One-pot Synthesis of 1,3-Disubstituted Allenes from 1-Alkynes, Aldehydes, and Morpholine

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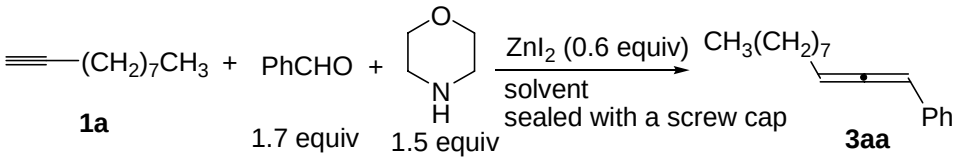
Supporting Information

Table of Contents

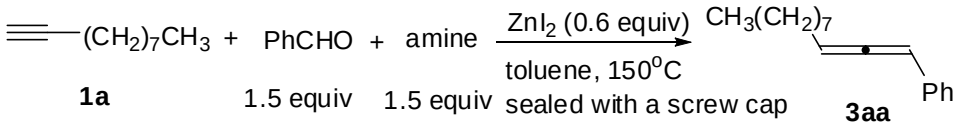
Table S1. Solvent effect on the ZnI_2-promoted one-pot synthesis of **3aa**[a]



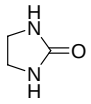
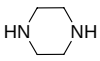
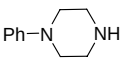
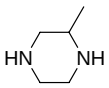
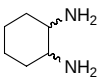
entry	solvent	temp (℃)	time (h)	NMR yield of **3aa** (%)
1	toluene	150	2.5	43
2	chlorobenzene	150	2.5	27
3	*o*-xylene	150	3	27
4	CH_3NO_2	150	6	-
5[b]	$CHCl_3$	90	12	-
6[b]	DCE	90	12	-
7	CH_3CN	120	22	-

[a] The reactions were carried out in 0.3 mmol scale in 0.9 mL of solvent. [b] The reaction proceeded at room temperature for 2 h, then at 60 ℃ for 2 h, and at 90 ℃ for 12 h.

Table S2. Amines screened for the ZnI_2 -promoted one-pot synthesis of **3aa**[a]

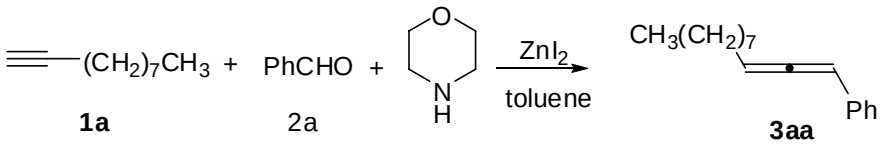


1a 1.5 equiv 1.5 equiv **3aa**

entry	amine	time (h)	NMR yield of **3aa** (%)
1		15	0
2		15	0
3		2	30
4		8.5	trace
5		8	0
6		7.7	trace
7		16	16
8		4.5	trace
9		2	36
10		8	0
11[b]	-	42	0

[a] The reactions were carried out in 0.3 mmol scale in 0.9 mL of toluene. [b] 1.8 equiv of PhCHO and 0.8 equiv of ZnI_2 were used.

Table S3. Optimization of reaction conditions for the ZnI_2-promoted one-pot synthesis of **3aa**[a]



1a 2a **3aa**

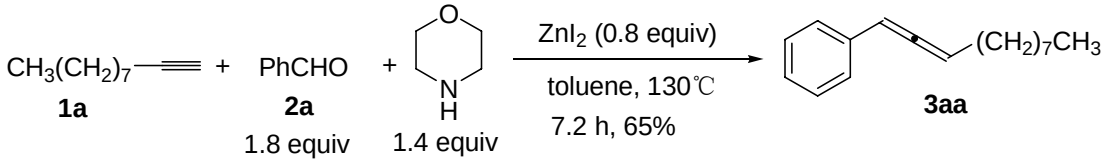
entry	PhCHO (equiv)	morpholine (equiv)	ZnI_2 (equiv)	temp. (℃)	time (h)	NMR yield of **3aa** (%)
1	1.7	1.5	0.6	150[b]	2.5	43
2	1.7	1.5	0.6	140	8	45
3	1.7	1.3	0.6	140	6	53
4	1.7	1.1	0.6	140	8	34
5	1.7	1.3	0.6	130	5	53
6	1.7	1.3	0.6	120	22	36
7	1.8	1.4	0.8	130	6.3	60

[a] The reactions were carried out in 0.3 mmol scale of **1a** in 0.9 mL of toluene. [b] The reaction was carried out in a reaction tube with a screw cap.

General. Liquid aromatic aldehydes were distilled before use. *i*-PrCHO and *n*-BuCHO were used as it is. ZnI_2 (98%) was purchased from Acros and used without further treatment. Toluene was dried over sodium wire with benzophenone as indicator and distilled freshly before use. Morpholine was dried over calcium hydride and distilled. All the temperatures are referred to the oil baths used.

1. General Procedure for the Synthesis of Allenes (3aa-3be and 5a-5c). To a dried reaction tube was added ZnI_2 (0.8 mmol). Then the reaction tube was dried under vacuum with a heating gun. Then aldehyde (1.8 mmol), alkyne (1.0 mmol), morpholine (1.4 mmol), and toluene (3 mL or 5 mL or 8 mL) were added sequentially into this dried reaction tube equipped with a reflux condenser under an argon atmosphere and the resulting mixture was stirred at 130 ℃. When the reaction was complete as monitored by TLC (or NMR), the reaction mixture was cooled to room temperature and then filtered. Evaporation and column chromatography on silica gel (eluent: petroleum ether or petroleum ether with ethyl acetate) afforded the corresponding 1,3-disubstituted allene.

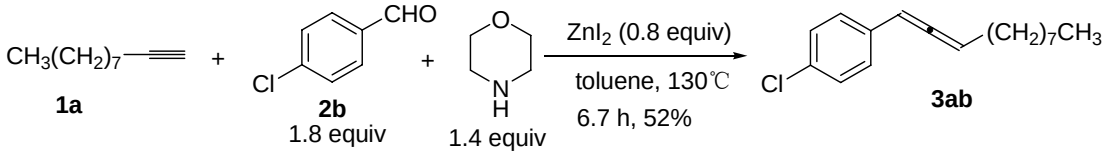
(1) Synthesis of 1-phenylundeca-1,2-diene (3aa)



The reaction of ZnI_2 (256 mg, 0.80 mmol), benzaldehyde (192 mg, 1.81 mmol), 1-decyne **1a** (139 mg, 1.00 mmol), and morpholine (122 mg, 1.40 mmol) in 5 mL of toluene afforded **3aa**[1] (148 mg, 65%) as a liquid (eluent: petroleum ether): ^{1}H NMR

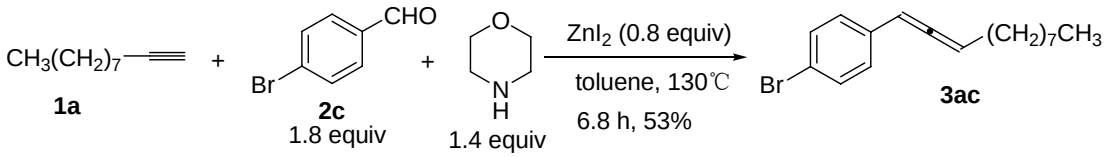
(300 MHz, $CDCl_3$) δ 7.38-7.27 (m, 4 H), 7.23-7.14 (m, 1 H), 6.15-6.07 (m, 1 H), 5.61-5.50 (m, 1 H), 2.20-2.05 (m, 2 H), 1.54-1.43 (m, 2 H), 1.42-1.20 (m, 10 H), 0.87 (t, *J* = 6.6 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.2, 135.2, 128.5, 126.6, 95.1, 94.5, 31.9, 29.4, 29.3, 29.19, 29.17, 28.8, 22.7, 14.1; MS (EI) *m/z* 228 (M^+, 4.81), 130 (100); IR (neat): 2959, 2926, 2853, 1949, 1599, 1494, 1460 cm^{-1}.

(2) Synthesis of 1-(4-chlorophenyl)undeca-1,2-diene (3ab)



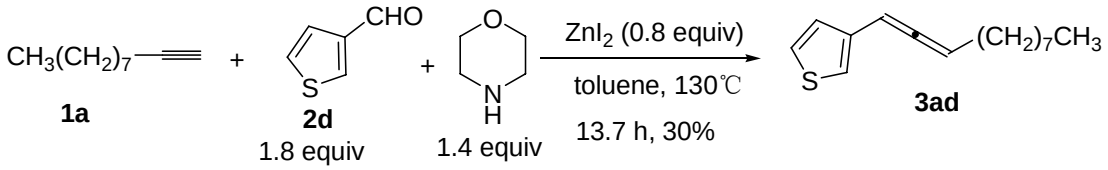
The reaction of ZnI_2 (255 mg, 0.80 mmol), 4-chlorobenzaldehyde (254 mg, 1.81 mmol), 1-decyne **1a** (136 mg, 0.99 mmol), and morpholine (122 mg, 1.40 mmol) in 3 mL of toluene afforded **3ab** (134 mg, 52%) as a liquid (eluent: petroleum ether): ^1H NMR (300 MHz, $CDCl_3$) δ 7.31-7.22 (m, 2 H), 7.22-7.15 (m, 2 H), 6.15-6.00 (m, 1 H) 5.63-5.50 (m, 1 H), 2.20-2.03 (m, 2 H), 1.53-1.41 (m, 2 H), 1.40-1.18 (m, 10 H), 0.88 (t, *J* = 6.3 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.2, 133.7, 132.1, 128.6, 127.7, 95.5, 93.7, 31.8, 29.4, 29.3, 29.2, 29.1, 28.6, 22.7, 14.1; MS (EI) m/z 264 (M^+(^{37}Cl), 0.19), 262 (M^+(^{35}Cl), 0.51), 129 (100); IR (neat): 2954, 2926, 2854, 1949, 1709, 1592, 1490, 1461, 1383, 1092, 1013 cm^{-1}; HRMS (EI) calcd for $C_{17}H_{23}{}^{35}Cl$ (M^+): 262.1488. Found: 262.1489.

(3) Synthesis of 1-(4-bromophenyl)undeca-1,2-diene (3ac)



The reaction of ZnI_2 (255 mg, 0.80 mmol), 4-bromobenzaldehyde (333 mg, 1.80 mmol), 1-decyne **1a** (138 mg, 1.00 mmol), and morpholine (122 mg, 1.40 mmol) in 3 mL of toluene afforded **3ac** (163 mg, 53%) as a liquid (eluent: petroleum ether): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.3 Hz, 2 H), 7.15 (d, *J* = 8.3 Hz, 2 H), 6.10-6.03 (m, 1 H), 5.62-5.52 (m, 1 H), 2.18-2.07 (m, 2 H), 1.53-1.41 (m, 2 H), 1.39-1.20 (m, 10 H), 0.88 (t, *J* = 6.3 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.3, 134.2, 131.6, 128.1, 120.2, 95.6, 93.8, 31.9, 29.4, 29.3, 29.2, 29.1, 28.6, 22.7, 14.1; MS (EI) *m/z* (%) 308 (M^+(^{81}Br), 0.39), 306 (M^+(^{79}Br), 0.39), 129 (100); IR (neat) 2957, 2925, 2854, 1949, 1487, 1461, 1383, 1070, 1009 cm^{-1}; Anal. calcd for $C_{17}H_{23}Br$: (%) C 66.45, H 7.54; Found: C 66.39, H 7.72; HRMS (EI) calcd for $C_{17}H_{23}{}^{81}Br$ (M^+): 308.0963. Found: 308.0963.

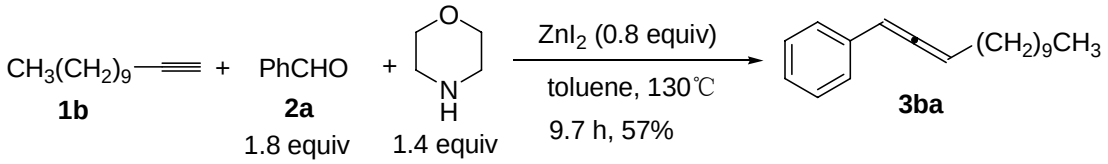
(4) Synthesis of 1-(3-thienyl)undeca-1,2-diene (3ad)



The reaction of ZnI_2 (256 mg, 0.80 mmol), thiophene-3-carbaldehyde (203 mg, 1.81 mmol), 1-decyne **1a** (138 mg, 1.00 mmol), and morpholine (122 mg, 1.40 mmol) in 3 mL of toluene afforded **3ad** (70 mg, 30%) as a liquid (eluent: petroleum ether): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26-7.16 (m, 1 H), 7.05 (d, *J* = 5.1 Hz, 1 H), 7.01 (s, 1 H), 6.25-6.13 (m, 1 H), 5.55-5.43 (m, 1 H), 2.28-2.03 (m, 2 H), 1.50-1.40 (m, 2 H), 1.38-1.20 (m, 10 H), 0.88 (t, *J* = 6.3 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.4, 136.6, 126.3, 125.6, 120.0, 94.2, 89.1, 31.9, 29.4, 29.3, 29.2, 29.1, 28.8, 22.7, 14.1;

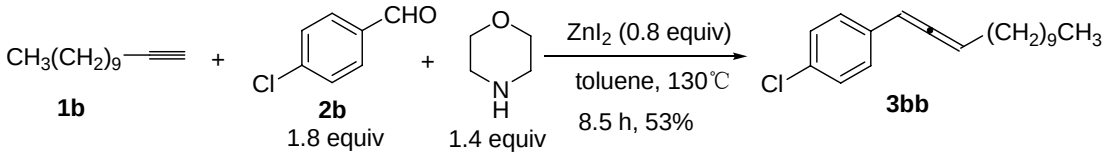
MS (EI) *m/z* (%) 234 (M^+, 2.00), 135 (100); IR (neat) 3104, 2955, 2925, 2854, 1951, 1465, 1378, 1264, 1233, 1078 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{22}S(M^+)$: 234.1442. Found: 234.1443.

(5) Synthesis of 1-phenyltrideca-1,2-diene (3ba)



The reaction of ZnI_2 (256 mg, 0.80 mmol), benzaldehyde (191 mg, 1.80 mmol), 1-dodecyne **1b** (167 mg, 1.01 mmol), and morpholine (123 mg, 1.41 mmol) in 5 mL of toluene afforded **3ba**[2] (148 mg, 57%) as a liquid (eluent: petroleum ether): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.27 (m, 4 H), 7.25-7.14 (m, 1 H), 6.20-6.08 (m, 1 H), 5.63-5.50 (m, 1 H), 2.22-2.07 (m, 2 H), 1.53-1.43 (m, 2 H), 1.40-1.20 (m, 14 H), 0.89 (t, *J* = 6.6 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.1, 135.2, 128.5, 126.5, 95.1, 94.5, 31.9, 29.63, 29.59, 29.4, 29.3, 29.19, 29.16, 28.8, 22.7, 14.1; MS (EI) *m/z* (%) 256 (M^+, 4.34), 130 (100); IR (neat) 3062, 3031, 2954, 2925, 2853, 1949, 1599, 1495, 1461 cm^{-1}.

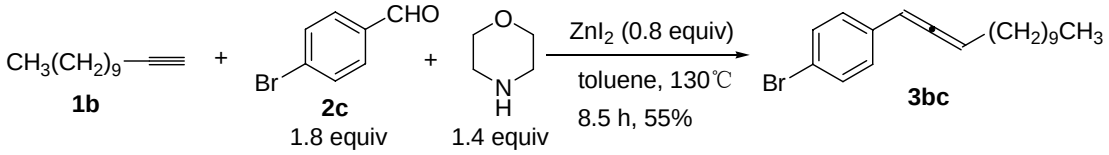
(6) Synthesis of 1-(4-chlorophenyl)trideca-1,2-diene (3bb)



The reaction of ZnI_2 (256 mg, 0.80 mmol), 4-chlorobenzaldehyde (253 mg, 1.80 mmol), 1-dodecyne **1b** (165 mg, 0.99 mmol), and morpholine (123 mg, 1.41 mmol) in 3 mL of toluene afforded **3bb** (152 mg, 53%) as a liquid (eluent: petroleum ether): ^{1}H

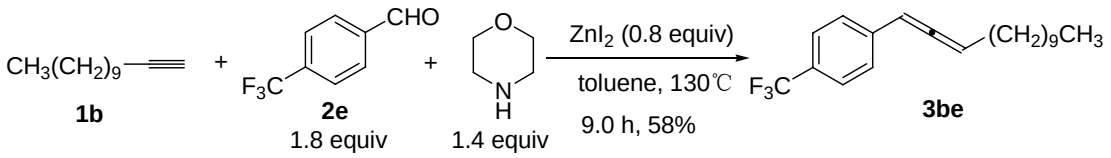
NMR (300 MHz, $CDCl_3$) δ 7.29-7.22 (m, 2 H), 7.22-7.16 (m, 2 H), 6.11-6.03 (m, 1 H) 5.63-5.50 (m, 1 H), 2.20-2.05 (m, 2 H), 1.52-1.40 (m, 2H), 1.38-1.20 (m, 14 H), 0.88 (t, *J* = 6.5 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.2, 133.7, 132.1, 128.6, 127.7, 95.5, 93.7, 31.9, 29.63, 29.59, 29.4, 29.3, 29.2, 29.1, 28.6, 22.7, 14.1; MS (EI) *m/z* 292 ($M^+(^{37}Cl)$, 0.27), 290 ($M^+(^{35}Cl)$, 0.77), 129 (100); IR (neat) 2955, 2925, 2854, 1950, 1491, 1465, 1092, 1013 cm^{-1}; HRMS (EI) calcd for $C_{19}H_{27}{}^{35}Cl$ (M^+): 290.1801. Found: 290.1798.

(7) Synthesis of 1-(4-bromophenyl)trideca-1,2-diene (3bc)



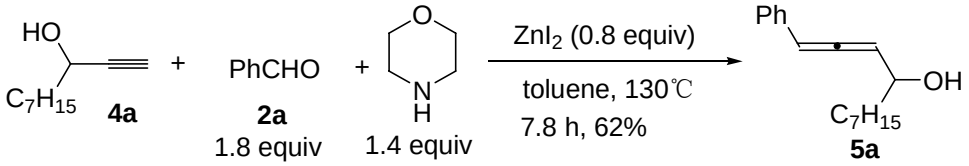
The reaction of ZnI_2 (255 mg, 0.80 mmol), 1-dodecyne **1b** (165 mg, 0.99 mmol), 4-bromobenzaldehyde (332 mg, 1.79 mmol), and morpholine (121 mg, 1.39 mmol) in 3 mL of toluene afforded **3bc** (183 mg, 55%) as a liquid (eluent: petroleum ether): ^1H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.4 Hz, 2 H), 7.15 (d, *J* = 8.4 Hz, 2 H), 6.12-6.02 (m, 1 H), 5.62-5.50 (m, 1 H), 2.20-2.05 (m, 2 H), 1.54-1.42 (m, 2 H), 1.40-1.20 (m, 14 H), 0.90 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.2, 134.2, 131.6, 128.1, 120.1, 95.6, 93.8, 31.9, 29.63, 29.59, 29.4, 29.3, 29.2, 29.1, 28.6, 22.7, 14.1; MS (EI) *m/z* (%) 336 ($M^+(^{81}Br)$, 0.96), 334 ($M^+(^{79}Br)$, 1.01), 129 (100); IR (neat) 2930, 2853, 1949, 1487, 1465, 1385, 1070, 1010 cm^{-1}; HRMS (EI) calcd for $C_{19}H_{27}{}^{79}Br$ (M^+): 334.1296. Found: 334.1296.

(8) Synthesis of 1-(4-trifluoromethylphenyl)trideca-1,2-diene (3be)



The reaction of ZnI_2 (256 mg, 0.80 mmol), 4-bromobenzaldehyde (311 mg, 1.79 mmol), 1-dodecyne **1b** (165 mg, 0.99 mmol), and morpholine (122 mg, 1.40 mmol) in 3 mL of toluene afforded **3be** (187 mg, 58%) as a liquid (eluent: petroleum ether): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.1 Hz, 2 H), 7.37 (d, *J* = 8.1 Hz, 2 H), 6.20-6.10 (m, 1 H) 5.69-5.58 (m, 1 H), 2.23-2.07 (m, 2 H), 1.55-1.42 (m, 2 H), 1.41-1.18 (m, 14 H), 0.88 (t, *J* = 6.6 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.1, 139.1, 128.4 (q, *J* = 32.0 Hz), 126.6, 125.4 (q, *J* = 3.75 Hz) 124.3 (q, *J* = 270.0 Hz) 95.7, 93.8, 31.9, 29.62, 29.58, 29.4, 29.3, 29.2, 29.0, 28.5, 22.7, 14.1; MS (EI) *m/z* (%) 324 (M^+, 4.24), 129 (100); IR (neat) 2927, 2856, 1949, 1616, 1461, 1325, 1169, 1122, 1067, 1017 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{27}F_3$ (M^+): 324.2065. Found: 324.2061.

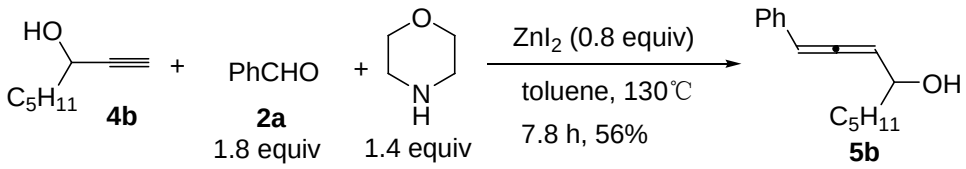
(9) Synthesis of 1-phenylundeca-1,2-dien-4-ol (5a)



The reaction of ZnI_2 (257 mg, 0.80 mmol), benzaldehyde (191 mg, 1.80 mmol), dec-1-yn-3-ol **4a** (154 mg, 1.00 mmol), and morpholine (122 mg, 1.40 mmol) in 8 mL of toluene afforded **5a** (152 mg, 62%) as a liquid as a pair of diastereoisomer (eluent: petroleum ether/ethyl acetate = 20:1, then 10:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.24 (m, 4 H), 7.24-7.14 (m, 1 H), 6.35-6.25 (m, 1 H), 5.75-5.60 (m, 1 H), 4.35-4.20 (m, 1 H), 2.04 (brs, 1H), 1.72-1.55 (m, 2 H), 1.53-1.38 (m, 2 H), 1.38-1.20

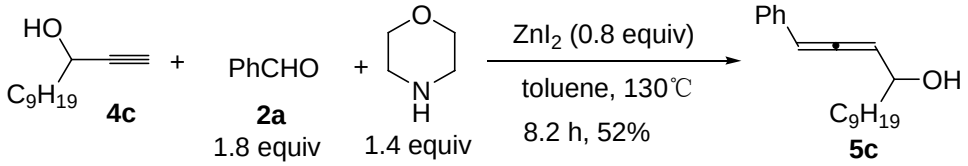
(m, 8 H), 0.88 (t, *J* = 6.2 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ [203.6, 203.4], [133.92, 133.88], 128.6, [127.09, 127.07], 126.7, [99.7, 99.5], [97.2, 96.8], [70.3, 69.8], [37.6, 37.5], 31.7, [29.41, 29.38], [29.23, 29.21], [25.5, 25.4], 22.6, 14.0; MS (EI) *m/z* (%) 244 (M^+, 1.07), 116 (100); IR (neat) 3364, 3031, 2956, 2929, 2854, 1950, 1705, 1600, 1494, 1460, 1126, 1069 cm^{-1}; HRMS (EI) calcd for $C_{17}H_{24}O$ (M^+): 244.1827. Found: 244.1828.

(10) Synthesis of 1-phenylnona-1,2-dien-4-ol (5b)



The reaction of ZnI_2 (255 mg, 0.80 mmol), benzaldehyde (191 mg, 1.80 mmol), dec-1-yn-3-ol **4b** (126 mg, 1.00 mmol), and morpholine (122 mg, 1.40 mmol) in 8 mL of toluene afforded **5b** (121 mg, 56%) as a liquid as a pair of diastereoisomer (eluent: petroleum ether/ethyl acetate = 20:1, then 10:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.27 (m, 4 H), 7.25-7.17 (m, 1 H), 6.35-6.25 (m, 1 H), 5.75-5.60 (m, 1 H), 4.35-4.20 (m, 1 H), 1.74 (brs, 1 H), 1.70-1.60 (m, 2 H), 1.53-1.38 (m, 2 H), 1.38-1.10 (m, 4 H), 0.89 (t, *J* = 6.3 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ [203.6, 203.4], [133.91, 133.86], 128.6, [127.1, 127.0], [127.67, 127.66], [99.7, 99.5], [97.2, 96.8], [70.3, 69.8], [37.5, 37.4], [31.62, 31.58], [25.1, 25.0], [22.57, 22.55], 13.9; MS (EI) *m/z* (%) 216 (M^+, 2.12), 116 (100); IR (neat) 3364, 2957, 2930, 2859, 1950, 1653, 1599, 1495, 1459, 1051, 1028 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{20}O$ (M^+): 216.1514. Found: 216.1514.

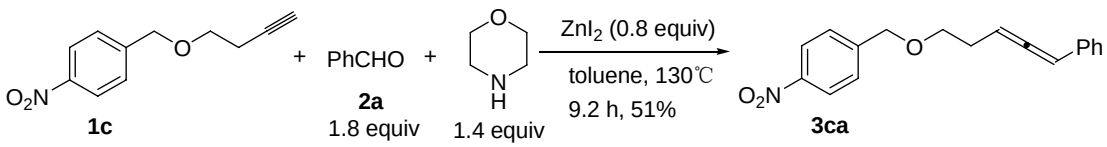
(11) Synthesis of 1-phenyltrideca-1,2-dien-4-ol (5c)



The reaction of ZnI_2 (256 mg, 0.80 mmol), benzaldehyde (192 mg, 1.81 mmol), dodec-1-yn-3-ol **4c** (181 mg, 0.99 mmol), and morpholine (122 mg, 1.40 mmol) in 8 mL of toluene afforded **5c** (142 mg, 52%) as a liquid (eluent: petroleum ether/ethyl acetate = 20:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.22 (m, 4 H), 7.22-7.13 (m, 1 H), 6.31-6.20 (m, 1 H), 5.72-5.58 (m, 1 H), 4.30-4.15 (m, 1 H), 2.23 (brs, 1 H), 1.70-1.55 (m, 2 H), 1.53-1.37 (m, 2 H), 1.35-1.18 (m, 12 H), 0.88 (t, *J* = 6.5 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ [203.6, 203.4], [133.92, 133.87], 128.5, [127.03, 127.01], 126.7, [99.7, 99.5], [97.1, 96.8], [70.3, 69.8], [37.6, 37.5], 31.8, [29.6, 29.54], 29.46, 29.42, 29.3, [25.5, 25.4], 22.6, 14.0; MS (EI) *m/z* (%) 272 (M^+, 1.01), 116 (100); IR (neat) 3385, 3062, 3031, 2925, 2853, 1950, 1695, 1600, 1494, 1460, 1127, 1069, 1007 cm^{-1}; HRMS (EI) calcd for $C_{19}H_{28}O$ (M^+): 272.2140. Found: 272.2138.

2. Allenes **3ca**, **3df**, **3dg**, **7a** and **7f** were prepared according to the following procedures.

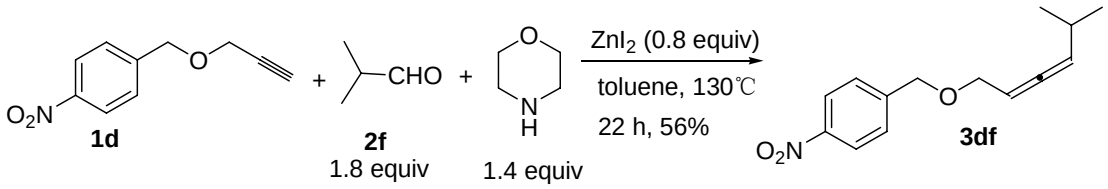
(1) Synthesis of 1-phenyl-5-(4-nitrobenzyloxy))-1,2-pentadiene (3ca)



To a dried reaction tube was added ZnI_2 (256 mg, 0.80 mmol). This reaction tube was then dried under vacuum with a heating gun. Then

1-((but-3-ynyloxy)methyl)-4-nitrobenzene **1c** (206 mg, 1.00 mmol), benzaldehyde (190 mg, 1.79 mmol), morpholine (123 mg, 1.41 mmol) and toluene (5 mL) were added sequentially into a dried reaction tube equipped with a reflux condenser under an argon atmosphere and the resulting mixture was stirred at 130 ℃. When the reaction was complete as monitored by TLC, the reaction mixture was cooled to room temperature and then filtered. Evaporation and column chromatography on silica gel afforded **3ca** (152 mg, 51%) as a liquid (eluent: petroleum ether/ethyl acetate=20:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.7 Hz, 2 H), 7.45 (d, *J* = 8.7 Hz, 2 H), 7.32-7.20 (m, 4 H), 7.20-7.0 (m, 1 H), 6.20-6.10 (m, 1 H), 5.70-5.55 (m, 1 H), 4.59 (s, 2 H), 3.67 (t, *J* = 6.5 Hz, 2 H), 2.55-2.40 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.6, 147.2, 146.0, 134.5, 128.5, 127.6, 126.8, 126.6, 123.5, 95.1, 91.5, 71.6, 70.0, 29.1; MS (EI) *m/z* (%) 295 (M^+, 6.96), 115 (100); IR (neat) 3107, 3073, 3062, 3031, 2914, 2859, 1950, 1603, 1519, 1456, 1344, 1205, 1101, 1013 cm^{-1}; HRMS (EI) calcd for $C_{18}H_{17}NO_3$ (M^+): 295.1208. Found: 295.1207.

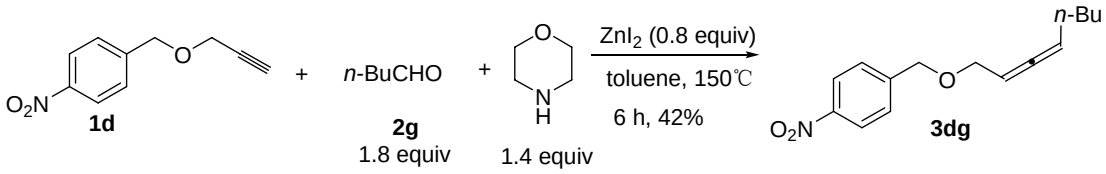
(2) Synthesis of 1-(4-nitrobenzyloxy)-5-methylhexa-2,3-diene (3df)



To a dried reaction tube with a screw cap was added ZnI_2 (256 mg, 0.80 mmol). This reaction tube was then dried under vacuum with a heating gun. Then propargyl 4-nitrobenzyl ether **1d** (191 mg, 1.00 mmol), isobutyraldehyde (131 mg, 1.82 mmol), morpholine (123 mg, 1.41 mmol), and toluene (5 mL) were added sequentially into

this dried reaction with a screw cap under an argon atmosphere and the resulting mixture sealed with the screw cap was stirred at 130 ℃. When the reaction was complete as monitored by TLC, the reaction mixture was cooled to room temperature and then filtered. Evaporation and column chromatography on silica gel afforded **3df** (138 mg, 56%) as a liquid (eluent: petroleum ether/ethyl acetate = 25:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.20 (d, *J* = 8.7 Hz, 2 H), 7.51 (d, *J* = 8.7 Hz, 2 H), 5.35-5.20 (m, 2 H), 4.63 (s, 2 H), 4.09 (dd, *J* = 6.5, 2.7 Hz, 2 H), 2.42-2.20 (m, 1 H), 1.02 (d, *J* = 6.6 Hz, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.7, 147.3, 146.1, 127.7, 123.5, 99.6, 89.2, 70.2, 69.4, 27.7, 22.4; MS (EI) *m/z* (%) 247 (M^+, 0.18), 81 (100); IR (neat) 2961, 2926, 2867, 1960, 1606, 1522, 1465, 1459, 1345, 1093, 1015; Anal. calcd. for $C_{14}H_{17}NO_3$: (%) C 68.00, H 6.93, N 5.66; Found: C 67.88, H 7.27, N 5.51.

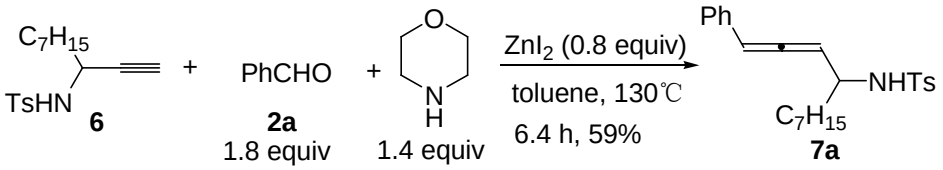
(3) Synthesis of 1-(4-nitrobenzyloxy)octa-2,3-diene (3dg)



To a dried reaction tube was added ZnI_2 (256 mg, 0.80 mmol). This reaction tube was dried under vacuum with a heating gun. Then propargyl 4-nitrobenzyl ether **1d** (191 mg, 1.00 mmol), valeraldehyde (155 mg, 1.80 mmol), morpholine (122 mg, 1.40 mmol), and toluene (5 mL) were added sequentially into this dried reaction tube with a screw tube under an argon atmosphere and the resulting mixture sealed with the screw tube was stirred at 150 ℃. When the reaction was complete as monitored by TLC, the reaction mixture was cooled to room temperature and then filtered.

Evaporation and column chromatography on silica gel afforded **3dg** (109 mg, 42%) as a liquid (eluent: petroleum ether/ethyl acetate=30:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (d, *J* = 8.7 Hz, 2 H), 7.50 (d, *J* = 8.7 Hz, 2 H), 5.30-5.15 (m, 2 H), 4.62 (s, 2 H), 4.13-4.02 (m, 2 H), 2.10-1.95 (m, 2 H), 1.45-1.27 (m, 4 H), 0.89 (t, *J* = 7.1 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.2, 147.2, 146.1, 127.7, 123.5, 92.2, 87.8, 70.2, 69.2, 31.2, 28.2, 22.0, 13.8; MS (EI) *m/z* (%) 261 (M^+, 0.09), 136 (100); IR (neat) 2957, 2928, 2859, 2362, 2336, 1963, 1605, 1522, 1460, 1346, 1094, 1013; Anal. calcd. for $C_{15}H_{19}NO_3$: (%) C 68.94, H 7.33, N 5.36; Found: C 69.17, H 7.47, N 5.21; HRMS (EI) calcd for $C_{15}H_{19}NO_3$ (M^+): 261.1365. Found: 261.1367.

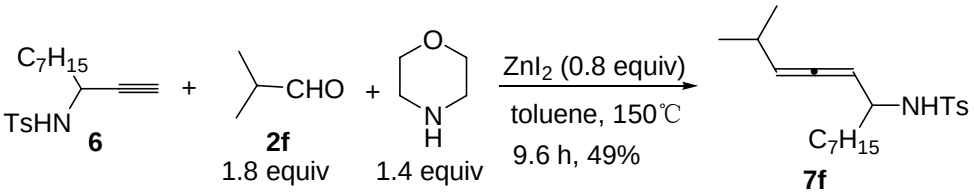
(4) Synthesis of *N*-(1-phenylundeca-1,2-dien-4-yl) *p*-tolylsulfonamide (7a)



To a dried reaction tube was added ZnI_2 (256 mg, 0.80 mmol). This reaction tube was dried under vacuum with a heating gun. Then *N*-(dec-1-yn-3-yl) *p*-tolylsulfonamide **6** (307 mg, 1.00 mmol), benzaldehyde (191 mg, 1.80 mmol), morpholine (122 mg, 1.40 mmol), and toluene (3 mL) were added sequentially into a dried reaction tube equipped with a reflux condenser under an argon atmosphere and the resulting mixture was stirred at 130 ℃. When the reaction was complete as monitored by TLC, the reaction mixture was cooled to room temperature and then filtered. Evaporation and column chromatography on silica gel afforded **7a** (235 mg, 59%) as a liquid as a pair of diastereoisomer (eluent: petroleum ether/ethyl acetate =

10:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84-7.68 (m, 2 H), 7.35-7.05 (m, 7 H), [6.13 (dd, *J* = 6.3, 2.1 Hz 0.44 H), 6.04 (dd, *J* = 6.3, 2.7 Hz 0.45 H)], [5.45 (t, *J* = 6.0 Hz, 0.46 H), 5.38 (t, *J* = 6.3 Hz, 0.45 H)], [5.16 (d, *J* = 8.1 Hz 0.46 H), 5.00 (d, *J* = 8.4 Hz 0.43 H)], 3.97-3.80 (m, 1 H), 2.39 (d, *J* = 4.8 Hz, 3 H), 1.70-1.45 (m, 2 H), 1.45-1.05 (m, 10 H), 0.85 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ [203.6, 203.4], [143.1, 143.0], [138.0, 137.8], 133.4, [129.5, 129.4], [128.56, 128.50], [127.25, 127.17], 127.08, [126.76, 126.73], [97.99, 97.53], [97.28, 96.88], [52.8, 52.5], [36.2, 36.0], 31.6, [29.1, 29.0], 25.3, 22.5, 21.4, 14.0; MS (EI) *m/z* (%) 397 (M^+, 4.03), 282 (100); IR (neat) 3275, 3062, 3032, 2926, 2855, 1952, 1598, 1495, 1458, 1328, 1159, 1093, 1069 cm^{-1}; HRMS (EI) calcd for $C_{24}H_{31}NO_2S$ (M^+): 397.2076. Found: 397.2077.

(5) **Synthesis of *N*-(2-methyluntrica-3,4-dien-6-yl) *p*-tolylsulfonamide (7f)**



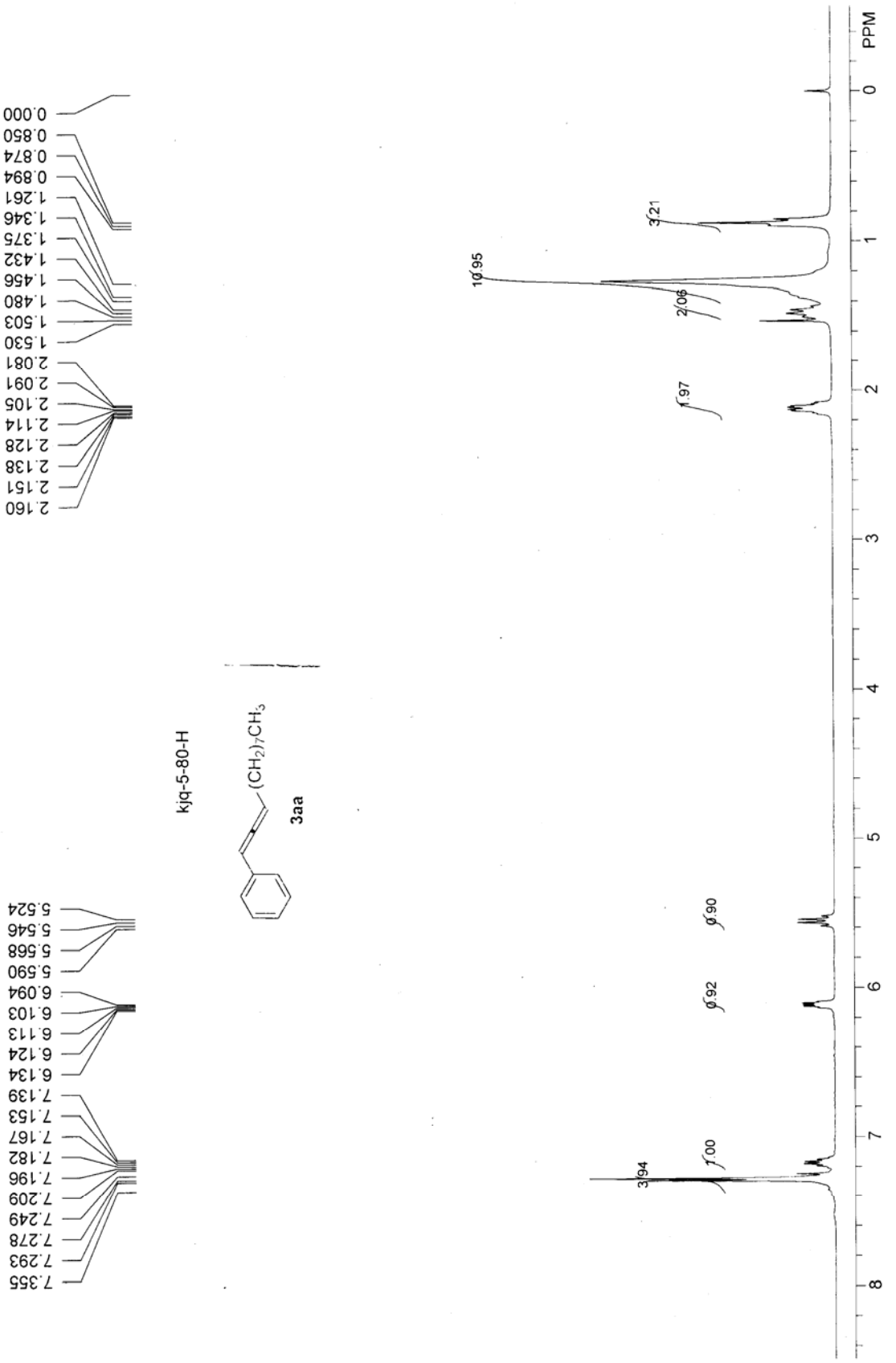
To a dried reaction tube was added ZnI_2 (256 mg, 0.80 mmol). This reaction tube was dried under vacuum with a heating gun. Then *N*-(dec-1-yn-3-yl) 4-methylbenzenesulfonamide **6** (306 mg, 1.00 mmol), isobutyraldehyde (130 mg, 1.81 mmol), morpholine (121 mg, 1.39 mmol), and toluene (5 mL) were added sequentially into a dried reaction tube with a screw cap under an argon atmosphere. The resulting mixture sealed with the screw cap was stirred at 150 ℃. When the reaction was complete as monitored by TLC, the reaction mixture was cooled to room

temperature and then filtered. Evaporation and column chromatography on silica gel afforded **7f** (177 mg, 49%) as a liquid as a pair of diastereoisomer (eluent: petroleum ether/ethyl acetate=8:1): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (dd, *J* = 8.3, 2.6 Hz, 2 H), 7.28 (d, *J* = 7.5 Hz, 2 H), 5.20-4.90 (m, 2 H), 4.68-4.50 (m, 1 H), 3.85-3.68 (m, 1 H), 2.42 (s, 3 H), 2.30-2.12 (m, 1 H), 1.60-1.40 (m, 2 H), 1.33-1.15 (m, 10 H), 1.00-0.83 (m, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ [200.9, 200.3], [143.0, 142.9], [138.2, 138.0], 129.4, 127.1, [102.6, 101.7], [94.3, 94.0], [52.9, 52.2], [36.2, 36.0], 31.7, [29.10, 29.05], 27.8, 25.2, 22.5, [22.31, 22.25], 21.4, 14.0; MS (EI) *m/z* (%) 363 (M^+, 1.07), 282 (100); IR (neat) 3277, 2958, 2927, 2858, 1963, 1916, 1599, 1496, 1461, 1328, 1161, 1093 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{33}NO_2S$ (M^+): 363.2232. Found: 363.2230.

References:

(1) (a) Elsevier, C. J.; Vermeer, P. *J. Org. Chem.* **1989,** *54*, 3726. (b) Larsen, C. H.; Anderson, K. W.; Tundel, R. E.; Buchwald, S. L. *Synlett* **2006**, 2941.

(2) Denis, J. N.; Krief, A. *Tetrahedron Lett.* **1982**, *23*, 3411.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PPM
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1.00
0.92
0.90
1.97
2.06
10.95
3.21
7.355
7.293
7.278
7.249
7.209
7.196
7.182
7.167
7.153
7.139
6.134
6.124
6.113
6.103
6.094
5.590
5.568
5.546
5.524
2.160
2.151
2.138
2.128
2.114
2.105
2.091
2.081
1.530
1.503
1.480
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1.432
1.375
1.346
1.261
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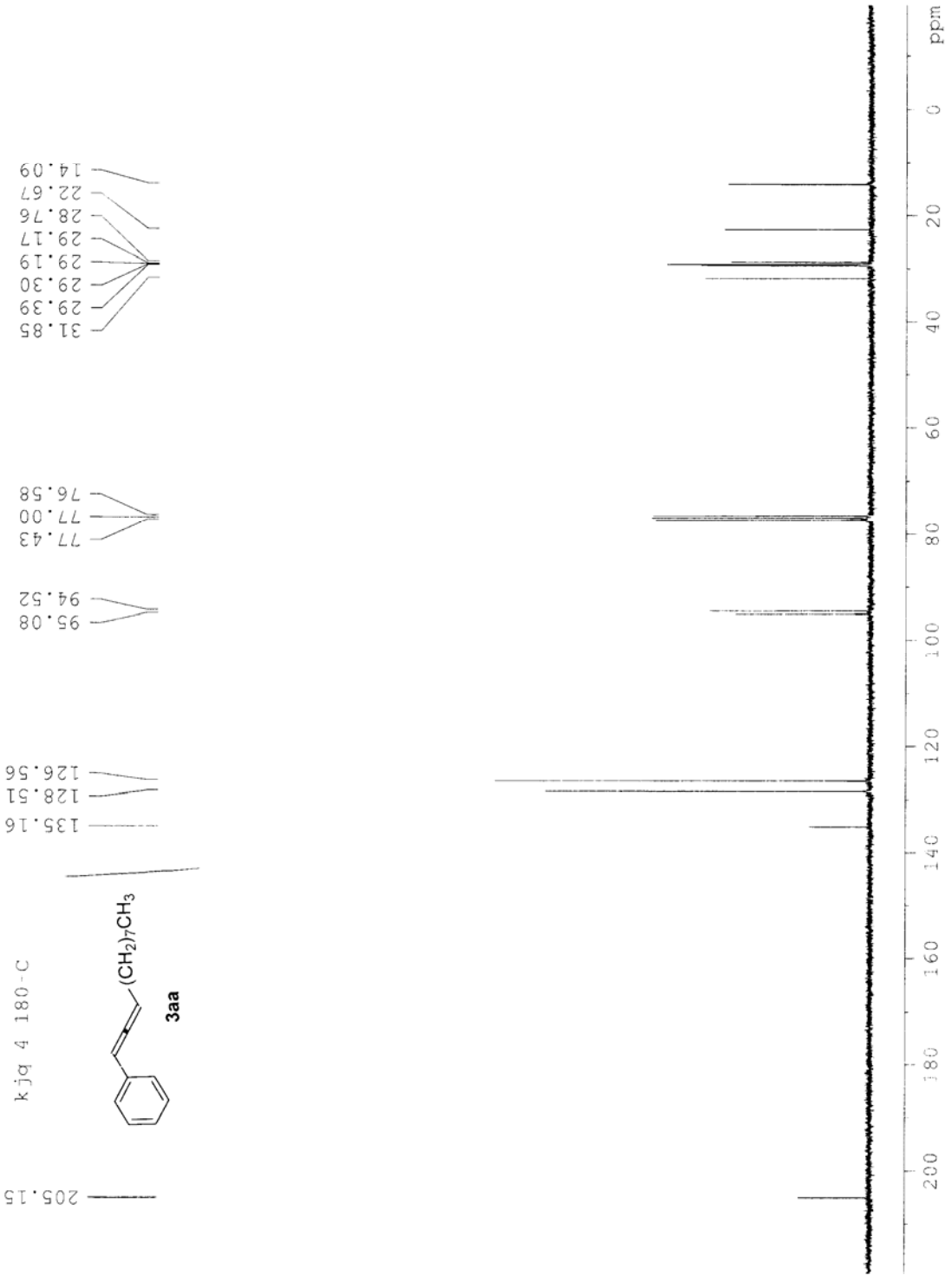
0.874
0.850
0.000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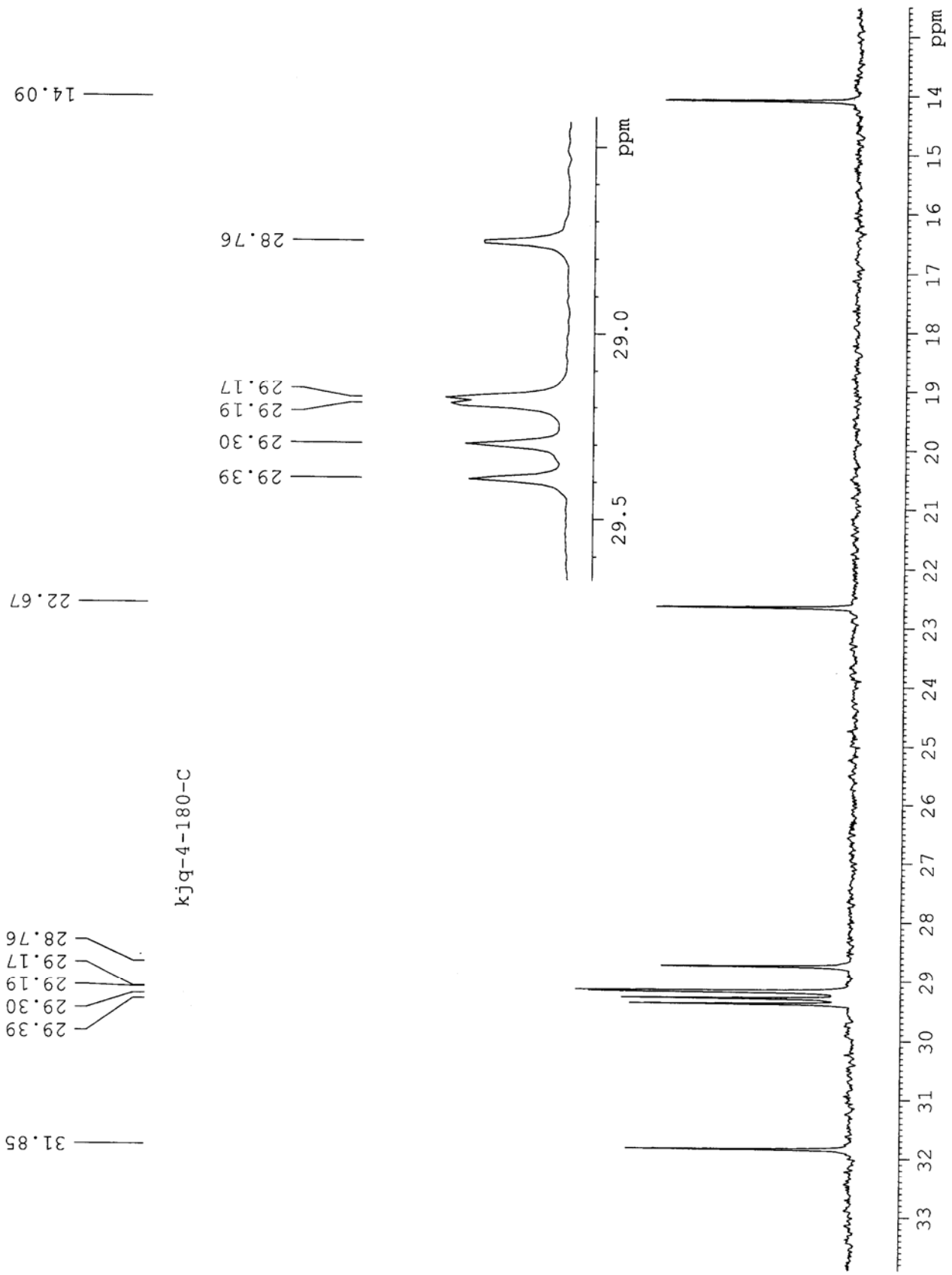
135.16
128.51
126.56
95.08
94.52
77.43
77.00
76.58
31.85
29.39
29.30
29.19
29.17
28.76
22.67
14.09
ppm


14.09
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29.17
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29.30
29.39
22.67
31.85
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ppm
29.5
29.0
28.76
29.17
29.19
29.30
29.39
ppm
14
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18
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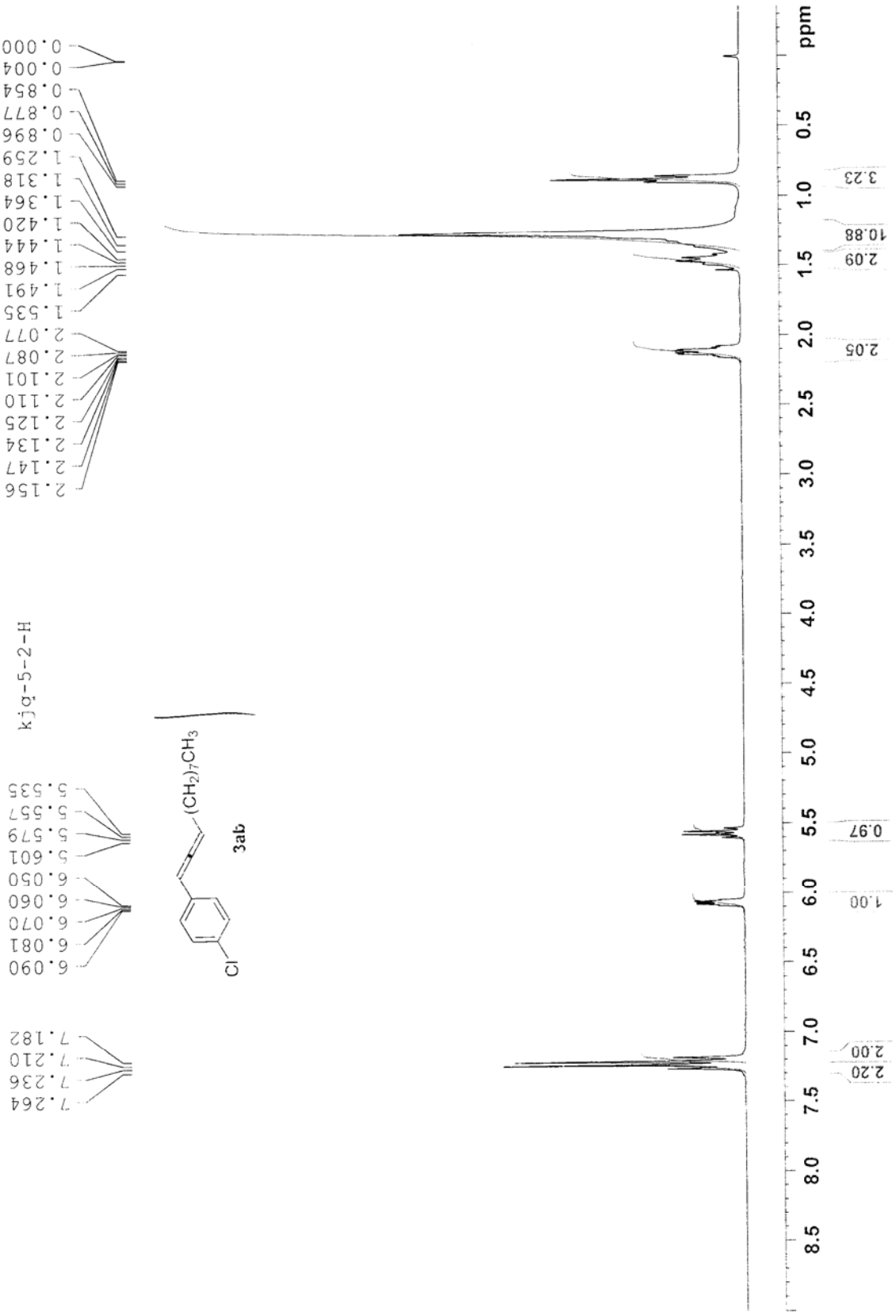
31
32
33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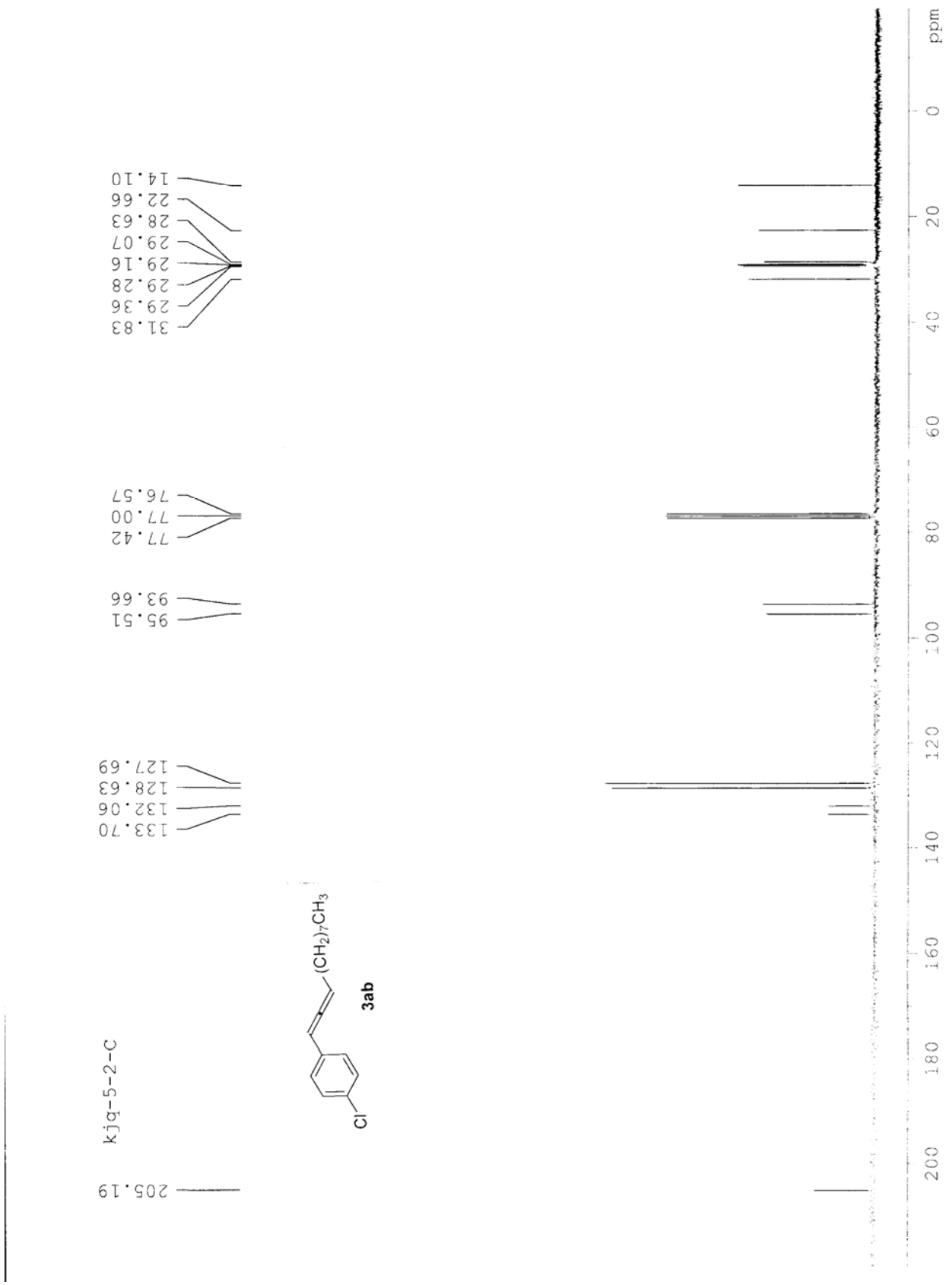
ppm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132.06
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127.69
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93.66
77.42
77.00
76.57
31.83
29.36
29.28
29.16
29.07
28.63
22.66
14.10
ppm
0
20
40
60
80
100
120
140
160
180
200
(CH2)7CH3
Cl
3ab

22.66
31.83
kjq-5-2-c
29.36
29.28
29.16
29.07
28.63
ppm
22
23
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27
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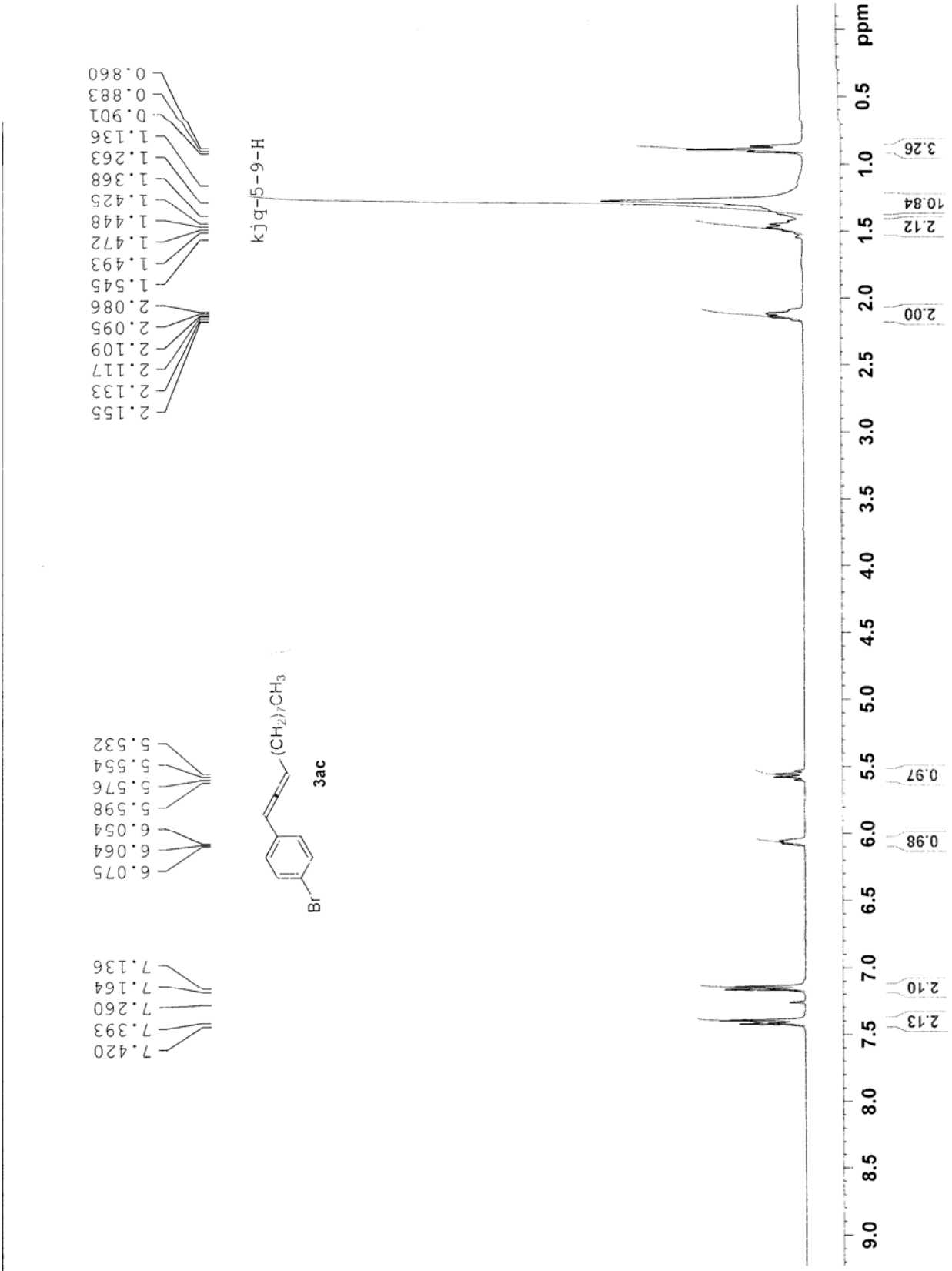
29
30
31
32
33
34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2.086
2.095
2.109
2.117
2.133
2.155
6.075
6.064
6.054
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5.576
5.554
5.532
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7.393
7.260
7.164
7.136
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Br
3ac
ppm
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2.0
2.5
3.0
3.5
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4.5
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5.5
6.0
6.5
7.0
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8.0
8.5
9.0
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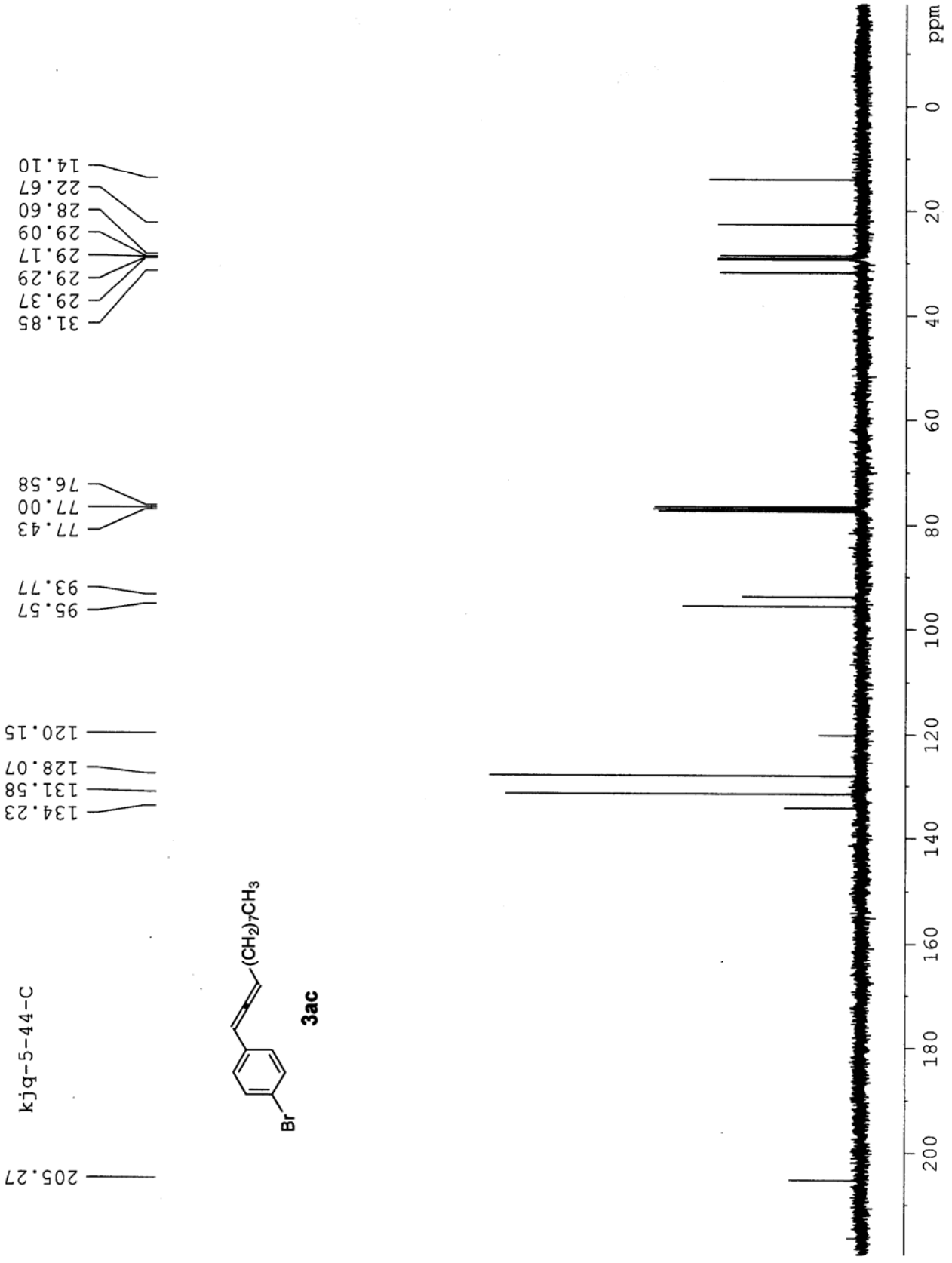
2.00
0.97
0.98
2.10
2.13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29.37
29.29
29.17
29.09
28.60
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14.10
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3ac
ppm
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60
80
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120
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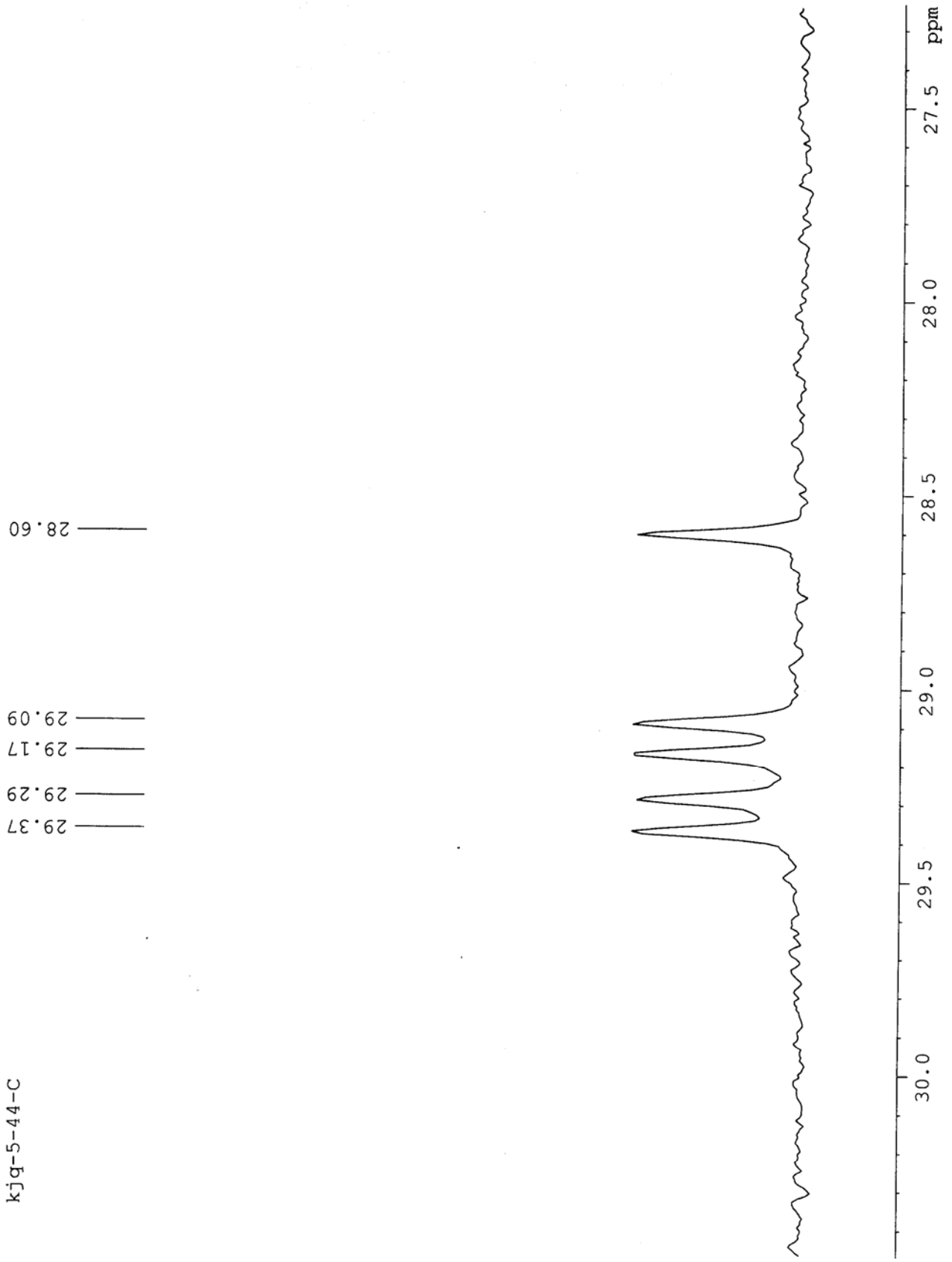
160
180
200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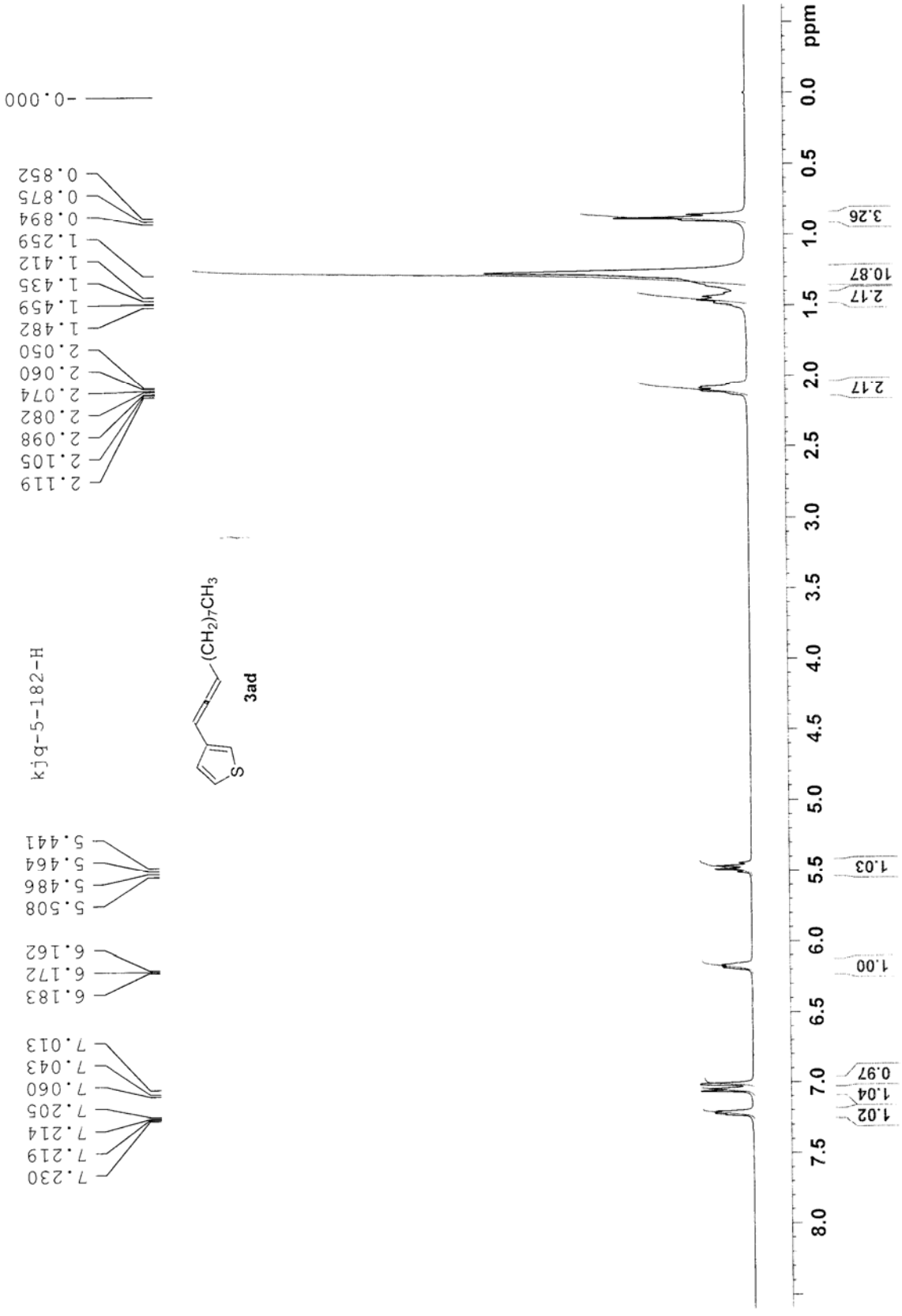
28.5
28.0
27.5
ppm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4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
3.26
10.87
2.17
2.17
1.03
1.00
0.97
1.04
1.02
-0.000
0.852
0.875
0.894
1.259
1.412
1.435
1.459
1.482
2.050
2.060
2.074
2.082
2.098
2.105
2.119
5.441
5.464
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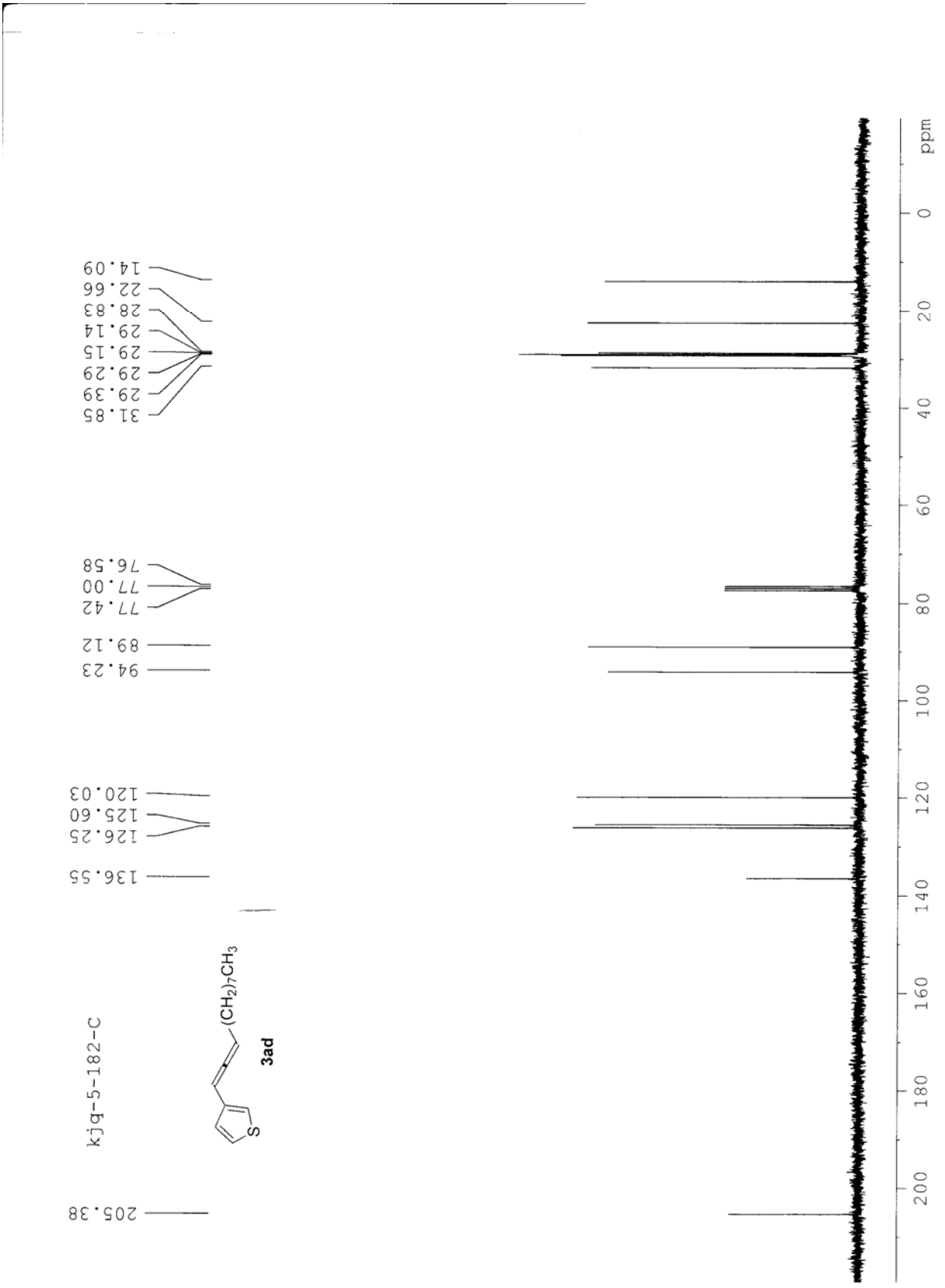
7.060
7.205
7.214
7.219
7.230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29.14
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22.66
14.09
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3ad
ppm
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20
40
60
80
100
120
140
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180
200

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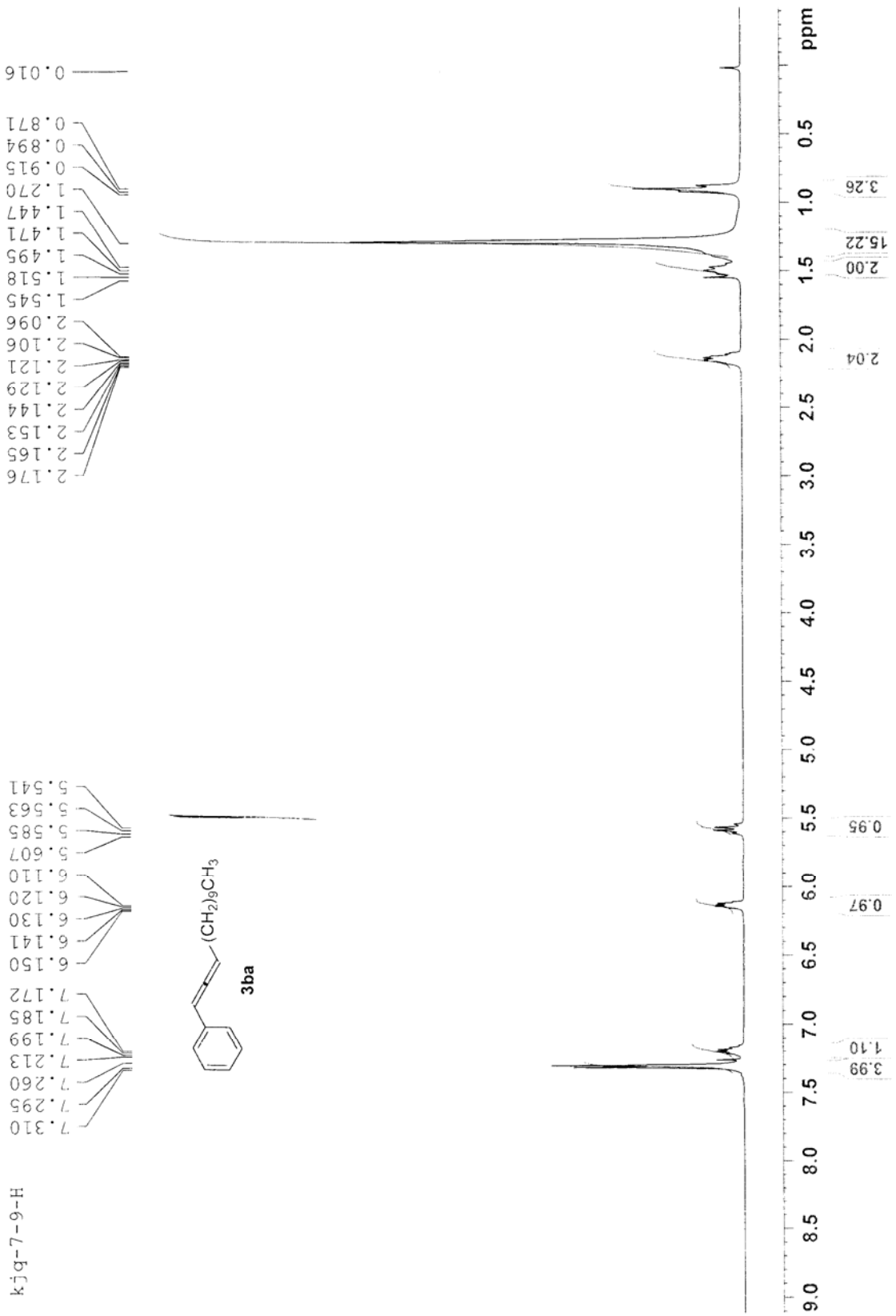
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2.153
2.144
2.129
2.121
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1.270
0.915
0.894
0.871
0.016
(CH2)9CH3
3ba
ppm
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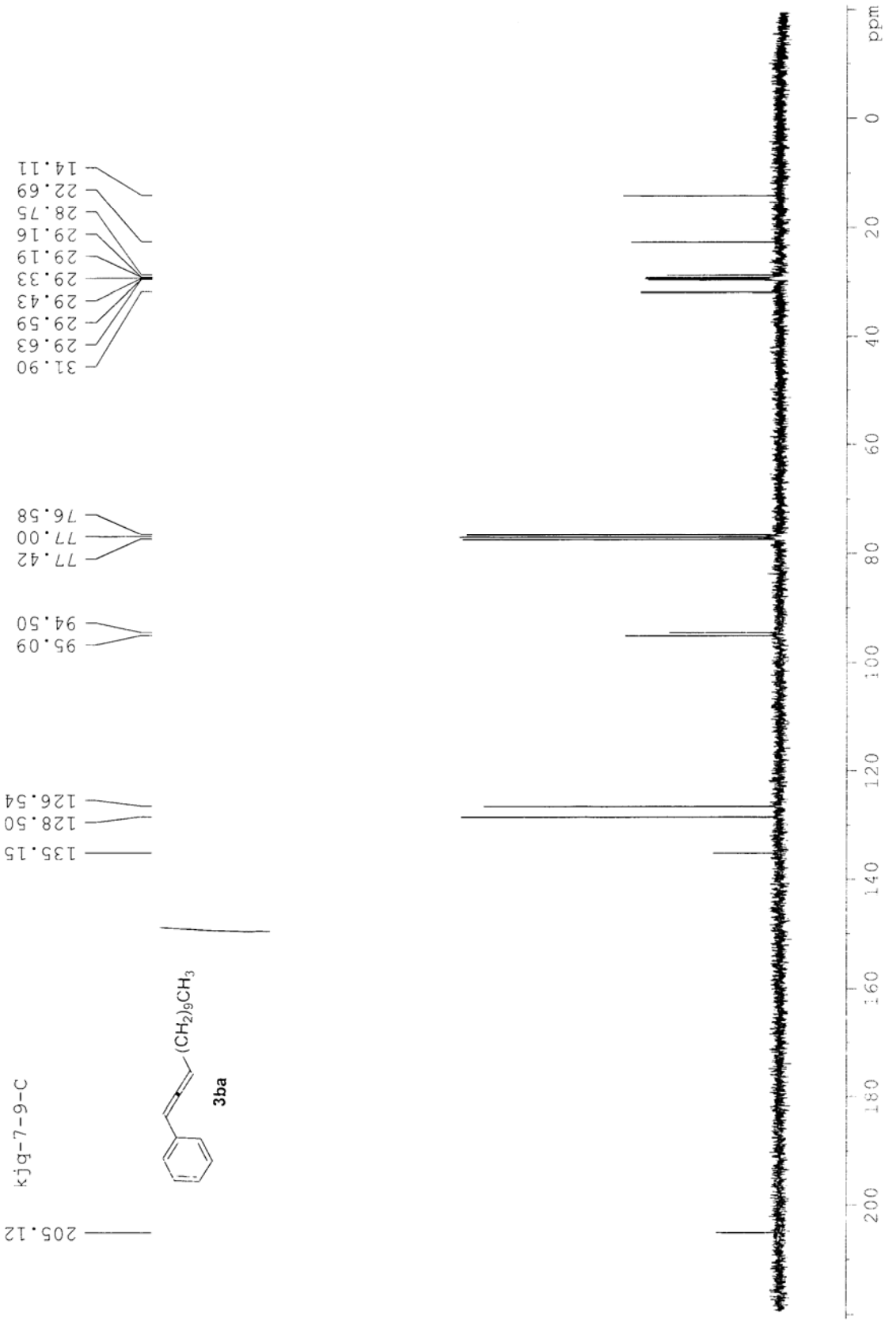
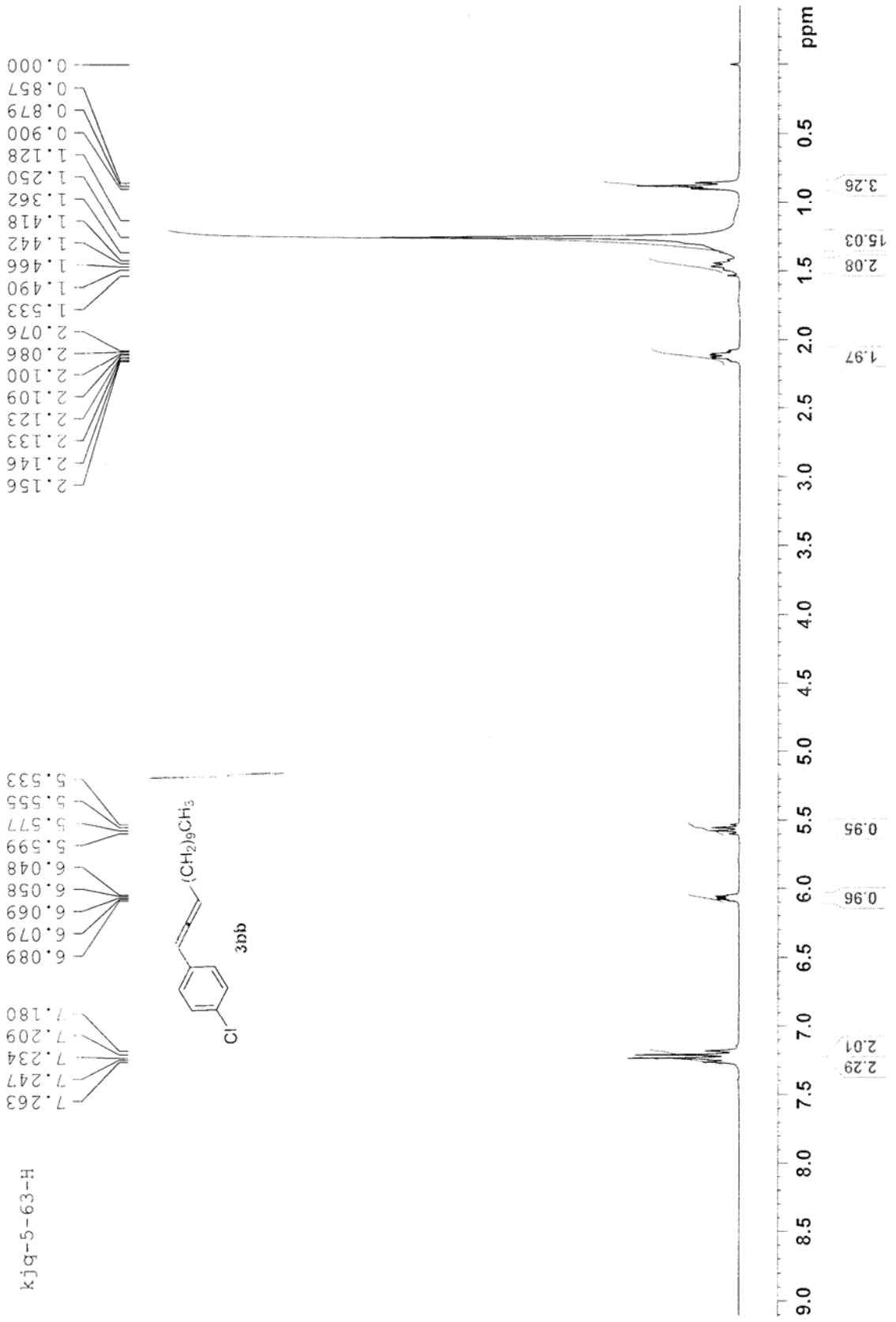
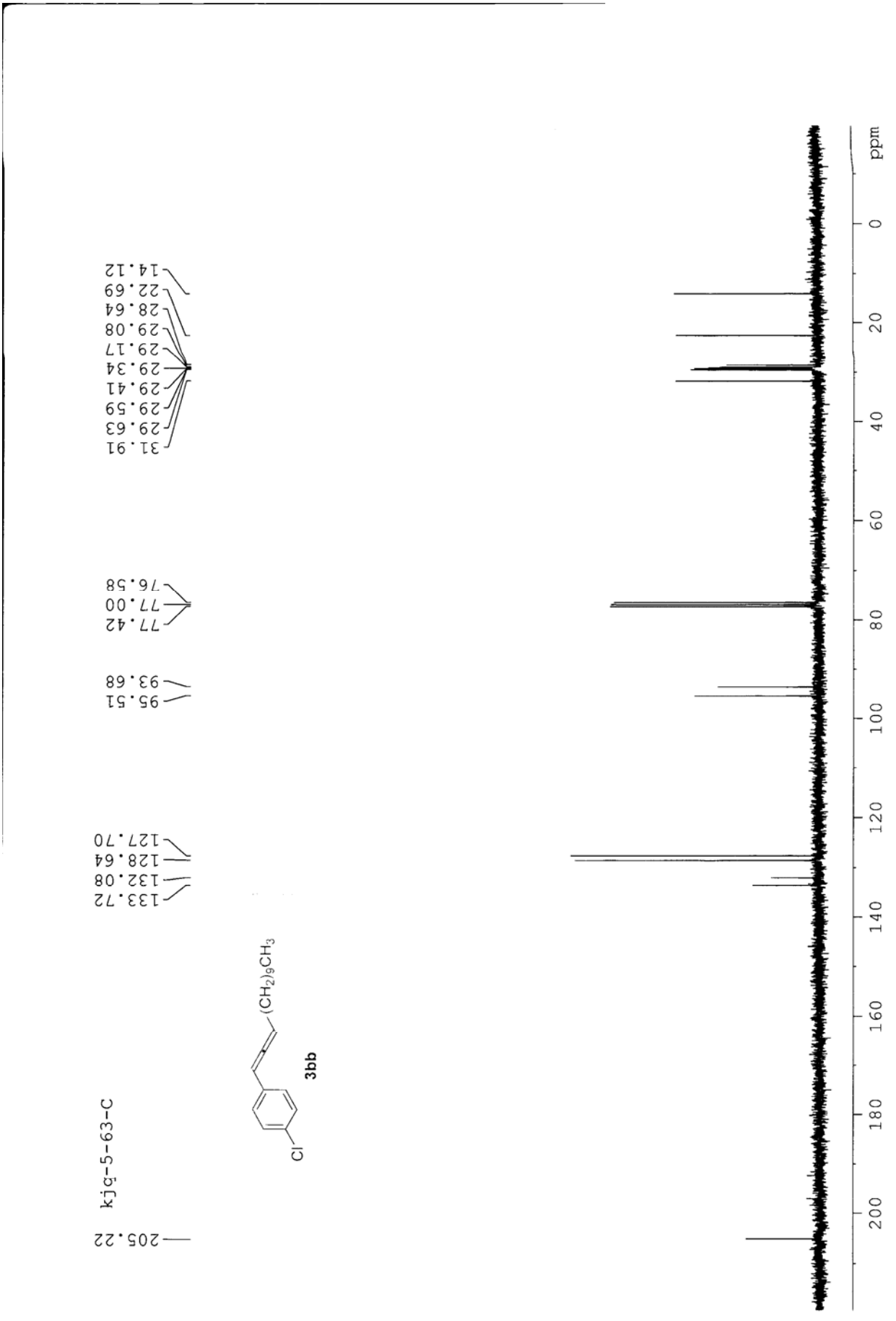
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
3.99
1.10
0.97
0.95
2.04
2.00
15.22
3.26

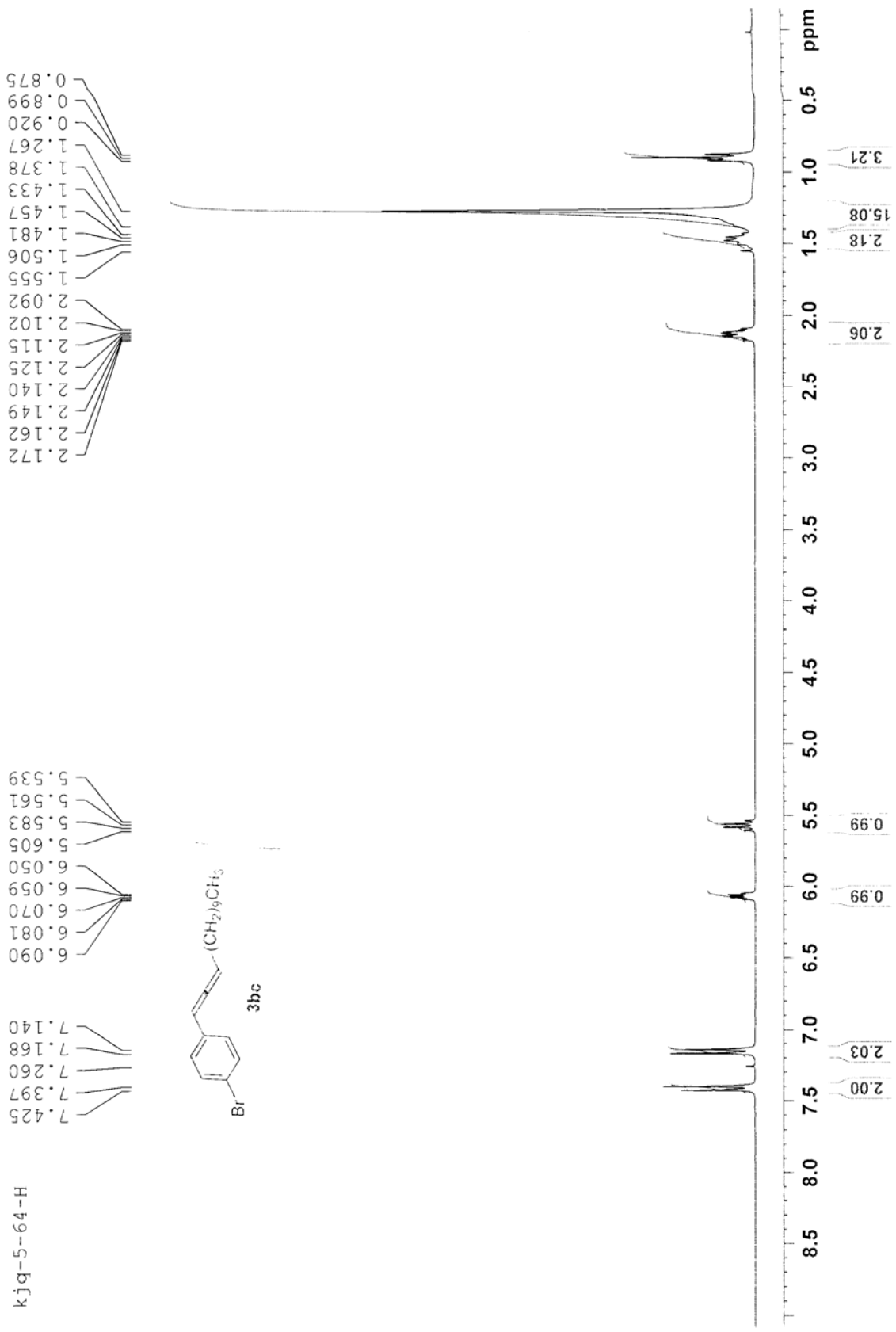

kjq-7-9-C
205.12
135.15
128.50
126.54
95.09
94.50
77.42
77.00
76.58
31.90
29.63
29.59
29.43
29.33
29.19
29.16
28.75
22.69
14.11
(CH2)9CH3
3ba
ppm
0
20
40
60
80
100
120
140
160
180
200

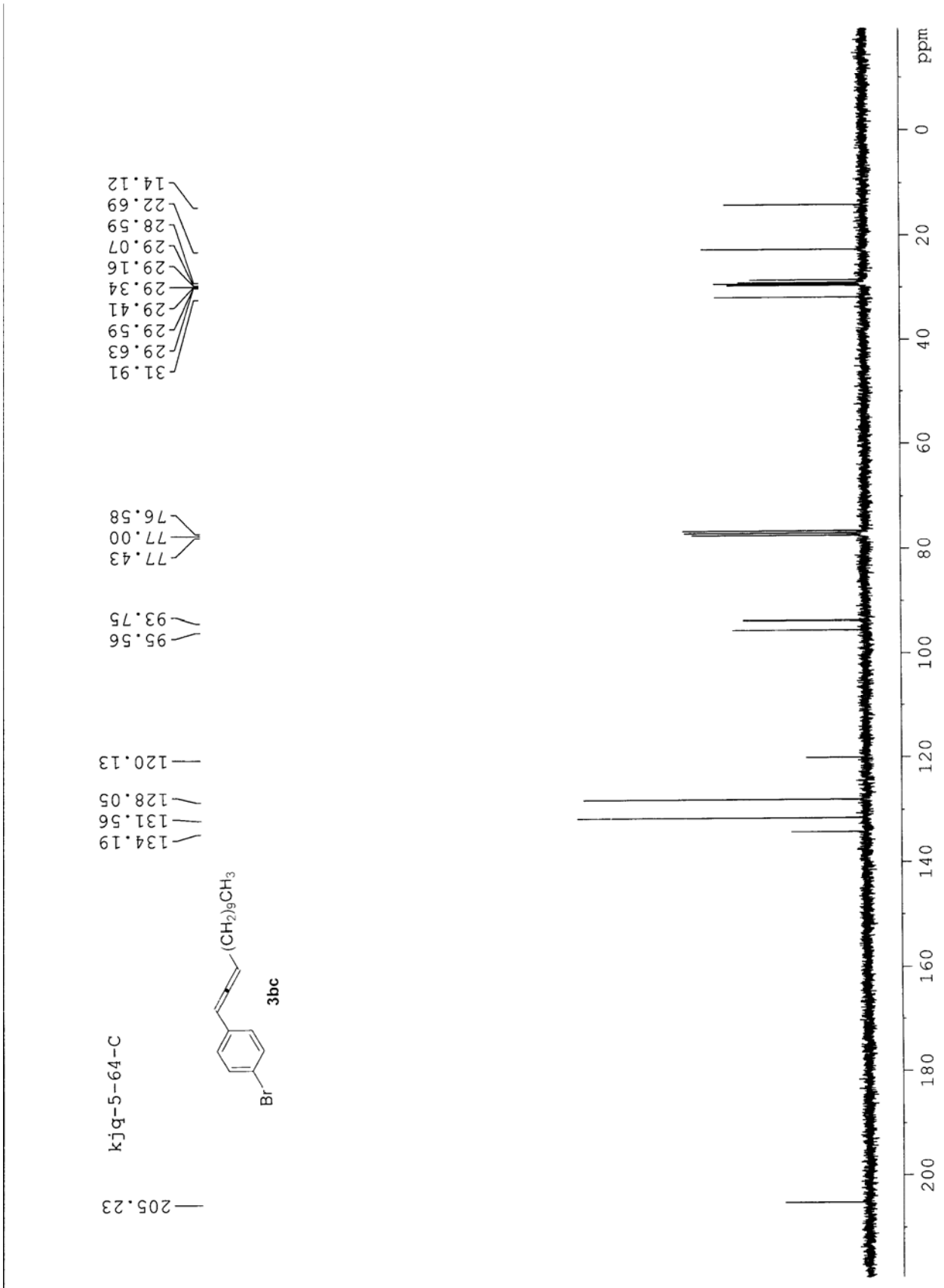
kjq-7-9-C
31.90
28.75
29.16
29.19
29.33
29.43
29.59
29.63
32.5
32.0
31.5
31.0
30.5
30.0
29.5
29.0
28.5
ppm

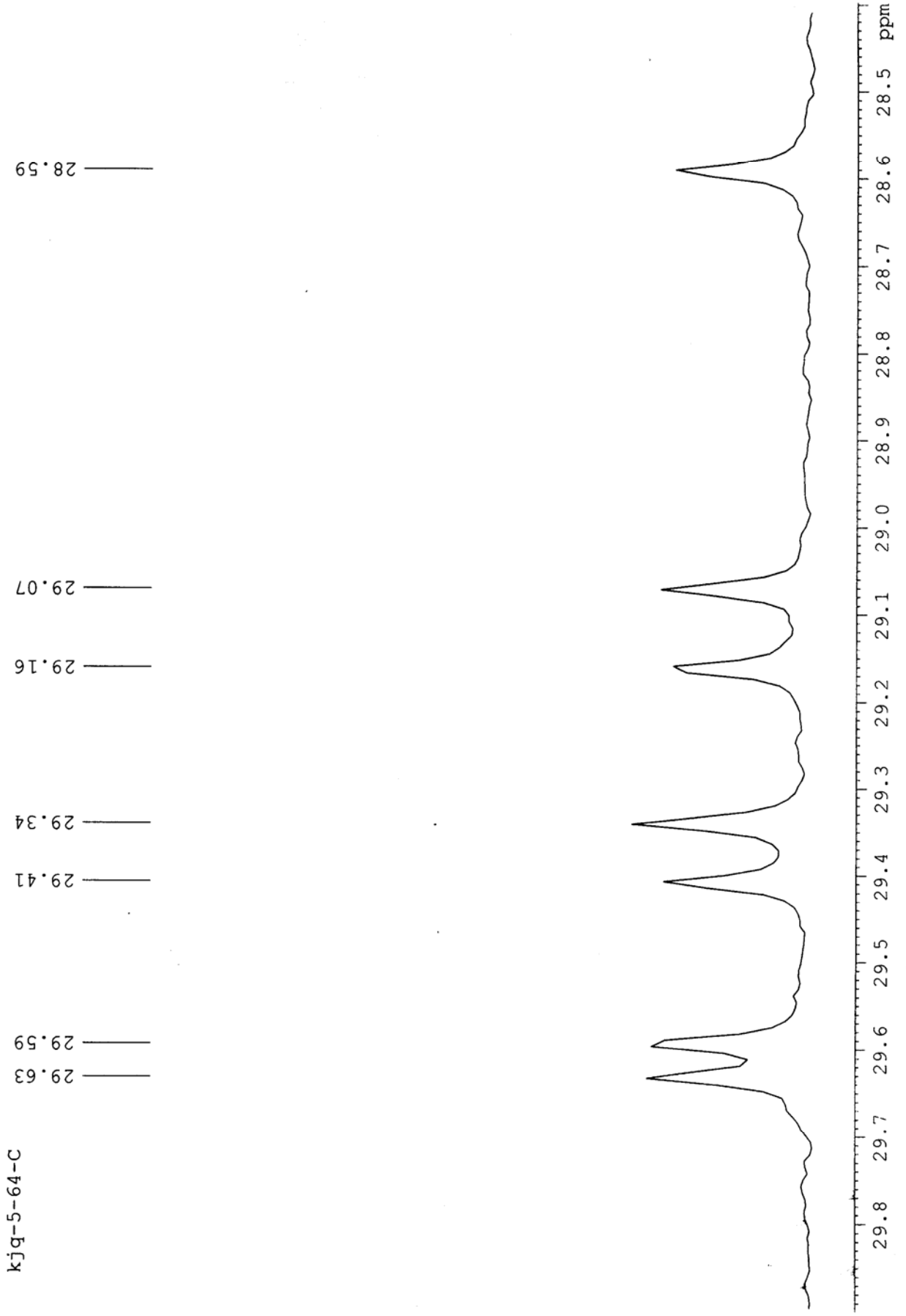

kjq-5-63-H
7.263
7.247
7.234
7.209
7.180
6.089
6.079
6.069
6.058
6.048
5.599
5.577
5.555
5.533
2.156
2.146
2.133
2.123
2.109
2.100
2.086
2.076
1.533
1.490
1.466
1.442
1.418
1.362
1.250
1.128
0.900
0.879
0.857
0.000
(CH2)9CH3
Cl
3bb
ppm
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
2.29
2.01
0.96
0.95
1.97
2.08
15.03
3.26

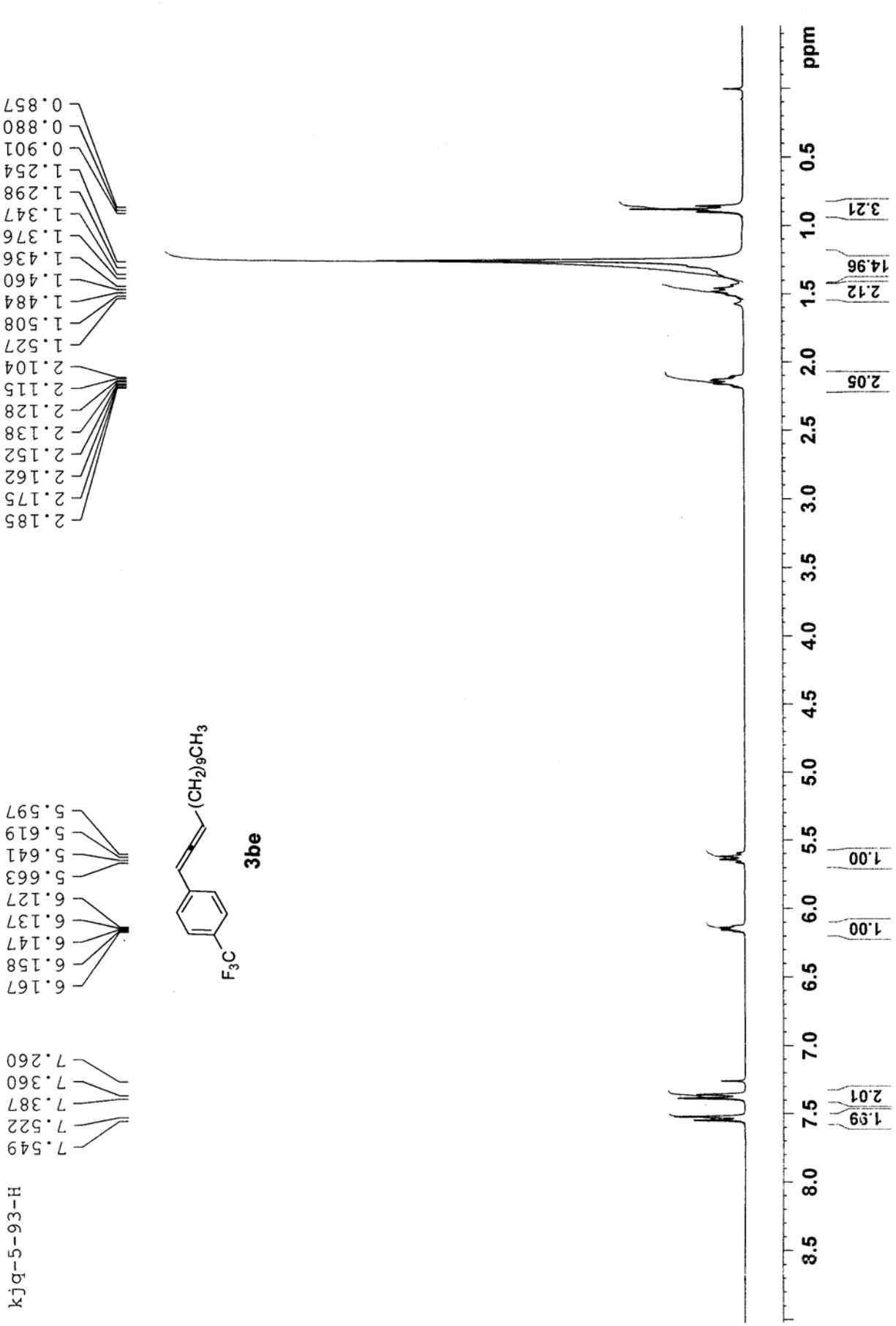

205.22
kjq-5-63-C
133.72
132.08
128.64
127.70
95.51
93.68
77.42
77.00
76.58
31.91
29.63
29.59
29.41
29.34
29.17
29.08
28.64
22.69
14.12
3bb
Cl
(CH2)9CH3
ppm
0
20
40
60
80
100
120
140
160
180
200

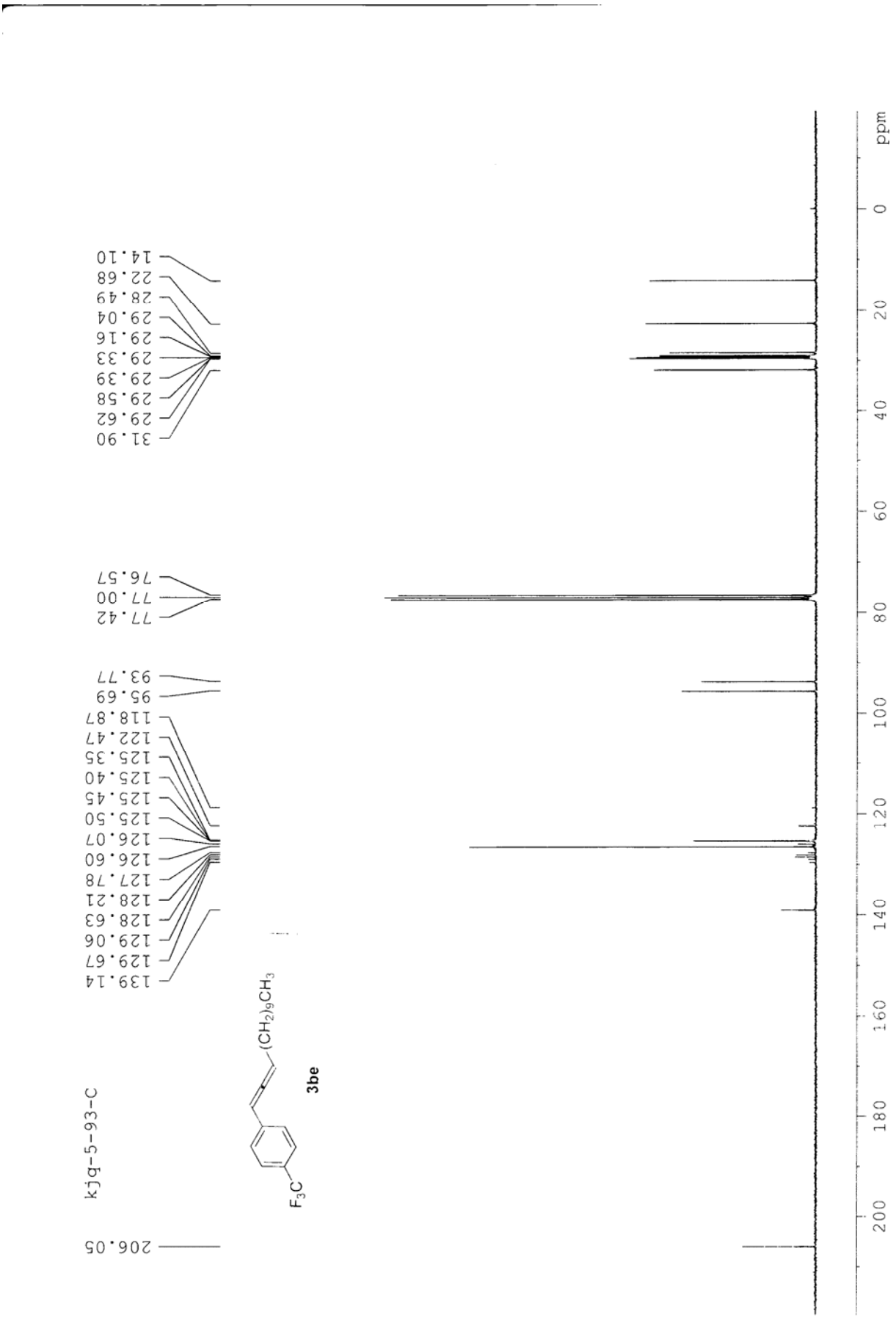
28.641
29.079
29.169
29.341
29.407
29.590
29.634
31.913
kjq-5-63-C
32.0
31.5
31.0
30.5
30.0
29.5
29.0
28.5
PPM

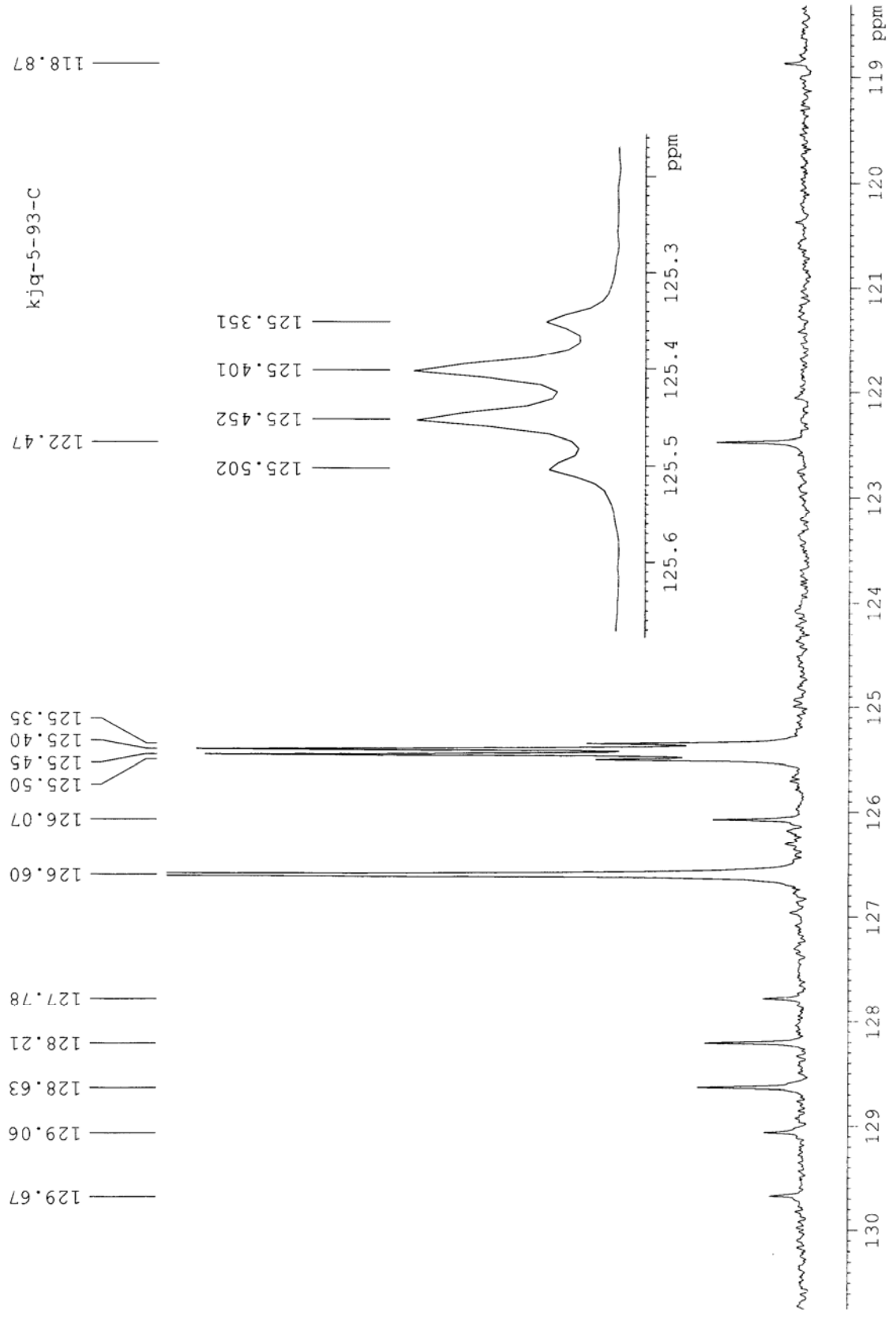

kjq-5-64-H
7.425
7.397
7.260
7.168
7.140
6.090
6.081
6.070
6.059
6.050
5.605
5.583
5.561
5.539
2.172
2.162
2.149
2.140
2.125
2.115
2.102
2.092
1.555
1.506
1.481
1.457
1.433
1.378
1.267
0.920
0.899
0.875
3bc
Br
(CH2)9CH3
ppm
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
2.00
2.03
0.99
0.99
2.06
2.18
15.08
3.21

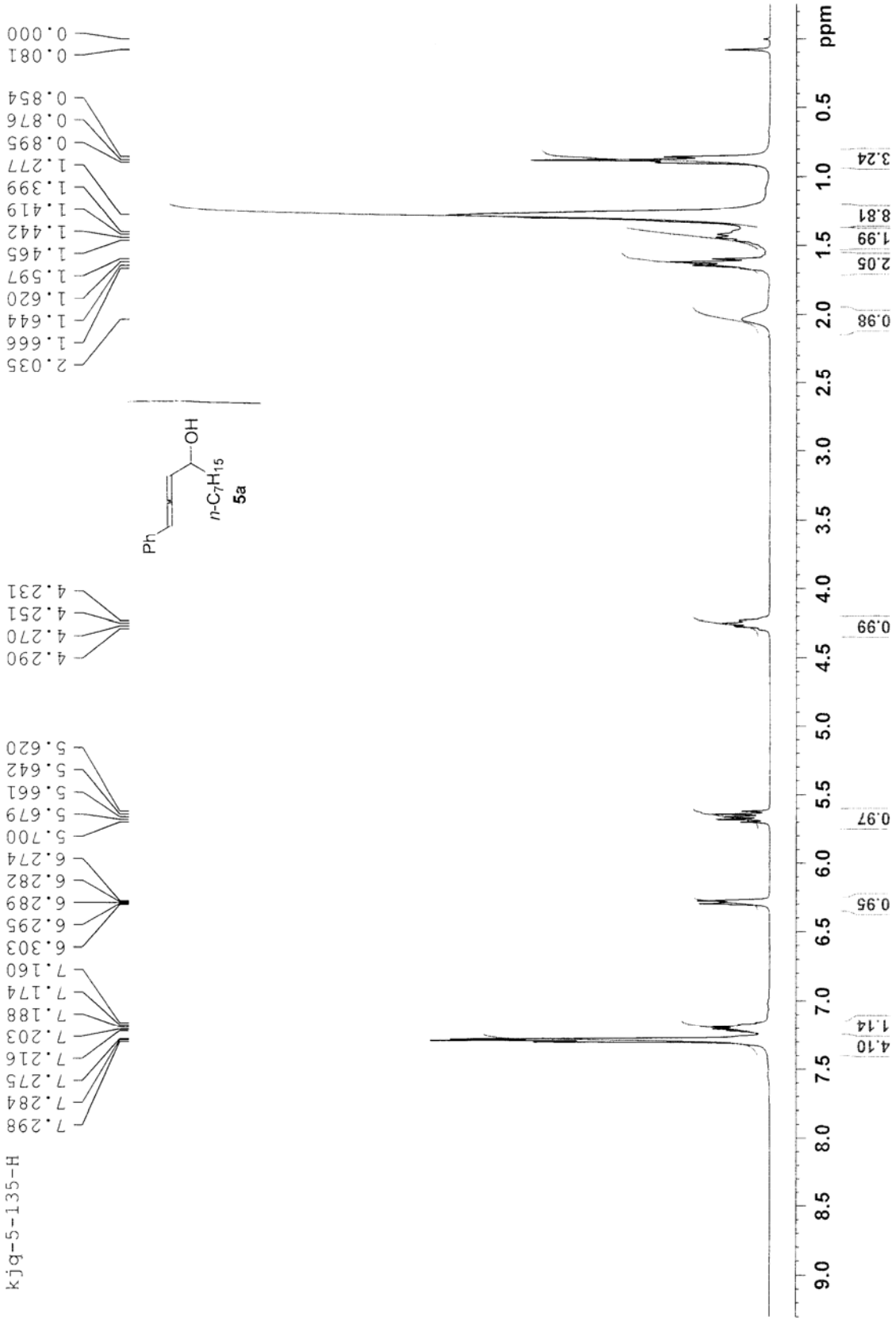

205.23
kjq-5-64-C
134.19
131.56
128.05
120.13
95.56
93.75
77.43
77.00
76.58
31.91
29.63
29.59
29.41
29.34
29.16
29.07
28.59
22.69
14.12
(CH2)9CH3
Br
3bc
ppm
0
20
40
60
80
100
120
140
160
180
200


kjq-5-64-C
29.63
29.59
29.41
29.34
29.16
29.07
28.59
29.8
29.7
29.6
29.5
29.4
29.3
29.2
29.1
29.0
28.9
28.8
28.7
28.6
28.5
ppm


kjq-5-93-H
7.549
7.522
7.387
7.360
7.260
6.167
6.158
6.147
6.137
6.127
5.663
5.641
5.619
5.597
2.185
2.175
2.162
2.152
2.138
2.128
2.115
2.104
1.527
1.508
1.484
1.460
1.436
1.376
1.347
1.298
1.254
0.901
0.880
0.857
3be
ppm
1.99
2.01
1.00
1.00
2.05
2.12
14.96
3.21

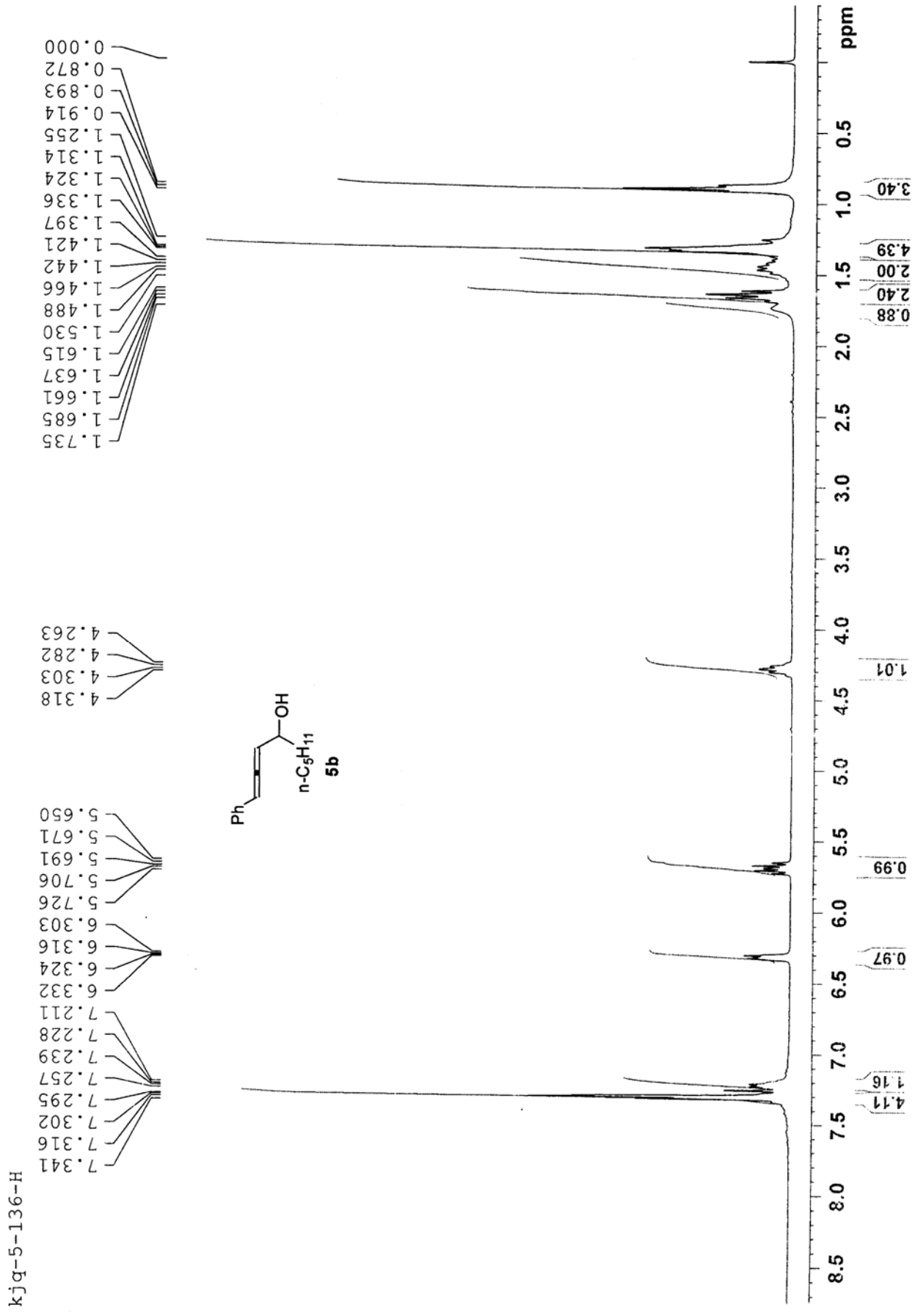

kjq-5-93-C
206.05
139.14
129.67
129.06
128.63
128.21
127.78
126.60
126.07
125.50
125.45
125.40
125.35
122.47
118.87
95.69
93.77
77.42
77.00
76.57
31.90
29.62
29.58
29.39
29.33
29.16
29.04
28.49
22.68
14.10
ppm
0
20
40
60
80
100
120
140
160
180
200
3be
$(CH_2)_9CH_3$
F_3C

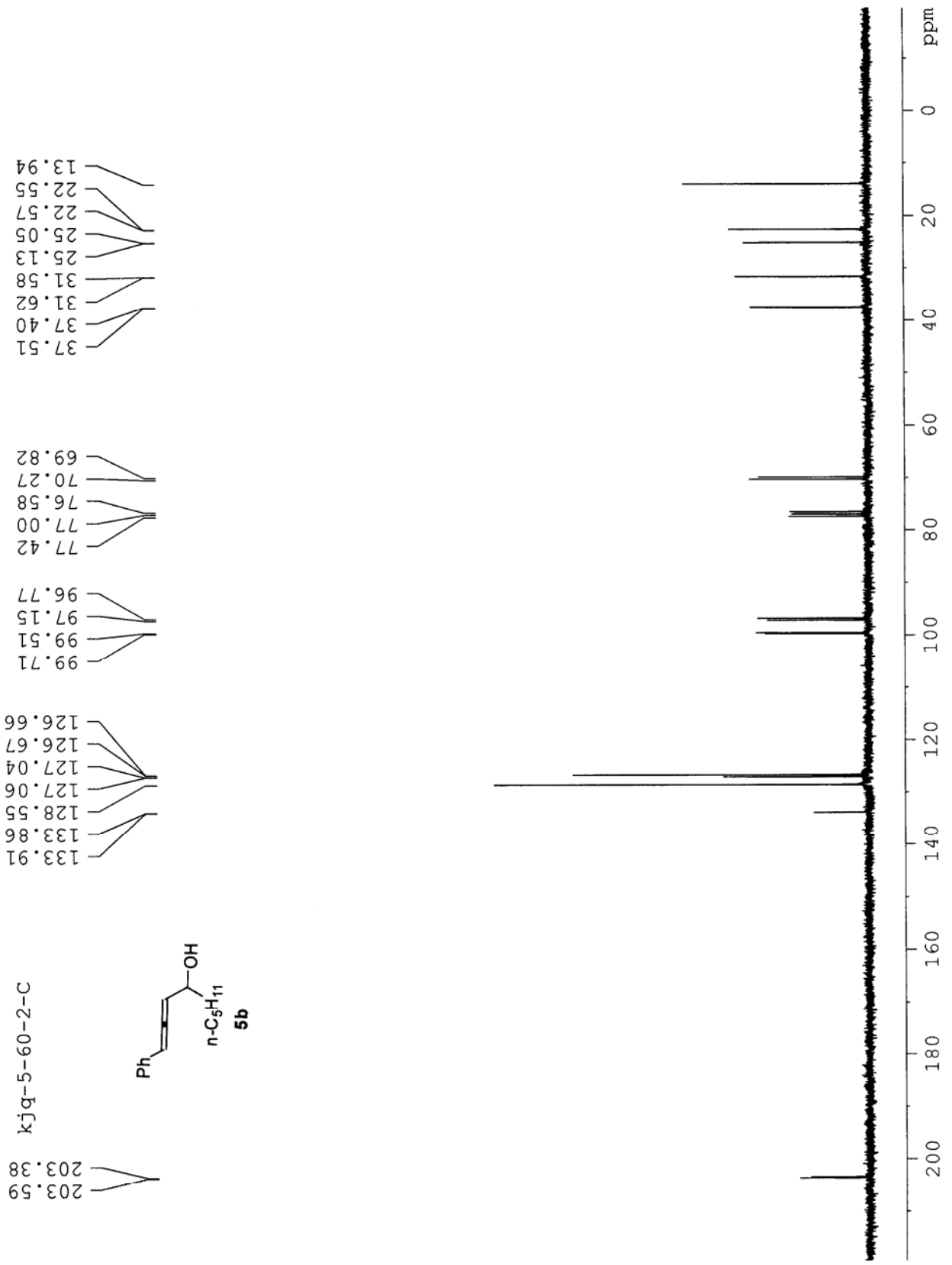

kjq-5-93-C
118.87
125.351
125.401
125.452
125.502
122.47
125.35
125.40
125.45
125.50
126.07
126.60
127.78
128.21
128.63
129.06
129.67
125.6
125.5
125.4
125.3
ppm
119
120
121
122
123
124
125
126
127
128
129
130
ppm

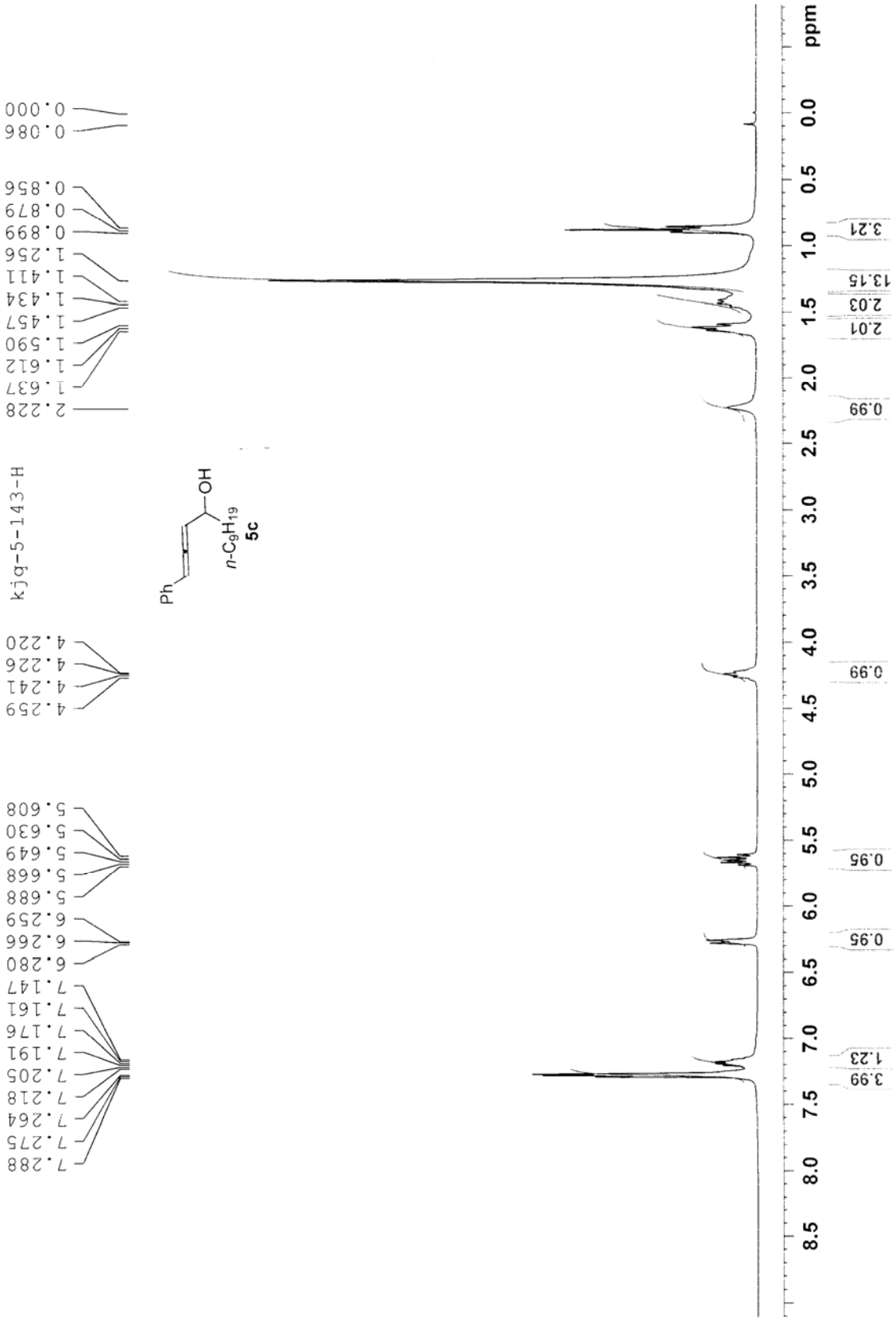

kjq-5-135-H
7.298
7.284
7.275
7.216
7.203
7.188
7.174
7.160
6.303
6.295
6.289
6.282
6.274
5.700
5.679
5.661
5.642
5.620
4.290
4.270
4.251
4.231
2.035
1.666
1.644
1.620
1.597
1.465
1.442
1.419
1.399
1.277
0.895
0.876
0.854
0.081
0.000
OH
n-C7H15
Ph
5a
ppm
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
3.24
8.81
1.99
2.05
0.98
0.99
0.97
0.95
1.14
4.10

kjq-5-135-C
203.60
203.38
133.92
133.88
128.57
127.09
127.07
126.69
99.74
99.54
97.23
96.85
77.42
77.00
76.57
70.28
69.85
37.60
37.48
31.72
29.41
29.38
29.23
29.21
25.46
25.39
22.59
14.03
OH
Ph
n-C7H15
5a
ppm
0
20
40
60
80
100
120
140
160
180
200

14.03
22.59
25.39
25.46
29.21
29.23
29.38
29.41
31.72
37.48
37.60
kjq-5-135-C
13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40
ppm

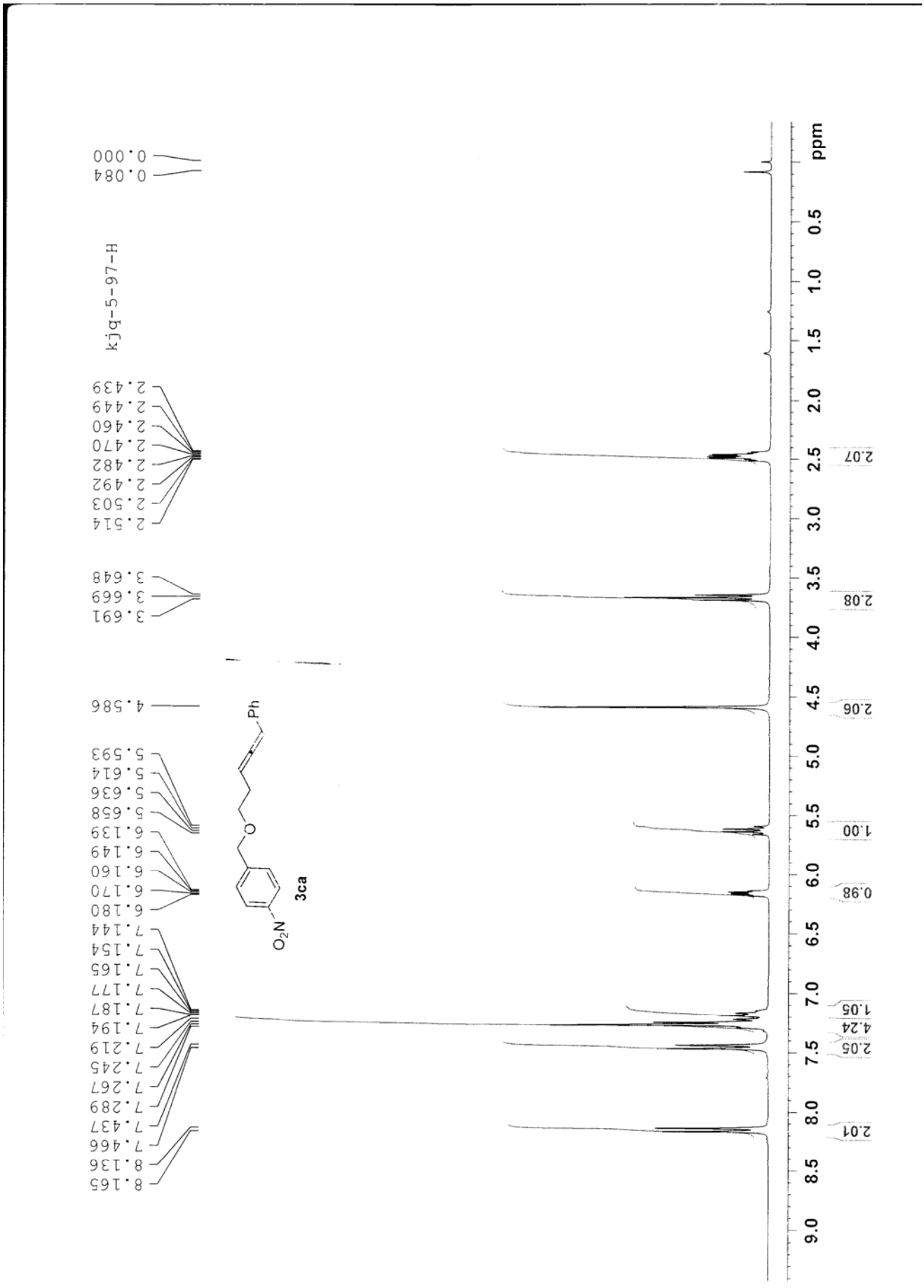

kjq-5-136-H
7.341
7.316
7.302
7.295
7.257
7.239
7.228
7.211
6.332
6.324
6.316
6.303
5.726
5.706
5.691
5.671
5.650
4.318
4.303
4.282
4.263
1.735
1.685
1.661
1.637
1.615
1.530
1.488
1.466
1.442
1.421
1.397
1.336
1.324
1.314
1.255
0.914
0.893
0.872
0.000
3.40
4.39
2.00
2.40
0.88
1.01
0.99
0.97
1.16
4.11
ppm
OH
n-C5H11
Ph
5b

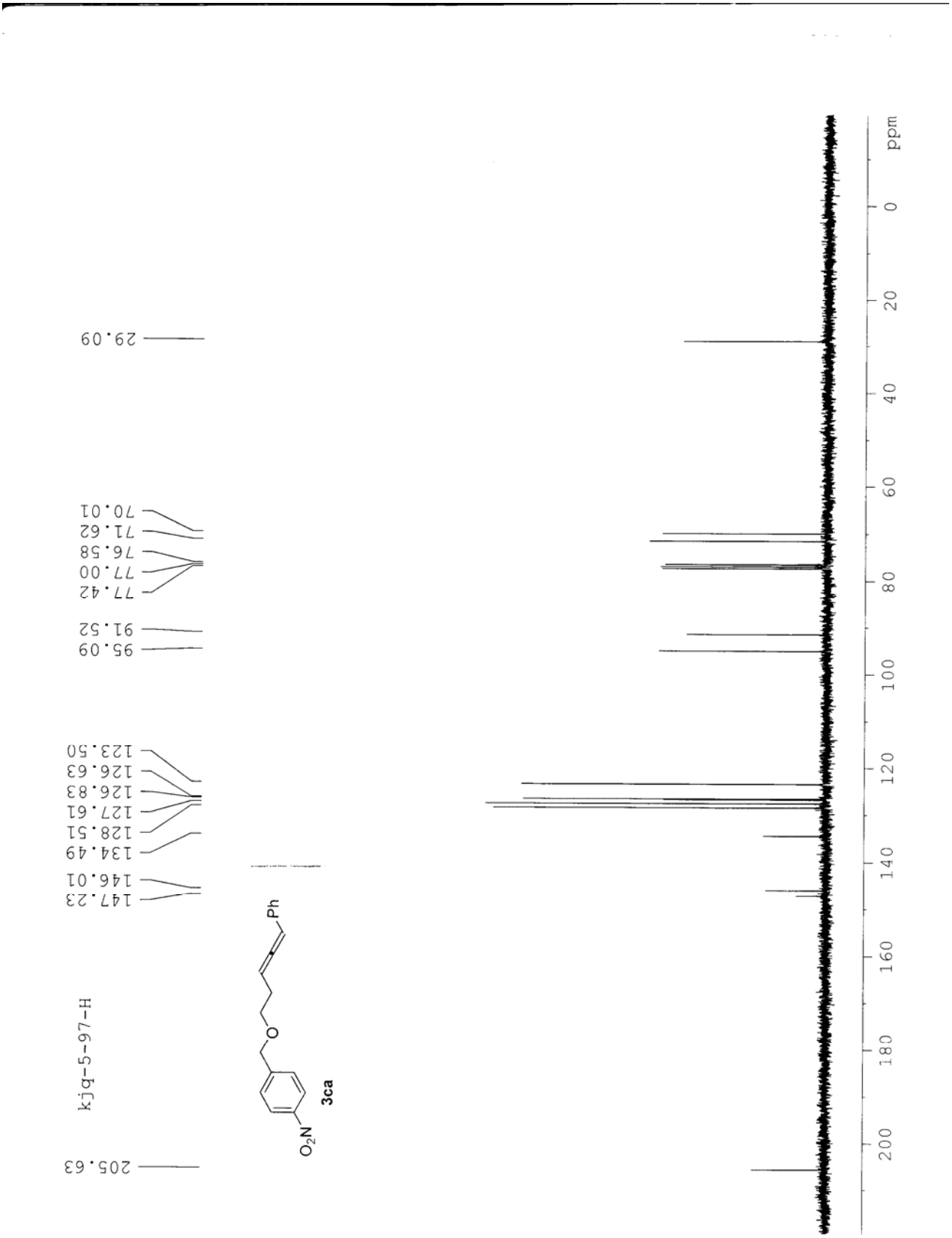

kjq-5-60-2-C
203.59
203.38
133.91
133.86
128.55
127.06
127.04
126.67
126.66
99.71
99.51
97.15
96.77
77.42
77.00
76.58
70.27
69.82
37.51
37.40
31.62
31.58
25.13
25.05
22.57
22.55
13.94
Ph
OH
n-C5H11
5b
ppm
0
20
40
60
80
100
120
140
160
180
200

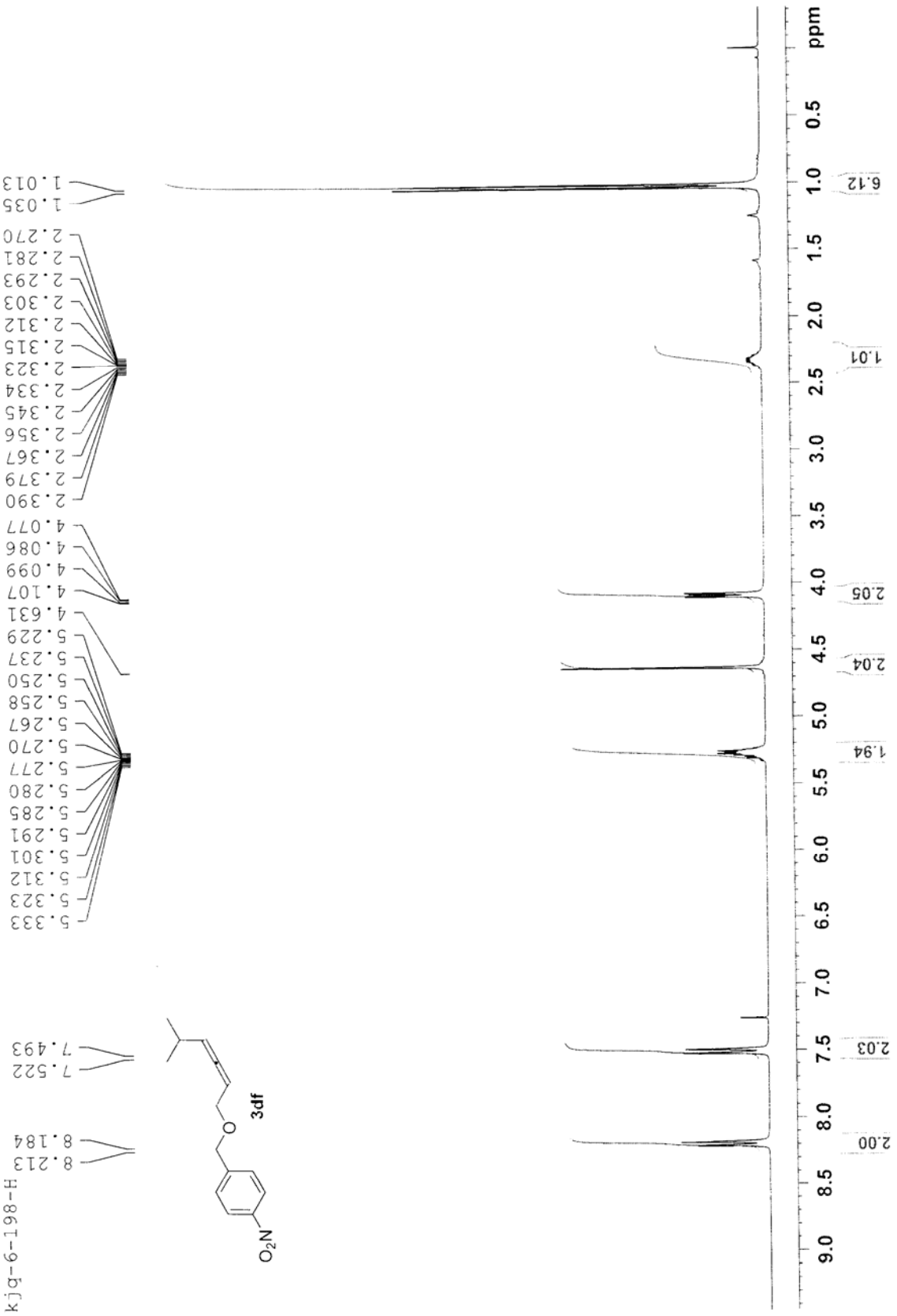

kjq-5-143-H
OH
n-C9H19
Ph
5c
ppm

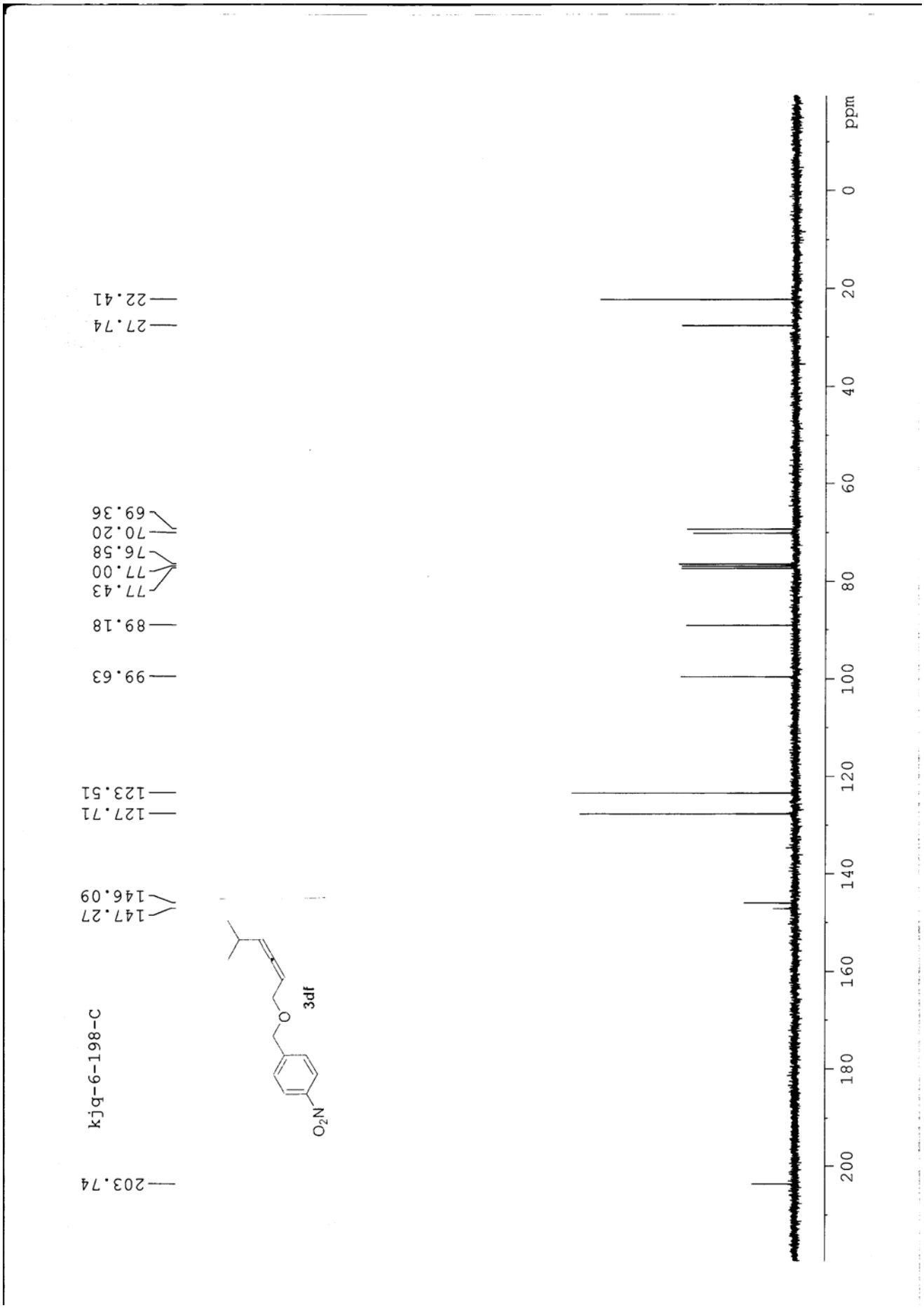
203.59
203.40
kjq-5-143-C
Ph
OH
n-C9H19
5c
133.92
133.87
128.53
127.03
127.01
126.66
99.69
99.49
97.11
96.75
77.42
77.00
76.57
70.26
69.83
37.56
37.45
31.81
29.57
29.54
29.46
29.42
29.25
25.46
25.39
22.60
14.03
ppm
0
20
40
60
80
100
120
140
160
180
200

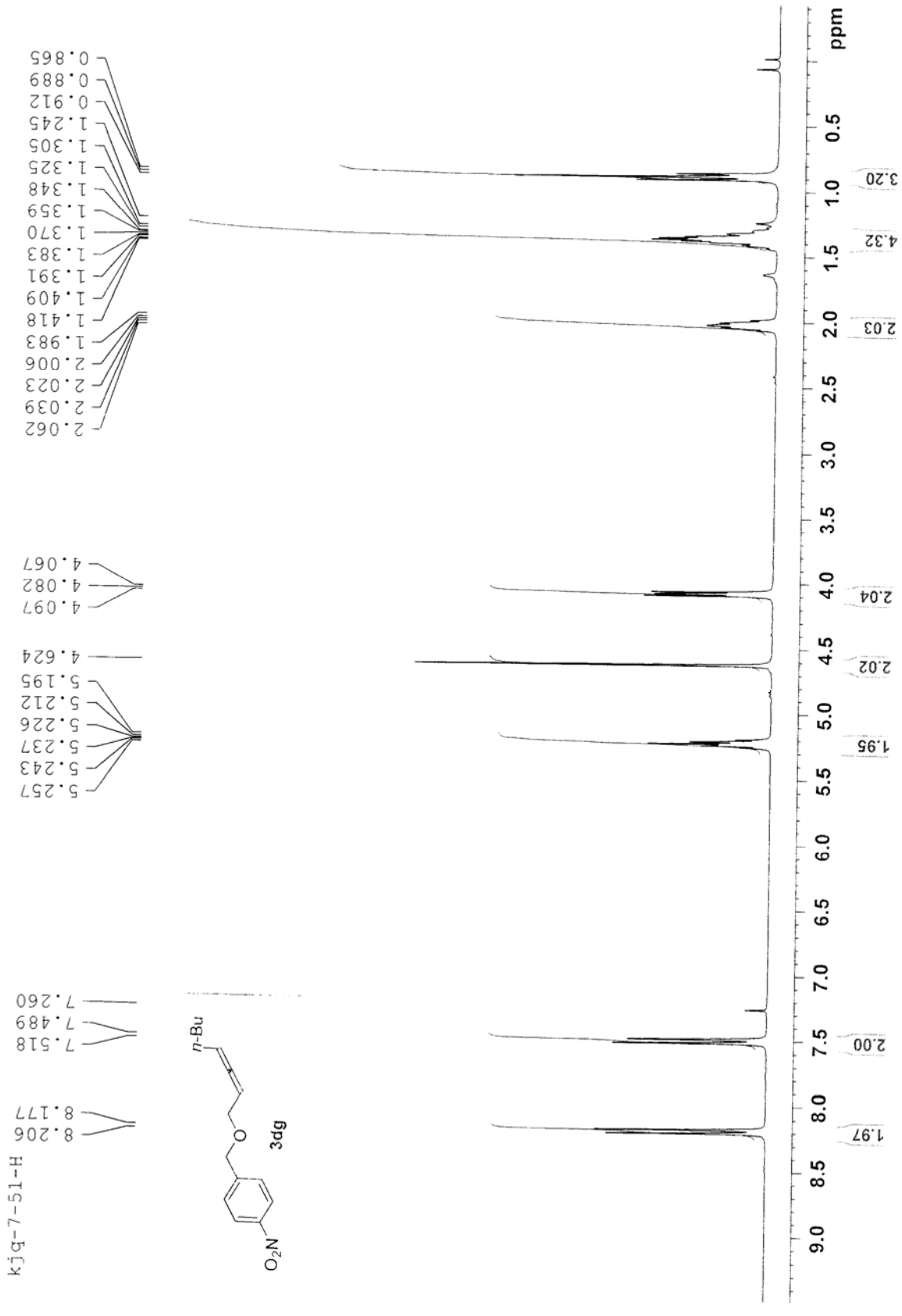
29.25
29.42
29.46
29.54
29.57
kjq-5-143-C
29.8
29.7
29.6
29.5
29.4
29.3
29.2
29.1
29.0
28.9
ppm

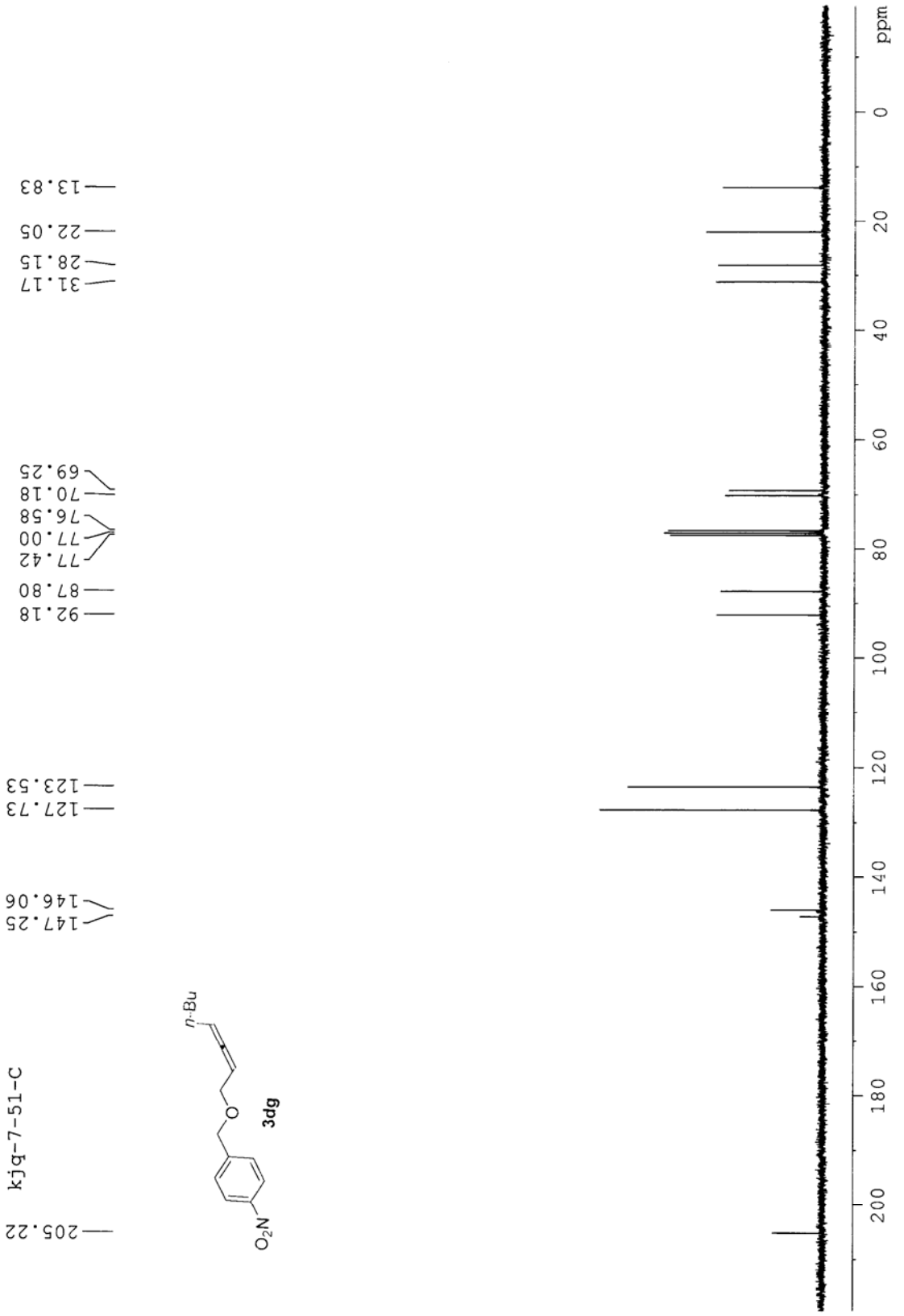

kjq-5-97-H
0.084
0.000
2.439
2.449
2.460
2.470
2.482
2.492
2.503
2.514
3.648
3.669
3.691
4.586
5.593
5.614
5.636
5.658
6.139
6.149
6.160
6.170
6.180
7.144
7.154
7.165
7.177
7.187
7.194
7.219
7.245
7.267
7.289
7.437
7.466
8.136
8.165
Ph
O
O_2N
3ca
ppm
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
2.07
2.08
2.06
1.00
0.98
1.05
4.24
2.05
2.01

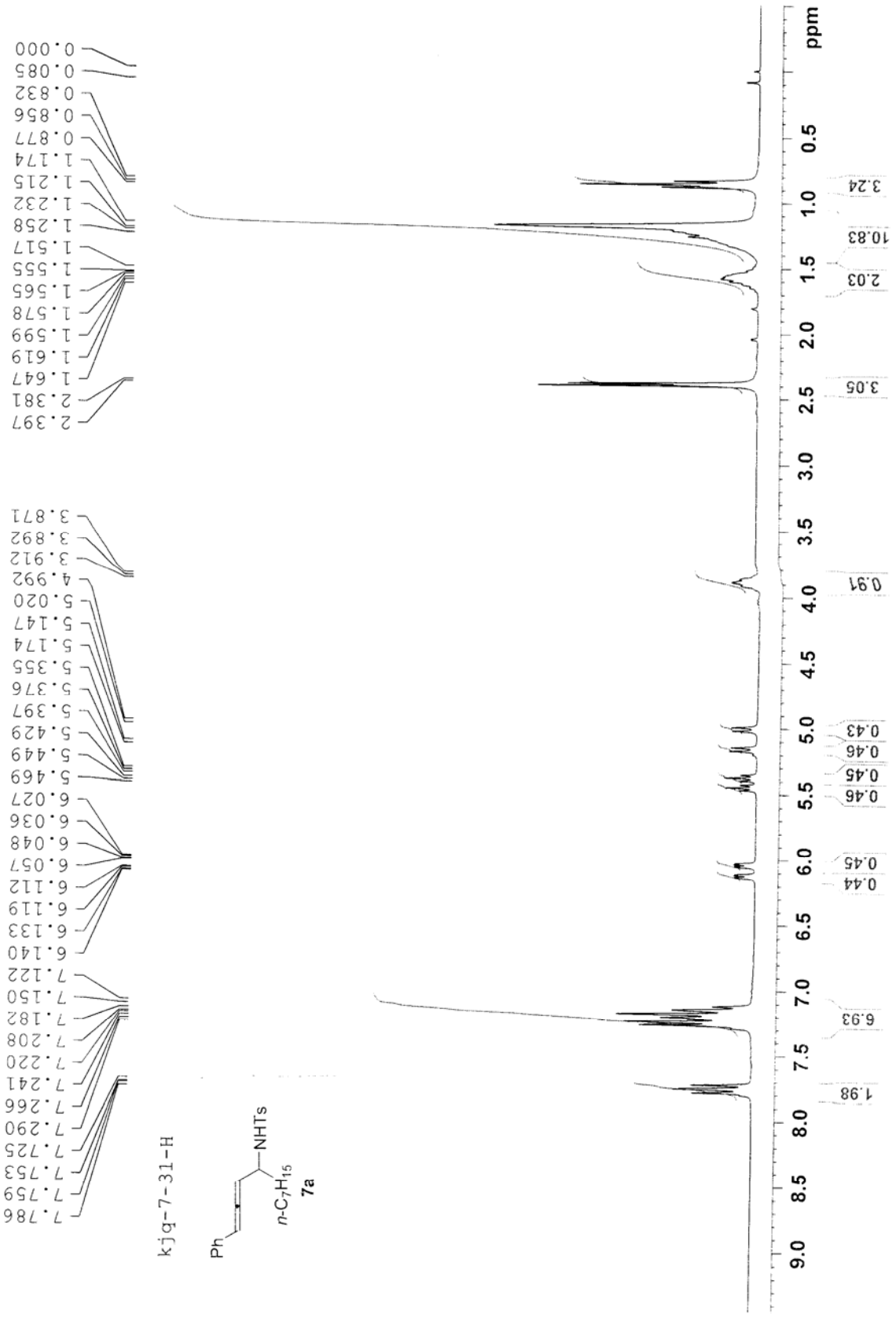

205.63
147.23
146.01
134.49
128.51
127.61
126.83
126.63
123.50
95.09
91.52
77.42
77.00
76.58
71.62
70.01
29.09
kjq-5-97-H
Ph
O
O2N
3ca
ppm
0
20
40
60
80
100
120
140
160
180
200


kjq-6-198-H
8.213
8.184
7.522
7.493
5.333
5.323
5.312
5.301
5.291
5.285
5.280
5.277
5.270
5.267
5.258
5.250
5.237
5.229
4.631
4.107
4.099
4.086
4.077
2.390
2.379
2.367
2.356
2.345
2.334
2.323
2.315
2.312
2.303
2.293
2.281
2.270
1.035
1.013
3df
O
O2N
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
6.12
1.01
2.05
2.04
1.94
2.03
2.00


kjq-6-198-C
3df
O2N
O
203.74
147.27
146.09
127.71
123.51
99.63
89.18
77.43
77.00
76.58
70.20
69.36
27.74
22.41
ppm
0
20
40
60
80
100
120
140
160
180
200


kjq-7-51-H
8.206
8.177
7.518
7.489
7.260
5.257
5.243
5.237
5.226
5.212
5.195
4.624
4.097
4.082
4.067
2.062
2.039
2.023
2.006
1.983
1.418
1.409
1.391
1.383
1.370
1.359
1.348
1.325
1.305
1.245
0.912
0.889
0.865
n-Bu
O
O2N
3dg
ppm
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
1.97
2.00
1.95
2.02
2.04
2.03
4.32
3.20

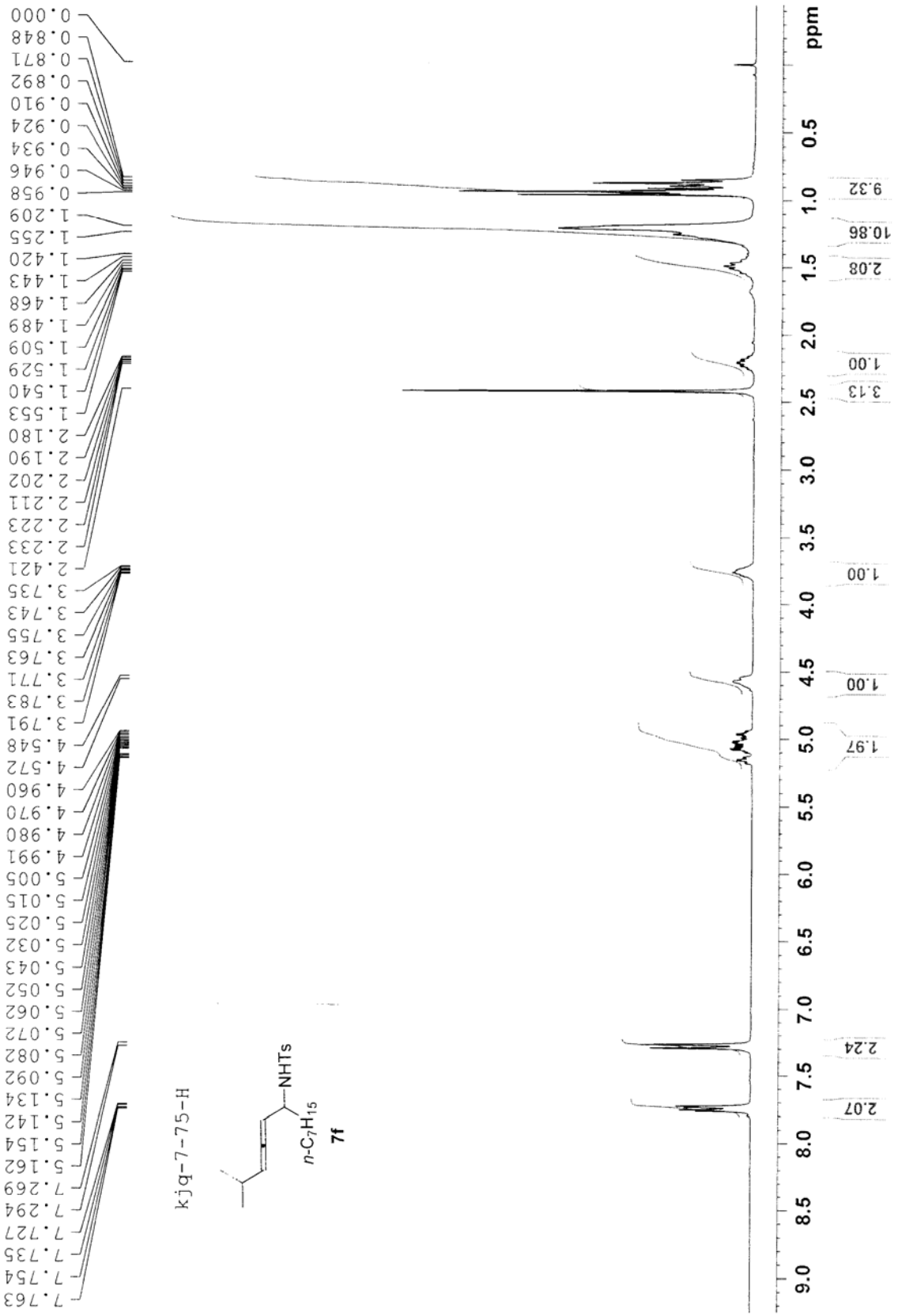

kjq-7-51-C
205.22
147.25
146.06
127.73
123.53
92.18
87.80
77.42
77.00
76.58
70.18
69.25
31.17
28.15
22.05
13.83
n-Bu
O
O2N
3dg
ppm
0
20
40
60
80
100
120
140
160
180
200


kjq-7-31-H
Ph
NHTs
n-C7H15
7a
ppm
0.000
0.085
0.832
0.856
0.877
1.174
1.215
1.232
1.258
1.517
1.555
1.565
1.578
1.599
1.619
1.647
2.381
2.397
3.871
3.892
3.912
4.992
5.020
5.147
5.174
5.355
5.376
5.397
5.429
5.449
5.469
6.027
6.036
6.048
6.057
6.112
6.119
6.133
6.140
7.122
7.150
7.182
7.208
7.220
7.241
7.266
7.290
7.725
7.753
7.759
7.786
1.98
6.93
0.44
0.45
0.46
0.45
0.46
0.43
0.91
3.05
2.03
10.83
3.24

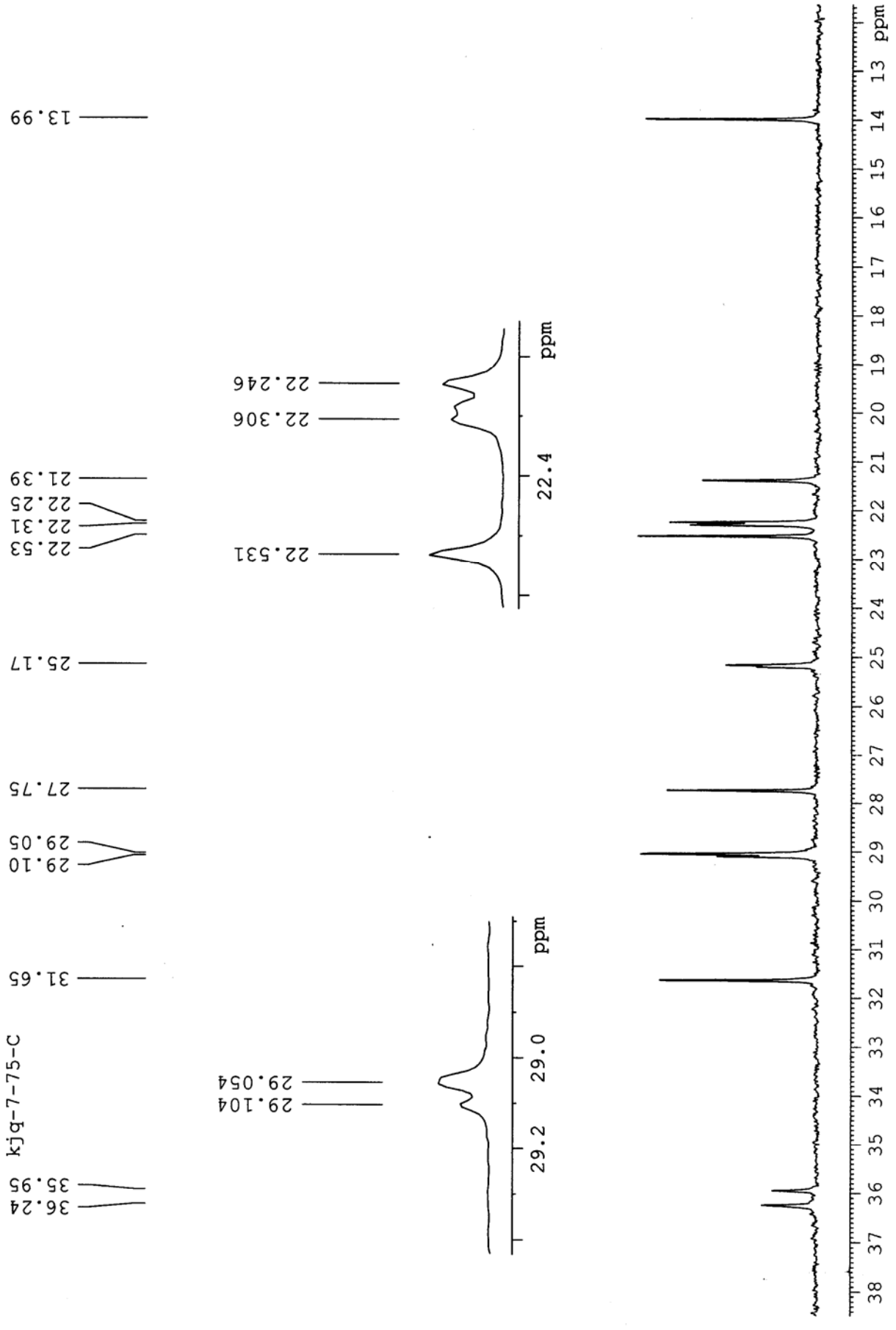
14.01
21.44
22.54
25.31
29.02
29.08
31.61
35.97
36.16
52.47
52.75
76.58
77.00
77.42
96.88
97.28
97.53
97.99
126.73
126.76
127.08
127.17
127.25
128.50
128.56
129.40
129.48
133.37
137.82
137.95
143.03
143.13
203.35
203.64
kjq-7-31-C
NHTs
n-C7H15
Ph
7a
ppm

143.13
143.03
137.95
137.82
kjq-7-31-C
133.37
129.48
129.40
128.56
128.50
127.25
127.17
127.08
126.76
126.73
145 144 143 142 141 140 139 138 137 136 135 134 133 132 131 130 129 128 127 126 ppm

14.01
21.44
22.54
25.31
29.02
29.08
31.61
35.97
36.16
kjq-7-31-C
ppm
15
20
25
30
35
40


kjq-7-75-H
NHTs
n-C7H15
7f
ppm
9.32
10.86
2.08
1.00
3.13
1.00
1.00
1.97
2.24
2.07

ppm
0
20
40
60
80
100
120
140
160
180
200
13.99
21.39
22.25
22.31
22.53
25.17
27.75
29.05
29.10
31.65
35.95
36.24
52.20
52.91
76.58
77.00
77.42
94.00
94.32
101.68
102.57
127.08
129.37
138.03
138.16
142.94
142.96
kjq-7-75-C
NHTs
n-C7H15
7f
200.30
200.88


kjq-7-75-C
13.99
22.246
22.306
22.531
21.39
22.25
22.31
22.53
25.17
27.75
29.10
29.05
31.65
29.104
29.054
36.24
35.95
ppm
22.4
29.2
29.0
38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22 21 20 19 18 17 16 15 14 13 ppm