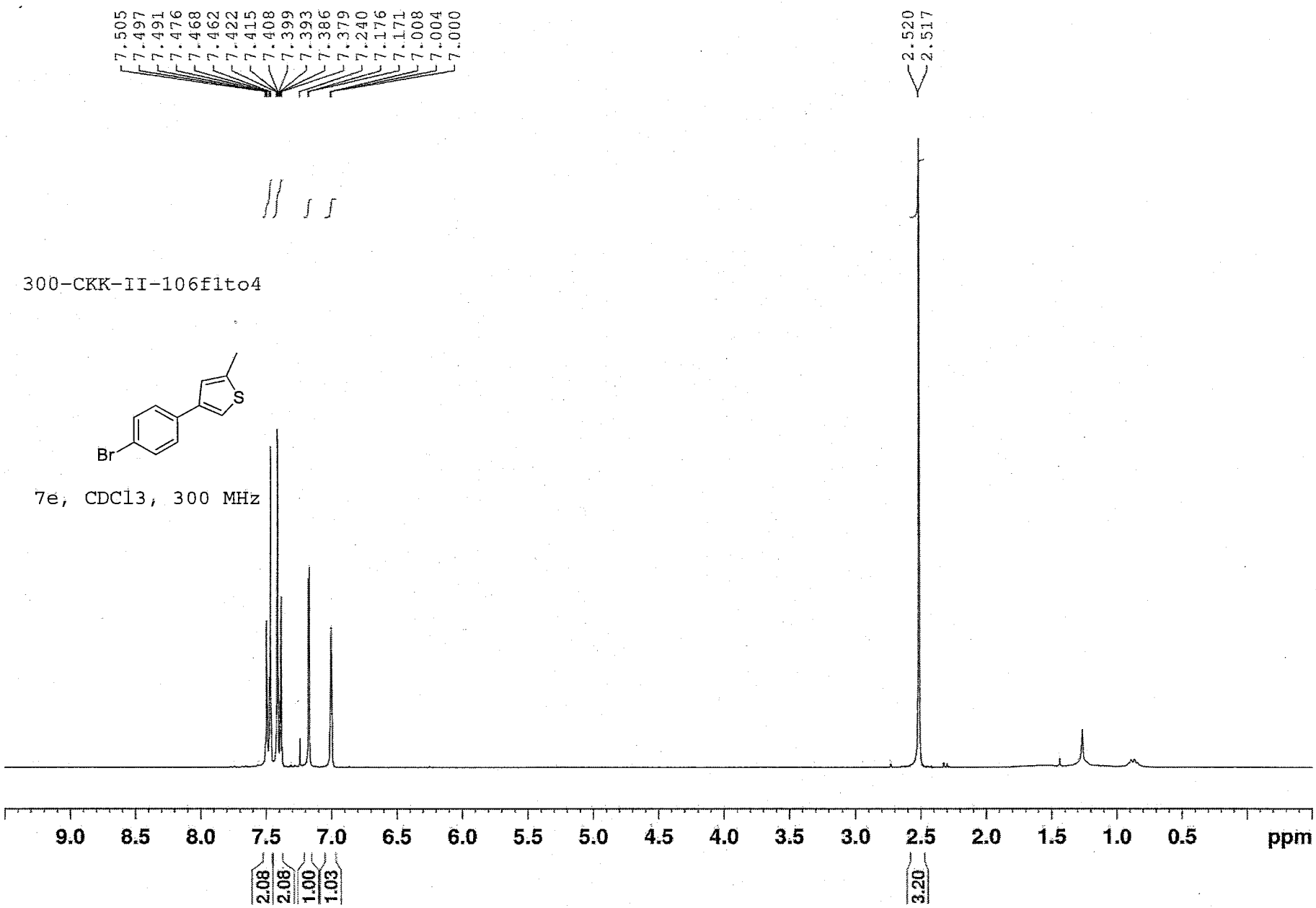
7c, CDCl3, 75 MHz




7.490
7.482
7.475
7.460
7.453
7.445
7.352
7.349
7.344
7.337
7.322
7.315
7.306
7.240
7.169
7.164
7.015
7.012
7.008
7.003
7.000
2.522
2.518
300-LYS-I-180flto2
S
Cl
7d, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.08
2.06
1.00
1.02
3.06

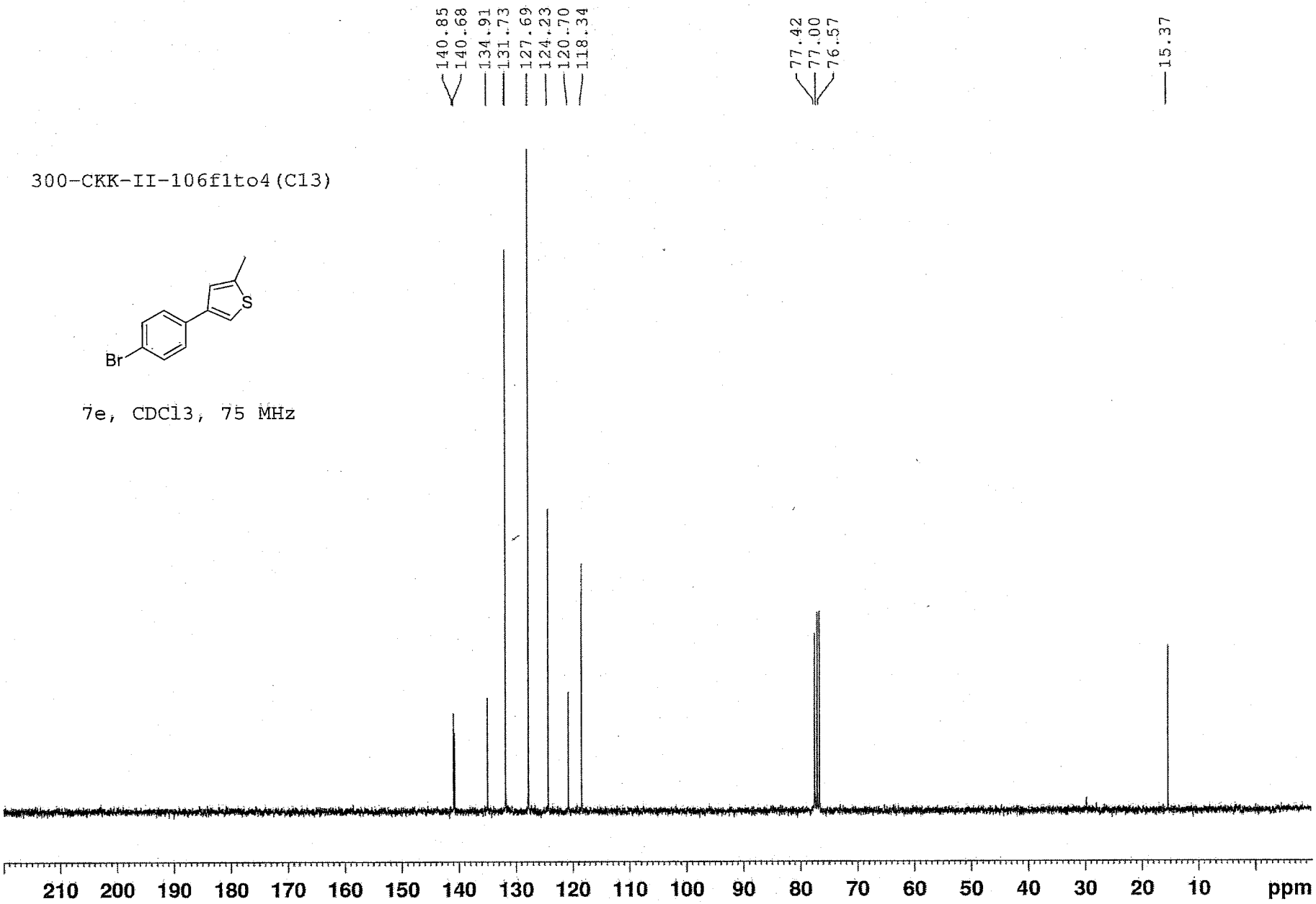

140.84
140.69
134.49
132.60
128.80
127.38
124.31
118.28
77.42
77.00
76.58
15.38
300-LYS-I-180f1to2(C13)
S
Cl
7d, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

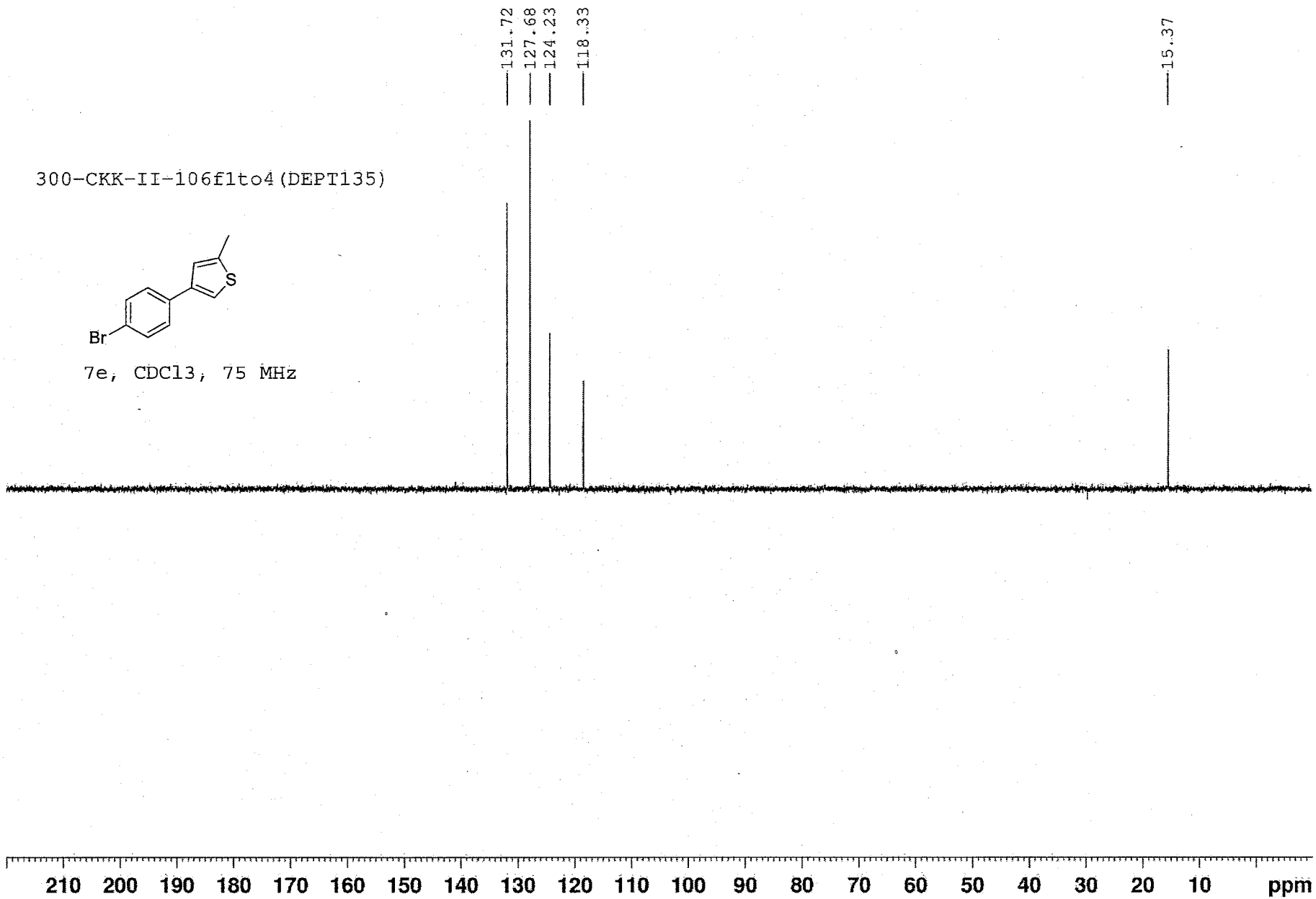

128.80
127.37
124.31
118.28
15.38
300-LYS-I-180f1to2(DEPT135)
S
Cl
7d, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

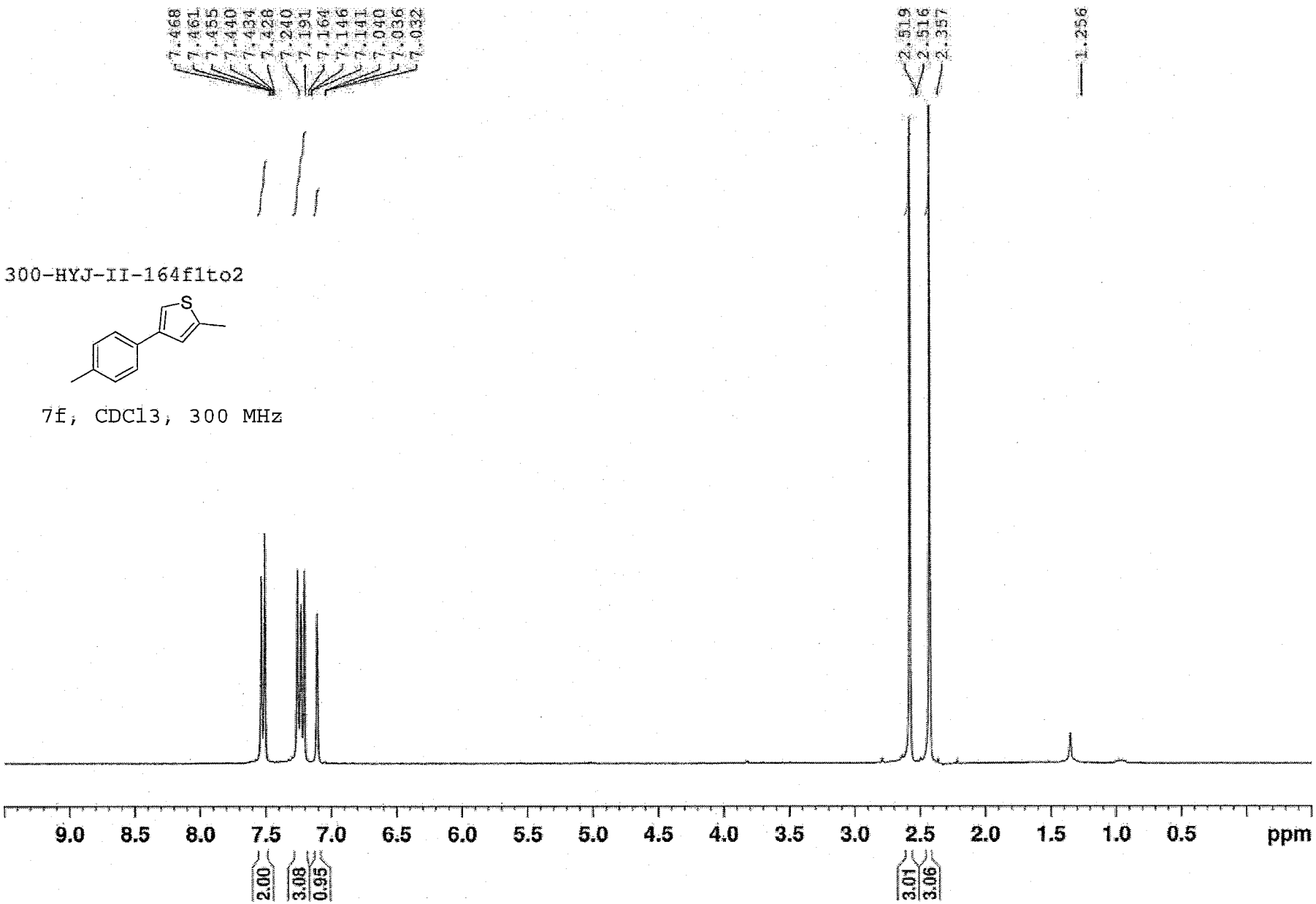

7.505
7.497
7.491
7.476
7.468
7.462
7.422
7.415
7.408
7.399
7.393
7.386
7.379
7.240
7.176
7.171
7.008
7.004
7.000
2.520
2.517
300-CKK-II-106f1to4
S
Br
7e, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.08
2.08
1.00
1.03
3.20

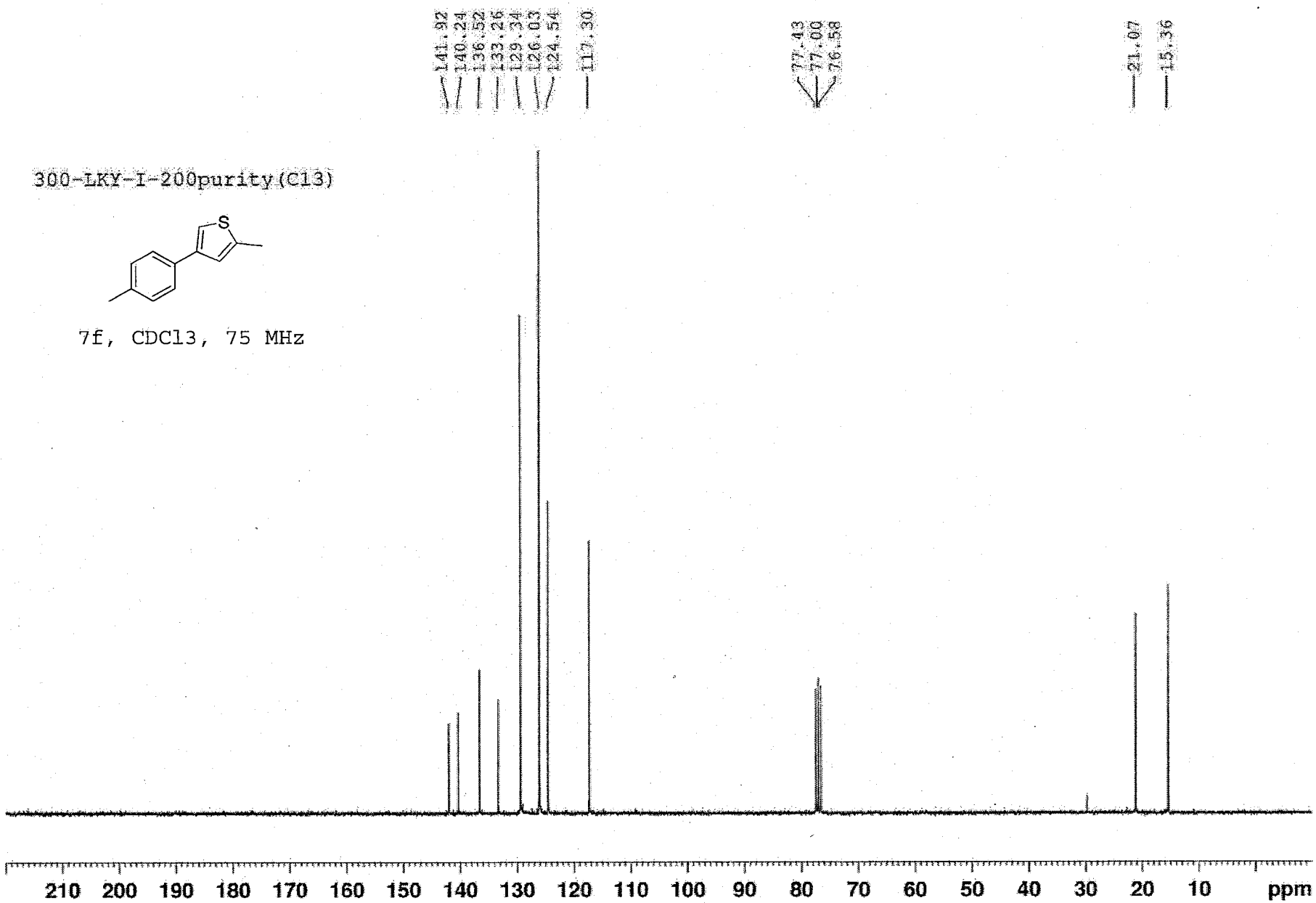
300-CKK-II-106f1to4(C13)

7e, CDCl3, 75 MHz



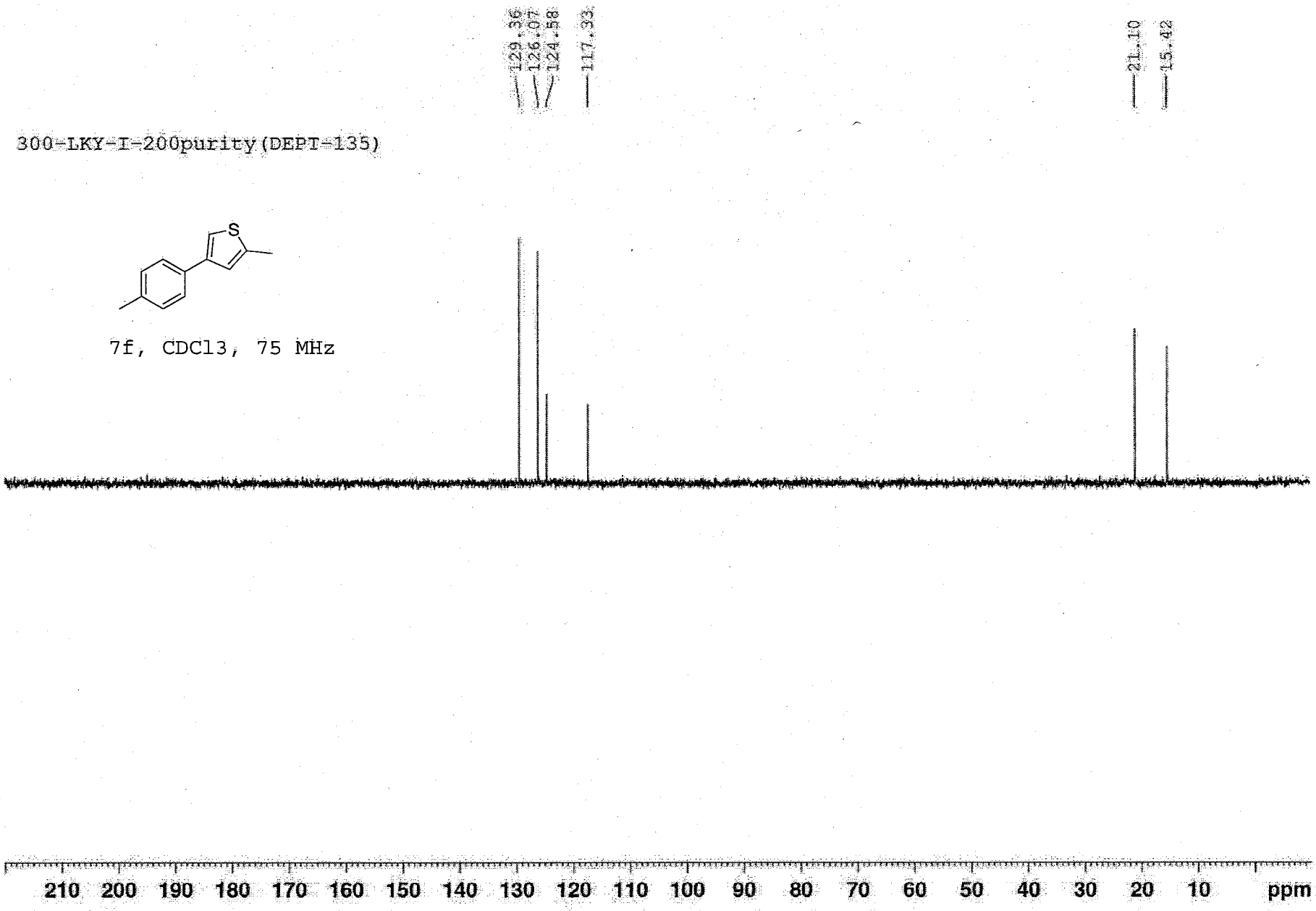

131.72
127.68
124.23
118.33
15.37
300-CKK-II-106f1to4(DEPT135)
S
Br
7e, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

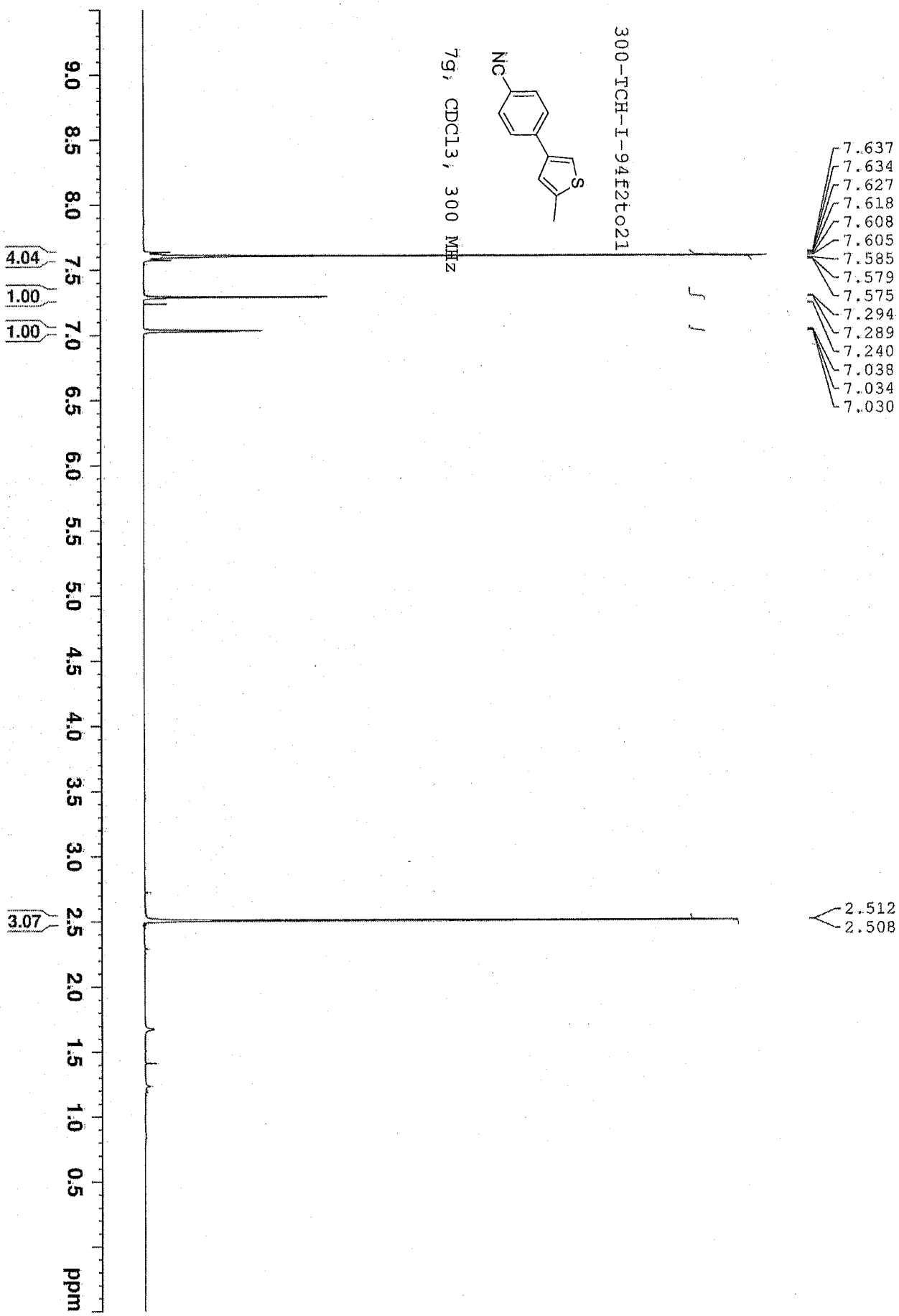

7.468
7.461
7.455
7.440
7.434
7.428
7.240
7.191
7.164
7.146
7.141
7.040
7.036
7.032
2.519
2.516
2.357
1.256
300-HYJ-II-164f1to2
S
7f, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.00
3.08
0.95
3.01
3.06

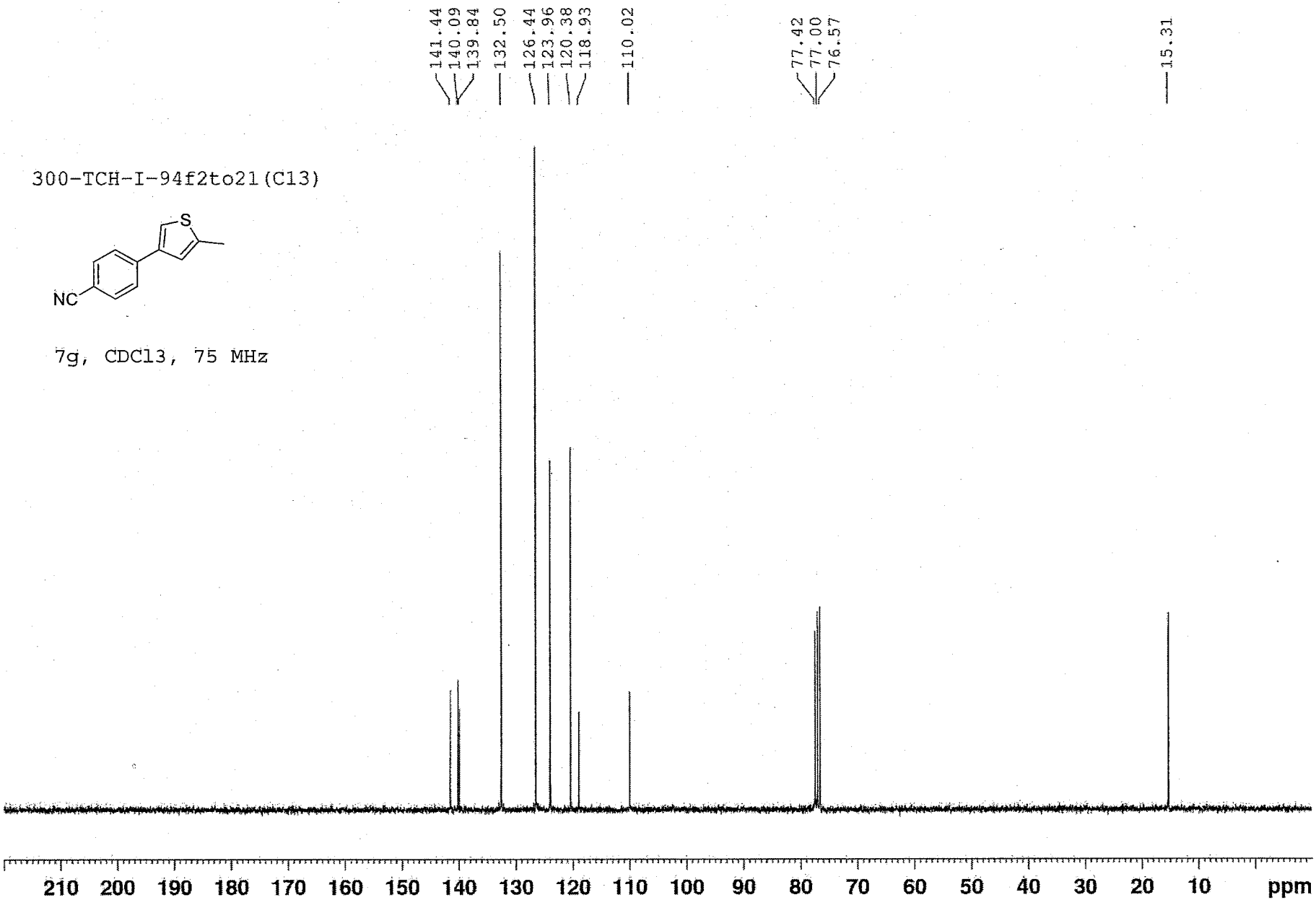

141.92
140.24
136.52
133.26
129.34
126.03
124.54
117.30
77.43
77.00
76.58
21.07
15.36
300-LKY-I-200purity(C13)
S
7f, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

300-LKY-I-200purity(DEPT-135)

7f, CDCl3, 75 MHz

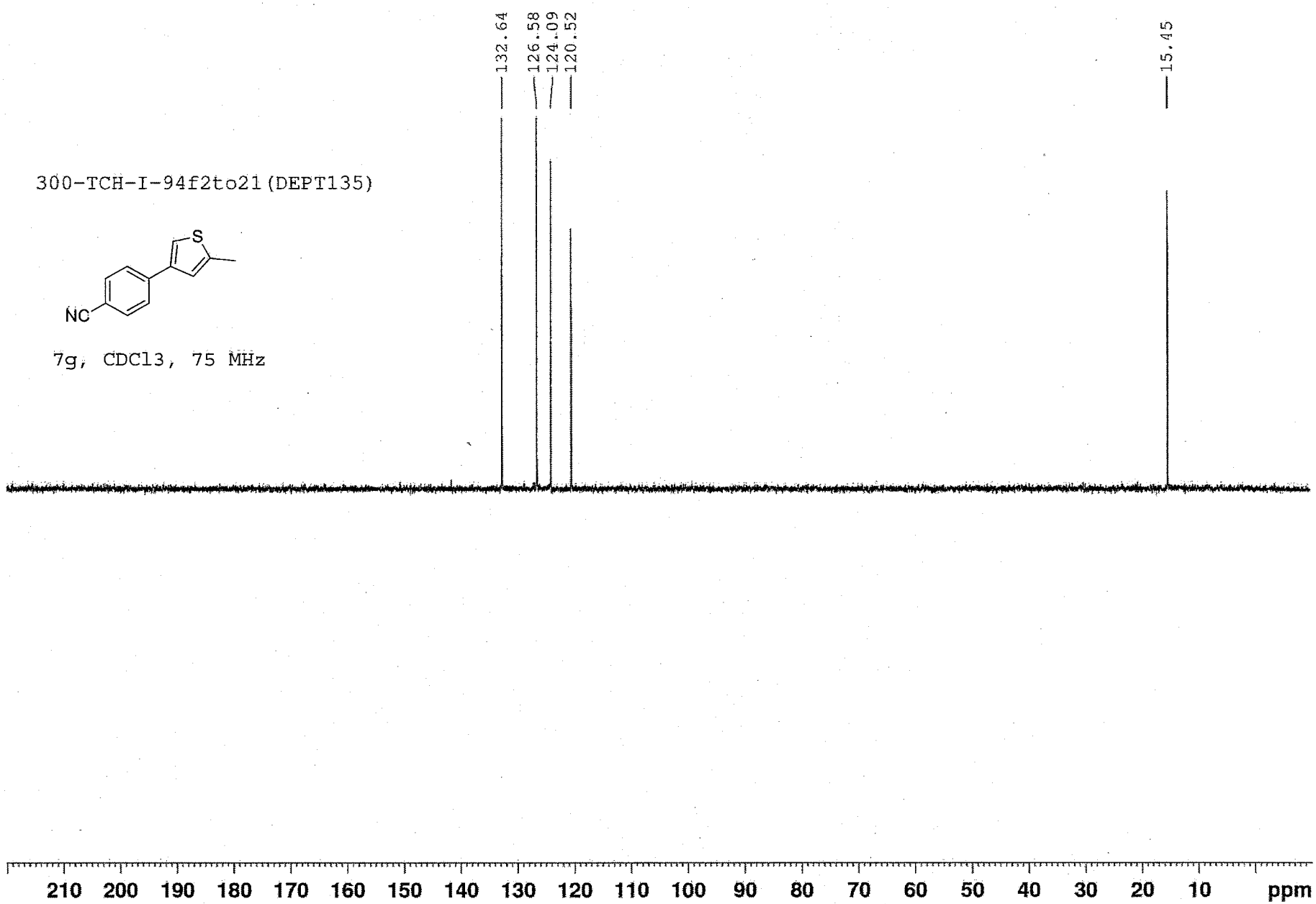


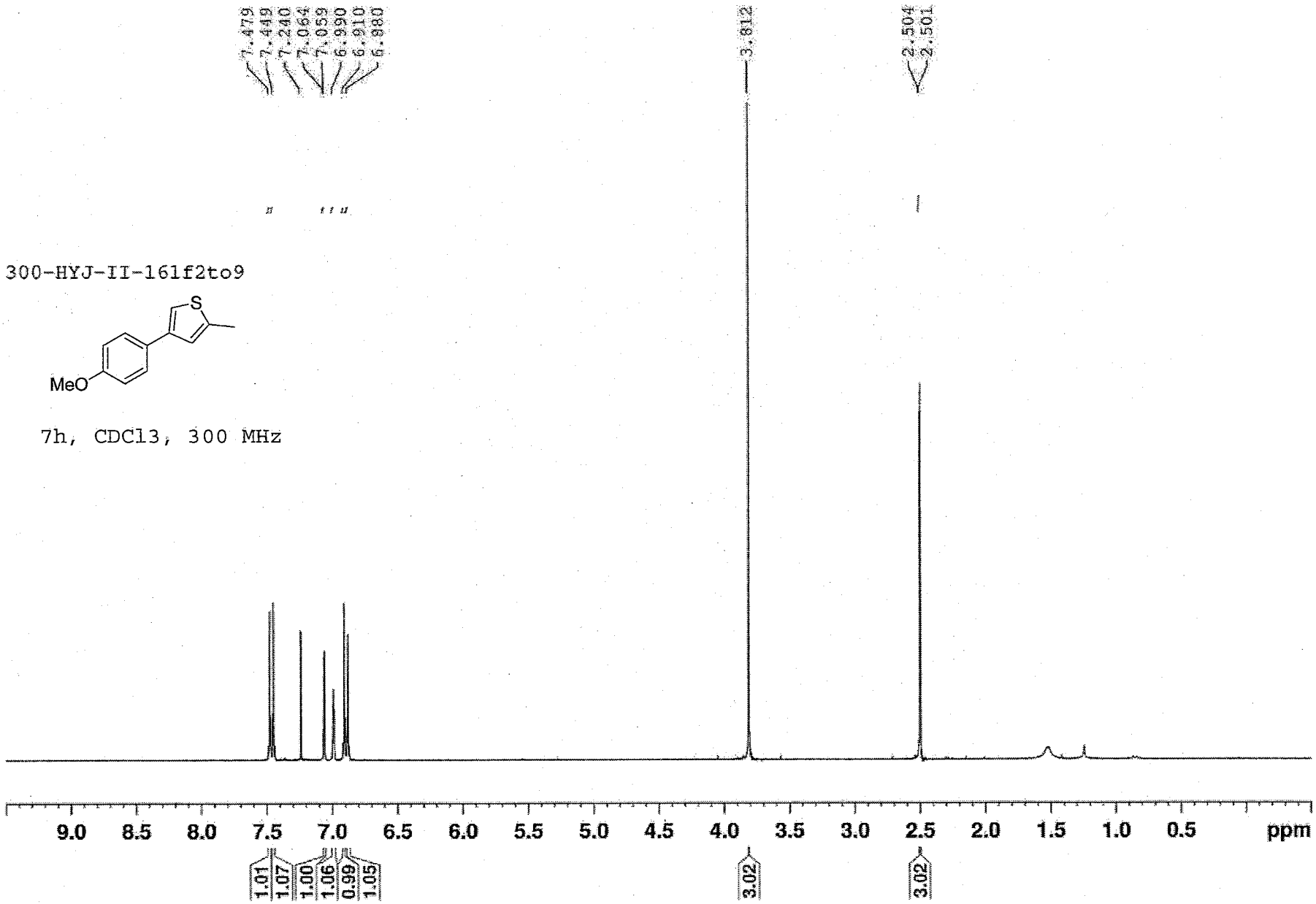

300-TCH-I-94f2to21
7g, CDCl3, 300 MHz
NC
S
7.637
7.634
7.627
7.618
7.608
7.605
7.585
7.579
7.575
7.294
7.289
7.240
7.038
7.034
7.030
2.512
2.508
4.04
1.00
1.00
3.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


141.44
140.09
139.84
132.50
126.44
123.96
120.38
118.93
110.02
77.42
77.00
76.57
15.31
300-TCH-I-94f2to21(C13)
S
NC
7g, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

300-TCH-I-94f2to21(DEPT135)

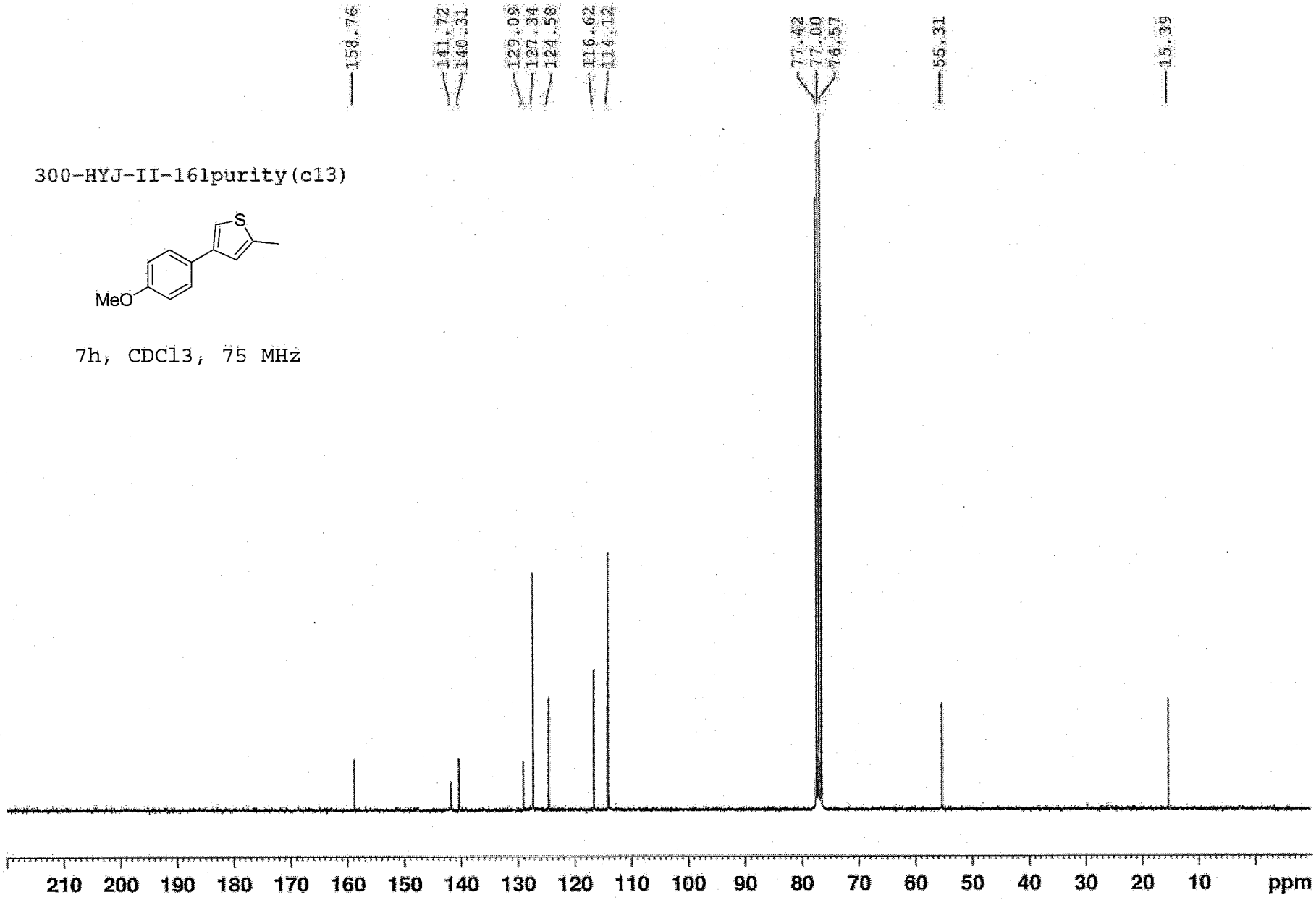
7g, CDCl3, 75 MHz

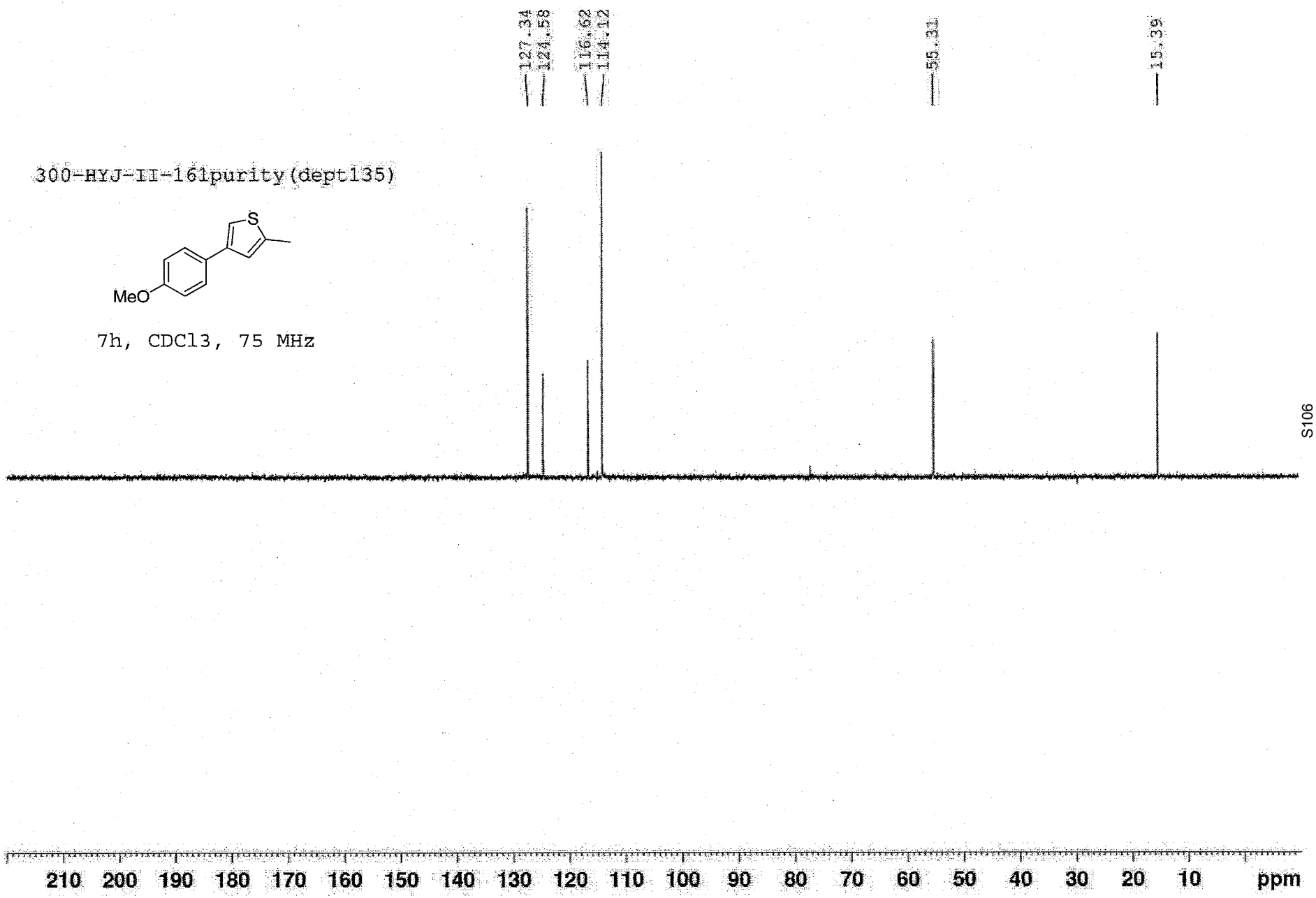


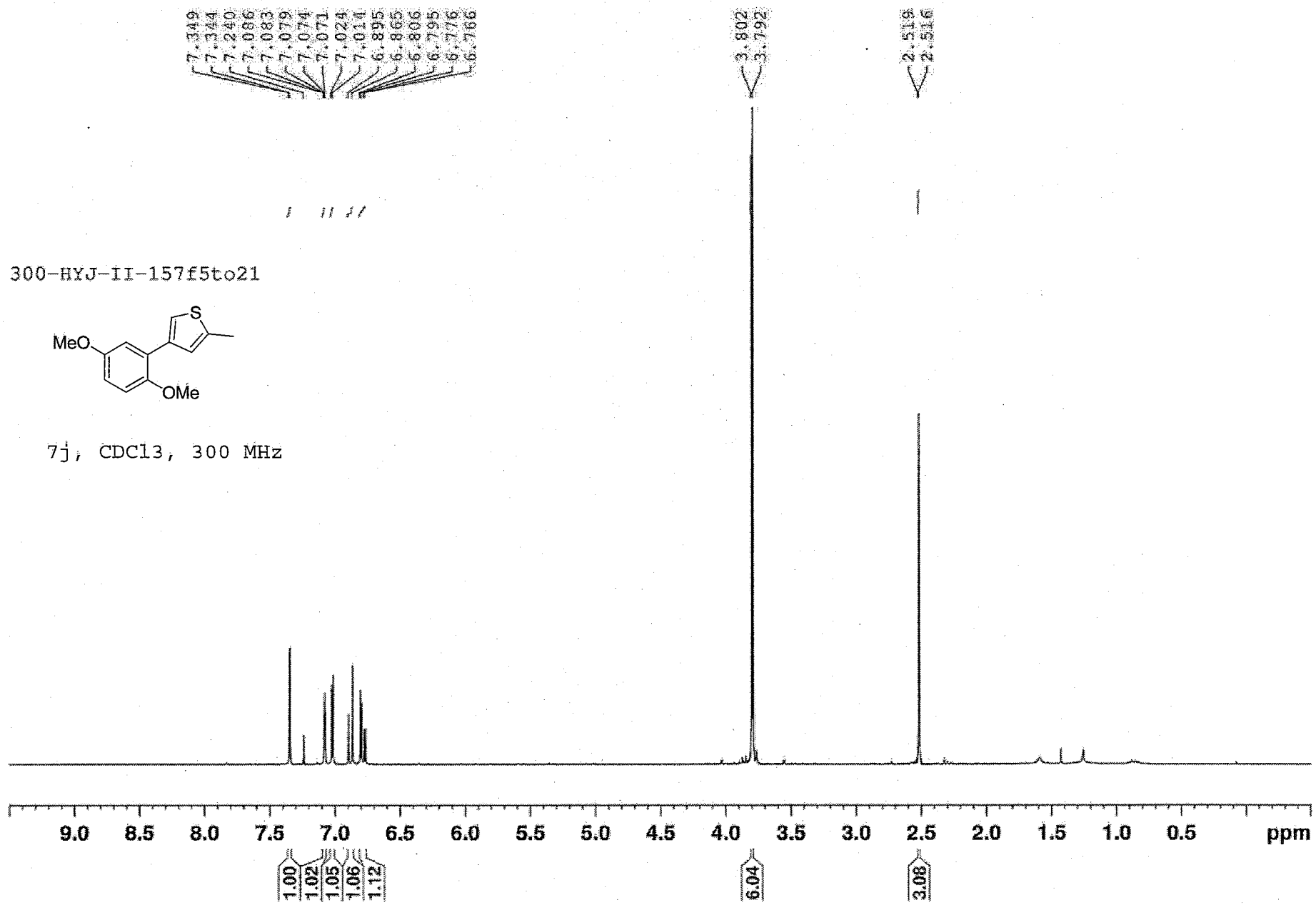

7.479
7.449
7.240
7.064
7.059
6.990
6.910
6.980
3.812
2.504
2.501
300-HYJ-II-161f2to9
S
MeO
7h, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.01
1.07
1.00
1.06
0.99
1.05
3.02
3.02

300-HYJ-II-161purity(c13)

7h, CDCl3, 75 MHz

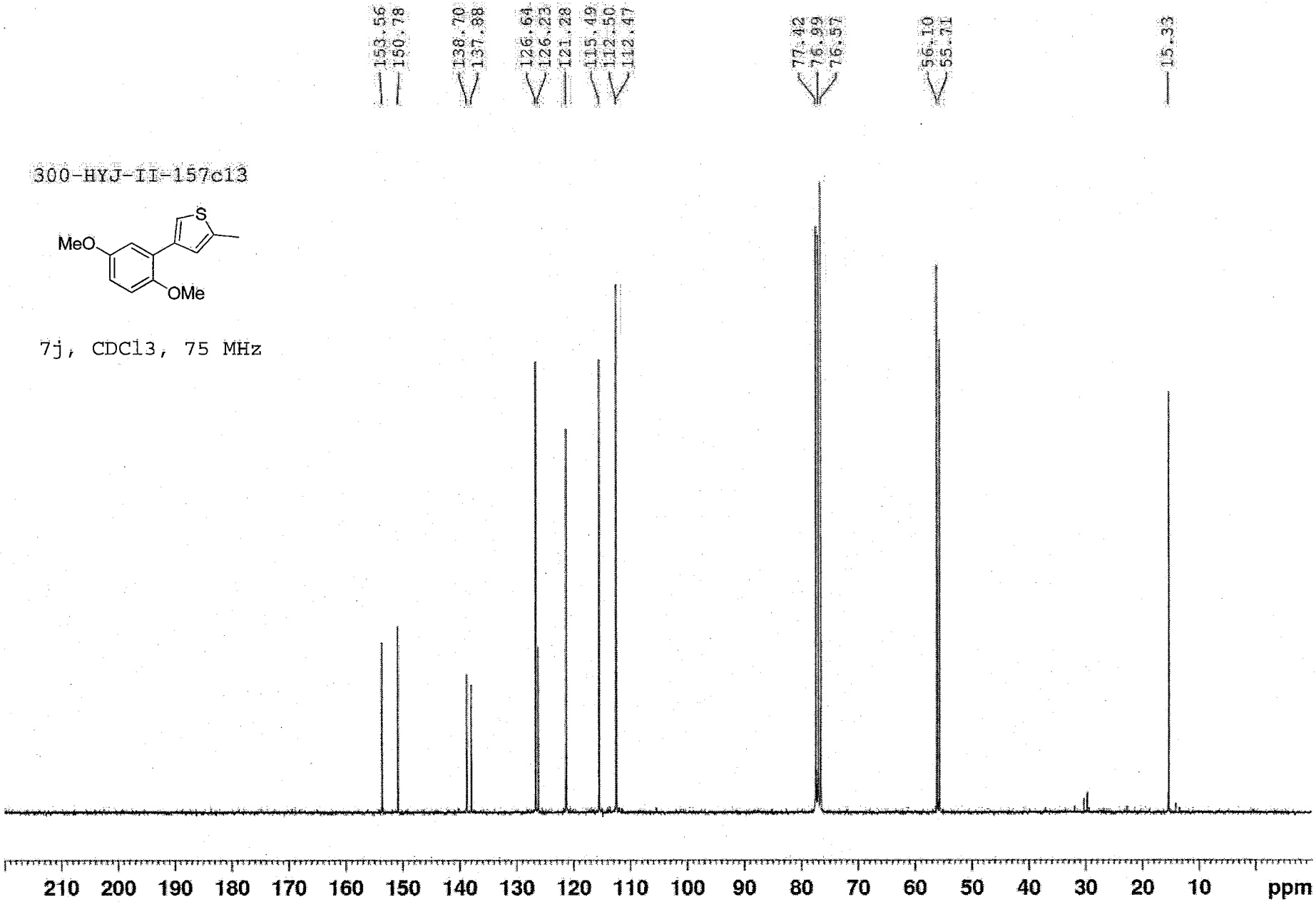


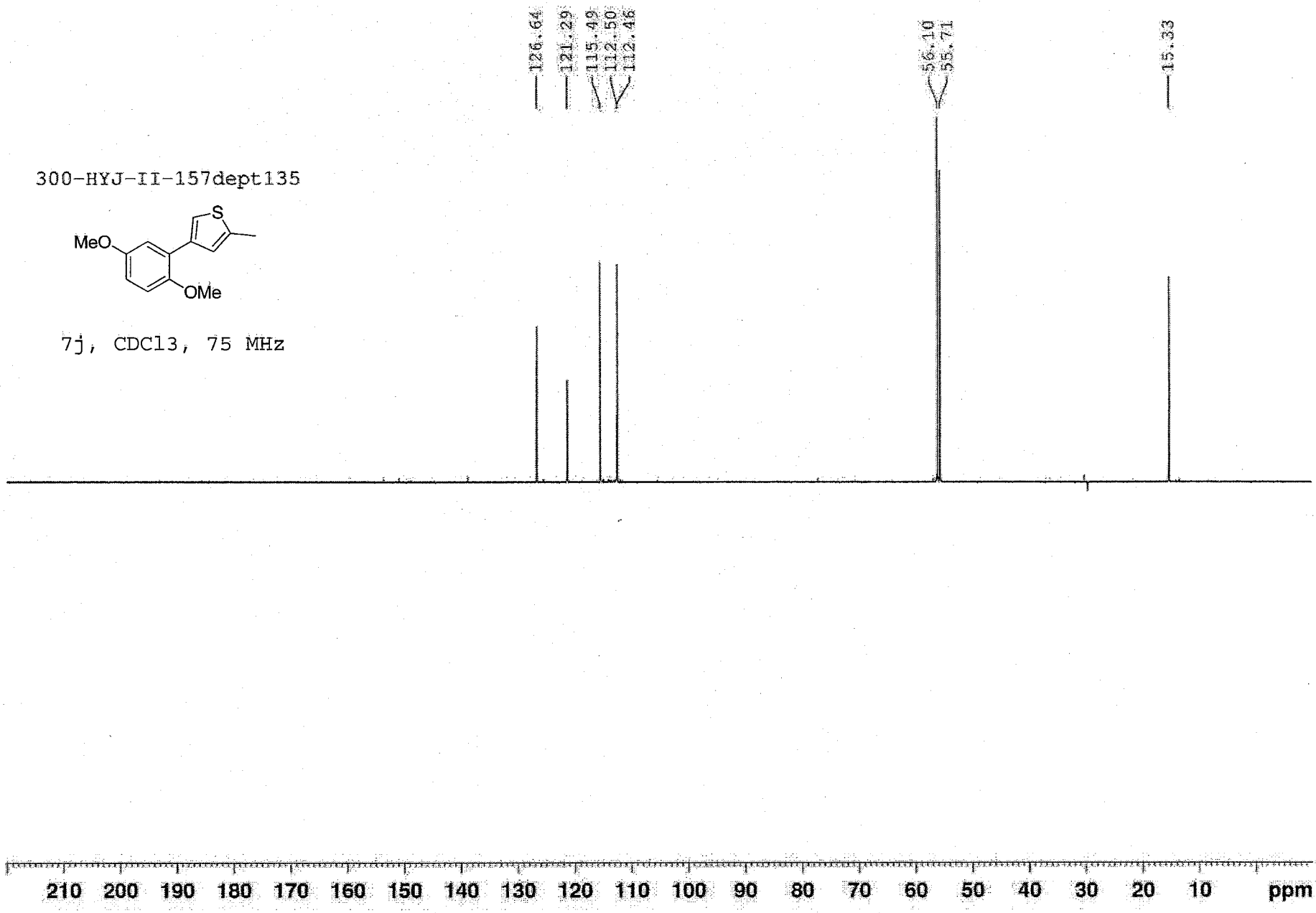

300-HYJ-II-161purity(dept135)
S
MeO
7h, CDCl3, 75 MHz
127.34
124.58
116.62
114.12
55.31
15.39
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

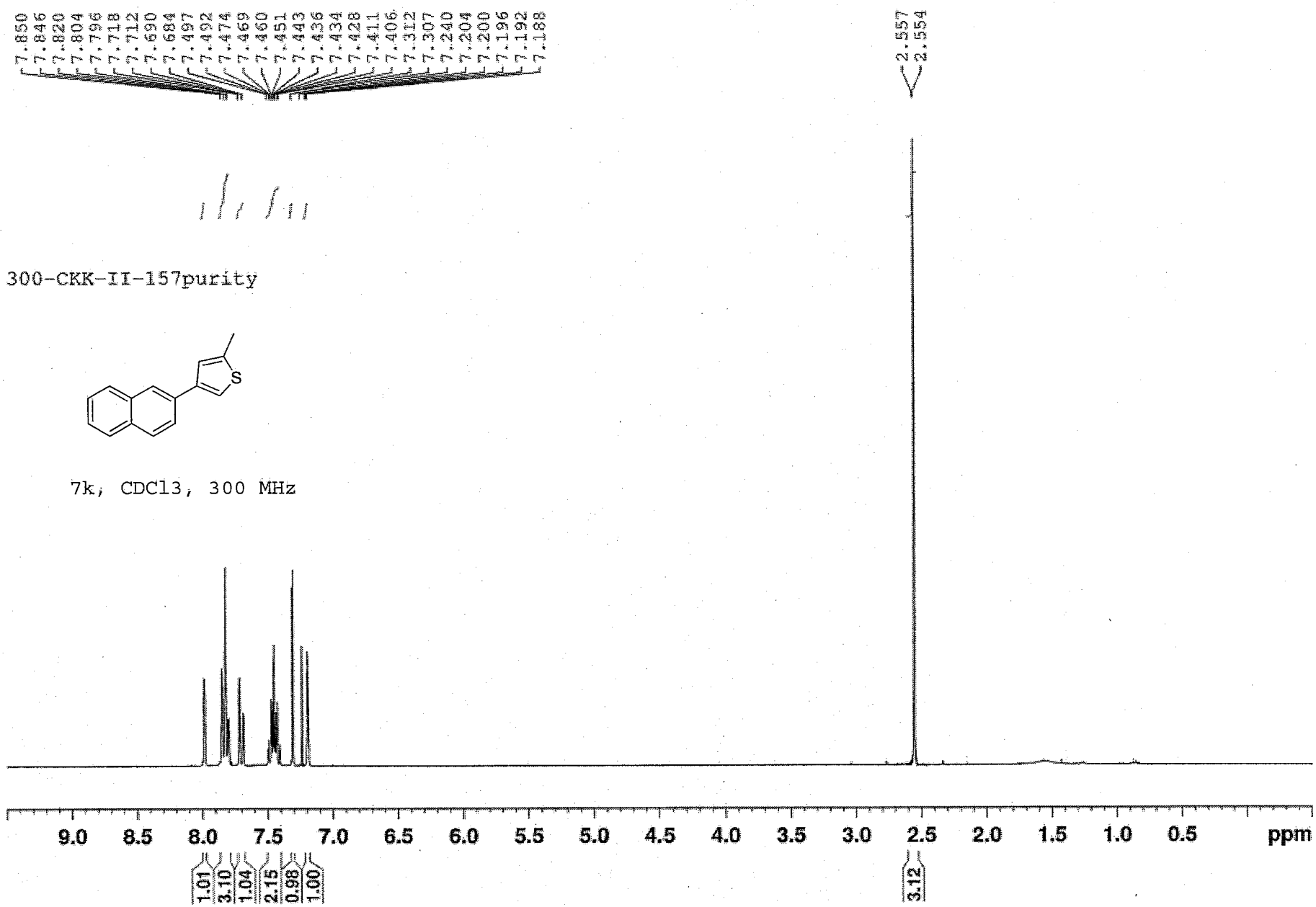

7.349
7.344
7.240
7.086
7.083
7.079
7.074
7.071
7.024
7.014
6.895
6.865
6.806
6.795
6.776
6.766
3.802
3.792
2.519
2.516
300-HYJ-II-157f5to21
MeO
OMe
S
7j, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.02
1.05
1.06
1.12
6.04
3.08

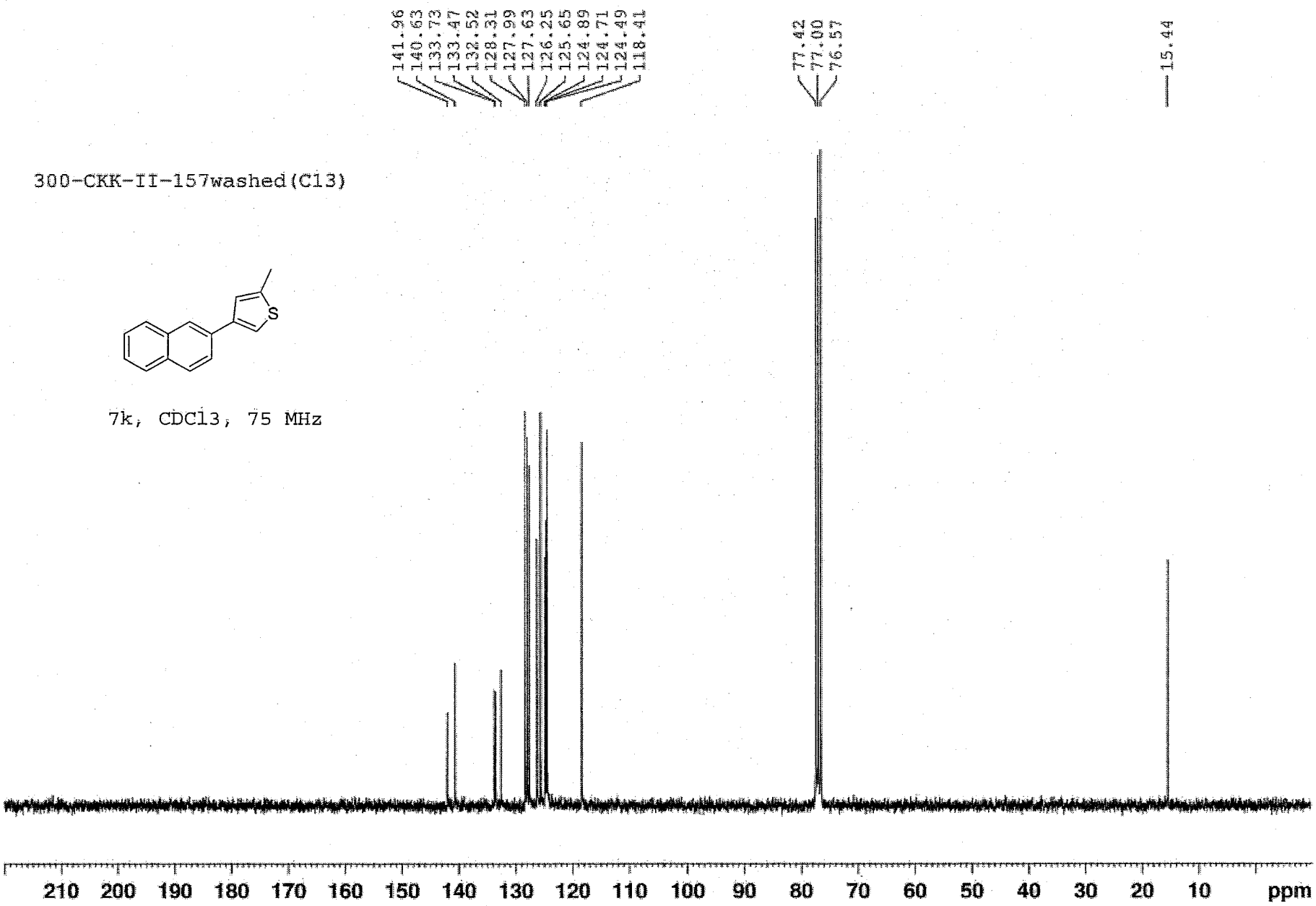
300-HYJ-II-157c13

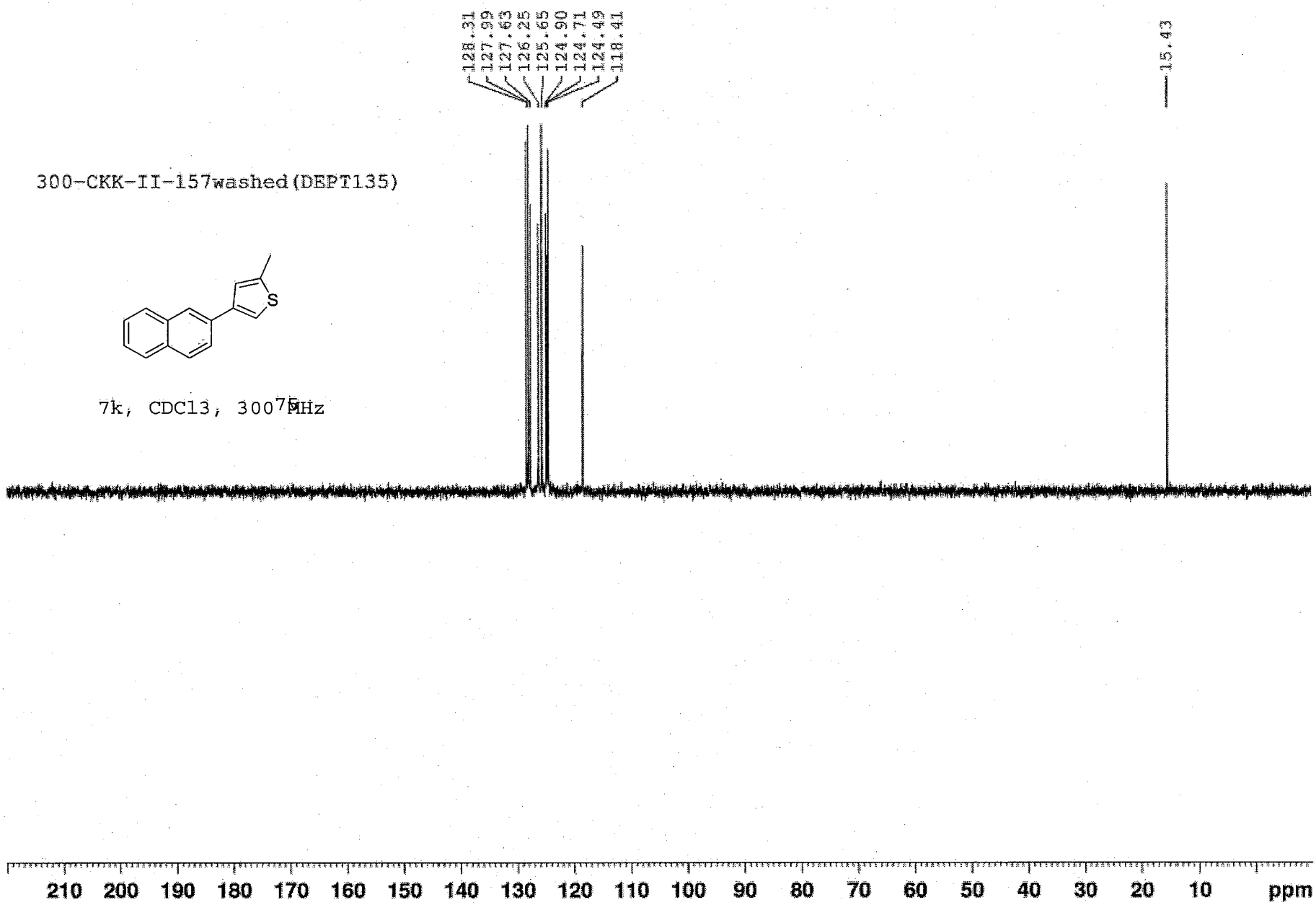
7j, CDCl3, 75 MHz

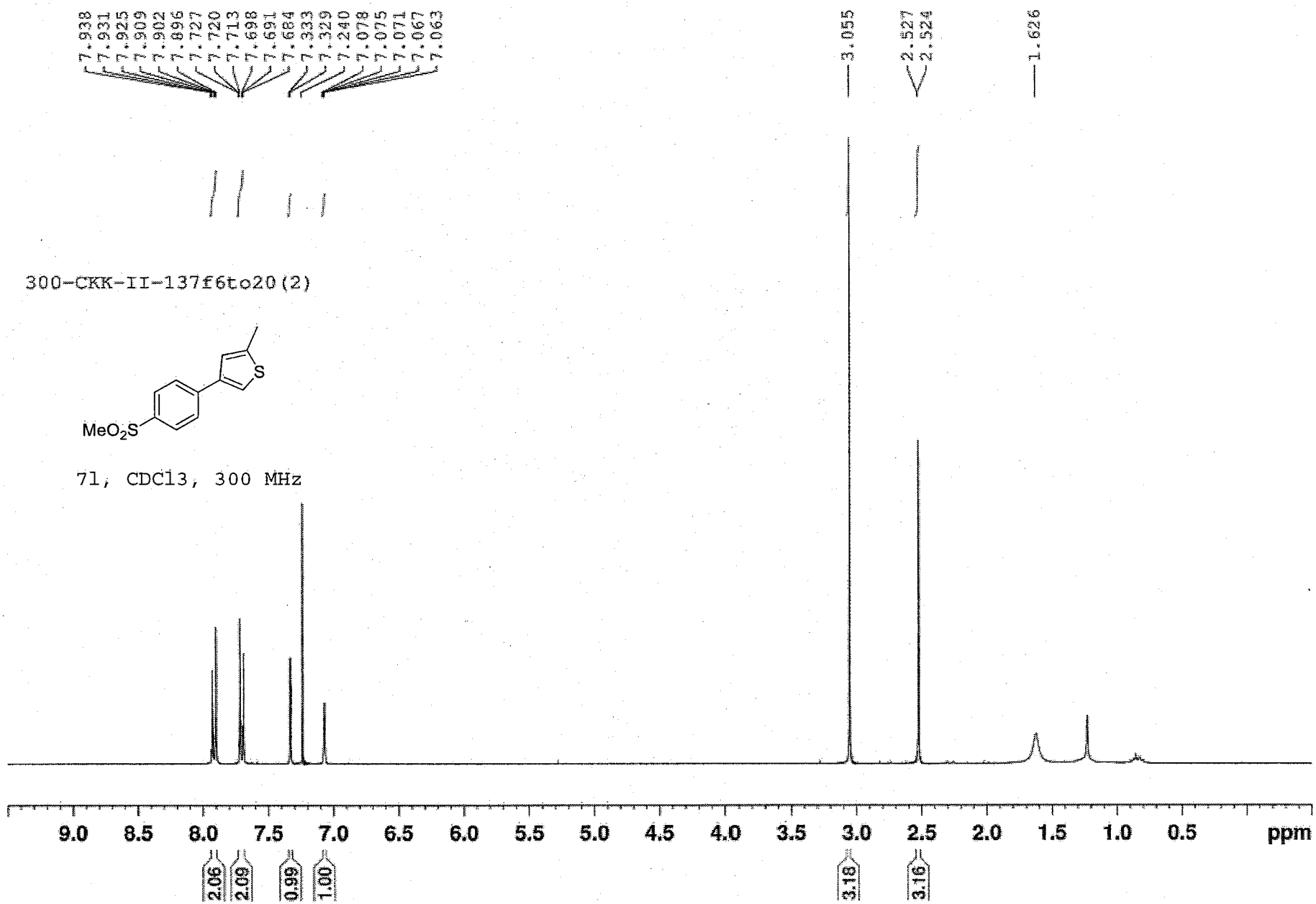


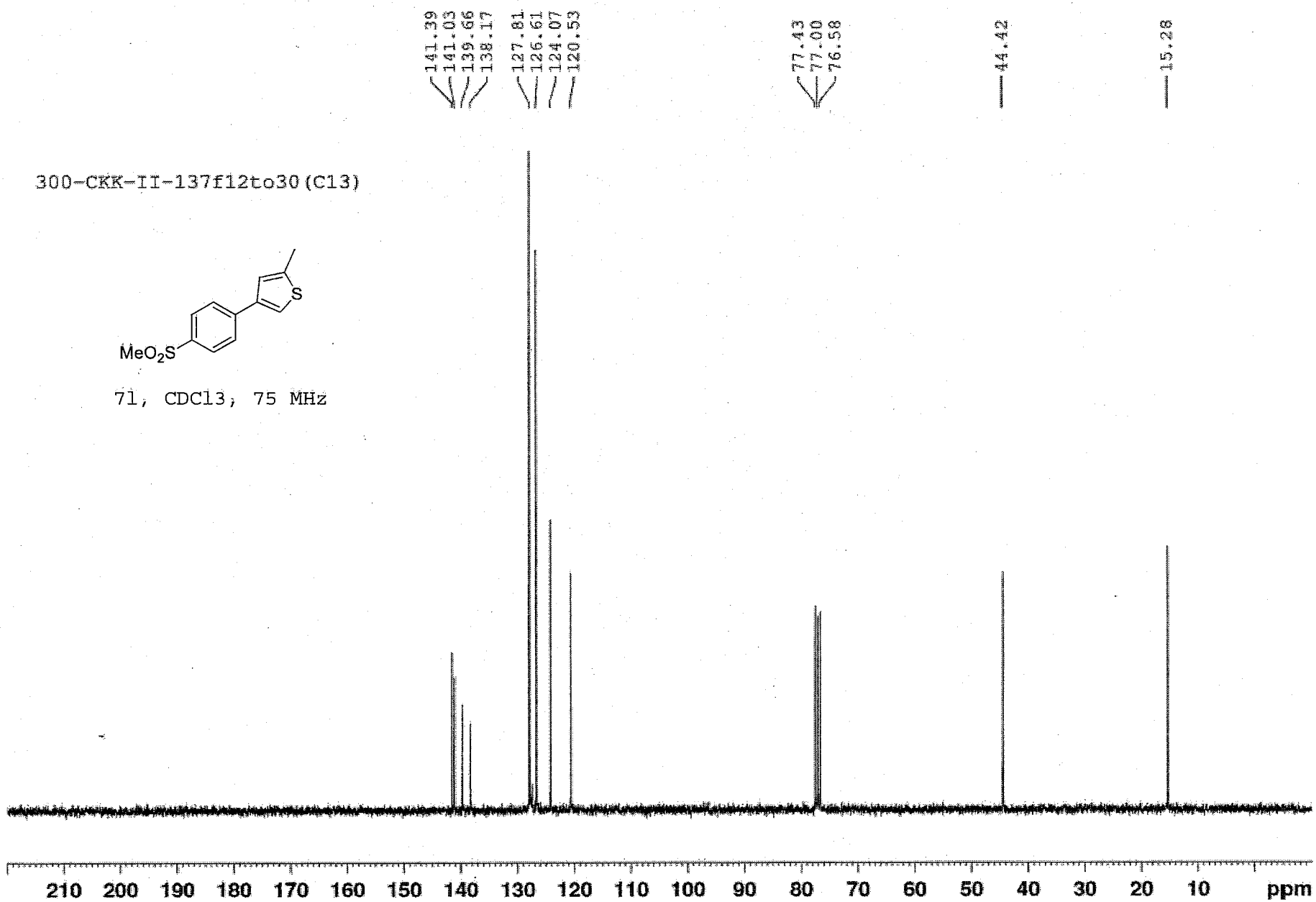

126.64
121.29
115.49
112.50
112.46
56.10
55.71
15.33
300-HYJ-II-157dept135
MeO
S
OMe
7j, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

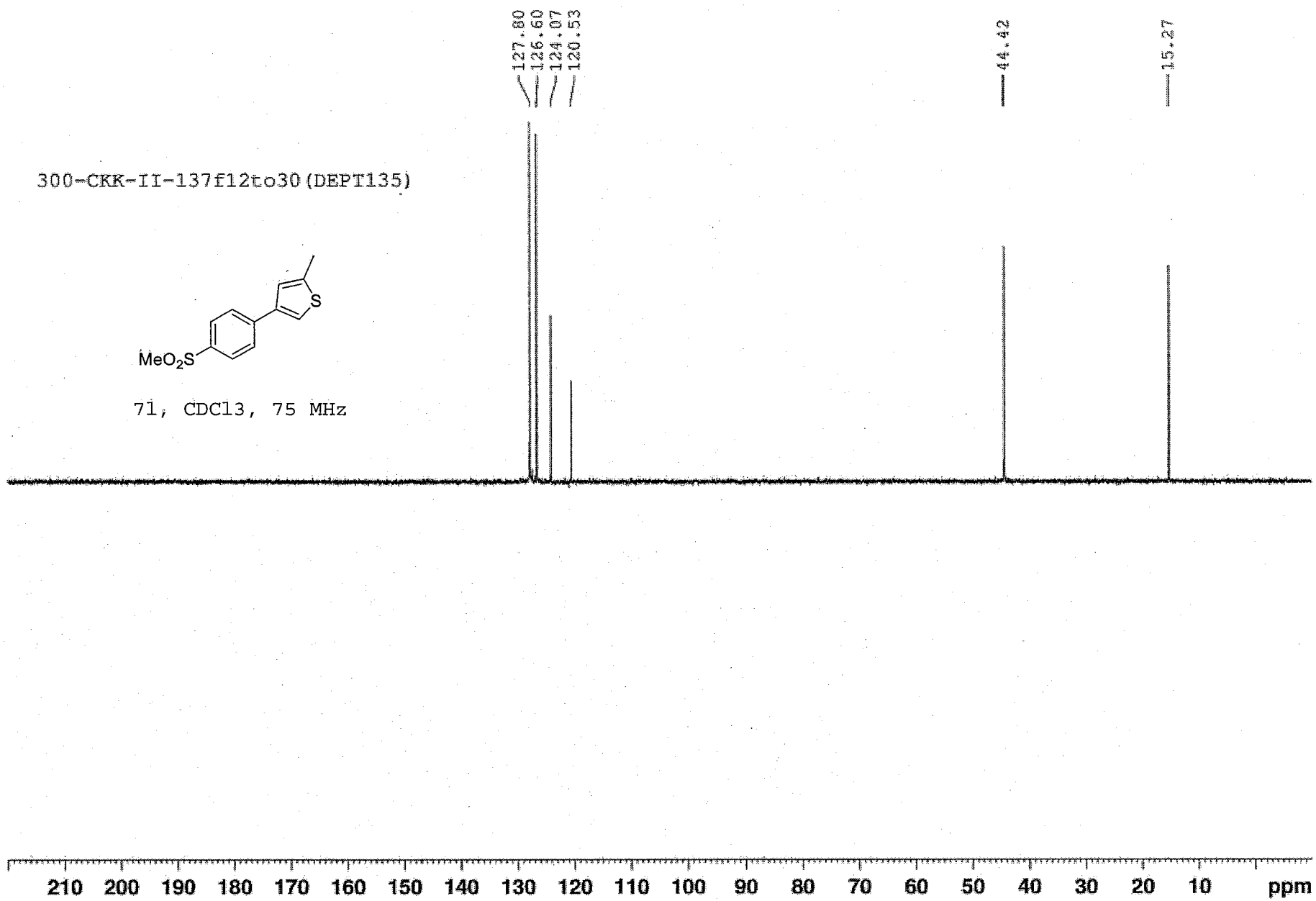

7.850
7.846
7.820
7.804
7.796
7.718
7.712
7.690
7.684
7.497
7.492
7.474
7.469
7.460
7.451
7.443
7.436
7.434
7.428
7.411
7.406
7.312
7.307
7.240
7.204
7.200
7.196
7.192
7.188
2.557
2.554
300-CKK-II-157purity
S
7k, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.01
3.10
1.04
2.15
0.98
1.00
3.12


141.96
140.63
133.73
133.47
132.52
128.31
127.99
127.63
126.25
125.65
124.89
124.71
124.49
118.41
77.42
77.00
76.57
15.44
300-CKK-II-157washed(C13)
S
7k, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


300-CKK-II-157washed(DEPT135)
7k, CDCl3, 300 MHz
128.31
127.99
127.63
126.25
125.65
124.90
124.71
124.49
118.41
15.43
S
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

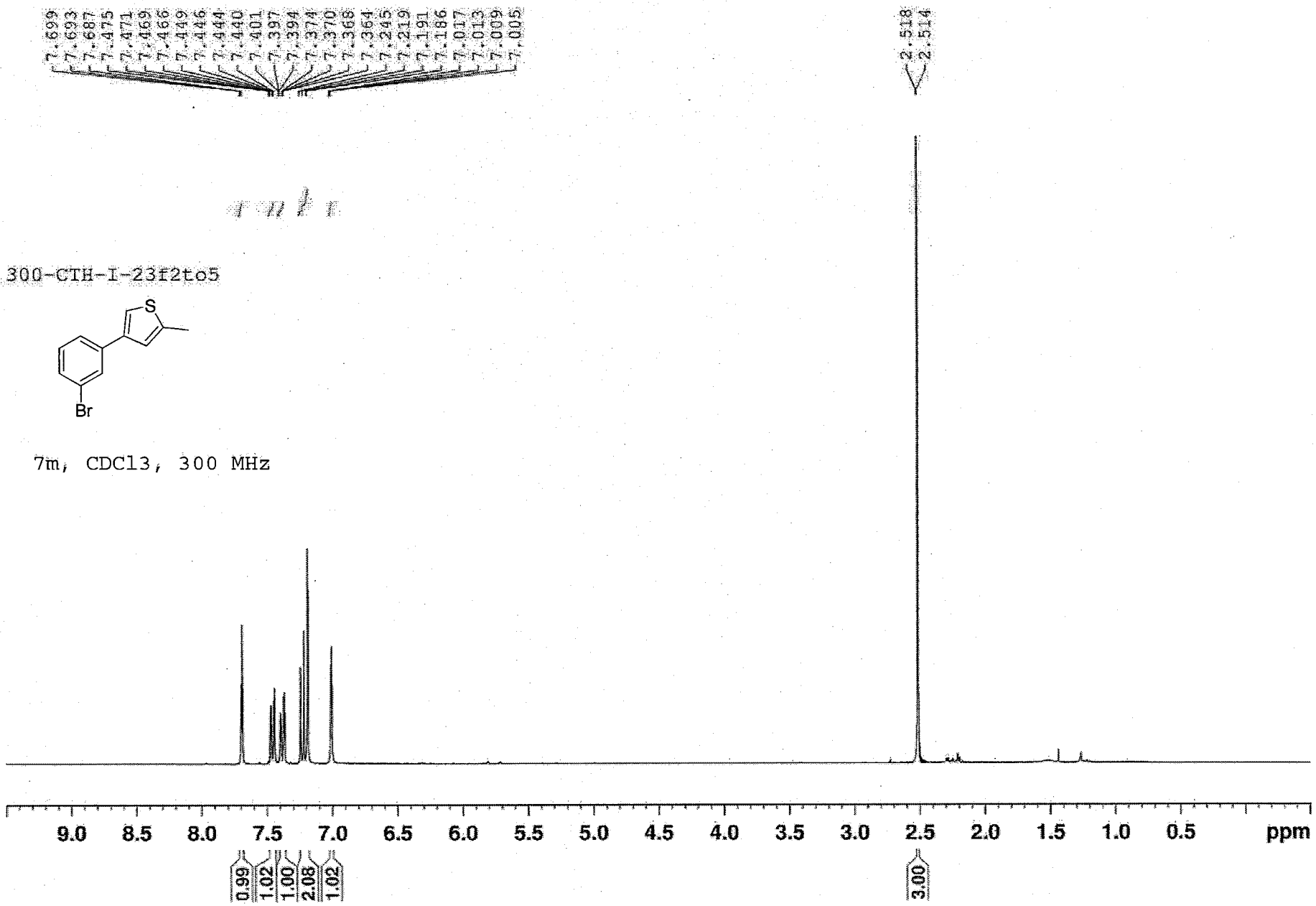

7.938
7.931
7.925
7.909
7.902
7.896
7.727
7.720
7.713
7.698
7.691
7.684
7.333
7.329
7.240
7.078
7.075
7.071
7.067
7.063
3.055
2.527
2.524
1.626
300-CKK-II-137f6to20(2)
S
MeO2S
7l, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.06
2.09
0.99
1.00
3.18
3.16


141.39
141.03
139.66
138.17
127.81
126.61
124.07
120.53
77.43
77.00
76.58
44.42
15.28
300-CKK-II-137f12to30(C13)
S
MeO2S
71, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


127.80
126.60
124.07
120.53
44.42
15.27
300-CKK-II-137f12to30(DEPT135)
S
MeO2S
7l, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

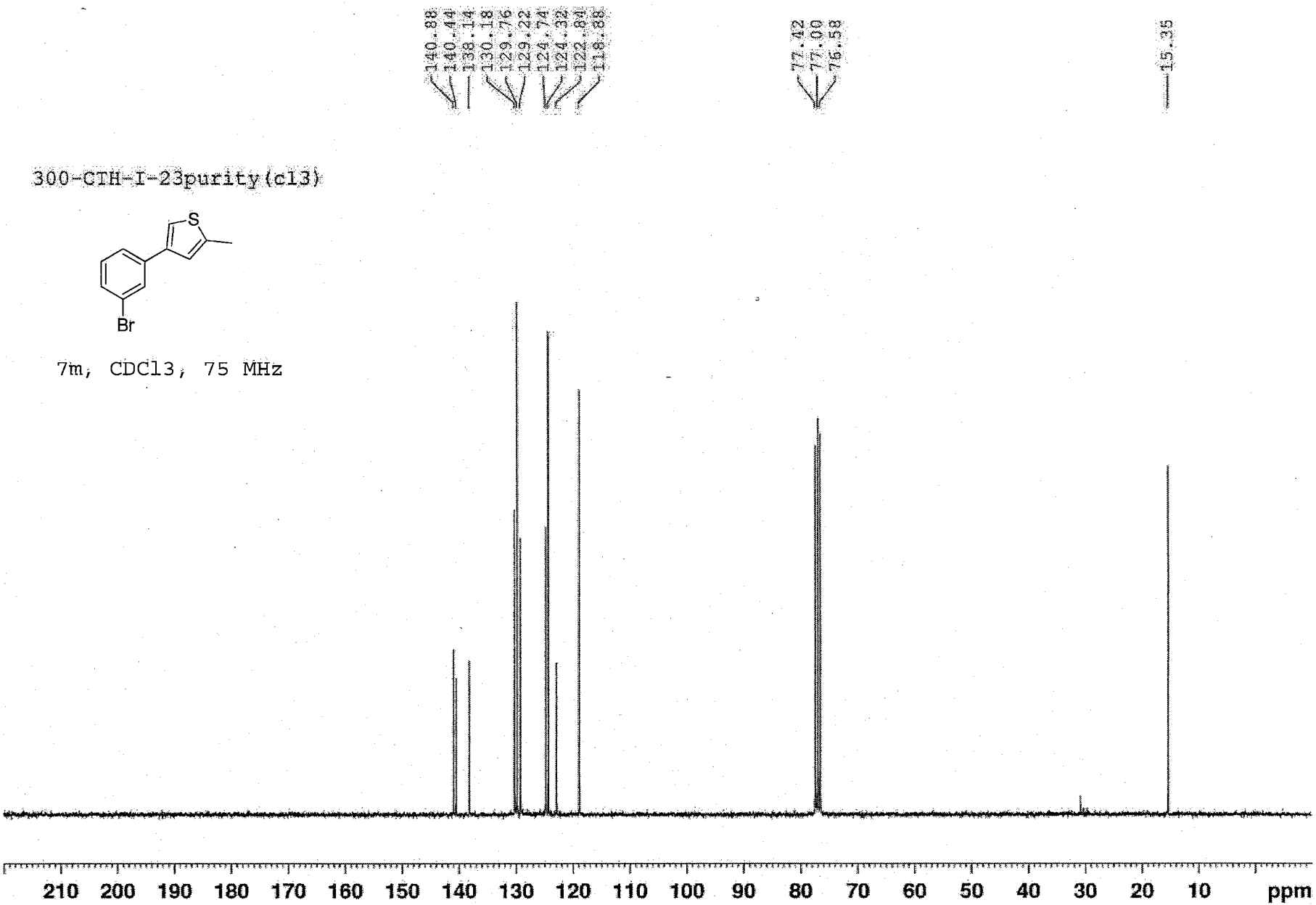
300-CTH-I-23f2to5

7m, CDCl3, 300 MHz



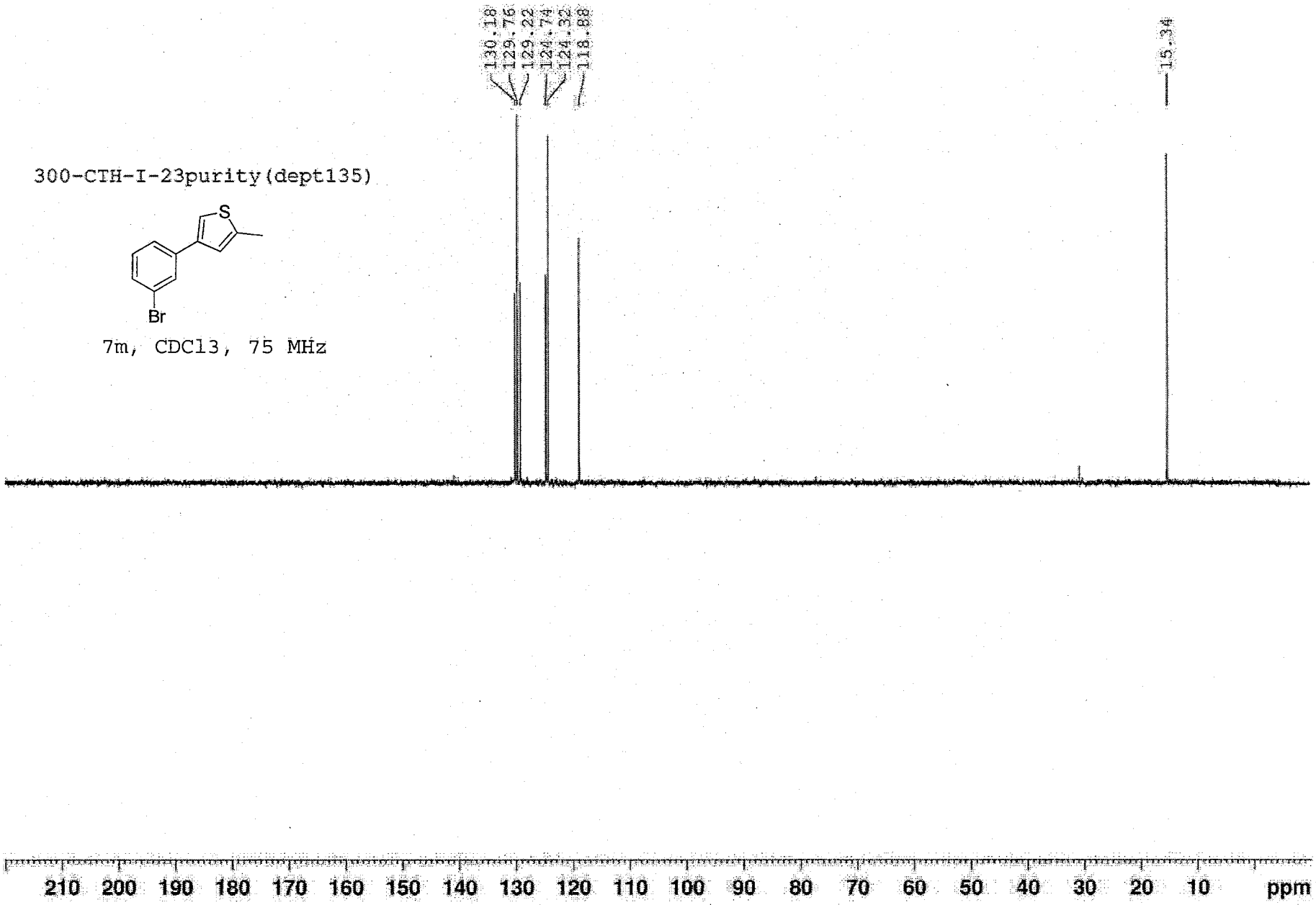
300-CTH-I-23purity(cl3)

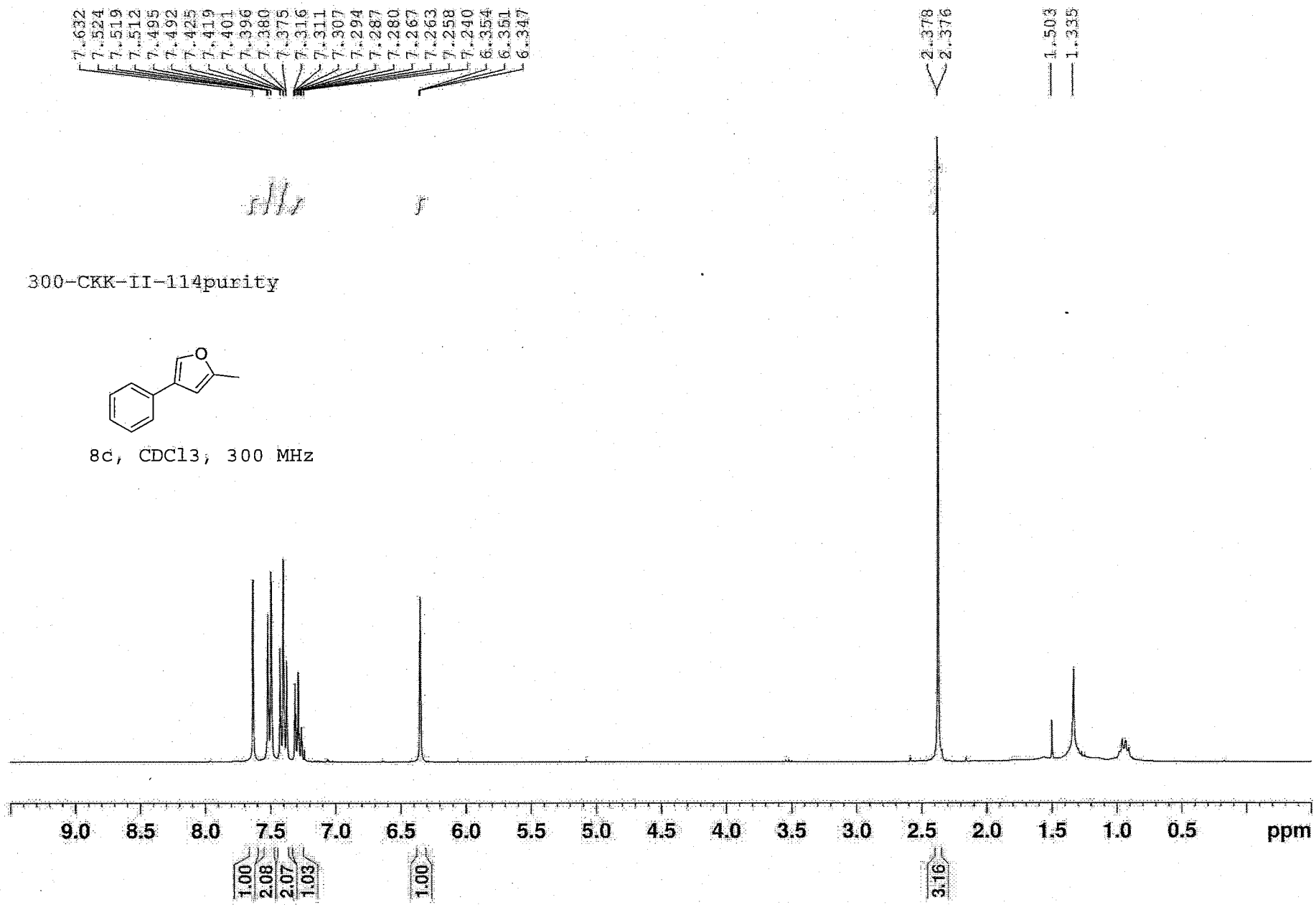
7m, CDCl3, 75 MHz

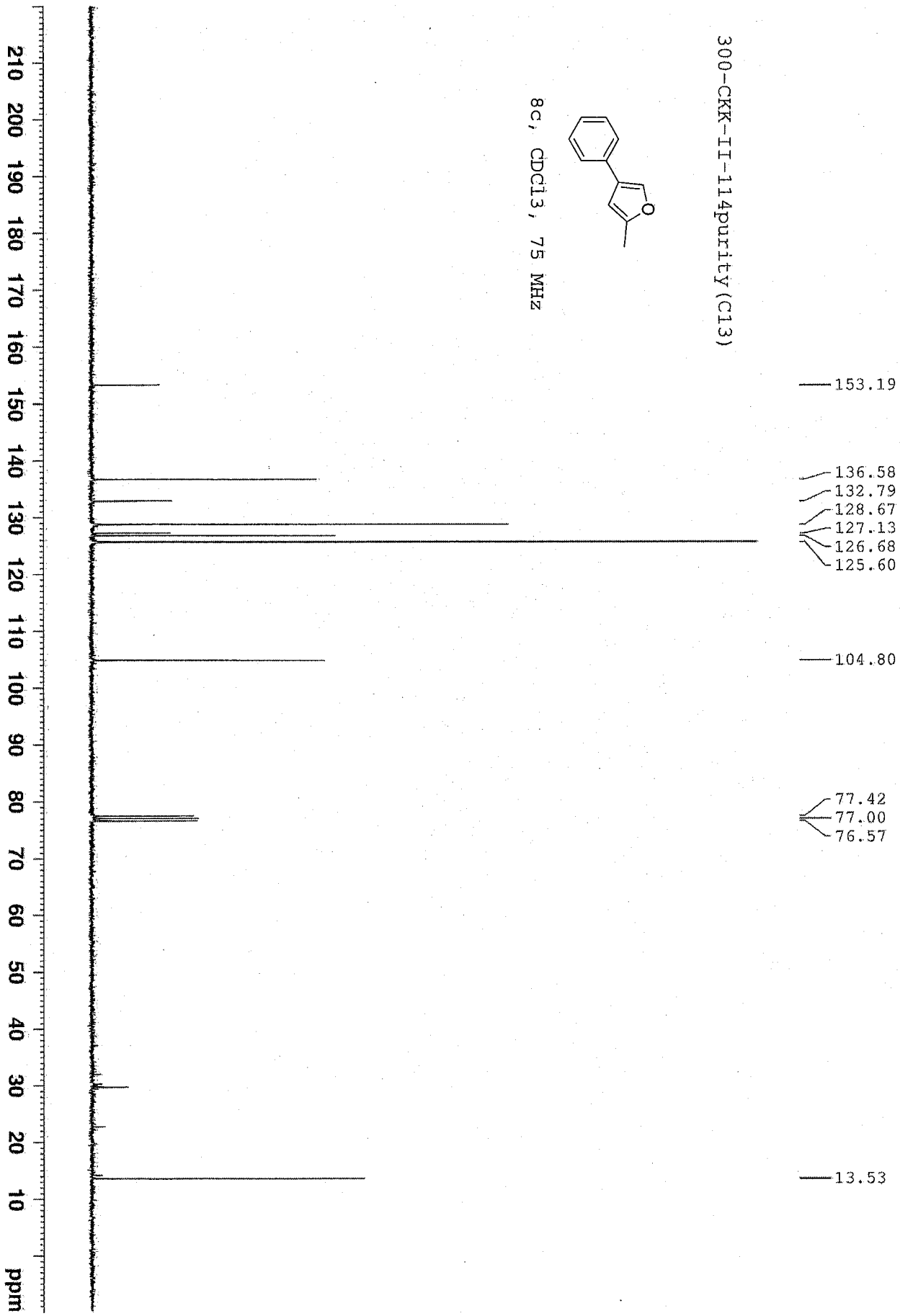


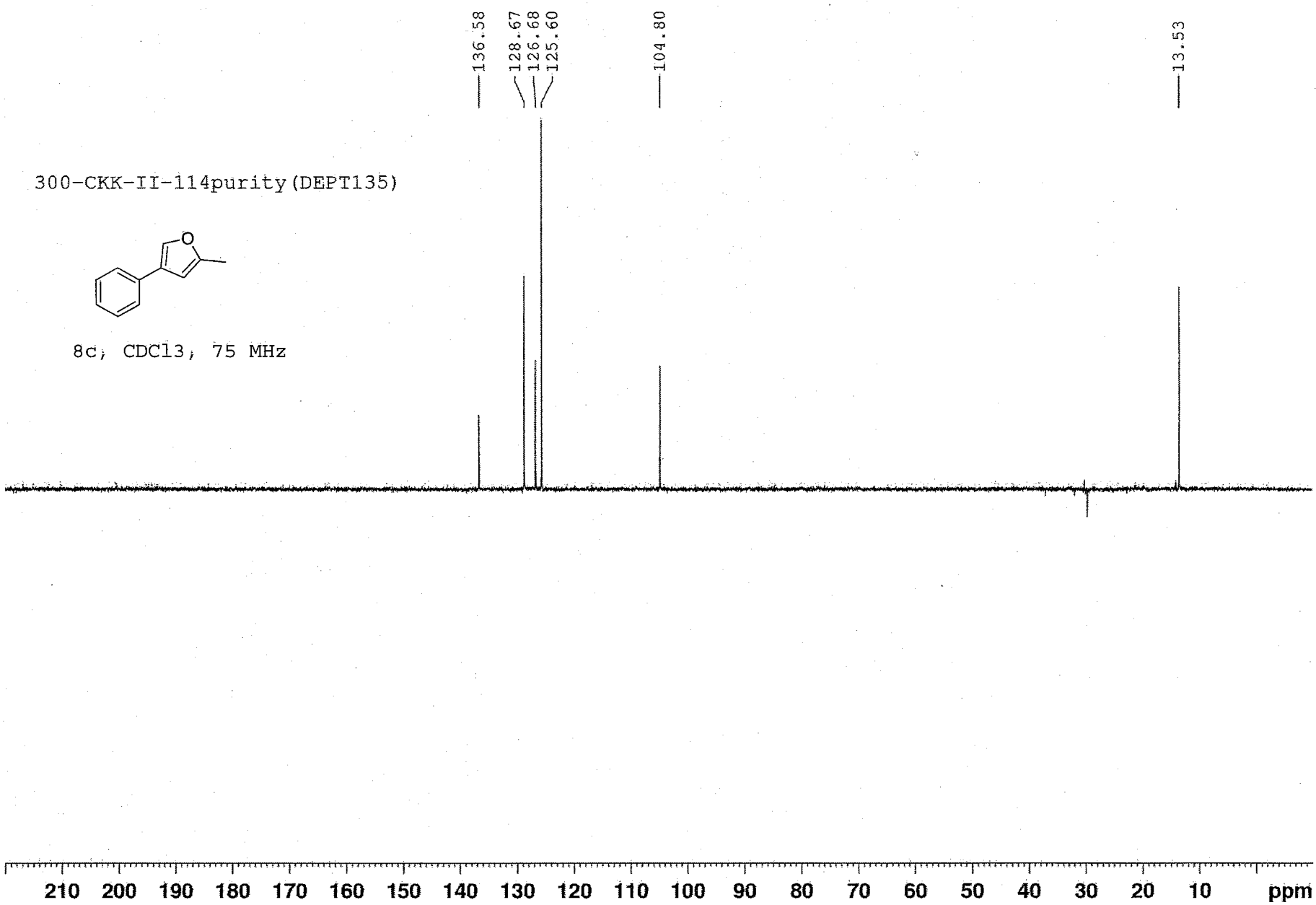
300-CTH-I-23purity(dept135)

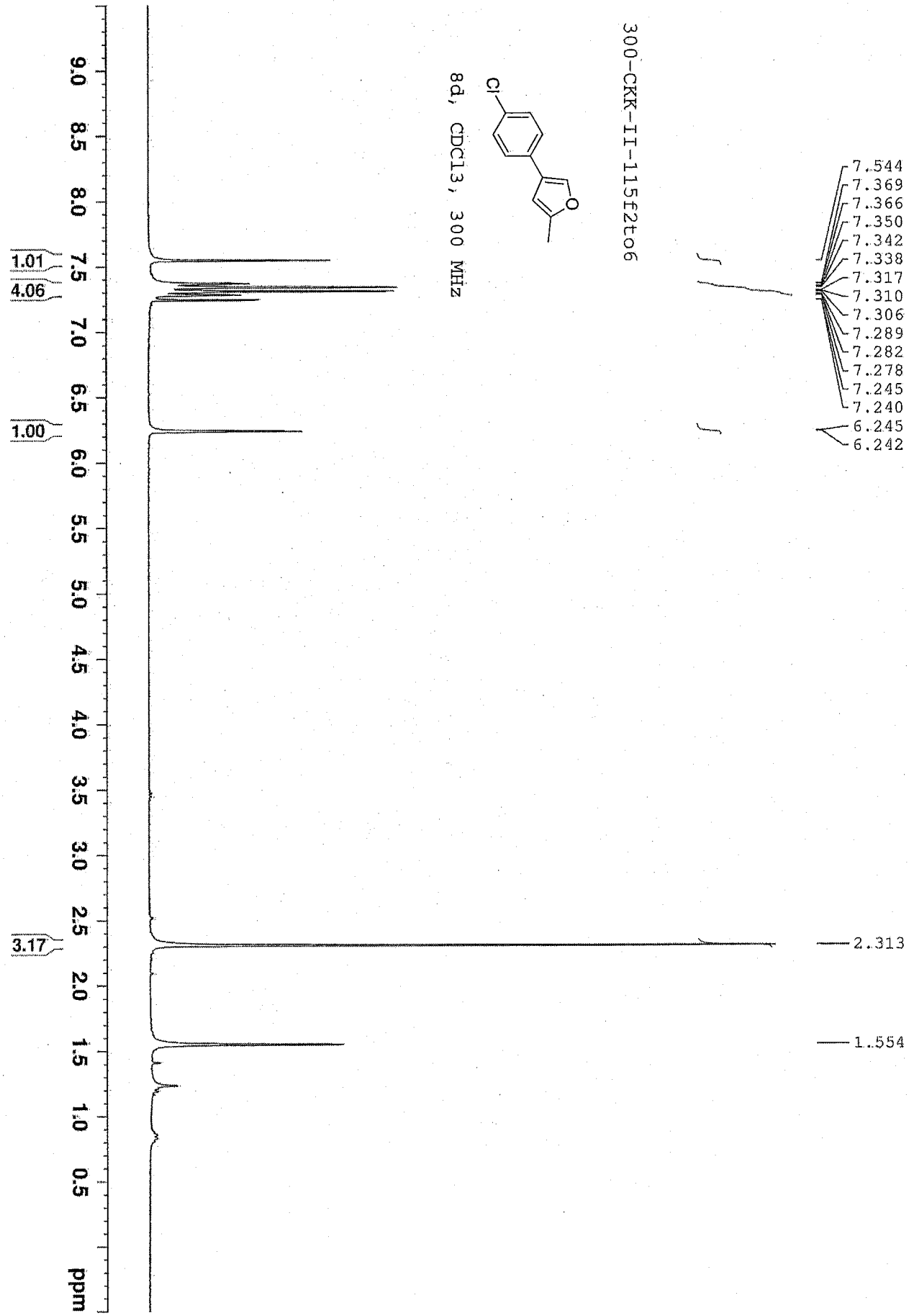
7m, CDCl3, 75 MHz

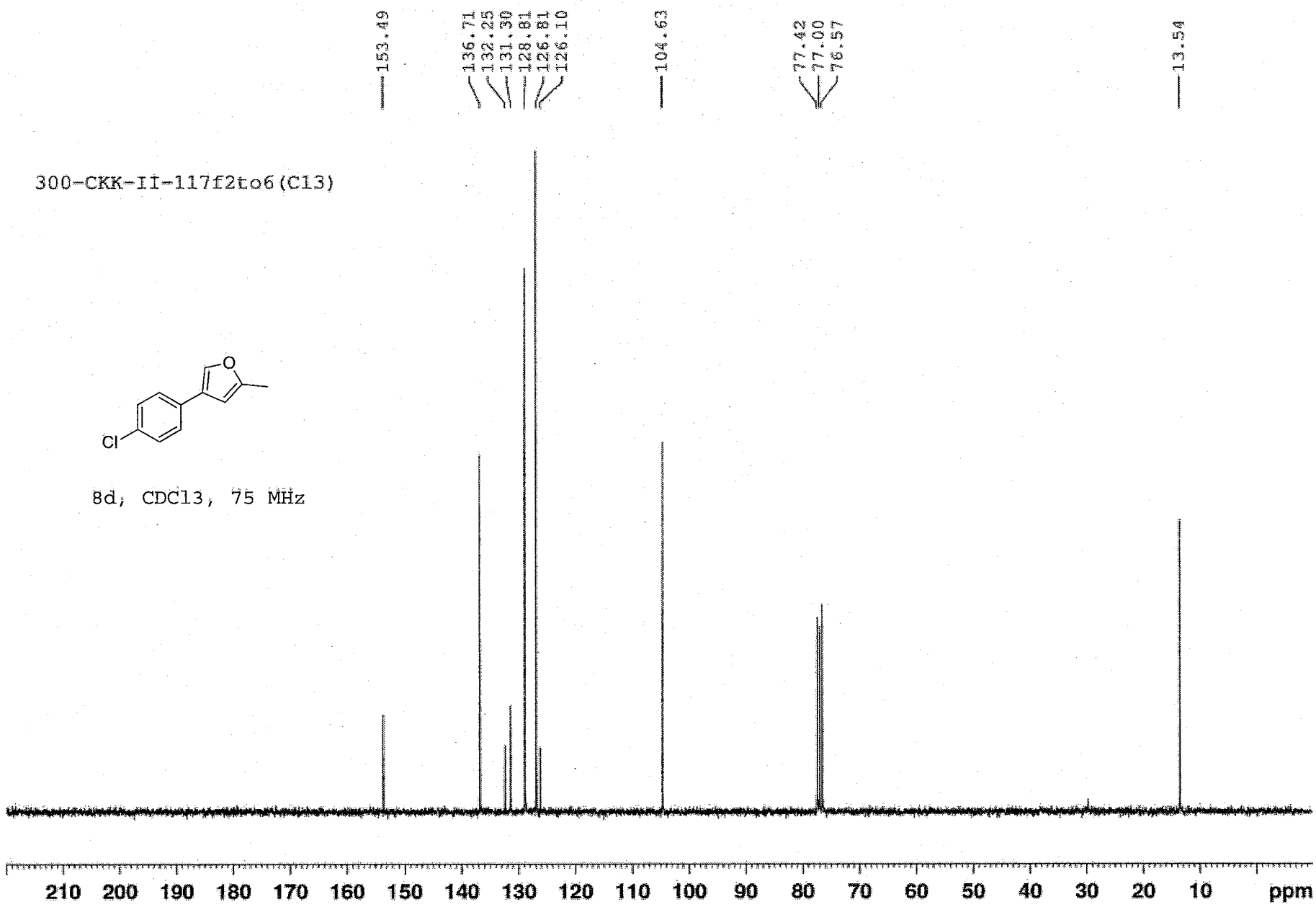


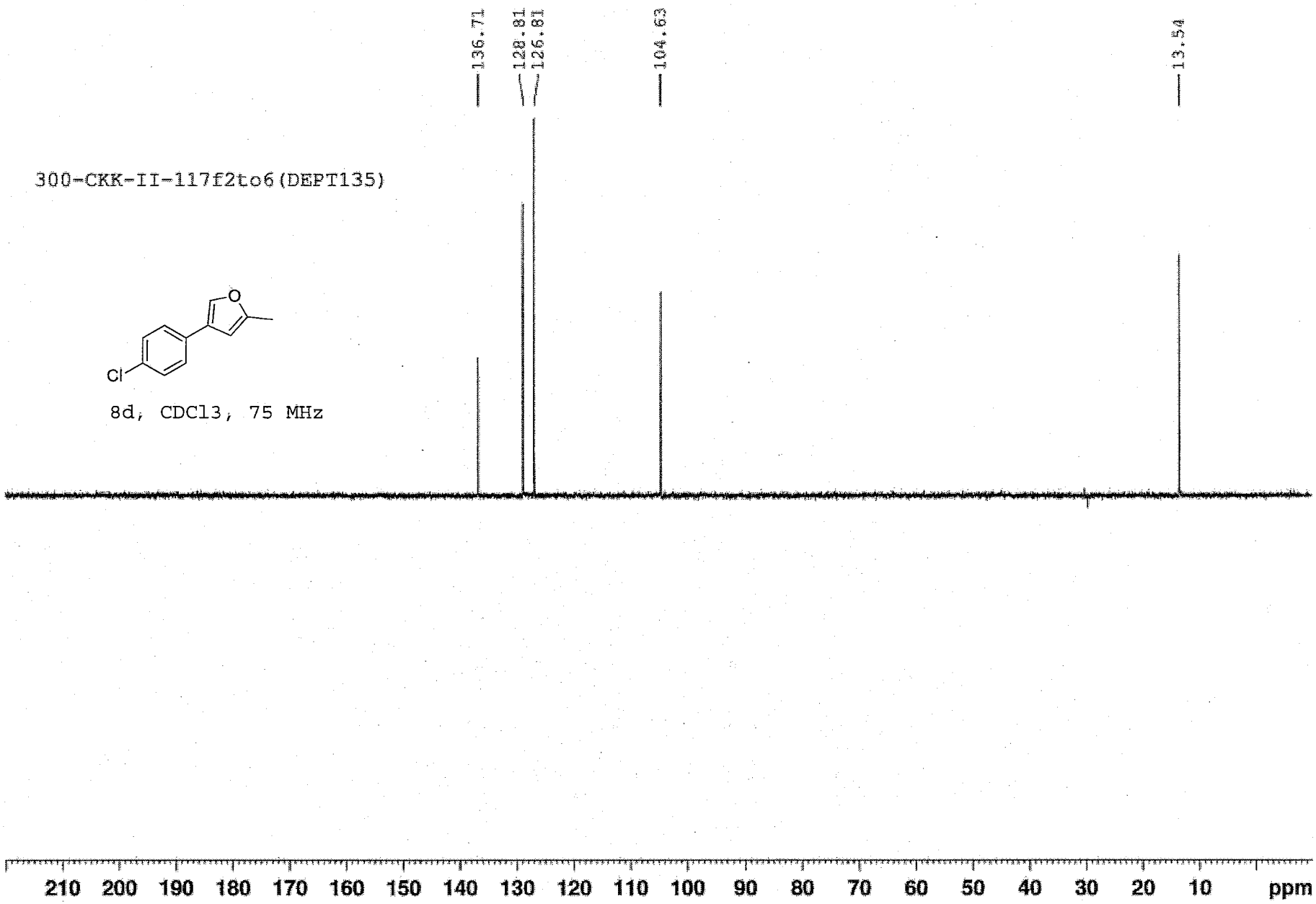

7.632
7.524
7.519
7.512
7.495
7.492
7.425
7.419
7.401
7.396
7.380
7.375
7.316
7.311
7.307
7.294
7.287
7.280
7.267
7.263
7.258
7.240
6.354
6.351
6.347
2.378
2.376
1.503
1.335
300-CKK-II-114purity
O
8c, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
2.08
2.07
1.03
1.00
3.16

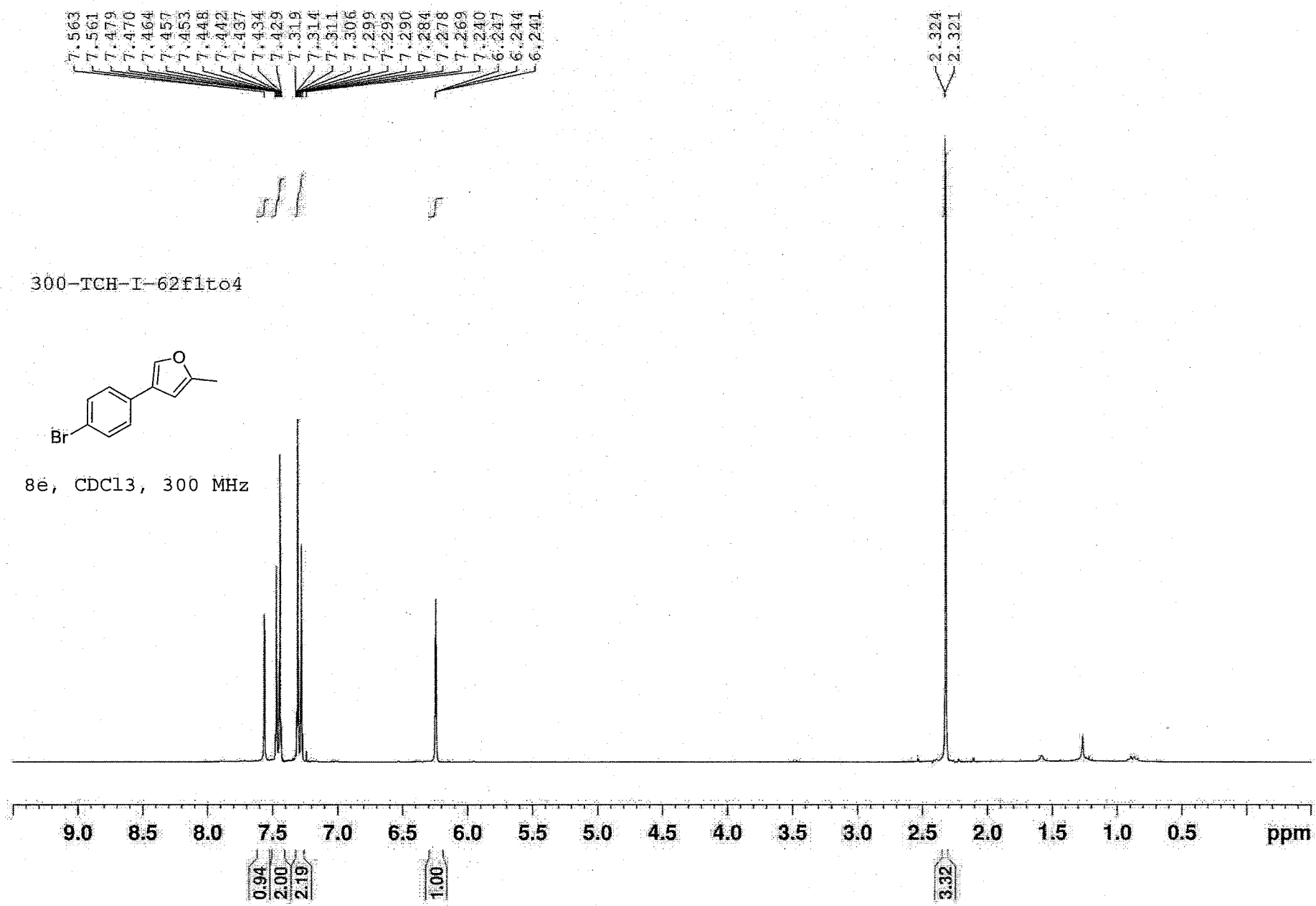

300-CKK-II-114purity(C13)
8c, CDCl3, 75 MHz
153.19
136.58
132.79
128.67
127.13
126.68
125.60
104.80
77.42
77.00
76.57
13.53
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

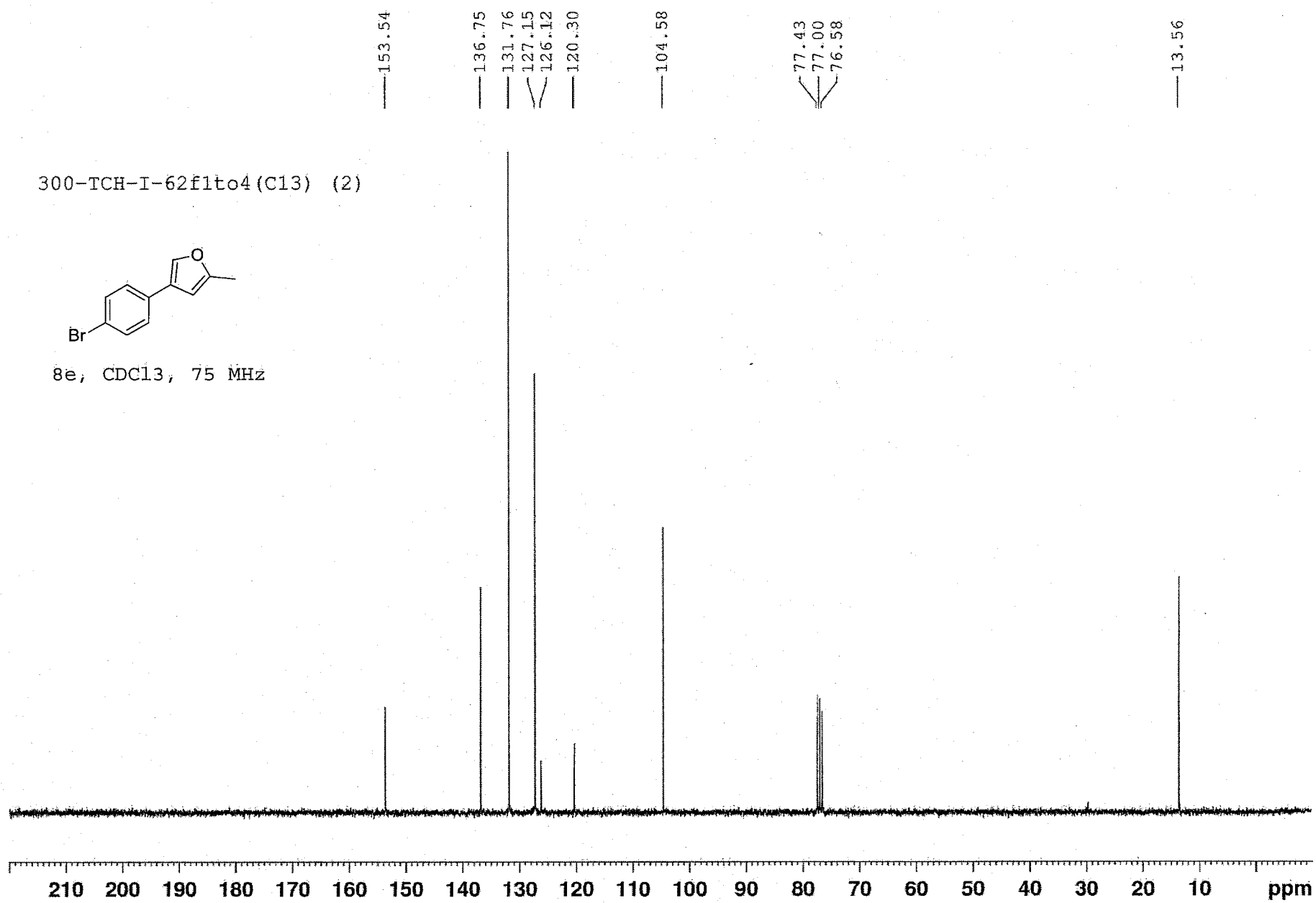

300-CKK-II-114purity(DEPT135)
8c, CDCl3, 75 MHz
136.58
128.67
126.68
125.60
104.80
13.53
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

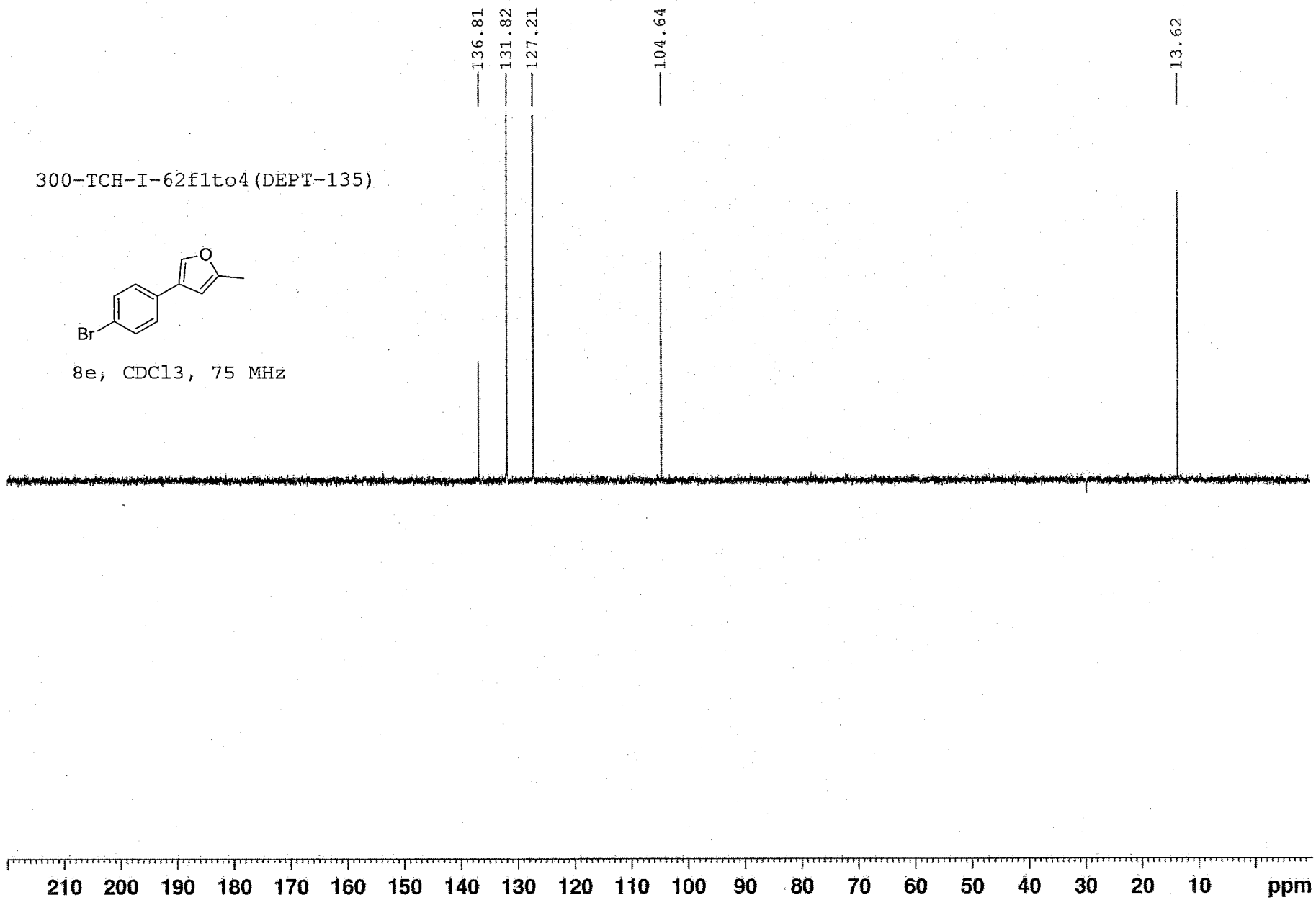

300-CKK-II-115f2to6
8d, CDCl3, 300 MHz
Cl
O
7.544
7.369
7.366
7.350
7.342
7.338
7.317
7.310
7.306
7.289
7.282
7.278
7.245
7.240
6.245
6.242
2.313
1.554
1.01
4.06
1.00
3.17
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

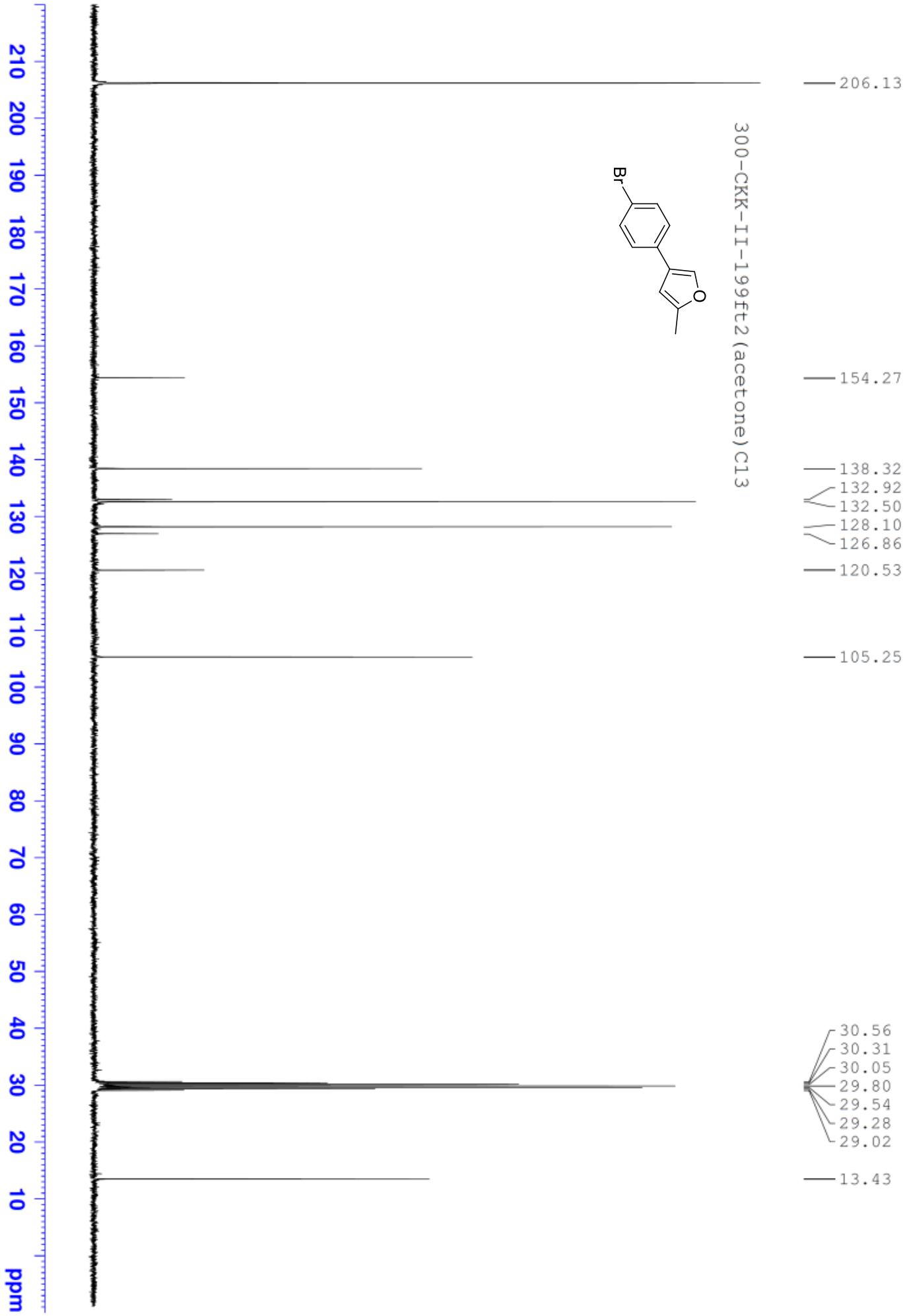

153.49
136.71
132.25
131.30
128.81
126.81
126.10
104.63
77.42
77.00
76.57
13.54
300-CKK-II-117f2to6(C13)
O
Cl
8d, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


136.71
128.81
126.81
104.63
13.54
300-CKK-II-117f2to6(DEPT135)
O
Cl
8d, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.563
7.561
7.479
7.470
7.464
7.457
7.453
7.448
7.442
7.437
7.434
7.429
7.319
7.314
7.311
7.306
7.299
7.292
7.290
7.284
7.278
7.269
7.240
6.247
6.244
6.241
2.324
2.321
300-TCH-I-62f1to4
O
Br
8e, CDCl3, 300 MHz
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.94
2.00
2.19
1.00
3.32


153.54
136.75
131.76
127.15
126.12
120.30
104.58
77.43
77.00
76.58
13.56
300-TCH-I-62f1to4(C13) (2)
O
Br
8e, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


136.81
131.82
127.21
104.64
13.62
300-TCH-I-62f1to4(DEPT-135)
O
Br
8e, CDCl3, 75 MHz
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


300-CKK-II-199ft2(acetone)C13
Br
O
206.13
154.27
138.32
132.92
132.50
128.10
126.86
120.53
105.25
30.56
30.31
30.05
29.80
29.54
29.28
29.02
13.43
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm