Electronic Supplementary Information

An Environment-Friendly Protocol for Oxidative Halocyclization of Tryptamine and Tryptophol Derivatives

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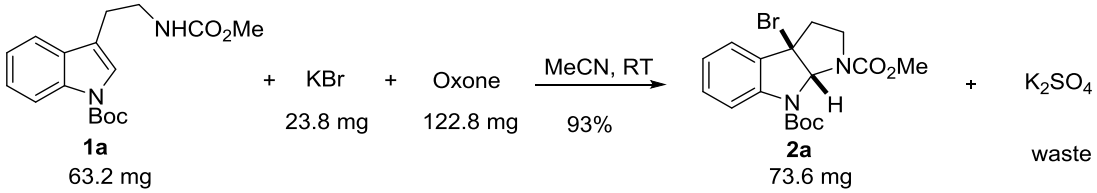
General Information

Reactions were carried out in oven or flame-dried glassware under a nitrogen atmosphere, unless otherwise noted. Tetrahydrofuran (THF) was freshly distilled before use from sodium using benzophenone as indicator. Dichloromethane was freshly distilled before use from calcium hydride (CaH_2). All other solvents were dried over 3Å or 4Å molecular sieves. Solvents used in workup, extraction and column chromatography were used as received from commercial suppliers without prior purification. Reactions were magnetically stirred and monitored by thin layer chromatography (TLC, 0.25 mm) on Merck pre-coated silica gel plates. Flash chromatography was performed with silica gel 60 (particle size 0.040 – 0.062 mm) supplied by Grace. Infrared spectra were collected on a Bruker model TENSOR27 spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AV-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C). Chemical shifts are reported in parts per million (ppm) as values relative to the internal chloroform (7.26 ppm for ^{1}H and 77.16 ppm for ^{13}C). Abbreviations for signal coupling are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Optical rotations were measured on a JASCO Perkin-Elmer model P-2000 polarimeter. High resolution mass spectra were measured at the Hong Kong University of Science and Technology Mass Spectrometry Service Center on either an Agilent GC/MS 5975C system or an API QSTAR XL System.

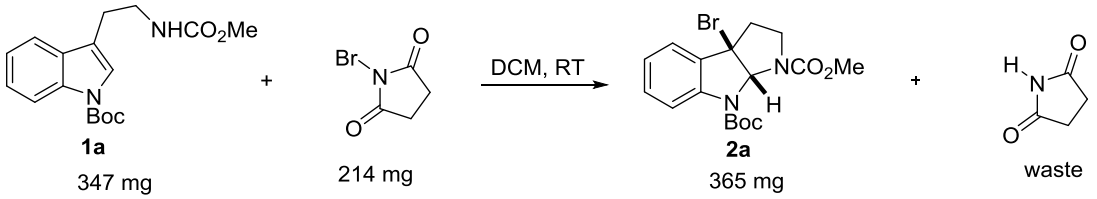
E-Factor Analysis

Example A (this work)



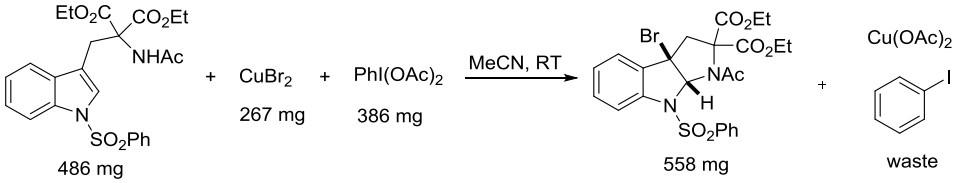
Total amount of reactants: 63.2 mg + 23.8 mg + 122.8 mg = 209.8 mg
Amount of final product: 73.6 mg
Amount of waste: 209.8 mg – 73.6 mg = 146.2 mg
E-Factor = Amount of waste/Amount of product = 146.2 /73.6 = 1.99

Example B (T. Newhouse, C. A. Lewis, K. J. Eastman and P. S. Baran, *J. Am. Chem. Soc.,* 2010, **132**, 7119)



Total amount of reactants: 347 mg + 214 mg = 561 mg
Amount of final product: 365 mg
Amount of waste: 561 mg – 365 mg = 196 mg
E-Factor = Amount of waste/Amount of product =196/365 = 0.54

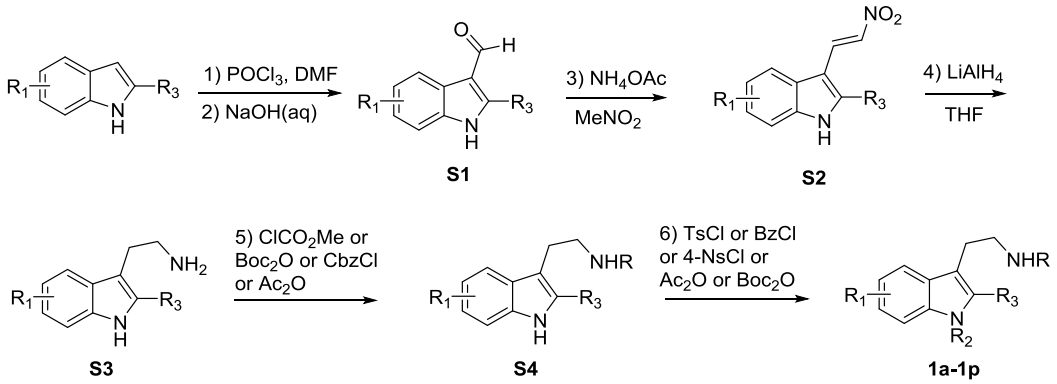
Example C (D. Tu, L. Ma, X. Tong, X. Deng and C. Xia, *Org. Lett*., 2012, **14**, 4830.)



Total amount of reactants: 486 mg + 267 mg + 386 mg = 1139 mg
Amount of final product: 558 mg
Amount of waste: 1139 mg – 558 mg = 581 mg
E-Factor = Amount of waste/Amount of product =581/558 = 1.04

Full Experimental Details and Spectroscopic Data

General Procedure A for Synthesis of the Substrates **1a-1p**.[1] Substrates **1a**, **1b** and **1e** are known compounds and reported in the related literature.[2] General Procedure B for Synthesis of the Substrates **1u-1ab**. Substrates **1u-1w**, **1y-1ab** are known compounds and reported in the related literature.[3] Substrates **1q**[4] and **1r**[5] are known compounds and prepared according to related literature. General Procedure C for the synthesis of products **2a-2ab**.



General Procedure A: To an oven-dried 250-mL round bottom flask was added dry DMF (20 mL) and a stir bar. The flask was cooled to 0 °C and $POCl_3$ (1.40 mL, 15mmol) was added dropwise. The solution was stirred for 15 min at this temperature before the addition of the substituted indole (10.0 mmol) dissolved in DMF (10 mL) over 15 min. The mixture was then allowed to warm to room temperature and stirred for 3 h, then ice water (20 mL) was carefully added, followed by aqueous 1 N NaOH (10 mL), with vigorous stirring. Additional 1 N NaOH was added until the reaction mixture maintained a yellow color. The reaction was then heated to reflux for 5 min before being allowed to cool to room temperature and stirred overnight. The mixture was then extracted with EtOAc (30 mL × 3), and the combined organic layers were washed with water, brine, dried over Na_2SO_4, filtered and concentrated to yield the aldehyde **S1** as a solid, which was used for the next step without further purification.

To an oven-dried 250-mL round bottom flask were added the crude aldehyde **S1**, nitromethane (20 mL), and ammonium acetate (1.69 g, 21.9 mmol). The reaction was then heated to reflux for 90 min with vigorous stirring. Then, the reaction mixture was concentrated by rotary evaporation and the residue was dissolved in EtOAc (30 mL). The organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated to afford the nitro alkene **S2**, which was used for the next step without further purification.

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1 (a) M. E. Muratore, C. A. Holloway, A. W. Pilling, R. I. Storer, G. Trevitt and D. J. Dixon, *J. Am. Chem. Soc*., 2009, **131**, 10796. (b) H. M. Nelson, S. H. Reisberg, H. P. Shunatona, J. S. Patel and F. D.Toste, *Angew. Chem., Int. Ed*., 2014, **53**, 5600

2 W. Xie, G. Jiang, H. Liu, J. Hu, X. Pan, H. Zhang, X. Wan, Y. Lai and D. Ma, *Angew. Chem., Int. Ed*., 2013, **52**, 12924

3 L. Han, C. Liu, W. Zhang, X.-X. Shi and S.-L. You, *Chem. Commun.*, 2014, **50**, 1231.

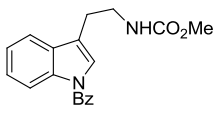
4 S. Zhou, D. Zhang, Y. Sun, R. Li, W. Zhang and A. Li, *Adv. Synth. Catal*., 2014, **356**, 2867.

5 V. R. Espejo and J. D. Rainier, *J. Am. Chem. Soc.*, 2008, **130**, 12894.

Under an inert nitrogen atmosphere, a THF solution (50 mL) of nitro olefin **S2** was added to a stirred slurry of $LiAlH_4$ powder (2.28 g, 60.6 mmol) in THF (50 mL) at 0 ^oC. The mixture was allowed to warm to room temperature and stirred for 36 h. The reaction was quenched by dropwise addition of water until effervescence ceased. The mixture was then diluted with diethyl ether before addition of a saturated aqueous solution of Rochelle's salt .The subsequent biphasic mixture was stirred for 24 h. The layers were separated and the organic layer was extracted with aqueous 1 N HCl. The aqueous phase was basified with 3 N KOH aqueous, and extracted with diethyl ether, dried over Na_2SO_4, filtered and concentrated *in vacuo* to provide the desired tryptamine derivative **S3**, which was used for the next step without further purification.

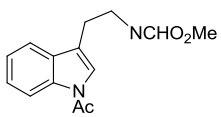
The tryptamine derivative **S3** was dissolved in CH_2Cl_2 (20 mL) and 10% aqueous Na_2CO_3 (10 mL), and then to the reaction mixture was added $ClCO_2Me$ or Boc_2O or CbzCl or Ac_2O (10.0 mmol). After vigorous stirring for 2 h, the reaction mixture was diluted with water. The organic layer was collected and the aqueous phase was extracted with EtOAc (10 mL × 3). The combined extracts were washed with brine, dried over Na_2SO_4, filtered and concentrated *in vacuo* to give the desired tryptamine derivative **S4**, which was used for the next step without further purification.

To a stirred solution of tryptamine derivative **S4** in CH_2Cl_2 (30 mL) were sequentially added Bu_4NHSO_4 (325 mg, 1 mmol) and NaOH (1.80 g, 45 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 5 min before addition of TsCl or BzCl or 4-NsCl or Ac_2O or Boc_2O (10.0 mmol). The reaction mixture was allowed to stir at room temperature for 5 h. Water was added, and the mixture was extracted with EtOAc (10 mL × 3). The combined organic phases were washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (EtOAc/hexane = 1:5) to give the tryptophan derivative **1a-1p.** The physical data for new compounds were provided below.



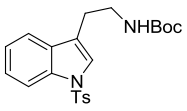
1c

1c: light yellow oil, 1.29 g, 40% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.38 (d, *J* = 8.1 Hz, 1H), 7.75-7.68 (m, 2H), 7.64-7.56 (m, 2H), 7.55-7.51 (m, 2H), 7.42-7.36 (m, 1H), 7.33 (td, *J* = 7.5, 1.1 Hz, 1H), 7.13 (s, 1H), 4.84 (s, 1H), 3.65 (s, 3H), 3.48 (dd, *J* = 13.2, 6.6 Hz, 2H), 2.90 (t, *J* = 7.0 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 168.5, 157.2, 136.6, 134.8, 133.4, 132.0, 130.8, 130.2, 129.2, 128.7, 128.5, 125.4, 125.07, 124.0, 119.0, 116.8, 52.2, 40.7, 25.8. IR (KBr) 2360.4, 2341.4, 1682.0, 1453.0,1357.9, 1219.2, 772.4 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{19}H_{18}N_2O_3$ $[M]^+$ 322.1317; found 322.1308.



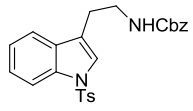
1d

1d: white solid, 936 mg, 36% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.37 (d, *J* = 5.1 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.25 (t, *J* = 7.3 Hz, 1H), 7.18 (s, 1H), 5.22 (s, 1H), 3.64 (s, 3H), 3.54-3.36 (m, 2H), 2.87 (t, *J* = 6.7 Hz, 2H), 2.46 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 168.5, 157.2, 135.8, 130.4, 125.3, 123.5, 122.6, 119.5, 118.8, 116.6, 52.0, 40.4, 25.6, 23.8. IR (KBr) 2360.4, 2341.4, 1699.7, 1537.6, 1453.6, 1389.6, 1253.4, 772.3 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{14}H_{16}N_2O_3$ $[M]^+$ 260.1161; found 260.1151.



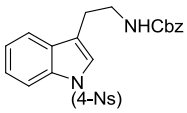
1f

1f: light yellow oil, 1.53 g, 37% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 8.3 Hz, 1H), 7.72 (d, *J* = 8.1 Hz, 2H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.29 (t, *J* = 7.7 Hz, 1H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 2H), 4.83 (s, 1H), 3.39 (d, *J* = 5.3 Hz, 2H), 2.84 (t, *J* = 6.8 Hz, 2H), 2.25 (s, 3H), 1.44 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 155.9, 144.8, 135.2, 135.1, 130.8, 129.8, 126.7, 124.7, 123.4, 123.1, 120.1, 119.5, 113.7, 79.2, 39.9, 28.4, 25.5, 21.4. IR (KBr) 2360.5, 2341.4, 1699.2, 1514.7, 1449.3, 1366.7, 1172.8, 772.0 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{22}H_{26}N_2O_4S$ $[M]^+$ 414.1613; found 414.1617.



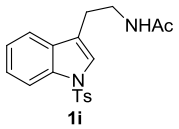
1g

1g: light yellow oil, 1.57 g, 35% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 8.3 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.48 (d, *J* = 7.7 Hz, 1H), 7.41-7.27 (m, 7H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 5.11 (s, 2H), 4.79 (s, 1H), 3.51-3.46 (m, 2H), 2.89 (t, *J* = 6.8 Hz, 2H), 2.29 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 156.4, 145.0, 136.6, 135.5, 135.3, 130.8, 123.0, 128.7, 128.3, 128.3, 126.9, 125.0, 123.7, 123.3, 119.8, 119.5, 114.0, 66.9, 40.5, 25.7, 21.7. IR (KBr) 2360.4, 2341.4, 1705.8, 1219.3, 1172.1, 772.5, 669.1 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{25}H_{24}N_2O_4S$ $[M]^+$ 448.1457; found 448.1453.



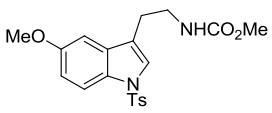
1h

1h: light yellow oil, 1.67 g, 35 % yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.16 (d, *J* = 8.7 Hz, 2H), 7.97 (dd, *J* = 7.1, 1.9 Hz, 3H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.38-7.27 (m, 8H), 5.10 (s, 2H), 4.96 (t, *J* = 5.7 Hz, 1H), 3.49 (dd, *J* = 13.1, 6.6 Hz, 2H), 2.90 (t, *J* = 6.7 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 156.4, 150.6, 143.1, 136.4, 135.2, 131.0, 128.7, 128.6, 128.3, 128.1, 128.0, 125.6, 124.5, 124.1, 123.2, 121.6, 119.9, 113.7, 66.8, 40.32, 25.59. IR (KBr) 2360.4, 2341.4, 1699.5, 1531.9, 1219.5, 1179.7, 772.6 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{24}H_{21}N_3O_6S$ $[M]^+$479.1151; found 479.1155.



1i

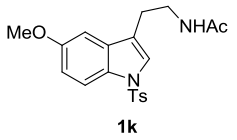
1i: pale yellow solid, 1.46 g, 41% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 8.3 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 7.7 Hz, 1H), 7.36 (s, 1H), 7.33-7.27 (m, 1H), 7.24-7.16 (m, 3H), 5.88 (s, 1H), 3.51 (dd, *J* = 13.0, 6.8 Hz, 2H), 2.86 (dd, *J* = 7.2, 6.6 Hz, 2H), 2.31 (s, 3H), 1.90 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.4, 145.0, 135.4, 135.2, 130.8, 129.9, 126.8, 125.0, 123.4, 123.3, 120.0, 119.5, 113.8, 39.0, 25.2, 23.3, 21.6. IR (KBr) 2924.8, 2854.8, 2360.4, 2341.4, 1648.3, 1448.4, 1367.4, 1219.5, 1172.7, 772.6 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{19}H_{21}N_2O_3S$ $[M+H]^+$ 357.1273; found 357.1264.



1j

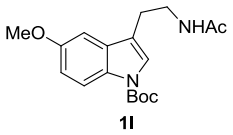
1j: light yellow oil, 1.69 g, 42% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.86 (d, *J* = 9.2 Hz, 1H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.32 (s, 1H), 7.18 (d, *J* = 8.1 Hz, 2H), 6.94-6.87 (m, 2H), 4.82 (s, 1H), 3.80 (s, 3H), 3.66 (s, 3H), 3.44 (dd, *J* = 12.6, 6.3 Hz, 2H), 2.82 (t, *J* = 6.8 Hz, 2H), 2.31 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 157.1, 156.6, 144.9, 135.2, 131.8, 130.1, 129.9, 126.8, 124.4, 120.1, 114.9, 113.9, 102.0, 55.8, 52.2, 40.4, 25.7, 21.6. IR (KBr) 2360.4, 2341.4,1765.1, 1705.1, 1474.7, 1387.1, 1219.8,

1171.2, 772.6, 669.7 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{20}H_{22}N_2O_5S$ $[M]^+$ 402.1249; found 402.1243.



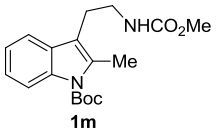
1k

1k: pale yellow solid, 1.20 g, 31% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.87 (d, *J* = 9.7 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 2H), 7.31 (s, 1H), 7.20 (d, *J* = 8.2 Hz, 2H), 6.93 (dt, *J* = 6.2, 3.1 Hz, 2H), 5.62 (s, 1H), 3.81 (s, 3H), 3.52 (q, *J* = 6.7 Hz, 2H), 2.83 (t, *J* = 6.9 Hz, 2H), 2.33 (s, 3H), 1.93 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.3, 156.6, 145.0, 135.3, 131.8, 130.1, 130.0, 126.8, 124.2, 120.0, 114.9, 114.0, 102.0, 55.8, 39.0, 25.3, 23.4, 21.7. IR (KBr) 2360.4, 2341.4, 1651.1, 1474.7, 1366.8, 1219.7, 1171.1, 772.4, 669.6 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{20}H_{23}N_2O_4S$ $[M+H]^+$ 387.1379; found 387.1364.



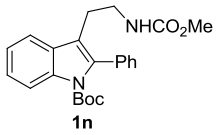
1l

1l: pale yellow solid, 1.13 g, 34% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.00 (s, 1H), 7.39 (s, 1H), 6.99 (d, *J* = 2.5 Hz, 1H), 6.93 (dd, *J* = 9.0, 2.5 Hz, 1H), 5.58 (s, 1H), 3.86 (s, 3H), 3.58 (q, *J* = 6.7 Hz, 2H), 2.88 (t, *J* = 6.8 Hz, 2H), 1.96 (s, 3H), 1.66 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.3, 156.0, 149.8, 131.3, 130.3, 123.8, 117.6, 116.2, 113.2, 101.8, 83.6, 55.8, 39.2, 28.3, 25.2, 23.4. IR (KBr) 2979.1, 2360.4,2341.5, 1729.1, 1650.7, 1478.6, 1387.3, 1257.6, 1160.2, 772.0 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{18}H_{24}N_2O_4$ $[M]^+$ 332.1736; found 332.1731.



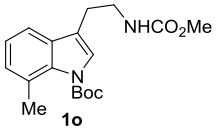
1m

1m: light yellow oil, 1.06 g, 32% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.11 (d, *J* = 7.4 Hz, 1H), 7.46 (d, *J* = 7.1 Hz, 1H), 7.33 -7.12 (m, 2H), 4.87 (s, 1H), 3.66 (s, 3H), 3.37 (dd, *J* = 13.1, 6.6 Hz, 2H), 2.89 (t, *J* = 6.9 Hz, 2H), 2.54 (s, 3H), 1.68 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 157.2, 150.8, 135.8, 134.3, 129.8, 123.6, 122.6, 117.7, 115.5, 115.0, 83.7, 52.1, 41.0, 28.3, 24.5, 13.9. IR (KBr) 2978.5,2360.5, 1728.3, 1459.5, 1525.3, 1459.5, 1358.0, 1356.2, 1221.0, 1137.4, 772.2 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{18}H_{24}N_2O_4$ $[M]^+$ 332.1736; found 332.1729.



1n

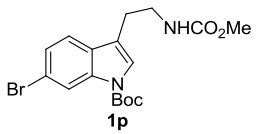
1n: light yellow oil, 1.38 g, 35% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.27 (d, *J* = 8.2 Hz, 1H), 7.62 (d, *J* = 7.5 Hz, 1H), 7.46-7.26 (m, 7H), 4.67 (s, 1H), 3.60 (s, 3H), 3.35 (dd, *J* = 13.0, 6.5 Hz, 2H), 2.77 (t, *J* = 6.9 Hz, 2H), 1.22 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 157.0, 150.9, 136.9, 136.6, 134.2, 129.9, 129.4, 128.1, 127.8, 124.8, 123.0, 118.9, 117.4, 115.5, 83.2, 52.1, 41.1, 27.6, 24.9. IR (KBr) 2980.2, 2360.4, 1227.6, 1523.5, 1475.5, 1360.0, 1327.8, 1154.5, 771.6 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{23}H_{26}N_2O_4$ $[M]^+$ 394.1893; found 394.1888.



1o

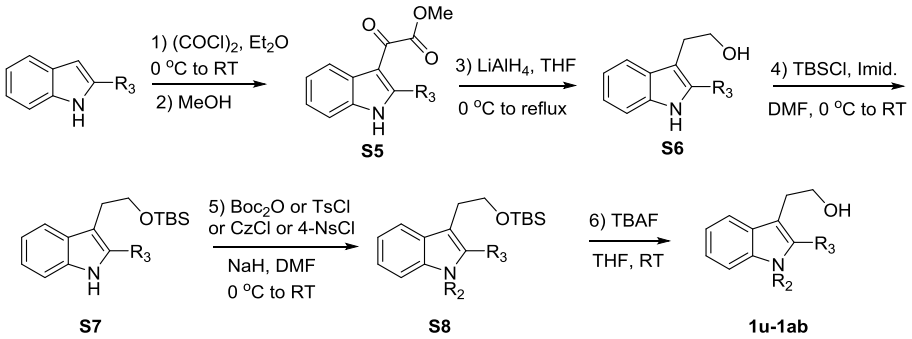
1o: light yellow oil, 1.19 g, 36 % yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.36 (d, *J* = 8.4 Hz, 2H), 7.20-7.08 (m, 2H), 4.87 (s, 1H), 3.67 (s, 3H), 3.50 (d, *J* = 6.4 Hz, 2H), 2.88 (t, *J* = 6.8 Hz, 2H), 2.63 (s, 3H), 1.64 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.2, 149.6, 135.3,

131.7, 128.0, 125.7, 125.6, 123.1, 117.3, 116.5, 83.3, 52.1, 40.7, 28.2, 25.7, 22.3. IR (KBr) 2979.1, 2360.4, 1717.0, 1524.6, 1456.0, 1350.9, 1256.0, 1221.0, 1156.8, 1046.6, 772.5 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{18}H_{24}N_2O_4$ $[M]^+$ 332.1736; found 332.1730.



1p

1p: light yellow oil, 1.42 g, 36% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.30 (s, 1H), 7.37-7.30 (m, 3H), 4.97 (s, 1H), 3.64 (s, 4H), 3.45 (dd, *J* = 12.7, 6.3 Hz, 22H), 2.85 (t, *J* = 6.8 Hz, 2H), 1.64 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 157.1, 149.3, 136.26, 129.2, 125.8, 123.6, 120.0, 118.5, 118.3, 117.5, 84.2, 52.1, 40.7, 28.2, 25.6. IR (KBr) 2979.7, 2360.4, 1732.1, 1530.8, 1454.8, 1372.4, 1310.7, 1254.1, 1157.7, 1091.8, 771.3 cm^{-1}; HRMS (CI^+) (m/z) calcd. for $C_{17}H_{21}BrN_2O_4$ $[M]^+$ 396.0685; found 396.0675.



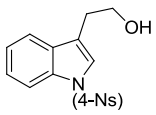
General Procedure B: To a solution of indole (10.0 mmol) in dry Et_2O (50 mL) at 0 °C was added dropwise oxalyl chloride (2.7 mL, 30.0 mmol). The ice bath was removed and the resultant yellow slurry was stirred at room temperature for 6 h and then cooled to 0 °C, followed by quenching with MeOH (2.0 mL, 50.0 mmol). The crude reaction mixture was filtered with celite and washed with cold Et_2O. The solid **S5** was used directly for the next step without further purification.

A solution of **S5** in THF (20 mL) was added dropwise to a suspension of $LiAlH_4$ (1.52 g, 40 mmol) in THF (40 mL) at 0 °C. The solution was stirred at 80 °C for 2 h and quenched carefully by H_2O (1.5 mL), 10% aqueous NaOH (3.0 mL), H_2O (4.5 mL) at 0 °C. The solution was then filtered and washed with EtOAc. The combined organic layers were dried over Na_2SO_4 and the solvent was removed under reduced pressure to give the crude product. The crude product was purified by silica gel column chromatography (EtOAc/hexane 1:3) to afford the tryptophol **S6**

TBSCl (1.66 g, 11 mmol) was added to a solution of **S6** (10.0 mmol) and imidazole (1.36 g, 20.0 mmol) in DMF (50 mL) at 0 °C. The ice bath was then removed and the reaction mixture was stirred at room temperature for 3 h. The mixture was quenched with water and extracted with EtOAc (30 mL × 3), then the combined organic layers were washed with water, brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was used directly for the next step without further purification.

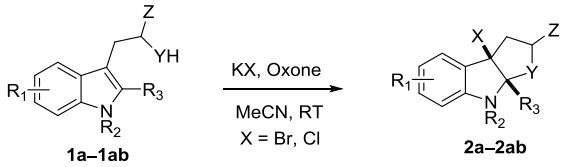
To a solution of **S7** in THF (50 mL) was added NaH (400 mg, 10.0 mmol) at 0 °C. After stirring at 0 °C for 15 min and then at room temperature for 1 h, the reaction

mixture was cooled to 0 °C, treated with Boc_2O or TsCl or CzCl or 4-NsCl (11.0 mmol), and then allowed to stir at room temperature for 6-12 h. After the reaction was complete (monitored by TLC), aqueous saturated $NaHCO_3$ (30 mL) was added slowly. The organic layer was separated and the aqueous layer was extracted with EtOAc (30 mL × 3). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product **S8** was further treated with *tetra-n*-butylammonium fluoride (15 mmol), after 24 h, the mixture was worked up and purified by silica gel column chromatography (EtOAc/hexane = 1:2) to afford the desired product **1u-1ab**. The physical data for new compounds were provided below.

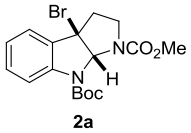


1x

1x: pale yellow solid, 1.48 g, 43% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.25 (d, *J* = 8.8 Hz, 2H), 8.04 (d, *J* = 8.8 Hz, 2H), 7.98 (d, *J* = 8.2 Hz, 1H), 7.52 (d, *J* = 7.7 Hz, 1H), 7.42 (s, 1H), 7.36 (t, *J* = 7.5 Hz, 1H), 7.29 (d, *J* = 7.5 Hz, 1H), 3.92 (t, *J* = 6.4 Hz, 2H), 2.93 (t, *J* = 6.3 Hz, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 150.7, 143.5, 135.3, 131.3, 128.2, 125.6, 124.6, 124.1, 123.4, 121.4, 120.0, 113.8, 61.8, 28.4. IR (KBr) 2360.3, 1636.0, 1531.2, 1349.6, 1219.5, 1173.6, 772.4 cm^{-1}; HRMS (CI$^+$) (m/z) calcd. for $C_{16}H_{14}N_2O_5S$ $[M]^+$ 346.0623; found 346.0621.

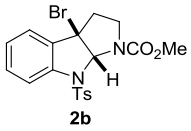


General Procedure C: To the solution of tryptamine and tryptophol derivative **1a–1ab** (0.20 mmol) and KBr (23.8 mg, 0.20 mmol) in MeCN (2.0 mL) was added oxone (122.8 mg, 0.20 mmol) at room temperature, and was stirred for 4–7 h. Saturated Na_2SO_3 aqueous solution (10 mL) was added to the reaction mixture, and the product was extracted with EtOAc (15 mL × 3). The combined extracts were washed with brine and dried over Na_2SO_4. The organic phase was concentrated under reduced pressure and the crude product was purified by silica gel column chromatography to give the HPIs/TFIs **2a–2ab**, respectively.



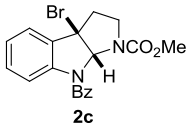
2a

2a (EtOAc/hexane = 1:5): light yellow oil, 73.6 mg, 93% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 1H), 7.31 (t, *J* = 7.0 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 6.40 (s, 1H), 3.81 (t, *J* = 8.0 Hz, 1H), 3.76 (s, 3H), 2.94-2.82 (m, 2H), 2.80-2.75 (m, 1H), 1.61 (s, 9H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.7, 152.2, 142.0, 132.4, 130.6, 124.3, 123.8, 117.4, 84.0, 82.2, 62.2, 52.8, 46.3, 41.1, 28.3. IR (KBr) 2979.8, 1711.6, 1478.2, 1446.0, 1390.4, 1367.2, 1331.2, 1288.9, 1152.9, 754.2 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{17}H_{21}BrN_2O_4$ $[M]^+$ 396.0685; found 396.0667.



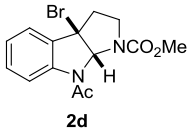
2b

2b (EtOAc/hexane = 1:5): white foam, 85.5 mg, 95% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, *J* = 7.8 Hz, 2H), 7.60 (d, *J* = 8.2 Hz, 1H), 7.32 (td, *J* = 7.9, 1.3 Hz, 1H), 7.30-7.26 (m, 1H), 7.17 (d, *J* = 7.9 Hz, 3H), 6.25 (s, 1H), 3.79 (s, 3H), 3.79-3.75 (m, 1H), 2.81 (td, *J* = 11.3, 5.1 Hz, 1H), 2.75-2.60 (m, 2H), 2.32 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.6, 144.4, 141.3, 135.6, 133.7, 130.8, 129.6, 128.0, 126.1, 124.3, 118.2, 86.8, 61.8, 53.1, 46.0, 42.2, 21.7. IR (KBr) 2360.4, 2341.5, 1714.6, 1714.6, 1447.6, 1383.6, 1219.7, 1170.9, 772.7 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{19}H_{19}BrN_2O_4S$ $[M]^+$ 450.0249; found 450.0250.



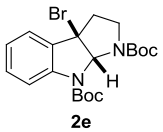
2c

2c (EtOAc/hexane = 1:5): white foam, 72.0 mg, 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.04 (d, *J* = 8.0 Hz, 1H), 7.85-7.72 (m, 2H), 7.56-7.40 (m, 4H), 7.36 (t, *J* = 7.8 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 6.37 (s, 1H), 3.70-3.68 (m, 1H), 3.45 (s, , 3H), 2.96-2.87 (m, 2H), 2.83-2.64 (m, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.4, 154.4, 142.5, 136.4, 132.3, 131.0, 131.0, 128.6, 128.4, 125.4, 123.6, 117.8, 84.5, 62.1, 52.6, 46.5, 39.5. IR (KBr) 2360.4, 1714.0, 1659.7, 1475.0, 1447.8, 1397.4, 772.0 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{19}H_{17}BrN_2O_3$ $[M]^-$ 400.0423; found 400.0416.



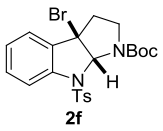
2d

2d (EtOAc/hexane = 1:5): light yellow oil, 60.8 mg, 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.97 (s, 1H), 7.41-7.27 (m, 2H), 7.14 (td, *J* = 7.5, 3.0 Hz, 1H), 6.12 (s, 1H), 3.69 (s, 4H), 2.94-2.80 (m, 2H), 2.77-2.71 (m, 1H), 2.60 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.6, 154.7, 141.9, 132.2, 130.6, 125.3, 123.3, 119.3, 85.3, 61.9, 52.9, 46.6, 40.2, 23.3. IR (KBr) 2955.8, 2360.4, 1708.2, 1475.7, 1450.0, 1386.7, 1348.0, 756.1 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{14}H_{15}BrN_2O_3$ $[M]^+$ 338.0266; found 338.0259.



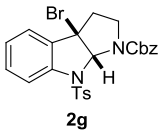
2e

2e (EtOAc/hexane = 1:5): white foam, 79.5 mg, 91% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.57 (s, 1H), 7.36 (d, *J* = 7.5 Hz, 1H), 7.33-7.21 (m, 2H), 7.09 (t, *J* = 7.0 Hz, 1H), 6.43 (s, 1H), 3.72 (t, *J* = 8.2 Hz, 1H), 2.97-2.56 (m, 3H), 1.58 (s, 9H), 1.48 (s, 9H).^{13}C-NMR (100 MHz, $CDCl_3$) δ: 153.6, 152.3, 142.2, 132.8, 130.4, 124.2, 123.9, 117.6, 84.0, 82.2, 80.9, 62.4 46.3,41.8, 28.5, 28.4. IR (KBr) 2978.1, 2981.4, 1714.0, 1478.4, 1393.4, 1366.9, 1152.7, 753.4 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{20}H_{28}BrN_2O_4$ $[M-H]^-$ 437.1154; found 437.1082.



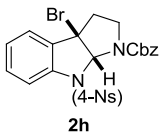
2f

2f (EtOAc/hexane = 1:5): light yellow oil, 89.5 mg, 91% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.68 (d, *J* = 7.1 Hz, 2H), 7.53 (d, *J* = 8.1 Hz, 1H), 7.32-7.25 (m, 2H), 7.19-7.10 (m, 3H), 6.29 (s, 1H), 3.76 (dd, *J* = 9.8, 6.4 Hz, 1H), 2.86-2.48 (m, 3H), 2.31 (s, 3H), 1.55 (s, 9H).^{13}C-NMR (100 MHz, $CDCl_3$) δ: 153.3, 144.2, 141.3, 135.8, 133.8, 130.6, 129.5, 127.9, 125.9, 124.4, 117.8, 86.8, 81.8, 62.2, 46.0, 42.6, 28.4, 21.6. IR (KBr)2979.1, 2360.7, 2341.3, 1701.3, 1475.6, 1462.8, 1390.9, 1367.2, 1171.8, 756.2, 662.2 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{22}H_{25}BrN_2O_4S$ $[M]^-$ 492.0718; found 492.0732.



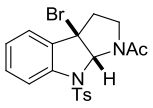
2g

2g (EtOAc/hexane = 1:5): light yellow oil, 100.2 mg, 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.62-7.60 (m, 3H), 7.49 (s, 1H), 7.42-7.26 (m, 6H), 7.20-7.08 (m, 3H), 6.30 (s, 1H), 5.30 (s, 1H), 5.18 (d, *J* = 11.0 Hz, 1H), 3.87-3.72 (m, 1H), 2.84-2.79 (m, 1H), 2.69 (m, 2H), 2.32 (s, 4H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 153.9, 144.4, 141.3, 136.3, 135.5, 133.6, 130.8, 129.5, 128.8, 128.6, 128.3, 128.0, 126.0, 124.4, 117.9, 86.9, 67.8, 61.8, 46.0, 42.5, 21.6. IR (KBr) 2360.4, 2341.4, 1714.0, 1407.8, 1355.1, 1171.7, 772.4, 662.4, 576.9 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{25}H_{23}BrN_2O_4S$ $[M]^-$ 526.0562; found 526.0569.



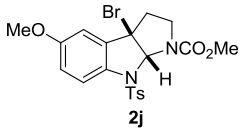
2h

2h (EtOAc/hexane = 1:5): light yellow oil, 90.4 mg, 86% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.17 (d, *J* = 8.4 Hz, 2H), 7.94 (s, 2H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.55-7.28 (m, 7H), 7.26-7.20 (m, 1H), 6.27 (s, 1H), 5.25-5.17 (m, 2H), 3.80 (dd, *J* = 10.7, 7.4 Hz, 1H), 2.85-2.80 (m, 1H), 2.78-2.59 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 153.8, 150.5, 144.0, 140.4, 136.1, 133.9, 131.2, 129.2, 128.7, 128.5, 127.0, 124.6, 124.2, 118.1, 87.0, 68.0, 61.6, 46.2, 41.9, 29.8. IR (KBr) 2360.6, 2341.4, 1713.9, 1531.0, 1176.6, 774.0, 738.6, 613.2, 564.8 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{24}H_{20}BrN_3O_6S$ $[M]^-$ 557.0256; found 557.0257.



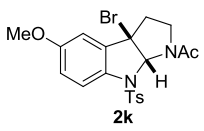
2i

2i (EtOAc/hexane = 1:5): white foam, 75.5 mg, 87% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, *J* = 7.9 Hz, 1H), 7.55 (d, *J* = 7.3 Hz, 2H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.31-7.26 (m, 1H), 7.24 -7.18 (m, 1H), 7.14 (d, *J* = 7.6 Hz, 2H), 6.14 (s, 1H), 4.06 (s,1H), 2.68 (d, *J* = 6.3 Hz, 1H), 2.55 (s, 2H), 2.52 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.8, 145.0, 140.9, 134.5, 130.9 129.6, 128.3, 127.1, 124.4, 119.1, 87.7, 62.1, 45.0, 41.6, 29.8, 22.9, 21.7. IR (KBr) 2360.4, 2341.4, 1660.9, 1402.4, 1366.1, 1170.1, 772.4 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{19}H_{19}BrN_2O_3S$ $[M]^-$ 434.0300; found 434.0314.



2j

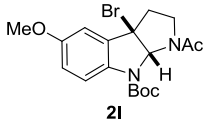
2j (EtOAc/hexane = 1:5): light yellow oil, 88.3 mg, 92% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.62 (d, *J* = 7.9 Hz, 2H), 7.54 (d, *J* = 8.9 Hz, 1H), 7.16 (d, *J* = 8.1 Hz, 2H), 6.88 (dd, *J* = 8.9, 2.6 Hz, 1H), 6.74 (d, *J* = 2.6 Hz, 1H), 6.18 (s, 1H), 3.79 (s, 3H), 3.78 (s, 3H), 3.76-3.72 (m, 1H), 2.83-2.76 (m, 1H), 2.70-2.62 (m, , 2H), 2.32 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 158.3, 154.5, 144.3, 135.5, 135.1, 134.6, 129.6, 128.2, 119.6, 117.1, 108.7, 87.0, 61.8, 55.8, 53.0, 45.8, 42.2, 21.6. IR (KBr) 2954.8, 2360.4, 2341.4, 1713.3, 1597.1, 1487.7, 1447.2, 1383.5, 1363.8, 772.9, 660.4 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{20}H_{21}BrN_2O_5S$ $[M]^-$ 480.0355; found 480.0347.



2k

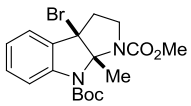
2k (EtOAc/hexane = 1:5): light yellow oil, 87.2 mg, 94% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, *J* = 8.9 Hz, 1H), 7.49 (d, *J* = 7.9 Hz, 2H), 7.12 (d, *J* = 7.9 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 1H), 6.72 (s, 1H), 6.05 (s, 1H), 4.00 (dd, *J* = 9.4, 6.5 Hz, 1H), 3.76 (s, 3H), 2.72-2.51 (m, 3H), 2.49 (s, 3H), 2.29 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ: 170.7, 158.9, 144.9, 135.9, 134.4, 133.9, 129.5, 128.3, 120.5, 117.2, 108.5, 87.7, 77.5, 77.4, 77.2,

76.8, 61.9, 55.8, 44.9, 41.3, 23.0, 21.6. IR (KBr) 3006.5, 2360.4, 1660.3, 1487.8, 1401.2, 1355.7, 1168.0, 991.2, 769.2, 661.6 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{20}H_{21}BrN_2O_4S$ $[M]^-$ 464.0405; found 464.0403.



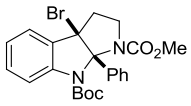
2l

2l (EtOAc/hexane = 1:5): white solid, 75.4 mg, 92% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.34 (s, 1H), 6.85 (d, *J* = 9.4 Hz, 3H), 6.32 (s, 1H), 4.08-3.96 (m, 1H), 3.78 (s, 3H), 2.87-2.57 (m, 3H), 2.31 (s, 3H), 1.55 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.6, 157.4, 152.9, 135.0, 134.1, 119.6, 116.5, 108.3, 85.3, 83.0, 77.5, 77.4, 77.2, 76.8, 62.6, 55.83, 45.2, 40.1, 28.3, 22.3. IR (KBr) 2360.5, 2341.4, 1647.0, 1219.5, 772.6 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{18}H_{23}BrN_2O_4$ $[M]^-$ 410.0841; found 410.0836.



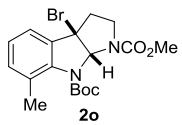
2m

2m (EtOAc/hexane = 1:5): light yellow oil, 74.6 mg, 91% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.75 (d, *J* = 4.8 Hz, 1H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.30 -7.23 (m, 1H), 7.07 (t, *J* = 7.5 Hz, 1H), 3.67 (s, 3H), 3.49 (t, *J* = 9.3 Hz, 1H), 3.01-2.79 (m, 2H), 2.78-2.58 (m, 1H), 2.14 (s, 3H), 1.61 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.2, 152.1, 142.2, 131.7, 130.4, 123.8, 123.2, 118.4, 88.4, 82.0, 70.4, 52.4, 45.9, 36.1, 28.5, 24.6. IR (KBr) 2360.4, 2341.4, 1713.6, 1370.7, 1219.4, 772.4 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{18}H_{23}BrN_2O_4$ $[M]^+$ 410.0841; found 410.0841.



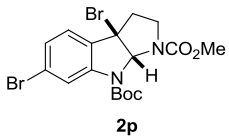
2n

2n (EtOAc/hexane = 1:5): white solid, 84.9 mg, 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.02 (d, *J* = 8.3 Hz, 1H), 7.69 (s, 1H), 7.46-7.27 (m, 5H), 7.17 (d, *J* = 5.5 Hz, 1H), 7.11 (t, *J* = 7.4 Hz, 1H), 3.73 (t, *J* = 9.7 Hz, 1H), 3.61 (s, 3H), 3.29-3.22 (m, 1H), 2.89-2.85 (m, 1H), 2.47-2.38 (m, 1H), 1.30 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.1, 151.8, 142.5, 135.9, 133.1, 131.5, 130.5, 128.4, 127.2, 125.8, 125.4, 123.6, 123.3, 118.4, 92.3, 81.6, 73.12, 52.6, 47.4, 35.1, 27.9. IR (KBr) 2360.4, 2341.4,1704.2, 1362.9, 1219.7, 1154.5, 772.5 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{23}H_{25}BrN_2O_4$ $[M]^+$ 472.0998; found 472.0995.



2o

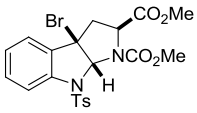
2o (EtOAc/hexane = 1:5): light yellow oil, 75.4 mg, 92% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.18 (d, *J* = 6.9 Hz, 1H), 7.13 -7.08(m, 2H), 6.27 (s, 1H), 3.74 (s, 3H), 3.63 (s, 1H), 2.99-2.54 (m, 3H), 2.28 (s, 3H), 1.54 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.9, 153.4, 141.4, 134.6, 132.6, 130.7, 126.1, 120.3, 86.2, 81.7, 62.2, 52.6, 46.0, 39.2, 28.2, 19.6. IR (KBr) 2979.7, 2360.4, 1710.3, 1448.1, 1387.8, 1157.1, 772.4 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{18}H_{23}BrN_2O_4$ $[M]^+$ 410.0841; found 410.0847.



2p

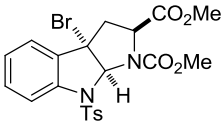
2p (EtOAc/hexane = 1:5): light yellow oil, 87.9 mg, 93% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.86 (s, 1H), 7.21 (s, 2H), 6.35 (s, 1H), 3.84-3.75 (m, 1H), 3.72 (s, 3H), 2.90-2.83 (m, 2H), 2.81-2.63 (m, 2H), 1.58 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.5, 151.8, 143.1, 131.4, 127.3, 125.0, 124.5, 120.5, 84.5, 82.8, 61.4, 52.9, 46.4, 41.2, 28.2. IR

(KBr) 2980.5, 2360.4, 2341.4, 1714.3, 1476.2, 1422.8, 1153.8, 772.1 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{17}H_{20}Br_2N_2O_4$ $[M]^+$ 473.9790; found 473.9793.



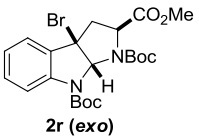
2q (*exo*)

2q (*exo*) (EtOAc/hexane = 1:5): white foam, 70.1 mg, 69% yield and **2q' (*endo*)** white foam, 23.3 mg, 23% yield. $[\alpha]^{25}_D$ = -153.5 (*c* = 1.0, $CHCl_3$); ^{1}H- NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, *J* = 7.0 Hz, 2H), 7.57 (d, *J* = 8.1 Hz, 1H), 7.40-7.30 (m, 1H), 7.27 (d, *J* = 5.8 Hz, 2H), 7.22-7.11 (m, 3H), 6.28 (s, 1H), 3.90 -3.77 (m, 3H), 3.75 (d, *J* = 5.7 Hz, 3H), 3.10 (dd, *J* = 12.7, 6.0 Hz, 1H), 2.81 (dd, *J* = 12.7, 10.5 Hz, 1H), 2.31 (s, 3H). ^{13}C- NMR (100 MHz, $CDCl_3$) δ: 170.7, 144.6, 140.5, 135.6, 133.8, 131.2, 129.5, 128.4, 126.4, 124.0, 118.9, 86.8, 59.4, 53.2, 52.8, 43.6, 21.7. IR (KBr) 2360.4, 2341.4, 1715.5, 1219.4, 1171.6, 772.6 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{21}H_{21}BrN_2O_6S$ $[M]^-$ 508.0304; found 508.0305.



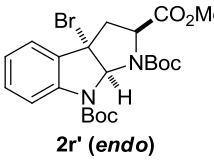
2q' (*endo*)

$[\alpha]^{25}_D$ = +86.9 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.70 (d, *J* = 8.1 Hz, 2H), 7.54 (d, *J* = 8.1 Hz, 1H), 7.36-7.29 (m, 1H), 7.24 (d, *J* = 7.8 Hz, 1H), 7.19 (d, *J* = 8.2 Hz, 2H), 7.13 (dd, *J* = 11.1, 4.0 Hz, 1H), 6.31 (s, 1H), 4.60 (d, *J* = 8.9 Hz, 1H), 3.71 (s, 3H), 3.25 (d, *J* = 13.0 Hz, 1H), 3.13 (s, 3H), 3.01 (dd, *J* = 13.0, 9.1 Hz, 1H), 2.34 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.2, 154.2, 144.3, 141.9, 136.6, 133.3, 131.2, 129.6, 127.6, 125.8, 124.6, 118.4, 87.1, 59.6, 53.2, 52.4, 44.5, 21.66. IR (KBr) 2924.5, 2360.4, 1716.0, 1447.3, 1382.6, 1220.1, 772.4 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{21}H_{21}BrN_2O_6S$ $[M]^-$ 508.0304; found 508.0308.



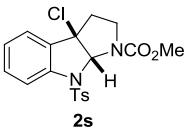
2r (*exo*)

2r (*exo*) (EtOAc/hexane = 1:5): white foam, 72.4 mg, 73% yield and **2r' (*endo*):** white foam, 19.8 mg, 20% yield. $[\alpha]^{25}_D$ = -138.0 (*c* = 1.0, $CHCl_3$); ^{1}H- NMR (400 MHz, $CDCl_3$) δ: 7.54 (s, 1H), 7.41-7.27 (m, 2H), 7.12 (t, *J* = 7.5 Hz, 1H), 6.39 (s, 1H), 3.88 (dd, *J* = 10.3, 6.3 Hz, 1H), 3.20 (dd, *J* = 12.6, 6.3 Hz, 1H), 2.81 (dd, *J* = 12.5, 10.4 Hz, 1H), 1.58 (s, 9H), 1.40 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 171.7, 152.3, 141.7, 133.0, 130.7, 124.5, 123.4, 118.7, 83.9, 82.4, 81.7, 59.7, 59.6, 52.5, 42.1, 28.4. IR (KBr) 2979.6, 1754.1, 1721.2, 1478.7, 1395.3, 1334.5, 1162.7, 754.0 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{22}H_{29}BrN_2O_6$ $[M]^+$ 496.1209; found 496.1203.



2r' (*endo*)

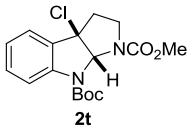
$[\alpha]^{25}_D$ = + 91.0 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.54 (s, 1H), 7.27 (dd, *J* = 7.3, 4.4 Hz, 3H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.43 (s, 1H), 4.53 (d, *J* = 8.4 Hz, 1H), 3.26 (d, *J* = 12.9 Hz, 1H), 3.11 (d, *J* = 10.0 Hz, 3H), 3.09-3.04 (m, 1H), 1.59 (s, 9H), 1.46 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.8, 152.8, 152.3, 142.6, 132.6, 130.8, 124.0, 118.2, 84.5, 82.2, 81.6, 60.5, 59.8, 52.1, 43.6, 28.5, 28.4. IR (KBr) 2978.8, 1715.2, 1478.9, 1393.2, 1160.8, 852.0, 753.8 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{22}H_{29}BrN_2O_6$ $[M]^+$ 496.1209; found 496.1195.



2s

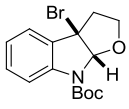
2s (EtOAc/hexane = 1:5): white foam, 65.7 mg, 81% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.63 (d, *J* = 8.1 Hz, 3H), 7.40 -7.29 (m, 1H), 7.27 (dd, *J* = 6.3, 1.0 Hz, 2H), 7.17 (t, *J* = 7.5 Hz, 3H), 6.14 (s, 1H),

3.92-3.82 (m, 1H), 3.80 (s, 3H), 2.87-2.82 (m, 1H), 2.67-2.48 (m, 2H), 2.32 (s, 3H).^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.7, 144.4, 141.8, 135.3, 132.6, 131.0, 129.6, 127.9, 126.10, 124.1, 118.2, 86.3, 73.0, 53.1, 45.9, 40.8, 21.7. IR (KBr) 2360.4, 2341.4, 1713.7, 1447.6, 1383.8, 1171.1, 771.4, 667.1cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{19}H_{19}ClN_2O_4S$ $[M]^-$ 406.0754; found 406.0753.



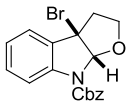
2t

2t (EtOAc/hexane = 1:5): white foam, 59.6 mg, 85% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, *J* = 8.1 Hz, 1H), 7.37-7.30 (m, 2H), 7.11 (td, *J* = 7.5, 0.8 Hz, 1H), 6.30 (s, 1H), 3.88 (dd, *J* = 11.1, 7.8 Hz, 1H), 3.74 (s, 3H), 2.89 (td, *J* = 11.7, 5.3 Hz, 1H), 2.77-2.57 (m, 2H), 1.58 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 155.0, 152.3, 142.5, 131.3, 130.79, 124.2, 123.7, 117.3, 83.5, 82.3, 72.9, 52.9, 46.3, 28.4. IR (KBr) 2979.6, 1714.28, 1479.3,1391.1, 1152.0, 754.8 cm^{-1}; HRMS (CI^-) (*m/z*) calcd. for $C_{17}H_{20}ClN_2O_4$ $[M-H]^-$ 351.1190; found 351.1121.



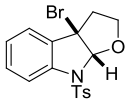
2u

2u (EtOAc/hexane = 1:5): white solid, 62.3 mg, 92% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.83 (s, 1H), 7.42 (d, *J* = 7.6 Hz, 1H), 7.35-7.22 (m, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.22 (s, 1H), 4.02 (t, *J* = 8.0 Hz, 1H), 3.63-3.40 (m, 1H), 3.04- 2.85 (m, 1H), 2.81 (dd, *J* = 12.2, 4.2 Hz, 1H), 1.62 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 152.0, 141.8, 131.9, 130.6, 124.95, 123.8, 115.0, 100.9, 82.3, 67.9, 61.8, 45.2, 28.5. IR (KBr) 2979.4, 2361.1, 1715.4, 1480.6, 1387.1, 1151.4, 1150.2, 754.0 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{15}H_{18}BrNO_3$ $[M]^+$339.0470; found 339.0467.



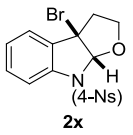
2v

2v (EtOAc/hexane = 1:5): white solid, 64.1 mg, 86% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.88 (s, 1H), 7.39 (m, 7H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.30 (s, 1H), 5.35 (dd, *J* = 28.9, 12.3 Hz, 2H), 4.24-3.89 (m, 1H), 3.51 (ddd, *J* = 11.2, 9.0, 4.8 Hz, 1H), 3.08-2.85 (m, 1H), 2.84-2.75 (m, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 152.5, 141.6, 135.9, 131.9, 130.7, 128.8, 128.4, 128.1, 125.0, 124.3, 115.1, 100.9, 68.1, 67.8, 61.8, 45.1. IR (KBr) 2360.5, 1718.5, 1482.6, 1403.0, 1358.4, 1219.0, 1051.0, 772.4 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{18}H_{16}BrNO_3$ $[M]^+$ 373.0314; found 373.0312.



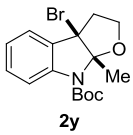
2w

2w (EtOAc/hexane = 1:5): light yellow foam, 70.7 mg, 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.79 (d, *J* = 8.3 Hz, 2H), 7.45 (d, *J* = 8.2 Hz, 1H), 7.41-7.31 (m, 1H), 7.30-7.20 (m, 3H), 7.09 (td, *J* = 7.6, 0.8 Hz, 1H), 6.24 (s, 1H), 4.02-3.98 (m, 1H), 3.45-3.39 (m, 1H), 2.94-2.77 (m, 1H), 2.72 (ddd, *J* = 12.5, 4.7, 1.1 Hz, 1H), 2.36 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 144.5, 140.6, 135.7, 132.5, 130.7, 129.8, 127.5, 125.4, 124.9, 114.3, 103.3, 68.1, 61.5, 44.8, 21.7. IR (KBr) 2360.4, 1463.3, 1358.7, 1219.3, 1169.9, 772.4, 663.7 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{17}H_{16}BrNO_3S$ $[M]^+$ 393.0034; found 393.0033.



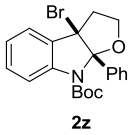
2x

2x (EtOAc/hexane = 1:5): light yellow foam, 76.1 mg, 90% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.31 (d, *J* = 8.9 Hz, 2H), 8.12 (d, *J* = 8.9 Hz, 2H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.38 (d, *J* = 7.7 Hz, 1H), 7.35-7.29 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.23 (s, 1H), 4.10- 3.90 (m, 1H), 3.38 (ddd, *J* = 11.2, 9.1, 4.8 Hz, 1H), 2.98-2.79 (m, 1H), 2.79-2.71 (m, 1H). ^{13}C- NMR (100 MHz, $CDCl_3$) δ: 150.6, 144.4, 139.8, 132.7, 131.1, 128.9, 125.8, 125.7, 124.4, 114.2, 103.4, 68.5, 61.1, 44.4. IR (KBr) 2360.5, 1531.0, 1361.3, 1349.1, 1219.3, 1173.6, 1021.8, 772.4 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{16}H_{13}BrN_2O_5S$ $[M]^+$ 423.9729; found 423.9723.



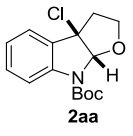
2y

2y (EtOAc/hexane = 1:10): white solid, 64.9 mg, 92% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.72 (d, *J* = 8.1 Hz, 1H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.30-7.14 (m, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 3.92 (t, *J* = 8.1 Hz, 1H), 3.42 (ddd, *J* = 11.3, 9.2, 5.0 Hz, 1H), 2.91-2.68 (m, 2H), 2.10 (s, 3H), 1.60 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 151.9, 141.9, 131.3, 130.3, 125.2, 123.4, 115.2, 103.9, 82.2, 71.5, 66.3, 46.3, 28.5, 26.5. IR (KBr) 2879.1, 2360.5, 1710.2, 1480.0, 1368.0, 1165.8, 771.8 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{16}H_{20}BrNO_3$ $[M]^+$ 353.0627; found 353.0624.



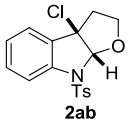
2z

2z (EtOAc/hexane = 1:10): white solid, 75.5 mg, 91% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 8.0 Hz, 1H), 7.66 (d, *J* = 5.7 Hz, 1H), 7.44 (d, *J* = 7.6 Hz, 2H), 7.37-7.28 (m, 3H), 7.22 (s, 1H), 7.10 (dd, *J* = 11.0, 4.0 Hz, 1H), 4.23 (m, 1H), 3.68 (m, 1H), 2.99-2.78 (m, 2H), 1.18 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 151.8, 142.5, 142.2, 131.1, 130.5, 128.1, 127.8, 127.1, 125.5, 123.7, 114.7, 106.8, 81.8, 72.5, 67.0, 46.9, 27.8. IR (KBr) 2979.6, 2360.4, 2341.4, 1707.8, 1480.0, 1367.0, 1219.3,1164.6, 1048.0, 772.4 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{21}H_{22}BrNO_3$ $[M]^+$ 415.0783; found 415.0787.



2aa

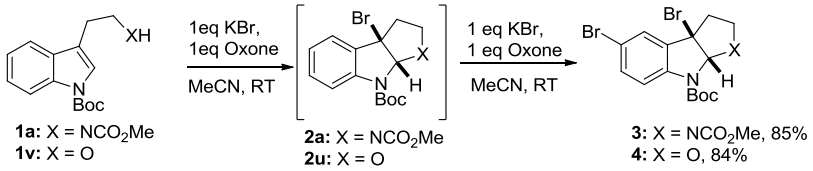
2aa (EtOAc/hexane = 1:5): white film, 47.6 mg, 80% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.84 (s, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.8 Hz, 1H), 7.08 (td, *J* = 7.6, 0.8 Hz, 1H), 6.08 (s, 1H), 4.13-4.00 (m, 1H), 3.57-3.51 (ddd, *J* = 11.1, 9.3, 4.8 Hz, 1H), 2.83-2.75 (m, 1H), 2.69 (ddd, *J* = 12.3, 4.7, 1.3 Hz, 1H), 1.60 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 152.0, 142.2, 130.7, 124.6, 123.8, 115.0, 100.2, 82.3, 72.8, 68.0, 44.2, 28.5. IR (KBr) 2360.4, 1715.0,1636.1, 1482.1, 1387.7,1151.8, 1057.5, 772.4 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{15}H_{18}ClNO_3$ $[M]^+$ 295.0975; found 295.0963.



2ab

2ab (EtOAc/hexane = 1:5): light yellow foam, 54.4 mg, 78% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.79 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.2 Hz, 1H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.30 (td, *J* = 8.1, 1.1 Hz, 1H), 7.24 (d, *J* = 8.3 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.12 (s, 1H), 4.16-4.04 (m, 1H), 3.48 (ddd, *J* = 11.2, 9.4, 4.8 Hz, 1H), 2.81 -2.67 (m, 1H), 2.63 (ddd, *J* = 12.4, 4.8, 1.5 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 144.5, 141.2, 135.8, 131.5, 130.9, 129.8, 127.5, 125.0, 124.9, 114.3, 102.7, 73.0, 68.2, 43.9, 21.7. IR (KBr) 2360.5, 1601.3, 1465.6, 1359.2,

1219.5, 1170.5, 1037.7, 772.5 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{17}H_{16}ClNO_3S$ $[M]^+$ 349.0539; found 349.0530.

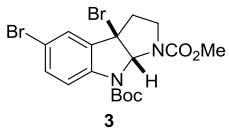


1a: X = NCO_2Me
1v: X = O

2a: X = NCO_2Me
2u: X = O

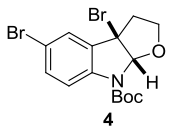
3: X = NCO_2Me, 85%
4: X = O, 84%

To the solution of **1a** or **1v** (0.20 mmol) and KBr (23.8 mg, 0.20 mmol) in MeCN (4 mL) was added oxone (122.8 mg, 0.20 mmol) at room temperature, and stirred for 4 h. Then, to the reaction mixture were added KBr (23.8 mg, 0.20 mmol) and oxone (122.8 mg, 0.20 mmol), and stirred for 5 h. Saturated Na_2SO_3 aqueous solution (10 mL) was added to quench the reaction. The organic layer was collected and the aqueous phase was extracted with EtOAc (15 mL × 3). The combined fractions were washed with brine and dried over Na_2SO_4, concentrated under reduced pressure. The residue was purified by silica gel column chromatography to give the desired products **3** and **4**, respectively.



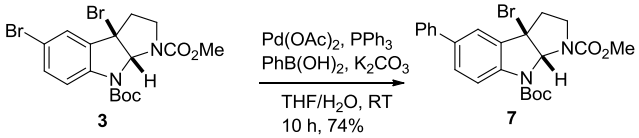
3

3 (EtOAc/hexane = 1:5): light yellow oil, 79.1 mg, 85% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.52 (d, *J* = 8.3 Hz, 1H), 7.45 (s, 1H), 7.37 (d, *J* = 8.7 Hz, 1H), 6.34 (s, 1H), 3.77 (dd, *J* = 10.1, 8.4 Hz, 1H), 3.70 (s, 3H), 2.90-2.83 (m, 1H), 2.80-2.62 (m, 2H), 1.55 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.6, 151.8, 141.1, 134.4, 133.4, 126.8, 118.8, 116.4, 84.3, 82.5, 61.0, 52.8, 46.3, 41.2, 28.2. IR (KBr) 2980.3, 1714.8, 1473.9, 1446.8, 1389.0, 1368.7, 1153.4, 755.6 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{17}H_{20}Br_2N_2O_4$ $[M]^+$ 473.9790; found 473.9781.



4

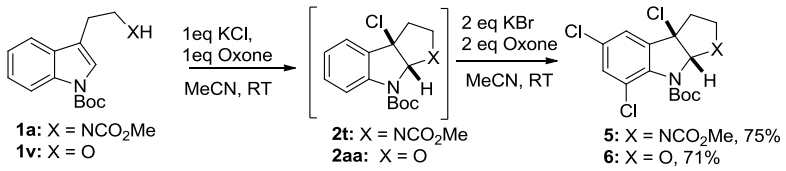
4 (EtOAc/hexane = 1:10): light yellow oil, 69.6 mg, 84% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.69 (s, 1H), 7.48 (d, *J* = 1.8 Hz, 1H), 7.35 (dd, *J* = 8.6, 1.9 Hz, 1H), 6.14 (s, 1H), 3.97 (t, *J* = 7.7 Hz, 1H), 3.62-3.29 (m, 1H), 2.97-2.77 (m, 1H), 2.77-2.60 (m, 1H), 1.56 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 151.5, 140.8, 133.9, 133.3, 127.9, 116.4, 115.8, 101.0, 82.46, 67.7, 60.4, 44.9, 28.3. IR (KBr) 2980.7, 2360.5, 1718.0, 1476.4, 1382.4, 1337.7, 1298.9, 1053.6 cm^{-1}; HRMS (CI^+) (*m/z*) calcd. for $C_{15}H_{17}Br_2NO_3$ $[M]^+$ 416.9575; found 416.9572.



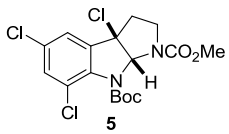
3 **7**

To a solution of **3** (60 mg, 0.13 mmol) in THF (2 mL) were sequentially added $PhB(OH)_2$ (30.8 mg, 0.26 mmol), K_2CO_3 (19.3 mg, 0.14 mmol), $Pd(OAc)_2$ (6.72 mg, 0.03 mmol), PPh_3 (7.86 mg, 0.03 mmol) and H_2O (50 μL) at room temperature. After bubbling with argon for 15 min, the resulting mixture was stirred at that temperature for 10 h. The mixture passed through a celite plug and washed with CH_2Cl_2 (10 mL × 3). The crude product was purified by silica gel column chromatography

(EtOAc/hexane = 1:10) to give the desired product **7** as light yellow foam (45.4 mg, 74% yield). ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.71 (d, *J* = 7.8 Hz, 1H), 7.56 (t, *J* = 8.1 Hz, 4H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 3.04-2.84 (m, 2H), 2.86-2.67 (m, 1H), 1.60 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 154.9, 152.2, 141.4, 140.4, 137.8, 133.1, 129.7, 129.0, 127.4, 127.0, 122.4, 117.6, 84.5, 82.4, 62.3, 52.9, 46.4, 41.4, 28.4. IR (KBr) 2926.0, 1717.1, 1477.9, 1389.6, 1153.0 cm^{-1}; HRMS (CI^{+}) (*m/z*) calcd. for $C_{23}H_{25}BrN_2O_4$ [M]$^{+}$ 472.0998; found 472.0992.

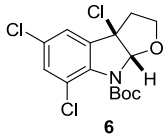


To the solution of **1a** or **1v** (0.20 mmol) and KBr (23.8 mg, 0.20 mmol) in MeCN (4 mL) was added oxone (122.8 mg, 0.20 mmol) at room temperature. The reaction mixture was stirred for 6 h before addition of KBr (47.6 mg, 0.40 mmol) and oxone (245.6 mg, 0.40 mmol). After stirring for additional 10 h. Saturated Na_2SO_3 aqueous solution (10 mL) was added to quench the reaction. The organic layer was collected and the aqueous layer was extracted with EtOAc (15 mL × 3). The combined extracts were washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography to give the desired products **5** and **6**, respectively.



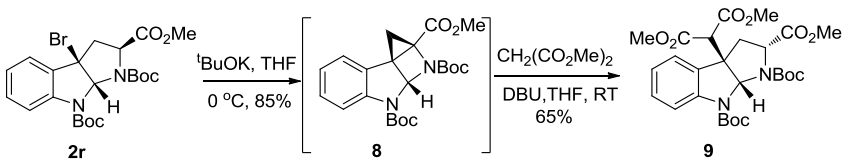
5

5 (EtOAc/hexane = 1:5): light yellow oil, 62.8 mg, 75% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.37 (d, *J* = 2.0 Hz, 1H), 7.26 (d, *J* = 1.6 Hz, 2H), 6.18 (s, 1H), 3.77 (s, 4H), 2.85 (m, 1H), 2.73-2.57 (m, 3H), 1.56 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 155.3, 152.3, 139.7, 137.5, 131.9, 131.7, 127.3, 122.0, 86.4, 83.0, 72.0, 53.0, 46.0, 38.0, 28.2. IR (KBr) 2957.3, 1722.8, 1453.0, 1384.8, 1298.9, 1155.2, 862.2 cm^{-1}; HRMS (CI^{+}) (*m/z*) calcd. for $C_{17}H_{19}Cl_3N_2O_4$ [M]$^{+}$ 420.0410; found 420.0412.

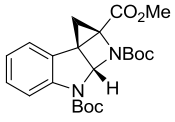


6

6 (EtOAc/hexane = 1:10): light yellow oil, 49.9 mg, 71% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.35 (d, *J* = 2.0 Hz, 1H), 7.29 (d, *J* = 2.0 Hz, 1H), 6.02 (s, 1H), 4.09-4.05 (m, 1H), 3.56-3.49 (m, *J* = 10.4, 9.3, 5.2 Hz, 1H), 2.80-2.72 (m, 1H), 2.66-2.61 (m, 1H), 1.55 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 151.6, 138.5, 137.2, 132.0, 131.0, 124.5, 123.0, 103.0, 83.4, 71.5, 68.0, 42.5, 28.1. IR (KBr) 2982.5, 2360.3, 1730.7, 1458.4,1369.9, 1331.3, 1219.0, 861.9, 61.3 cm^{-1}; HRMS (CI^{+}) (*m/z*) calcd. for $C_{15}H_{16}Cl_3NO_3$[M]$^{+}$ 363.0196; found 363.0192.

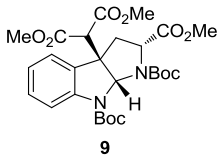


To a solution of bromide **2r** (100 mg, 0.20 mmol) in THF (2 mL) at 0 °C was added KO^{t}Bu (0.24 mL, 1.0 M solution in THF, 0.24 mmol) dropwise over 1 h. The reaction was quenched with aq. NH_4Cl and the resulting mixture was extracted with CH_2Cl_2 (15 mL × 3). The combined extracts were washed with brine and dried over Na_2SO_4. The organic phase was concentrated under reduced pressure and the crude product was purified by silica gel column chromatography (EtOAc/hexane = 1:10) to give the desired cyclopropane **8** as white foam (70.7 mg, 85% yield). To a solution of cyclopropane **8** in THF (2 mL) were added dimethyl malonate (45.7 μL, 0.40 mmol) and DBU (35.8 μL, 0.24 mmol). The reaction mixture was stirred for 10 h at room temperature. The reaction mixture passed through a plug of silica gel and concentrated under reduced pressure. The residue was purified by flash chromatography (EtOAc/hexane = 1:10) on silica gel to afford **9** (55.5 mg, 60% yield) as white foam.



8

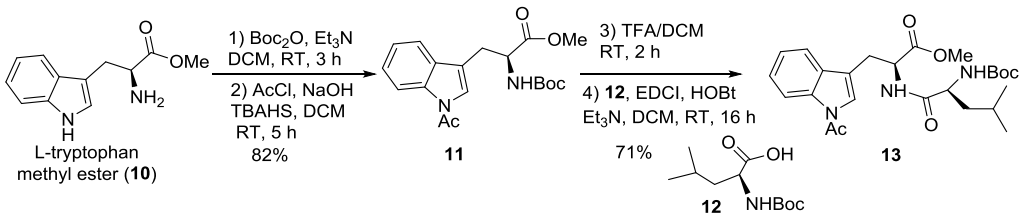
$[\alpha]^{25}_D$ = +290.0 (*c* = 1.0, $CHCl_3$). ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.77 (s, 1H), 7.17 (t, *J* = 7.5 Hz, 1H), 6.92 (m, 2H), 5.84 (s, 1H), 3.61 (s, 3H), 2.80 (d, *J* = 7.0 Hz, 1H), 2.22 (d, *J* = 6.9 Hz, 1H), 1.58 (s, 9H), 1.47 (s, 9H).^{13}C-NMR (100 MHz, $CDCl_3$) δ: 166.98, 156.4, 152.2, 147.8, 129.0, 122.7, 121.7, 115.1, 82.6, 81.3, 79.8, 56.49, 52.3, 40.0, 28.5, 28.2, 25.6. IR (KBr) 2979.2, 1731.8, 1456.2, 1370.9, 1255.9, 1157.9 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{22}H_{28}N_2O_6[M]^+$ 416.1947; found 416.1958.



9

$[\alpha]^{25}_D$ = -30.0 (*c* = 1.0, $CHCl_3$). ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.52 (s, 1.2H), 7.30-7.14 (m, 2.0H), 7.09 (d, *J* = 7.5 Hz, 0.5 H), 7.02 (t, *J* = 7.5 Hz, 0.5 H), 6.94 (t, *J* = 7.4 Hz, 1.0H), 6.50 (s, 0.7H), 6.36 (s, 0.3H), 4.61 (d, *J* = 8.0 Hz, 1.0H), 3.90 (dd, *J* = 9.9, 6.8 Hz, 1.0H), 3.76-3.72 (m, 1.0H), 3.71 (d, *J* = 4.2 Hz, 1.0H), 3.67 (s, 3.0H), 3.62 (s, 3.0H), 3.12 (s, 1.0H), 2.89-2.71 (m,2.3H), 2.56-2.40 (m, 0.5H), 1.73(s, 2.2H), 1.58 (s, 9.0H), 1.46 (s, 9.0H).1.38(s, 4.0H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 171.8, 167.2, 167.1, 152.4, 143.4, 142.4, 131.3, 129.6, 124.4, 123.1, 81.6, 79.8, 59.3, 56.6, 55.0, 52.9, 52.8, 52.8, 52.7, 52.3, 52.0, 37.3, 36.2, 28.5, 28.4. IR (KBr) 2955.6, 1739.1, 1481.7, 1395.6, 1159.5 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{27}H_{36}N_2O_{10}$ $[M]^+$ 548.2370; found 548.2366.

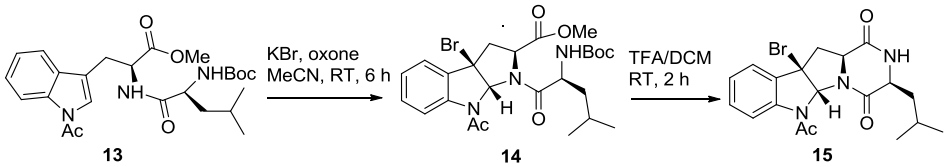
Total Synthesis of Protubonines A and B



To a solution of L-tryptophan methyl ester **10** (10 g, 39.4 mmol) in CH_2Cl_2 (200 mL) were added Et_3N (10.9 mL, 78.8 mmol) and Boc_2O (9.09 mL, 39.4 mmol). The reaction mixture was stirred at room temperature for 3 h. The reaction was quenched with water and extracted with EtOAc (150 mL × 3), then the combined organic layers

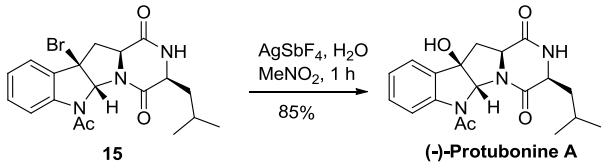
were washed by brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was used directly for the next step without further purification. BuN_4HSO_4 (TBAHS, 1.34 g, 3.94 mmol) and finely grounded NaOH (7.88 g, 197 mmol) were added to a solution of the above methyl (2-(1H-indol-3-yl)ethyl)carbamate in CH_2Cl_2 (200 mL) at room temperature and the reaction mixture was stirred for 10 min. AcCl (8.38 mL, 118.2 mmol) was added via syringe. The mixture was stirred for 5 h at room temperature and quenched with addition of 100 mL H_2O. Then the organic layer was collected and the aqueous layer was extracted with EtOAc (100 mL × 3). The combined extracts were washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (EtOAc/hexane = 1:5) to give the known product **11** as white solid (11.8 g, 82% yield).

To a stirred solution of **11** (3 g, 8.24 mmol) in CH_2Cl_2 (80 mL) was added TFA (16 mL) at room temperature. The reaction mixture was stirred for 2 h and then concentrated *in vacuo*. The resulting viscous residue was dissolved in CH_2Cl_2 (100 mL) and cooled to 0 °C. To this solution were added Et_3N (5.13 mL, 37.1 mmol), HOBt•H_2O (1.67 g, 12.4 mmol), N-Boc-L-Leucine **12** (3.81 g, 16.5 mmol), and EDCI•HCl (2.37 g, 12.4 mmol). The reaction mixture was stirred vigorously for 16 h before quenched with addition of 1N HCl. The organic layer was collected and the aqueous layer was extracted with CH_2Cl_2 (100 mL × 3). The combined organics were then washed with saturated aqueous $NaHCO_3$, and the aqueous layer was extracted again with CH_2Cl_2 (100 mL × 3). The combined extracts were washed by brine and dried over Na_2SO_4. The organic phase was concentrated under reduced pressure and the crude product was purified by column chromatography on silica gel (CH_2Cl_2/MeOH = 20:1) to give the desired product **13** (2.77 g, 71% yield) as white solid. $[\alpha]^{25}_D = +122.3$ ($c = 0.9$, $CHCl_3$). ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.38 (d, $J = 7.4$ Hz, 1H), 7.44 (d, $J = 8.1$ Hz, 2H), 7.35-7.18 (m, 3H), 6.96 (d, $J = 7.4$ Hz, 1H), 5.12 (d, $J = 7.2$ Hz, 1H), 5.00-4.84 (m, 1H), 4.12 (d, $J = 4.9$ Hz, 1H), 3.65 (s, 3H), 3.24 (d, $J = 4.5$ Hz, 2H), 2.56 (s, 3H), 1.73-1.52 (m, 2H), 1.53 -1.43 (m, 1H), 1.39 (s, 9H), 0.90 (t, $J = 5.7$ Hz, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 172.7, 171.7, 168.9, 155.7, 135.7, 130.4, 125.3, 124.4, 123.5, 118.6, 116.7, 116.4, 79.9, 53.4, 52.5, 51.9, 41.0, 28.2, 27.3, 24.7, 23.9, 22.9, 21.9. IR (KBr) 2957.7, 2360.5, 1746.0, 1698.2, 1519.9, 1219.6, 1169.0, 772.1 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{25}H_{35}N_3O_6$ $[M]^+$ 473.2526; found 473.2526.



To a solution of **13** (2 g, 4.23 mmol) and KBr (503 mg, 4.23 mmol) in MeCN (40 mL) was added oxone (2.6 g, 4.23 mmol) at room temperature and the reaction mixture was stirred for 6 h. Saturated Na_2SO_3 aqueous solution was added to quench the reaction. The organic layer was collected and aqueous layer was extracted with EtOAc (50 mL × 3). The combined extracts were washed by brine and dried over

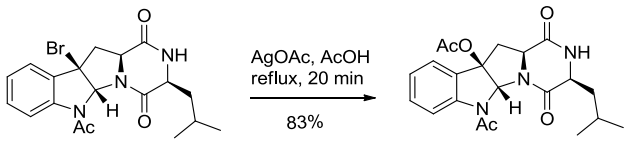
Na_2SO_4, concentrated under reduced pressure. The residue was purified by silica gel column chromatography (CH_2Cl_2/MeOH = 20:1) to give the product **14** (*exo*/*endo* 3.5： 1). To a stirred solution of **14** in CH_2Cl_2 (40 mL) was added TFA (6 mL) at room temperature. The reaction mixture was stirred for 2 h before it was quenched with saturated aq. $NaHCO_3$ solution. The organic layer was collected and the aqueous layer was extracted with CH_2Cl_2 (2 x 40 mL). The combined extracts were washed with brine and dried over Na_2SO_4, concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (CH_2Cl_2/MeOH = 20:1) to give the desired product **15** (1.07 g, 61% yield) as white solid. m.p. 119–120 °C. $[\alpha]^{25}_D$ = -182.3 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.00 (d, *J* = 7.8 Hz, 1H), 7.44 (d, *J* = 7.6 Hz, 1H), 7.36 (dd, *J* = 11.4, 4.2 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 6.69 (s, 1H), 6.25 (s, 1H), 3.99-3.82 (m, 2H), 3.35 (dd, *J* = 12.9, 5.7 Hz, 1H), 2.94 (dd, *J* = 12.7, 11.6 Hz, 1H), 2.04-1.95 (m, 1H), 1.78-1.71 (m, 1H), 1.56-1.49 (m, 1H), 0.99 (d, *J* = 6.5 Hz, 3H), 0.90 (d, *J* = 6.5 Hz, 3H).^{13}C-NMR (100 MHz, $CDCl_3$) δ: 170.7, 168.0, 166.4, 142.0, 131.6, 131.2, 125.8, 123.4, 119.7, 85.1, 59.2, 58.8, 53.2, 43.3, 38.6, 24.4, 23.6, 23.4, 21.2. IR (KBr) 2959.3, 2360.3, 2341.1, 1682.1, 1406.2, 1219.3, 772.3cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{19}H_{23}BrN_3O_3$ $[M+H]^+$ 420.0923; found 420.0918.



15 **(-)-Protubonine A**

To a round-bottomed flask were sequentially added **15** (50 mg, 0.12 mmol), nitromethane (2 mL) and de-ionized water (22 μL, 1.20 mmol) at 23 °C. The flask was opened to air and a solution of silver(I) hexafluoroantimonate (61.7 mg, 0.18 mmol) in nitromethane (1 mL) was added slowly by glass pipette over 2 min. The reaction mixture was stirred for 1 h before addition of brine and dichloromethane. The organic layer was collected and the aqueous layer was extracted with CH_2Cl_2 (3 × 10 mL). The organic layers were combined, dried over Na_2SO_4, filtered and concentrated under reduced pressure to give a brown residue. This residue was purified by flash column chromatography on silica gel (CH_2Cl_2/MeOH = 20:1) to furnish protubonine A as a white solid (36.5 mg, 85% yield). m.p. 149–150 °C; $[\alpha]^{25}_D$ = –145.0 (*c* = 1.0, CH_3OH); (When the concentration is 12 mg/mL) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.90 (d, *J* = 8.1 Hz, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.36-7.28 (m, 1H), 7.13 (t, *J* = 7.4 Hz, 1H), 5.98 (s, 1H), 5.86 (s, 1H), 3.87 (dd, *J* = 9.6, 3.3 Hz, 1H), 3.71 (dd, *J* = 11.8, 5.6 Hz, 1H), 2.83 (dd, *J* = 12.6, 5.8 Hz, 1H), 2.60 (s, 3H), 2.54 (t, *J* = 12.2 Hz, 1H), 1.97 (ddd, *J* = 14.0, 9.9, 3.7 Hz, 1H), 1.74-1.69 (m, 2H), 1.56-1.49 (m, 2H), 0.99 (d, *J* = 6.5 Hz, 3H), 0.91 (d, *J* = 6.5 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 171.8, 168.5, 166.6, 143.2, 131.9, 131.0, 125.5, 123.4, 119.8, 83.7, 82.2, 58.8, 53.1, 39.0, 24.5, 23.8, 23.5, 21.2. (When the concentration is 6 mg/mL) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 8.1 Hz, 1H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 1H), 7.17 (t, *J* = 7.5 Hz, 1H), 5.91 (s, 1H), 5.84 (s, 1H), 3.91 (dd, J = 9.6, 3.3 Hz, 1H), 3.80 (dd, J = 11.8, 5.6 Hz, 1H), 2.88 (dd, *J*

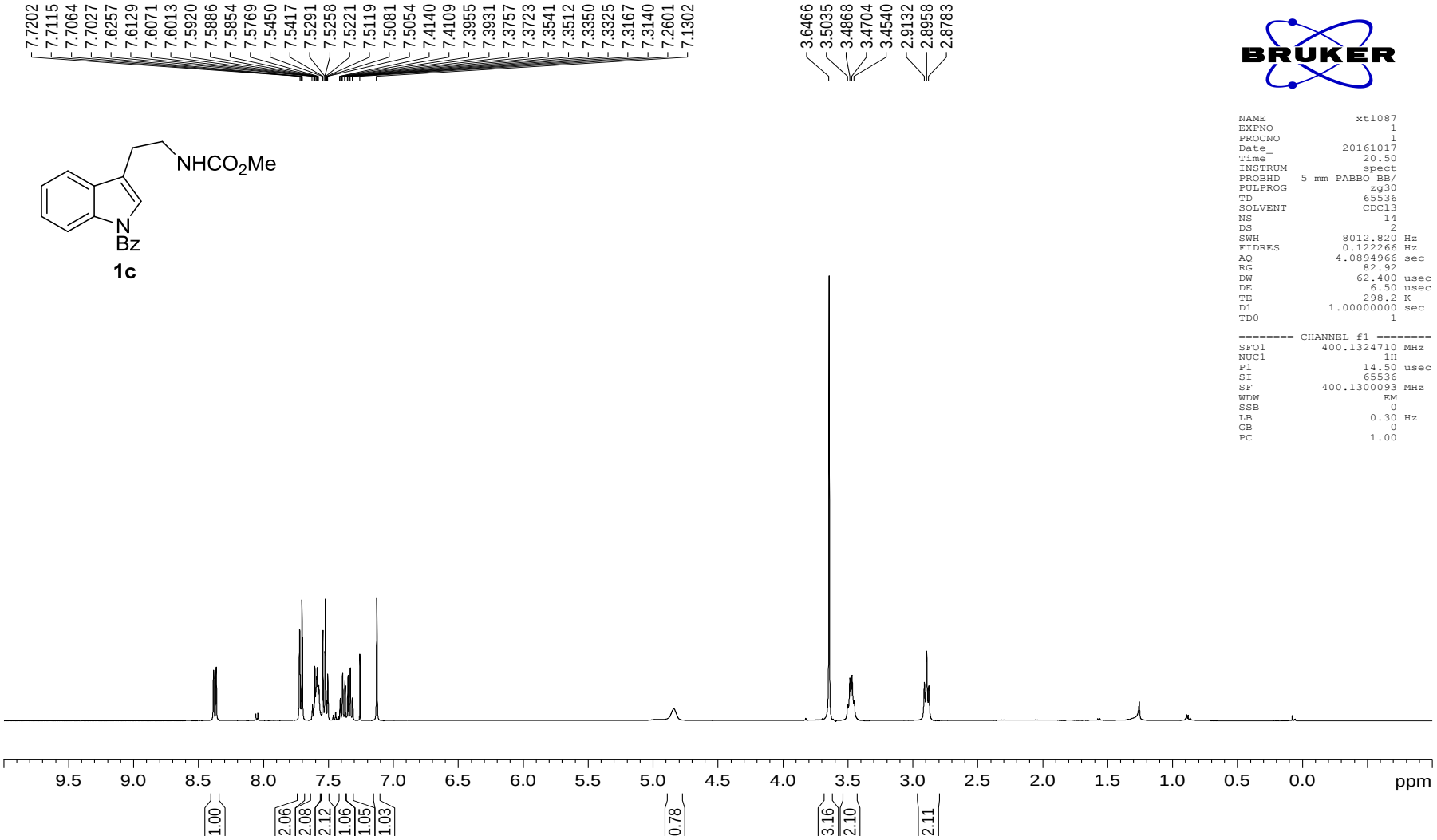
= 12.6, 5.7 Hz, 1H), 2.62 (s, 3H), 2.57 (t, J = 12.2 Hz, 1H), 1.99 (ddd, *J* = 14.1, 10.0, 3.7 Hz, 1H), 1.76-1.65 (m, 1H), 1.58-1.48 (m, 1H), 1.00 (d, *J* = 6.5 Hz, 3H), 0.91 (d, *J* = 6.5 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 168.3, 166.6, 143.3, 131.7, 131.2, 125.5, 123.3, 119.8, 83.8, 82.2, 58.9, 53.1, 39.1, 39.0, 24.6, 23.8, 23.5, 21.2. IR (KBr) 2957.1, 1678.5, 1479.1, 1380.7, 1230.6, 1053.3, 757.1 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{19}H_{24}N_3O_4$ $[M+H]^+$ 358.1767; found 358.1753.

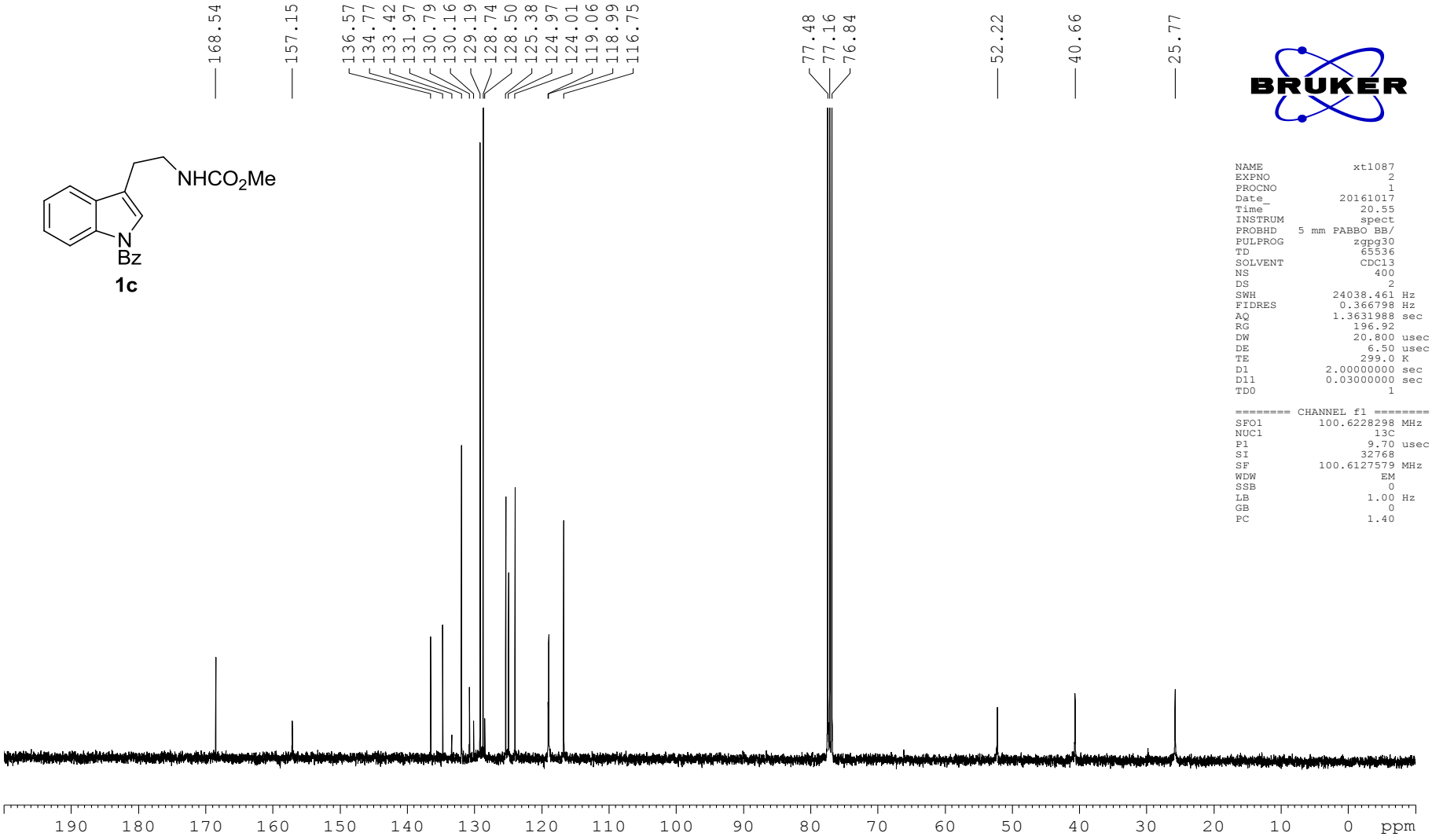


15 **(-)-Protubonine B**

To a solution of **15** (50 mg, 0.12 mmol) in AcOH (1 mL) was added silver acetate (22.1 mg, 0.13 mmol), and the reaction mixture was heated at reflux for 20 min. After the mixture was cooled to room temperature, Et_2O was added. The mixture was filtered and washed with Et_2O. The organic volatile solvents were removed under reduced pressure, and the residue was purified by column chromatography on silica gel (CH_2Cl_2/MeOH = 20:1) to afford protubonine B as a white solid (39.8 mg, 83% yield). m.p. 117–118 °C; $[\alpha]^{25}_D$ = –198.0 (*c* = 1.0, CH_3OH). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.04 (d, *J* = 7.9 Hz, 1H), 7.53 (d, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.17 (t, *J* = 7.5 Hz, 1H), 6.43 (s, 1H), 6.38 (s, 1H), 4.00 (dd, *J* = 9.5, 3.3 Hz, 1H), 3.92 (dd, *J* = 11.9, 5.6 Hz, 1H), 3.24 (dd, *J* = 12.6, 5.7 Hz, 1H), 2.68 (t, *J* = 12.3 Hz, 1H), 2.64 (s, 3H), 2.03 (s, 3H), 2.02-1.94 (m, 1H), 1.79-1.72 (m, 1H), 1.61-1.54 (m, 1H), 0.99 (d, *J* = 6.5 Hz, 3H), 0.91 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 171.4, 169.6, 167.8, 166.2, 144.6, 131.4, 127.9, 125.2, 125.0, 119.3, 88.0, 80.1, 58.1, 53.3, 39.5, 39.4, 24.5, 23.7, 23.4, 21.4, 21.2. IR (KBr) 2957.0, 1749.1, 1679.8, 1478.9, 1379.8, 1230.6, 1053.3 cm^{-1}; HRMS (CI$^+$) (*m/z*) calcd. for $C_{21}H_{26}N_3O_5$ $[M+H]^+$ 400.1872; found 400.1867.

Copies of ^{1}H- and ^{13}C-NMR Spectra

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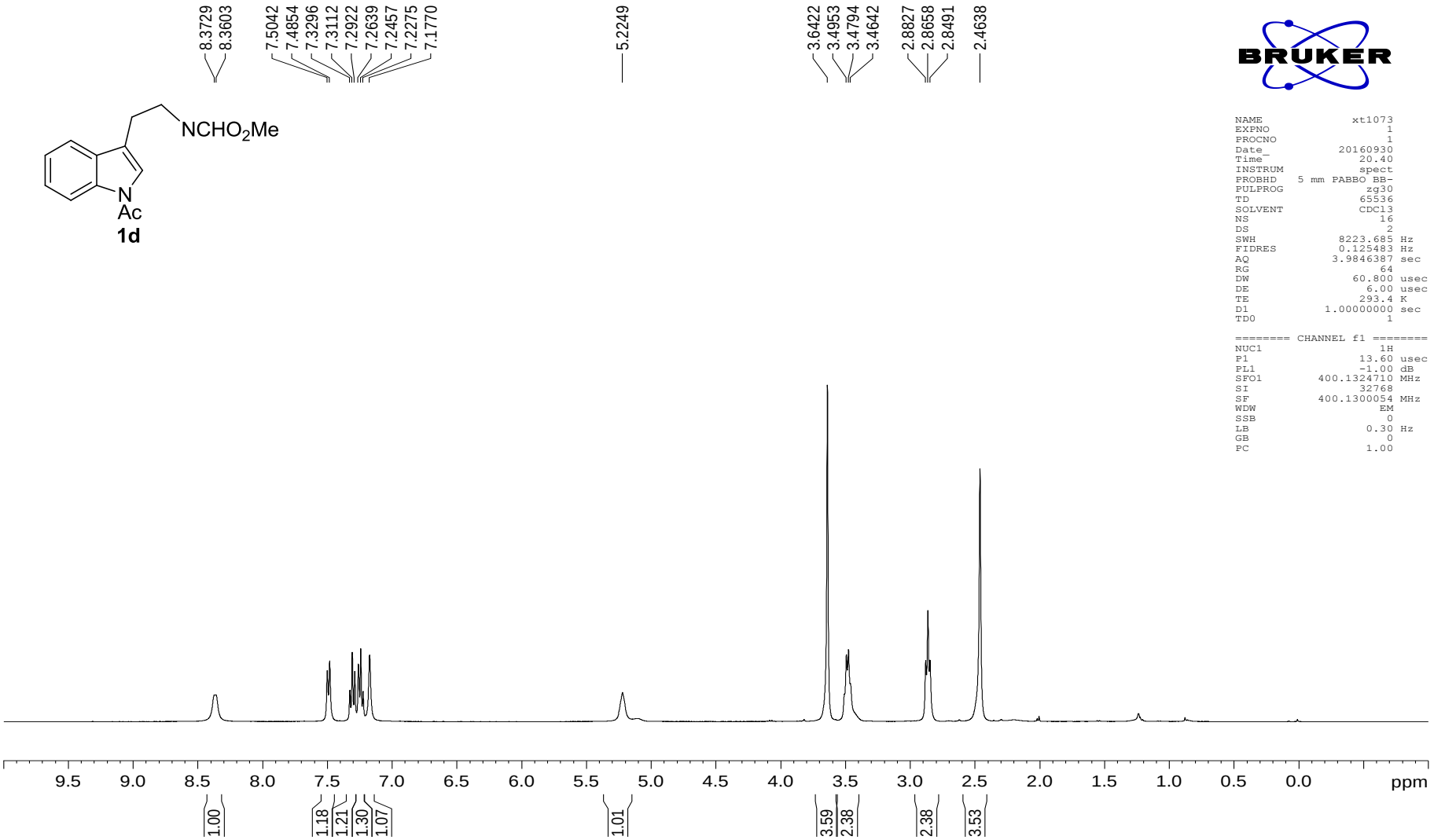
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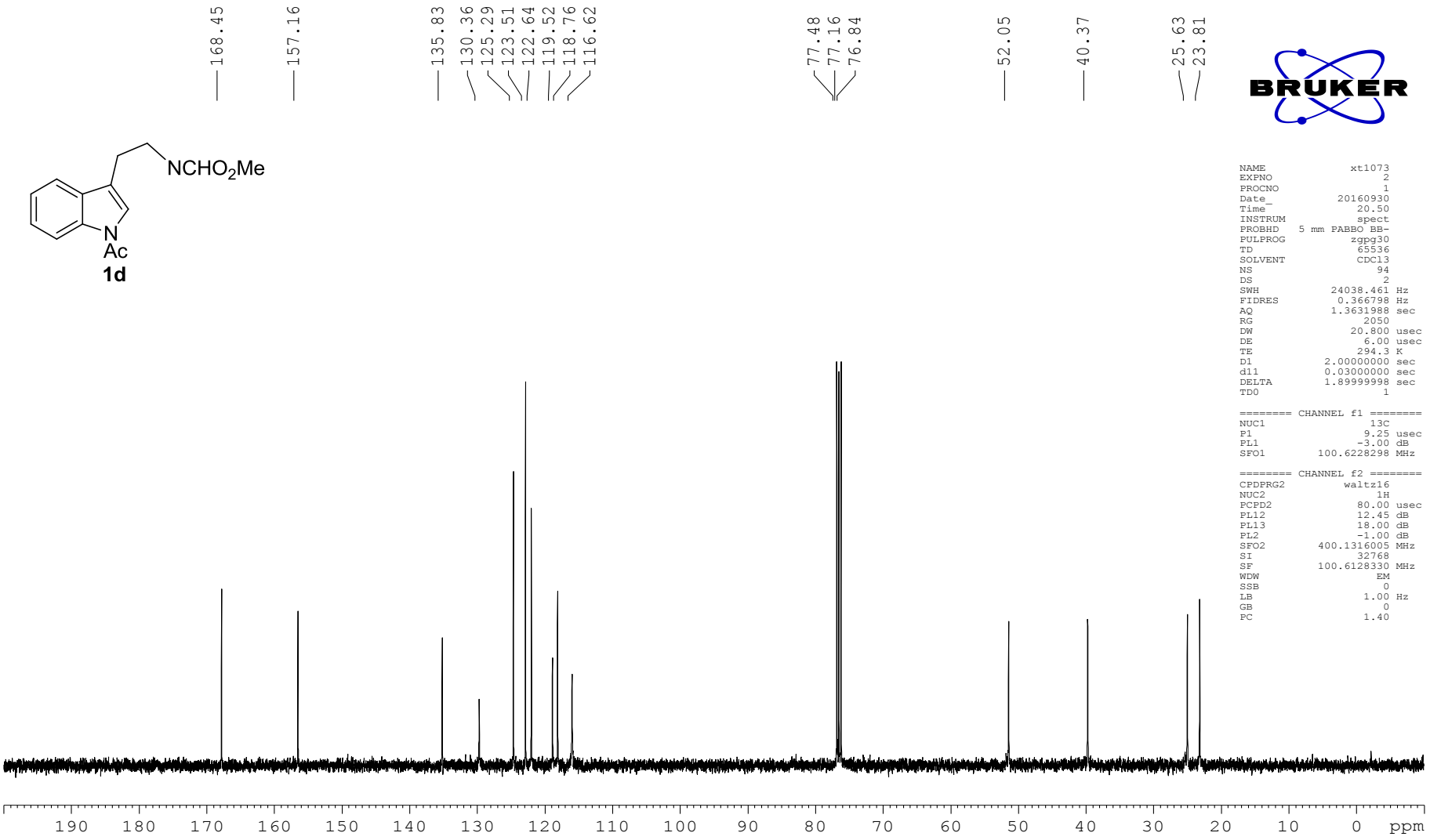
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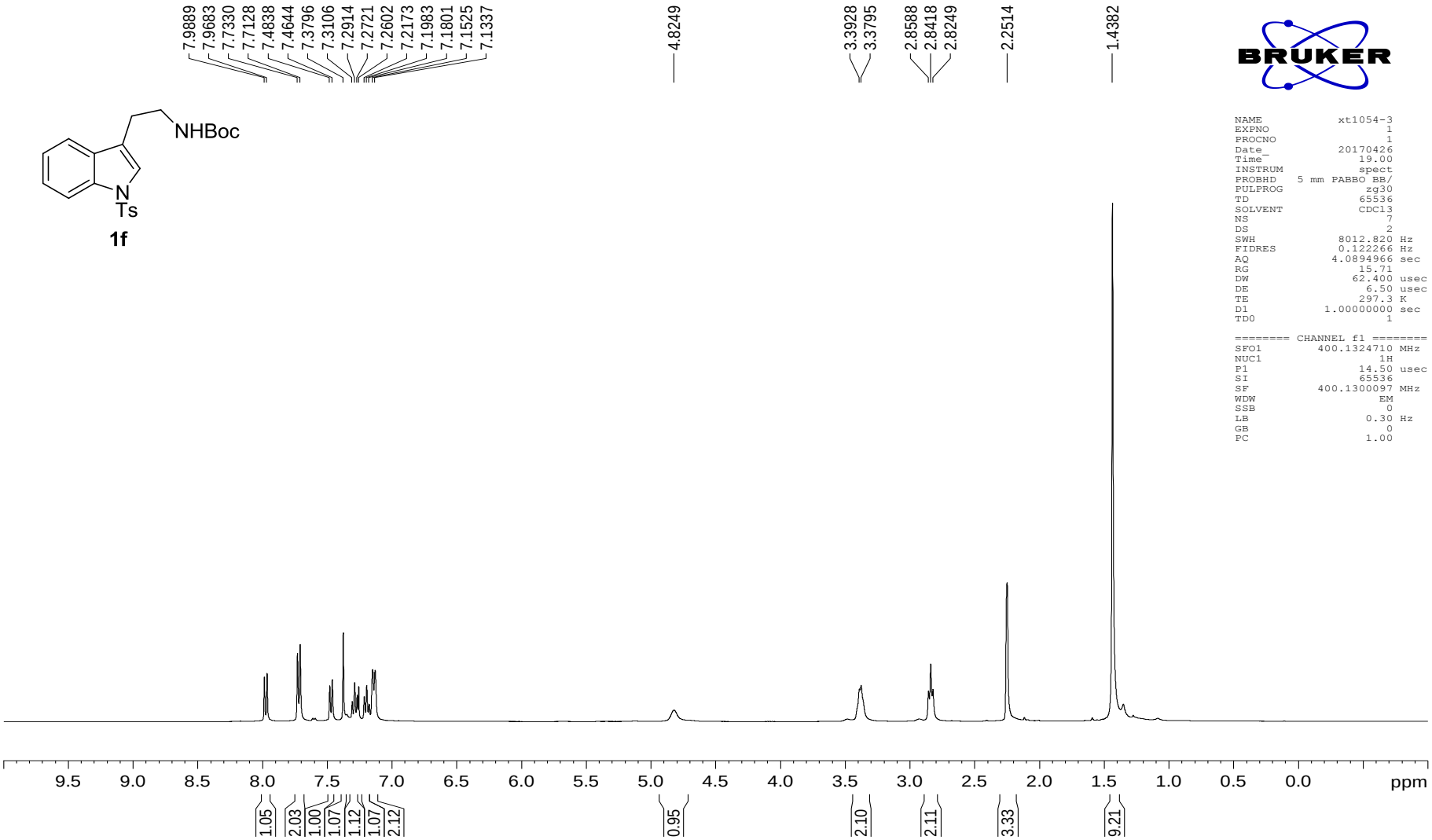
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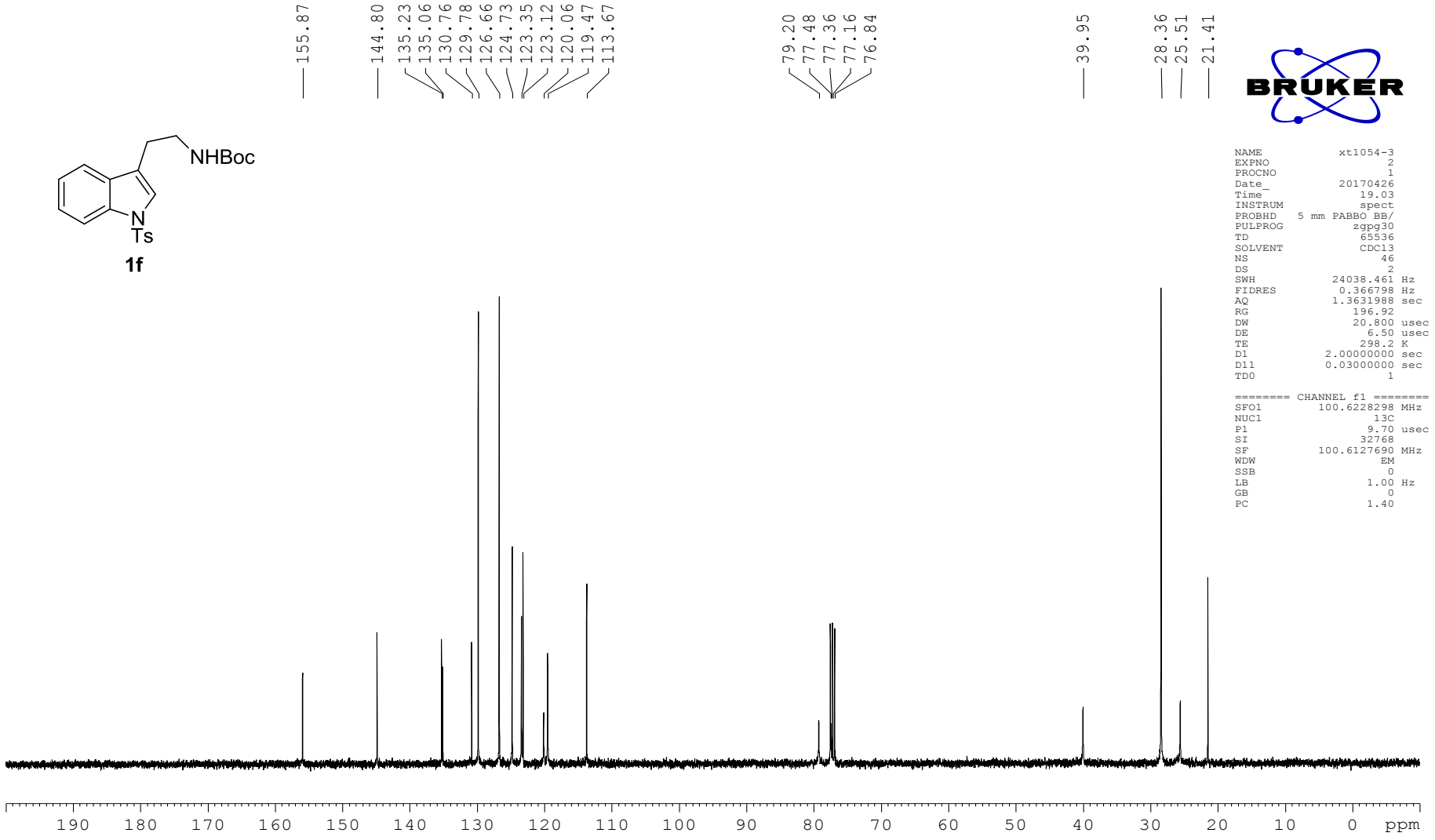
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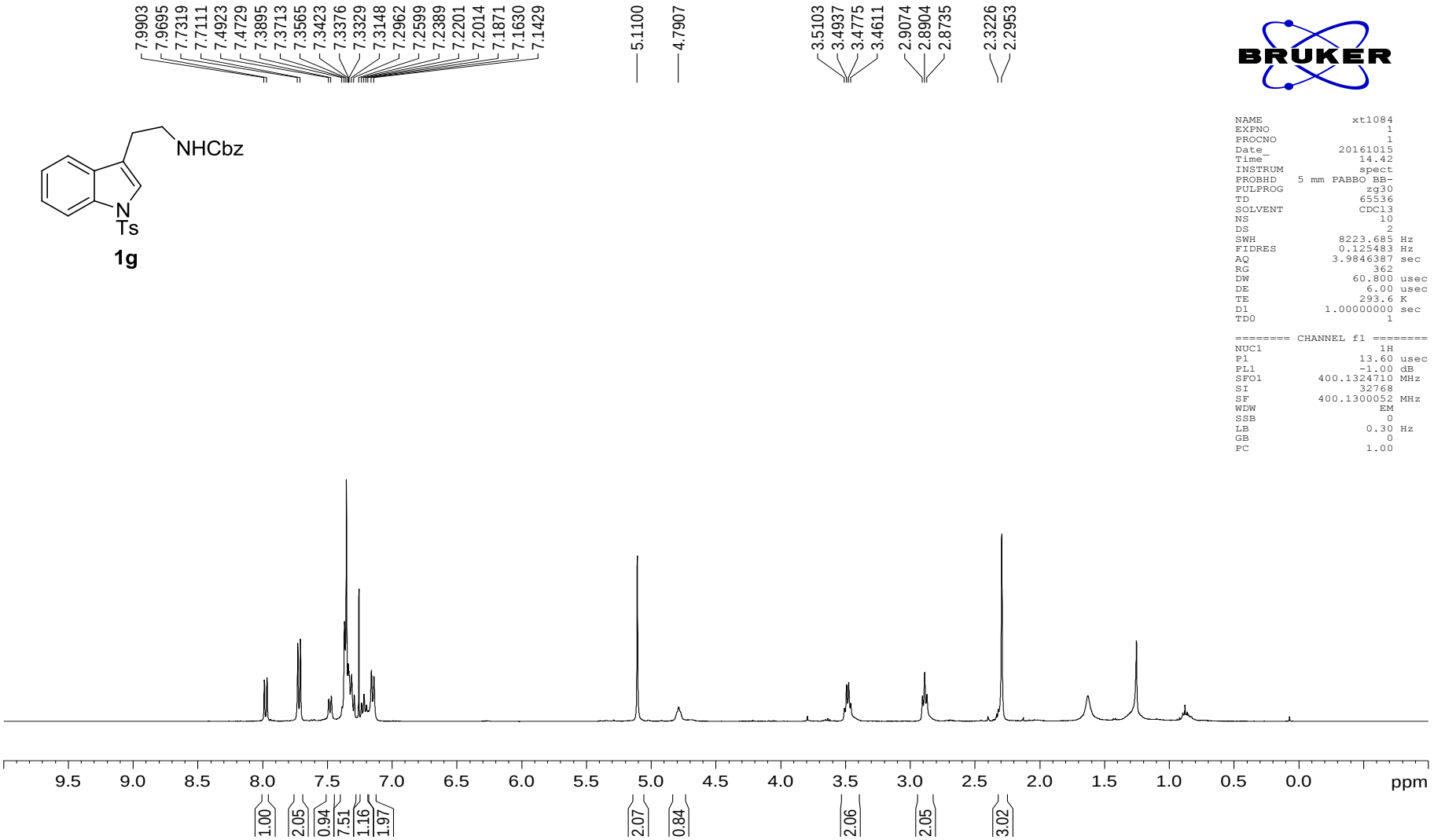
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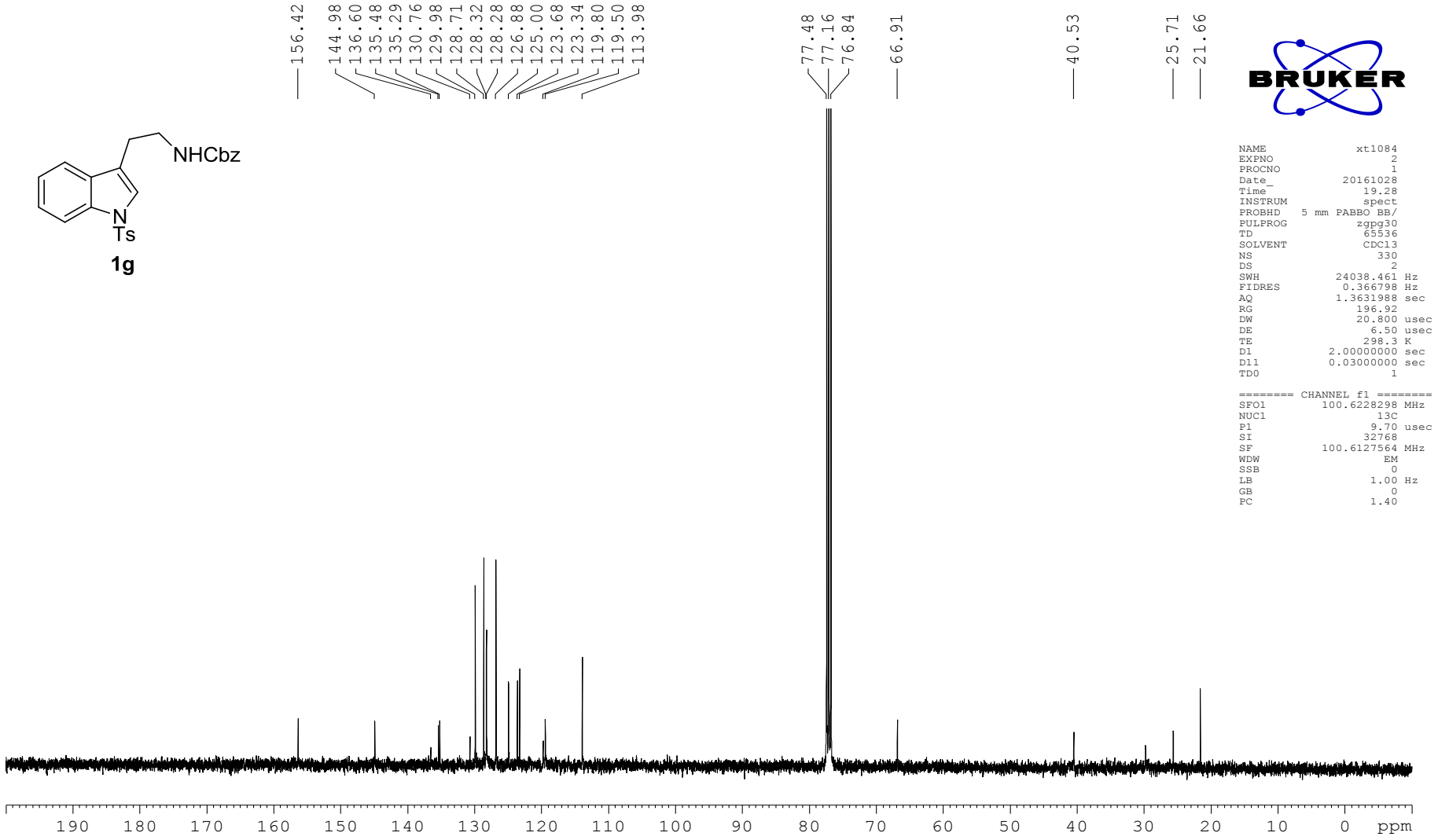
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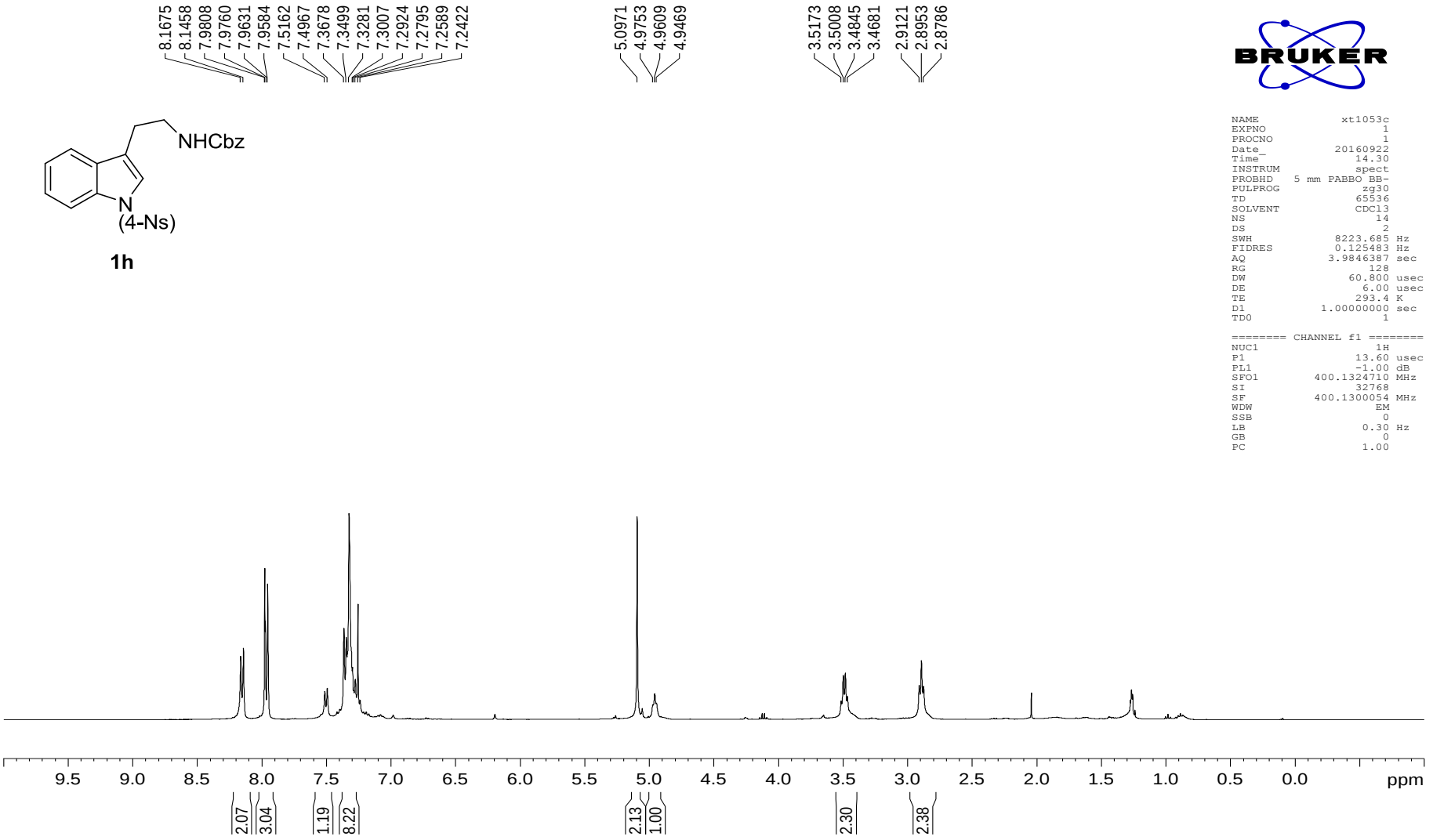
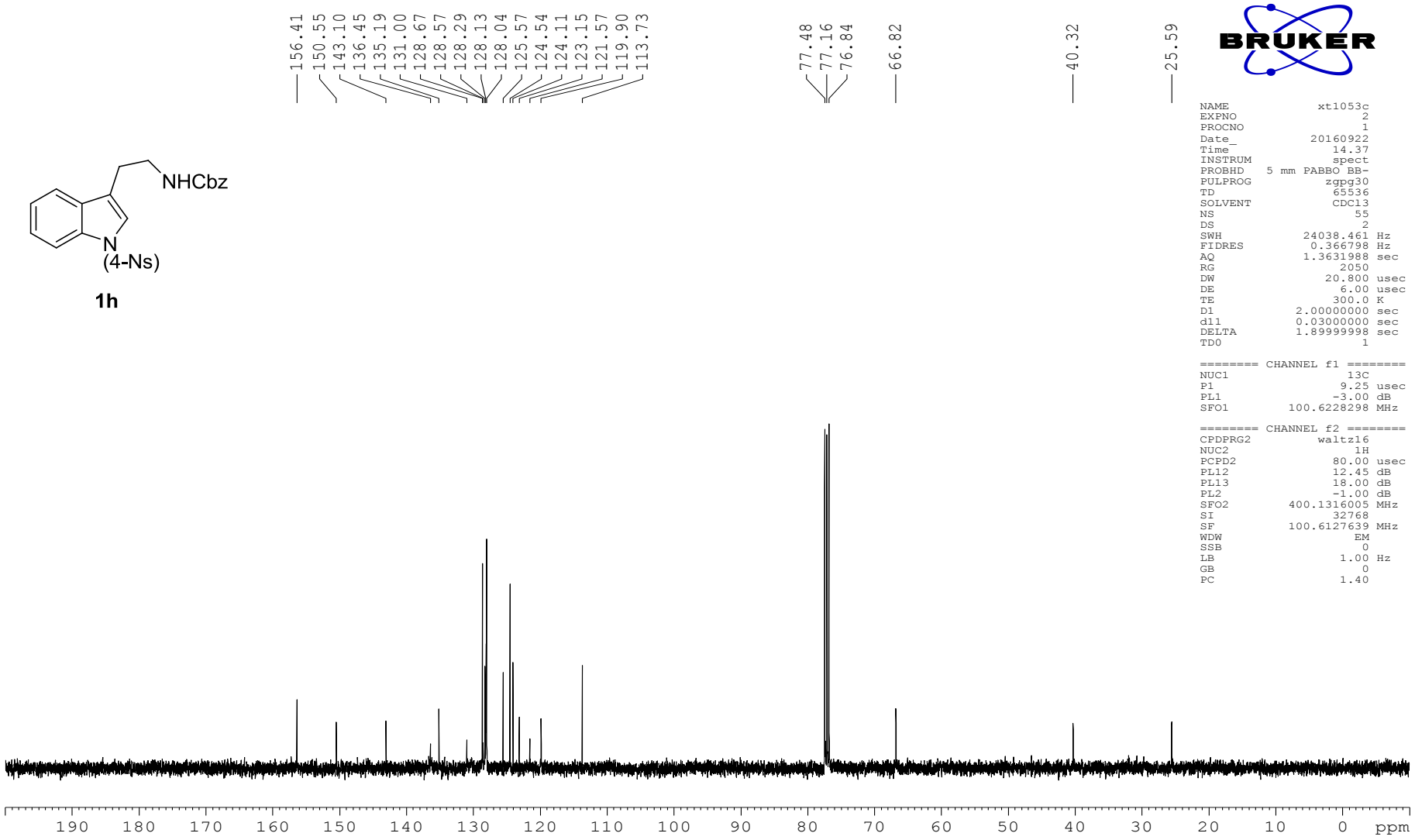
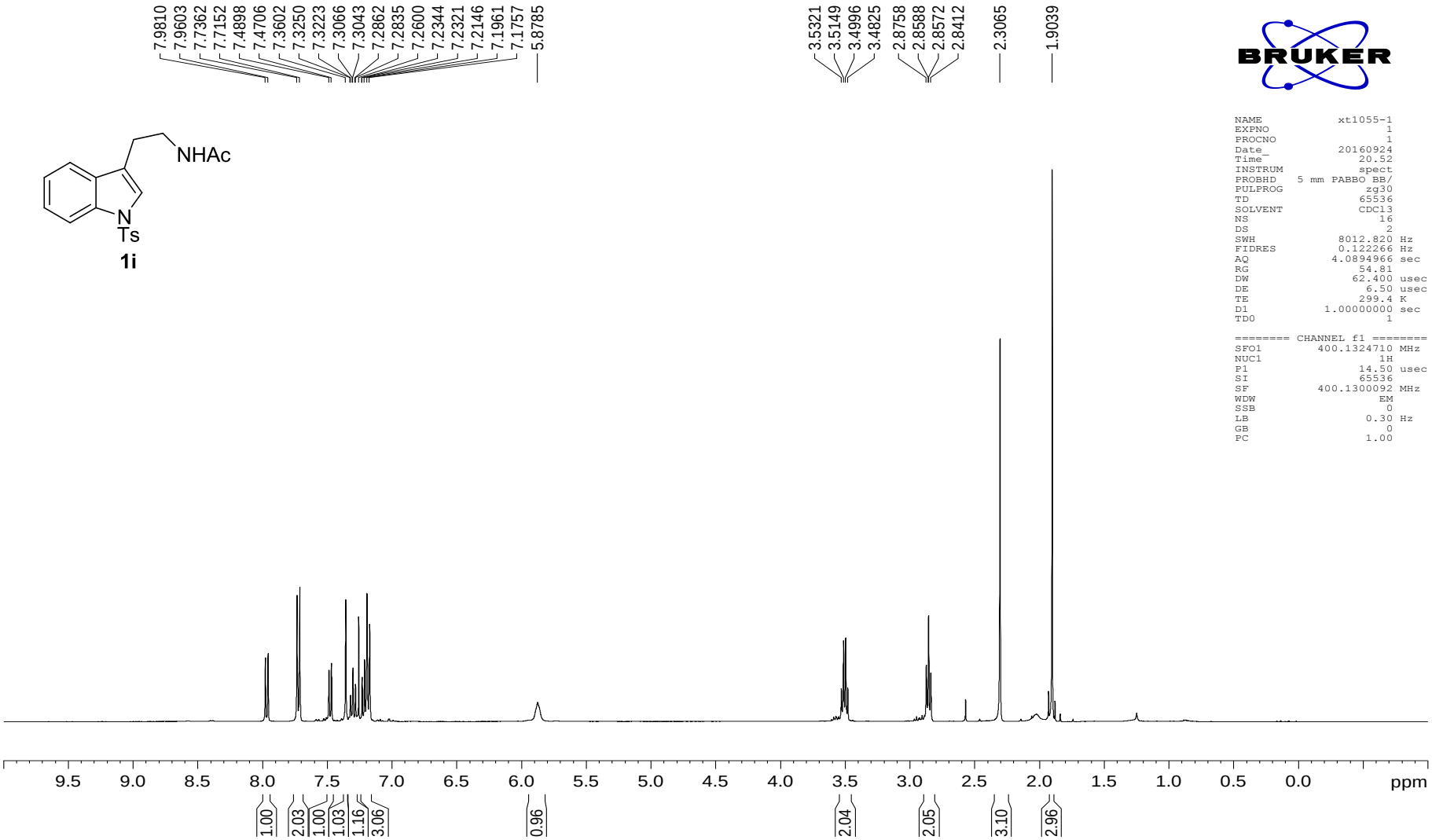
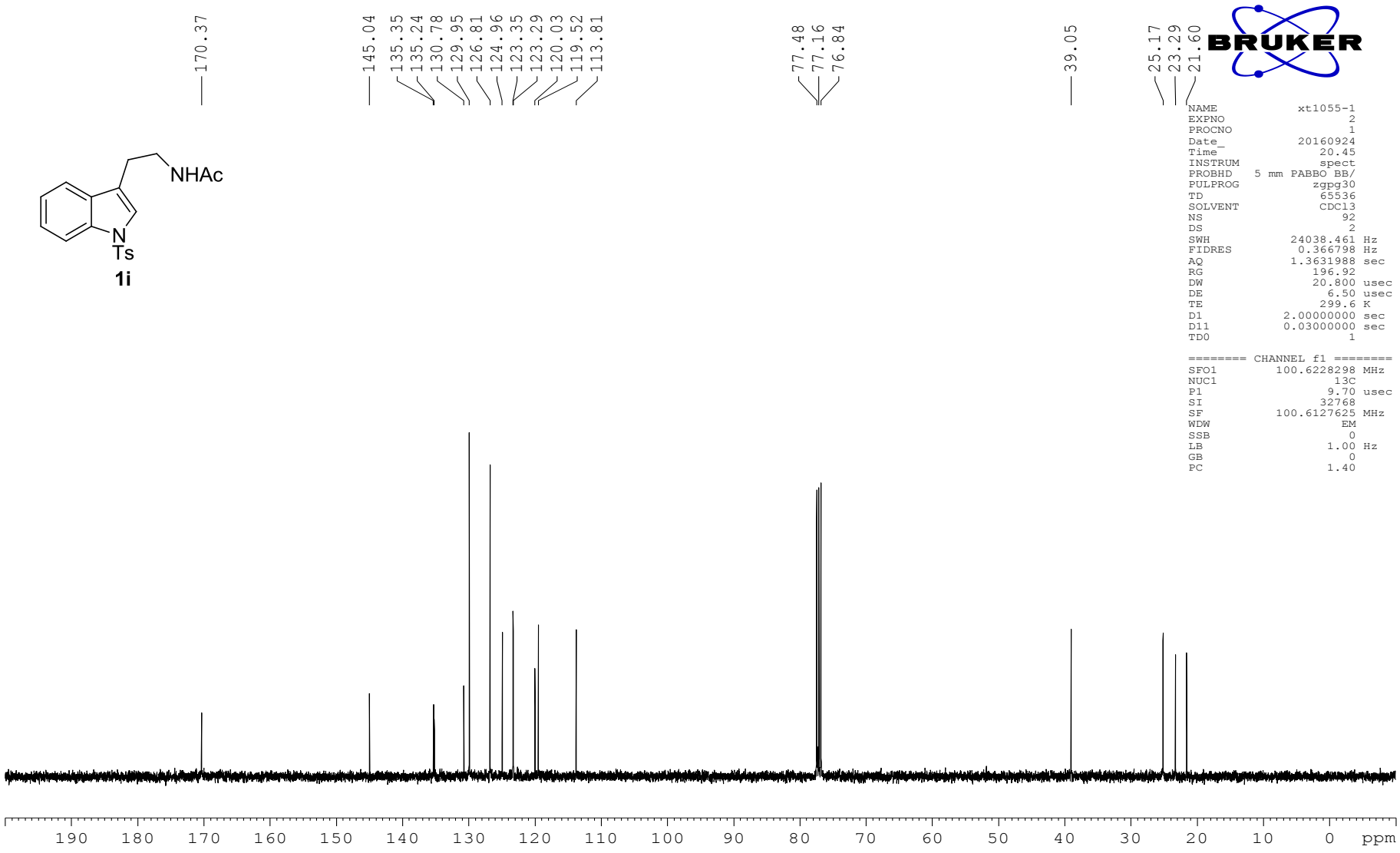
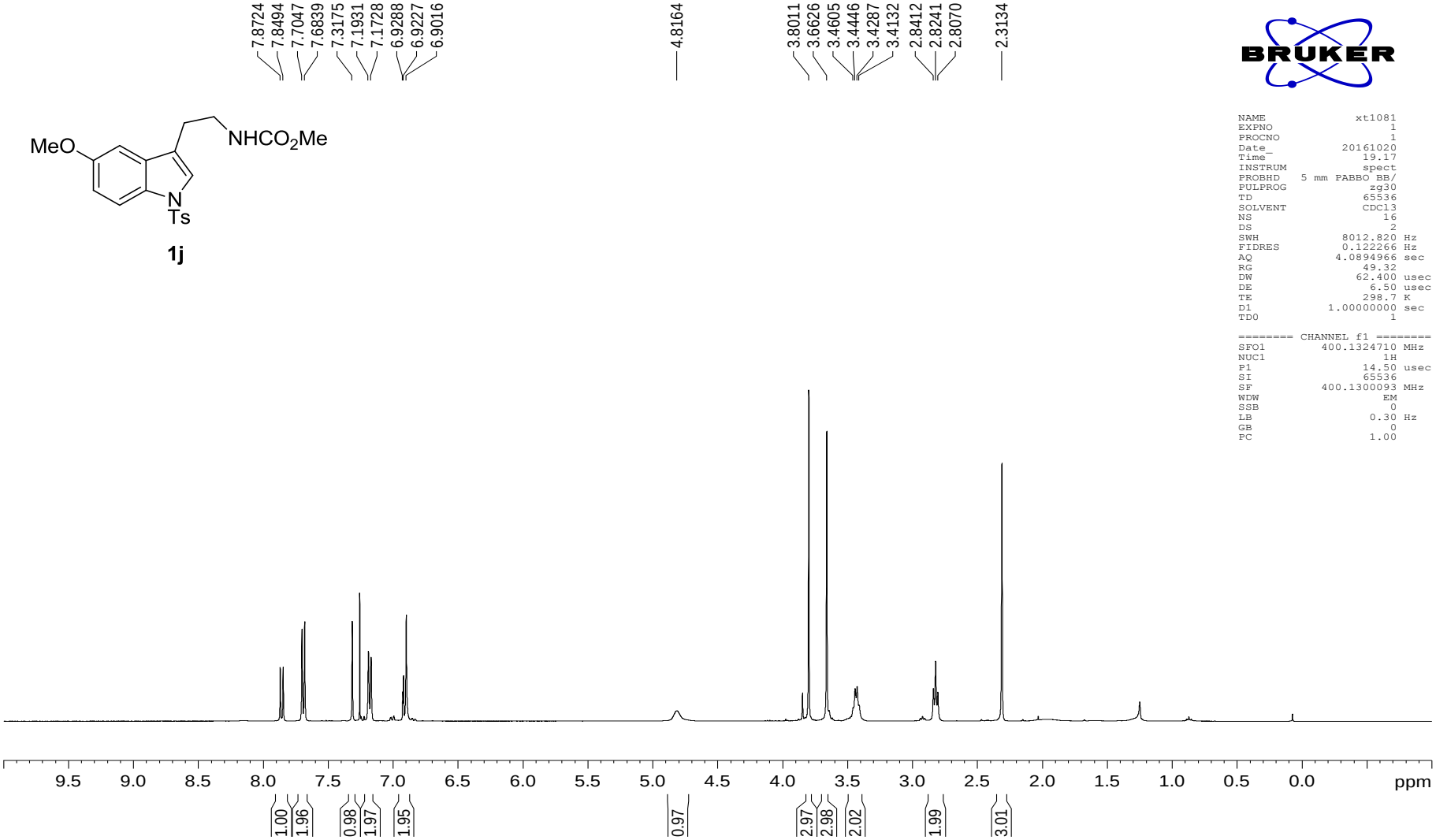
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 330
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127564 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

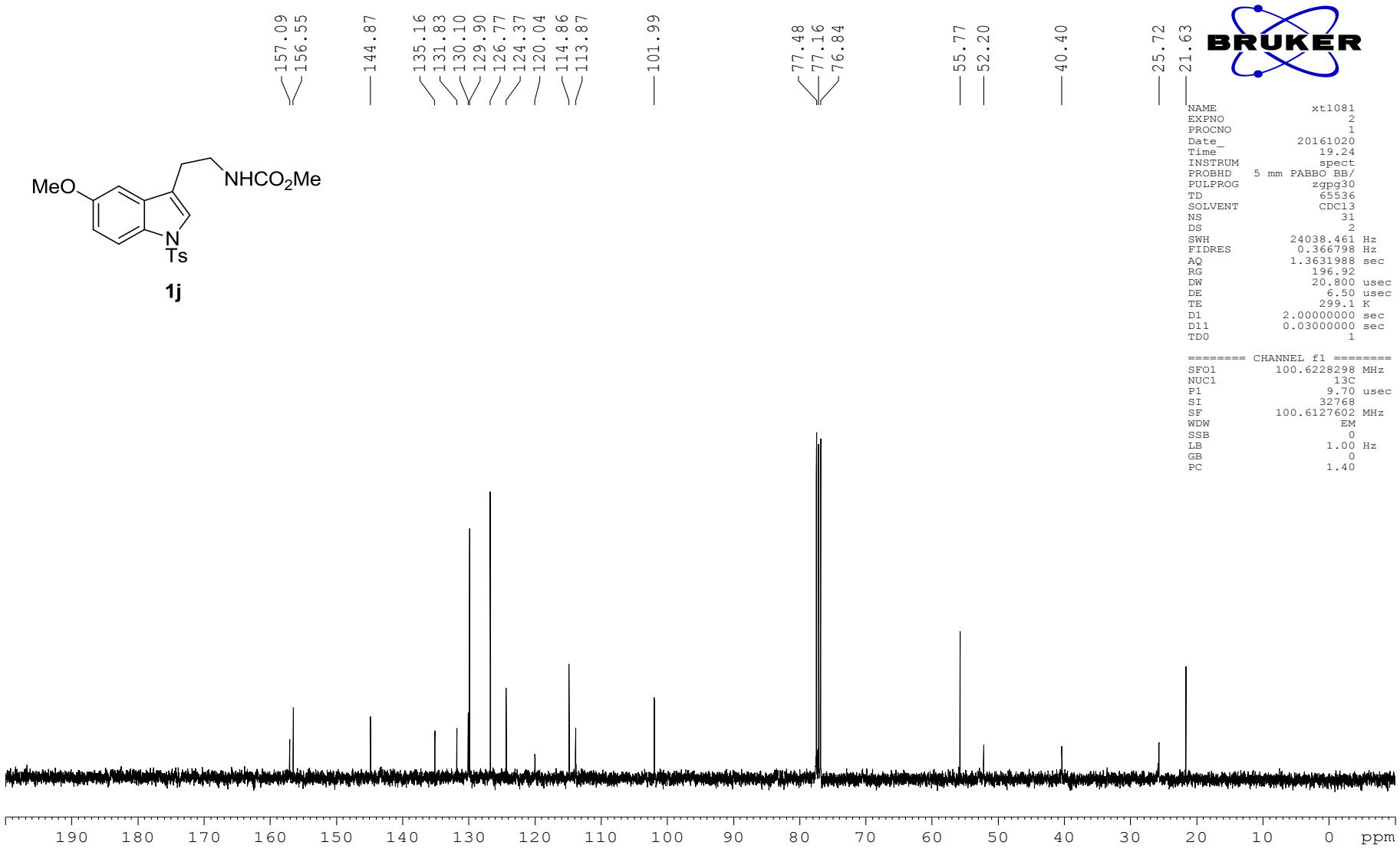

8.1675
8.1458
7.9808
7.9760
7.9631
7.9584
7.5162
7.4967
7.3678
7.3499
7.3281
7.3007
7.2924
7.2795
7.2589
7.2422
5.0971
4.9753
4.9609
4.9469
3.5173
3.5008
3.4845
3.4681
2.9121
2.8953
2.8786
NHCbz
N
(4-Ns)
1h
BRUKER
NAME xt1053c
EXPNO 1
PROCNO 1
Date_ 20160922
Time_ 14.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 14
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.07
3.04
1.19
8.22
2.13
1.00
2.30
2.38

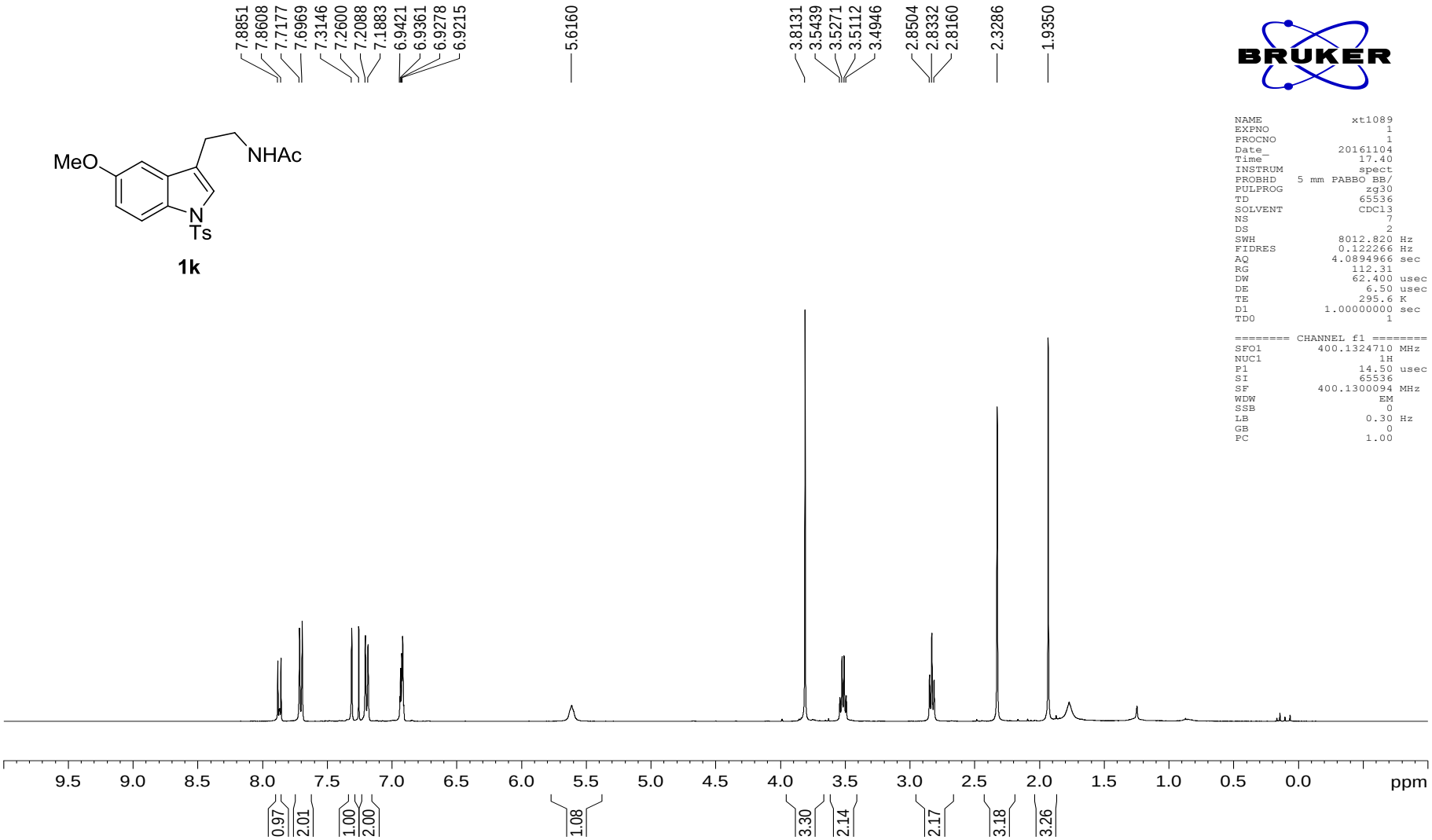

156.41
150.55
143.10
136.45
135.19
131.00
128.67
128.57
128.29
128.13
128.04
125.57
124.54
124.11
123.15
121.57
119.90
113.73
77.48
77.16
76.84
66.82
40.32
25.59
NHCbz
N
(4-Ns)
1h
NAME xt1053c
EXPNO 2
PROCNO 1
Date_ 20160922
Time 14.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 55
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127639 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

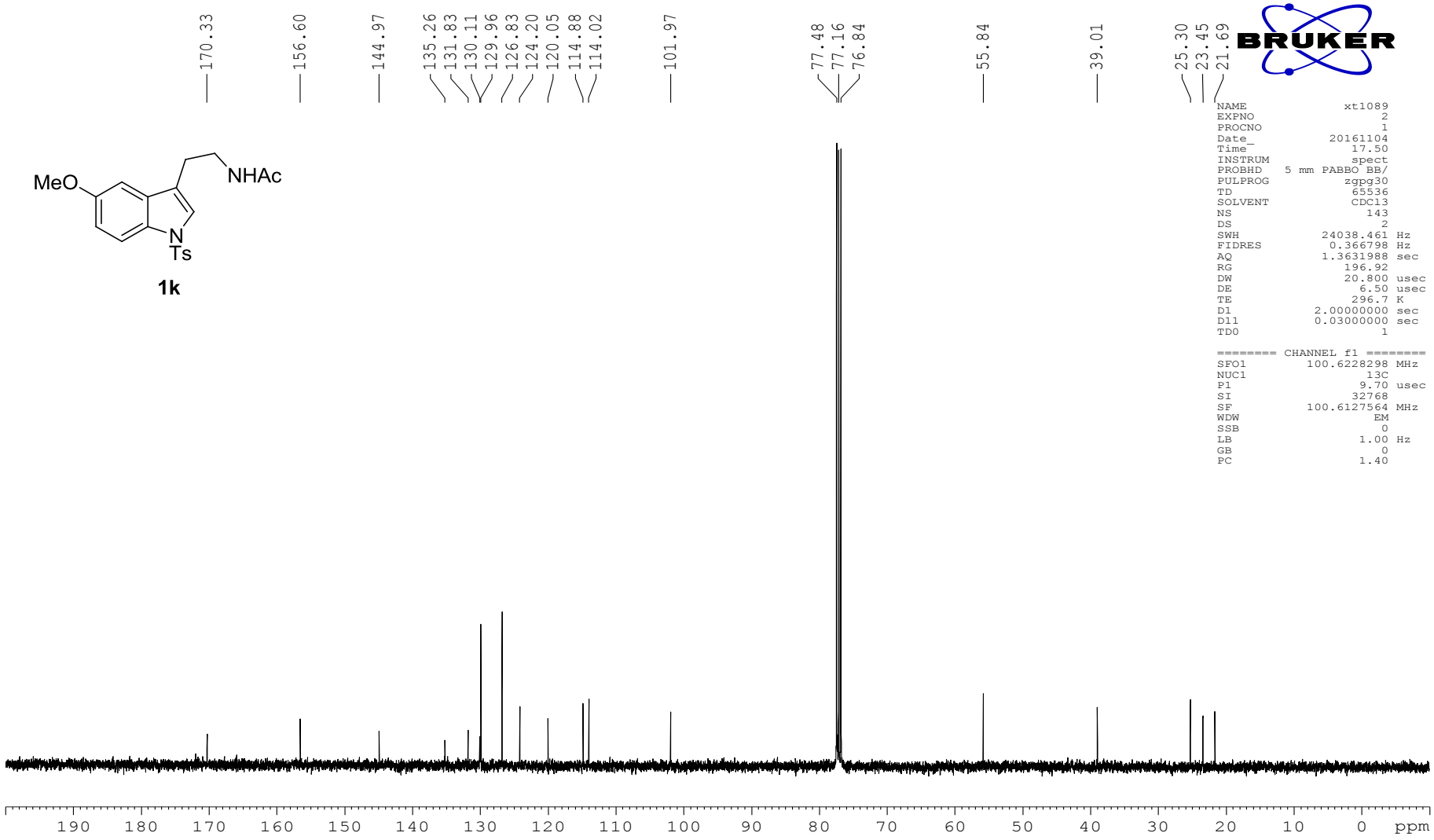

7.9810
7.9603
7.7362
7.7152
7.4898
7.4706
7.3602
7.3250
7.3223
7.3066
7.3043
7.2862
7.2835
7.2600
7.2344
7.2321
7.2146
7.1961
7.1757
5.8785
3.5321
3.5149
3.4996
3.4825
2.8758
2.8588
2.8572
2.8412
2.3065
1.9039
NHAc
N
Ts
1i
BRUKER
NAME xt1055-1
EXPNO 1
PROCNO 1
Date_ 20160924
Time_ 20.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 54.81
DW 62.400 usec
DE 6.50 usec
TE 299.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.03
1.00
1.03
1.16
3.06
0.96
2.04
2.05
3.10
2.96

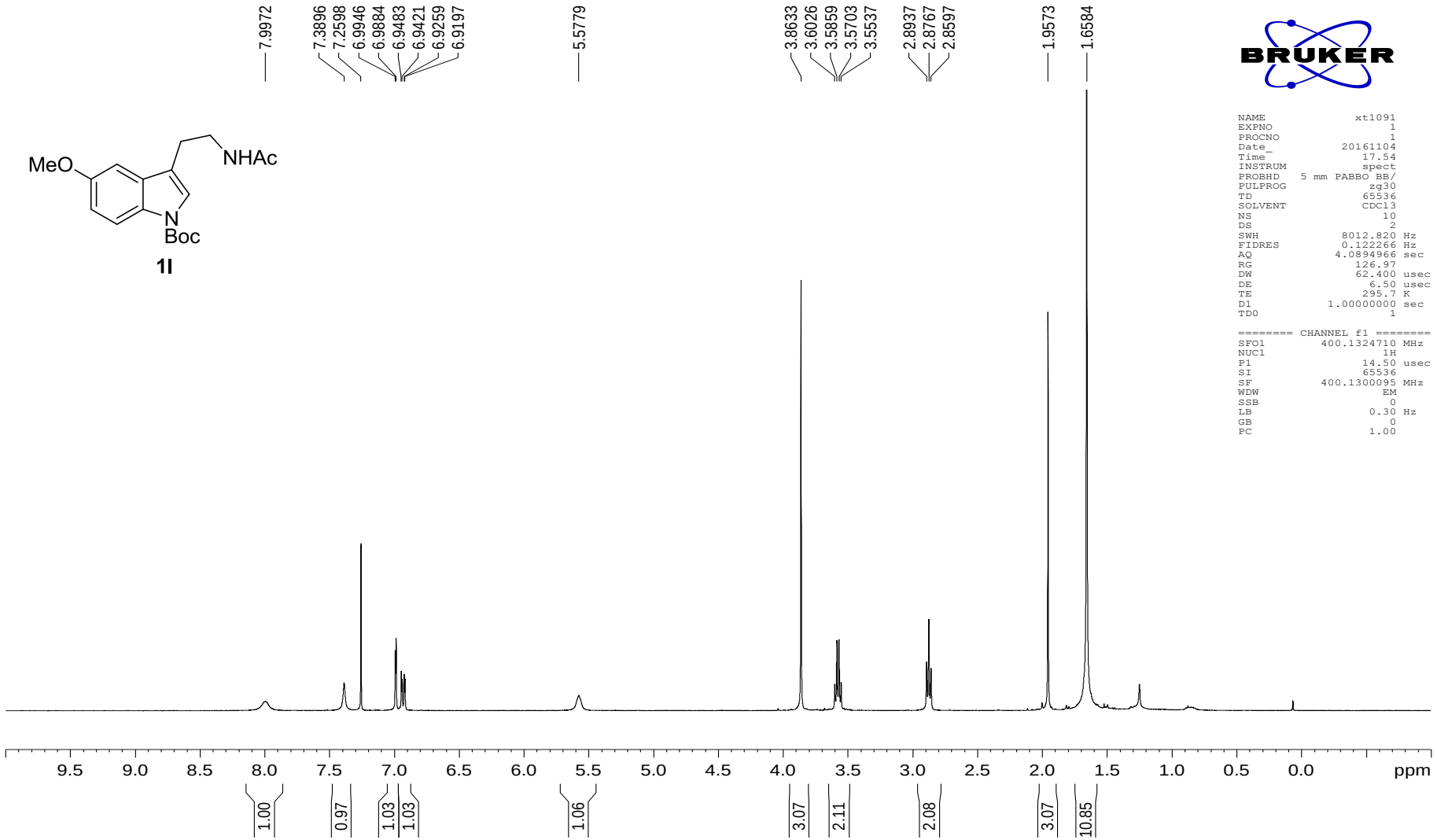

170.37
145.04
135.35
135.24
130.78
129.95
126.81
124.96
123.35
123.29
120.03
119.52
113.81
77.48
77.16
76.84
39.05
25.17
23.29
21.60
NHAc
N
Ts
1i
BRUKER
NAME xt1055-1
EXPNO 2
PROCNO 1
Date_ 20160924
Time 20.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 92
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127625 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

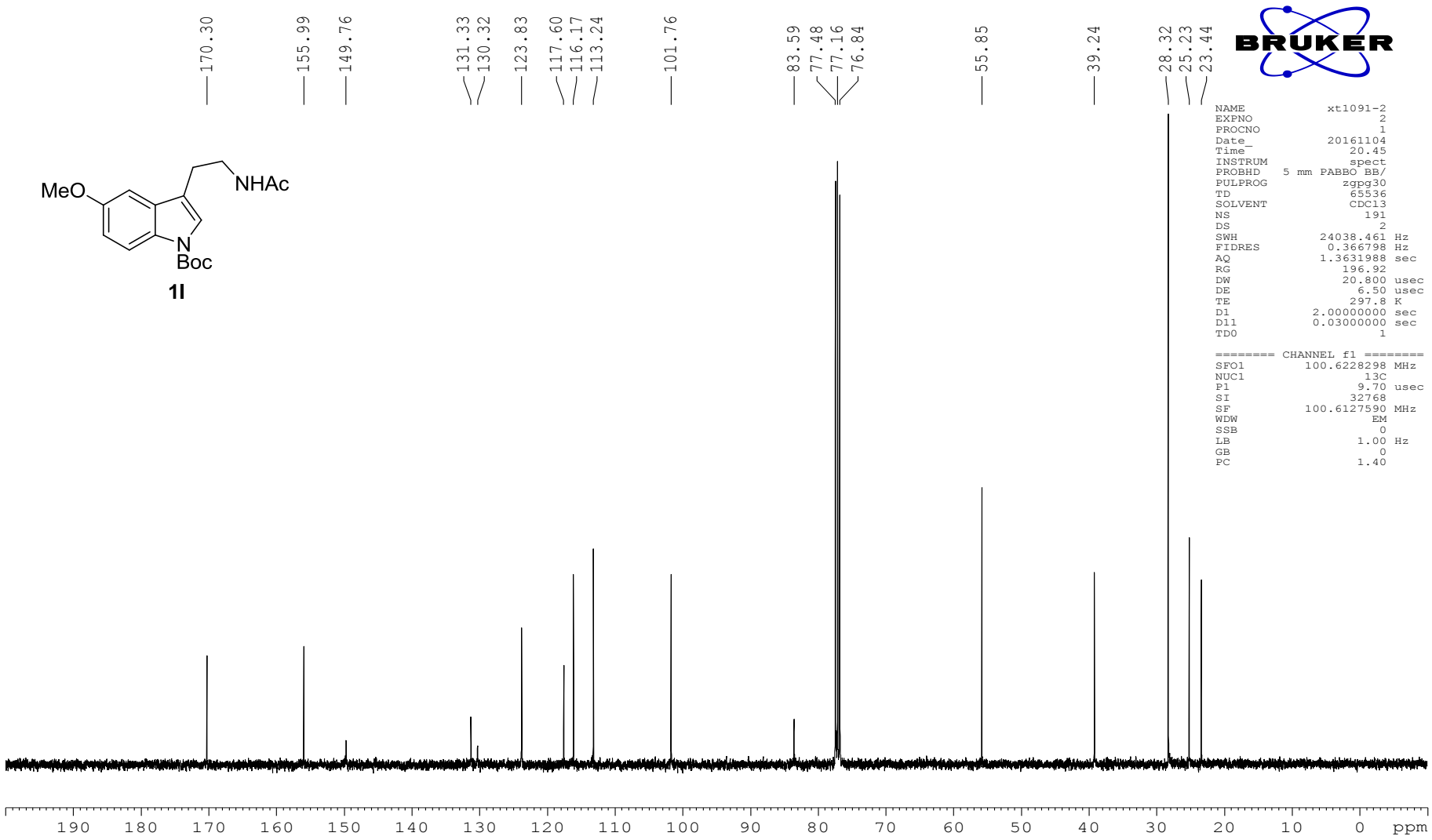

7.8724
7.8494
7.7047
7.6839
7.3175
7.1931
7.1728
6.9288
6.9227
6.9016
4.8164
3.8011
3.6626
3.4605
3.4446
3.4287
3.4132
2.8412
2.8241
2.8070
2.3134
MeO
NHCO2Me
N
Ts
1j
BRUKER
NAME xt1081
EXPNO 1
PROCNO 1
Date_ 20161020
Time_ 19.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 49.32
DW 62.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm
1.00
1.96
0.98
1.97
1.95
0.97
2.97
2.98
2.02
1.99
3.01

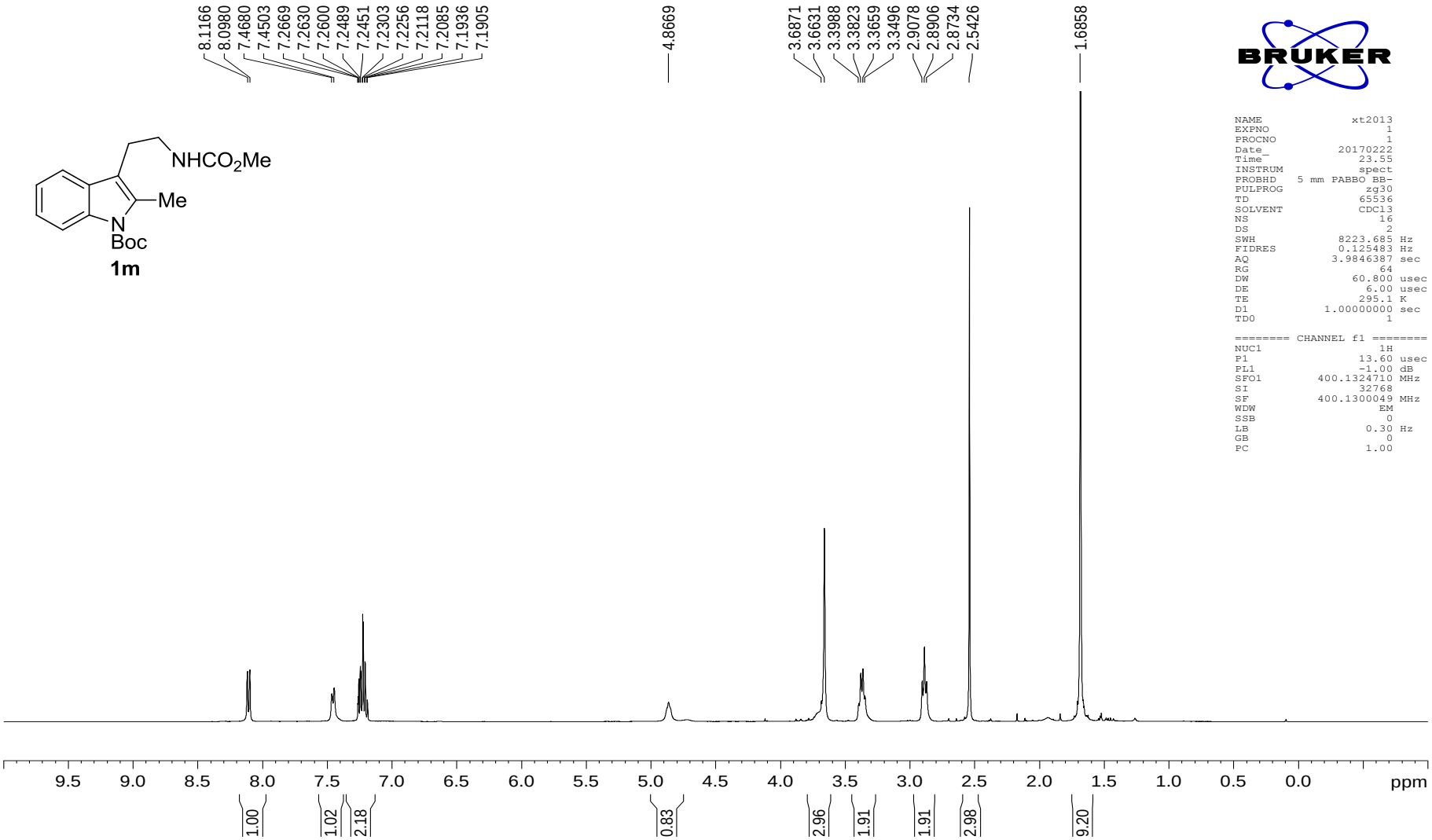

157.09
156.55
144.87
135.16
131.83
130.10
129.90
126.77
124.37
120.04
114.86
113.87
101.99
77.48
77.16
76.84
55.77
52.20
40.40
25.72
21.63
BRUKER
MeO
NHCO2Me
N
Ts
1j
NAME xt1081
EXPNO 2
PROCNO 1
Date_ 20161020
Time 19.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 31
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127602 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

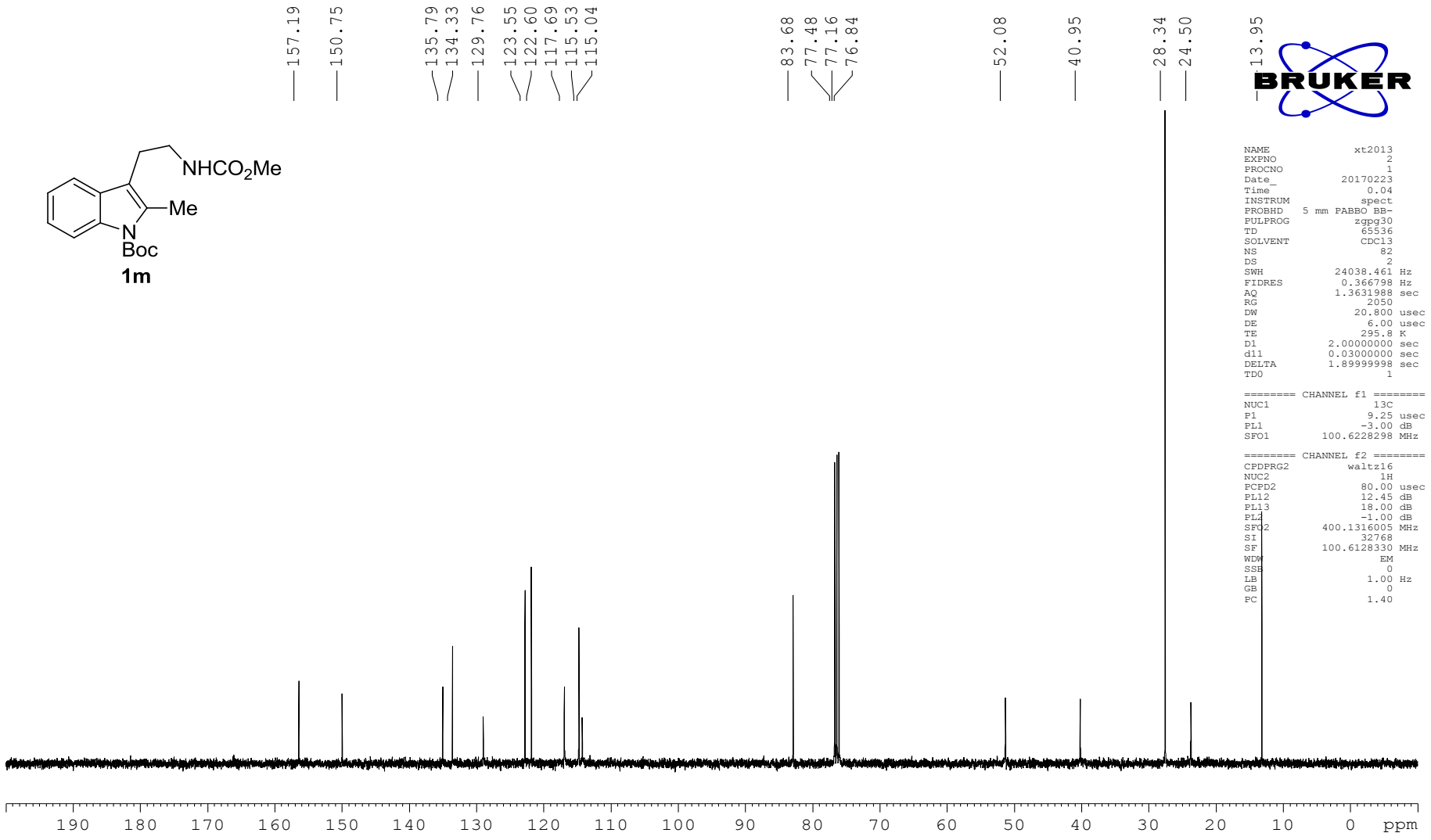

7.8851
7.8608
7.7177
7.6969
7.3146
7.2600
7.2088
7.1883
6.9421
6.9361
6.9278
6.9215
5.6160
3.8131
3.5439
3.5271
3.5112
3.4946
2.8504
2.8332
2.8160
2.3286
1.9350
MeO
NHAc
N
Ts
1k
NAME xt1089
EXPNO 1
PROCNO 1
Date_ 20161104
Time_ 17.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm
0.97
2.01
1.00
2.00
1.08
3.30
2.14
2.17
3.18
3.26

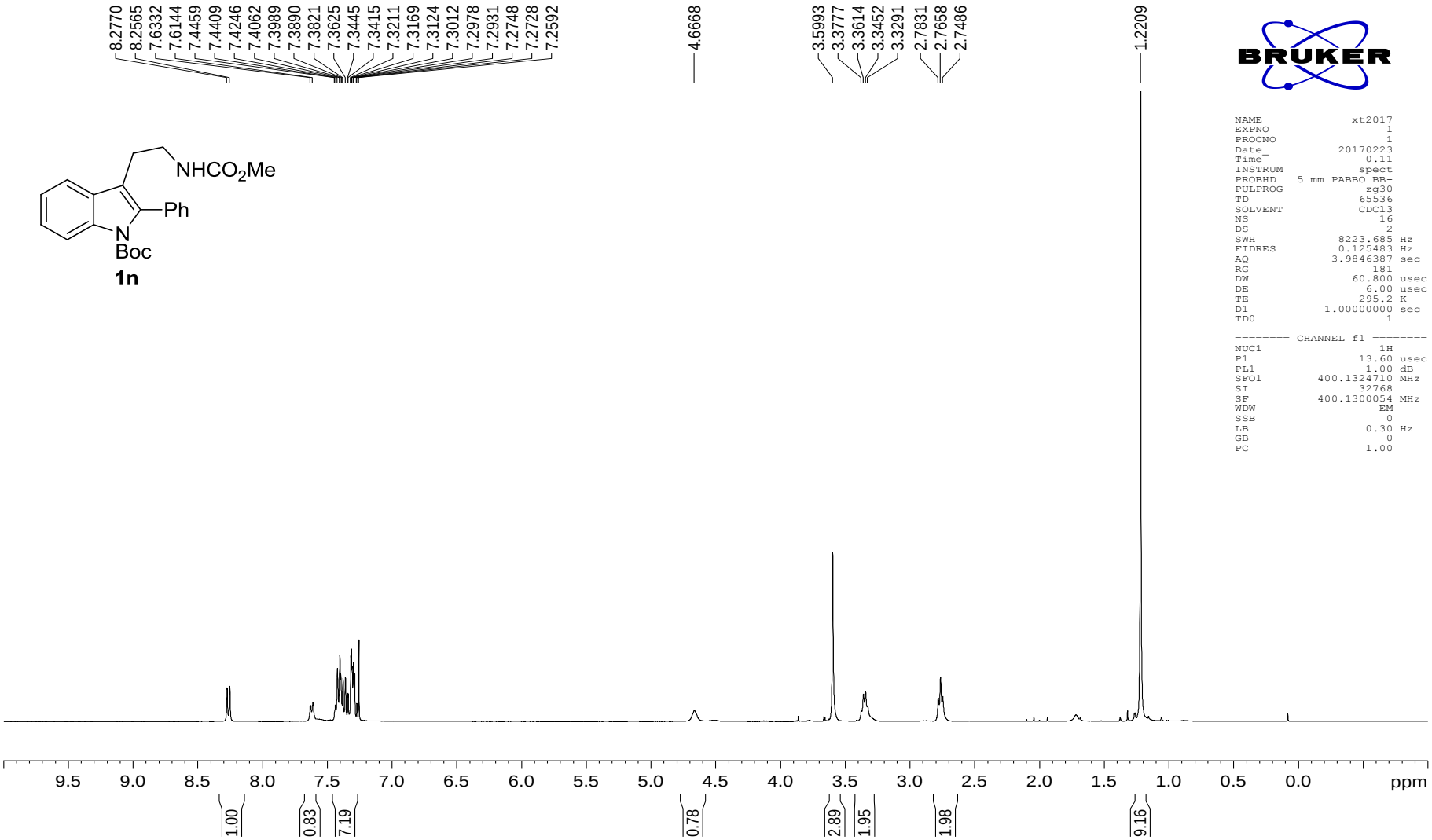

170.33
156.60
144.97
135.26
131.83
130.11
129.96
126.83
124.20
120.05
114.88
114.02
101.97
77.48
77.16
76.84
55.84
39.01
25.30
23.45
21.69
BRUKER
MeO
NHAc
N
Ts
1k
NAME xt1089
EXPNO 2
PROCNO 1
Date_ 20161104
Time 17.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 143
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127564 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

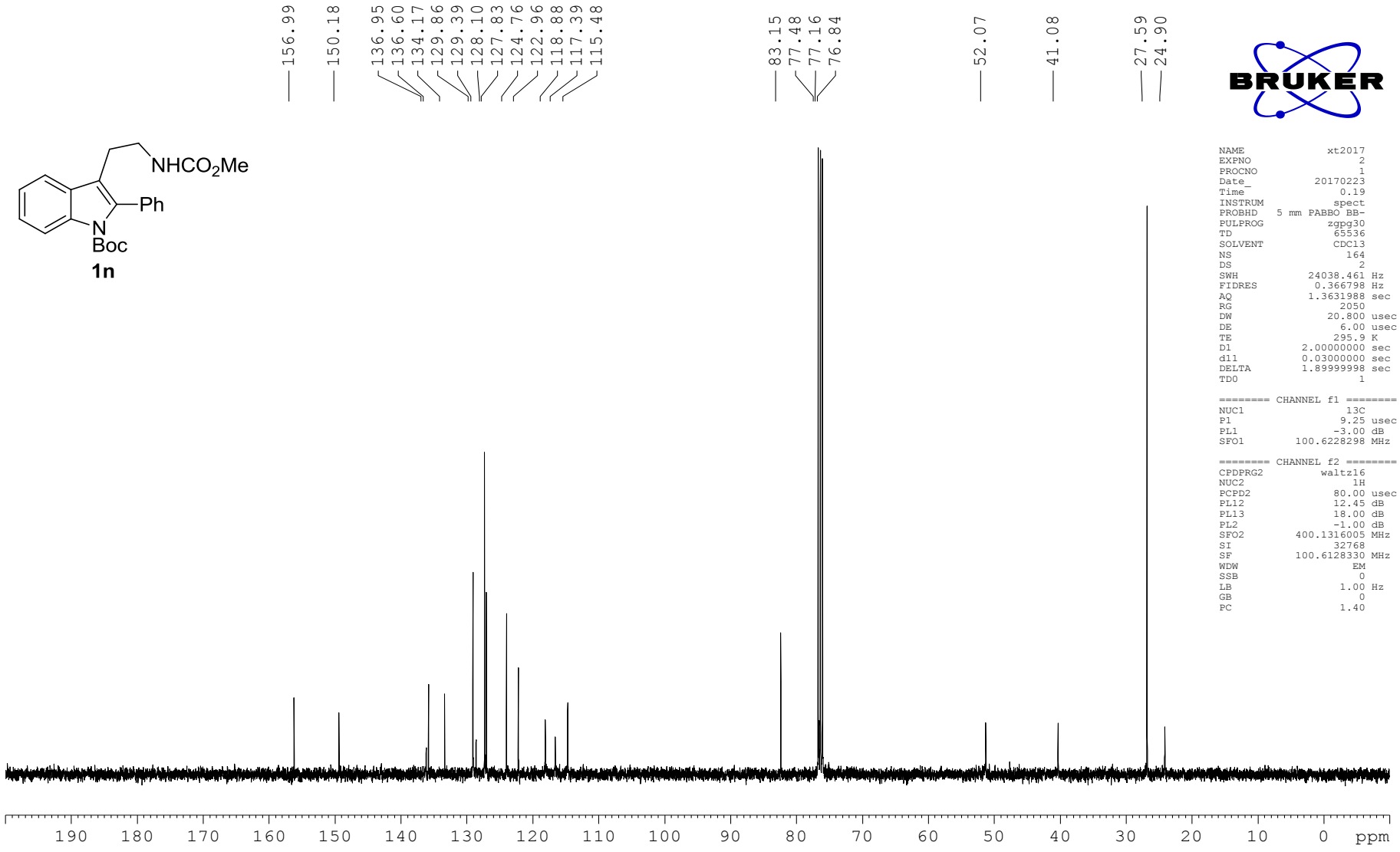

7.9972
7.3896
7.2598
6.9946
6.9884
6.9483
6.9421
6.9259
6.9197
5.5779
3.8633
3.6026
3.5859
3.5703
3.5537
2.8937
2.8767
2.8597
1.9573
1.6584
MeO
NHAc
N
Boc
1l
BRUKER
NAME xt1091
EXPNO 1
PROCNO 1
Date_ 20161104
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
0.97
1.03
1.03
1.06
3.07
2.11
2.08
3.07
10.85

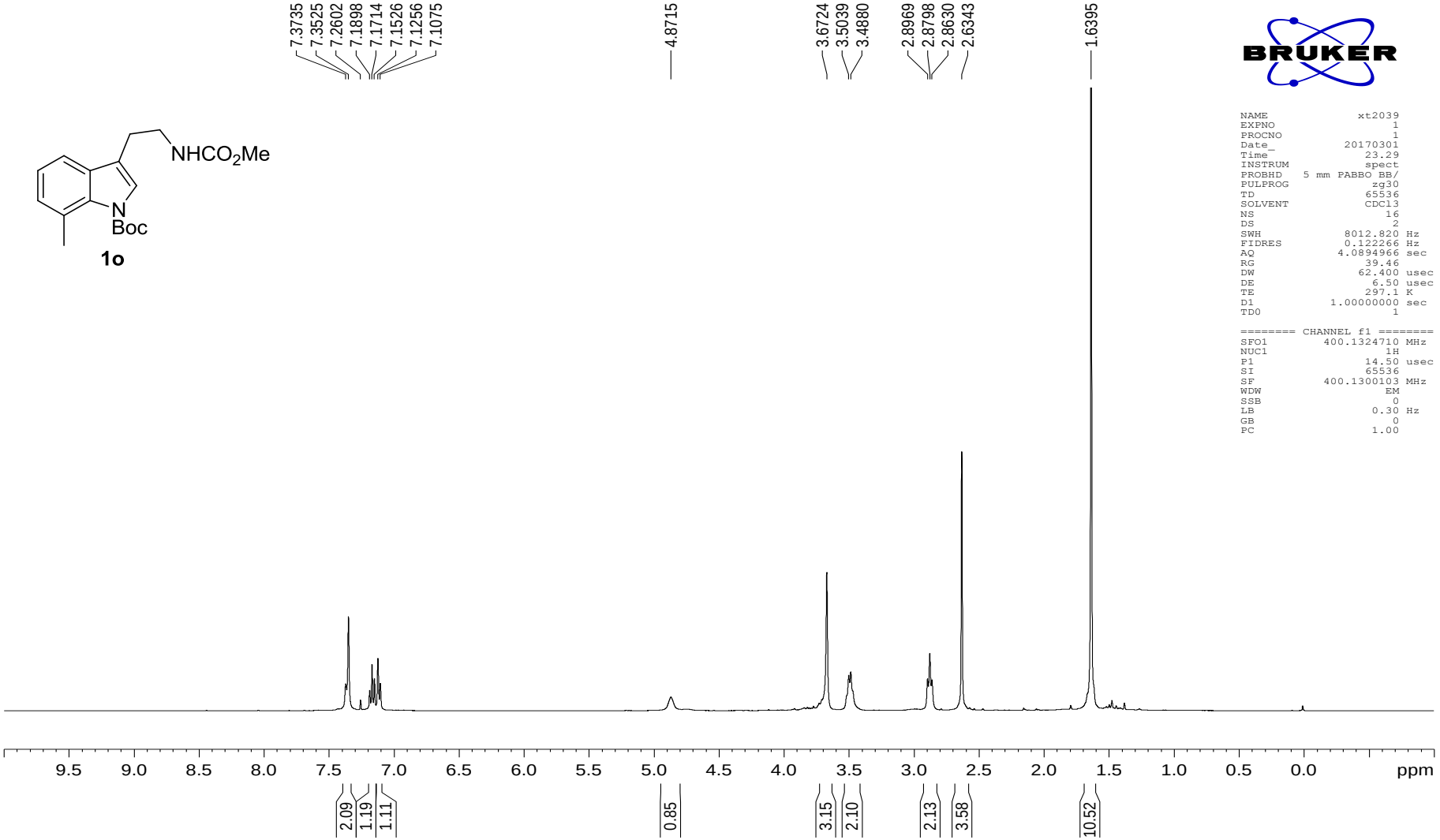

170.30
155.99
149.76
131.33
130.32
123.83
117.60
116.17
113.24
101.76
83.59
77.48
77.16
76.84
55.85
39.24
28.32
25.23
23.44
MeO
NHAc
N
Boc
1l
NAME xt1091-2
EXPNO 2
PROCNO 1
Date_ 20161104
Time 20.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 191
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

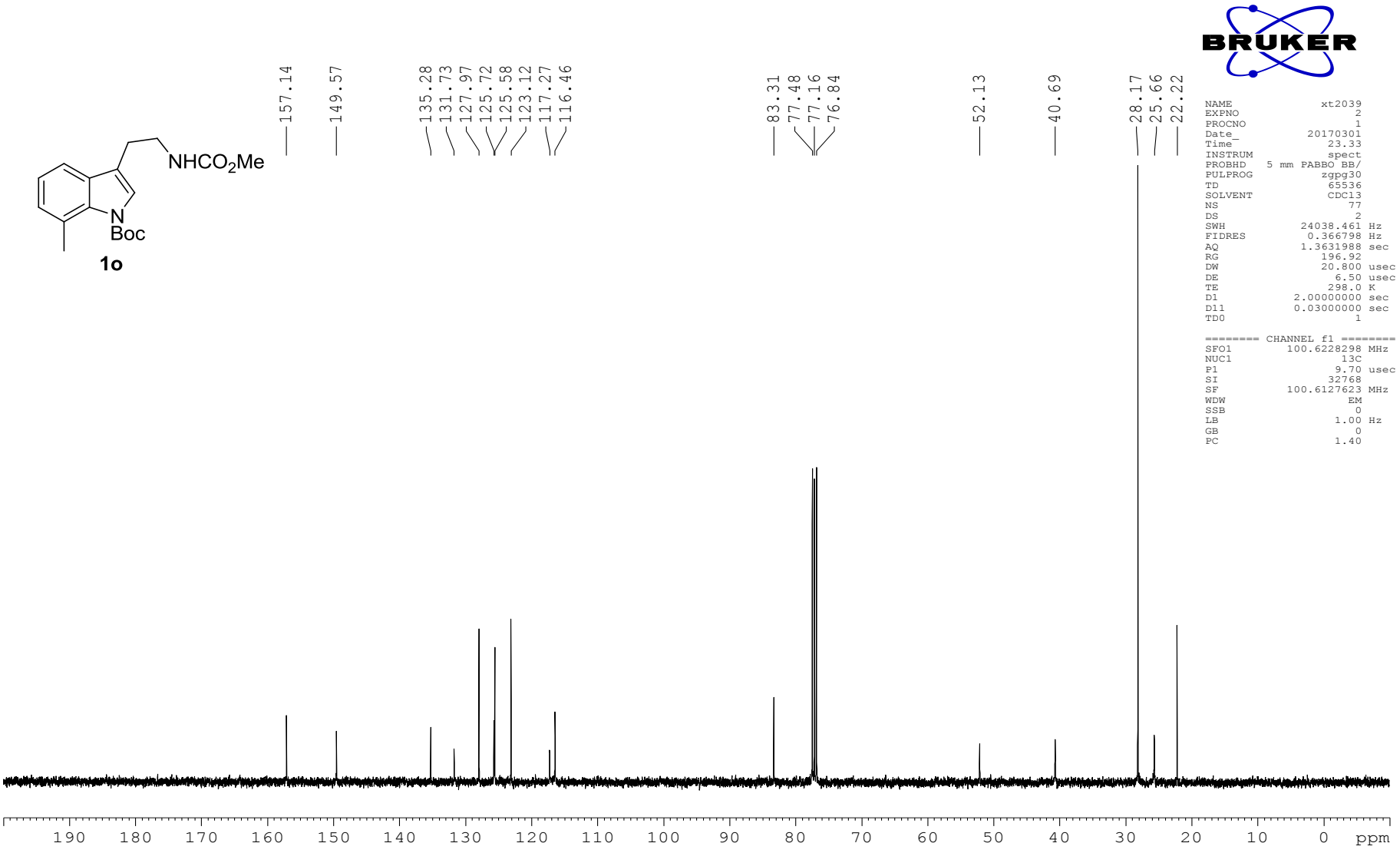

8.1166
8.0980
7.4680
7.4503
7.2669
7.2630
7.2600
7.2489
7.2451
7.2303
7.2256
7.2118
7.2085
7.1936
7.1905
4.8669
3.6871
3.6631
3.3988
3.3823
3.3659
3.3496
2.9078
2.8906
2.8734
2.5426
1.6858
NHCO2Me
Me
N
Boc
1m
NAME xt2013
EXPNO 1
PROCNO 1
Date_ 20170222
Time_ 23.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 64
DW 60.800 usec
DE 6.00 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.02
2.18
0.83
2.96
1.91
1.91
2.98
9.20

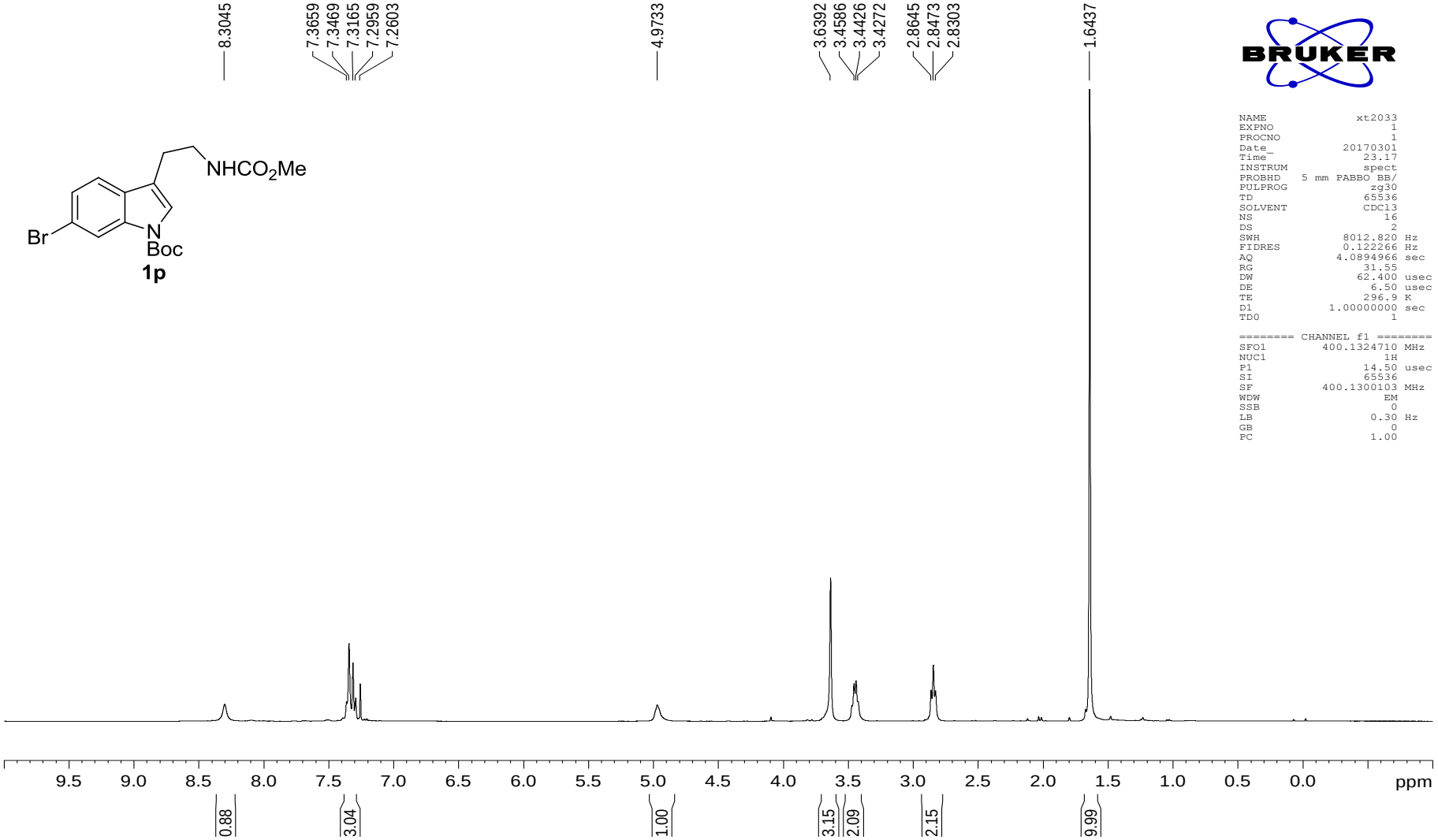

157.19
150.75
135.79
134.33
129.76
123.55
122.60
117.69
115.53
115.04
83.68
77.48
77.16
76.84
52.08
40.95
28.34
24.50
13.95
NHCO2Me
Me
N
Boc
1m
BRUKER
NAME xt2013
EXPNO 2
PROCNO 1
Date_ 20170223
Time_ 0.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 82
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

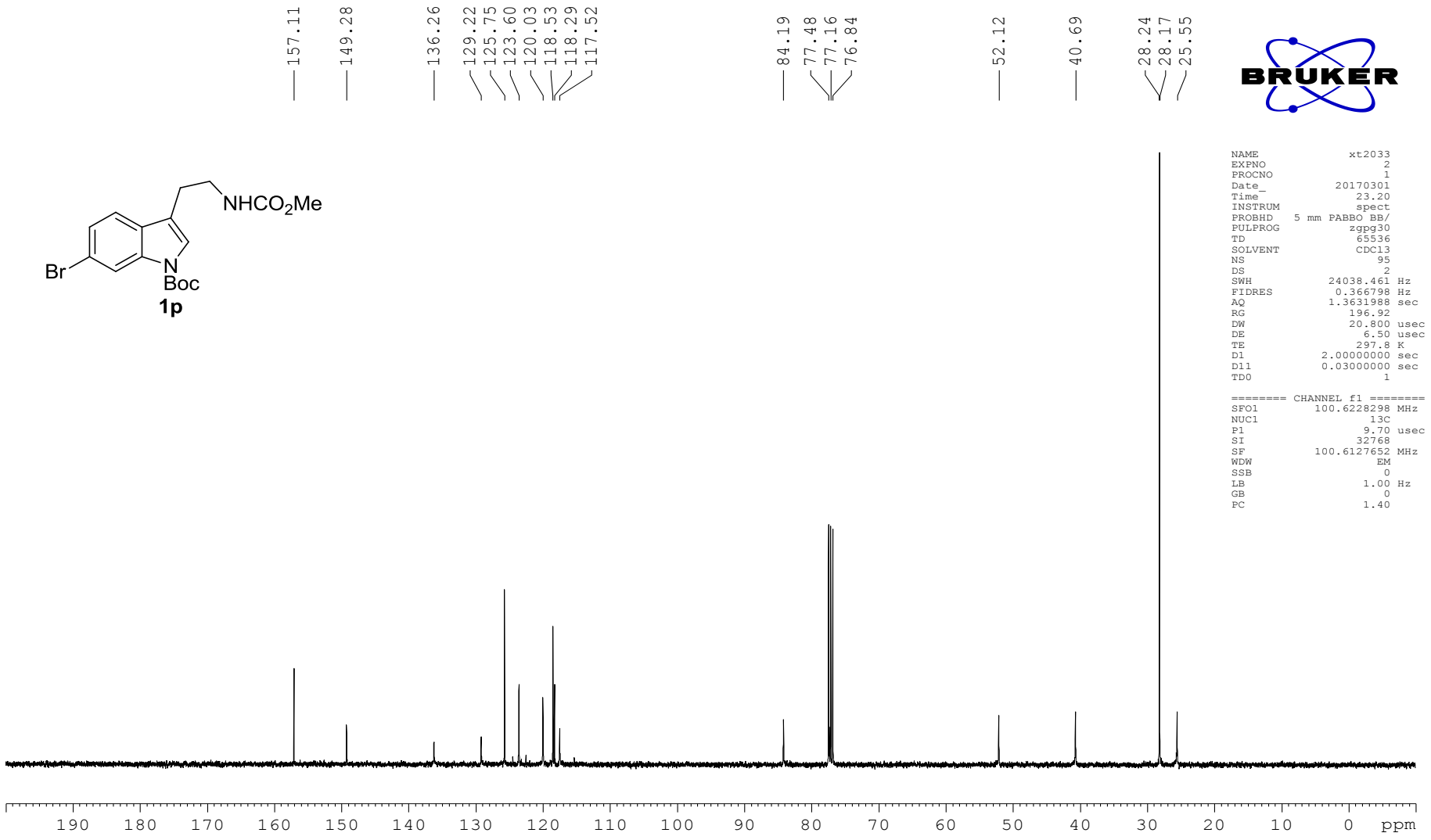

8.2770
8.2565
7.6332
7.6144
7.4459
7.4409
7.4246
7.4062
7.3989
7.3890
7.3821
7.3625
7.3445
7.3415
7.3211
7.3169
7.3124
7.3012
7.2978
7.2931
7.2748
7.2728
7.2592
4.6668
3.5993
3.3777
3.3614
3.3452
3.3291
2.7831
2.7658
2.7486
1.2209
NHCO2Me
Ph
N
Boc
1n
BRUKER
NAME xt2017
EXPNO 1
PROCNO 1
Date_ 20170223
Time_ 0.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.83
7.19
0.78
2.89
1.95
1.98
9.16

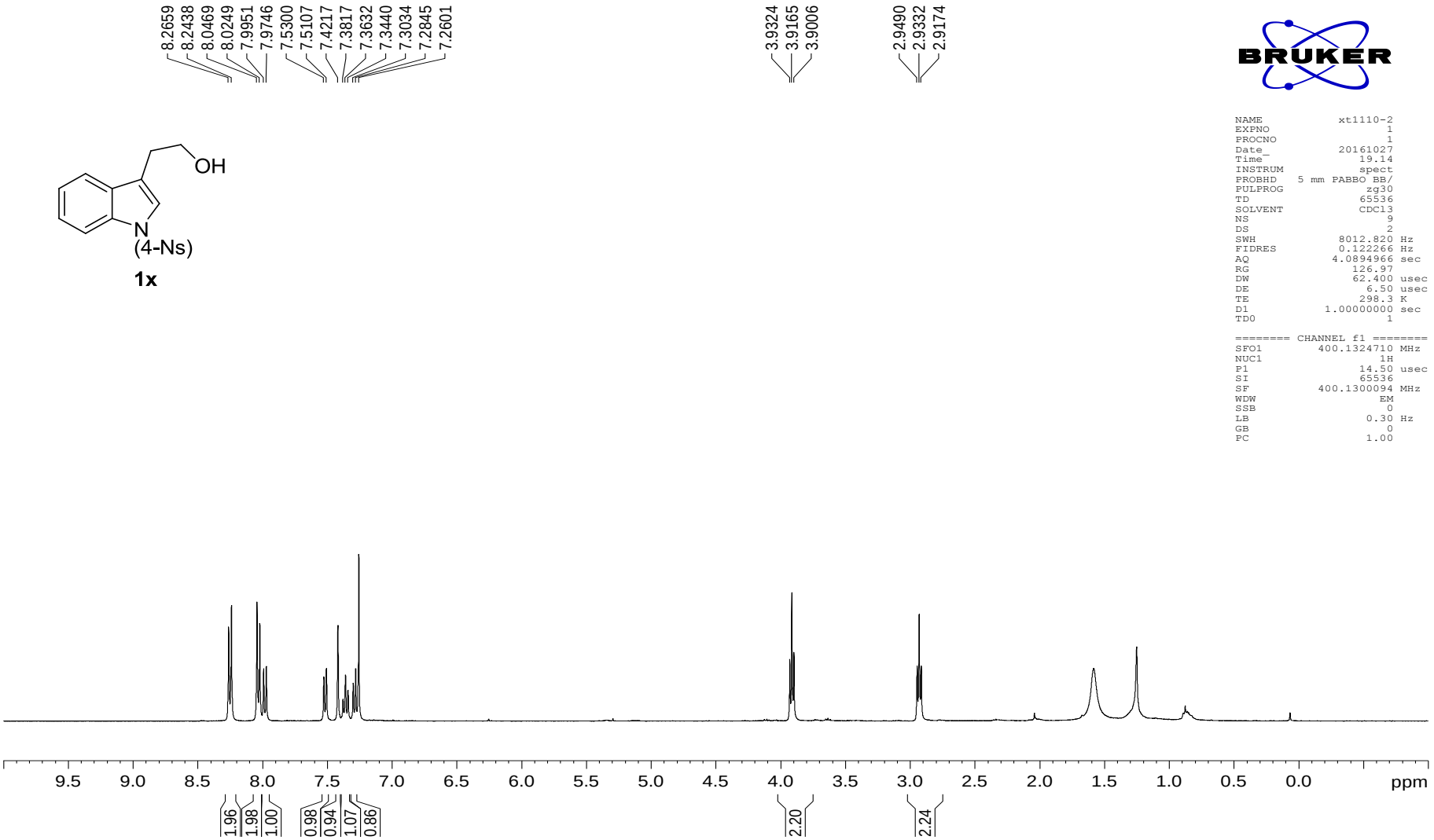

156.99
150.18
136.95
136.60
134.17
129.86
129.39
128.10
127.83
124.76
122.96
118.88
117.39
115.48
83.15
77.48
77.16
76.84
52.07
41.08
27.59
24.90
NHCO2Me
Ph
N
Boc
1n
BRUKER
NAME xt2017
EXPNO 2
PROCNO 1
Date_ 20170223
Time 0.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 164
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.3735
7.3525
7.2602
7.1898
7.1714
7.1526
7.1256
7.1075
4.8715
3.6724
3.5039
3.4880
2.8969
2.8798
2.8630
2.6343
1.6395
NHCO2Me
N
Boc
1o
BRUKER
NAME xt2039
EXPNO 1
PROCNO 1
Date_ 20170301
Time 23.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 39.46
DW 62.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.09
1.19
1.11
0.85
3.15
2.10
2.13
3.58
10.52


NHCO2Me
N
Boc
1o
157.14
149.57
135.28
131.73
127.97
125.72
125.58
123.12
117.27
116.46
83.31
77.48
77.16
76.84
52.13
40.69
28.17
25.66
22.22
BRUKER
NAME xt2039
EXPNO 2
PROCNO 1
Date_ 20170301
Time 23.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 77
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127623 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.3045
7.3659
7.3469
7.3165
7.2959
7.2603
4.9733
3.6392
3.4586
3.4426
3.4272
2.8645
2.8473
2.8303
1.6437
NHCO2Me
Br
N
Boc
1p
NAME xt2033
EXPNO 1
PROCNO 1
Date_ 20170301
Time 23.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 31.55
DW 62.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.88
3.04
1.00
3.15
2.09
2.15
9.99


157.11
149.28
136.26
129.22
125.75
123.60
120.03
118.53
118.29
117.52
84.19
77.48
77.16
76.84
52.12
40.69
28.24
28.17
25.55
BRUKER
NHCO2Me
Br
N
Boc
1p
NAME xt2033
EXPNO 2
PROCNO 1
Date_ 20170301
Time 23.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 95
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127652 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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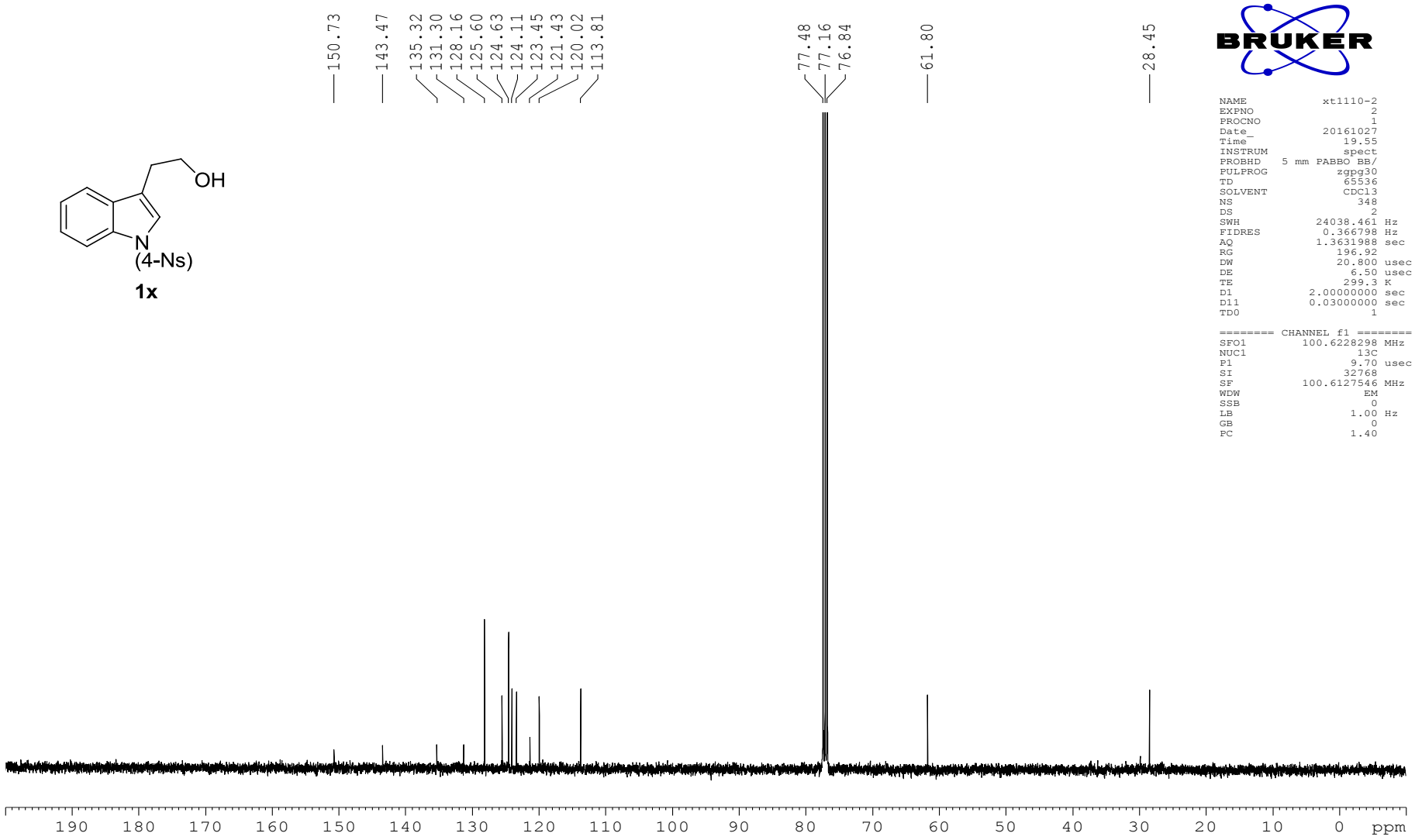
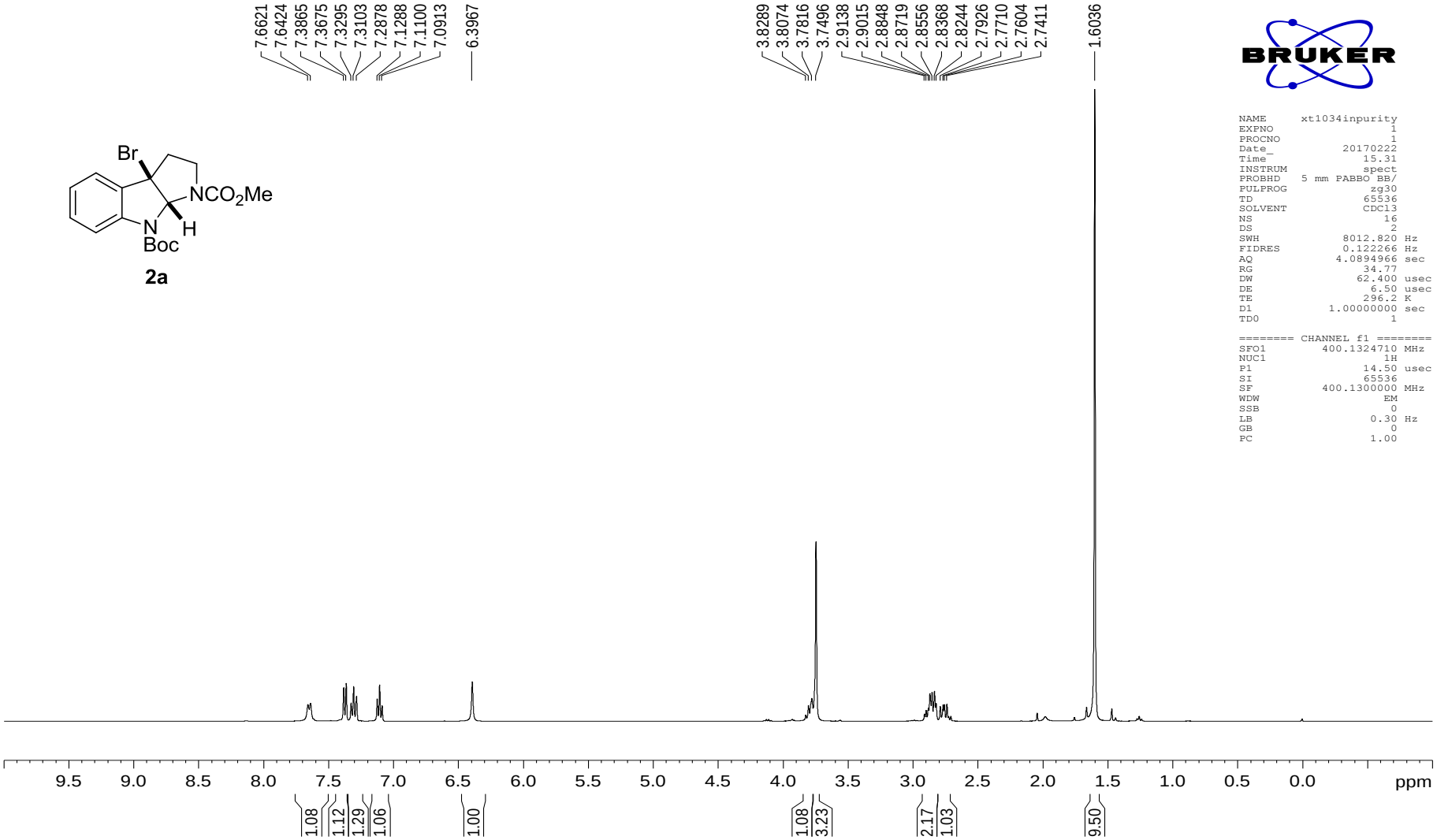
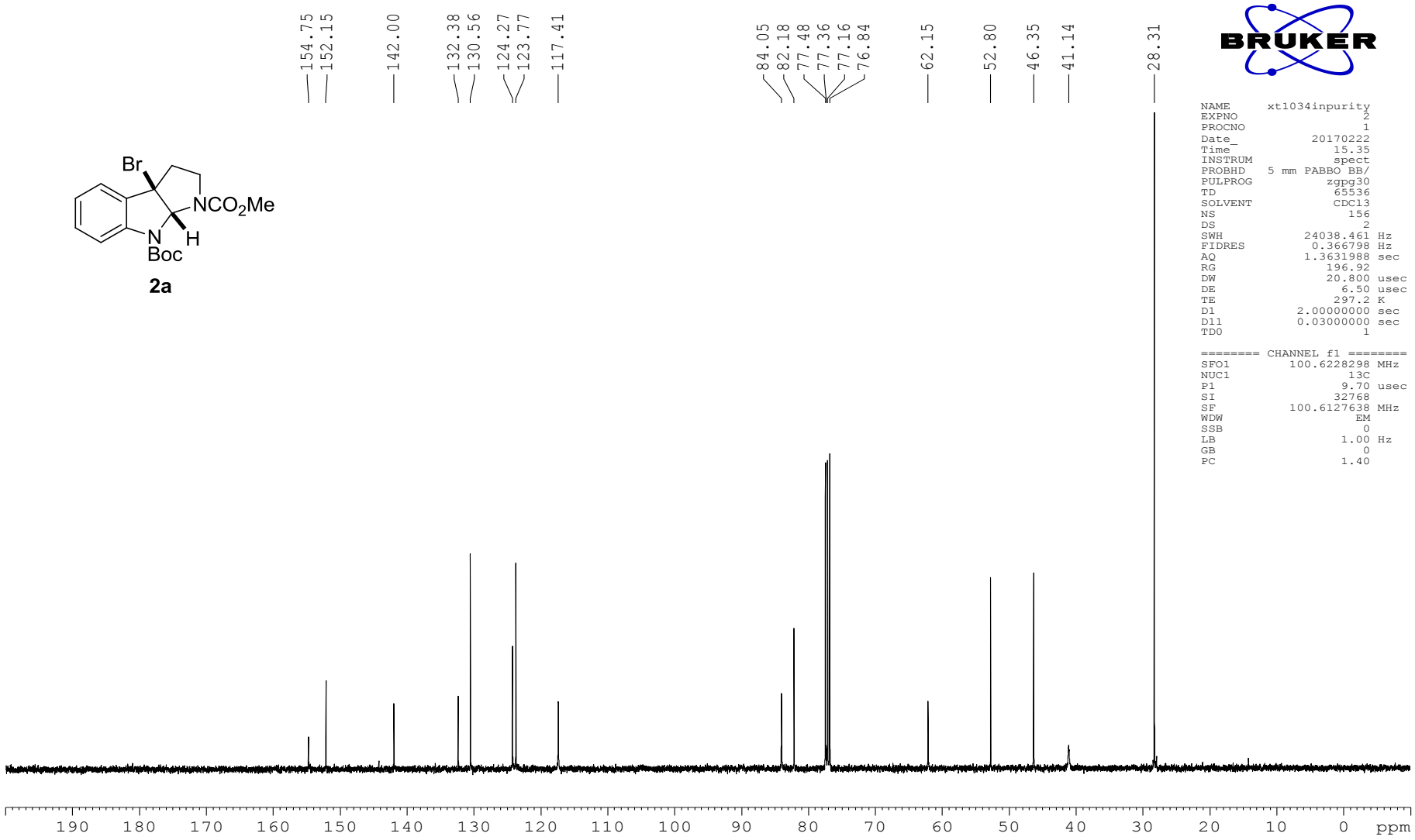
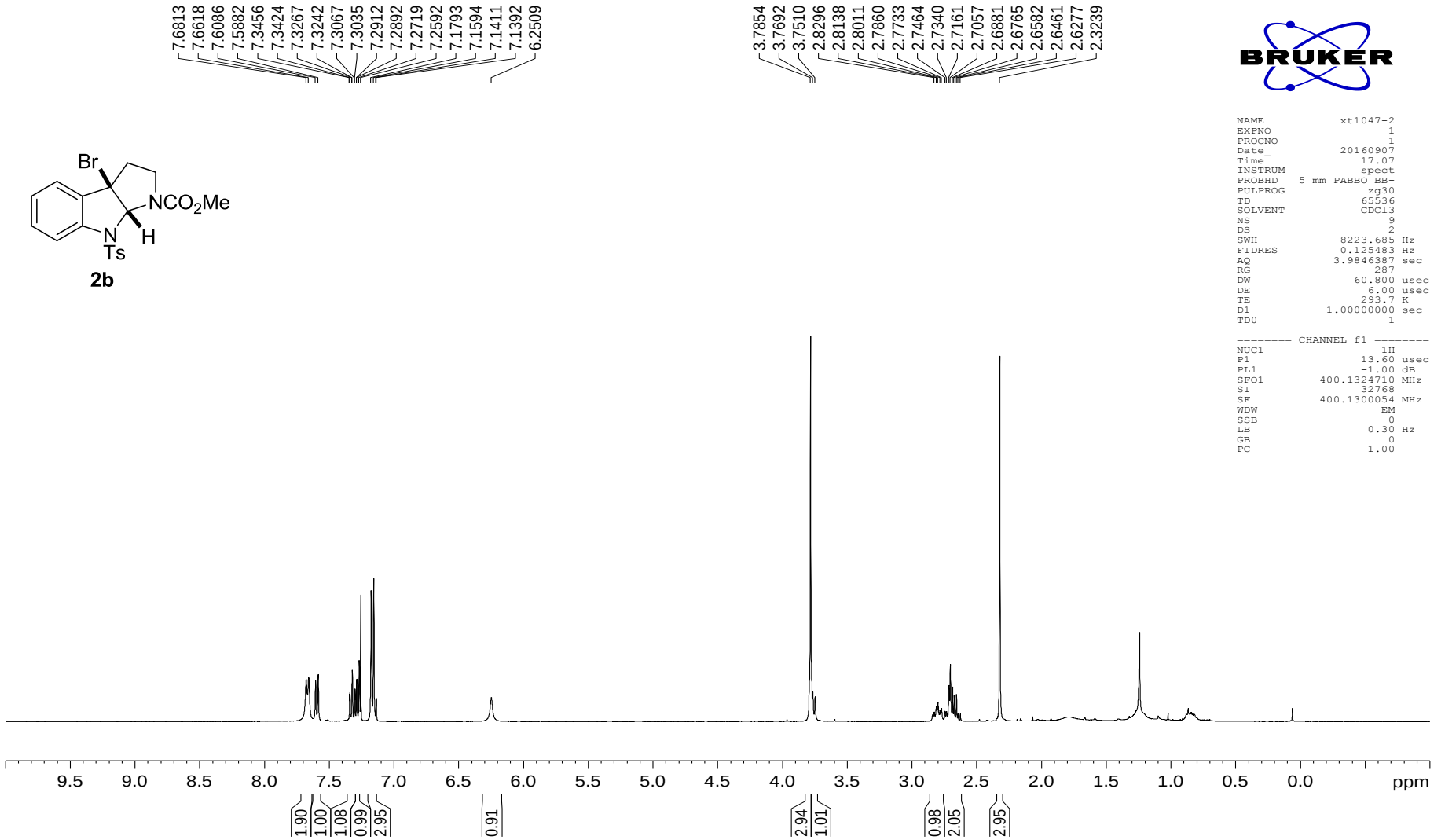
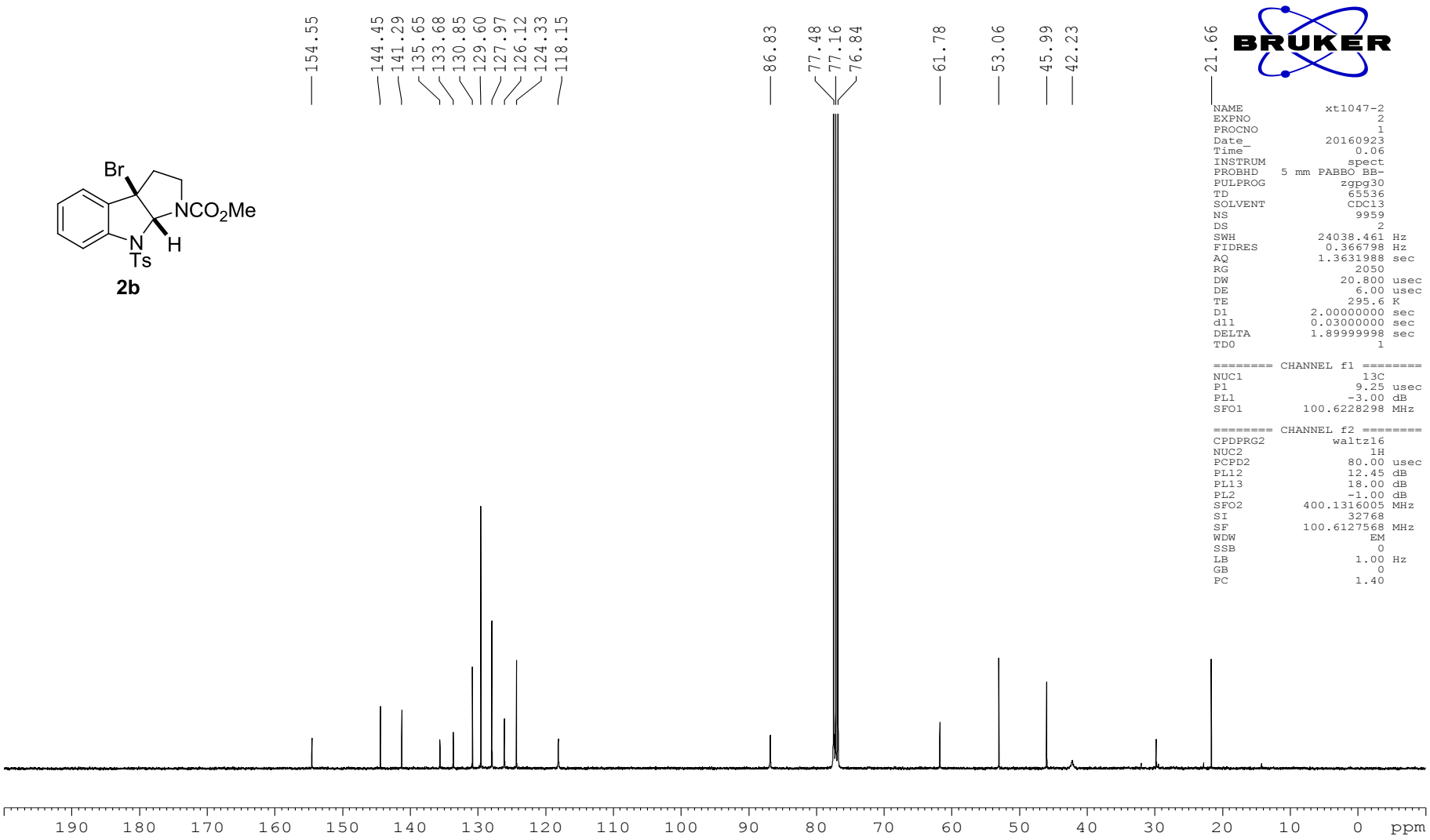
8.2438
8.0469
8.0249
7.9951
7.9746
7.5300
7.5107
7.4217
7.3817
7.3632
7.3440
7.3034
7.2845
7.2601
3.9324
3.9165
3.9006
2.9490
2.9332
2.9174
OH
N
(4-Ns)
1x
BRUKER
NAME xt1110-2
EXPNO 1
PROCNO 1
Date_ 20161027
Time_ 19.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 126.97
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.96
1.98
1.00
0.98
0.94
1.07
0.86
2.20
2.24


150.73
143.47
135.32
131.30
128.16
125.60
124.63
124.11
123.45
121.43
120.02
113.81
77.48
77.16
76.84
61.80
28.45
OH
N
(4-Ns)
1x
BRUKER
NAME xt1110-2
EXPNO 2
PROCNO 1
Date_ 20161027
Time 19.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 348
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.6621
7.6424
7.3865
7.3675
7.3295
7.3103
7.2878
7.1288
7.1100
7.0913
6.3967
3.8289
3.8074
3.7816
3.7496
2.9138
2.9015
2.8848
2.8719
2.8556
2.8368
2.8244
2.7926
2.7710
2.7604
2.7411
1.6036
Br
NCO2Me
N
H
Boc
2a
BRUKER
NAME xt1034inpurity
EXPNO 1
PROCNO 1
Date_ 20170222
Time 15.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 34.77
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.08
1.12
1.29
1.06
1.00
1.08
3.23
2.17
1.03
9.50


154.75
152.15
142.00
132.38
130.56
124.27
123.77
117.41
84.05
82.18
77.48
77.36
77.16
76.84
62.15
52.80
46.35
41.14
28.31
Br
NCO2Me
N
H
Boc
2a
NAME xt1034inpurity
EXPNO 2
PROCNO 1
Date_ 20170222
Time_ 15.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 156
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127638 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.6813
7.6618
7.6086
7.5882
7.3456
7.3424
7.3267
7.3242
7.3067
7.3035
7.2912
7.2892
7.2719
7.2592
7.1793
7.1594
7.1411
7.1392
6.2509
3.7854
3.7692
3.7510
2.8296
2.8138
2.8011
2.7860
2.7733
2.7464
2.7340
2.7161
2.7057
2.6881
2.6765
2.6582
2.6461
2.6277
2.3239
Br
NCO2Me
N
H
Ts
2b
NAME xt1047-2
EXPNO 1
PROCNO 1
Date_ 20160907
Time 17.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.00 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.90
1.00
1.08
0.99
2.95
0.91
2.94
1.01
0.98
2.05
2.95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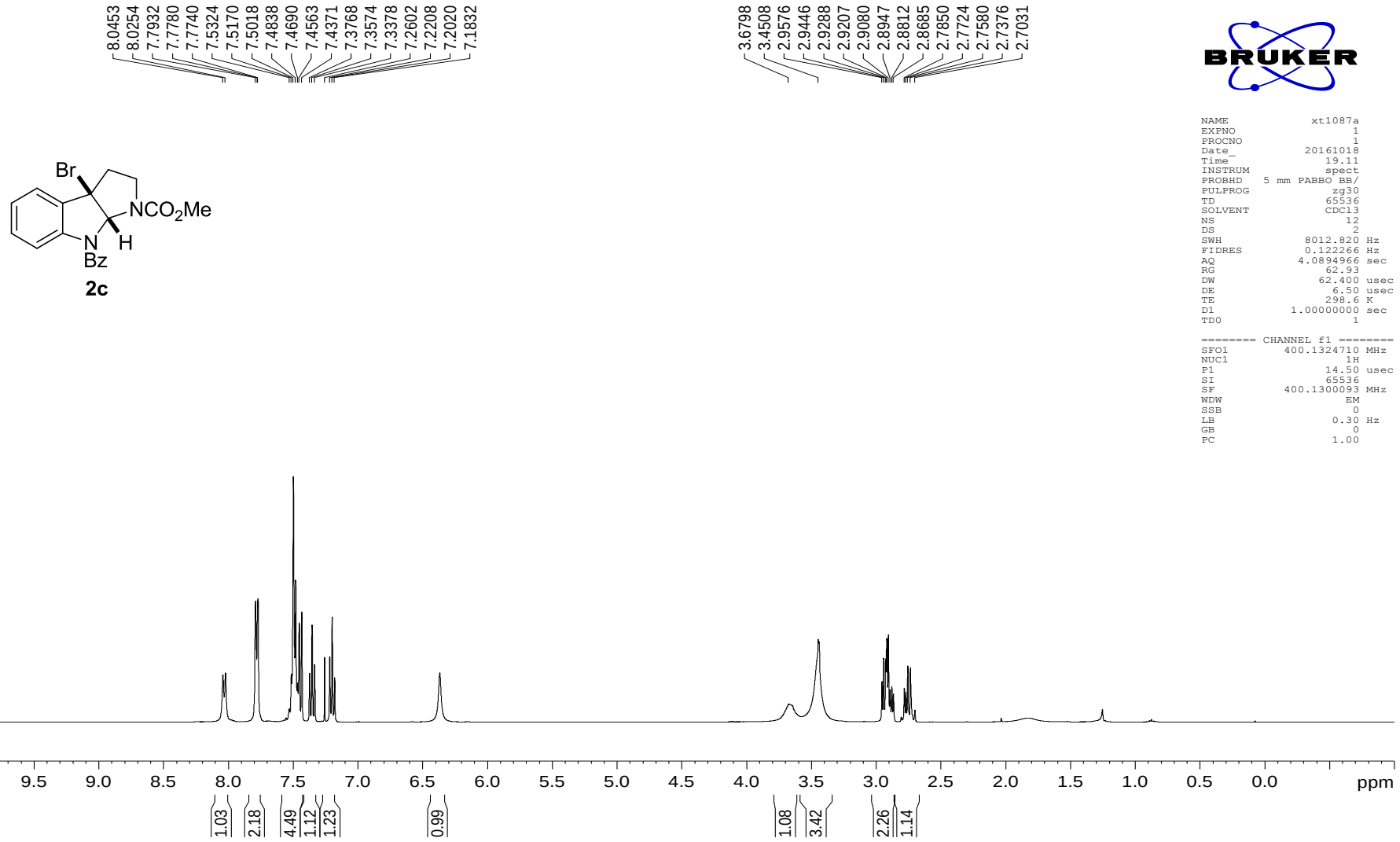
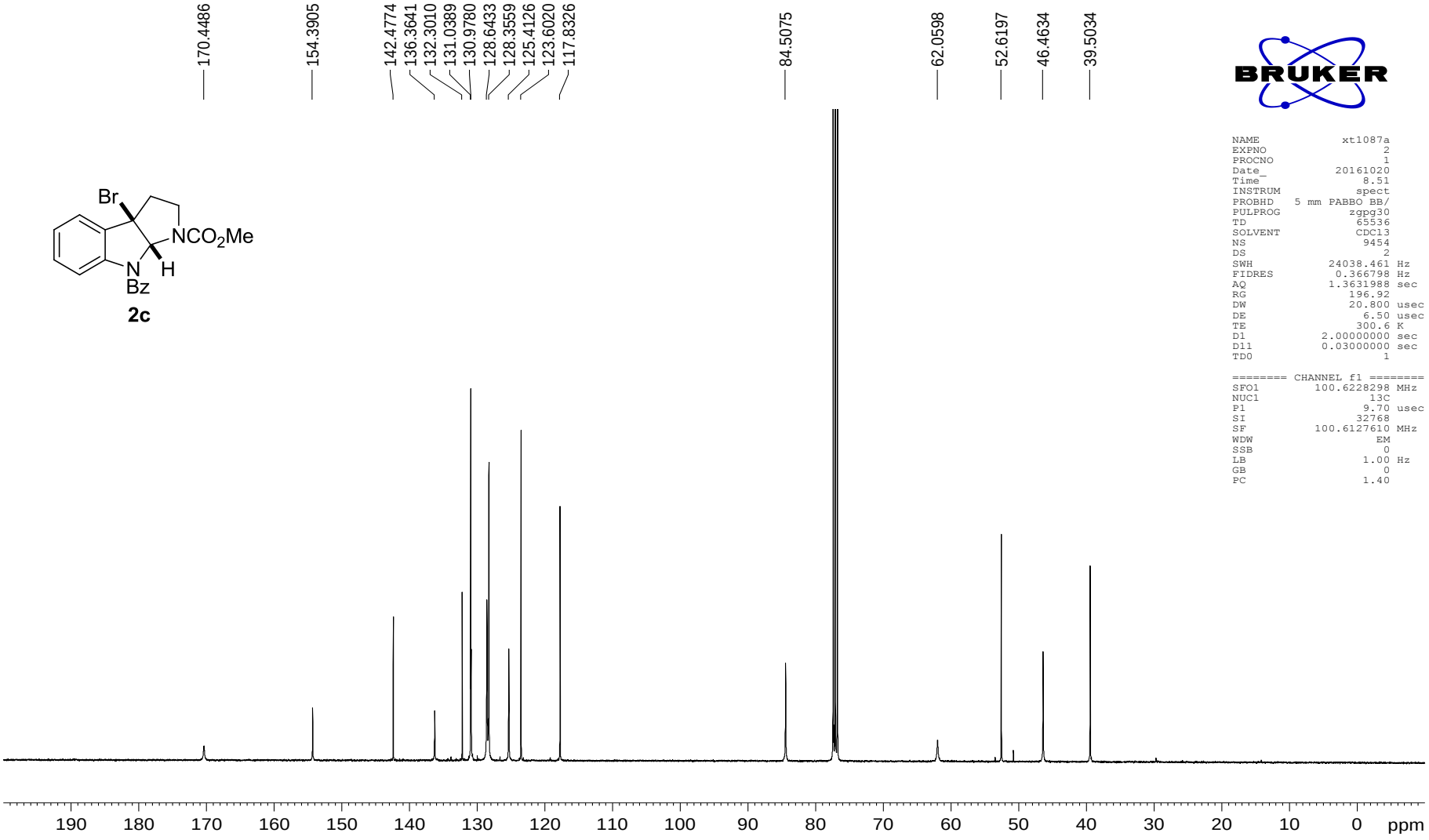
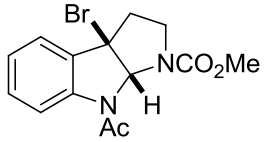
144.45
141.29
135.65
133.68
130.85
129.60
127.97
126.12
124.33
118.15
86.83
77.48
77.16
76.84
61.78
53.06
45.99
42.23
21.66
Br
NCO2Me
N
H
Ts
2b
BRUKER
NAME xt1047-2
EXPNO 2
PROCNO 1
Date_ 20160923
Time 0.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9959
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127568 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

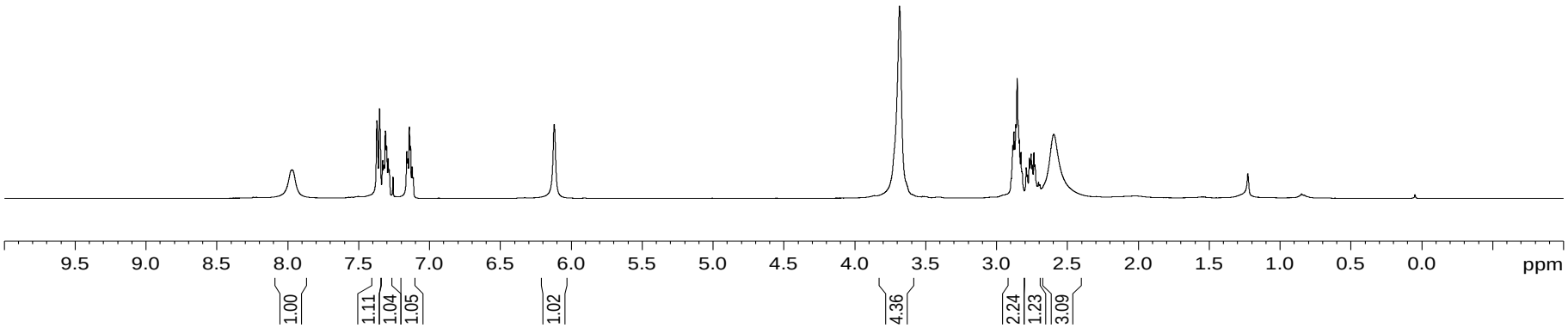

8.0453
8.0254
7.7932
7.7780
7.7740
7.5324
7.5170
7.5018
7.4838
7.4690
7.4563
7.4371
7.3768
7.3574
7.3378
7.2602
7.2208
7.2020
7.1832
3.6798
3.4508
2.9576
2.9446
2.9288
2.9207
2.9080
2.8947
2.8812
2.8685
2.7850
2.7724
2.7580
2.7376
2.7031
Br
NCO2Me
N
H
Bz
2c
NAME xt1087a
EXPNO 1
PROCNO 1
Date_ 20161018
Time 19.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 62.93
DW 62.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.03
2.18
4.49
1.12
1.23
0.99
1.08
3.42
2.26
1.14

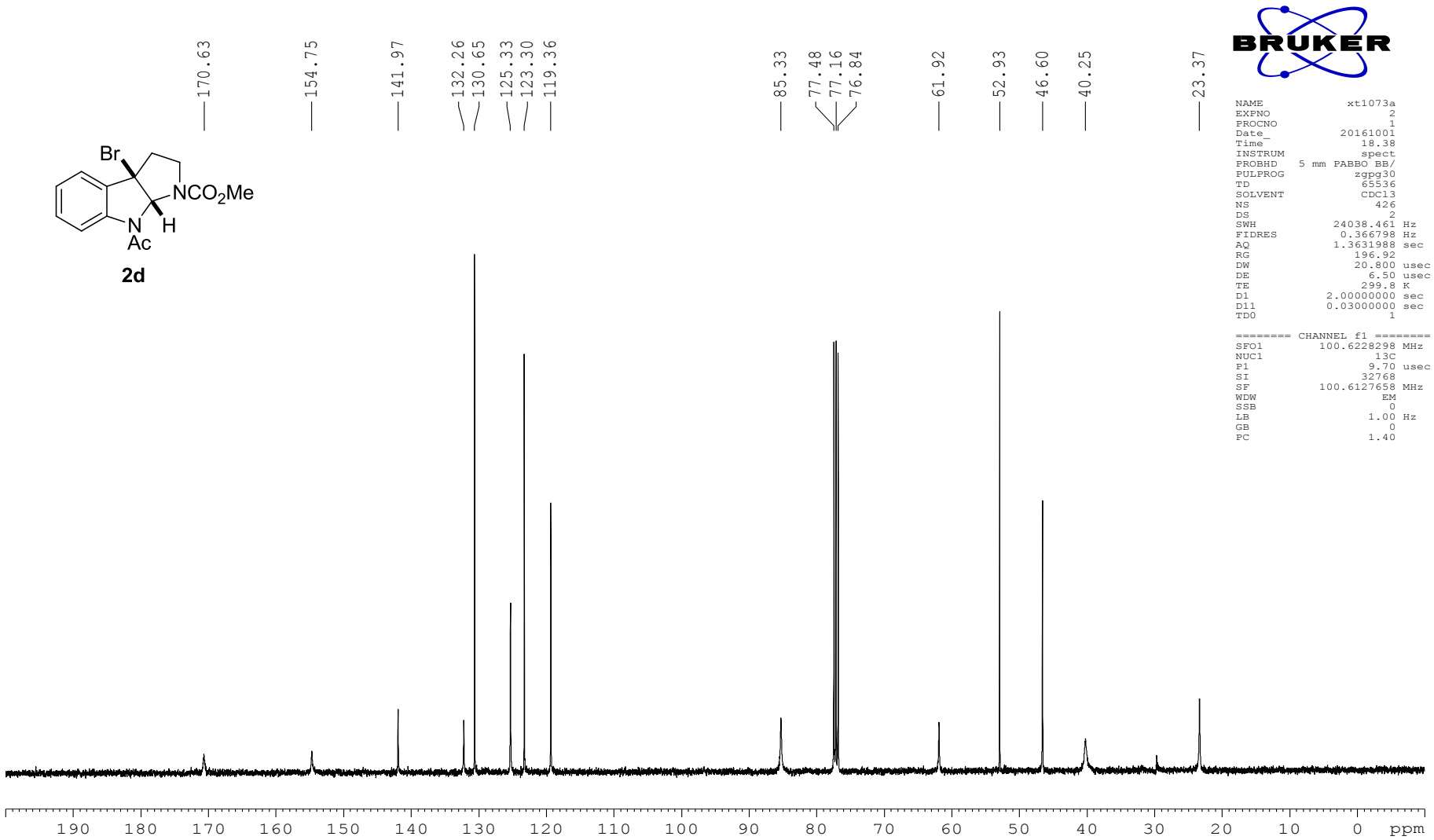

170.4486
154.3905
142.4774
136.3641
132.3010
131.0389
130.9780
128.6433
128.3559
125.4126
123.6020
117.8326
84.5075
62.0598
52.6197
46.4634
39.5034
Br
NCO2Me
N
H
Bz
2c
BRUKER
NAME xt1087a
EXPNO 2
PROCNO 1
Date_ 20161020
Time 8.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9454
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127610 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

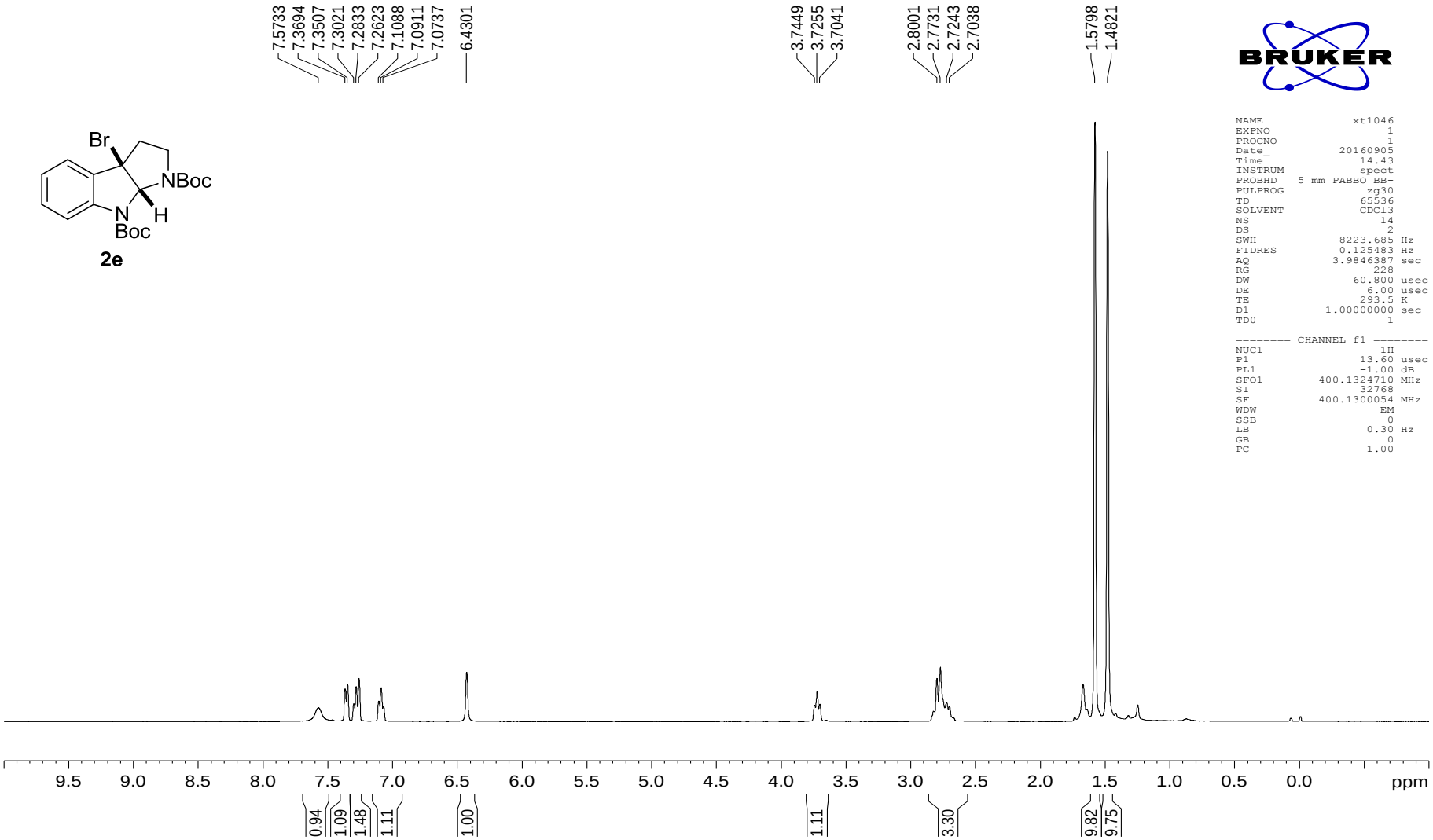


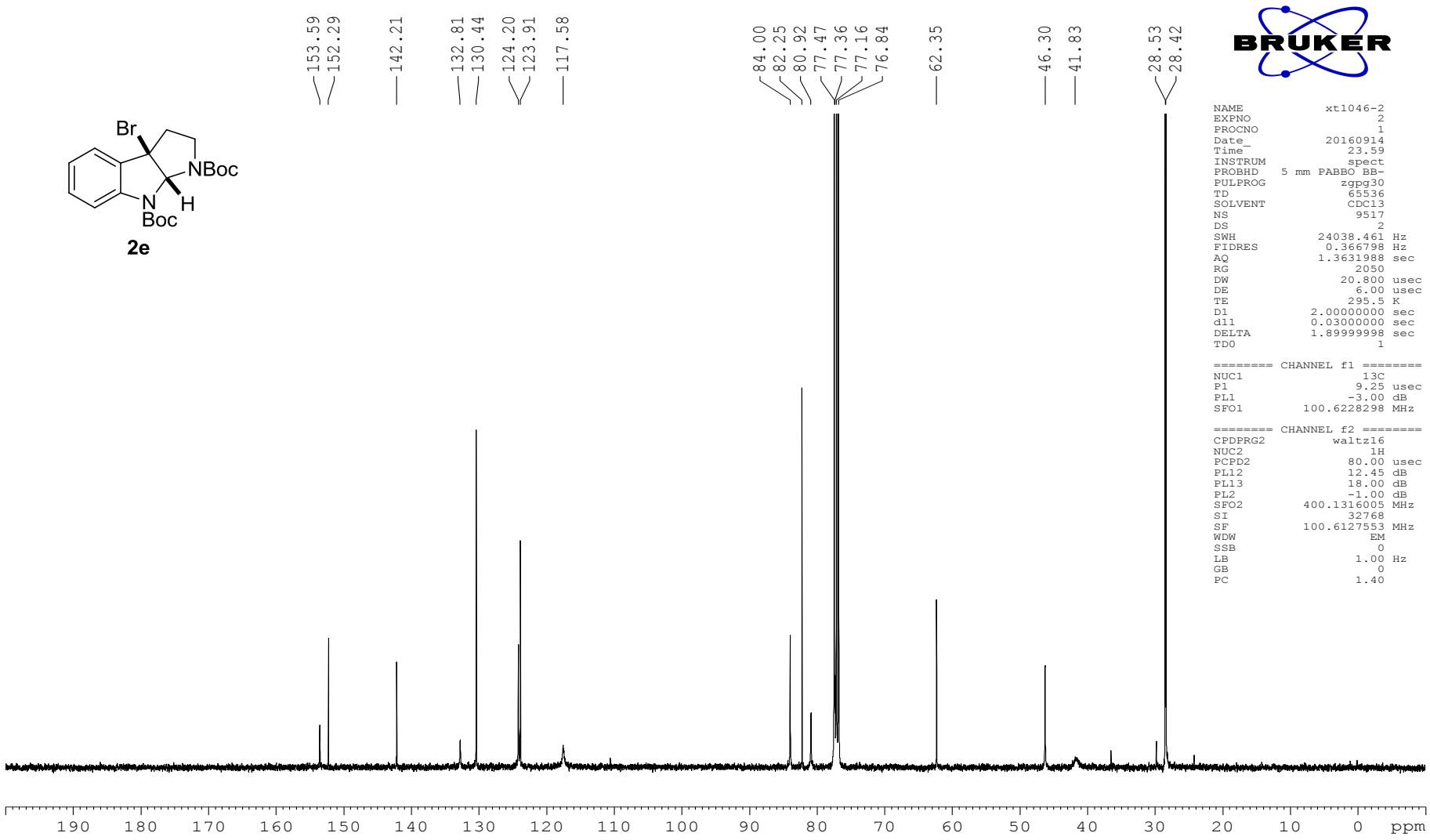
2d

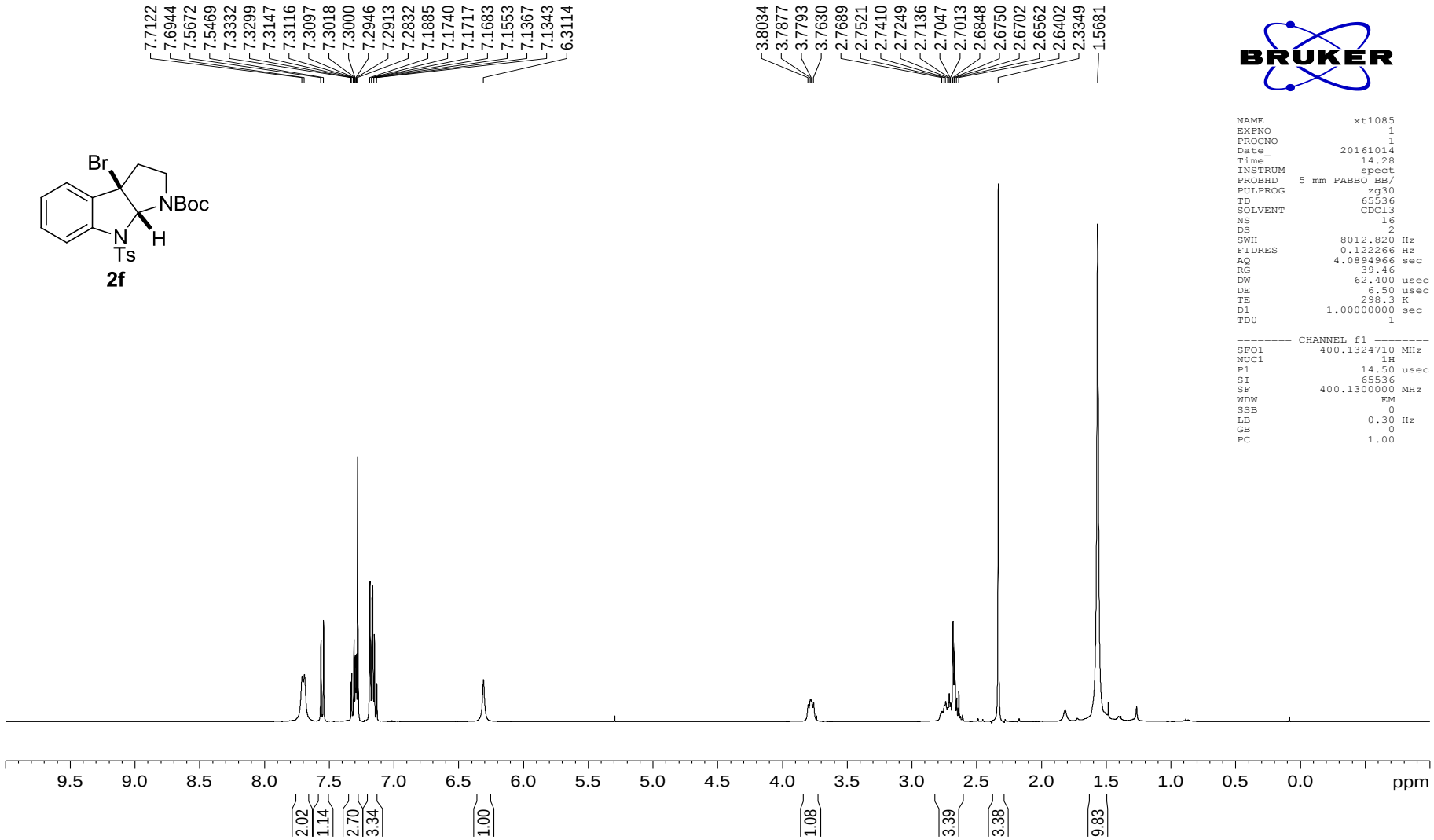
```
NAME                xt1073a
EXPNO                     1
PROCNO                    1
Date_              20161001
Time                  18.32
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       15
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                     45.67
DW                    62.400 usec
DE                     6.50 usec
TE                    298.8 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                    1H
P1                    14.50 usec
SI                    65536
SF             400.1300089 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

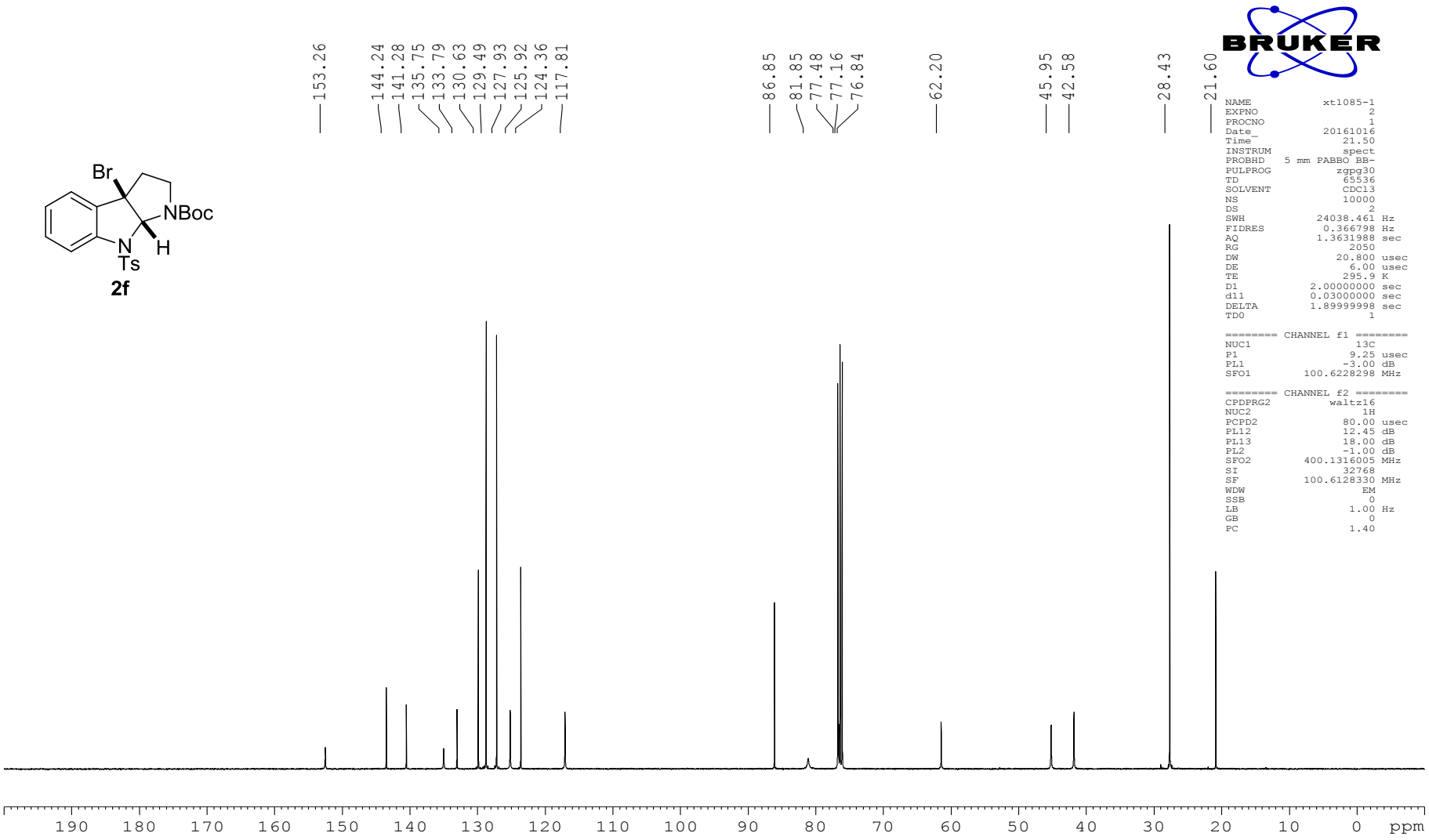


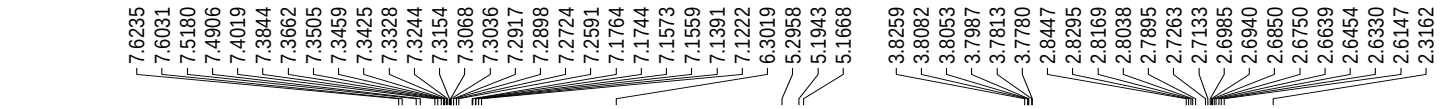

170.63
154.75
141.97
132.26
130.65
125.33
123.30
119.36
85.33
77.48
77.16
76.84
61.92
52.93
46.60
40.25
23.37
Br
NCO2Me
N
Ac
H
2d
BRUKER
NAME xt1073a
EXPNO 2
PROCNO 1
Date_ 20161001
Time 18.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 426
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127658 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

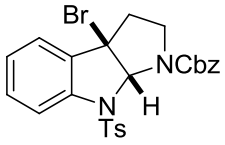

7.5733
7.3694
7.3507
7.3021
7.2833
7.2623
7.1088
7.0911
7.0737
6.4301
3.7449
3.7255
3.7041
2.8001
2.7731
2.7243
2.7038
1.5798
1.4821
Br
NBoc
N
H
Boc
2e
BRUKER
NAME xt1046
EXPNO 1
PROCNO 1
Date_ 20160905
Time 14.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 14
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 228
DW 60.800 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.94
1.09
1.48
1.11
1.00
1.11
3.30
9.82
9.75


153.59
152.29
142.21
132.81
130.44
124.20
123.91
117.58
84.00
82.25
80.92
77.47
77.36
77.16
76.84
62.35
46.30
41.83
28.53
28.42
Br
NBoc
N
H
Boc
2e
BRUKER
NAME xt1046-2
EXPNO 2
PROCNO 1
Date_ 20160914
Time 23.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9517
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.7122
7.6944
7.5672
7.5469
7.3332
7.3299
7.3147
7.3116
7.3097
7.3018
7.3000
7.2946
7.2913
7.2832
7.1885
7.1740
7.1717
7.1683
7.1553
7.1367
7.1343
6.3114
3.8034
3.7877
3.7793
3.7630
2.7689
2.7521
2.7410
2.7249
2.7136
2.7047
2.7013
2.6848
2.6750
2.6702
2.6562
2.6402
2.3349
1.5681
BRUKER
NAME xt1085
EXPNO 1
PROCNO 1
Date_ 20161014
Time 14.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 39.46
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
NBoc
N
H
Ts
2f
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.02
1.14
2.70
3.34
1.00
1.08
3.39
3.38
9.83

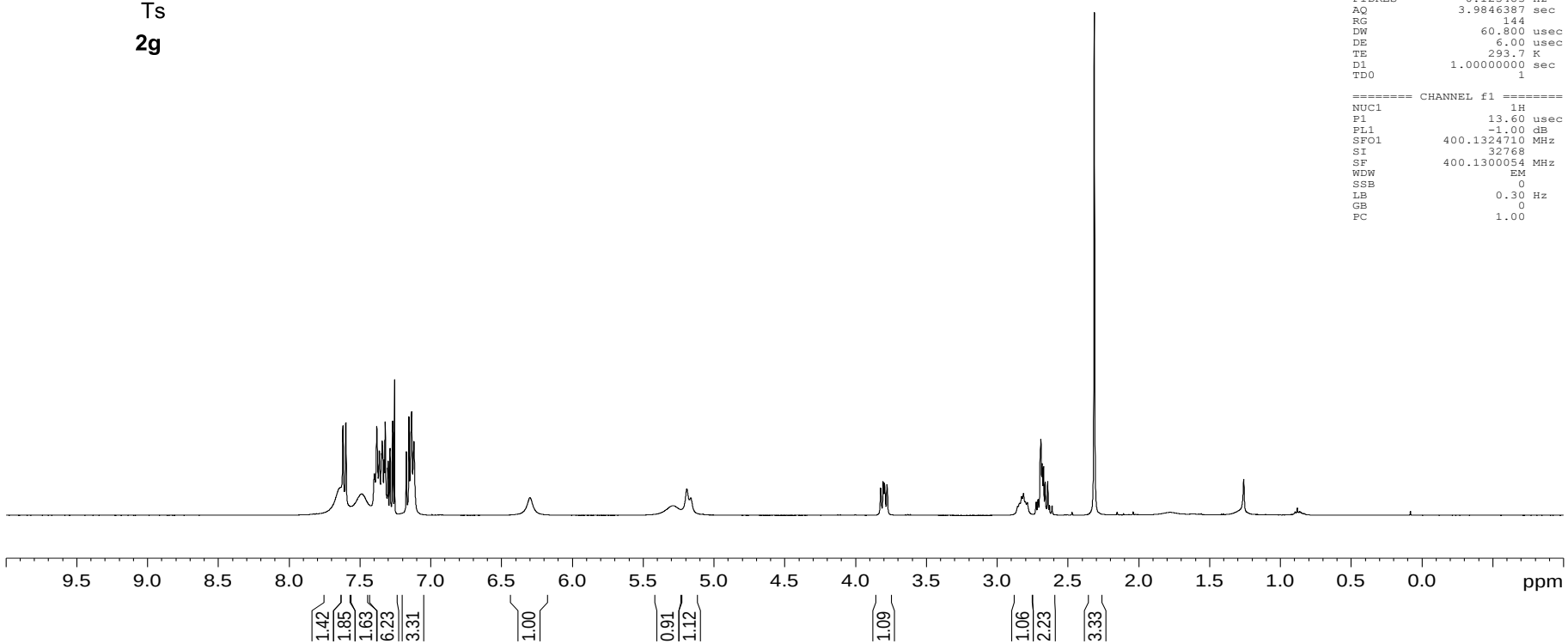

153.26
144.24
141.28
135.75
133.79
130.63
129.49
127.93
125.92
124.36
117.81
86.85
81.85
77.48
77.16
76.84
62.20
45.95
42.58
28.43
21.60
Br
NBoc
N
H
Ts
2f
BRUKER
NAME xt1085-1
EXPNO 2
PROCNO 1
Date_ 20161016
Time 21.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10000
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6128330 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

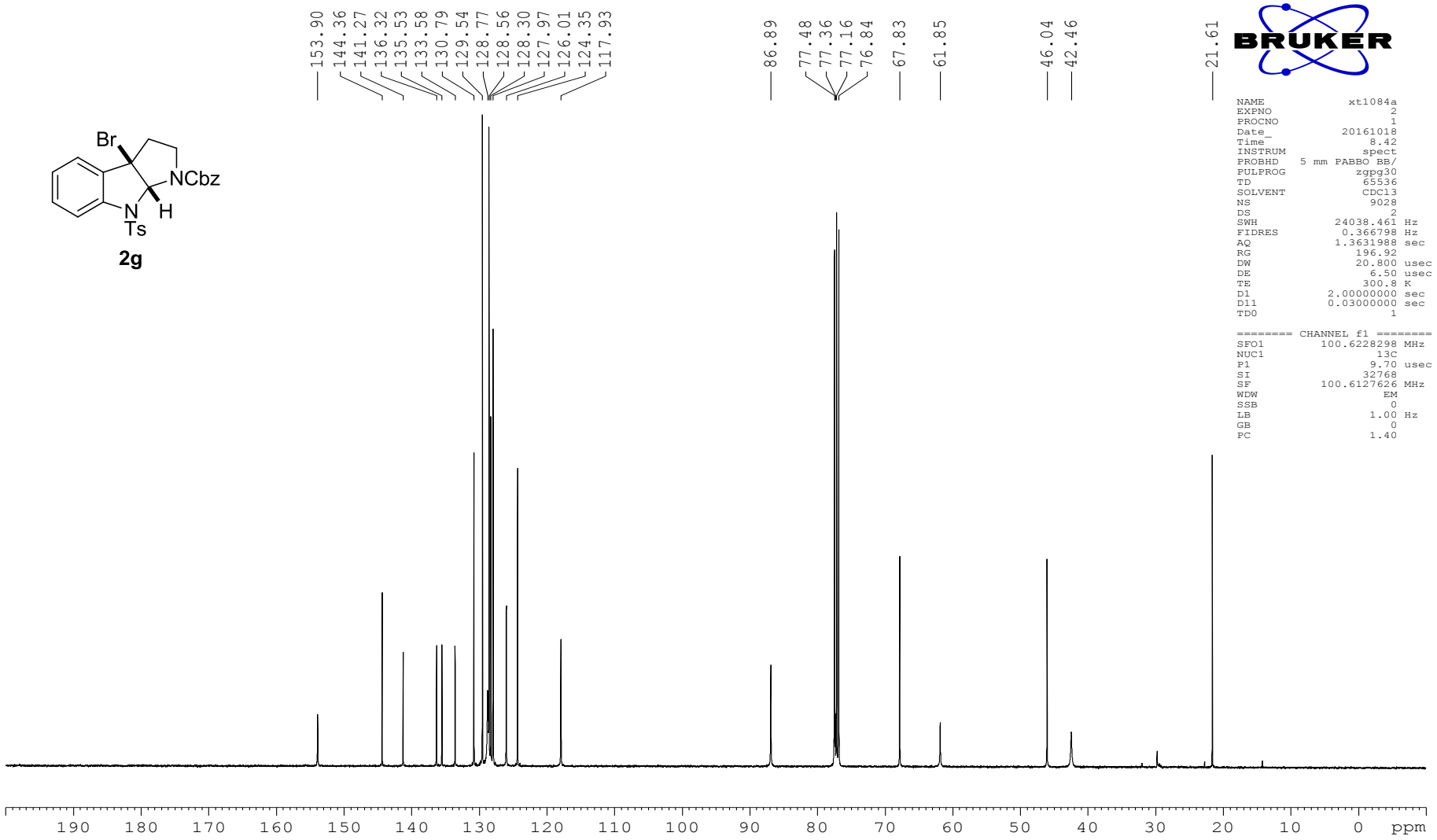


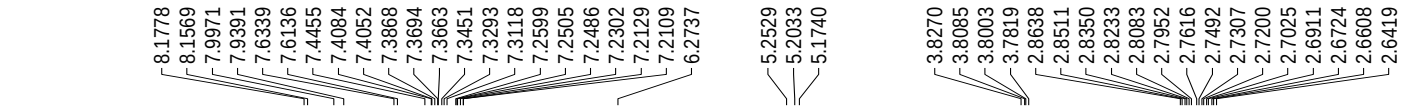


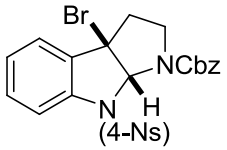
2g

```
NAME                xt1084a
EXPNO                     1
PROCNO                    1
Date_              20161015
Time_                 14.54
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      144
DW                   60.800 usec
DE                    6.00 usec
TE                   293.7 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.60 usec
PL1                  -1.00 dB
SFO1          400.1324710 MHz
SI                    32768
SF            400.1300054 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



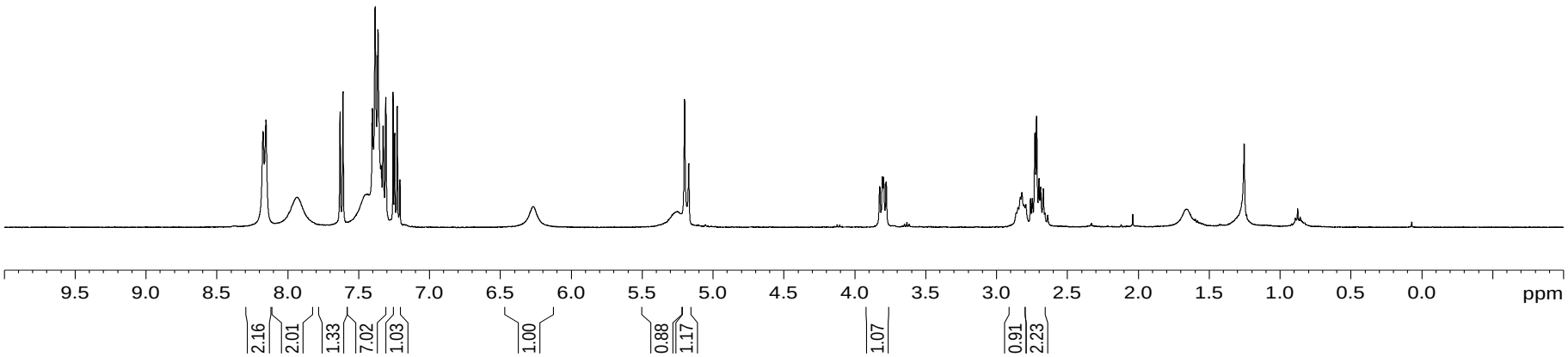

153.90
144.36
141.27
136.32
135.53
133.58
130.79
129.54
128.77
128.56
128.30
127.97
126.01
124.35
117.93
86.89
77.48
77.36
77.16
76.84
67.83
61.85
46.04
42.46
21.61
Br
NCbz
N
H
Ts
2g
BRUKER
NAME xt1084a
EXPNO 2
PROCNO 1
Date_ 20161018
Time_ 8.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9028
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127626 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

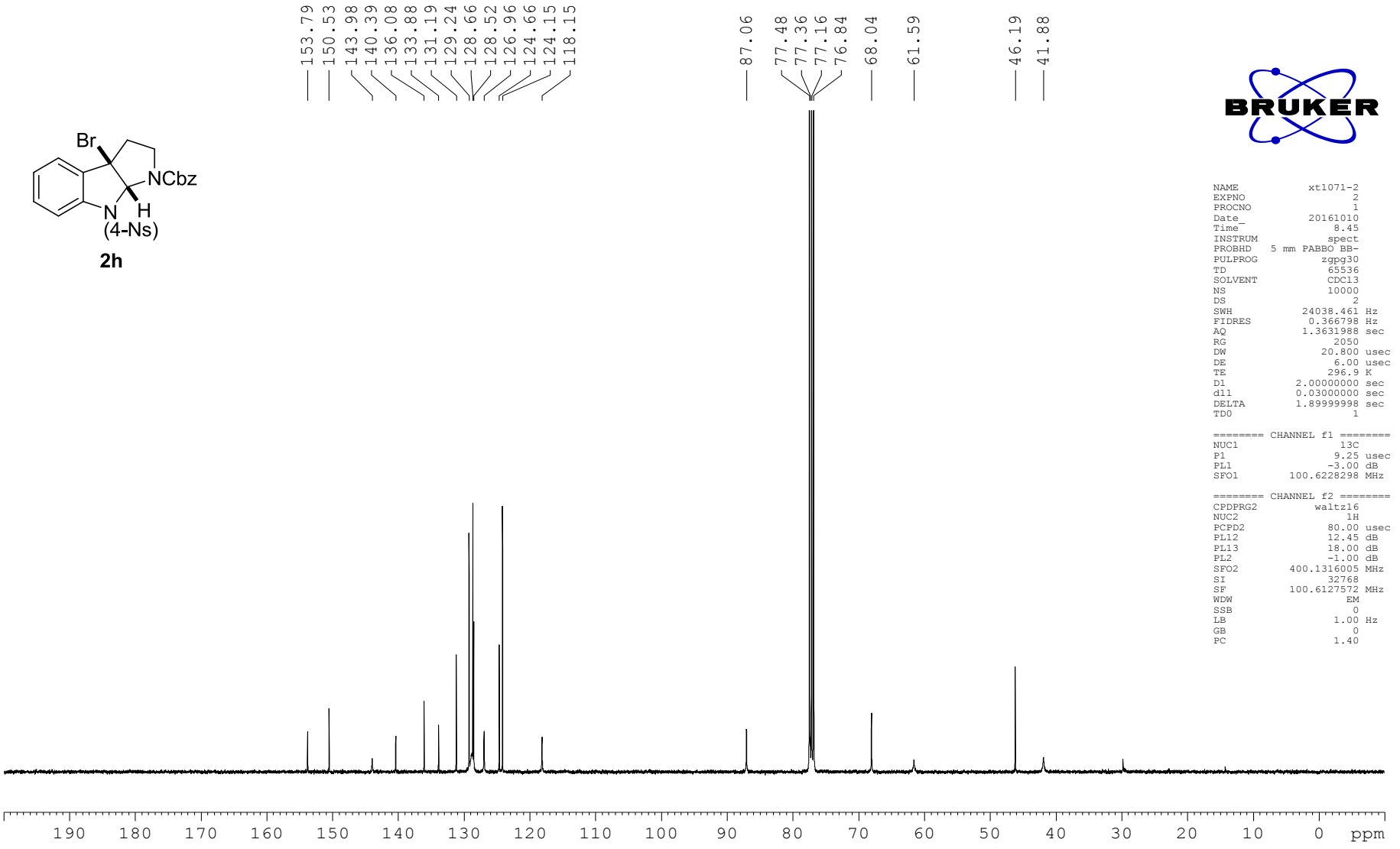


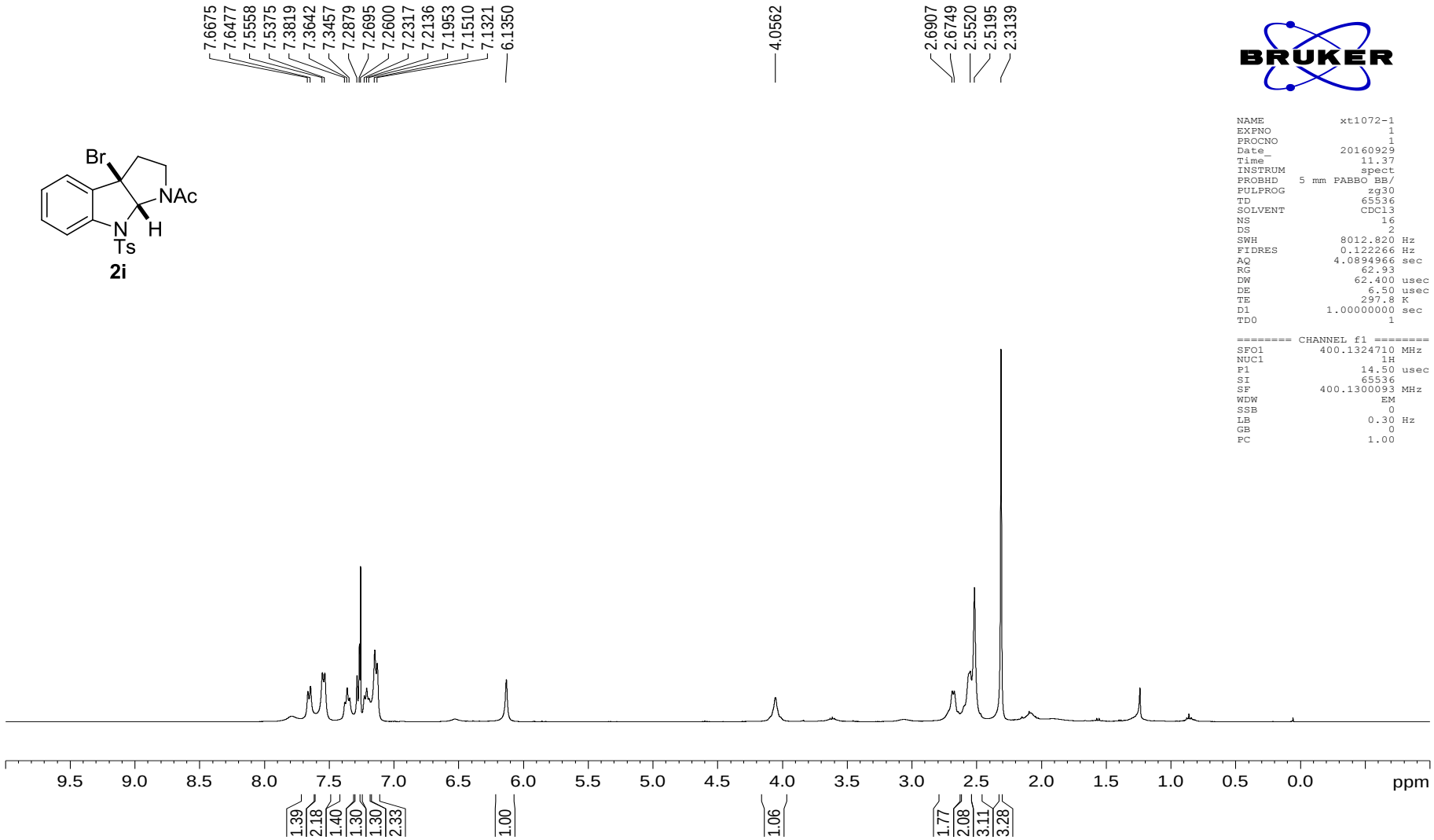


2h

```
NAME                xt1071
EXPNO                    1
PROCNO                   1
Date_             20161005
Time                 11.12
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                    88.84
DW                  62.400 usec
DE                   6.50 usec
TE                  298.3 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                   1H
P1                  14.50 usec
SI                  65536
SF             400.1300093 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```




153.79
150.53
143.98
140.39
136.08
133.88
131.19
129.24
128.66
128.52
126.96
124.66
124.15
118.15
87.06
77.48
77.36
77.16
76.84
68.04
61.59
46.19
41.88
Br
NCbz
N H
(4-Ns)
2h
BRUKER
NAME xt1071-2
EXPNO 2
PROCNO 1
Date_ 20161010
Time 8.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10000
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127572 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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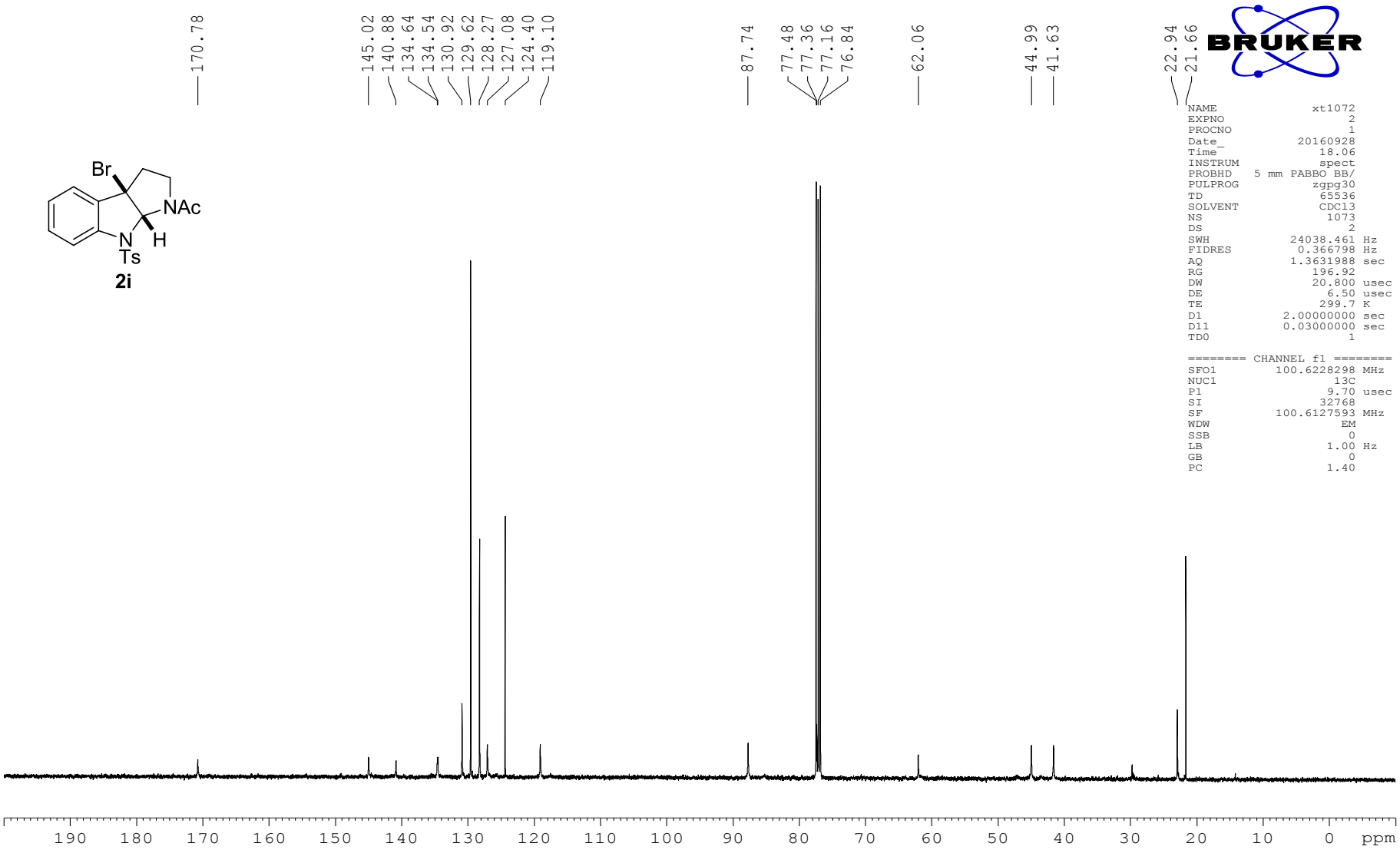
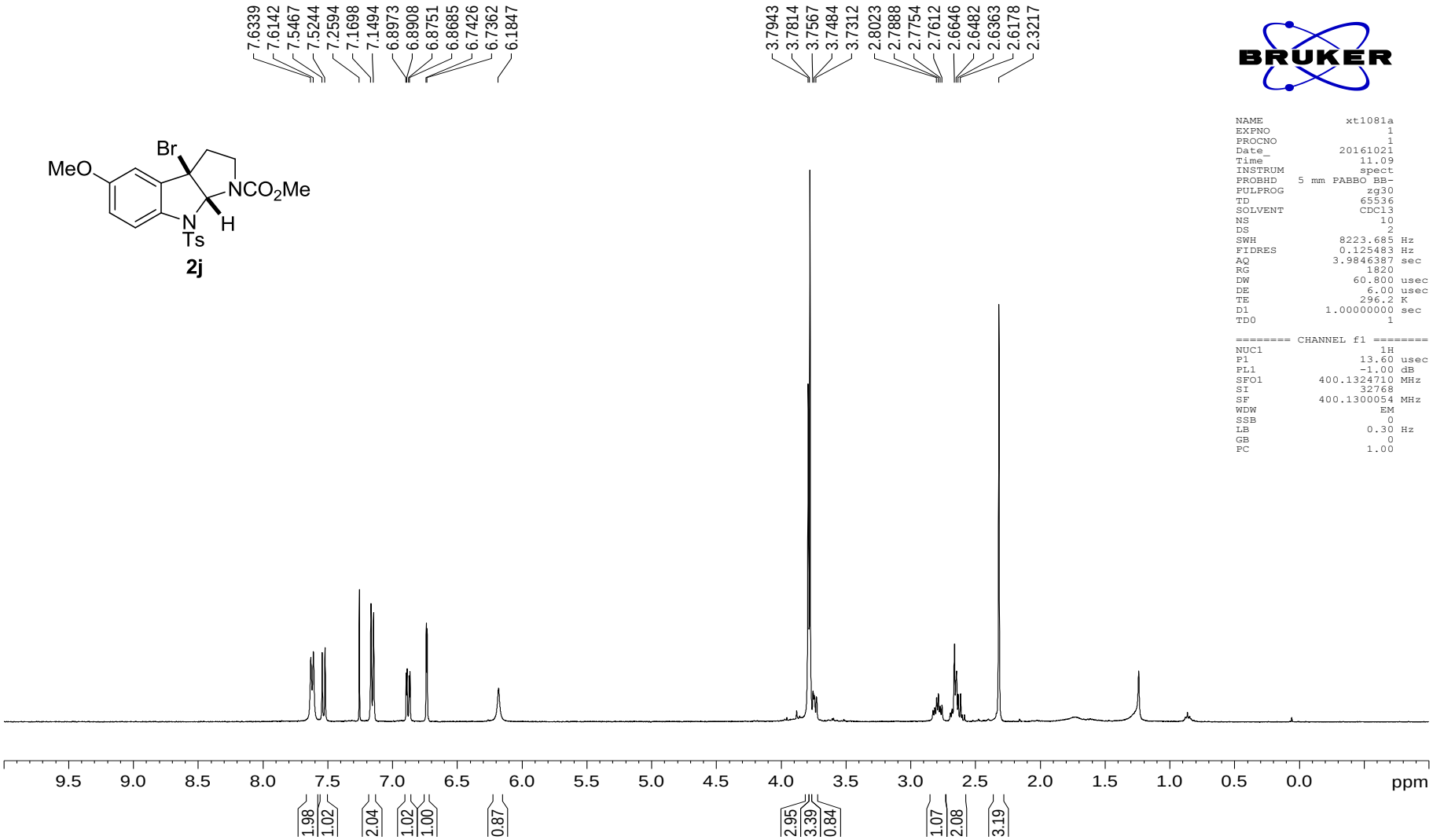
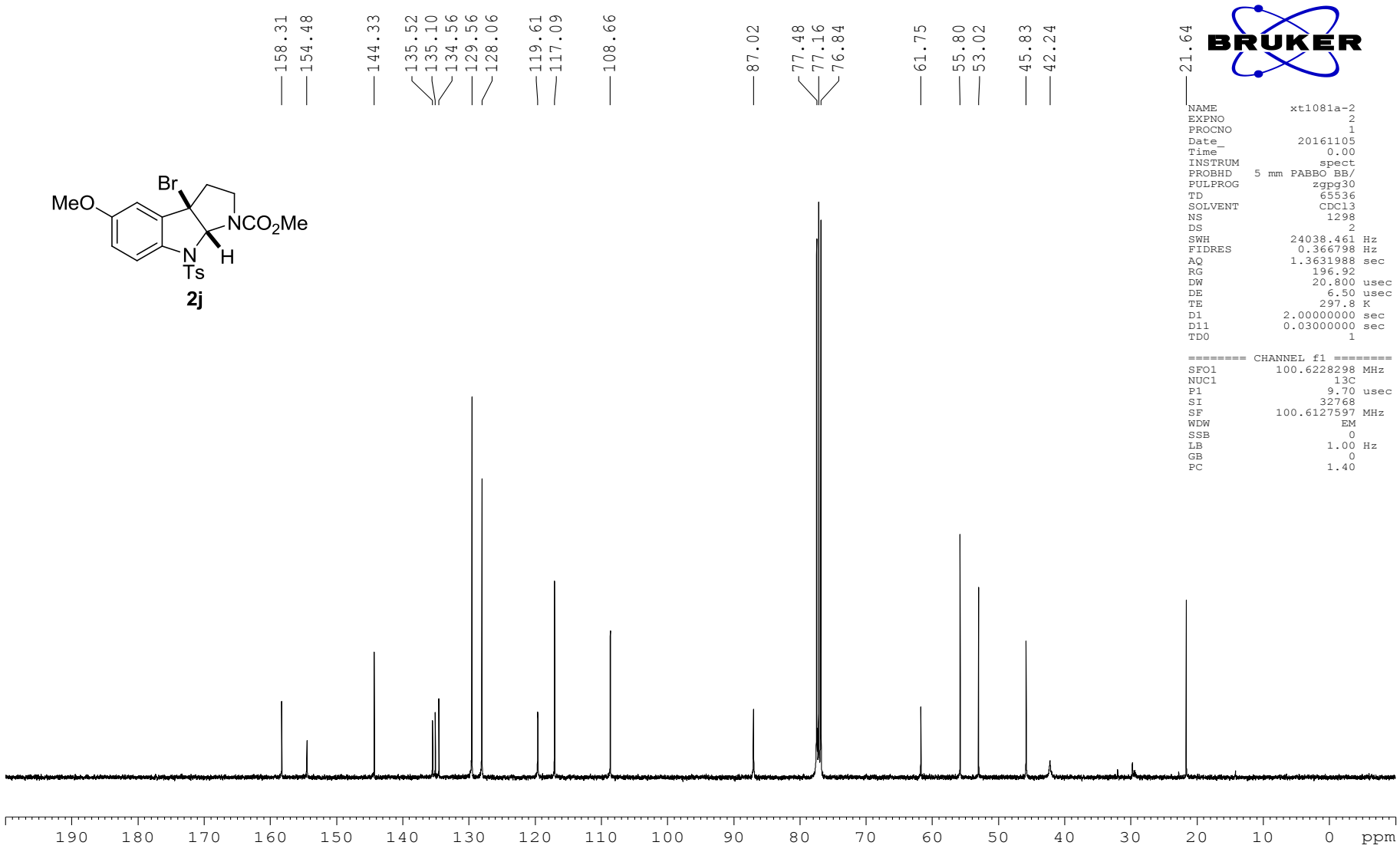
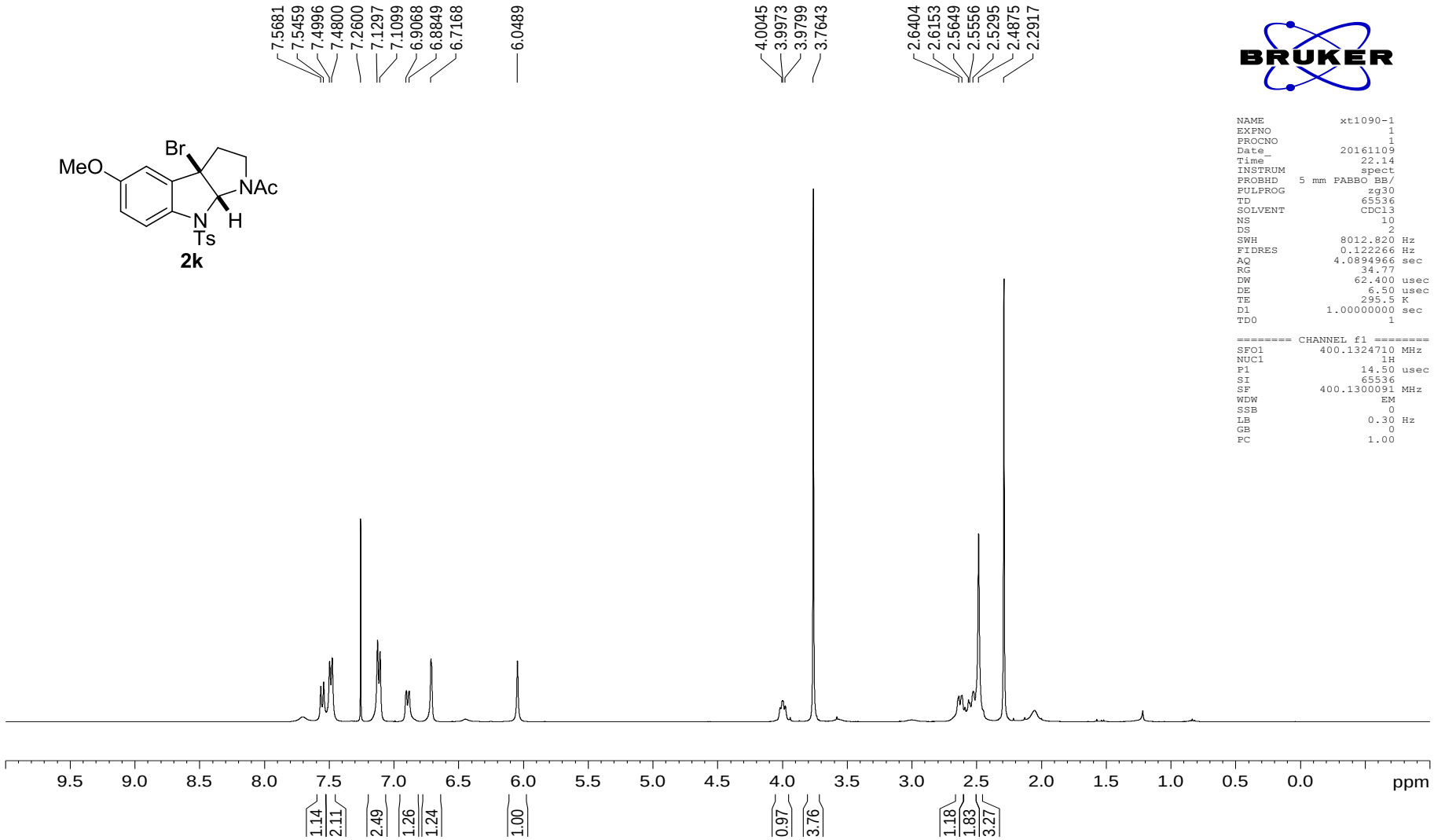
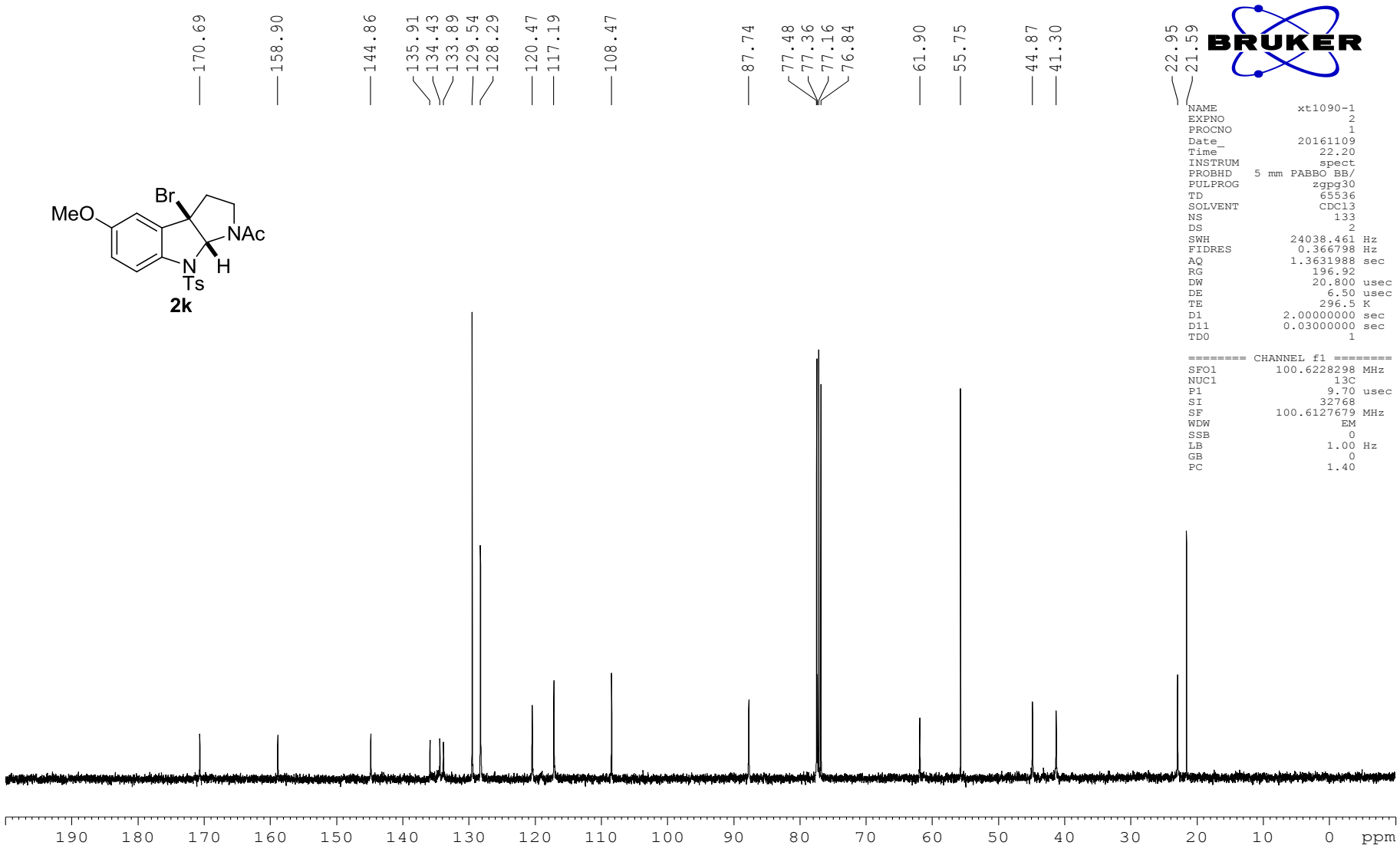
7.6477
7.5558
7.5375
7.3819
7.3642
7.3457
7.2879
7.2695
7.2600
7.2317
7.2136
7.1953
7.1510
7.1321
6.1350
4.0562
2.6907
2.6749
2.5520
2.5195
2.3139
Br
NAc
N
H
Ts
2i
BRUKER
NAME xt1072-1
EXPNO 1
PROCNO 1
Date_ 20160929
Time 11.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 62.93
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.39
2.18
1.40
1.30
1.30
2.33
1.00
1.06
1.77
2.08
3.11
3.28

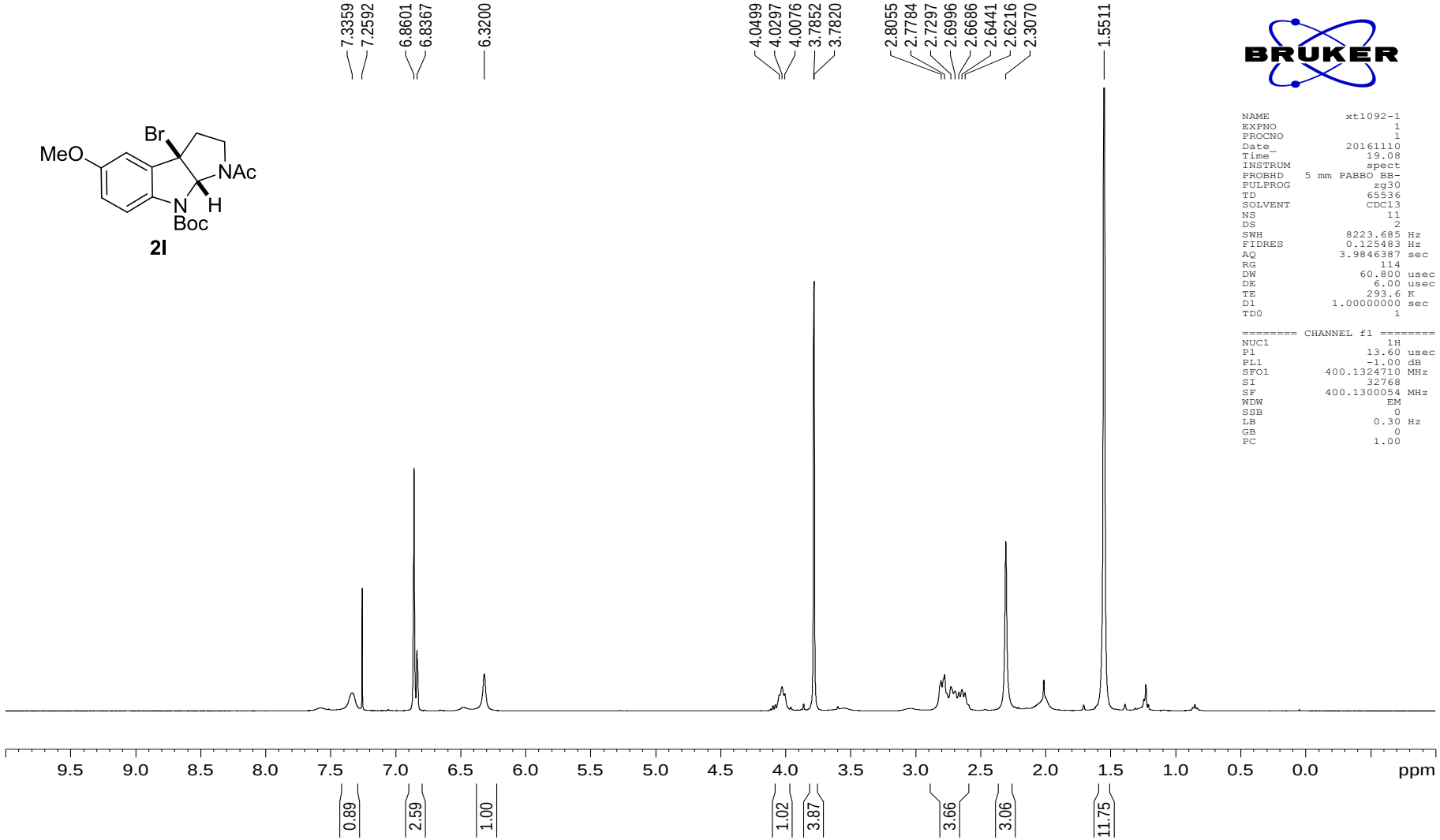

170.78
145.02
140.88
134.64
134.54
130.92
129.62
128.27
127.08
124.40
119.10
87.74
77.48
77.36
77.16
76.84
62.06
44.99
41.63
22.94
21.66
Br
NAc
N
H
Ts
2i
BRUKER
NAME xt1072
EXPNO 2
PROCNO 1
Date_ 20160928
Time 18.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1073
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127593 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

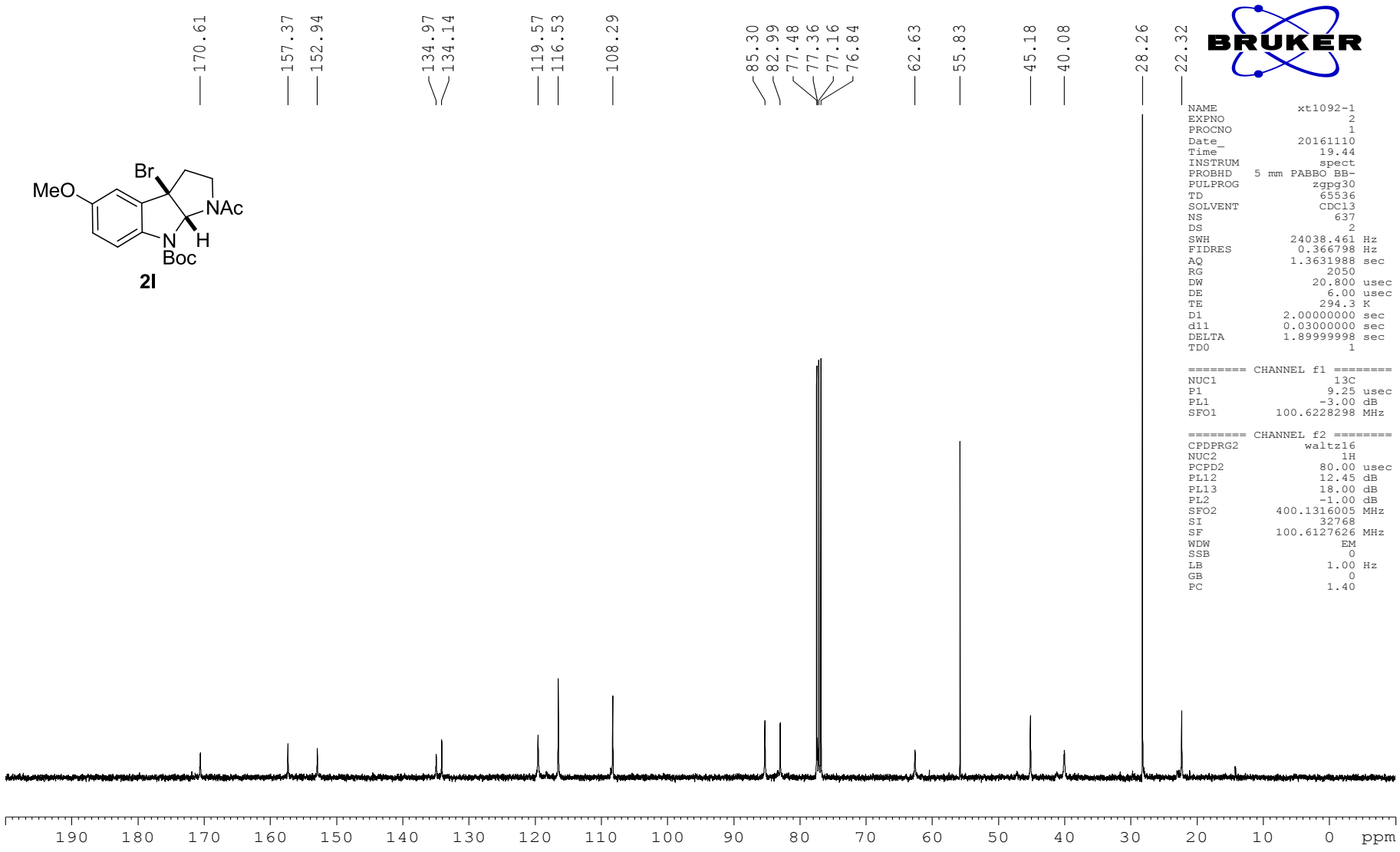

7.6339
7.6142
7.5467
7.5244
7.2594
7.1698
7.1494
6.8973
6.8908
6.8751
6.8685
6.7426
6.7362
6.1847
3.7943
3.7814
3.7567
3.7484
3.7312
2.8023
2.7888
2.7754
2.7612
2.6646
2.6482
2.6363
2.6178
2.3217
MeO
Br
NCO2Me
N
Ts
H
2j
NAME xt1081a
EXPNO 1
PROCNO 1
Date_ 20161021
Time 11.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 1820
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.98
1.02
2.04
1.02
1.00
0.87
2.95
3.39
0.84
1.07
2.08
3.19

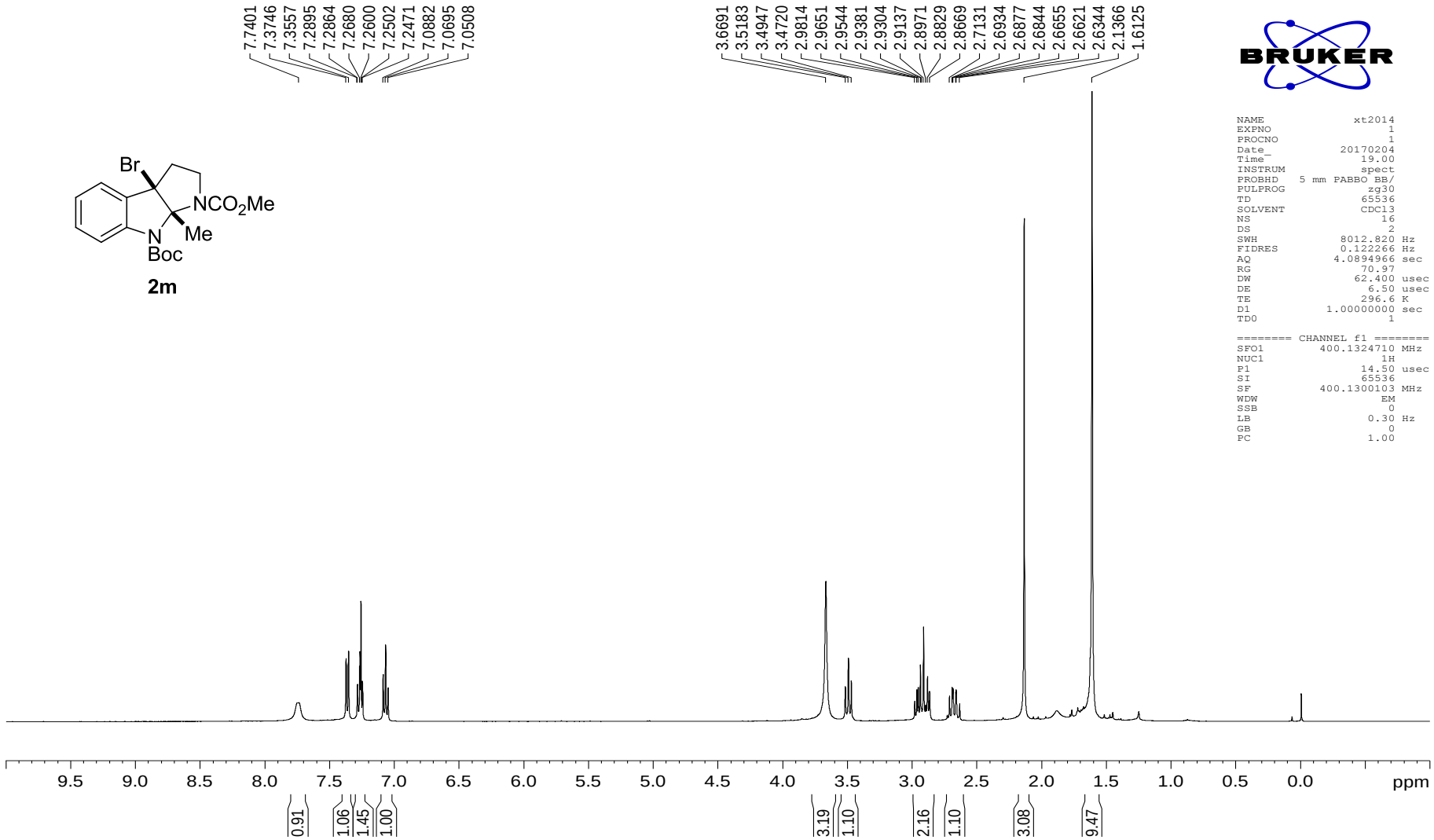

158.31
154.48
144.33
135.52
135.10
134.56
129.56
128.06
119.61
117.09
108.66
87.02
77.48
77.16
76.84
61.75
55.80
53.02
45.83
42.24
21.64
MeO
Br
NCO2Me
N
H
Ts
2j
NAME xt1081a-2
EXPNO 2
PROCNO 1
Date_ 20161105
Time 0.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1298
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127597 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

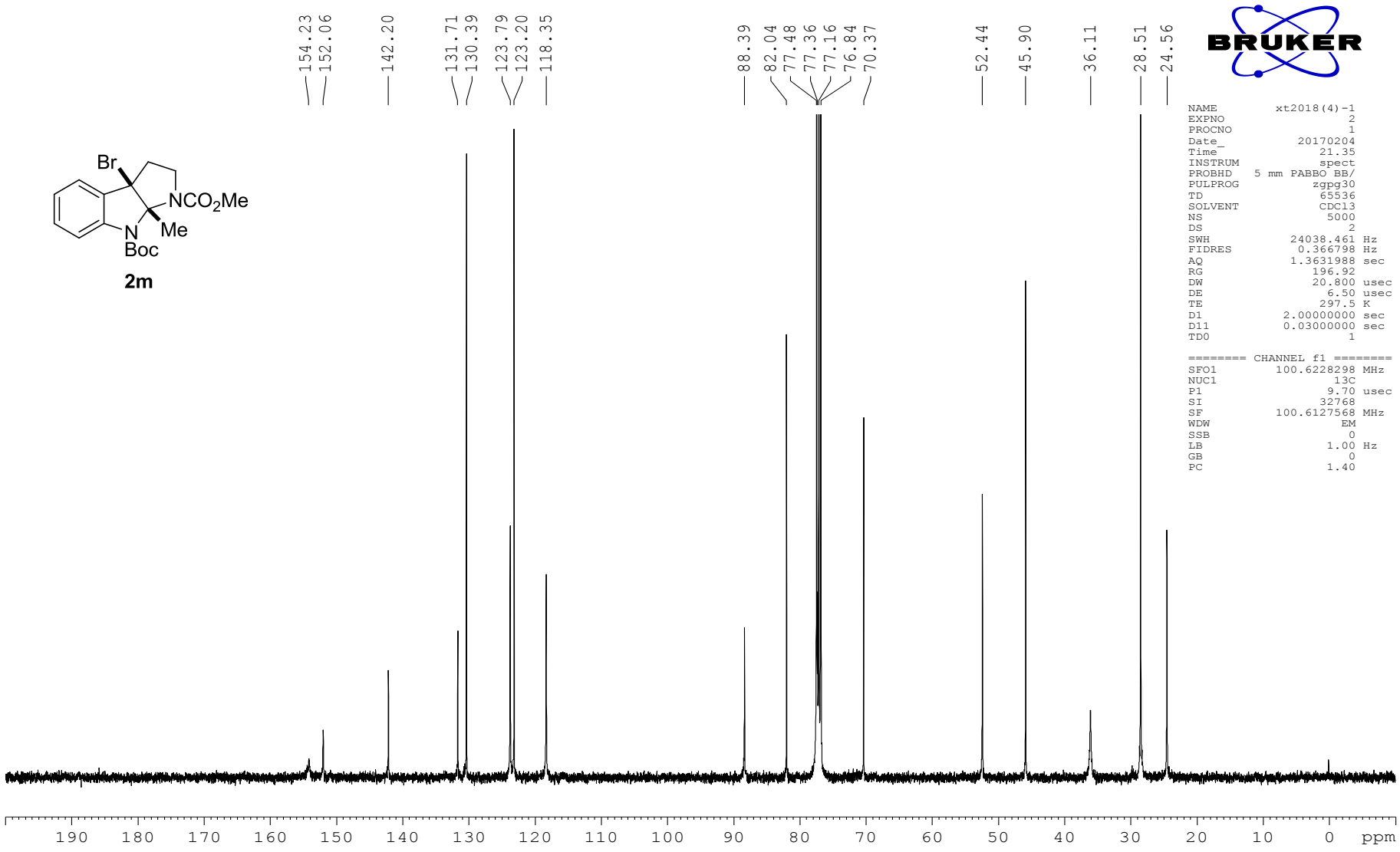

7.5681
7.5459
7.4996
7.4800
7.2600
7.1297
7.1099
6.9068
6.8849
6.7168
6.0489
4.0045
3.9973
3.9799
3.7643
2.6404
2.6153
2.5649
2.5556
2.5295
2.4875
2.2917
BRUKER
MeO
Br
NAc
N
H
Ts
2k
NAME xt1090-1
EXPNO 1
PROCNO 1
Date_ 20161109
Time 22.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 34.77
DW 62.400 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.14
2.11
2.49
1.26
1.24
1.00
0.97
3.76
1.18
1.83
3.27

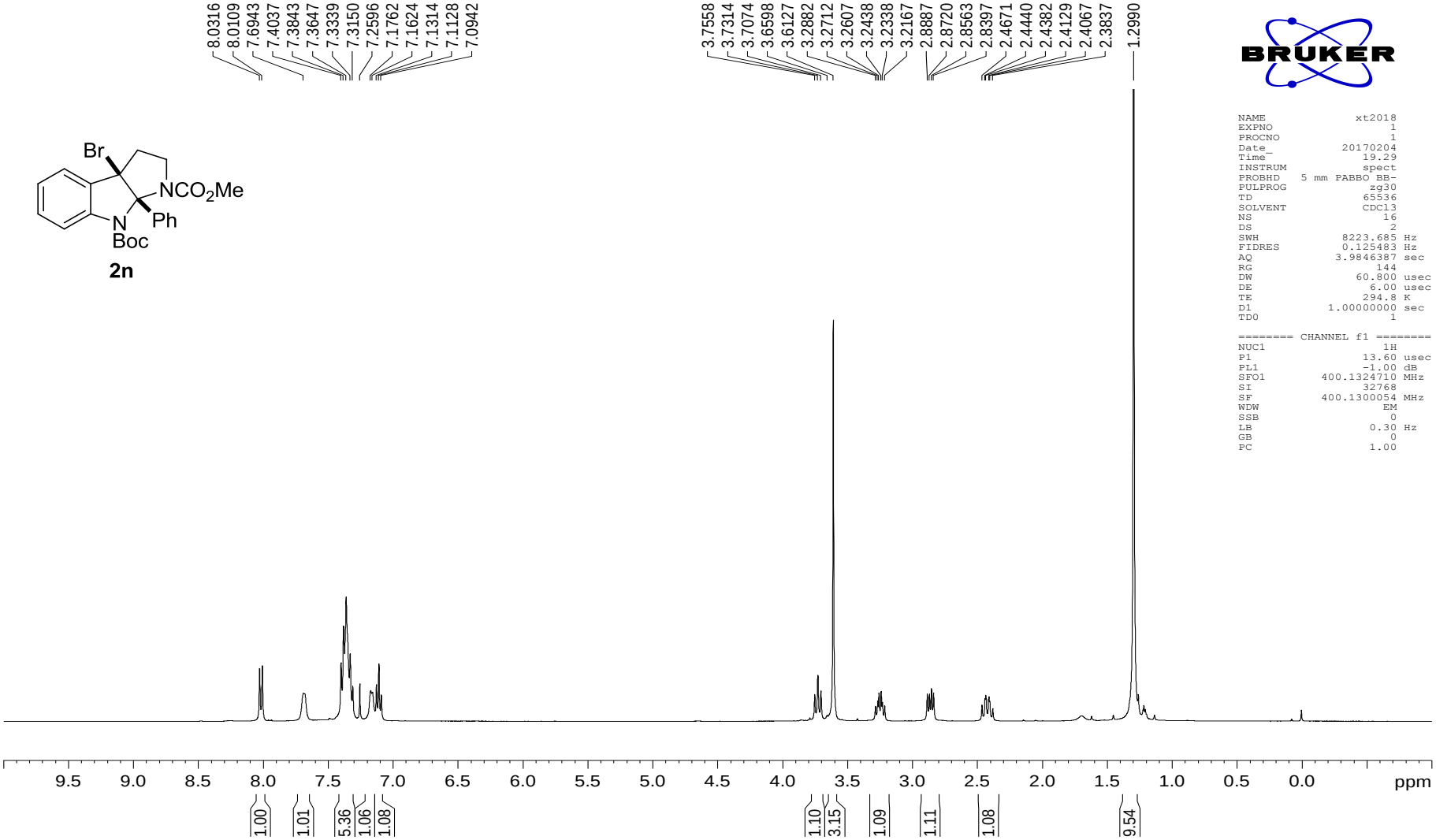

170.69
158.90
144.86
135.91
134.43
133.89
129.54
128.29
120.47
117.19
108.47
87.74
77.48
77.36
77.16
76.84
61.90
55.75
44.87
41.30
22.95
21.59
BRUKER
NAME xt1090-1
EXPNO 2
PROCNO 1
Date_ 20161109
Time 22.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 133
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127679 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
Br
NAc
N
H
Ts
2k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

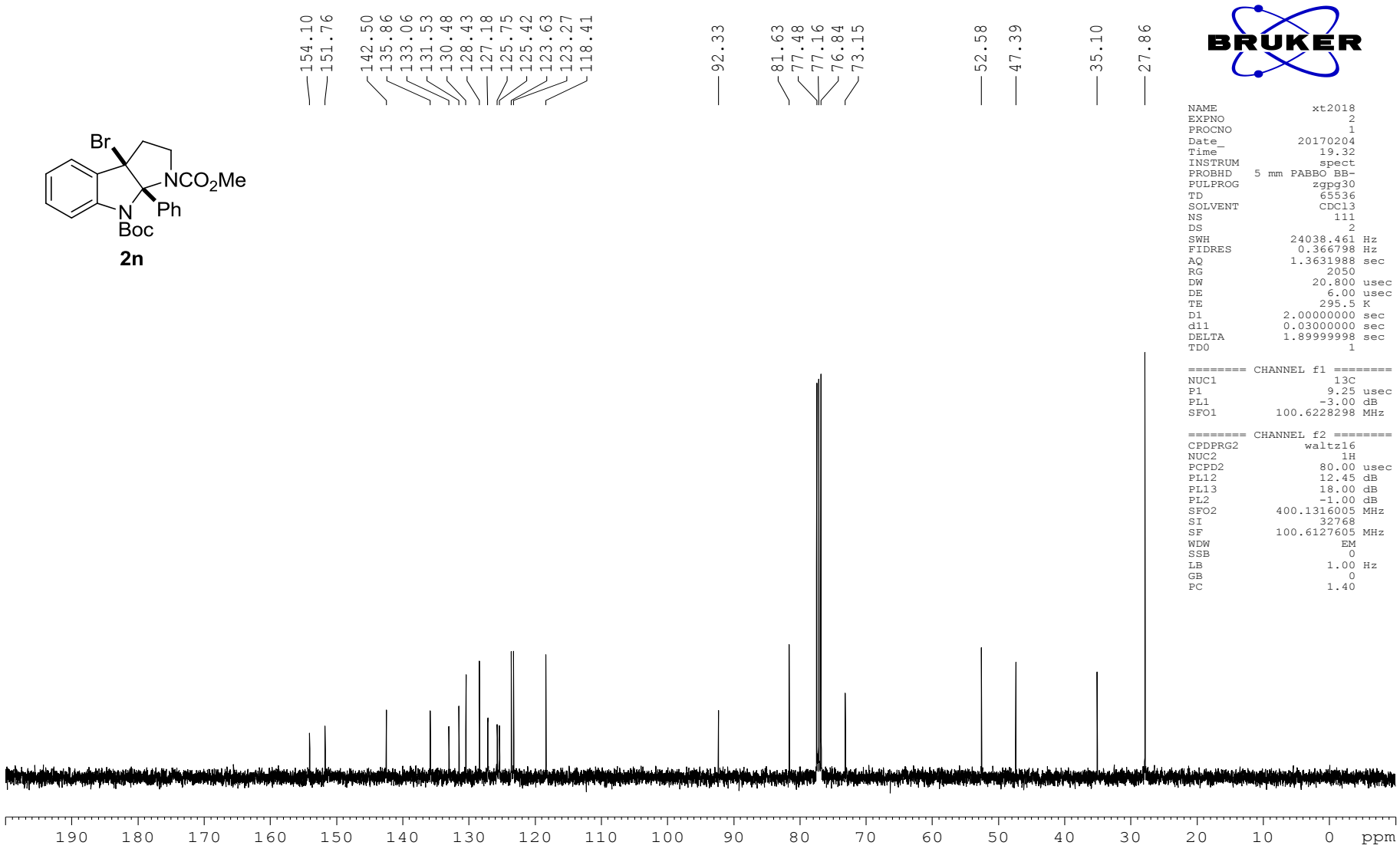

7.3359
7.2592
6.8601
6.8367
6.3200
4.0499
4.0297
4.0076
3.7852
3.7820
2.8055
2.7784
2.7297
2.6996
2.6686
2.6441
2.6216
2.3070
1.5511
MeO
Br
NAc
N
H
Boc
2l
BRUKER
NAME xt1092-1
EXPNO 1
PROCNO 1
Date_ 20161110
Time_ 19.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.89
2.59
1.00
1.02
3.87
3.66
3.06
11.75

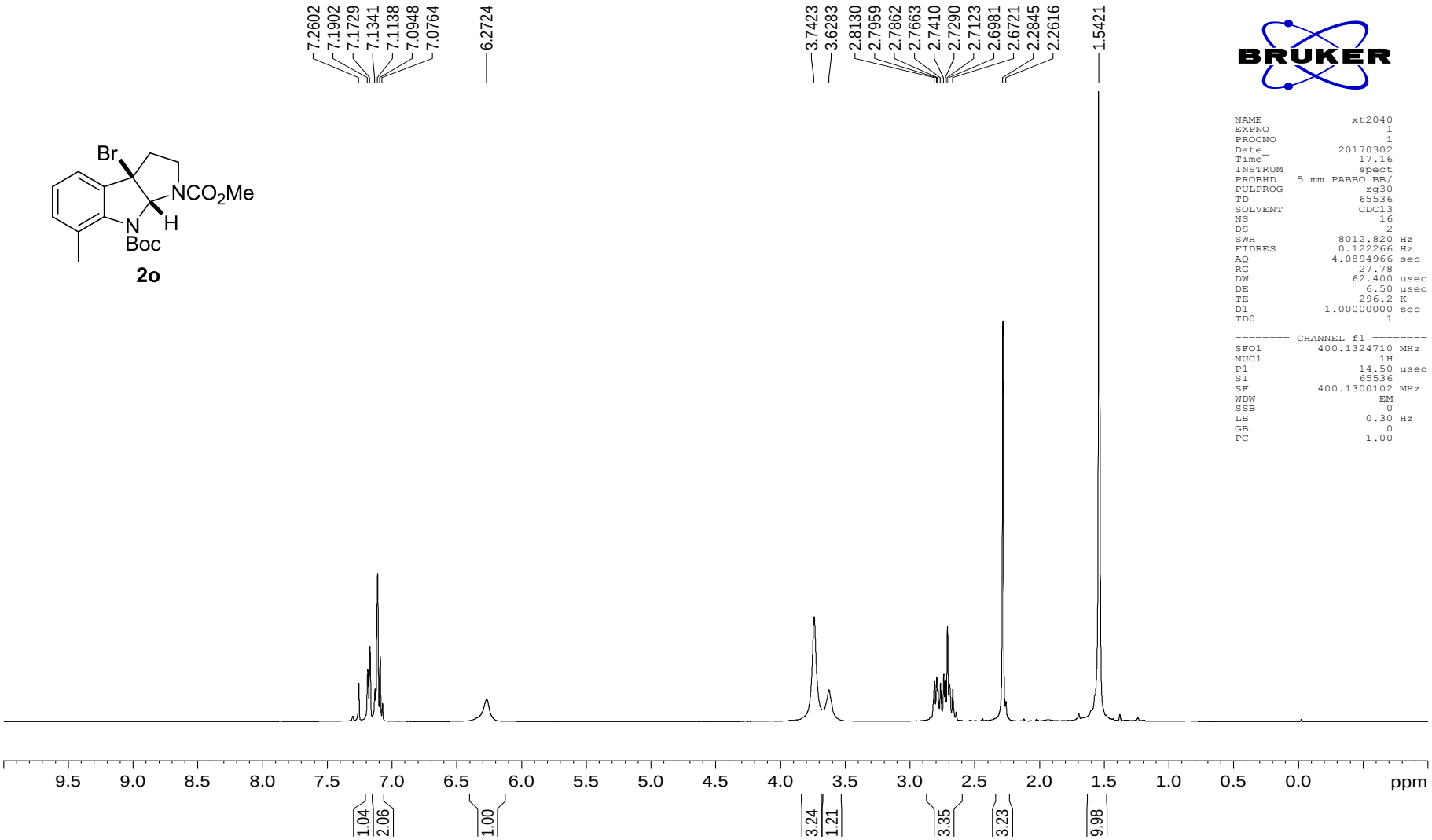

170.61
157.37
152.94
134.97
134.14
119.57
116.53
108.29
85.30
82.99
77.48
77.36
77.16
76.84
62.63
55.83
45.18
40.08
28.26
22.32
Br
MeO
NAc
N
H
Boc
2l
NAME xt1092-1
EXPNO 2
PROCNO 1
Date_ 20161110
Time 19.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 637
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 294.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127626 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

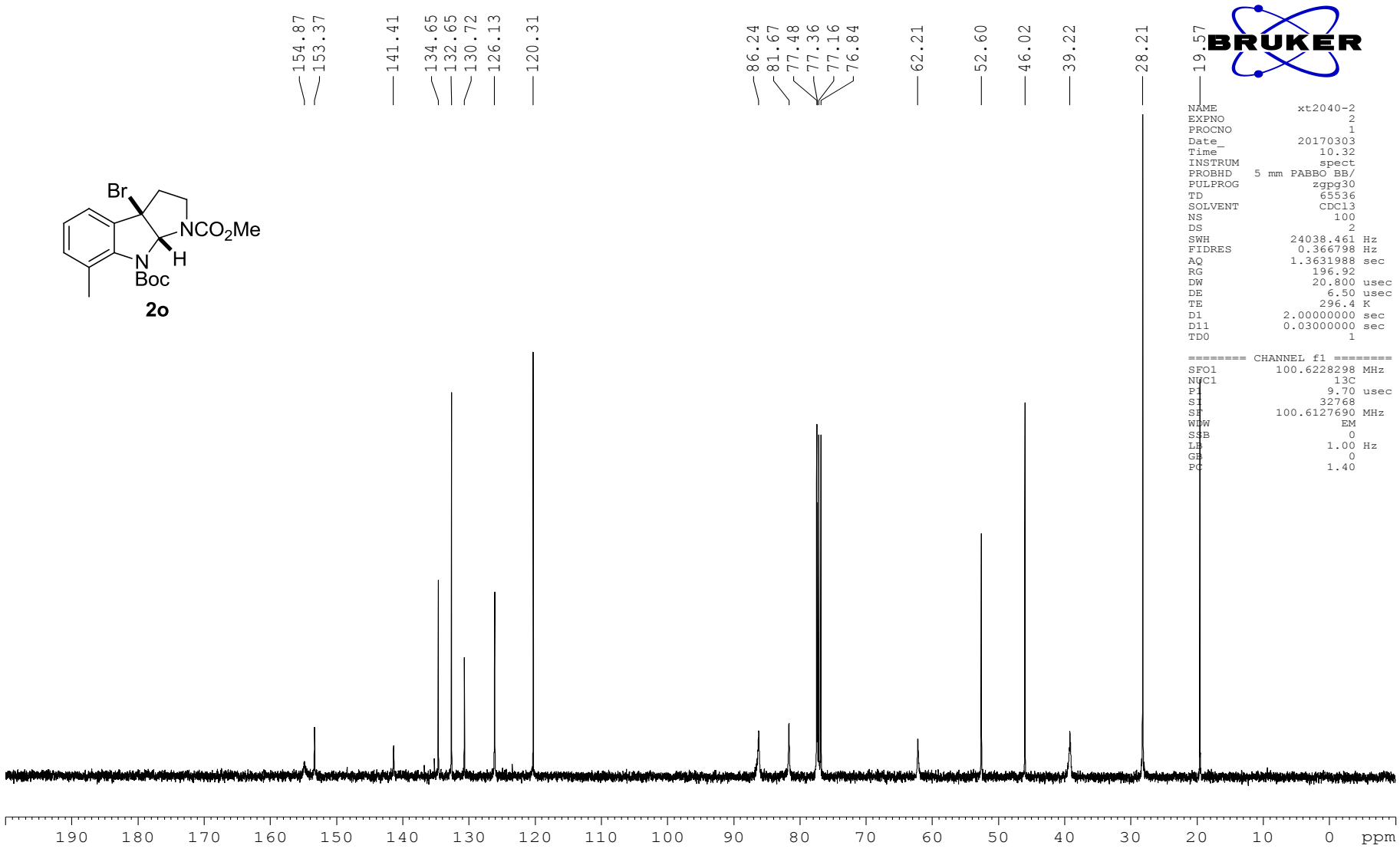

7.7401
7.3746
7.3557
7.2895
7.2864
7.2680
7.2600
7.2502
7.2471
7.0882
7.0695
7.0508
3.6691
3.5183
3.4947
3.4720
2.9814
2.9651
2.9544
2.9381
2.9304
2.9137
2.8971
2.8829
2.8669
2.7131
2.6934
2.6877
2.6844
2.6655
2.6621
2.6344
2.1366
1.6125
Br
NCO2Me
N
Me
Boc
2m
BRUKER
NAME xt2014
EXPNO 1
PROCNO 1
Date_ 20170204
Time_ 19.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 70.97
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.91
1.06
1.45
1.00
3.19
1.10
2.16
1.10
3.08
9.47

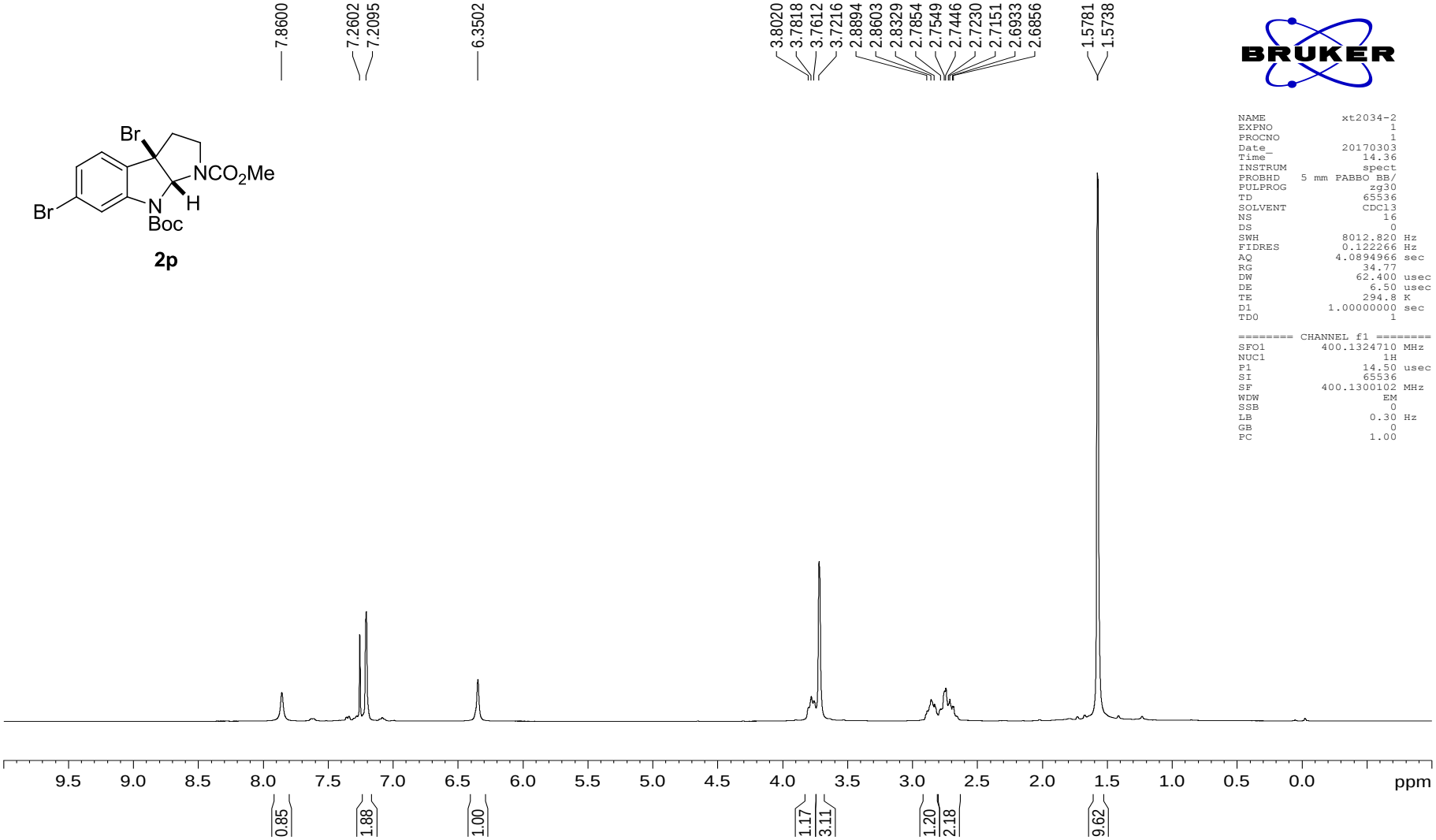

154.23
152.06
142.20
131.71
130.39
123.79
123.20
118.35
88.39
82.04
77.48
77.36
77.16
76.84
70.37
52.44
45.90
36.11
28.51
24.56
Br
NCO2Me
N
Me
Boc
2m
BRUKER
NAME xt2018(4)-1
EXPNO 2
PROCNO 1
Date_ 20170204
Time 21.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5000
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127568 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

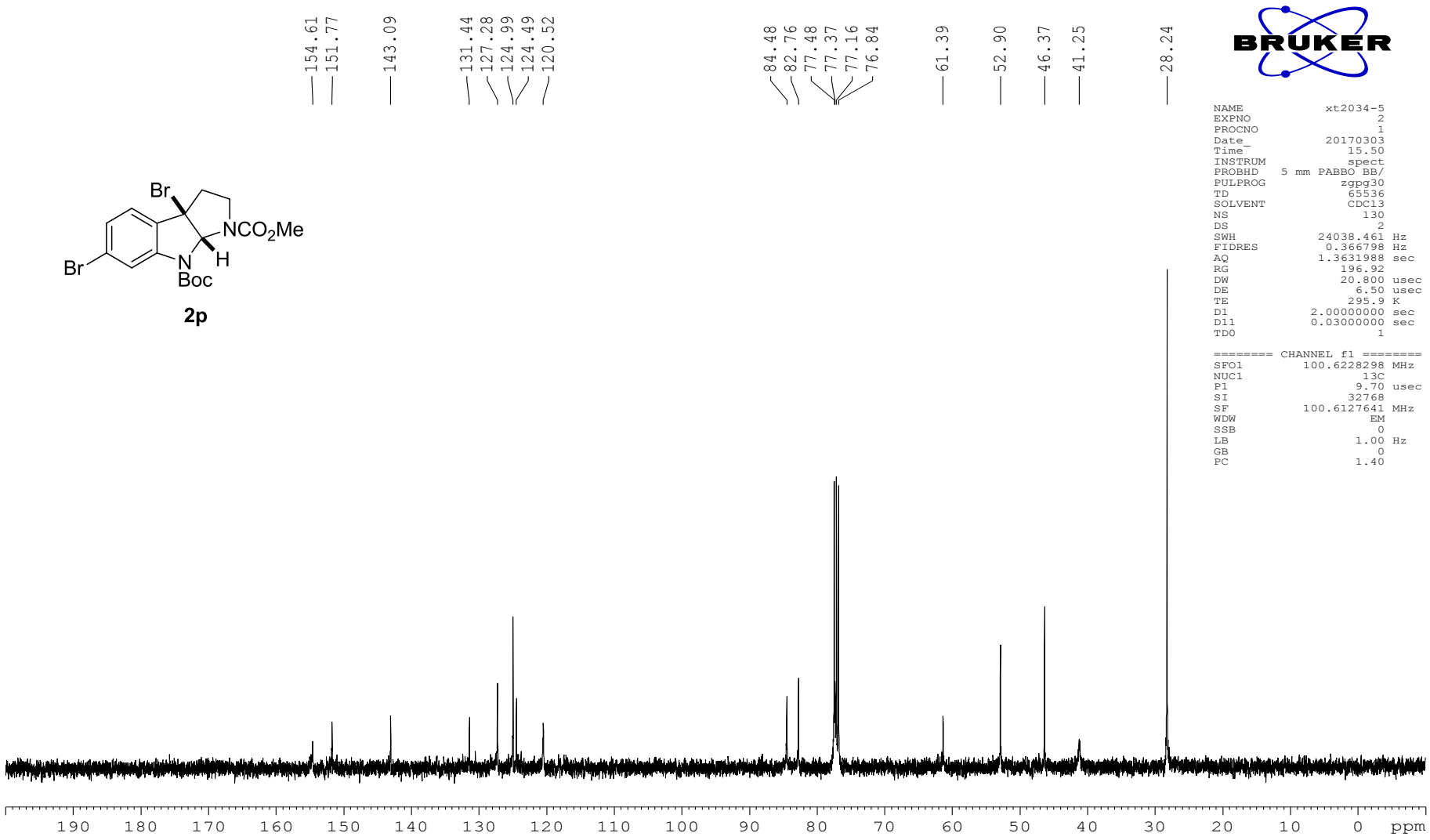

8.0316
8.0109
7.6943
7.4037
7.3843
7.3647
7.3339
7.3150
7.2596
7.1762
7.1624
7.1314
7.1128
7.0942
3.7558
3.7314
3.7074
3.6598
3.6127
3.2882
3.2712
3.2607
3.2438
3.2338
3.2167
2.8887
2.8720
2.8563
2.8397
2.4671
2.4440
2.4382
2.4129
2.4067
2.3837
1.2990
Br
NCO2Me
Ph
N
Boc
2n
BRUKER
NAME xt2018
EXPNO 1
PROCNO 1
Date_ 20170204
Time_ 19.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
5.36
1.06
1.08
1.10
3.15
1.09
1.11
1.08
9.54

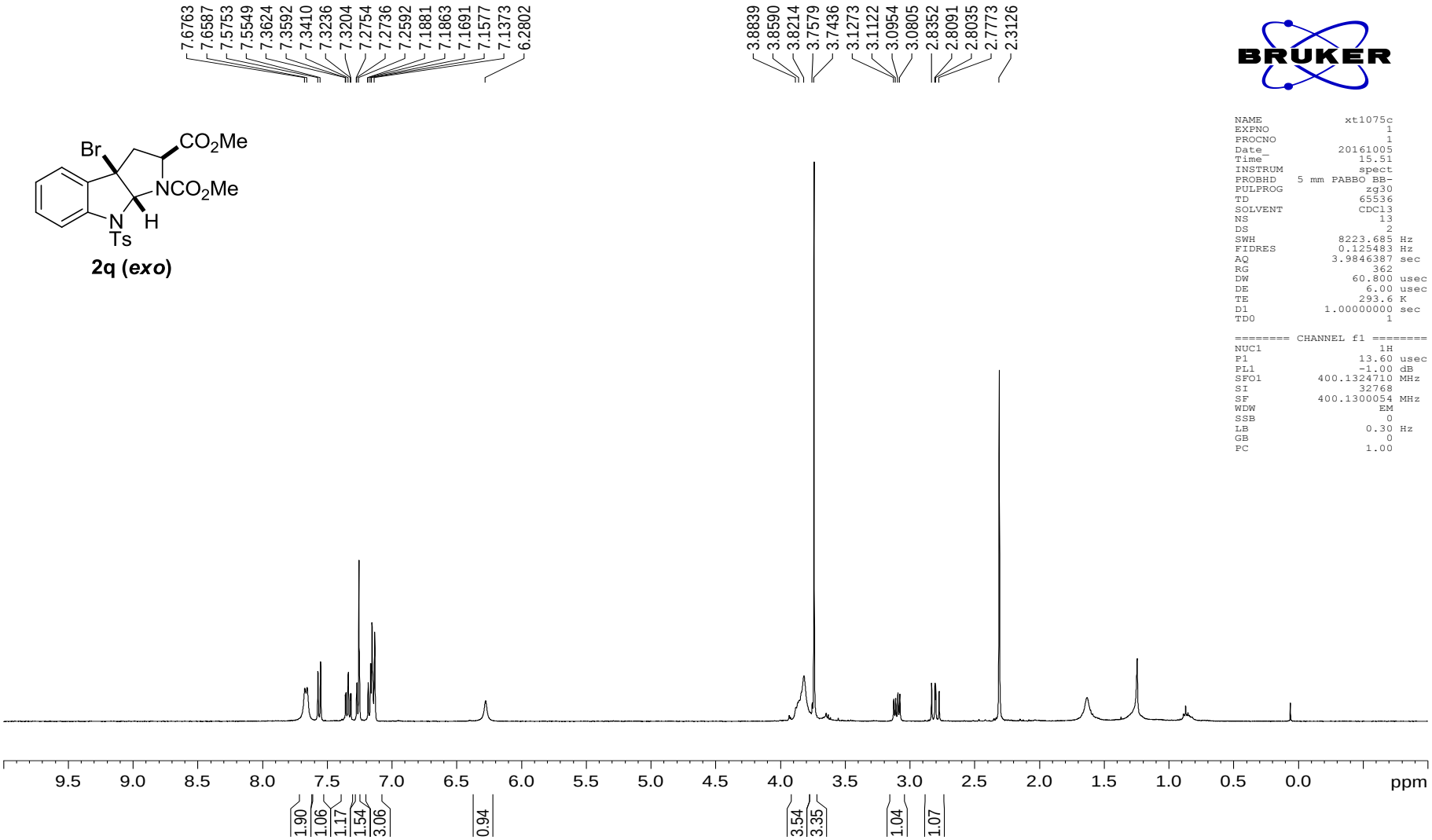

154.10
151.76
142.50
135.86
133.06
131.53
130.48
128.43
127.18
125.75
125.42
123.63
123.27
118.41
92.33
81.63
77.48
77.16
76.84
73.15
52.58
47.39
35.10
27.86
Br
NCO2Me
N
Ph
Boc
2n
BRUKER
NAME xt2018
EXPNO 2
PROCNO 1
Date_ 20170204
Time 19.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 111
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127605 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

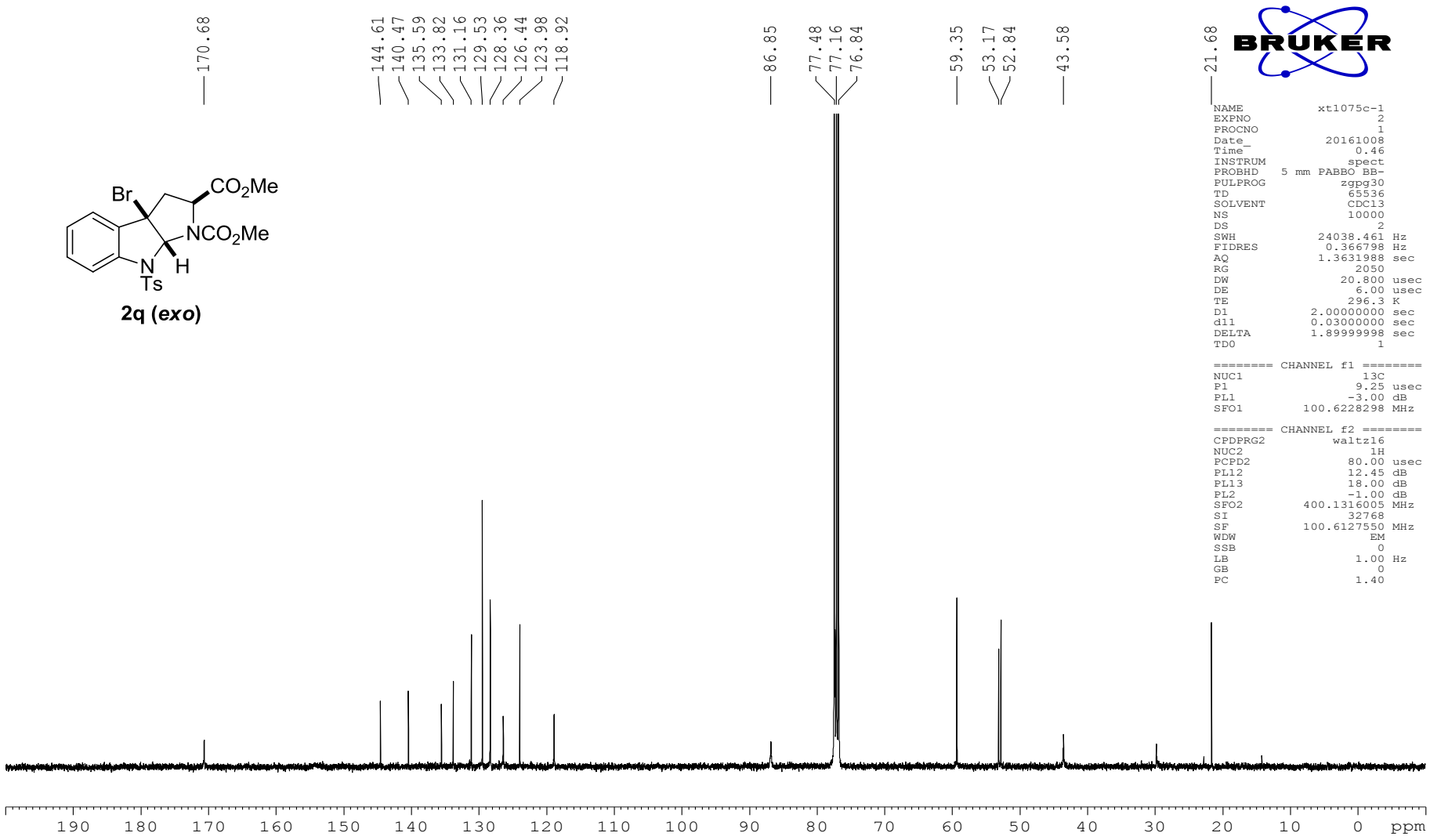

7.2602
7.1902
7.1729
7.1341
7.1138
7.0948
7.0764
6.2724
3.7423
3.6283
2.8130
2.7959
2.7862
2.7663
2.7410
2.7290
2.7123
2.6981
2.6721
2.2845
2.2616
1.5421
Br
NCO2Me
N
H
Boc
2o
BRUKER
NAME xt2040
EXPNO 1
PROCNO 1
Date_ 20170302
Time_ 17.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 27.78
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.04
2.06
1.00
3.24
1.21
3.35
3.23
9.98

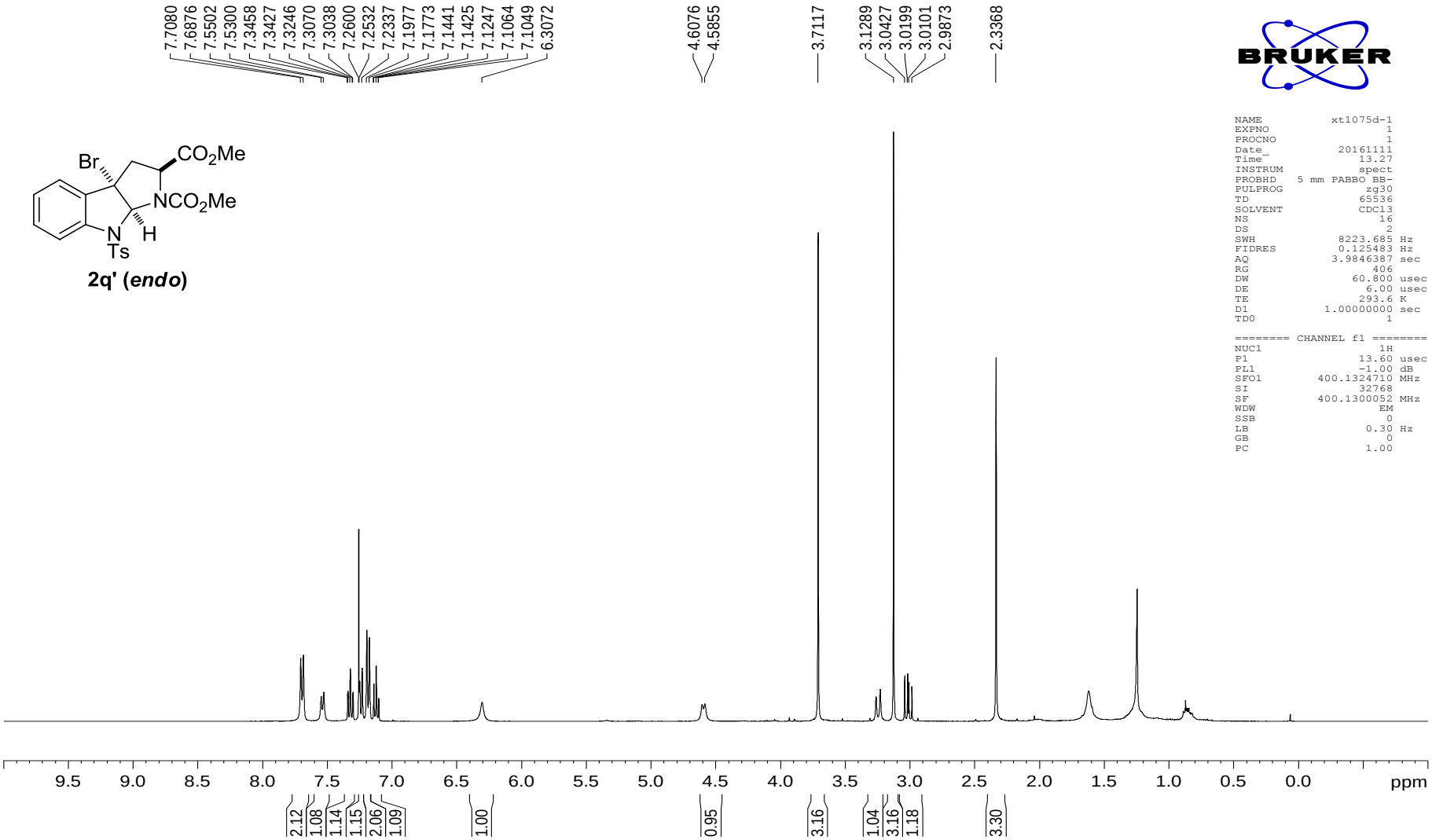

154.87
153.37
141.41
134.65
132.65
130.72
126.13
120.31
86.24
81.67
77.48
77.36
77.16
76.84
62.21
52.60
46.02
39.22
28.21
19.57
BRUKER
NAME xt2040-2
EXPNO 2
PROCNO 1
Date_ 20170303
Time 10.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Br
NCO2Me
N
H
Boc
2o
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

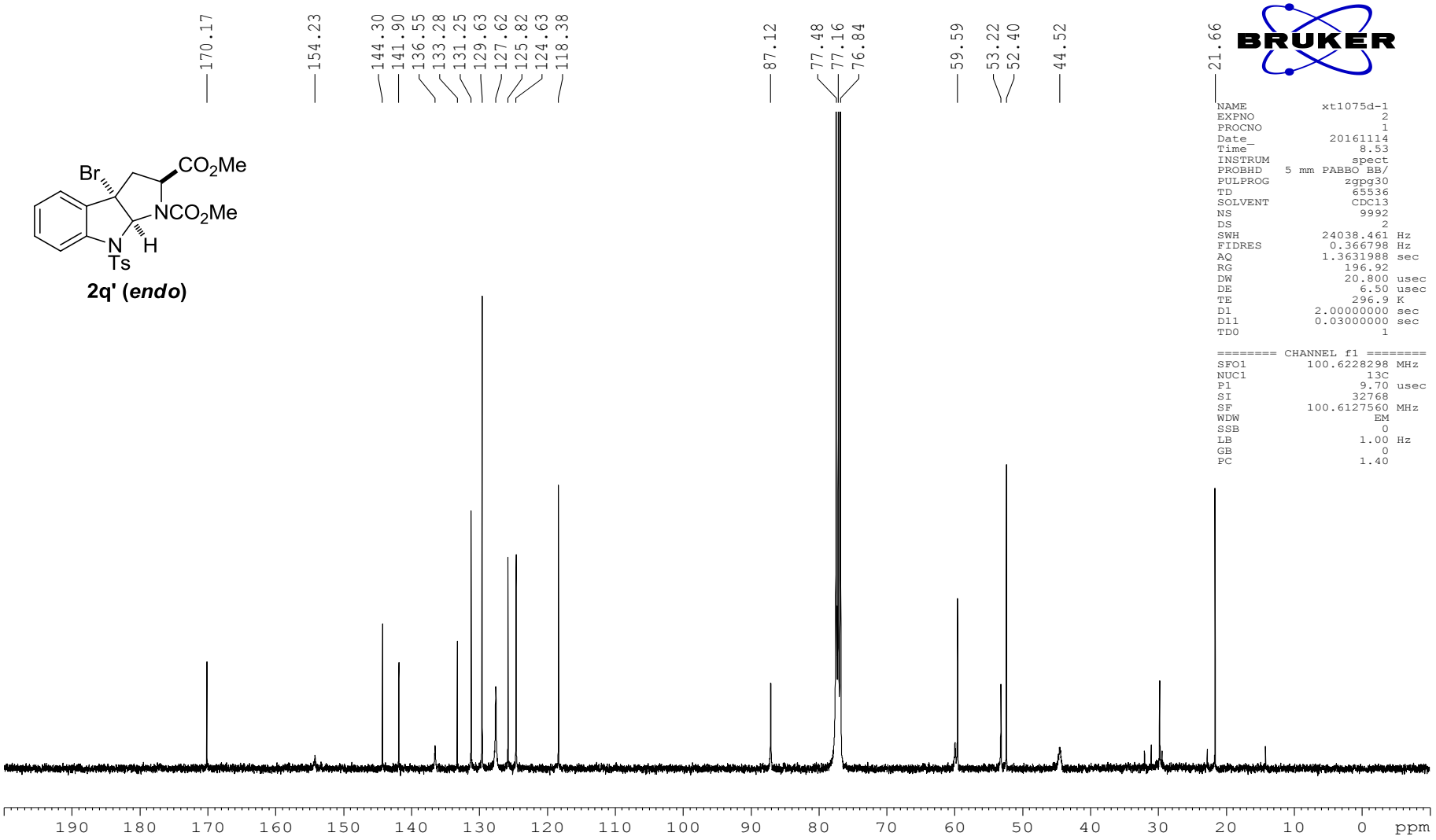

7.8600
7.2602
7.2095
6.3502
3.8020
3.7818
3.7612
3.7216
2.8894
2.8603
2.8329
2.7854
2.7549
2.7446
2.7230
2.7151
2.6933
2.6856
1.5781
1.5738
Br
NCO2Me
Br
N
H
Boc
2p
NAME xt2034-2
EXPNO 1
PROCNO 1
Date_ 20170303
Time_ 14.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 34.77
DW 62.400 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.85
1.88
1.00
1.17
3.11
1.20
2.18
9.62

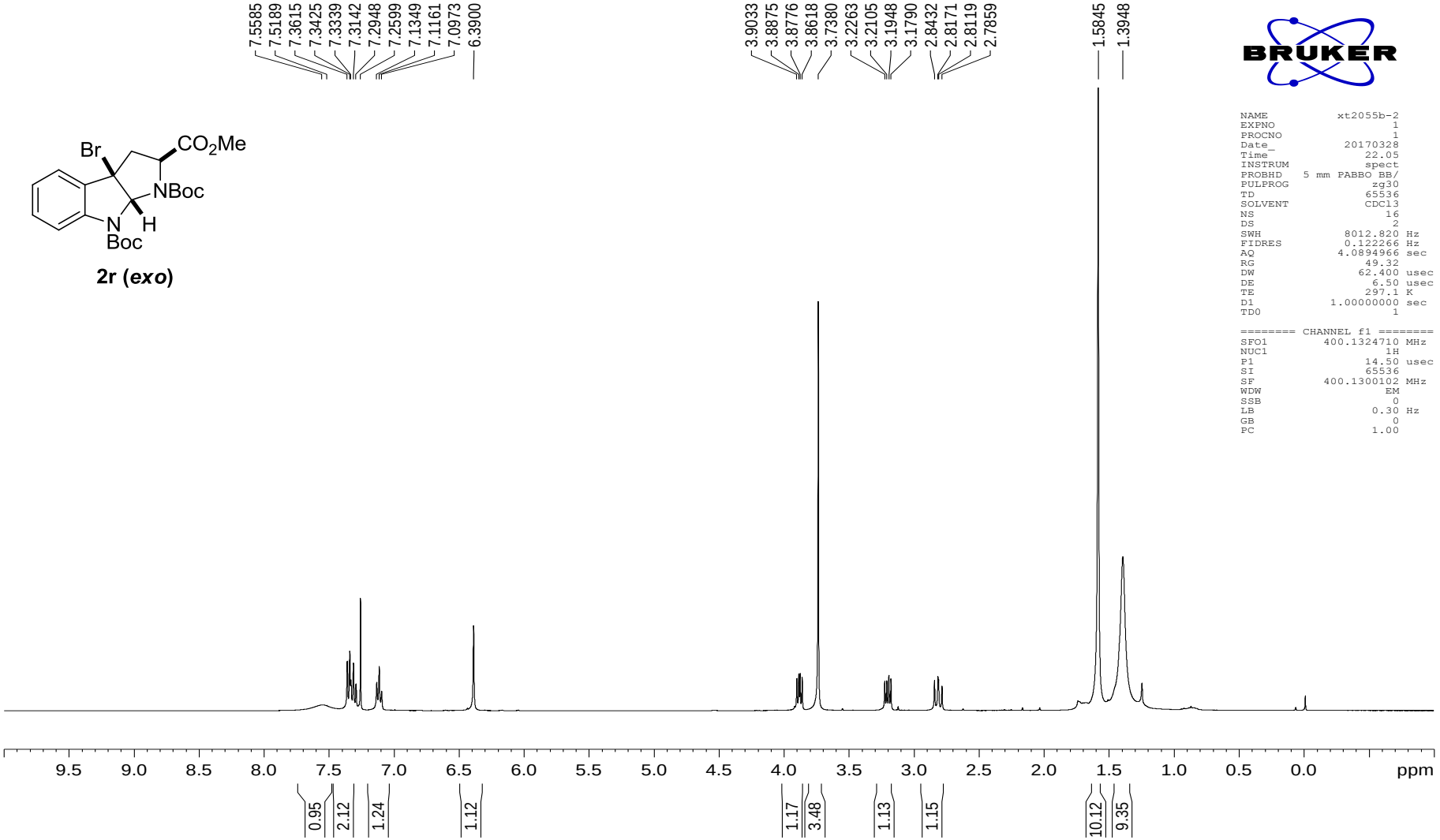

154.61
151.77
143.09
131.44
127.28
124.99
124.49
120.52
84.48
82.76
77.48
77.37
77.16
76.84
61.39
52.90
46.37
41.25
28.24
BRUKER
NAME xt2034-5
EXPNO 2
PROCNO 1
Date_ 20170303
Time 15.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 130
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127641 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Br
NCO2Me
Br
N
H
Boc
2p
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

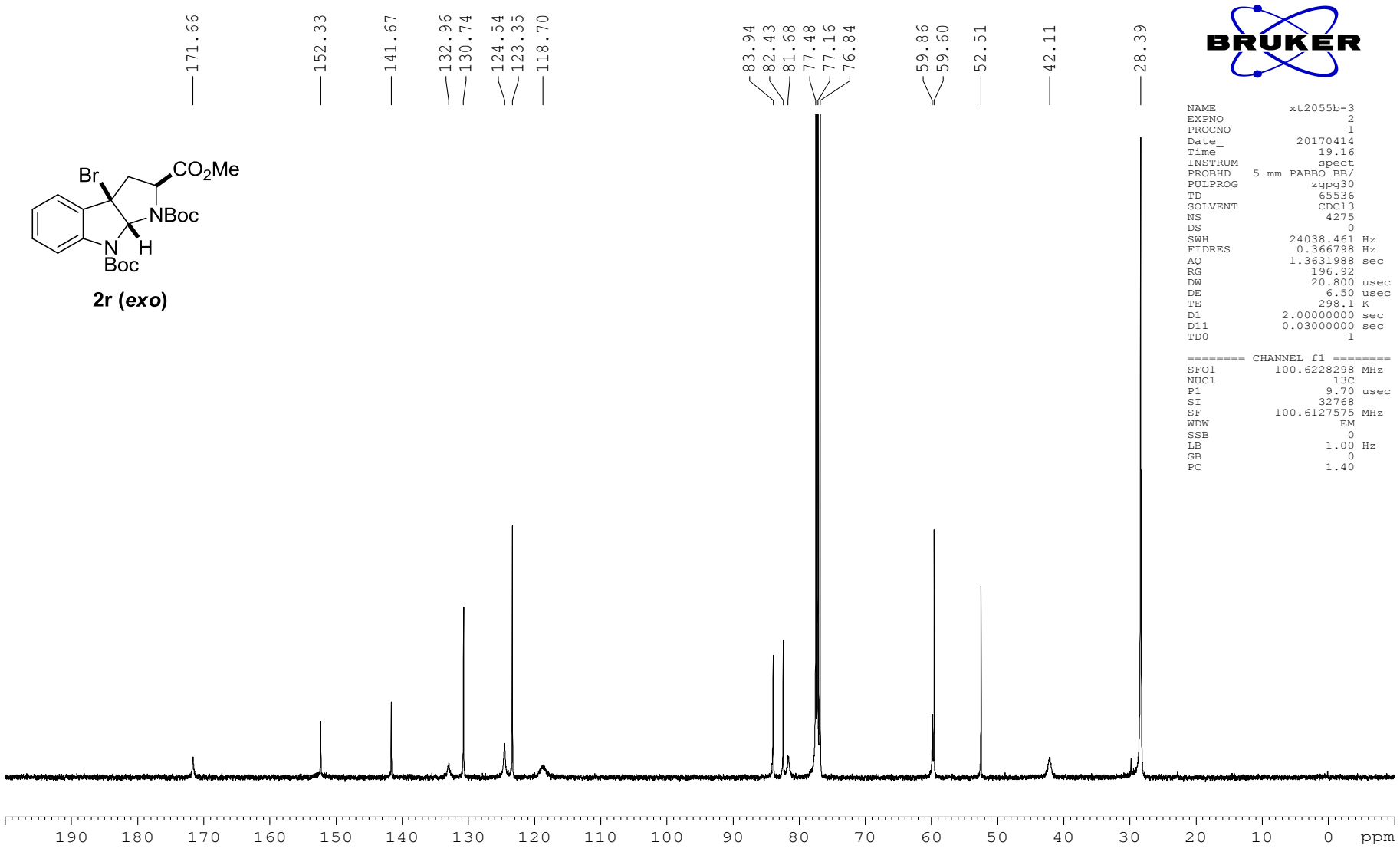

7.6763
7.6587
7.5753
7.5549
7.3624
7.3592
7.3410
7.3236
7.3204
7.2754
7.2736
7.2592
7.1881
7.1863
7.1691
7.1577
7.1373
6.2802
3.8839
3.8590
3.8214
3.7579
3.7436
3.1273
3.1122
3.0954
3.0805
2.8352
2.8091
2.8035
2.7773
2.3126
Br
CO2Me
NCO2Me
N
H
Ts
2q (exo)
NAME xt1075c
EXPNO 1
PROCNO 1
Date_ 20161005
Time_ 15.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 362
DW 60.800 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.90
1.06
1.17
1.54
3.06
0.94
3.54
3.35
1.04
1.07

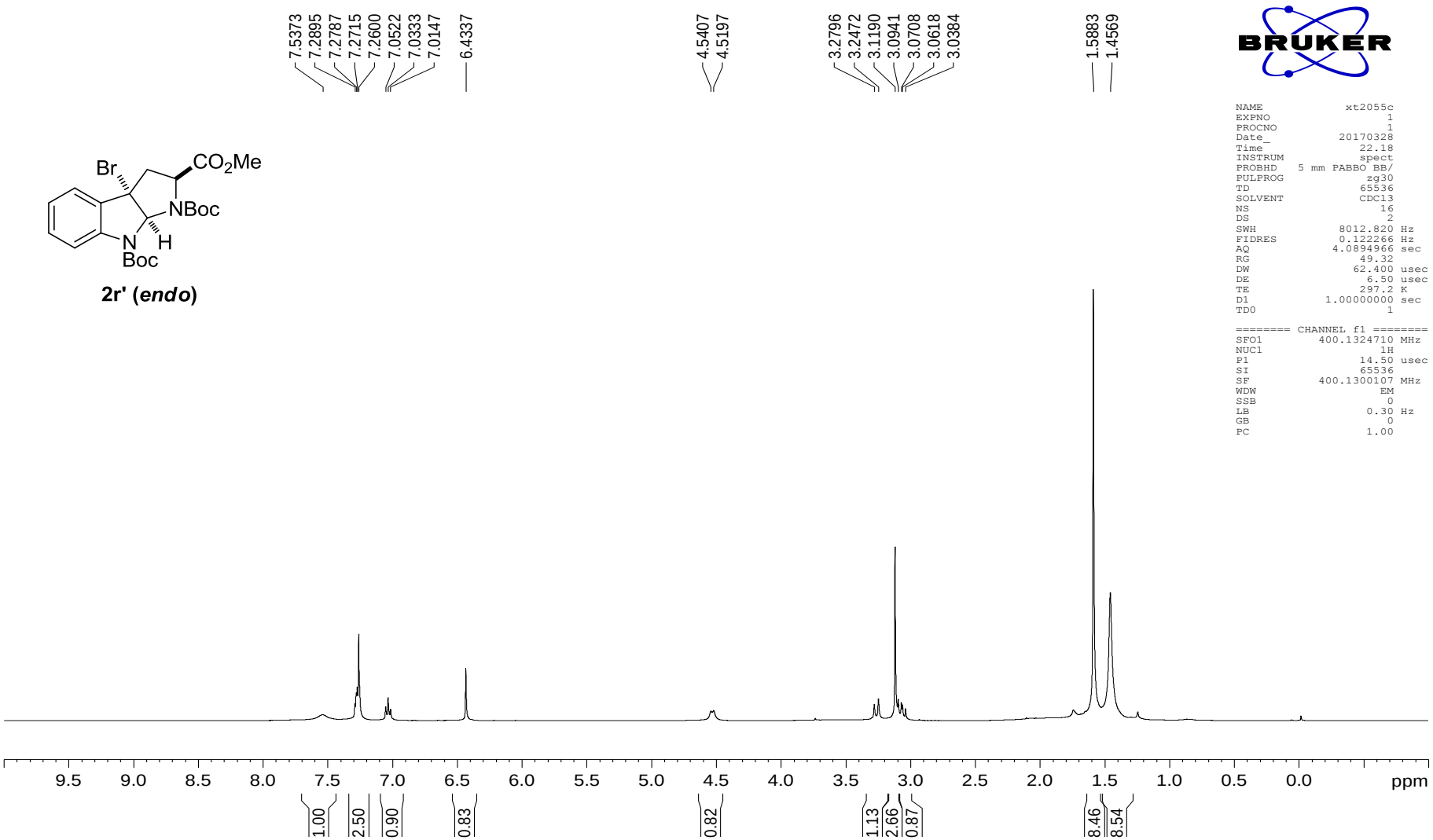

170.68
144.61
140.47
135.59
133.82
131.16
129.53
128.36
126.44
123.98
118.92
86.85
77.48
77.16
76.84
59.35
53.17
52.84
43.58
21.68
Br
CO2Me
NCO2Me
N
H
Ts
2q (exo)
BRUKER
NAME xt1075c-1
EXPNO 2
PROCNO 1
Date_ 20161008
Time 0.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10000
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

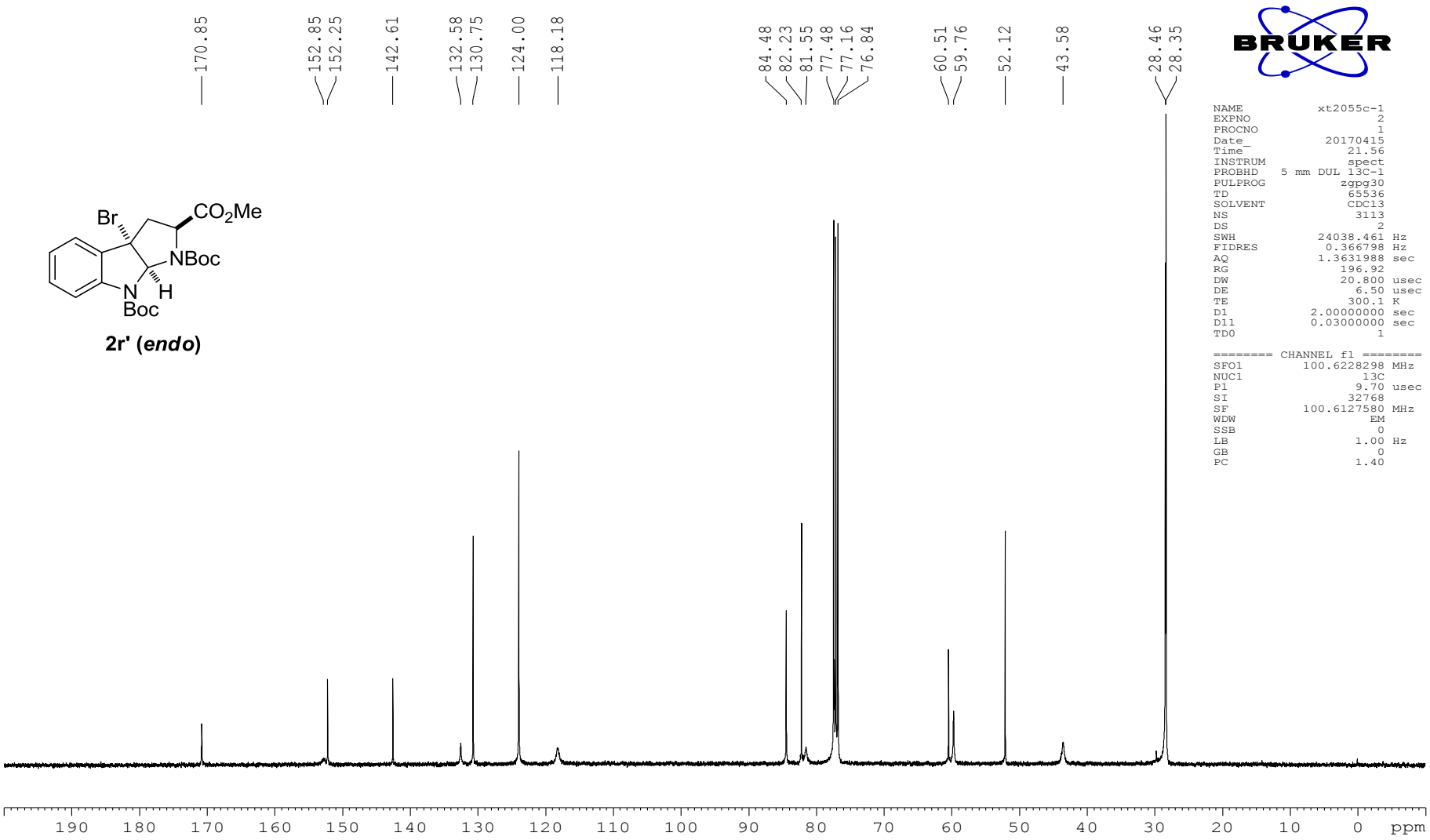

7.7080
7.6876
7.5502
7.5300
7.3458
7.3427
7.3246
7.3070
7.3038
7.2600
7.2532
7.2337
7.1977
7.1773
7.1441
7.1425
7.1247
7.1064
7.1049
6.3072
4.6076
4.5855
3.7117
3.1289
3.0427
3.0199
3.0101
2.9873
2.3368
Br
CO2Me
NCO2Me
N
H
Ts
2q' (endo)
NAME xt1075d-1
EXPNO 1
PROCNO 1
Date_ 20161111
Time_ 13.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 406
DW 60.800 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.12
1.08
1.14
1.15
2.06
1.09
1.00
0.95
3.16
1.04
3.16
1.18
3.30

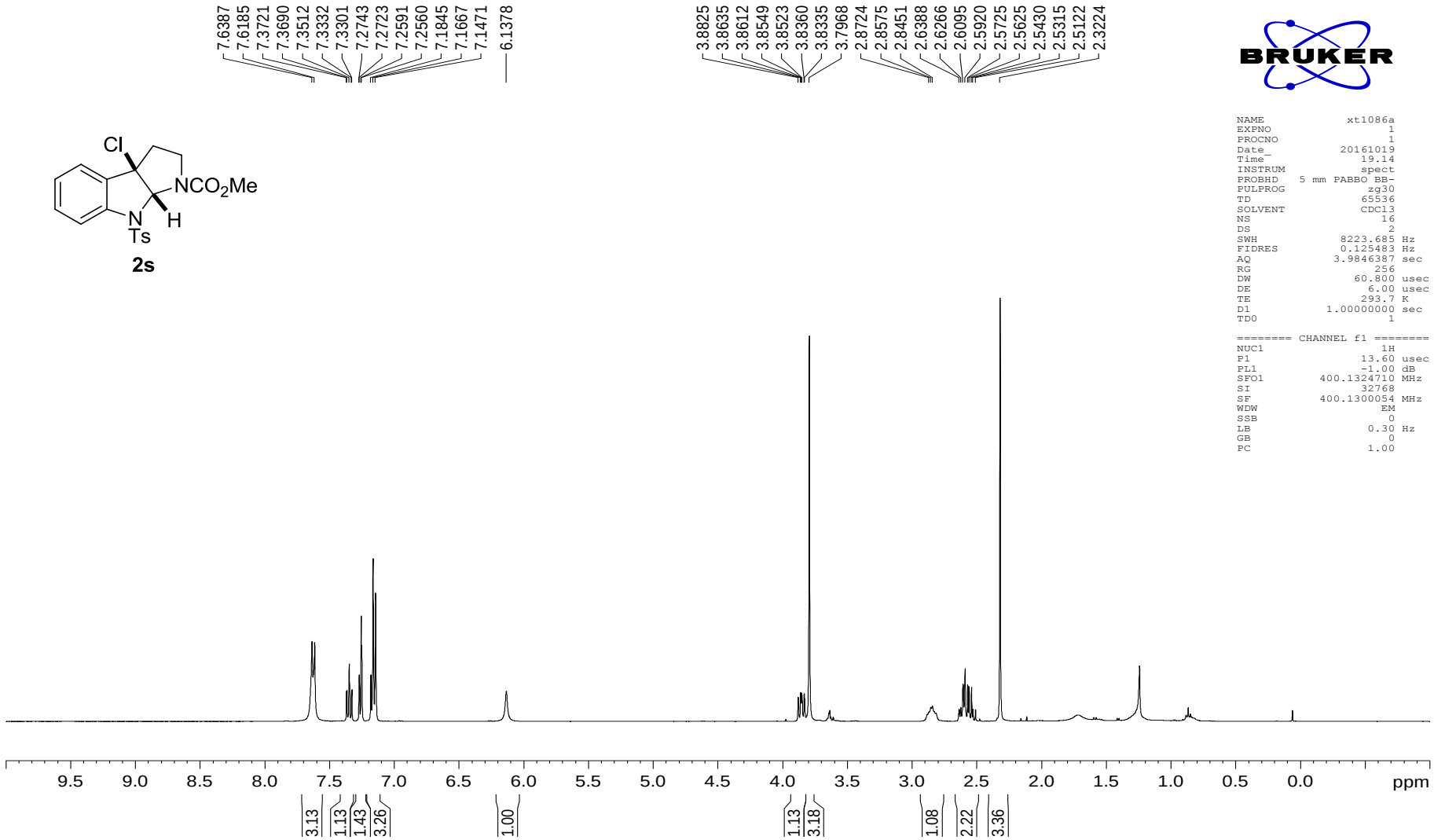

170.17
154.23
144.30
141.90
136.55
133.28
131.25
129.63
127.62
125.82
124.63
118.38
87.12
77.48
77.16
76.84
59.59
53.22
52.40
44.52
21.66
Br
CO2Me
NCO2Me
N
H
Ts
2q' (endo)
BRUKER
NAME xt1075d-1
EXPNO 2
PROCNO 1
Date_ 20161114
Time 8.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9992
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

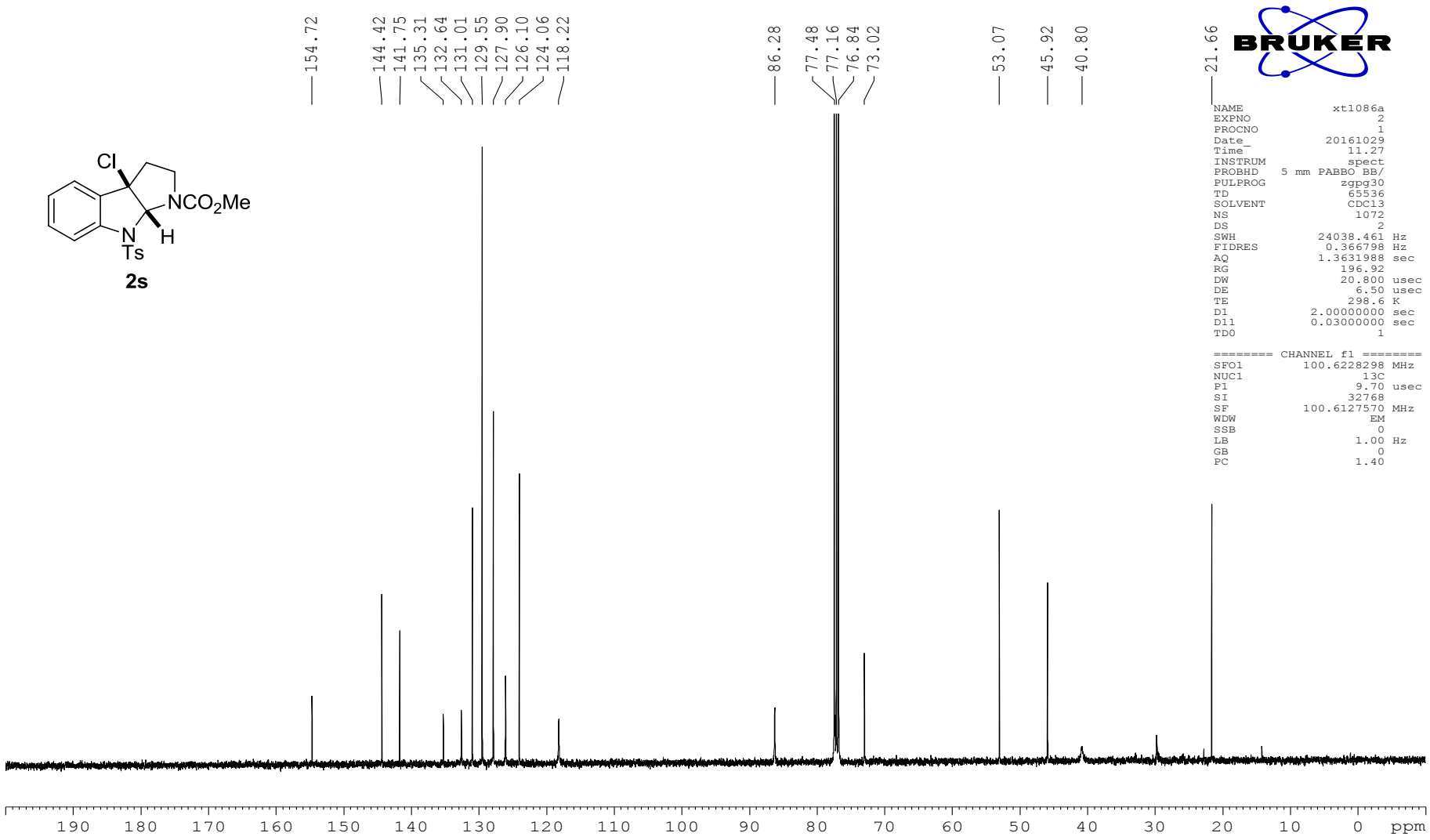

7.5585
7.5189
7.3615
7.3425
7.3339
7.3142
7.2948
7.2599
7.1349
7.1161
7.0973
6.3900
3.9033
3.8875
3.8776
3.8618
3.7380
3.2263
3.2105
3.1948
3.1790
2.8432
2.8171
2.8119
2.7859
1.5845
1.3948
Br
CO2Me
NBoc
N
H
Boc
2r (exo)
BRUKER
NAME xt2055b-2
EXPNO 1
PROCNO 1
Date_ 20170328
Time 22.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 49.32
DW 62.400 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.95
2.12
1.24
1.12
1.17
3.48
1.13
1.15
10.12
9.35

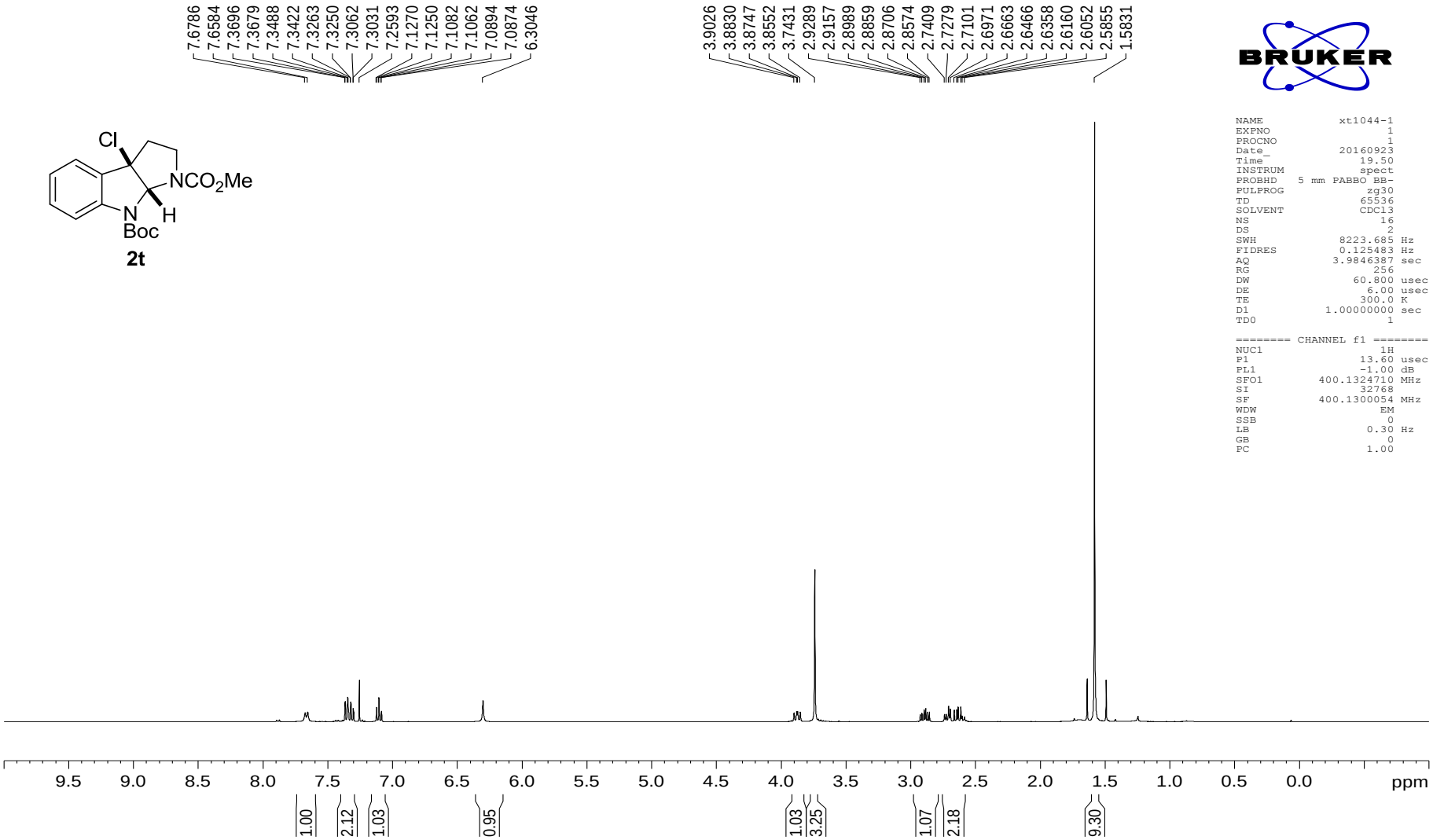

171.66
152.33
141.67
132.96
130.74
124.54
123.35
118.70
83.94
82.43
81.68
77.48
77.16
76.84
59.86
59.60
52.51
42.11
28.39
Br
CO2Me
NBoc
N
H
Boc
2r (exo)
BRUKER
NAME xt2055b-3
EXPNO 2
PROCNO 1
Date_ 20170414
Time 19.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4275
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

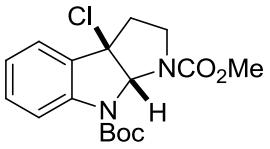

7.5373
7.2895
7.2787
7.2715
7.2600
7.0522
7.0333
7.0147
6.4337
4.5407
4.5197
3.2796
3.2472
3.1190
3.0941
3.0708
3.0618
3.0384
1.5883
1.4569
Br
CO2Me
NBoc
N H
Boc
2r' (endo)
NAME xt2055c
EXPNO 1
PROCNO 1
Date_ 20170328
Time_ 22.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 49.32
DW 62.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300107 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
2.50
0.90
0.83
0.82
1.13
2.66
0.87
8.46
8.54


170.85
152.85
152.25
142.61
132.58
130.75
124.00
118.18
84.48
82.23
81.55
77.48
77.16
76.84
60.51
59.76
52.12
43.58
28.46
28.35
Br
CO2Me
NBoc
N
H
Boc
2r' (endo)
NAME xt2055c-1
EXPNO 2
PROCNO 1
Date_ 20170415
Time 21.56
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3113
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127580 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.6387
7.6185
7.3721
7.3690
7.3512
7.3332
7.3301
7.2743
7.2723
7.2591
7.2560
7.1845
7.1667
7.1471
6.1378
3.8825
3.8635
3.8612
3.8549
3.8523
3.8360
3.8335
3.7968
2.8724
2.8575
2.8451
2.6388
2.6266
2.6095
2.5920
2.5725
2.5625
2.5430
2.5315
2.5122
2.3224
BRUKER
Cl
NCO2Me
N
H
Ts
2s
NAME xt1086a
EXPNO 1
PROCNO 1
Date_ 20161019
Time_ 19.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.00 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.13
1.13
1.43
3.26
1.00
1.13
3.18
1.08
2.22
3.36

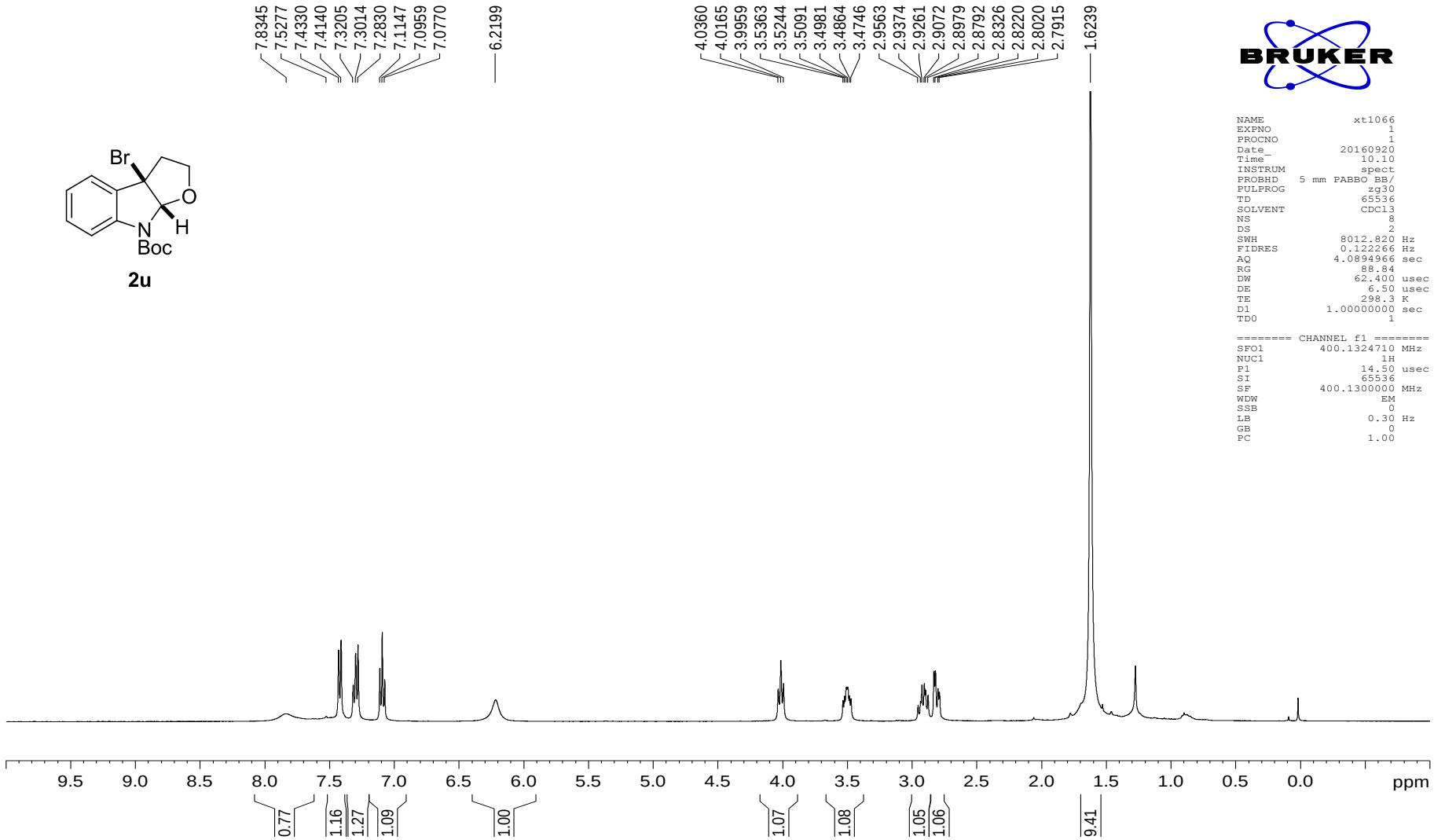

154.72
144.42
141.75
135.31
132.64
131.01
129.55
127.90
126.10
124.06
118.22
86.28
77.48
77.16
76.84
73.02
53.07
45.92
40.80
21.66
Cl
NCO2Me
N
H
Ts
2s
BRUKER
NAME xt1086a
EXPNO 2
PROCNO 1
Date_ 20161029
Time 11.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1072
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

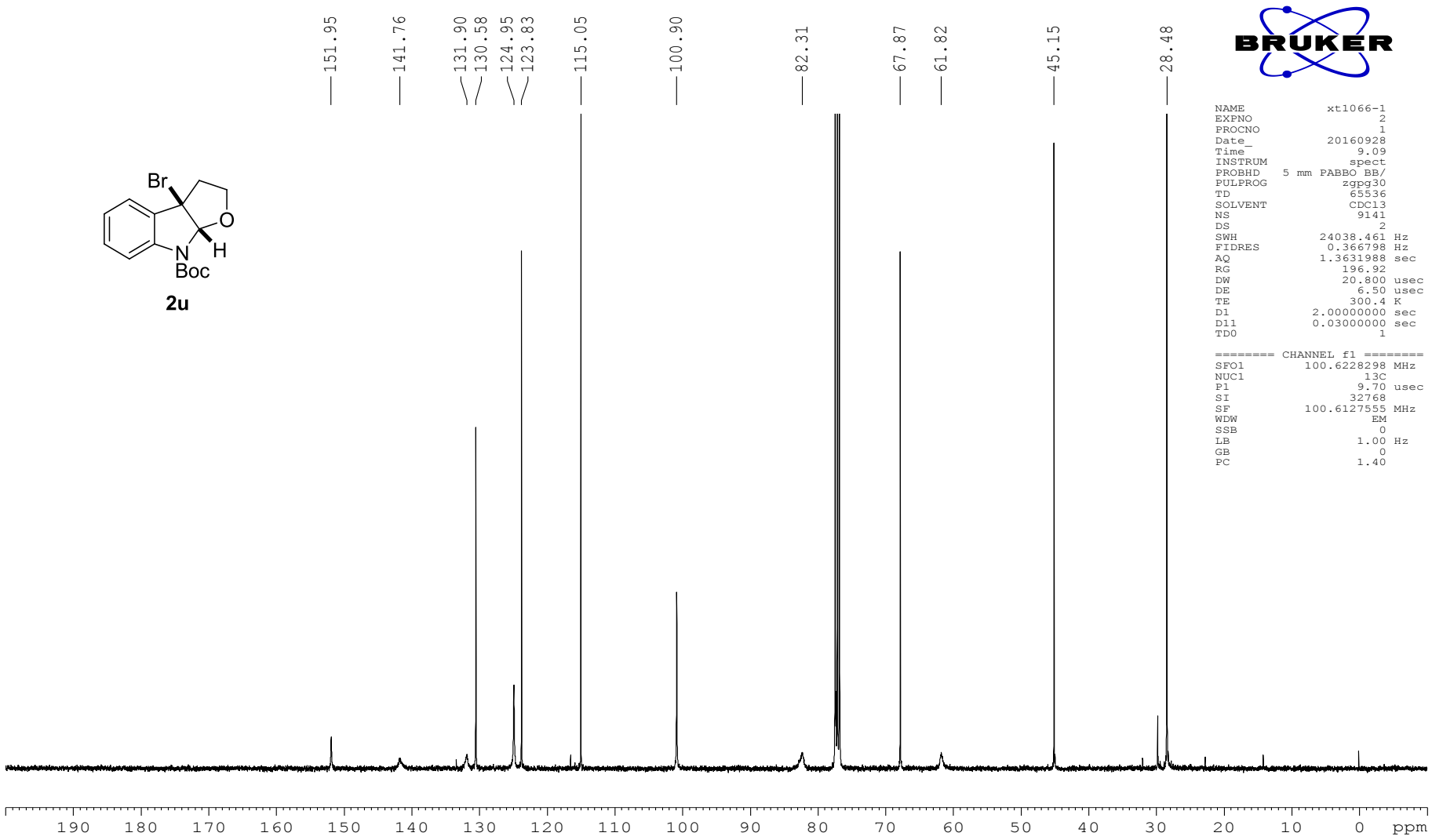

7.6786
7.6584
7.3696
7.3679
7.3488
7.3422
7.3263
7.3250
7.3062
7.3031
7.2593
7.1270
7.1250
7.1082
7.1062
7.0894
7.0874
6.3046
3.9026
3.8830
3.8747
3.8552
3.7431
2.9289
2.9157
2.8989
2.8859
2.8706
2.8574
2.7409
2.7279
2.7101
2.6971
2.6663
2.6466
2.6358
2.6160
2.6052
2.5855
1.5831
Cl
NCO2Me
N
H
Boc
2t
BRUKER
NAME xt1044-1
EXPNO 1
PROCNO 1
Date_ 20160923
Time 19.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.12
1.03
0.95
1.03
3.25
1.07
2.18
9.30

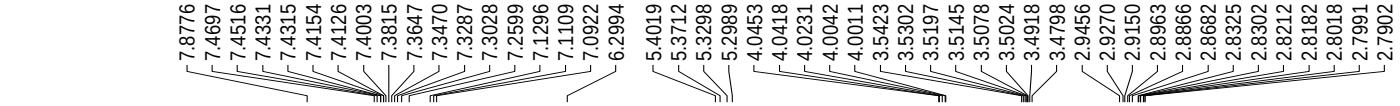

Cl
NCO2Me
N
H
Boc

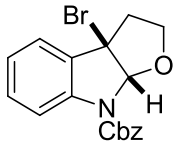
2t

155.03
152.26
142.53
131.33
130.79
124.24
123.66
117.30
83.48
82.28
77.48
77.16
76.84
72.89
52.89
46.31
40.41
28.40
BRUKER
NAME xt1044-2
EXPNO 2
PROCNO 1
Date_ 20160925
Time 22.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12000
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.8345
7.5277
7.4330
7.4140
7.3205
7.3014
7.2830
7.1147
7.0959
7.0770
6.2199
4.0360
4.0165
3.9959
3.5363
3.5244
3.5091
3.4981
3.4864
3.4746
2.9563
2.9374
2.9261
2.9072
2.8979
2.8792
2.8326
2.8220
2.8020
2.7915
1.6239
Br
O
N
H
Boc
2u
BRUKER
NAME xt1066
EXPNO 1
PROCNO 1
Date_ 20160920
Time_ 10.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.77
1.16
1.27
1.09
1.00
1.07
1.08
1.05
1.06
9.41

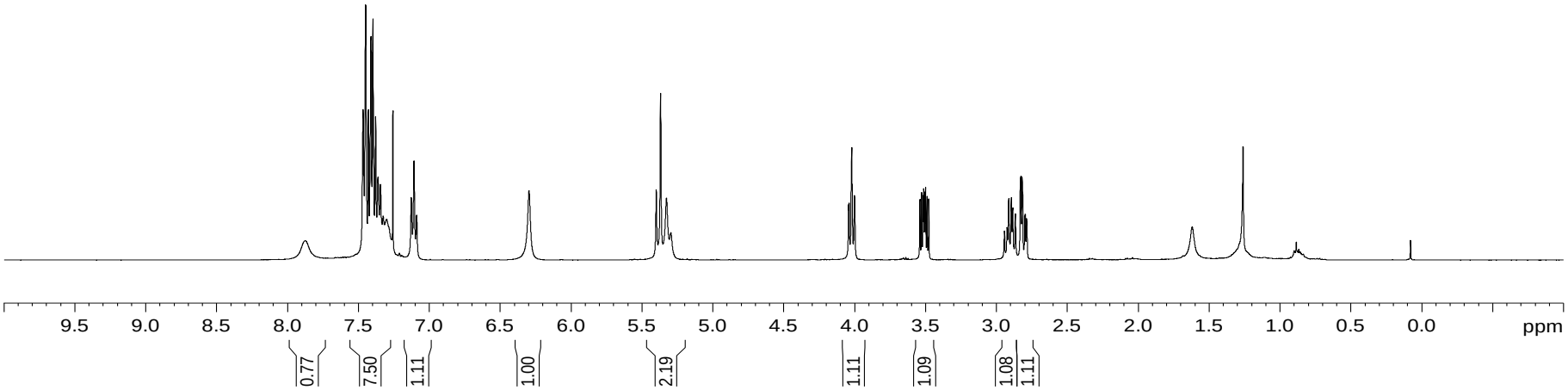

151.95
141.76
131.90
130.58
124.95
123.83
115.05
100.90
82.31
67.87
61.82
45.15
28.48
Br
O
N
H
Boc
2u
BRUKER
NAME xt1066-1
EXPNO 2
PROCNO 1
Date_ 20160928
Time 9.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9141
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

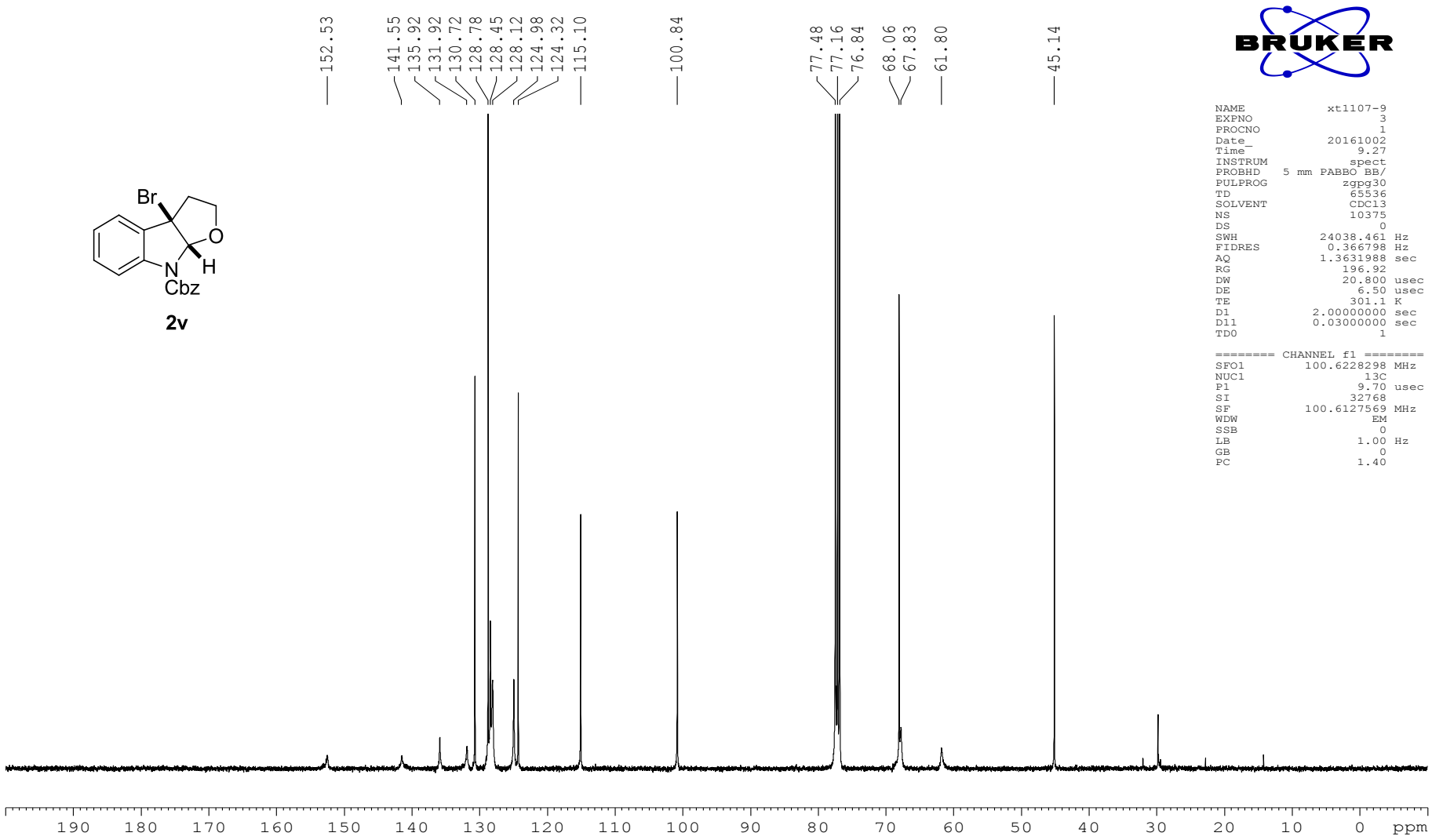


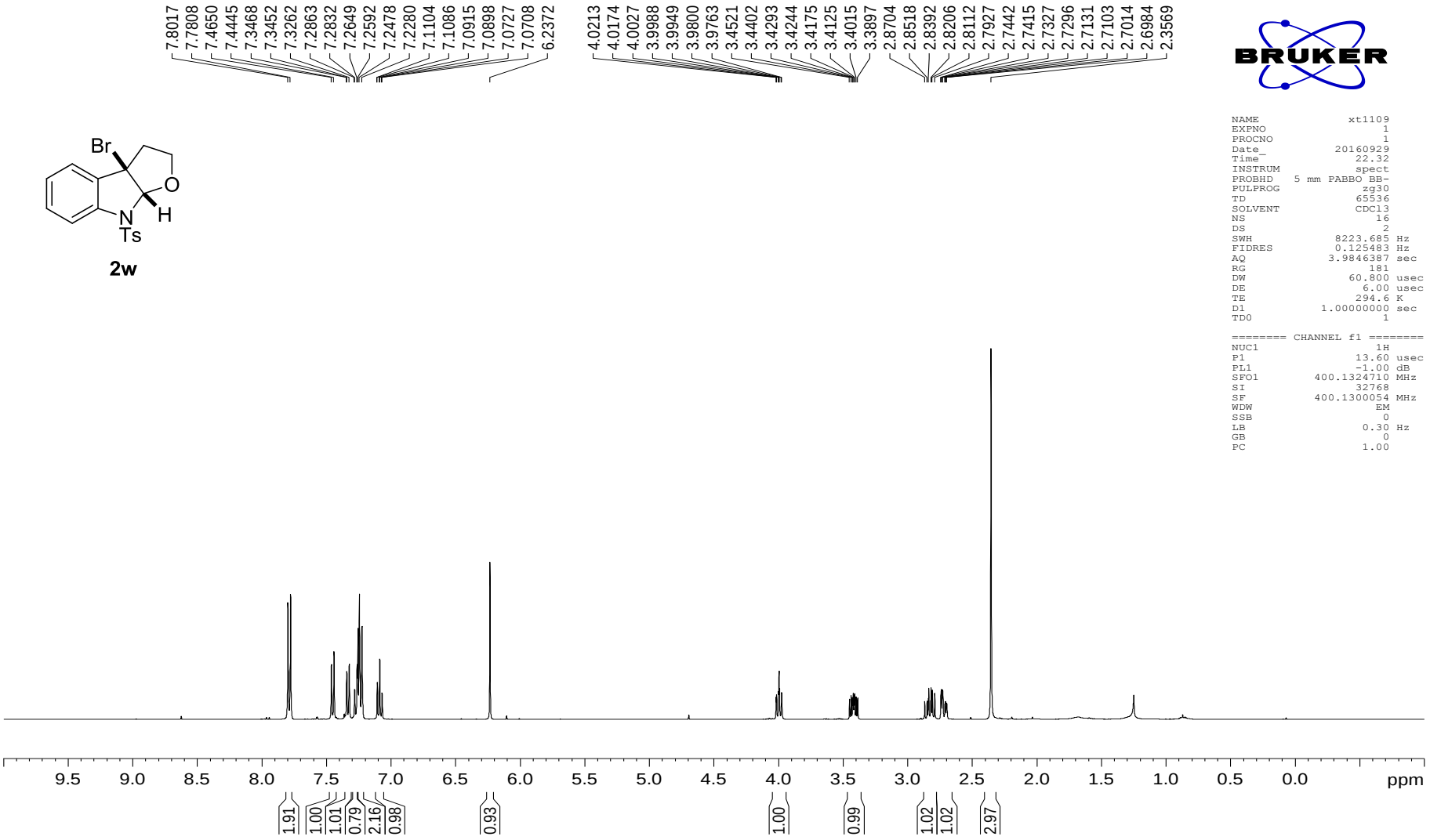


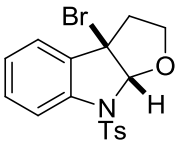
2v

```
NAME                xt1107
EXPNO                    1
PROCNO                   1
Date_             20160929
Time_                11.44
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                     88.84
DW                    62.400 usec
DE                     6.50 usec
TE                    297.8 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                    14.50 usec
SI                    65536
SF            400.1300094 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




152.53
141.55
135.92
131.92
130.72
128.78
128.45
128.12
124.98
124.32
115.10
100.84
77.48
77.16
76.84
68.06
67.83
61.80
45.14
Br
O
N
H
Cbz
2v
BRUKER
NAME xt1107-9
EXPNO 3
PROCNO 1
Date_ 20161002
Time 9.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10375
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 301.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127569 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.8017
7.7808
7.4650
7.4445
7.3468
7.3452
7.3262
7.2863
7.2832
7.2649
7.2592
7.2478
7.2280
7.1104
7.1086
7.0915
7.0898
7.0727
7.0708
6.2372
4.0213
4.0174
4.0027
3.9988
3.9949
3.9800
3.9763
3.4521
3.4402
3.4293
3.4244
3.4175
3.4125
3.4015
3.3897
2.8704
2.8518
2.8392
2.8206
2.8112
2.7927
2.7442
2.7415
2.7327
2.7296
2.7131
2.7103
2.7014
2.6984
2.3569
Br
O
N
H
Ts
2w
BRUKER
NAME xt1109
EXPNO 1
PROCNO 1
Date_ 20160929
Time_ 22.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.00 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.91
1.00
1.01
0.79
2.16
0.98
0.93
1.00
0.99
1.02
1.02
2.97

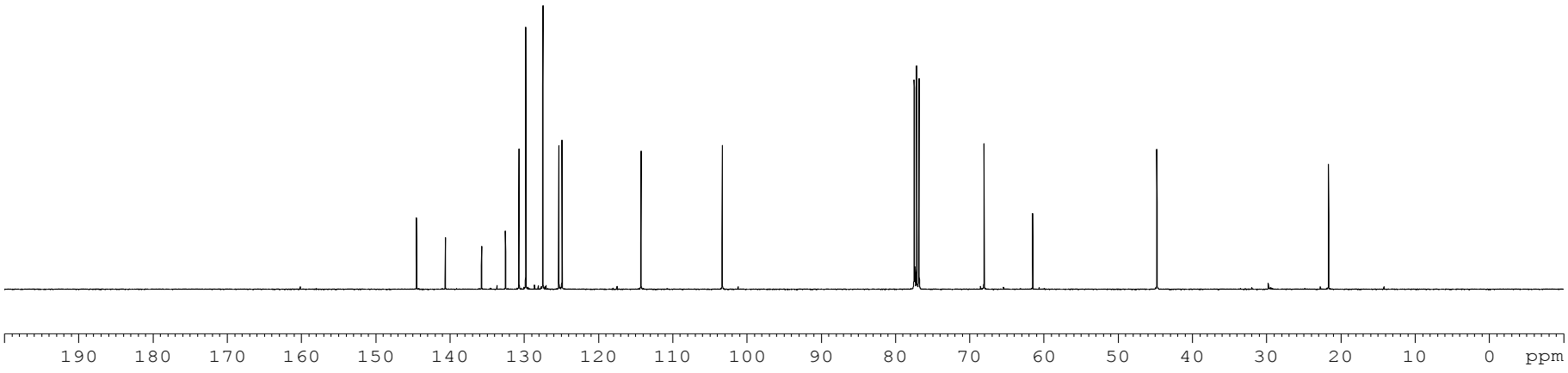


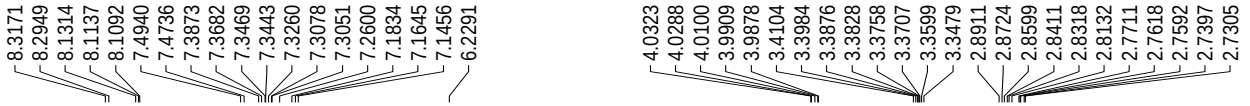
2w

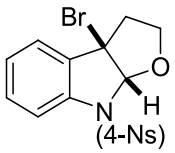
144.53
140.65
135.77
132.55
130.74
129.82
127.52
125.36
124.93
114.30
103.35
77.48
77.36
77.16
76.84
68.08
61.52
44.79
21.66

BRUKER

NAME	xt1109-1
EXPNO	2
PROCNO	1
Date_	20161002
Time	21.45
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	12000
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	196.92
DW	20.800 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228298 MHz
NUC1	13C
P1	9.70 usec
SI	32768
SF	100.6127597 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

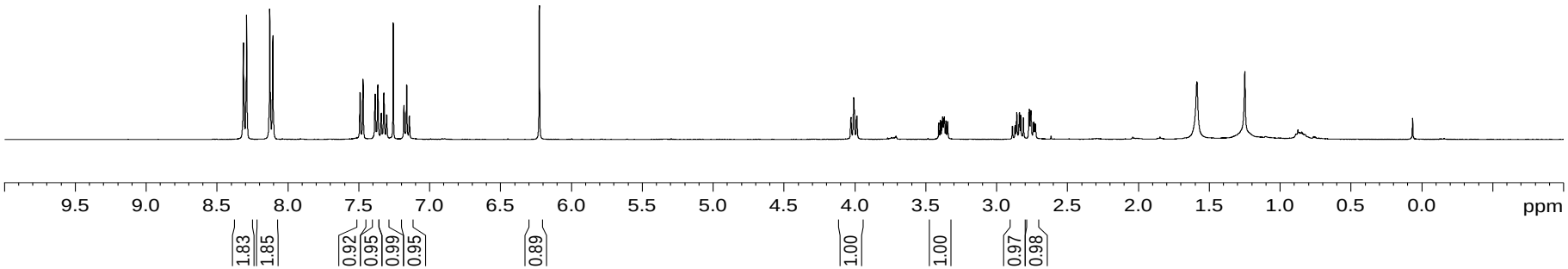


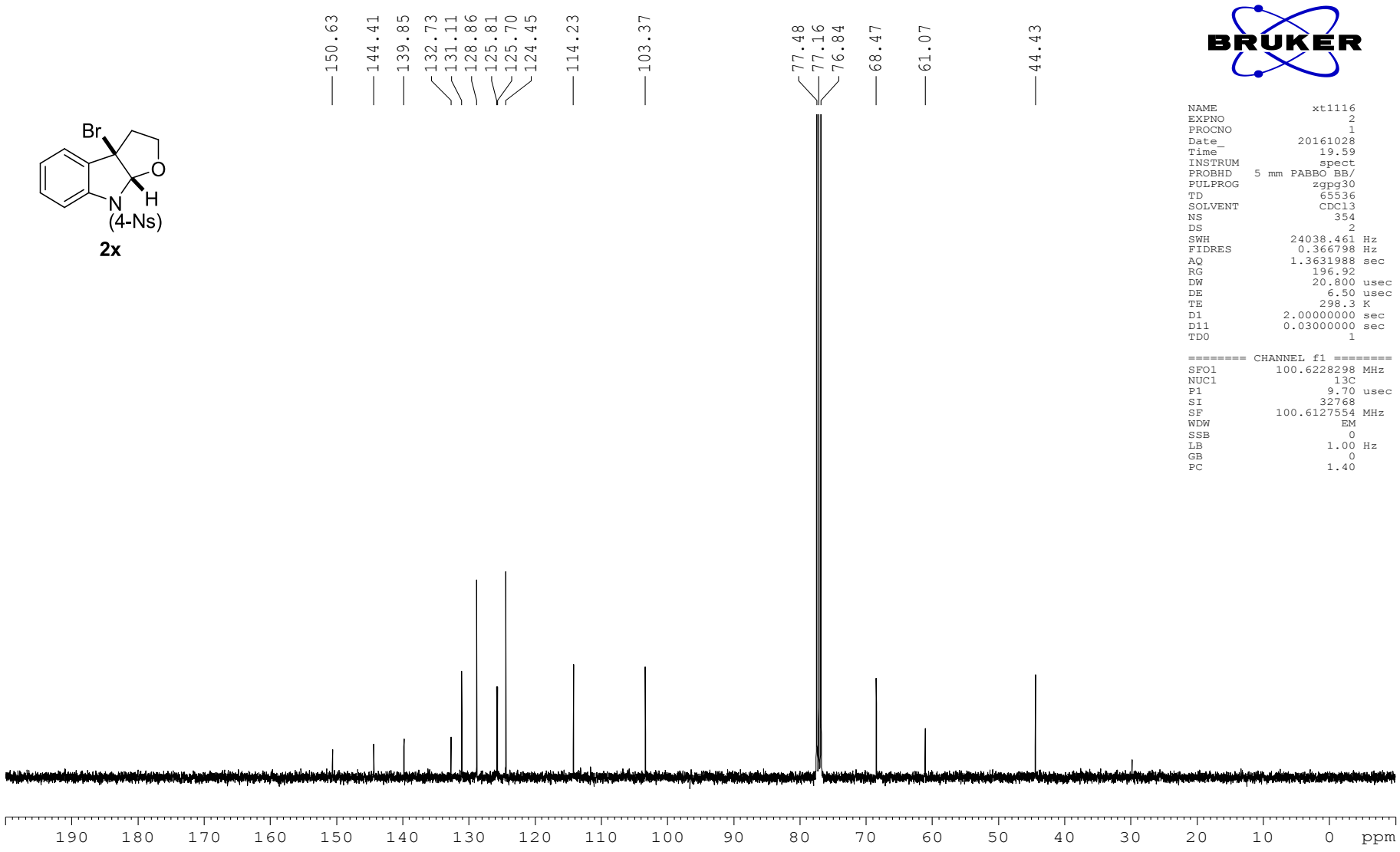


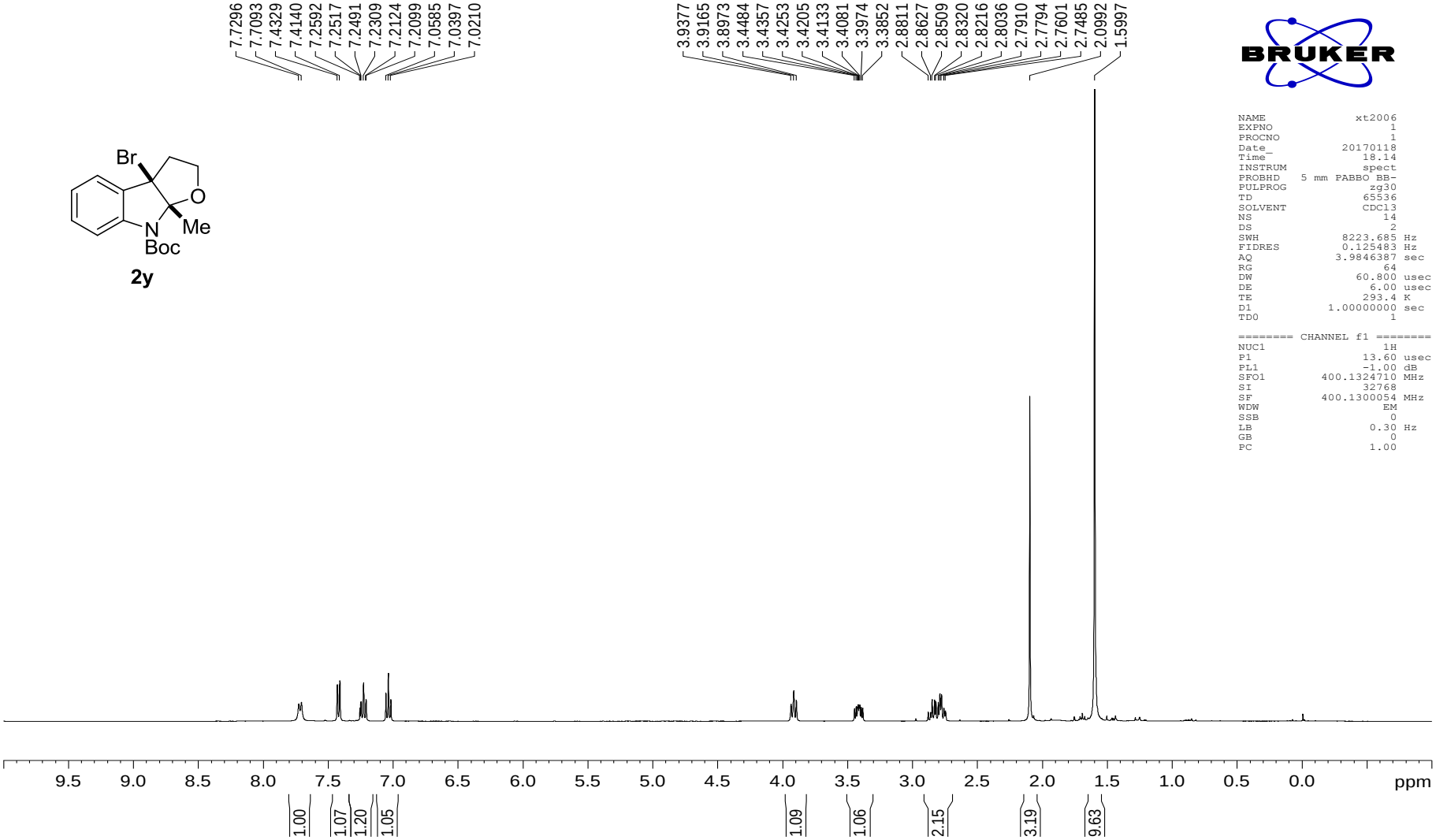


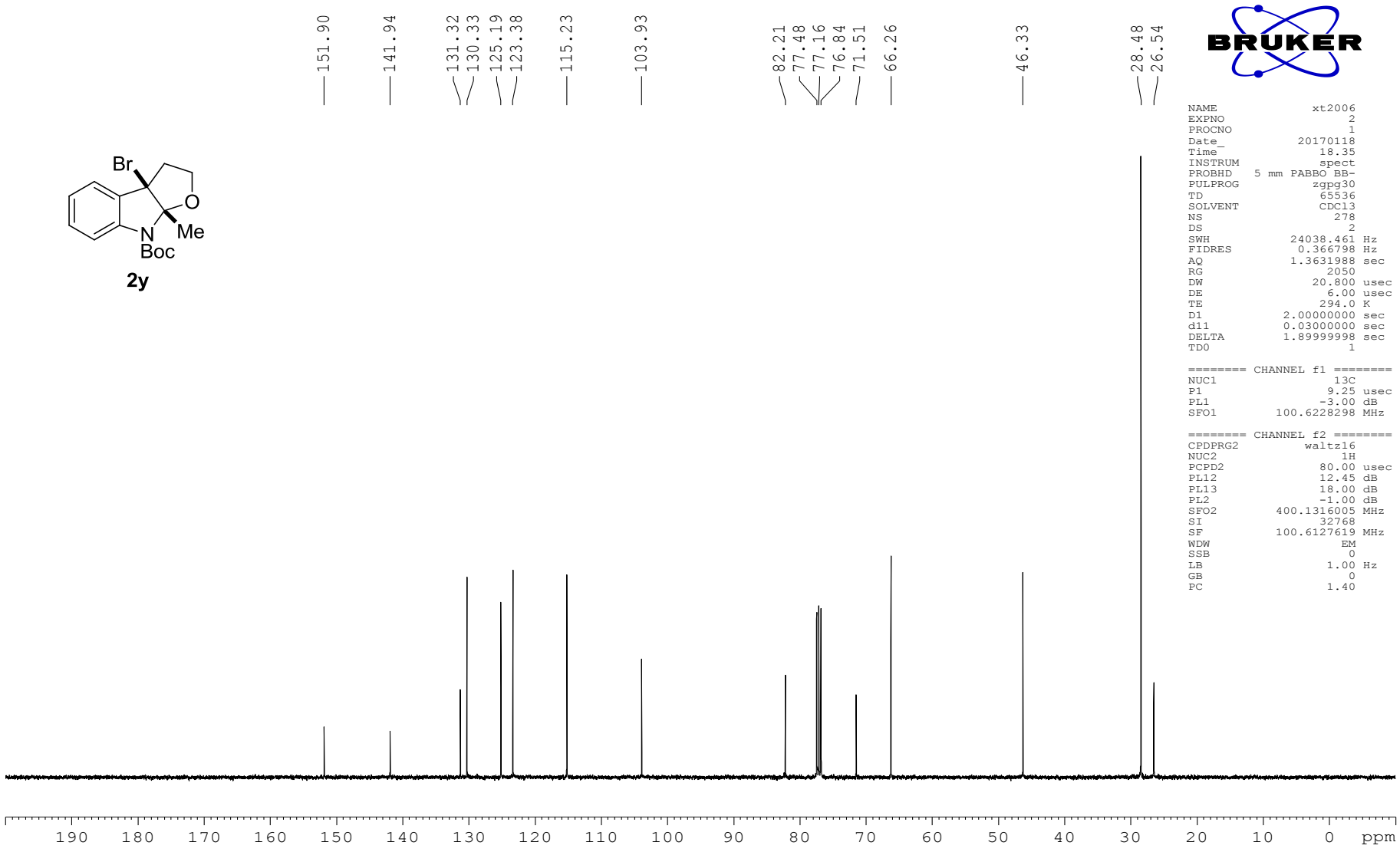
2x

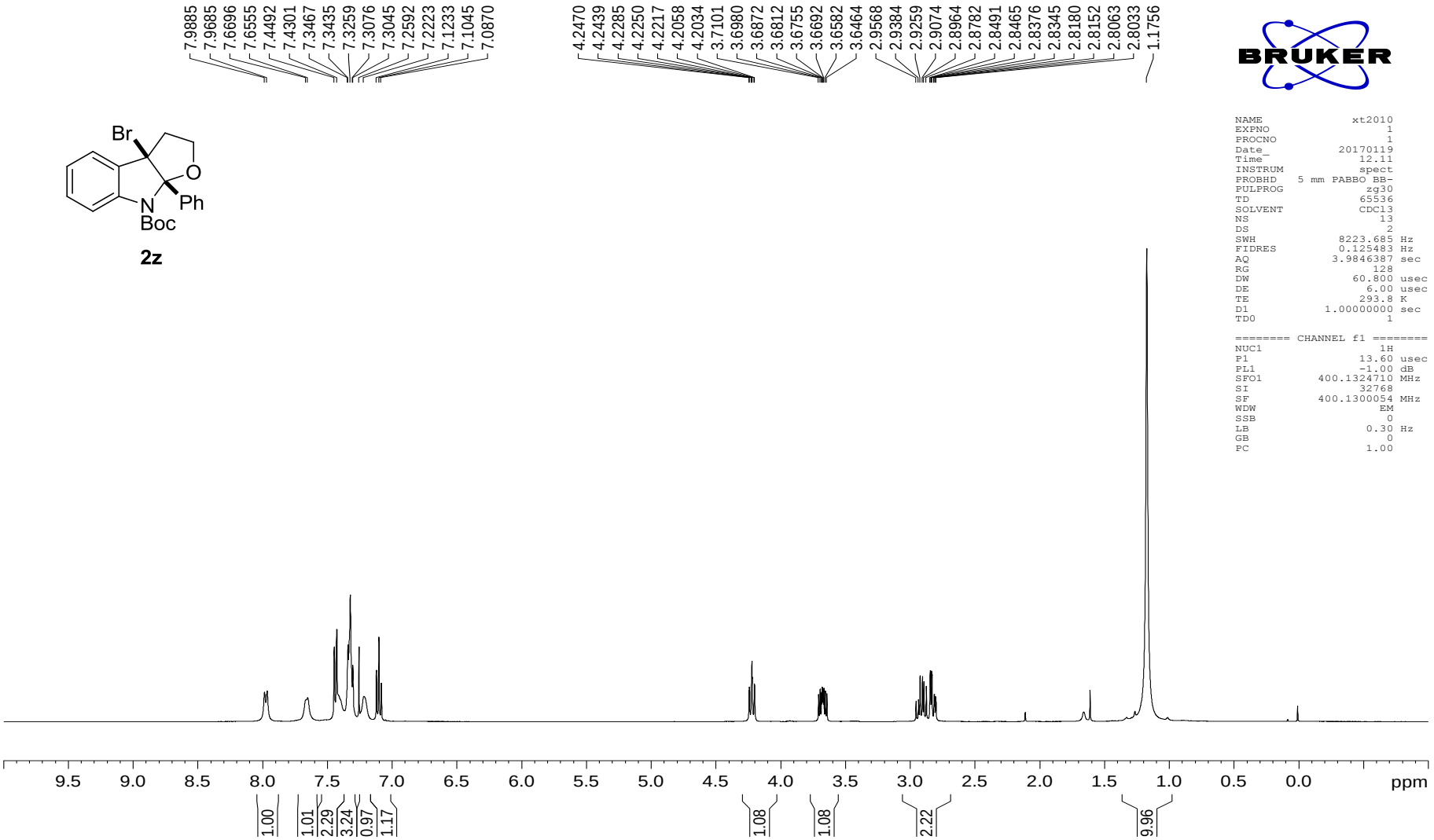
```
NAME                xt1116-2
EXPNO                      1
PROCNO                     1
Date_               20161028
Time                   19.03
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                         7
DS                         2
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                     126.97
DW                    62.400 usec
DE                     6.50 usec
TE                    296.8 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                      1H
P1                     14.50 usec
SI                     65536
SF             400.1300094 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00
```

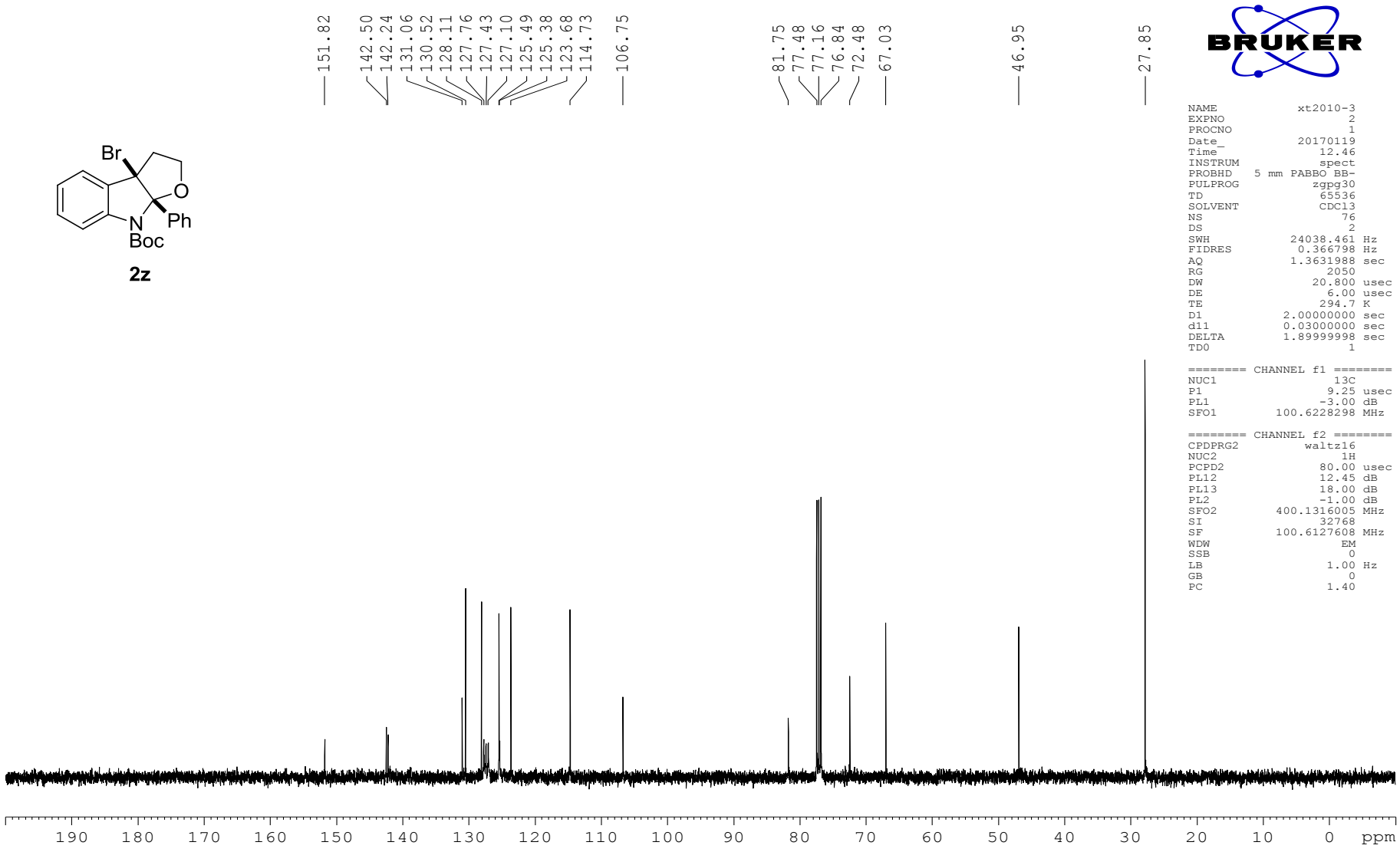


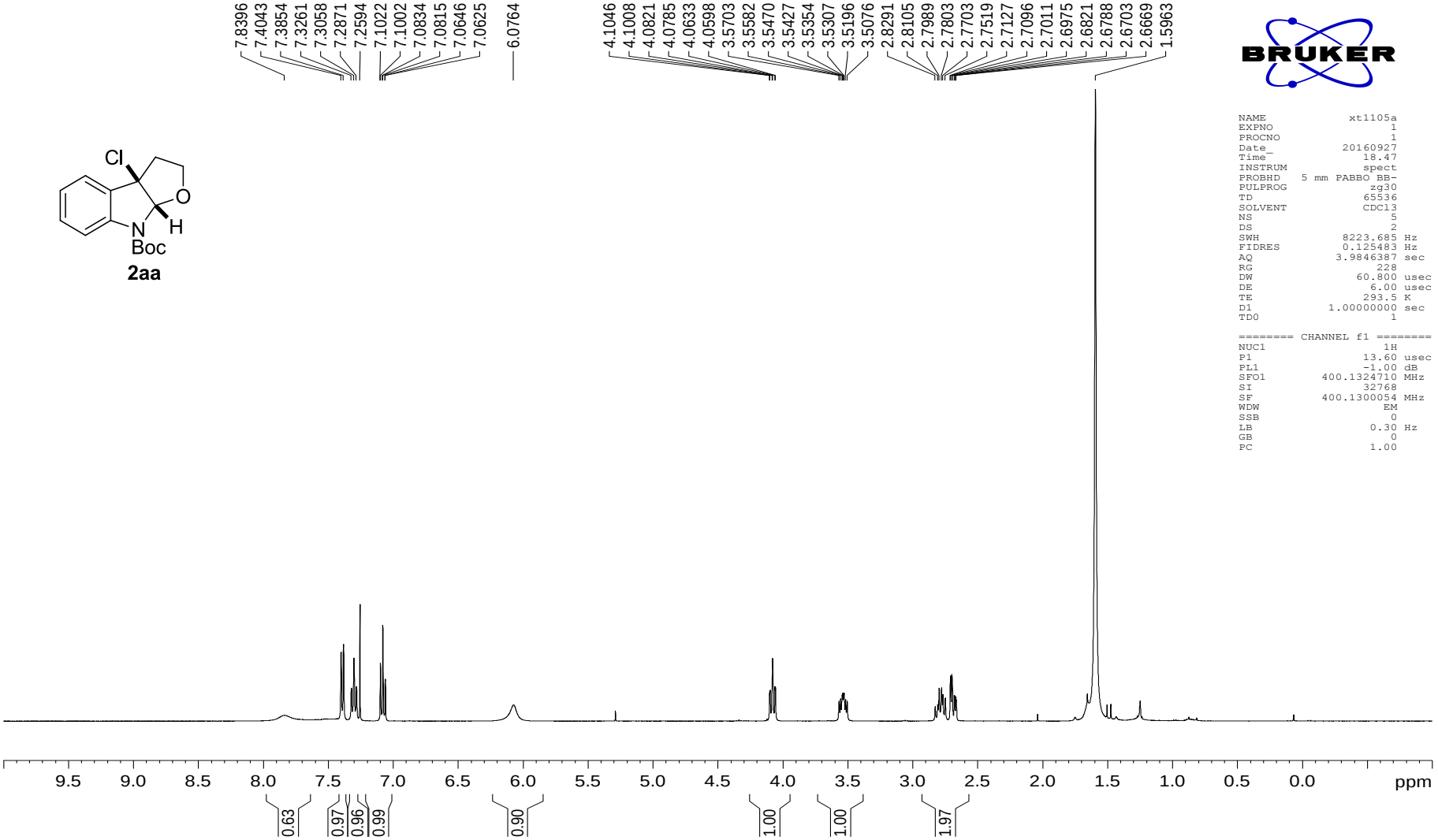

150.63
144.41
139.85
132.73
131.11
128.86
125.81
125.70
124.45
114.23
103.37
77.48
77.16
76.84
68.47
61.07
44.43
Br
O
N
H
(4-Ns)
2x
BRUKER
NAME xt1116
EXPNO 2
PROCNO 1
Date_ 20161028
Time 19.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 354
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127554 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

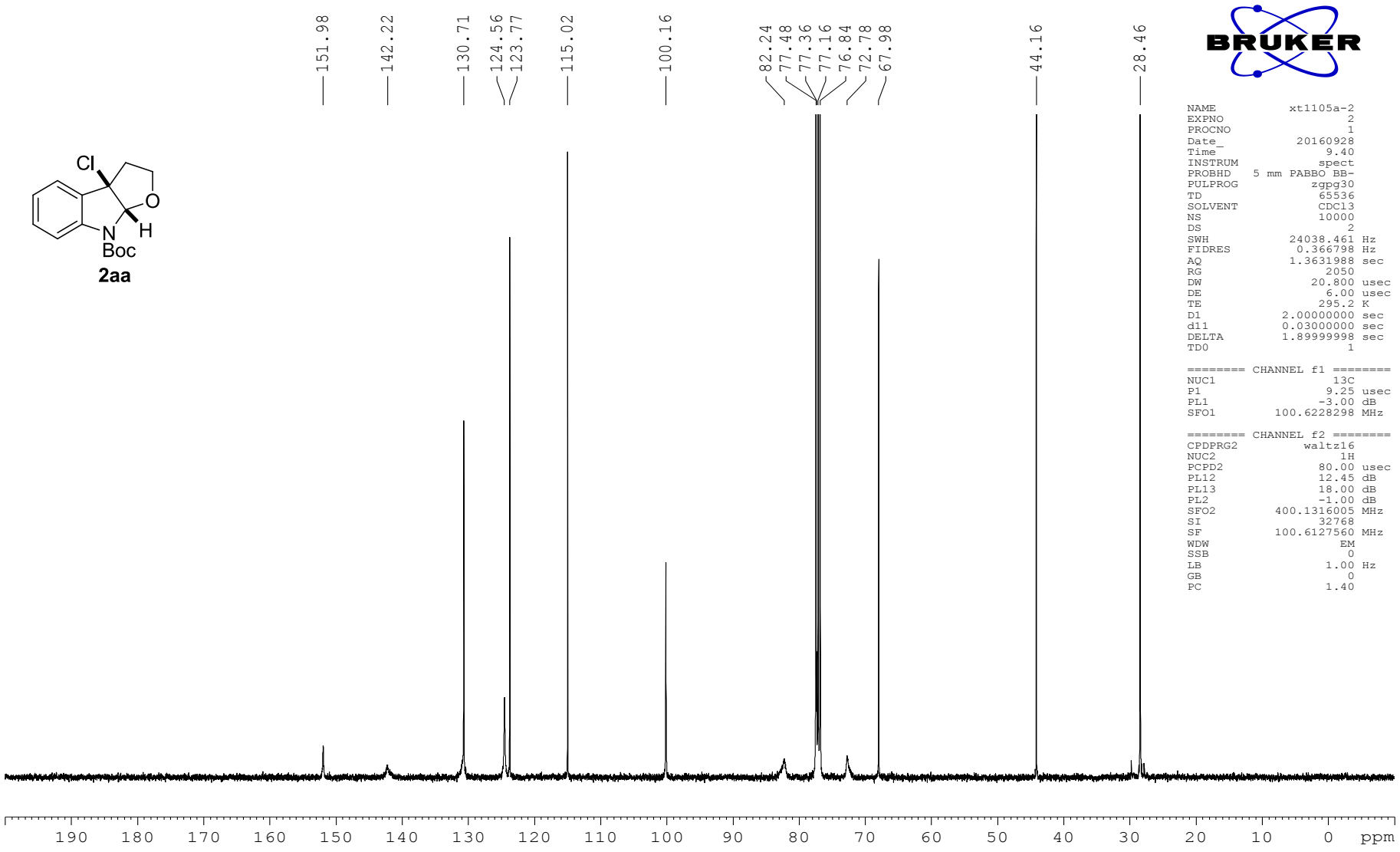

7.7296
7.7093
7.4329
7.4140
7.2592
7.2517
7.2491
7.2309
7.2124
7.2099
7.0585
7.0397
7.0210
3.9377
3.9165
3.8973
3.4484
3.4357
3.4253
3.4205
3.4133
3.4081
3.3974
3.3852
2.8811
2.8627
2.8509
2.8320
2.8216
2.8036
2.7910
2.7794
2.7601
2.7485
2.0992
1.5997
Br
O
N
Me
Boc
2y
BRUKER
NAME xt2006
EXPNO 1
PROCNO 1
Date_ 20170118
Time_ 18.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 14
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 64
DW 60.800 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.07
1.20
1.05
1.09
1.06
2.15
3.19
9.63

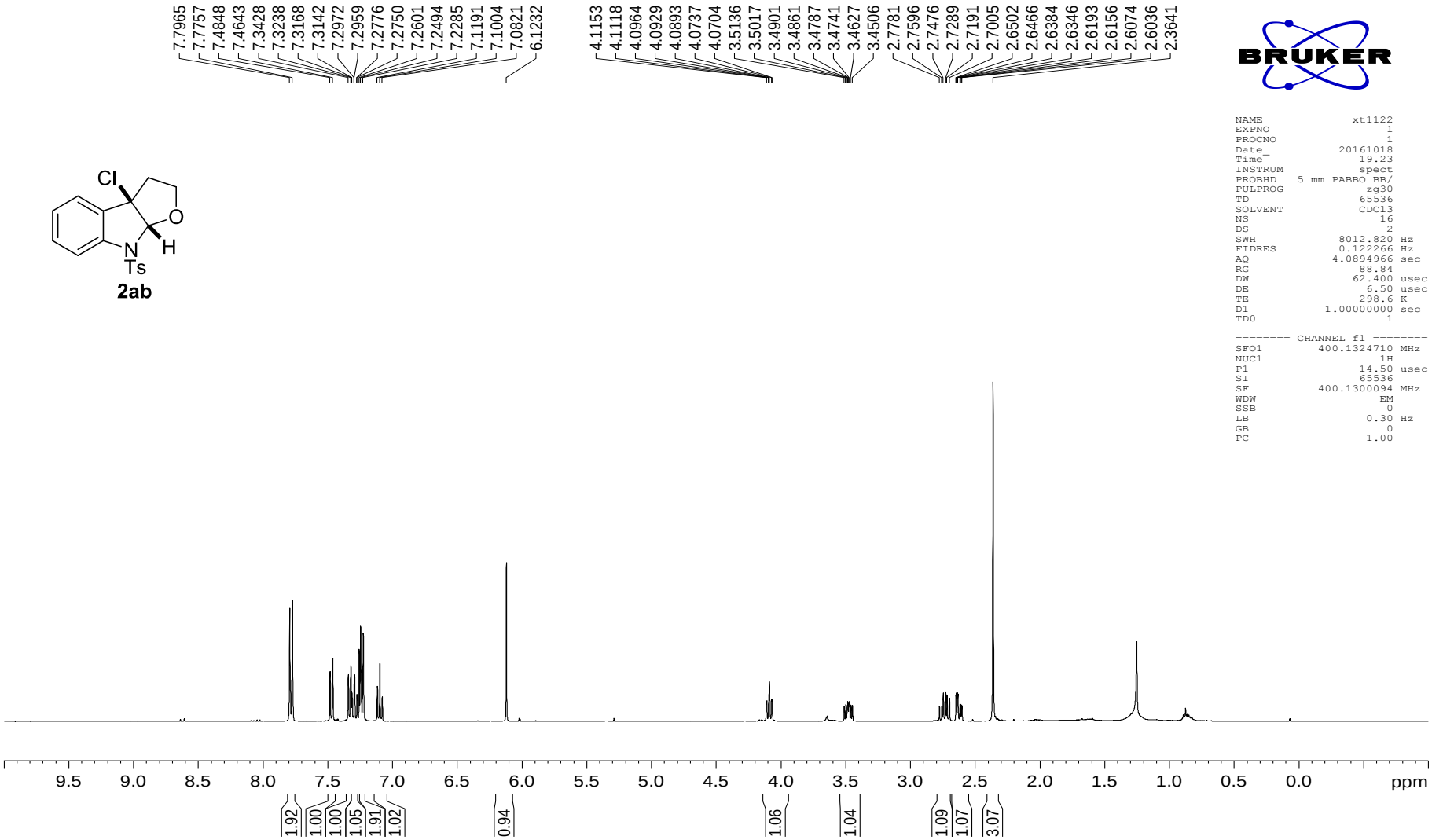

151.90
141.94
131.32
130.33
125.19
123.38
115.23
103.93
82.21
77.48
77.16
76.84
71.51
66.26
46.33
28.48
26.54
Br
O
N
Me
Boc
2y
BRUKER
NAME xt2006
EXPNO 2
PROCNO 1
Date_ 20170118
Time 18.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 278
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 294.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127619 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

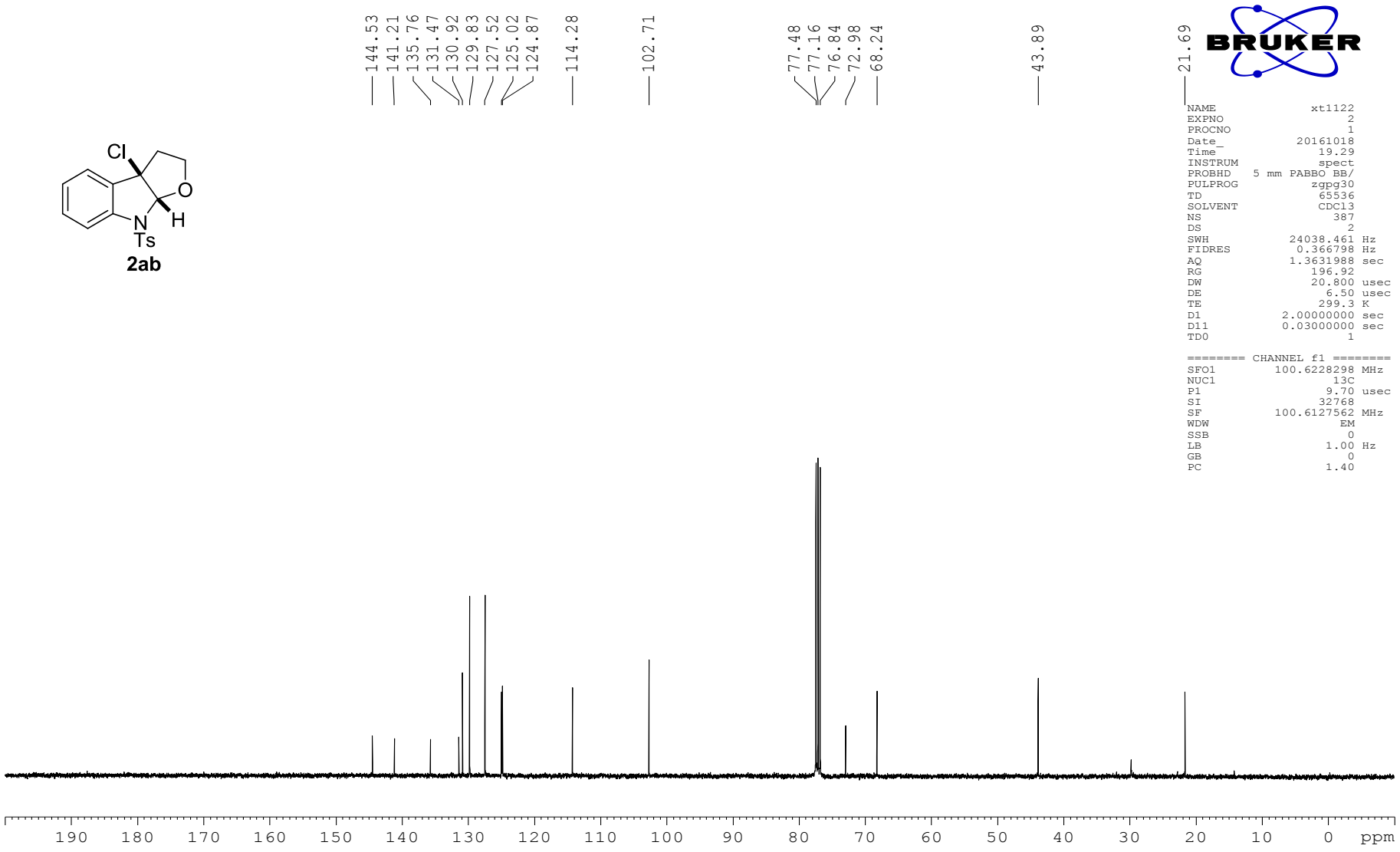

7.9885
7.9685
7.6696
7.6555
7.4492
7.4301
7.3467
7.3435
7.3259
7.3076
7.3045
7.2592
7.2223
7.1233
7.1045
7.0870
4.2470
4.2439
4.2285
4.2250
4.2217
4.2058
4.2034
3.7101
3.6980
3.6872
3.6812
3.6755
3.6692
3.6582
3.6464
2.9568
2.9384
2.9259
2.9074
2.8964
2.8782
2.8491
2.8465
2.8376
2.8345
2.8180
2.8152
2.8063
2.8033
1.1756
Br
O
N
Ph
Boc
2z
BRUKER
NAME xt2010
EXPNO 1
PROCNO 1
Date_ 20170119
Time_ 12.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 128
DW 60.800 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
2.29
3.24
0.97
1.17
1.08
1.08
2.22
9.96

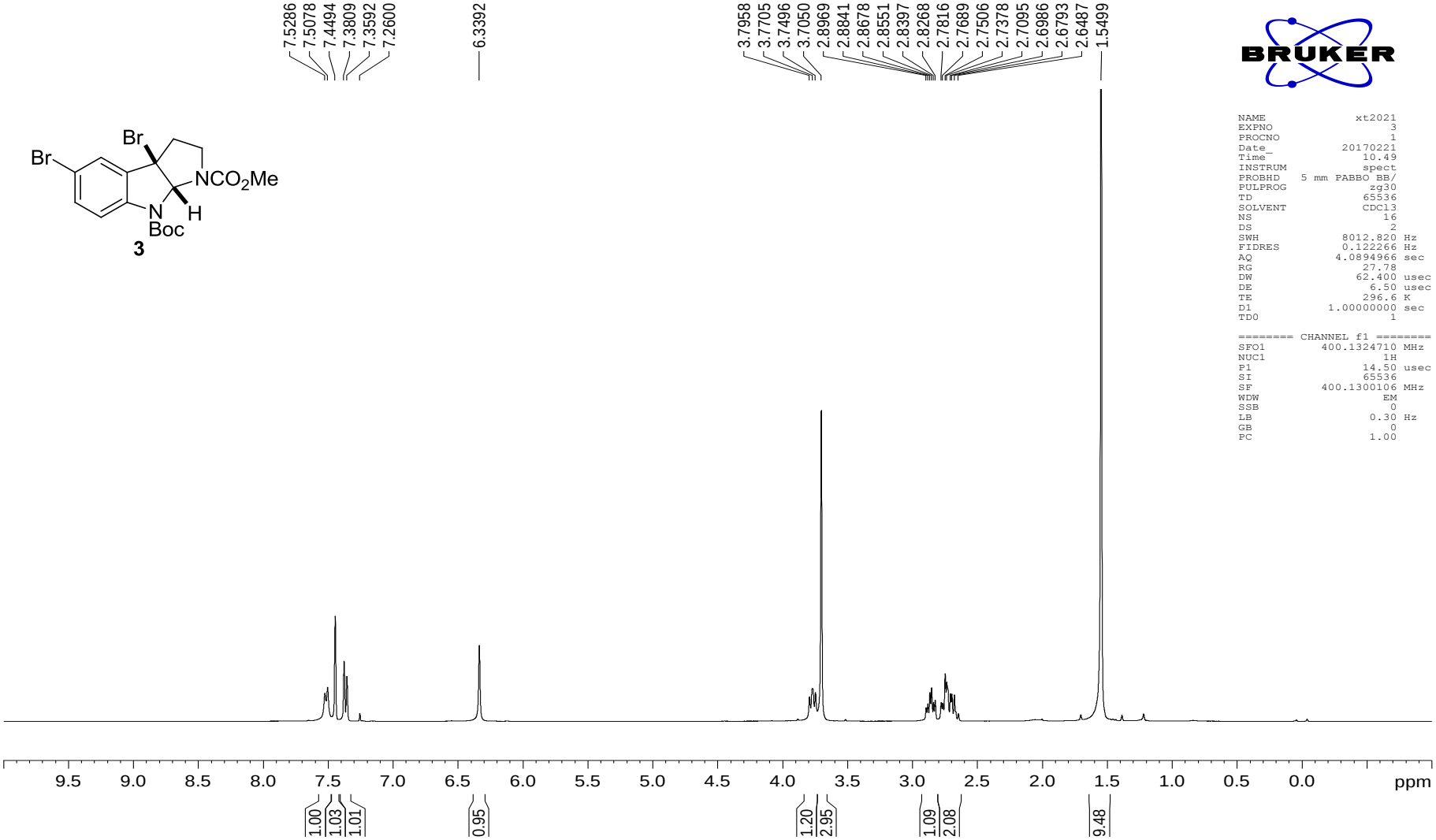

151.82
142.50
142.24
131.06
130.52
128.11
127.76
127.43
127.10
125.49
125.38
123.68
114.73
106.75
81.75
77.48
77.16
76.84
72.48
67.03
46.95
27.85
Br
O
N
Ph
Boc
2z
BRUKER
NAME xt2010-3
EXPNO 2
PROCNO 1
Date_ 20170119
Time 12.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 76
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127608 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

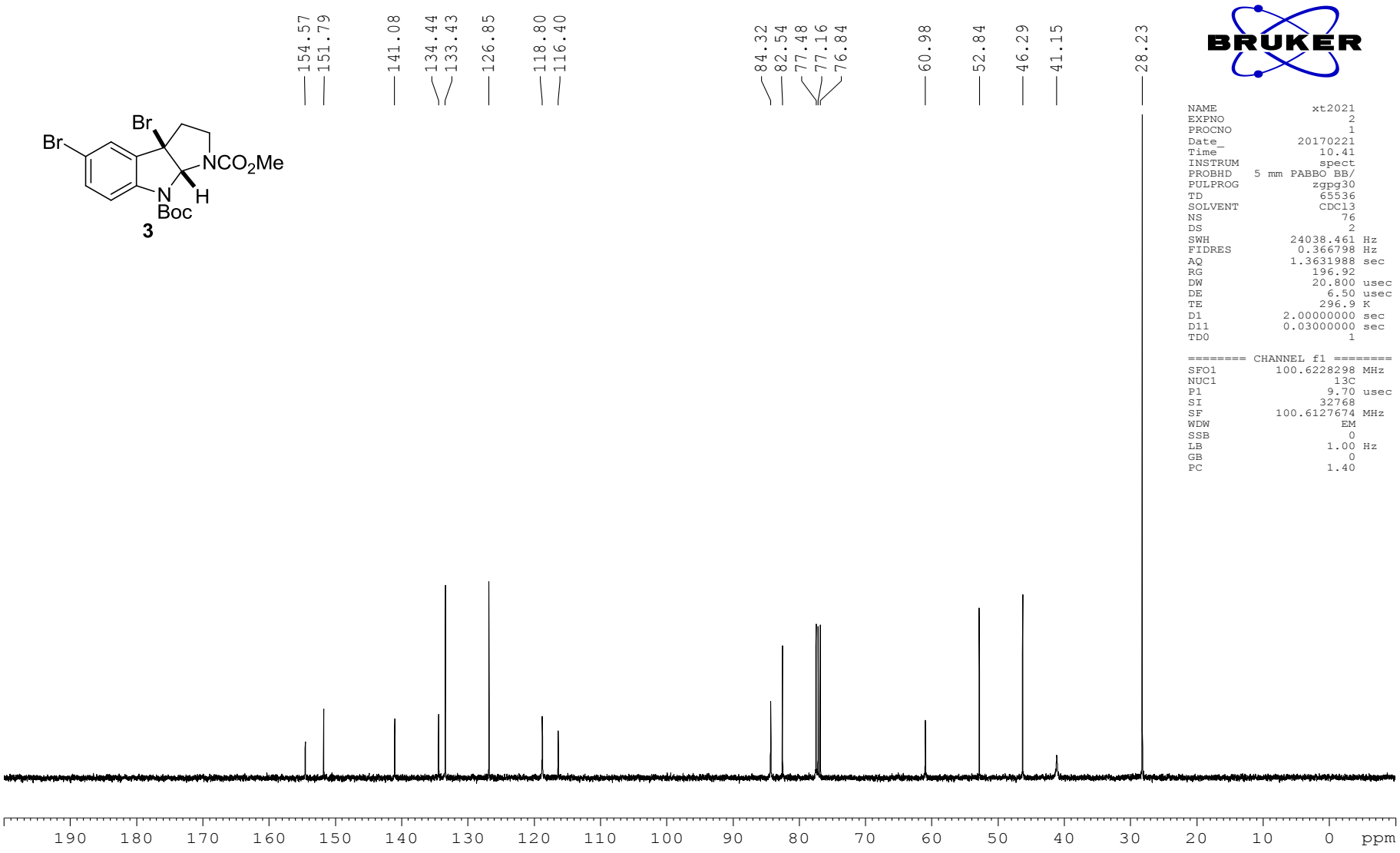

7.8396
7.4043
7.3854
7.3261
7.3058
7.2871
7.2594
7.1022
7.1002
7.0834
7.0815
7.0646
7.0625
6.0764
4.1046
4.1008
4.0821
4.0785
4.0633
4.0598
3.5703
3.5582
3.5470
3.5427
3.5354
3.5307
3.5196
3.5076
2.8291
2.8105
2.7989
2.7803
2.7703
2.7519
2.7127
2.7096
2.7011
2.6975
2.6821
2.6788
2.6703
2.6669
1.5963
Cl
O
N
H
Boc
2aa
BRUKER
NAME xt1105a
EXPNO 1
PROCNO 1
Date_ 20160927
Time_ 18.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 228
DW 60.800 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.63
0.97
0.96
0.99
0.90
1.00
1.00
1.97

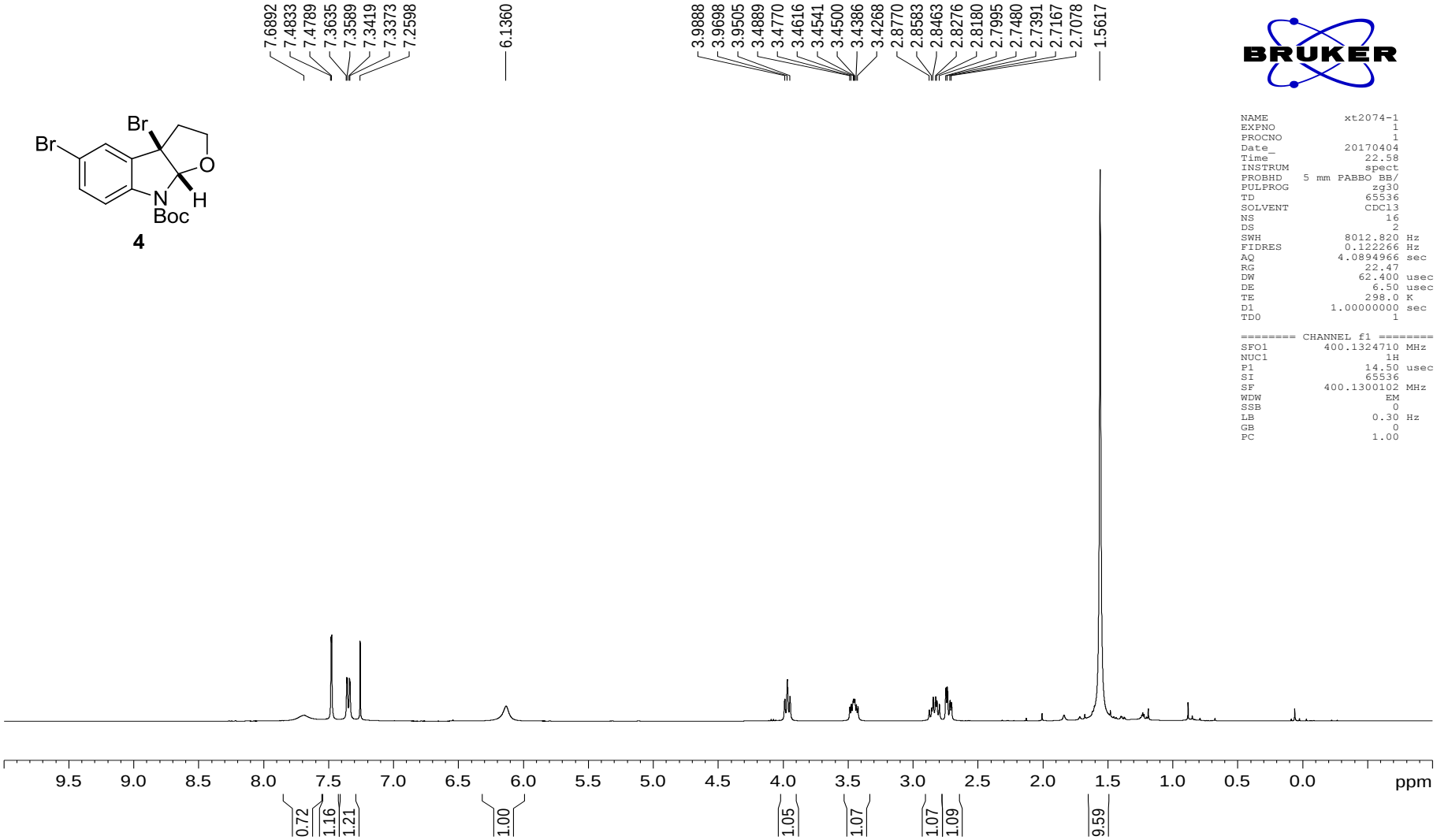

151.98
142.22
130.71
124.56
123.77
115.02
100.16
82.24
77.48
77.36
77.16
76.84
72.78
67.98
44.16
28.46
Cl
O
N
H
Boc
2aa
BRUKER
NAME xt1105a-2
EXPNO 2
PROCNO 1
Date_ 20160928
Time 9.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10000
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

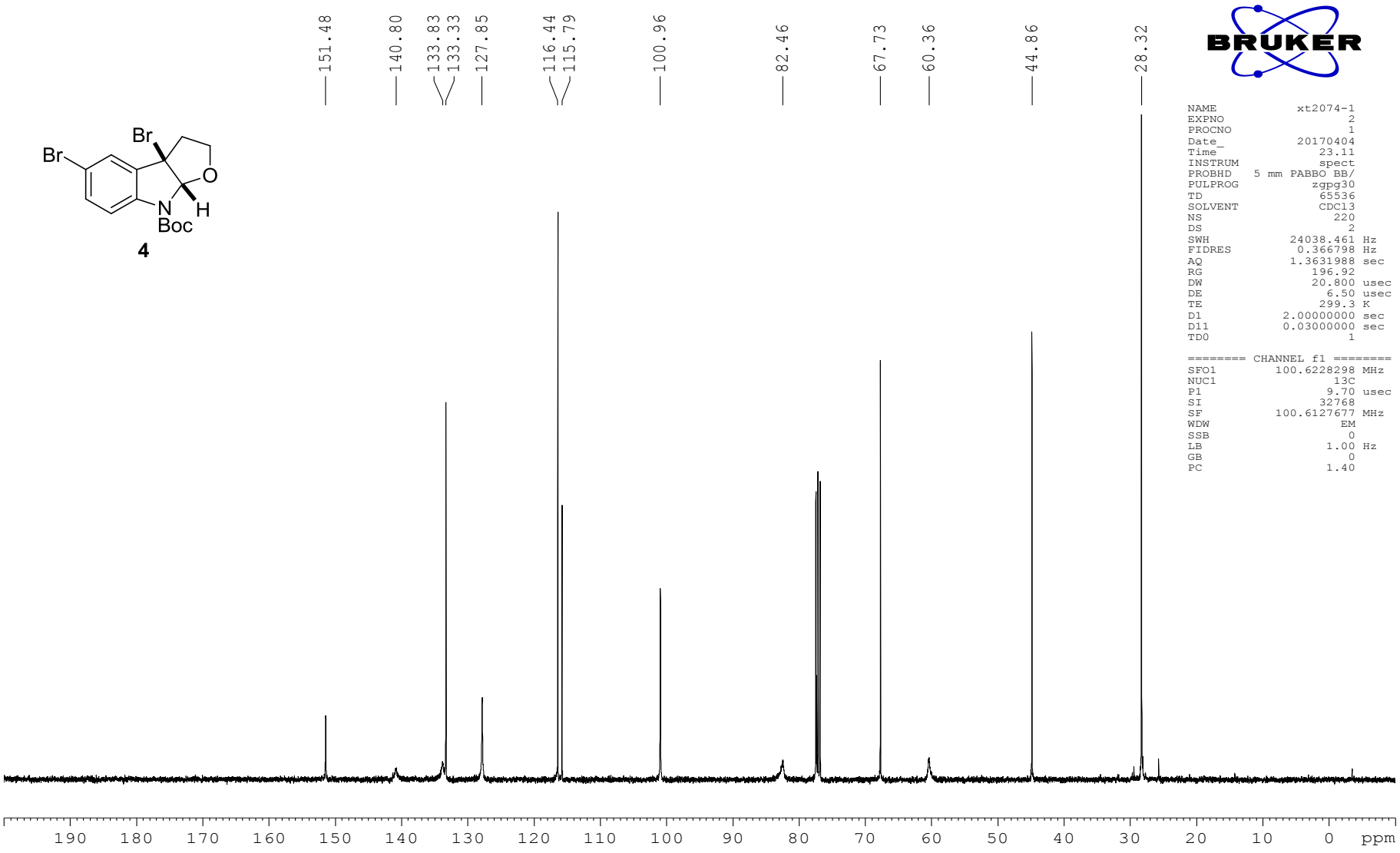

7.7965
7.7757
7.4848
7.4643
7.3428
7.3238
7.3168
7.3142
7.2972
7.2959
7.2776
7.2750
7.2601
7.2494
7.2285
7.1191
7.1004
7.0821
6.1232
4.1153
4.1118
4.0964
4.0929
4.0893
4.0737
4.0704
3.5136
3.5017
3.4901
3.4861
3.4787
3.4741
3.4627
3.4506
2.7781
2.7596
2.7476
2.7289
2.7191
2.7005
2.6502
2.6466
2.6384
2.6346
2.6193
2.6156
2.6074
2.6036
2.3641
BRUKER
Cl
O
H
N
Ts
2ab
NAME xt1122
EXPNO 1
PROCNO 1
Date_ 20161018
Time_ 19.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 88.84
DW 62.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.92
1.00
1.00
1.05
1.91
1.02
0.94
1.06
1.04
1.09
1.07
3.07

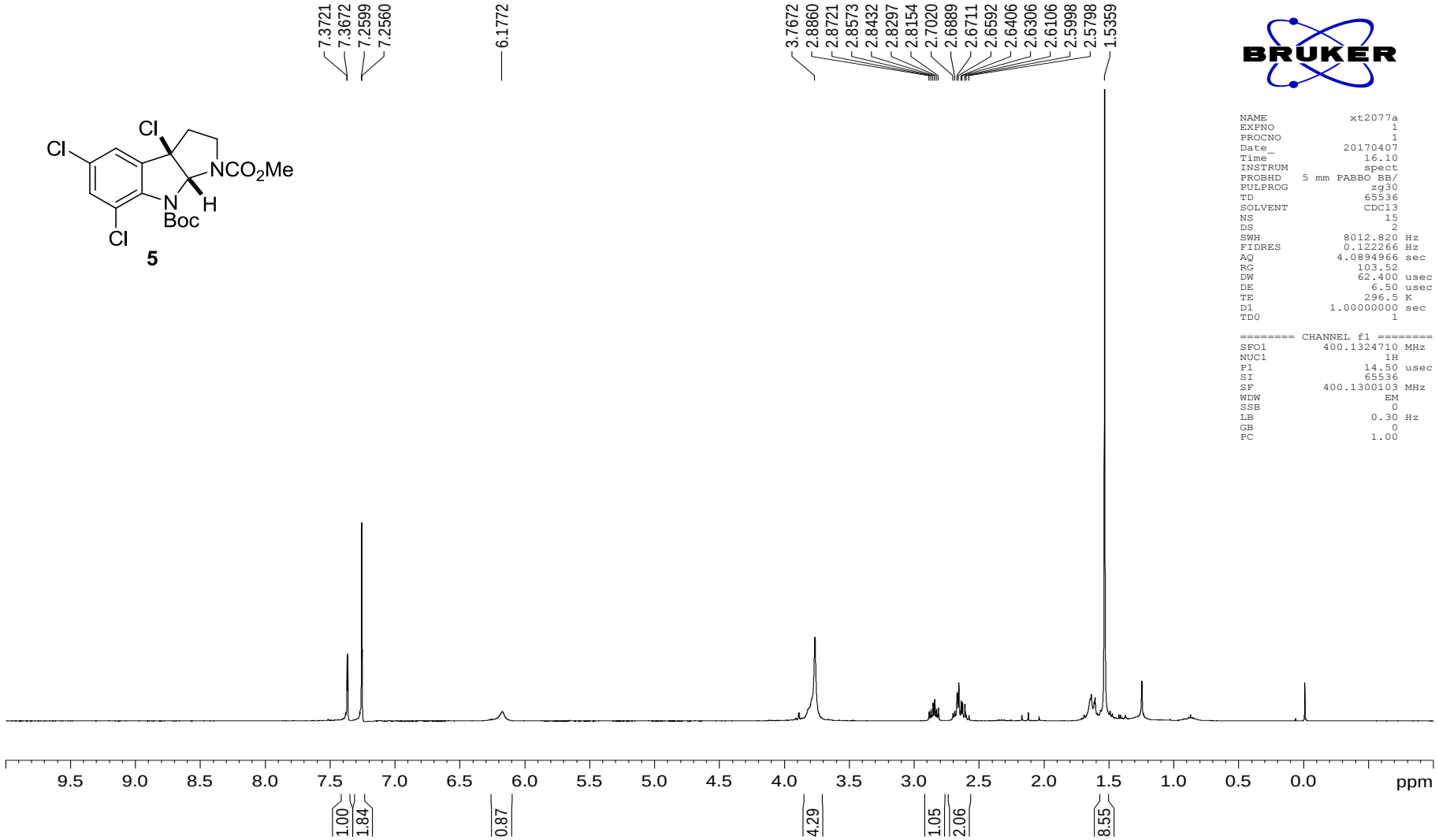

144.53
141.21
135.76
131.47
130.92
129.83
127.52
125.02
124.87
114.28
102.71
77.48
77.16
76.84
72.98
68.24
43.89
21.69
Cl
O
H
N
Ts
2ab
BRUKER
NAME xt1122
EXPNO 2
PROCNO 1
Date_ 20161018
Time 19.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 387
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

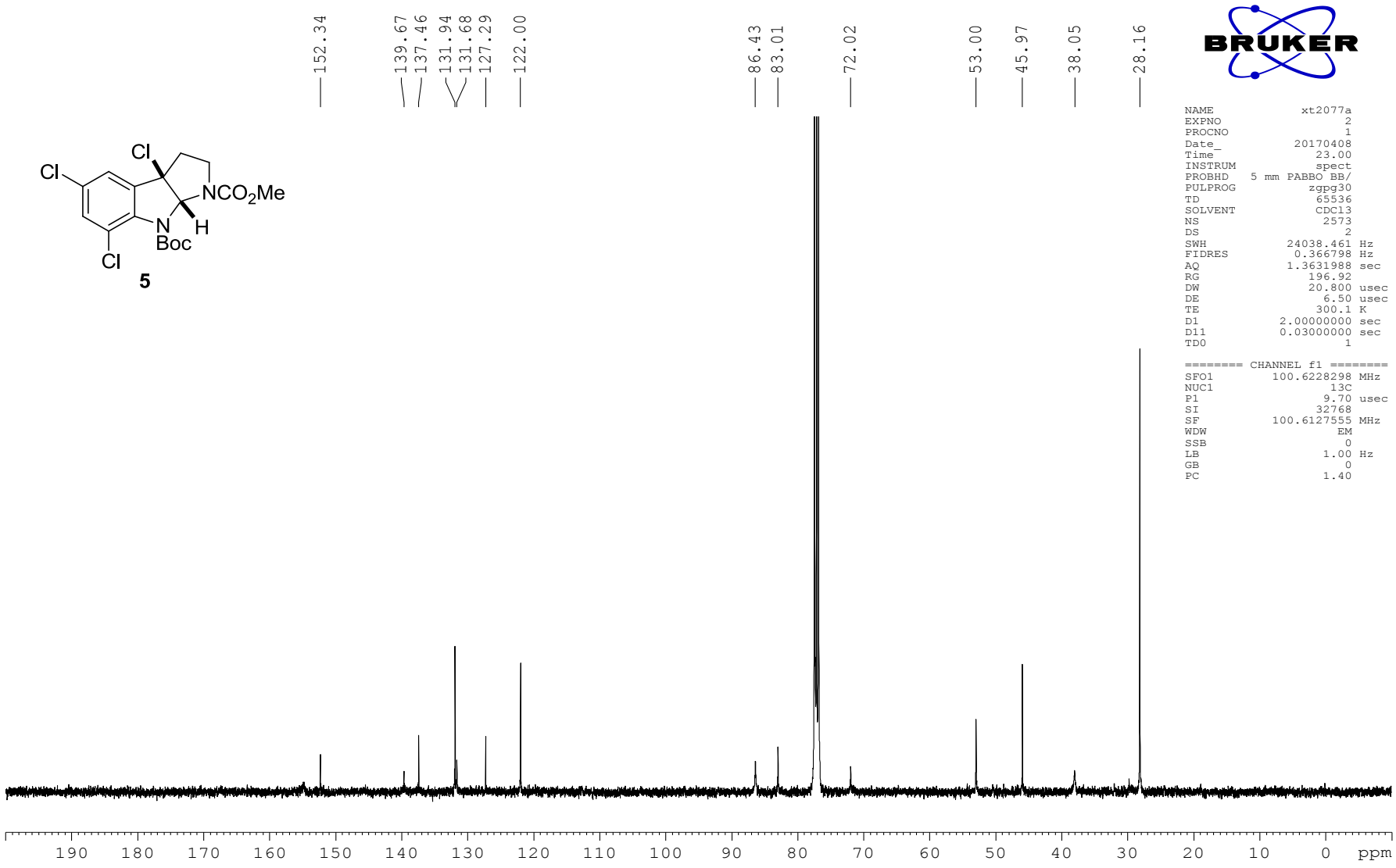

7.5286
7.5078
7.4494
7.3809
7.3592
7.2600
6.3392
3.7958
3.7705
3.7496
3.7050
2.8969
2.8841
2.8678
2.8551
2.8397
2.8268
2.7816
2.7689
2.7506
2.7378
2.7095
2.6986
2.6793
2.6487
1.5499
Br
Br
NCO2Me
N
H
Boc
3
BRUKER
NAME xt2021
EXPNO 3
PROCNO 1
Date_ 20170221
Time_ 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 27.78
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.03
1.01
0.95
1.20
2.95
1.09
2.08
9.48

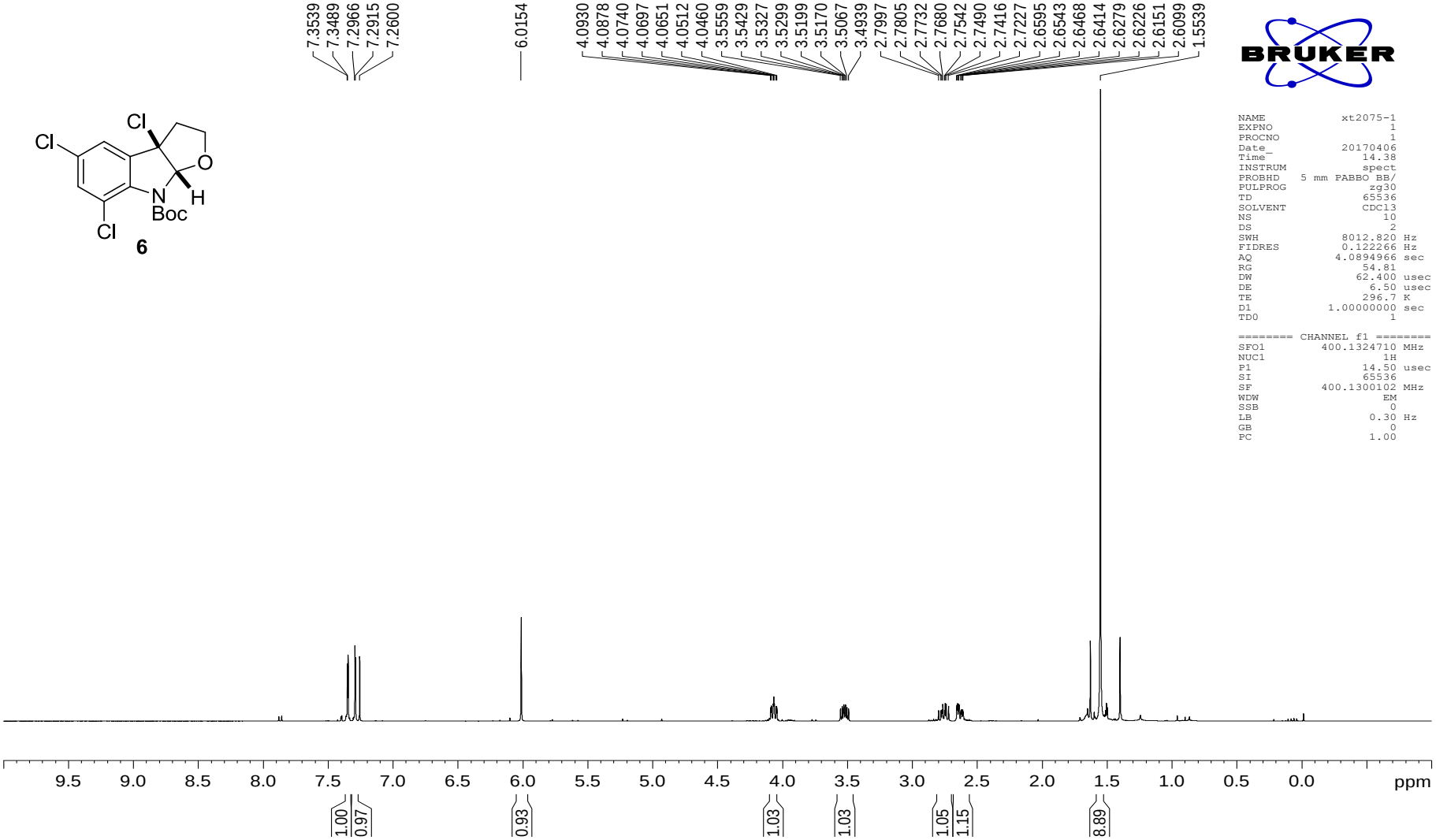

154.57
151.79
141.08
134.44
133.43
126.85
118.80
116.40
84.32
82.54
77.48
77.16
76.84
60.98
52.84
46.29
41.15
28.23
Br
Br
NCO2Me
N
H
Boc
3
NAME xt2021
EXPNO 2
PROCNO 1
Date_ 20170221
Time 10.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 76
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127674 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

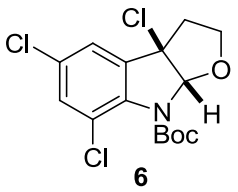

7.6892
7.4833
7.4789
7.3635
7.3589
7.3419
7.3373
7.2598
6.1360
3.9888
3.9698
3.9505
3.4889
3.4770
3.4616
3.4541
3.4500
3.4386
3.4268
2.8770
2.8583
2.8463
2.8276
2.8180
2.7995
2.7480
2.7391
2.7167
2.7078
1.5617
Br
Br
O
N
H
Boc
4
BRUKER
NAME xt2074-1
EXPNO 1
PROCNO 1
Date_ 20170404
Time 22.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 22.47
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.72
1.16
1.21
1.00
1.05
1.07
1.07
1.09
9.59


151.48
140.80
133.83
133.33
127.85
116.44
115.79
100.96
82.46
67.73
60.36
44.86
28.32
Br
Br
O
N
H
Boc
4
BRUKER
NAME xt2074-1
EXPNO 2
PROCNO 1
Date_ 20170404
Time 23.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 220
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127677 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.3721
7.3672
7.2599
7.2560
6.1772
3.7672
2.8860
2.8721
2.8573
2.8432
2.8297
2.8154
2.7020
2.6889
2.6711
2.6592
2.6406
2.6306
2.6106
2.5998
2.5798
1.5359
Cl
Cl
Cl
NCO2Me
N
Boc
H
5
BRUKER
NAME xt2077a
EXPNO 1
PROCNO 1
Date_ 20170407
Time 16.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 103.52
DW 62.400 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm
1.00
1.84
0.87
4.29
1.05
2.06
8.55

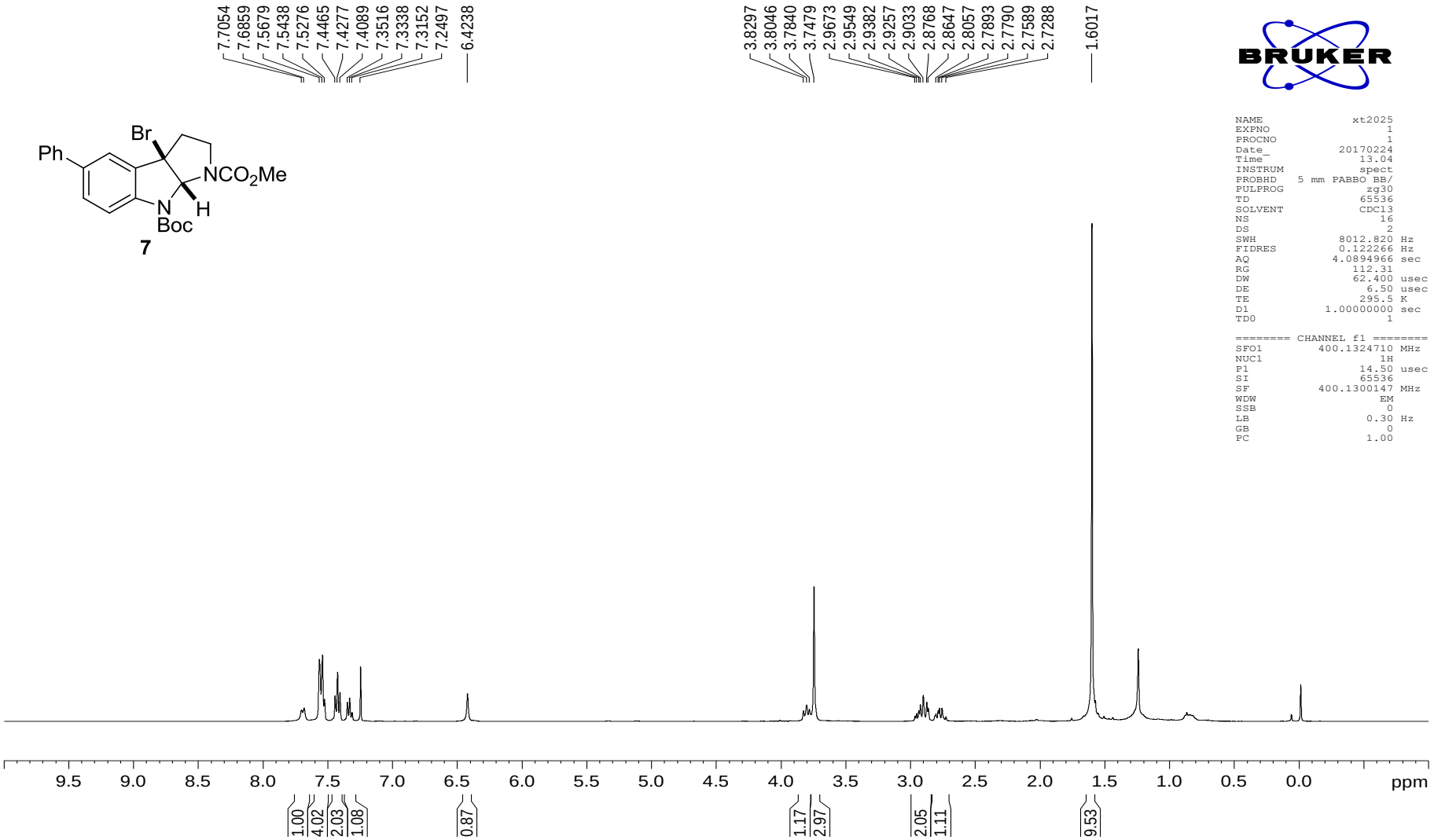

152.34
139.67
137.46
131.94
131.68
127.29
122.00
86.43
83.01
72.02
53.00
45.97
38.05
28.16
Cl
Cl
NCO2Me
N
H
Boc
Cl
5
BRUKER
NAME xt2077a
EXPNO 2
PROCNO 1
Date_ 20170408
Time 23.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2573
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

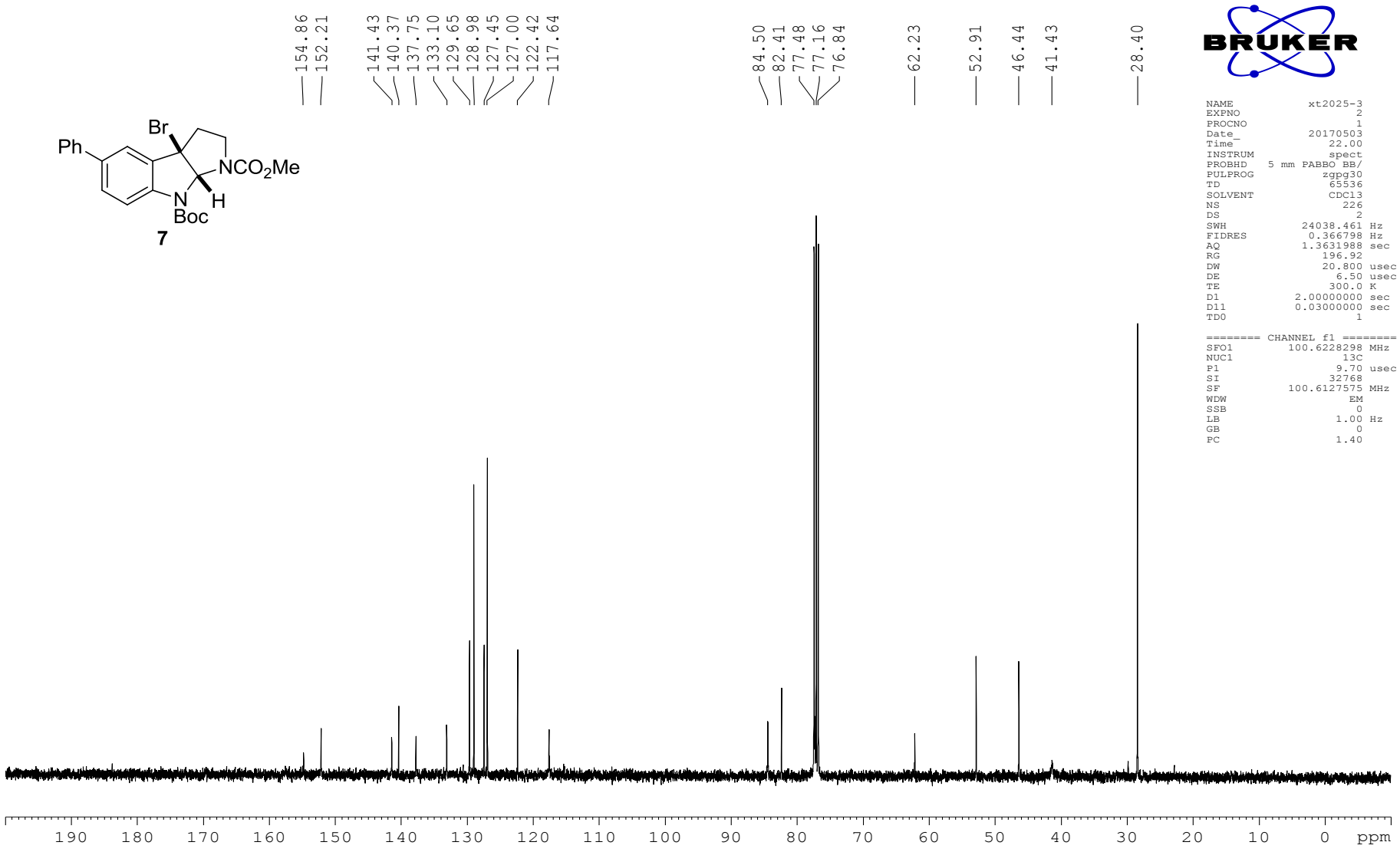

7.3539
7.3489
7.2966
7.2915
7.2600
6.0154
4.0930
4.0878
4.0740
4.0697
4.0651
4.0512
4.0460
3.5559
3.5429
3.5327
3.5299
3.5199
3.5170
3.5067
3.4939
2.7997
2.7805
2.7732
2.7680
2.7542
2.7490
2.7416
2.7227
2.6595
2.6543
2.6468
2.6414
2.6279
2.6226
2.6151
2.6099
1.5539
BRUKER
NAME xt2075-1
EXPNO 1
PROCNO 1
Date_ 20170406
Time_ 14.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 54.81
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
Cl
Cl
O
N
H
Boc
6
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.97
0.93
1.03
1.03
1.05
1.15
8.89

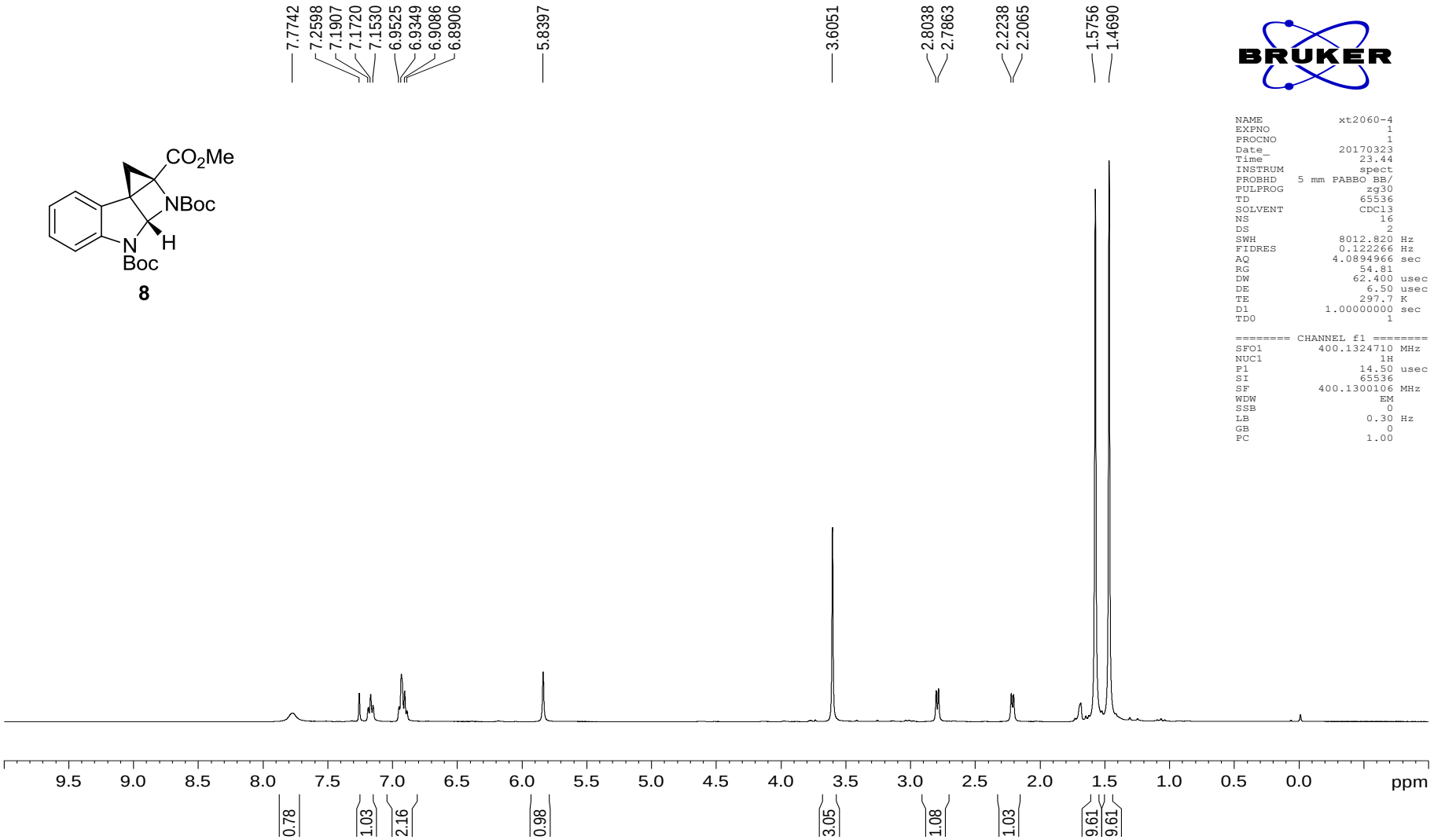

Cl
Cl
O
N
H
Boc
Cl

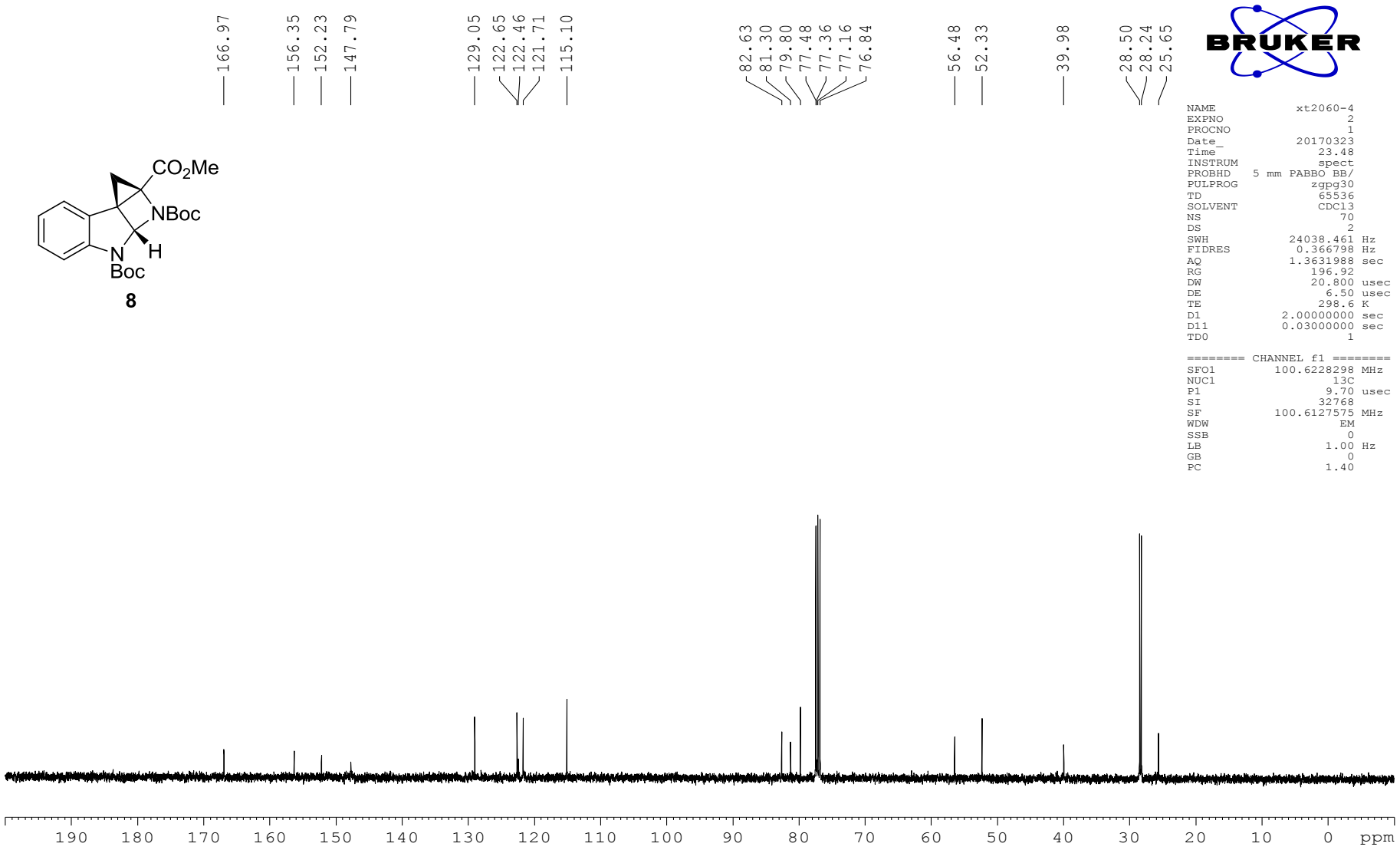
6

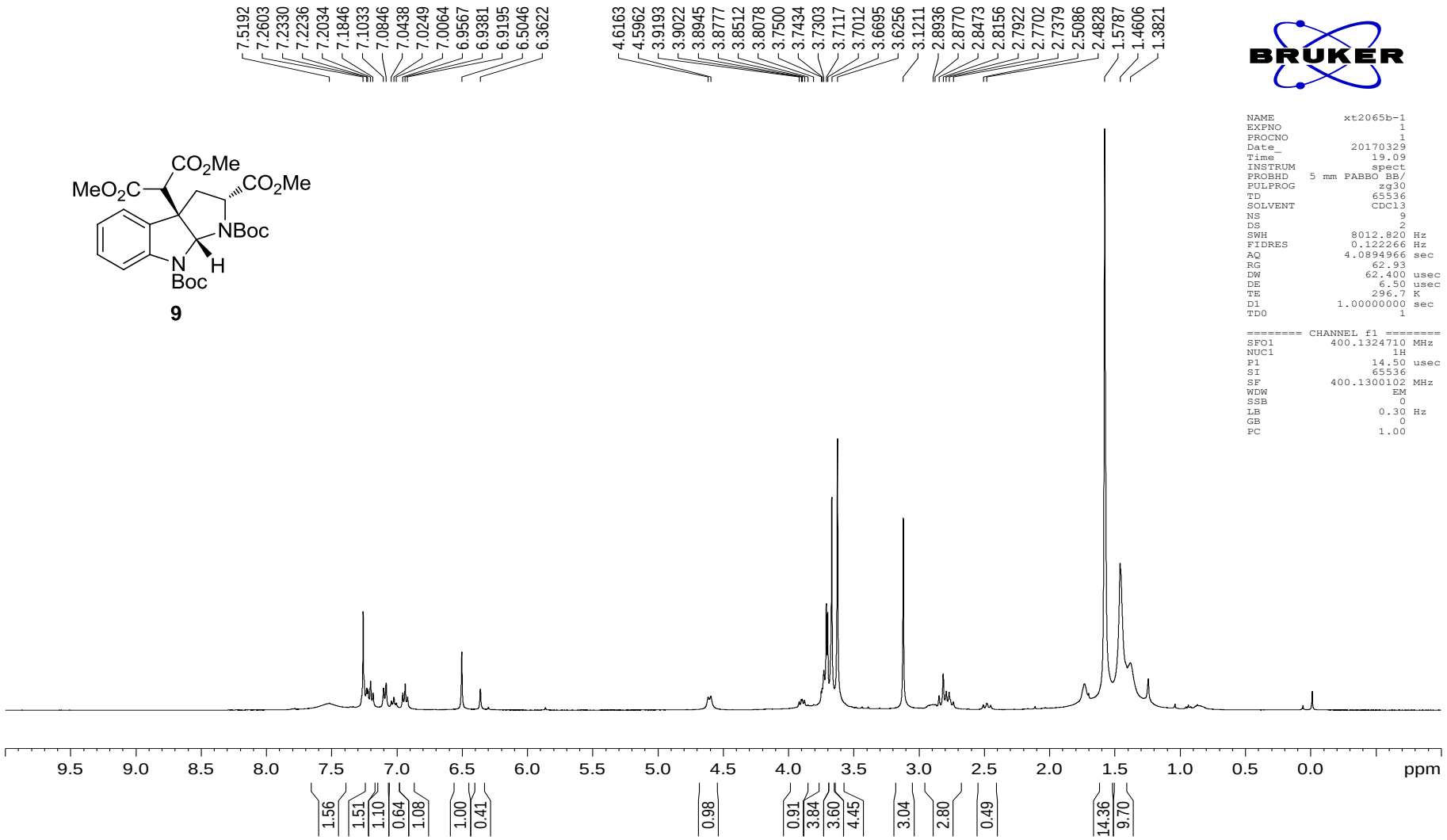
151.61
138.54
137.19
131.98
130.96
124.50
122.96
102.76
83.43
77.48
77.16
76.84
71.46
68.03
42.48
28.14
BRUKER
NAME xt2075-1
EXPNO 2
PROCNO 1
Date_ 20170406
Time 14.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127582 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.7054
7.6859
7.5679
7.5438
7.5276
7.4465
7.4277
7.4089
7.3516
7.3338
7.3152
7.2497
6.4238
3.8297
3.8046
3.7840
3.7479
2.9673
2.9549
2.9382
2.9257
2.9033
2.8768
2.8647
2.8057
2.7893
2.7790
2.7589
2.7288
1.6017
Br
Ph
NCO2Me
N
H
Boc
7
NAME xt2025
EXPNO 1
PROCNO 1
Date_ 20170224
Time_ 13.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 112.31
DW 62.400 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300147 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
4.02
2.03
1.08
0.87
1.17
2.97
2.05
1.11
9.53


154.86
152.21
141.43
140.37
137.75
133.10
129.65
128.98
127.45
127.00
122.42
117.64
84.50
82.41
77.48
77.16
76.84
62.23
52.91
46.44
41.43
28.40
Br
Ph
NCO2Me
N
H
Boc
7
BRUKER
NAME xt2025-3
EXPNO 2
PROCNO 1
Date_ 20170503
Time 22.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 226
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.7742
7.2598
7.1907
7.1720
7.1530
6.9525
6.9349
6.9086
6.8906
5.8397
3.6051
2.8038
2.7863
2.2238
2.2065
1.5756
1.4690
CO2Me
NBoc
N
H
Boc
8
NAME xt2060-4
EXPNO 1
PROCNO 1
Date_ 20170323
Time_ 23.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 54.81
DW 62.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.78
1.03
2.16
0.98
3.05
1.08
1.03
9.61
9.61


166.97
156.35
152.23
147.79
129.05
122.65
122.46
121.71
115.10
82.63
81.30
79.80
77.48
77.36
77.16
76.84
56.48
52.33
39.98
28.50
28.24
25.65
BRUKER
NAME xt2060-4
EXPNO 2
PROCNO 1
Date_ 20170323
Time 23.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 70
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
CO2Me
NBoc
N
H
Boc
8
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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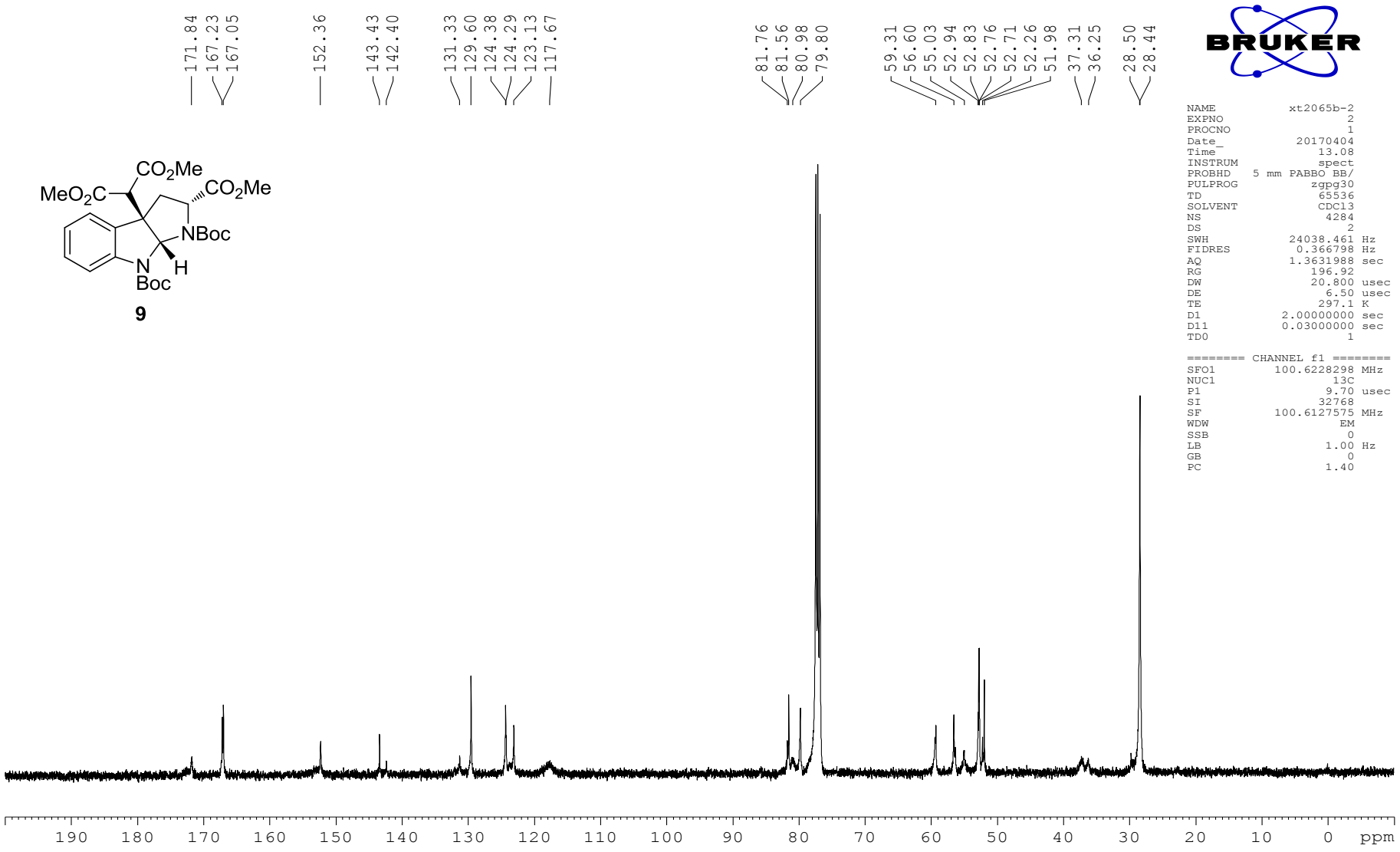
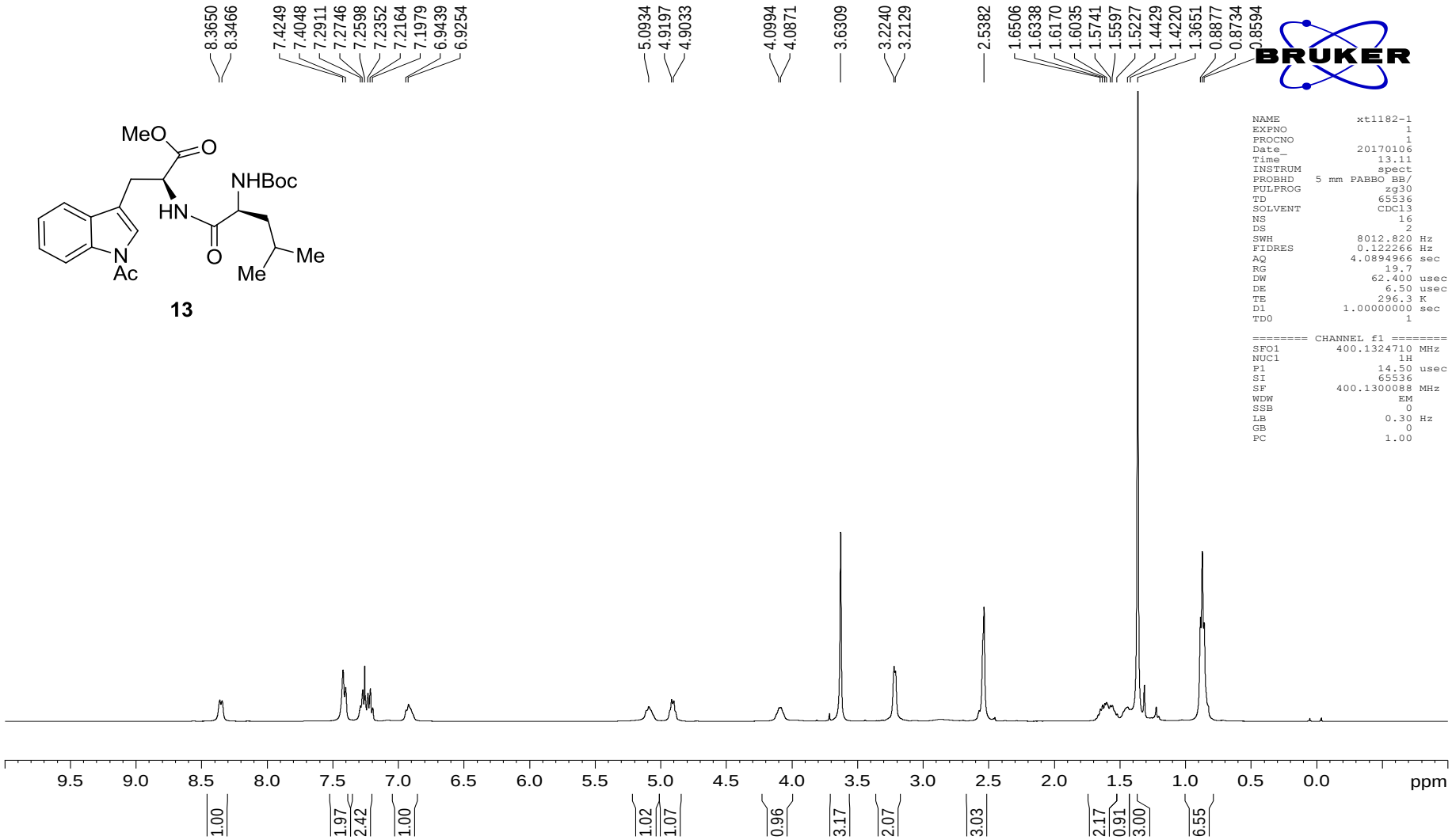
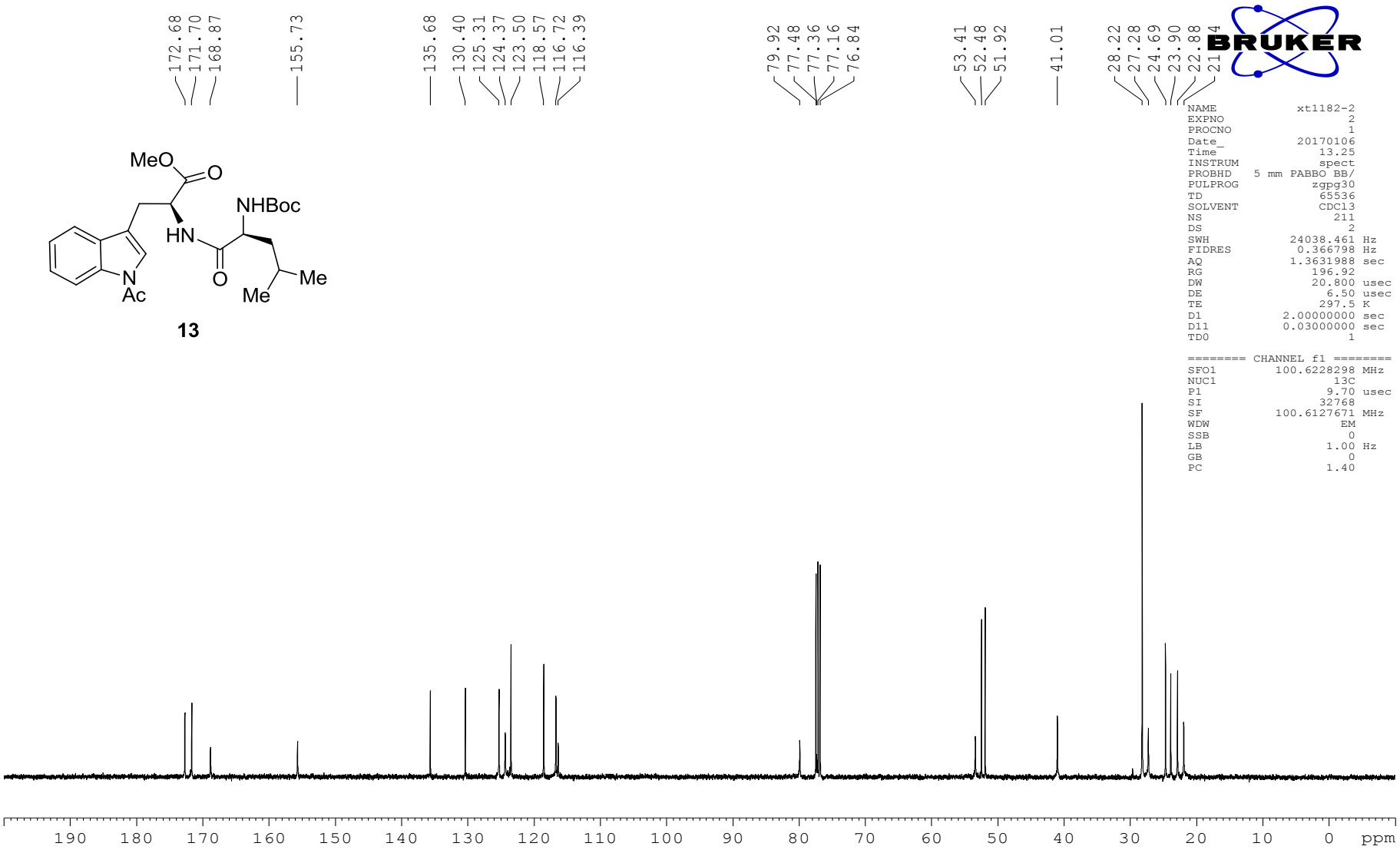
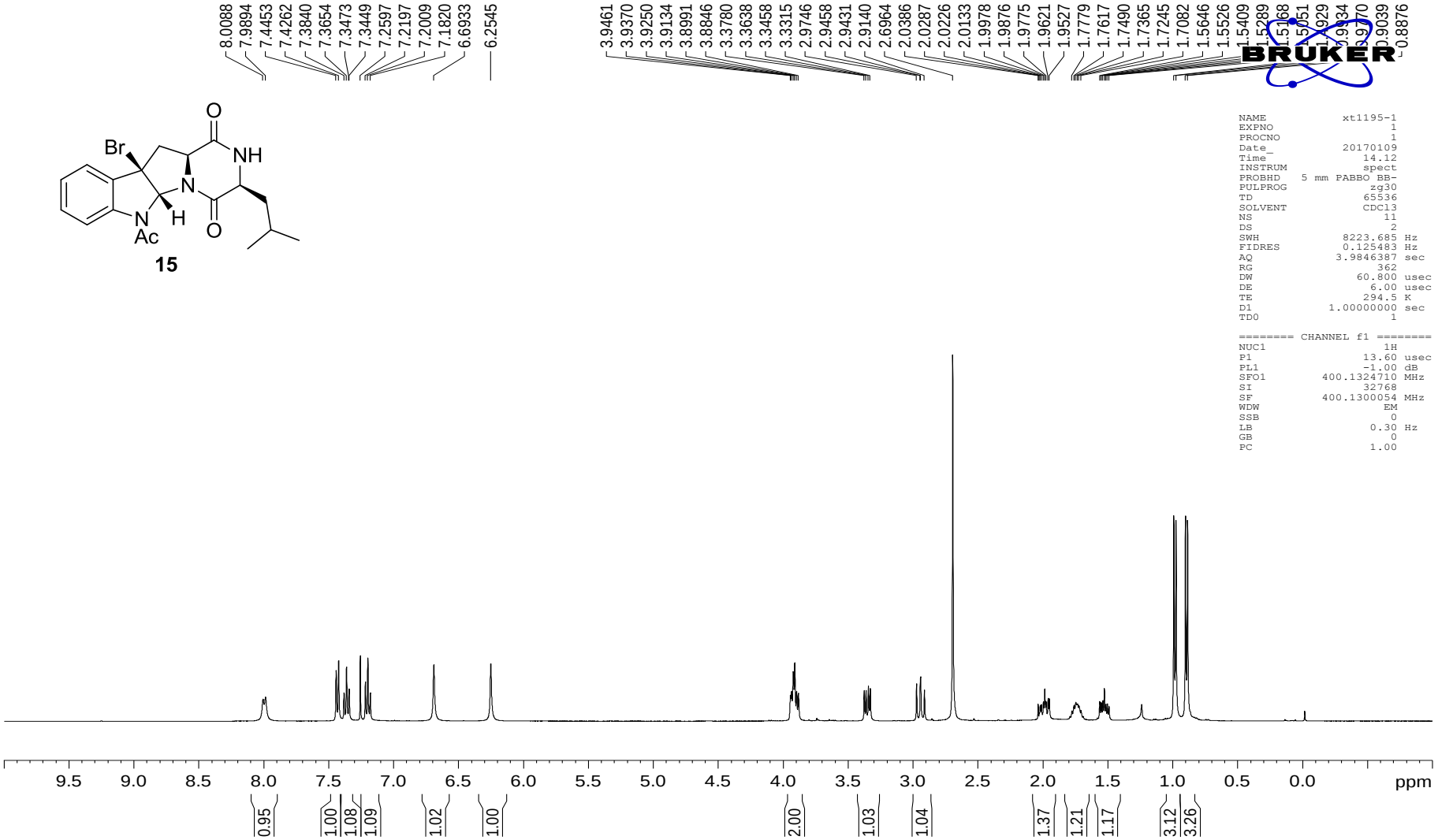
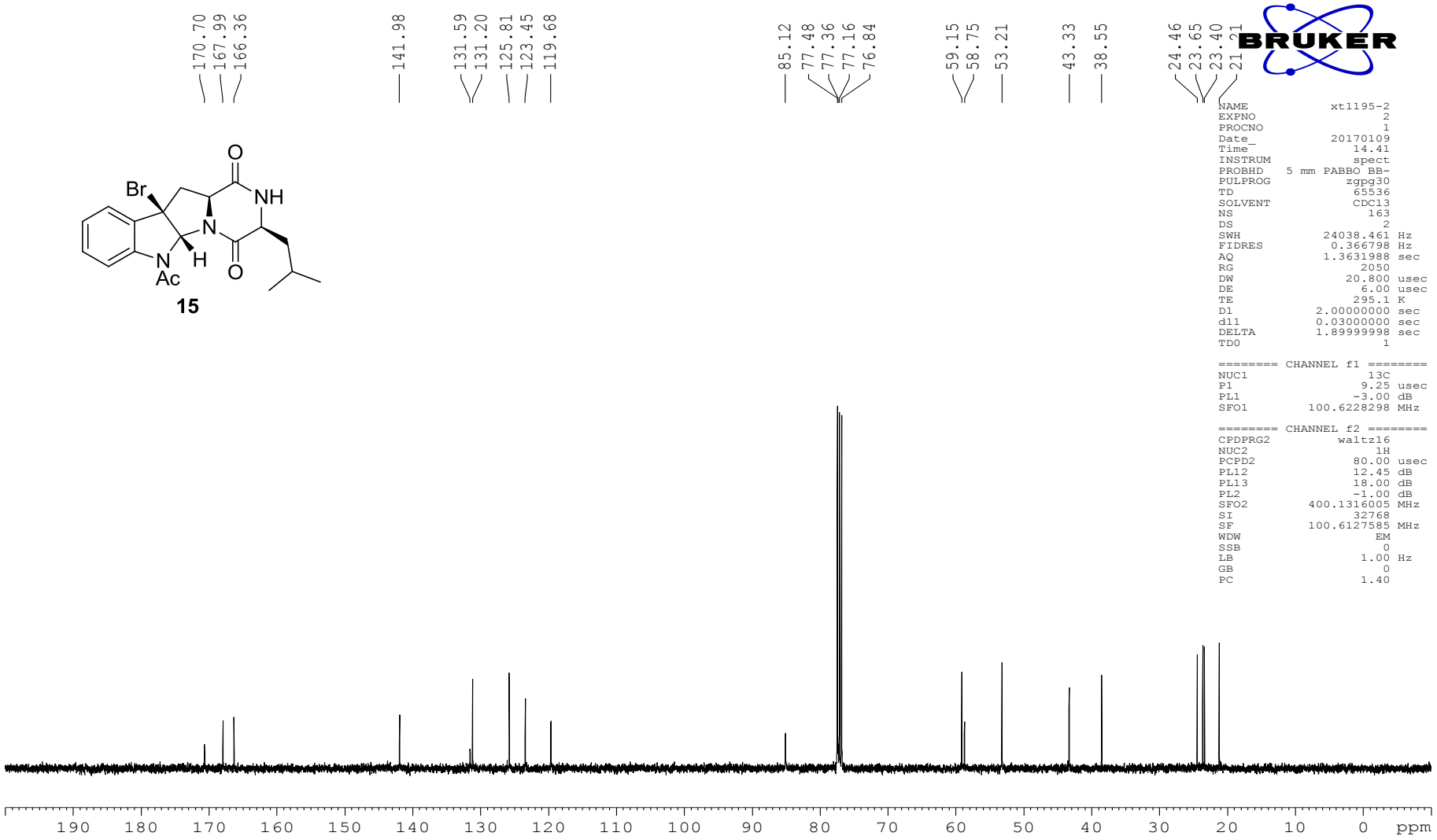
7.2603
7.2330
7.2236
7.2034
7.1846
7.1033
7.0846
7.0438
7.0249
7.0064
6.9567
6.9381
6.9195
6.5046
6.3622
4.6163
4.5962
3.9193
3.9022
3.8945
3.8777
3.8512
3.8078
3.7500
3.7434
3.7303
3.7117
3.7012
3.6695
3.6256
3.1211
2.8936
2.8770
2.8473
2.8156
2.7922
2.7702
2.7379
2.5086
2.4828
1.5787
1.4606
1.3821
BRUKER
NAME xt2065b-1
EXPNO 1
PROCNO 1
Date_ 20170329
Time 19.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 62.93
DW 62.400 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
CO2Me
MeO2C
CO2Me
NBoc
N
H
Boc
9
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.56 1.51 1.10 0.64 1.08 1.00 0.41 0.98 0.91 3.84 3.60 4.45 3.04 2.80 0.49 14.36 9.70

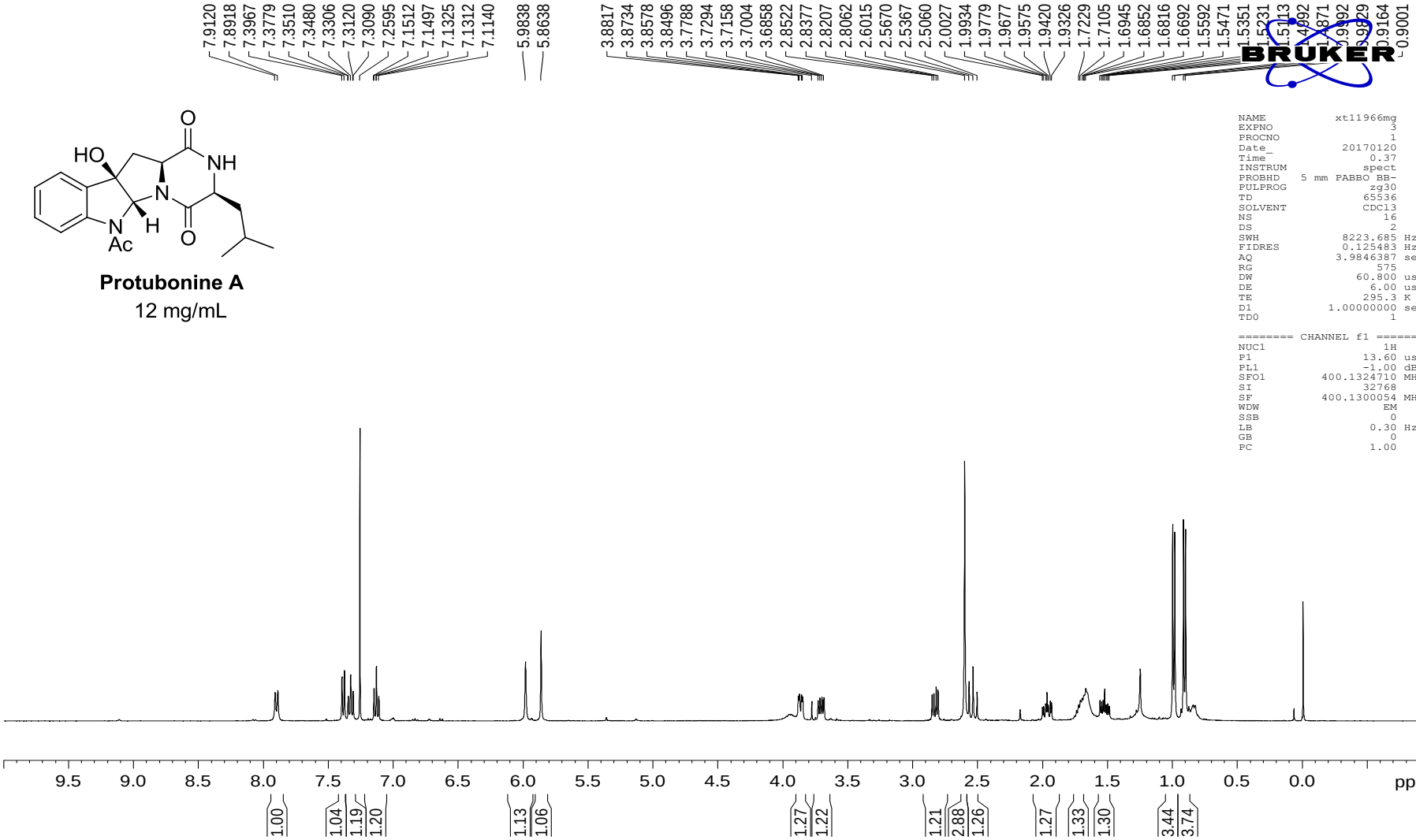

171.84
167.23
167.05
152.36
143.43
142.40
131.33
129.60
124.38
124.29
123.13
117.67
81.76
81.56
80.98
79.80
59.31
56.60
55.03
52.94
52.83
52.76
52.71
52.26
51.98
37.31
36.25
28.50
28.44
BRUKER
NAME xt2065b-2
EXPNO 2
PROCNO 1
Date_ 20170404
Time 13.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4284
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO2C
CO2Me
CO2Me
NBoc
N
Boc
H
9
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

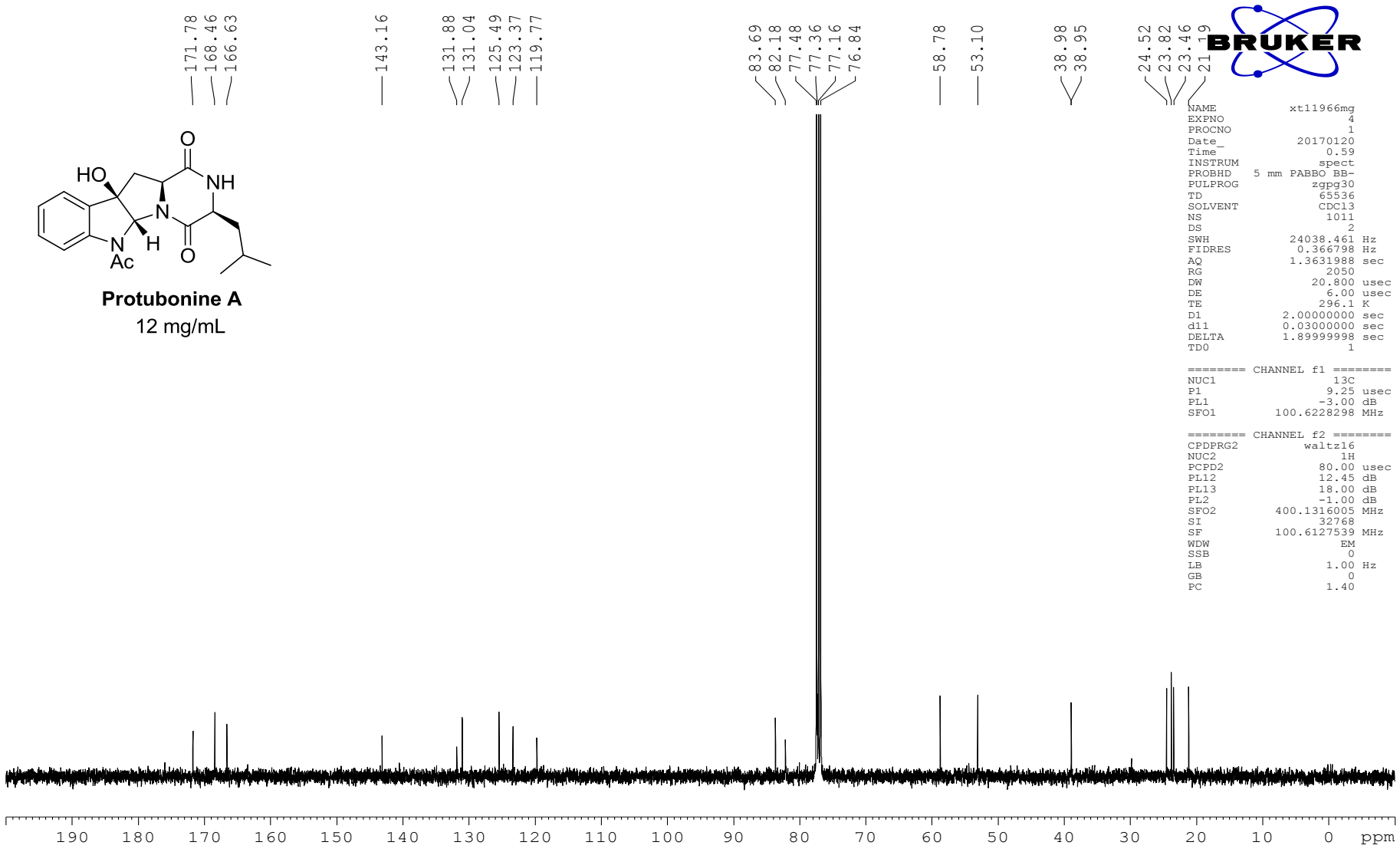

8.3650
8.3466
7.4249
7.4048
7.2911
7.2746
7.2598
7.2352
7.2164
7.1979
6.9439
6.9254
5.0934
4.9197
4.9033
4.0994
4.0871
3.6309
3.2240
3.2129
2.5382
1.6506
1.6338
1.6170
1.6035
1.5741
1.5597
1.5227
1.4429
1.4220
1.3651
0.8877
0.8734
0.8594
BRUKER
MeO
O
NHBoc
HN
O
Me
Me
N
Ac
13
NAME xt1182-1
EXPNO 1
PROCNO 1
Date_ 20170106
Time 13.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 19.7
DW 62.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.97
2.42
1.00
1.02
1.07
0.96
3.17
2.07
3.03
2.17
0.91
3.00
6.55

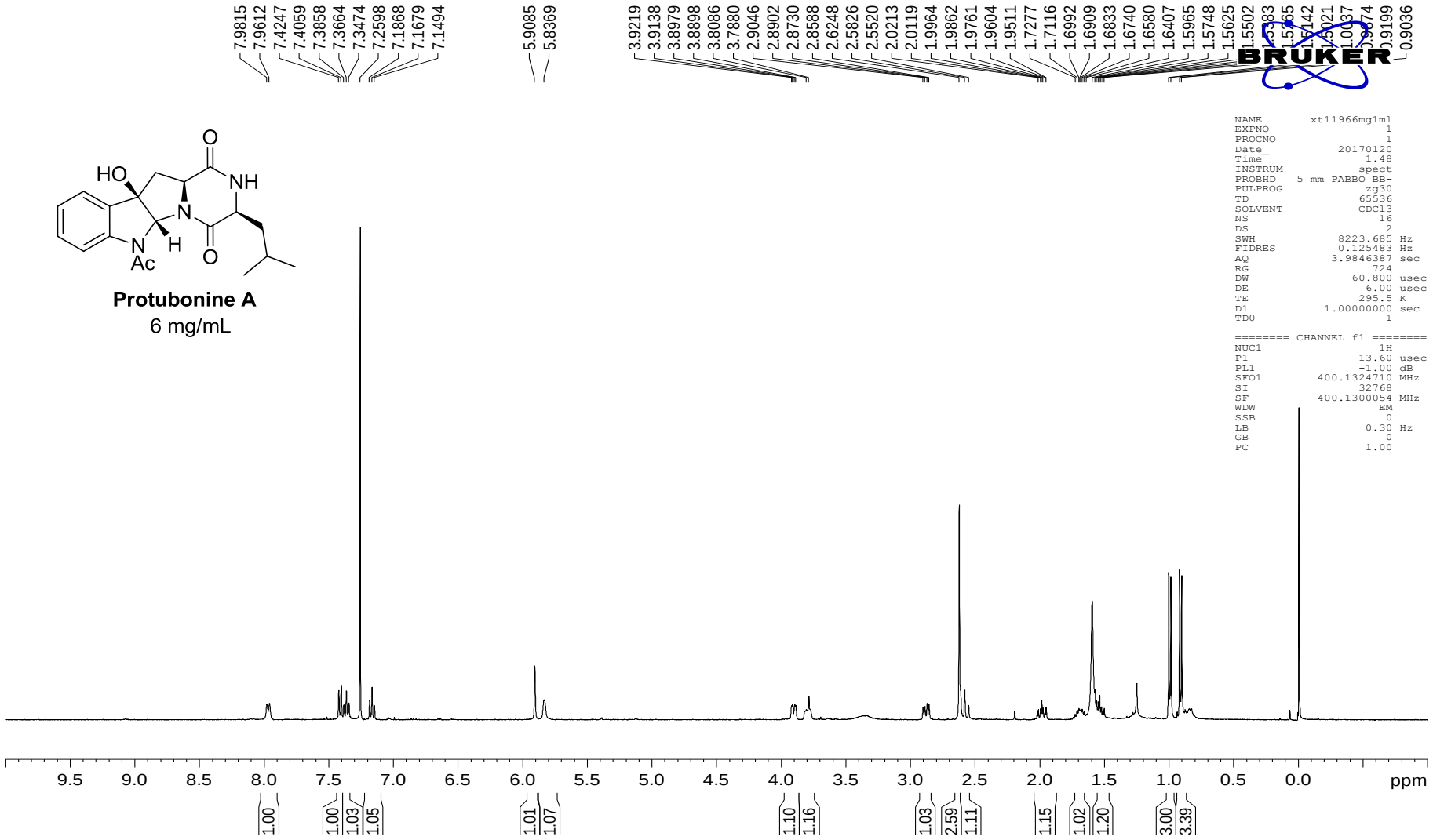

172.68
171.70
168.87
155.73
135.68
130.40
125.31
124.37
123.50
118.57
116.72
116.39
79.92
77.48
77.36
77.16
76.84
53.41
52.48
51.92
41.01
28.22
27.28
24.69
23.90
22.88
21.14
BRUKER
MeO
O
NHBoc
HN
O
N
Ac
Me
Me
13
NAME xt1182-2
EXPNO 2
PROCNO 1
Date_ 20170106
Time 13.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 211
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127671 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

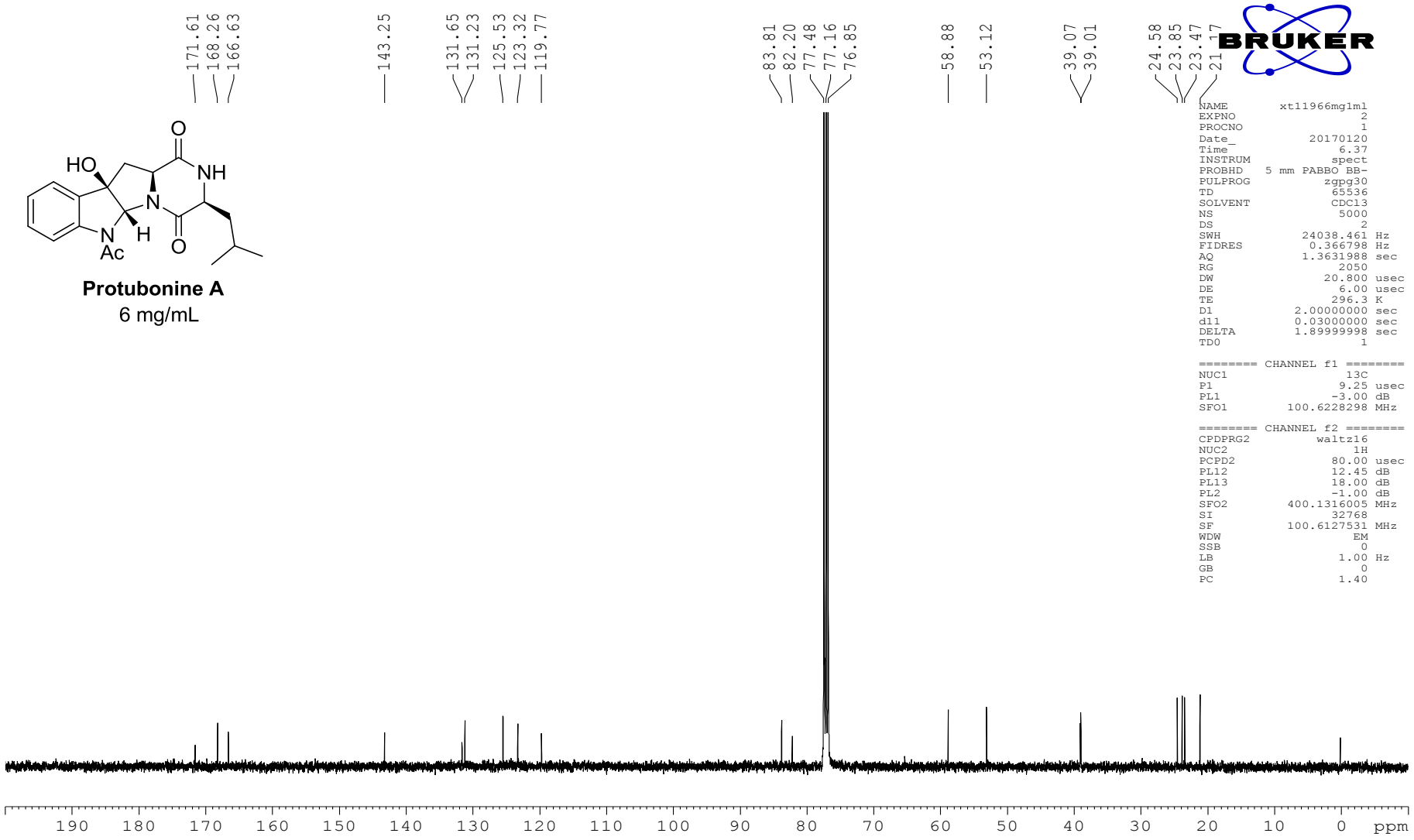

8.0088
7.9894
7.4453
7.4262
7.3840
7.3654
7.3473
7.3449
7.2597
7.2197
7.2009
7.1820
6.6933
6.2545
3.9461
3.9370
3.9250
3.9134
3.8991
3.8846
3.3780
3.3638
3.3458
3.3315
2.9746
2.9458
2.9431
2.9140
2.6964
2.0386
2.0287
2.0226
2.0133
1.9978
1.9876
1.9775
1.9621
1.9527
1.7779
1.7617
1.7490
1.7365
1.7245
1.7082
1.5646
1.5526
1.5409
1.5289
1.5168
1.5051
1.4929
0.9934
0.9770
0.9039
0.8876
BRUKER
Br
O
NH
N
N
H
Ac
O
15
NAME xt1195-1
EXPNO 1
PROCNO 1
Date_ 20170109
Time 14.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 362
DW 60.800 usec
DE 6.00 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
0.95
1.00
1.08
1.09
1.02
1.00
2.00
1.03
1.04
1.37
1.21
1.17
3.12
3.26

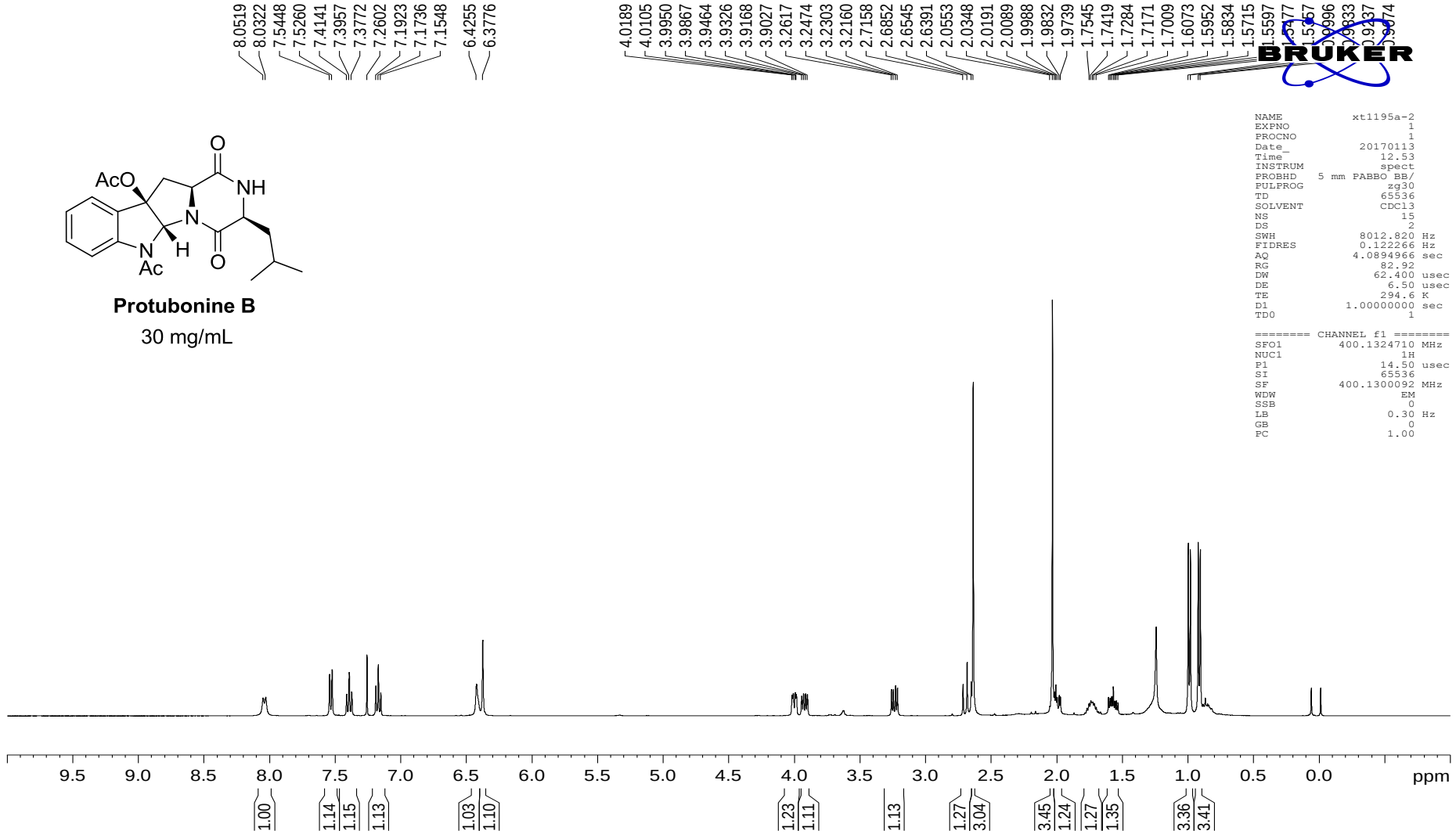

170.70
167.99
166.36
141.98
131.59
131.20
125.81
123.45
119.68
85.12
77.48
77.36
77.16
76.84
59.15
58.75
53.21
43.33
38.55
24.46
23.65
23.40
21.21
BRUKER
Br
O
NH
N
N
H
Ac
O
15
NAME xt1195-2
EXPNO 2
PROCNO 1
Date_ 20170109
Time 14.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 163
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127585 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

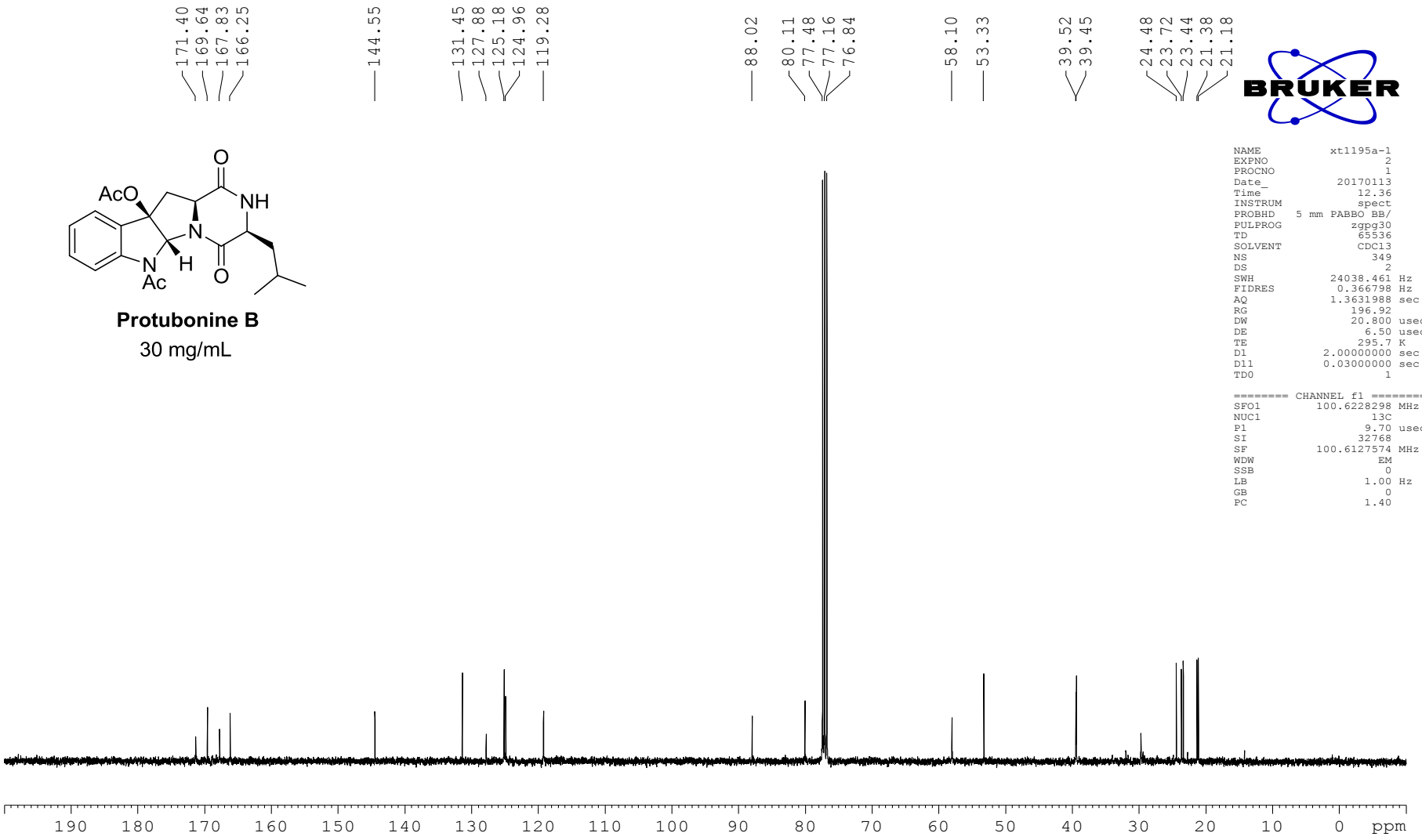

7.9120
7.8918
7.3967
7.3779
7.3510
7.3480
7.3306
7.3120
7.3090
7.2595
7.1512
7.1497
7.1325
7.1312
7.1140
5.9838
5.8638
3.8817
3.8734
3.8578
3.8496
3.7788
3.7294
3.7158
3.7004
3.6858
2.8522
2.8377
2.8207
2.8062
2.6015
2.5670
2.5367
2.5060
2.0027
1.9934
1.9779
1.9677
1.9575
1.9420
1.9326
1.7229
1.7105
1.6945
1.6852
1.6816
1.6692
1.5592
1.5471
1.5351
1.5231
1.5113
1.4992
1.4871
0.9992
0.9164
0.9001
BRUKER
HO
O
NH
N
N
H
Ac
O
Protubonine A
12 mg/mL
NAME xt11966mg
EXPNO 3
PROCNO 1
Date_ 20170120
Time_ 0.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 575
DW 60.800 usec
DE 6.00 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.04
1.19
1.20
1.13
1.06
1.27
1.22
1.21
2.88
1.26
1.27
1.33
1.30
3.44
3.74


171.78
168.46
166.63
143.16
131.88
131.04
125.49
123.37
119.77
83.69
82.18
77.48
77.36
77.16
76.84
58.78
53.10
38.98
38.95
24.52
23.82
23.46
21.19
HO
O
NH
N
H
N
Ac
O
Protubonine A
12 mg/mL
BRUKER
NAME xt11966mg
EXPNO 4
PROCNO 1
Date_ 20170120
Time 0.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1011
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127539 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.9815
7.9612
7.4247
7.4059
7.3858
7.3664
7.3474
7.2598
7.1868
7.1679
7.1494
5.9085
5.8369
3.9219
3.9138
3.8979
3.8898
3.8086
3.7880
2.9046
2.8902
2.8730
2.8588
2.6248
2.5826
2.5520
2.0213
2.0119
1.9964
1.9862
1.9761
1.9604
1.9511
1.7277
1.7116
1.6992
1.6909
1.6833
1.6740
1.6580
1.6407
1.5965
1.5748
1.5625
1.5502
1.5383
1.5265
1.5142
1.5021
1.0037
0.9674
0.9199
0.9036
BRUKER
HO
O
NH
N
N
H
Ac
O
Protubonine A
6 mg/mL
NAME xt11966mg1ml
EXPNO 1
PROCNO 1
Date_ 20170120
Time_ 1.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 724
DW 60.800 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.00
1.03
1.05
1.01
1.07
1.10
1.16
1.03
2.59
1.11
1.15
1.02
1.20
3.00
3.39


171.61
168.26
166.63
143.25
131.65
131.23
125.53
123.32
119.77
83.81
82.20
77.48
77.16
76.85
58.88
53.12
39.07
39.01
24.58
23.85
23.47
21.17
BRUKER
HO
O
NH
N
H
N
Ac
O
Protubonine A
6 mg/mL
NAME xt11966mg1ml
EXPNO 2
PROCNO 1
Date_ 20170120
Time 6.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5000
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.45 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
SI 32768
SF 100.6127531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.0519
8.0322
7.5448
7.5260
7.4141
7.3957
7.3772
7.2602
7.1923
7.1736
7.1548
6.4255
6.3776
4.0189
4.0105
3.9950
3.9867
3.9464
3.9326
3.9168
3.9027
3.2617
3.2474
3.2303
3.2160
2.7158
2.6852
2.6545
2.6391
2.0553
2.0348
2.0191
2.0089
1.9988
1.9832
1.9739
1.7545
1.7419
1.7284
1.7171
1.7009
1.6073
1.5952
1.5834
1.5715
1.5597
BRUKER
AcO
O
NH
N
H
N
Ac
O
Protubonine B
30 mg/mL
NAME xt1195a-2
EXPNO 1
PROCNO 1
Date_ 20170113
Time 12.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 82.92
DW 62.400 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
SI 65536
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.14
1.15
1.13
1.03
1.10
1.23
1.11
1.13
1.27
3.04
3.45
1.24
1.27
1.35
3.36
3.41


171.40
169.64
167.83
166.25
144.55
131.45
127.88
125.18
124.96
119.28
88.02
80.11
77.48
77.16
76.84
58.10
53.33
39.52
39.45
24.48
23.72
23.44
21.38
21.18
BRUKER
AcO
O
NH
N
N
H
Ac
O
Protubonine B
30 mg/mL
NAME xt1195a-1
EXPNO 2
PROCNO 1
Date_ 20170113
Time 12.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 349
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 196.92
DW 20.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 9.70 usec
SI 32768
SF 100.6127574 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm