Electronic Supplementary Information

Regioselective Arene Homologation through Rhenium-Catalyzed Deoxygenative Aromatization of 7-Oxabicyclo[2.2.1]hepta-2,5-diene

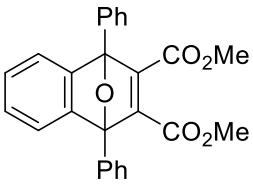
Masahito Murai,* Takuya Ogita, and Kazuhiko Takai*

Division of Applied Chemistry, Graduate School of Natural Science and Technology, Okayama University, 3-1-1 Tsushimanaka, Kita-ku, Okayama 700-8530, Japan.

E-mail: masahito.murai@okayama-u.ac.jp
ktakai@cc.okayama-u.ac.jp

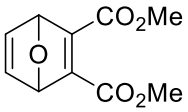
Table of Contents

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. Toluene was purchased from Wako Pure Chemical Industries, and was 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. NH_4ReO_4, Re_2O_7, and $P(OPh)_3$ were purchased from Wako, Sigma-Aldrich, and Tokyo Chemical Industry, respectively. 7-Oxabicyclo-[2.2.1]heptadiene derivatives **1a**,[1] **1b**,[2] **1c**,[3] **1d**,[4] **1e**,[5] **1f**,[6] **1g**,[7] **1h**,[8] **1i**,[8,9] and **1k**[10] were synthesized according to the reported methods. ^{1}H (400 or 300 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; s: singlet, d: doublet, t: triplet, q: quartet, sept: septet, 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.

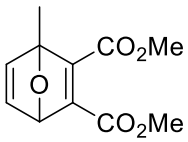


2. Preparation of Dimethyl 1,4-Diphenyl-1,4-dihydro-1,4-epoxy-naphthalene-2,3-dicarboxylate (1j). To a stirred solution of 1,3-diphenylisobenzofuran[11] (540.7 mg, 2.0 mmol) in toluene (10 mL) was added dimethyl acetylenedicarboxylate (625.2 mg, 4.4 mmol), and the resulting mixture was stirred at reflux for 3 h. The solvent was removed under the reduced pressure, and the residue was subjected to flash column chromatography on silica gel with hexane / EtOAc (*v* / *v* = 10 / 1) as the eluent to afford dimethyl 1,4-diphenyl-1,4-dihydro-1,4-epoxy-naphthalene-2,3-dicarboxylate (**1j**) as a colorless solid (593.9 mg, 1.4 mmol, 72% yield).

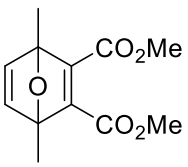
m.p. 156.8-157.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ3.69 (s, 6H), 7.14-7.18 (m, 2H), 7.38-7.52 (m, 6H), 7.54-7.57 (m, 2H), 7.79 (d, J = 7.6 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 52.3, 94.0, 122.2, 126.0, 128.0, 128.6, 129.0, 133.1, 149.1, 153.9, 164.1. IR (KBr / cm^{-1}): 3073, 3034, 3009, 2955, 1721, 1638, 1501, 1456, 1435, 1317, 1294, 1256, 1192, 1128, 1006, 982, 773, 748. HRMS (FAB^+): calcd for $C_{26}H_{21}O_5$ ($[M+H]^+$) 413.1384; found. 413.1375.



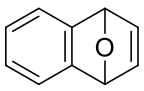
Dimethyl 7-oxabicyclo[2.2.1]hepta-2,5-diene-2,3-dicarboxylate (1a): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ3.82 (s, 6H), 5.68 (s, 2H), 7.22 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ52.4, 85.1, 143.2, 152.9, 163.2. HRMS (FAB^+): calcd for $C_{10}H_{11}O_5$ ($[M+H]^+$) 211.0601; found. 211.0610. The analytical data match those reported in the literature.[1]



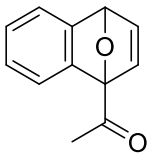
Dimethyl 1-methyl-7-oxabicyclo[2.2.1]hepta-2,5-diene-2,3-dicarboxylate (1b): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ1.79 (s, 3H), 3.78 (s, 3H), 3.85 (s, 3H), 5.60 (d, J = 2.0 Hz, 1H), 6.98 (d, J = 5.6 Hz, 1H), 7.18 (dd, J = 2.0, 5.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ15.1, 52.2, 52.3, 83.3, 93.8, 144.6, 145.9, 151.2, 156.5, 162.8, 164.9. HRMS (FAB^+): calcd for $C_{11}H_{13}O_5$ ($[M+H]^+$) 225.0757; found. 225.0761. The analytical data match those reported in the literature.[2]



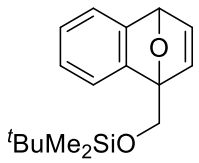
Dimethyl 1,4-dimethyl-7-oxabicyclo[2.2.1]hepta-2,5-diene-2,3-dicarboxylate (1c): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ1.79 (s, 6H), 3.79 (s, 6H), 6.95 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ15.3, 52.1, 92.0, 147.2, 154.8, 164.3. HRMS (FAB^+): calcd for $C_{12}H_{15}O_5$ ($[M+H]^+$) 239.0914; found. 239.0921. The analytical data match those reported in the literature.[3]



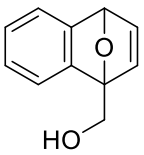
1,4-Dihydro-1,4-epoxynaphthalene (1d): Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.71 (s, 2H), 6.97 (dd, J = 2.8, 5.2 Hz, 2H), 7.03 (s, 2H), 7.25 (dd, J = 2.8, 5.2 Hz, 2H). The analytical data match those reported in the literature.[4]



1-Acetyl-1,4-dihydro-1,4-epoxynaphthalene (1e): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.40 (s, 3H), 5.80 (d, J = 2.0 Hz, 1H), 6.96-7.03 (m, 3H), 7.03-7.07 (m, 1H), 7.24-7.29 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 26.8, 82.3, 95.7, 119.5, 120.5, 125.2, 125.6, 142.2, 143.4, 147.4, 148.0, 205.2. HRMS (FAB^+): calcd for $C_{12}H_{11}O_2$ ($[M+H]^+$) 187.0754; found. 187.0749. The analytical data match those reported in the literature.[5]

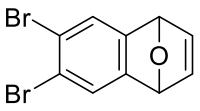


1-[(*tert*-Butyldimethylsiloxy)methyl]-1,4-dihydro-1,4-epoxynaphthalene (1f): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.17 (s, 6H), 0.97 (s, 9H), 4.33 (d, J = 10.8 Hz, 1H), 4.48 (d, J = 10.8 Hz, 1H), 5.69 (d, J =1.5 Hz, 1H), 6.96-6.99 (m, 3H), 7.04 (dd, J = 1.5, 5.6 Hz, 1H), 7.21-7.24 (m, 1H), 7.27-7.30 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ -5.4, 18.4, 25.9, 61.5, 82.1, 92.7, 119.8 (overlapped), 124.8, 124.9, 143.1, 143.8, 149.4, 150.5. HRMS (FAB^+): calcd for $C_{17}H_{25}O_2Si$ ($[M+H]^+$) 289.1618; found. 289.1610. The analytical data match those reported in the literature.[6]

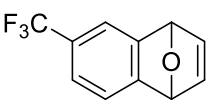


1-(Hydroxymethyl)-1,4-dihydro-1,4-epoxynaphthalene (1f'): Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.57 (br s, 1H), 4.44 (d, J = 12.8 Hz, 1H), 4.48 (d, J = 12.8 Hz, 1H), 5.73 (d, J =2.0 Hz, 1H), 6.89 (d, J =5.6 Hz, 1H), 6.98-7.01 (m, 2H), 7.09 (dd, J = 1.6, 5.6 Hz, 1H), 7.17-7.20 (m, 1H), 7.24-7.26 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 60.3, 82.1, 93.6, 119.5, 120.2, 125.0, 125.1, 142.3, 144.9, 147.7, 150.4. HRMS (FAB^+): calcd for $C_{11}H_{11}O_2$ ($[M+H]^+$) 175.0754; found.

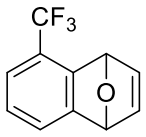
175.0747. The analytical data match those reported in the literature.[6a]



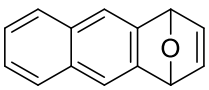
6,7-Dibromo-1,4-dihydro-1,4-epoxynaphthalene (1g): Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.66 (s, 2H), 6.99 (s, 2H), 7.47 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 81.8, 120.6, 125.5, 142.7, 150.2. HRMS (FAB^+): calcd for $C_{10}H_6Br_2O$ ($[M]^+$) 299.8785; found. 299.8747. The analytical data match those reported in the literature.[7]



6-(Trifluoromethyl)-1,4-dihydro-1,4-epoxynaphthalene (1h): Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.85 (d, J =2.0 Hz, 2H), 7.13 (dd, J = 2.0, 5.6 Hz, 1H), 7.15 (dd, J = 2.0, 5.6 Hz, 1H), 7.38 (d, J = 7.6 Hz, 1H), 7.42 (d, J = 7.6 Hz, 1H), 7.55 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 82.1 (two peaks overlapped), 116.8 (q, J = 3.3 Hz), 120.0, 123.0 (q, J = 3.8 Hz), 124.2 (q, J = 270.8 Hz), 127.4 (q, J = 32.5 Hz), 142.7, 143.1, 150.3, 153.2. HRMS (FAB^+): calcd for $C_{11}H_8F_3O$ ($[M+H]^+$) 213.0522; found. 213.0522. The analytical data match those reported in the literature.[8]

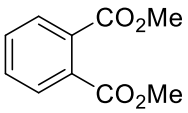


5-Trifluoromethyl-1,4-dihydro-1,4-epoxynaphthalene (1i): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.78 (s, 1H), 6.01 (s, 1H), 7.04 (dd, J = 2.0, 5.6 Hz, 1H), 7.07 (dd, J = 2.0, 5.6 Hz, 1H), 7.09 (t, J = 6.8 Hz, 1H), 7.18 (d, J = 6.8 Hz, 1H), 7.39 (d, J = 6.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 81.5, 81.9, 121.1 (q, J = 4.8 Hz), 123.0, 123.6 (q, J = 33.4 Hz), 123.9 (q, J = 275.5 Hz), 125.6, 142.4, 143.6, 148.0, 150.5. HRMS (FAB^+): calcd for $C_{11}H_8F_3O$ ($[M+H]^+$) 213.0522; found. 213.0524. The analytical data match those reported in the literature.[9]

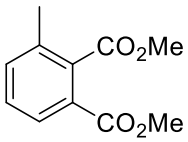


1,4-Dihydro-1,4-epoxyanthracene (1k): Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.81 (s, 2H), 6.97 (s, 2H), 7.43 (dd, J = 3.2, 6.0 Hz, 2H), 7.59 (s, 2H), 7.71 (dd, J = 3.2, 6.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 81.8, 118.6, 126.1, 128.1, 131.9, 141.7, 144.1. HRMS (FAB^+): calcd for $C_{14}H_{10}O$ ($[M]^+$) 194.0732; found. 194.0743. The analytical data match those reported in the literature.[10]

3. General Procedure for Rhenium-Catalyzed Deoxygenation of 7-Oxabicyclo[2.2.1]-heptadiene Derivatives. A flame-dried test tube was charged with Re_2O_7 (1.8 mg, 3.75 μmol) or NH_4ReO_4 (2.0 mg, 7.5 μmol), $P(OPh)_3$ (111.7 mg, 0.36 mmol) and toluene (0.90 mL). After stirring for 10 min, 7-oxabicyclo[2.2.1]heptadiene derivatives **1** (0.30 mmol) was added, and further stirred at the temperature specified in the text for 18 h. The solvent was removed under the reduced pressure, and the residue was subjected to flash column chromatography on silica gel with hexane / EtOAc as eluents to afford the corresponding aromatic compounds.

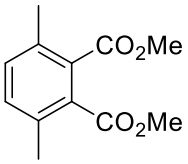


Dimethyl phthlate (2a): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.89 (s, 6H), 7.50-7.54 (m, 2H), 7.69-7.72 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 52.6, 128.8, 131.1, 131.9, 168.0. HRMS (FAB^+): calcd for $C_{10}H_{11}O_4$ ($[M+H]^+$) 195.0657; found. 195.0655. The analytical data match those reported in the literature (P. Dawar, M. Bhagavan Raju, R. A. Ramakrishana, *Tetrahedron Lett.* 2011, **52**, 4262).

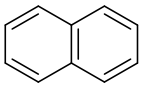


Dimethyl 3-methylphthalate (2b): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.33 (s, 3H), 3.87 (s, 3H), 3.93 (s, 3H), 7.31-7.41 (m, 2H), 7.79-7.83 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.9, 52.4, 52.5, 127.2, 127.4, 129.0, 134.4, 135.3, 135.7, 166.3, 169.9. IR (neat / cm^{-1}): 3001, 2953, 1732, 1595, 1456, 1435, 1282, 1198, 1153, 1117, 1022, 960, 767. HRMS (FAB^+): calcd for $C_{11}H_{13}O_4$

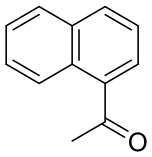
([M+H]$^+$) 209.0808; found. 209.0830.



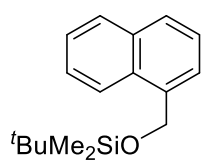
Dimethyl 3,6-dimethylphthlate (2c): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.38 (s, 6H), 3.87 (s, 6H), 7.19 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.8, 52.2, 131.7, 132.5, 134.3, 168.9. IR (neat / cm^{-1}): 3022, 2953, 1732, 1589, 1489, 1435, 1402, 1294, 1263, 1203, 1161, 1111, 1036, 962, 756. HRMS (FAB$^+$): calcd for $C_{12}H_{15}O_4$ ([M+H]$^+$) 223.0965; found. 223.0946.



Naphthalene (2d) [CAS 91-20-3]: Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (dd, J = 3.2, 6.4 Hz, 4H), 7.87 (dd, J = 3.2, 6.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 126.0, 127.9, 133.4. The analytical data match those measured for commercial sample.

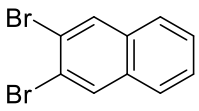


1-Acetylnaphthalene (2e): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.75 (s, 3H), 7.50 (t, J = 7.6 Hz, 1H), 7.54 (t, J = 7.6 Hz, 1H), 7.61 (dt, J = 1.2, 7.6 Hz, 1H), 7.88 (d, J = 8.4 Hz, 1H), 7.94 (dd, J = 2.0, 7.6 Hz, 1H), 7.99 (d, J = 8.4 Hz, 1H), 8.76 (d, J = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 29.9, 124.3, 126.0, 126.4, 128.0, 128.4, 128.6, 130.1, 133.0, 133.9, 135.4, 201.8. HRMS (FAB$^+$): calcd for $C_{12}H_{11}O$ ([M+H]$^+$) 171.0810; found. 171.0805. The analytical data match those reported in the literature (J. Ruan, J. A. Iggo, N. G. Berry. J. Xiao, *J. Am. Chem. Soc.* 2010, **132**, 16689).

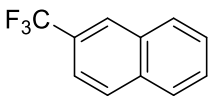


1-[(*tert*-Butyldimethylsiloxy)methyl]naphthalene (2f): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.16 (s, 6H), 0.99 (s, 9H), 5.24 (s, 2H), 7.46-7.57 (m, 3H), 7.62 (d, J = 6.8 Hz, 1H), 7.79 (d, J = 8.4 Hz, 1H), 7.89 (dd, J = 2.0, 7.2 Hz, 1H), 8.02 (dd, J = 2.0, 7.2 Hz, 1H). ^{13}C NMR (100 MHz,

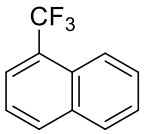
$CDCl_3$): δ-5.2, 18.4, 26.0, 63.4, 123.3, 123.7, 125.4, 125.5, 125.8, 127.5, 128.6, 130.8, 133.5, 136.7. HRMS (FAB^+): calcd for $C_{17}H_{25}OSi$ ($[M+H]^+$) 273.1675; found. 273.1654. The analytical data match those reported in the literature (P. Patschinski, C. Zhang, H. Zipse, *J. Org. Chem.* 2014, **79**, 8348).



2,3-Dibromonaphthalene (2g): Colorless solid; ^1H NMR (400 MHz, $CDCl_3$): δ7.50-7.53 (m, 2H), 7.72-7.75 (m, 2H), 8.15 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ122.1, 127.0, 127.3, 132.4, 133.2. HRMS (FAB^+): calcd for $C_{10}H_6Br_2$ ($[M]^+$) 283.8836; found. 283.8851. The analytical data match those reported in the literature (G. London, M. von Wantoch Rekowski, O. Dumele, W. B. Schweizer, J.-P. Gisselbrecht, C. Boudon, F. Diederich, *Chem. Sci.* 2014, **5**, 965).

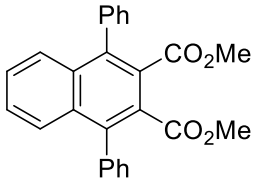


2-(Trifluoromethyl)naphthalene (2h): Colorless oil; ^1H NMR (400 MHz, $CDCl_3$): δ7.59 (dd, J = 1.6, 5.6 Hz, 1H), 7.62 (dd, J = 1.6, 5.6 Hz, 1H), 7.65 (dd, J = 1.6, 8.4 Hz, 1H), 7.89-7.93 (m, 2H), 7.96 (d, J = 8.4 Hz, 1H), 8.16 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ124.4 (q, J = 3.1 Hz), 125.7 (q, J = 4.6 Hz), 127.1, 127.5 (q, J = 30.0 Hz), 127.8, 128.0, 128.8, 128.9 (q, J = 276.9 Hz), 129.0, 132.2, 134.5. HRMS (FAB^+): calcd for $C_{11}H_8F_3$ ($[M+H]^+$) 197.0578; found. 197.0577. The analytical data match those reported in the literature (T. Liu, Q. Shen, *Org. Lett*. 2011, **13**, 2342).

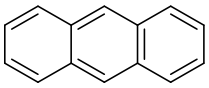


1-(Trifluoromethyl)naphthalene (2i): Colorless solid; ^1H NMR (400 MHz, $CDCl_3$): δ7.51 (t, J = 7.6 Hz, 1H), 7.58 (dt, J = 1.2, 8.0 Hz, 1H), 7.64 (dd, J = 1.2, 8.0 Hz, 1H), 7.88 (d, J = 7.2 Hz, 1H), 7.93 (d, J = 7.2 Hz, 1H), 8.03 (d, J = 8.0 Hz, 1H), 8.20 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ124.1, 124.2 (q, J = 2.3 Hz), 124.7 (q, J = 6.1 Hz), 124.8 (q, J = 271.7 Hz), 126.0 (q, J = 24.8 Hz), 126.6, 127.6, 128.7, 128.9, 132.7, 133.9. HRMS (FAB^+): calcd for $C_{11}H_8F_3$ ($[M+H]^+$) 197.0578;

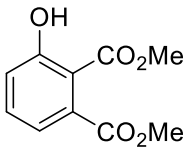
found. 197.0560. The analytical data match those reported in the literature (J. Xu, D.-F. Luo, B. Xiao, Z.-J. Liu, T.-J. Gong, Y. Fu, L. Liu, *Chem. Commun.* 2011, **47**, 4300).



Dimethyl 1,4-diphenylnaphthalene-2,3-dicarboxylate (2j): Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ3.51 (s, 6H), 7.36-7.41 (m, 4H), 7.42-7.52 (m, 8H), 7.62-7.67 (dd, J = 2.8, 6.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ52.2, 127.4, 127.6, 127.9, 128.1, 128.7, 129.9, 132.7, 137.5, 139.1, 168.9. HRMS (FAB^+): calcd for $C_{26}H_{20}O_4$ ($[M]^+$) 396.1362; found. 396.1367. The analytical data match those reported in the literature.[11b]

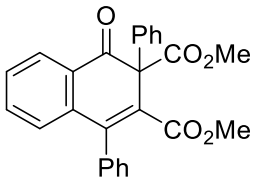


Anthracene (2k) [CAS 120-12-7]: Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ7.47 (dd, J = 3.2, 6.4 Hz, 4H), 8.01 (dd, J = 3.2, 6.4 Hz, 4H), 8.43 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ125.5, 126.3, 128.3, 131.8. The analytical data match those measured for commercial sample.



Dimethyl 3-hydroxyphthalate (3a): Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ3.88 (s, 3H), 3.91 (s, 3H), 6.95 (dd, J = 1.2, 7.2 Hz, 1H), 7.07 (dd, J = 1.2, 7.6 Hz, 1H), 7.44 (dt, J = 1.2, 7.6 Hz, 1H), 10.5 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ52.6, 52.8, 110.1, 119.0, 119.8, 134.5, 135.4, 161.0, 169.3, 169.4. IR (neat / cm^{-1}): 3401, 2955, 1732, 1681, 1605, 1581, 1454, 1330, 1283, 1199, 1145, 1115, 1009, 820, 760. HRMS (FAB^+): calcd for $C_{10}H_{11}O_5$ ($[M+H]^+$) 211.0601; found. 211.0603.

Dimethyl 1-oxo-2,4-diphenyl-1,2-dihydronaphthalene-2,3-dicarboxylate (3j): Pale yellow crystal; mp 199.8－200.2 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ3.36 (s, 3H), 3.79 (s, 3H), 6.93 (dd, J = 0.8, 7.6 Hz, 1H), 7.27-7.35 (m, 4H), 7.38 (dt, J = 2.0, 8.0 Hz, 1H),

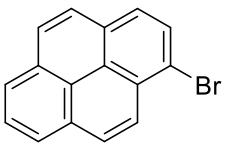


7.42-7.53 (m, 5H), 7.67 (dd, J = 2.0, 7.2 Hz, 2H), 7.92 (dd, J = 1.2, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 51.8, 53.1, 68.0, 127.7, 128.2 (two peaks overlapped), 128.39, 128.40, 128.7, 128.92, 128.93, 129.0, 130.1, 130.5, 134.0, 134.4, 137.5, 137.6, 144.2, 167.0, 169.5, 194.0. IR (KBr / cm^{-1}): 2954, 1763, 1703, 1686, 1439, 1271, 1225, 1140, 719. HRMS (FAB^+): calcd for $C_{26}H_{20}O_5$ ($[M]^+$) 412.1311; found. 412.1315.

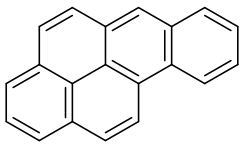
4. Regioselective Benzannulative π-Extension of PAHs. A flame-dried test tube was charged with NBS (106.7 mg, 0.60 mmol), $AuCl_3$ (1.5 mg, 5.0 μmol), $ClCH_2CH_2Cl$ (1.0 mL), and aromatic compounds (benzene, naphthalene, and phenanthrene, 0.50 mmol), and the resulting mixture was stirred at 80 ^oC for 24 h. The solvent was removed under reduced pressure, and the residue was subjected to flash column chromatography on silica gel with hexane / EtOAc as the eluent to afford the corresponding aryl bromides. Because the above procedure was not applicable to bromination of pyrene and chrysene (complex mixture was obtained), 1-bromopyrene and 6,12-dibromochrysene were synthesized by the treatment of pyrene and chrysene with bromine in $CHCl_3$ at 80 ^oC for 24 h.

To a stirred solution of $NaNH_2$ (17.6 mg, 0.45 mmol) and ^{t}BuOK (1.7 mg, 15 μmol (30 μmol for the synthesis of **4b**)) in THF (1.0 mL) was added aryl bromides (0.30 mmol) and furan (306.3 mg, 4.5 mmol (9.0 mmol for the synthesis of **4b**)), and the resulting mixture was stirred at 50 ^oC for 15 h. The reaction mixture was diluted with Et_2O (5 mL), and passed through short silica gel pad. The filtrate was washed with brine, and extracted with Et_2O for three times. The combined organic layer was dried over $MgSO_4$, filtered, and the solvent was removed under reduced pressure. The residue was treated with Re_2O_7 (1.8 mg, 3.75 μmol), $P(OPh)_3$ (111.7 mg, 0.36 mmol), and toluene (0.90 mL), and the resulting mixture was stirred at 80 ^oC for 18 h. The solvent was removed under the reduced pressure, and the residue was subjected to flash column chromatography on silica gel with hexane as

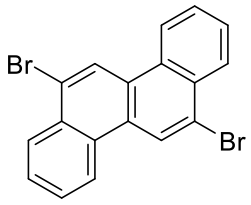
the eluent to afford the corresponding aromatic compounds **4**.



1-Bromopyrene: Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J = 8.4 Hz, 1H), 7.90 (d, J = 8.4 Hz, 1H), 7.97 (d, J = 7.6 Hz, 1H), 7.99 (d, J = 9.2 Hz, 1H), 8.06 (d, J = 9.2 Hz, 1H), 8.12 -8.18 (m, 3H), 8.34 (d, J = 9.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 119.7, 123.8, 125.3, 125.4, 125.5, 125.6, 125.7, 126.3, 126.9, 127.5, 128.7, 129.4, 129.8, 130.4, 130.7, 130.9. The analytical data match those reported in the literature (R. S. Kathayat, N. S. Finney, *J. Am. Chem. Soc.* 2013, **135**, 12612; W. Wu, X. Wu, J. Zhao, M. Wu, *J. Mater. Chem. C.* 2015, **3**, 2291).

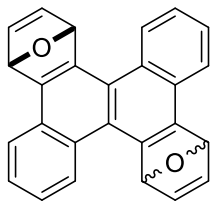


Benzo[*a*]pyrene (4a): A pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (dt, J = 1.2, 7.2 Hz, 1H), 7.82 (dt, J = 1.2, 7.2 Hz, 1H), 7.90 (d, J = 8.8 Hz, 1H), 7.97 (d, J = 9.2 Hz, 1H), 7.98 (d, J = 7.6 Hz, 1H), 8.08 (d, J = 7.6 Hz, 1H), 8.22 (d, J = 7.6 Hz, 1H), 8.27 (d, J =6.4 1H), 8.29 (d, J =9.2 1H), 8.47 (s, 1H), 9.00 (d, J = 7.2 Hz, 1H), 9.01 (d, J = 7.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 122.1, 122.9, 123.7, 124.7, 124.8, 125.3, 125.5, 125.9, 126.0, 126.1, 127.3, 127.4, 127.7, 128.0, 128.1, 128.8, 129.8, 131.2, 131.3, 131.5. IR (KBr / cm^{-1}): 3071, 3036, 1616, 1558, 1508, 1474, 1314, 1182, 883, 839, 758, 743, 691. HRMS (FAB^{+}): calcd for $C_{20}H_{12}$ ($[M]^{+}$) 252.0939; found. 252.0944.

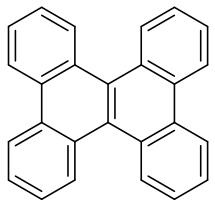


6,12-Dibromochrysene: A colorless solid; m.p. 287.9-288.4 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (t, J = 7.2 Hz, 2H), 7.79 (t, J = 7.2 Hz, 2H), 8.45 (dd, J = 2.4, 7.2 Hz, 2H), 8.71 (dd, J = 2.4, 7.2 Hz, 2H), 9.01 (s, 2H). ^{1}H NMR data match those reported in the literature (Bock, H.; Huet, S.; Dechambenoit, P.; Hillard, E. A.; Durola, F. *Eur. J. Org. Chem.* **2015**, 1033). ^{13}C NMR (100 MHz, $CDCl_3$): δ 123.3, 123.4, 125.4, 127.90, 127.94, 128.3, 128.4, 130.7, 131.0. IR

(KBr / cm^{-1}): 3040, 1576, 1508, 1396, 916, 864, 752. HRMS (FAB^+): calcd for $C_{18}H_{10}Br_2$ ($[M]^+$) 383.9149; found. 383.9148.

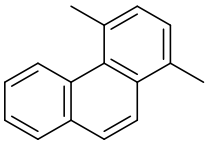


1,4,9,12-Tetrahydro-1,4:9,12-diepoxydibenzo[*g*,*p*]chrysene: A yellow solid (Obtained as a mixture of two stereoisomers. The ratio was determined to be 60 / 40 by ^1H NMR analysis of the crude product); ^1H NMR for major isomer (400 MHz, $CDCl_3$): δ6.40 (s, 2H), 6.89 (s, 2H), 7.46 (dd, J = 1.6, 5.6 Hz, 2H), 7.61 (dd, J = 1.6, 5.6 Hz, 2H), 7.68-7.71 (m, 4H), 8.02-8.05 (m, 2H), 8.39-8.43 (m, 2H). ^1H NMR for minor isomer (400 MHz, $CDCl_3$): δ6.43 (s, 2H), 7.00 (s, 2H), 7.40 (dd, J = 1.6, 5.6 Hz, 2H), 7.52 (dd, J = 1.6, 5.6 Hz, 2H), 7.68-7.71 (m, 4H), 8.03-8.08 (m, 2H), 8.64-8.67 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ80.9, 81.2, 85.4, 85.9, 122.8, 123.1, 124.0, 124.5, 125.9, 126.0, 126.3 (two peaks overlapped), 126.6, 126.7, 127.3, 128.0, 128.3, 143.7, 144.0, 144.3, 144.4, 146.9, 147.1, 147.2, 147.3. IR (KBr / cm^{-1}): 3003, 1541, 1273, 1038, 889, 841, 768. HRMS (FAB^+): calcd for $C_{26}H_{16}O_2$ ($[M]^+$) 360.1150; found. 360.1158.

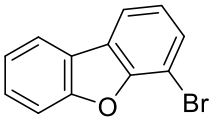


Dibenzo[*g*,*p*]chrysene (4b): A pale yellow solid; ^1H NMR (400 MHz, $CDCl_3$): δ7.64 (t, J = 8.0 Hz, 4H), 7.69 (t, J = 8.0 Hz, 4H), 8.68-8.73 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$): δ123.6, 126.6 (two peaks overlapped), 128.9, 129.2, 130.9, 131.6. The analytical data match those reported in the literature (Y. Kurata, S. Otsuka, N. Fukui, K. Nogi, H. Yorimitsu, A. Osuka, *Org. Lett.* 2017, **19**, 1274).

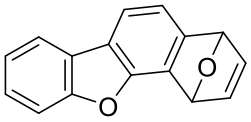
1,4-Dimethylphenanthrene (4c): Colorless crystal; ^1H NMR (400 MHz, $CDCl_3$): δ2.76 (s, 3 H), 3.14 (s, 3 H), 7.34 (d, J = 7.2 Hz, 1H), 7.38 (d, J = 7.2 Hz, 1H), 7.58-7.64 (m, 2H), 7.78 (d, J = 9.2 Hz, 1H), 7.93 (d, J = 7.6 Hz, 1H), 7.98 (d, J = 9.2 Hz, 1H), 8.91 (d, J = 9.2



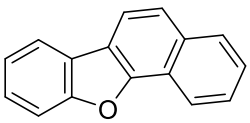
Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.3, 27.4, 123.5, 125.3, 125.7, 126.8, 127.3, 127.7, 128.5, 130.3, 130.7, 131.9, 132.2, 132.8, 133.1, 133.3. The analytical data match those reported in the literature (S. Nandi, K. Panda, J. R. Suresh, H. Ilaa, H. Junjappa, *Tetrahedron* 2004, **60**, 3663).



4-Bromodibenzo[*b*,*d*]furan: This compound was synthesized according to the reported procedure,[12] and obtained as a mixture of 12% of dibenzofuran after the purification by column chromatography on silica gel with hexane as the eluent. The analytically pure sample was obtained by further purification with GPC. A colorless crystal; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (t, J = 7.6 Hz, 1H), 7.38 (dt, J = 0.8, 7.6 Hz, 1H), 7.50 (dt, J = 1.6, 7.6 Hz, 1H), 7.62 (d, J = 1.6, 7.6 Hz, 1H), 7.66 (d, J = 7.6 Hz, 1H), 7.90 (dd, J = 0.8, 7.6 Hz, 1H), 7.94 (dd, J = 0.8, 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 104.5, 112.1, 119.6, 121.0, 123.3, 123.9, 124.2, 125.7, 127.8, 130.0, 153.3, 156.0. The analytical data match those reported in the literature.[12]

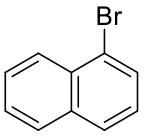


Benzo[*b*]naphtho[2,1-*d*]furan: Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.89 (s, 1H), 6.24 (s, 1H), 7.14-7.15 (m, 2H), 7.31 (d, J = 7.6 Hz, 1H), 7.32 (t, J = 7.6 Hz, 1H), 7.44 (t, J = 7.6 Hz, 1H), 7.54 (d, J = 7.6 Hz, 1H), 7.59 (d, J = 7.6 Hz, 1H), 7.90 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 80.1, 82.9, 111.5, 115.4, 117.3, 120.7, 122.8, 123.5, 124.2, 127.3, 131.1, 142.7, 143.9, 149.0, 150.0, 156.6. HRMS (FAB$^+$): calcd for $C_{16}H_{10}O_2$ ([M]$^+$) 234.0681; found. 234.0671.

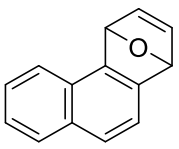


Benzo[*b*]naphtho[2,1-*d*]furan (4d): Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (dt, J = 1.2, 7.6 Hz, 1H), 7.49 (dt, J = 1.2, 7.6 Hz, 1H), 7.58 (dt, J = 1.2, 7.2 Hz, 1H), 7.66 (dt, J = 1.2, 7.2 Hz, 1H), 7.73 (d, J = 8.4

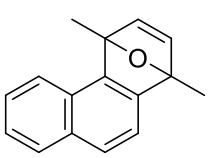
Hz, 1H), 7.79 (d, J = 8.4 Hz, 1H), 7.99-8.03 (m, 3H), 8.47 (d, J = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 111.8, 118.5, 119.2, 120.3, 120.9, 121.4, 122.9, 123.3, 125.1, 126.1, 126.2, 126.5, 128.4, 133.1, 152.0, 156.0. The analytical data match those reported in the literature (S. Maetani, T. Fukuyama, I. Ryu, *Org. Lett.* 2013, **15**, 2754; T. Okazaki, M. Nakagawa, T. Kitagawa, K. K. Laali, *Bull. Chem. Soc. Jpn.* 2014, **87**, 1235).



1-Bromonaphthalene [CAS 90-11-9]: Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (t, J = 7.2 Hz, 1H), 7.54 (t, J = 7.2 Hz, 1H), 7.61 (t, J = 7.2 Hz, 1H), 7.85 (d, J = 7.6 Hz, 1H), 7.82 (d, J = 8.8 Hz, 1H), 7.85 (d, J = 8.8 Hz, 1H), 8.26 (d, J = 8.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 122.8, 126.2, 126.7, 127.1, 127.3, 127.9, 128.3, 129.9, 132.0, 134.6. The analytical data match those measured for commercial sample.

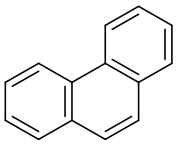


1,4-Dihydro-1,4-epoxyphenanthrene: Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.93 (s, 1H), 6.27 (s, 1H), 7.21 (s, 2H), 7.38 (dt, J = 0.8, 7.2 Hz, 1H), 7.46 (dt, J = 0.8, 7.2 Hz, 1H), 7.54 (d, J = 8.0 Hz, 1H), 7.58 (d, J = 8.0 Hz, 1H), 7.82 (t, J = 9.2 Hz, 1H), 7.83 (t, J = 9.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 81.2, 83.4, 119.3, 122.6, 125.1, 125.4, 126.2, 127.6, 128.7, 131.7, 143.4, 144.9, 147.8, 148.3. HRMS (FAB$^+$): calcd for $C_{14}H_{11}O$ ([M+H]$^+$) 195.0809; found. 195.0801. The analytical data match those reported in the literature (S. K. Sundalam, A. Nilova, T. L. Seidl, D. R. Stuart, *Angew. Chem. Int. Ed.* 2016, **55**, 8431).

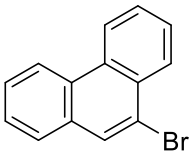


1,4-Dimethyl-1,4-dihydro-1,4-epoxyphenanthrene: Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.02 (s, 3H), 2.31 (s, 3H), 6.92 (d, J = 5.6 Hz, 1H), 7.02 (d, J = 5.6 Hz, 1H), 7.35 (t, J = 7.6 Hz, 1H), 7.42 (d, J = 8.4 Hz, 1H), 7.45 (t, J = 8.0 Hz, 1H), 7.60 (d, J = 8.0 Hz, 1H), 7.85 (d, J = 8.4 Hz, 1H),

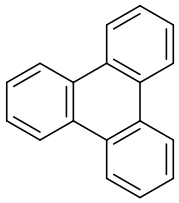
8.04 (d, J = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 15.4, 18.8, 88.9, 90.7, 117.5, 122.4, 124.5, 125.9, 126.1, 127.8, 129.2, 132.1, 148.2, 148.3, 149.4, 152.4. The analytical data match those reported in the literature (K.-y. Jung, M. Koreeda, *J. Org. Chem.* 1998, **54**, 5667).



Phenanthrene (4e) [CAS 85-01-8]: Colorless crystal; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (t, 7.6 Hz, 2H), 7.67 (dt, J = 2.0, 6.8 Hz, 2H), 7.76 (s, 2H), 7.90 (dt, J = 2.0, 7.6 Hz, 2H), 8.71 (d, J = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 122.6, 126.5 (overlapped), 126.9, 128.6, 130.3, 132.0. The analytical data match those reported in the literature (R. Wakabayashi, T. Kurahashi, S. Matsubara, *Synlett* 2013, **24**, 2297).



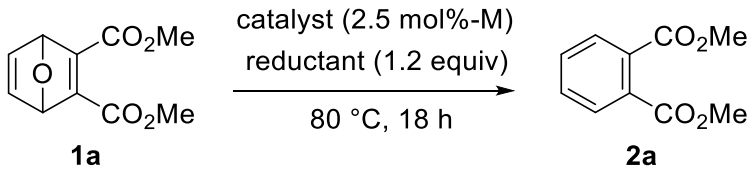
9-Bromophenanthrene [CAS 573-17-1]: Colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (dt, J = 1.2, 6.8 Hz, 1H), 7.65-7.73 (m, 3H), 7.81 (dd, J = 0.8, 7.6 Hz, 1H), 8.12 (s, 1H), 8.36-8.39 (m, 1H), 8.66 (d, J = 8.0 Hz, 1H), 8.68-8.71 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 121.7, 122.7, 122.8, 127.1, 127.2, 127.4, 127.5, 127.8, 128.1, 129.8, 130.4, 130.5, 131.3, 132.2. The analytical data match those measured for commercial sample.



Triphenylene (4f) [CAS 217-59-4]: Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (dd, J = 3.2, 6.4 Hz, 6H), 8.65 (dd, J = 3.2, 6.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 123.3, 127.2, 129.8. The analytical data match those reported in the literature (J.-C. Hsieh, C.-H. Cheng, *Chem. Commun.* 2008, 2992).

5. Optimization of Reaction Conditions

Table S1. Optimization of Reaction Conditions



1a **2a**

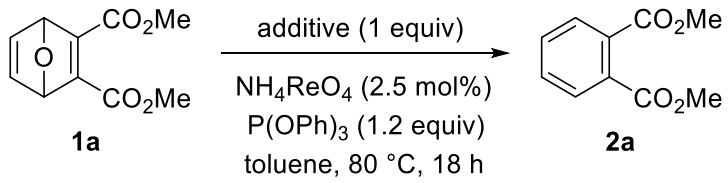
Entry	catalyst	solvent	reductant	Yield of **2a**[a] / %	Recov. of **1a**[a] / %
1	$MeReO_3$	toluene	$P(OPh)_3$	84	0
2	$ReOCl_3(PPh_3)_2$	toluene	$P(OPh)_3$	88	0
3	$ReIO_2(PPh_3)_2$	toluene	$P(OPh)_3$	82	0
4	Re_2O_7	toluene	$P(OPh)_3$	94 (93)	0
5	$ReCl_5$	toluene	$P(OPh)_3$	89	0
6	NH_4ReO_4	toluene	$P(OPh)_3$	92 (90)	0
7[b]	NH_4ReO_4	toluene	$P(OPh)_3$	85	0
8	$ReCl_3(PPhMe_2)_3$	toluene	$P(OPh)_3$	67	0
9	MoO_2Cl_2	toluene	$P(OPh)_3$	54	0
10	NH_4ReO_4	toluene	$P(OEt)_3$	0	0
11	NH_4ReO_4	toluene	PPh_3	0	38
12	NH_4ReO_4	toluene	P(*o*-tol)$_3$	10	72
13	NH_4ReO_4	toluene	P(2-furyl)$_3$	62	62
14	NH_4ReO_4	toluene	P^{n}Bu$_3$	0	19
15	NH_4ReO_4	toluene	dppe	37	0
16[c]	NH_4ReO_4	toluene	3-pentanol	74	0
17	NH_4ReO_4	toluene	^{i}PrOH	54	0
18[c]	NH_4ReO_4	toluene	Zn	23	21
19[c]	NH_4ReO_4	toluene	Na_2SO_3	2	32
20[c]	NH_4ReO_4	toluene	——	0	80
21	NH_4ReO_4	C_6H_5Cl	$P(OPh)_3$	41	26
22	NH_4ReO_4	^{n}decane	$P(OPh)_3$	48	42
23	NH_4ReO_4	cyclohexane	$P(OPh)_3$	50	10
24	NH_4ReO_4	$ClCH_2CH_2Cl$	$P(OPh)_3$	28	18
25	NH_4ReO_4	1,4-dioxane	$P(OPh)_3$	31	40
26	NH_4ReO_4	MeCN	$P(OPh)_3$	0	81

[a]Determined by ^{1}H NMR. Values in parentheses are the isolated yields. [b]NH_4ReO_4 (1 mol%) for 36 h. [c]At 120 °C.

2a was not obtained with the following catalysts: $TiCl_4$, Cr_2O_7, WCl_6, MnO_2, $MnBr_2$, $Mn(acac)_2$, $Re_2(CO)_{10}$, $[ReBr(CO)_3(thf)]_2$, $RuCl_3$, $Rh_2(OAc)_4$, $NiCl_2$, Cu_2O.

6. Tolerance of Functional Groups in Deoxygenative Aromatization of 1a

Table S2. Study of the Functional Group Tolerance



Entry	additive	Recov. of additive[a] / %	Yield[a] / %
1	$Ph(CH_2)_2COMe$	94	86
2	$Ph(CH_2)_2CO_2Et$	>95	84
3	$1,3\text{-}(MeO)_2C_6H_4$	>95	86
4	$4\text{-}^{n}BuC_6H_4Br$	>95	81
5	$Ph(CH_2)_2Br$	86	86
6	$Ph(CH_2)_2CN$	84	87
7	$Ph(CH_2)_2CHO$	62	87
8	$MeO_2C{-}{\equiv}{-}CO_2Me$	91	88

[a]Determined by ^1H NMR.

7. Plausible Reaction Mechanism

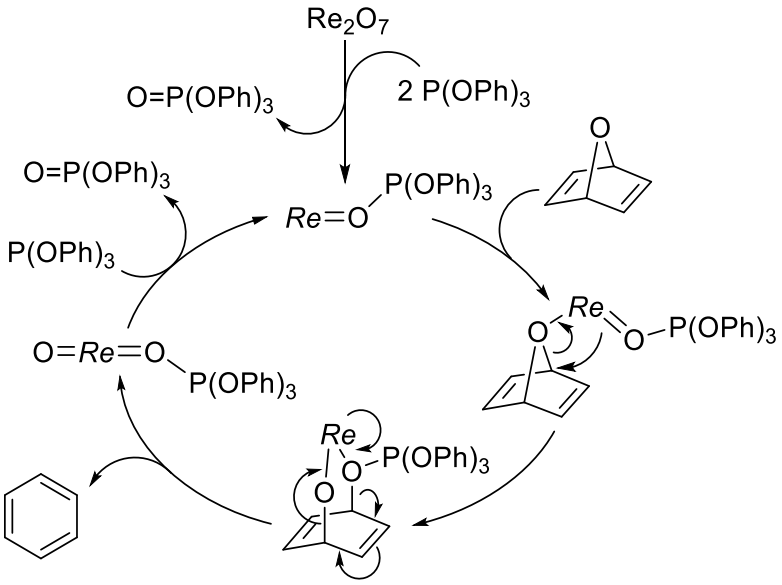
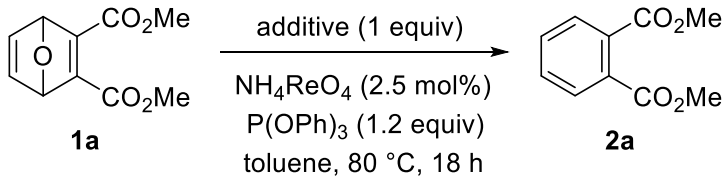


Figure S1. Plausible Reaction Mechanism

8. Comparison with the Reported Deoxygenation Protocols

Table S3. Deoxygenation of **1a** by Various Methods



Entry	conditions	Yield[a]	reference
1	$TiCl_4$ (6.8 equiv), $LiAlH_4$ (2.7 equiv) Et_3N (1.1 equiv), THF, 0 to 65 °C, 24 h	45% (lit. 64%)	*J. Org. Chem.* 1982, **47**, 140.
2	$Mo(CO)_6$ (1.1 equiv) cyclohexane, reflux, 48 h	21% (lit. 46%)	*Heterocycles* 1998, **27**, 217.
3	PhMgBr (10 equiv) THF, reflux, 2 h	0%	*Tetrahedron Lett.* 1997, **38**, 4761.
4	Me_3SiCl (3 equiv), NaI (3 equiv) CH_3CN, rt, 1 h	0%	*J. Org. Chem.* 1989, **54**, 5667.
5	Zn (15 equiv), CH_3CO_2H toluene, 100 °C, 18 h, 8 h	16%	*Tetrahedron Lett.* 1980, **21**, 3627.
6	NH_4ReO_4 (2.5 mol%) $P(OPh)_3$ (1.2 equiv), toluene, 80 °C, 18 h	92% (90%)	**This work**

Determined by ^1H NMR. Value in parentheses is the isolated yield. "lit." means yields reported in the reference.

9. References

1. N. Sultan, R. Guillot, L. Blanco, S. Deloisy, *Synthesis* 2013, **45**, 2018.

2. (a) R. R. Burton, W. Tam, *Tetrahedron Lett.* 2006, **47**, 7185; (b) M. G. Constantino, V. Aragão, G. V. J. da Silva, *Tetrahedron Lett.* 2008, **49**, 1393.

3. (a) Y. D. Xing, N. Z. Huang, *J. Org. Chem.* 1982, **47**, 140; (b) D. P. OMalley, K. Li, M. Maue, A. L. Zografos, P. S. Baran, *J. Am. Chem. Soc.* 2007, **129**, 4762.

4. (a) T. Nishimura, T. Kawamoto, K. Sasaki, E. Tsurumaki, T. Hayahsi, *J. Am. Chem. Soc.* 2007, **129**, 1492; (b) I. Rose, M. Carta, R. Malpass-Evans, M.-C. Ferrari, P. Bernardo, G. Clarizia, J. C. Jansen, N. B. McKeown, *ACS Macro Lett.* 2015, **4**, 912.

5. (a) K. Villeneuve, W. Tam, *Eur. J. Org. Chem.* 2006, 5449; (b) R. R. Burton, W. Tam, *J. Org. Chem.* 2007, **72**, 7333.

6. (a) R. Webster, C. Böing, M. Lautens, *J. Am. Chem. Soc.* 2009, **131**, 444; (b) Y. Sawama, T. Kawajiri, S. Asai, N. Yasukawa, Y. Shishido, Y. Monguchi, H. Sajiki, *J. Org. Chem.* 2015, **80**, 5556.

7. (a) R. Luo, J. Liao, L. Xie, W. Tang, A. S. C. Chan, *Chem. Commun*. 2013, **49**, 9959; (b) H. Haneda, S. Eda, M. Aratani, T. Hamura, *Org. Lett.* 2014, **16**, 286.

8. F. Bailly, F. Cottet, M. Schlosser, *Synthesis* 2005, 791.

9. (a) M. Lautens, G. A. Schmid, A. Chau, *J. Org. Chem.* 2002, **67**, 8043; (b) I. Sapountzis, W. Lin, M. Fischer, P. Knochel, *Angew. Chem. Int. Ed.* 2004, **43**, 4364.

10. (a) C. S. LeHoullier, G. W. Gribble, *J. Org. Chem.* 1983, **48**, 2364; (b) Y.-L. Chen, J.-Q. Sun, X. Wei, W.-Y. Wong, A. W. M. Lee, *J. Org. Chem.* 2004, **69**, 7190.

11. (a) Y. Kuninobu, Y. Nishina, C. Nakagawa, K. Takai, *J. Am. Chem. Soc.* 2006, **128**, 12376; (b) Y. Kuninobu, Y. Nishina, K. Takai, *Tetrahedron* 2007, **63**, 8463.

12. (a) Y. Unger, D. Meyer, O. Molt, C. Schildknecht, I. Münster, G. Wagenblast, T. Strassner, *Angew. Chem. Int. Ed.* 2010, **49**, 10214; (b) T. Okazaki, M. Nakagawa, T. Kitagawa, K. K. Laali, *Bull. Chem. Soc. Jpn.* 2014, **87**, 1235.

10. X-ray Crystallographic Studies of Isomerized Product 3j (CCDC 1873272): Pale yellow crystal of **3j** suitable for X-ray analysis was obtained by recrystallization from CH_2Cl_2 at 25 ^oC. All measurements were made on a Rigaku R-AXIS imaging plate area detector with multi-layer monochromated Mo-Kα radiation. Details of crystal and data collection parameters are summarized in Table S4. The positions of non-hydrogen atoms were determined by direct methods (SHELXS97) and subsequent Fourier syntheses. An ORTEP drawing is shown in Figure S2.

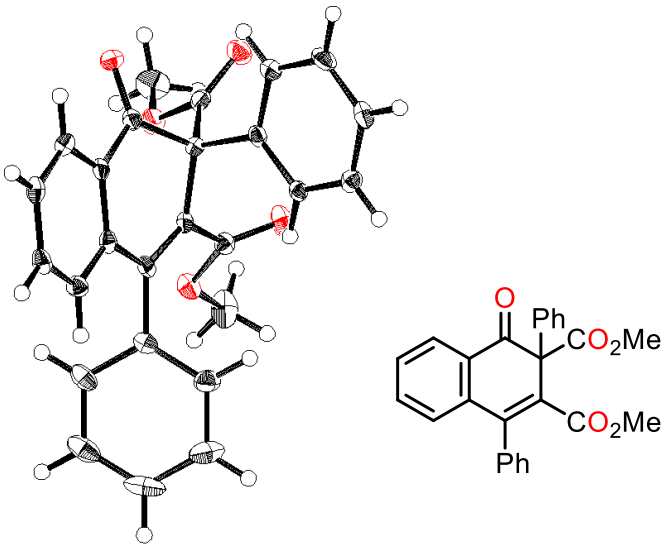
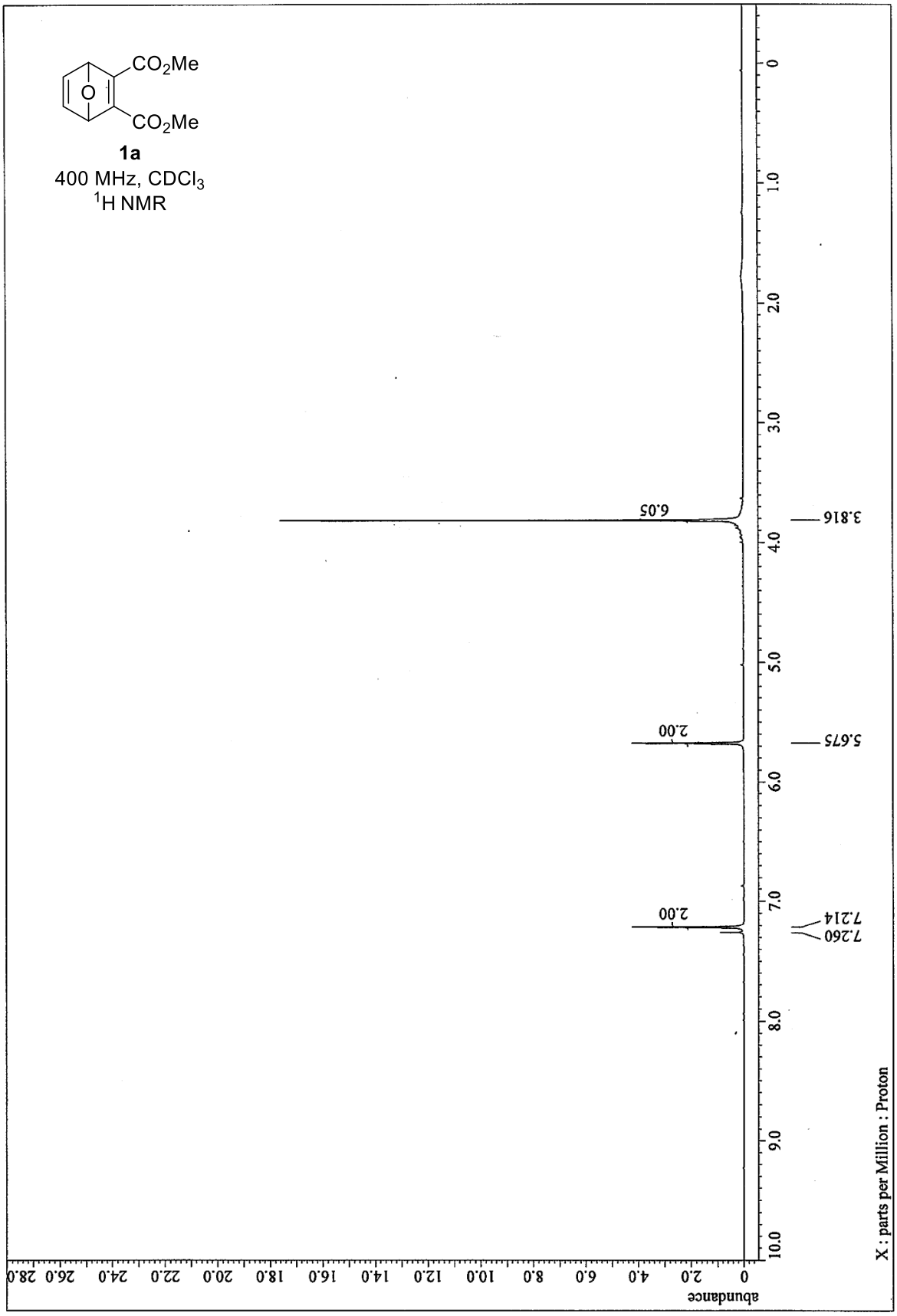


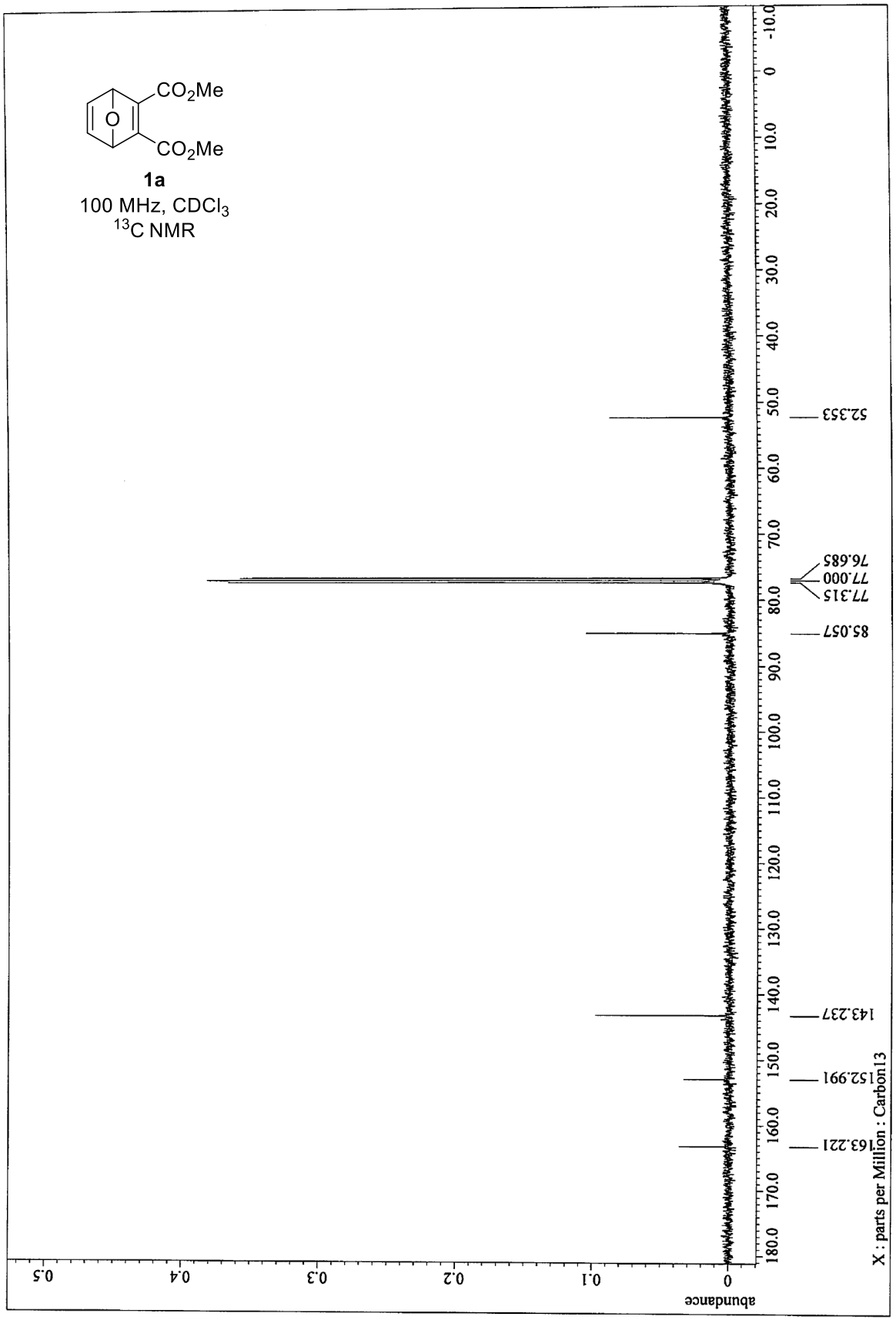
Figure S2. ORTEP drawing of borylcyclopropane **3j**. Thermal ellipsoids are drawn at the 50% probability level.

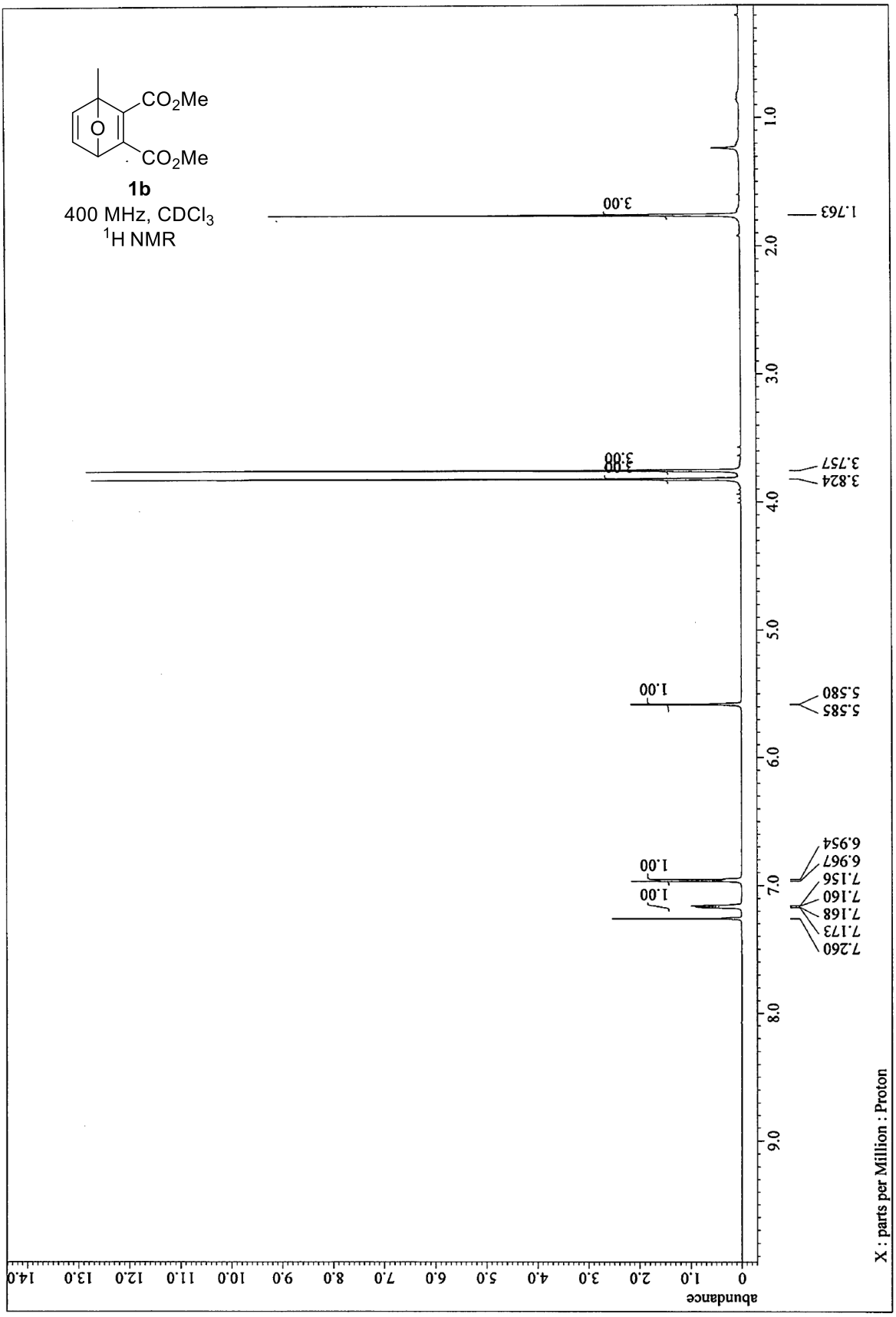
Table S4. Summary of Crystallographic Data of Borylcyclopropane **3j**

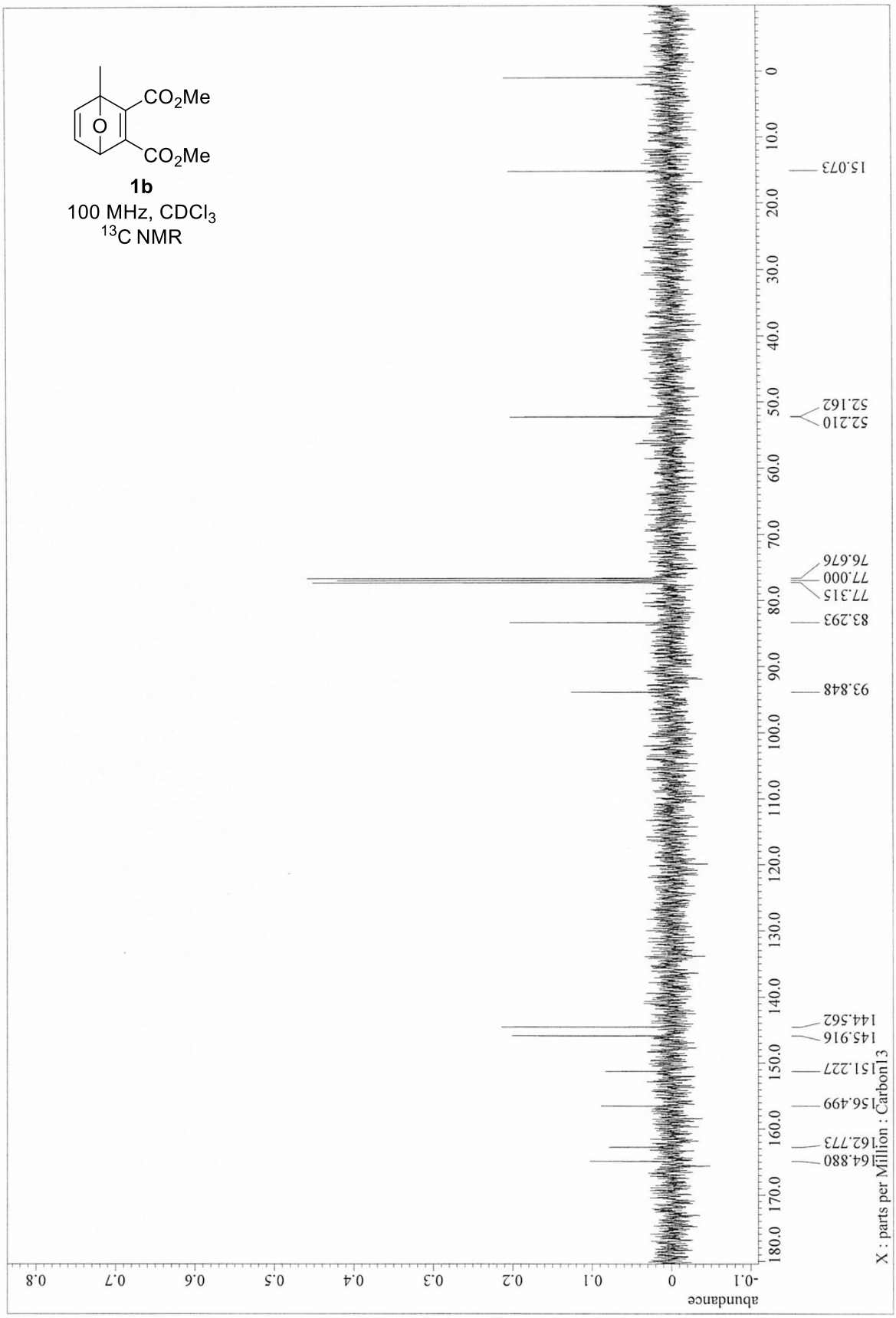
Empirical formula: $C_{26}H_{20}O_5$
Formula weight: 412.44
Crystal system: monoclinic
Space group: $P2_1/c$ (#14)
Crystal color: yellow
Lattice parameters:
a (Å) = 15.7487(10), *b* (Å) = 7.427(5), *c* (Å) = 17.812(11)
β (°) = 96.731(8), V (Å^3) = 2069.0(19), Z = 4
D_{calc} (g cm^{-3}): 1.324
μ (Mo K α) (cm^{-1}): 0.915
Goodness of fit (GOF) = 1.242
F(000): 864.00
Diffractometer: Saturn724
Radiation: MoK α (λ = 0.71075 Å), Multi-layer Mirror Monochromated
Temp (°C): -163.0
Scan type: ω–2 θ
Max. 2 θ (°): 55.0
No. of reflections measured total: 32142
No. of observns (*I* > 3.00 σ (*I*)): 4746
Structure solution: Direct Methods (SHELXS97)
Refinement: Full-Matrix Least-Squares on F^2
No. of variables: 280
Reflection/parameter ratio: 16.95
Residuals: R = 0.1148, wR2 = 0.2221
Max Shift/Error in Final Cycle: 0.000
Maximum peak in Final Diff Map (e (Å^{-3}): 0.26
Minimum peak in Final Diff Map (e (Å^{-3}): -0.27

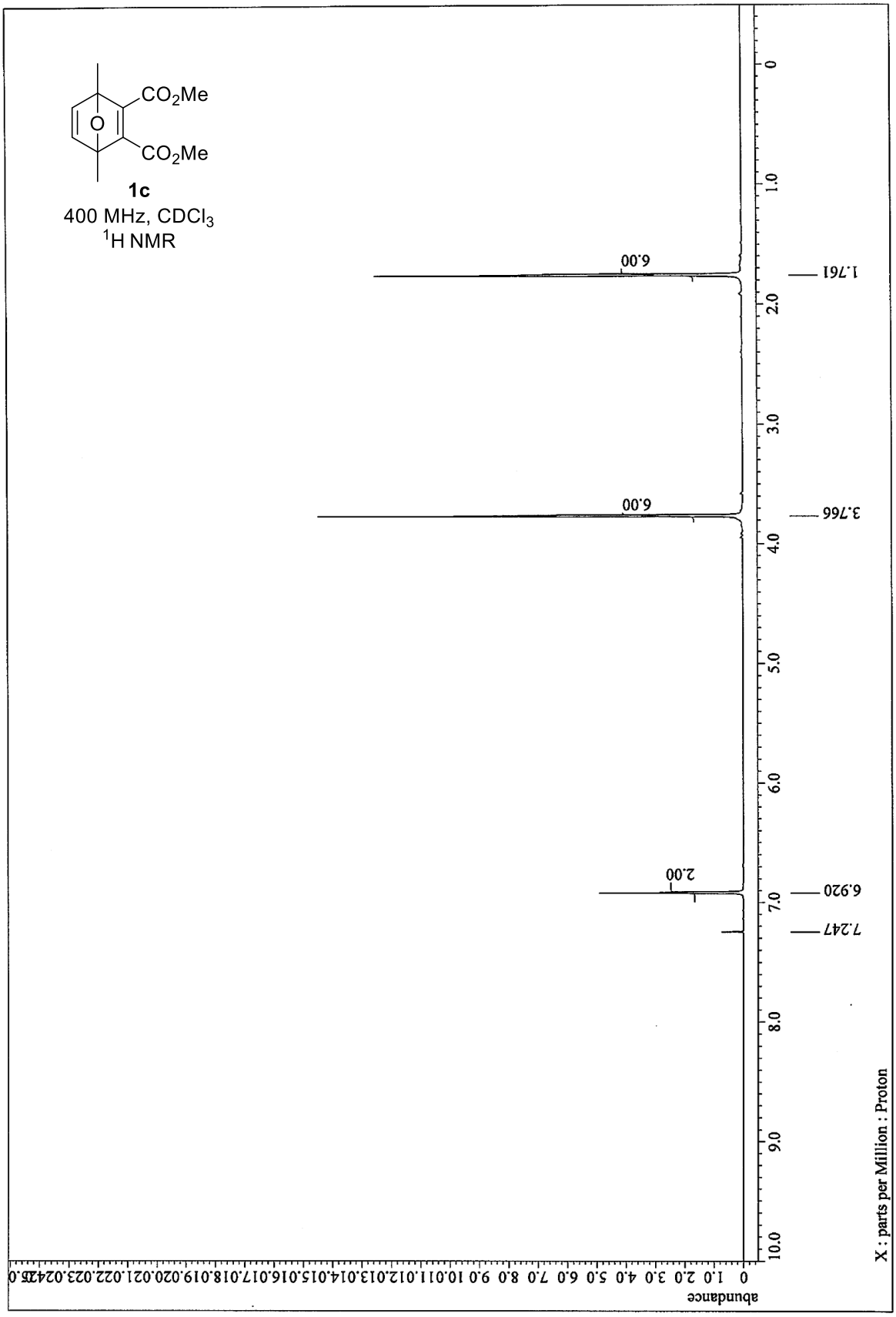
11. ^{1}H NMR and ^{13}C NMR Spectra of Products

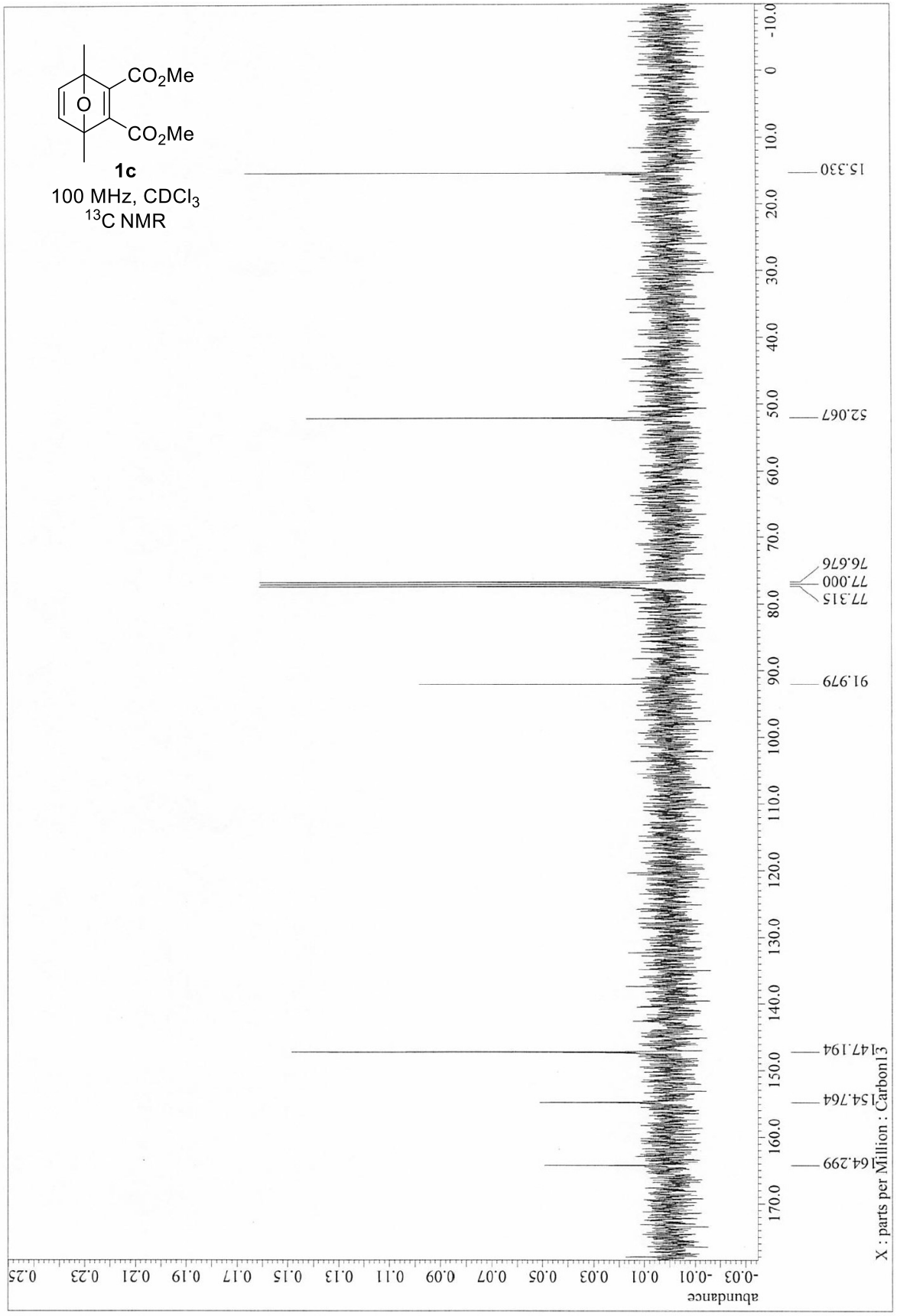


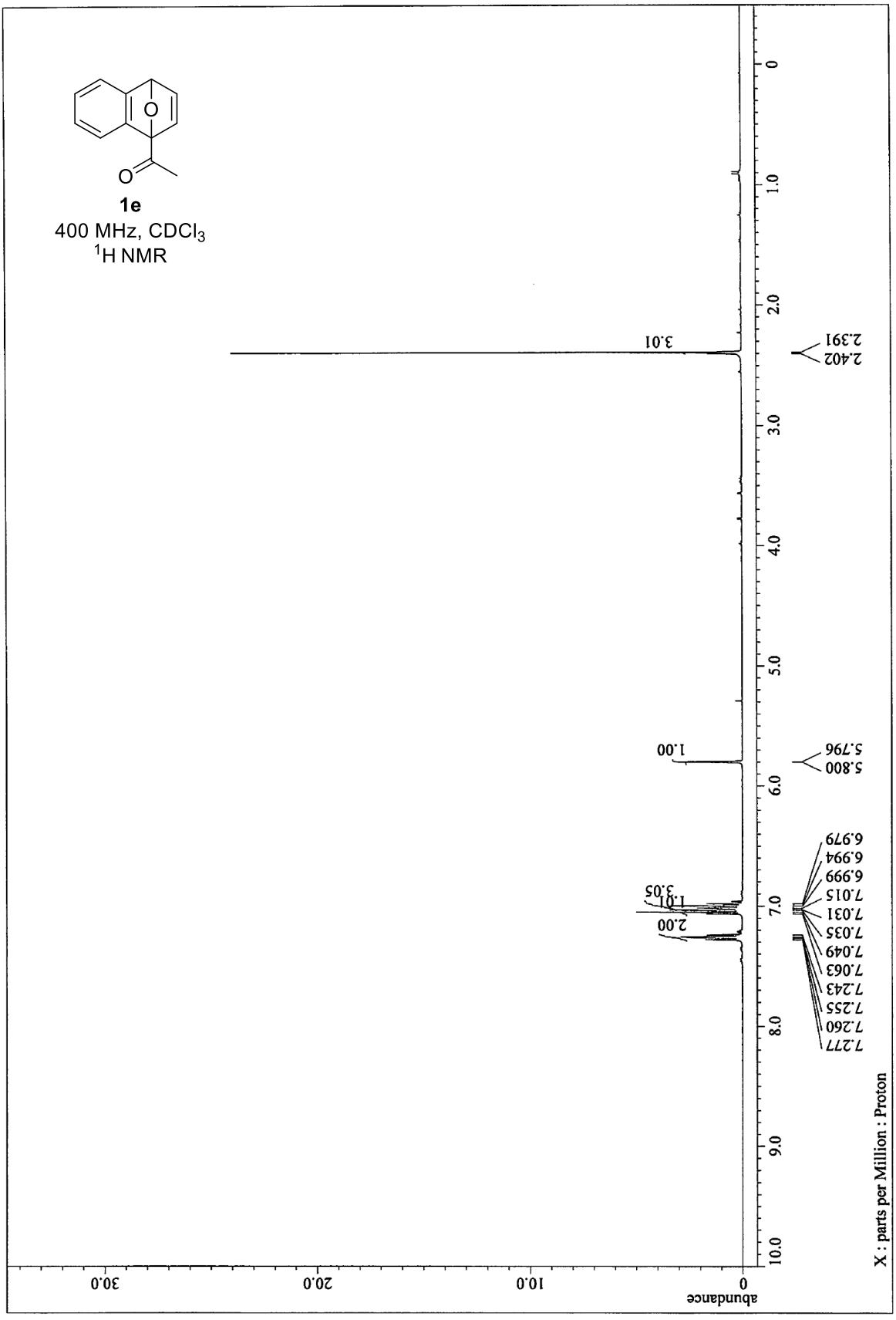

CO2Me
O
CO2Me
1a
100 MHz, CDCl3
13C NMR
52.353
76.685
77.000
77.315
85.057
143.237
152.991
163.221
X : parts per Million : Carbon13
abundance

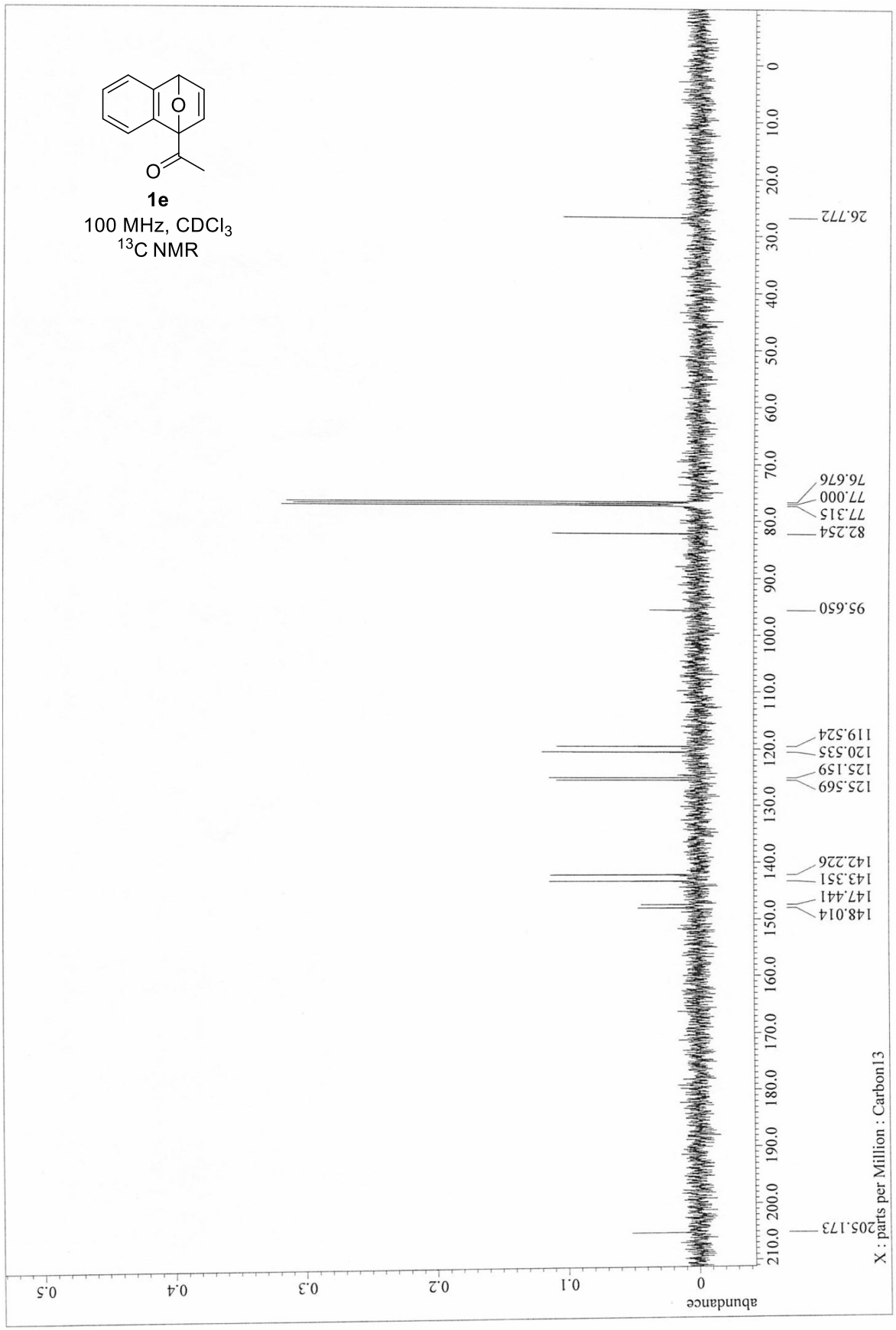

CO2Me
O
CO2Me
1b
400 MHz, CDCl3
1H NMR
3.00
1.763
3.88
3.757
3.824
1.00
5.585
5.580
1.00
1.00
7.260
7.173
7.168
7.160
7.156
6.967
6.954
abundance
X : parts per Million : Proton

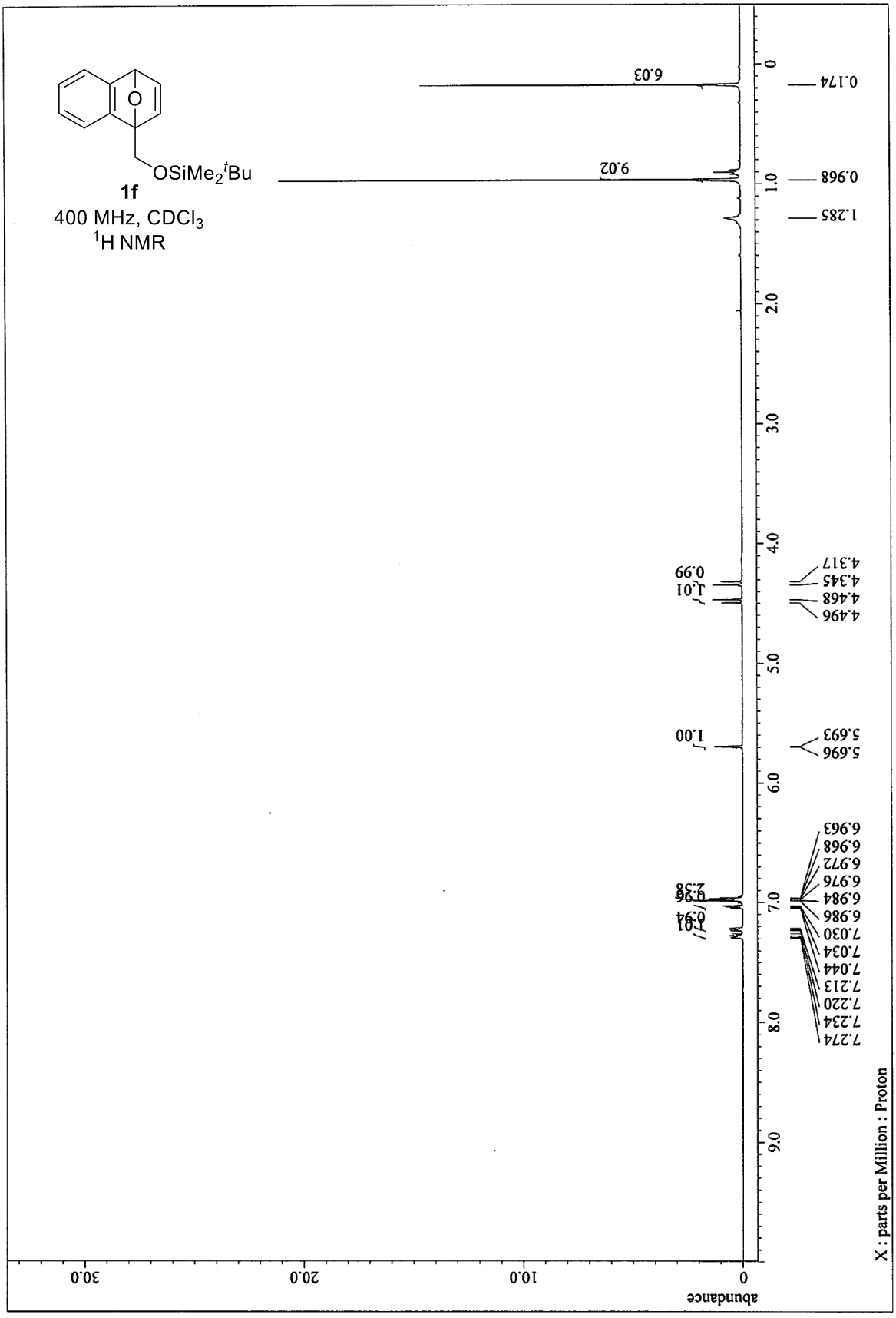

CO2Me
O
CO2Me
1b
100 MHz, CDCl3
13C NMR
15.073
52.210
52.162
76.676
77.000
77.315
83.293
93.848
144.562
145.916
151.227
156.499
162.773
164.880
X : parts per Million : Carbon13
abundance

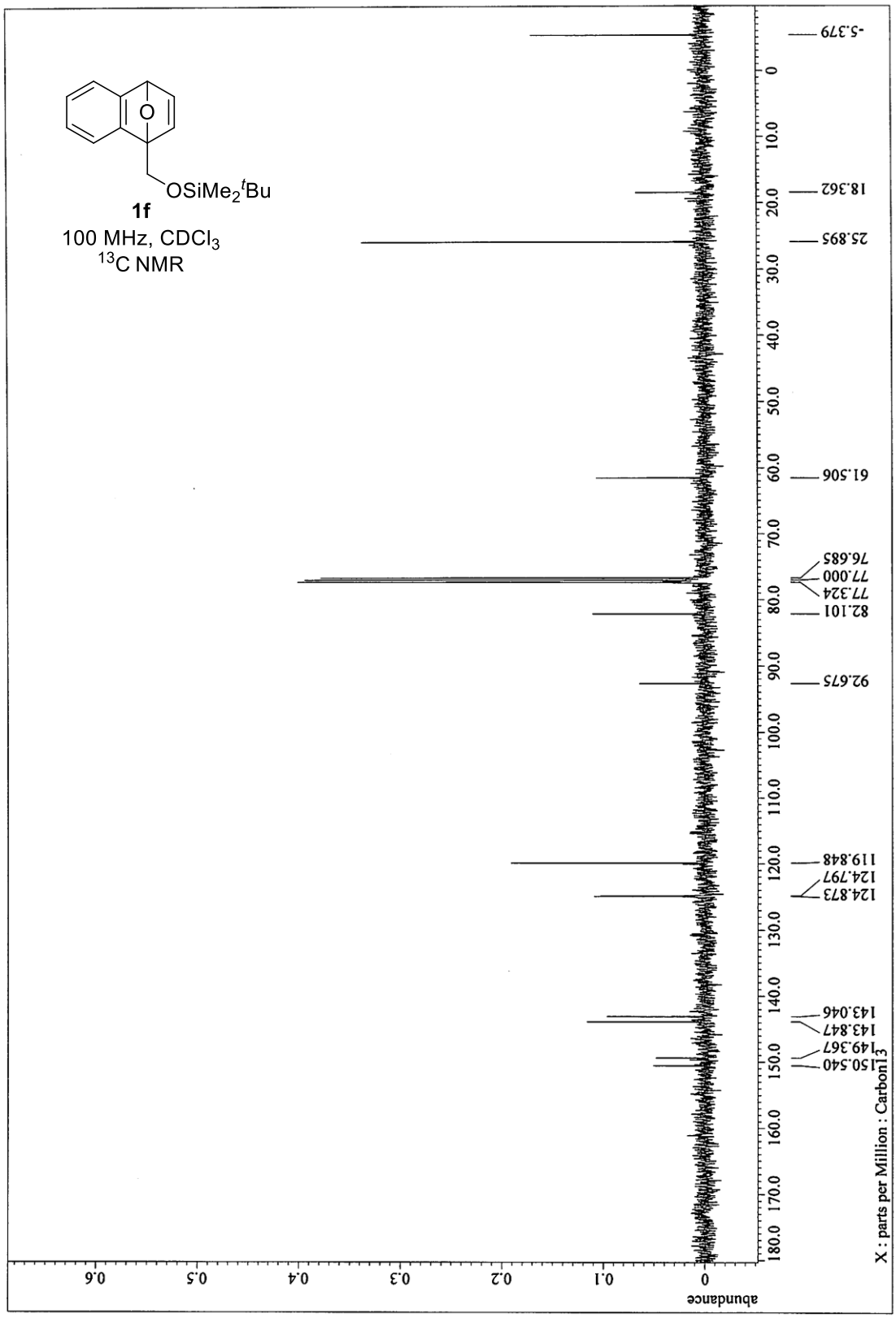

CO2Me
O
CO2Me
1c
400 MHz, CDCl3
1H NMR
6.00
6.00
2.00
1.761
3.766
6.920
7.247
abundance
X : parts per Million : Proton

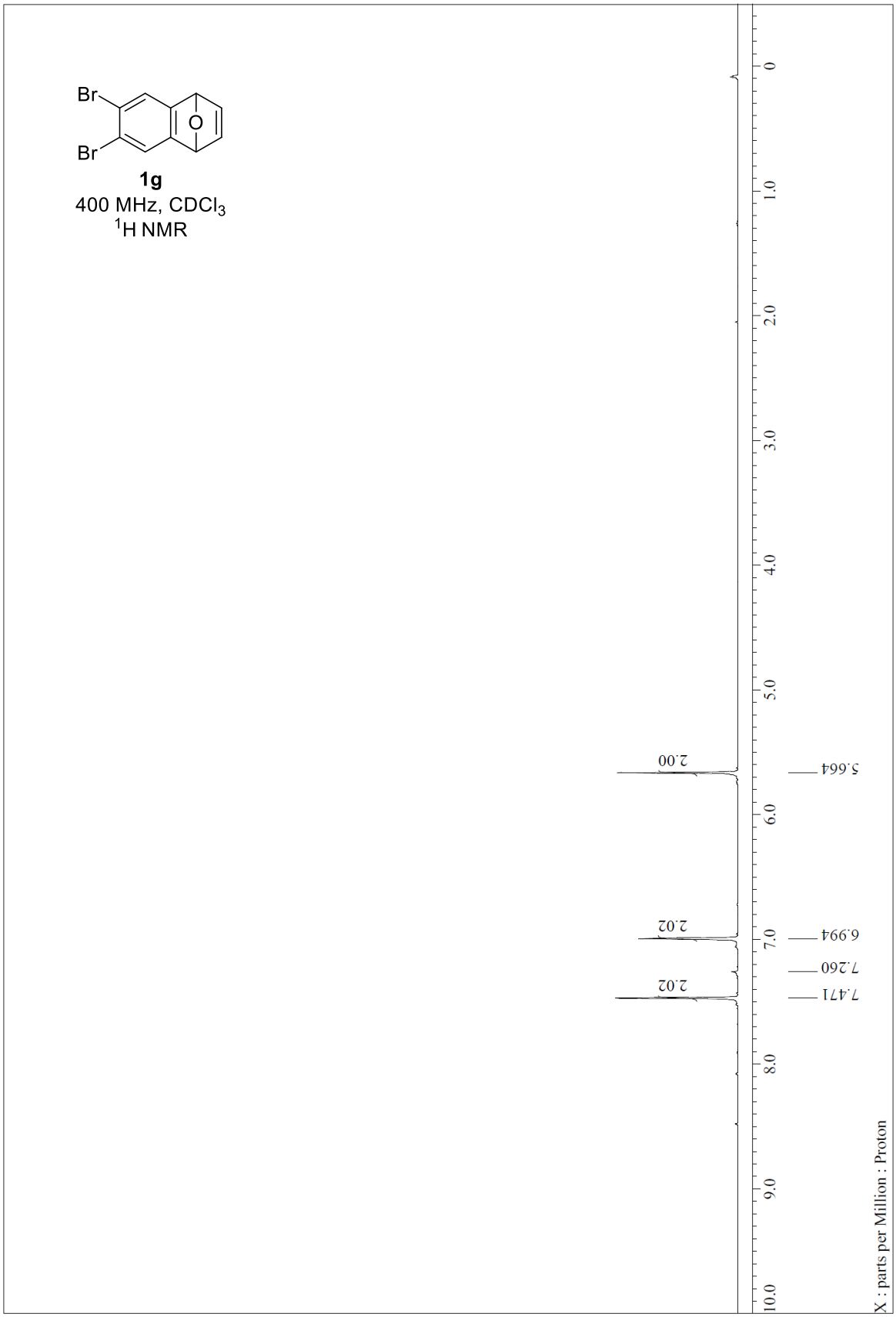

CO2Me
O
CO2Me
1c
100 MHz, CDCl3
13C NMR
15.330
52.067
76.676
77.000
77.315
91.979
147.194
154.764
164.299
X : parts per Million : Carbon13
abundance

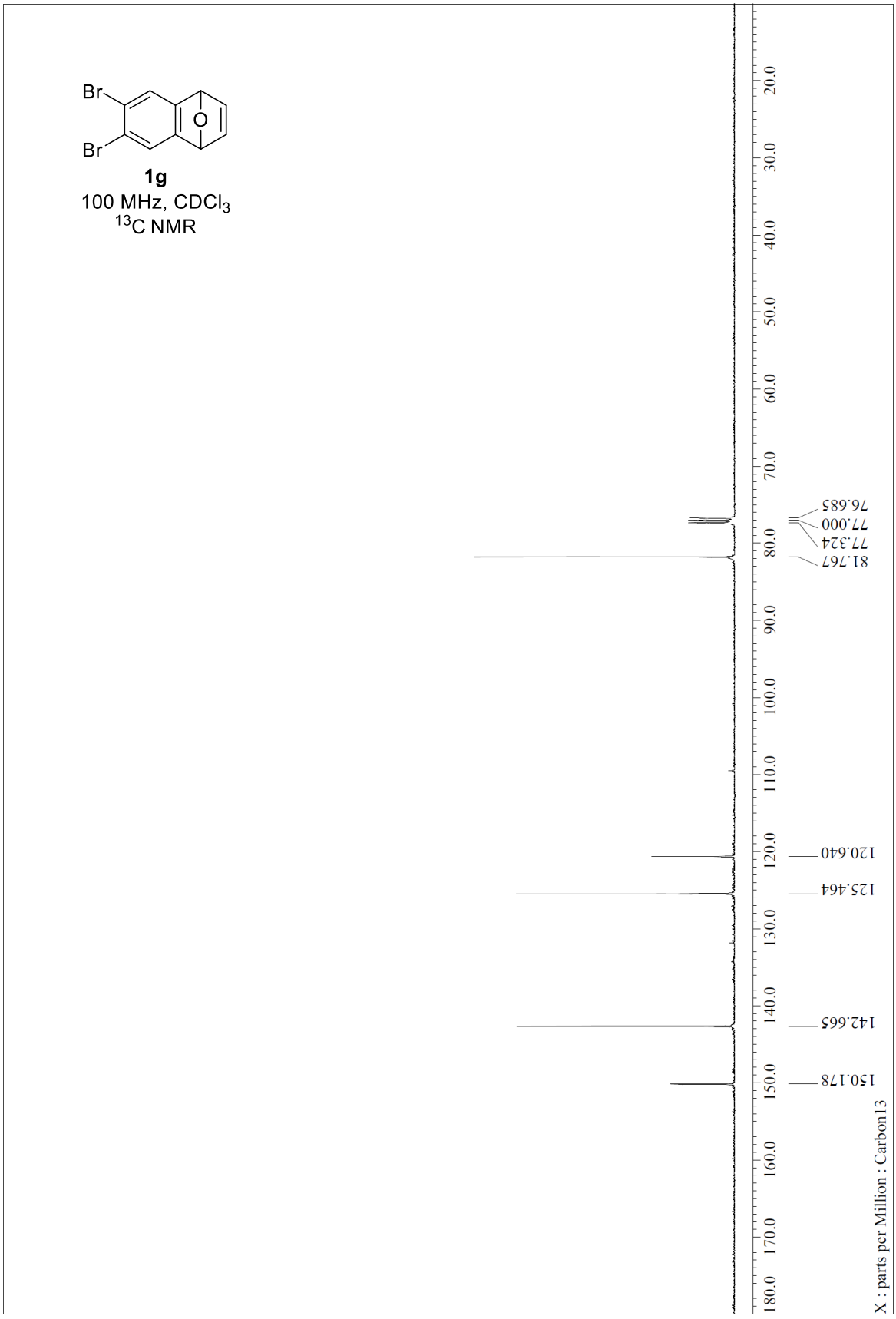

1e
400 MHz, CDCl3
1H NMR
3.01
2.402
2.391
1.00
5.800
5.796
2.00
1.01
3.05
7.277
7.260
7.255
7.243
7.063
7.049
7.035
7.031
7.015
6.999
6.994
6.979
abundance
X : parts per Million : Proton

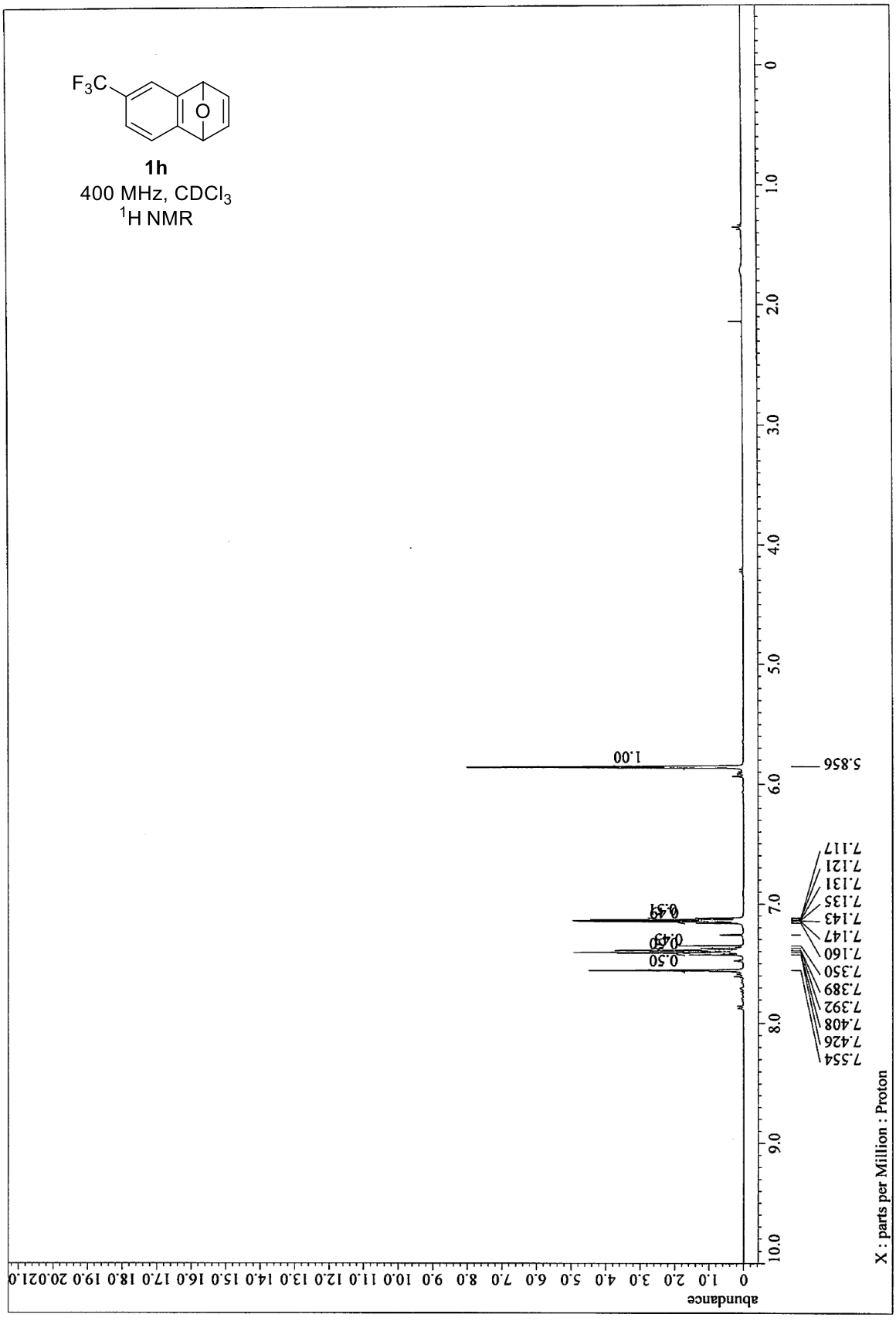

1e
100 MHz, CDCl3
13C NMR
26.772
76.676
77.000
77.315
82.254
95.650
119.524
120.535
125.159
125.569
142.226
143.351
147.441
148.014
205.173
X : parts per Million : Carbon13
abundance

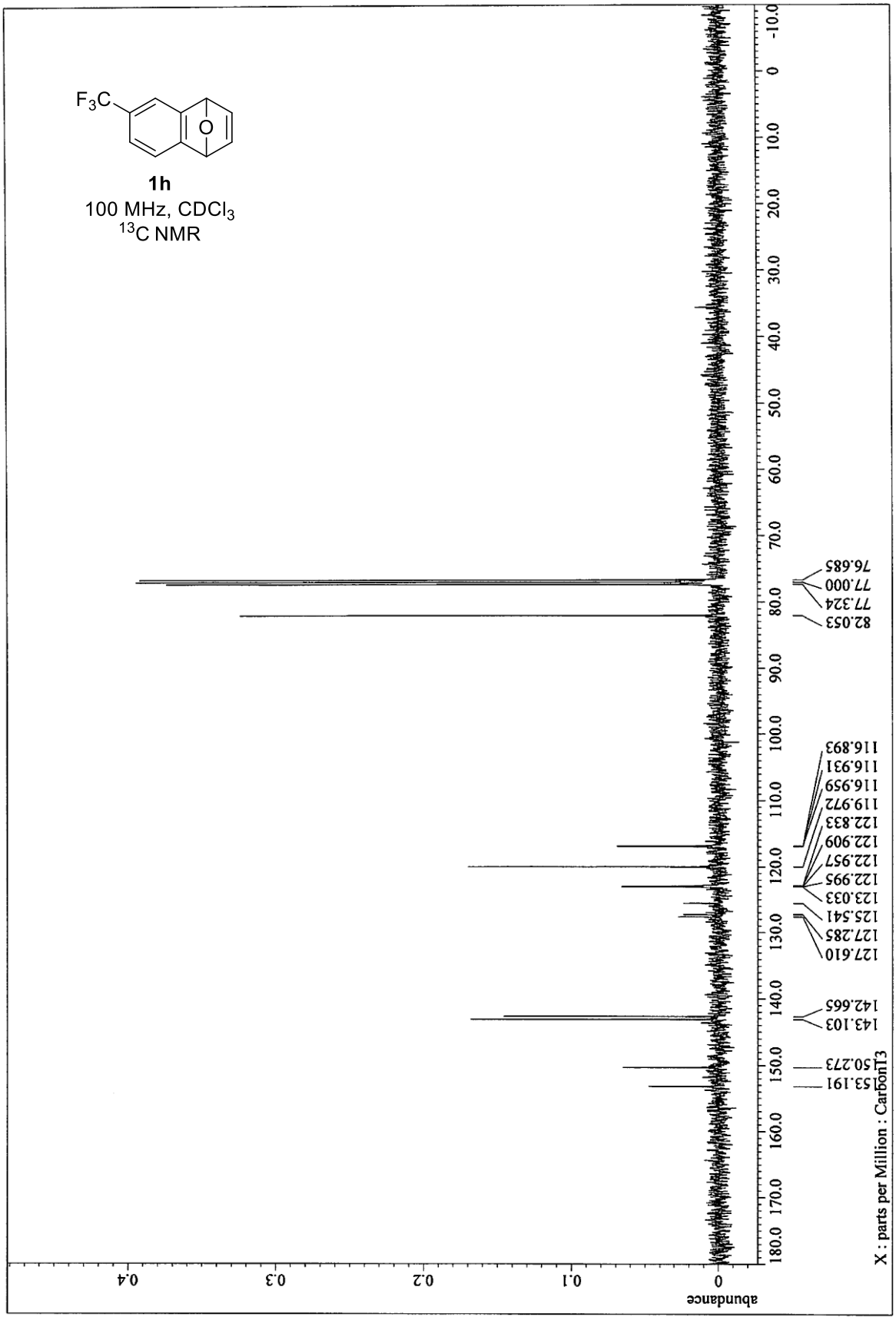

OSiMe2tBu
1f
400 MHz, CDCl3
1H NMR
6.03
9.02
0.99
1.01
1.00
2.38
0.96
0.94
0.174
0.968
1.285
4.317
4.345
4.468
4.496
5.693
5.696
6.963
6.968
6.972
6.976
6.984
6.986
7.030
7.034
7.044
7.213
7.220
7.234
7.274
abundance
X : parts per Million : Proton

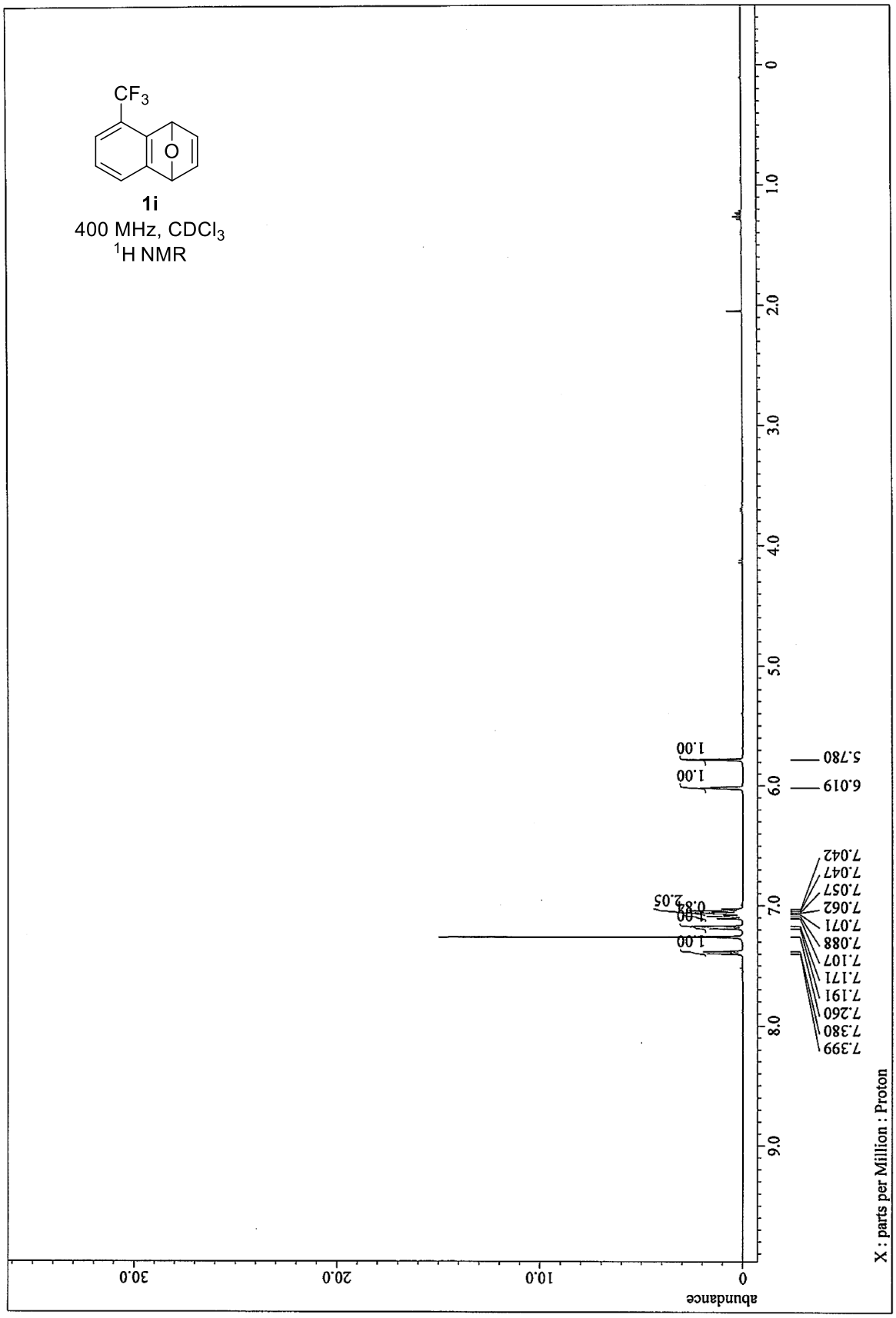

OSiMe2tBu
1f
100 MHz, CDCl3
13C NMR
-5.379
18.362
25.895
61.506
76.685
77.000
77.324
82.101
92.675
119.848
124.797
124.873
143.046
143.847
149.367
150.540
X : parts per Million : Carbon13
abundance

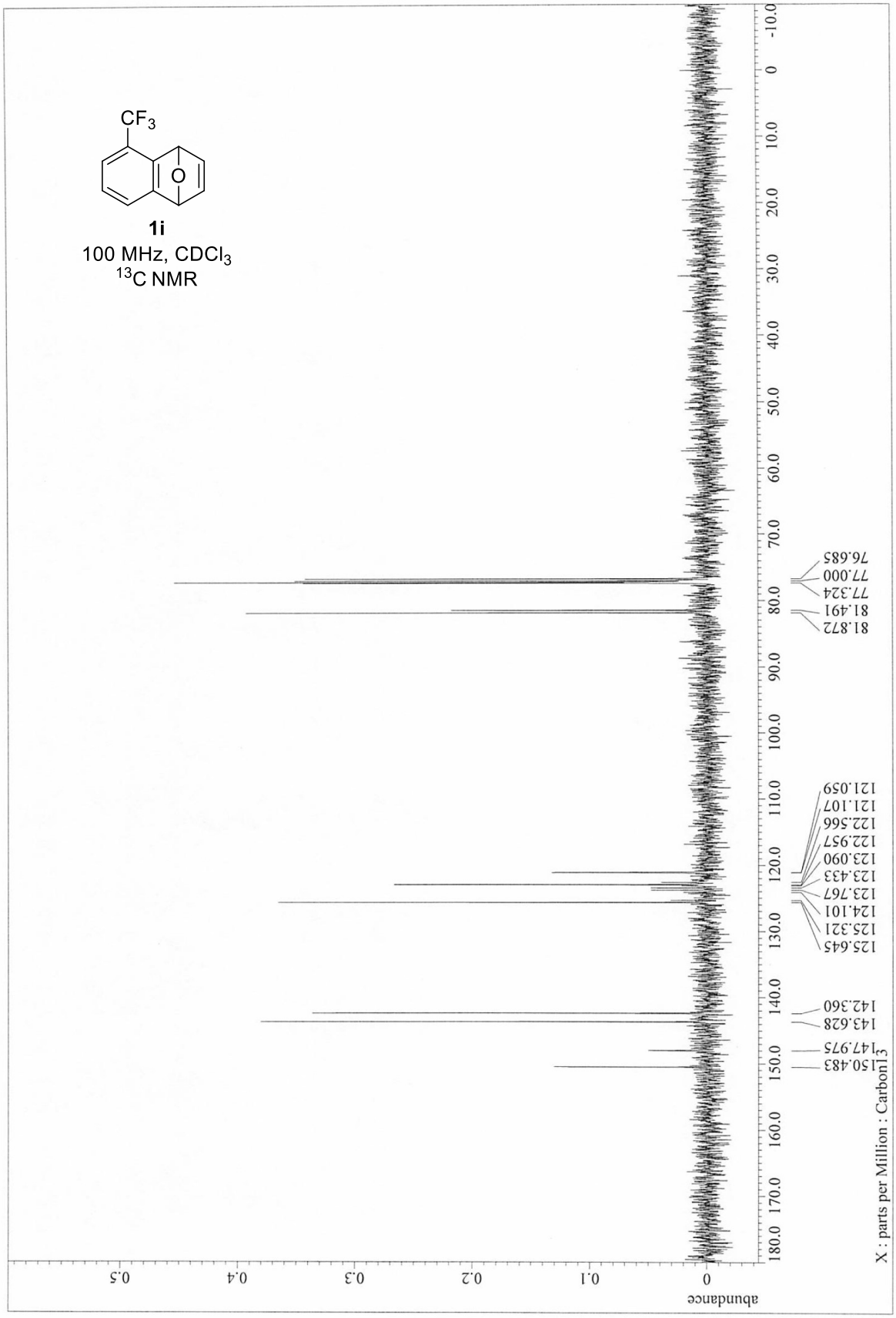

Br
Br
O
1g
400 MHz, CDCl3
1H NMR
2.00
2.02
2.02
5.664
6.994
7.260
7.471
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
X : parts per Million : Proton

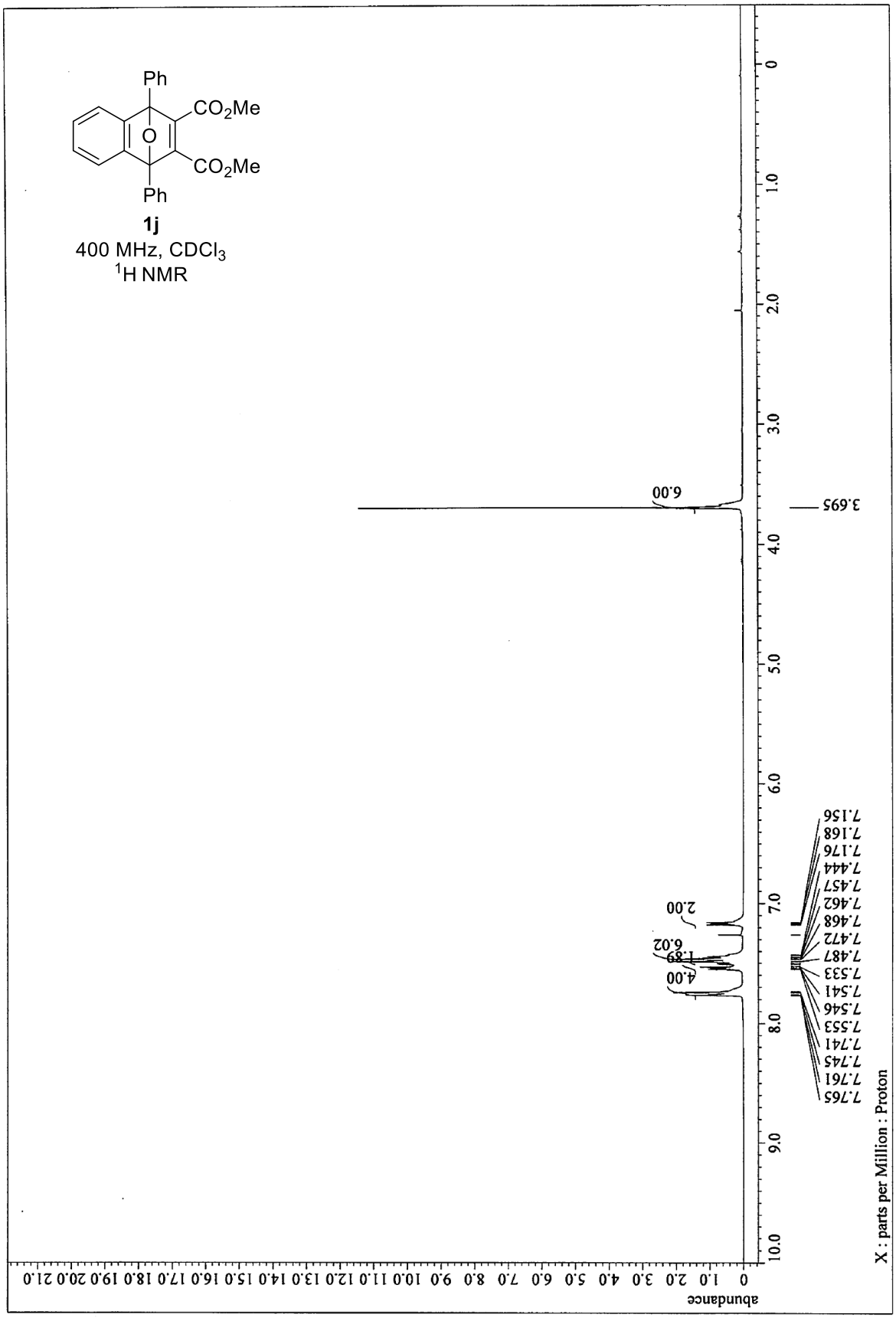

Br
Br
O
1g
100 MHz, CDCl3
13C NMR
81.767
77.324
77.000
76.685
120.640
125.464
142.665
150.178
X : parts per Million : Carbon13

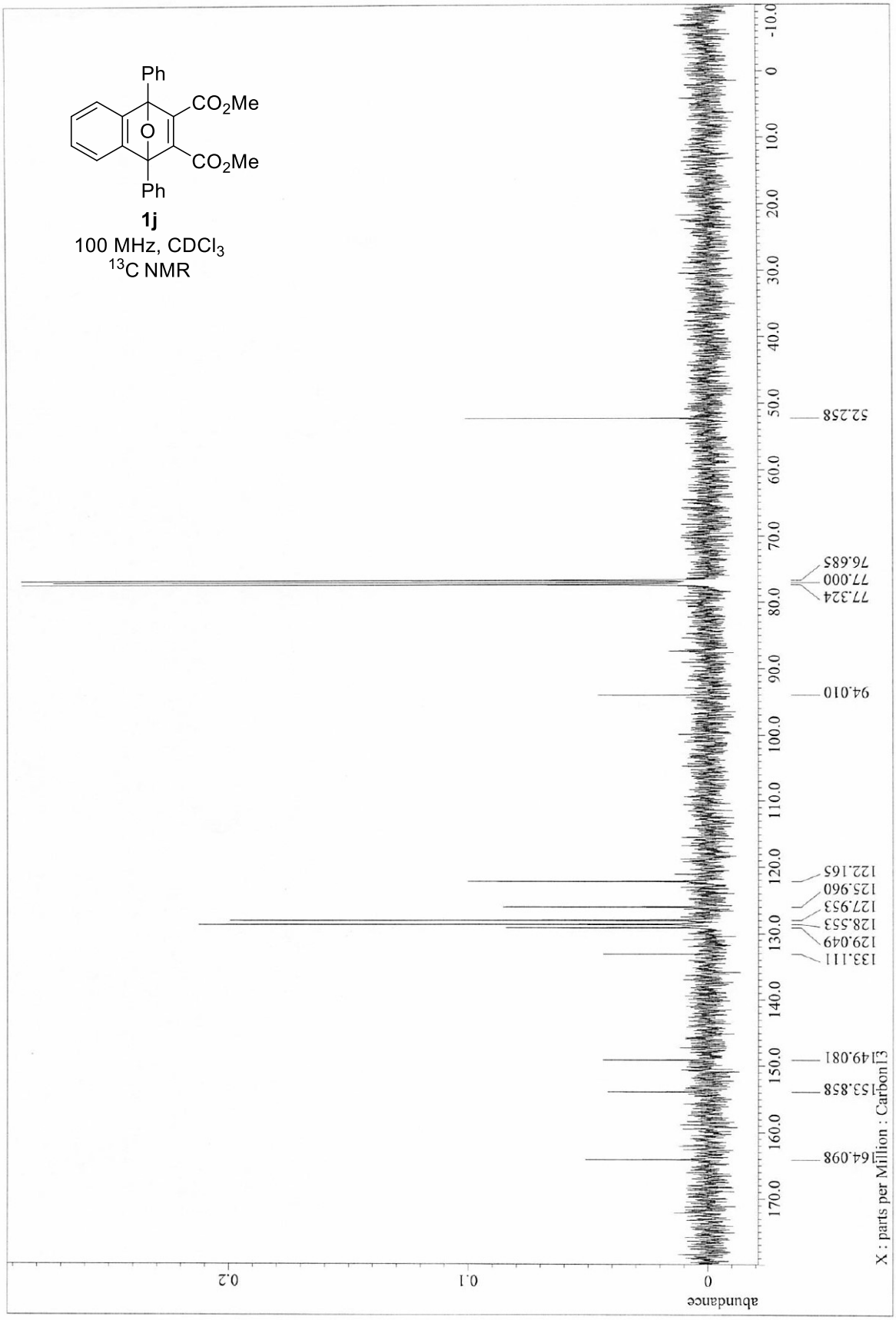

F3C
O
1h
400 MHz, CDCl3
1H NMR
1.00
0.50
5.856
7.554
7.426
7.408
7.392
7.389
7.350
7.160
7.147
7.143
7.135
7.131
7.121
7.117
X : parts per Million : Proton

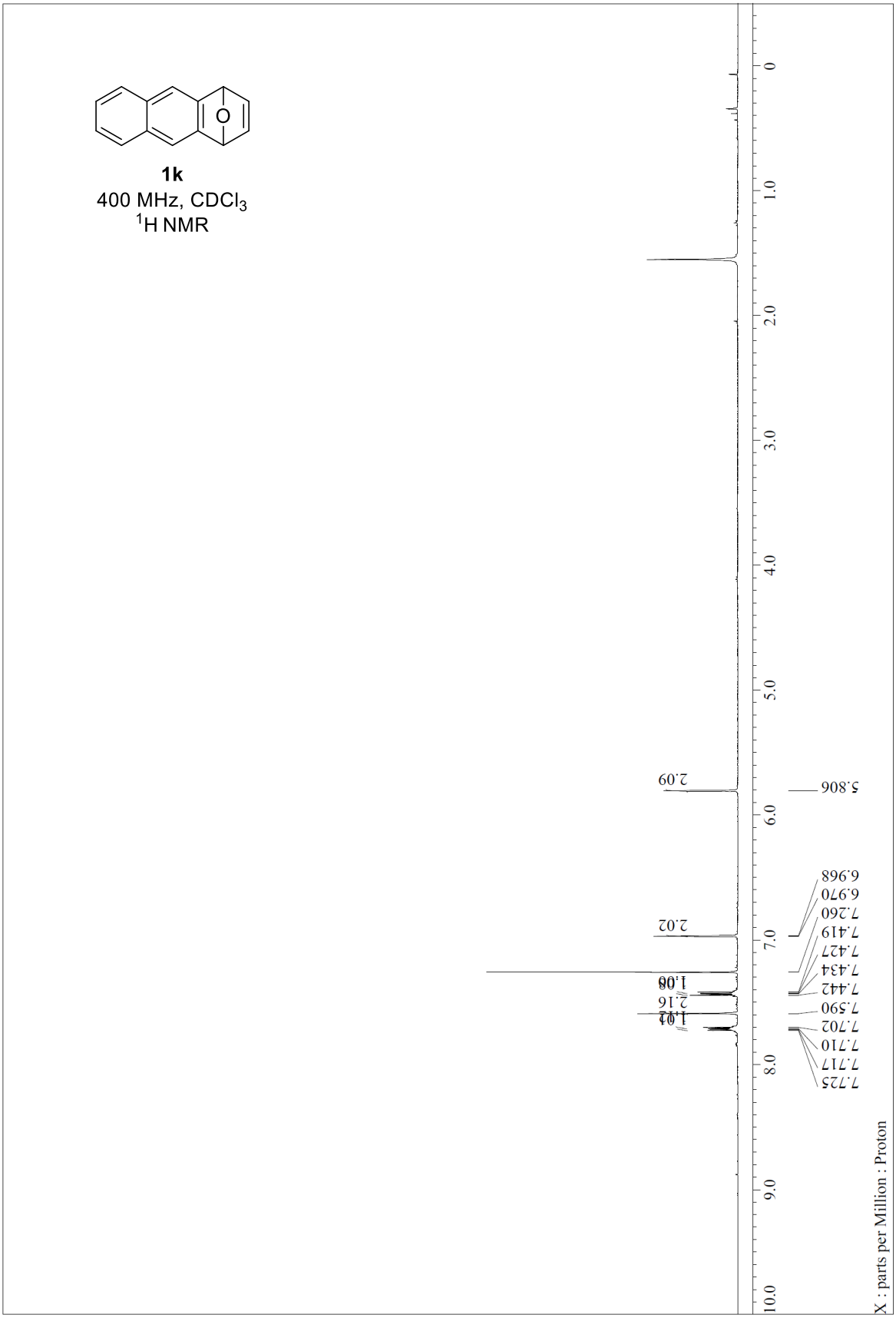

F3C
O
1h
100 MHz, CDCl3
13C NMR
abundance
X : parts per Million : Carbon13
82.053
77.324
77.000
76.685
127.610
127.285
125.541
123.033
122.995
122.957
122.909
122.833
119.972
116.959
116.931
116.893
143.103
142.665
150.273
153.191

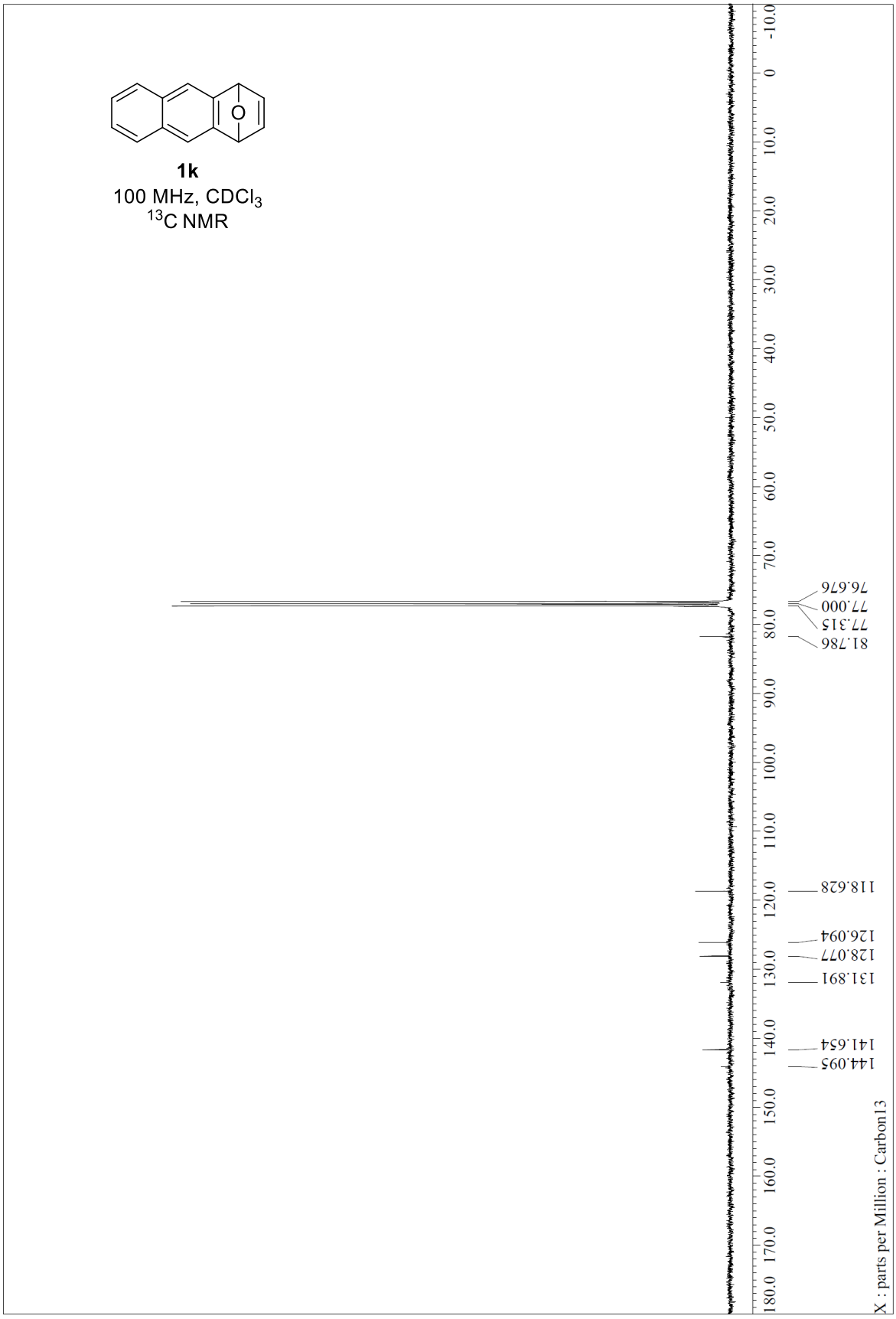

CF3
O
1i
400 MHz, CDCl3
1H NMR
abundance
X : parts per Million : Proton
5.780
6.019
7.042
7.047
7.057
7.062
7.071
7.088
7.107
7.171
7.191
7.260
7.380
7.399
1.00
1.00
0.84
2.05
1.00

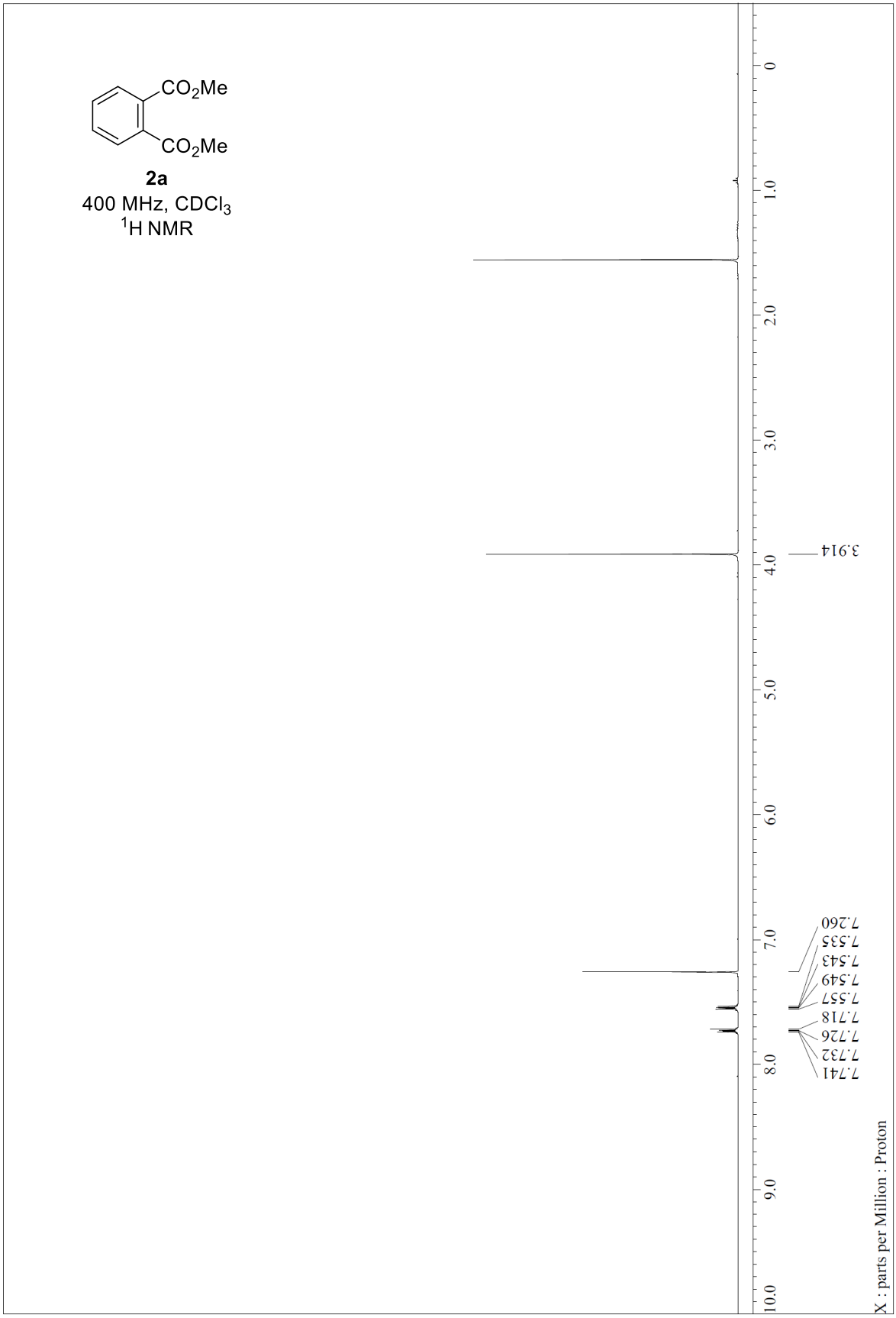

CF3
O
1i
100 MHz, CDCl3
13C NMR
abundance
X : parts per Million : Carbon13
76.685
77.000
77.324
81.491
81.872
121.059
121.107
122.566
122.957
123.090
123.433
123.767
124.101
125.321
125.645
142.360
143.628
147.975
150.483

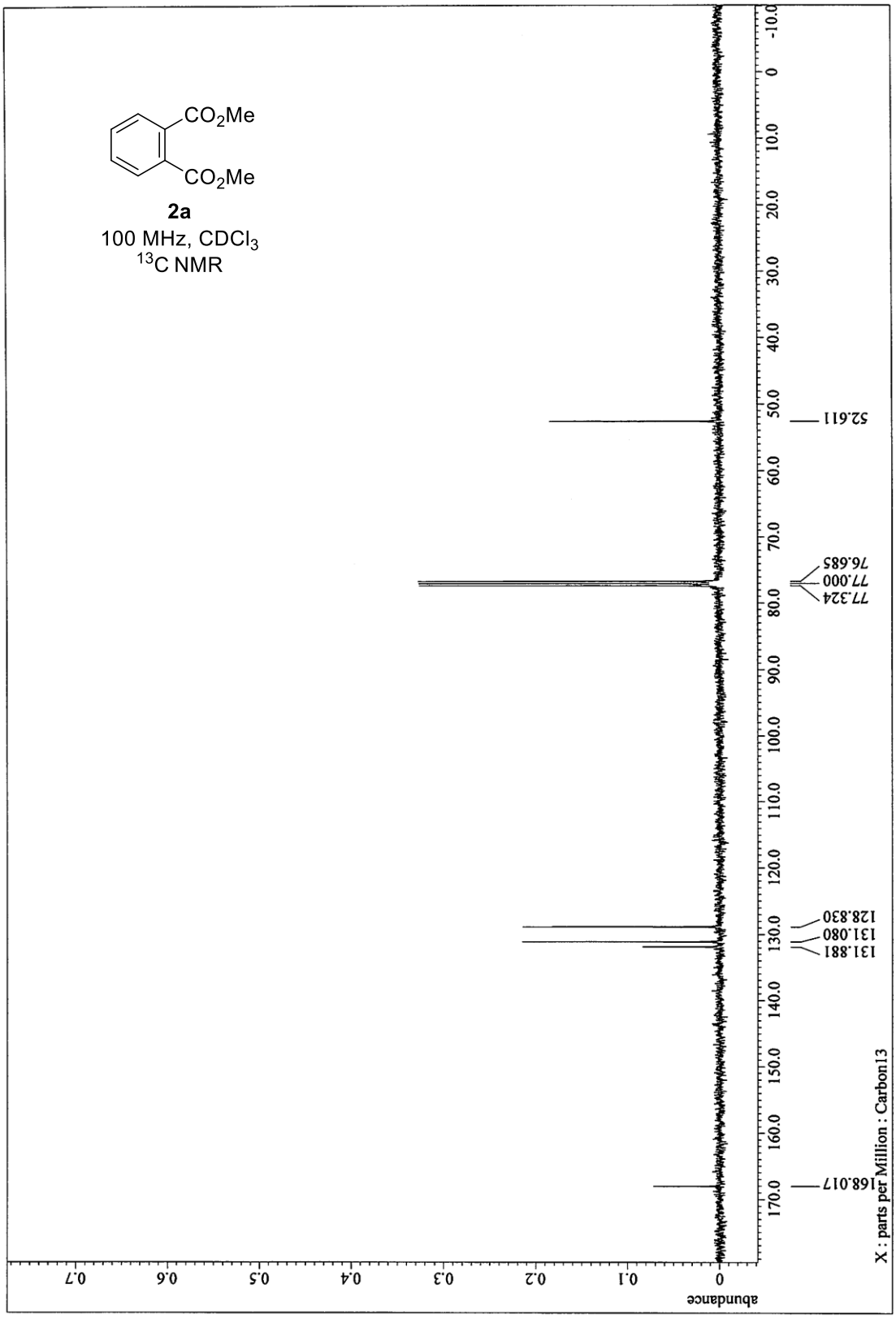

Ph
CO2Me
O
CO2Me
Ph
1j
400 MHz, CDCl3
1H NMR
3.695
6.00
2.00
6.02
1.89
4.00
7.765
7.761
7.745
7.741
7.553
7.546
7.541
7.533
7.487
7.472
7.468
7.462
7.457
7.444
7.176
7.168
7.156
X : parts per Million : Proton
abundance

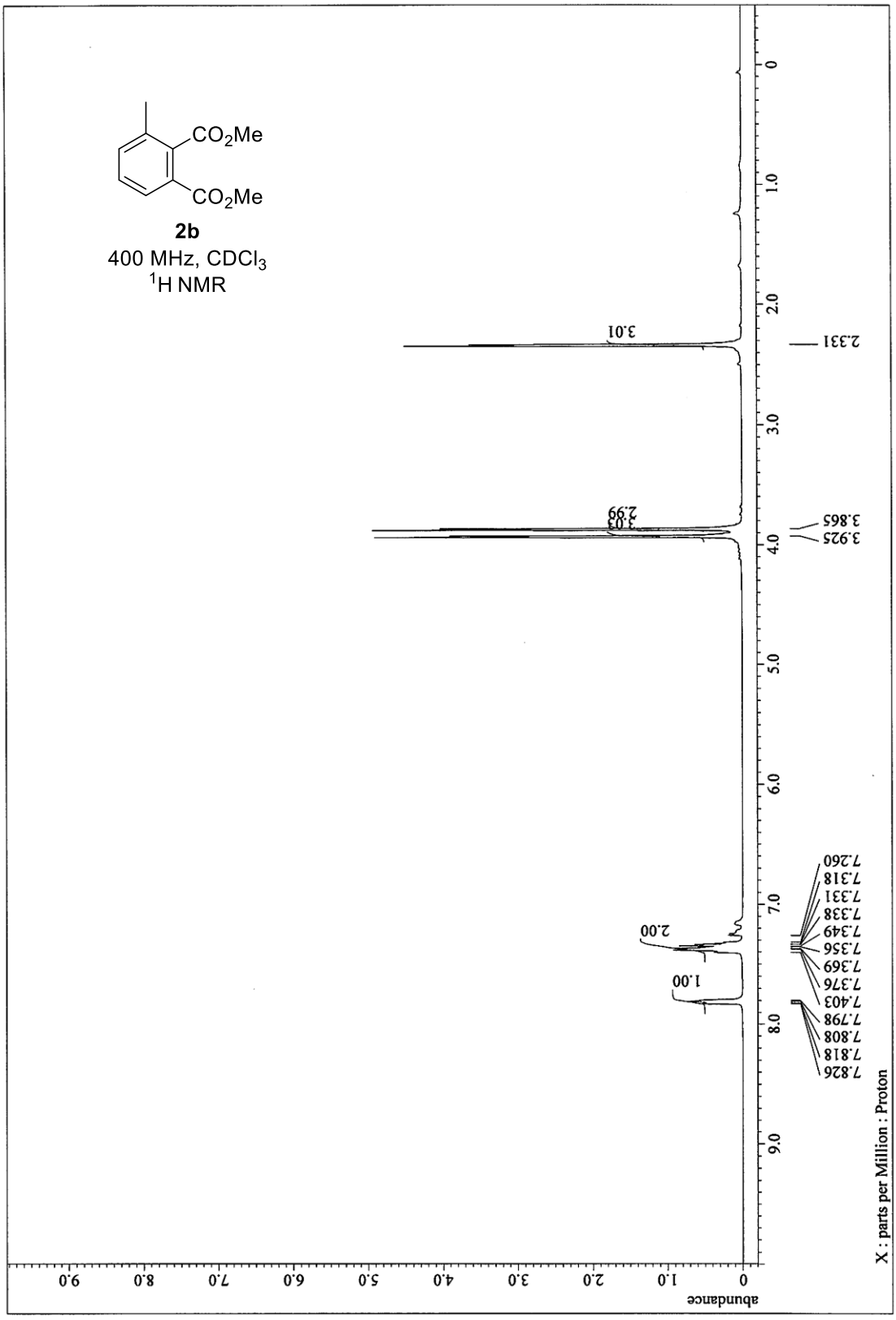

Ph
CO2Me
O
CO2Me
Ph
1j
100 MHz, CDCl3
13C NMR
52.258
76.685
77.000
77.324
94.010
122.165
125.960
127.953
128.553
129.049
133.111
149.081
153.858
164.098
X : parts per Million : Carbon13
abundance

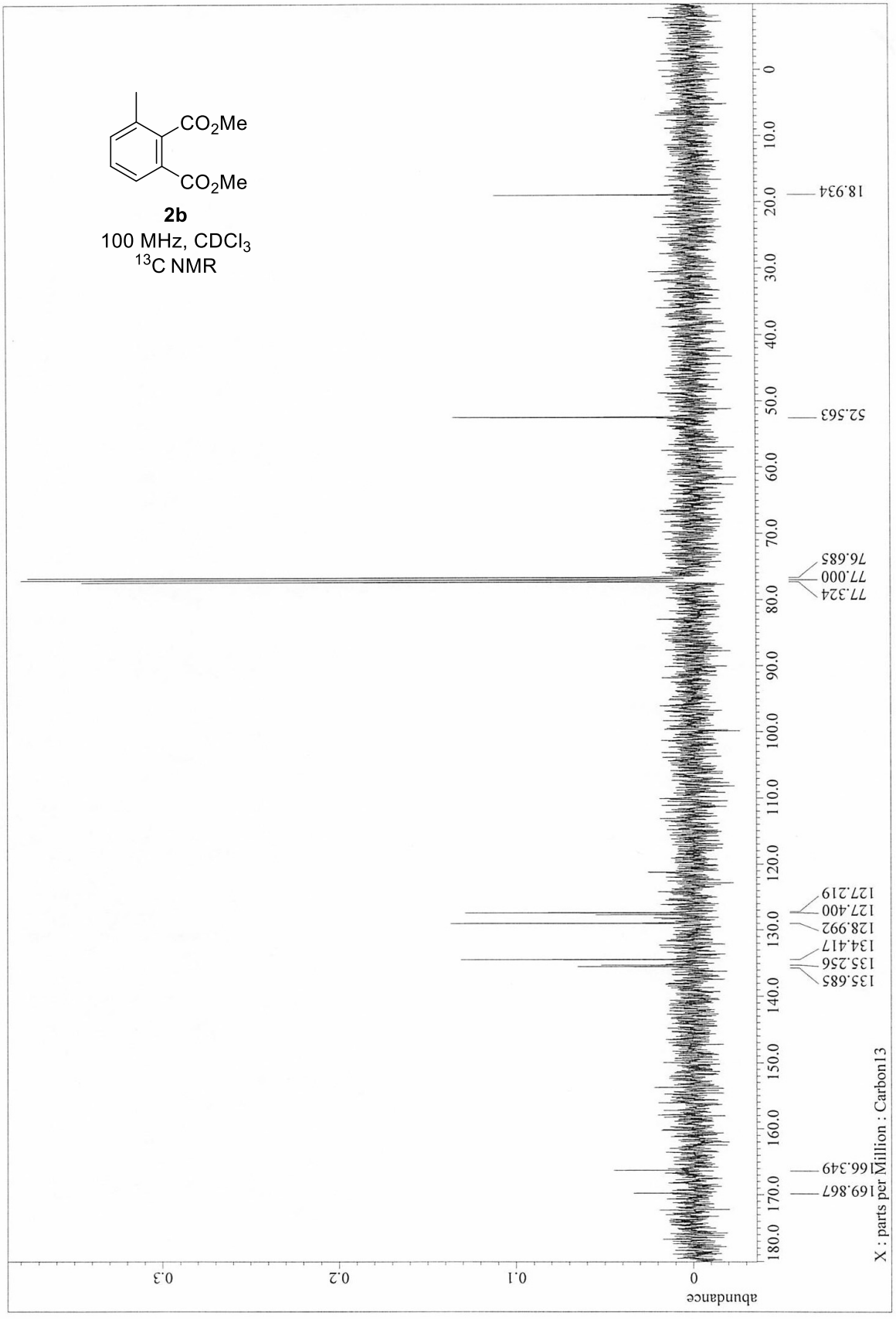

O
1k
400 MHz, CDCl3
1H NMR
2.09
2.02
1.08
2.16
1.02
5.806
6.968
6.970
7.260
7.419
7.427
7.434
7.442
7.590
7.702
7.710
7.717
7.725
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
X : parts per Million : Proton

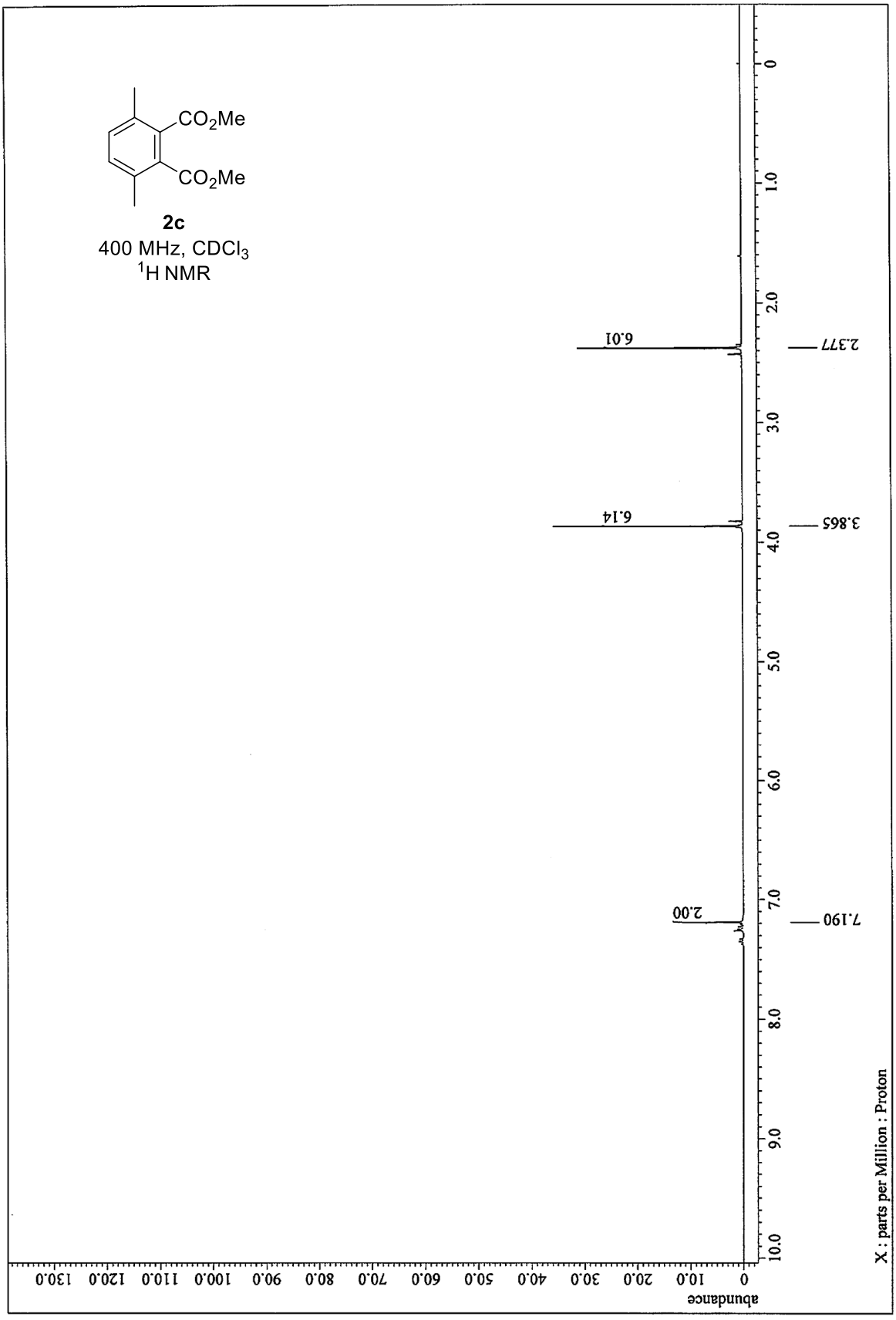

O
1k
100 MHz, CDCl3
13C NMR
76.676
77.000
77.315
81.786
118.628
126.094
128.077
131.891
141.654
144.095
X : parts per Million : Carbon13

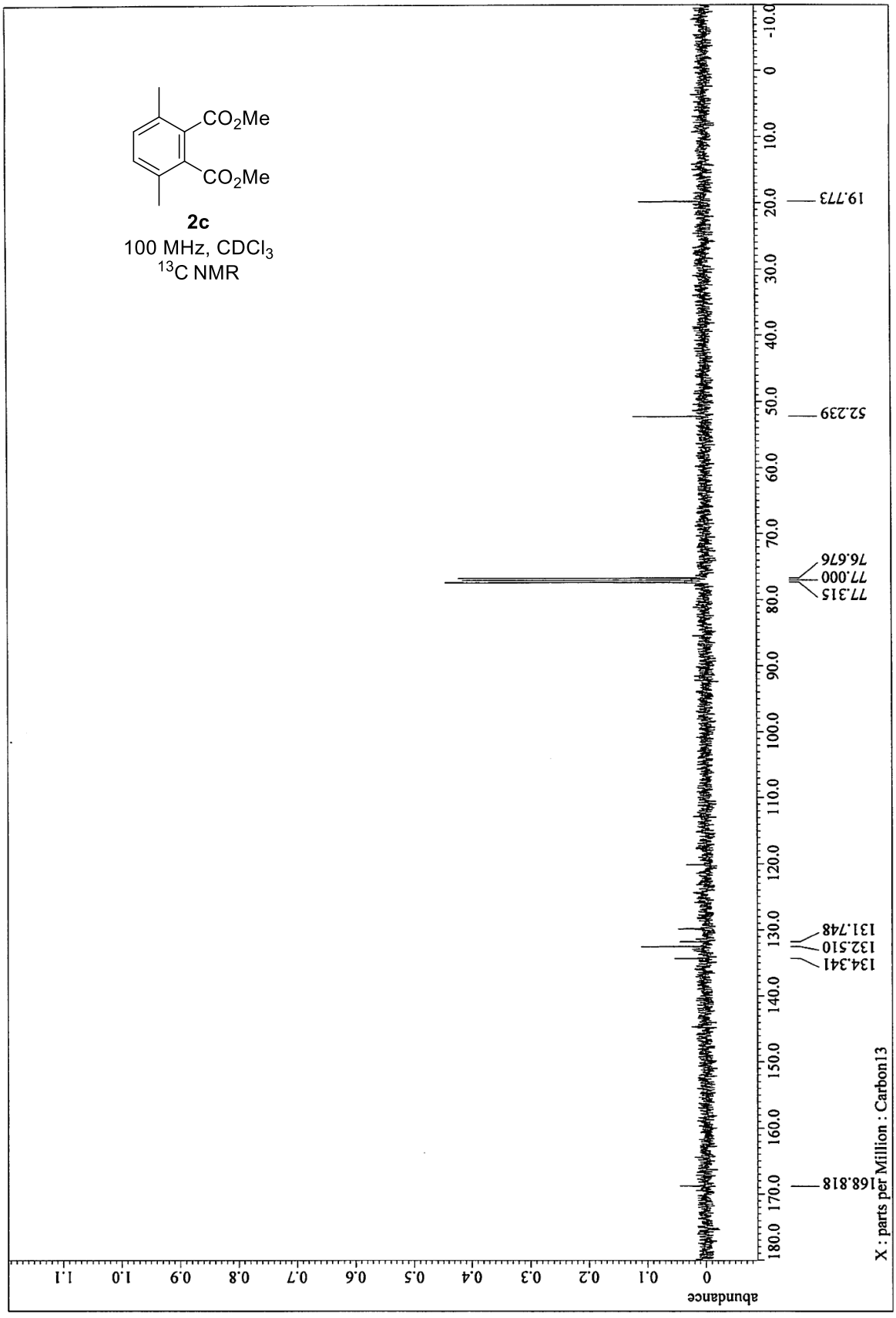

CO2Me
CO2Me
2a
400 MHz, CDCl3
1H NMR
3.914
7.260
7.535
7.543
7.549
7.557
7.718
7.726
7.732
7.741
X : parts per Million : Proton

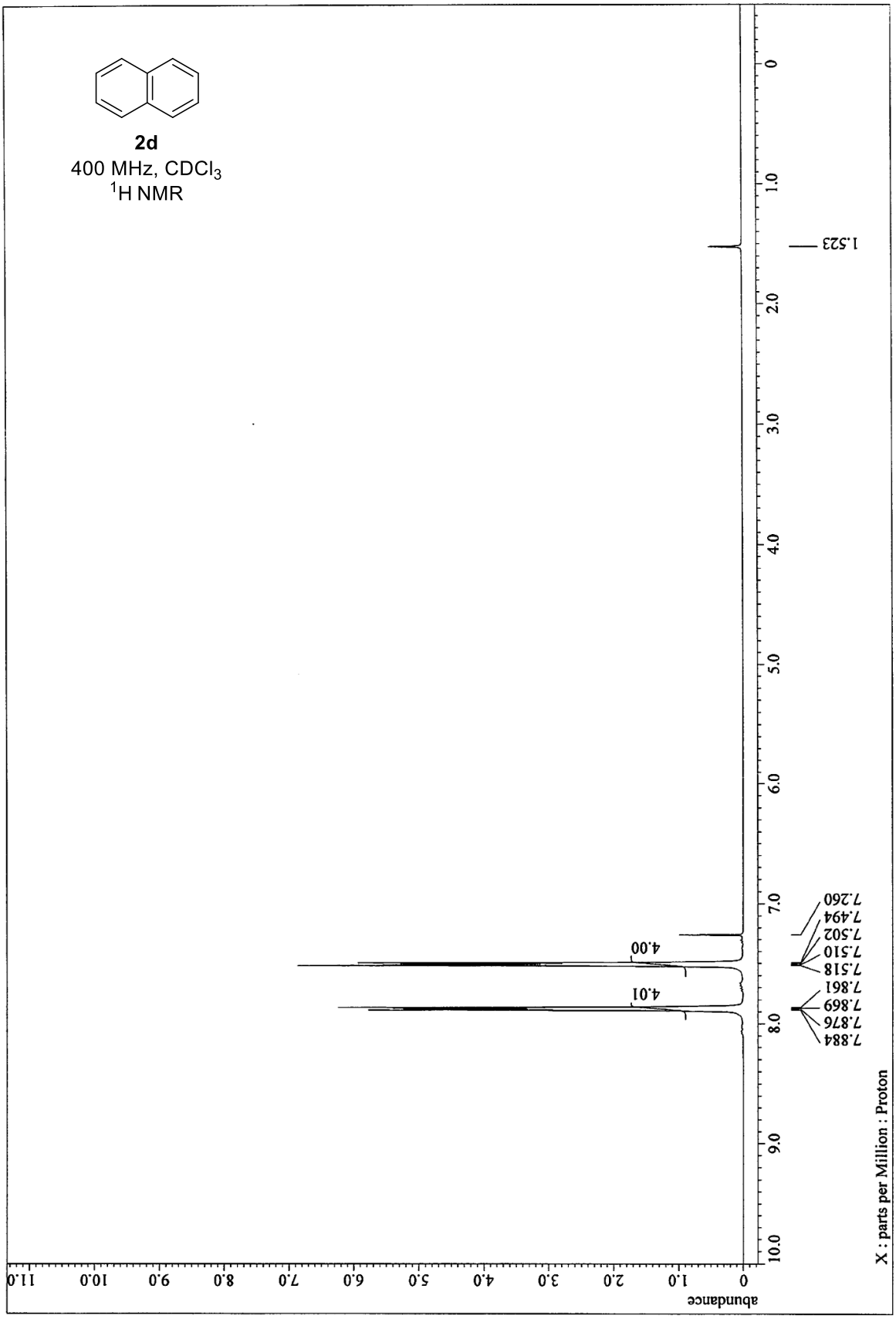

CO2Me
CO2Me
2a
100 MHz, CDCl3
13C NMR
52.611
76.685
77.000
77.324
128.830
131.080
131.881
168.017
X : parts per Million : Carbon13
abundance

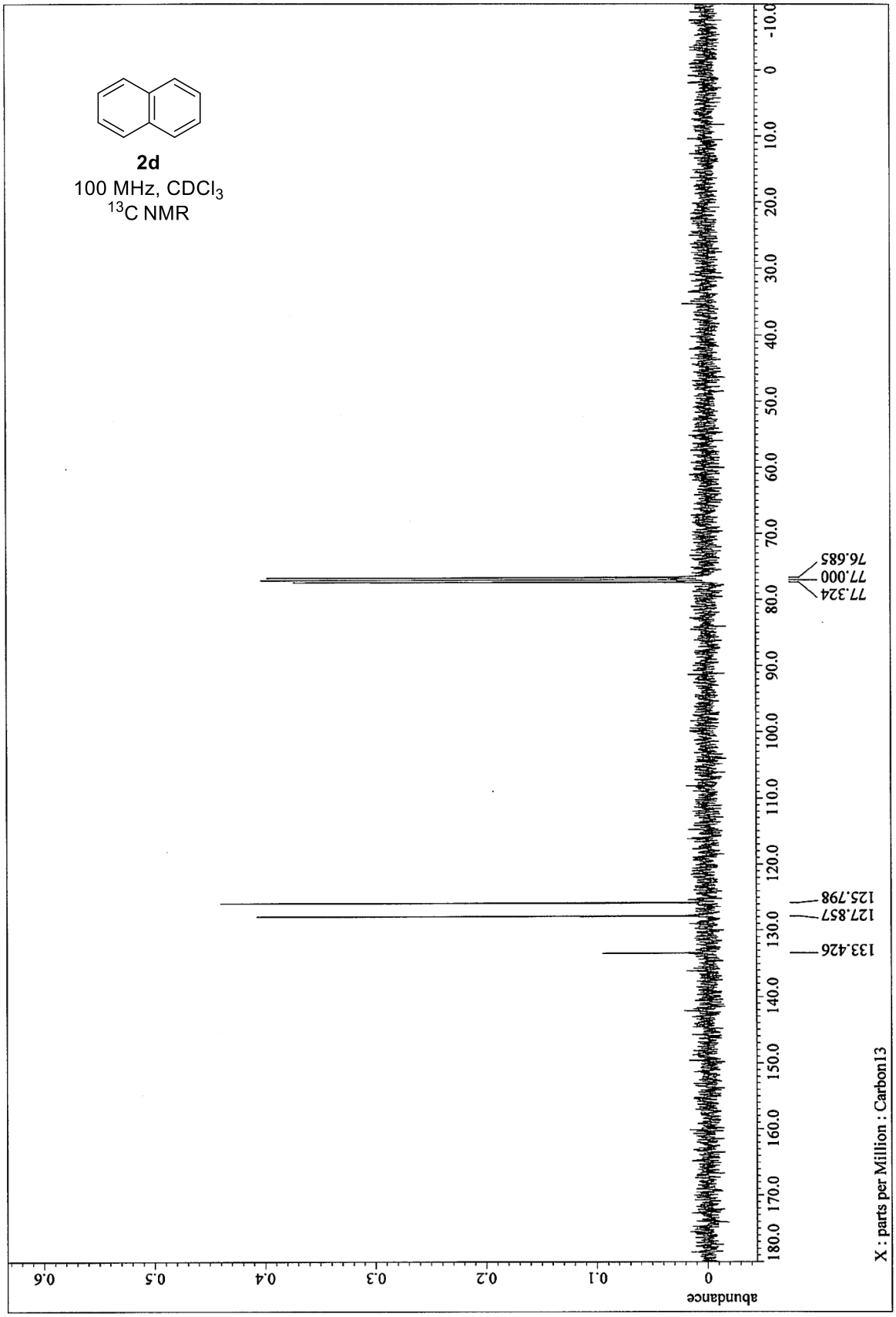

CO2Me
CO2Me
2b
400 MHz, CDCl3
1H NMR
3.01
2.99
2.00
1.00
2.331
3.865
3.925
7.260
7.318
7.331
7.338
7.349
7.356
7.369
7.376
7.403
7.798
7.808
7.818
7.826
abundance
X : parts per Million : Proton

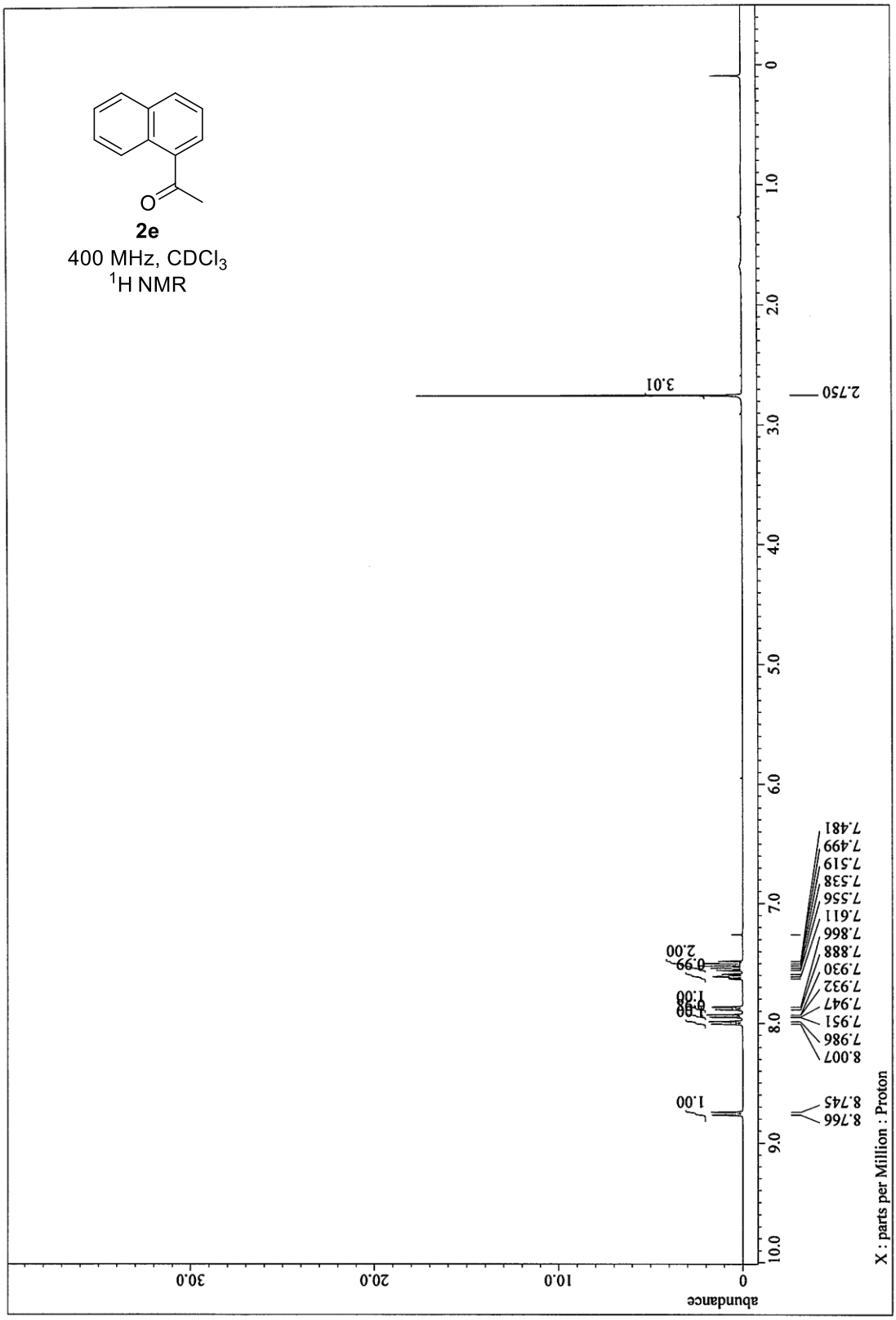

CO2Me
CO2Me
2b
100 MHz, CDCl3
13C NMR
18.934
52.563
76.685
77.000
77.324
127.219
127.400
128.992
134.417
135.256
135.685
166.349
169.867
X : parts per Million : Carbon13
abundance

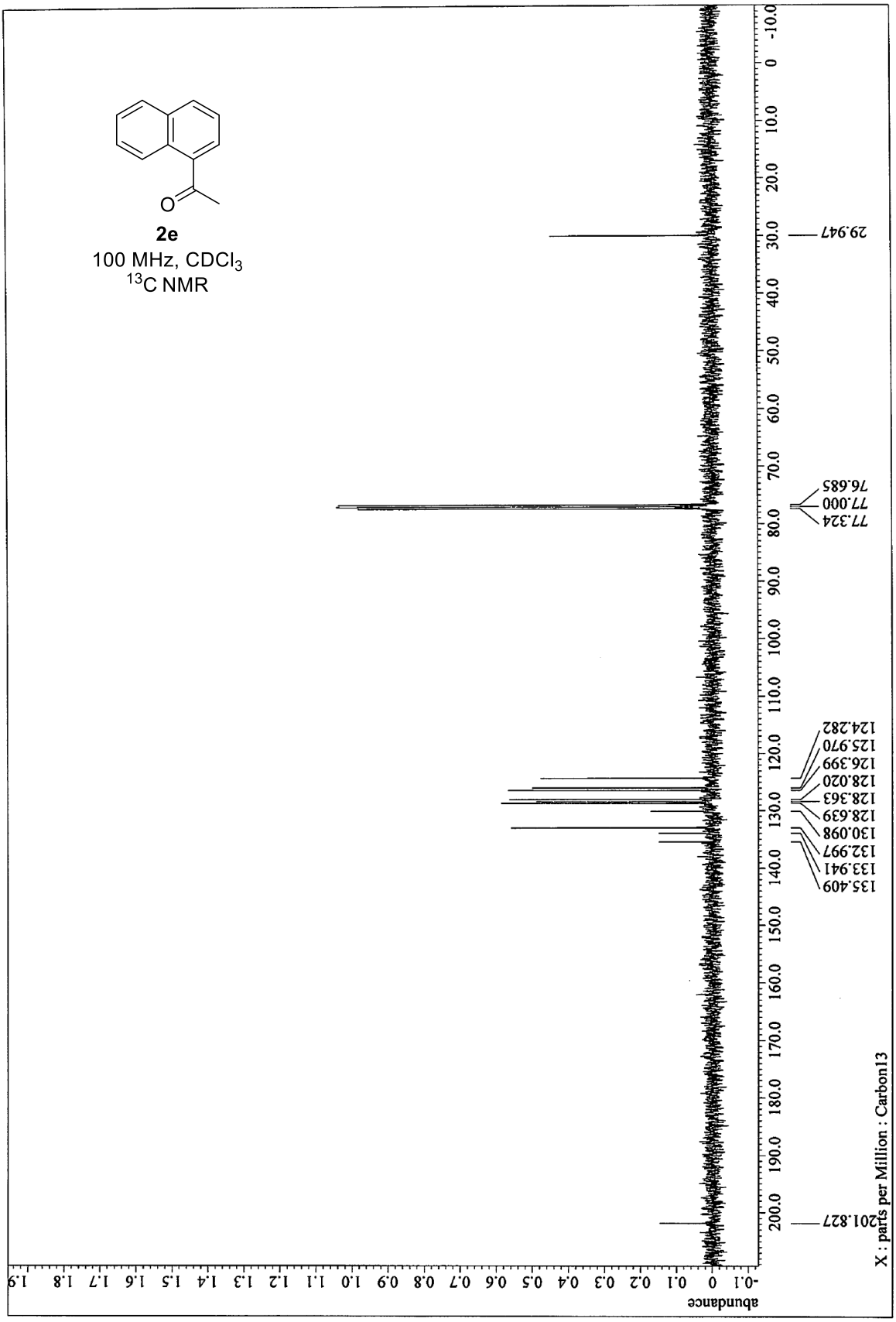

CO2Me
CO2Me
2c
400 MHz, CDCl3
1H NMR
7.190
3.865
2.377
2.00
6.14
6.01
X : parts per Million : Proton
abundance

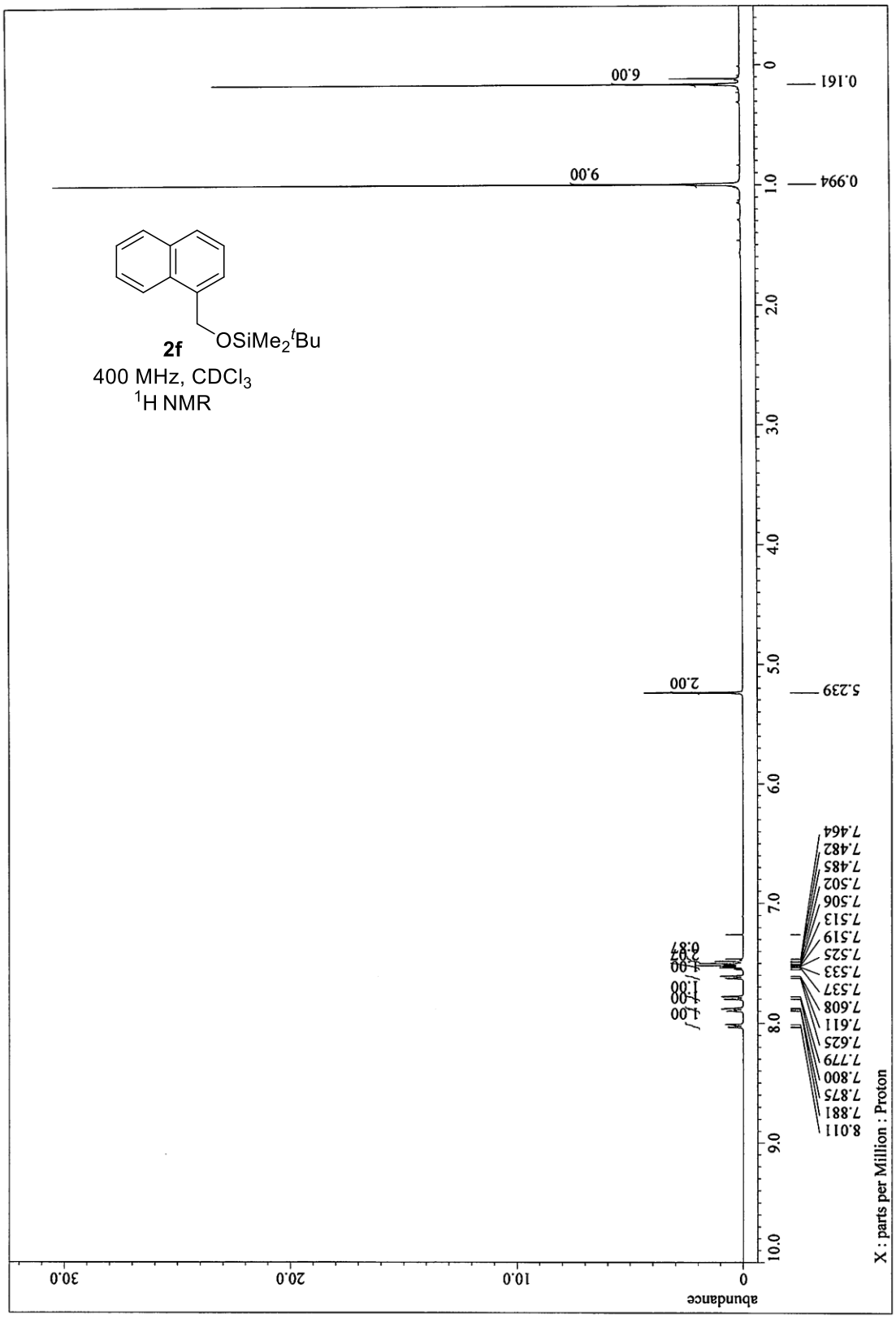

CO2Me
CO2Me
2c
100 MHz, CDCl3
13C NMR
19.773
52.239
76.676
77.000
77.315
131.748
132.510
134.341
168.818
X : parts per Million : Carbon13
abundance

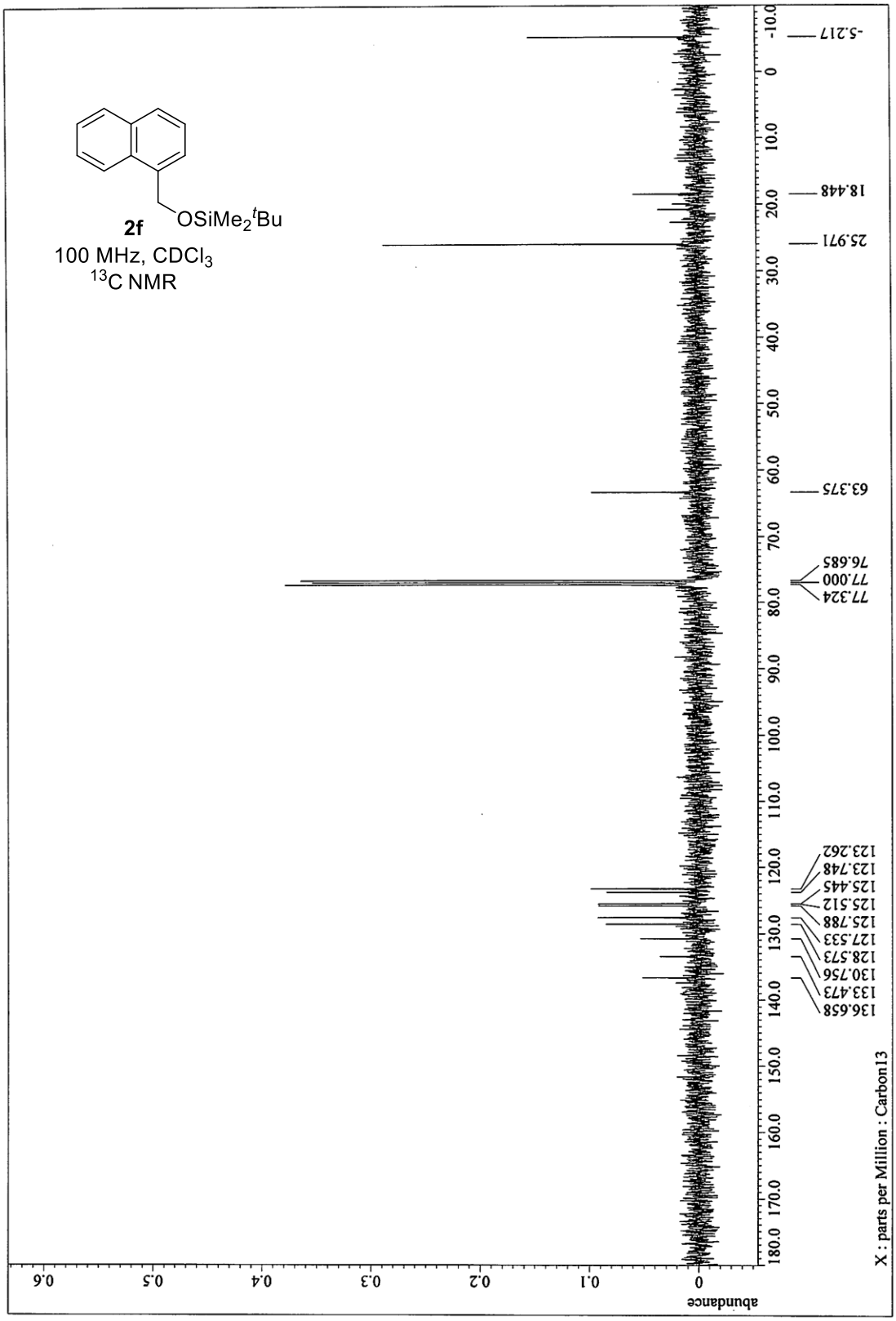

2d
400 MHz, CDCl3
1H NMR
1.523
4.00
4.01
7.260
7.494
7.502
7.510
7.518
7.861
7.869
7.876
7.884
X : parts per Million : Proton
abundance

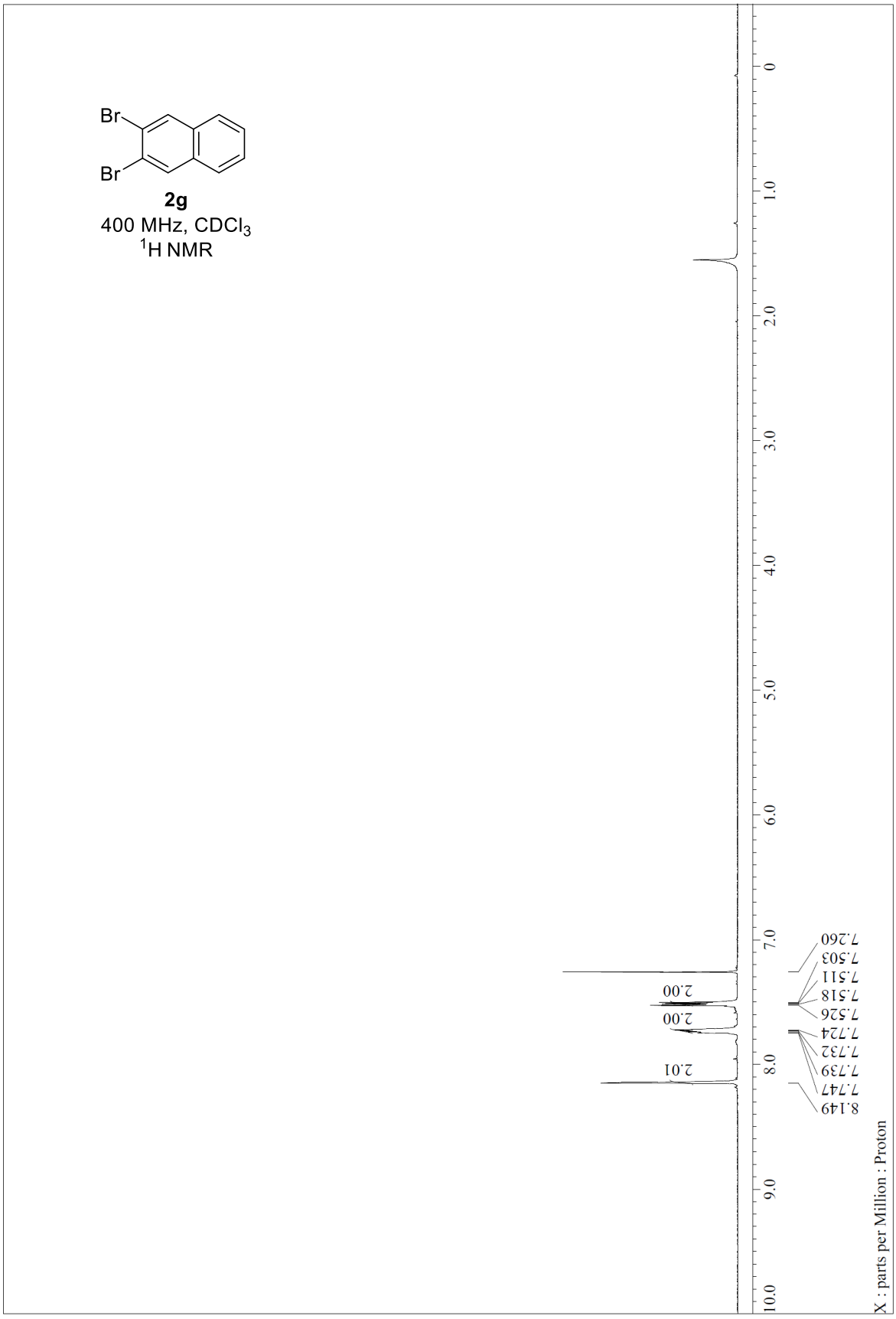

2d
100 MHz, CDCl3
13C NMR
133.426
127.857
125.798
77.324
77.000
76.685
X : parts per Million : Carbon13
abundance

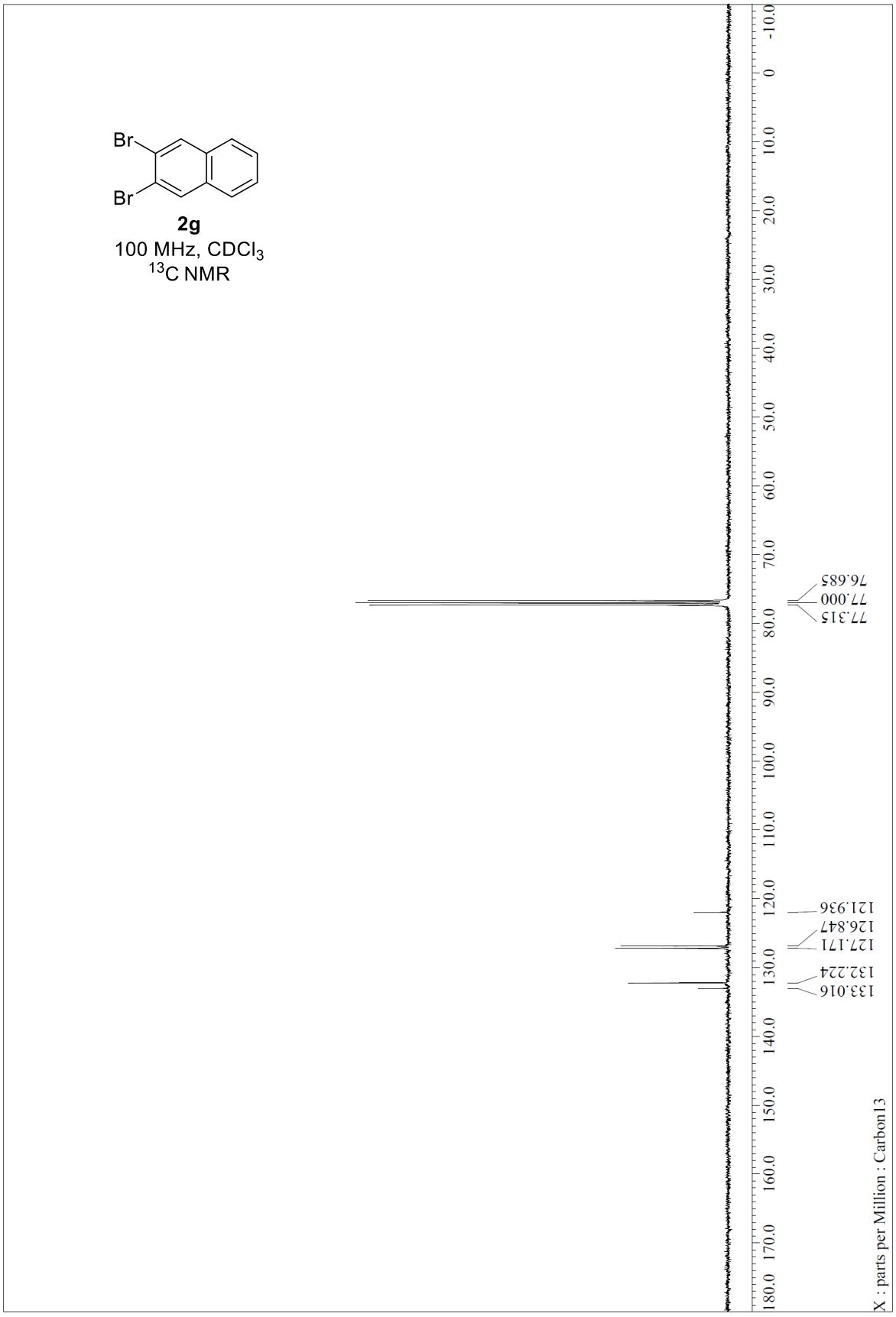

O
2e
400 MHz, CDCl3
1H NMR
3.01
2.750
2.00
0.99
1.00
1.00
1.00
8.007
7.986
7.951
7.947
7.932
7.930
7.888
7.866
7.611
7.556
7.538
7.519
7.499
7.481
8.766
8.745
1.00
abundance
X : parts per Million : Proton

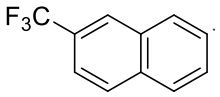

O
2e
100 MHz, CDCl3
13C NMR
29.947
76.685
77.000
77.324
124.282
125.970
126.399
128.020
128.363
128.639
130.098
132.997
133.941
135.409
201.827
X : parts per Million : Carbon13
abundance


OSiMe2tBu
2f
400 MHz, CDCl3
1H NMR
X : parts per Million : Proton
abundance

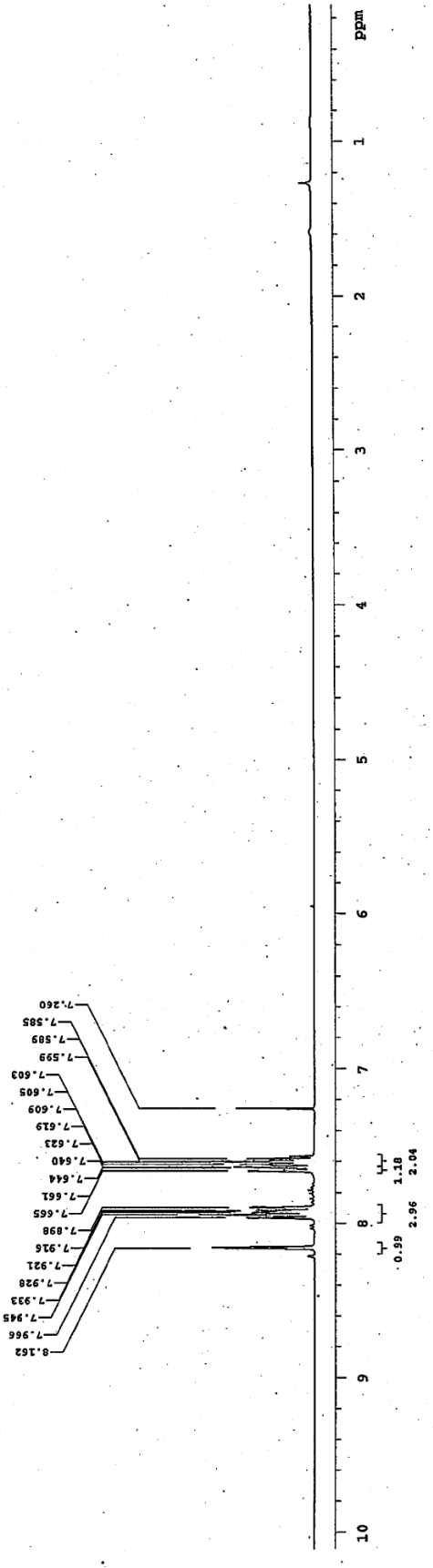

OSiMe2tBu
2f
100 MHz, CDCl3
13C NMR
-5.217
18.448
25.971
63.375
76.685
77.000
77.324
123.262
123.748
125.445
125.512
125.788
127.533
128.573
130.756
133.473
136.658
X : parts per Million : Carbon13
abundance

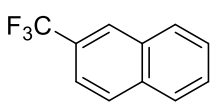

Br
Br
2g
400 MHz, CDCl3
1H NMR
2.01
2.00
2.00
8.149
7.747
7.739
7.732
7.724
7.526
7.518
7.511
7.503
7.260
X : parts per Million : Proton


Br
Br
2g
100 MHz, CDCl3
13C NMR
76.685
77.000
77.315
121.936
126.847
127.171
132.224
133.016
X : parts per Million : Carbon13



2h
400 MHz, $CDCl_3$
^1H NMR

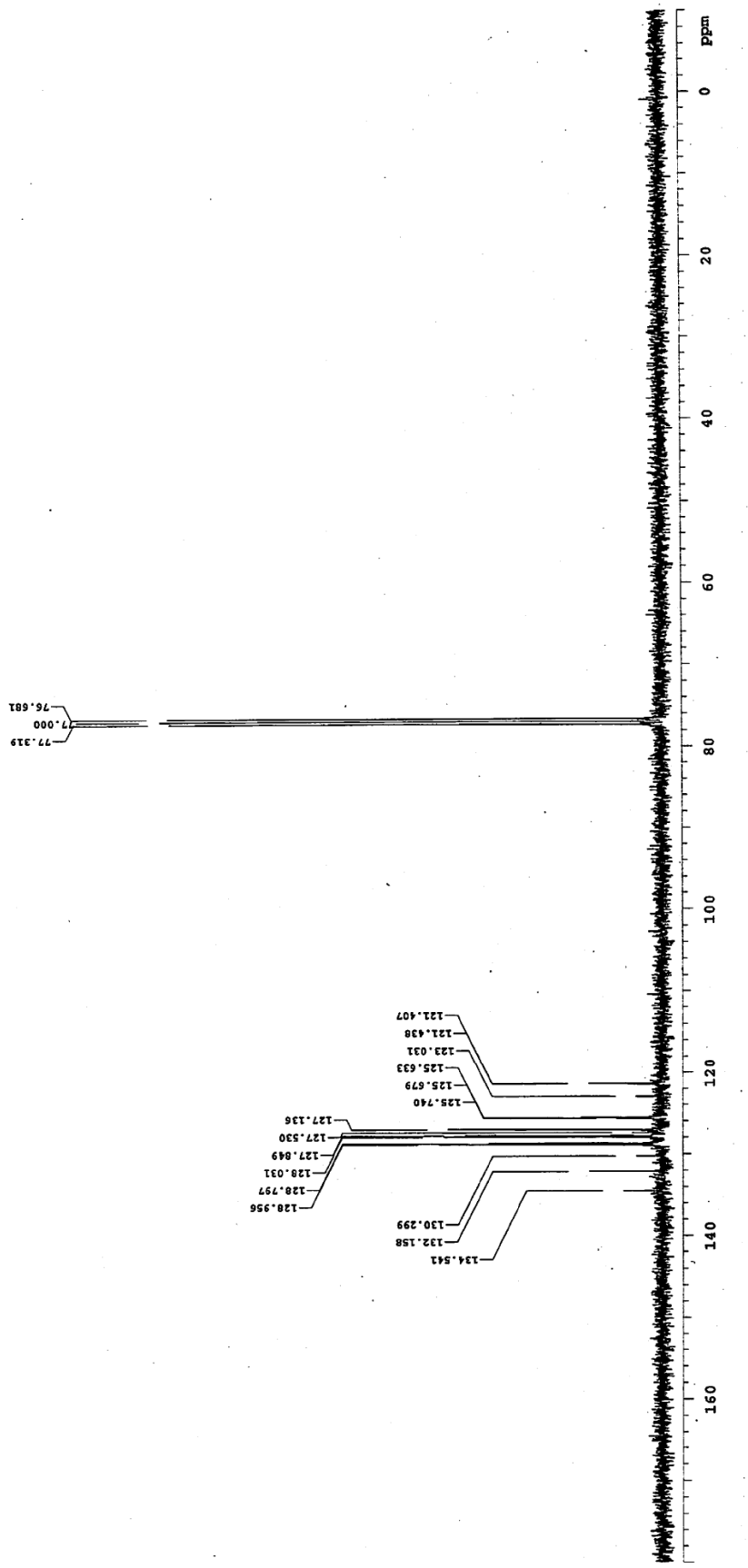


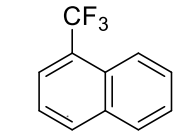


2h

100 MHz, $CDCl_3$

^{13}C NMR

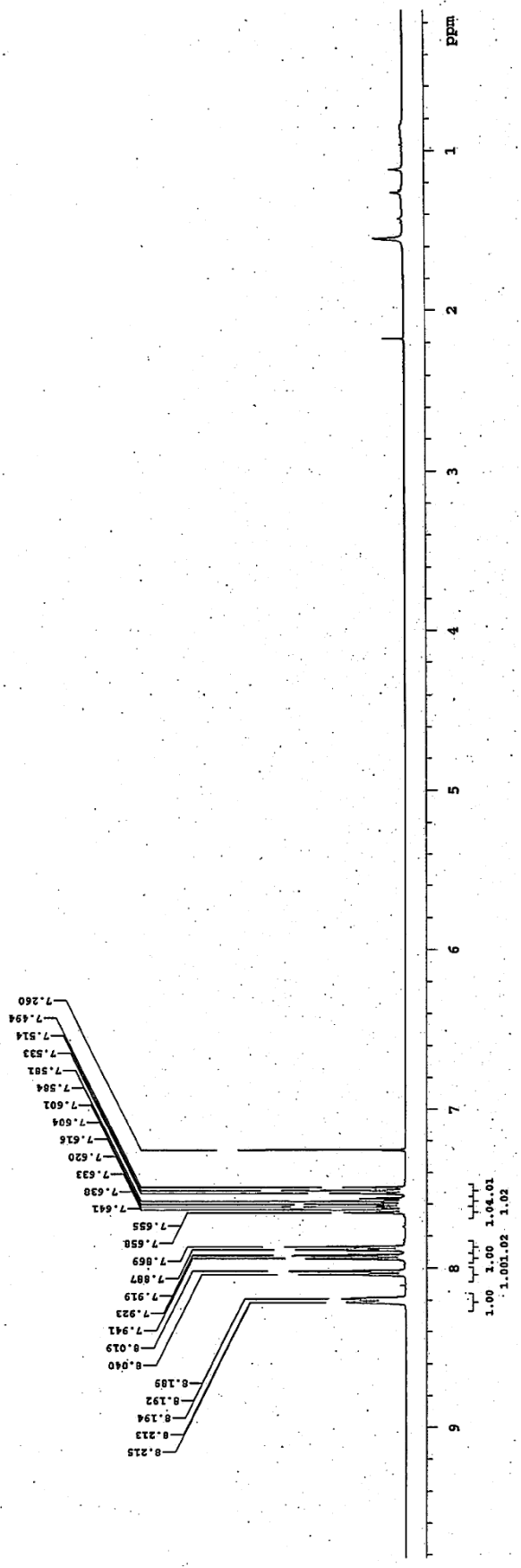


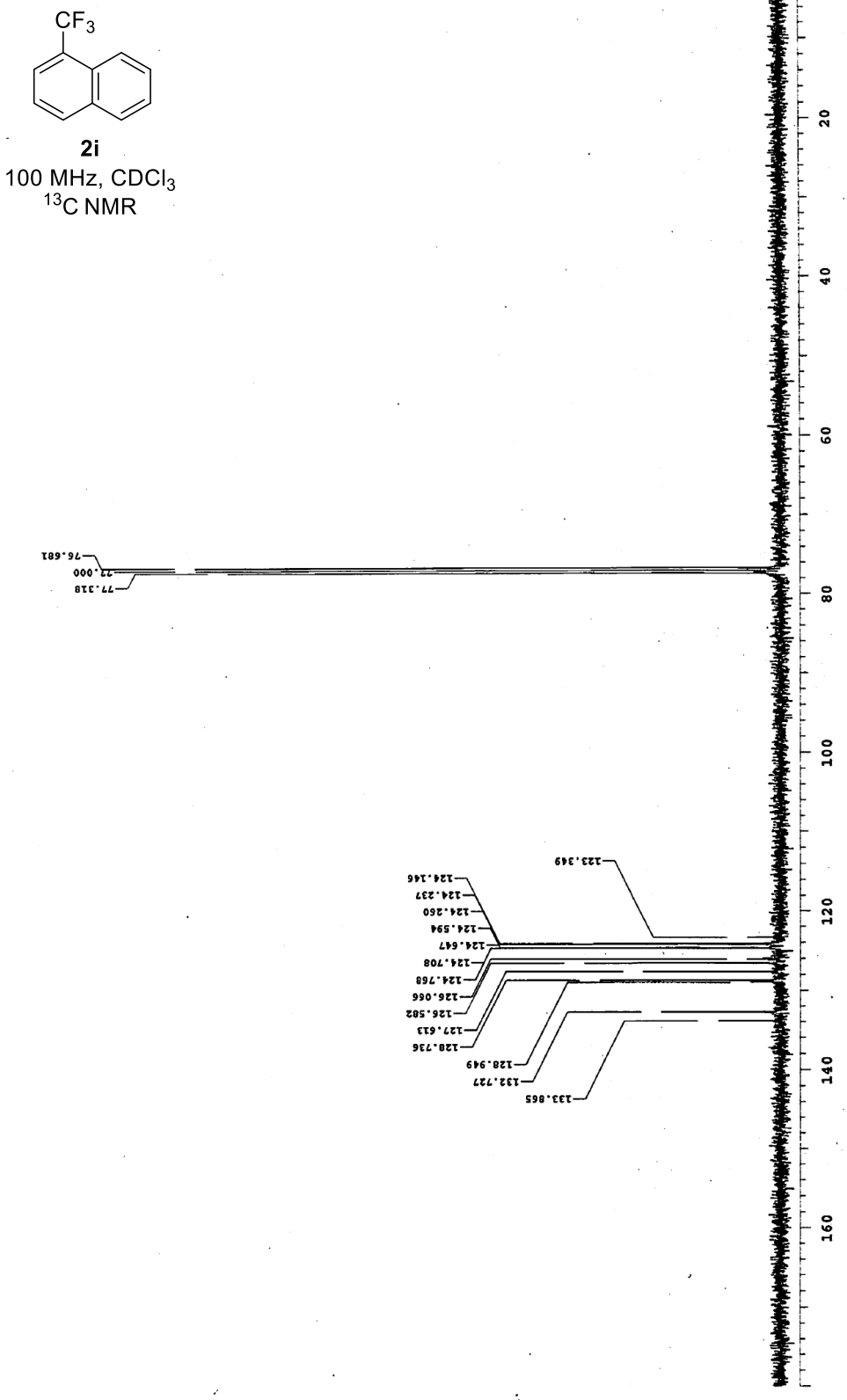


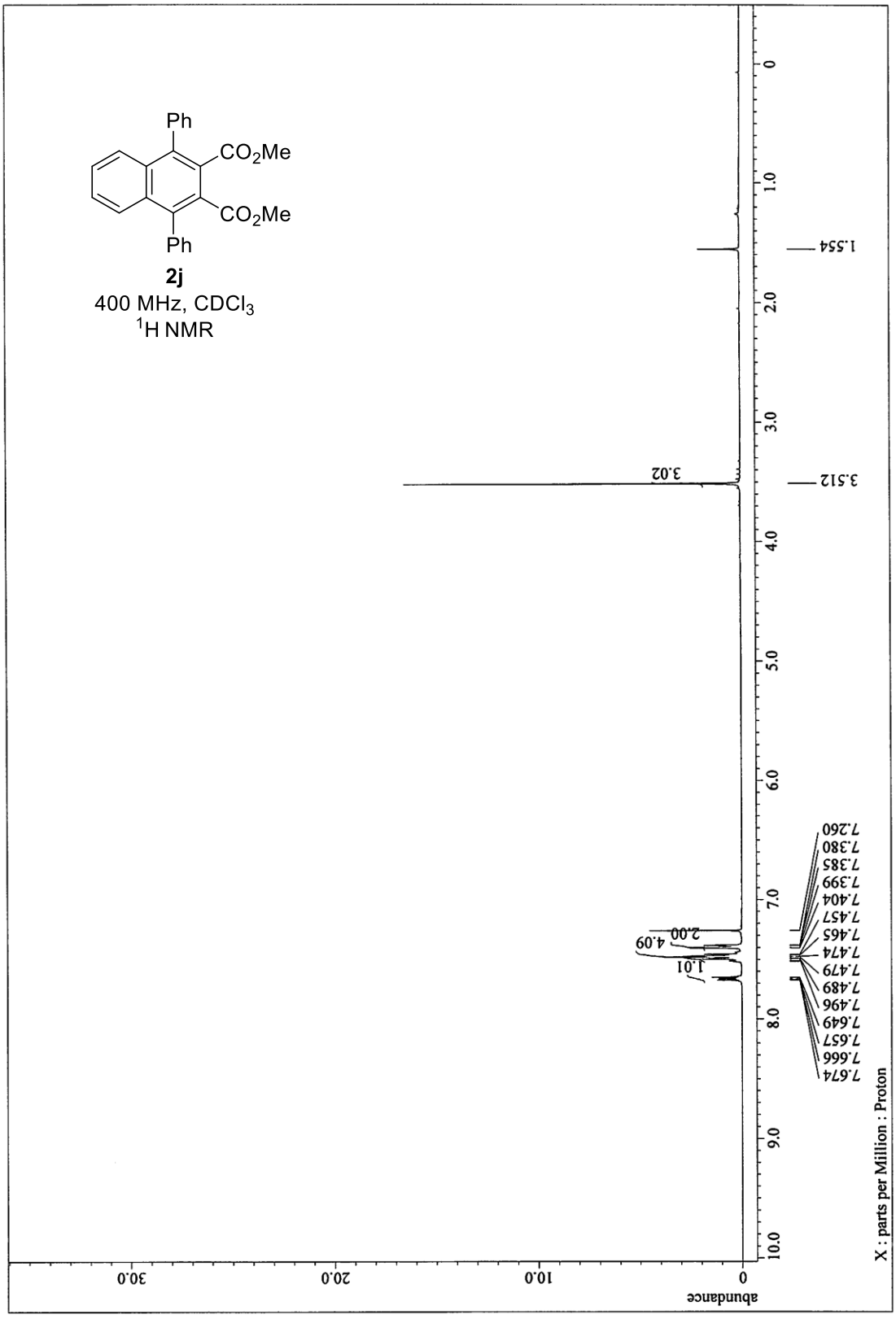
2i

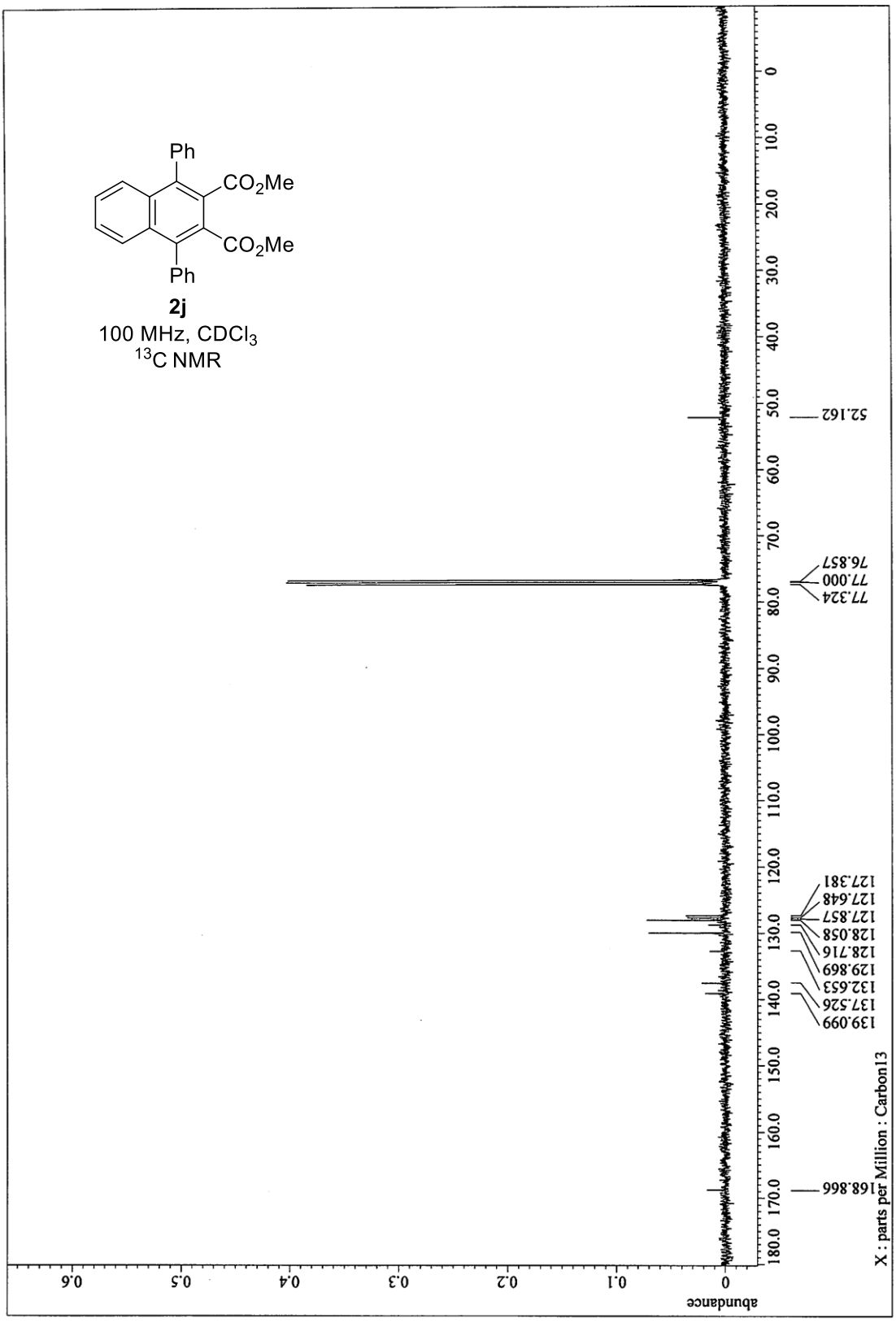
400 MHz, $CDCl_3$

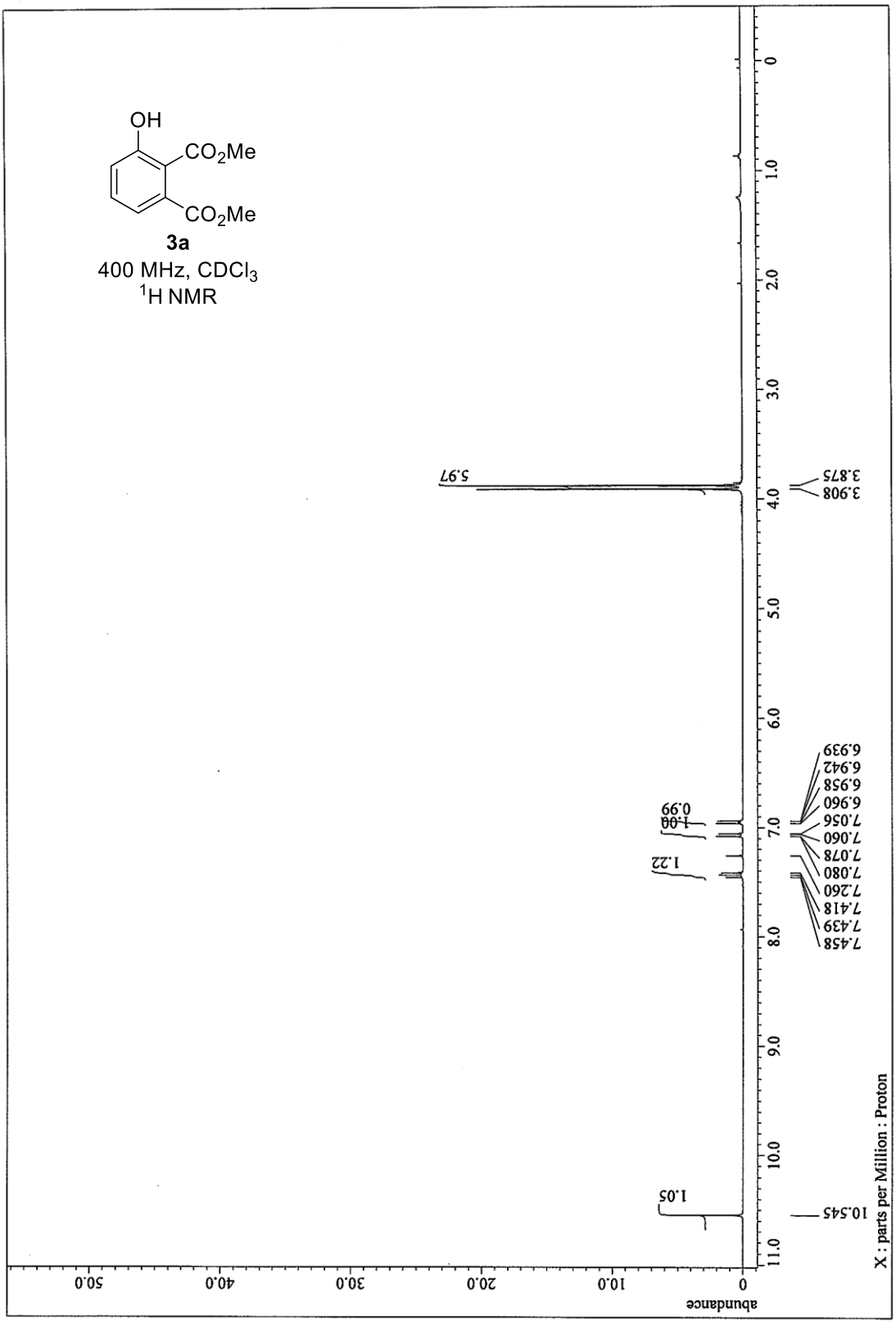
^1H NMR

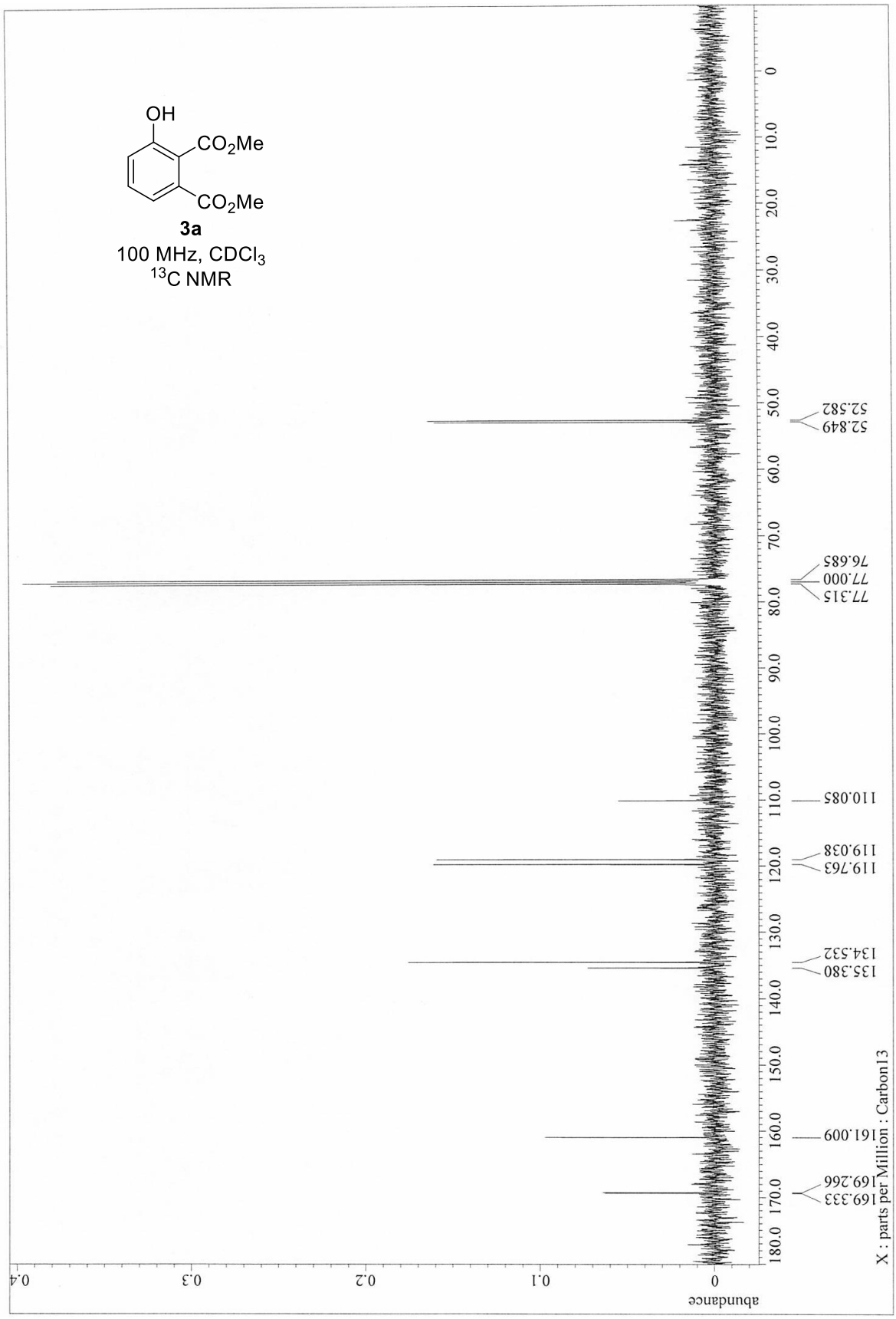


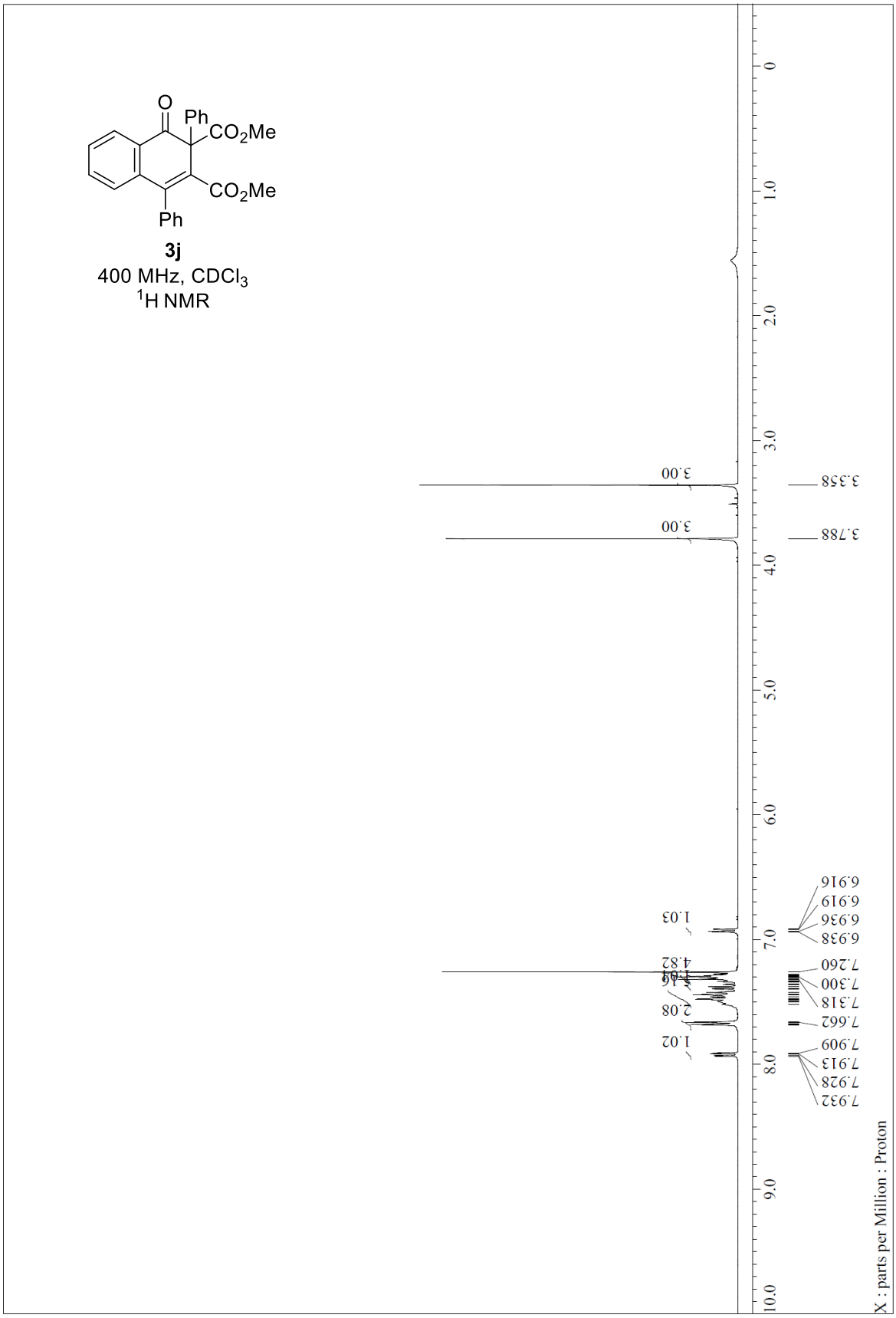

CF3
2i
100 MHz, CDCl3
13C NMR
ppm
0
20
40
60
80
100
120
140
160
76.681
77.000
77.318
123.349
124.146
124.237
124.360
124.594
124.647
124.708
124.768
126.066
126.582
127.613
128.736
128.949
132.727
133.865

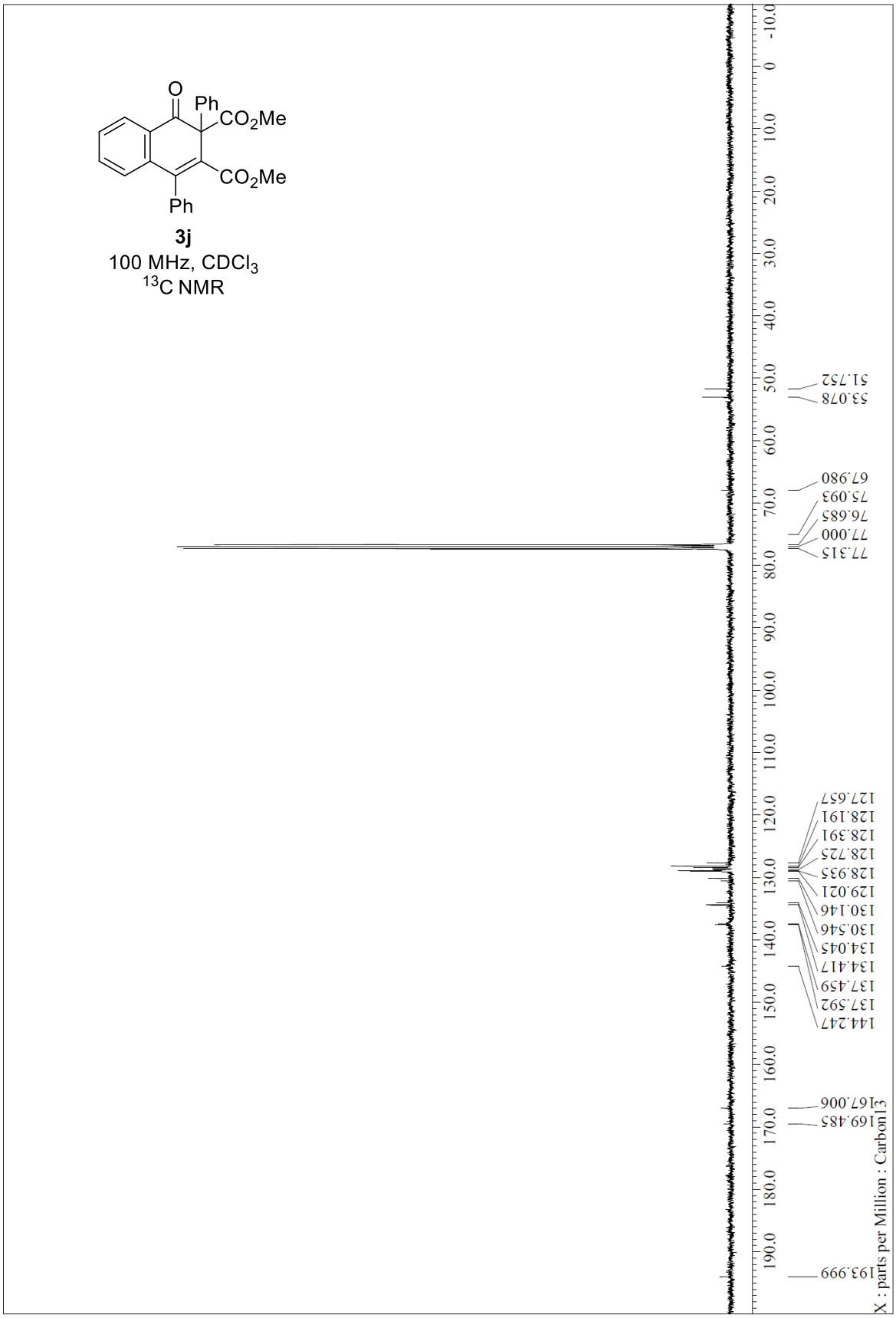

Ph
CO2Me
CO2Me
Ph
2j
400 MHz, CDCl3
1H NMR
1.554
3.512
3.02
2.00
4.09
1.01
7.260
7.380
7.385
7.399
7.404
7.457
7.465
7.474
7.479
7.489
7.496
7.649
7.657
7.666
7.674
X : parts per Million : Proton
abundance

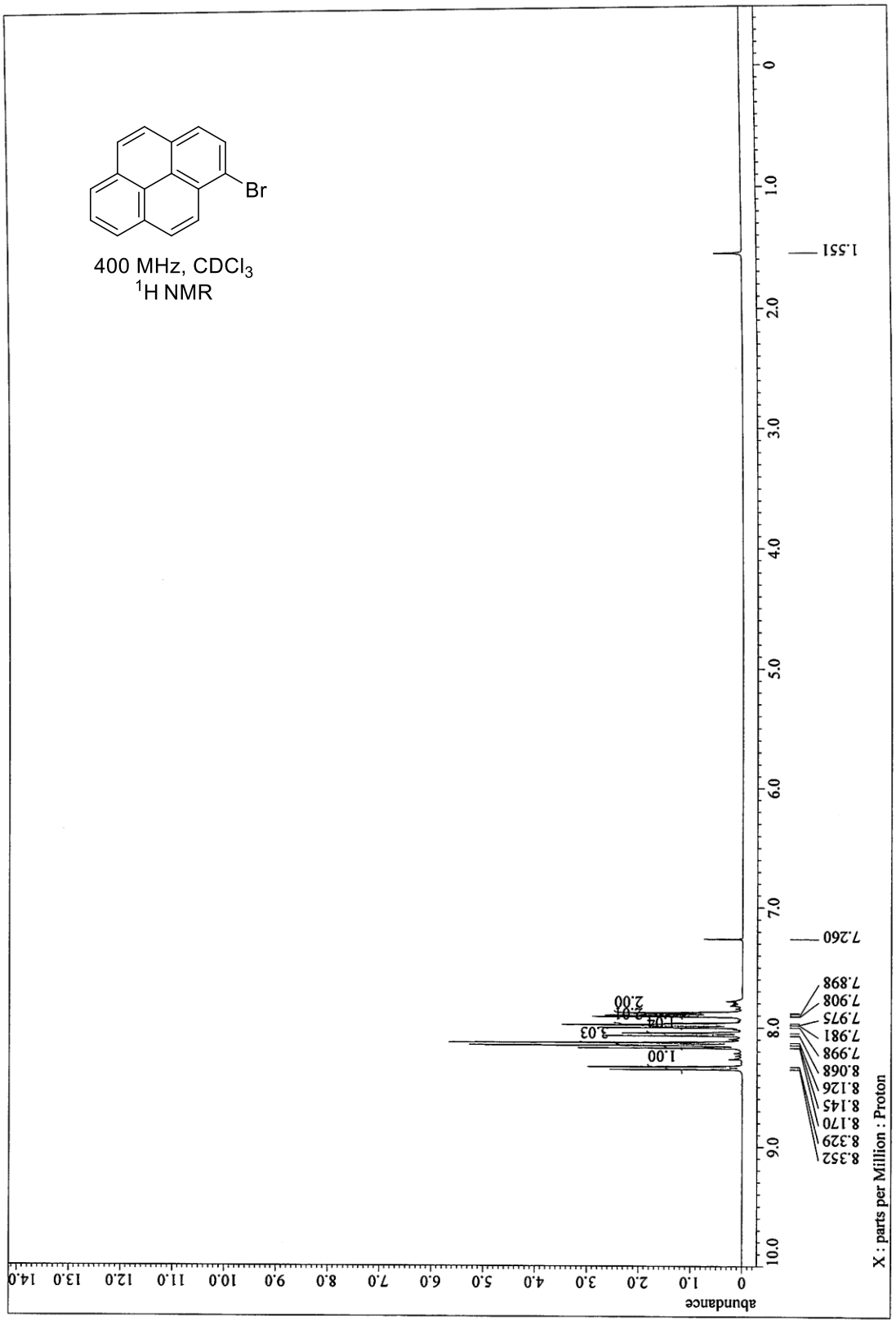

Ph
CO2Me
CO2Me
Ph
2j
100 MHz, CDCl3
13C NMR
52.162
76.857
77.000
77.324
127.381
127.648
127.857
128.058
128.716
129.869
132.653
137.526
139.099
168.866
X : parts per Million : Carbon13
abundance

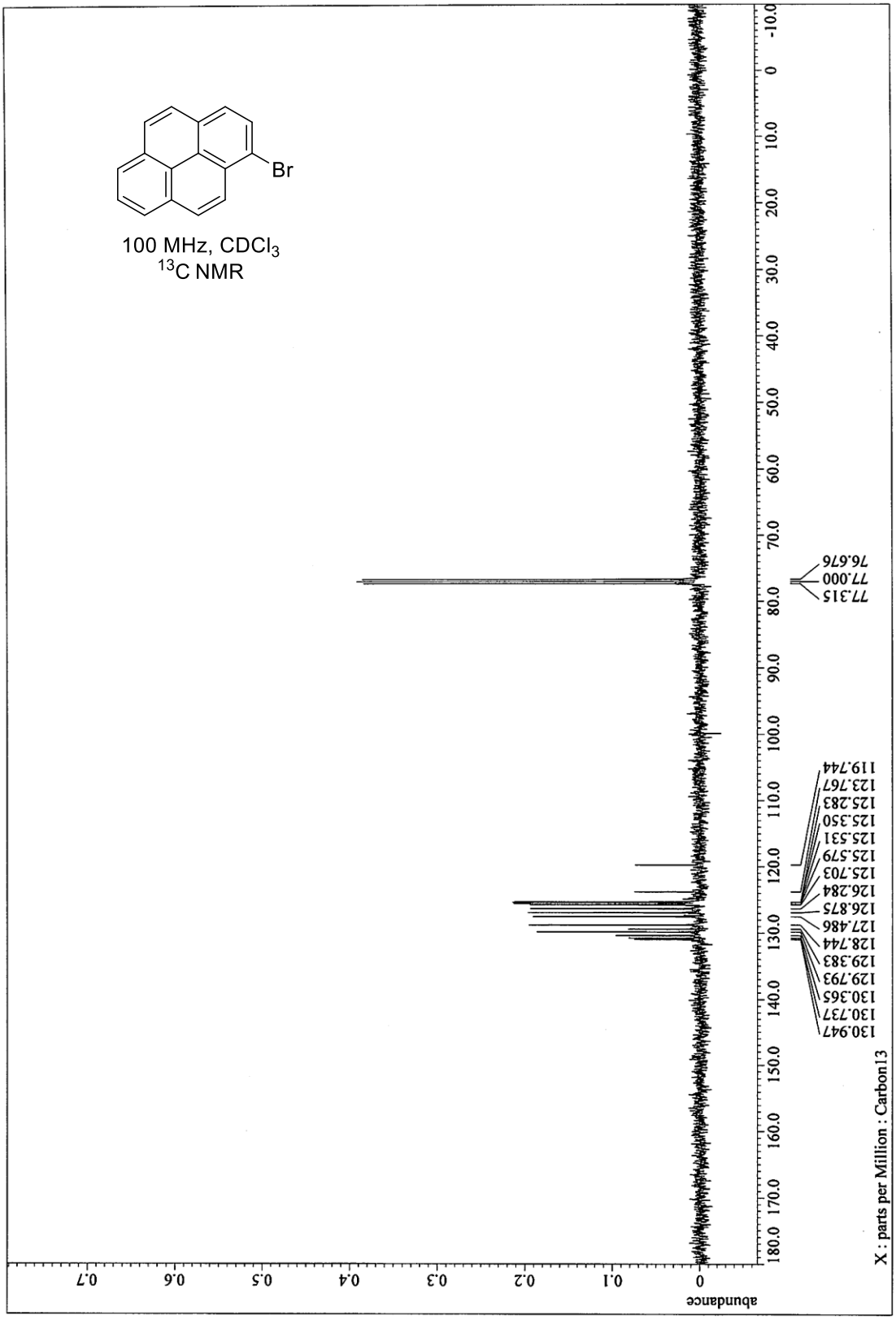

OH
CO2Me
CO2Me
3a
400 MHz, CDCl3
1H NMR
X : parts per Million : Proton
abundance
10.545
7.458
7.439
7.418
7.260
7.080
7.078
7.060
7.056
6.960
6.958
6.942
6.939
5.97
3.908
3.875
1.05
1.22
1.00
0.99

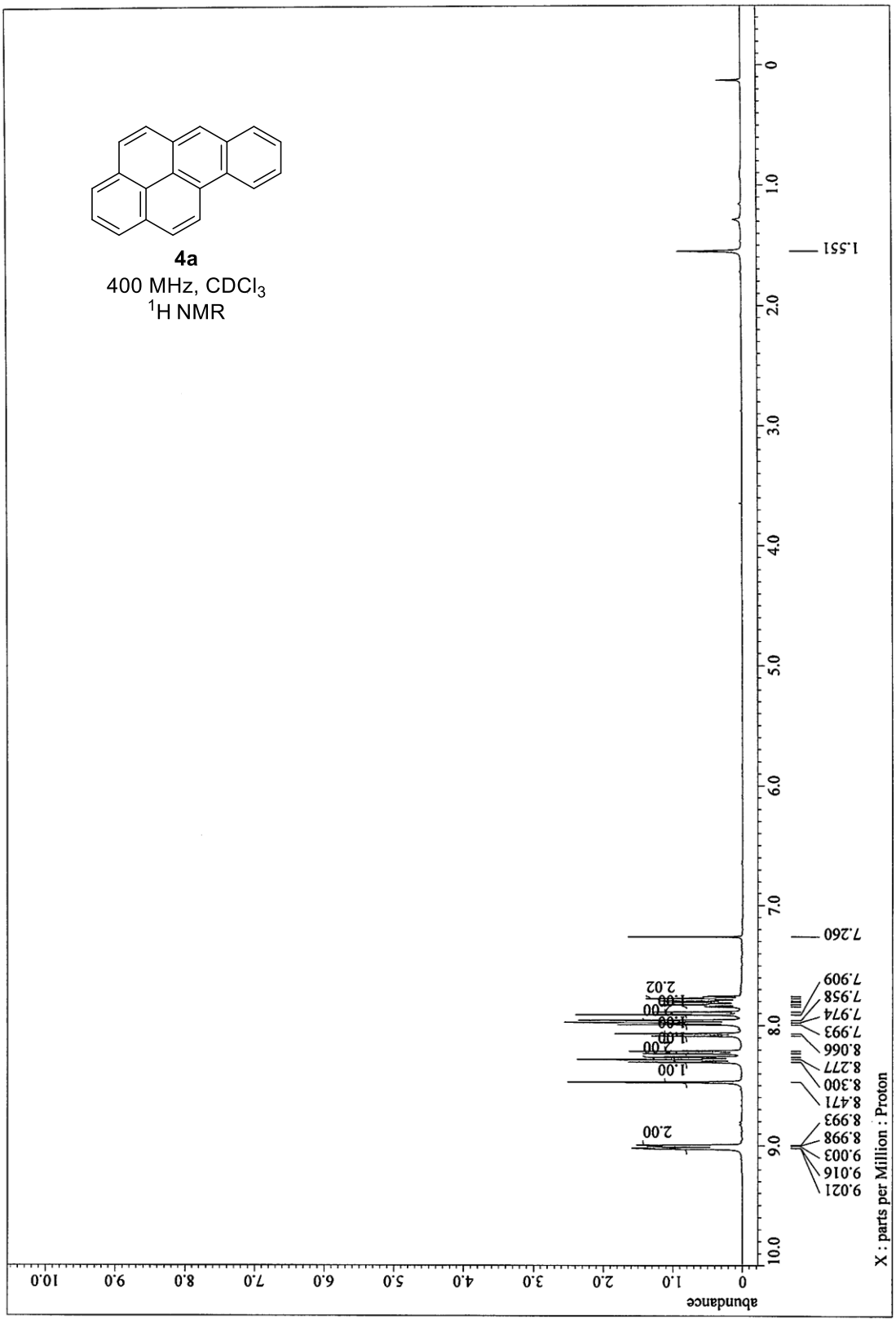

OH
CO2Me
CO2Me
3a
100 MHz, CDCl3
13C NMR
52.849
52.582
77.315
77.000
76.685
110.085
119.763
119.038
135.380
134.532
161.009
169.333
169.266
X : parts per Million : Carbon13
abundance

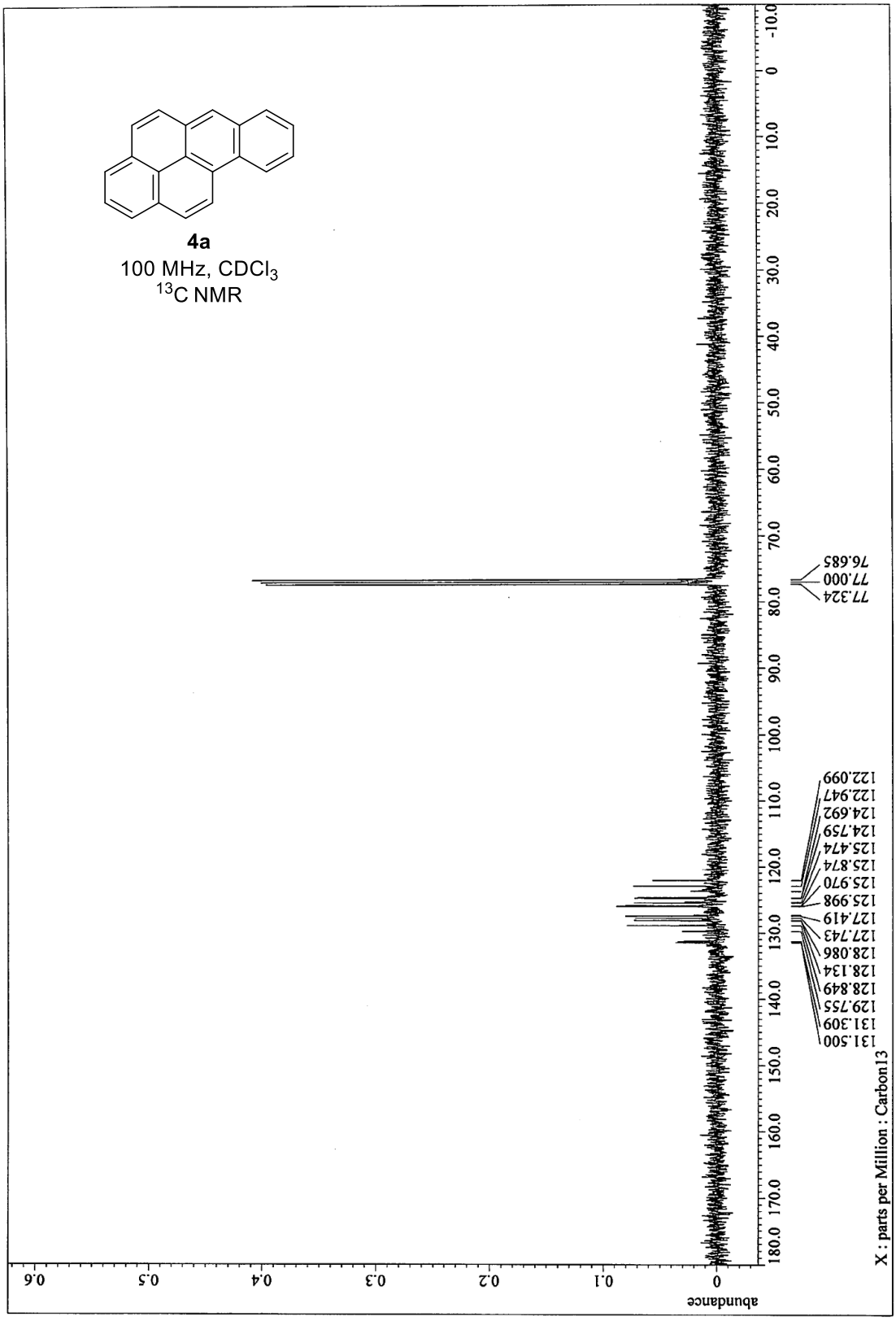

O
Ph
CO2Me
CO2Me
Ph
3j
400 MHz, CDCl3
1H NMR
3.00
3.00
1.03
4.82
2.08
1.02
3.358
3.788
6.916
6.919
6.936
6.938
7.260
7.300
7.318
7.662
7.909
7.913
7.928
7.932
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
X : parts per Million : Proton

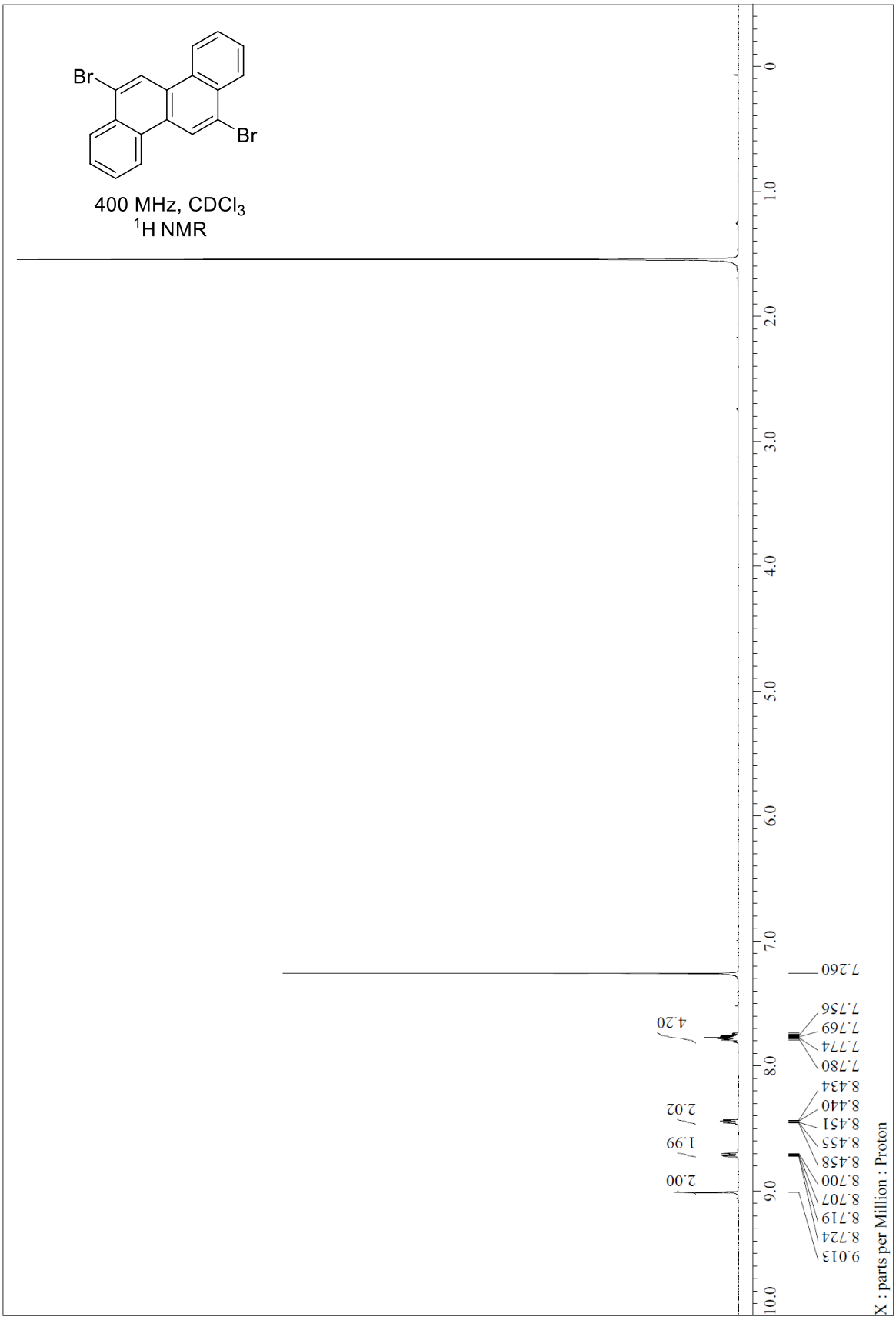

O
Ph
CO2Me
CO2Me
Ph
3j
100 MHz, CDCl3
13C NMR
X : parts per Million : Carbon13

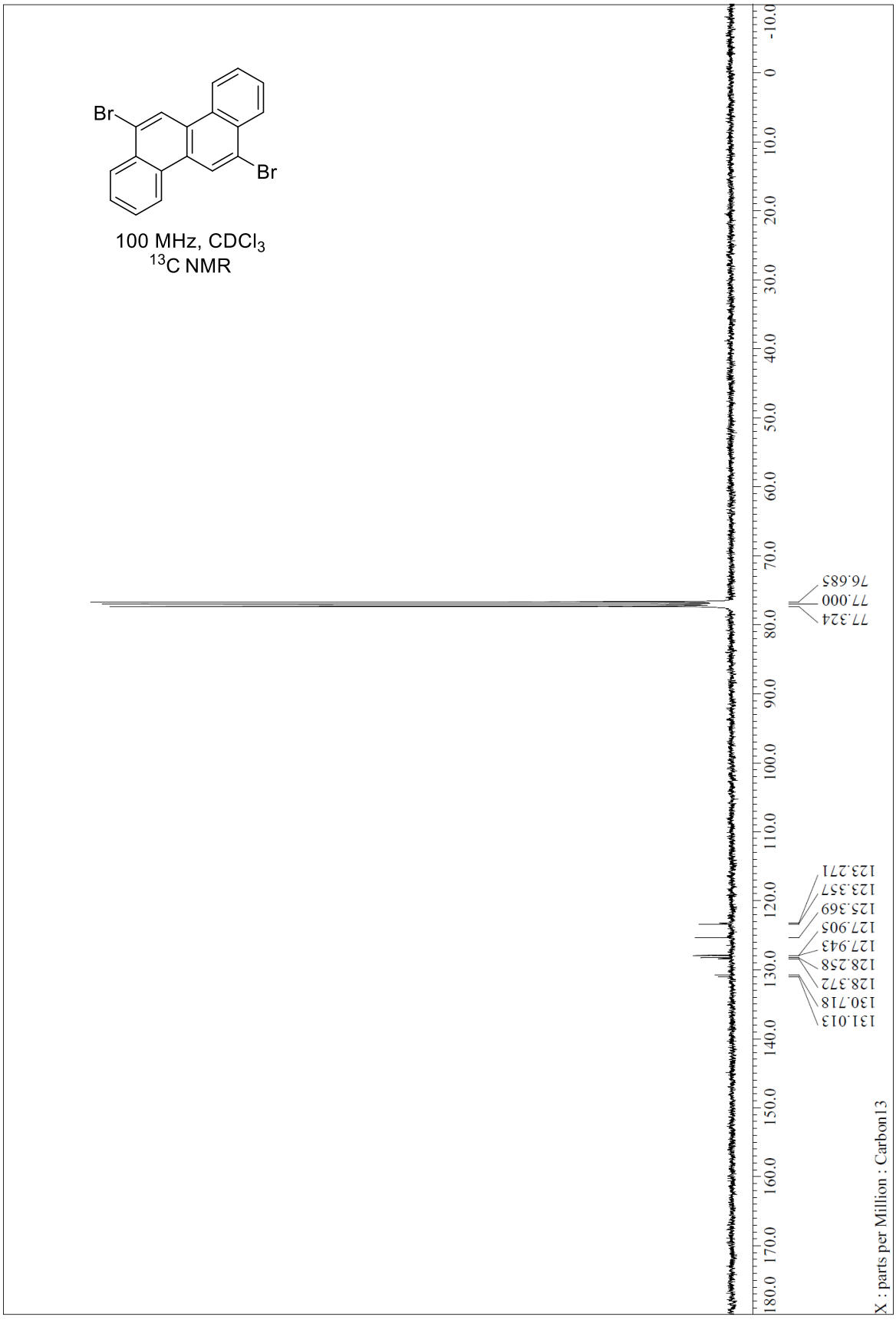

Br
400 MHz, CDCl3
1H NMR
1.551
7.260
8.352
8.329
8.170
8.145
8.126
8.068
7.998
7.981
7.975
7.908
7.898
1.00
3.03
1.04
2.00
X : parts per Million : Proton
abundance

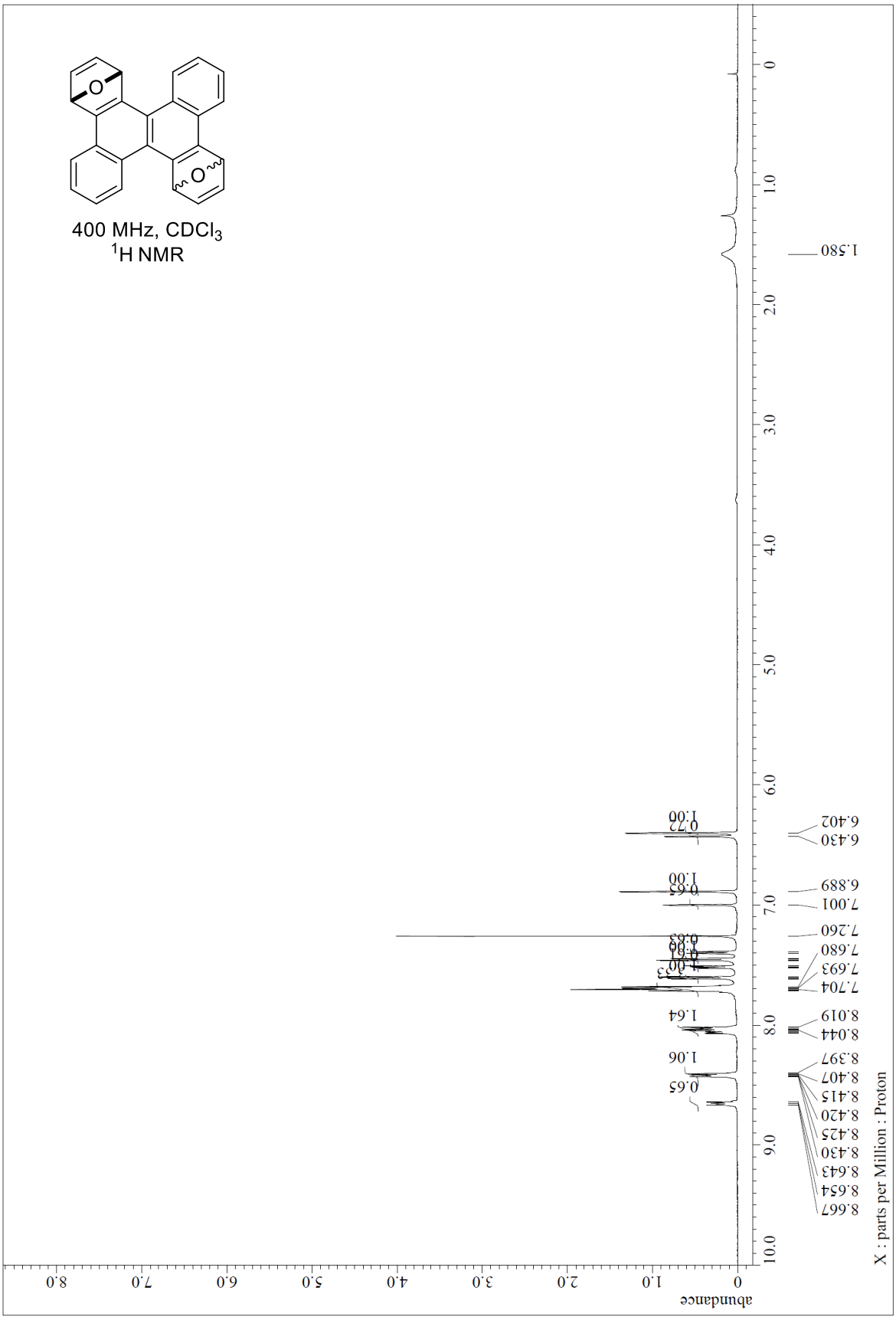

Br
100 MHz, CDCl3
13C NMR
abundance
X : parts per Million : Carbon13
77.315
77.000
76.676
130.947
130.737
130.365
129.793
129.383
128.744
127.486
126.875
126.284
125.703
125.579
125.531
125.350
125.283
123.767
119.744

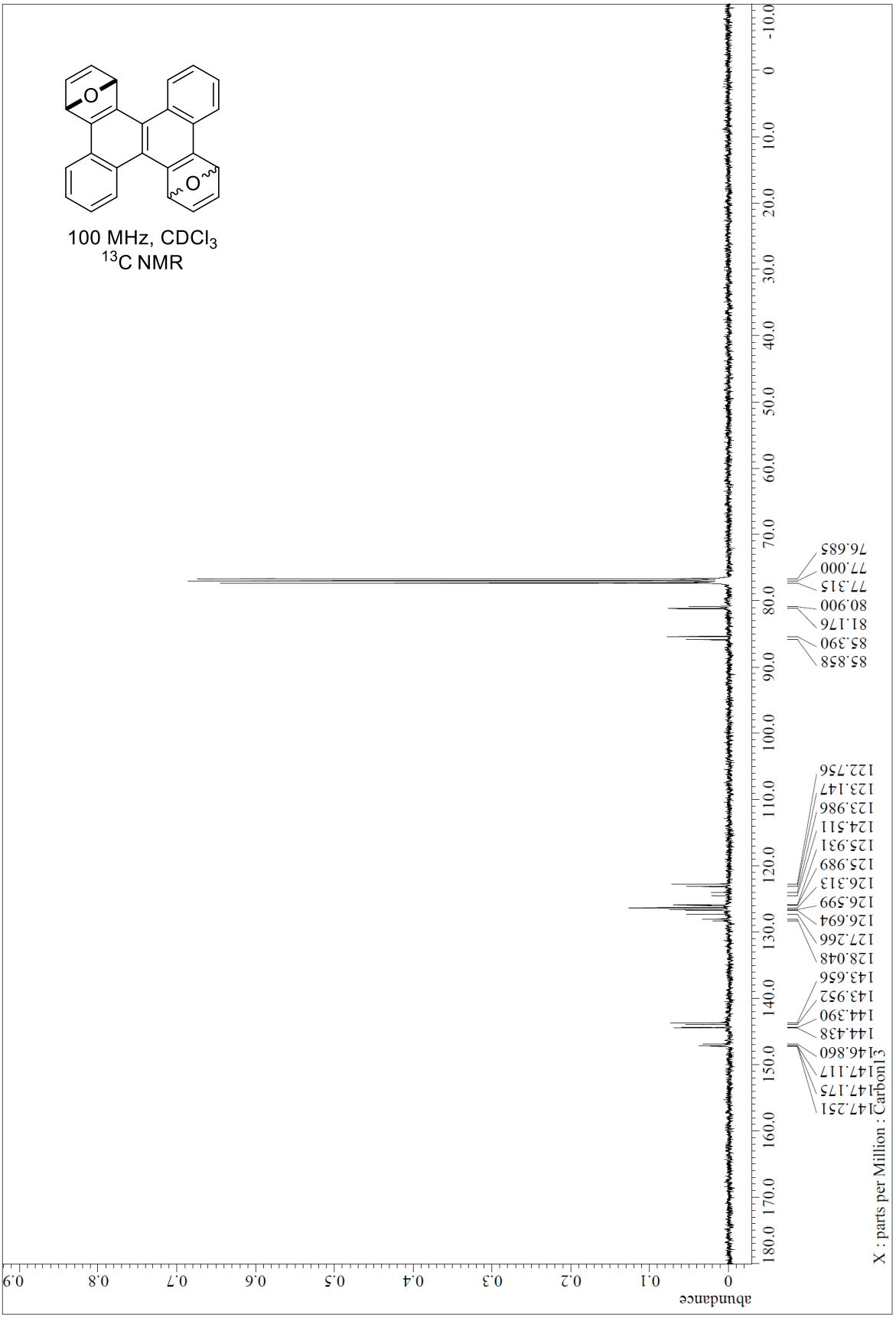

4a
400 MHz, CDCl3
1H NMR
1.551
7.260
7.909
7.958
7.974
7.993
8.066
8.277
8.300
8.471
8.993
8.998
9.003
9.016
9.021
2.00
1.00
1.00
2.00
1.00
1.00
2.02
X : parts per Million : Proton
abundance

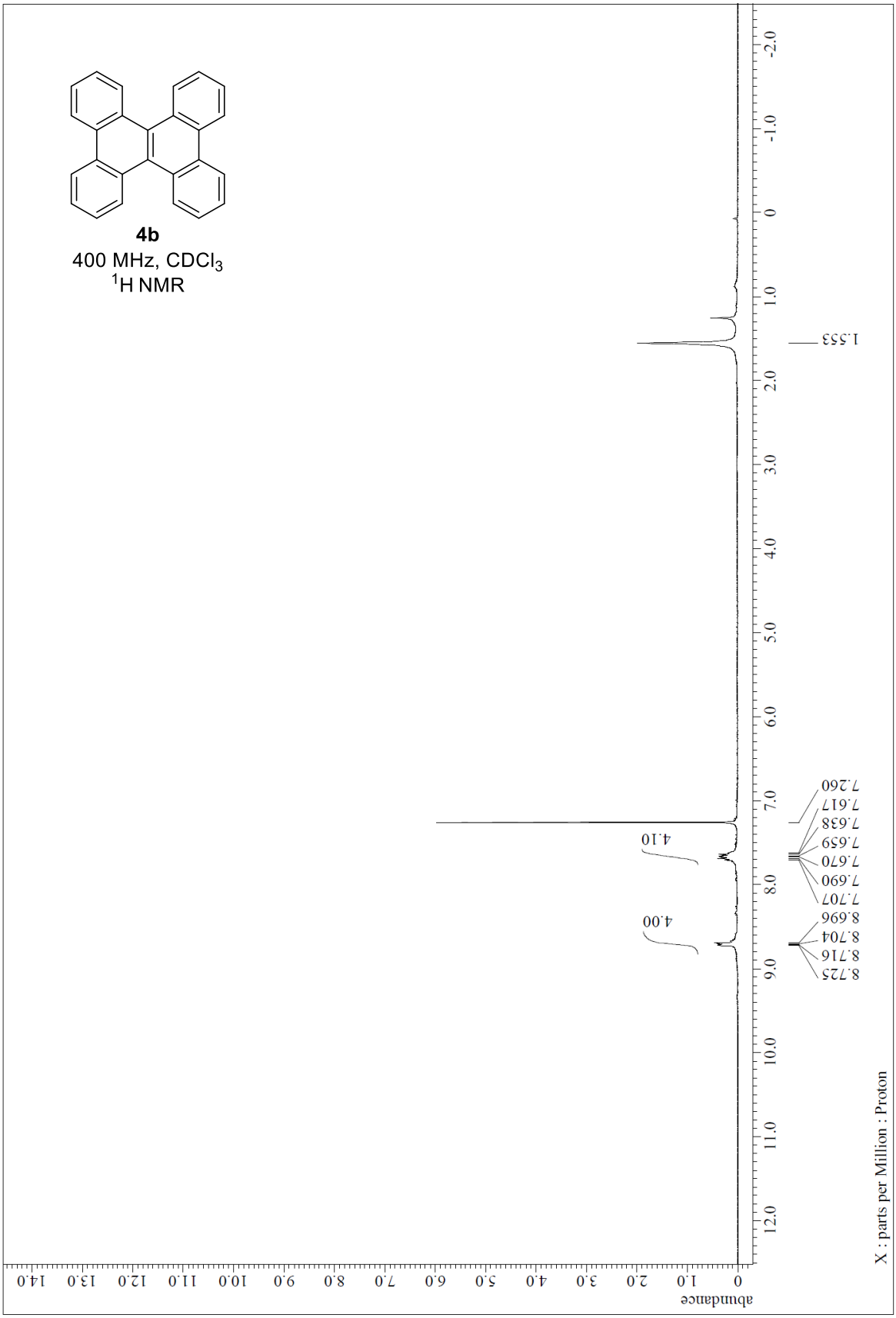

4a
100 MHz, CDCl3
13C NMR
abundance
X : parts per Million : Carbon13
131.500
131.309
129.755
128.849
128.134
128.086
127.743
127.419
125.998
125.970
125.874
125.474
124.759
124.692
122.947
122.099
77.324
77.000
76.685

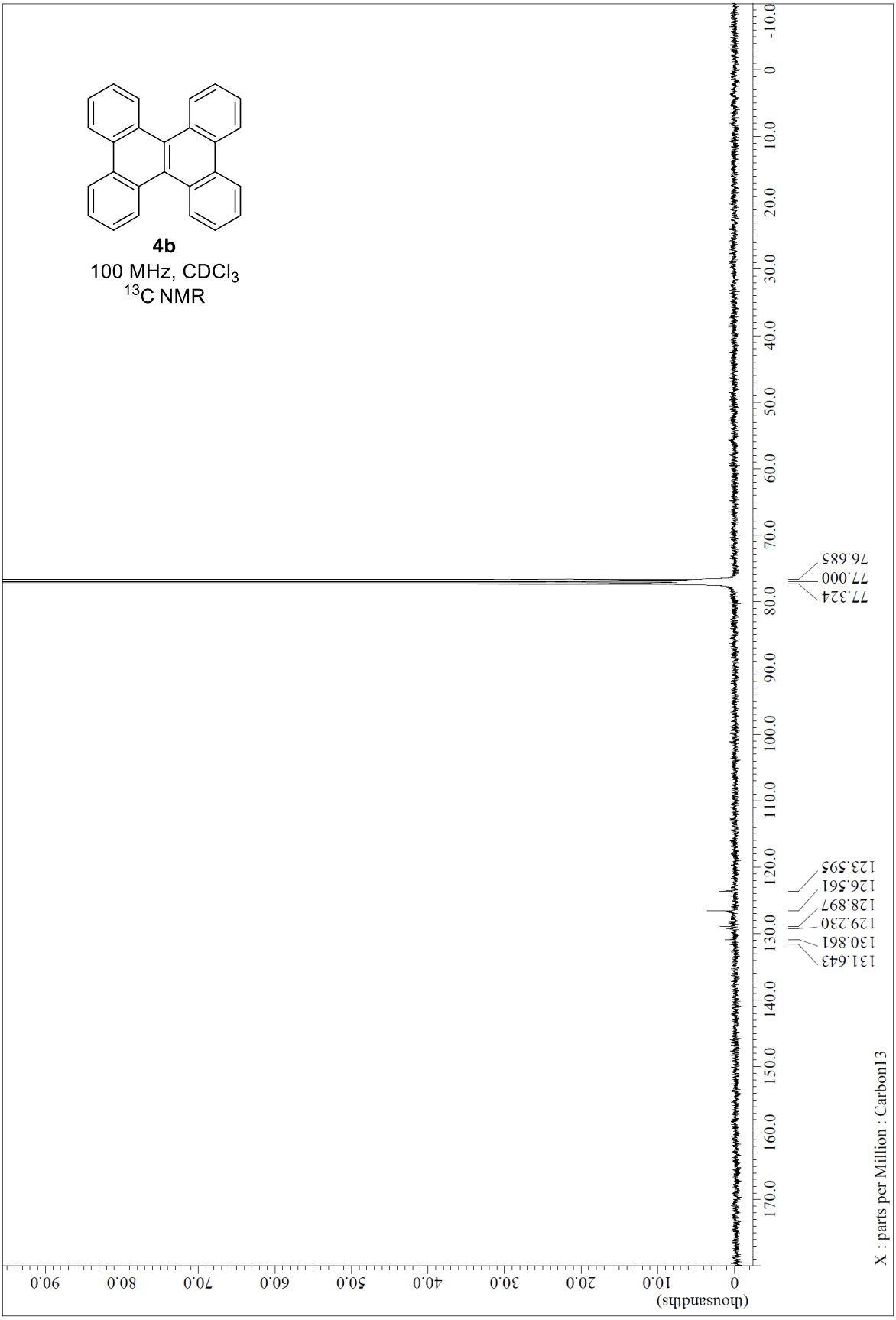

Br
Br
400 MHz, CDCl3
1H NMR
9.013
8.724
8.719
8.707
8.700
8.458
8.455
8.451
8.440
8.434
7.780
7.774
7.769
7.756
7.260
4.20
2.02
1.99
2.00
X : parts per Million : Proton

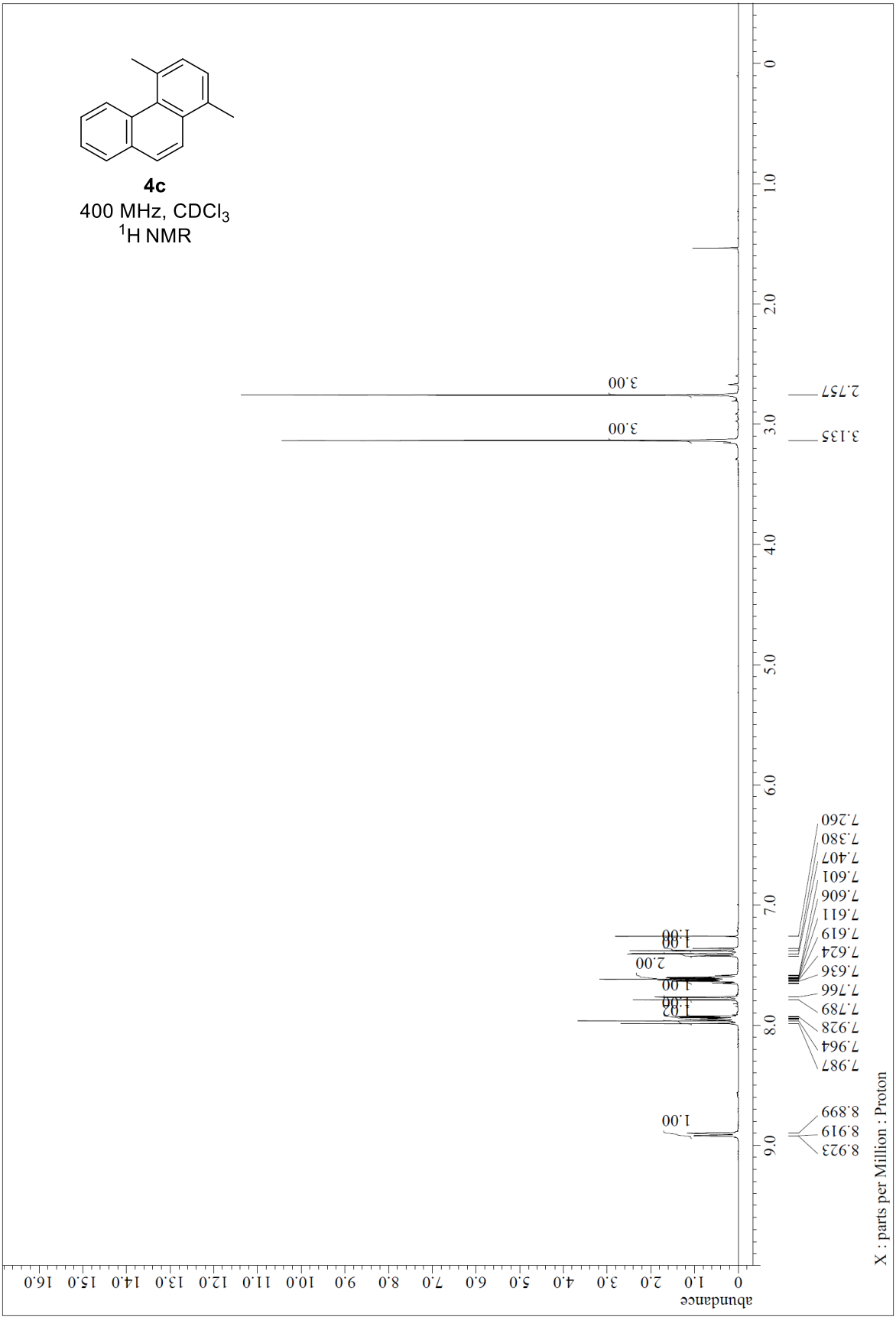

Br
Br
100 MHz, CDCl3
13C NMR
-10.0
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
X : parts per Million : Carbon13
76.685
77.000
77.324
123.271
123.357
123.369
127.905
127.943
128.258
128.372
130.718
131.013

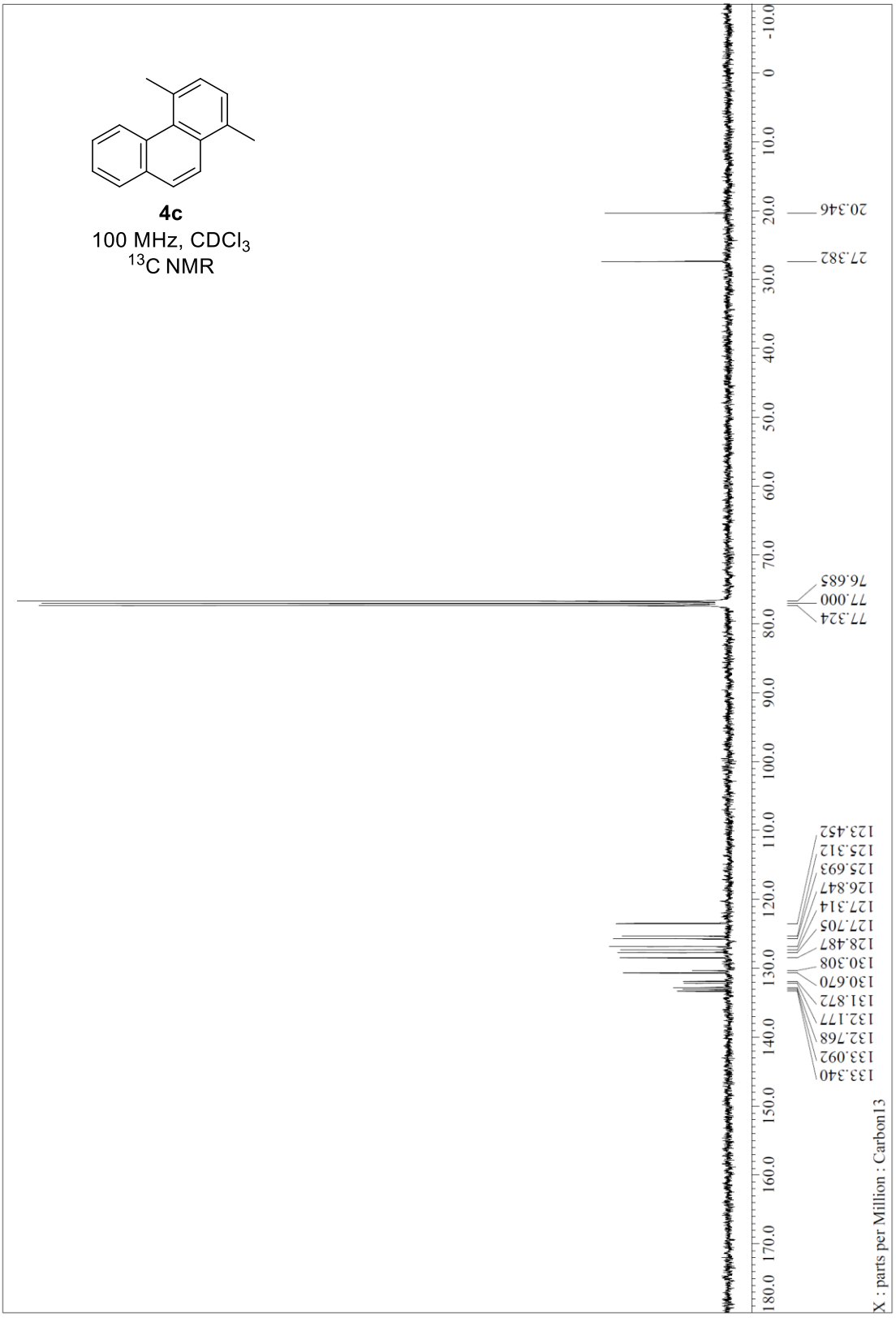

400 MHz, CDCl3
1H NMR
O
O
1.580
6.402
6.430
6.889
7.001
7.260
7.680
7.693
7.704
8.019
8.044
8.397
8.407
8.415
8.420
8.425
8.430
8.643
8.654
8.667
0.65
1.06
1.64
0.72
1.00
0.85
1.00
X : parts per Million : Proton
abundance

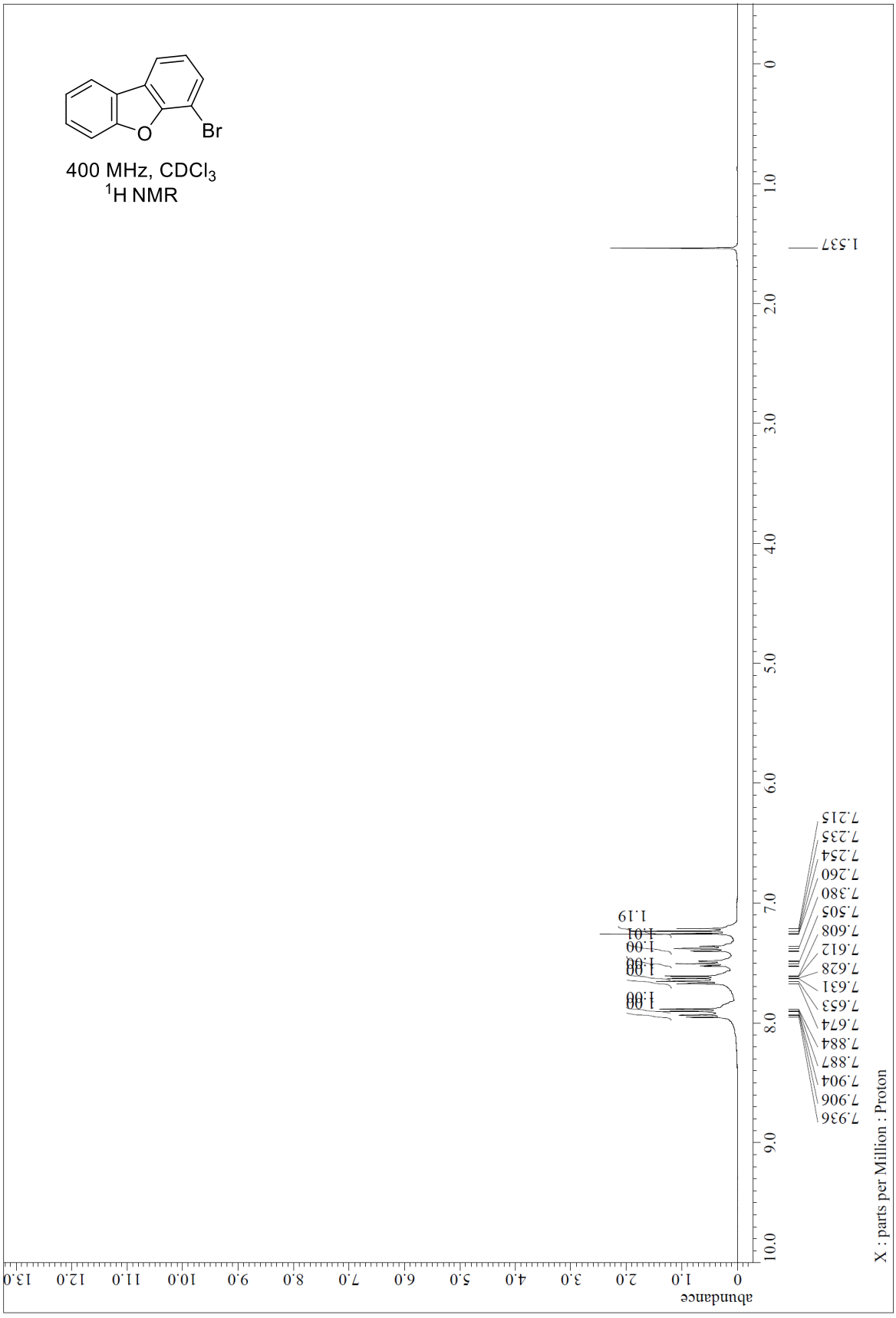

100 MHz, CDCl3
13C NMR
O
O
X : parts per Million : Carbon13
abundance
85.858
85.390
81.176
80.900
77.315
77.000
76.685
147.251
147.175
147.117
146.860
144.438
144.390
143.952
143.656
128.048
127.266
126.694
126.599
126.313
125.989
125.931
124.511
123.986
123.147
122.756

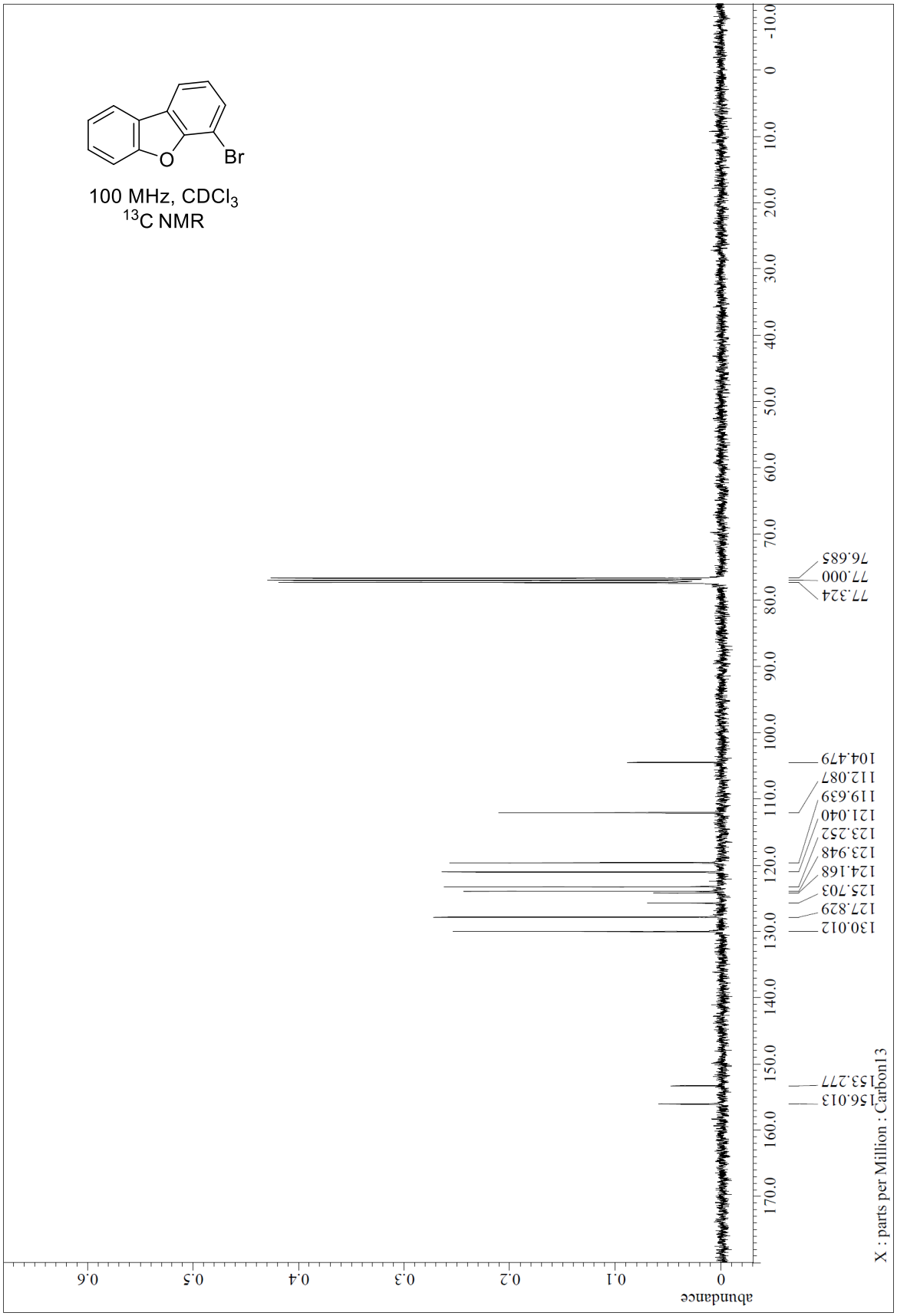

4b
400 MHz, CDCl3
1H NMR
abundance
X : parts per Million : Proton
1.553
4.10
4.00
8.725
8.716
8.704
8.696
7.707
7.690
7.670
7.659
7.638
7.617
7.260

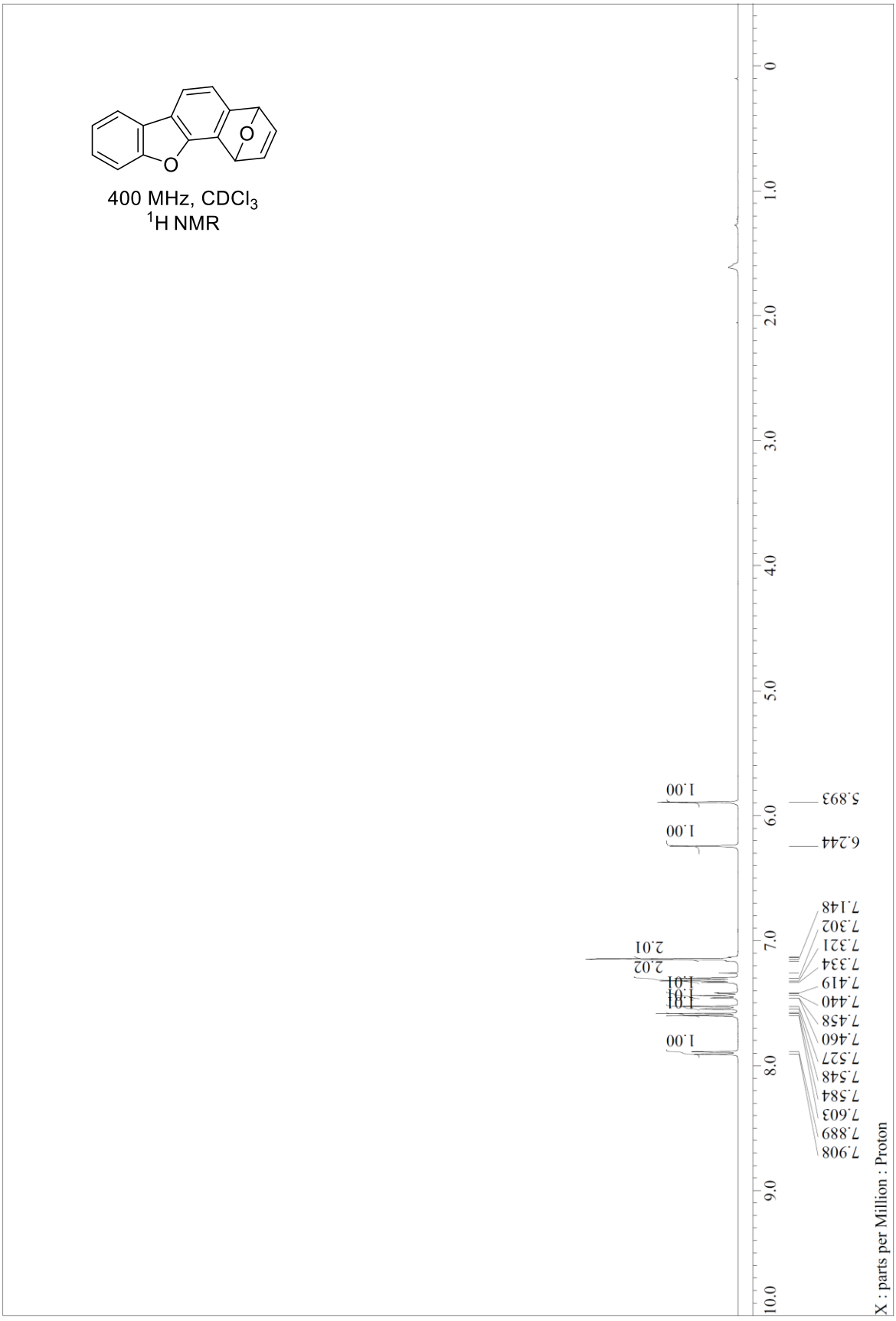

4b
100 MHz, CDCl3
13C NMR
77.324
77.000
76.685
131.643
130.861
129.230
128.897
126.561
123.595
X : parts per Million : Carbon13
(thousandths)

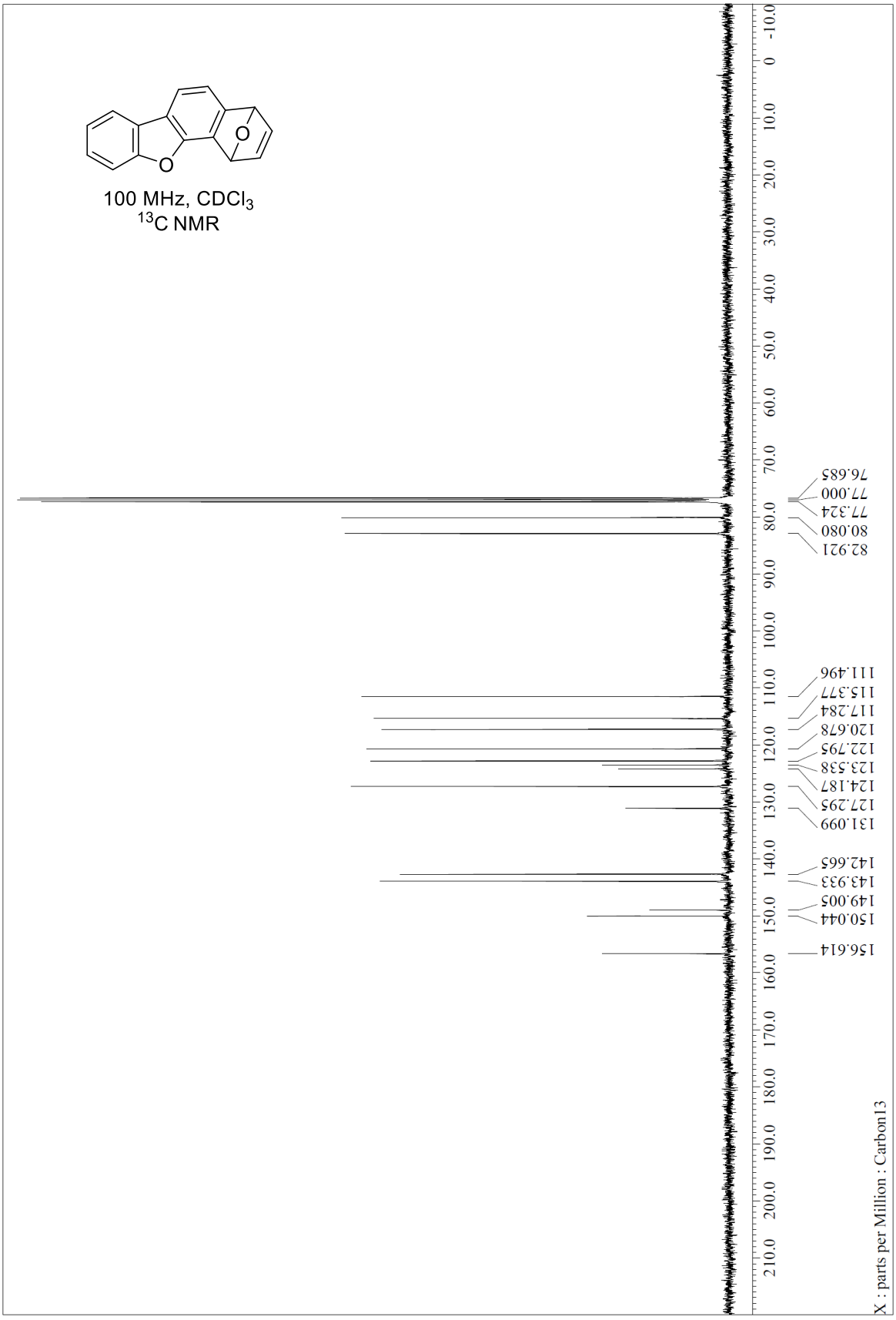

4c
400 MHz, CDCl3
1H NMR
abundance
X : parts per Million : Proton
2.757
3.135
8.923
8.919
8.899
7.987
7.964
7.928
7.789
7.766
7.636
7.624
7.619
7.611
7.606
7.601
7.407
7.380
7.260
3.00
3.00
1.00
2.00
1.00
1.02
1.00
1.00

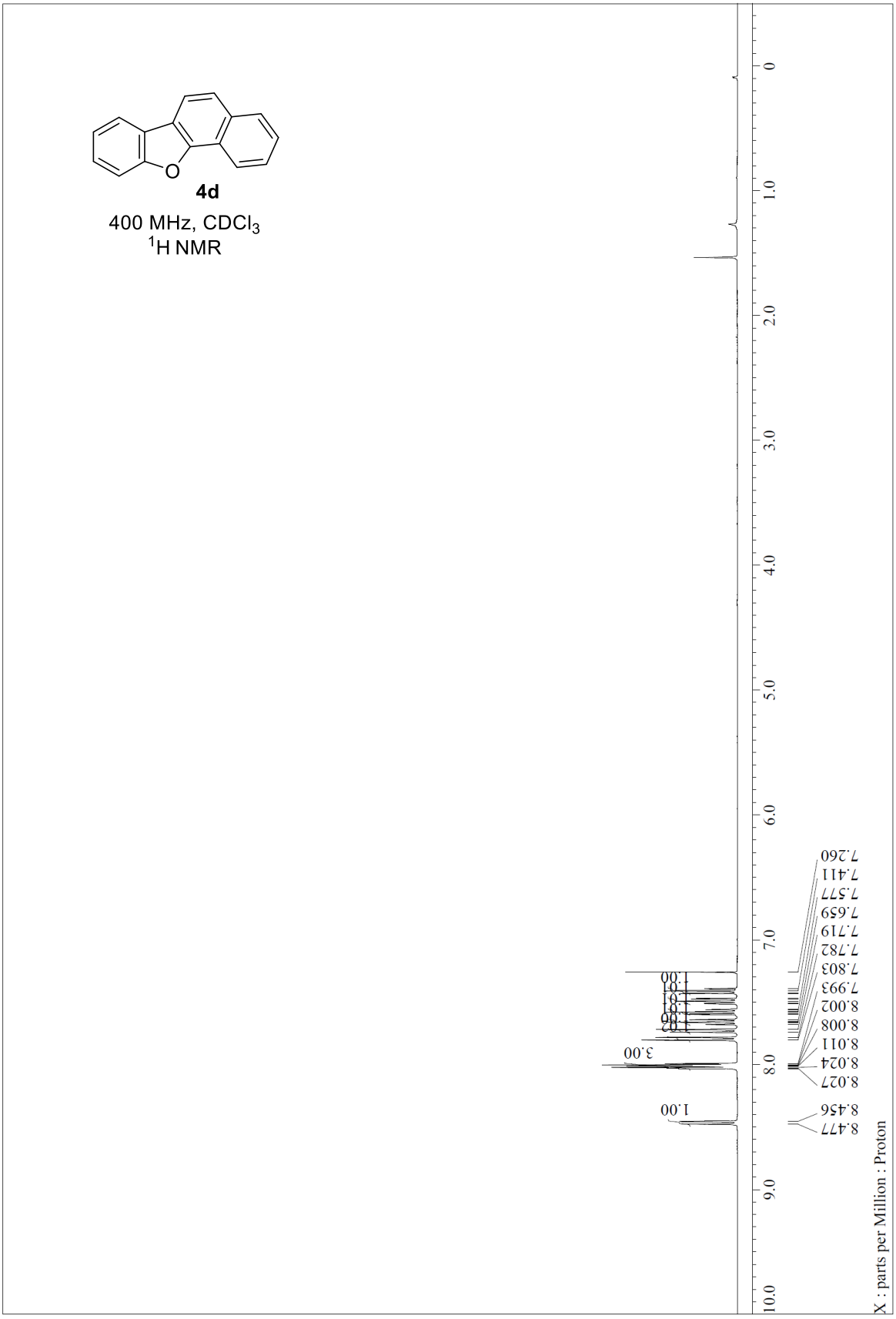

4c
100 MHz, CDCl3
13C NMR
20.346
27.382
76.685
77.000
77.324
123.452
125.312
125.693
126.847
127.314
127.705
128.487
130.308
130.670
131.872
132.177
132.768
133.092
133.340
X : parts per Million : Carbon13

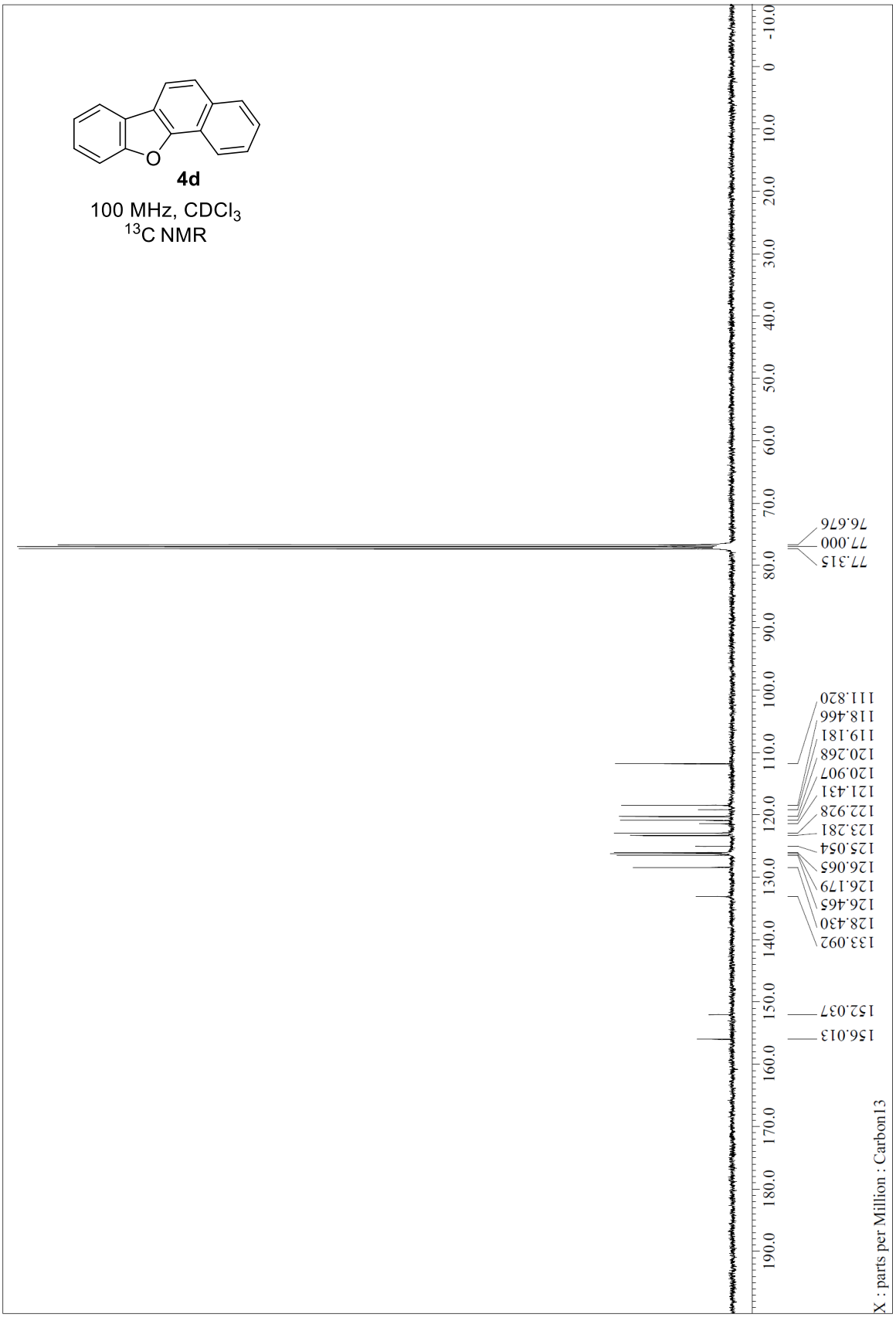

Br
O
400 MHz, CDCl3
1H NMR
1.537
1.19
1.01
1.00
1.00
1.00
1.00
7.936
7.906
7.904
7.887
7.884
7.674
7.653
7.631
7.628
7.612
7.608
7.505
7.380
7.260
7.254
7.235
7.215
X : parts per Million : Proton
abundance

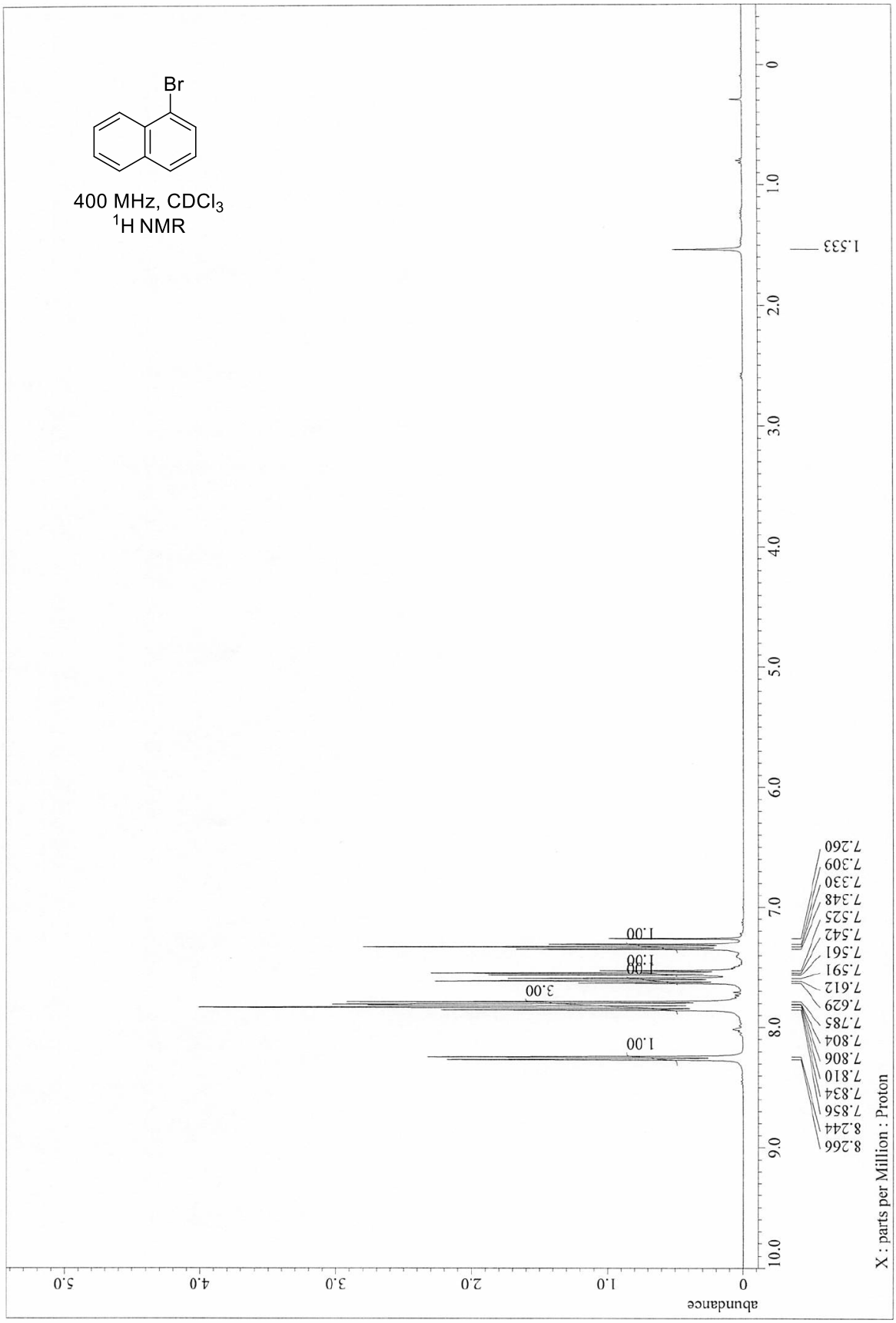

Br
O
100 MHz, CDCl3
13C NMR
abundance
X : parts per Million : Carbon13
153.277
156.013
130.012
127.829
125.703
124.168
123.948
123.252
121.040
119.639
112.087
104.479
77.324
77.000
76.685

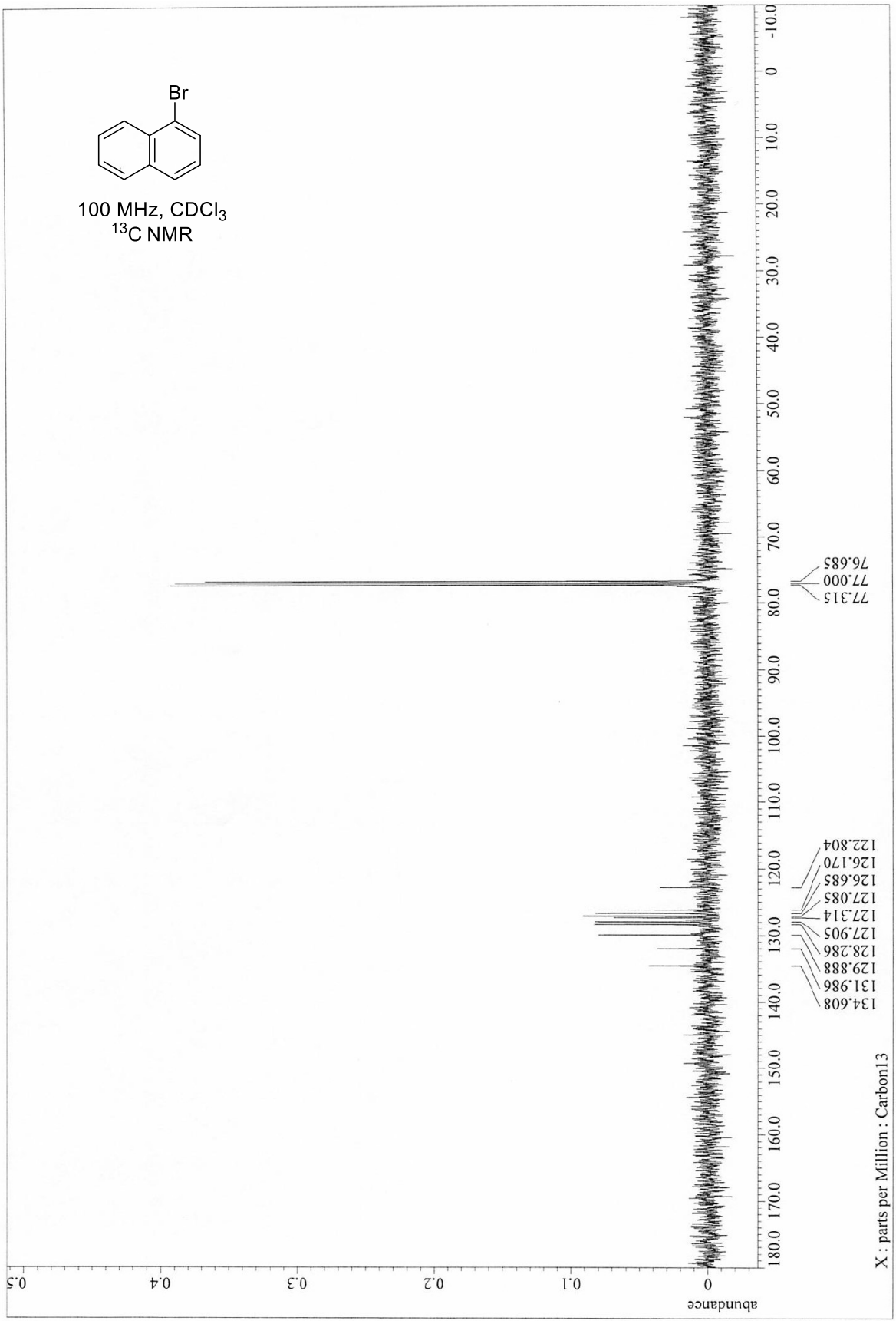

400 MHz, CDCl3
1H NMR
1.00
1.00
2.01
2.02
1.01
1.01
1.01
1.00
5.893
6.244
7.148
7.302
7.321
7.334
7.419
7.440
7.458
7.460
7.527
7.548
7.584
7.603
7.889
7.908
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
X : parts per Million : Proton

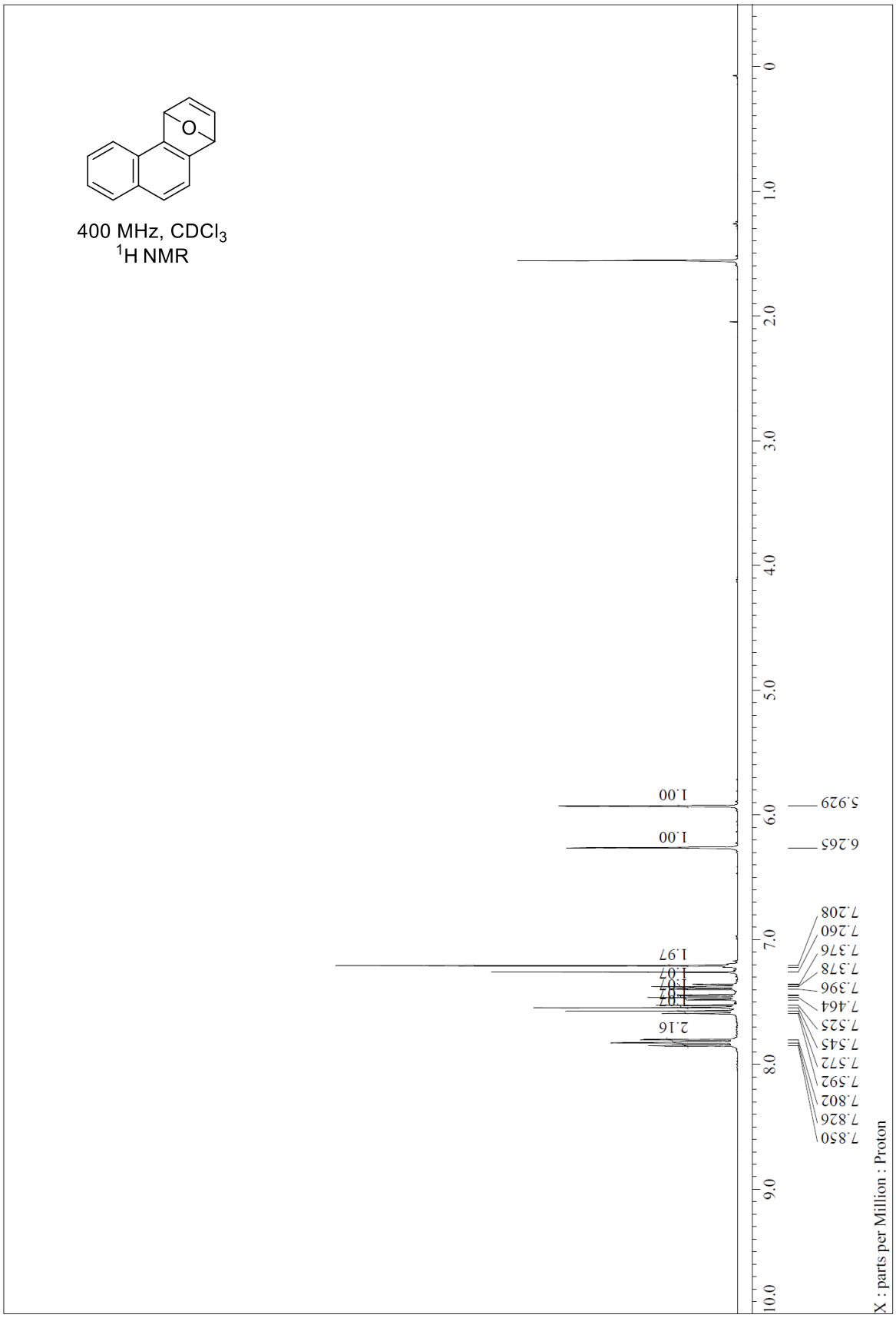

100 MHz, CDCl3
13C NMR
O
O
-10.0
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
210.0
76.685
77.000
77.324
80.080
82.921
111.496
115.377
117.284
120.678
122.795
123.538
124.187
127.295
131.099
142.665
143.933
149.005
150.044
156.614
X : parts per Million : Carbon13

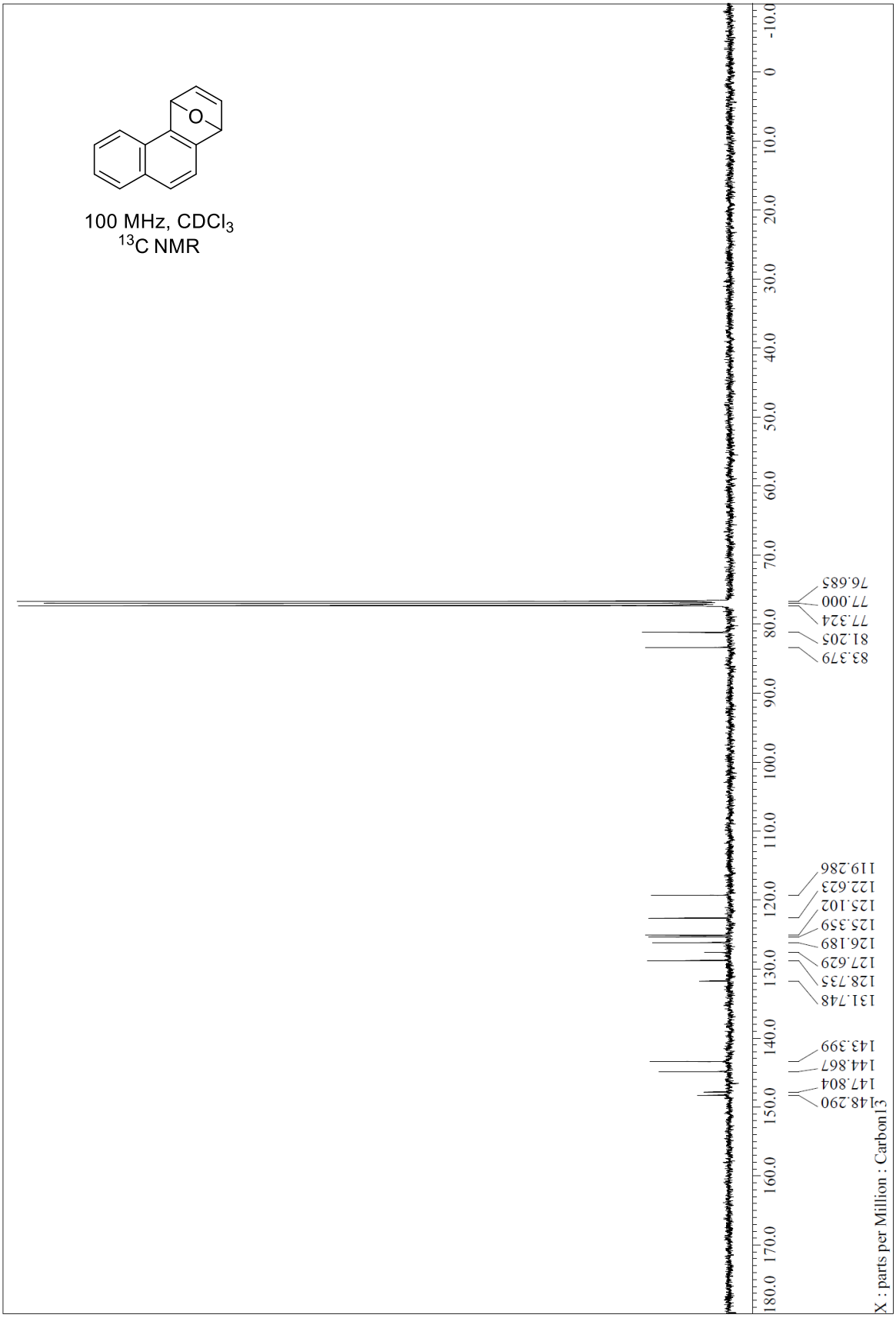

4d
400 MHz, CDCl3
1H NMR
8.477
8.456
8.027
8.024
8.011
8.008
8.002
7.993
7.803
7.782
7.719
7.659
7.577
7.411
7.260
1.00
3.00
X : parts per Million : Proton

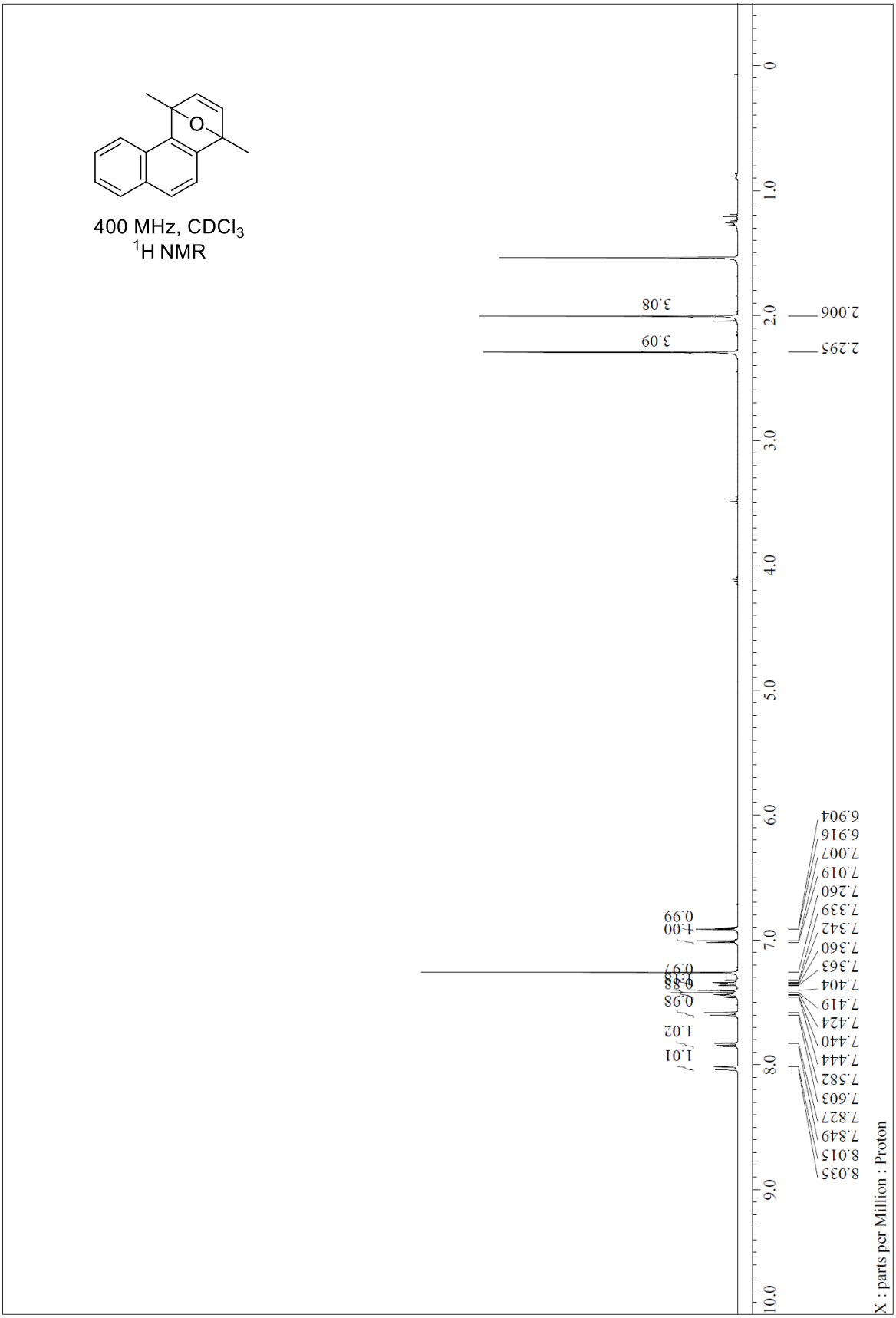

4d
100 MHz, CDCl3
13C NMR
X : parts per Million : Carbon13
156.013
152.037
133.092
128.430
126.465
126.179
126.065
125.054
123.281
122.928
121.431
120.907
120.268
119.181
118.466
111.820
77.315
77.000
76.676

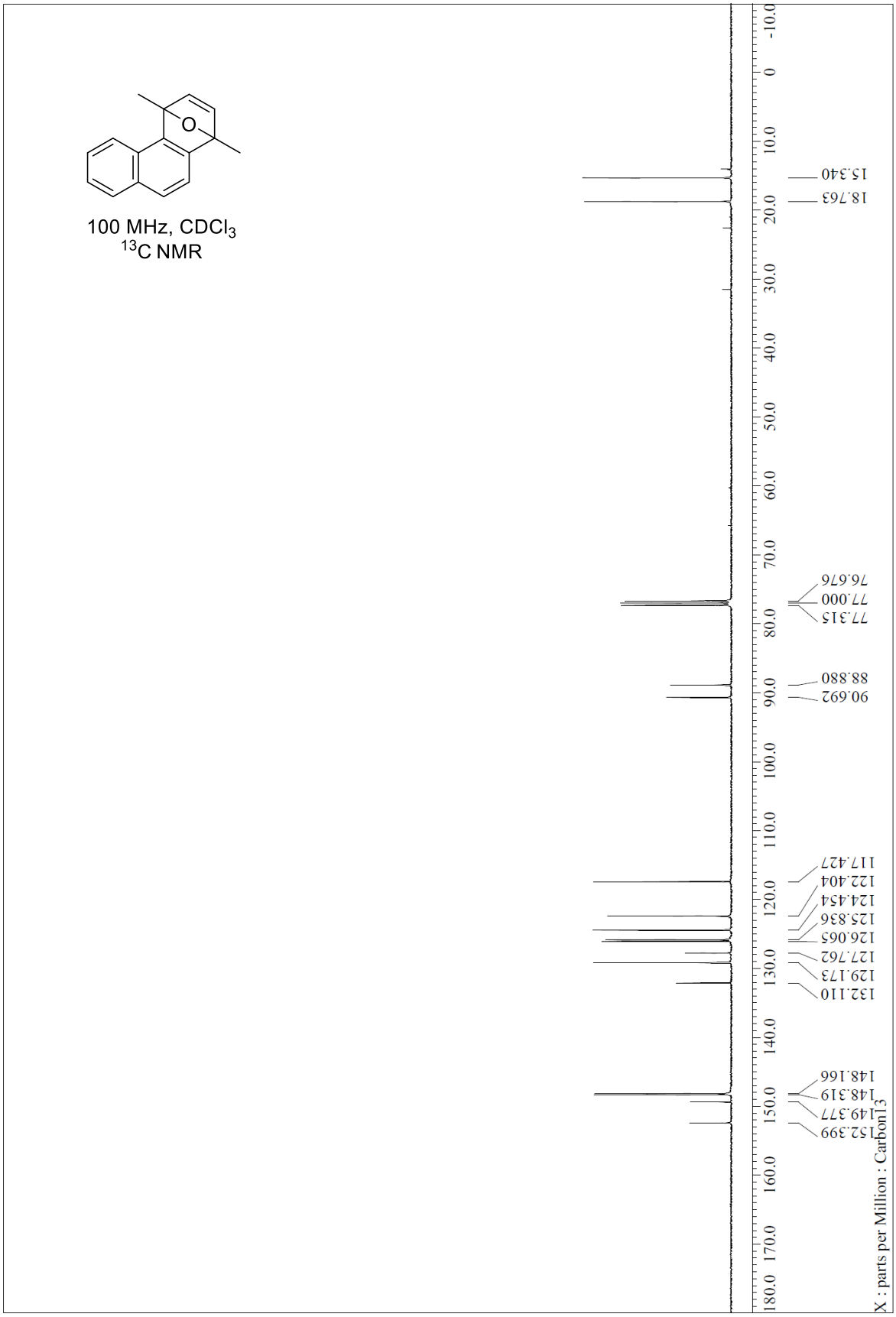

Br
400 MHz, CDCl3
1H NMR
1.533
7.260
7.309
7.330
7.348
7.525
7.542
7.561
7.591
7.612
7.629
7.785
7.804
7.806
7.810
7.834
7.856
8.244
8.266
1.00
3.00
1.00
1.00
1.00
abundance
X : parts per Million : Proton

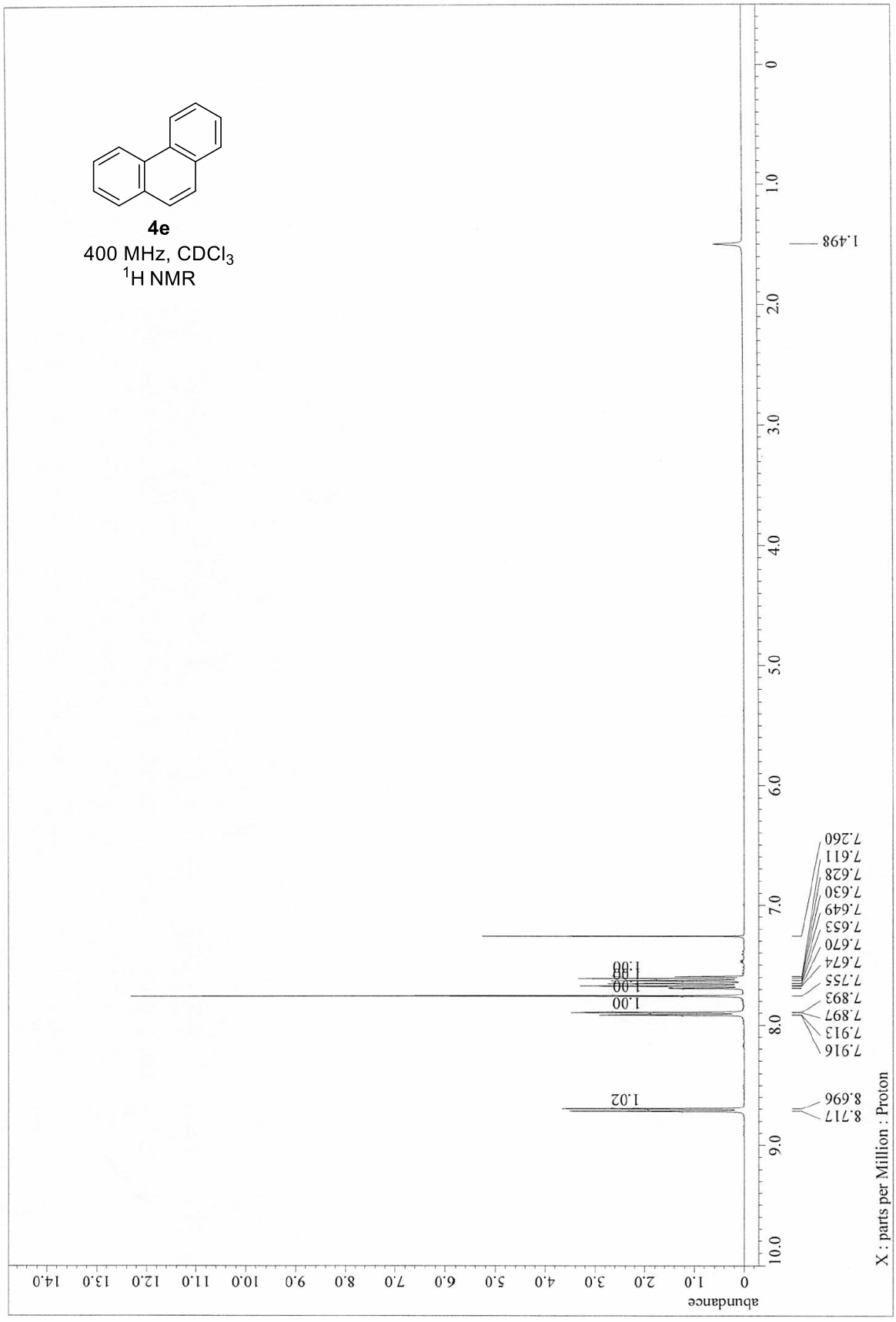

Br
100 MHz, CDCl3
13C NMR
X : parts per Million : Carbon13
abundance
134.608
131.986
129.888
128.286
127.905
127.314
127.085
126.685
126.170
122.804
77.315
77.000
76.685

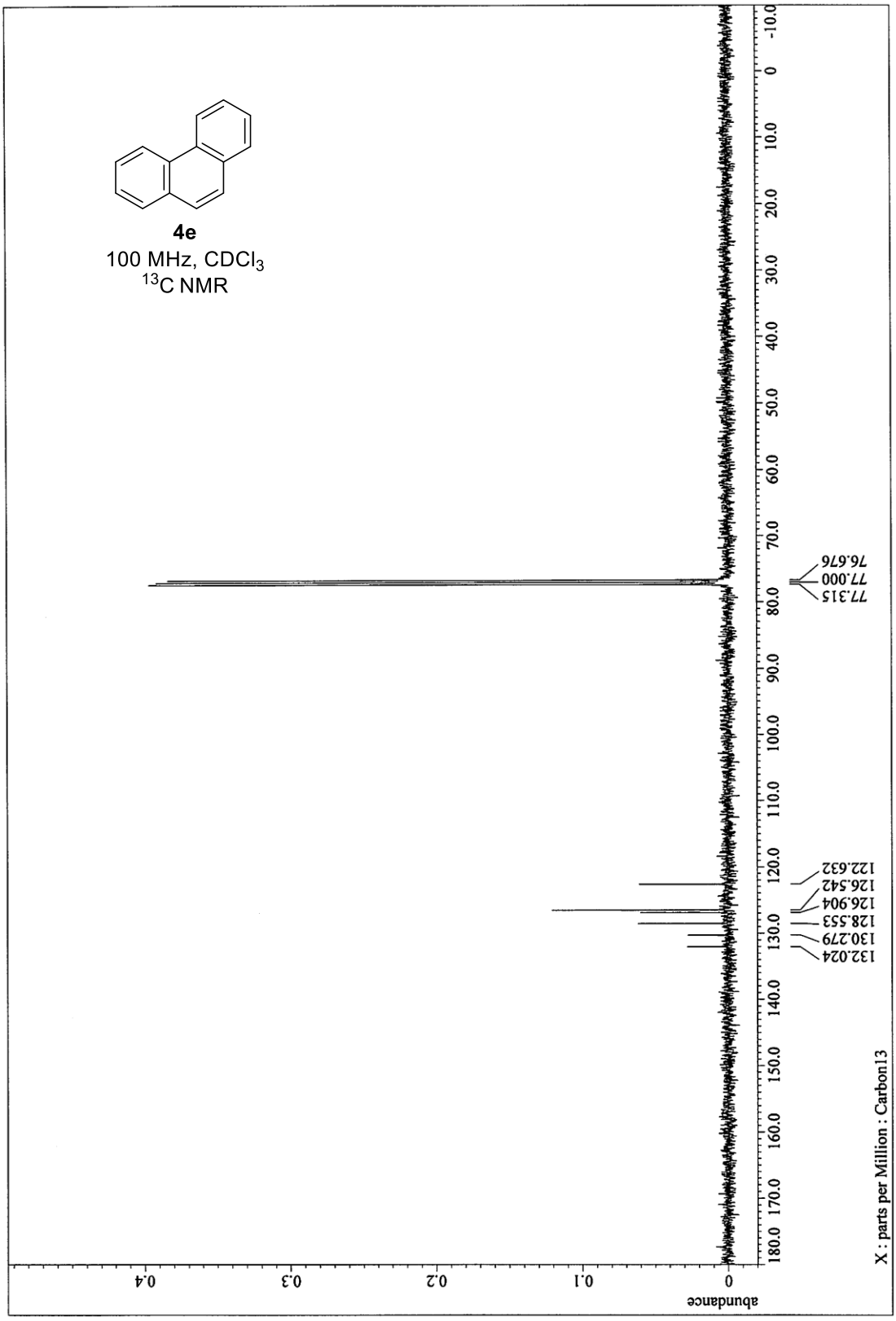

O
400 MHz, CDCl3
1H NMR
1.00
1.00
1.97
2.16
5.929
6.265
7.208
7.260
7.376
7.378
7.396
7.464
7.525
7.545
7.572
7.592
7.802
7.826
7.850
X : parts per Million : Proton

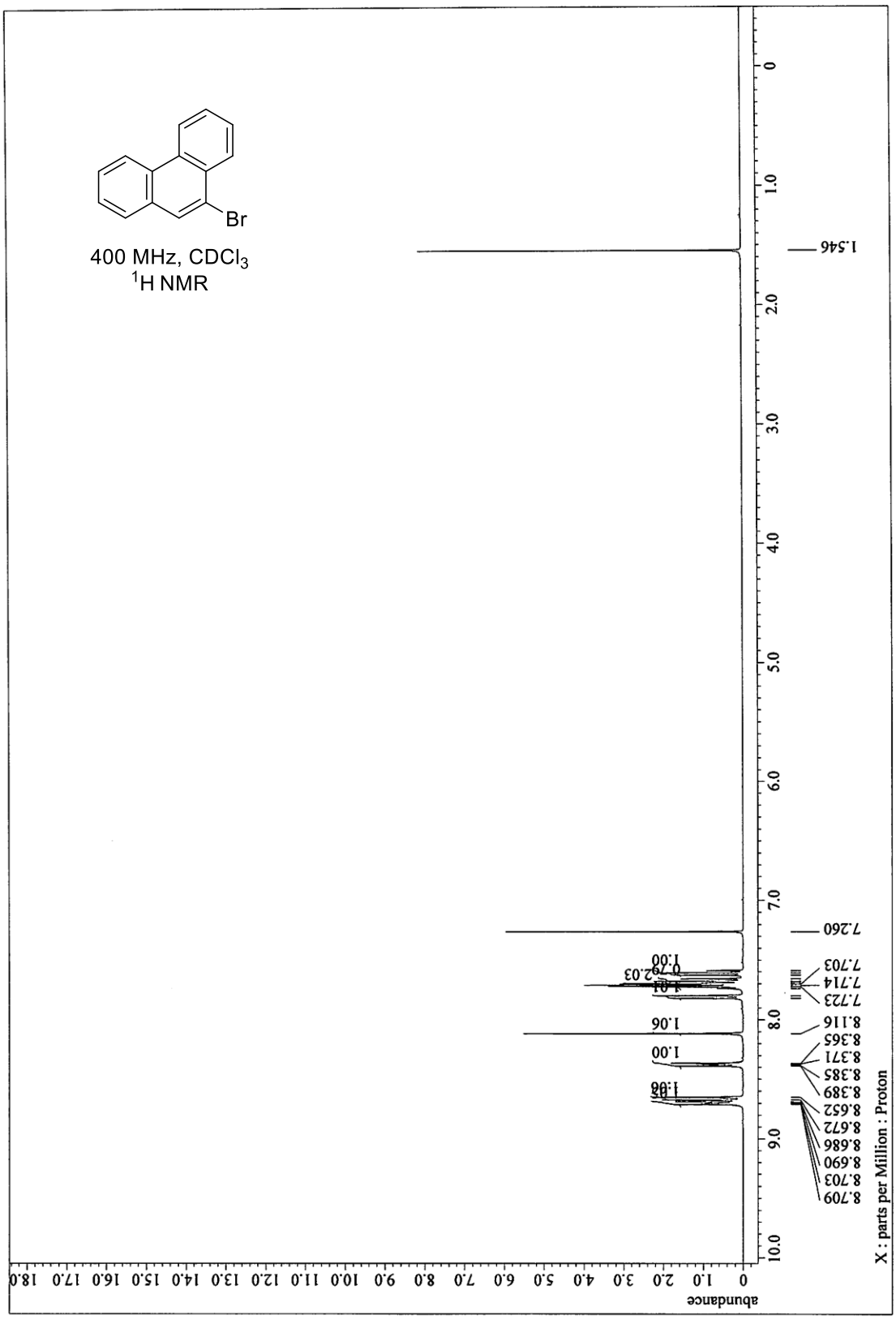

O
100 MHz, CDCl3
13C NMR
76.685
77.000
77.324
81.205
83.379
119.286
122.623
125.102
125.359
126.189
127.629
128.735
131.748
143.399
144.867
147.804
148.290
X : parts per Million : Carbon13

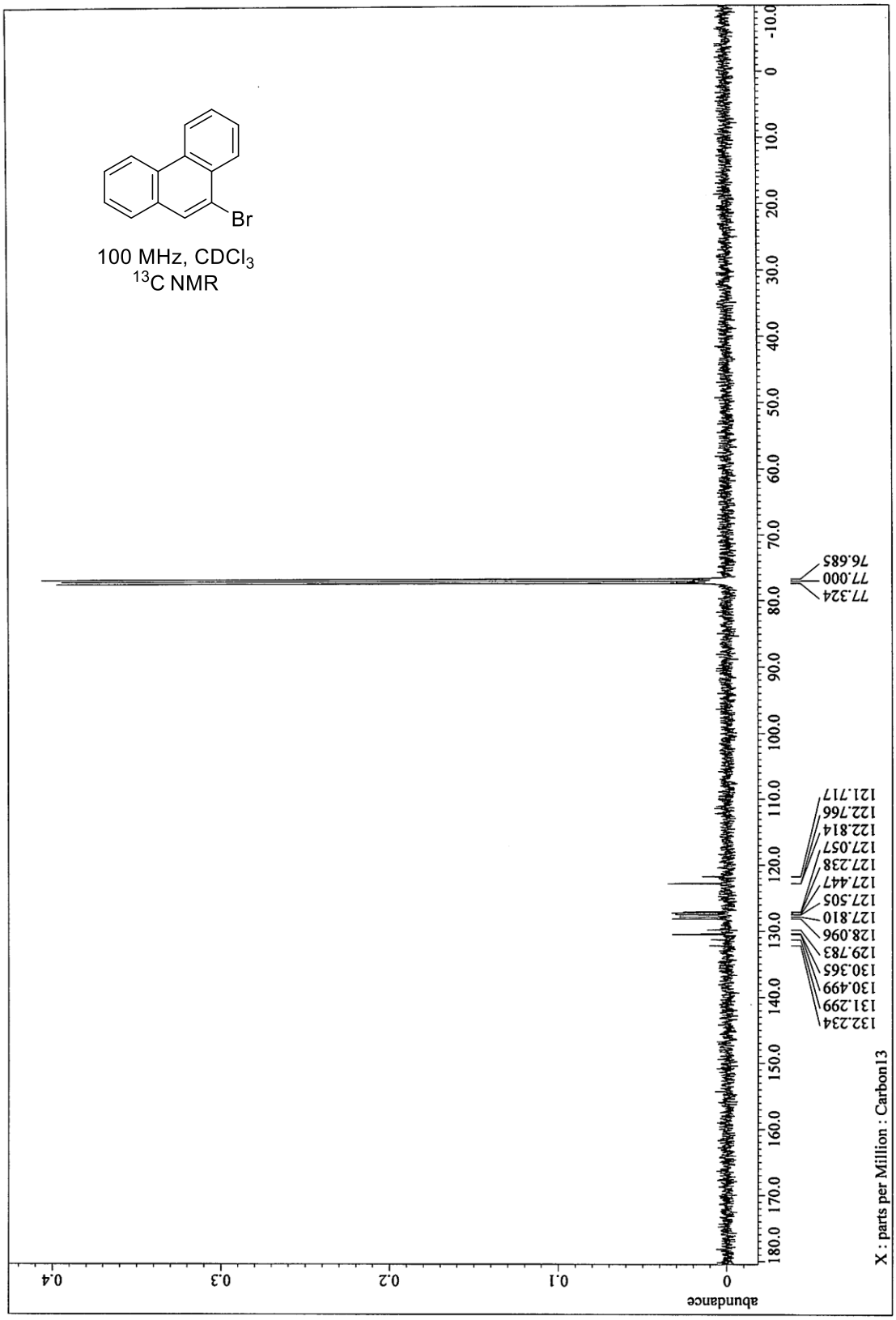

O
400 MHz, CDCl3
1H NMR
3.08
3.09
2.006
2.295
1.00
0.99
0.97
0.98
1.02
1.01
8.035
8.015
7.849
7.827
7.603
7.582
7.444
7.440
7.424
7.419
7.404
7.363
7.360
7.342
7.339
7.260
7.019
7.007
6.916
6.904
X : parts per Million : Proton

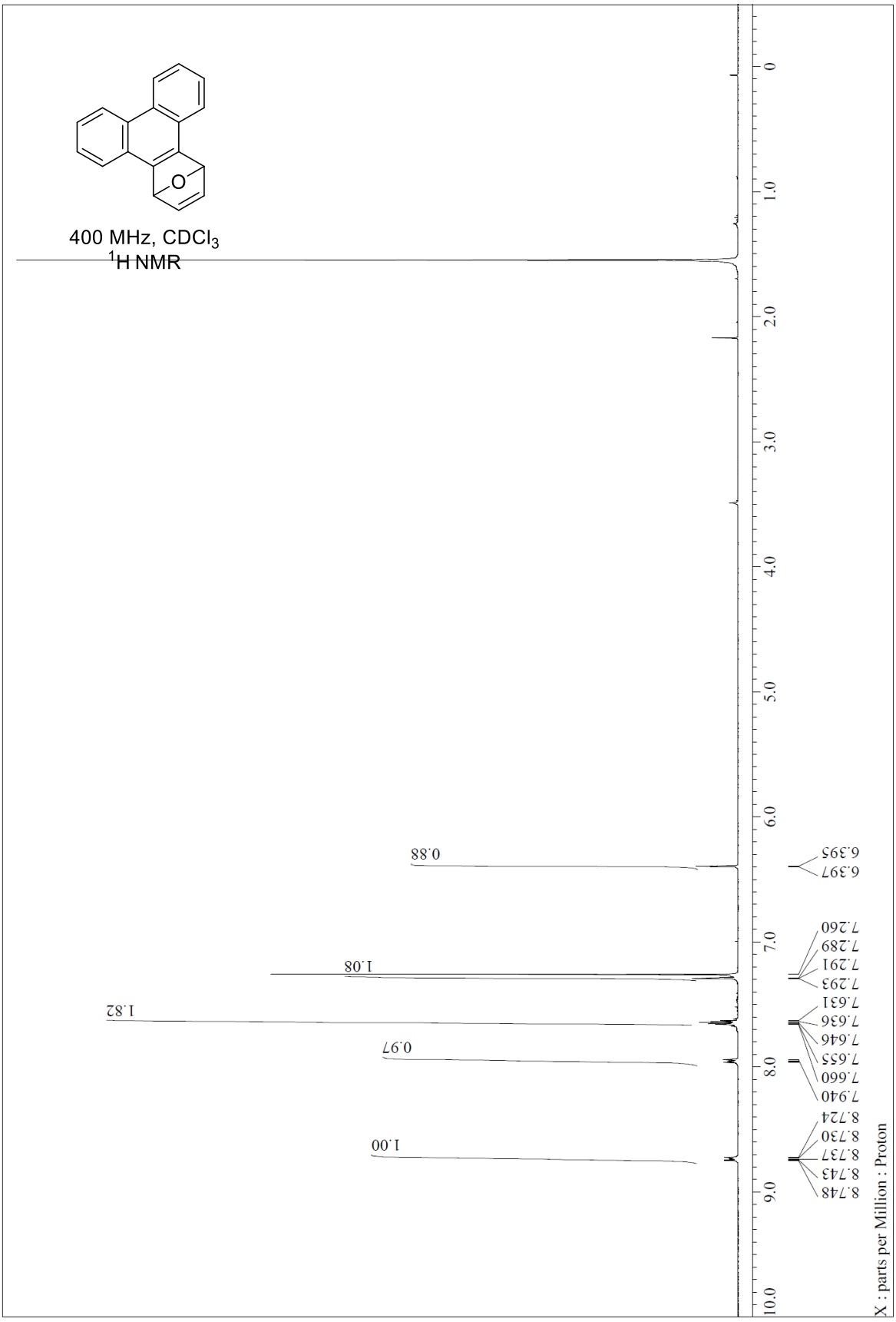

O
100 MHz, CDCl3
13C NMR
15.340
18.763
76.676
77.000
77.315
88.880
90.692
117.427
122.404
124.454
125.836
126.065
127.762
129.173
132.110
148.166
148.319
149.377
152.399
X : parts per Million : Carbon13

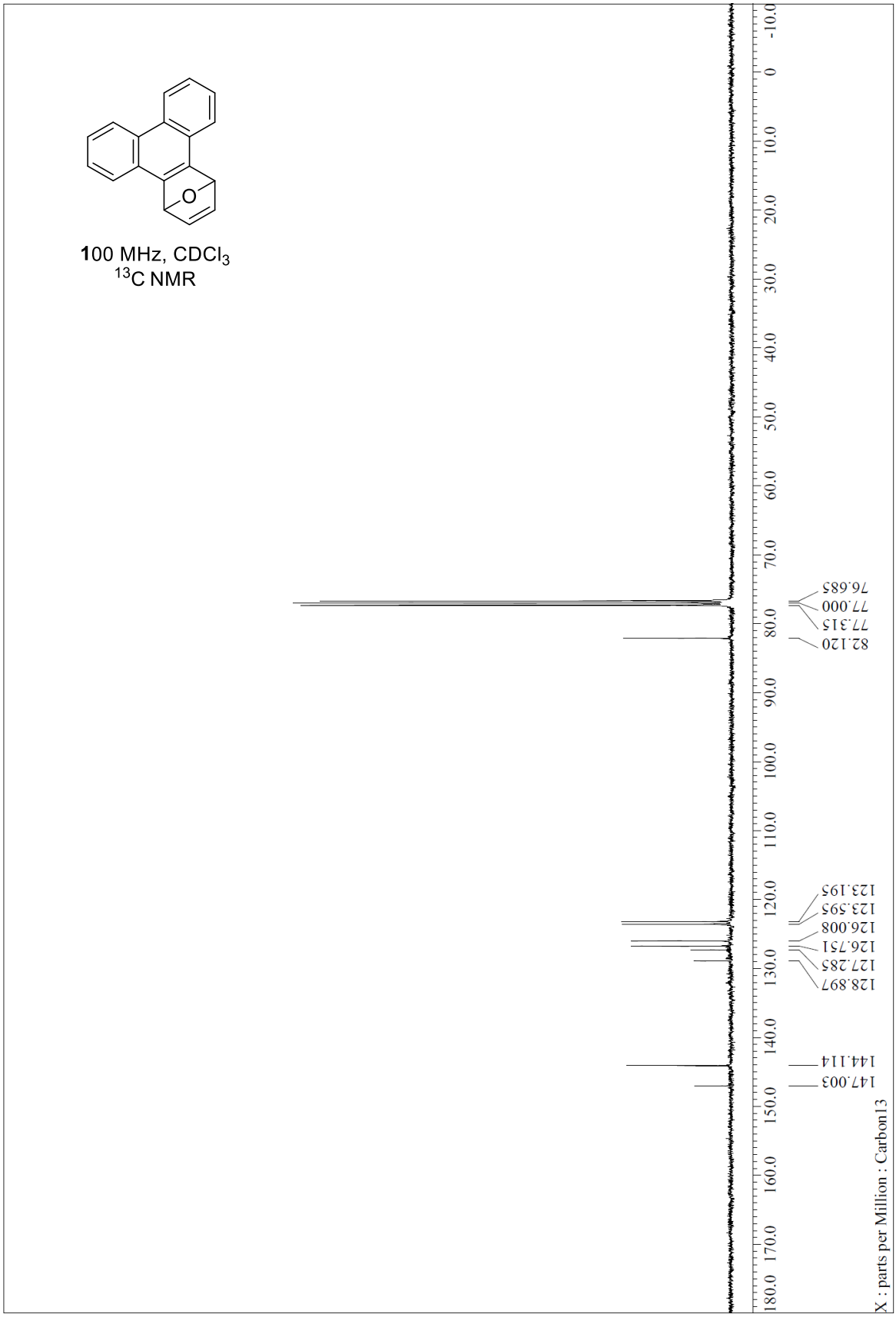

4e
400 MHz, CDCl3
1H NMR
1.498
7.260
7.611
7.628
7.630
7.649
7.653
7.670
7.674
7.755
7.893
7.897
7.913
7.916
8.696
8.717
1.00
1.02
X : parts per Million : Proton
abundance

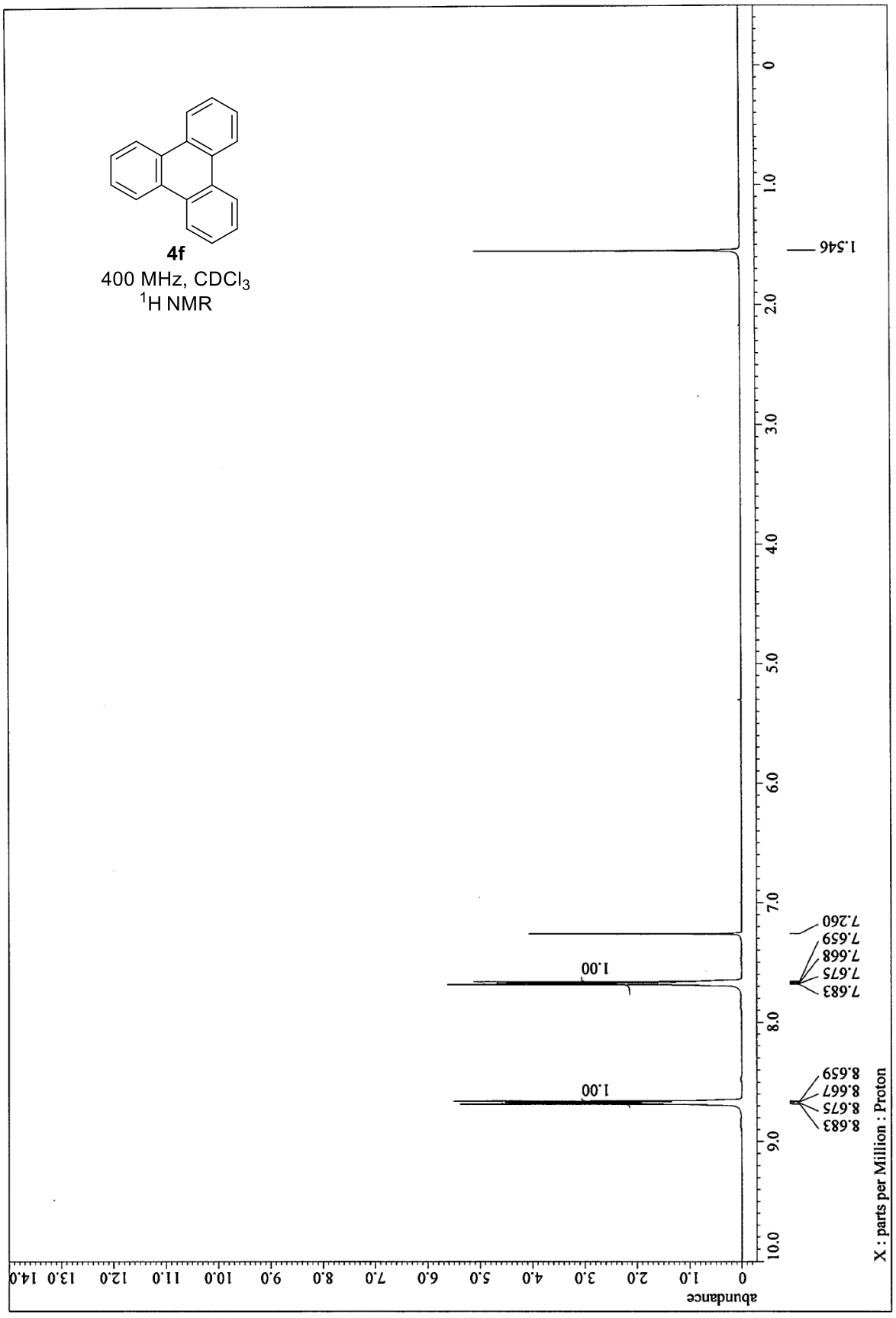

4e
100 MHz, CDCl3
13C NMR
abundance
X : parts per Million : Carbon13
132.024
130.279
128.553
126.904
126.542
122.632
77.315
77.000
76.676

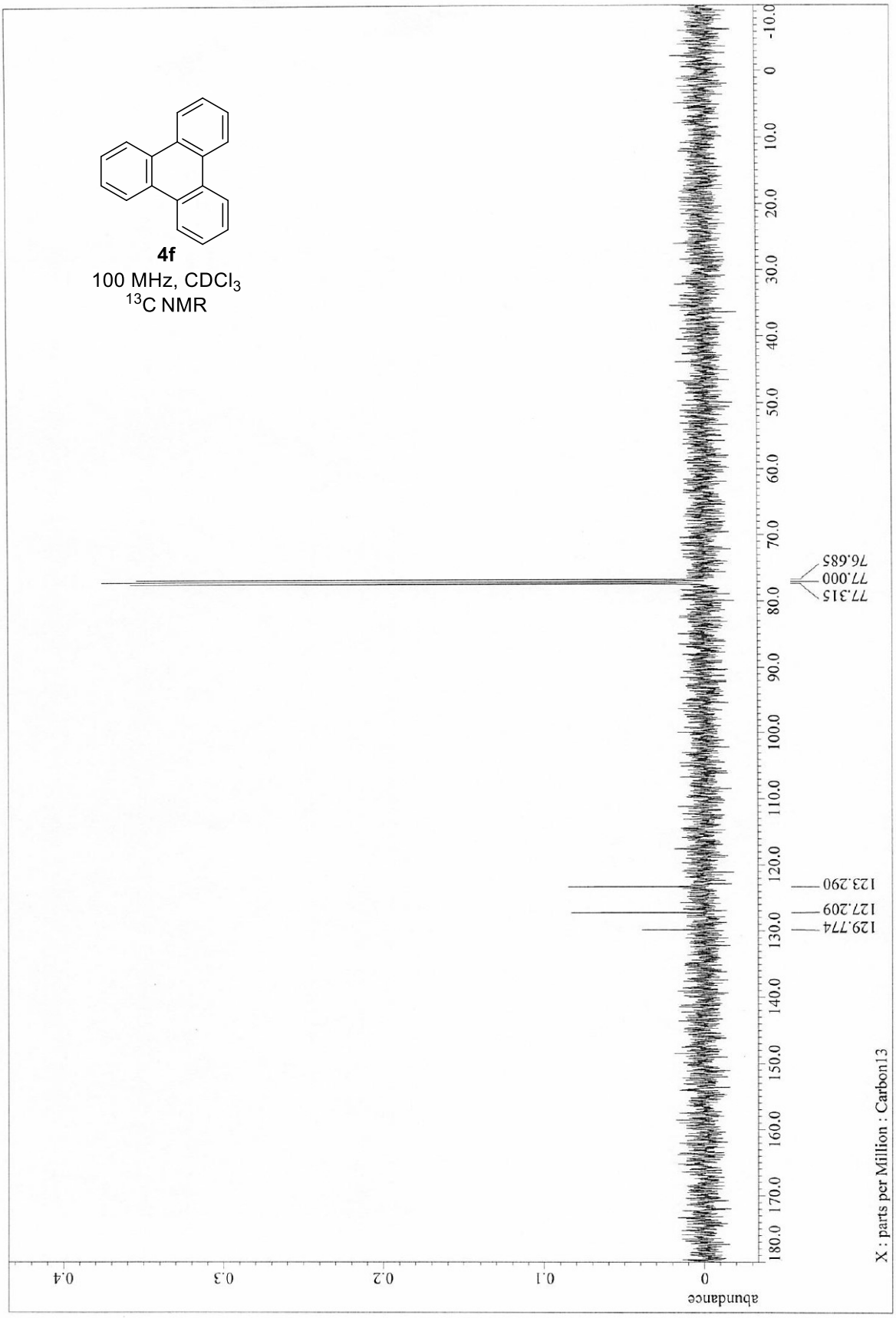

Br
400 MHz, CDCl3
1H NMR
1.546
7.260
7.703
7.714
7.723
8.116
8.365
8.371
8.385
8.389
8.652
8.672
8.686
8.690
8.703
8.709
1.06
1.00
2.03
1.00
X : parts per Million : Proton
abundance


Br
100 MHz, CDCl3
13C NMR
abundance
X : parts per Million : Carbon13
132.234
131.299
130.499
130.365
129.783
128.096
127.810
127.505
127.447
127.238
127.057
122.814
122.766
121.717
77.324
77.000
76.685


O
400 MHz, CDCl3
1H NMR
0.88
1.08
1.82
0.97
1.00
6.395
6.397
7.260
7.289
7.291
7.293
7.631
7.636
7.646
7.655
7.660
7.940
8.724
8.730
8.737
8.743
8.748
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
X : parts per Million : Proton


O
100 MHz, CDCl3
13C NMR
76.685
77.000
77.315
82.120
123.195
123.595
126.008
126.751
127.285
128.897
144.114
147.003
X : parts per Million : Carbon13


4f
400 MHz, CDCl3
1H NMR
1.546
7.260
7.659
7.668
7.675
7.683
8.659
8.667
8.675
8.683
1.00
1.00
X : parts per Million : Proton
abundance


4f
100 MHz, CDCl3
13C NMR
77.315
77.000
76.685
123.290
127.209
129.774
X : parts per Million : Carbon13
abundance