Supporting Information

Amine-Promoted *anti*-Markovnikov Addition of 1,3-Dicarbonyl Compounds with Terminal Alkynes under Rhenium Catalysis

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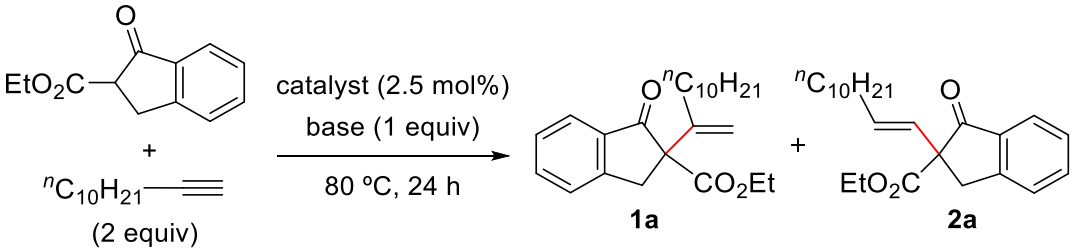
1. General Methods. All reactions were carried out in dry solvent under an argon atmosphere. Unless otherwise noted, other chemicals obtained from commercial suppliers were used without further purification. Octane was purchased from Wako Pure Chemical Industries, dried by the usual methods, and degassed with an argon gas for 20 min before use. Column chromatography was performed with silica gel 60N (neutral, 40-50 μm) purchased from Kanto Chemical. $Re_2(CO)_{10}$ and cyclohexylacethylene were purchased from Sigma-Aldrich. Ethyl 2-oxocyclopentanecarboxylate, 2-acetylcyclohexanone, 2-acetylcyclopentanone, 1-dodecyne, and phenylacetylene were purchased from Tokyo Chemical Industry. Ethyl 2-oxocyclohexanecarboxylate and 3-methyl-2,4-pentanedione were purchased from Wako Pure Chemical Industries. Ethyl 1-indanone-2-carboxylate,[1] ethyl 5-methoxy-1-indanone-2-carboxylate,[2] ethyl 1-tetralone-2-carboxylate,[3] ethyl 4-oxochroman- 3-carboxylate,[4] ethyl 2-oxotetrahydro-*2H*-pyran-3-carboxylate,[5] 2-benzoylcyclohexanone,[6] and $[ReBr(CO)_3(thf)]_2$[7] were synthesized according to the reported methods. ^1H (400 MHz) and ^{13}C (100 MHz) NMR spectra were recorded on a JEOL JNN-LA400 spectrometer. Proton chemical shifts are reported in ppm based on the solvent resonance resulting from incomplete deuteration ($CDCl_3$ at 7.26 ppm) as the internal standard. ^{13}C NMR was recorded with complete proton decoupling and the chemical shifts are reported relative to $CDCl_3$ at 77.00 ppm. The following abbreviations are used; brs: broad singlet, s: singlet, d: doublet, t: triplet, q: quartet, quint: quintet, m: multiplet. IR spectra were recorded on a SHIMADZU IRAFFINITY-1 100V J. High-resolution mass spectra (HRMS) was measured with JEOL JMS-700 MStation FAB-MS. Melting points were measured on a Yanaco micromelting point apparatus and are uncorrected.

2. Optimization of Reaction Conditions

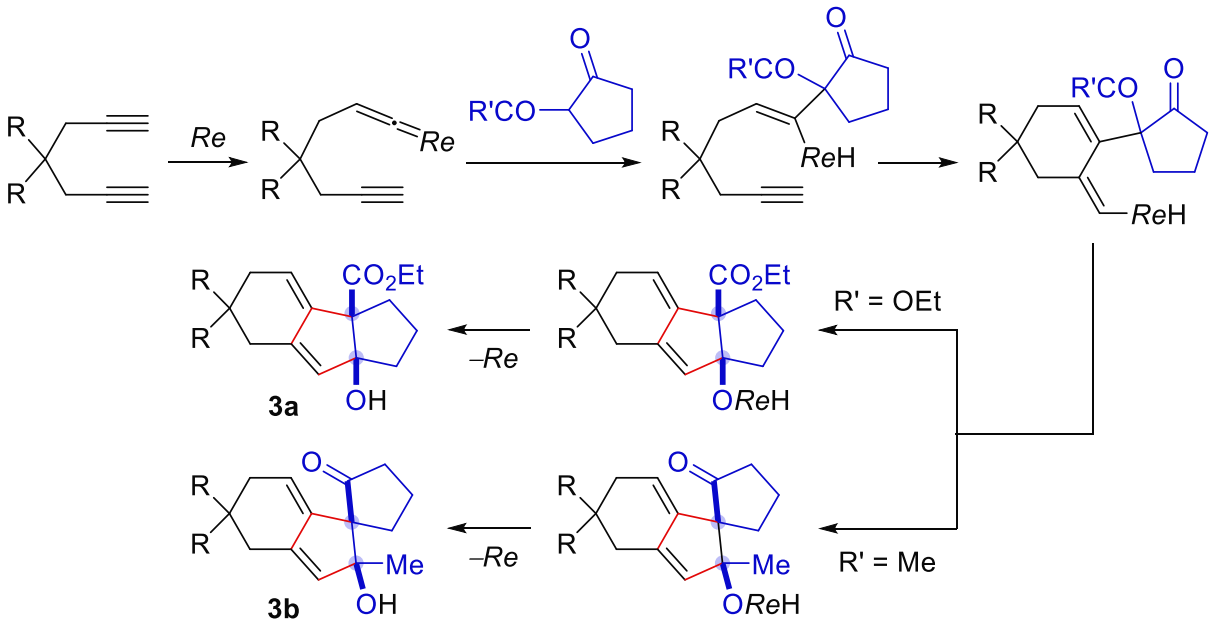
Table S1. Optimization of Reaction Conditions



Entry	catalyst	base	solvent	Yield / %	
				1a[a]	**2a**[a]
1	$[HRe(CO)_4]_n$	———	toluene (1 M)	27	31
2	$[HRe(CO)_4]_n$	———	octane (1 M)	57	28
3	$Re_2(CO)_{10}$	———	toluene (1 M)	0	2
4	$[ReBr(CO)_3(thf)]_2$	———	toluene (1 M)	94	0
5	$ReBr(CO)_5$	———	toluene (1 M)	92	0
6	$[HRe(CO)_4]_n$	Et_3N	toluene (1 M)	0	68
7	$Re_2(CO)_{10}$	Et_3N	toluene (1 M)	0	68
8	$[ReBr(CO)_3(thf)]_2$	Et_3N	toluene (1 M)	0	68
9	$ReBr(CO)_5$	Et_3N	toluene (1 M)	0	68
10	$ReI(CO)_5$	Et_3N	toluene (1 M)	0	62
11	$[ReBr(CO)_3(thf)]_2$	Et_3N	C_6H_5Cl (1 M)	0	61
12	$[ReBr(CO)_3(thf)]_2$	Et_3N	decane (1 M)	0	71
13	$[ReBr(CO)_3(thf)]_2$	Et_3N	^{n}octane (1 M)	0	78
14	$[ReBr(CO)_3(thf)]_2$	Et_3N	cyclohexane (1 M)	0	75
15	$[ReBr(CO)_3(thf)]_2$	Et_3N	$ClCH_2CH_2Cl$ (1 M)	0	26
16	$[ReBr(CO)_3(thf)]_2$	Et_3N	1,4-dioxane (1 M)	0	57
17	$[ReBr(CO)_3(thf)]_2$	Et_3N	MeCN (1 M)	1	21
18	$[ReBr(CO)_3(thf)]_2$	Et_3N	DMF (1 M)	0	7
19	$[ReBr(CO)_3(thf)]_2$	Et_3N	neat (1 M)	0	75
20	$[ReBr(CO)_3(thf)]_2$	$^n Bu_3N$	^{n}octane (1 M)	0	78
21	$[ReBr(CO)_3(thf)]_2$	$^i Pr_2NEt$	^{n}octane (1 M)	0	80
22	$[ReBr(CO)_3(thf)]_2$	DABCO	^{n}octane (1 M)	0	1
23	$[ReBr(CO)_3(thf)]_2$	Cy_2NH	^{n}octane (1 M)	0	71
24	$[ReBr(CO)_3(thf)]_2$	$^i Pr_2NH$	^{n}octane (1 M)	0	77
25	$[ReBr(CO)_3(thf)]_2$	$^n Bu_2NH$	^{n}octane (1 M)	0	2
26	$[ReBr(CO)_3(thf)]_2$	Et_2NH	^{n}octane (1 M)	0	2
27	$[ReBr(CO)_3(thf)]_2$	pyridine	^{n}octane (1 M)	0	0

28	$[ReBr(CO)_3(thf)]_2$	AcONa	^{n}octane (1 M)	34	34
29	$[ReBr(CO)_3(thf)]_2$	$NaHCO_3$	^{n}octane (1 M)	83	5
30	$[ReBr(CO)_3(thf)]_2$	Na_2CO_3	^{n}octane (1 M)	23	31
31	$[ReBr(CO)_3(thf)]_2$	^{t}BuOK	^{n}octane (1 M)	0	0
32	$[ReBr(CO)_3(thf)]_2$	P^{t}Bu$_3$	^{n}octane (1 M)	0	0
33	$[ReBr(CO)_3(thf)]_2$	PPh_3	^{n}octane (1 M)	0	0
34	$[ReBr(CO)_3(thf)]_2$	$P(OPh)_3$	^{n}octane (1 M)	0	0
35	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt (0.5 equiv)	^{n}octane (1 M)	2	78
36	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt (0.2 equiv)	^{n}octane (1 M)	5	77
37	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt (0.1 equiv)	^{n}octane (1 M)	8	63
38	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt	^{n}octane (2 M)	0	81
39	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt	^{n}octane (1 M)	0	80
40	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt	^{n}octane (0.5 M)	0	79
41	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt	^{n}octane (0.25 M)	0	86
42	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt	^{n}octane (0.125 M)	0	82
43[b]	$[ReBr(CO)_3(thf)]_2$	^{i}Pr$_2$NEt	^{n}octane (0.25 M)	0	96 (94)
44[b]	$[ReBr(CO)_3(thf)]_2$	——	^{n}octane (0.25 M)	96 (94)	0

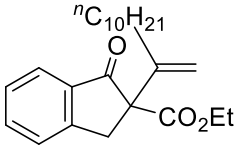
[a]Determined by ^{1}H NMR. Values in parentheses are isolated yields. [b]0.5 equiv of 1-dodecyne was added at the beginning and additional 1-dodecyne was added three times (0.5 equiv each) at intervals of 3 h.



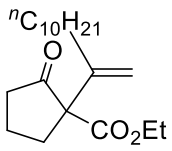
Scheme S1. Reaction Mechanism for Sequential Cyclization Initiated by *anti*-Markovnikov Addition to 1,6-Diynes (see Scheme 2 in the main text, R = CO_2Et)

3. General Procedure for Rhenium-Catalyzed *anti*-Markovnikov Addition Reaction of Terminal Alkynes with Carbon Nucleophiles

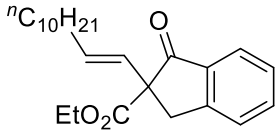
A flame dried test tube was charged with $[ReBr(CO)_3(thf)]_2$ (4.2 mg, 0.010 mmol), carbon nucleophiles (0.20 mmol), terminal alkynes (0.10 mmol), *N*,*N*-diisopropylethylamine (0.20 mmol, 25.9 mg), and octane (0.80 mL), and then the resulting mixture was stirred at 80 ^{o}C for 2 h. Additional alkyne was added three times at the interval of 2 h (0.10 mmol×3). The solvent was removed under reduced pressure and the residue was subjected to flash column chromatography on silica gel with hexane / ethyl acetate (*v* / *v* = 20 / 1) as the eluent to afford the corresponding *anti*-Markovnikov adducts **2**.



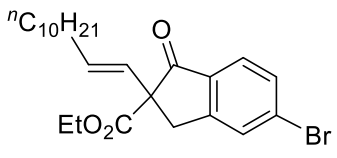
Ethyl 2-((*E*)-1-dodecen-2-yl)-1-indanone-2-carboxylate (1a): Following the general procedure using ethyl 1-indanone-2-carboxylate (40.8 mg, 0.20 mmol) and 1-dodecyne (66.5 mg, 0.40 mmol) without *N*,*N*-diisopropylethyl- amine provided 69.5 mg (0.19 mmol, 94% yield) of **1a** as a yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, *J* = 7.0 Hz, 3H), 1.20-1.32 (m, 17H), 1.46-1.54 (m, 2H), 2.10 (t, *J* = 8.0 Hz, 2H), 3.25 (d, *J* = 17.2 Hz, 1H), 3.98 (d, *J* = 17.2 Hz, 1H), 4.16-4.28 (m, 2H), 5.00 (s, 1H), 5.04 (t, *J* = 1.6 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.62 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.78 (d, *J* = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 22.7, 28.0, 29.3, 29.4, 29.5, 29.6 (two peaks overlapped), 31.9, 33.2, 38.3, 62.0, 67.6, 112.1, 124.9, 126.2, 127.7, 135.2, 135.4, 146.6, 152.2, 169.9, 200.1. IR (neat / cm^{-1}): 2955, 2926, 2853, 1746, 1715, 1607, 1464, 1273, 1246, 1211, 1179, 1055, 901, 750. HRMS (FAB^{+}): calcd for $C_{24}H_{35}O_3$ ($[M+H]^{+}$) 371.2586; found. 371.2574.



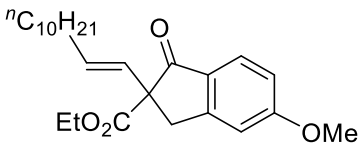
Ethyl 2-oxo-1-((*E*)-1-dodecen-2-yl)cyclopentanecarboxylate (1d): Following the general procedure using ethyl 2-oxocyclopentanecarboxylate (31.2 mg, 0.20 mmol) and 1-dodecyne (66.5 mg, 0.40 mmol) without *N*,*N*-diisopropylethylamine provided 61.2 mg (0.19 mmol, 95% yield) of **1d** as a yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 7.2 Hz, 3H), 1.20-1.31 (m, 17H), 1.42-1.48 (m, 2H), 1.89-1.98 (m, 2H), 2.02 (dd, J = 7.2, 12.8 Hz, 2H), 2.24 (quintet, J = 6.8 Hz, 1H), 2.33 (t, J = 8.0 Hz, 2H), 2.56 (quintet, J = 6.8 Hz, 1H), 4.15-4.23 (m, 2H), 4.94 (s, 1H), 5.08 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 19.2, 22.7, 28.1, 29.1, 29.3, 29.4, 29.5, 29.6, 31.9, 32.7, 33.1, 37.8, 61.6, 67.4, 113.0, 144.6, 170.4, 212.5. IR (neat / cm^{-1}): 2955, 2926, 2855, 1751, 1724, 1636, 1458, 1248, 1159, 1134, 1096, 1026, 899. HRMS (FAB^+): calcd for $C_{20}H_{35}O_3$ ($[M+H]^+$) 323.2586; found. 323.2584.



Ethyl 2-((*E*)-1-dodecen-1-yl)-1-indanone-2-carboxylate (2a): Following the general procedure using ethyl 1-indanone-2-carboxylate (40.8 mg, 0.20 mmol) and 1-dodecyne (66.5 mg, 0.40 mmol) provided 69.6 mg (0.19 mmol, 94% yield) of **2a** as a yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, J = 6.8 Hz, 3H), 1.20-1.40 (m, 19H), 2.03 (q, J = 7.6 Hz, 2H), 3.33 (d, J = 17.2 Hz, 1H), 3.83 (d, J = 17.2 Hz, 1H), 4.18 (q, J = 3.2 Hz, 2H), 5.60 (dt, J = 15.6, 6.8 Hz, 1H), 5.98 (dt, J = 1.6, 15.6 Hz, 1H), 7.39 (t, J = 7.2 Hz, 1H), 7.48 (d, J = 7.2 Hz, 1H), 7.62 (td, J = 7.2, 0.8 Hz, 1H), 7.77 (d, J = 7.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 22.7, 29.0, 29.1, 29.3, 29.4, 29.5, 29.6, 31.9, 32.6, 37.8, 61.8, 62.6, 125.1, 126.2, 126.8, 127.7, 132.6, 134.5, 135.2, 152.6, 170.6, 200.6. IR (neat / cm^{-1}): 2955, 2926, 2855, 1744, 1717, 1608, 1464, 1271, 1250, 1211, 1188, 1096, 1011, 970, 750. HRMS (FAB^+): calcd for $C_{24}H_{35}O_3$ ($[M+H]^+$) 371.2586; found. 371.2574.

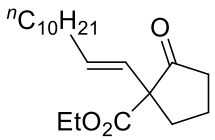


Ethyl 5-bromo-2-((*E*)-1-dodecen-1-yl)-1-indanone-2-carboxylate (2b): Following the general procedure using ethyl 5-bromo-1-indanone-2-carboxylate (56.6 mg, 0.20 mmol) and 1-dodecyne (66.5 mg, 0.40 mmol) provided 79.7 mg (0.18 mmol, 89% yield) of **2b** as a pale yellow solid after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). mp 42.5-43.4 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, J = 6.8 Hz, 3H), 1.18-1.36 (m, 19H), 2.03 (q, J = 6.8 Hz, 2H), 3.30 (d, J = 17.2 Hz, 1H), 3.79 (d, J = 17.2 Hz, 1H), 4.18 (q, J = 7.2 Hz, 2H), 5.58 (dt, J = 6.8, 15.6 Hz, 1H), 5.96 (d, J = 15.6 Hz, 1H), 7.53 (d, J = 8.0 Hz, 1H), 7.62 (d, J = 8.0 Hz, 1H), 7.67 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 22.7, 28.9, 29.1, 29.3, 29.4, 29.5, 29.6, 31.9, 32.6, 37.3, 62.0, 62.6, 126.2, 126.4, 129.6, 130.7, 131.5, 132.9, 133.3, 154.1, 170.2, 199.3. IR (KBr / cm^{-1}): 2957, 2922, 2853, 1717, 1595, 1558, 1508, 1466, 1233, 1182, 1159, 899. HRMS (FAB$^+$): calcd for $C_{24}H_{33}BrO_3$ ([M+H]$^+$) 449.1691; found. 449.1706.

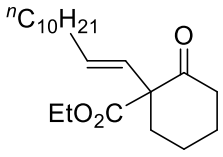


Ethyl 2-((*E*)-1-dodecen-1-yl)-5-methoxy-1-indanone-2-carboxylate (2c): Following the general procedure using ethyl 5-methoxy-1-indanone-2-carboxylate (46.8 mg, 0.20 mmol) and 1-dodecyne (66.5 mg, 0.40 mmol) provided 50.4 mg (0.13 mmol, 63% yield) of **2c** as a pale yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). In this reaction, the corresponding Markovnikov adduct, ethyl 2-((*E*)-1-dodecen-2-yl)-5-methoxy-1-indanone-2- carboxylate, was also obtained in 7% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, J = 6.8 Hz, 3H), 1.19-1.31 (m, 19H), 2.03 (d, J = 7.2 Hz, 2H), 3.26 (d, J = 17.2 Hz, 1H), 3.76 (d, J = 17.2 Hz, 1H), 3.88 (s, 3H), 4.18 (q, J = 7.6 Hz, 2H), 5.59 (dt, J = 7.2, 15.6 Hz, 1H), 6.00 (dt, J = 1.2, 15.6 Hz, 1H), 6.90 (s, 1H), 6.91 (d, J = 6.8 Hz, 1H), 7.70 (d, J = 6.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 22.7, 29.0, 29.1, 29.3, 29.4, 29.6 (two peaks overlapped), 31.9, 32.6, 37.7, 55.7, 61.8, 62.8, 109.4, 115.8, 126.8, 127.2,

127.5, 132.1, 155.7, 165.7, 170.8, 198.8. IR (neat / cm^{-1}): 2926, 2853, 1742, 1709, 1601, 1491, 1447, 1339, 1300, 1263, 1188, 1092, 1026, 930, 845. HRMS (FAB^+): calcd for $C_{25}H_{37}O_4$ ($[M+H]^+$) 401.26918; found. 401.2705.

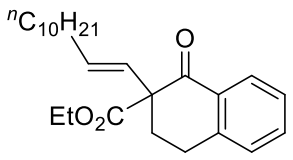


Ethyl 2-oxo-1-((*E*)-1-dodecen-1-yl)cyclopentanecarboxylate (2d): Following the general procedure using ethyl 2-oxocyclopentanecarboxylate (31.2 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and *N*,*N*-diisopropylethylamine (1.0 mmol, 129.3 mg) provided 36.1 mg (0.11 mmol, 56% yield) of **2d** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^1H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, J = 7.2 Hz, 3H), 1.22-1.29 (m, 17H), 1.29-1.37 (m, 2H), 1.86-2.02 (m, 2H), 2.04 (t, J = 7.2 Hz, 1H), 2.06 (t, J = 7.2 Hz, 1H), 2.13 (dt, J = 7.2, 13.2 Hz, 1H), 2.25-2.42 (m, 2H), 2.57 (dt, J = 7.2, 13.2 Hz, 1H), 4.16 (q, J = 7.2 Hz, 2H), 5.54-5.65 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 19.4, 22.7, 28.9, 29.0, 29.3, 29.4, 29.6 (two peaks overlapped), 31.9, 32.6, 33.6, 37.5, 61.5, 62.6, 125.9, 133.5, 170.7, 212.8. IR (neat / cm^{-1}): 2953, 2926, 2855, 1734, 1717, 1558, 1506, 1458, 1418, 1236, 1177, 1096, 1024. HRMS (FAB^+): calcd for $C_{20}H_{34}O_3$ ($[M+H]^+$) 323.2586; found. 323.2581.

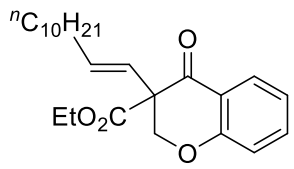


Ethyl 2-oxo-1-((*E*)-1-dodecen-1-yl)cyclohexanecarboxylate (2e): Following the general procedure using ethyl 2-oxocyclohexanecarboxylate (34.0 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and *N*,*N*-diisopropylethylamine (1.0 mmol, 129.3 mg) provided 43.7 mg (0.13 mmol, 65% yield) of **2e** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^1H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, J = 6.8 Hz, 3H), 1.22-1.36 (m, 19H), 1.61-1.82 (m, 4H), 1.94-2.01 (m, 1H), 2.05 (q, J = 6.8 Hz, 2H), 2.35-2.43 (m, 1H), 2.47-2.52 (m, 1H), 2.58-2.61 (m, 1H), 4.14-4.25 (m, 2H), 5.51 (dt, J = 6.8, 16.0 Hz, 1H), 5.85 (d, J = 16.0, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 22.5, 22.7, 27.3, 29.0, 29.1, 29.3, 29.4, 29.6 (two

peaks overlapped), 31.9, 32.8, 36.2, 40.8, 61.4, 62.5, 127.2, 133.0, 170.7, 206.7. IR (neat / cm^{-1}): 2926, 2855, 1717, 1464, 1452, 1439, 1366, 1310, 1238, 1201, 1090, 1022, 970. HRMS (FAB^+): calcd for $C_{21}H_{37}O_3$ ($[M+H]^+$) 337.2743; found. 337.2759.

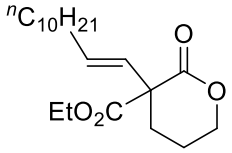


Ethyl 2-((*E*)-1-dodecen-1-yl)-1-tetralone-2-carboxylate (2f): Following the general procedure using ethyl 1-tetralone-2-carboxylate (43.6 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and *N*,*N*-diisopropylethylamine (1.0 mmol, 129.3 mg) provided 43.0 mg (0.11 mmol, 56% yield) of **2f** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^1H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 6.8 Hz, 3H), 1.18-1.38 (m, 19H), 2.07 (q, J = 6.8 Hz, 2H), 2.17-2.22 (m, 2H), 2.94-3.08 (m, 2H), 4.16 (q, J = 7.2 Hz, 2H), 5.54 (dt, J = 6.4, 16.0 Hz, 1H), 5.85 (d, J = 16.0, 1H), 7.21 (d, J = 7.6 Hz, 1H), 7.31 (t, J = 7.6 Hz, 1H), 7.47 (t, J = 7.6 Hz, 1H), 8.07 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 22.7, 25.7, 28.9, 29.0, 29.1, 29.3, 29.4, 29.6 (two peaks overlapped), 31.9, 32.8, 59.8, 61.5, 126.3, 126.7, 128.0, 128.6, 131.9, 133.5, 133.9, 143.1, 171.1, 194.4. IR (neat / cm^{-1}): 2955, 2924, 2853, 1732, 1684, 1601, 1456, 1298, 1240, 1226, 1020, 918, 741. HRMS (FAB^+): calcd for $C_{25}H_{37}O_3$ ($[M+H]^+$) 385.2743; found. 385.2753.

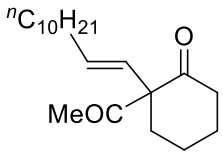


Ethyl 3-((*E*)-1-dodecen-1-yl)-4-oxochroman-3-carboxylate (2g): Following the general procedure using ethyl 4-oxochroman-3-carboxylate (44.0 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and *N*,*N*-diisopropylethylamine (1.0 mmol, 129.3 mg) provided 43.2 mg (0.13 mmol, 64% yield) of **2g** as a yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^1H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 6.8 Hz, 3H), 1.18 (t, J = 7.2 Hz, 3H), 1.22-1.37 (m, 16H), 2.07 (t, J = 6.8 Hz, 1H), 2.08 (t, J = 6.8 Hz, 1H), 4.18 (q, J = 7.2 Hz, 2H), 4.37 (d, J = 11.2 Hz, 1H), 4.92 (d, J = 11.2 Hz, 1H), 5.72-5.83 (m, 2H), 6.95 (d, J = 8.0 Hz, 1H), 7.03 (dt, J = 1.2, 8.0 Hz, 1H), 7.47 (ddd, J = 1.6, 7.2, 8.6 Hz,

1H), 7.94 (dd, J = 1.6, 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9, 14.1 22.7, 28.8, 29.0, 29.3, 29.4, 29.5, 29.6, 31.9, 32.9, 58.6, 62.1, 72.0, 117.7, 120.0, 121.7, 122.0, 127.9, 135.9, 136.0, 160.8, 168.5, 188.5. IR (neat / cm^{-1}): 2955, 2926, 2855, 1738, 1699, 1607, 1479, 1464, 1304, 1217, 1038, 1020, 760. HRMS (FAB^+): calcd for $C_{24}H_{34}O_4$ ([M]) 336.2457; found. 336.2456.

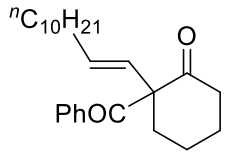


Ethyl 3-((*E*)-1-dodecen-1-yl)-2-oxotetrahydro-*2H*-pyran-3-carboxylate (2h): Following the general procedure using ethyl 2-oxotetrahydro-*2H*-pyran-3-carboxylate (34.4 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and 1,4-dioxane (0.20 mL) at 120 ^{o}C provided 27.1 mg (0.08 mmol, 40% yield) of **2h** as a yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 10 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 7.2 Hz, 3H), 1.25-1.31 (m, 17H), 1.31-1.39 (m, 2H), 1.88-1.98 (m, 3H), 2.07 (t, J = 6.8 Hz, 1H), 2.09 (t, J = 6.8 Hz, 1H), 2.52-2.59 (m, 1H), 4.23 (q, J = 7.2 Hz, 2H), 4.24-4.39 (m, 2H), 5.61 (dt, J = 6.8, 16.0 Hz, 1H), 5.83 (d, J = 16.0, Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 14.1, 19.8, 22.7, 28.9, 29.0, 29.1, 29.3, 29.4, 29.6 (two peaks overlapped), 31.9, 32.6, 56.3, 62.2, 68.9, 127.2, 133.7, 169.2, 170.2. IR (neat / cm^{-1}): 2955, 2926, 2855, 1734, 1701, 1558, 1506, 1456, 1261, 1242, 1207, 1155, 1088, 1043, 968, 729. HRMS (FAB^+): calcd for $C_{20}H_{34}O_4$ ([M]) 338.2457; found. 338.2470.

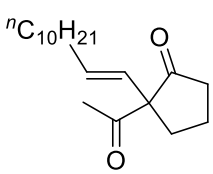


2-Acetyl-2-((*E*)-1-dodecen-1-yl)cyclohexanone (2i): Following the general procedure using 2-acetylcyclohexanone (28.0 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and octane (0.20 mL) provided 49.0 mg (0.16 mmol, 80% yield) of **2i** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 6.8 Hz, 3H), 1.24-1.40 (m, 16H), 1.65-1.79 (m, 4H), 1.86-1.96 (m, 1H), 2.07 (q, J = 6.8 Hz, 2H), 2.10 (s,

3H), 2.22-2.29 (m, 1H), 2.46-2.58 (m, 2H), 5.47 (dt, J = 6.8, 16.0 Hz, 1H), 5.89 (dt, J = 1.6, 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 22.1, 22.7, 26.9, 27.1, 29.0, 29.1, 29.3, 29.4, 29.6 (two peaks overlapped), 31.9, 32.9, 34.8, 41.2, 69.3, 127.6, 134.6, 204.6, 208.9. IR (neat / cm^{-1}): 2926, 2854, 1701, 1458, 1433, 1354, 1314, 1233, 1194, 1173, 1126, 974. HRMS (FAB$^+$): calcd for $C_{20}H_{35}O_2$ ([M+H]$^+$) 307.2637; found. 307.2644.

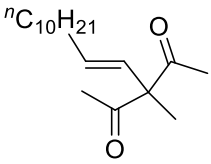


2-Benzoyl-2-((*E*)-1-dodecen-1-yl))cyclohexanone (2j): Following the general procedure using 2-benzoylcyclohexanone (40.4 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and octane (0.20 mL) provided 44.2 mg (0.12 mmol, 60% yield) of **2j** as a pale yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, J = 6.8 Hz, 3H), 1.10-1.34 (m, 16H), 1.59-1.72 (m, 3H), 1.80-1.90 (m, 1H), 1.96-2.06 (m, 4H), 2.31-2.37 (m, 1H), 2.76-2.83 (m, 1H), 5.38 (dt, J = 6.8, 16.0 Hz, 1H), 6.44 (d, J = 16.0, 1H), 7.36 (t, J = 7.6 Hz, 2H), 7.48 (t, J = 7.6 Hz, 1H), 7.82 (d, J = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 22.7 (two peaks overlapped), 28.8, 28.9, 29.0, 29.3, 29.4, 29.6 (two peaks overlapped), 31.9, 32.9, 40.3, 42.7, 68.0, 128.1, 128.4, 129.5, 132.8, 133.8, 136.0, 195.7, 209.9. IR (neat / cm^{-1}): 2924, 2853, 1717, 1678, 1597, 1580, 1449, 1435, 1240, 1213, 1182, 1225, 974, 770, 733, 690. HRMS (FAB$^+$): calcd for $C_{25}H_{36}O_2$ ([M]$^+$) 368.2715; found. 368.2725.

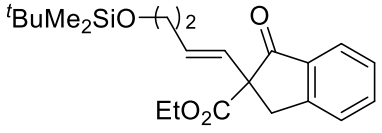


2-Acetyl-2-((*E*)-1-dodecen-1-yl)cyclopentanone (2k): Following the general procedure using 2-acetylcyclopentanone (25.2 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and octane (0.20 mL) provided 40.9 mg (0.14 mmol, 70% yield) of **2k** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 7.2 Hz, 3H), 1.22-1.37 (m, 16H), 1.83-1.96 (m, 3H), 2.05 (q, J = 7.2 Hz, 2H), 2.21 (s, 3H), 2.28 (t, J = 7.2

Hz, 2H), 2.65-2.72 (m, 1H), 5.58 (dt, J = 15.6, 6.8 Hz, 1H), 5.68 (d, J = 15.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 19.2, 22.7, 26.8, 29.0, 29.1, 29.3, 29.4, 29.6 (two peaks overlapped), 31.7, 31.9, 32.7, 38.1, 70.9, 127.2, 134.4, 203.3, 214.1. IR (neat / cm^{-1}): 2957, 2926, 2855, 1742, 1709, 1458, 1354, 1207, 1152, 1134, 1105, 978. HRMS (FAB$^+$): calcd for $C_{19}H_{33}O_2$ ([M+H]$^+$) 293.2481; found. 293.2469.

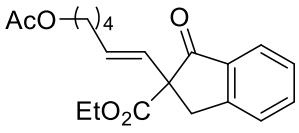


3-((*E*)-1-Dodecen-1-yl)-3-methyl-2,4-pentanedione (2l): Following the general procedure using 3-methyl-2,4-pentanedione (22.8 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and octane (0.20 mL) provided 11.3 mg (0.040 mmol, 20% yield) of **2l** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 7.2 Hz, 3H), 1.20-1.30 (m, 14H), 1.35-1.39 (m, 2H), 1.43 (s, 3H), 2.09 (q, J = 7.2 Hz, 2H), 2.13 (s, 6H), 5.58 (dt, J = 6.8, 16.0 Hz, 1H), 5.95 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 18.6, 22.7, 26.9, 29.0, 29.1, 29.3, 29.4, 29.6 (two peaks overlapped), 31.9, 32.9, 68.2, 127.4, 134.3, 206.3. IR (neat / cm^{-1}): 2926, 2855, 1717, 1701, 1684, 1653, 1558, 1506, 1456, 1362, 1217, 1089, 974. HRMS (FAB$^+$): calcd for $C_{18}H_{32}O_2$ ([M+H]$^+$) 281.2481; found. 281.2472.

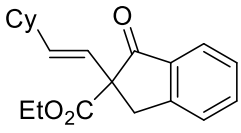


Ethyl 2-((*E*)-4-(*tert*-butyldimethylsilyl)oxy-1-buten-1-yl)-1-indanone-2-carboxylate (2m): Following the general procedure using ethyl 1-indanone-2-carboxylate (40.8 mg, 0.20 mmol) and (3-butyn-1-yloxy)(*tert*-butyl)dimethylsilane (73.6 mg, 0.40 mmol) provided 72.7 mg (0.19 mmol, 94% yield) of **2m** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ -0.03 (s, 3H), -0.05 (s, 3H) , 0.82 (t, J = 2.8 Hz, 9H), 1.23 (t, J = 7.2 Hz, 3H), 2.27 (qd, J = 6.8, 1.6 Hz, 2H), 3.33 (d, J = 16.8 Hz, 1H), 3.53-3.63 (m, 2H), 3.80 (d, J = 16.8 Hz, 1H), 4.18 (q, J = 7.2

Hz, 2H), 5.61 (dt, J = 6.8, 16.0 Hz, 1H), 6.07 (d, J = 16.0 Hz, 1H), 7.39 (t, J = 7.6 Hz, 1H), 7.48 (d, J = 7.6 Hz, 1H), 7.61 (dt, J = 0.8, 7.6 Hz, 1H), 7.76 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ -5.4, 14.0, 18.2, 25.9, 36.2, 37.7, 61.9, 62.6 (two peaks overlapped), 125.2, 126.2, 127.8, 128.7, 128.8, 134.3, 135.3, 152.5, 170.5, 200.4. IR (neat / cm^{-1}): 2953, 2928, 2857, 1744, 1717, 1609, 1456, 1387, 1362, 1254, 1209, 1188, 1099, 853, 777. HRMS (FAB^+): calcd for $C_{22}H_{33}O_4Si$ ($[M+H]^+$) 388.2148; found. 388.2167.

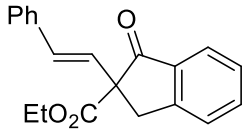


Ethyl 2-((*E*)-6-acetoxy-1-hexen-1-yl)-1-indanone-2-carboxylate (2n): Following the general procedure using ethyl 1-indanone-2-carboxylate (40.8 mg, 0.20 mmol) and 5-hexyn-1-yl acetate (56.0 mg, 0.40 mmol) provided 55.1 mg (0.16 mmol, 80% yield) of **2n** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 5 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.22 (t, J = 6.8 Hz, 3H), 1.39-1.44 (m, 2H), 1.54-1.61 (m, 2H), 2.02 (s, 3H), 2.08 (q, J = 7.2 Hz, 2H), 3.32 (d, J = 17.2 Hz, 1H), 3.81 (d, J = 17.2 Hz, 1H), 4.01 (t, J = 7.2 Hz, 2H), 4.18 (q, J = 7.2 Hz, 2H), 5.59 (dt, J = 16.0, 6.8 Hz, 1H), 6.01 (dt, J = 16.0, 1.2 Hz, 1H), 7.40 (d, J = 7.6 Hz, 1H), 7.49 (d, J = 7.6 Hz, 1H), 7.62 (t, J = 7.6 Hz, 1H), 7.77 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 21.0, 25.3, 28.0, 32.1, 37.8, 61.9, 62.5, 64.3, 125.1, 126.2, 127.5, 127.8, 131.7, 134.4, 135.3, 152.5, 170.6, 171.2, 200.5. IR (neat / cm^{-1}): 2980, 2938, 2860, 1738, 1717, 1607, 1464, 1437, 1387, 1366, 1244, 1211, 1188, 1038, 972, 910, 752. HRMS (FAB^+): calcd for $C_{20}H_{25}O_5$ ($[M+H]^+$) 345.1702; found. 345.1699.

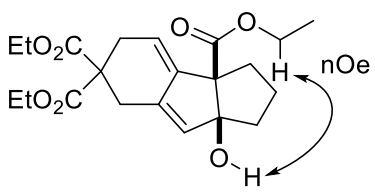


Ethyl 2-((*E*)-2-cyclohexylvinyl)-1-indanone-2-carboxylate (2o): Following the general procedure using ethyl 1-indanone-2-carboxylate (40.8 mg, 0.20 mmol) and cyclohexylacethylene (43.2 mg, 0.40 mmol) provided 43.7 mg (0.14 mmol, 70% yield) of **2o** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.98-1.28

(m, 6H), 1.22 (t, J = 7.2 Hz, 3H), 1.58-1.72 (m, 4H), 1.94-2.03 (m, 1H), 3.33 (d, J = 17.6 Hz, 1H), 3.80 (d, J = 17.6 Hz, 1H), 4.12-4.22 (m, 2H), 5.54 (dd, J = 6.8, 16.0 Hz, 1H), 5.97 (dd, J = 1.6, 16.0 Hz, 1H), 7.39 (t, J = 8.0 Hz, 1H), 7.48 (d, J = 8.0 Hz, 1H), 7.62 (t, J = 8.0 Hz, 1H), 7.77 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 25.9 (two peaks overlapped), 26.1, 32.6 (two peaks overlapped), 37.7, 40.7, 61.8, 62.5, 124.7, 125.1, 126.2, 127.7, 134.5, 135.2, 137.9, 152.6, 170.7, 200.7. IR (neat / cm^{-1}): 2924, 2851, 1746, 1732, 1715, 1609, 1447, 1248, 1211, 1188, 1011, 968, 926, 750. HRMS (FAB^+): calcd for $C_{20}H_{24}O_3$ ($[M+H]^+$) 313.1804; found. 313.1794.

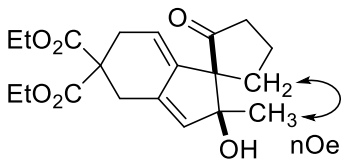


Ethyl 2-((*E*)-styryl)-1-indanone-2-carboxylate (2p): Following the general procedure using ethyl 1-indanone-2-carboxylate (40.8 mg, 0.20 mmol) and phenylacethylene (40.8 mg, 0.40 mmol) provided 30.6 mg (0.14 mmol, 50% yield) of **2p** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 5 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.25 (t, J = 7.2 Hz, 3H), 3.49 (d, J = 16.8 Hz, 1H), 3.94 (d, J = 16.8 Hz, 1H), 4.22 (q, J = 7.2 Hz, 2H), 6.52 (d, J = 16.0 Hz, 1H), 6.76 (d, J = 16.0, 1H), 7.22 (t, J = 7.6 Hz, 1H), 7.29 (t, J = 7.6 Hz, 2H), 7.37 (d, J = 7.6 Hz, 2H), 7.42 (t, J = 7.6 Hz, 1H), 7.54 (d, J = 7.6 Hz, 1H), 7.65 (t, J = 7.6 Hz, 1H), 7.81 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 37.7, 62.1, 62.9, 125.3, 126.3, 126.6, 126.7, 127.8, 127.9, 128.5, 130.9, 134.3, 135.5, 136.4, 152.5, 170.3, 200.0. IR (neat / cm^{-1}): 3057, 3026, 2982, 2938, 1715, 1607, 1589, 1464, 1449, 1271, 1246, 1211, 1188, 1065, 966, 743, 694. HRMS (FAB^+): calcd for $C_{20}H_{18}O_3$ ($[M]^+$) 306.1256; found. 306.1265.



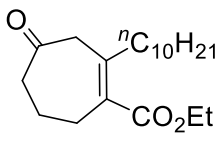
Tricycle 3a: Following the general procedure using ethyl 2-oxocyclopentanecarboxylate (62.5 mg, 0.40 mmol) and diethyl 2,2-di(prop-2-yn-1-yl)malonate (47.2 mg, 0.20 mmol) provided 61.1 mg (0.16 mmol, 78% yield) of **3a** as a pale yellow oil after purification by flash

chromatography (eluent: hexane / EtOAc = 5 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.08-1.17 (m, 1H), 1.21 (t, J = 7.2 Hz, 3H), 1.24 (t, J = 7.2 Hz, 3H), 1.25 (t, J = 7.2 Hz, 3H), 1.59-1.66 (m, 1H), 1.72-1.94 (m, 3H), 2.31 (dt, J = 6.0, 12.6 Hz, 1H), 2.65 (dd, J = 3.4, 17.0 Hz, 1H), 2.78 (dd, J = 3.4, 17.0 Hz, 1H), 2.82 (dd, J = 2.0, 15.2 Hz, 1H), 3.03 (d, J = 15.2 Hz, 1H), 4.11-4.20 (m, 6H), 5.47 (t, J = 4.0 Hz, 1H), 5.58 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0 (two peaks overlapped), 14.2, 22.2, 30.7, 30.8, 36.4, 39.2, 54.1, 61.0, 61.5, 61.6, 63.3, 93.7, 115.8, 133.4, 139.5, 147.1, 170.4, 170.9, 173.4. IR (neat / cm^{-1}): 3524, 3447, 2980, 2963, 2909, 1732, 1449, 1368, 1302, 1250, 1184, 1096, 1067, 1016, 862. HRMS (FAB^{+}): calcd for $C_{21}H_{28}O_7$ ([M]$^{+}$) 392.1835.

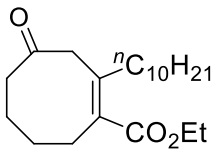


Spirocycle 3b: Following the general procedure using 2-acetylcyclopentanone (31.2 mg, 0.20 mmol) and diethyl 2,2-di(prop-2-yn-1-yl)malonate (94.5 mg, 0.40 mmol) provided 63.7 mg (0.18 mmol, 88% yield, d.r. = 57 / 43) of **3b** as a pale brown oil after purification by flash chromatography (eluent: hexane / EtOAc = 5 / 1). The diastereomeric ratio was determined to be 54 / 46 by ^{1}H NMR analysis of the crude product. These diastereomers can be separable by flash column chromatography, and major isomer was eluted faster than minor isomer. For major diastereomer; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.18 (s, 3H), 1.21 (t, J = 7.2 Hz, 3H), 1.24 (t, J = 7.2 Hz, 3H), 1.98-2.14 (m, 3H), 2.24-2.32 (m, 2H), 2.42-2.50 (m, 1H), 2.60 (dd, J = 17.2, 3.6 Hz, 1H), 2.76 (dd, J = 17.2, 3.6 Hz, 1H), 2.81 (d, J = 15.6 Hz, 1H), 2.94 (d, J = 15.6 Hz, 1H), 3.00 (s, 1H), 4.08-4.24 (m, 4H), 5.47 (t, J = 3.6 Hz, 1H), 5.71 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9, 14.0, 19.2, 24.4, 30.3, 30.6, 30.9, 37.3, 54.0, 61.4, 61.7, 65.8, 83.6, 114.2, 136.3, 137.0, 144.7, 170.3, 170.8, 218.4. IR (neat / cm^{-1}): 2980, 2940, 2907, 1734, 1558, 1447, 1368, 1300, 1248, 1194, 1057, 1022, 916, 864, 733. HRMS (FAB^{+}): calcd for $C_{20}H_{27}O_6$ ([M+H]$^{+}$) 363.1808; found. 363.1821. For minor diastereomer; ^{1}H NMR for minor diastereomer (400 MHz, $CDCl_3$): δ 1.21 (t, J = 7.2 Hz, 3H), 1.24 (t, J = 7.2 Hz, 3H), 1.27 (s, 3H), 1.41 (brs, 1H), 1.94-2.05 (m, 3H), 2.22-2.40 (m, 2H), 2.42-2.50 (m, 1H), 2.69

(dd, J = 4.0, 17.6 Hz, 1H), 2.75 (dd, J = 4.0, 17.6 Hz, 1H), 2.88 (dd, J = 1.2, 15.2 Hz, 1H), 2.97 (d, J = 15.2 Hz, 1H), 4.10-4.21 (m, 4H), 5.40 (t, J = 4.0 Hz, 1H), 5.69 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9, 14.0, 19.6, 21.8, 30.6, 30.9, 31.1, 38.9, 54.1, 61.5, 61.7, 65.1, 83.4, 115.9, 134.7, 139.9, 146.4, 170.6, 218.0. HRMS (FAB$^+$): calcd for $C_{20}H_{26}O_6$ ([M]$^+$) 362.1729; found. 362.1746.

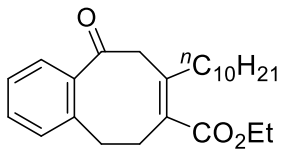


Ethyl 2-decyl-4-oxo-1-cycloheptene-1-carboxylate (4d): Following the general procedure using ethyl 2-oxocyclopentanecarboxylate (31.2 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and sodium bicarbonate (16.8 mg, 0.20 mmol) provided 52.2 mg (0.16 mmol, 81% yield) of **4d** as a colorless oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 6.8 Hz, 3H), 1.22-1.34 (m, 14H), 1.30 (t, J = 7.2 Hz, 3H), 1.40-1.48 (m, 2H), 1.97 (quint, J = 6.8 Hz, 2H), 2.37 (t, J = 7.2 Hz, 2H), 2.57 (t, J = 6.8 Hz, 4H), 3.25 (s, 2H), 4.21 (t, J = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 14.3, 22.7, 25.2, 27.8, 28.6, 29.3, 29.5, 29.6 (two peaks overlapped), 29.7, 31.9, 36.6, 43.6, 49.6, 60.5, 129.8, 144.3, 168.9, 207.3. IR (neat / cm^{-1}): 2955, 2926, 2855, 1769, 1732, 1717, 1558, 1506, 1456, 1238, 1098, 1026. HRMS (FAB$^+$): calcd for $C_{20}H_{35}O_3$ ([M+H]$^+$) 323.2586; found. 323.2589.



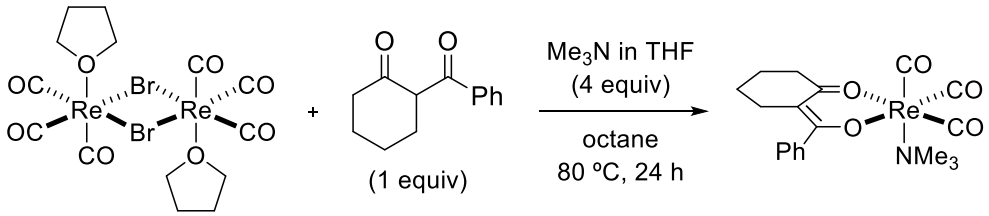
Ethyl 2-decyl-4-oxo-1-cyclooctene-1-carboxylate (4e): Following the general procedure using ethyl 2-oxocyclohexanecarboxylate (34.0 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and sodium bicarbonate (16.8 mg, 0.20 mmol) provided 63.8 mg (0.19 mmol, 95% yield) of **4e** as a pale yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, J = 6.8 Hz, 3H), 1.20-1.32 (m, 14H), 1.31 (t, J = 7.2 Hz, 3H), 1.40-1.46 (m, 2H), 1.76-1.77 (m, 4H), 2.24-2.29 (m, 2H), 2.42-2.47 (m, 4H), 3.26 (s, 2H), 4.21 (q, J = 7.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 14.3, 22.7, 24.5, 27.7, 28.1, 29.3, 29.4, 29.5, 29.6, 29.7, 29.8, 31.9, 36.1, 42.3, 49.1, 60.4, 129.5, 142.9, 169.3, 210.8. IR

(neat / cm^{-1}): 2953, 2926, 2855, 1730, 1717, 1705, 1558, 1506, 1456, 1364, 1261, 1198, 1096, 1028. HRMS (FAB^+): calcd for $C_{21}H_{36}O_3$ ($[M]^+$) 336.2665; found. 336.2657.



Ethyl (*Z*)-8-dodecyl-10-oxo-5,6,9,10-tetrahydrobenzocyclooctene-7-carboxylate (4f): Following the general procedure using ethyl 1-tetralone-2-carboxylate (43.6 mg, 0.20 mmol), 1-dodecyne (66.5 mg, 0.40 mmol), and sodium bicarbonate (16.8 mg, 0.20 mmol) provided 53.8 mg (0.14 mmol, 70% yield) of **4f** as a yellow oil after purification by flash chromatography (eluent: hexane / EtOAc = 20 / 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88 (t, J = 6.8 Hz, 3H), 1.07-1.10 (m, 2H), 1.18-1.34 (m, 17H), 2.15 (t, J = 7.2 Hz, 2H), 2.81 (t, J = 7.2 Hz, 2H), 3.13 (t, J = 7.2 Hz, 2H), 3.58 (s, 2H), 4.10 (q, J = 7.2 Hz, 2H), 7.13 (d, J = 7.6 Hz, 1H), 7.25 (t, J = 7.6 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.41 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1, 14.2, 22.7, 27.9, 29.3, 29.4 (two peaks overlapped), 29.5 (two peaks overlapped), 29.6, 31.9, 33.7, 36.4, 51.4, 60.3, 126.8, 127.2, 129.2, 130.0, 131.5, 137.6, 138.8, 143.7, 169.2, 205.4. IR (neat / cm^{-1}): 2955, 2926, 2855, 1717, 1684, 1559, 1506, 1447, 1373, 1233, 1193, 1096, 1026, 768, 731. HRMS (FAB^+): calcd for $C_{25}H_{37}O_3$ ($[M+H]^+$) 385.2743; found. 385.2751..

4. Isolation of Oxarhenacycle Intermediate



In this study, Me_3N was used in place of ^{i}Pr$_2$NEt due to its volatile nature. A flame dried Schlenk flask was charged with $[ReBr(CO)_3(thf)]_2$ (21.1 mg, 0.025 mmol), 2-benzoylcyclohexanone (10.1 mg, 0.050 mmol), trimethylamine (2.0 M in THF, 0.20 mmol, 100 μL), and octane (1.0 mL), and then the resulting mixture was stirred at 80 °C for 24 h.

The solvent was removed under reduced pressure to afford the oxarhenacycle intermediate as a yellow solid (27.0 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.50-1.62 (m, 2H), 1.62-1.70 (m, 1H), 1.76-1.82 (m, 1H), 2.20-2.38 (m, 3H), 2.45 (dd, J = 7.2, 18.8 Hz, 1H), 2.54 (s, 9H), 7.31-7.34 (m, 2H), 7.36-7.40 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.5, 23.8, 28.6, 36.7, 45.1, 53.8, 109.5, 126.8, 128.0, 129.1, 140.9, 185.9, 190.9, 196.4, 198.8, 199.0. IR (KBr / cm^{-1}): 2016, 1888, 1869, 1553, 1456, 1333, 1319.

5. References

1. (a) Smith, A. M. R.; Billen, D.; Hii, K. K. M. Palladium-Catalysed Enantioselective α-Hydroxylation of β-Ketoesters. *Chem. Commun.* **2009**, *26*, 3925-3927. (b) Duan, Y.-N.; Cui, L.-Q.; Zuo, L.-H.; Zhang, C. Recyclable Hypervalent-Iodine-Mediated Dehydrogenative α, β'-Bifunctionalization of β-Keto Esters Under Metal-Free Conditions. *Chem. Eur. J.* **2015**, *21*, 13052-13057.
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3. Smith, A. M. R.; Rzepa, H. S.; White, A. J. P.; Billen, D.; Hii, K. K. M. Delineating Origins of Stereocontrol in Asymmetric Pd-Catalyzed α-Hydroxylation of 1,3-Ketoesters. *J. Org. Chem.* **2010**, *75*, 3085-3096.
4. Kenny, M.; Christensen, J.; Coles, S. J.; Franckevicius, V. Regioswitchable Palladium-Catalyzed Decarboxylative Coupling of 1,3-Dicarbonyl Compounds. *Org. Lett.* **2015**, *17*, 3926-3929.
5. Szostak, M.; Spain, M.; Choquette, K. A.; Flowers II, R. A.; Procter, D. J. Substrate-Directable Electron Transfer Reactions. Dramatic Rate Enhancement in the

Chemoselective Reduction of Cyclic Esters Using SmI_2–H_2O: Mechanism, Scope, and Synthetic Utility. *J. Am. Chem. Soc.* **2013**, *135*, 15702-15705.

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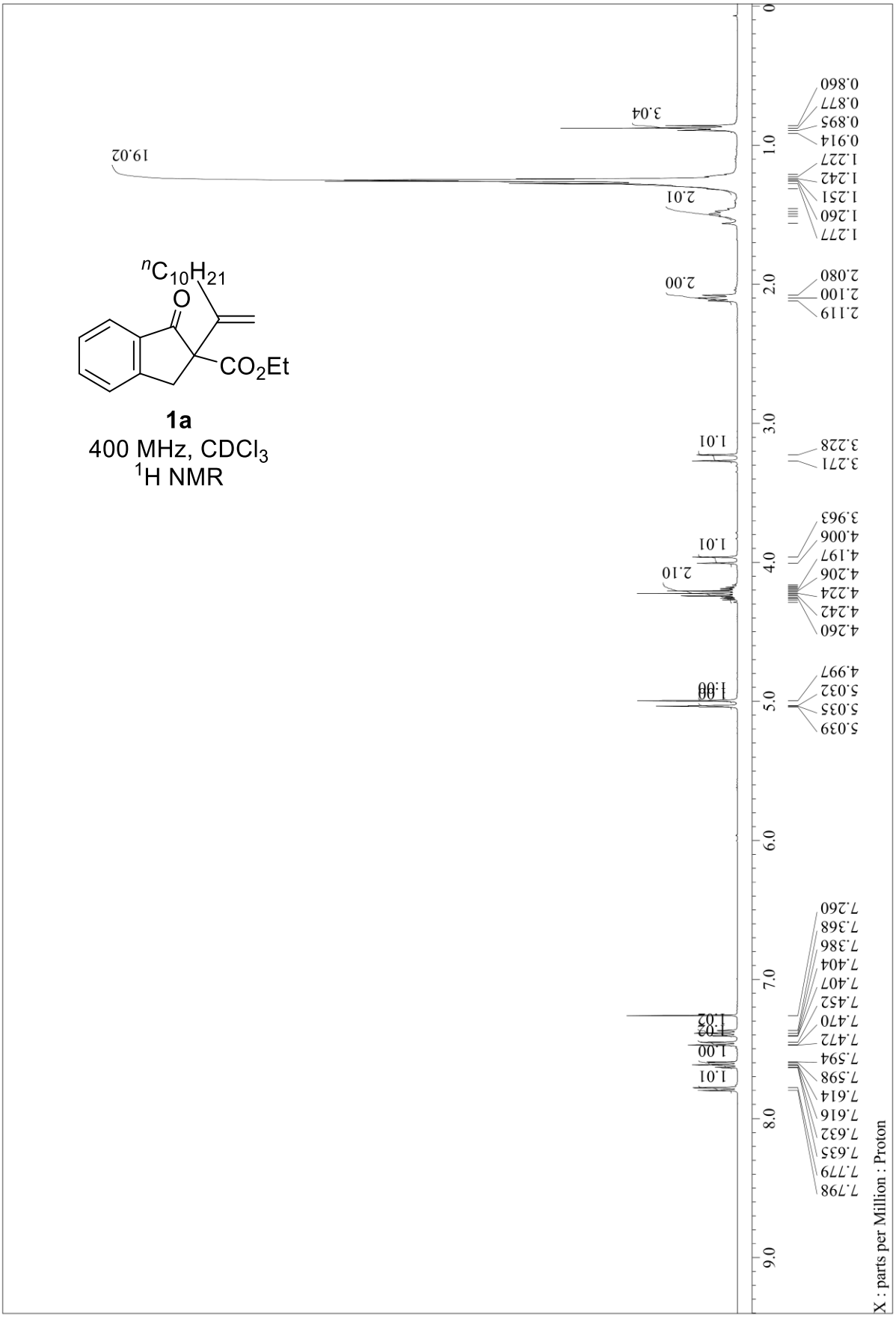
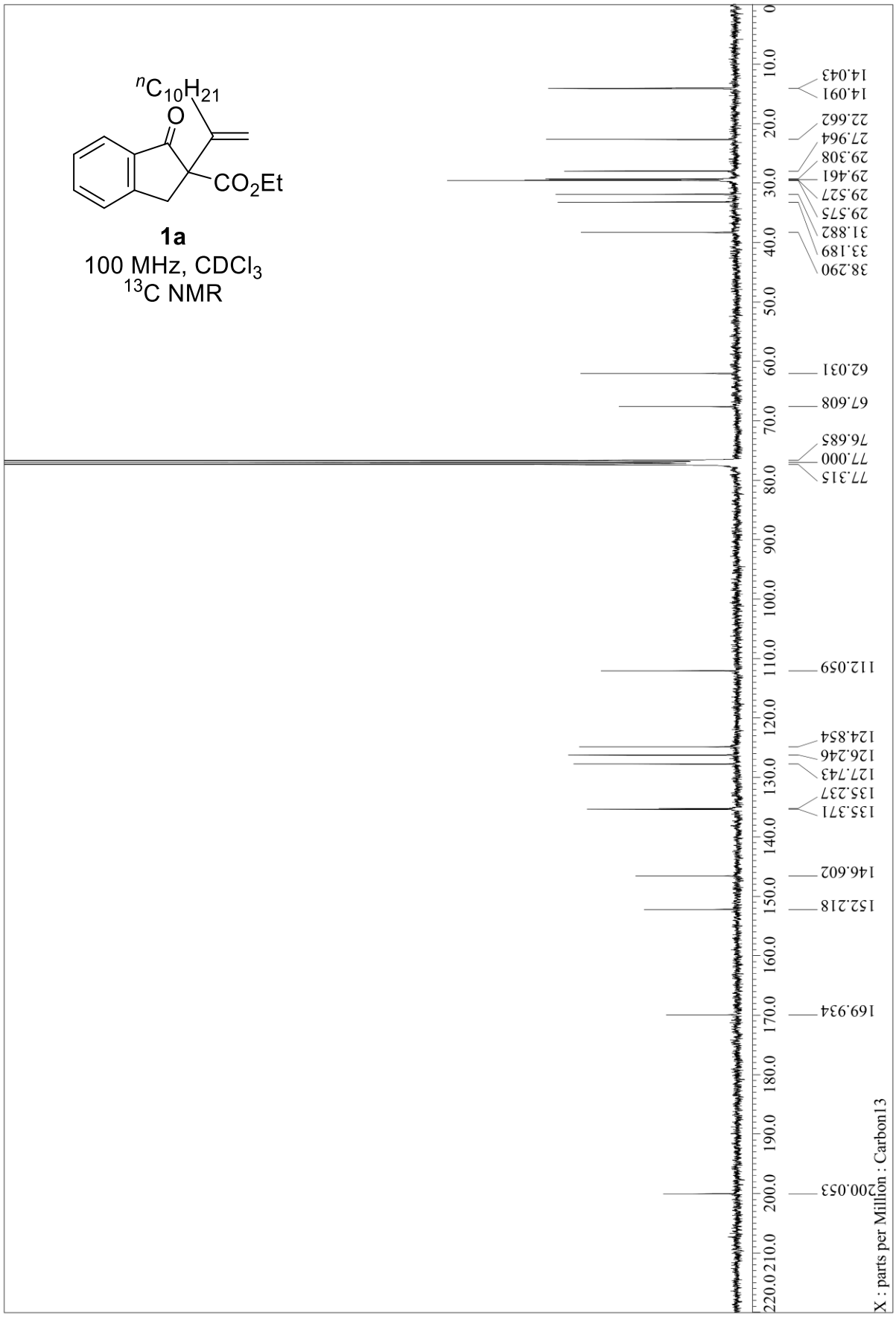
7. (a) Vitali, D.; Calderazzo, F. Reactions of Halocarbonyls of Rhenium(I). *Gazz. Chim. Ital.* **1972**, *102*, 587-596. (b) Du, S.; Kautz, J. A.; McGrath, T. D.; Stone, F. G. A. Synthesis and Structure of the Novel 11-Vertex Rhenacarborane Dianion $[1,1,1\text{-}(CO)_3\text{-}2\text{-Ph-closo-}1,2\text{-}ReCB_9H_9]^{2-}$ and Its Reactivity toward Cationic Transition Metal Fragments. *Organometallics* **2003**, *22*, 2842-2850.

6. ^{1}H NMR and ^{13}C NMR Spectra of New Compounds

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29.308
29.461
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31.882
33.189
38.290
62.031
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77.000
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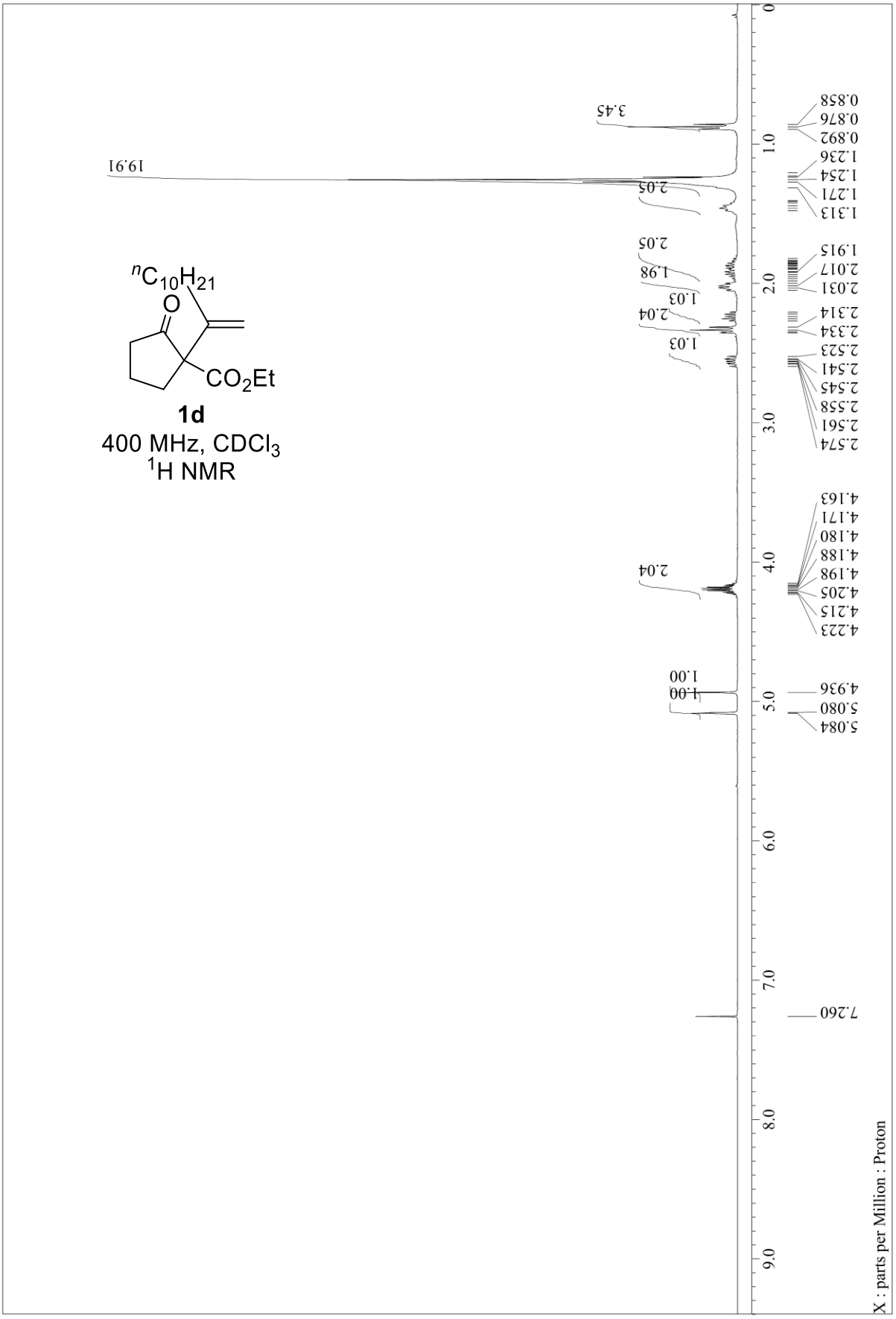
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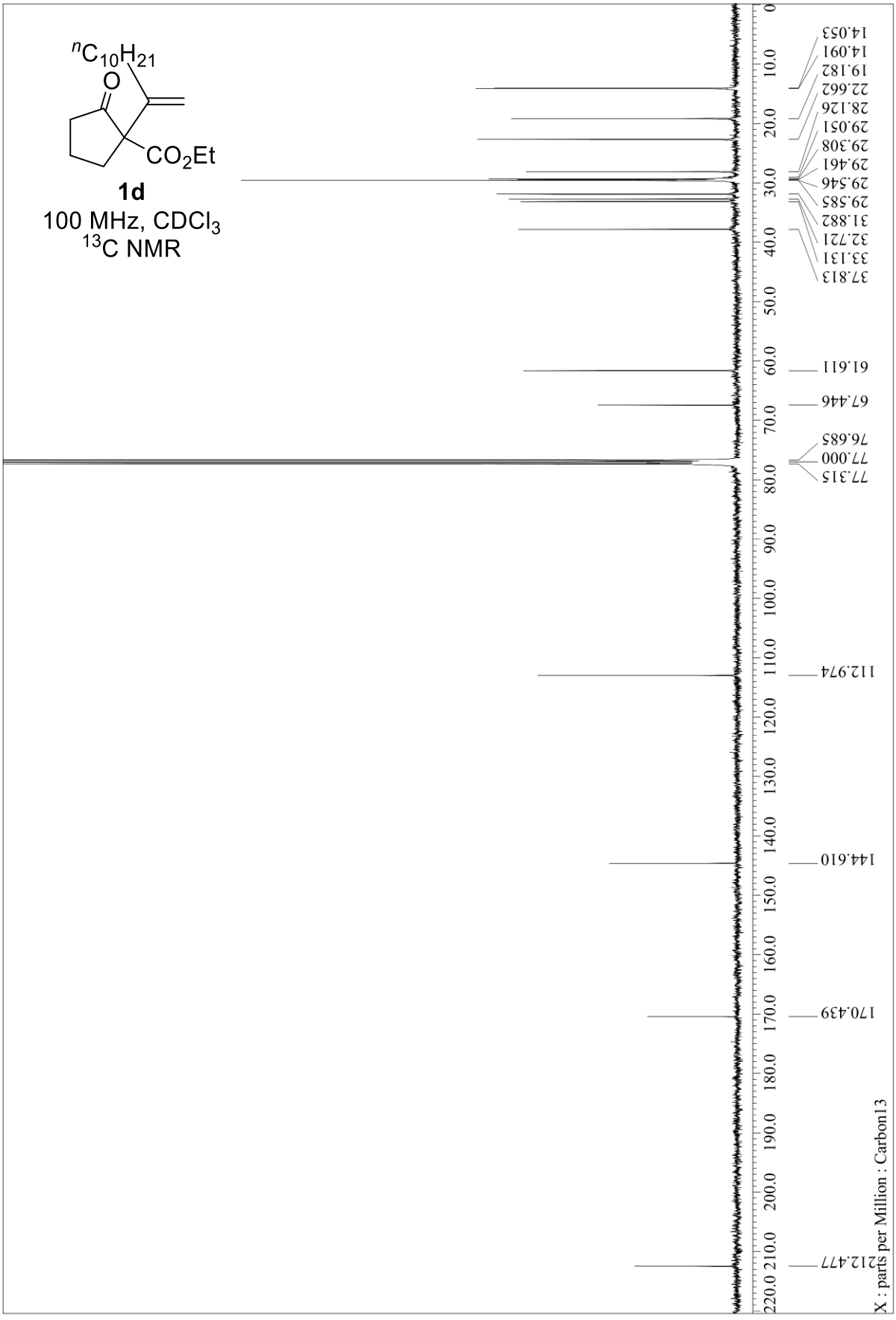
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CO_2Et
1d
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28.126
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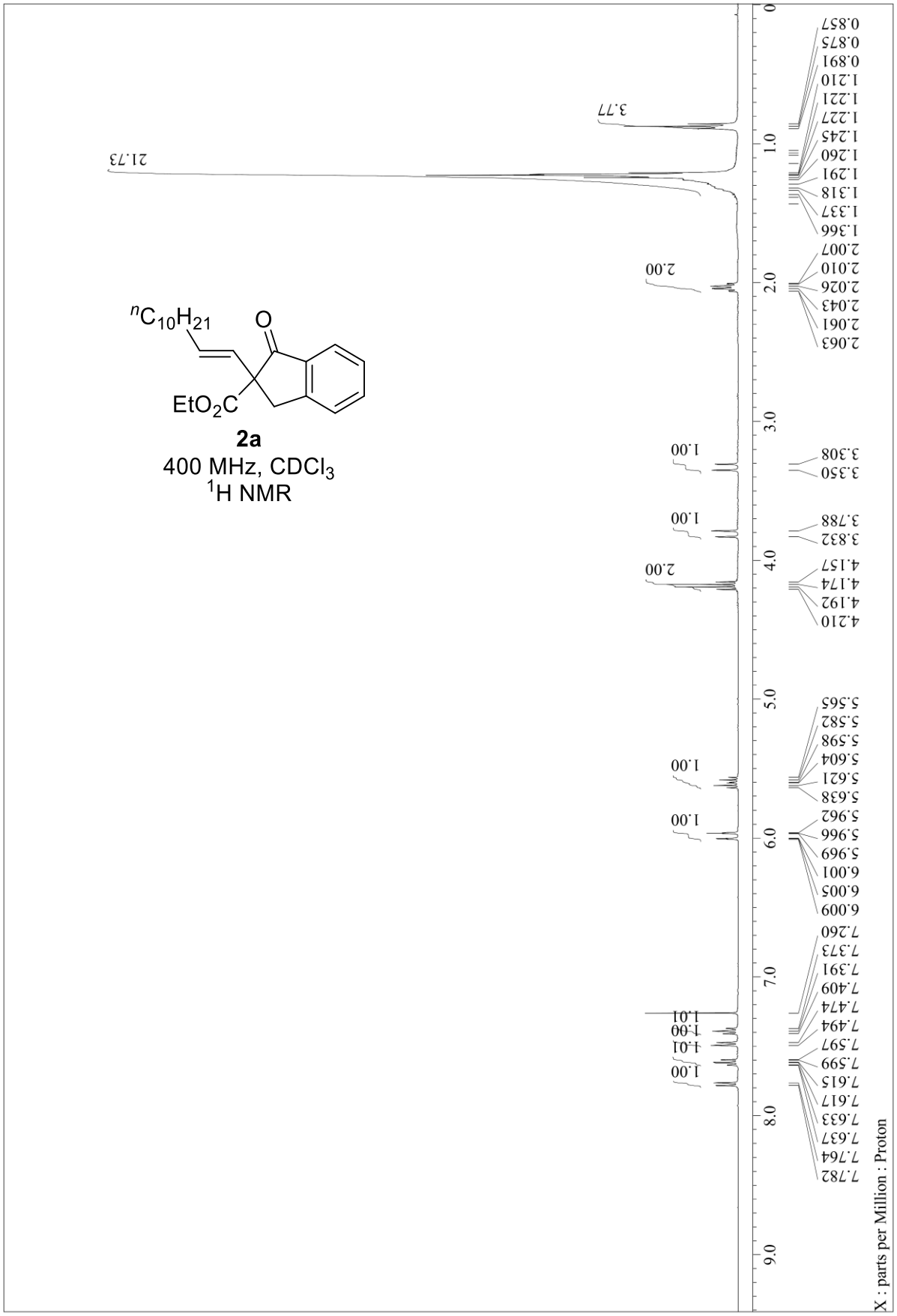
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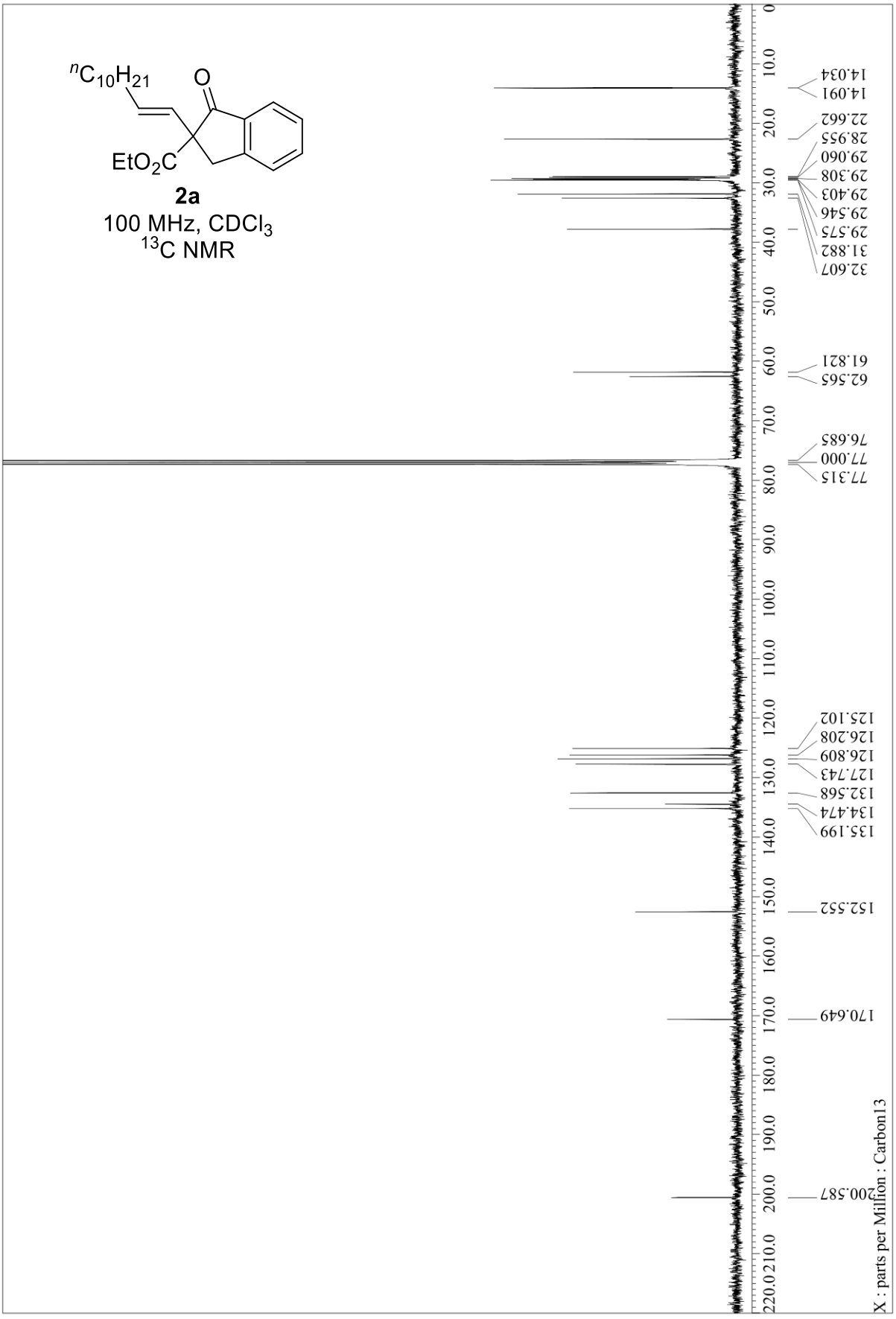
EtO_2C
2a
400 MHz, $CDCl_3$
^{1}H NMR
X : parts per Million : Proton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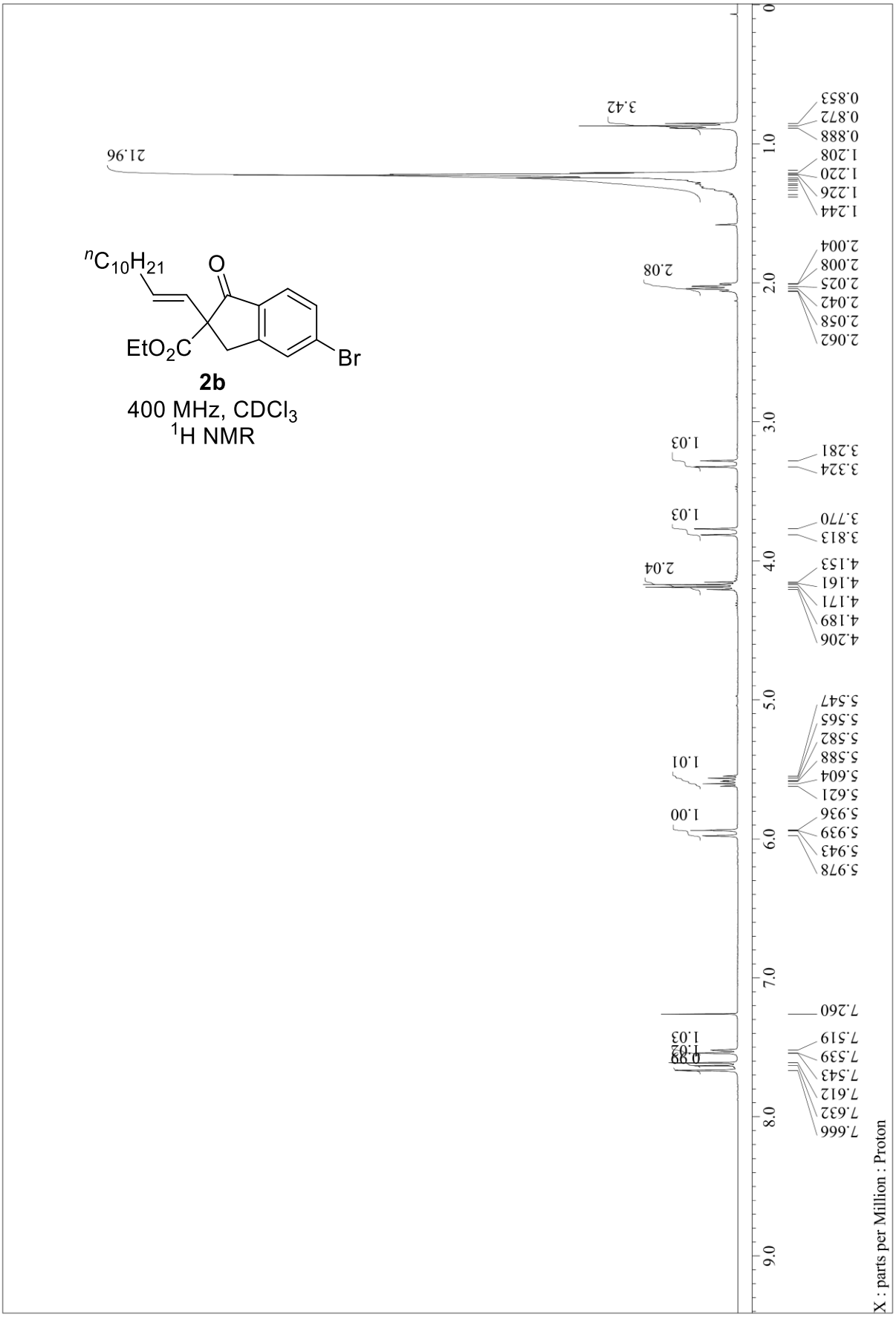
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1.03
1.03
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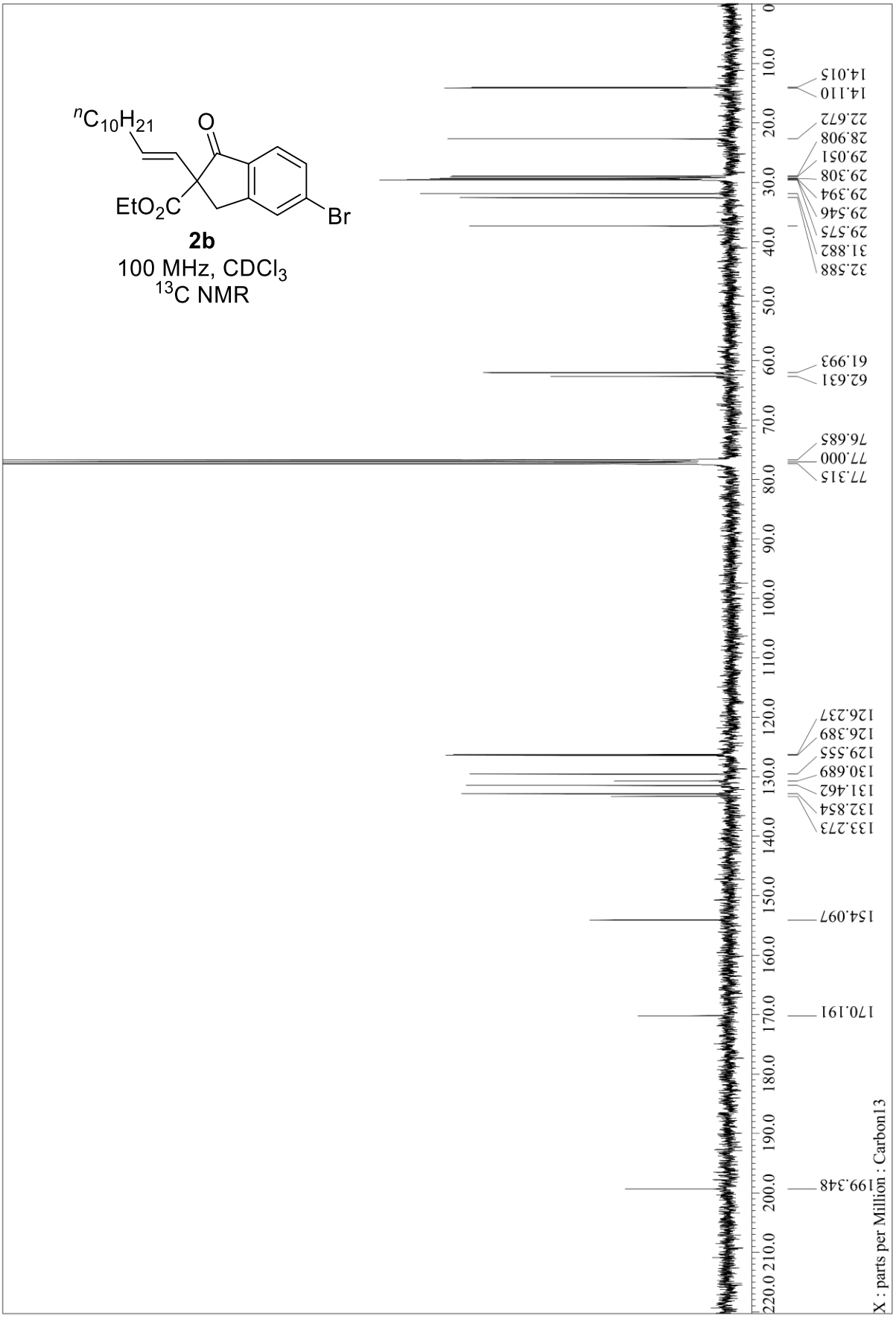
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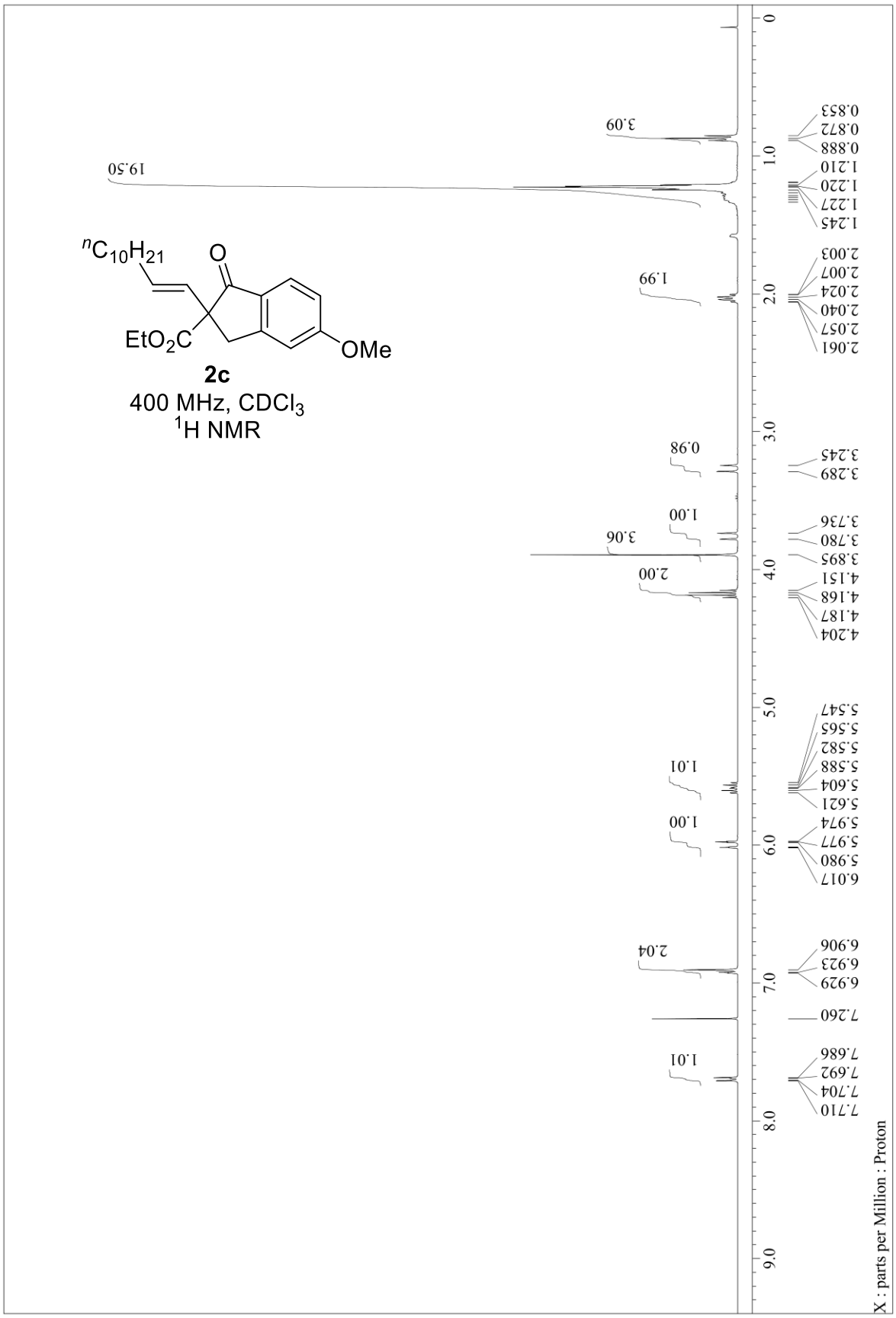
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EtO_2C
OMe
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1.00
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2.00
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1.00
2.04
1.01
0.853
0.872
0.888
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1.220
1.227
1.245
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2.007
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3.245
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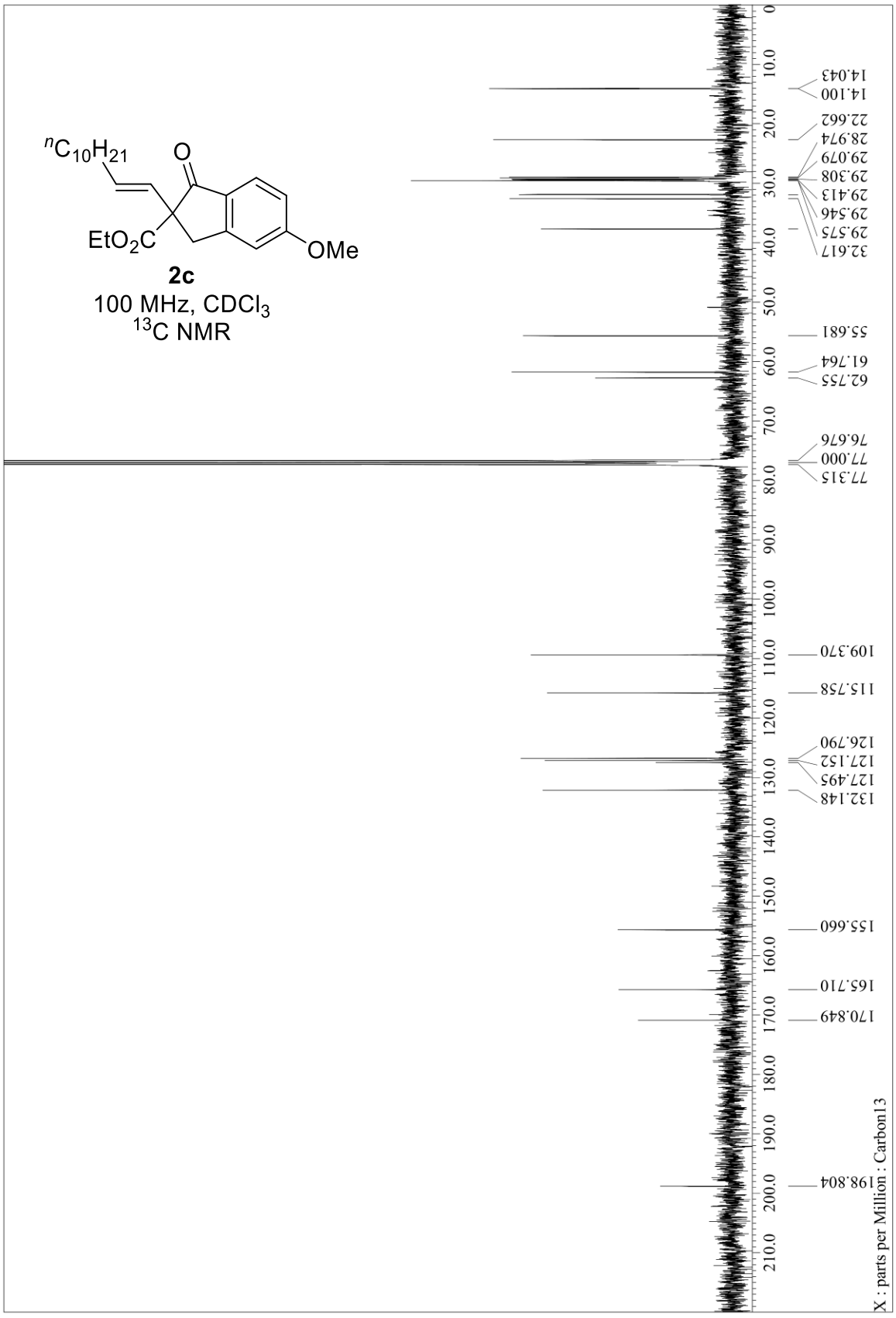
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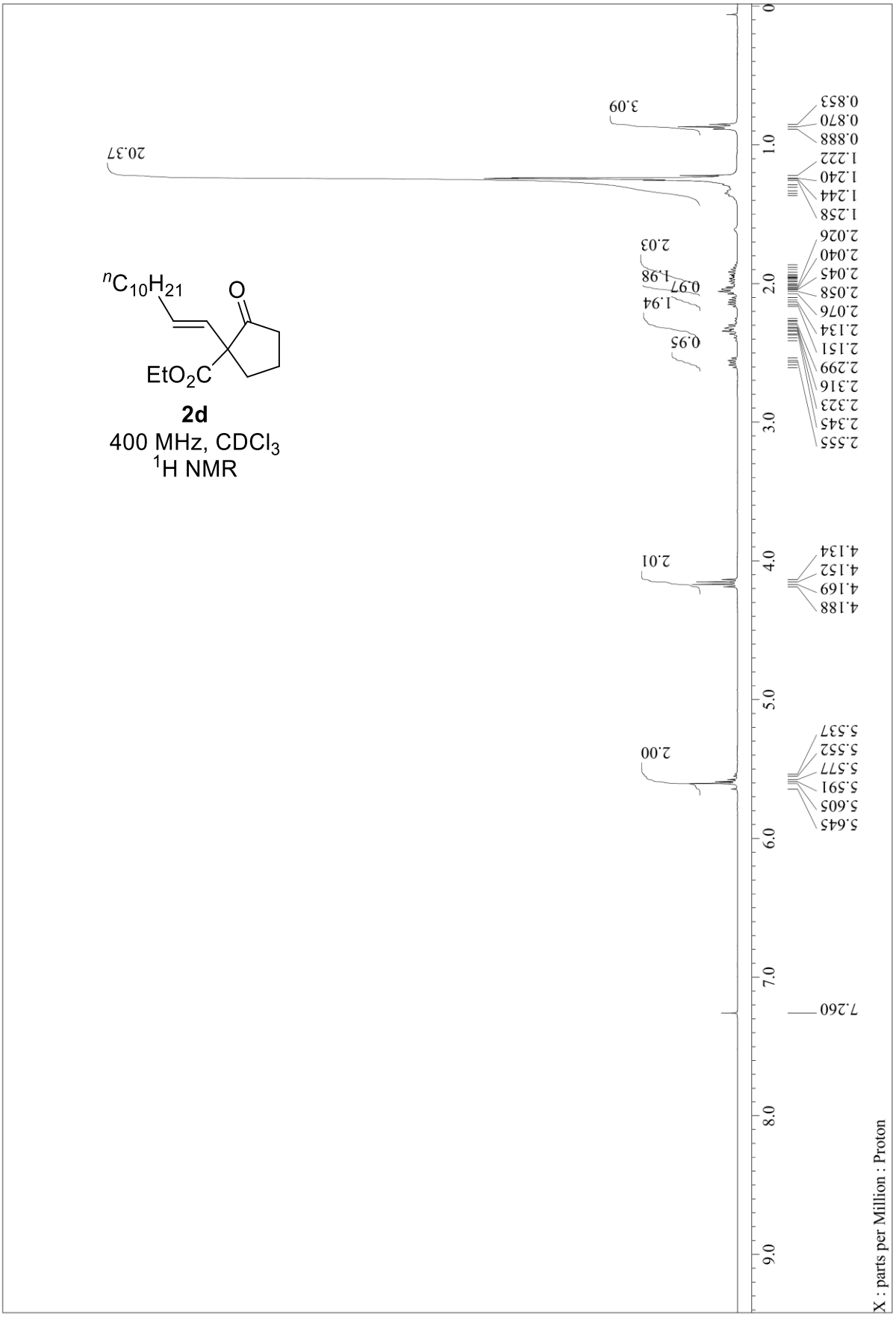
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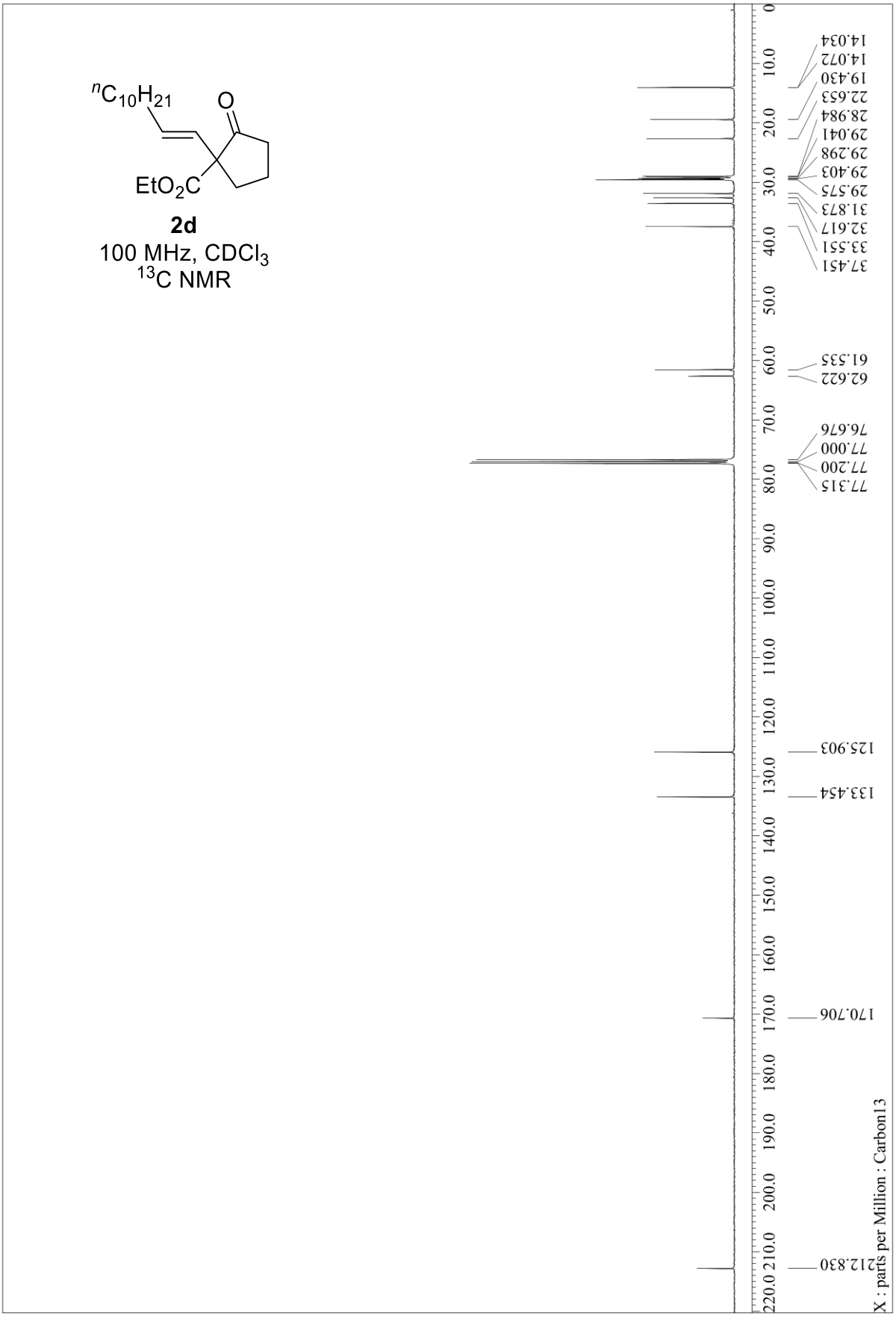
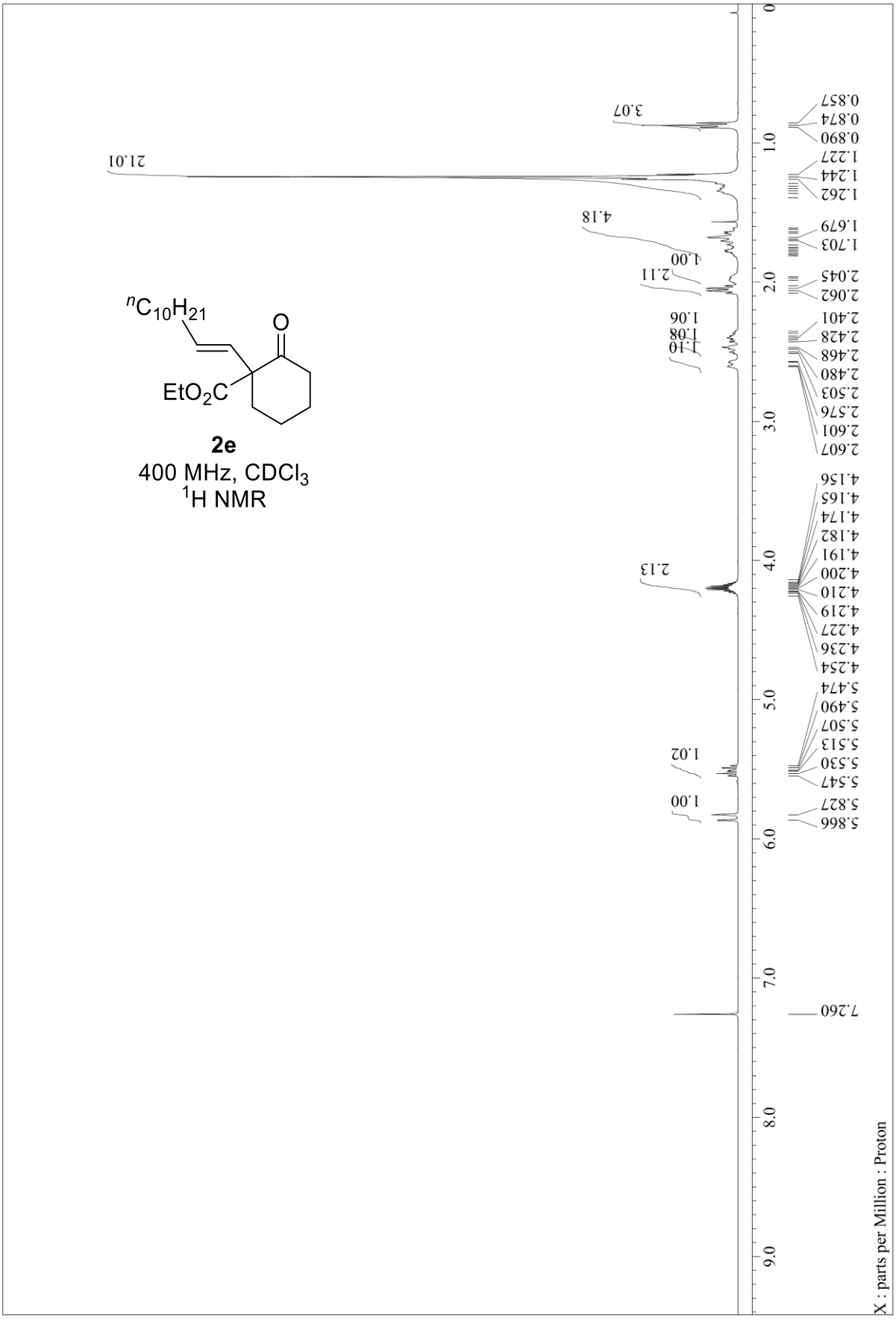
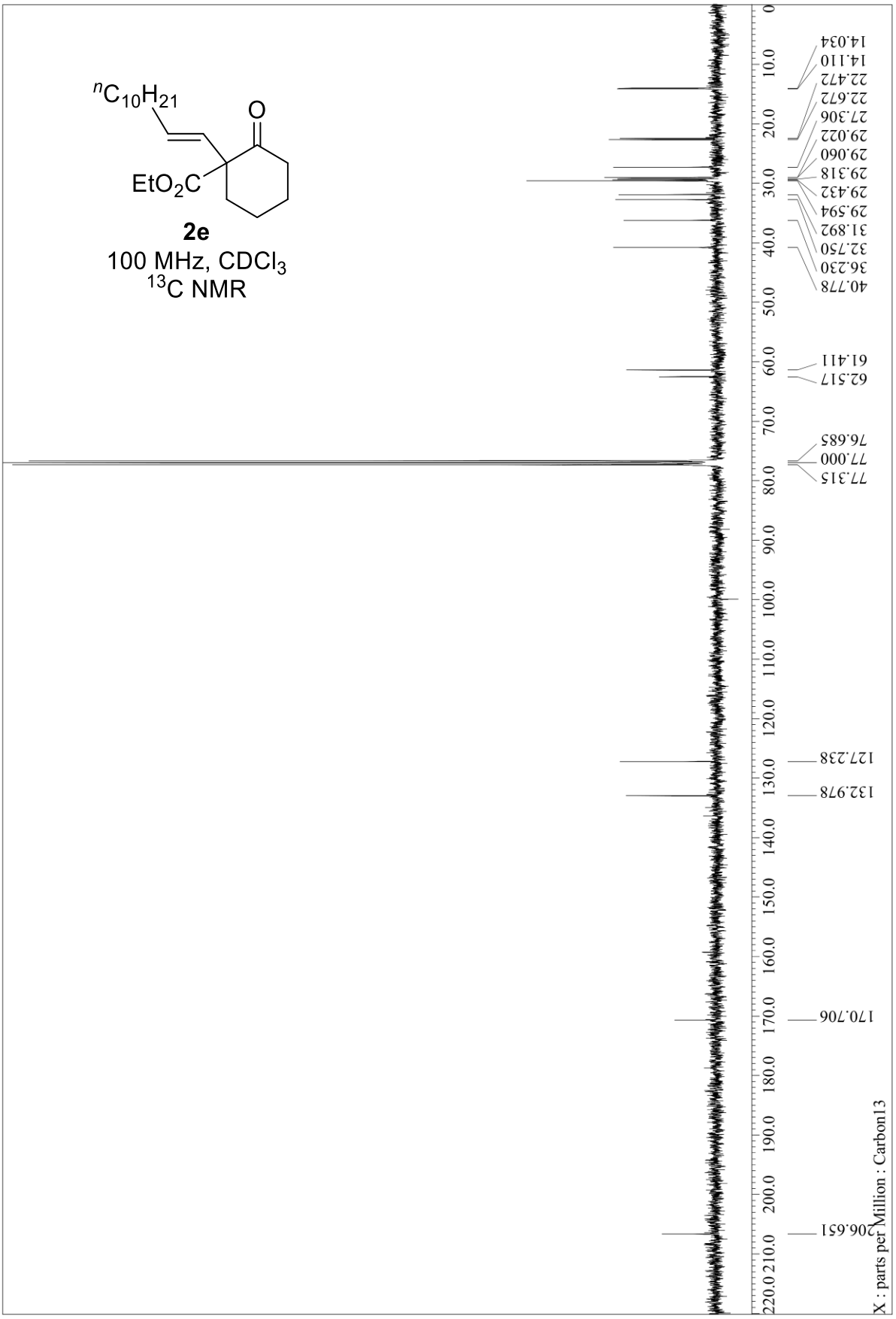
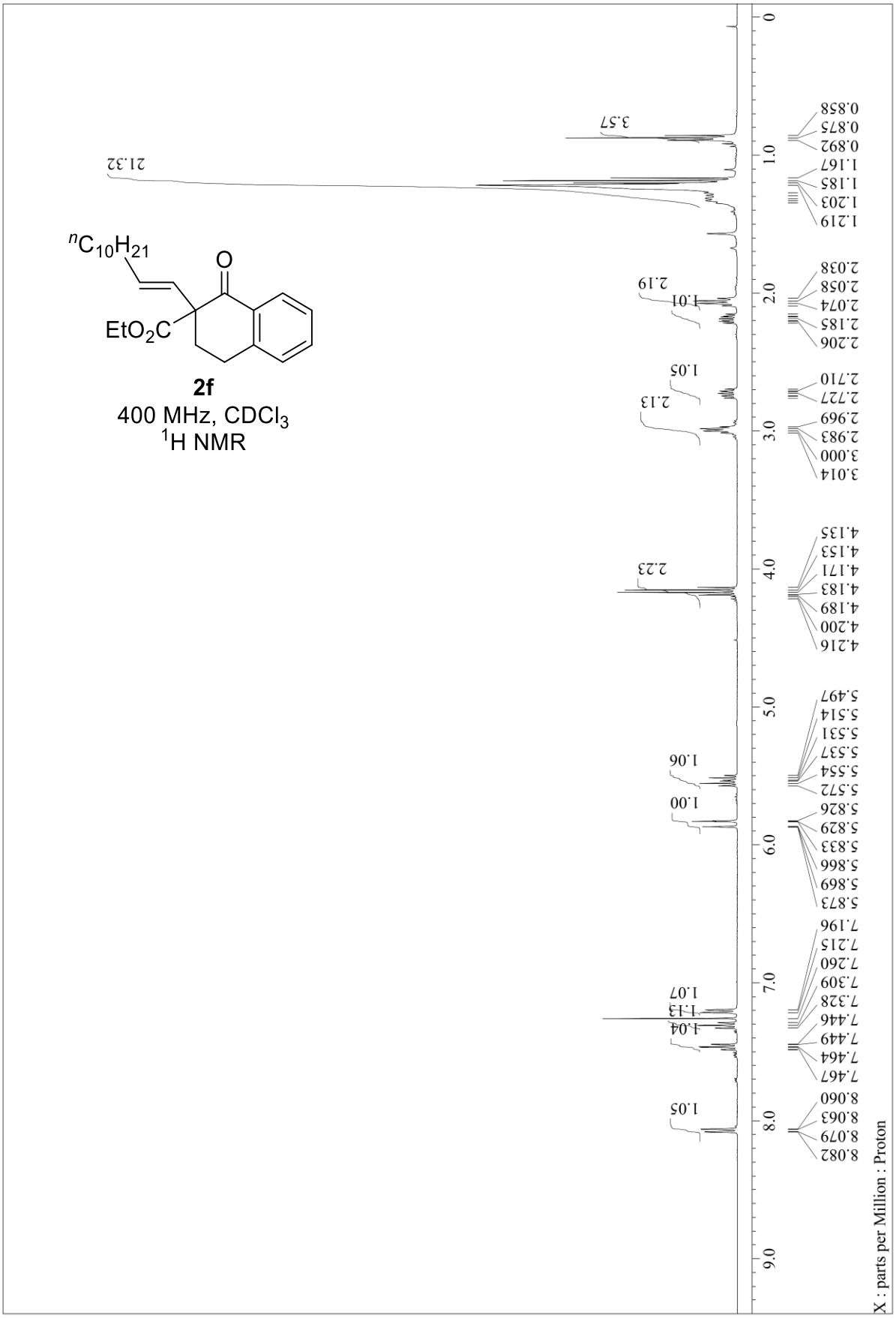
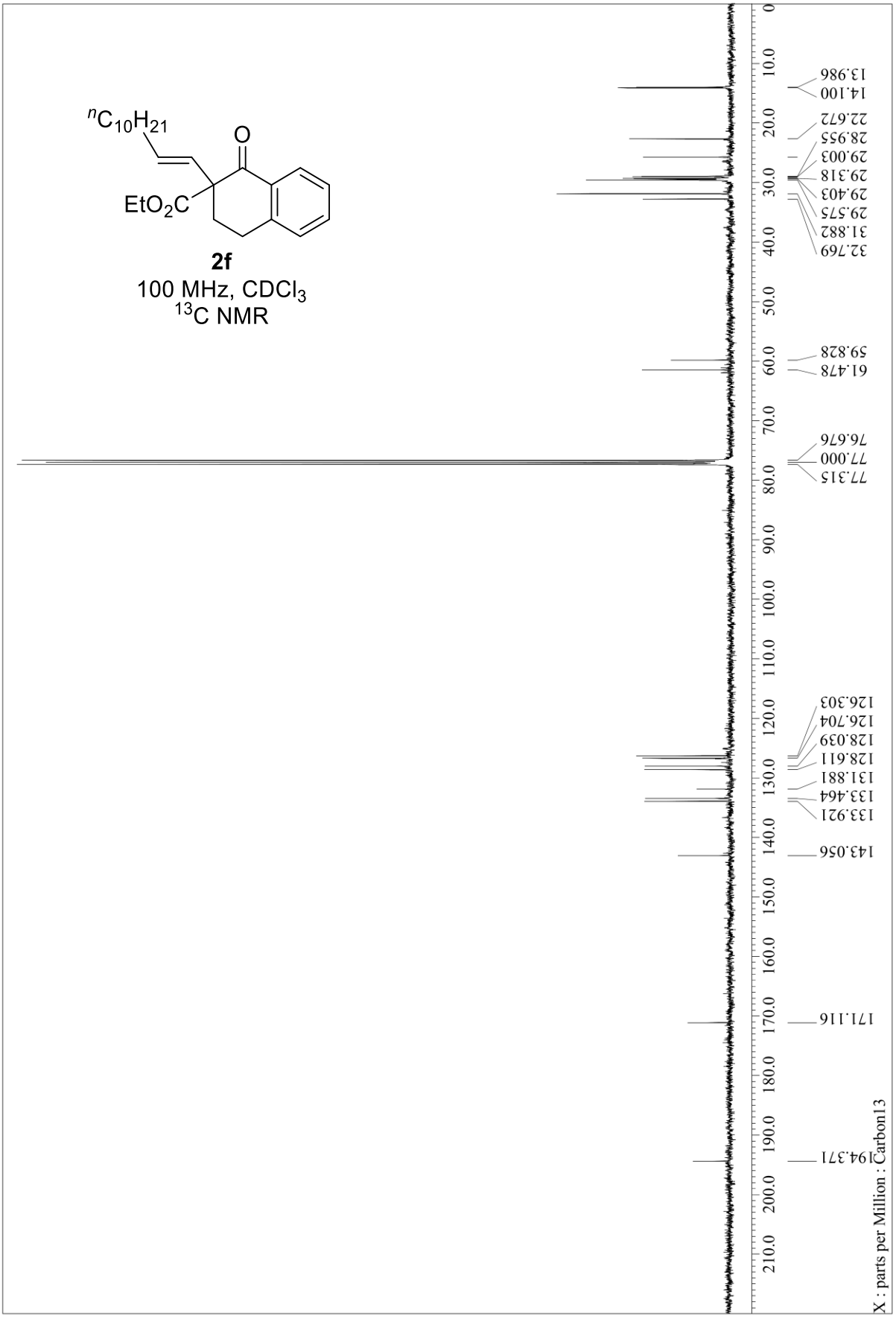
7.260
X : parts per Million : Proton

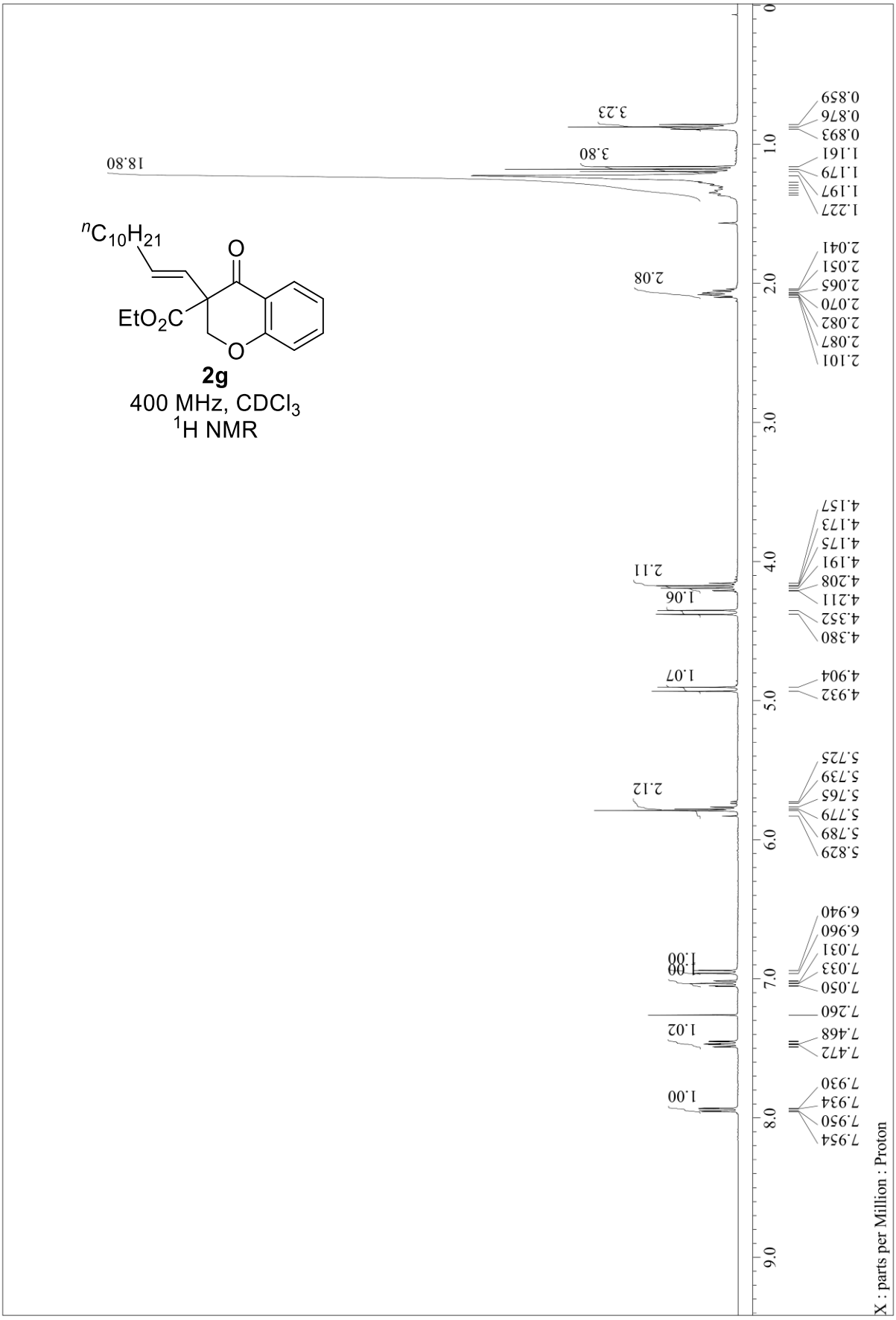

$^{n}C_{10}H_{21}$
O
EtO_2C
2d
100 MHz, $CDCl_3$
^{13}C NMR
14.034
14.072
19.430
22.653
28.984
29.041
29.298
29.403
29.575
31.873
32.617
33.551
37.451
61.535
62.622
76.676
77.000
77.200
77.315
125.903
133.454
170.706
212.830
X : parts per Million : Carbon13

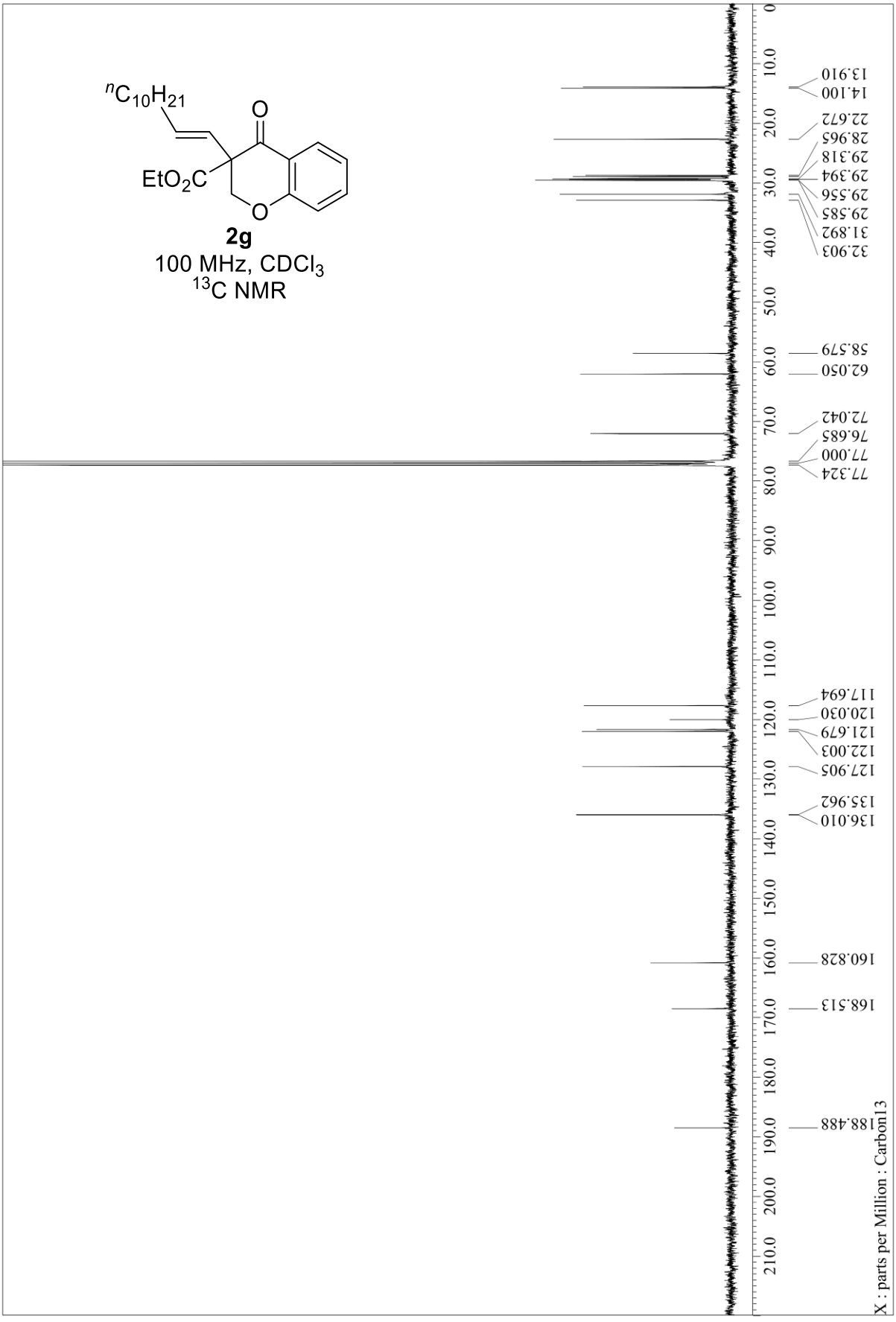

$^{n}C_{10}H_{21}$
O
EtO_2C
2e
400 MHz, $CDCl_3$
^{1}H NMR
21.01
3.07
4.18
1.00
2.11
1.06
1.08
1.10
2.13
1.02
1.00
7.260
5.866
5.827
5.547
5.530
5.513
5.507
5.490
5.474
4.254
4.236
4.227
4.219
4.210
4.200
4.191
4.182
4.174
4.165
4.156
2.607
2.601
2.576
2.503
2.480
2.468
2.428
2.401
2.062
2.045
1.703
1.679
1.262
1.244
1.227
0.890
0.874
0.857
X : parts per Million : Proton

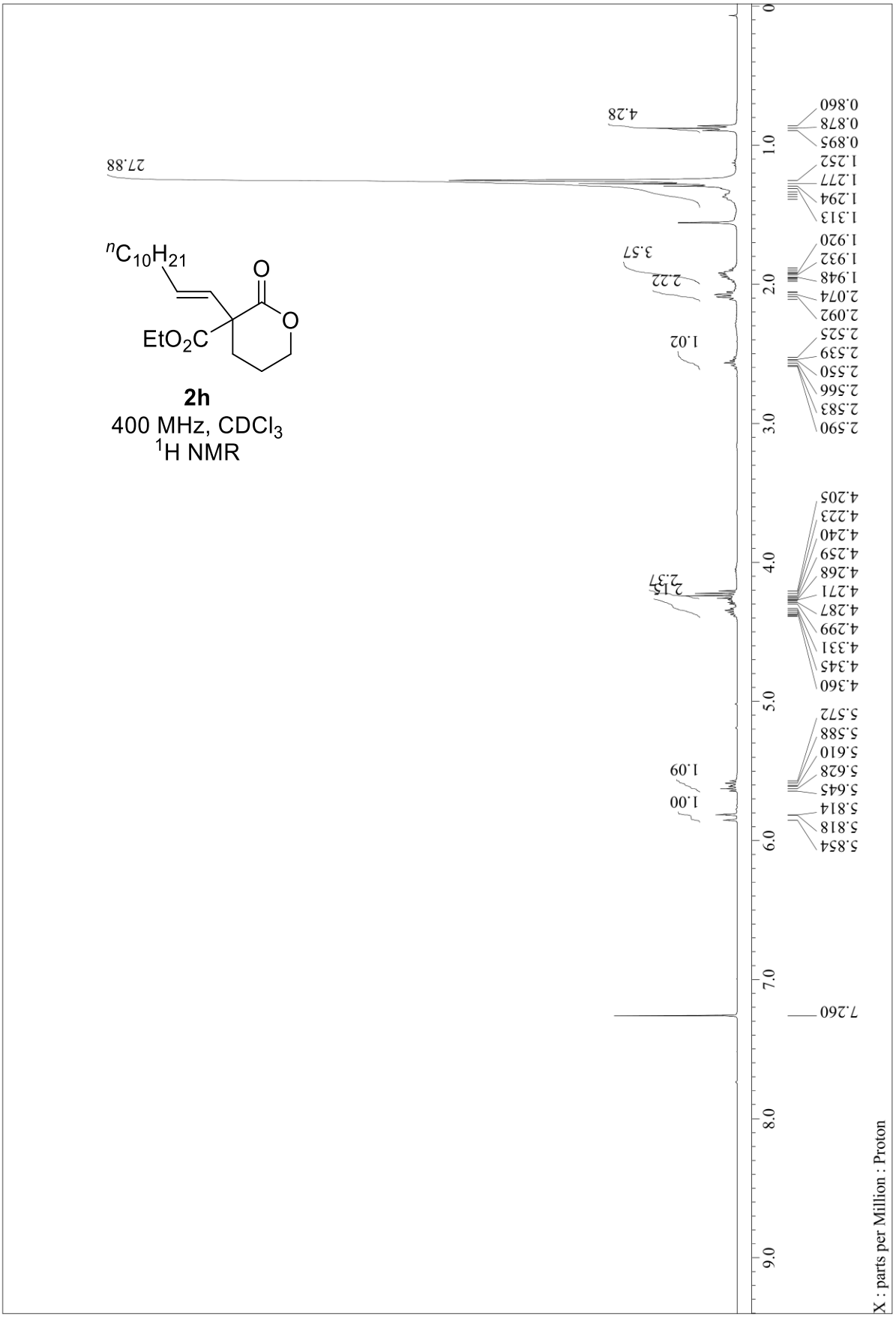

$^{n}C_{10}H_{21}$
O
EtO_2C
2e
100 MHz, $CDCl_3$
^{13}C NMR
14.034
14.110
22.472
22.672
27.306
29.022
29.060
29.318
29.432
29.594
31.892
32.750
36.230
40.778
61.411
62.517
76.685
77.000
77.315
127.238
132.978
170.706
206.651
X : parts per Million : Carbon13

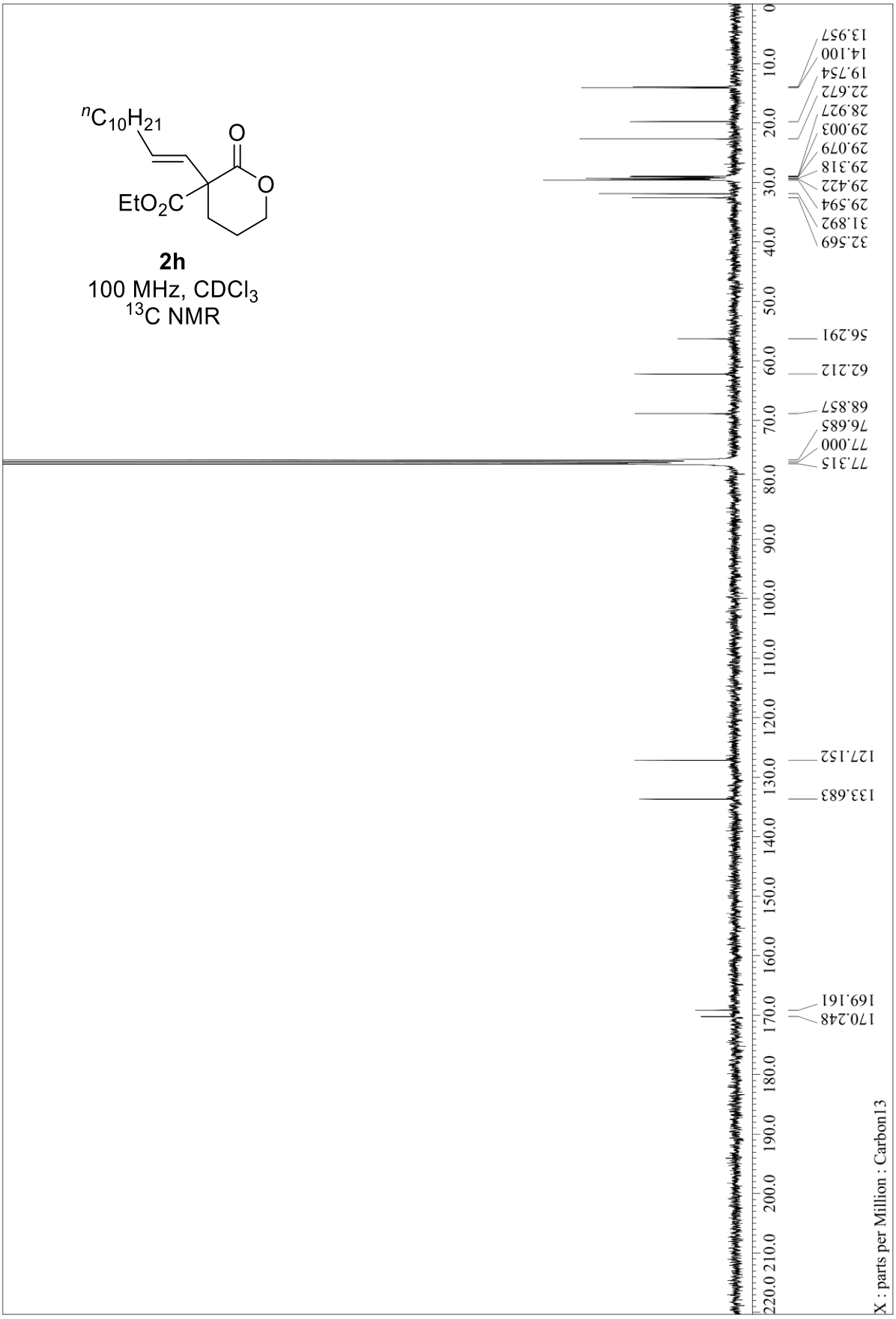

$^{n}C_{10}H_{21}$
O
EtO_2C
2f
400 MHz, $CDCl_3$
^{1}H NMR
X : parts per Million : Proton

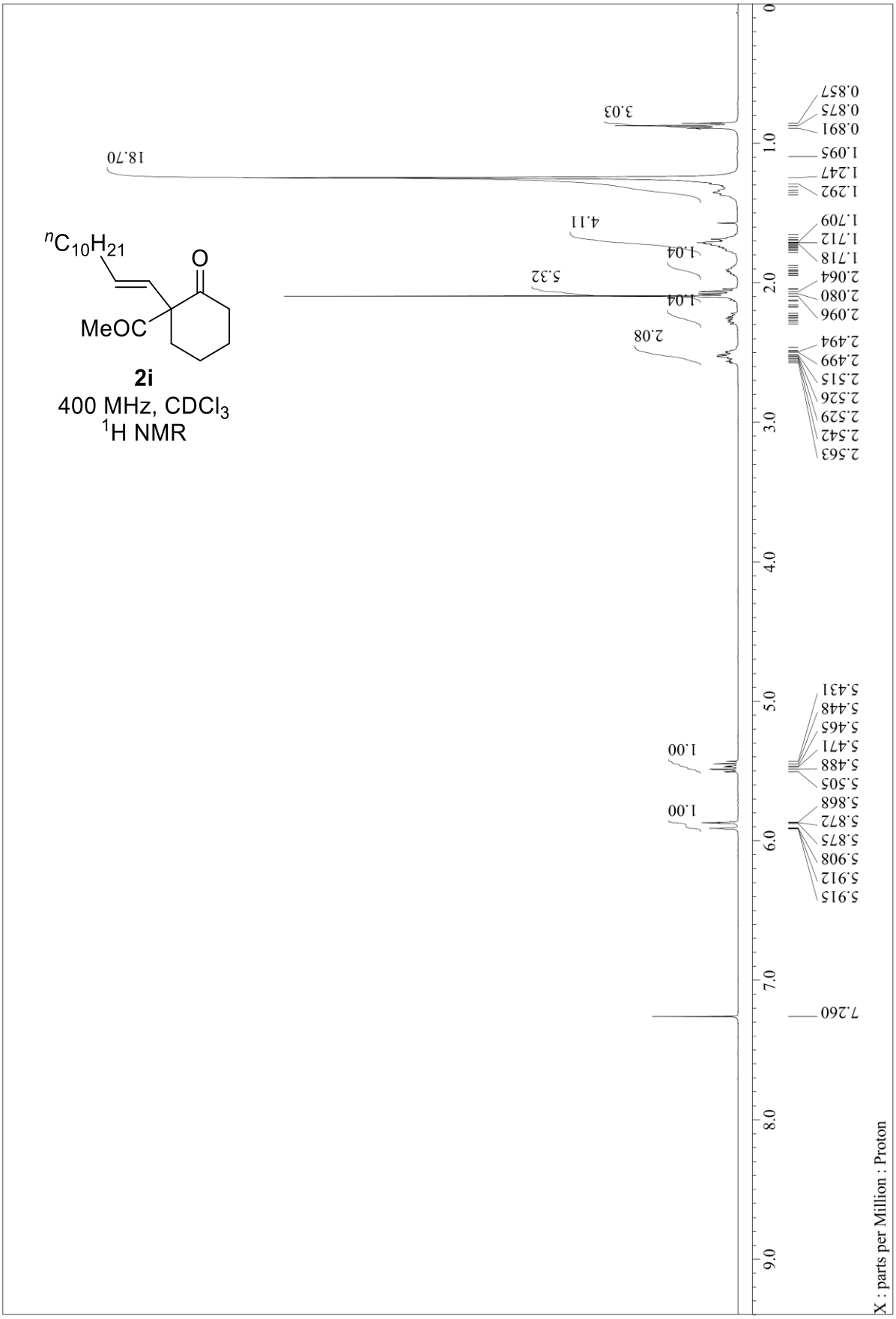

$^{n}C_{10}H_{21}$
O
EtO_2C
2f
100 MHz, $CDCl_3$
^{13}C NMR
13.986
14.100
22.672
28.955
29.003
29.318
29.403
29.575
31.882
32.769
59.828
61.478
76.676
77.000
77.315
126.303
126.704
128.039
128.611
131.881
133.464
133.921
143.056
171.116
194.371
X : parts per Million : Carbon13

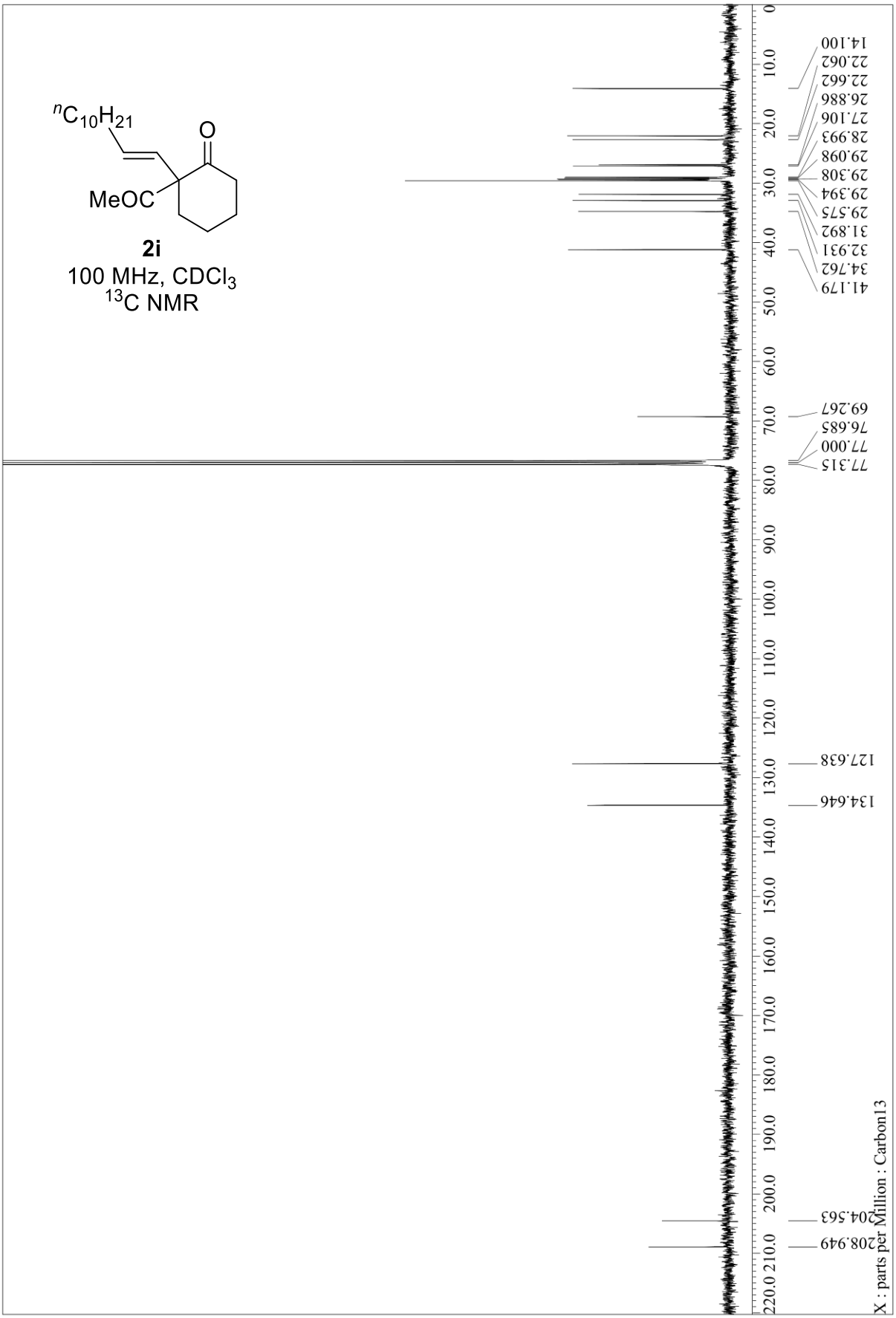

$^{n}C_{10}H_{21}$
O
EtO_2C
O
2g
400 MHz, $CDCl_3$
^{1}H NMR
18.80
3.23
3.80
2.08
2.11
1.06
1.07
2.12
1.00
1.00
1.02
1.00
0.859
0.876
0.893
1.161
1.179
1.197
1.227
2.041
2.051
2.065
2.070
2.082
2.087
2.101
4.157
4.173
4.175
4.191
4.208
4.211
4.352
4.380
4.904
4.932
5.725
5.739
5.765
5.779
5.789
5.829
6.940
6.960
7.031
7.033
7.050
7.260
7.468
7.472
7.930
7.934
7.950
7.954
X : parts per Million : Proton

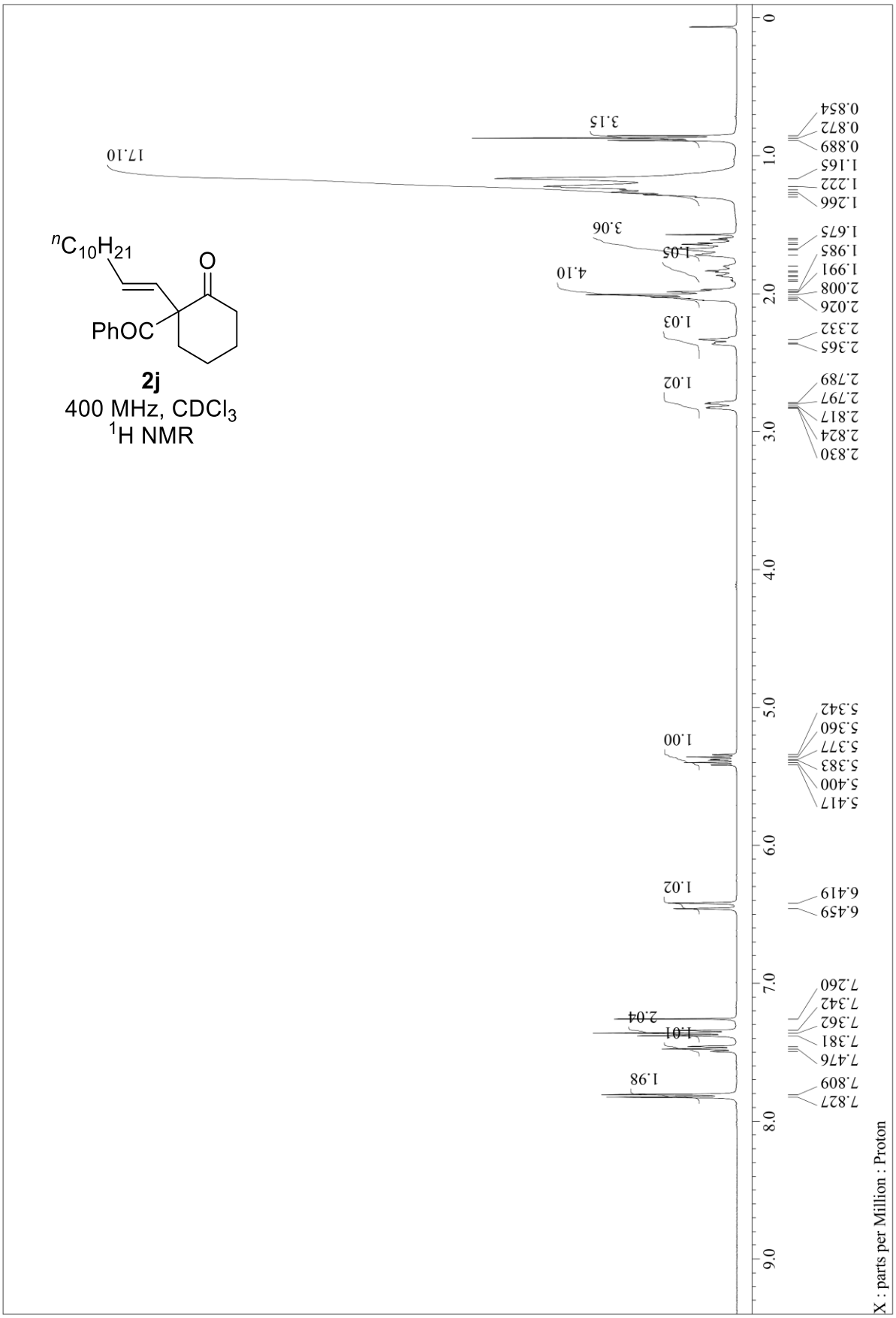

$^{n}C_{10}H_{21}$
O
EtO_2C
O
2g
100 MHz, $CDCl_3$
^{13}C NMR
13.910
14.100
22.672
28.965
29.318
29.394
29.556
29.585
31.892
32.903
58.579
62.050
72.042
76.685
77.000
77.324
117.694
120.030
121.679
122.003
127.905
135.962
136.010
160.828
168.513
188.488
X : parts per Million : Carbon13

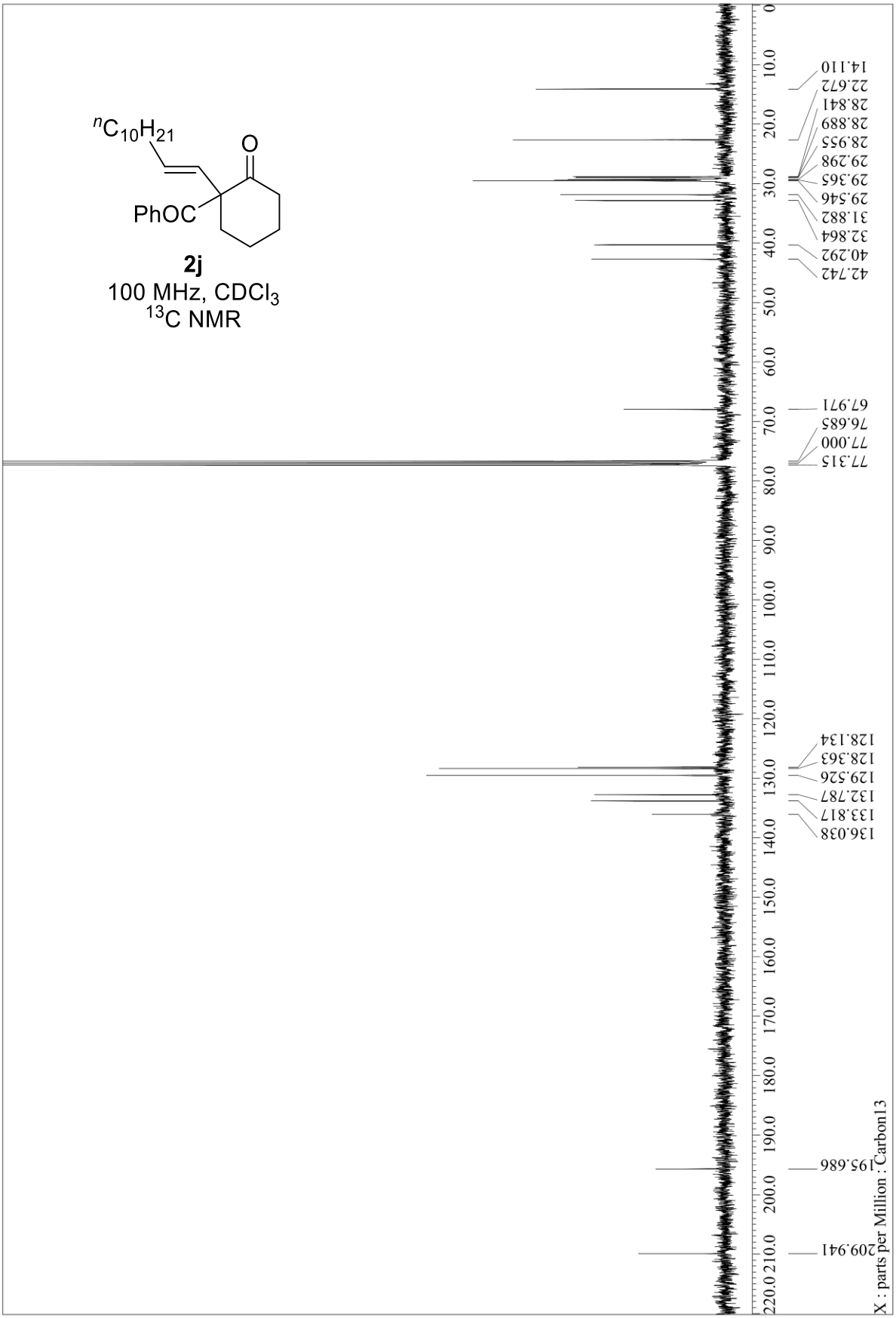

$^{n}C_{10}H_{21}$
O
O
EtO_2C
2h
400 MHz, $CDCl_3$
^{1}H NMR
27.88
4.28
3.57
2.22
1.02
2.37
1.09
1.00
7.260
X : parts per Million : Proton

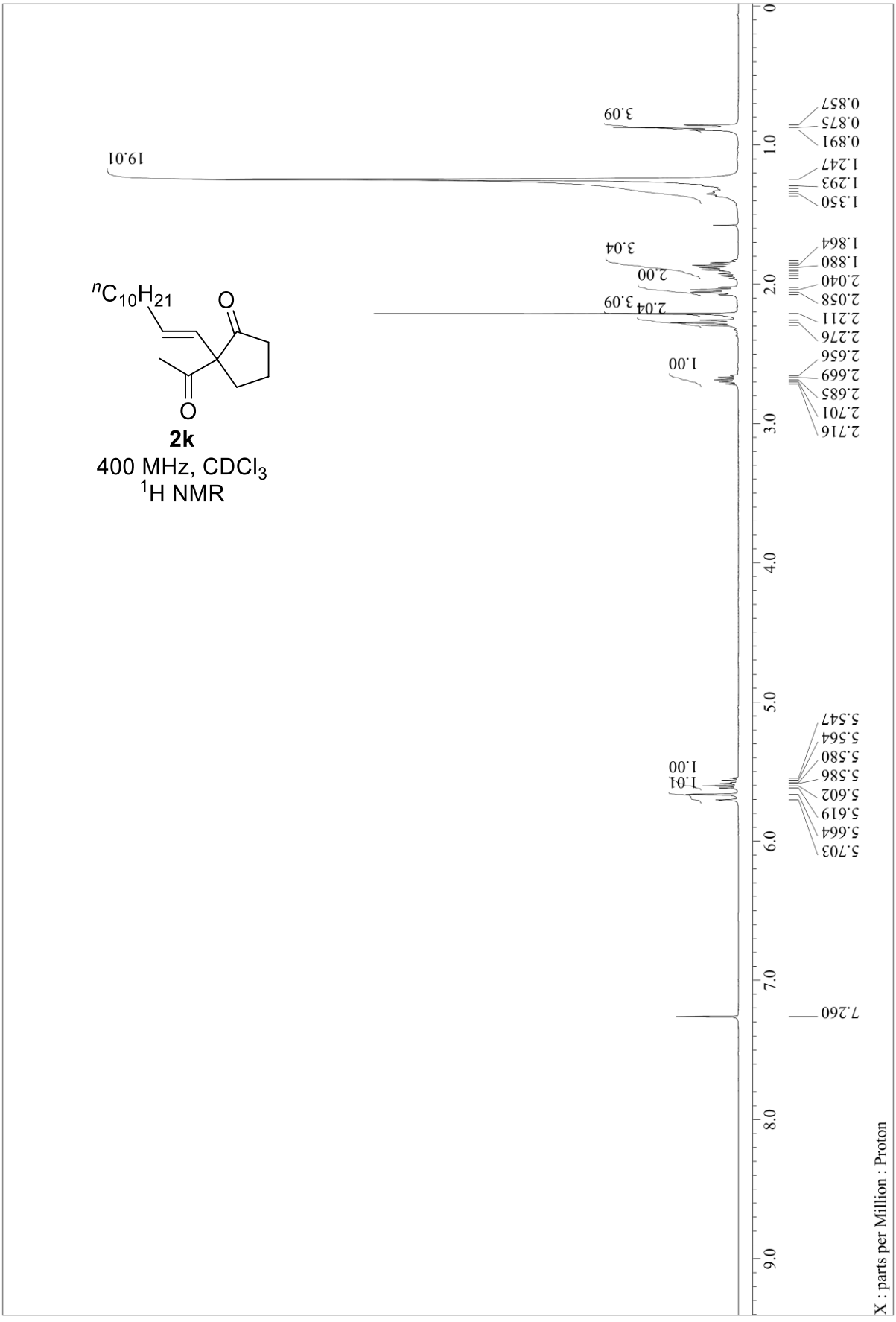

$^{n}C_{10}H_{21}$
O
O
EtO_2C
2h
100 MHz, $CDCl_3$
^{13}C NMR
13.957
14.100
19.754
22.672
28.927
29.003
29.079
29.318
29.422
29.594
31.892
32.569
56.291
62.212
68.857
76.685
77.000
77.315
127.152
133.683
169.161
170.248
X : parts per Million : Carbon13

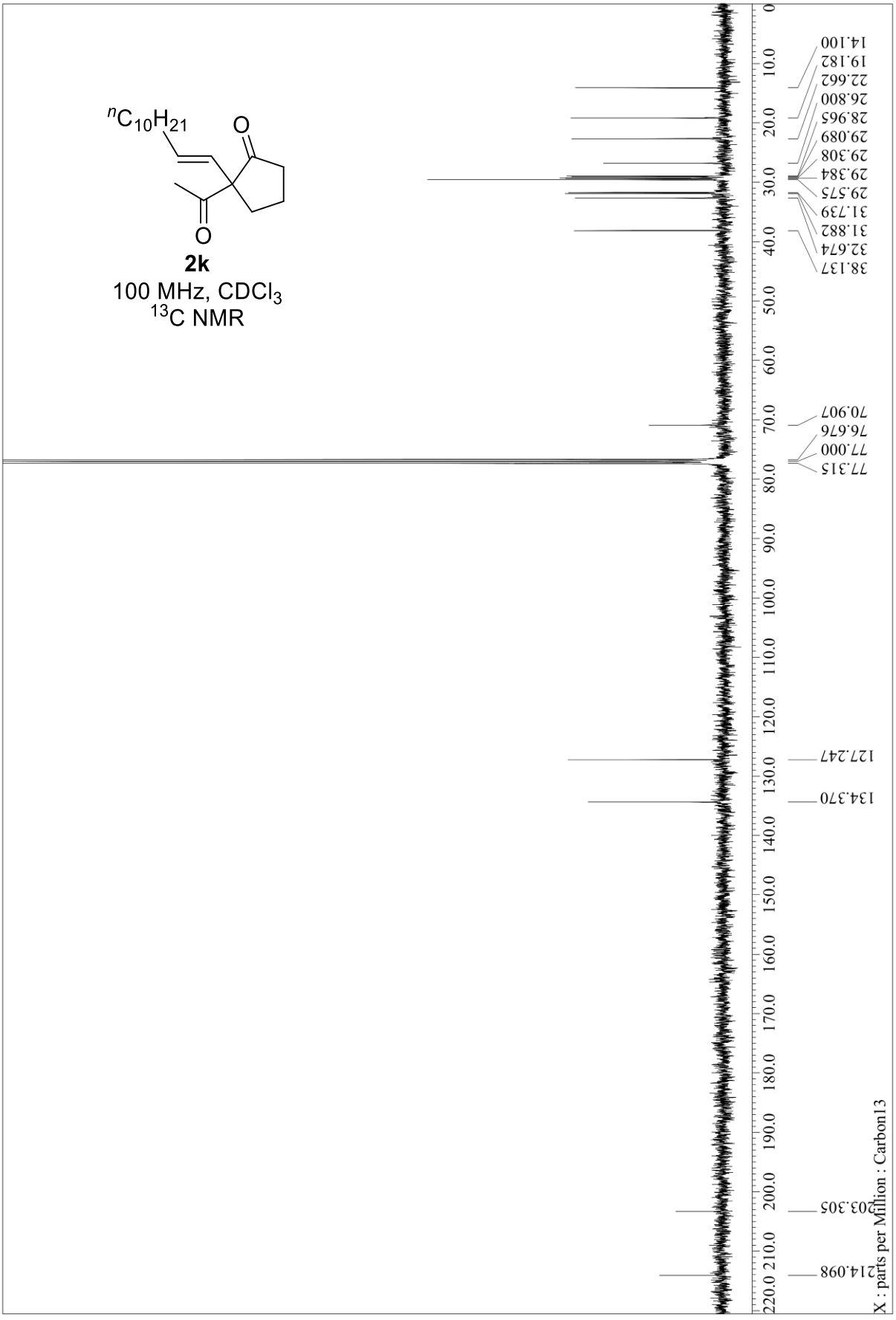

$^{n}C_{10}H_{21}$
O
MeOC
2i
400 MHz, CDCl3
1H NMR
18.70
3.03
4.11
1.04
5.32
1.04
2.08
1.00
1.00
7.260
X : parts per Million : Proton

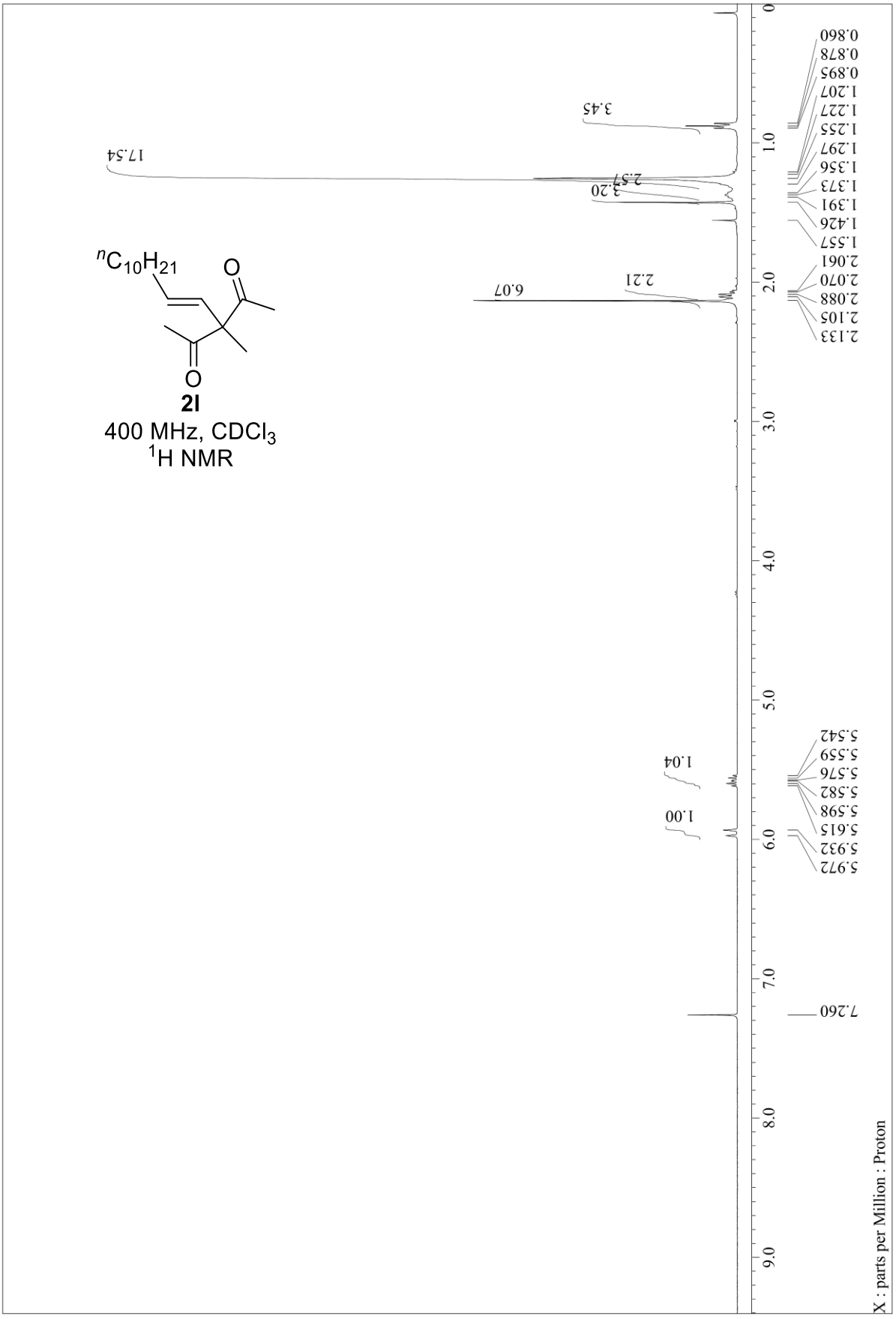

$^{n}C_{10}H_{21}$
O
MeOC
2i
100 MHz, $CDCl_3$
^{13}C NMR
14.100
22.062
22.662
26.886
27.106
28.993
29.098
29.308
29.394
29.575
31.892
32.931
34.762
41.179
69.267
76.685
77.000
77.315
127.638
134.646
204.563
208.949
X : parts per Million : Carbon13

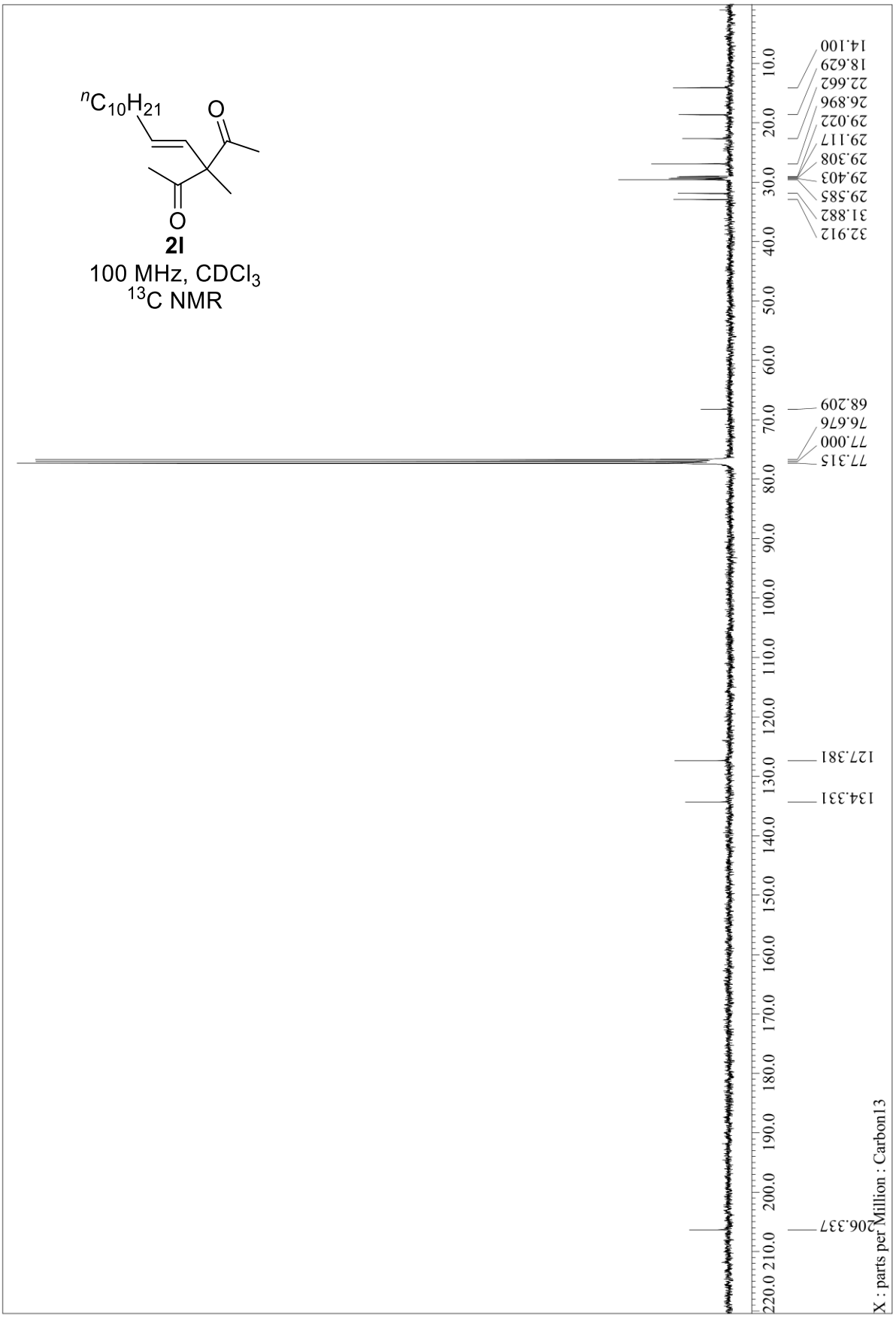

$^{n}C_{10}H_{21}$
O
PhOC
2j
400 MHz, $CDCl_3$
^{1}H NMR
17.10
3.15
3.06
1.05
4.10
1.03
1.02
1.00
1.02
2.04
1.01
1.98
0.854
0.872
0.889
1.165
1.222
1.266
1.675
1.985
1.991
2.008
2.026
2.332
2.365
2.789
2.797
2.817
2.824
2.830
5.342
5.360
5.377
5.383
5.400
5.417
6.419
6.459
7.260
7.342
7.362
7.381
7.476
7.809
7.827
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
X : parts per Million : Proton

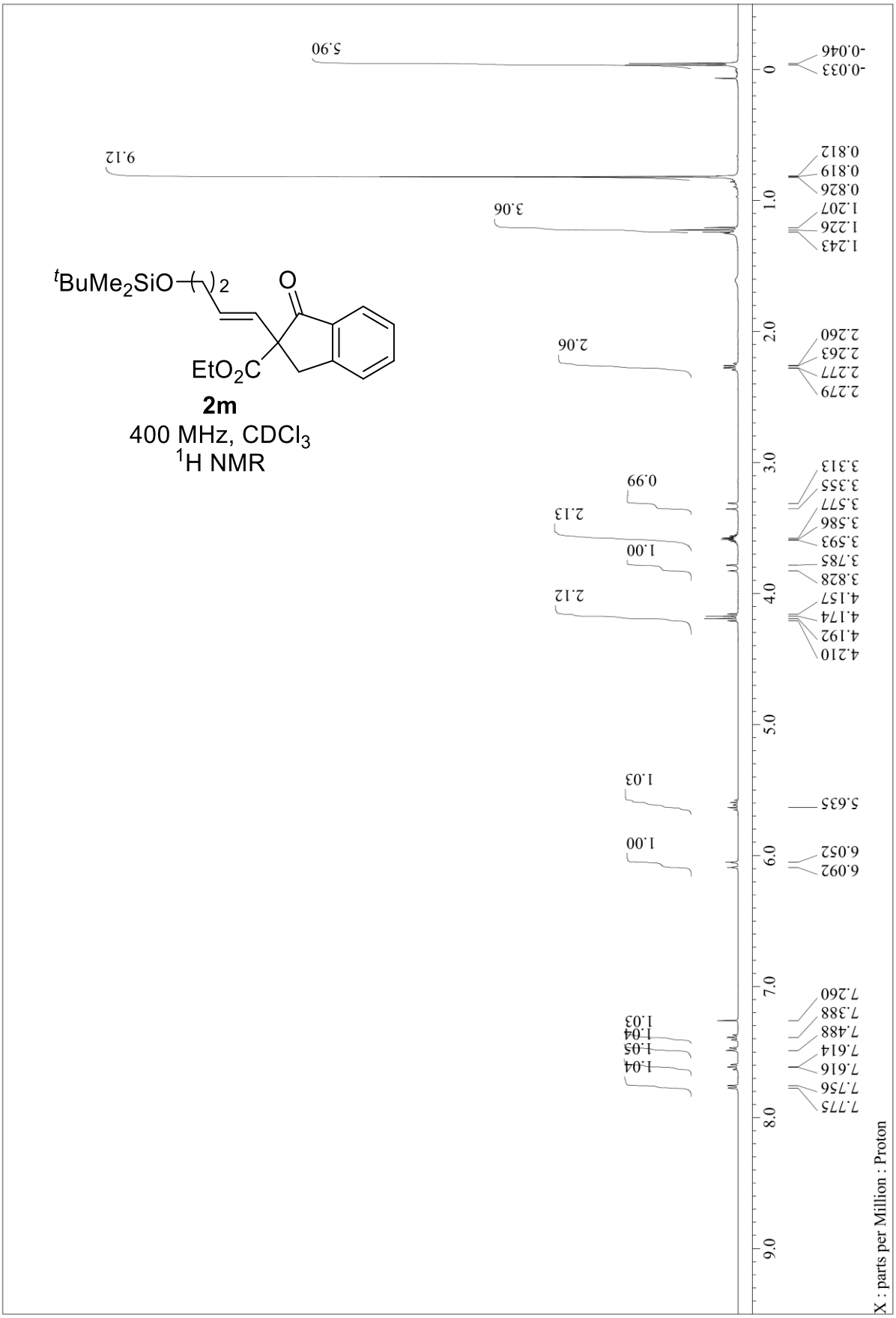

$^{n}C_{10}H_{21}$
O
PhOC
2j
100 MHz, CDCl3
13C NMR
14.110
22.672
28.841
28.889
28.955
29.298
29.365
29.546
31.882
32.864
40.292
42.742
67.971
76.685
77.000
77.315
128.134
128.363
129.526
132.787
133.817
136.038
195.686
209.941
X : parts per Million : Carbon13

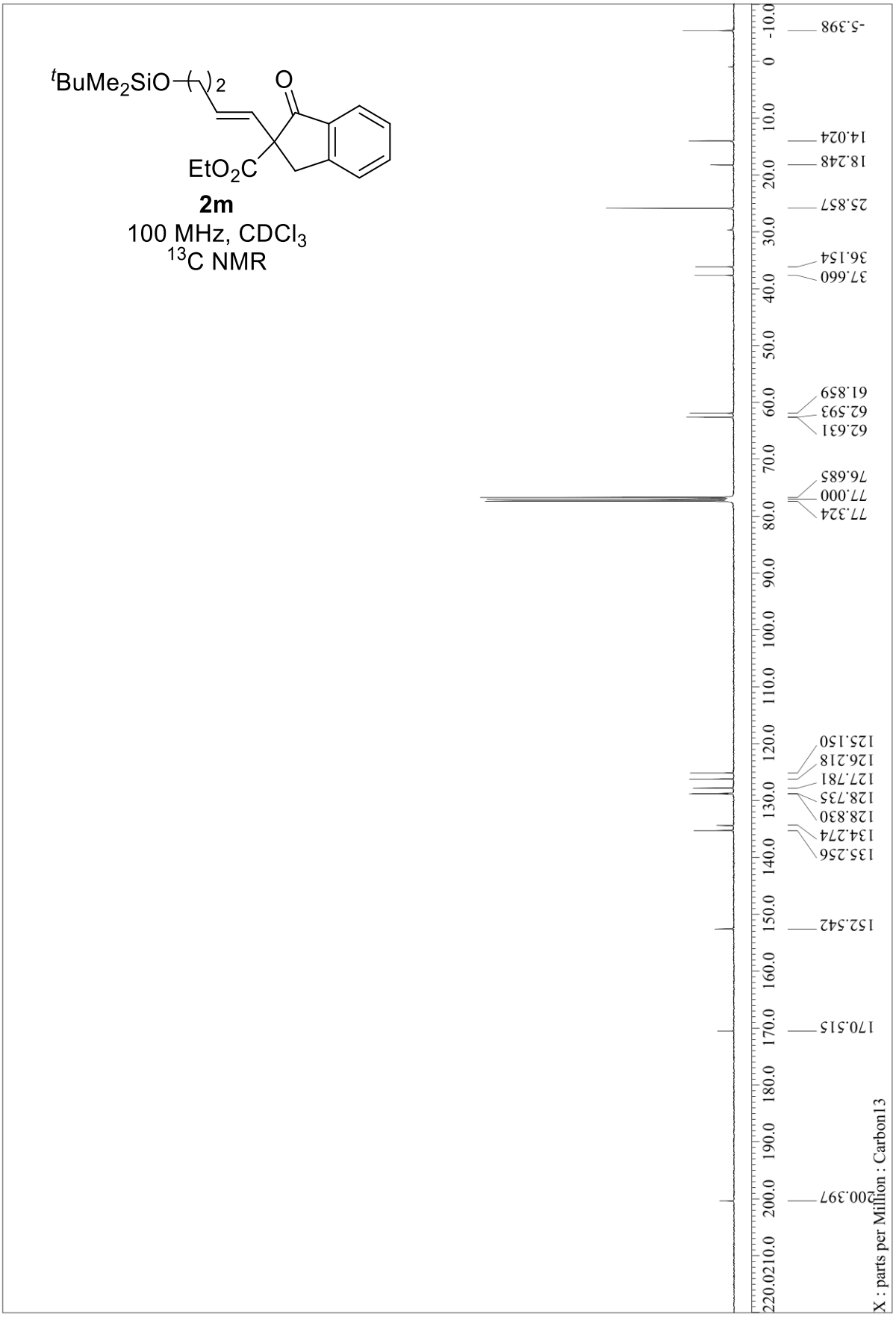

$^{n}C_{10}H_{21}$
O
O
2k
400 MHz, $CDCl_3$
^{1}H NMR
19.01
3.09
3.04
2.00
2.04
3.09
1.00
1.01
1.00
7.260
0.857
0.875
0.891
1.247
1.293
1.350
1.864
1.880
2.040
2.058
2.211
2.276
2.656
2.669
2.685
2.701
2.716
5.547
5.564
5.580
5.586
5.602
5.619
5.664
5.703
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
X : parts per Million : Proton

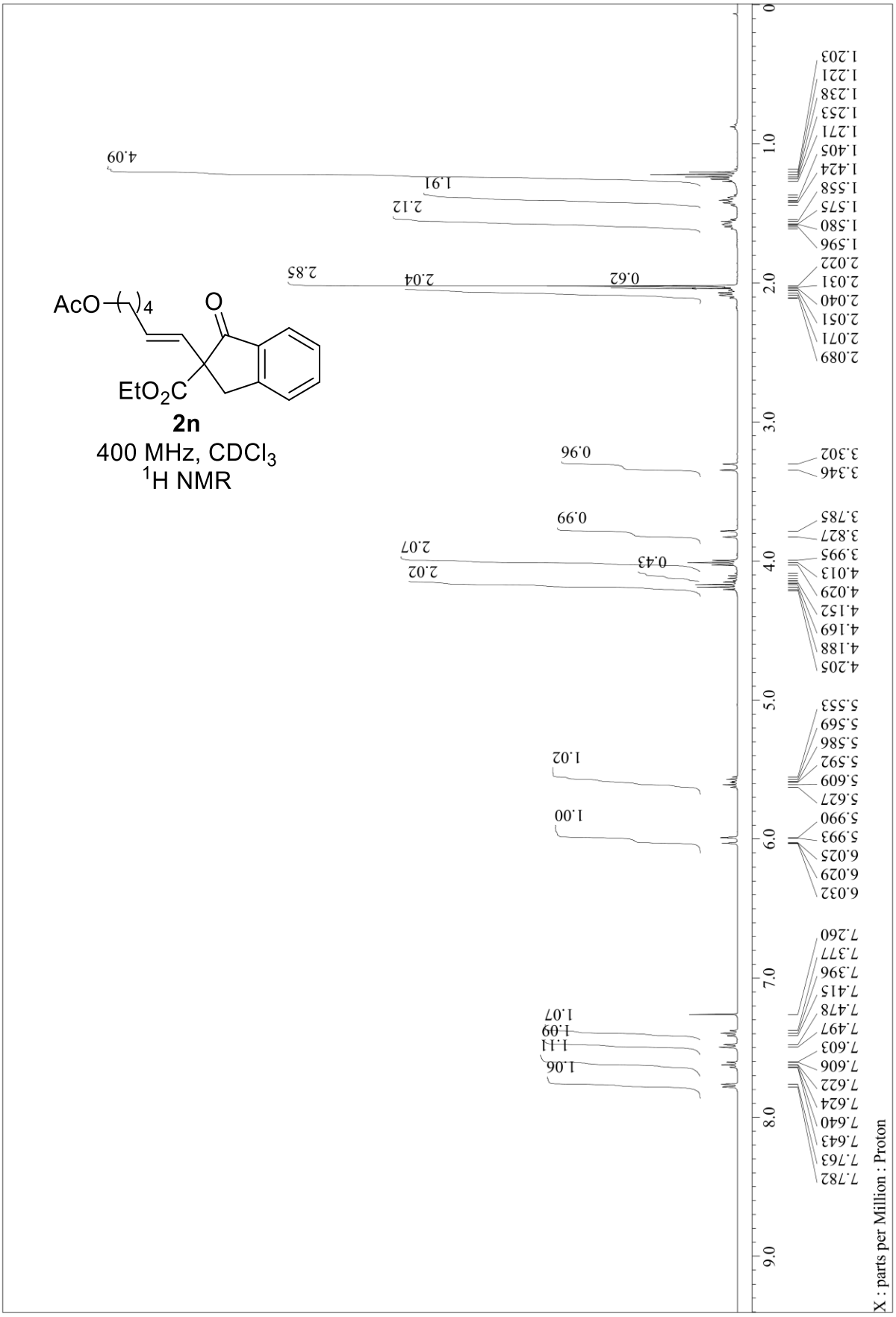

$^{n}C_{10}H_{21}$
O
O
2k
100 MHz, $CDCl_3$
^{13}C NMR
14.100
19.182
22.662
26.800
28.965
29.089
29.308
29.384
29.575
31.739
31.882
32.674
38.137
70.907
76.676
77.000
77.315
127.247
134.370
203.305
214.098
X : parts per Million : Carbon13

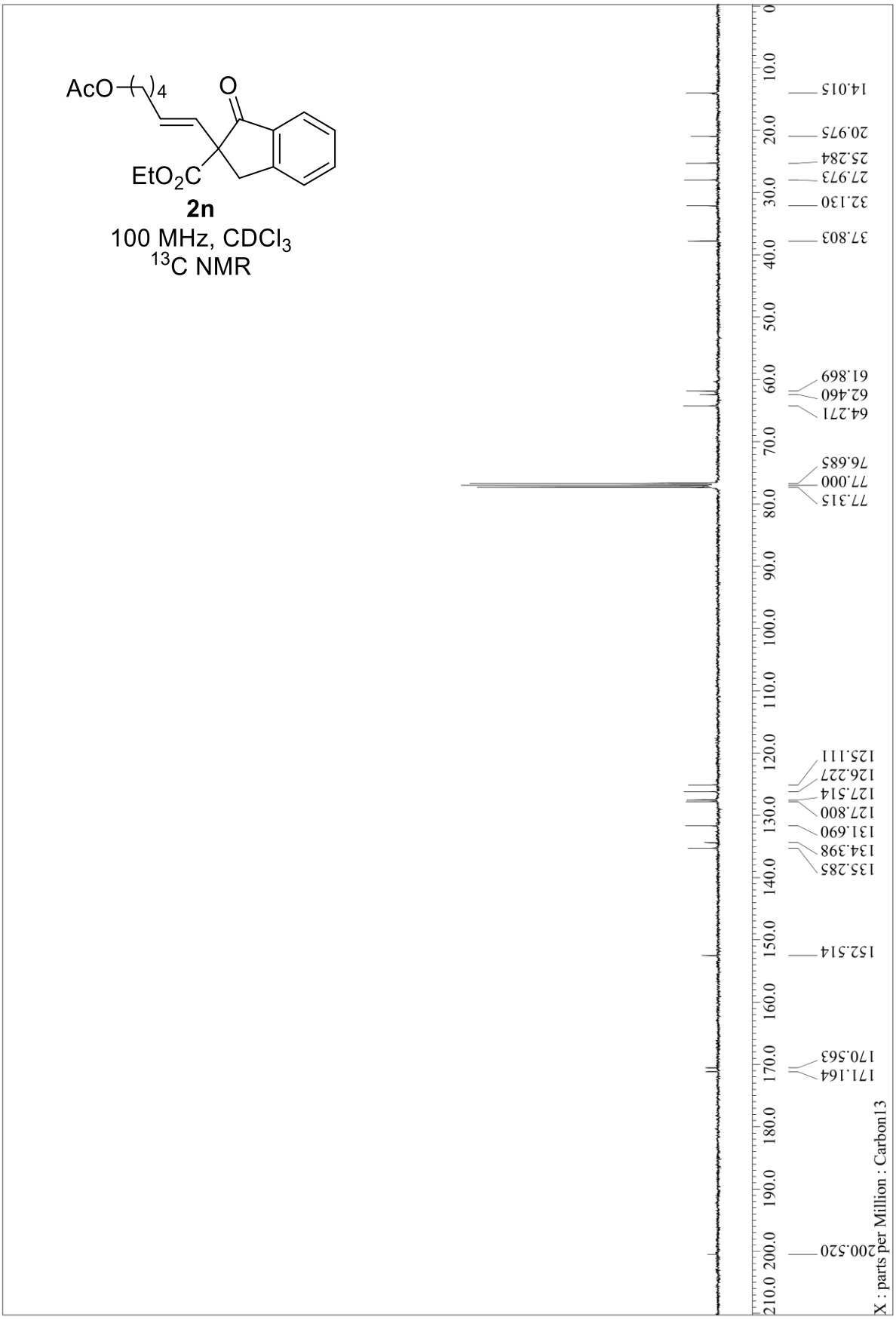

$^{n}C_{10}H_{21}$
O
O
2l
400 MHz, $CDCl_3$
^{1}H NMR
17.54
3.45
3.20
2.57
2.21
6.07
1.04
1.00
7.260
5.972
5.932
5.615
5.598
5.582
5.576
5.559
5.542
2.133
2.105
2.088
2.070
2.061
1.557
1.426
1.391
1.373
1.356
1.297
1.255
1.227
1.207
0.895
0.878
0.860
X : parts per Million : Proton

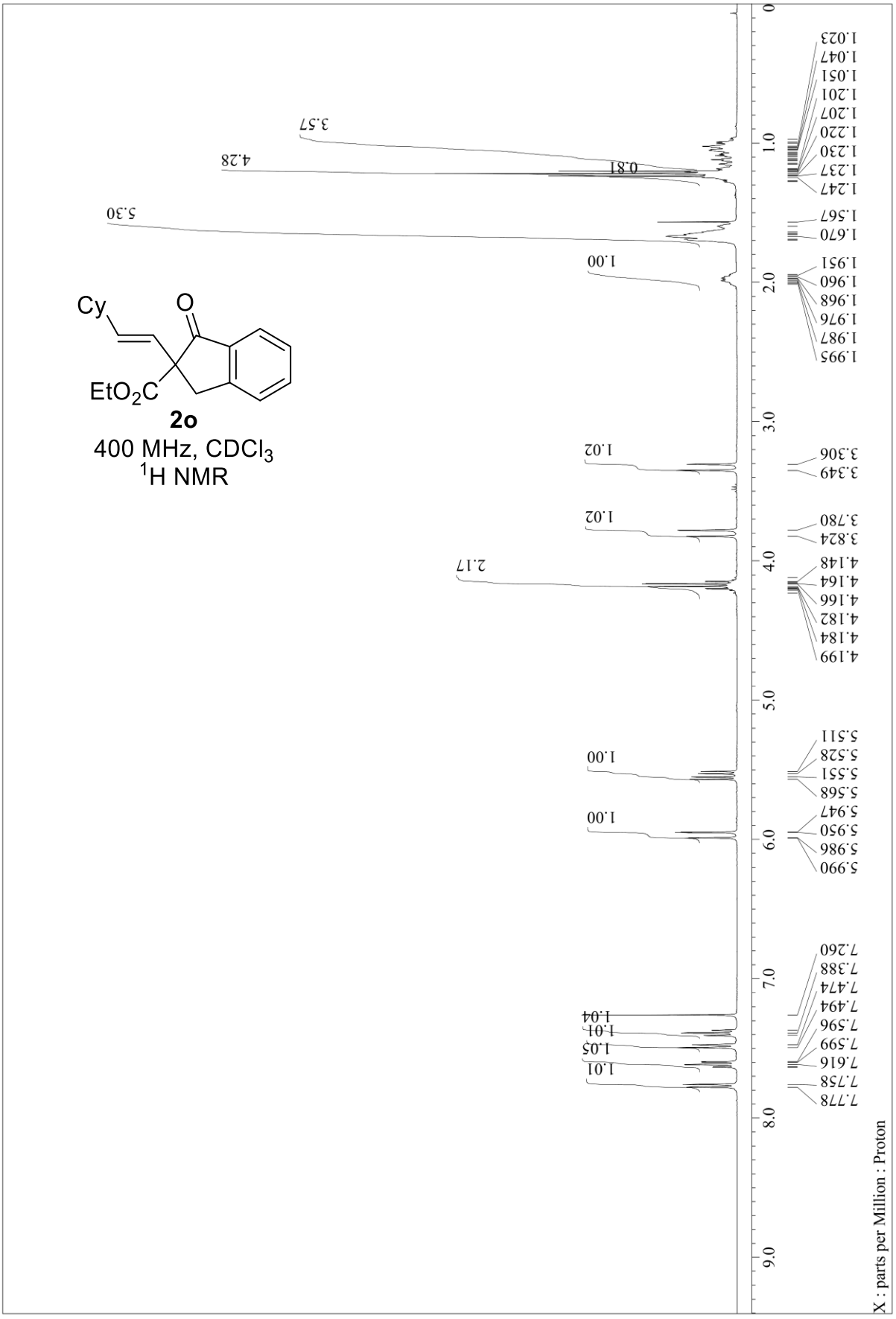

$^{n}C_{10}H_{21}$
O
O
2l
100 MHz, $CDCl_3$
^{13}C NMR
14.100
18.629
22.662
26.896
29.022
29.117
29.308
29.403
29.585
31.882
32.912
68.209
76.676
77.000
77.315
127.381
134.331
206.337
X : parts per Million : Carbon13

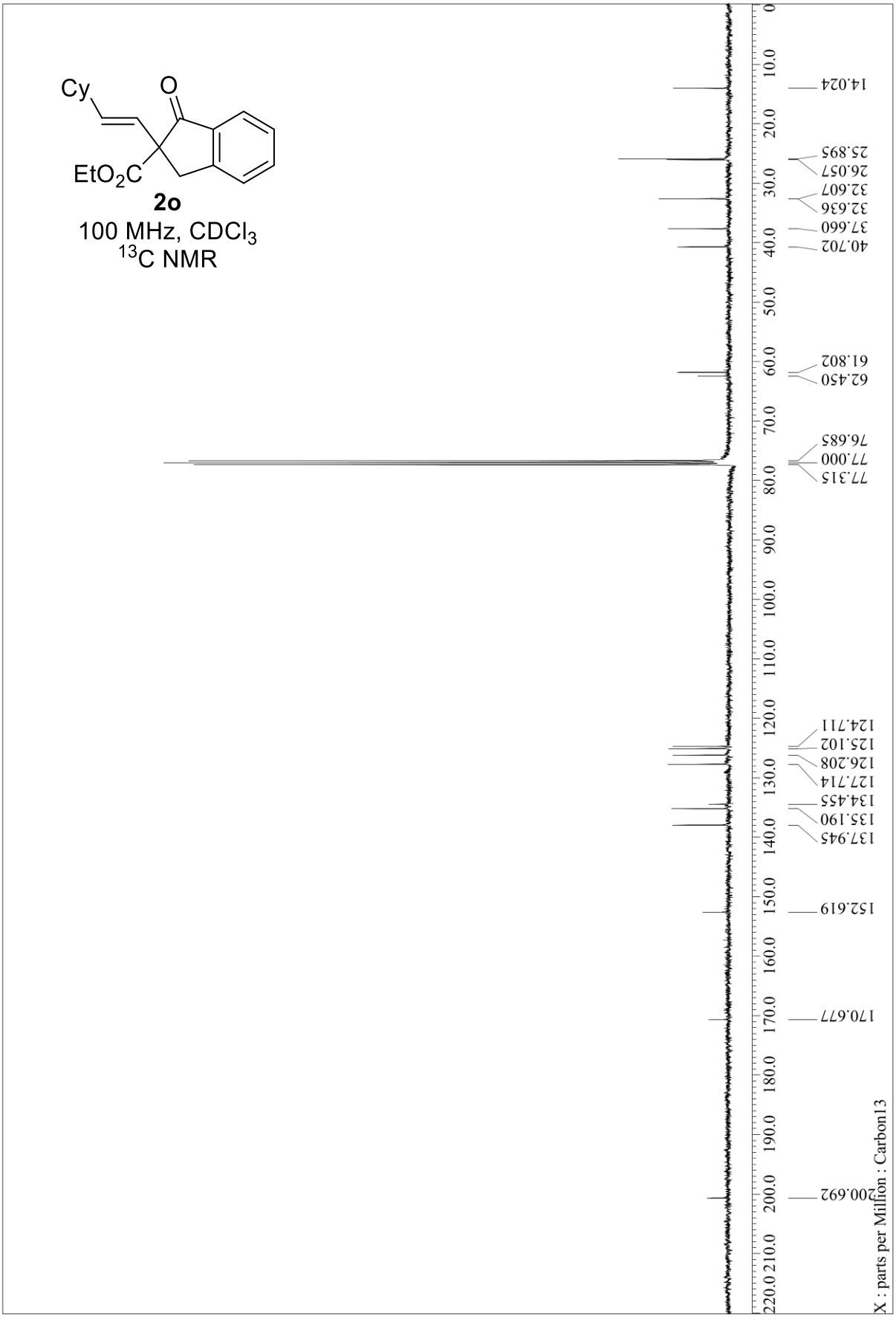

^{t}BuMe$_2$SiO
2
O
EtO$_2$C
2m
400 MHz, CDCl$_3$
^{1}H NMR
X : parts per Million : Proton

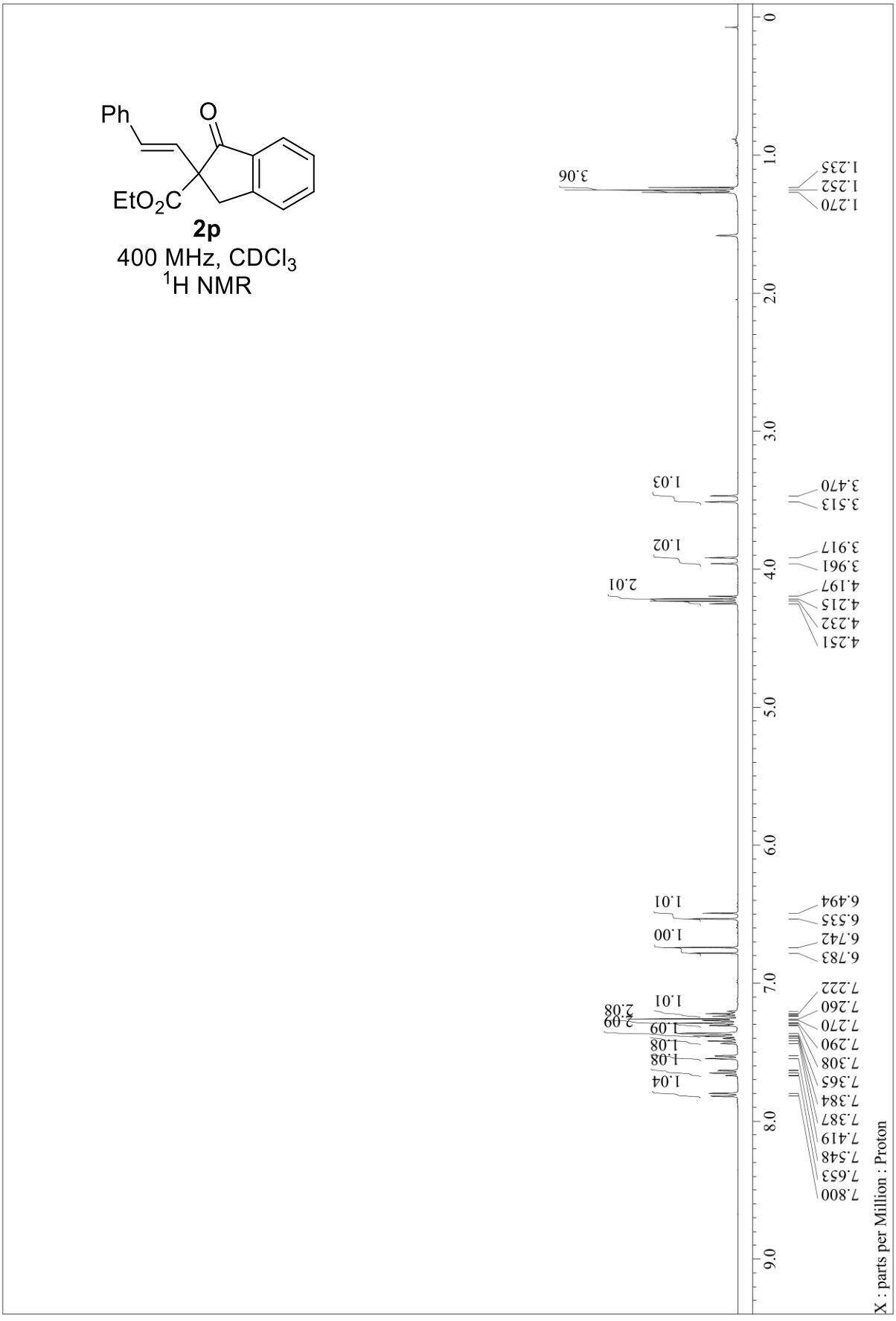

^{t}BuMe$_2$SiO
2
O
EtO$_2$C
2m
100 MHz, CDCl$_3$
^{13}C NMR
-5.398
14.024
18.248
25.857
36.154
37.660
61.859
62.593
62.631
76.685
77.000
77.324
125.150
126.218
127.781
128.735
128.830
134.274
135.256
152.542
170.515
200.397
X : parts per Million : Carbon13

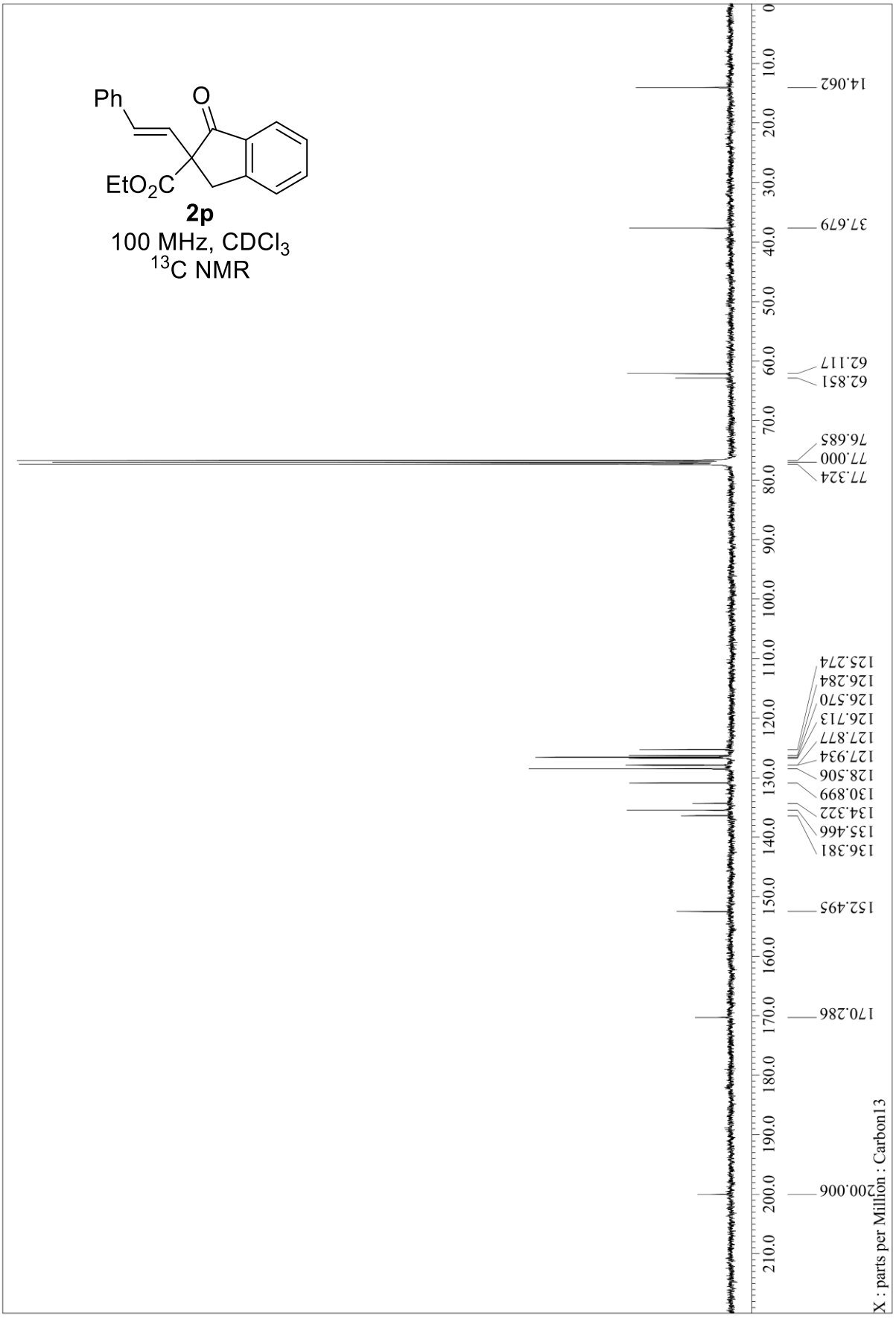

AcO
4
O
EtO2C
2n
400 MHz, CDCl3
1H NMR
X : parts per Million : Proton

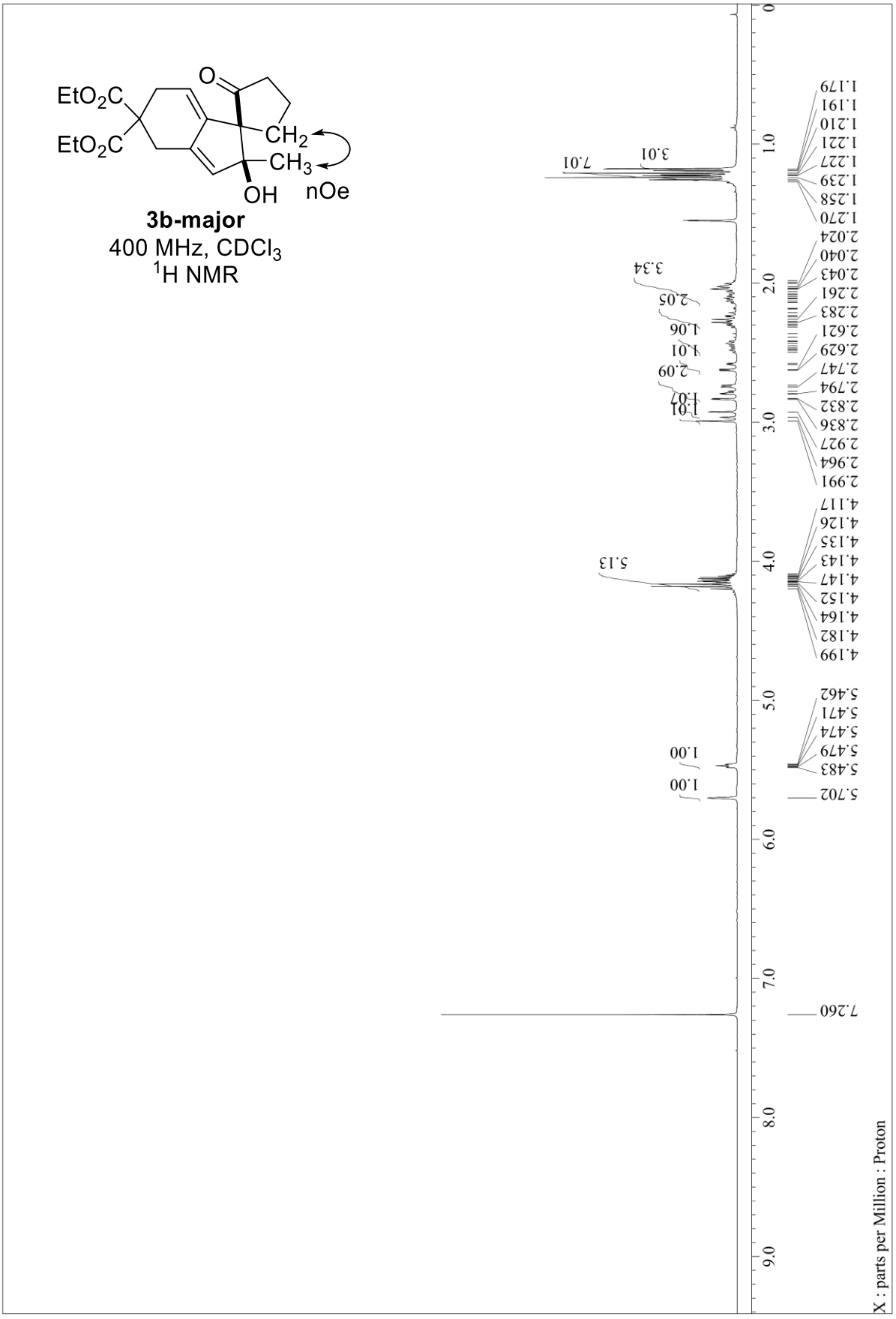

AcO
4
O
EtO2C
2n
100 MHz, CDCl3
13C NMR
14.015
20.975
25.284
27.973
32.130
37.803
61.869
62.460
64.271
76.685
77.000
77.315
125.111
126.227
127.514
127.800
131.690
134.398
135.285
152.514
170.563
171.164
200.520
X : parts per Million : Carbon13

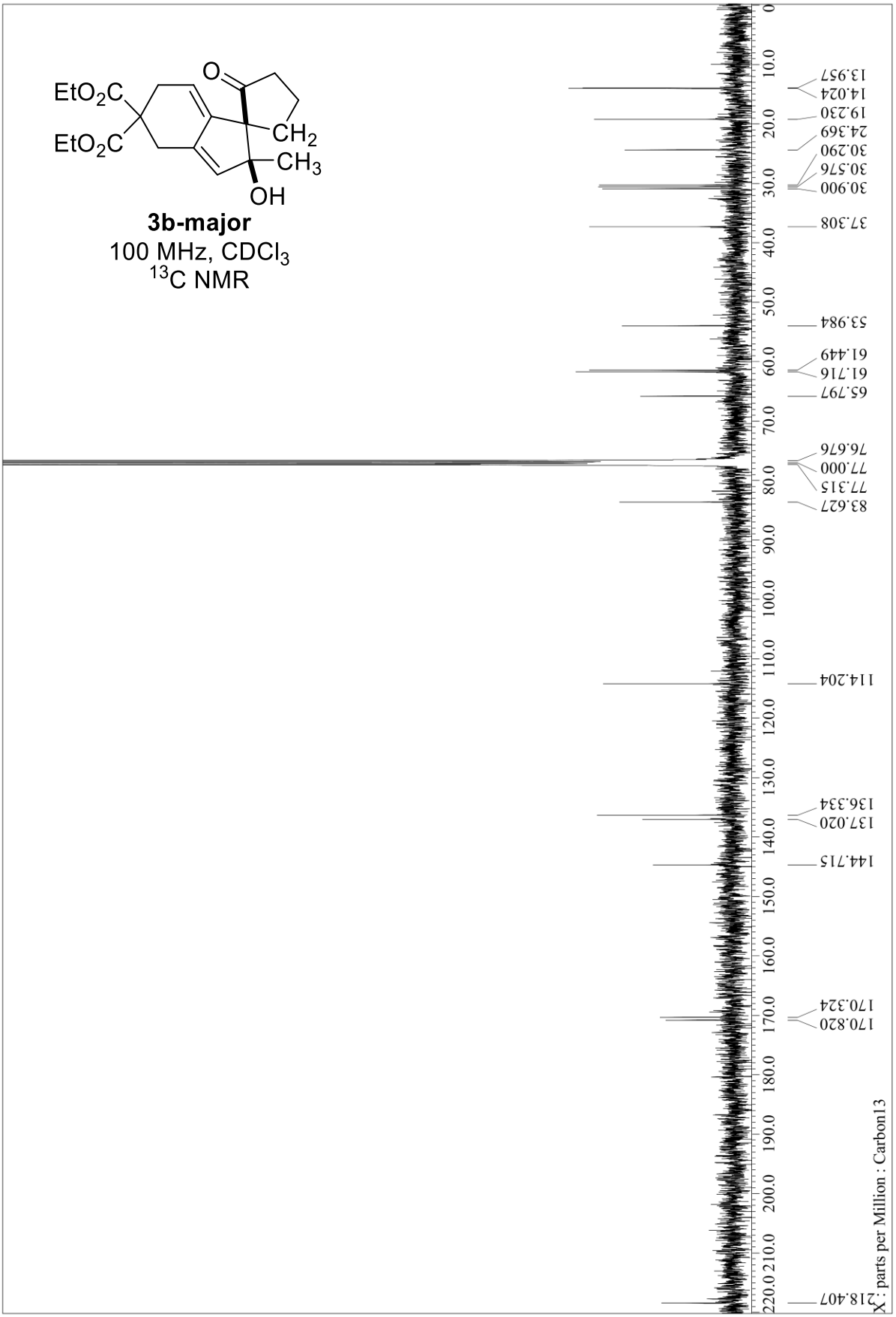

Cy
O
EtO2C
2o
400 MHz, CDCl3
1H NMR
X : parts per Million : Proton

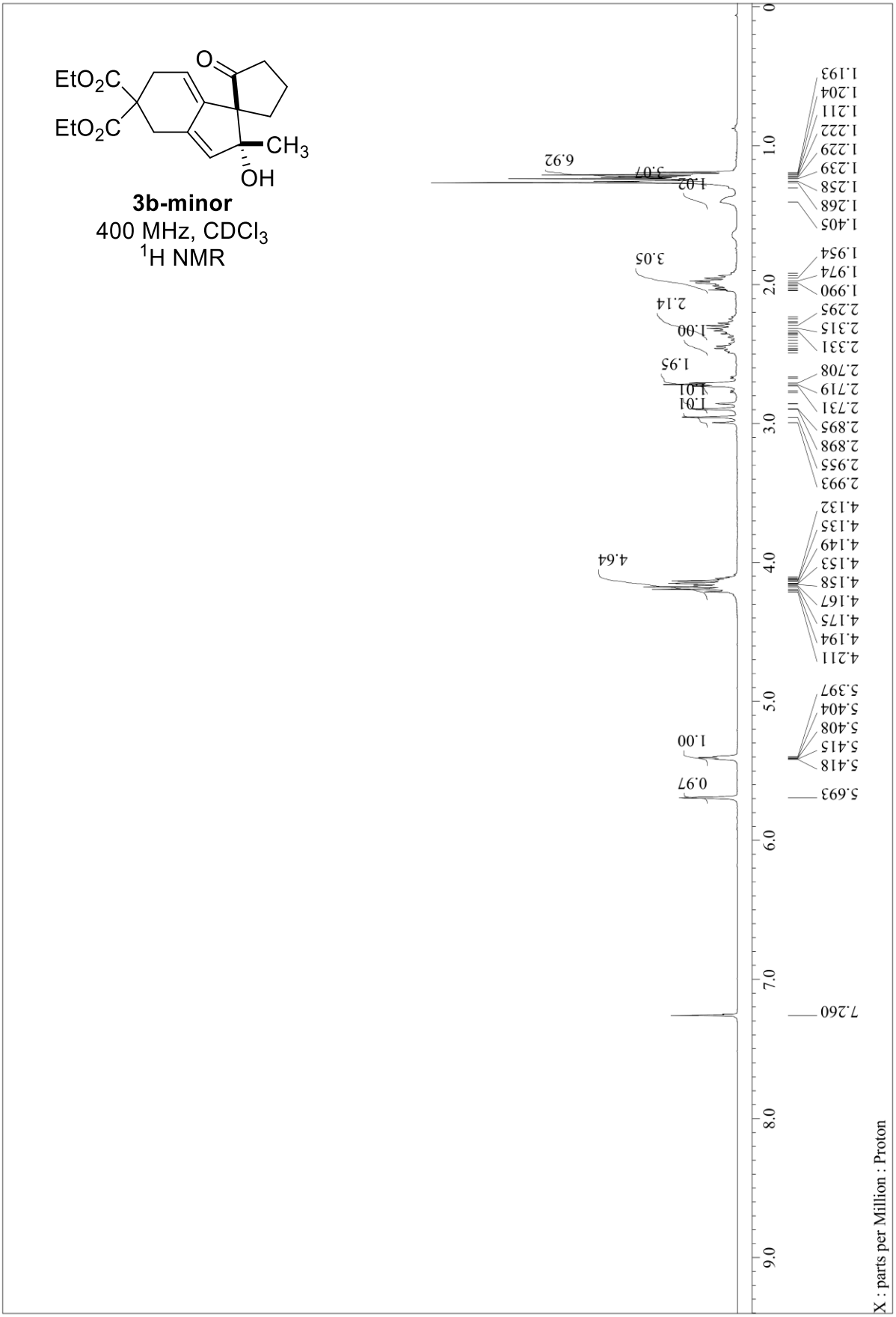

Cy
O
EtO2C
2o
100 MHz, CDCl3
13C NMR
14.024
25.895
26.057
32.607
32.636
37.660
40.702
61.802
62.450
76.685
77.000
77.315
124.711
125.102
126.208
127.714
134.455
135.190
137.945
152.619
170.677
200.692
X : parts per Million : Carbon13

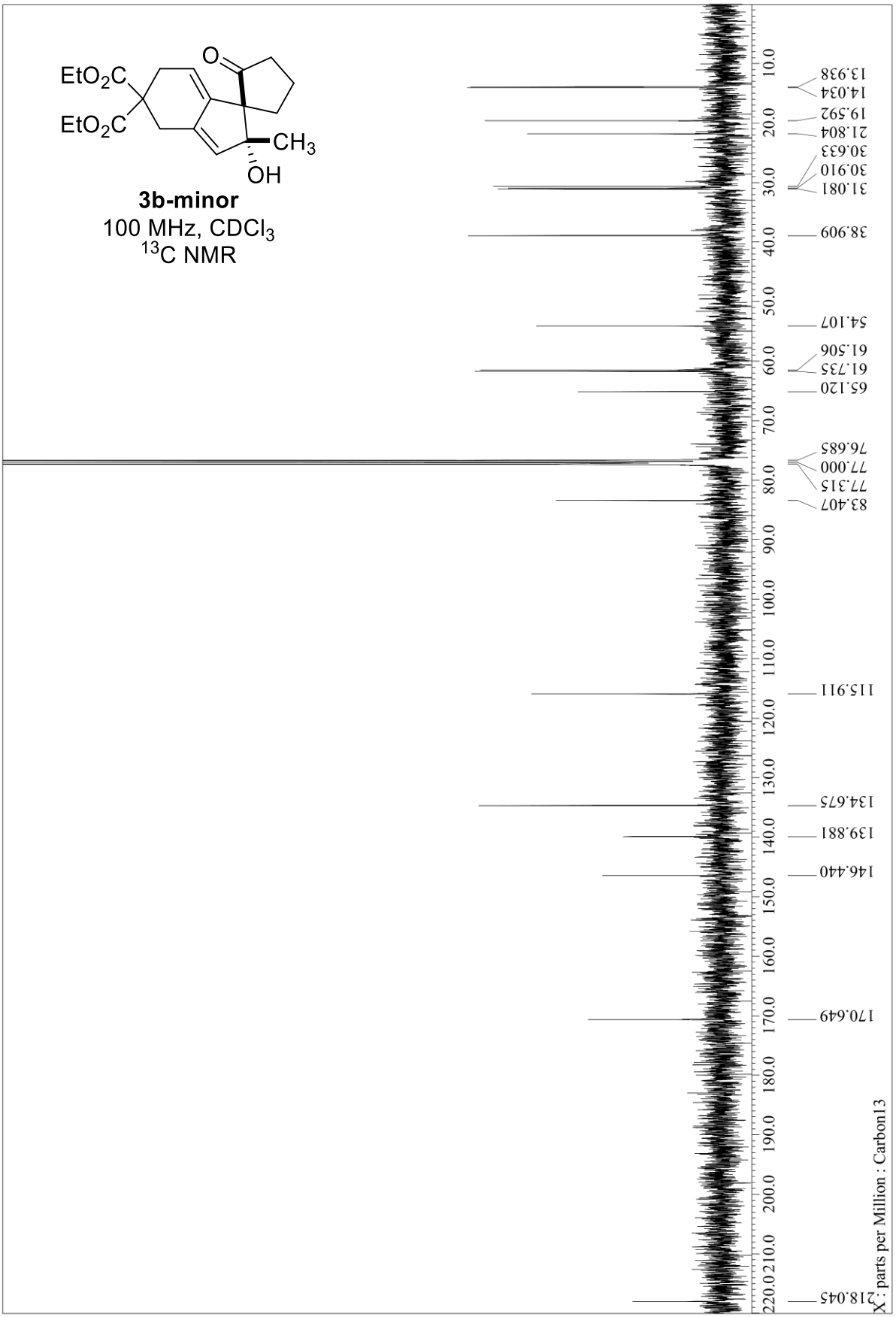

Ph
O
EtO2C
2p
400 MHz, CDCl3
1H NMR
3.06
1.270
1.252
1.235
1.03
3.470
3.513
1.02
3.917
3.961
2.01
4.197
4.215
4.232
4.251
1.01
6.494
6.535
1.00
6.742
6.783
1.01
2.08
2.09
1.09
1.08
1.08
1.04
7.222
7.260
7.270
7.290
7.308
7.365
7.384
7.387
7.419
7.548
7.653
7.800
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
X : parts per Million : Proton

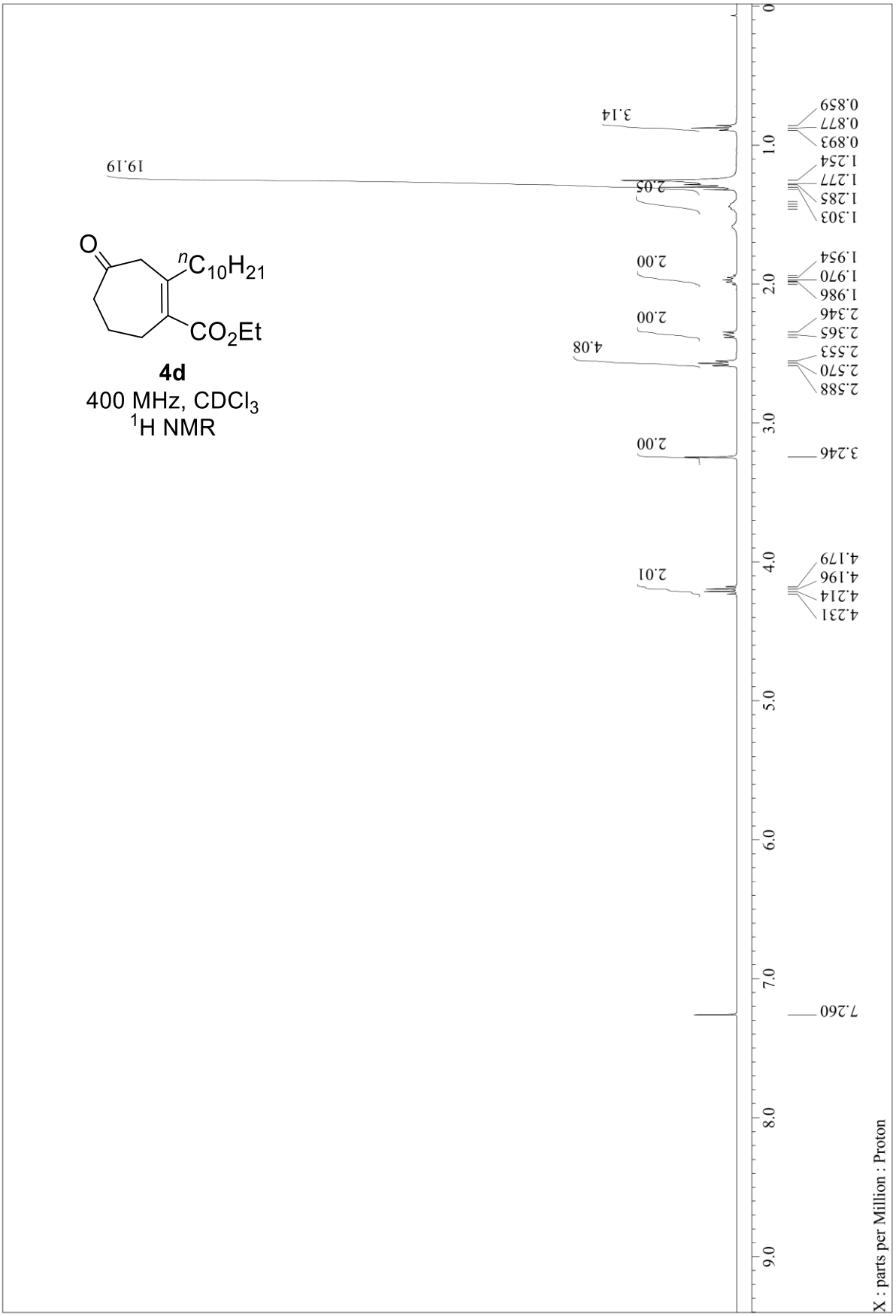

Ph
O
EtO2C
2p
100 MHz, CDCl3
13C NMR
14.062
37.679
62.117
62.851
76.685
77.000
77.324
125.274
126.284
126.570
126.713
127.877
127.934
128.506
130.899
134.322
135.466
136.381
152.495
170.286
200.006
X : parts per Million : Carbon13

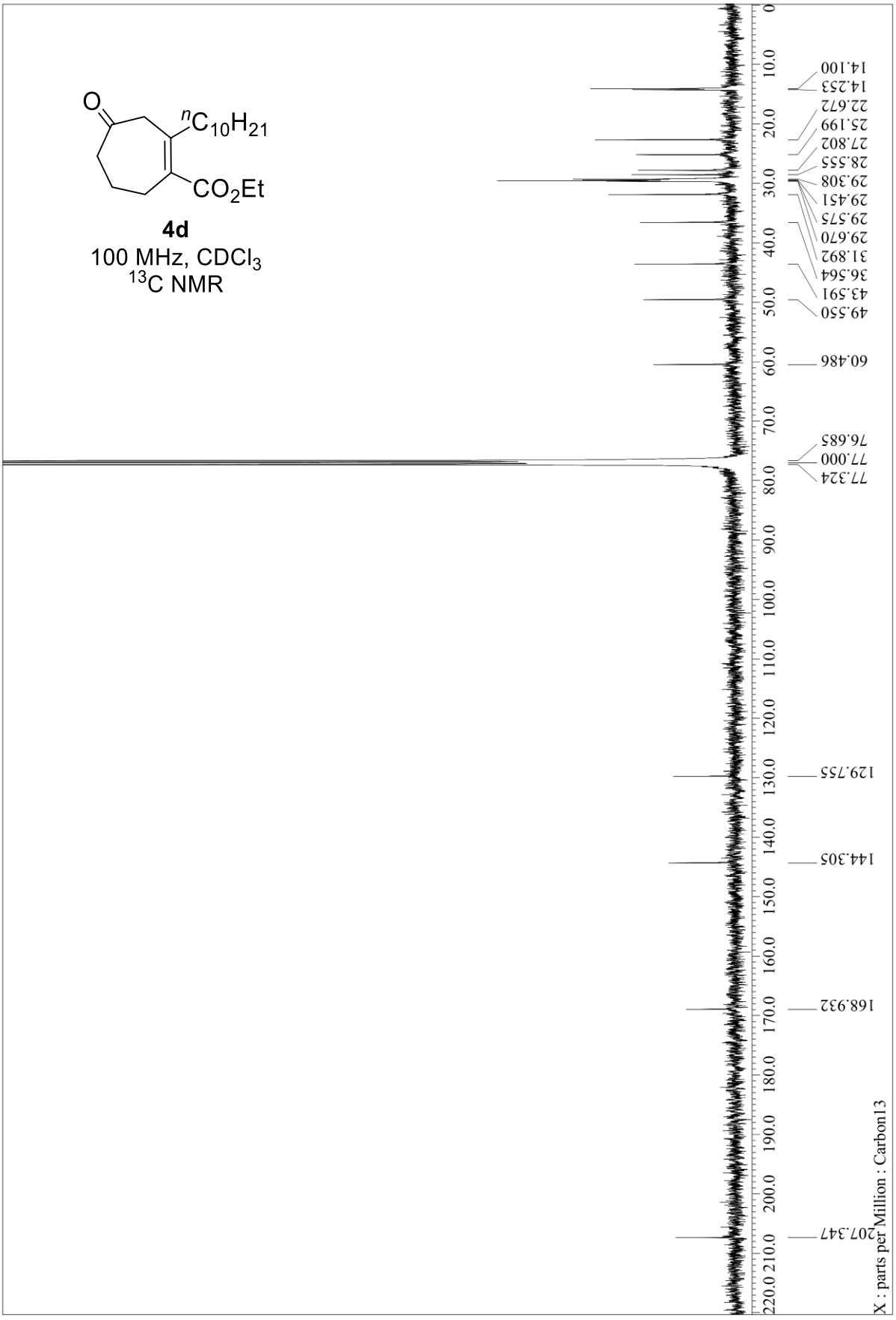

EtO2C
EtO2C
O
CH2
CH3
OH
nOe
3b-major
400 MHz, CDCl3
1H NMR
7.260
5.702
5.483
5.479
5.474
5.471
5.462
4.199
4.182
4.164
4.152
4.147
4.143
4.135
4.126
4.117
2.991
2.964
2.927
2.836
2.832
2.794
2.747
2.629
2.621
2.283
2.261
2.043
2.040
2.024
1.270
1.258
1.239
1.227
1.221
1.210
1.191
1.179
7.01
3.01
3.34
2.05
1.06
1.01
2.09
1.07
1.01
5.13
1.00
1.00
X : parts per Million : Proton

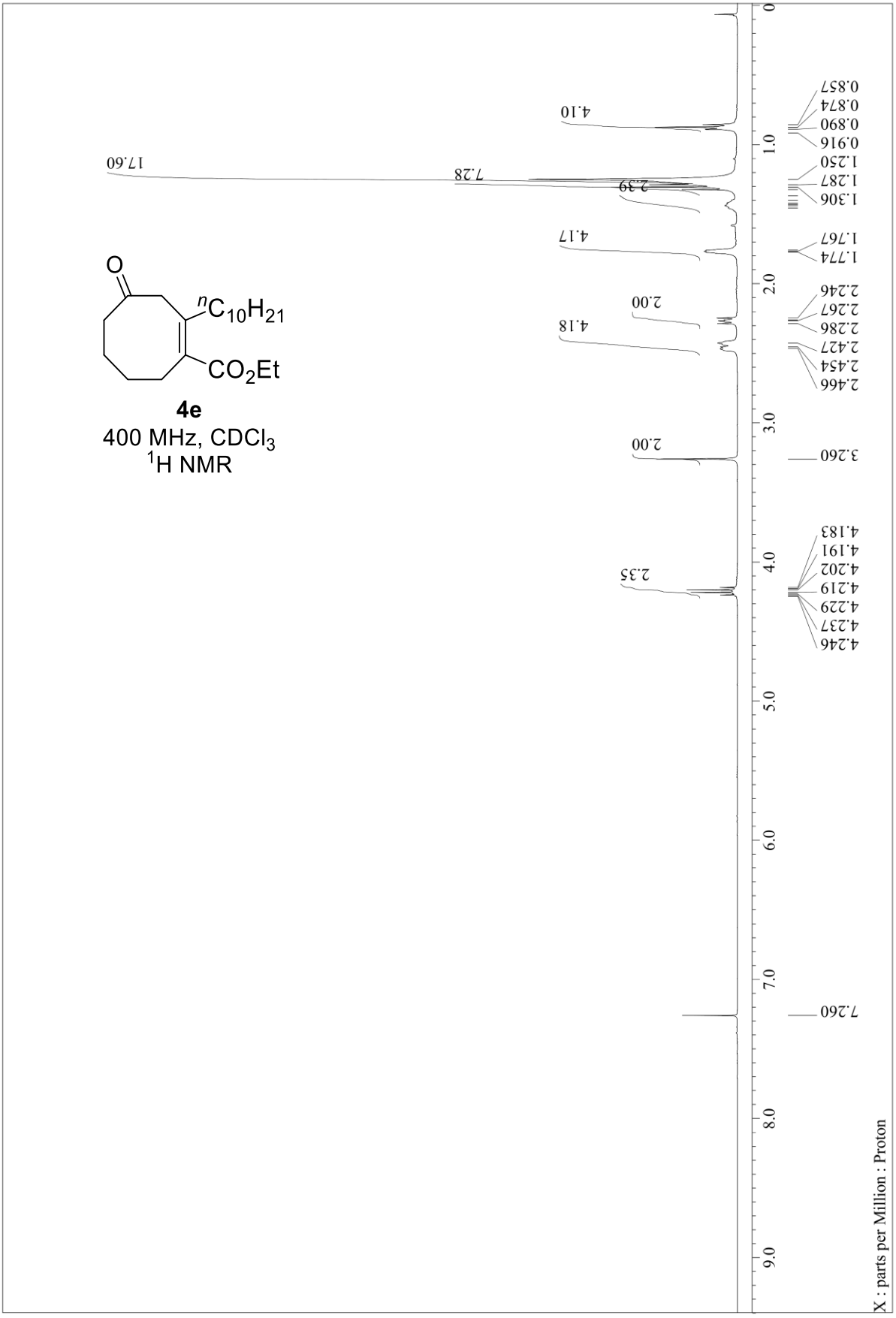

EtO2C
EtO2C
O
CH2
CH3
OH
3b-major
100 MHz, CDCl3
13C NMR
13.957
14.024
19.230
24.369
30.290
30.576
30.900
37.308
53.984
61.449
61.716
65.797
76.676
77.000
77.315
83.627
114.204
136.334
137.020
144.715
170.324
170.820
218.407
X : parts per Million : Carbon13

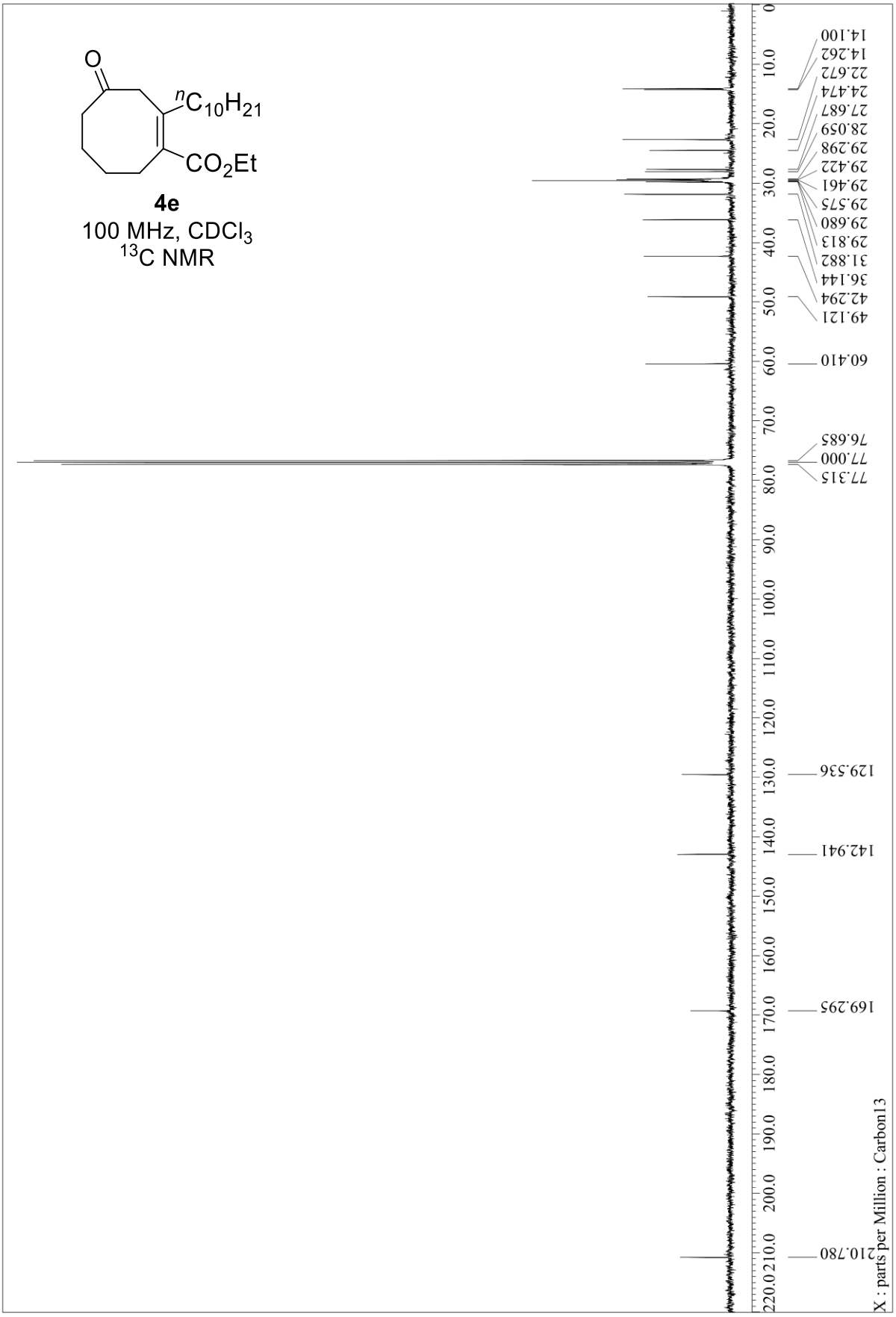

EtO2C
EtO2C
O
CH3
OH
3b-minor
400 MHz, CDCl3
1H NMR
X : parts per Million : Proton

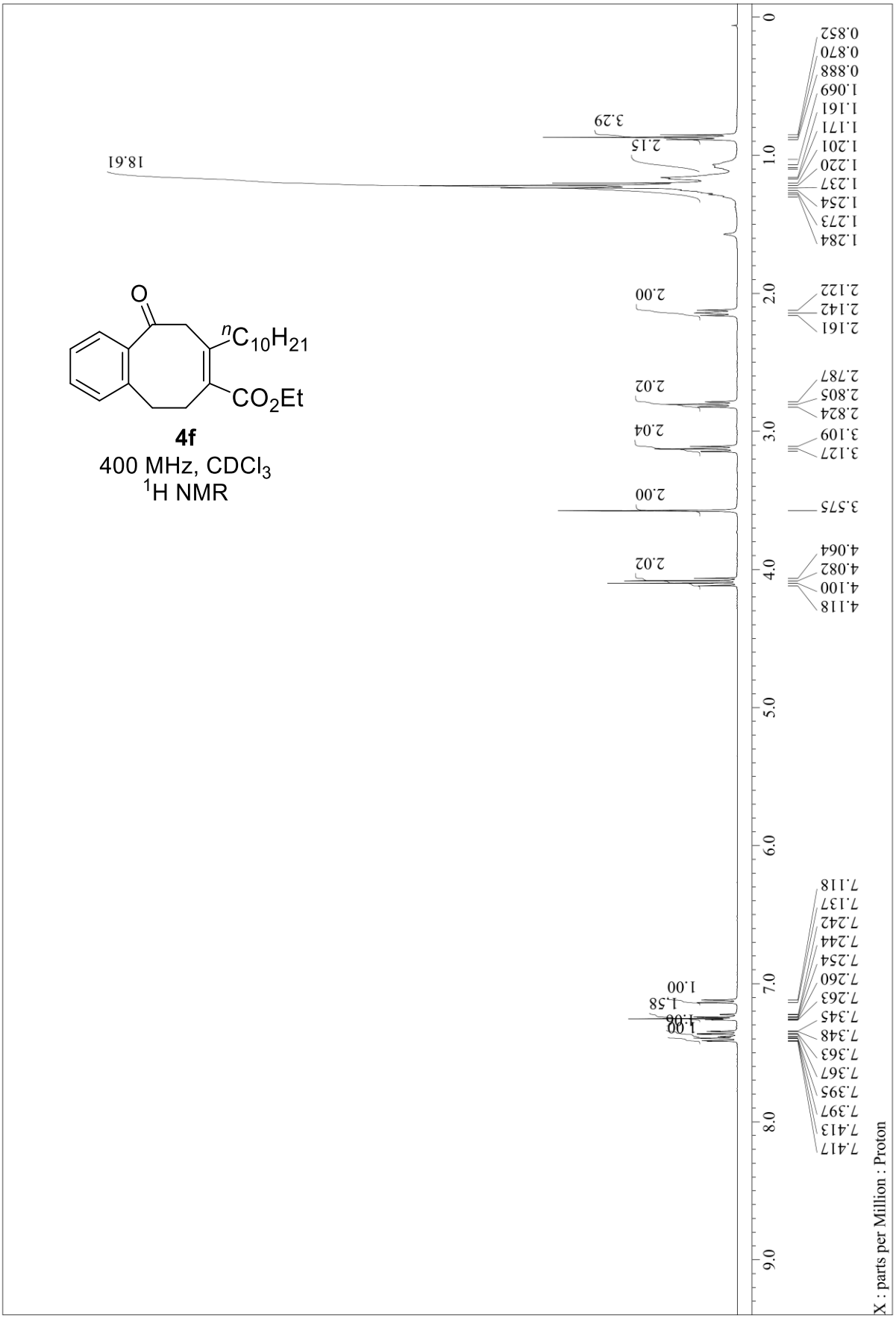

EtO2C
EtO2C
O
CH3
OH
3b-minor
100 MHz, CDCl3
13C NMR
13.938
14.034
19.592
21.804
30.633
30.910
31.081
38.909
54.107
61.506
61.735
65.120
76.685
77.000
77.315
83.407
115.911
134.675
139.881
146.440
170.649
218.045
X: parts per Million : Carbon13

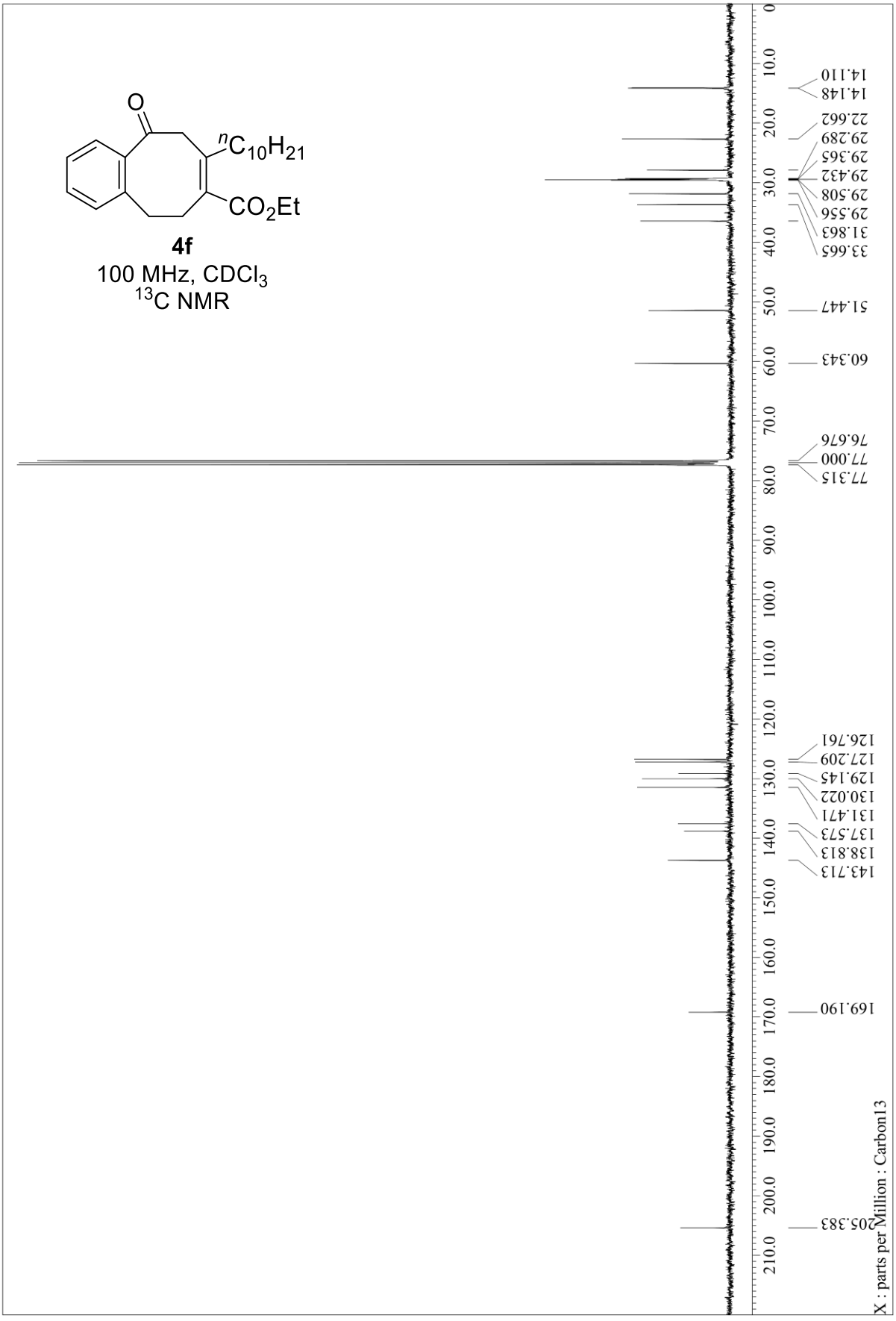

O
$^{n}C_{10}H_{21}$
CO_2Et
4d
400 MHz, $CDCl_3$
^{1}H NMR
19.19
3.14
2.05
2.00
2.00
4.08
2.00
2.01
0.859
0.877
0.893
1.254
1.277
1.285
1.303
1.954
1.970
1.986
2.346
2.365
2.553
2.570
2.588
3.246
4.179
4.196
4.214
4.231
7.260
X : parts per Million : Proton


O
nC10H21
CO2Et
4d
100 MHz, CDCl3
13C NMR
14.100
14.253
22.672
25.199
27.802
28.555
29.308
29.451
29.575
29.670
31.892
36.564
43.591
49.550
60.486
76.685
77.000
77.324
129.755
144.305
168.932
207.347
X : parts per Million : Carbon13


O
nC10H21
CO2Et
4e
400 MHz, CDCl3
1H NMR
17.60
7.28
4.10
2.39
4.17
2.00
4.18
2.00
2.35
7.260
3.260
X : parts per Million : Proton


O
nC10H21
CO2Et
4e
100 MHz, CDCl3
13C NMR
14.100
14.262
22.672
24.474
27.687
28.059
29.298
29.422
29.461
29.575
29.680
29.813
31.882
36.144
42.294
49.121
60.410
76.685
77.000
77.315
129.536
142.941
169.295
210.780
X : parts per Million : Carbon13


O
nC10H21
CO2Et
4f
400 MHz, CDCl3
1H NMR
X : parts per Million : Proton


O
nC10H21
CO2Et
4f
100 MHz, CDCl3
13C NMR
14.110
14.148
22.662
29.289
29.365
29.432
29.508
29.556
31.863
33.665
51.447
60.343
76.676
77.000
77.315
126.761
127.209
129.145
130.022
131.471
137.573
138.813
143.713
169.190
205.383
X : parts per Million : Carbon13