

Supporting Information

Efficient Catalytic Synthesis of Condensed Isoxazole Derivatives via Intramolecular Oxidative Cycloaddition of Aldoximes

Irina A. Mironova,¹ Valentine G. Nenajdenko,² Pavel S. Postnikov,¹ Akio Saito,³ Mekhman S. Yusubov,^{*1} and Akira Yoshimura^{*4}

¹ Research School of Chemistry and Applied Biomedical Sciences, Tomsk Polytechnic University, 634050 Tomsk, Russia; irina190793@mail.ru (I.A.M.); pavelpostnikov@gmail.com (P.S.P.)

² Department of Chemistry, M. V. Lomonosov Moscow State University, 119991 Moscow, Russia; nenajdenko@gmail.com

³ Division of Applied Chemistry, Institute of Engineering, Tokyo University of Agriculture and Technology, Tokyo 184-8588, Japan; akio-sai@cc.tuat.ac.jp

⁴ Faculty of Pharmaceutical Sciences, Aomori University, 2-3-1 Kobata, Aomori 030-0943, Japan

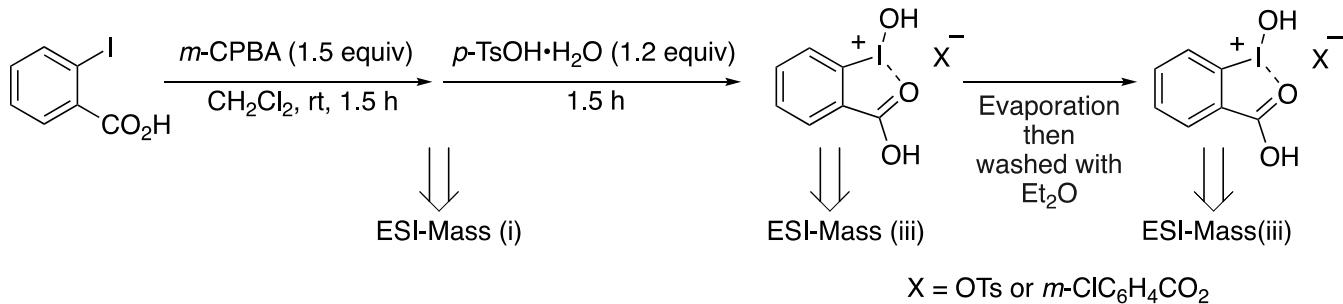
* Correspondence: yusubov@mail.ru (M.S.Y.); ayoshimura@aomori-u.ac.jp (A.Y.)

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1. General experimental remarks

All commercial reagents were ACS grade reagents and used without further purification from freshly opened containers. All solvents were distilled in prior to use. Melting points were determined in an open capillary tube with Buchi M-580 melting point apparatus. Infrared spectra were recorded as ATR on a P Agilent Cary 630 FT-IR spectrophotometer. NMR spectra were recorded on a Bruker BioSpin NMR spectrometer at 400 or 600 MHz (¹H NMR), 101 or 150 MHz (¹³C NMR), 376 MHz (¹⁹F NMR). Chemical shifts are reported in parts per million (ppm). High-resolution mass spectrometric measurements were performed using a Shimadzu LCMS-9030 Q-TOF mass spectrometer, coupled with LC-30 UHPLC system. X-ray crystal analysis was performed by Rigaku XtaLAB Synergy, Single source at home/near, HyPix using CuK α radiation ($\lambda = 1.54184 \text{ \AA}$) at 105 K. Please see the supporting information or the cif file for more detailed crystallography information. (*E*)-2-(Prop-2-yn-1-yloxy)benzaldehyde O-methyl oxime **5** was prepared according to the reported procedure.¹

2. ESI-Mass Study



Scheme S1. ESI-Mass study of the generation of active species for intramolecular oxidative cycloaddition of aldoximes

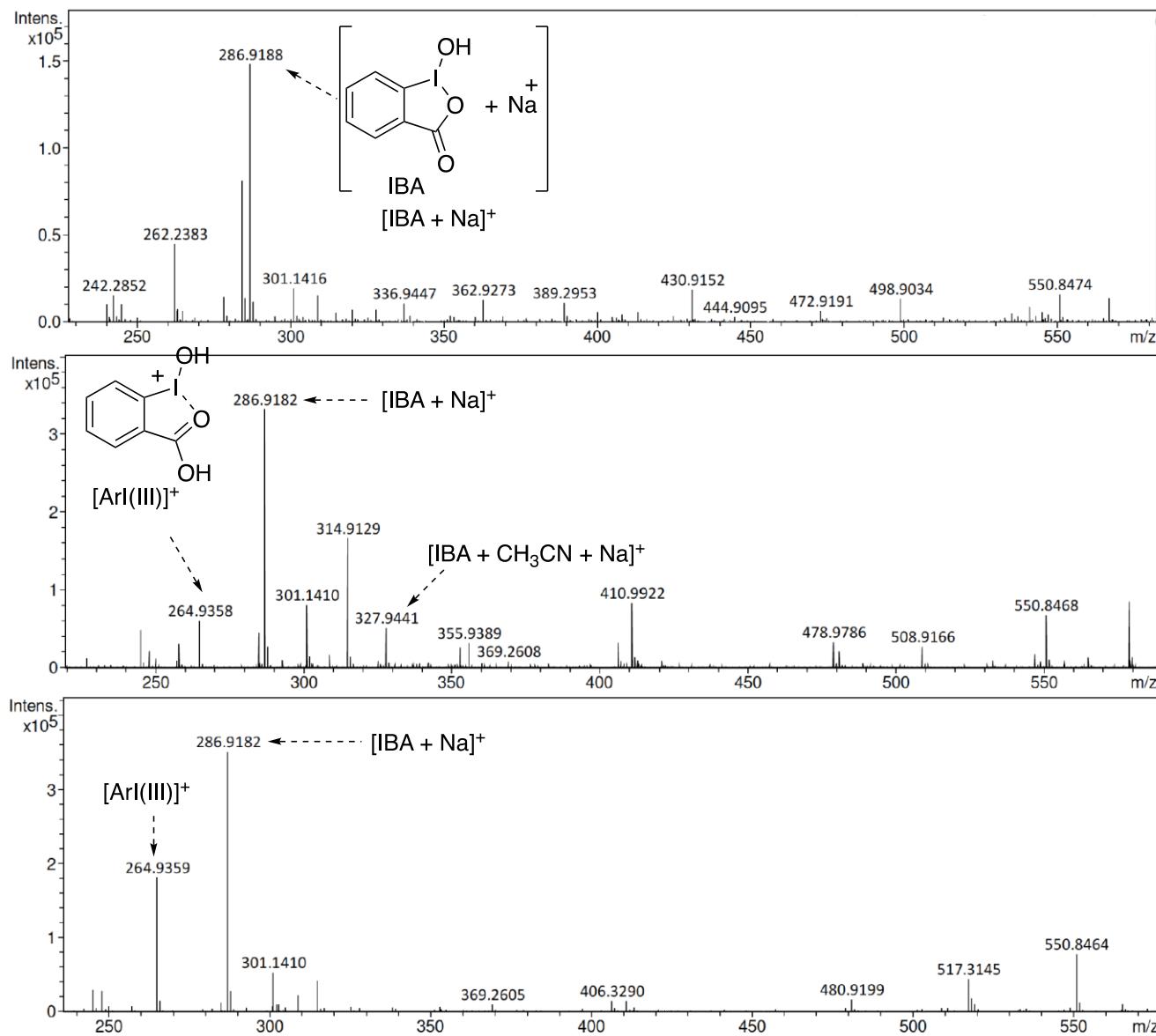


Figure S1. ESI-Mass study of the generation of active species for intramolecular oxidative cycloaddition of aldoximes

3. ^1H NMR Spectroscopy Study

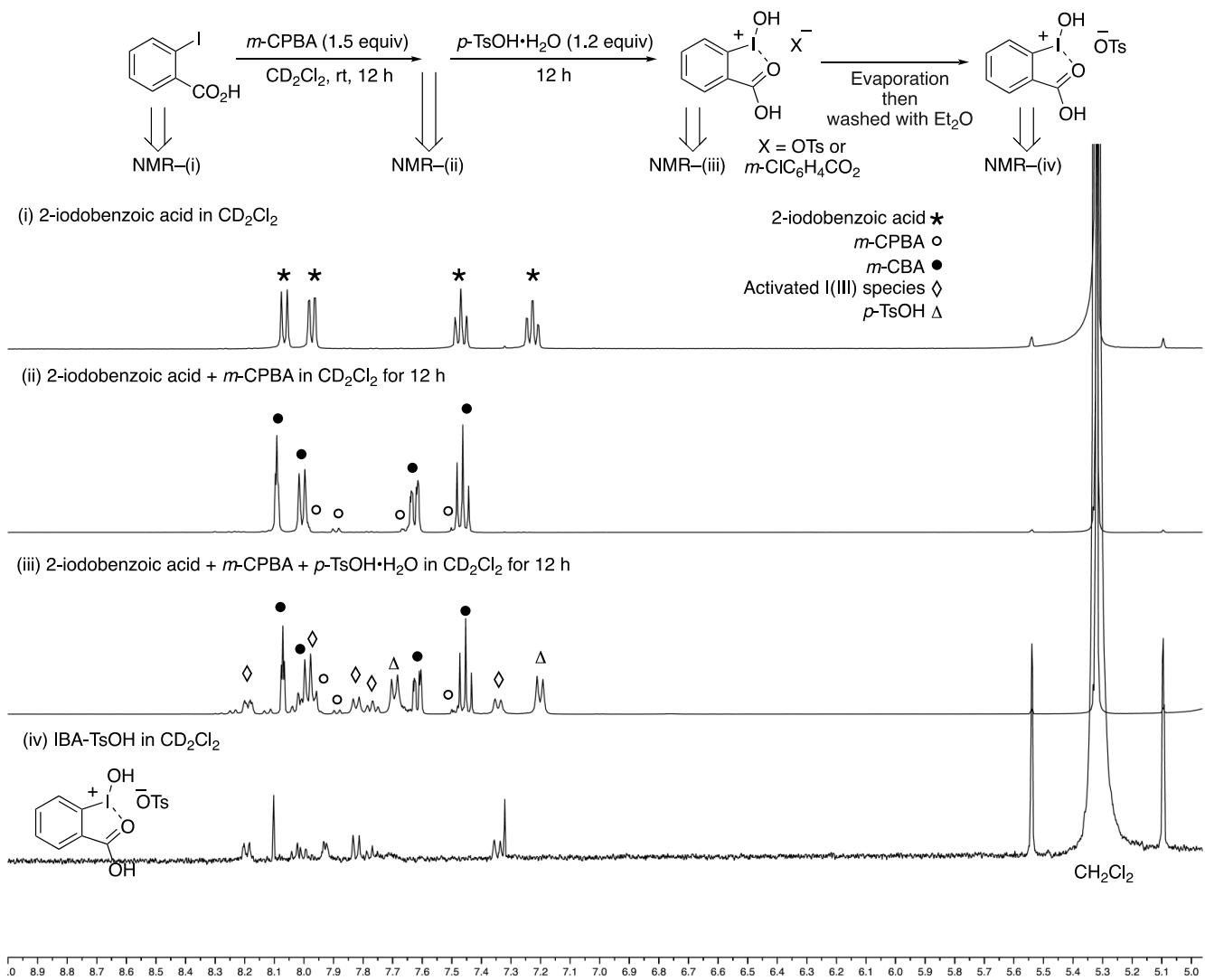


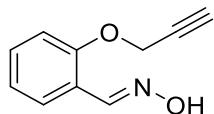
Figure S2. ^1H NMR spectroscopy study of the generation of active species for intramolecular oxidative cycloaddition of aldoximes 1 from 2-hydroxybenzaldehydes

4. General procedure for the synthesis of aldoximes 1 from 2-hydroxybenzaldehydes

In a 50 mL round-bottom flask, dissolve 2-hydroxybenzaldehydes (2.0 mmol) in acetonitrile (20 mL). To this solution is added propargyl bromide (2.4 mmol, 286 mg) or allyl bromide (2.4 mmol, 290 mg), followed by anhydrous K_2CO_3 (4.0 mmol, 553 mg) and the reaction mixture was stirred under reflux for 8 h. The mixture was cooled to ambient temperature and water (20 ml) was added to the reaction mixture. The resulted mixture was extracted by dichloromethane, dried over MgSO_4 and evaporated under reduced pressure. Then, the crude 2-alkoxybenzaldehyde was dissolved in a mixed solution ($\text{MeOH-H}_2\text{O} = 3.5 : 1$ v/v)

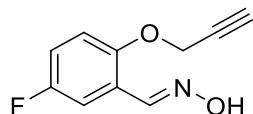
followed by hydroxylamine hydrochloride (2.8 mmol, 195 mg) and AcONa (4.0 mmol, 328 mg) were added. The mixture was stirred at room temperature for 24 h. After completion of the reaction, water (10 mL) was added, and the mixture was extracted with dichloromethane. The organic layer was dried with MgSO₄ and concentrated under reduced pressure. The residue was purified by column chromatography (dichloromethane-hexane = 1 : 1) to afford the pure products **1**.

(E)-2-(Prop-2-yn-1-yloxy)benzaldehyde oxime (1a)²



Reaction of 2-hydroxybenzaldehyde (2.0 mmol, 244 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 284 mg (overall yield in 2 steps 81%) of product **1a**, isolated as colorless solid: mp 86.0 °C (lit.², mp 86 °C); IR (ATR) cm⁻¹: 3256, 3147, 3017, 2935, 2113, 1600, 1491, 1459, 1449, 1221, 1026, 747 657; ¹H NMR (400 MHz, CD₃OD): δ 8.37 (s, 1H), 7.71 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.34-7.27 (m, 1H), 7.08 (d, *J* = 8.4 Hz, 1H), 6.98-6.90 (m, 1H), 4.77 (d, *J* = 2.4 Hz, 2H), 2.95 (t, *J* = 2.4 Hz, 1H); ¹³C NMR (101 MHz, CD₃OD): δ 156.8, 145.8, 131.7, 127.1, 123.2, 122.5, 114.2, 79.5, 77.1, 57.2.

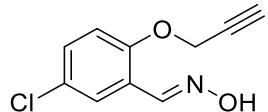
(E)-5-Fluoro-2-(prop-2-yn-1-yloxy)benzaldehyde oxime (1b)



Reaction of 5-fluoro-2-hydroxybenzaldehyde (2.0 mmol, 280 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general afforded procedure 355 mg (overall yield in 2 steps 92%) of product **1b**, isolated as colorless solid: mp 106.7-107.8 °C; IR (ATR) cm⁻¹: 3303, 3147, 3022, 2933, 2124, 1590, 1491, 1432, 1260, 1182, 1017, 799, 660.; ¹H NMR (400 MHz, CDCl₃): δ 8.47 (s, 1H), 7.46 (dd, *J* = 9.0, 2.6 Hz, 1H), 7.11-6.95 (m, 2H), 4.73 (d, *J* = 1.6 Hz, 2H), 2.56-2.50 (m, 1H); ¹³C NMR (101 MHz, CDCl₃): δ 157.6 (d, ¹J_{CF} = 241.4 Hz), 151.9 (d, ⁴J_{CF} = 2.0 Hz), 145.7 (d, ⁴J_{CF} = 1.0 Hz), 123.0 (d, ³J_{CF} = 8.1 Hz), 117.6 (d, ²J_{CF} = 23.7 Hz), 114.7

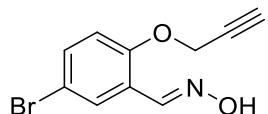
(d, $^3J_{CF} = 8.2$ Hz), 113.1 (d, $^2J_{CF} = 24.7$ Hz), 78.0, 76.4, 57.3.: ^{19}F NMR (376 MHz, CDCl_3): δ -121.5; HRMS (ESI-positive mode): calcd for $\text{C}_{10}\text{H}_9\text{FNO}_2$ ([M+H] $^+$): 194.0612, found: 194.0614.

(E)-5-Chloro-2-(prop-2-yn-1-yloxy)benzaldehyde oxime (1c)³



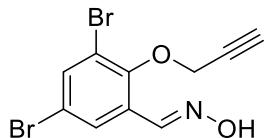
Reaction of 5-chloro-2-hydroxybenzaldehyde (2.0 mmol, 313 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 306 mg (overall yield in 2 steps 73%) of product **1c**, isolated as colorless solid: mp 120.0-121.0 °C; IR (ATR) cm^{-1} : 3248, 3191, 2983, 2118, 1594, 1489, 1453, 1227, 1106, 1005, 740, 684; ^1H NMR (400 MHz, CDCl_3): δ 8.43 (s, 1H), 7.71 (d, $J = 2.4$ Hz, 1H), 7.31 (dd, $J = 8.8, 2.4$ Hz, 1H), 6.98 (d, $J = 8.8$ Hz, 1H), 4.74 (d, $J = 2.4$ Hz, 2H), 2.54 (t, $J = 2.4$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ 154.1, 145.5, 130.8, 127.2, 126.7, 122.9, 114.4, 77.8, 76.5, 56.8.; HRMS (ESI-positive mode): calcd for $\text{C}_{10}\text{H}_9^{35}\text{ClNO}_2$ ([M+H] $^+$): 210.0316, found: 210.0319.

(E)-5-Bromo-2-(prop-2-yn-1-yloxy)benzaldehyde oxime (1d)⁴



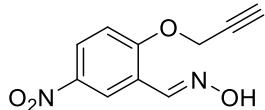
Reaction of 5-bromo-2-hydroxybenzaldehyde (2.0 mmol, 402 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure 295 mg (overall yield in 2 steps 58%) of product **1d**, isolated as colorless solid: mp 127.0-128.0 °C; IR (ATR) cm^{-1} : 3243, 3186, 2979, 2920, 2117, 1590, 1486, 1453, 1225, 1005, 799, 671.; ^1H NMR (400 MHz, CDCl_3): δ 8.43 (s, 1H), 7.87 (d, $J = 2.4$ Hz, 1H), 7.45 (dd, $J = 8.8, 2.4$ Hz, 1H), 6.93 (d, $J = 8.8$ Hz, 1H), 4.74 (d, $J = 2.3$ Hz, 2H), 2.54 (t, $J = 2.3$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ 154.7, 145.4, 133.7, 129.6, 123.3, 114.7, 114.5, 77.7, 76.6, 56.7.; HRMS (ESI-positive mode): calcd for $\text{C}_{10}\text{H}_9^{79}\text{BrNO}_2$ ([M+H] $^+$): 253.9811, found: 253.9812.

(E)-3,5-Dibromo-2-(prop-2-yn-1-yloxy)benzaldehyde oxime (1e)



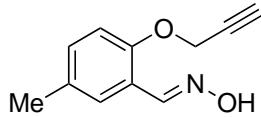
Reaction of 3,5-dibromo-2-hydroxybenzaldehyde (2.0 mmol, 560 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 373 mg (overall yield in 2 steps 56%) of product **1e**, isolated as colorless solid: mp 122.0-123.0 °C; IR (ATR) cm^{-1} : 3490, 3289, 3236, 3067, 3001, 2926, 2119, 1544, 1438, 1221, 1088, 716, 665; ^1H NMR (400 MHz, CDCl_3): δ 8.46 (s, 1H), 7.92 (d, J = 2.4 Hz, 1H), 7.72 (d, J = 2.4 Hz, 1H), 4.74 (d, J = 2.4 Hz, 2H), 2.56 (t, J = 2.4 Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3): δ 152.4, 145.6, 137.0, 130.2, 128.6, 118.7, 118.6, 77.6, 61.7.; HRMS (ESI-positive mode): calcd for $\text{C}_{10}\text{H}_8^{79}\text{Br}_2\text{NO}_2$ ([M+H] $^+$: 331.8916, found: 331.8921.

(E)-5-Nitro-2-(prop-2-yn-1-yloxy)benzaldehyde oxime (1f)



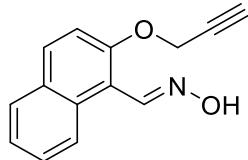
Reaction of 2-hydroxy-5-nitrobenzaldehyde (2.0 mmol, 334 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 366 mg (overall yield in 2 steps 83%) of product **1f**, isolated as yellowish solid: mp 157.6-158.5 °C; IR (ATR) cm^{-1} : 3282, 3193, 2994, 2949, 2123, 1582, 1495, 1338, 1232, 1006, 839, 746, 682.; ^1H NMR (400 MHz, CD_3OD): δ 8.59 (d, J = 2.8 Hz, 1H), 8.34 (s, 1H), 8.23 (dd, J = 9.2, 2.8 Hz, 1H), 7.29 (d, J = 9.2 Hz, 1H), 4.97 (d, J = 2.3 Hz, 2H), 3.10 (t, J = 2.3 Hz, 1H); ^{13}C NMR: (101 MHz, CD_3OD): δ 160.8, 143.5, 143.3, 126.7, 124.2, 122.3, 114.2, 78.3, 78.3, 57.9.; HRMS (ESI-positive mode): calcd for $\text{C}_{10}\text{H}_9\text{N}_2\text{O}_4$ ([M+H] $^+$: 221.0557, found: 221.0558.

(E)-5-Methyl-2-(prop-2-yn-1-yloxy)benzaldehyde oxime (1g)⁵



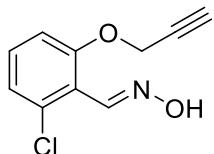
Reaction of 2-hydroxy-5-methylbenzaldehyde (2.0 mmol, 272 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 352 mg (overall yield in 2 steps 93%) of product **1g**, isolated as colorless solid: mp 102.8-103.9 °C (lit.^[5], mp 107-108 °C); IR (ATR) cm⁻¹: 3262, 3195, 2975, 2924, 2118, 1580, 1500, 1457, 1218, 1006, 926, 797, 697.; ¹H NMR (400 MHz, CDCl₃): δ 8.49 (s, 1H), 7.52 (d, *J* = 2.1 Hz, 1H), 7.17 (dd, *J* = 8.7, 2.1 Hz, 1H), 6.95 (d, *J* = 8.7 Hz, 1H), 4.73 (d, *J* = 2.4 Hz, 2H), 2.52 (t, *J* = 2.4 Hz, 1H), 2.30 (s, 3H).; ¹³C NMR (101 MHz, CDCl₃): δ 153.7, 146.7, 131.8, 131.3, 127.5, 121.0, 113.1, 78.4, 76.0, 56.6, 20.6.

(E)-2-(Prop-2-yn-1-yloxy)-1-naphthaldehyde oxime (1h)⁶



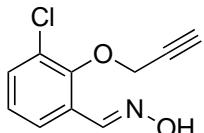
Reaction of 2-hydroxy-1-naphthaldehyde (2.0 mmol, 344 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 342 mg (overall yield in 2 steps 76%) of product **1h**, isolated as colorless solid: mp 127.5-128.5 °C (lit.^[6], mp 133-134 °C); IR (ATR) cm⁻¹: 3242, 3181, 2934, 2117, 1592, 1514, 1457, 1265, 1213, 1092, 1023, 749, 687.; ¹H NMR (400 MHz, CD₃OD): δ 8.87 (d, *J* = 8.6 Hz, 1H), 8.75 (s, 1H), 7.89 (d, *J* = 9.2 Hz, 1H), 7.81 (d, *J* = 8.4 Hz, 1H), 7.51-7.46 (m, 1H), 7.45 (d, *J* = 8.4 Hz, 1H), 7.41-7.35 (m, 1H), 4.93 (d, *J* = 2.4 Hz, 3H), 2.99 (t, *J* = 2.4 Hz, 1H).; ¹³C NMR (101 MHz, CD₃OD): δ 156.2, 147.5, 132.8, 132.5, 131.2, 129.3, 128.5, 127.2, 125.4, 116.7, 115.7, 79.6, 77.3, 58.2.

(E)-2-Chloro-6-(prop-2-yn-1-yloxy)benzaldehyde oxime (1i)



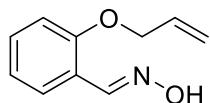
Reaction of 2-chloro-6-hydroxybenzaldehyde (2.0 mmol, 313 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 268 mg (overall yield in 2 steps 64%) of product **1i**, isolated as colorless solid: mp 157.0-158.0 °C (decomp.); IR (ATR) cm⁻¹: 3290, 3103, 3010, 2921, 2854, 2115, 1569, 1449, 1275, 1208, 1025, 767, 666.; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.5 (s, 1H), 8.15 (s, 1H), 7.38 (t, *J* = 8.2 Hz, 1H), 7.18-7.11 (m, 2H), 4.89 (d, *J* = 2.4 Hz, 2H), 3.64 (t, *J* = 2.4 Hz, 1H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 156.6, 142.7, 132.7, 130.5, 122.8, 120.3, 112.1, 78.9, 78.8, 56.5.; HRMS (ESI-positive mode): calcd for C₁₀H₉³⁵ClNO₂ ([M+H])⁺: 210.0316, found: 210.0319.

(E)-3-Chloro-2-(prop-2-yn-1-yloxy)benzaldehyde oxime (1j)



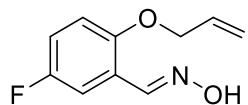
Reaction of 3-chloro-2-hydroxybenzaldehyde (2.0 mmol, 313 mg), propargyl bromide (2.4 mmol, 286 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 277 mg (overall yield in 2 steps 66%) of product **1j**, isolated as colorless solid: mp 98.5-100.3 °C; IR (ATR) cm⁻¹: 3291, 3249, 3075, 3015, 2937, 2868, 2123, 1560, 1438, 1217, 1075, 777, 691.; ¹H NMR (400 MHz, CDCl₃): δ 8.56 (s, 1H), 7.71 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.42 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.11 (t, *J* = 8.0 Hz, 1H), 4.77 (d, *J* = 2.4 Hz, 2H), 2.55 (t, *J* = 2.4 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 152.2, 146.6, 132.0, 128.6, 128.5, 125.9, 125.0, 77.9, 77.1, 61.4.; HRMS (ESI-positive mode): calcd for C₁₀H₉³⁵ClNO₂ ([M+H])⁺: 210.0316, found: 210.0317.

(E)-2-(Allyloxy)benzaldehyde oxime (1k)⁷



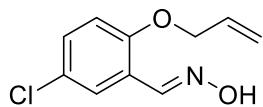
Reaction of 2-hydroxybenzaldehyde (2.0 mmol, 244 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 248 mg (overall yield in 2 steps 70%) of product **1k**, isolated as yellowish oil; IR (ATR) cm⁻¹: 3235, 3078, 3005, 2868, 1599, 1489, 1449, 1245, 994, 947, 751.; ¹H NMR (400 MHz, CDCl₃): δ 8.59 (s, 1H), 7.72 (d, J = 7.7, 1H), 7.37-7.29 (m, 1H), 6.97 (t, J = 7.7 Hz, 1H), 6.90 (d, J = 8.4 Hz, 1H), 6.14-5.98 (m, 1H), 5.42 (dd, J = 17.2, 1.2 Hz, 1H), 5.30 (d, J = 10.8 Hz, 1H), 4.59 (d, J = 5.2 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃): δ 156.7, 146.6, 132.9, 131.3, 127.0, 121.1, 120.8, 117.8, 112.5, 69.2.

(E)-2-(Allyloxy)-5-fluorobenzaldehyde oxime (1l)



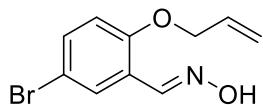
Reaction of 5-fluoro-2-hydroxybenzaldehyde (2.0 mmol, 280 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 355 mg (overall yield in 2 steps 91%) of product **1l**, isolated as colorless oil; IR (ATR) cm⁻¹: 3292, 3080, 3009, 2920, 2871, 1625, 1588, 1490, 1425, 1258, 1187, 1020, 937, 759.; ¹H NMR (400 MHz, CDCl₃): δ 8.64 (s, 1H), 8.51 (d, J = 1.6 Hz, 1H), 7.43 (dd, J = 9.0, 3.0 Hz, 1H), 7.06-6.98 (m, 1H), 6.84 (dd, J = 9.0, 4.2 Hz, 1H), 6.09-5.97 (m, 1H), 5.40 (dq, J = 17.2, 1.6 Hz, 1H), 5.34-5.26 (m, 1H), 4.55 (dt, J = 5.2, 1.6 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃): δ 157.1 (d, ¹J_{CF} = 240.3 Hz), 153.0 (d, ⁴J_{CF} = 2.0 Hz), 145.9 (d, ⁴J_{CF} = 2.4 Hz), 132.8, 122.2 (d, ³J_{CF} = 7.9 Hz), 118.1, 117.6 (d, ²J_{CF} = 23.6 Hz), 114.0 (d, ³J_{CF} = 8.2 Hz), 113.0 (d, ²J_{CF} = 24.6 Hz), 70.1.; ¹⁹F NMR (376 MHz, CDCl₃): δ -122.6. HRMS (ESI-positive mode): calcd for C₁₀H₉³⁵ClNO₂ ([M+H]⁺: 196.0768, found: 196.0772.

(E)-2-(Allyloxy)-5-chlorobenzaldehyde oxime (1m)⁸



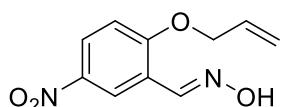
Reaction of 5-chloro-2-hydroxybenzaldehyde (2.0 mmol, 313 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 394 mg (overall yield in 2 steps 93%) of product **1m**, isolated as colorless solid: mp 80.4-80.9 °C (lit.⁸, mp 100.7-101.7 °C); IR (ATR) cm⁻¹: 3400, 3167, 3080, 3003, 2861, 1594, 1477, 1451, 1255, 1129, 1002, 928, 758.; ¹H NMR (400 MHz, CDCl₃): δ 8.48 (s, 1H), 7.70 (d, J = 2.6 Hz, 1H), 7.27 (dd, J = 8.8, 2.6 Hz, 1H), 6.83 (d, J = 8.8 Hz, 1H), 6.13-5.94 (m, 1H), 5.40 (dd, J = 17.2, 1.5 Hz, 1H), 5.31 (dd, J = 10.6, 1.5 Hz, 1H), 4.57 (d, J = 5.2 Hz, 2H).; ¹³C NMR (101 MHz, CDCl₃): δ 155.2, 145.6, 132.5, 130.9, 126.4, 126.3, 122.3, 118.3, 113.9, 69.67.

(E)-2-(Allyloxy)-5-bromobenzaldehyde oxime (1n)⁸



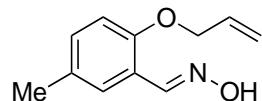
Reaction of 5-bromo-2-hydroxybenzaldehyde (2.0 mmol, 402 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 405 mg (overall yield in 2 steps 79%) of product **1n**, isolated as yellow solid: mp 84.3-85.3 °C (lit.⁸, mp 87.0-88.5 °C); IR (ATR) cm⁻¹: 3242, 3138, 3080, 3019, 2915, 1589, 1484, 1454, 1249, 1018, 948, 796.; ¹H NMR (400 MHz, CDCl₃): δ 8.47 (s, 1H), 7.84 (d, J = 2.4 Hz, 1H), 7.41 (dd, J = 8.8, 2.4 Hz, 1H), 6.78 (d, J = 8.8 Hz, 1H), 6.08-5.96 (m, 1H), 5.40 (dq, J = 17.2, 1.6 Hz, 1H), 5.34-5.28 (m, 1H), 4.56 (dt, J = 5.2, 1.6 Hz, 2H).; ¹³C NMR (101 MHz, CDCl₃): δ 155.7, 145.5, 133.8, 132.5, 129.4, 122.8, 118.3, 114.3, 113.6, 69.6.

(E)-2-(Allyloxy)-5-nitrobenzaldehyde oxime (1o)⁹



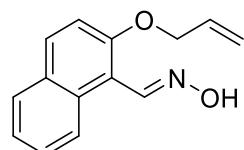
Reaction of 2-hydroxy-5-nitrobenzaldehyde (2.0 mmol, 334 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 258 mg (overall yield in 2 steps 58%) of product **1o**, isolated as colorless solid: mp 89.0-90.0 °C; IR (ATR) cm⁻¹: 3424, 3246, 3090, 3019, 2902, 2860, 1611, 1578, 1510, 1421, 1331, 1265, 1076, 926, 747.; ¹H NMR (400 MHz, CDCl₃): δ 8.65 (d, *J* = 2.8 Hz, 1H), 8.50 (s, 1H), 8.23 (dd, *J* = 9.2, 2.8 Hz, 1H), 6.97 (d, *J* = 9.2 Hz, 1H), 6.14-5.94 (m, 1H), 5.44 (d, *J* = 16.8 Hz, 1H), 5.38 (d, *J* = 10.4 Hz, 1H), 4.70 (d, *J* = 5.2 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃): δ 160.9, 144.8, 141.7, 131.6, 126.7, 122.7, 121.8, 119.2, 112.2, 70.0.

(E)-2-(Allyloxy)-5-methylbenzaldehyde oxime (1p)¹⁰



Reaction of 2-hydroxy-5-methylbenzaldehyde (2.0 mmol, 272 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 325 mg (overall yield in 2 steps 58%) of product **1p**, isolated as colorless solid: mp 67.2-68.2 °C; IR (ATR) cm⁻¹: 3216, 3081, 3019, 2923, 2872, 1610, 1497, 1426, 1250, 1025, 948, 799, 727.; ¹H NMR (400 MHz, CDCl₃): δ 8.54 (s, 1H), 7.53 (s, 1H), 7.14 (d, *J* = 8.2 Hz, 1H), 6.80 (d, *J* = 8.2 Hz, 1H), 6.13-5.96 (m, 1H), 5.41 (dt, *J* = 17.2, 1.8 Hz, 1H), 5.29 (dt, *J* = 10.8, 1.7 Hz, 1H), 4.56 (d, *J* = 4.8 Hz, 2H), 2.29 (s, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 154.8, 146.8, 133.1, 132.0, 130.4, 127.2, 120.5, 117.8, 112.7, 69.5, 20.6.

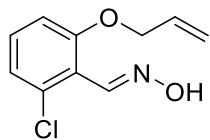
(E)-2-(Allyloxy)-1-naphthaldehyde oxime (1q)⁶



Reaction of 2-hydroxy-1-naphthaldehyde (2.0 mmol, 344 mg), allyl bromide (2.4 mmol, 290 mg) and

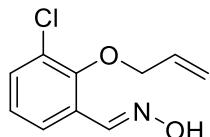
hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 327 mg (overall yield in 2 steps 72%) of product **1q**, isolated as colorless solid: mp 110.3-111.0 °C (lit.⁶, mp 133.0-134.0 °C); IR (ATR) cm⁻¹: 3227, 3077, 2998, 2923, 2869, 1591, 1508, 1427, 1243, 1056, 1002, 946, 751.; ¹H NMR (400 MHz, CDCl₃): δ 8.95 (s, 1H), 8.78 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 8.8 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.64-7.55 (m, 1H), 7.47-7.39 (m, 1H), 7.29 (d, *J* = 8.0 Hz, 1H), 6.21-6.07 (m, 1H), 5.48 (d, *J* = 17.6, 1H), 5.36 (d, *J* = 10.8, 1H), 4.81 (d, *J* = 4.8 Hz, 2H).; ¹³C NMR (101 MHz, CDCl₃): δ 156.3, 147.8, 133.0, 132.4, 131.8, 129.3, 128.5, 128.1, 125.4, 124.4, 118.2, 114.2, 113.7, 70.5.

(E)-2-(Allyloxy)-6-chlorobenzaldehyde (**1r**)



Reaction of 2-chloro-6-hydroxybenzaldehyde (2.0 mmol, 313 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 364 mg (overall yield in 2 steps 86%) of product **1r**, isolated as colorless solid: mp 84.0-85.0 °C; IR (ATR) cm⁻¹: 3178, 2951, 2896, 1587, 1568, 1459, 1419, 1269, 1207, 1021, 970, 932, 770.; ¹H NMR (400 MHz, CDCl₃): δ 8.57 (s, 1H), 7.35-7.23 (m, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 6.15-5.95 (m, 1H), 5.42 (d, *J* = 17.2 Hz, 1H), 5.33 (d, *J* = 10.4 Hz, 1H), 4.81-4.67 (m, 2H).; ¹³C NMR (101 MHz, CDCl₃): δ 158.5, 152.9, 145.1, 135.6, 132.2, 131.8, 122.9, 118.8, 111.5, 70.3; HRMS (ESI-positive mode): calcd for C₁₀H₁₀³⁵ClNO₂ ([M+H]⁺): 212.0472, found: 212.0476.

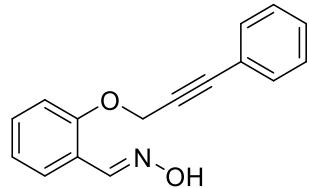
(E)-2-(Allyloxy)-3-chlorobenzaldehyde oxime (**1s**)



Reaction of 3-chloro-2-hydroxybenzaldehyde (2.0 mmol, 313 mg), allyl bromide (2.4 mmol, 290 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 402 mg (overall

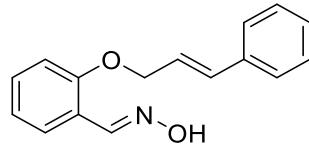
yield in 2 steps 86%) of product **1s**, isolated as colorless solid: mp 75.6-76.6 °C; IR (ATR) cm⁻¹: 3245, 3152, 3069, 3017, 2912, 2868, 1590, 1477, 1436, 1262, 1224, 1015, 964, 740.; ¹H NMR (400 MHz, CDCl₃): δ 8.43 (s, 1H), 7.73-7.67 (m, 1H), 7.43 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.08 (t, *J* = 8.0 Hz, 1H), 6.19-6.01 (m, 1H), 5.44 (dq, *J* = 17.2, 1.2 Hz, 1H), 5.31 (dq, *J* = 10.4, 1.2 Hz, 1H), 4.51 (dt, *J* = 6.0, 1.2 Hz, 2H).; ¹³C NMR (101 MHz, CDCl₃): δ 153.5, 146.2, 132.7, 132.1, 128.7, 127.6, 125.3, 125.1, 119.2, 75.6.; HRMS (ESI-positive mode): calcd for C₁₀H₁₀³⁵ClNO₂ ([M+H])⁺: 212.0472, found: 212.0476.

(E)-2-((3-Phenylprop-2-yn-1-yl)oxy)benzaldehyde oxime (**1t**)



Reaction of 2-hydroxybenzaldehyde (2.0 mmol, 244 mg), (3-chloroprop-1-yn-1-yl)benzene (2.4 mmol, 362 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 402 mg (overall yield in 2 steps 80%) of product **1t**, isolated as pale solid: mp 77.6-78.6 °C; IR (ATR) cm⁻¹: 3239, 3052, 2968, 2879, 2238, 1598, 1489, 1454, 1281, 1222, 1128, 1007, 970, 749, 689.; ¹H NMR (400 MHz, CDCl₃) δ 8.94 (s, 1H), 8.58 (s, 1H), 7.73 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.47-7.42 (m, 2H), 7.42-7.36 (m, 1H), 7.36-7.27 (m, 3H), 7.14 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.03 (m, 1H), 4.98 (s, 2H).; ¹³C NMR (101 MHz, CDCl₃): δ 156.0, 146.7, 131.9, 131.2, 128.9, 128.4, 127.2, 122.2, 121.8, 121.4, 113.2, 87.7, 83.6, 57.3.; HRMS (ESI-positive mode): calcd for C₁₆H₁₃NO₂ ([M+H])⁺: 252.1018, found: 252.1026.

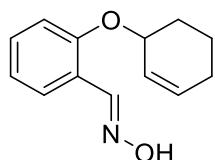
(E)-2-(Cinnamyoxy)benzaldehyde oxime (**1u**)¹¹



Reaction of 2-hydroxybenzaldehyde (2.0 mmol, 244 mg), (*E*)-(3-bromoprop-1-en-1-yl)benzene (2.4 mmol, 473 mg)

and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 441 mg (overall yield in 2 steps 87%) of product **1u**, isolated as colorless solid: mp 129.7-130.7 °C (lit.¹¹ mp 104.0-106.0 °C); IR (ATR) cm⁻¹: 3285, 3076, 3013, 2913, 2861, 1597, 1490, 1446, 1295, 1236, 1107, 1001, 960, 751; ¹H NMR (400 MHz, DMSO-d₆) δ 11.26 (s, 1H), 8.38 (s, 1H), 7.68 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.50 (d, *J* = 7.2 Hz, 2H), 7.40-7.32 (m, 3H), 7.28 (t, *J* = 7.4 Hz, 1H), 7.14 (d, *J* = 8.4 Hz, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 6.77 (d, *J* = 16.0 Hz, 1H), 6.54 (dt, *J* = 16.0, 5.8 Hz, 1H), 4.78 (dd, *J* = 5.8, 1.6 Hz, 2H). ¹³C NMR (101 MHz, DMSO-d₆) δ 155.9, 143.5, 136.1, 132.6, 130.7, 128.7, 128.0, 126.6, 125.5, 124.8, 121.3, 120.9, 113.2, 68.7.

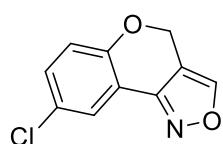
(E)-2-(Cyclohex-2-en-1-yloxy)benzaldehyde oxime (**1v**)

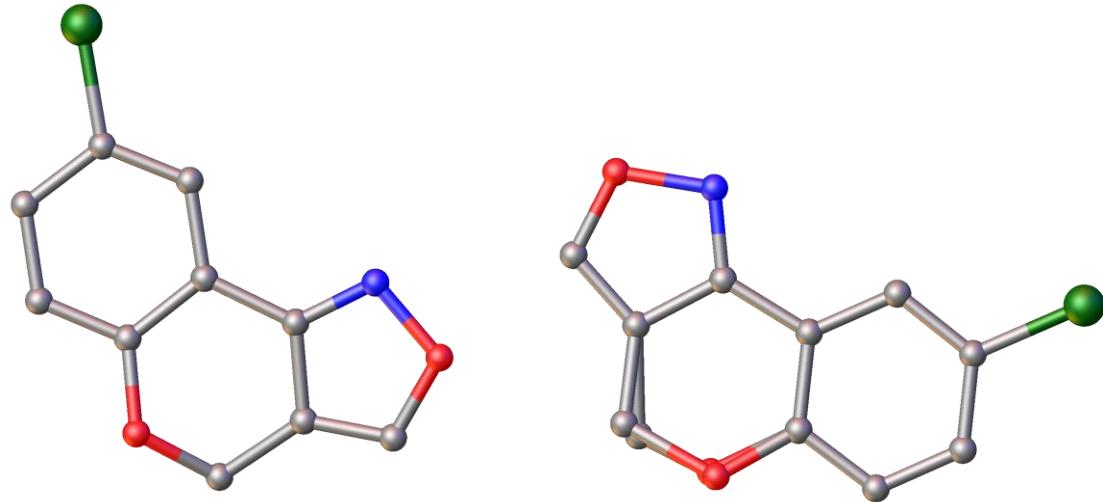


Reaction of 2-hydroxybenzaldehyde (2.0 mmol, 244 mg), 3-bromocyclohex-1-ene (2.4 mmol, 233 mg) and hydroxylamine hydrochloride (2.8 mmol, 195 mg) according to the general procedure afforded 195 mg (overall yield in 2 steps 45%) of product **1v**, isolated as colorless solid: mp 79.7-81.3 °C; IR (ATR) cm⁻¹: 3247, 3148, 3028, 2945, 2921, 2865, 1596, 1577, 1488, 1454, 1439, 1236, 1057, 947, 735; ¹H NMR (400 MHz, CDCl₃): δ 8.57 (s, 1H), 7.75 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.36-7.28 (m, 1H), 6.99-6.92 (m, 2H), 6.02-5.93 (m, 1H), 5.91-5.82 (m, 1H), 4.88-4.77 (m, 1H), 2.21-2.09 (m, 1H), 2.09-1.80 (m, 4H), 1.71-1.60 (m, 1H).; ¹³C NMR (101 MHz, CDCl₃) δ 156.3, 146.9, 132.5, 131.2, 126.7, 126.0, 121.9, 121.0, 114.1, 72.0, 28.6, 25.2, 19.1.; HRMS (ESI-positive mode): calcd for C₁₃H₁₅NO₂ ([M+H]⁺): 218.1175, found: 218.1179.

5. X-Ray single crystal data of compound **3c**

8-Chloro-4*H*-chromeno[4,3-*c*]isoxazole (**3c**)²





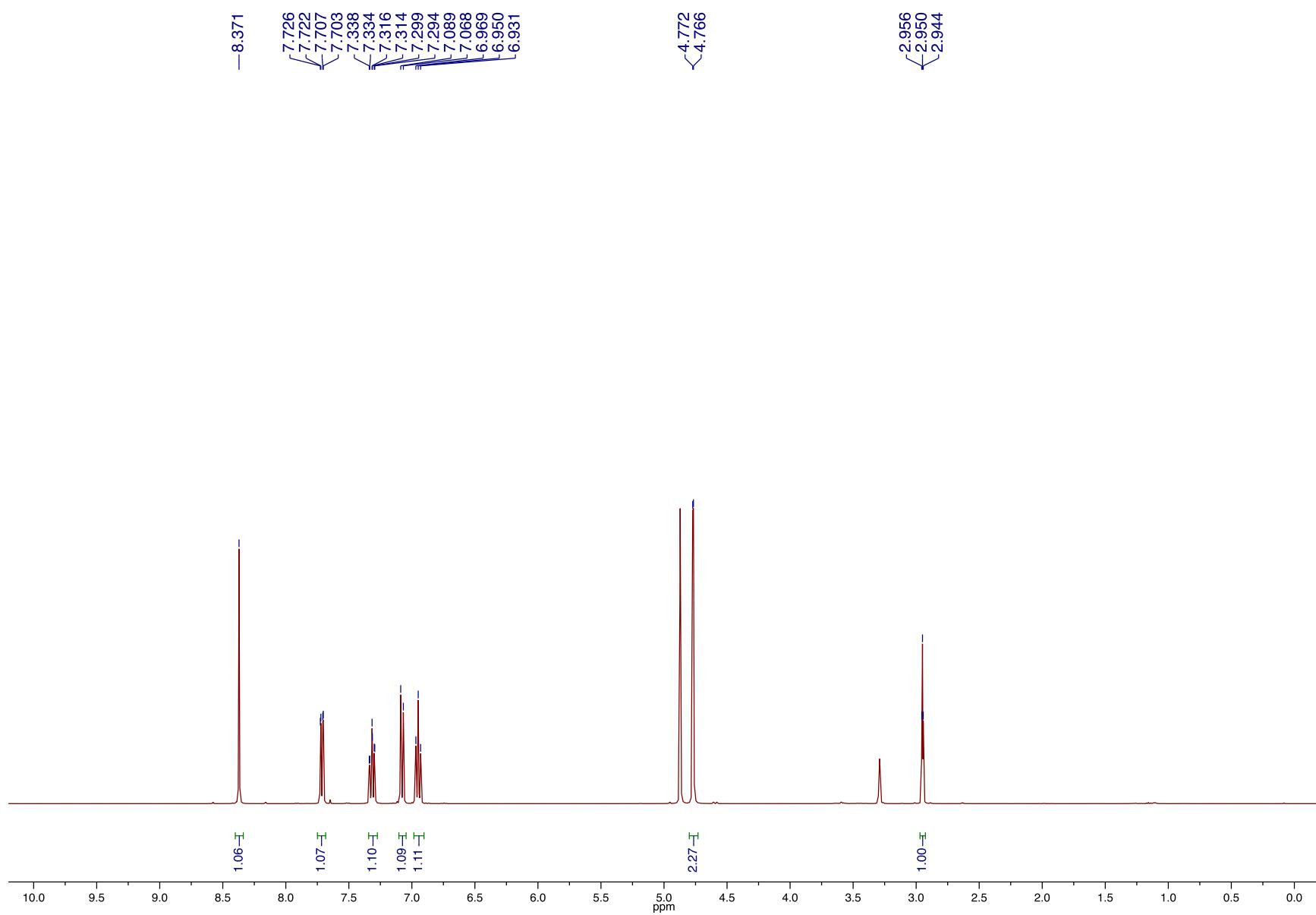
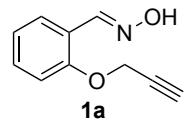
Single crystals of product **3c** suitable for X-ray crystallographic analysis were obtained by slow evaporation of acetonitrile solution. For details on crystal structure of compound **3c** see the CIF file in Supporting Information. Selected crystallographic data for **3c**: size 0.15 mm × 0.13 mm × 0.12 mm, orthorhombic, space group Pca21 (no. 29), $a = 13.4924(6)$ Å, $b = 3.89250(18)$ Å, $c = 32.3452(14)$ Å, $V = 1698.75(13)$ Å³, $Z = 4$, $T = 105(9)$ K, $\mu(\text{Cu K}\alpha) = 3.732$ mm⁻¹, $D_{\text{calc}} = 1.624$ g/cm³, 5818 reflections measured ($5.464^\circ \leq 2\Theta \leq 139.996^\circ$), 2510 unique ($R_{\text{int}} = 0.0587$, $R_{\text{sigma}} = 0.0620$) which were used in all calculations. The final R_1 was 0.0644 ($I > 2\sigma(I)$) and wR_2 was 0.1678 (all data). Using Olex2,¹² the structure was solved with the ShelXT¹³ structure solution program using Intrinsic Phasing and refined with the ShelXL¹⁴ refinement package using Least Squares minimization. Supplementary crystallographic data for this paper have been deposited at Cambridge Crystallographic Data Centre (CCDC number 21216235) and can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

6. References:

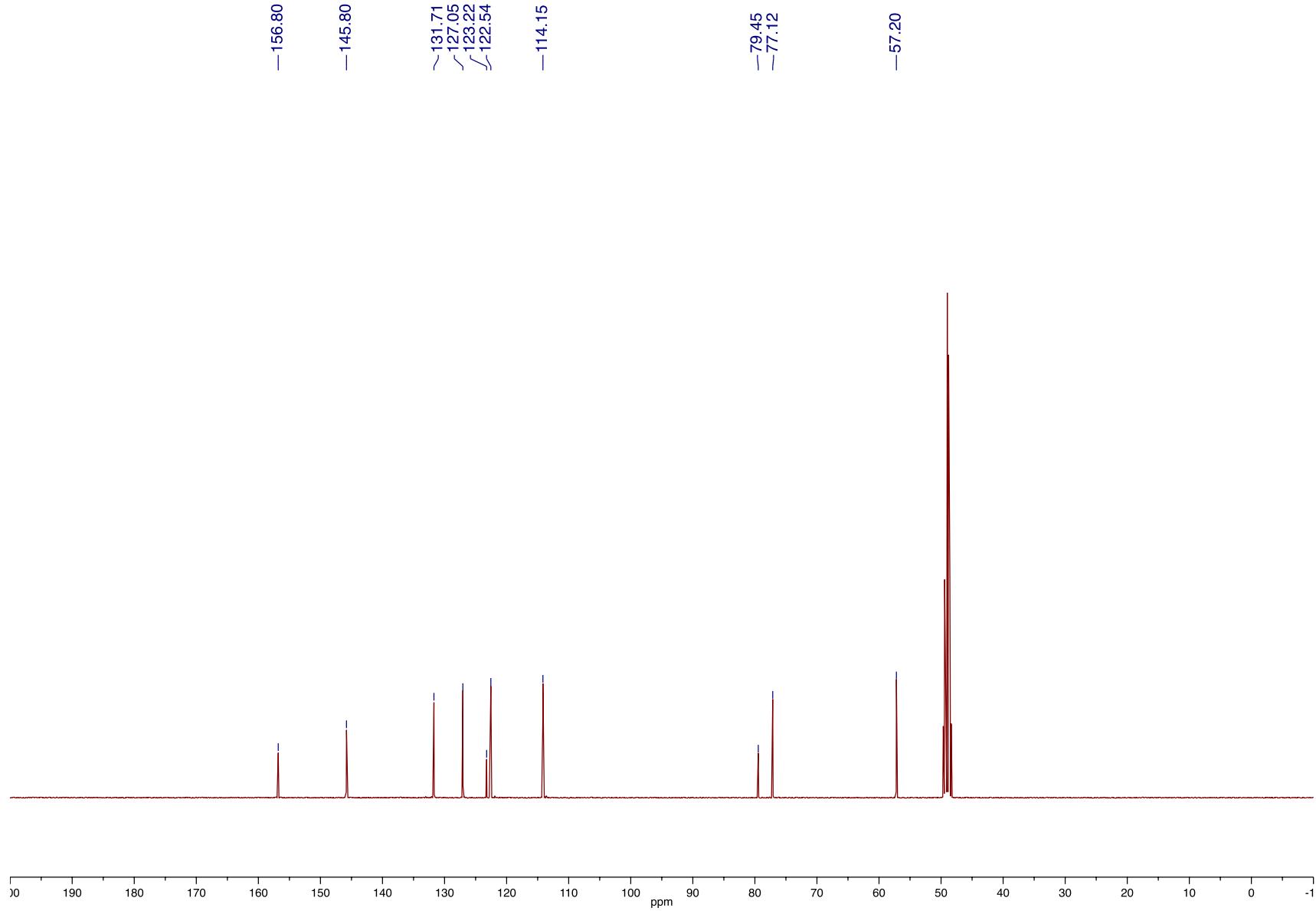
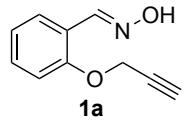
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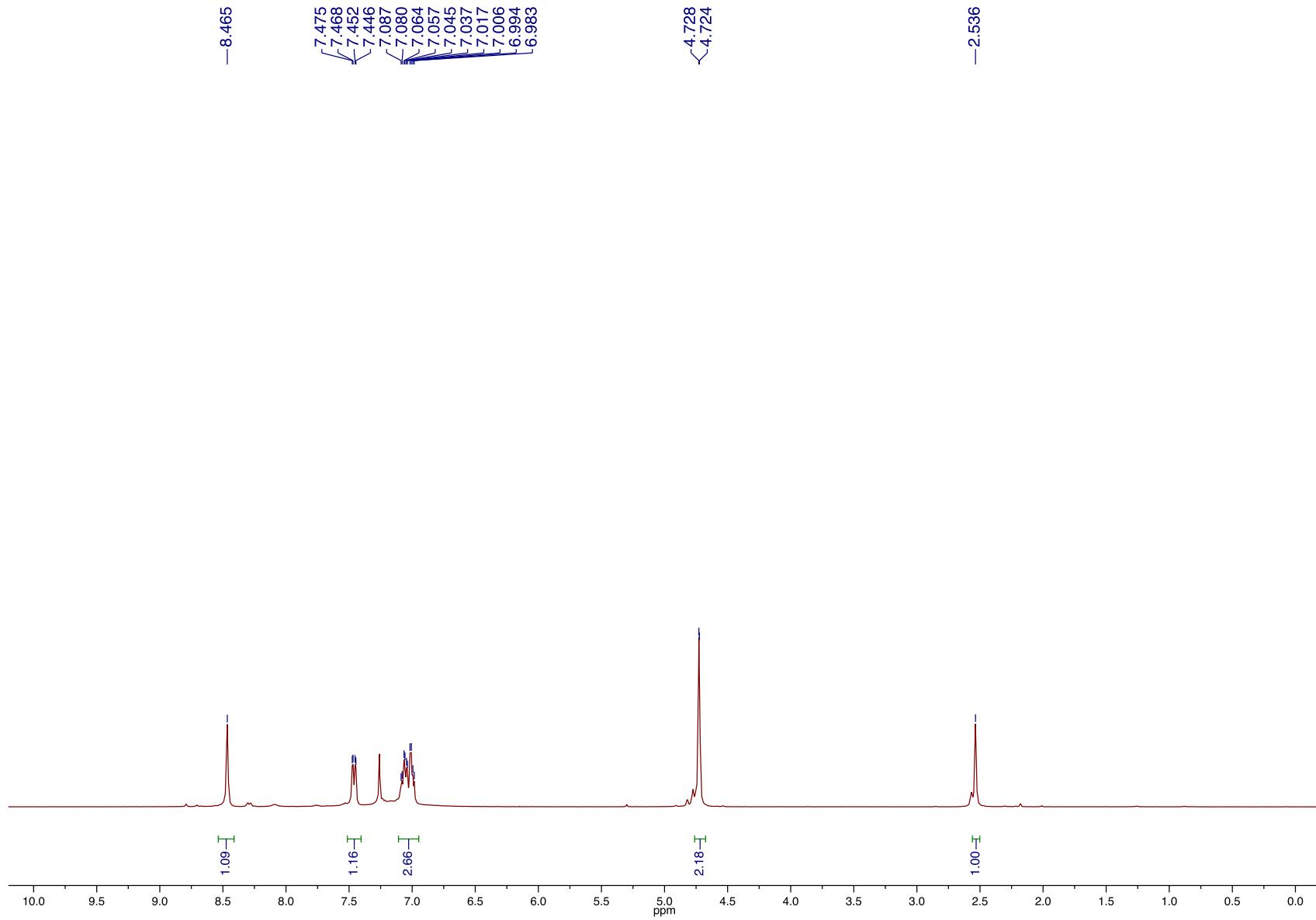
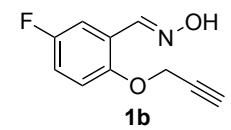
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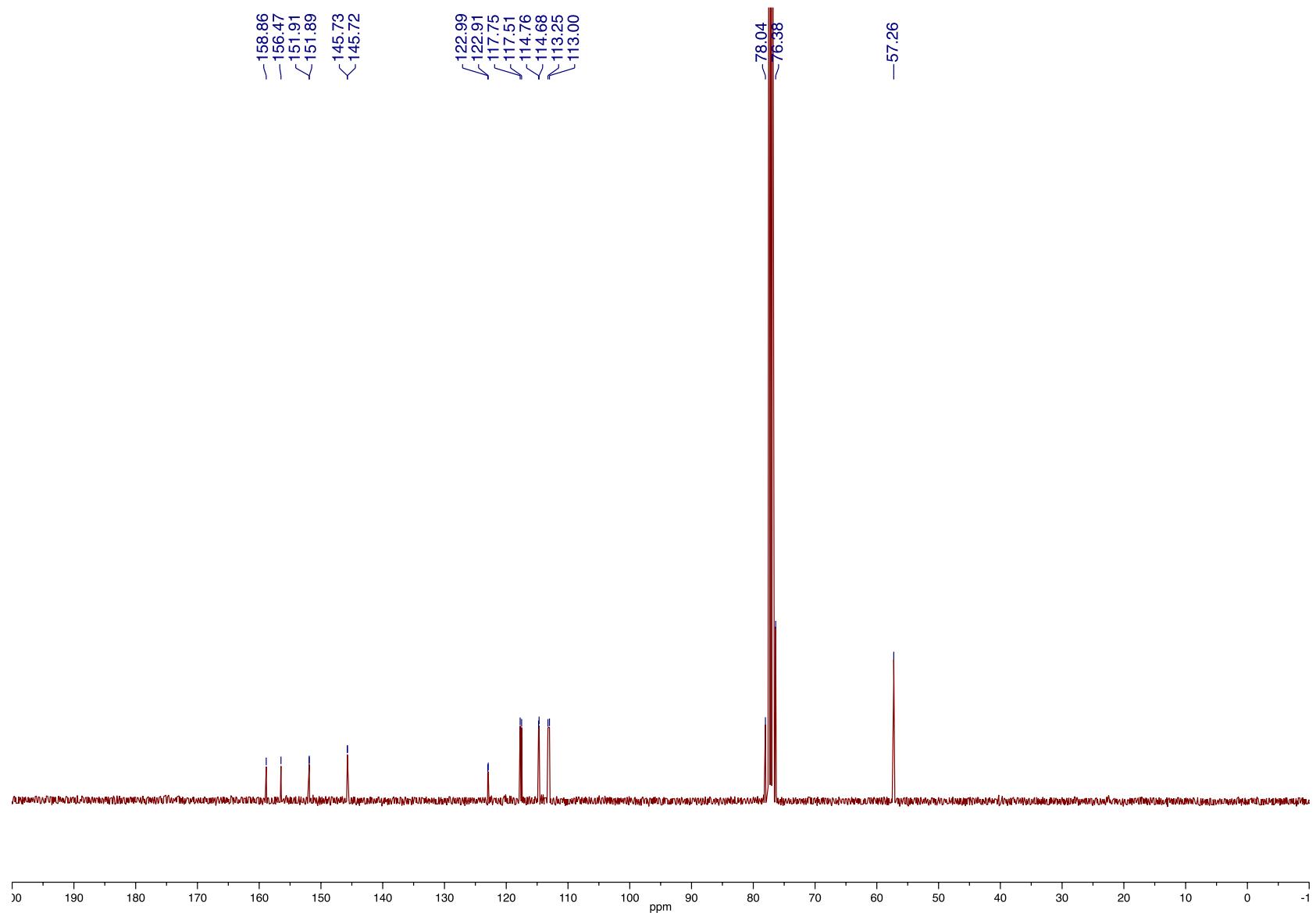
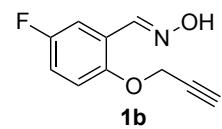
¹³C NMR (101 MHz, CD₃OD)



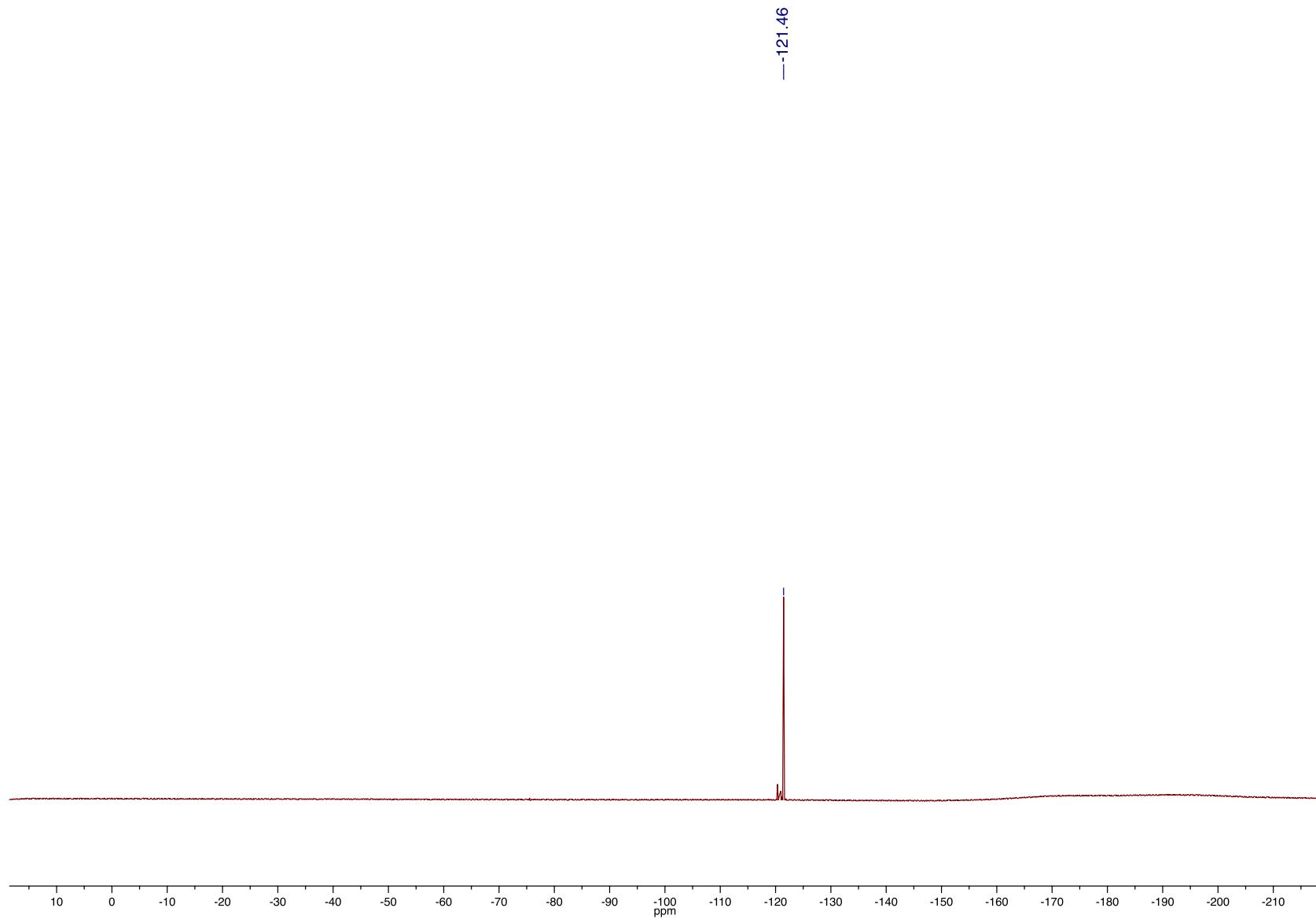
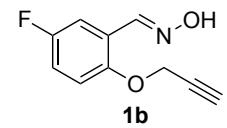
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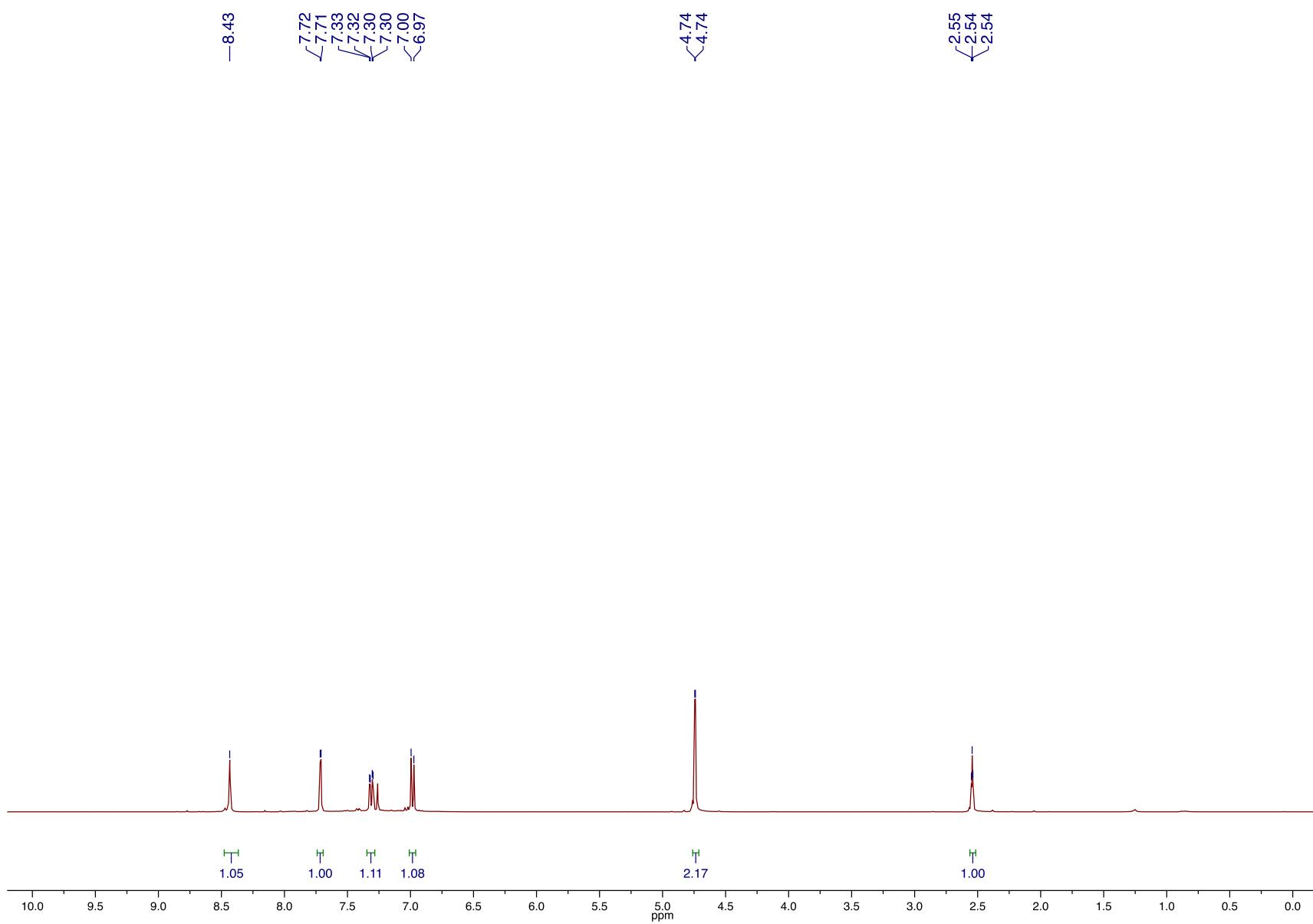
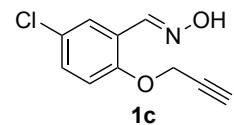
¹³C NMR (101 MHz, CDCl₃)



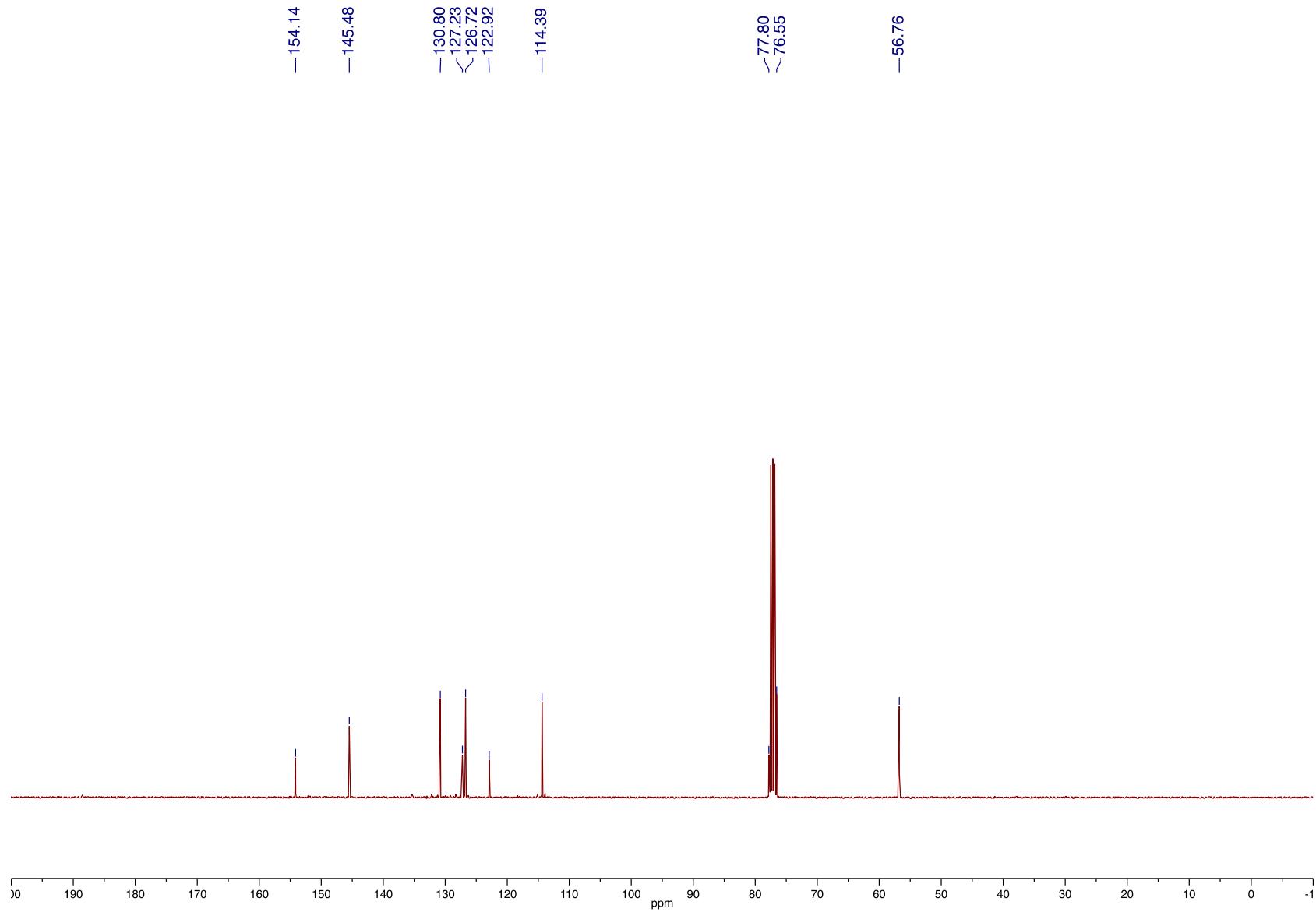
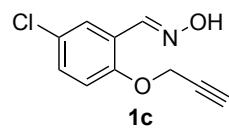
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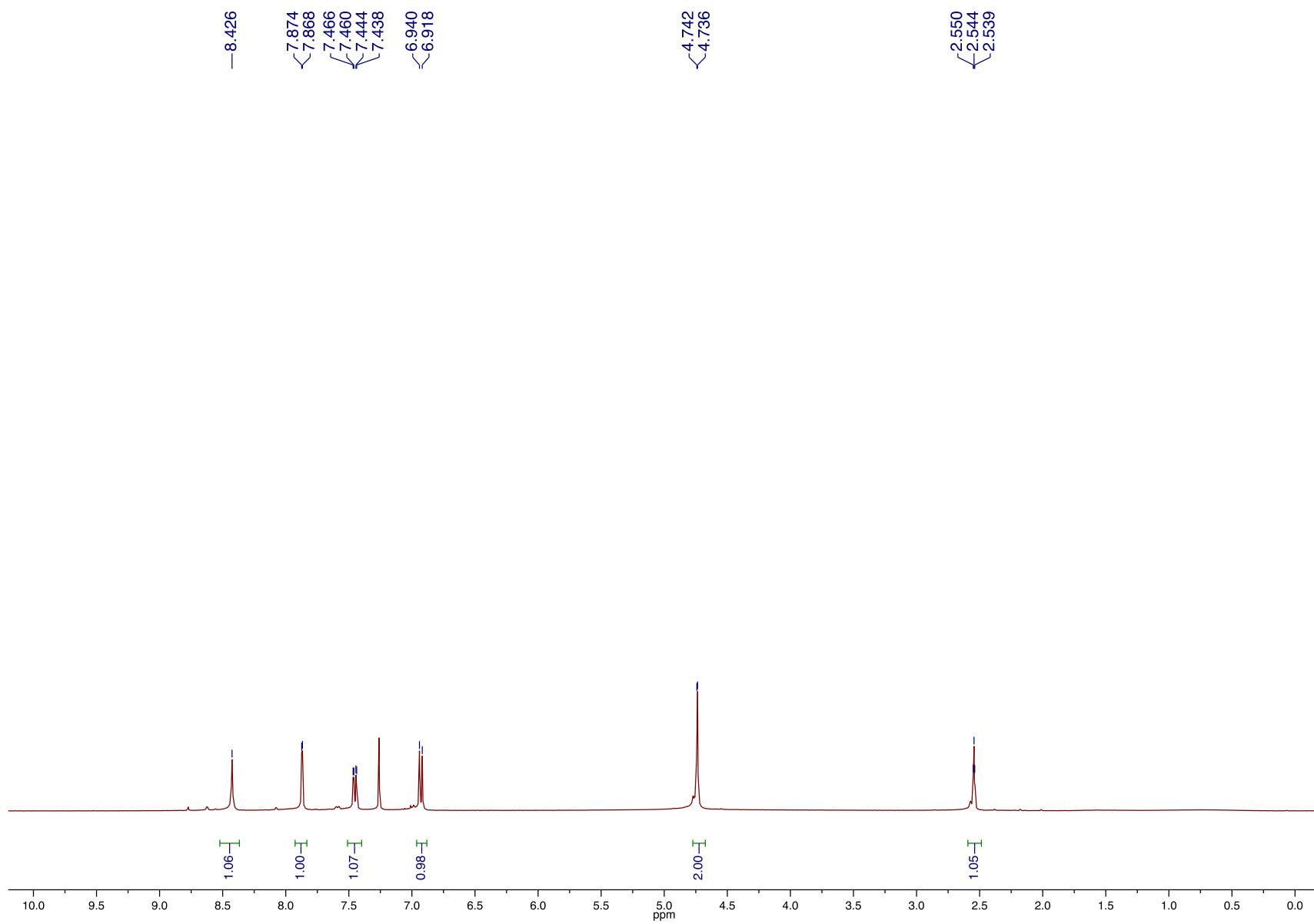
¹H NMR (400 MHz, CDCl₃)



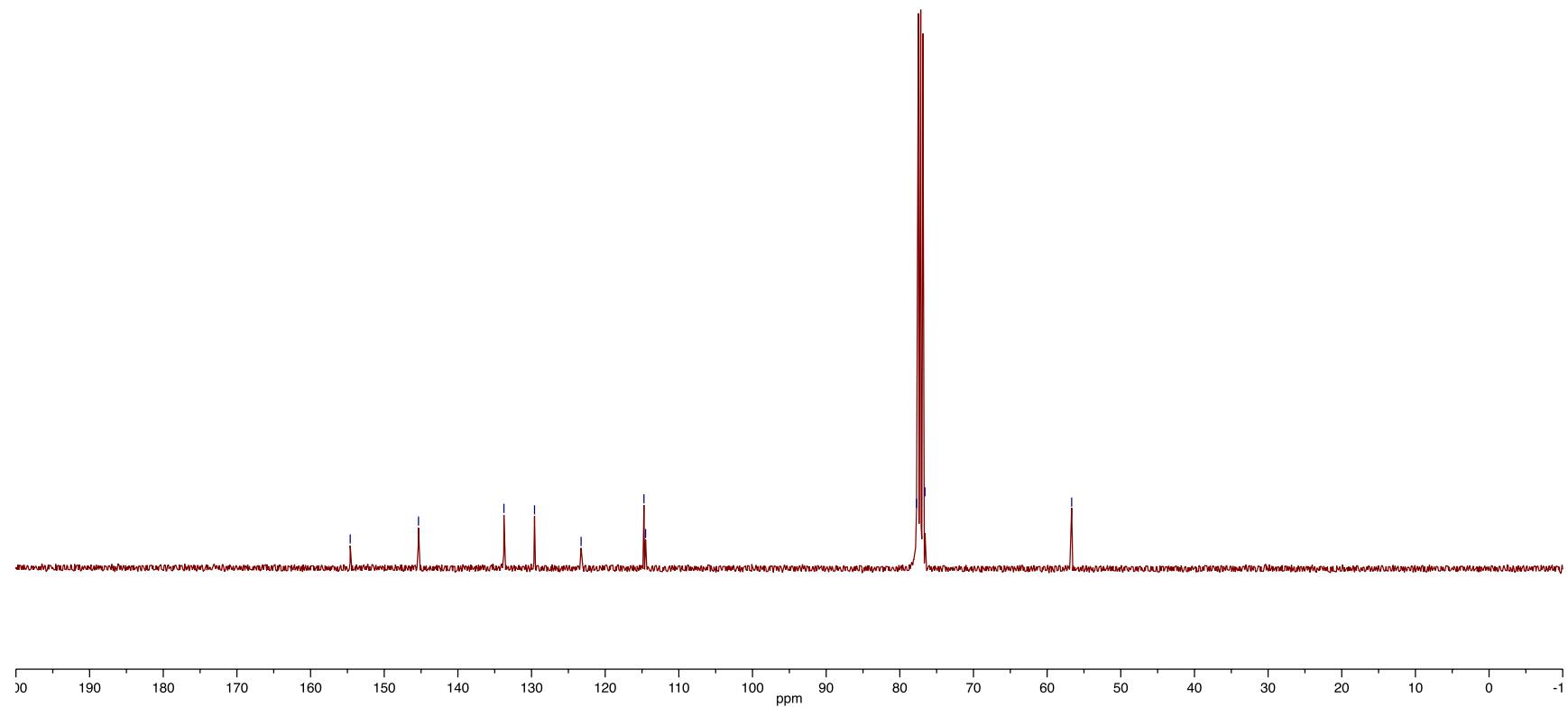
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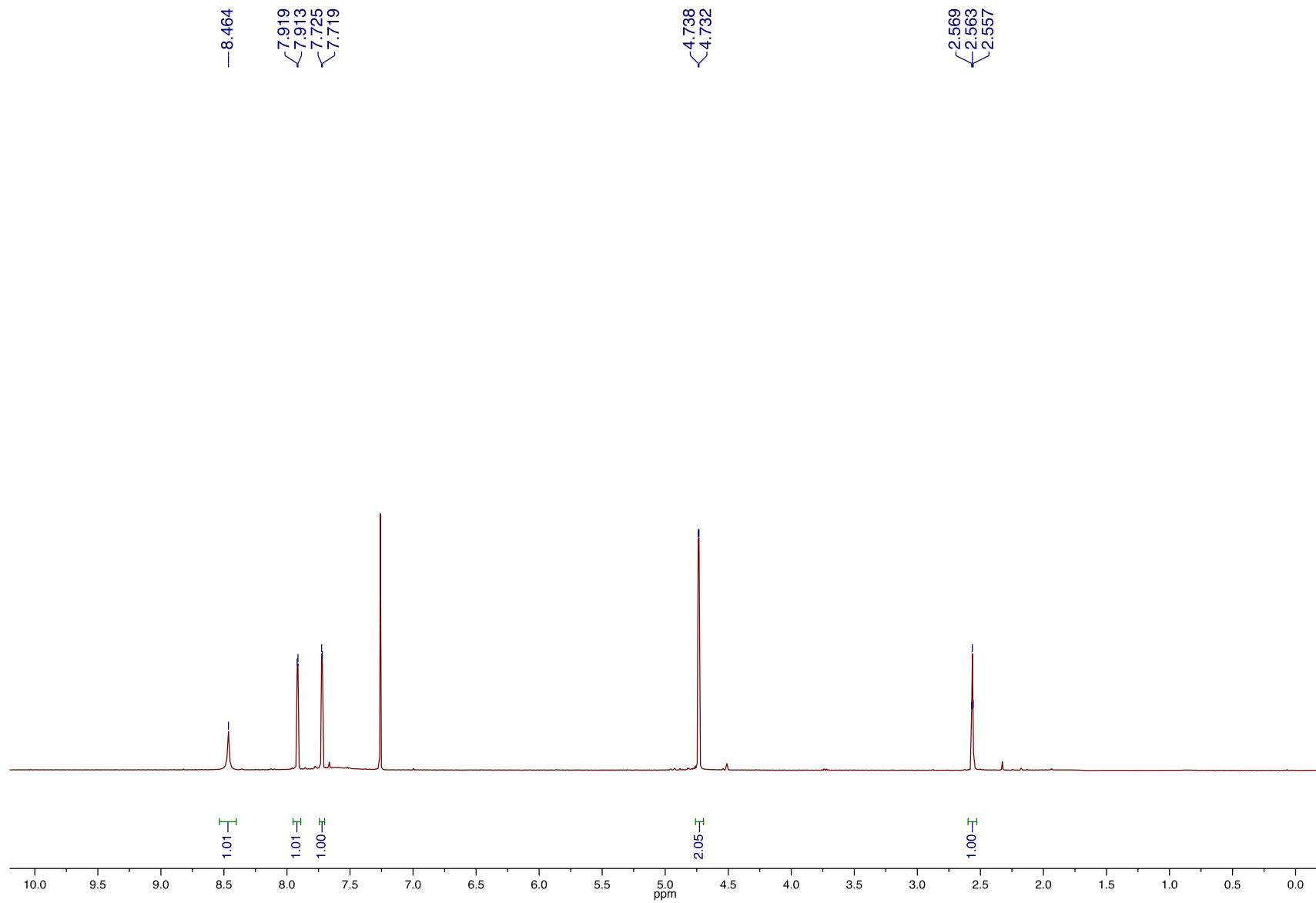
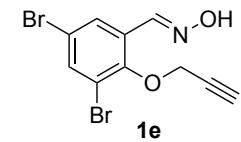
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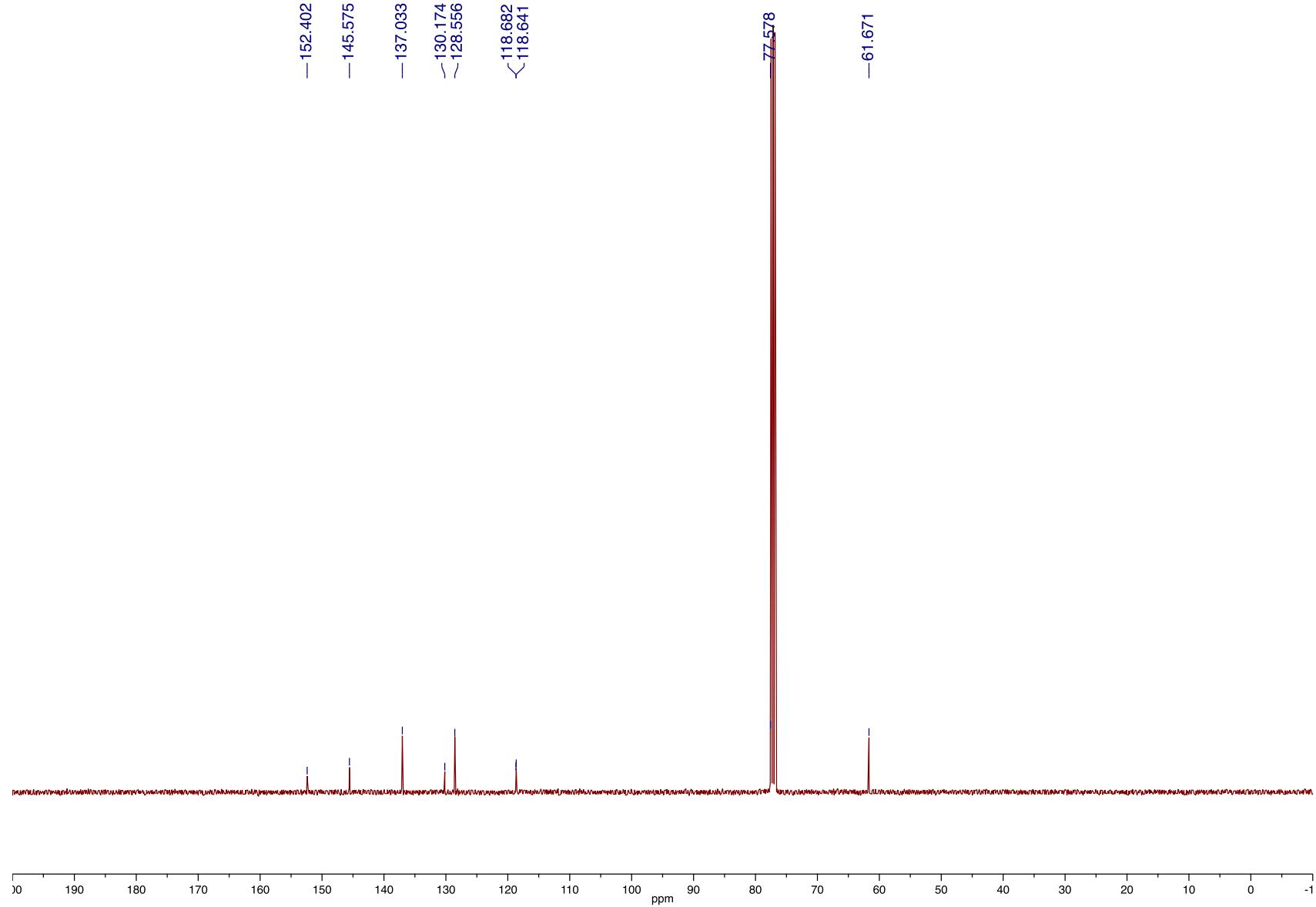
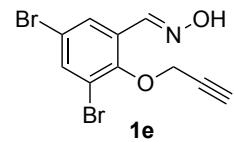
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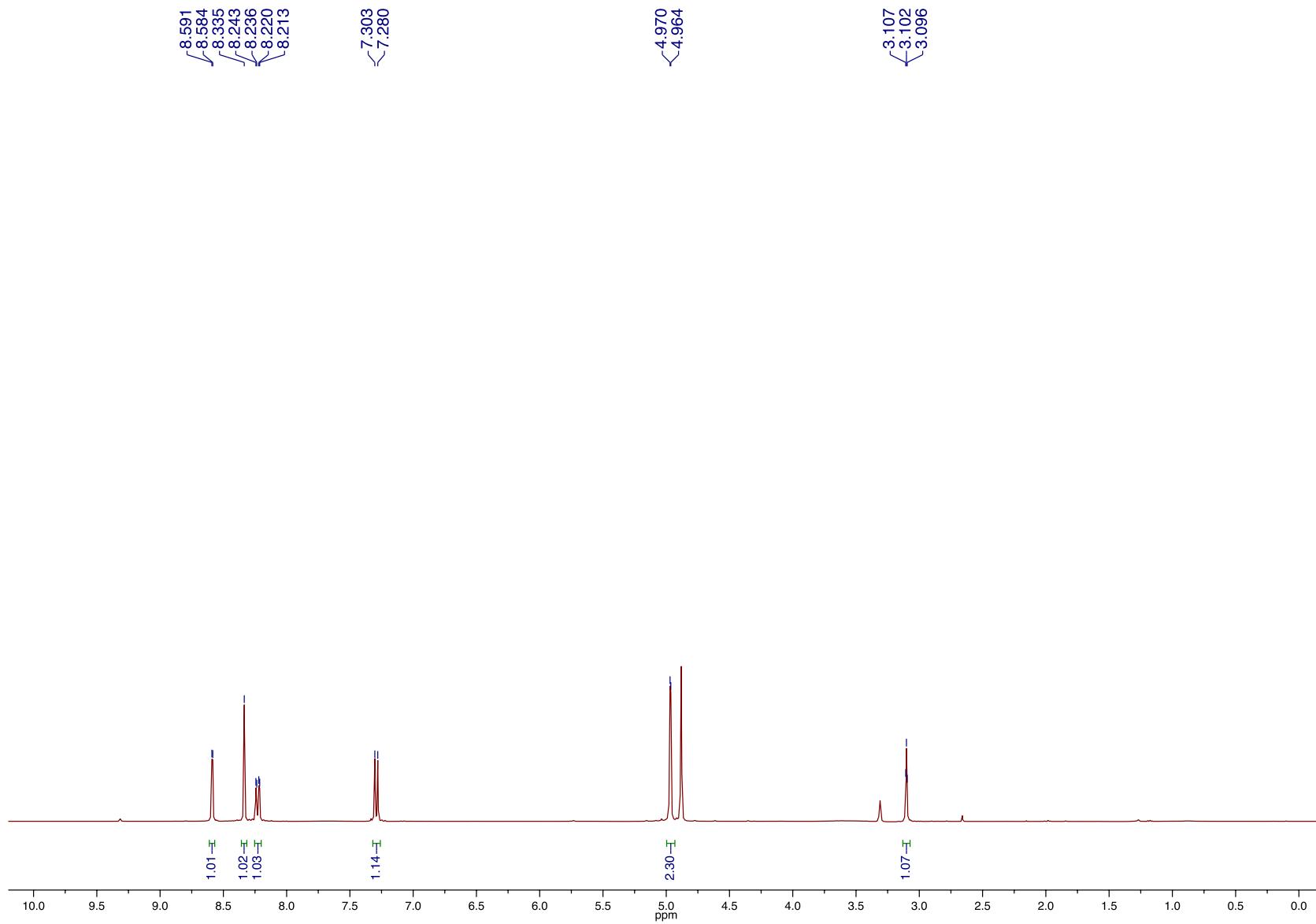
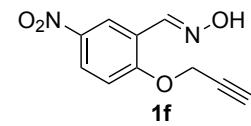
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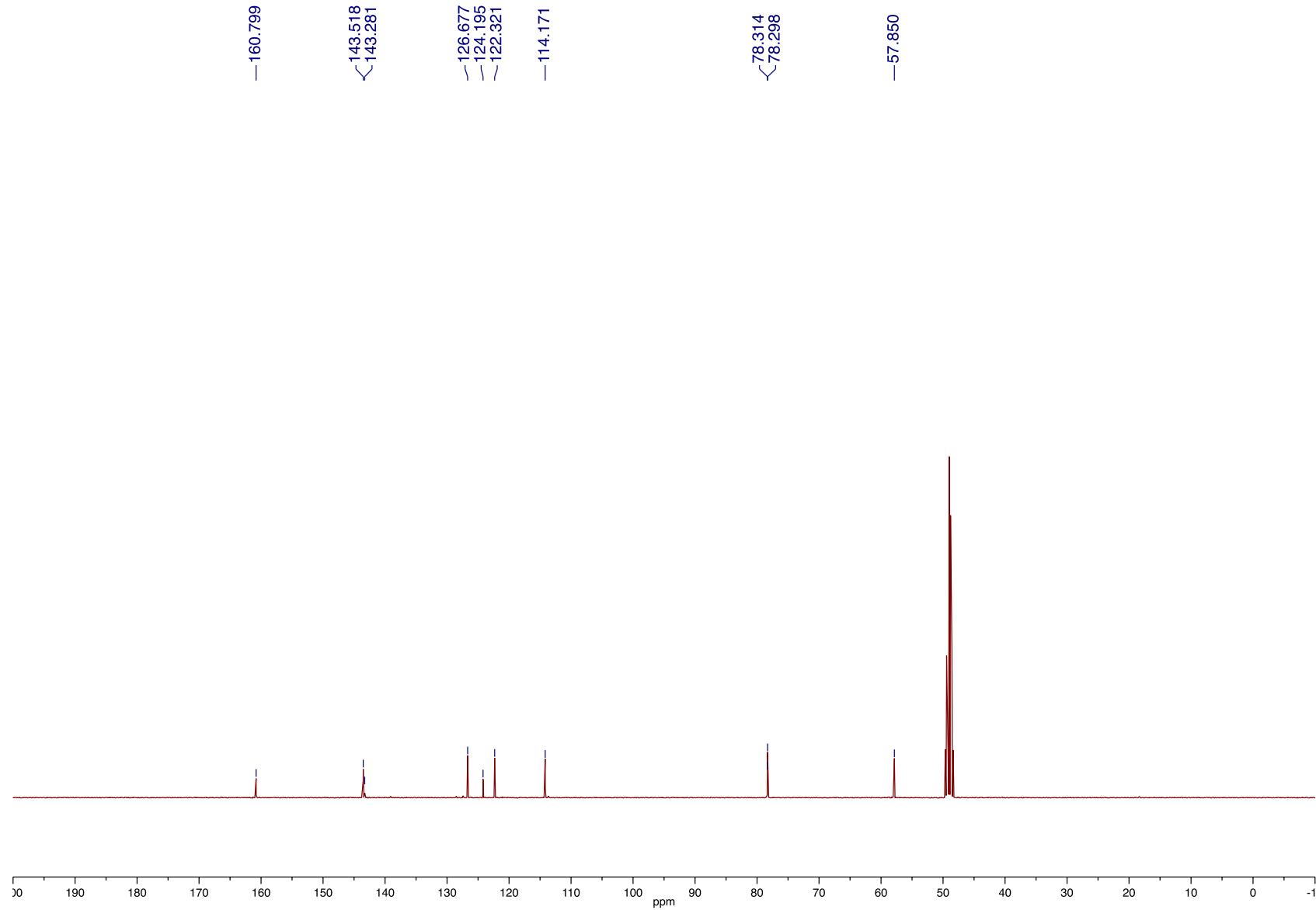
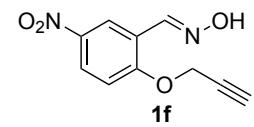
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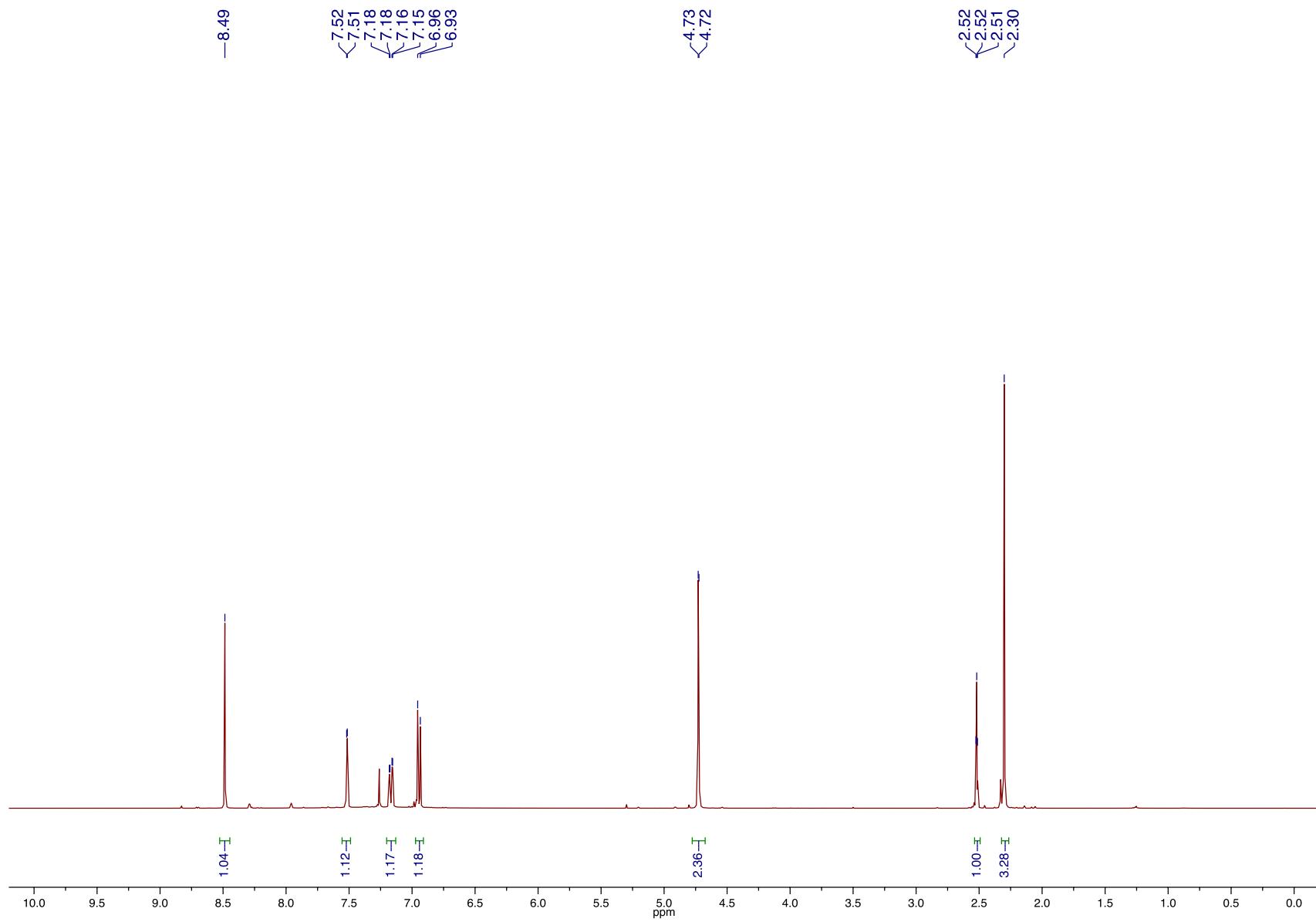
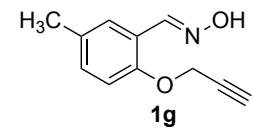
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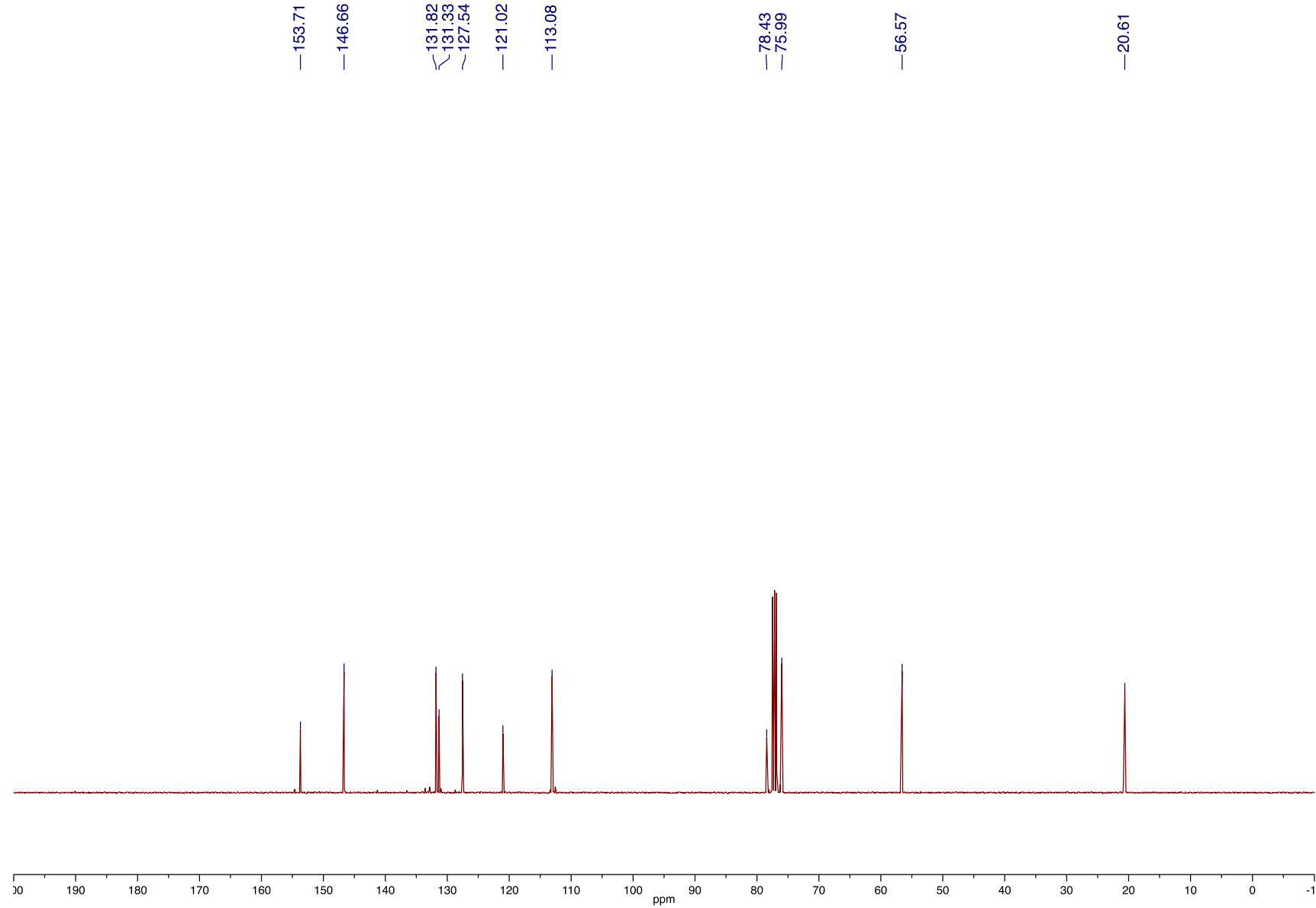
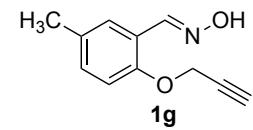
¹³C NMR (101 MHz, CD₃OD)



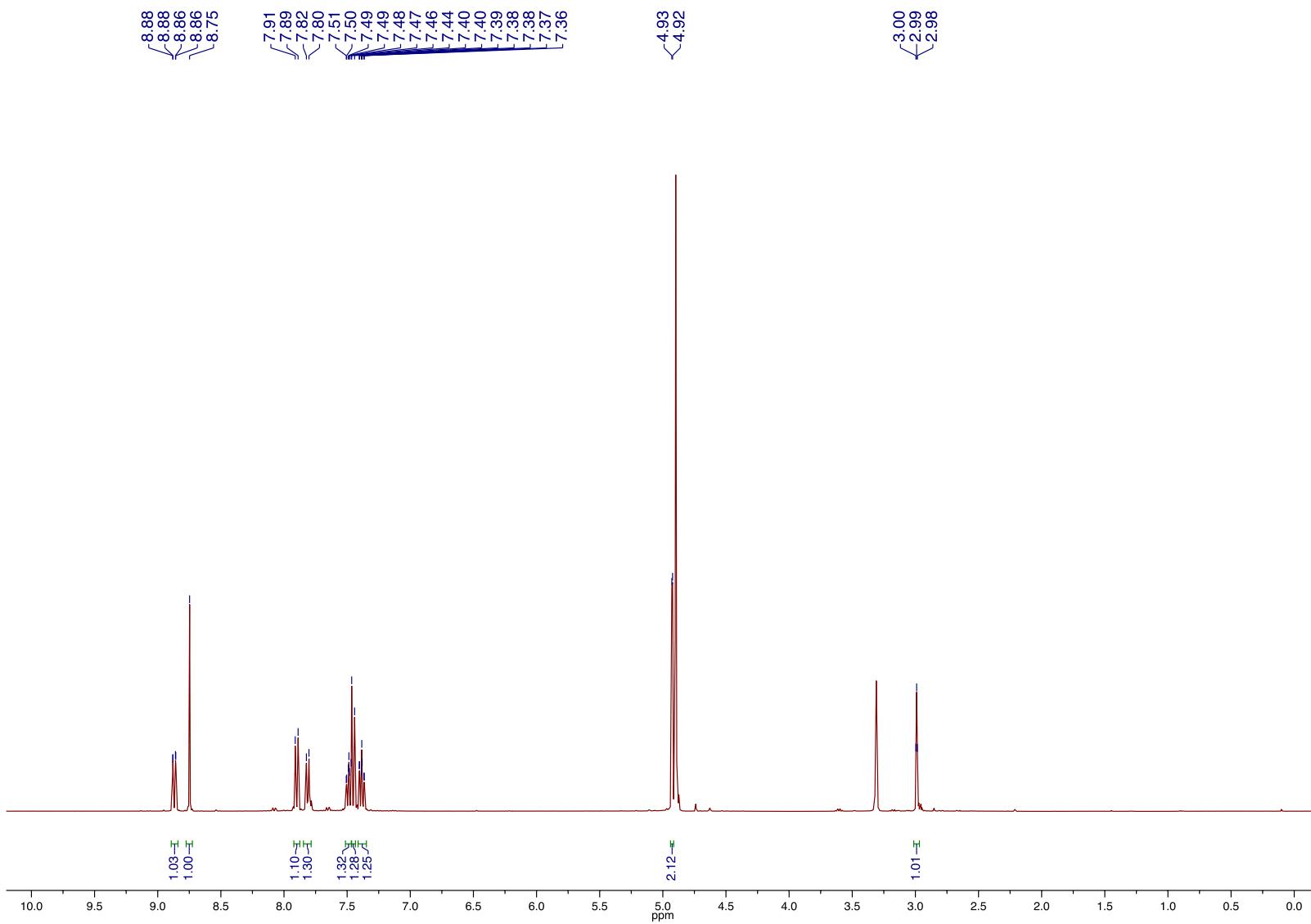
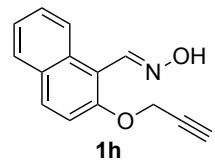
¹H NMR (400 MHz, CDCl₃)



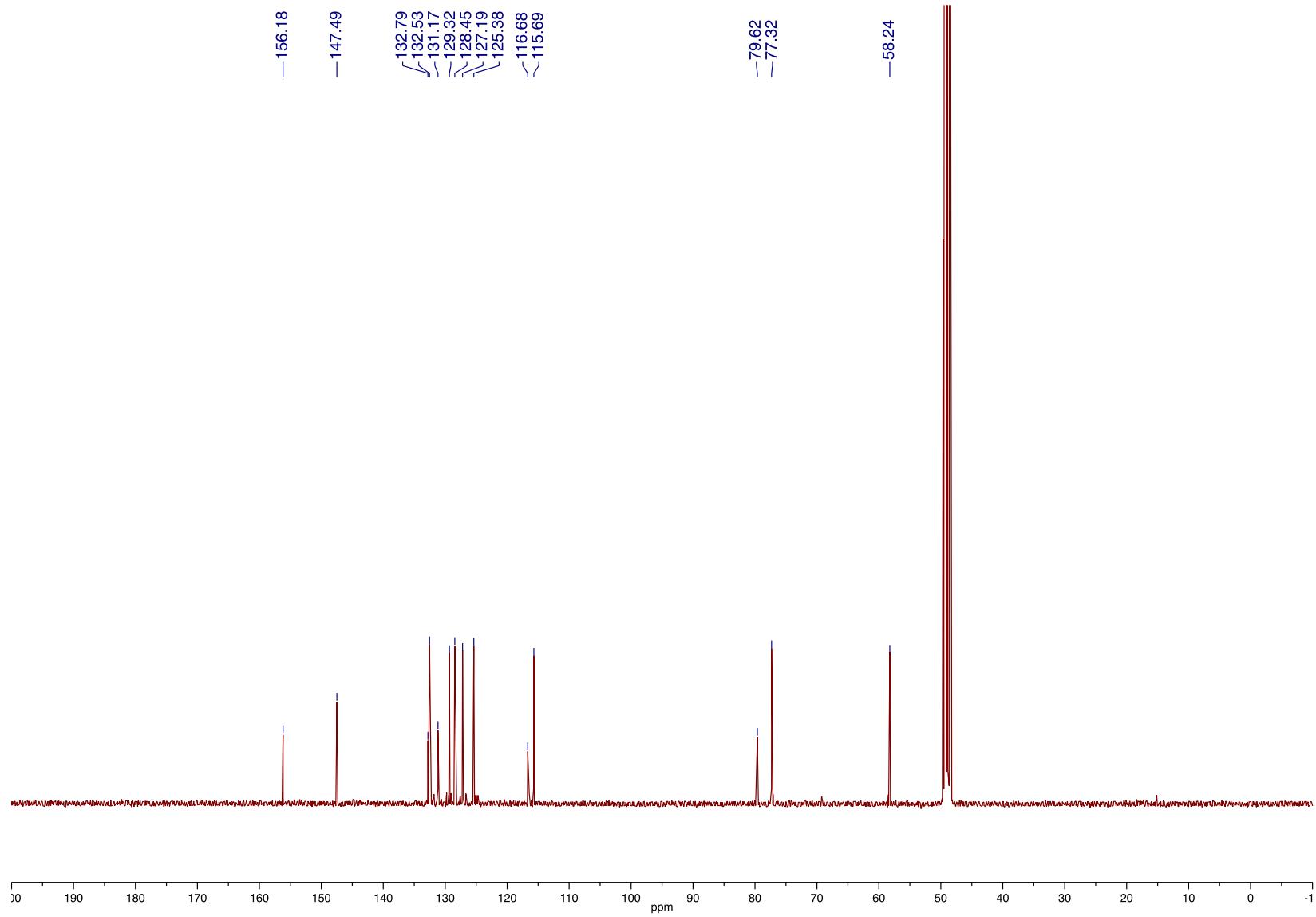
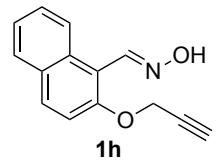
¹³C NMR (101 MHz, CDCl₃)



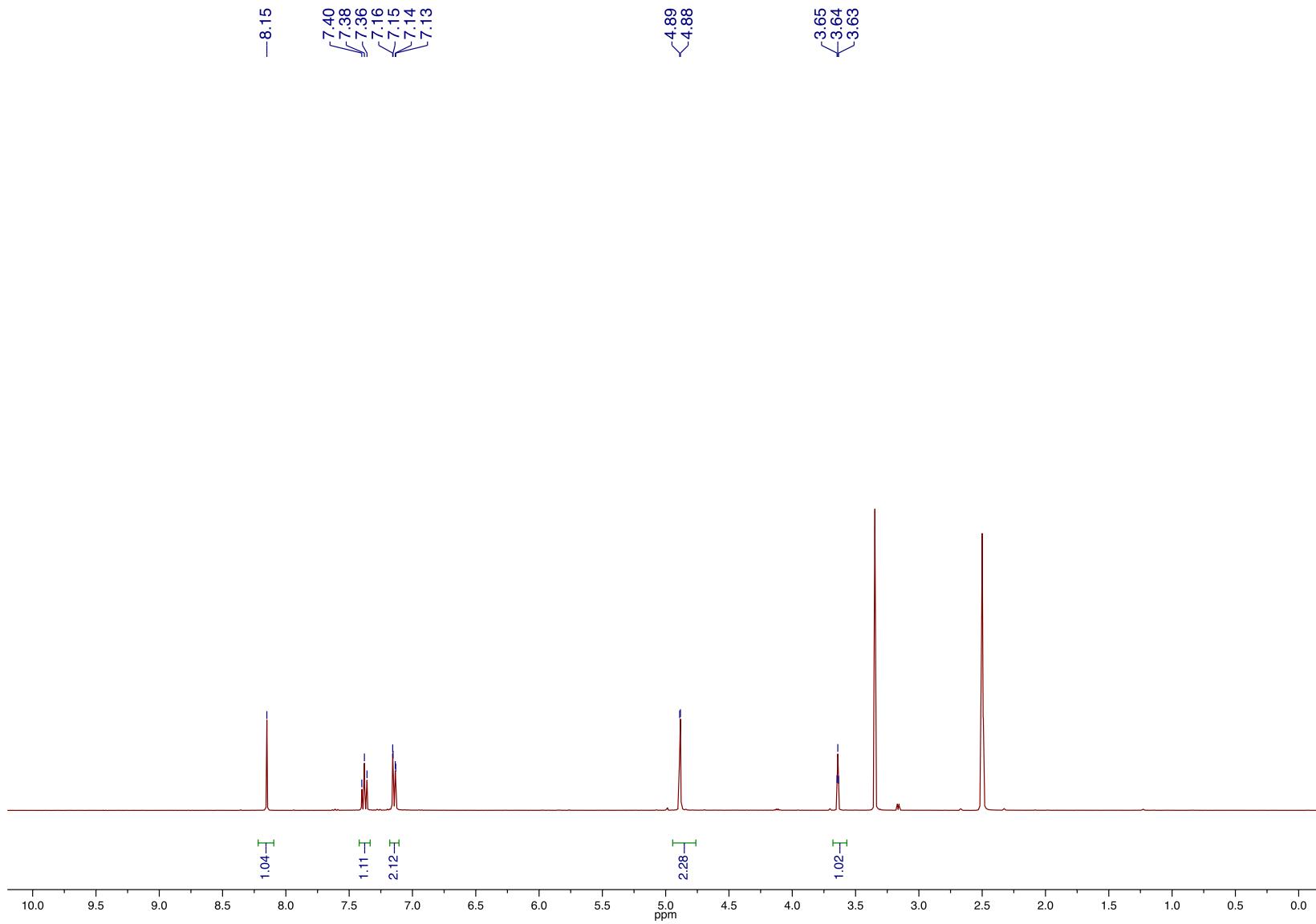
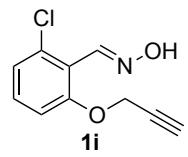
¹H NMR (400 MHz, CD₃OD)



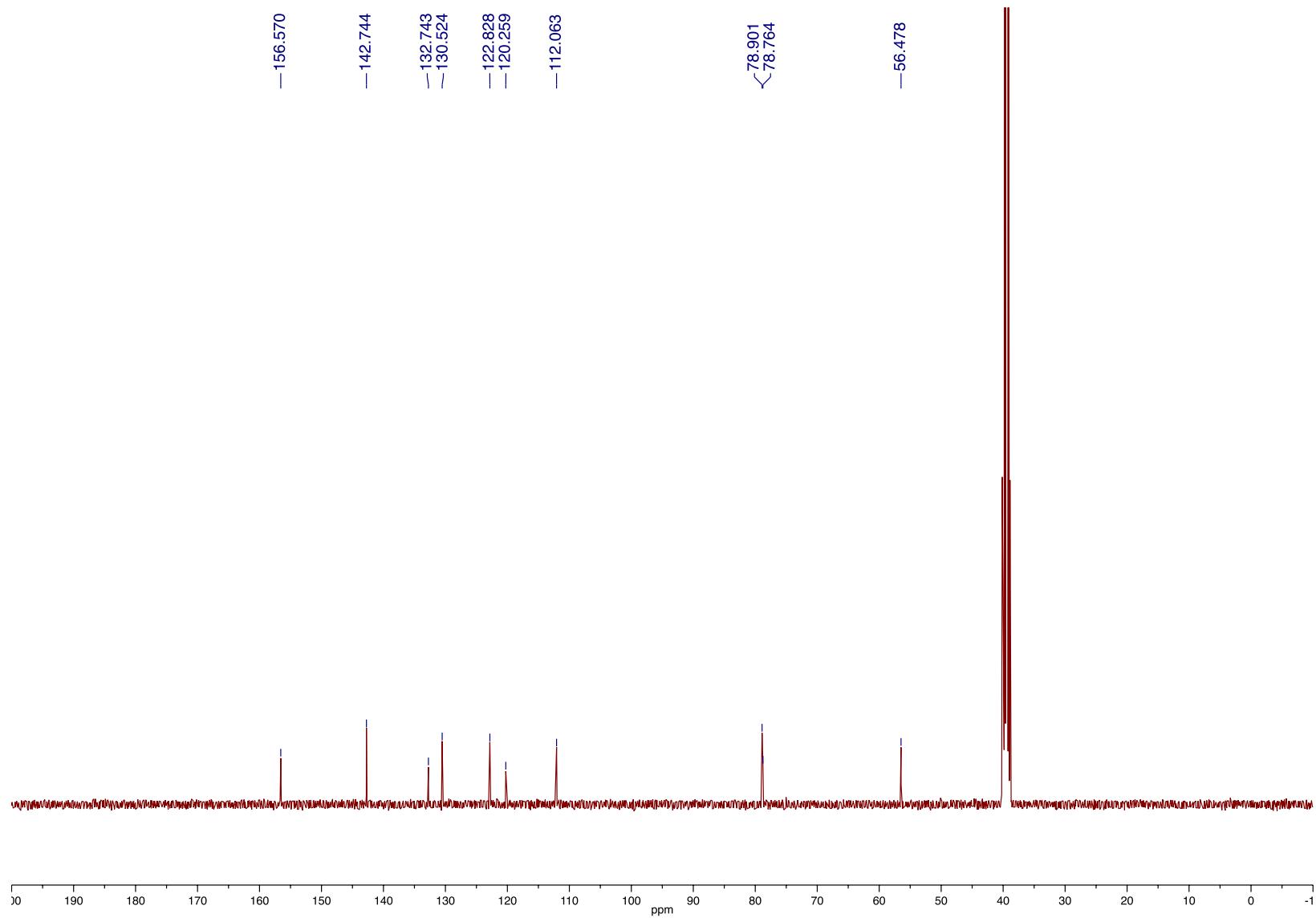
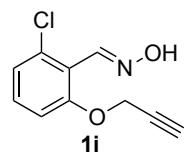
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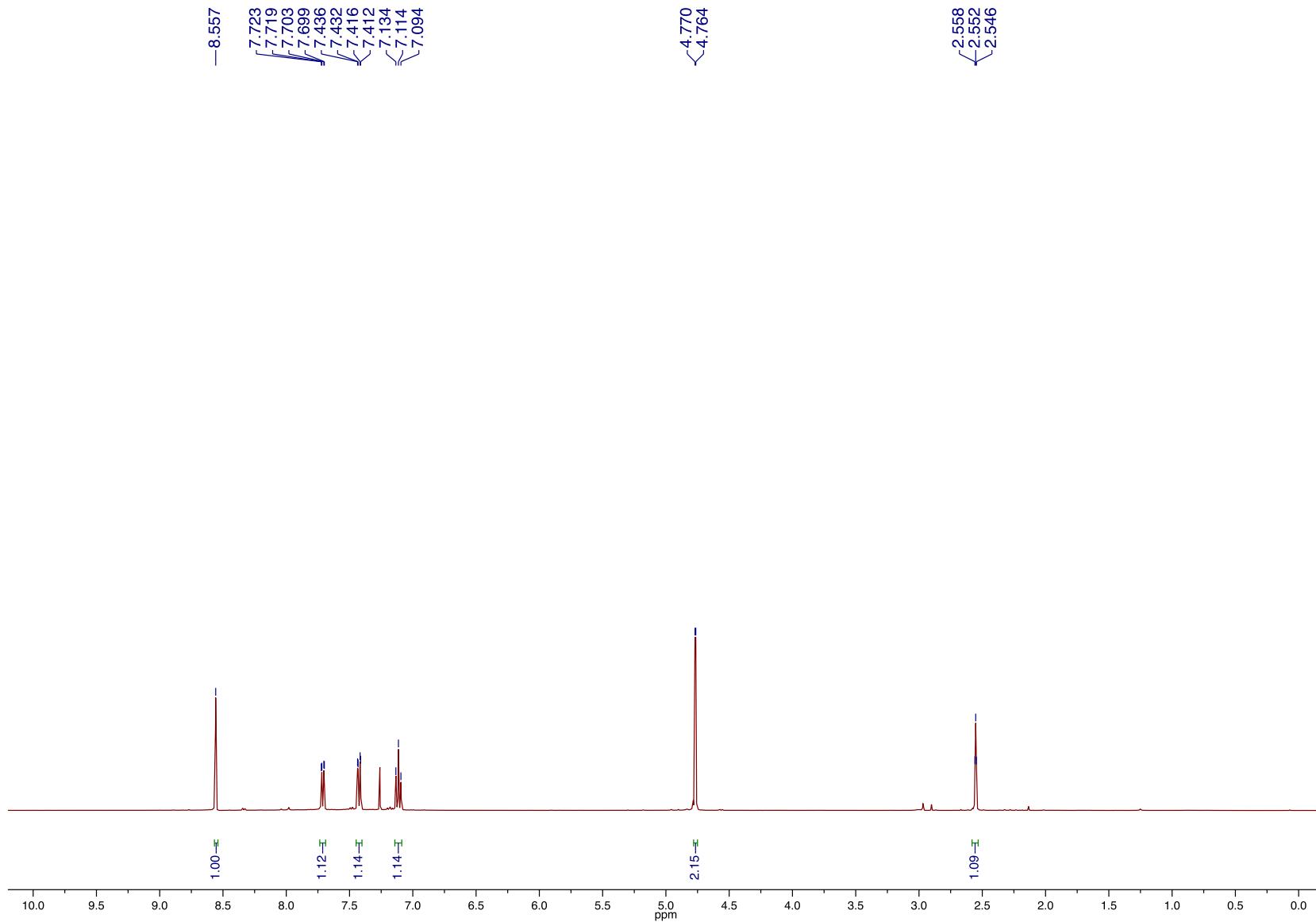
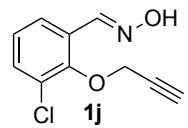
¹H NMR (400 MHz, DMSO-d₆)



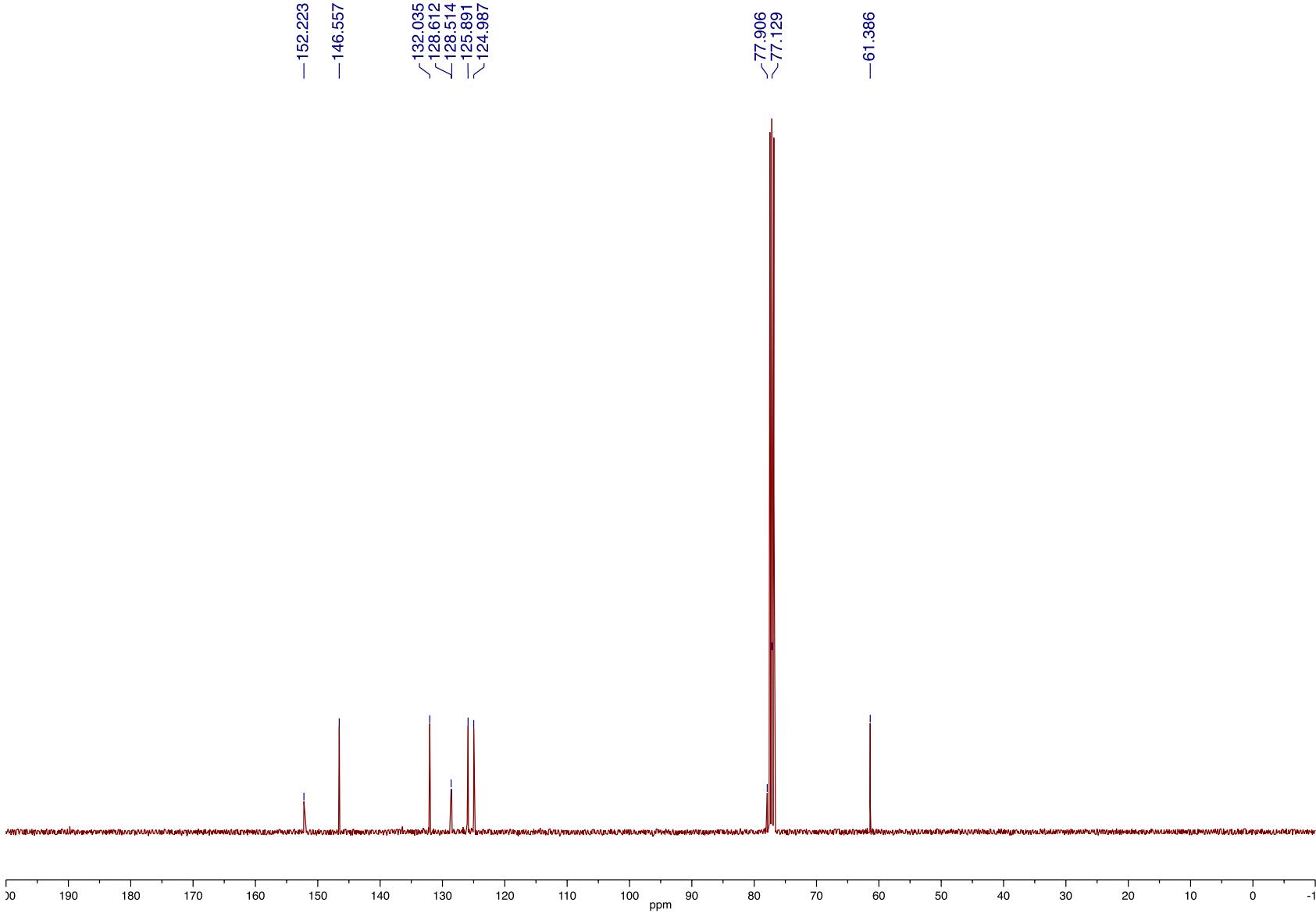
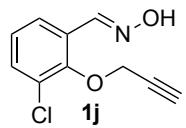
¹³C NMR (101 MHz, DMSO-d₆)



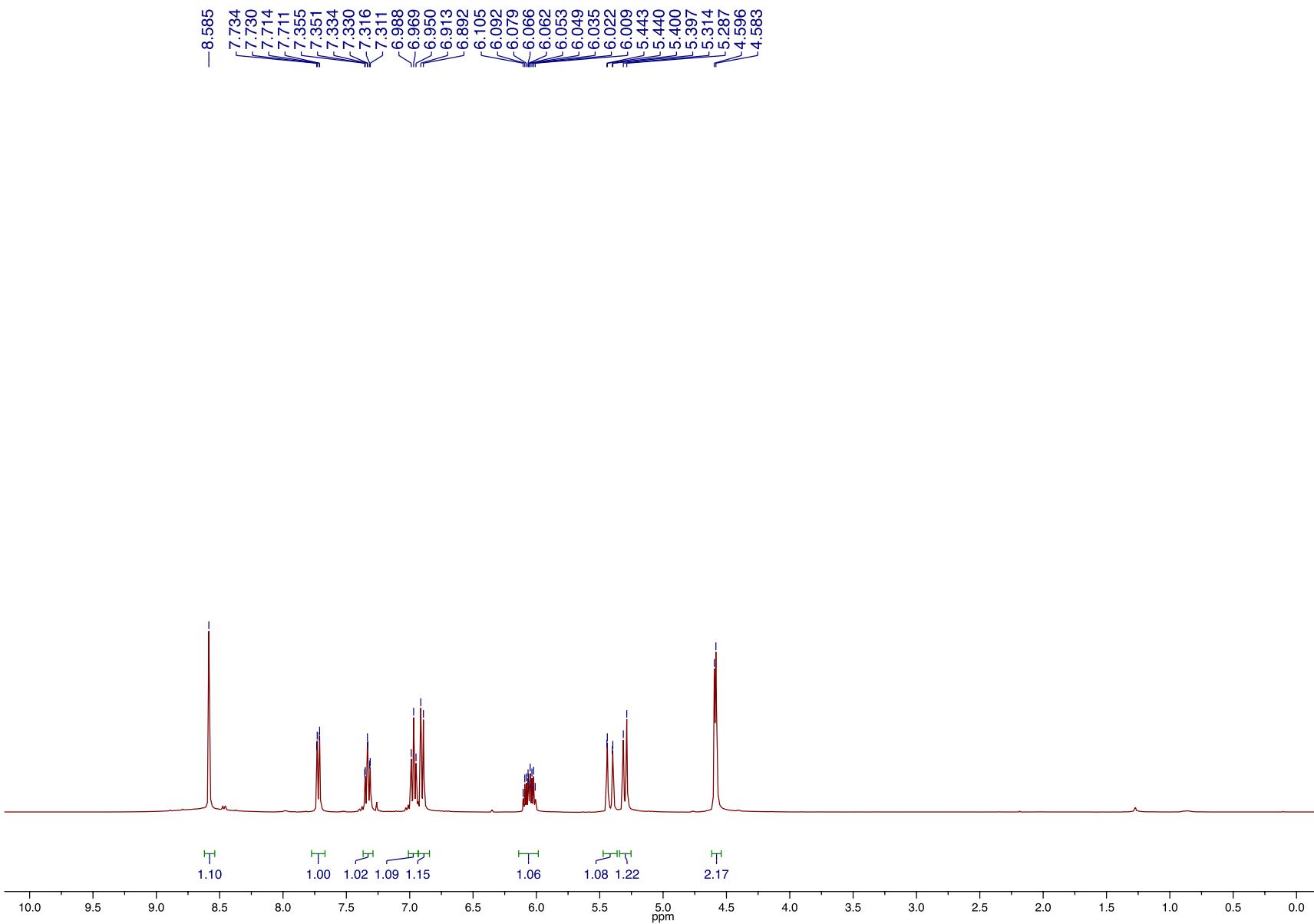
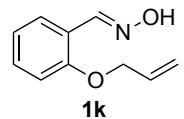
¹H NMR (500 MHz, CDCl₃)



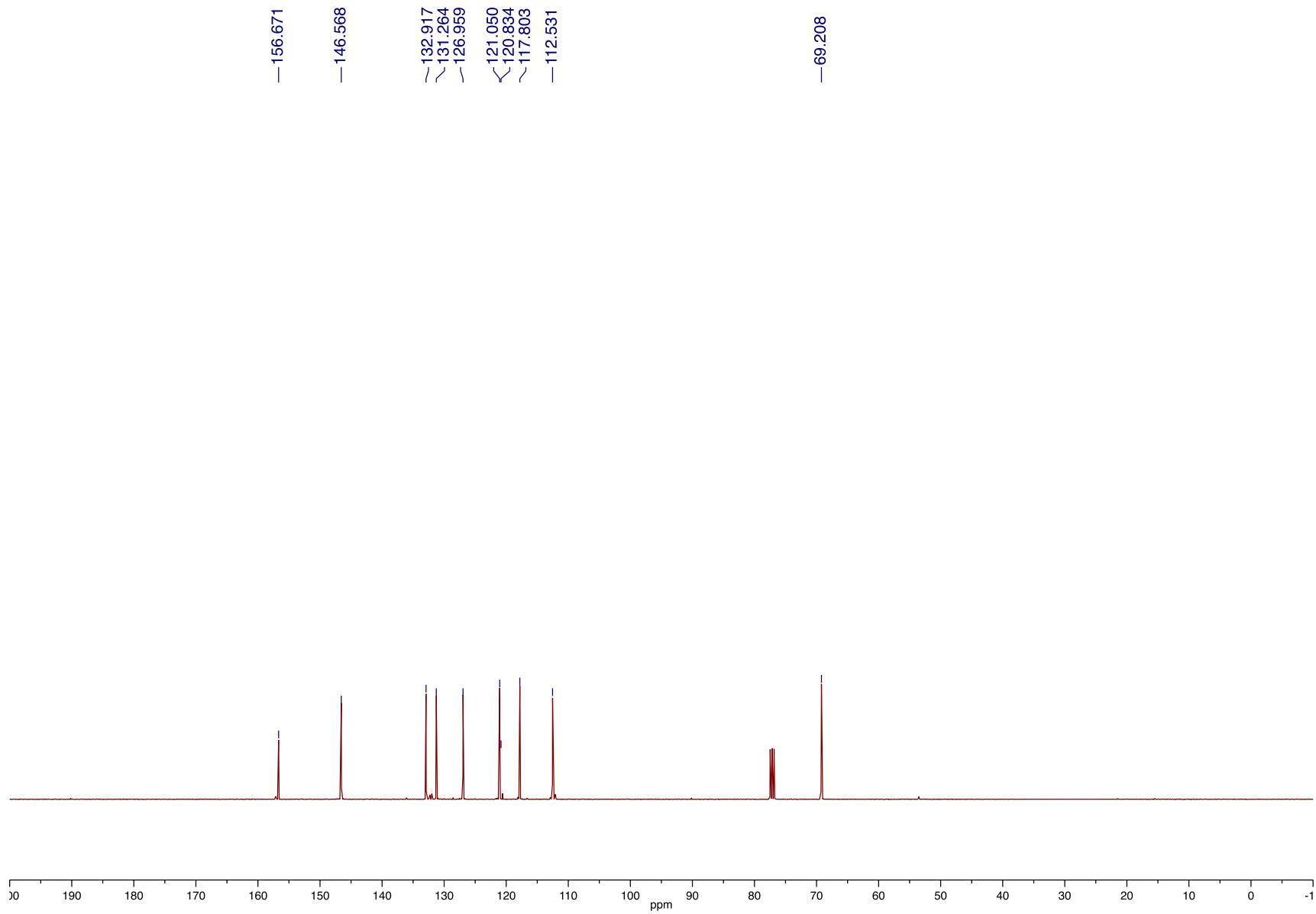
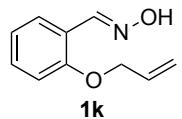
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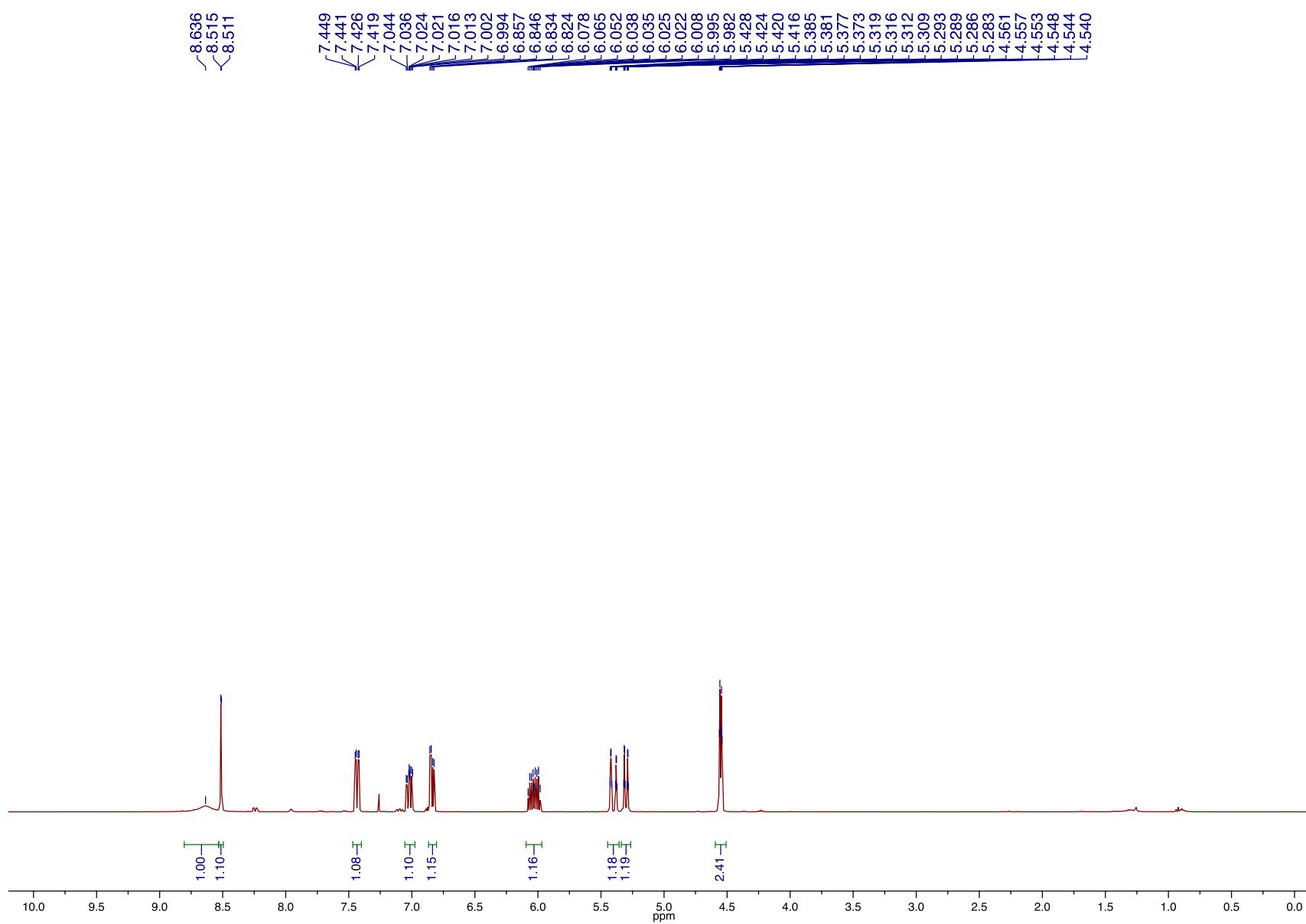
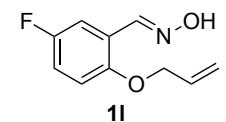
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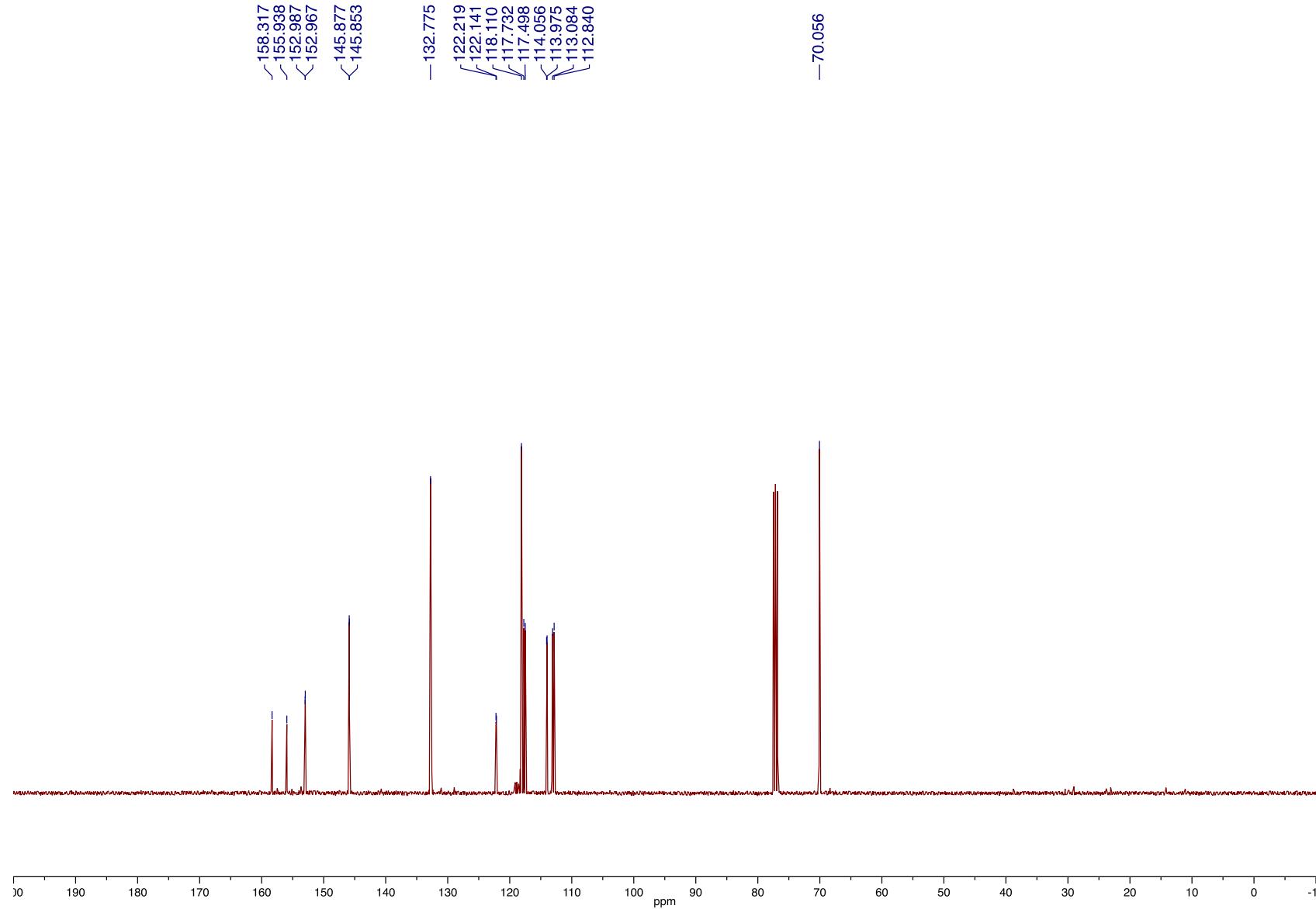
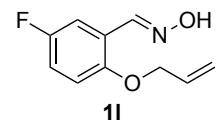
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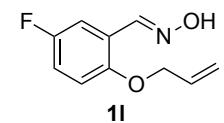
¹H NMR (400 MHz, CDCl₃)



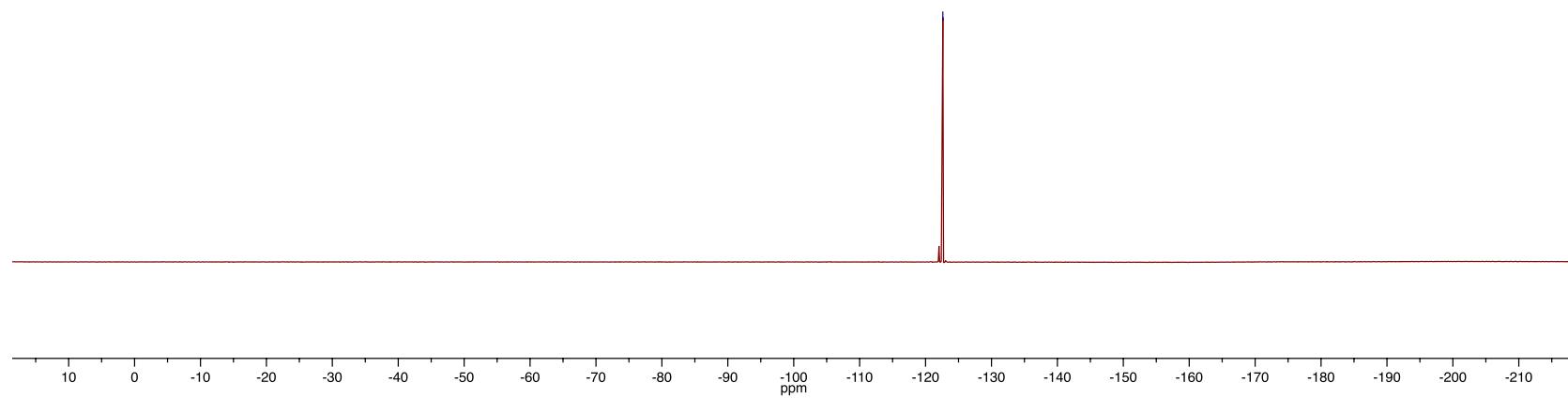
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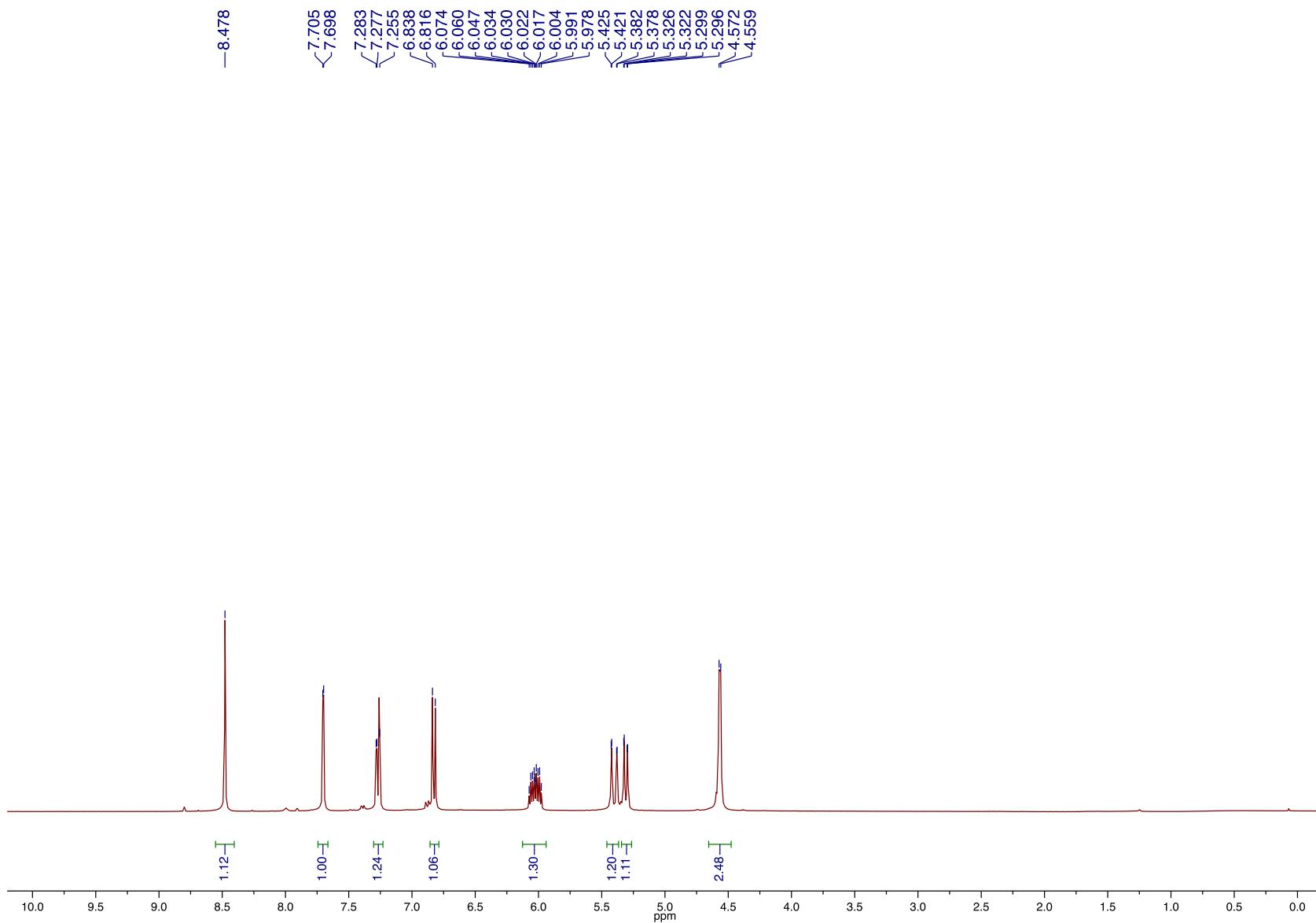
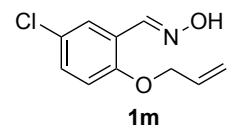
¹⁹F NMR (376 MHz, CDCl₃)



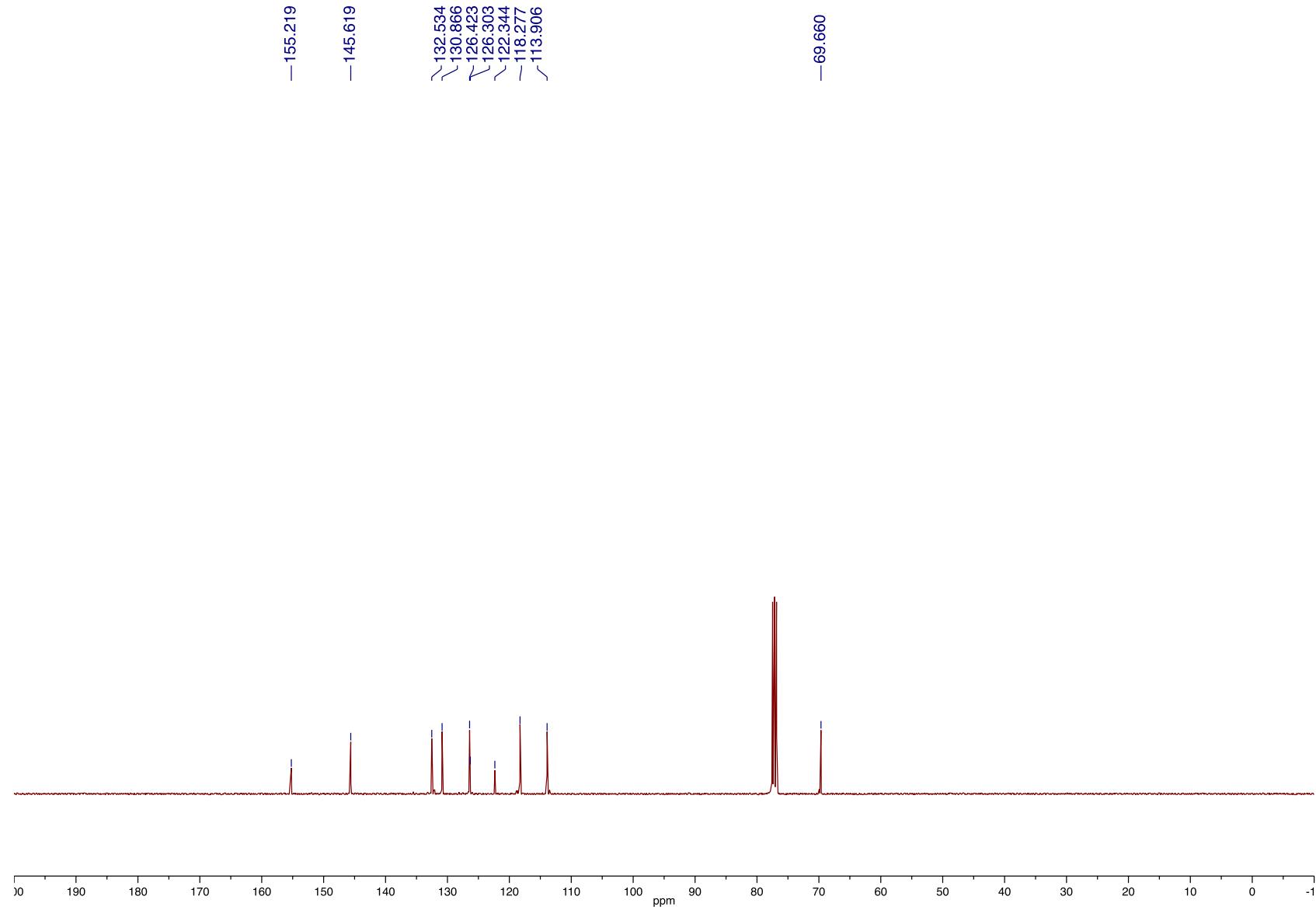
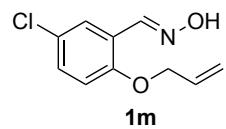
-122.622



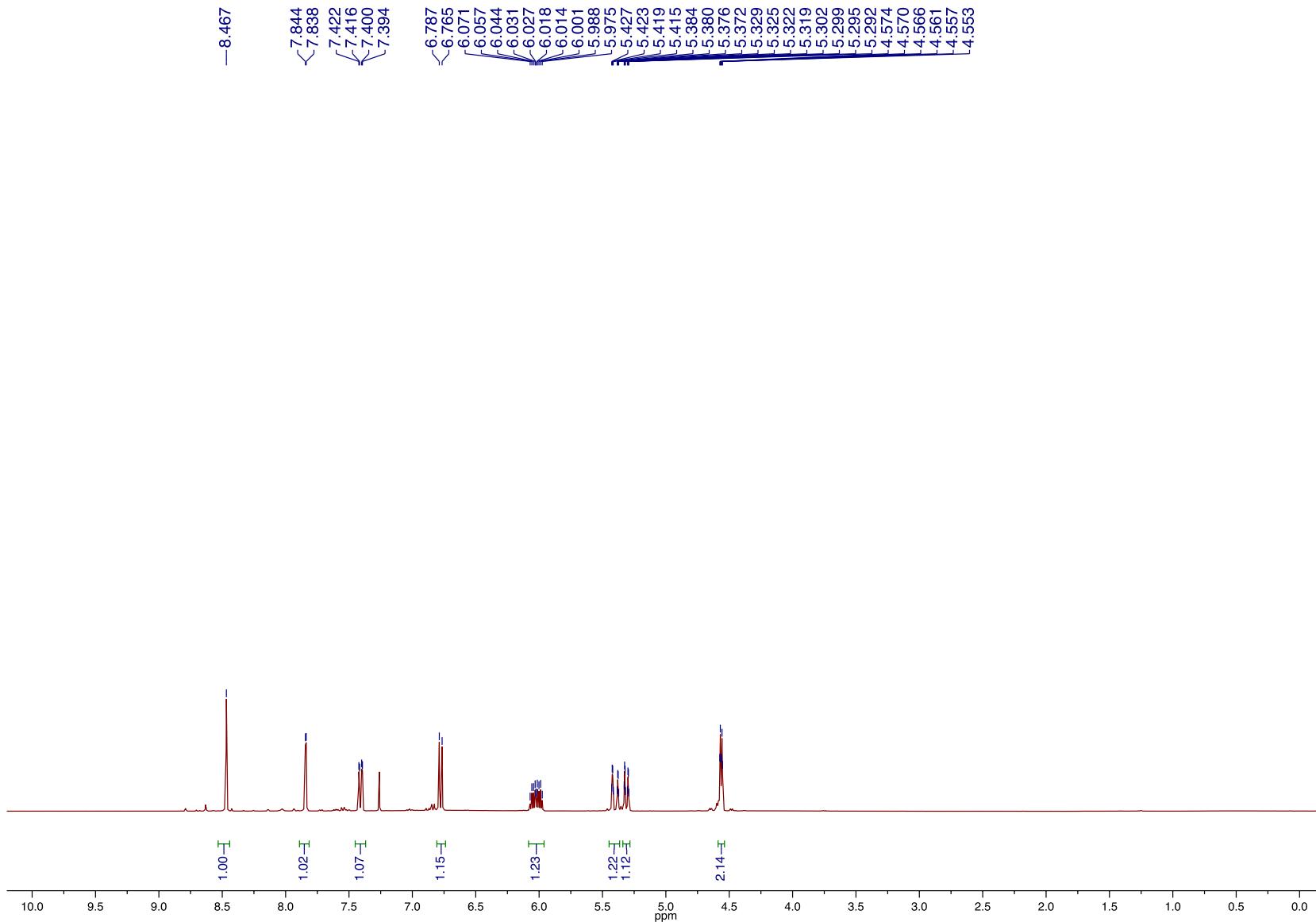
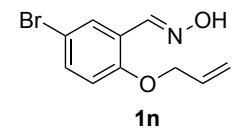
¹H NMR (400 MHz, CDCl₃)



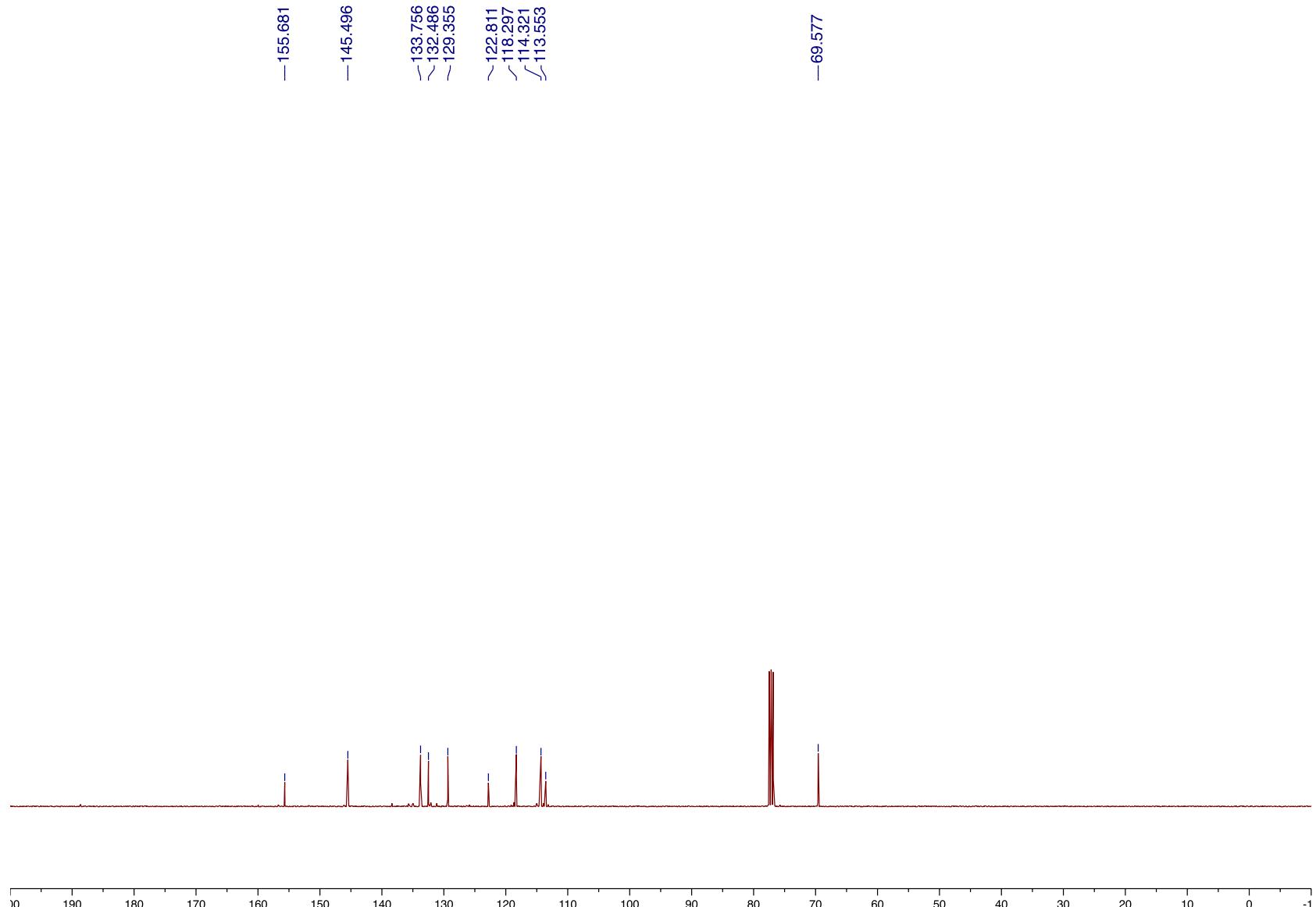
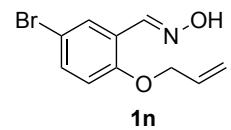
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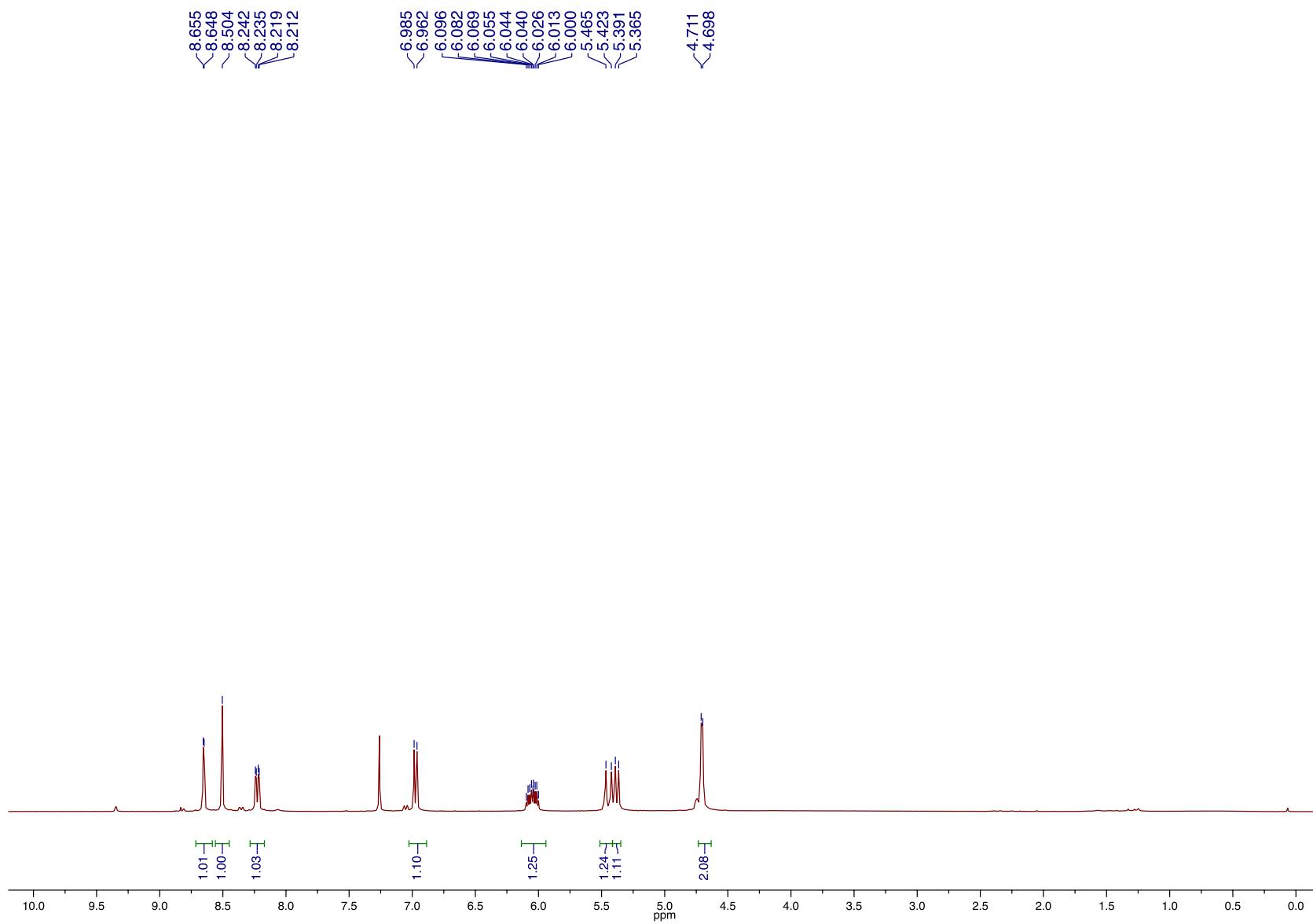
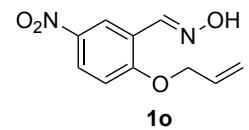
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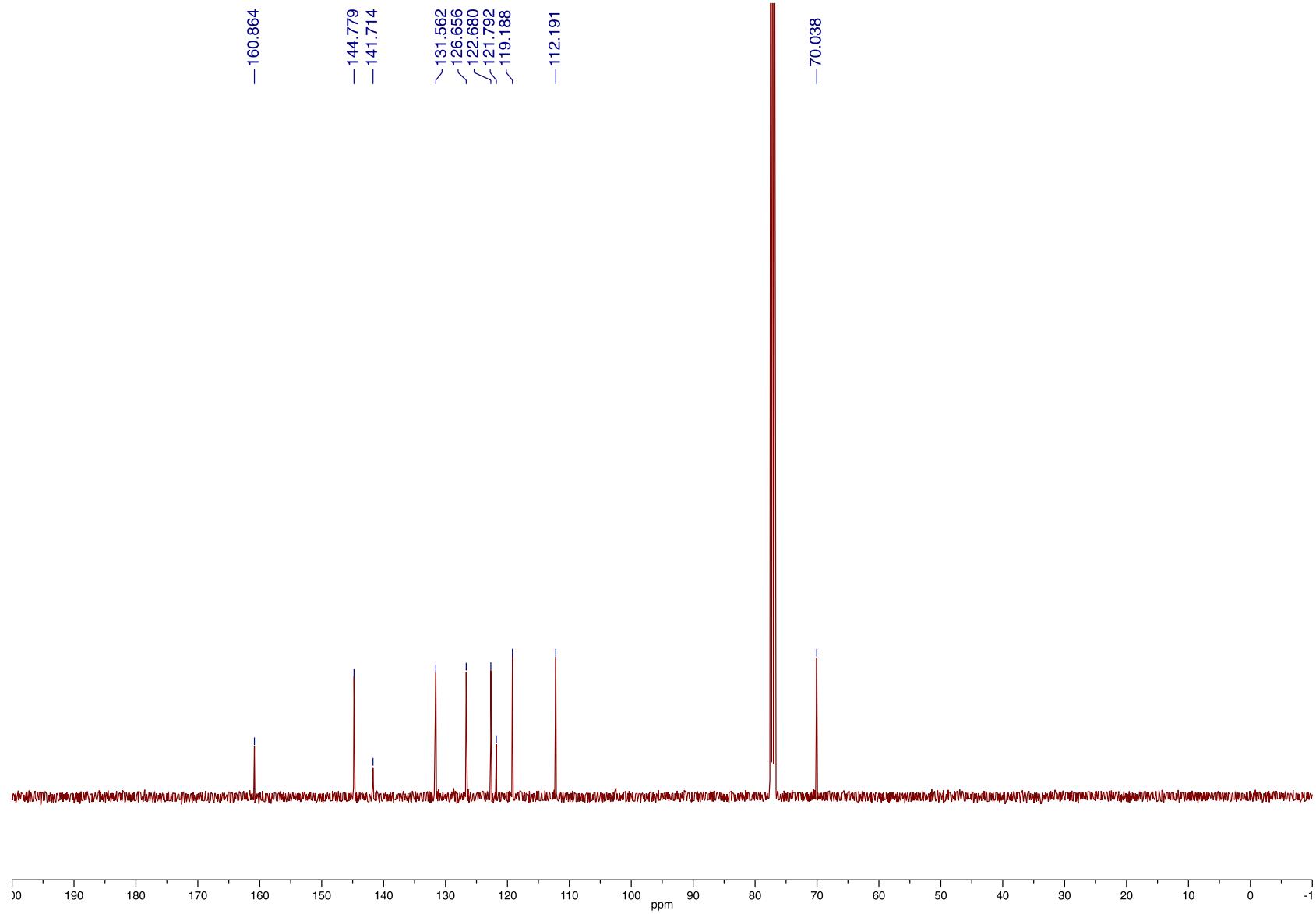
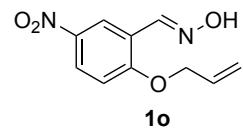
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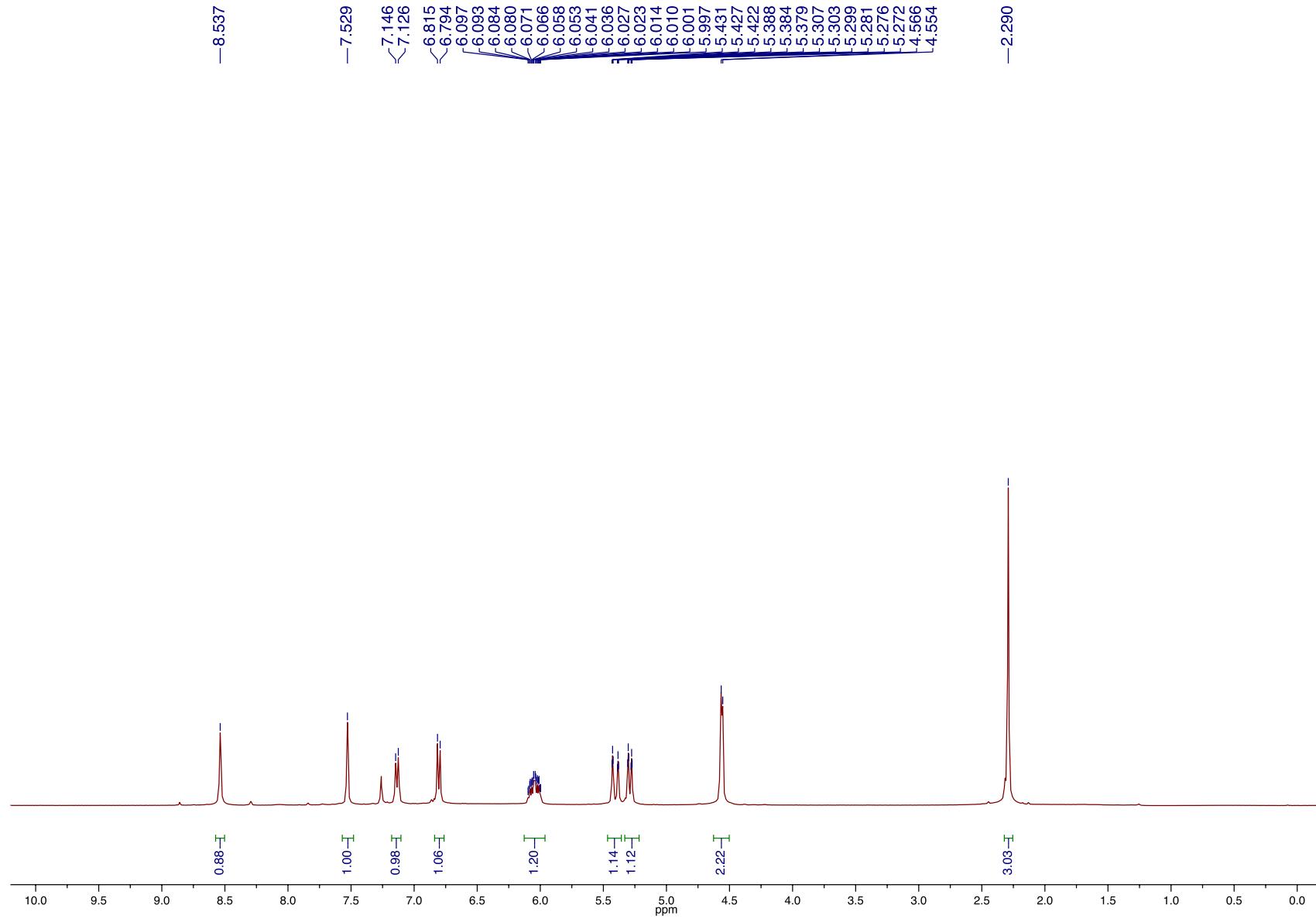
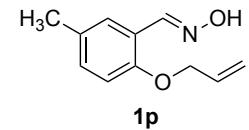
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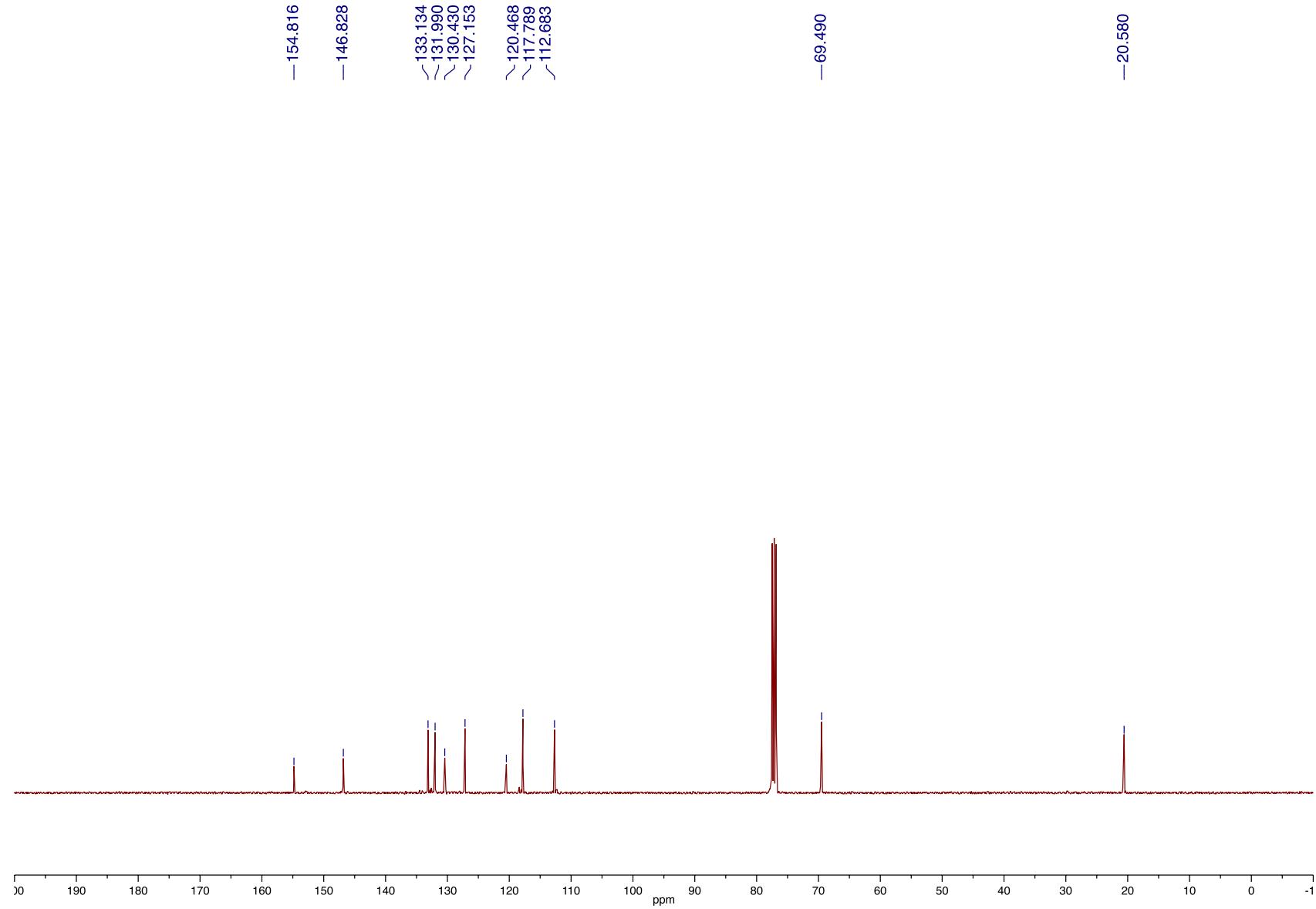
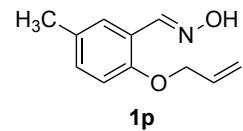
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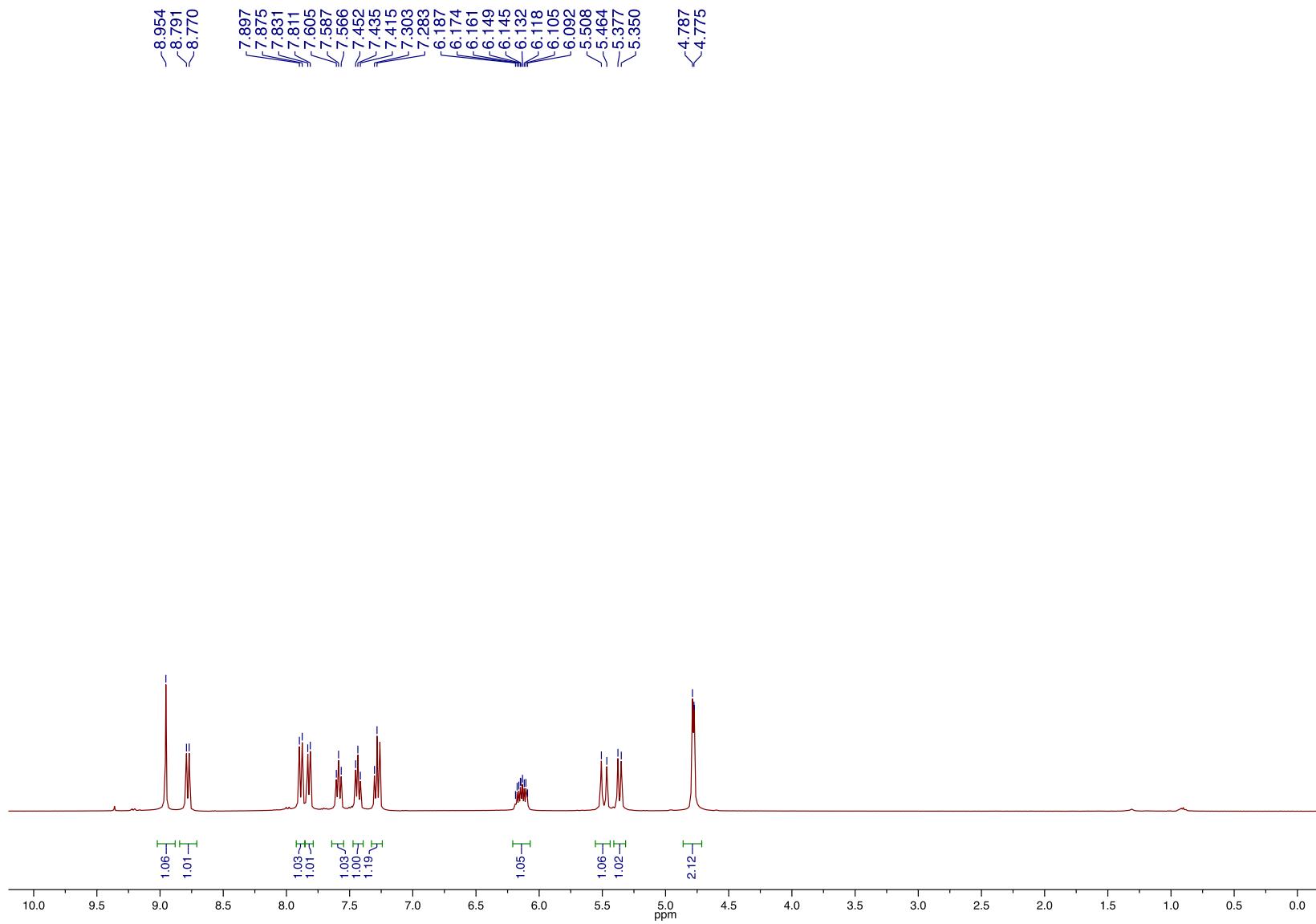
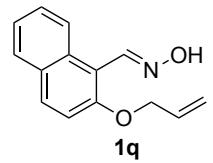
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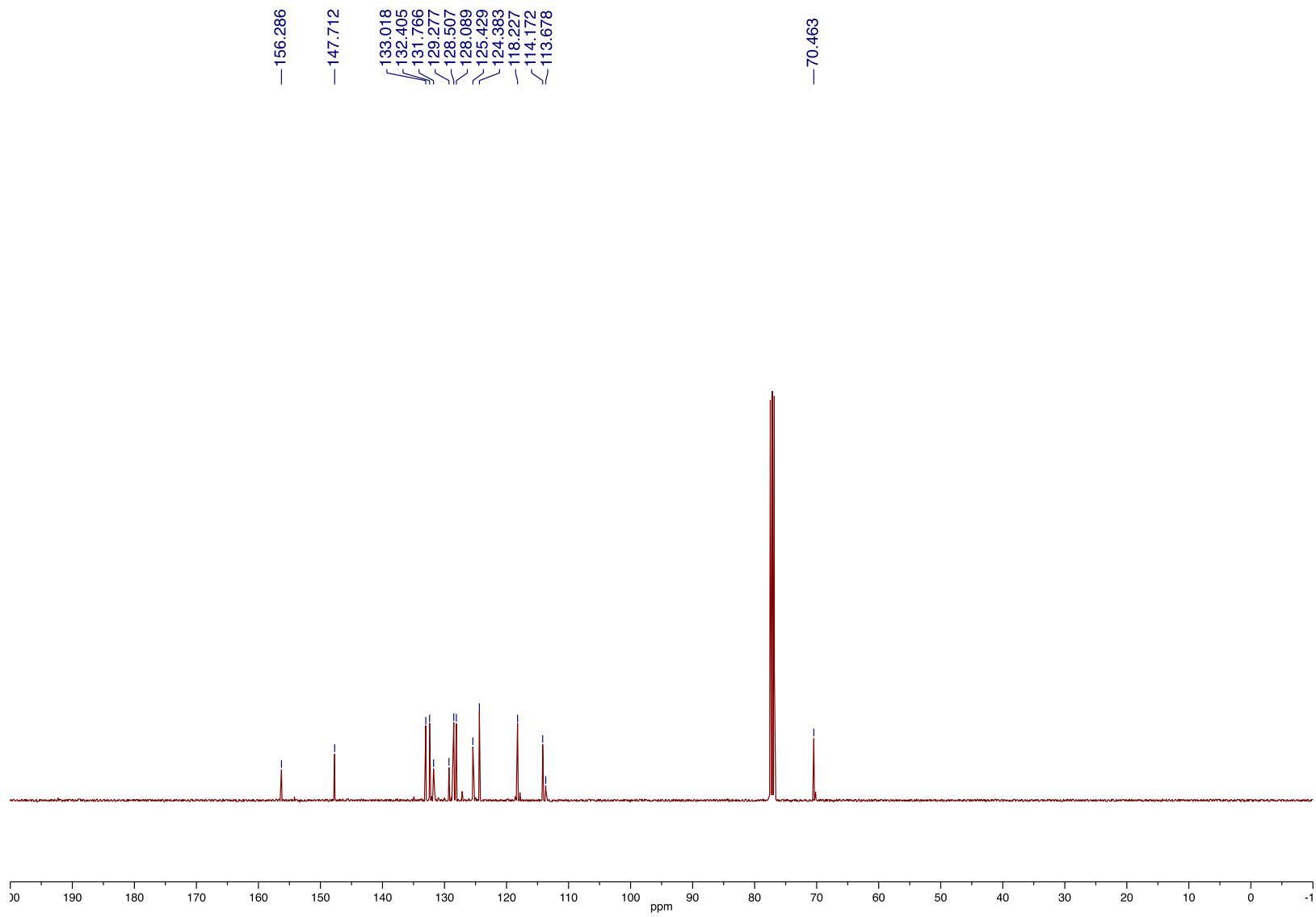
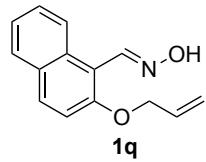
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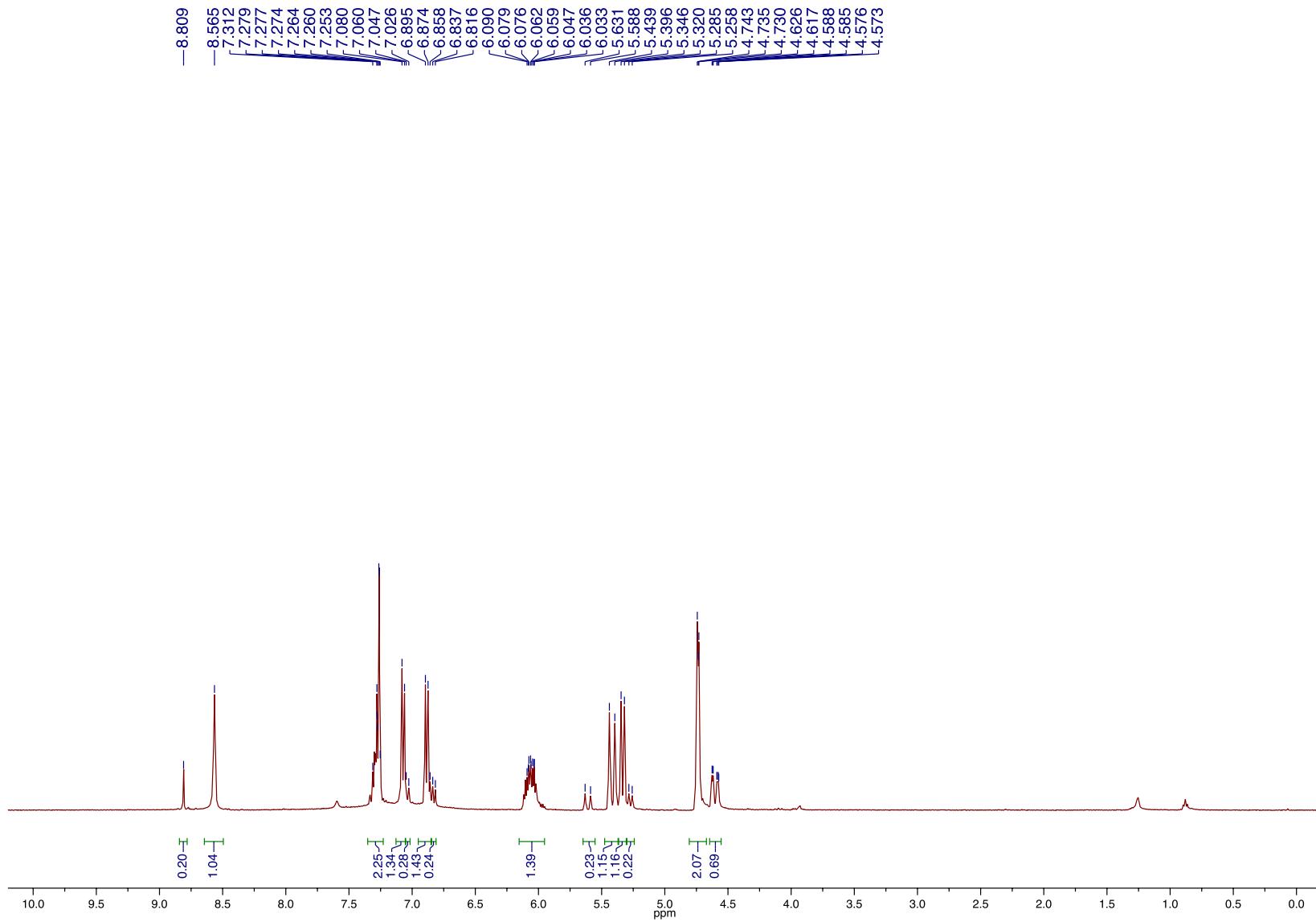
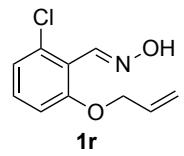
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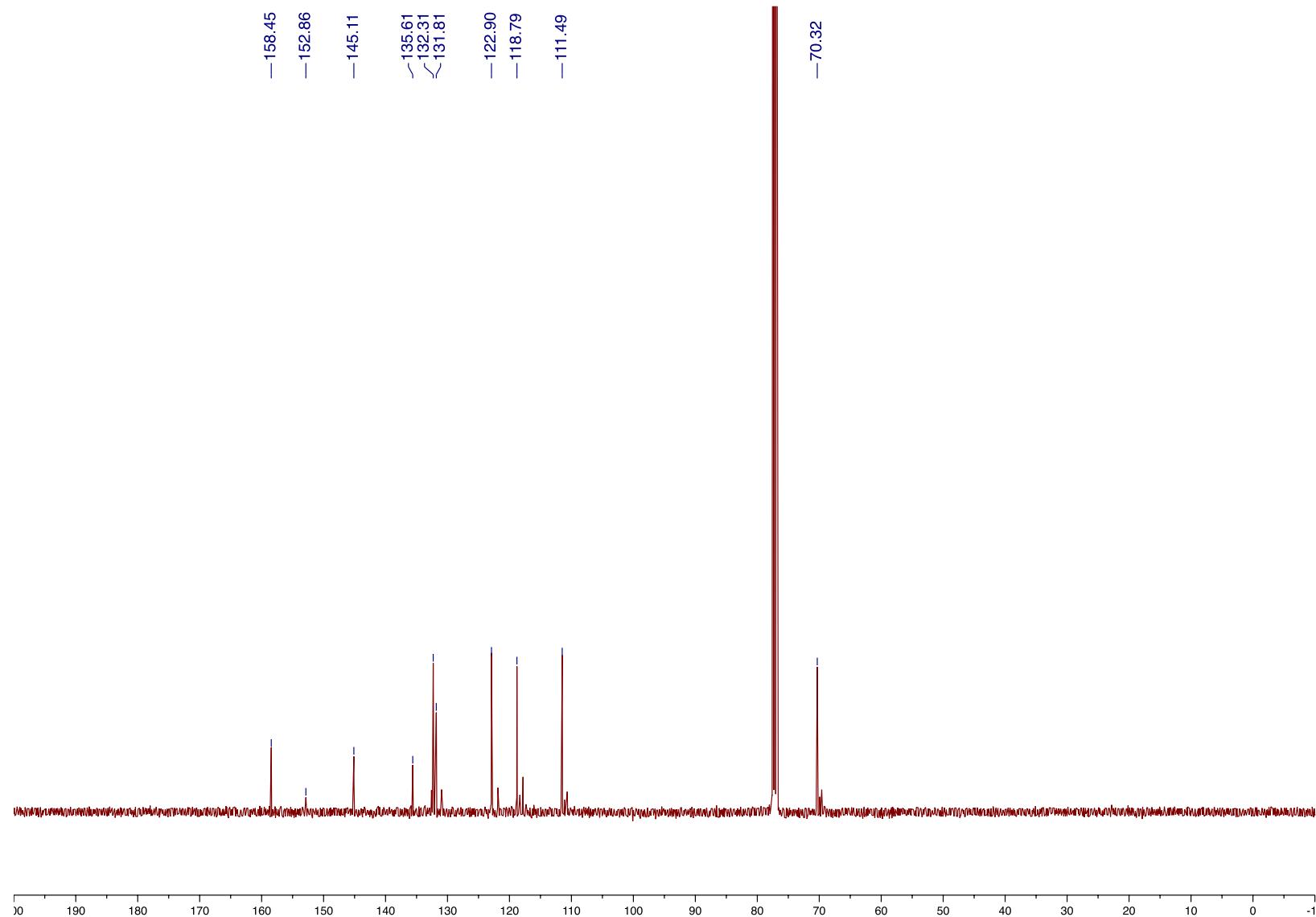
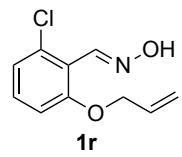
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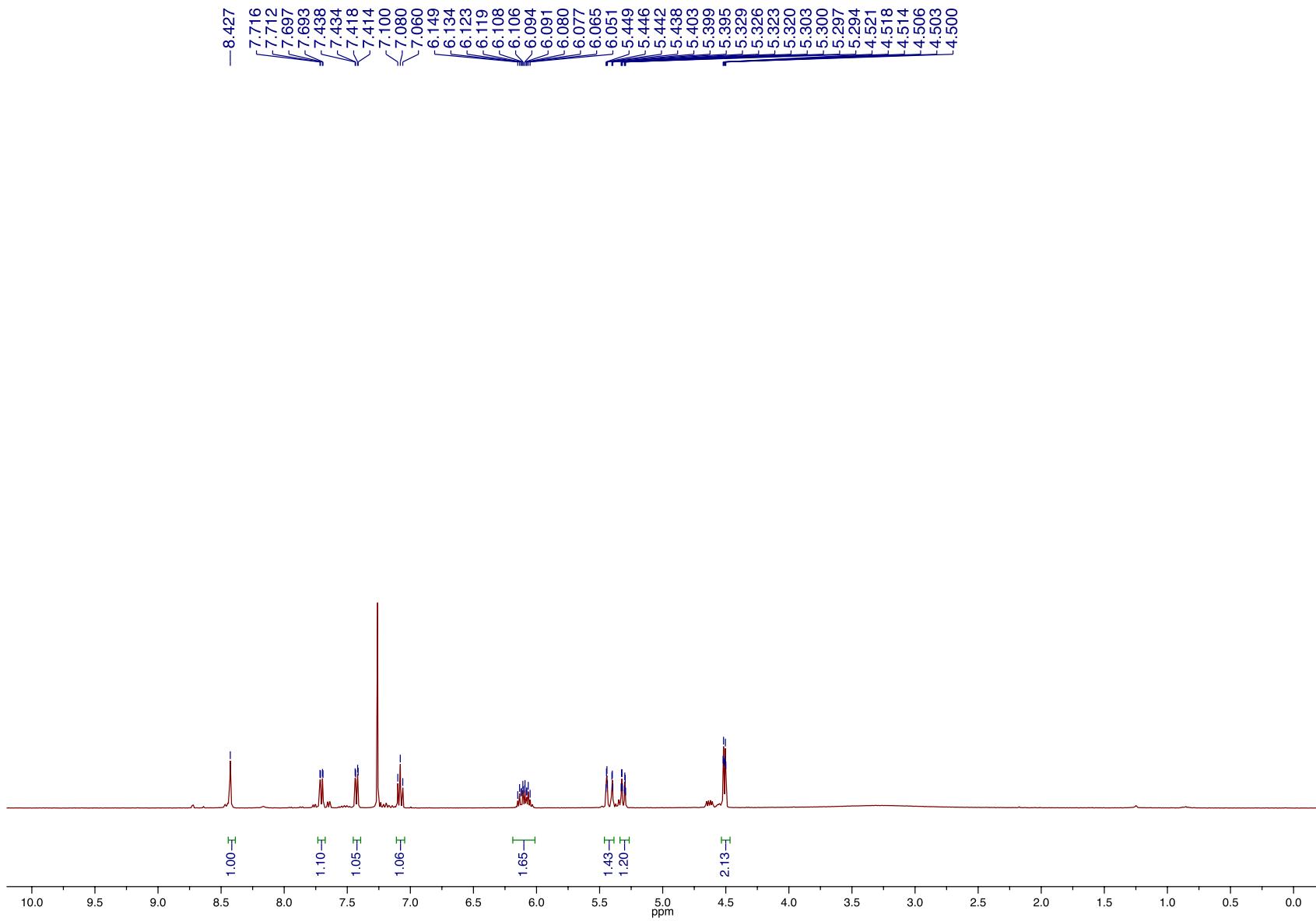
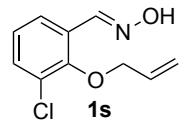
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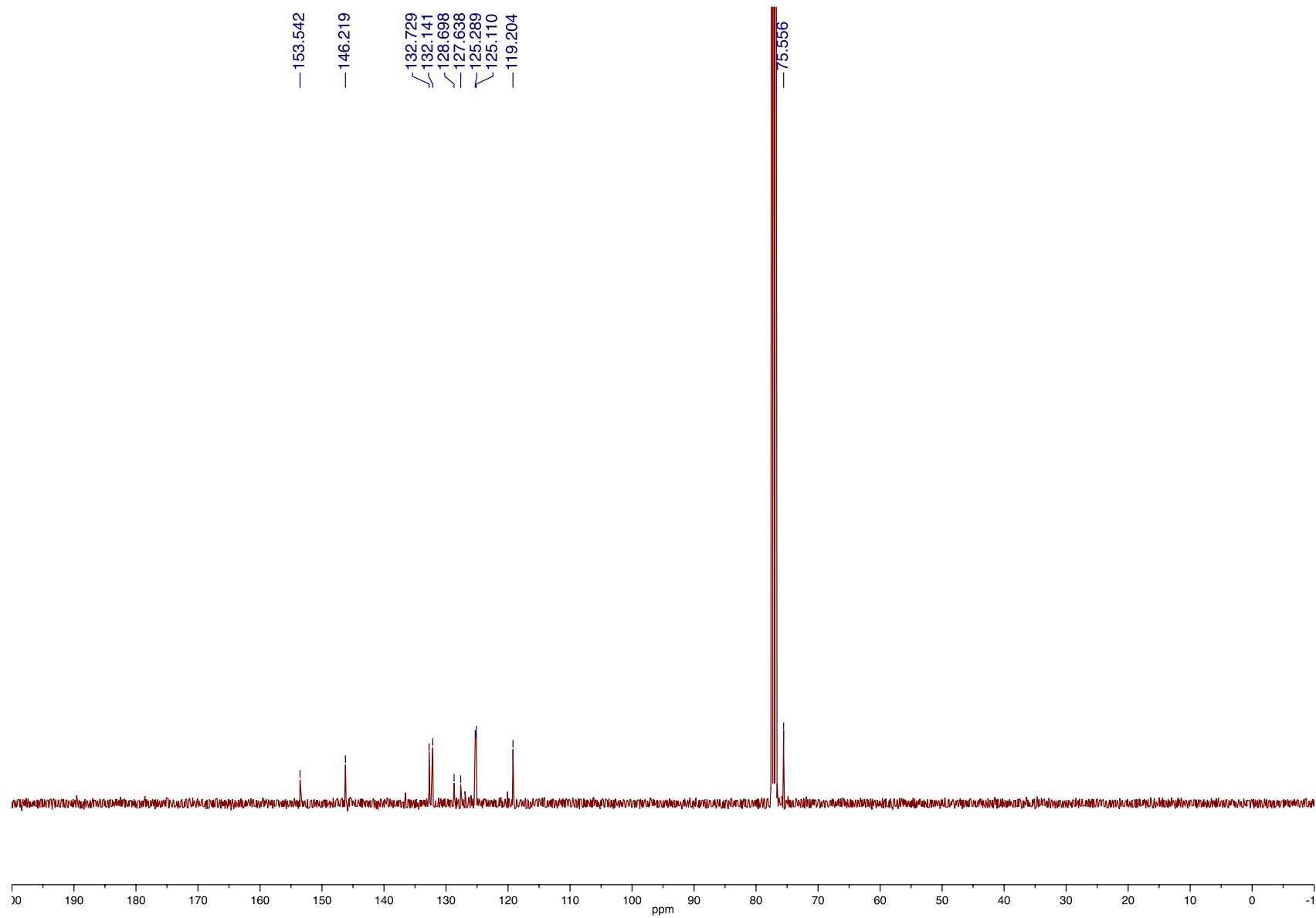
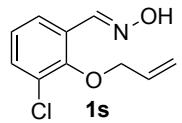
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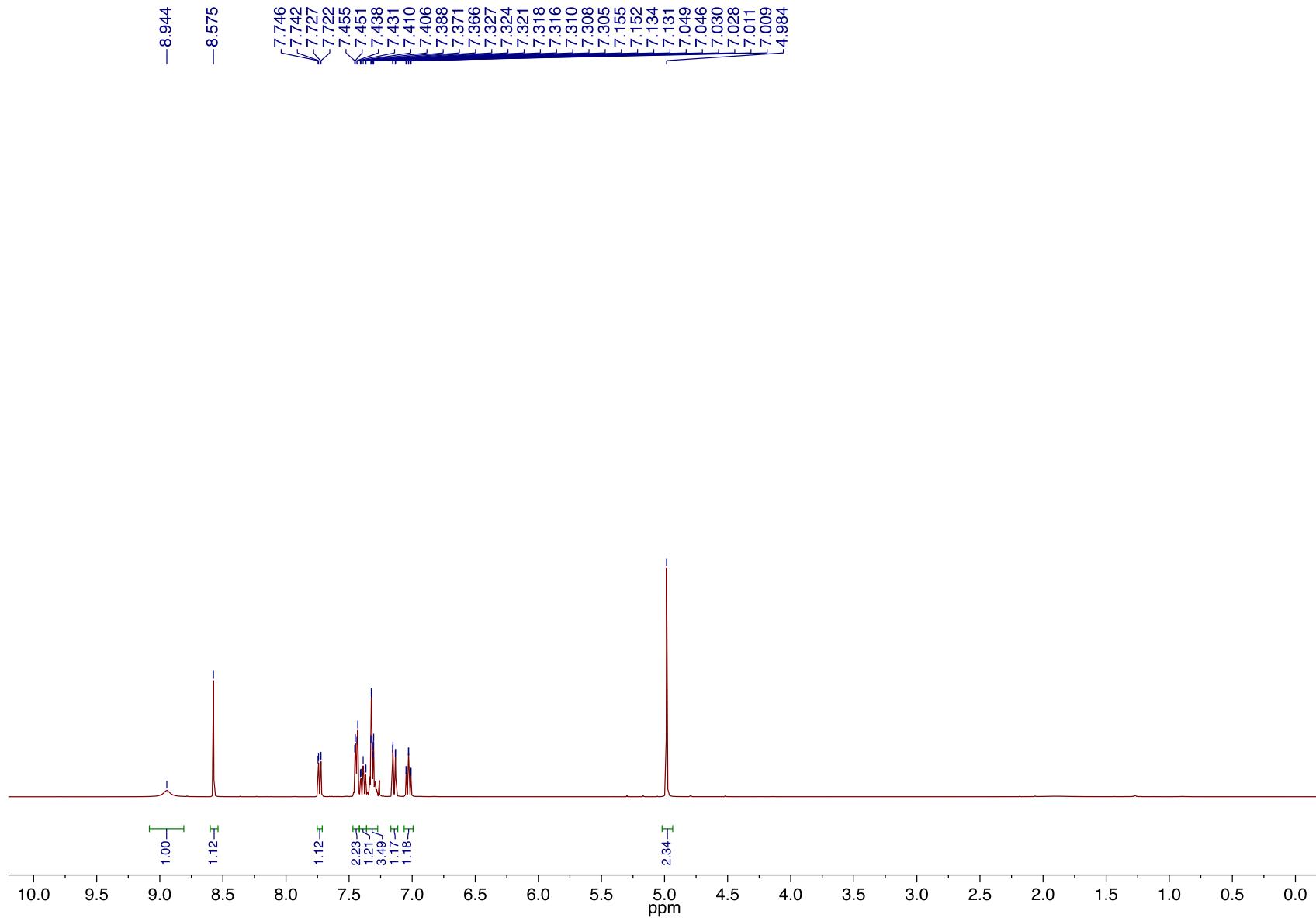
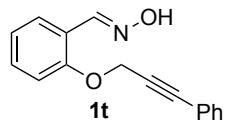
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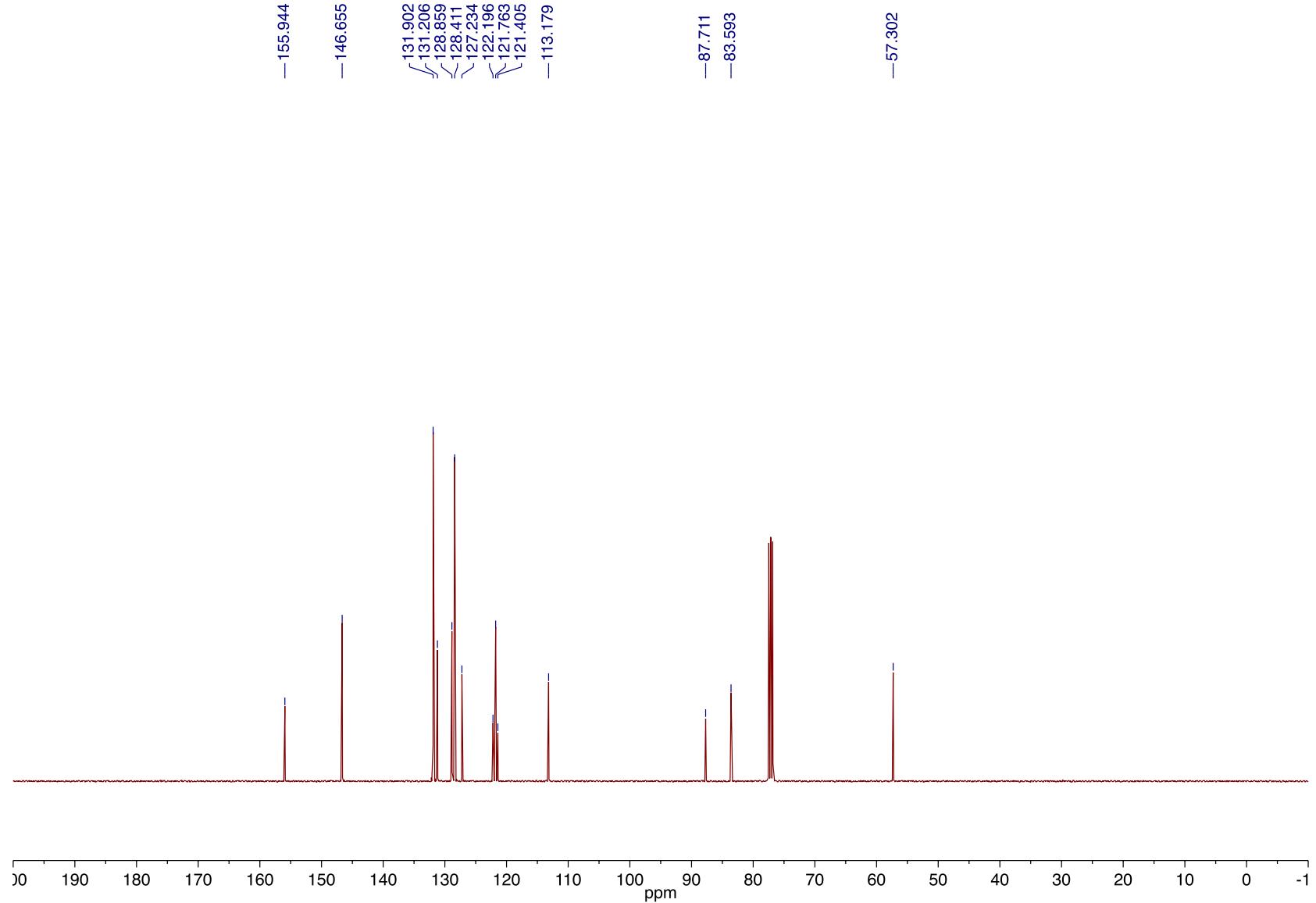
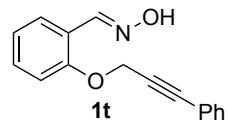
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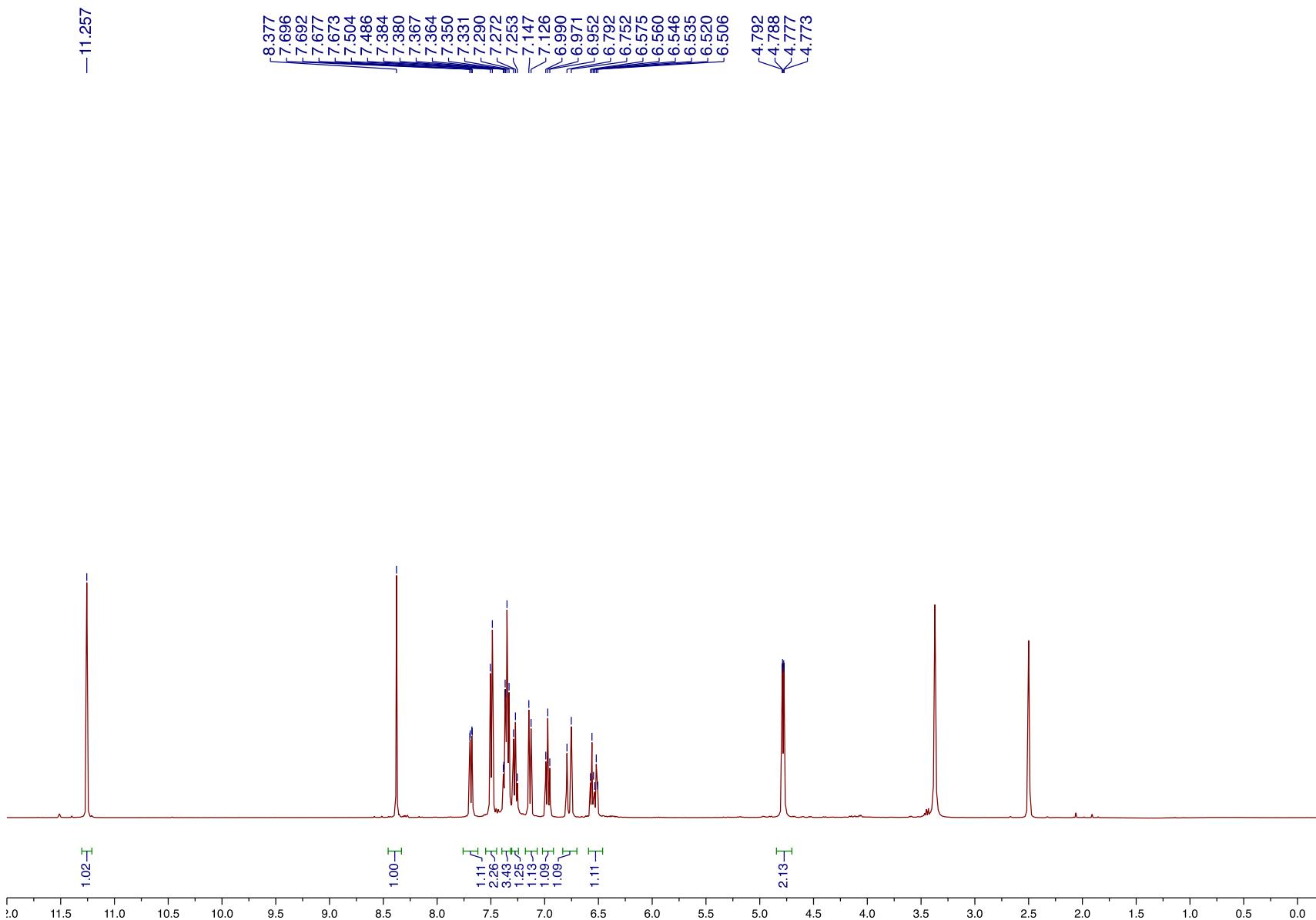
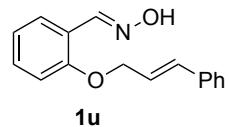
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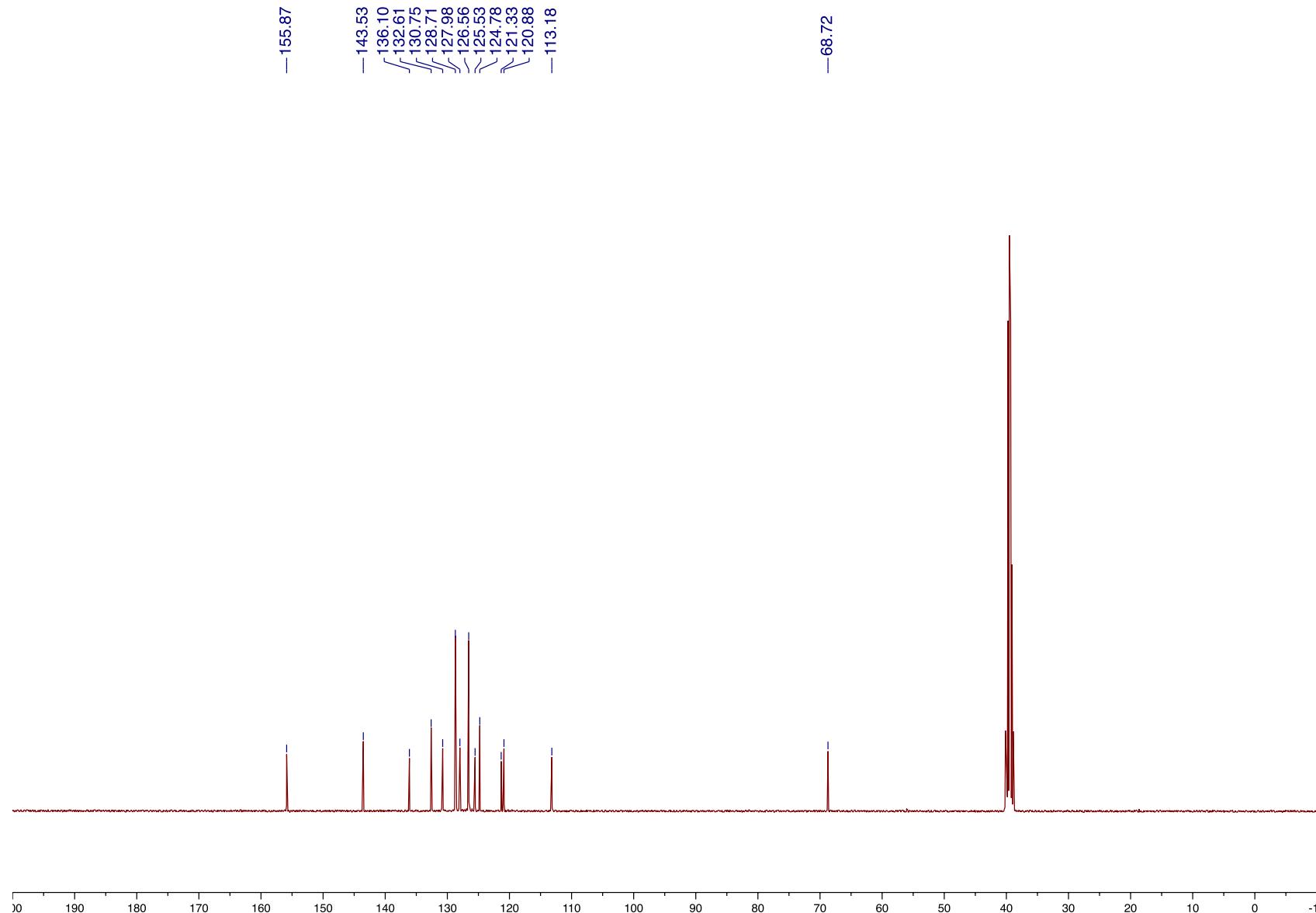
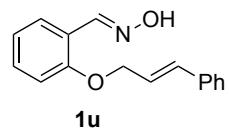
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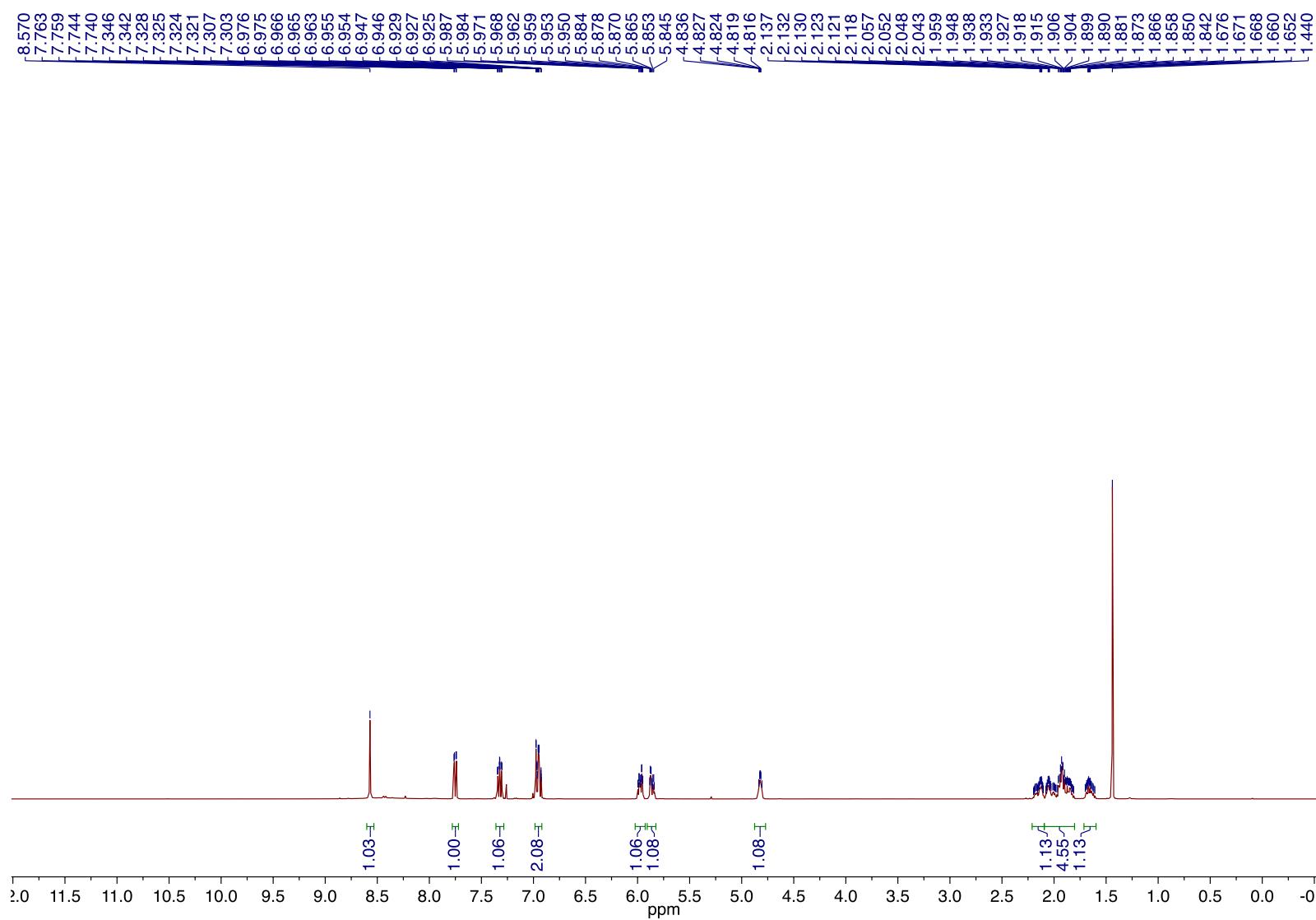
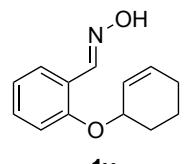
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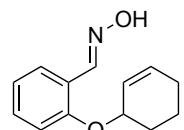
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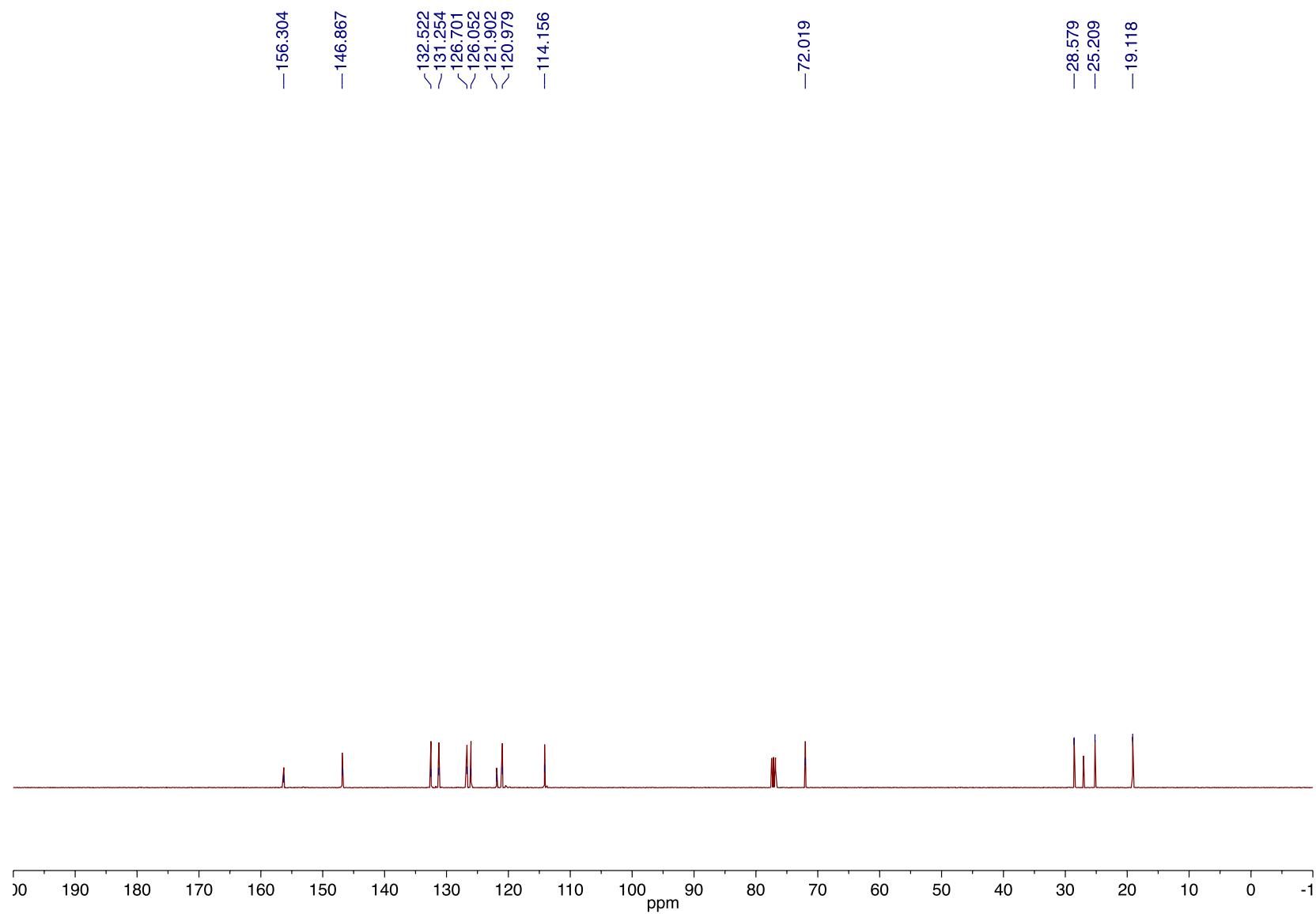
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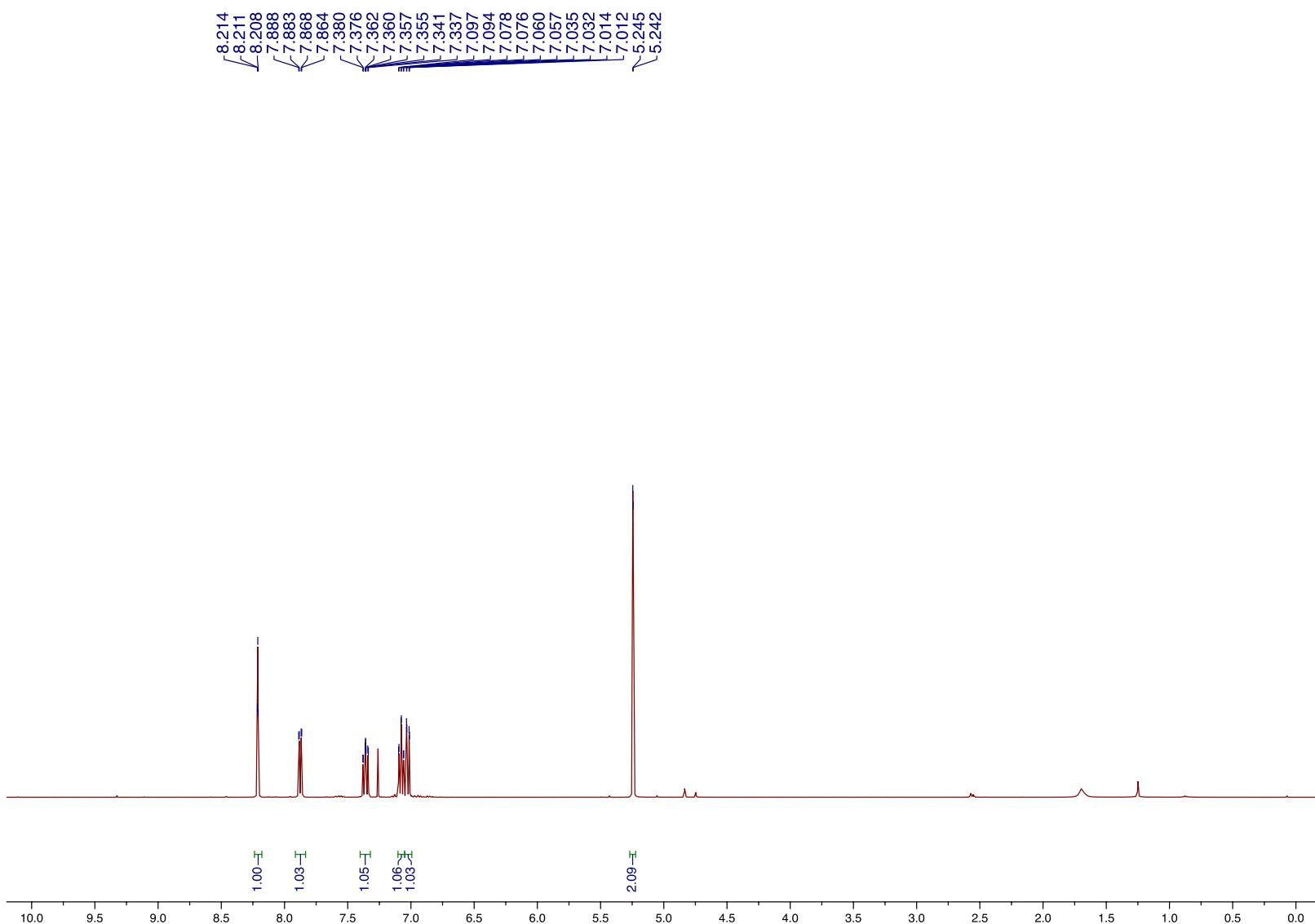
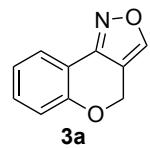
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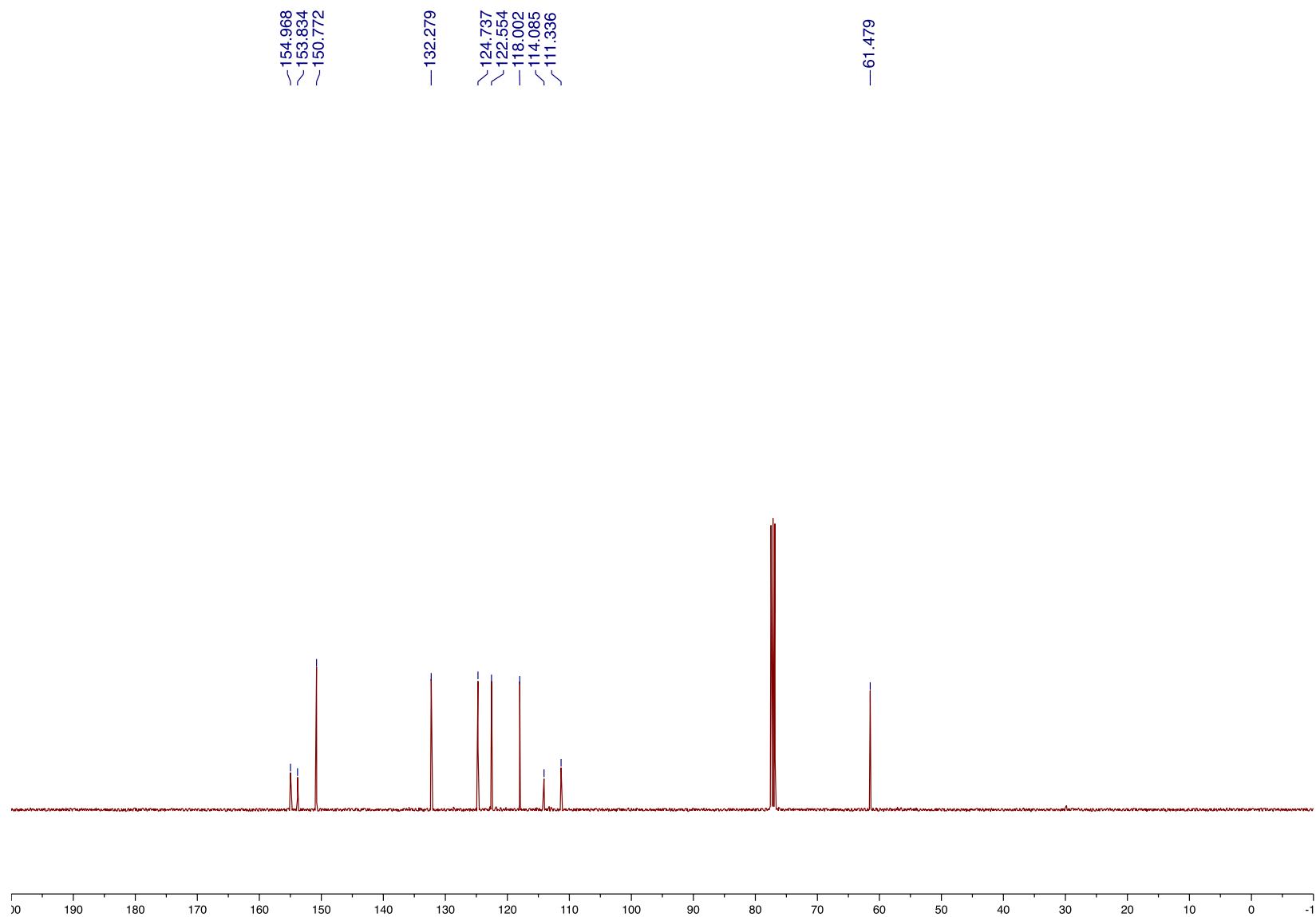
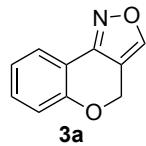
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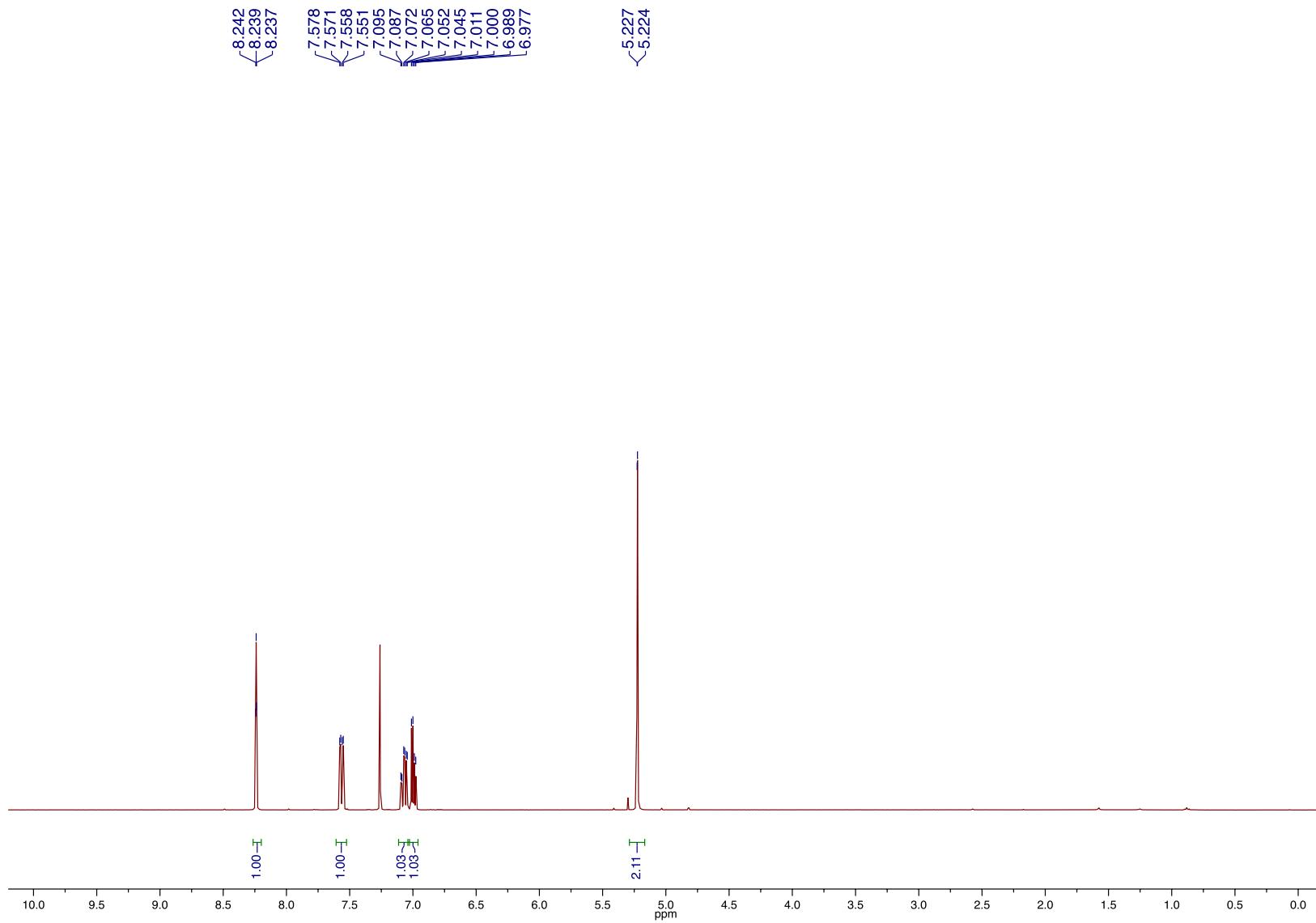
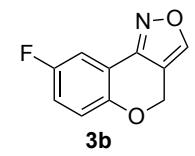
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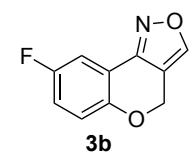
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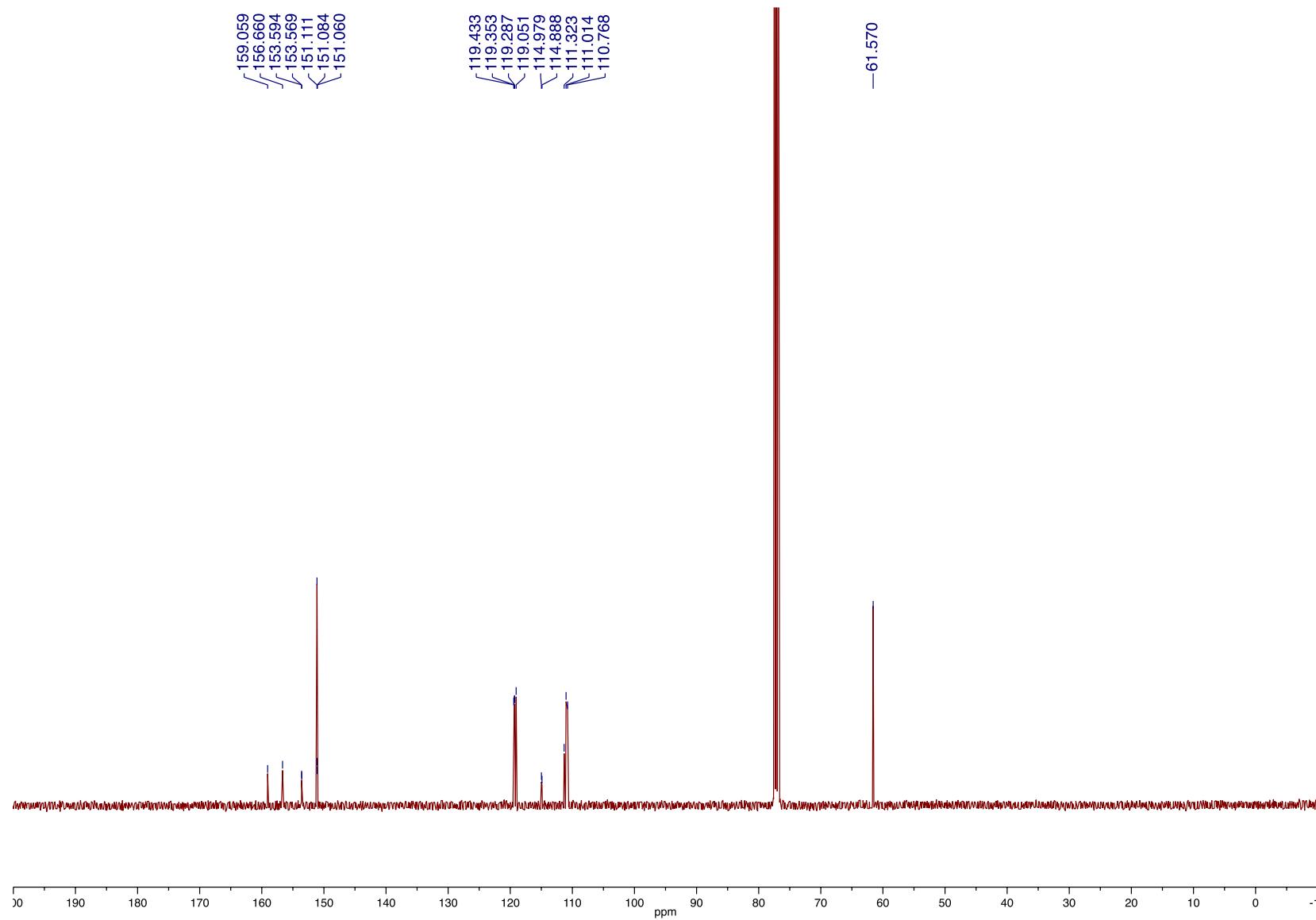
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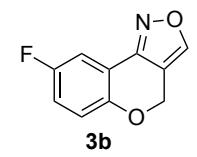
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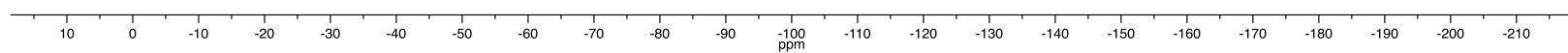
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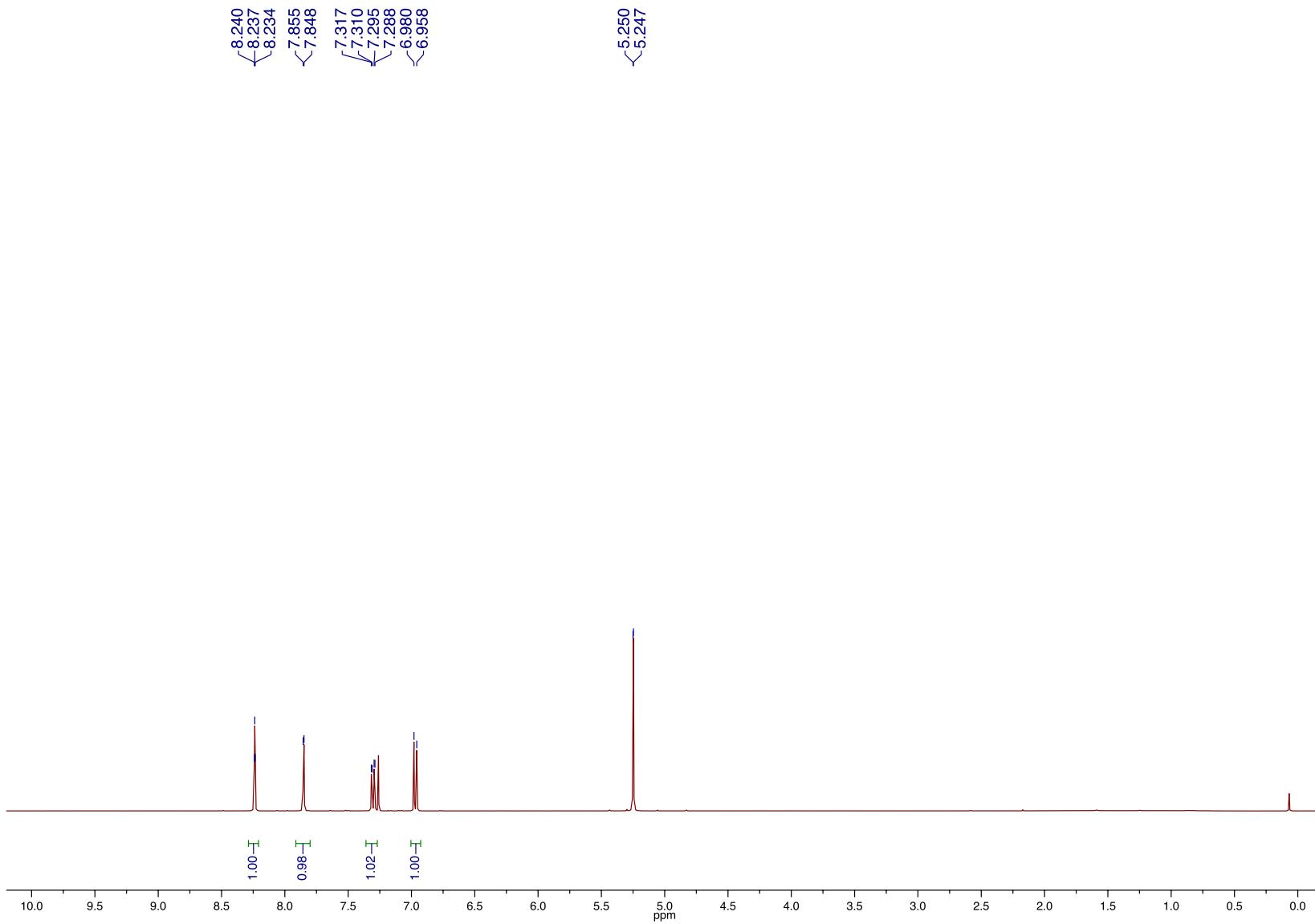
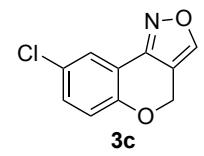
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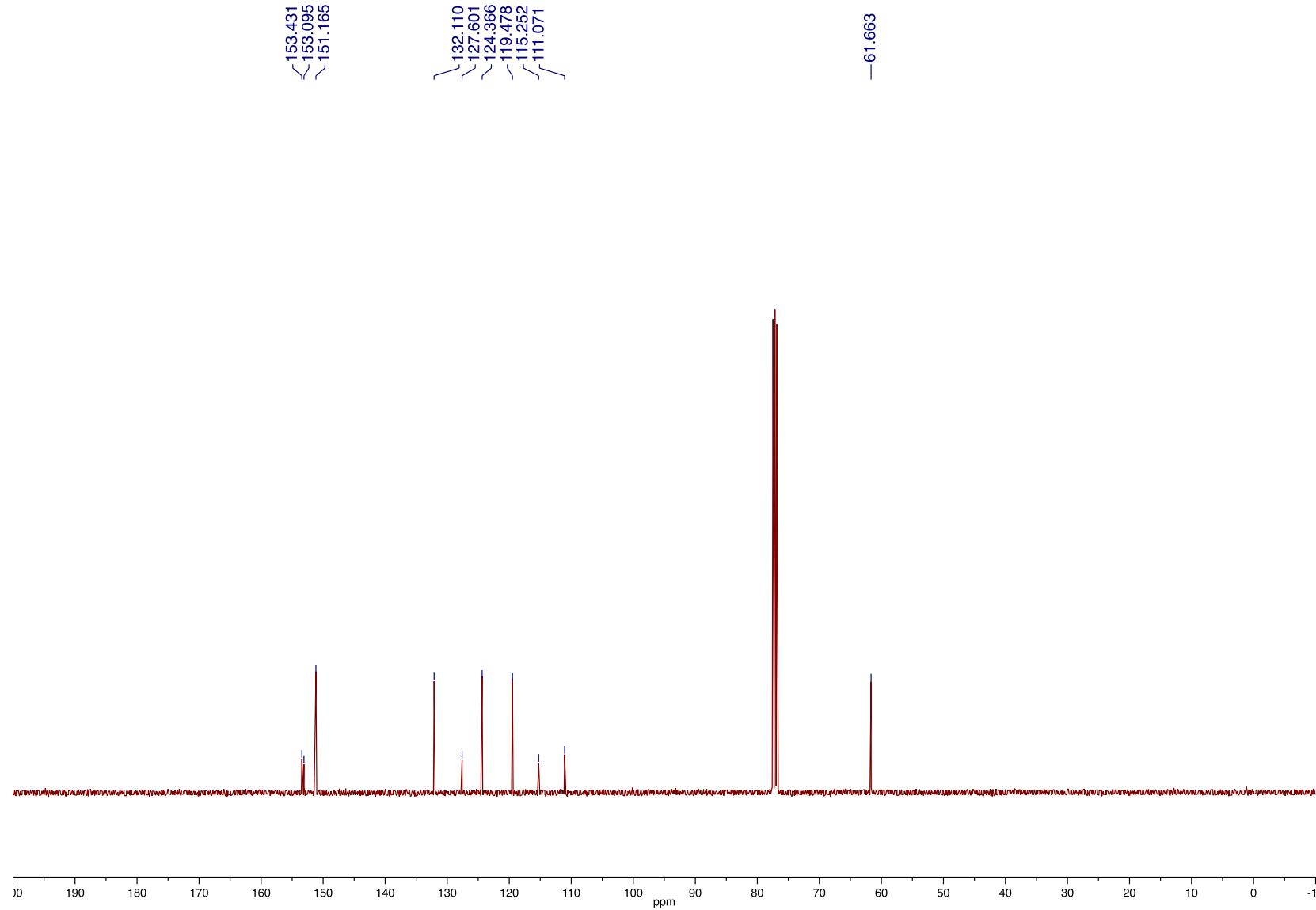
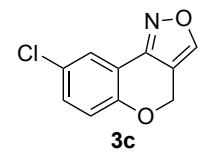
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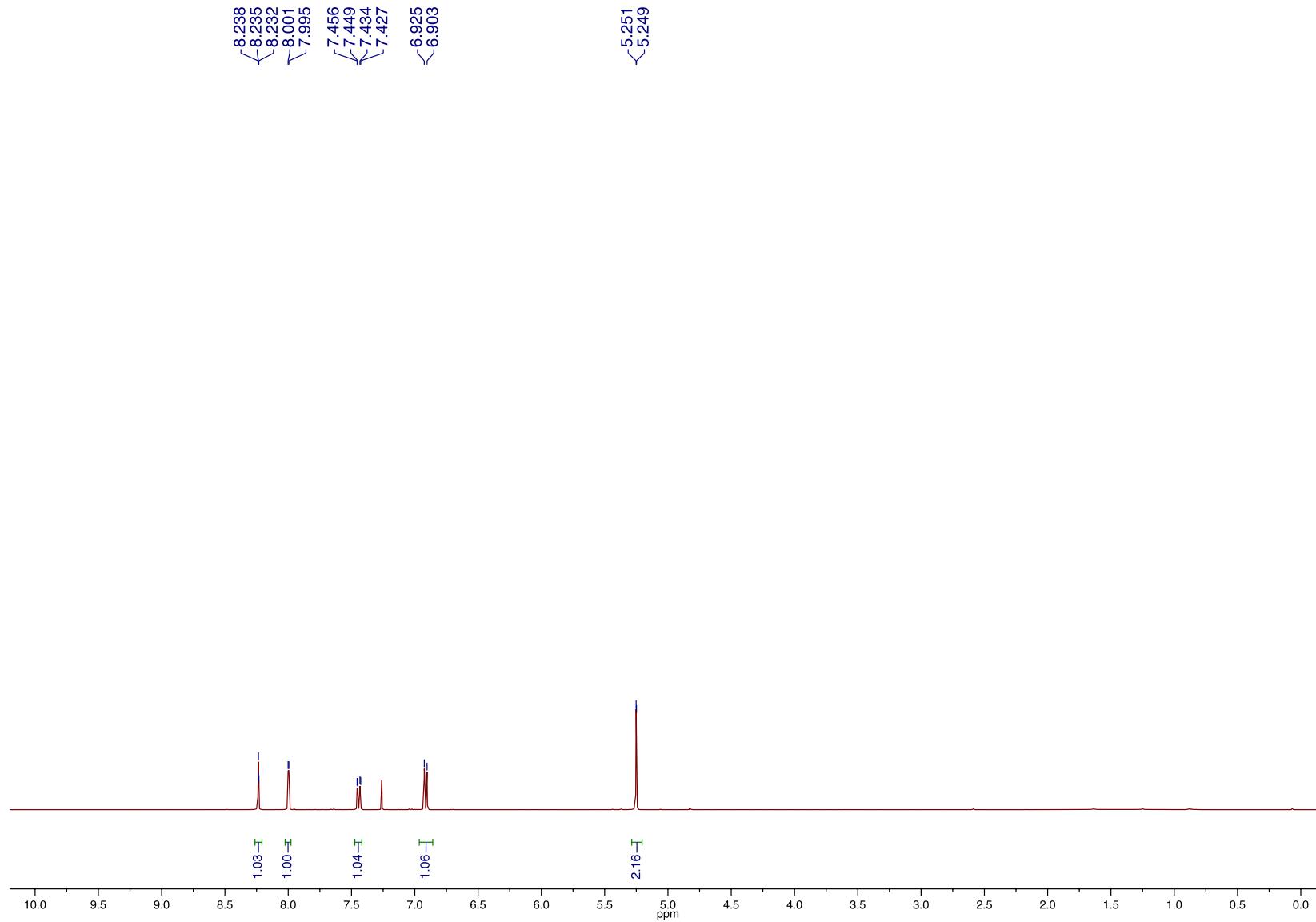
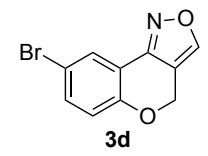
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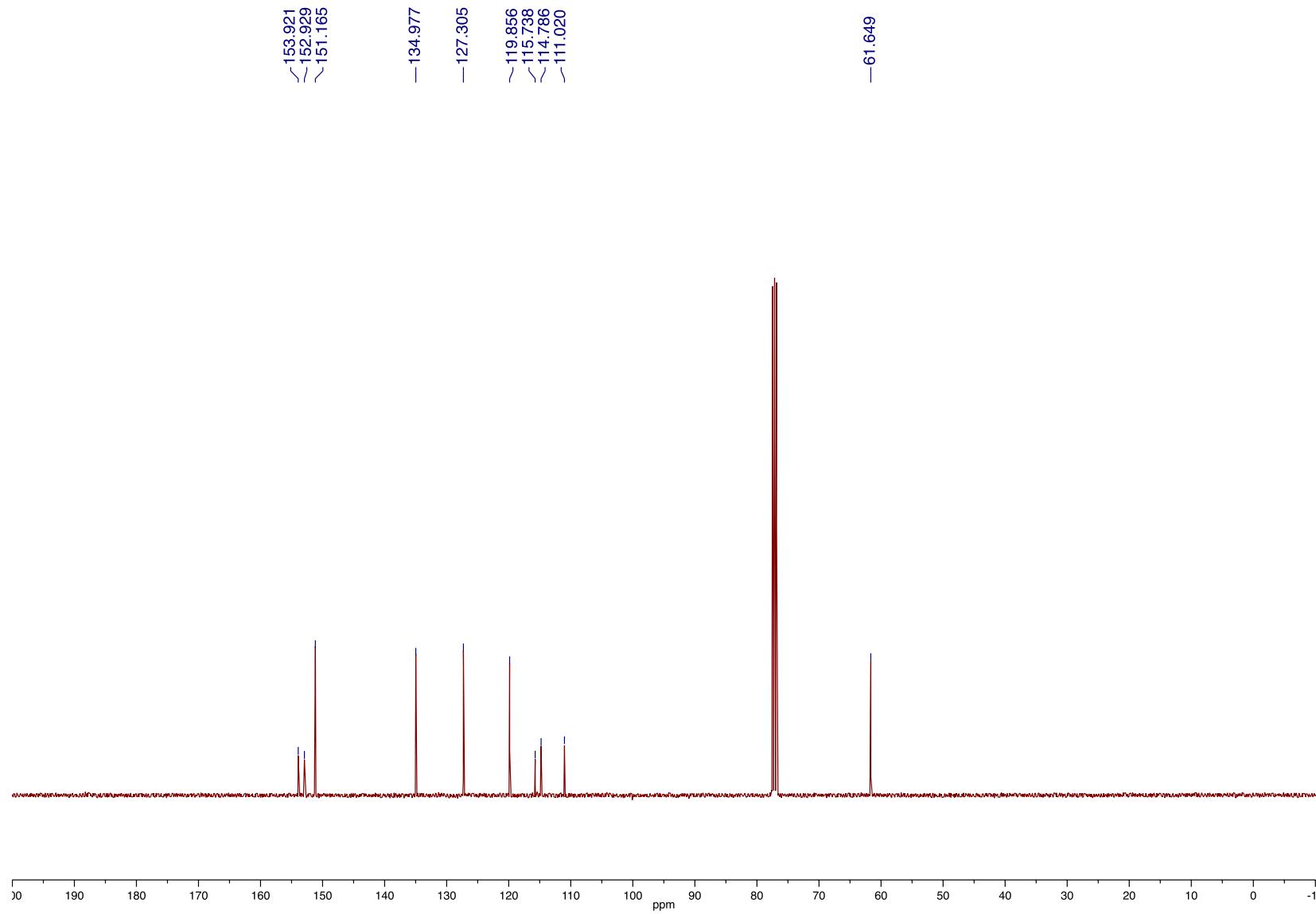
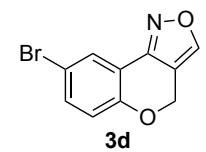
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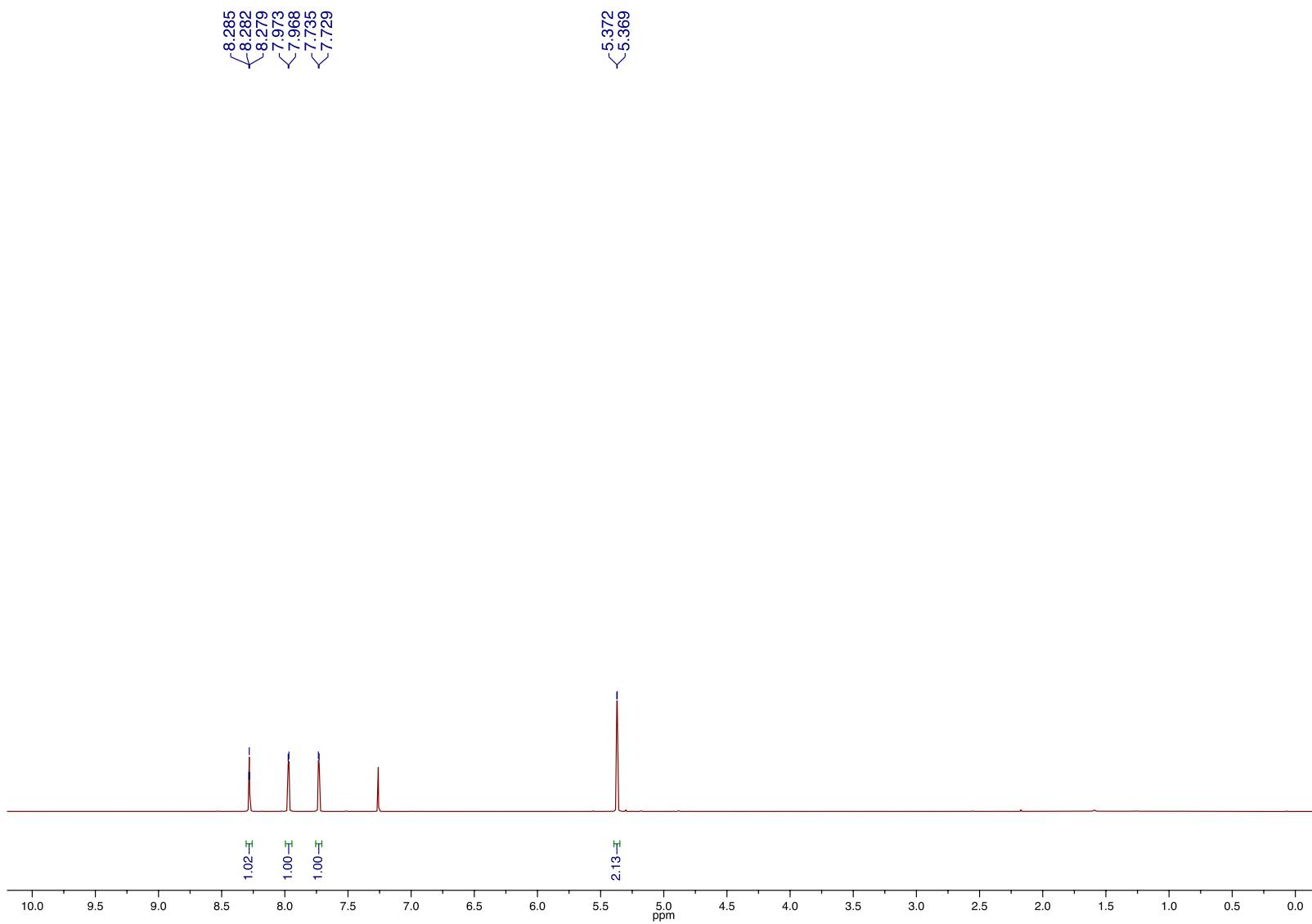
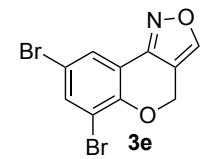
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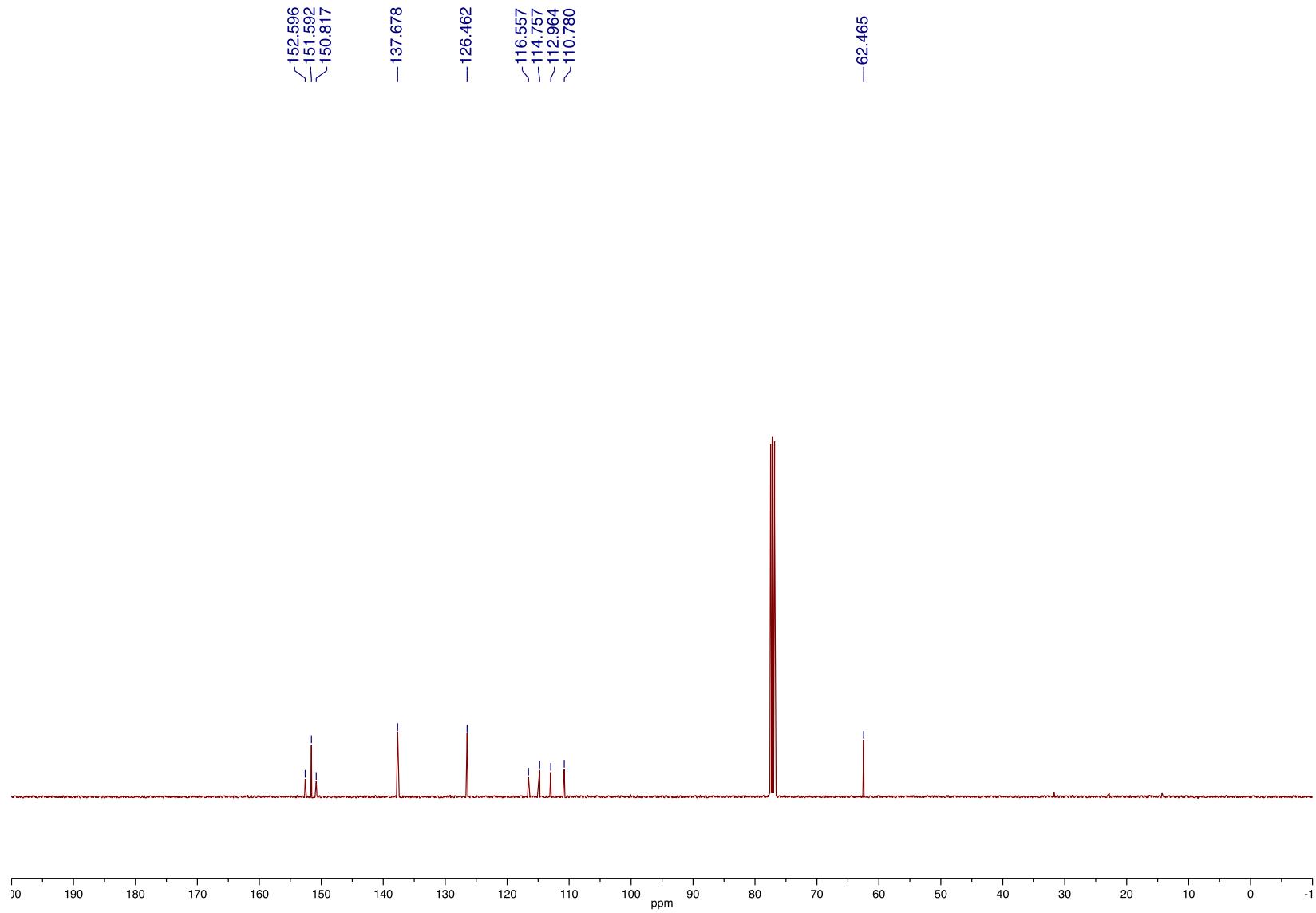
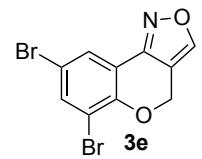
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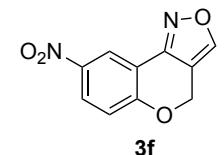
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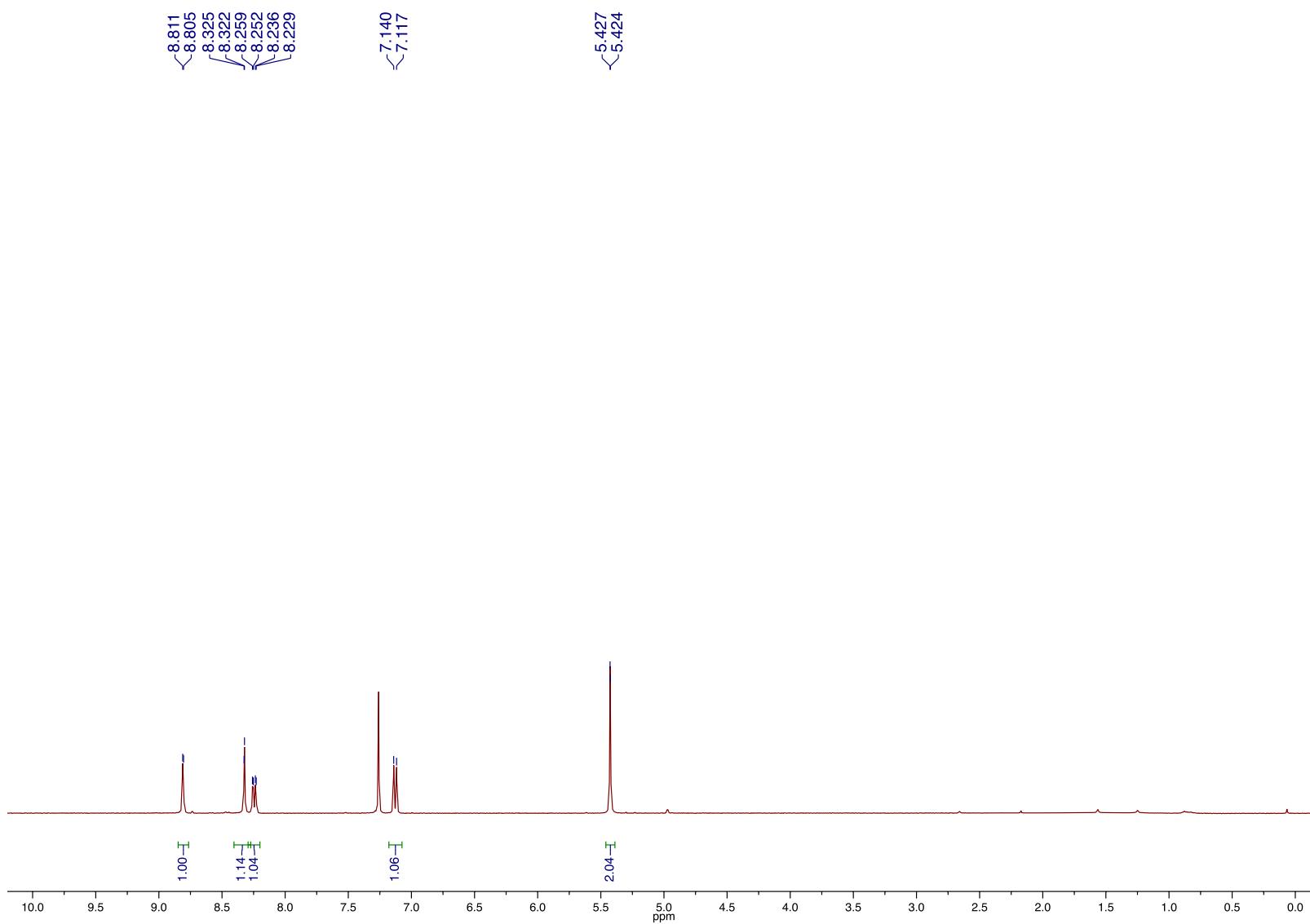
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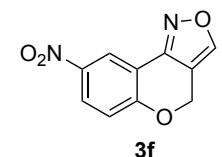
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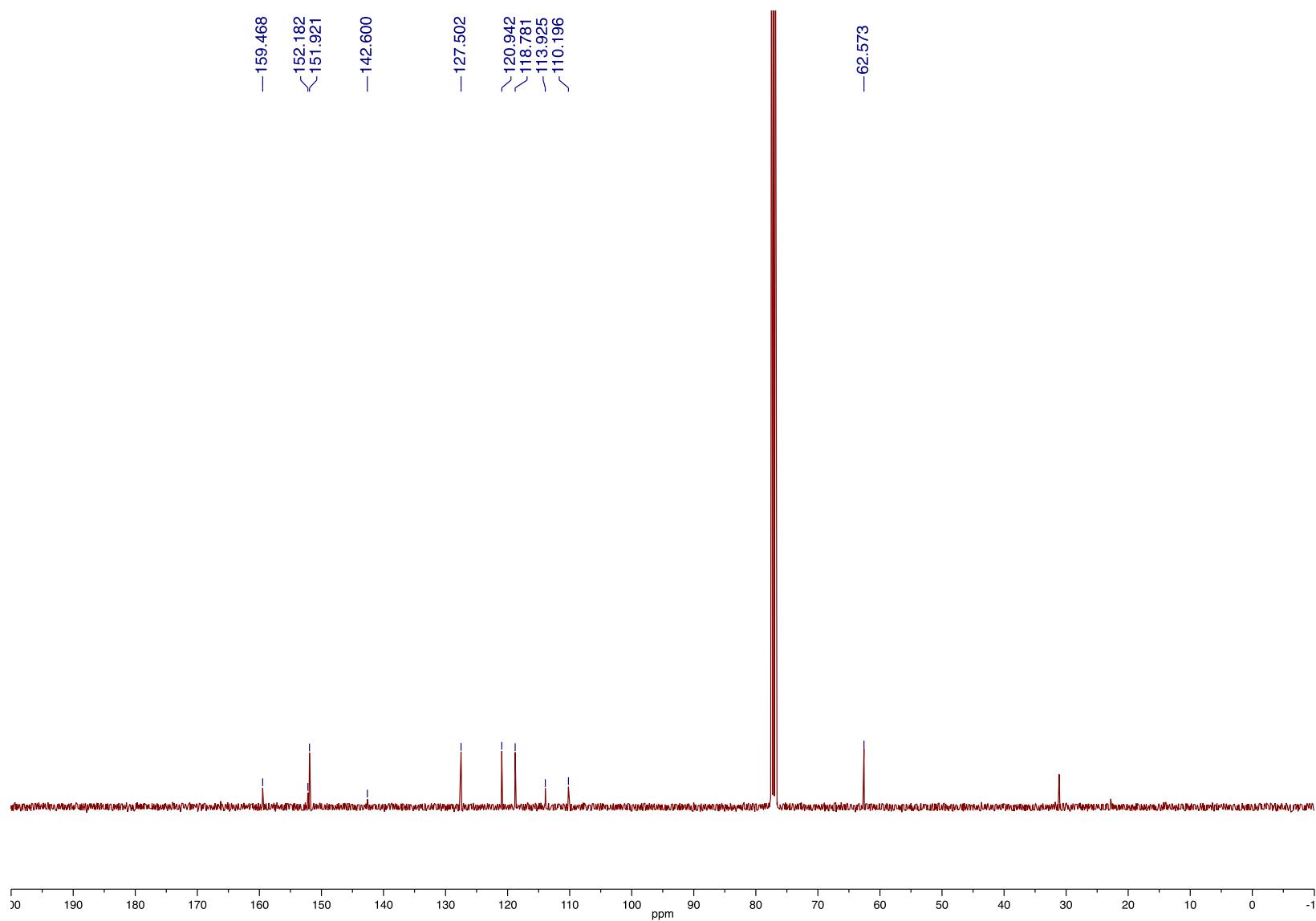
3f



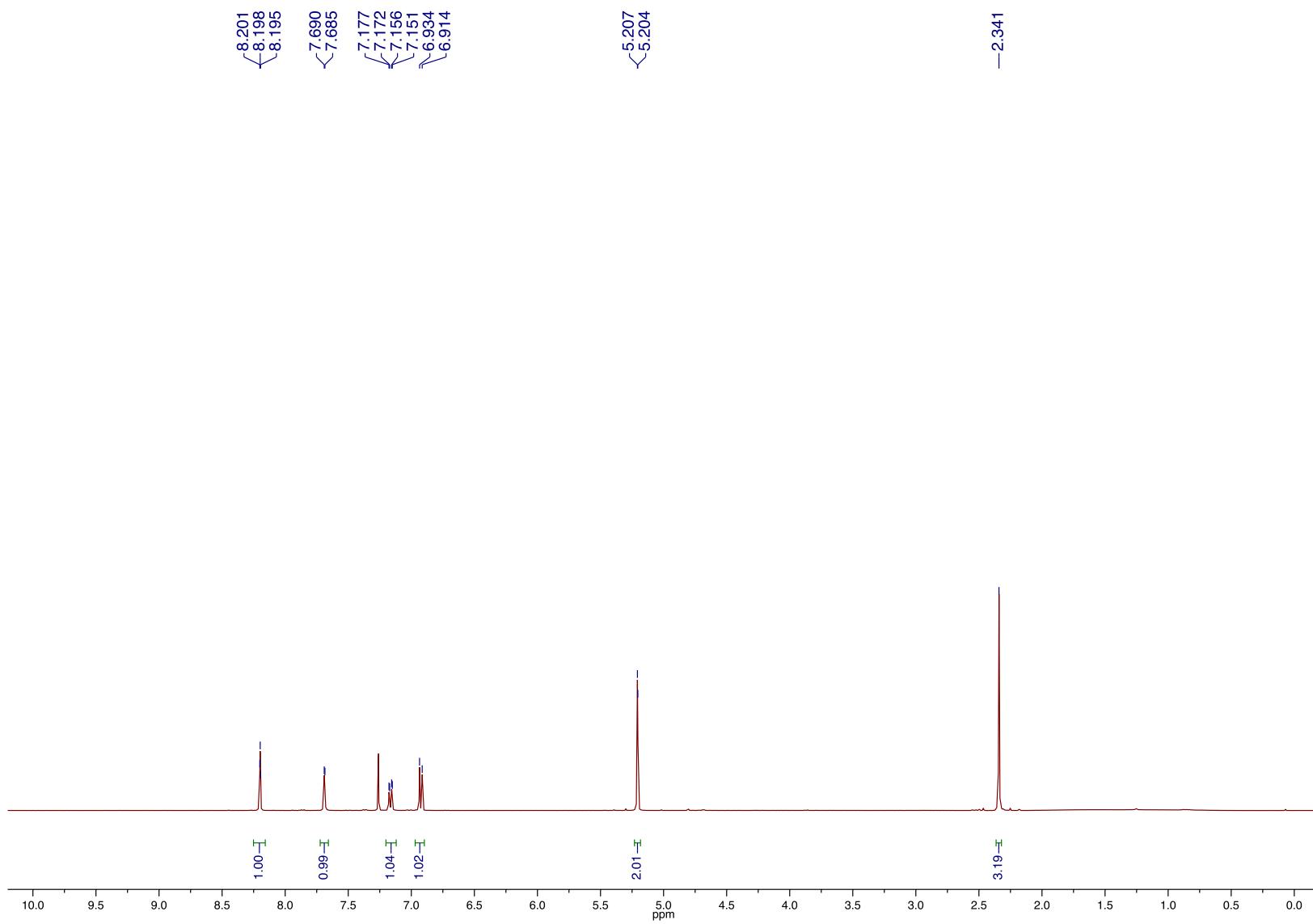
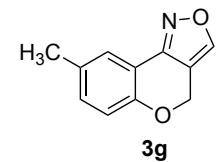
¹³C NMR (101 MHz, CDCl₃)



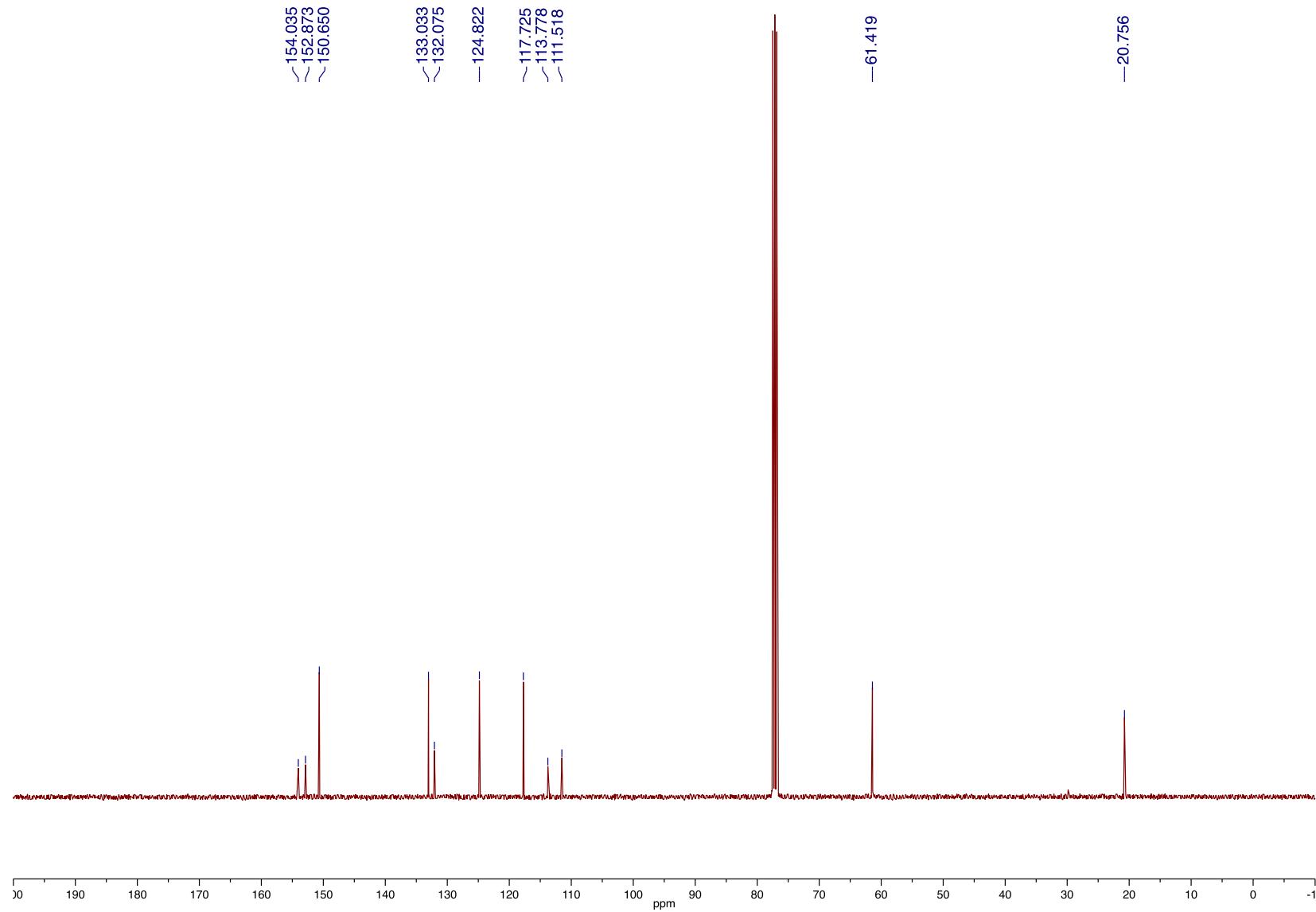
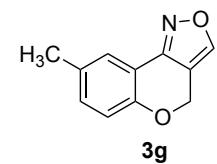
3f



¹H NMR (400 MHz, CDCl₃)



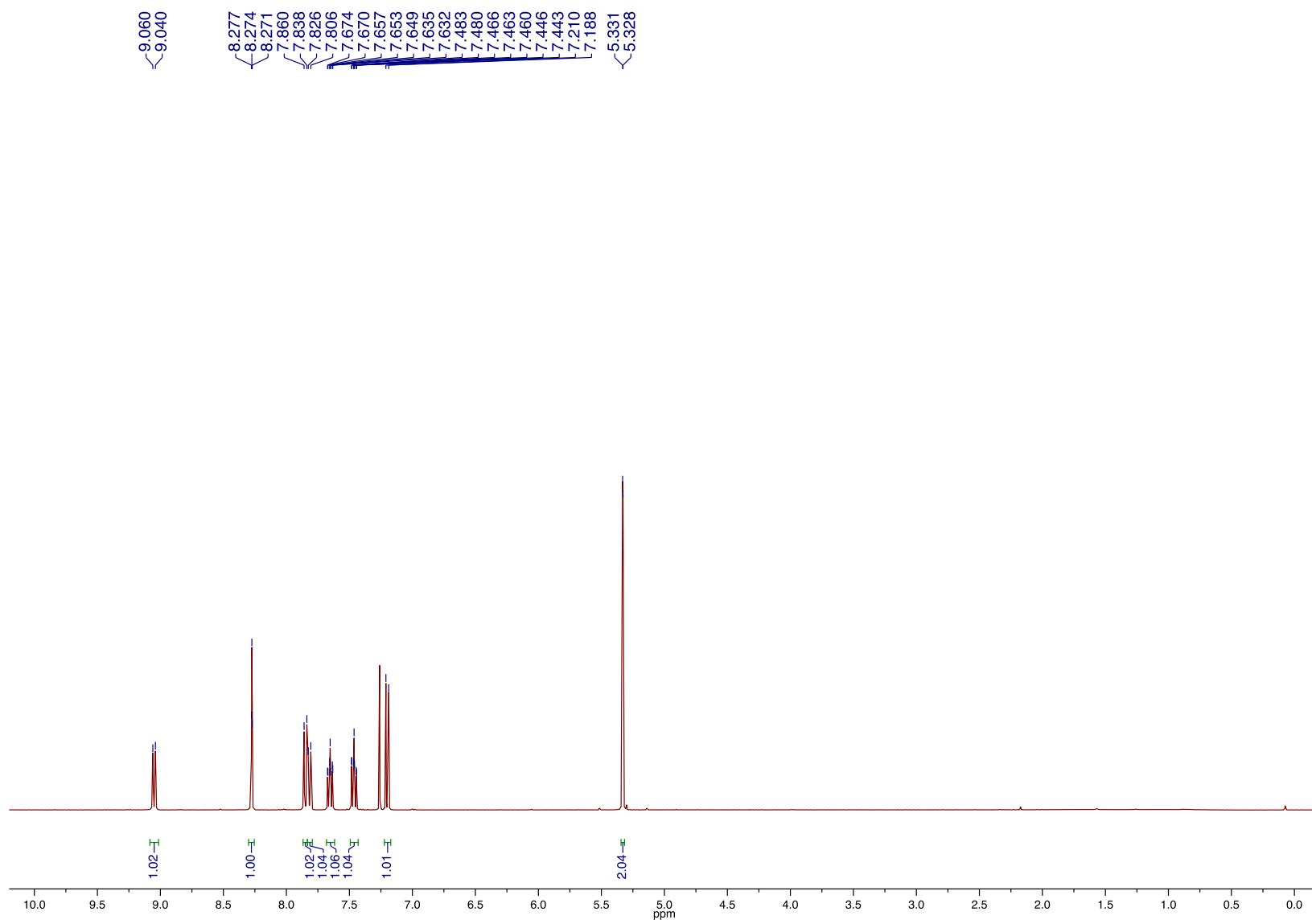
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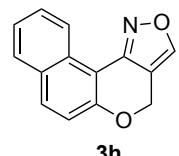
¹H NMR (400 MHz, CDCl₃)



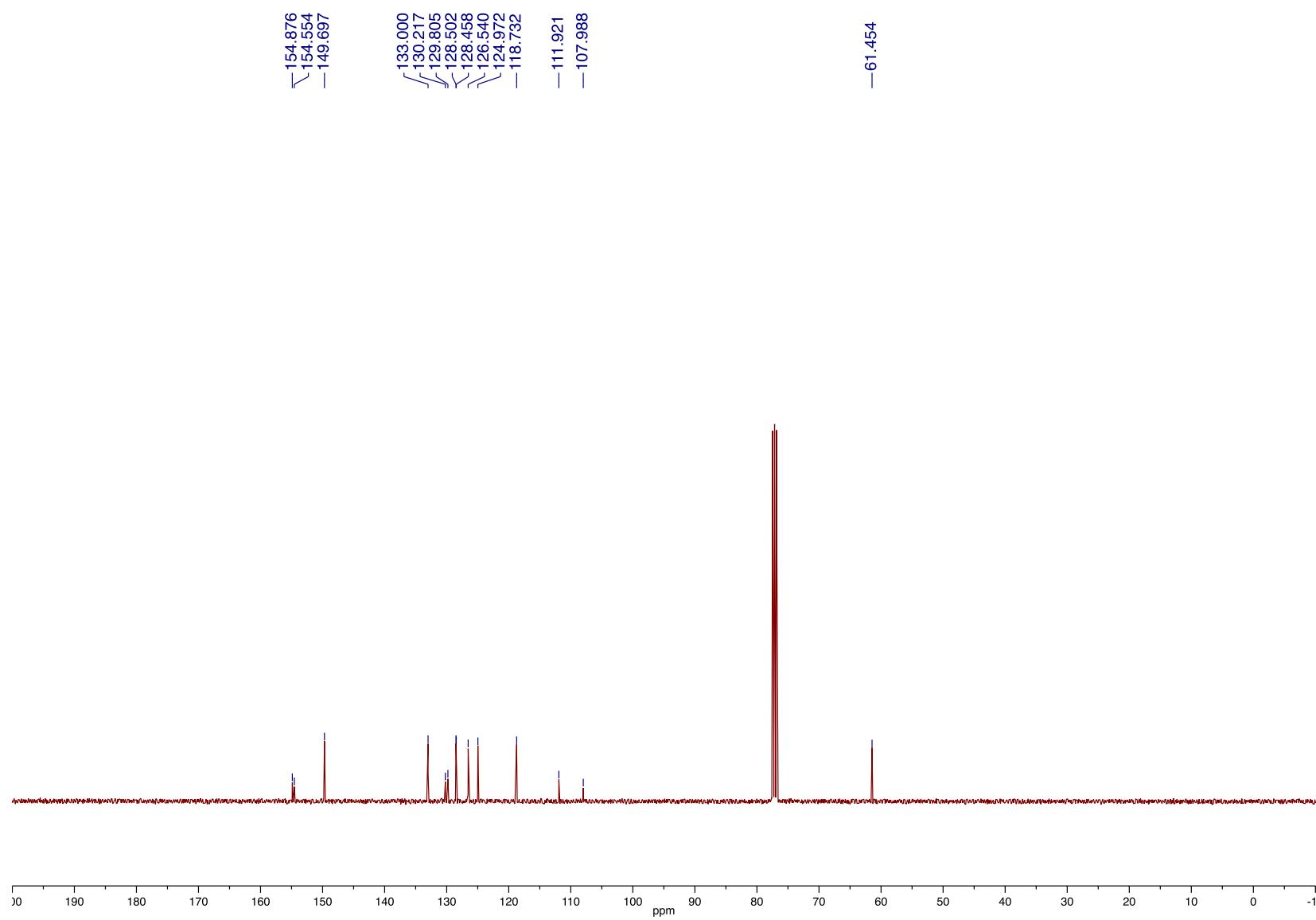
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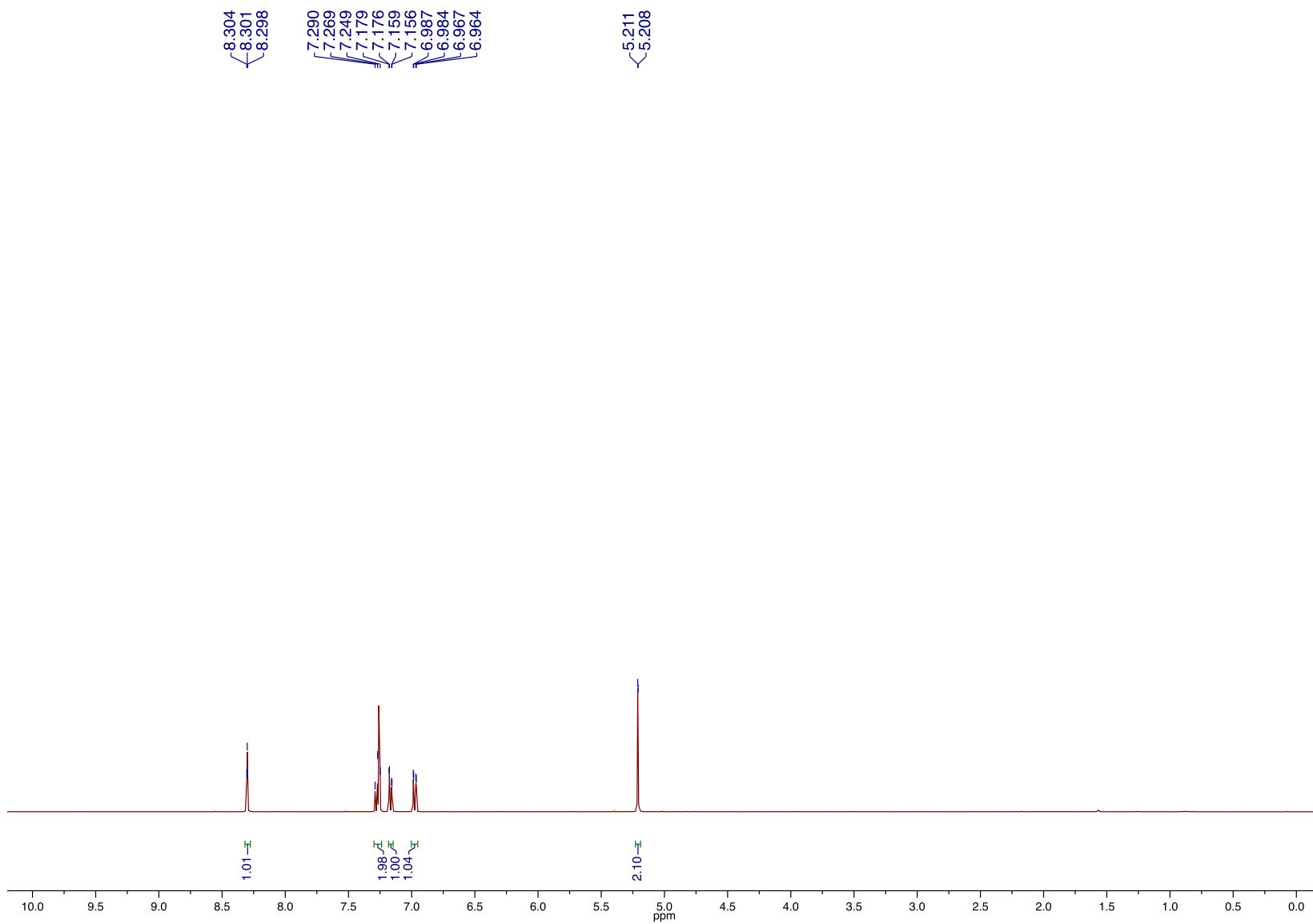
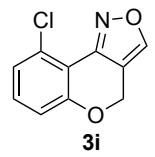
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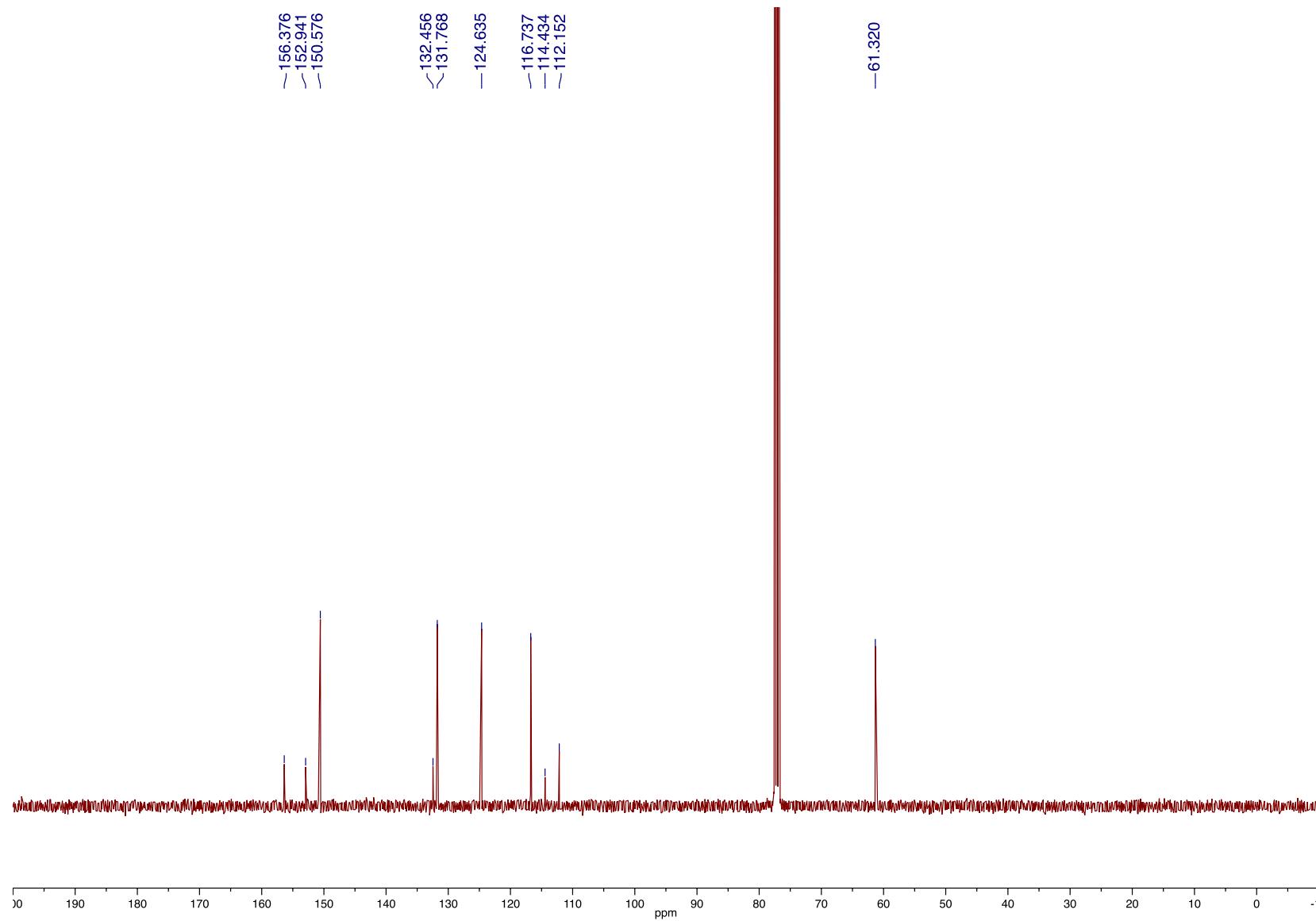
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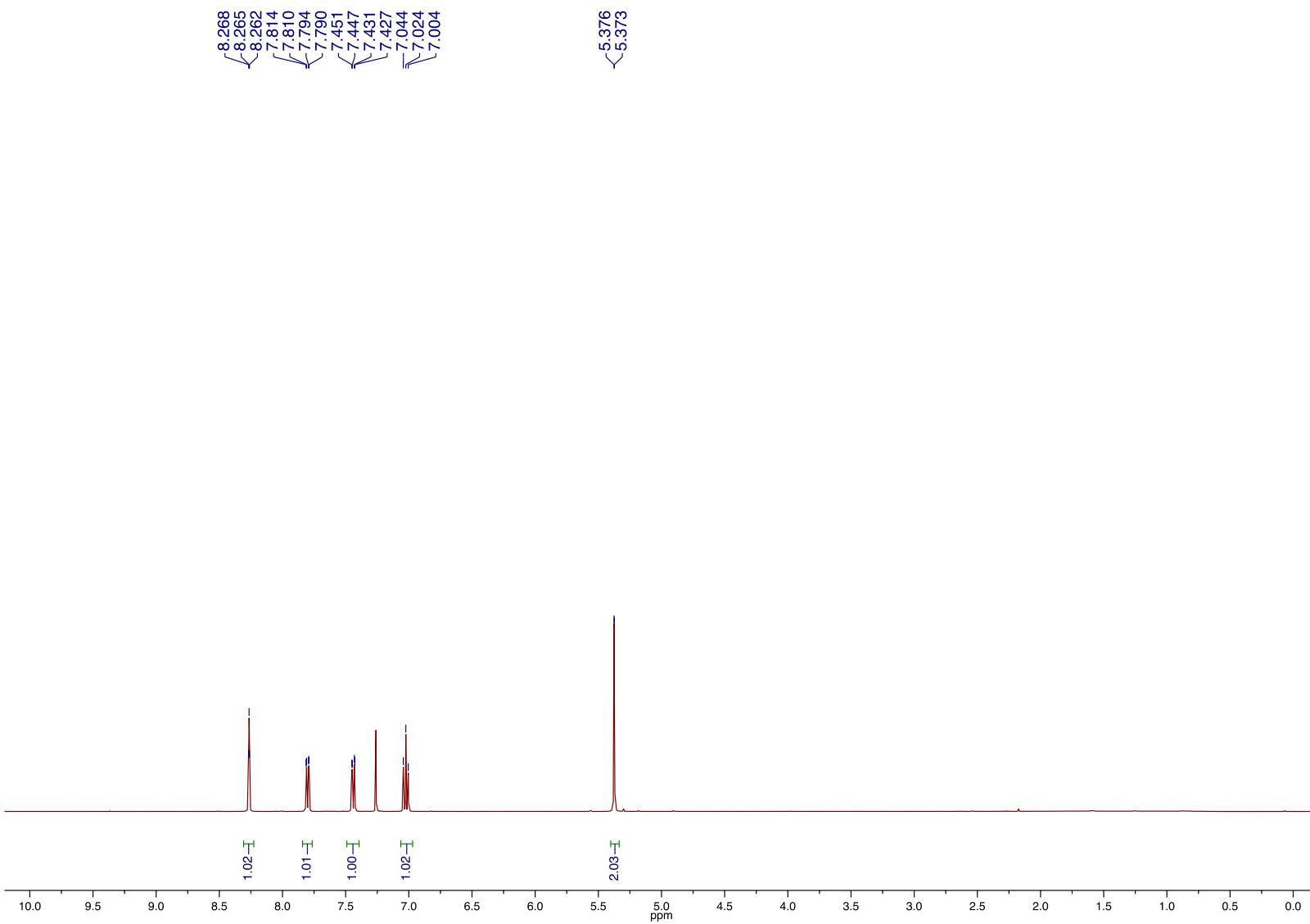
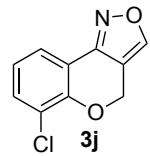
¹H NMR (400 MHz, CDCl₃)



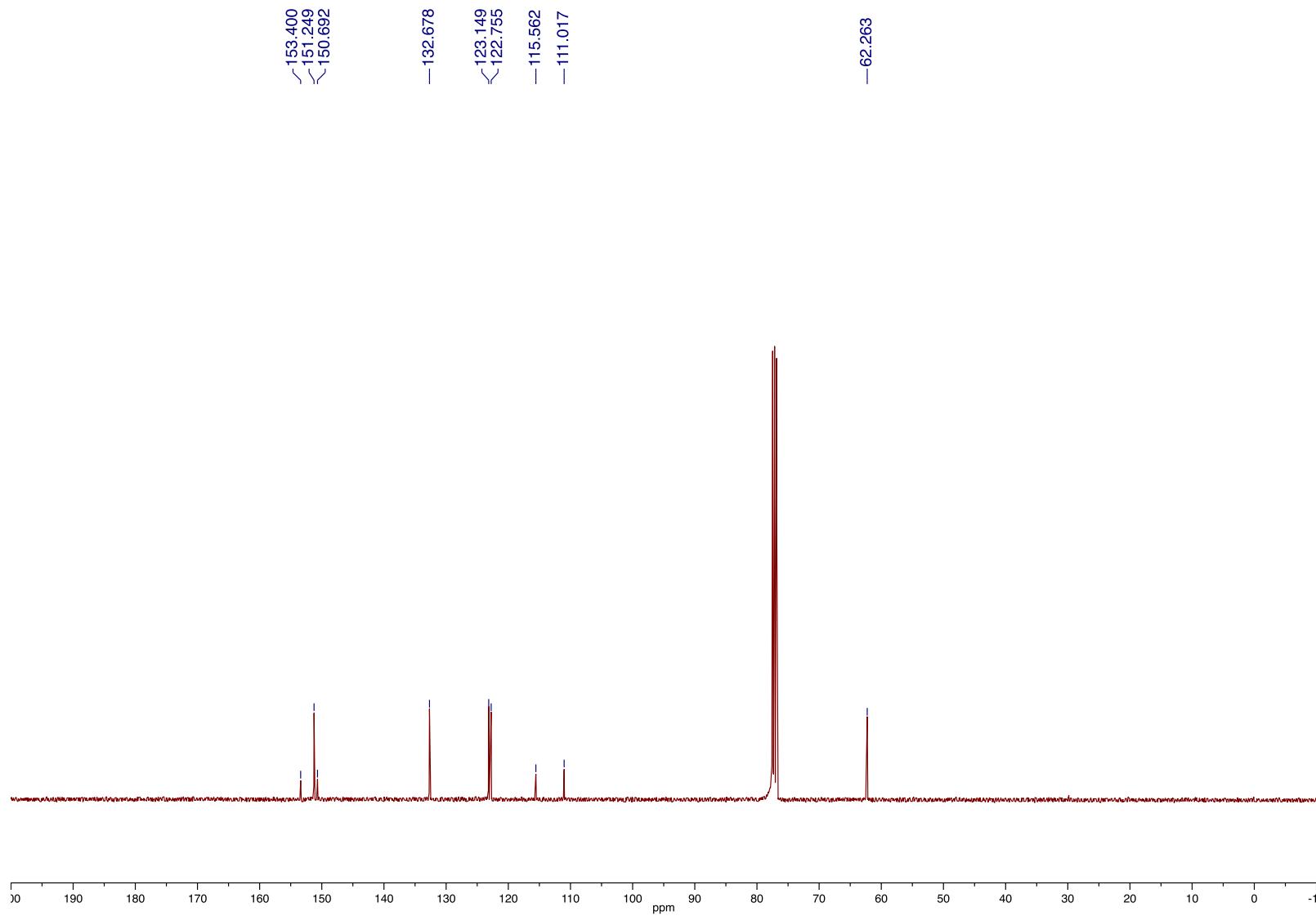
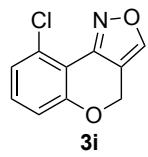
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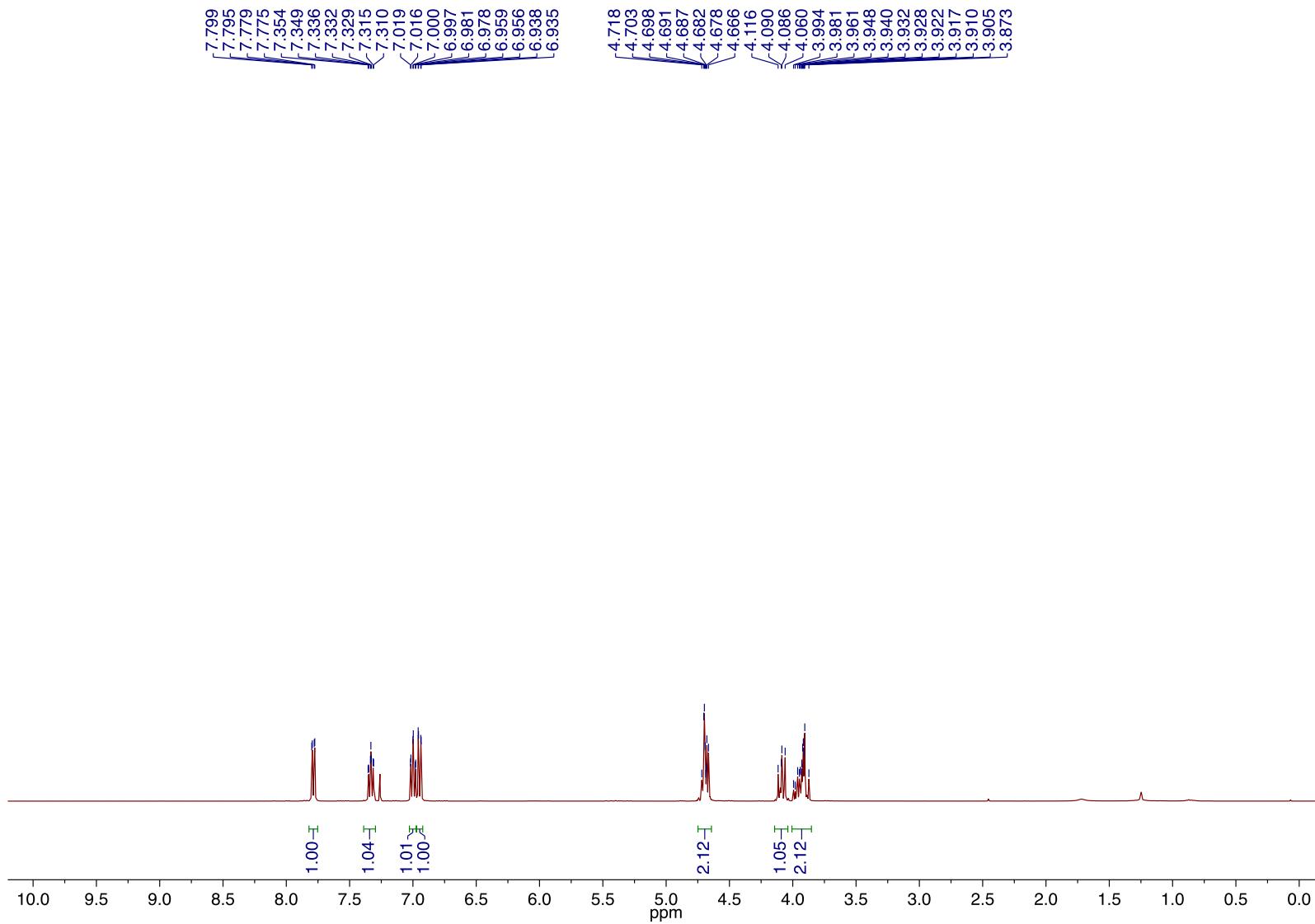
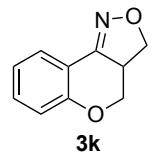
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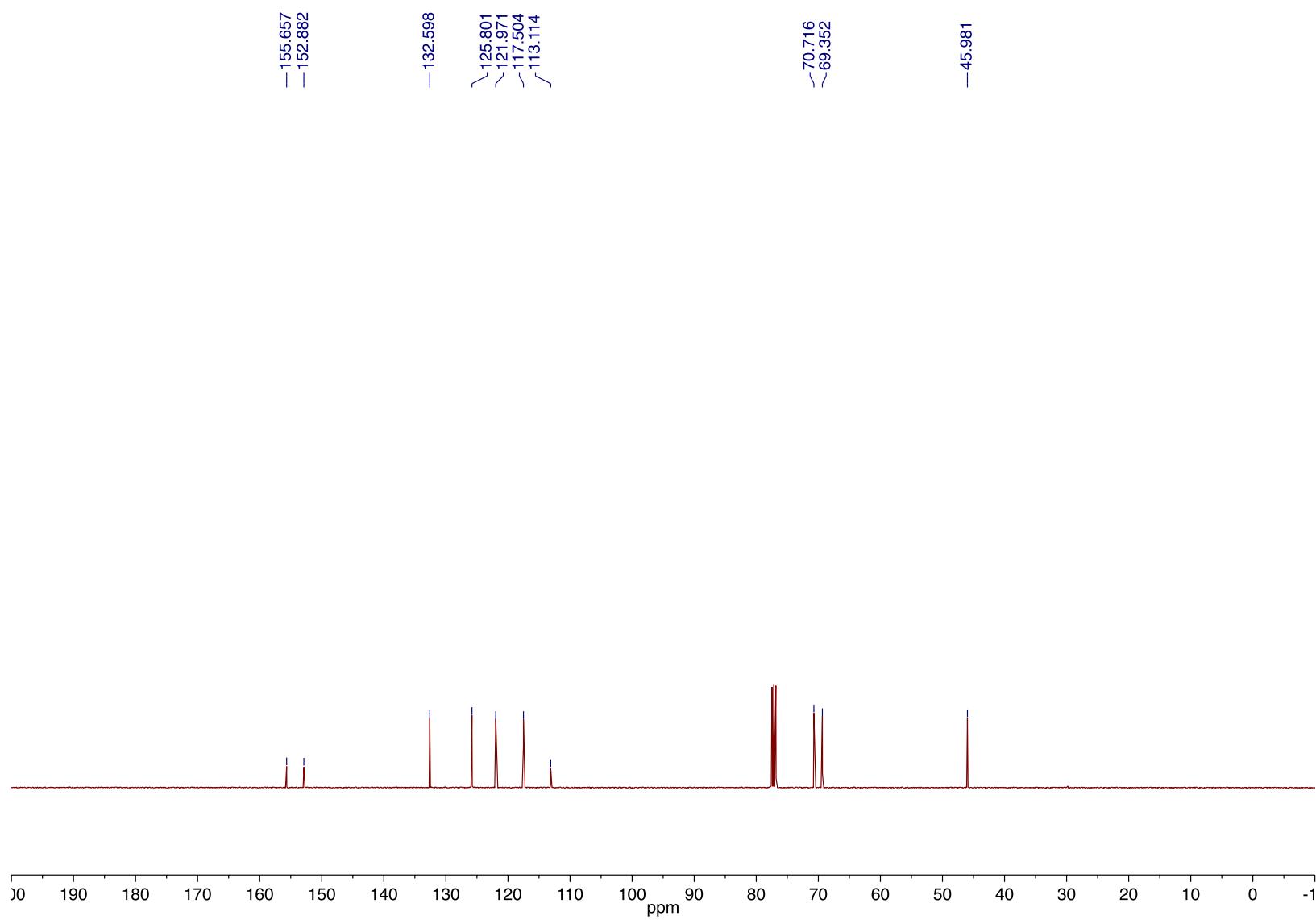
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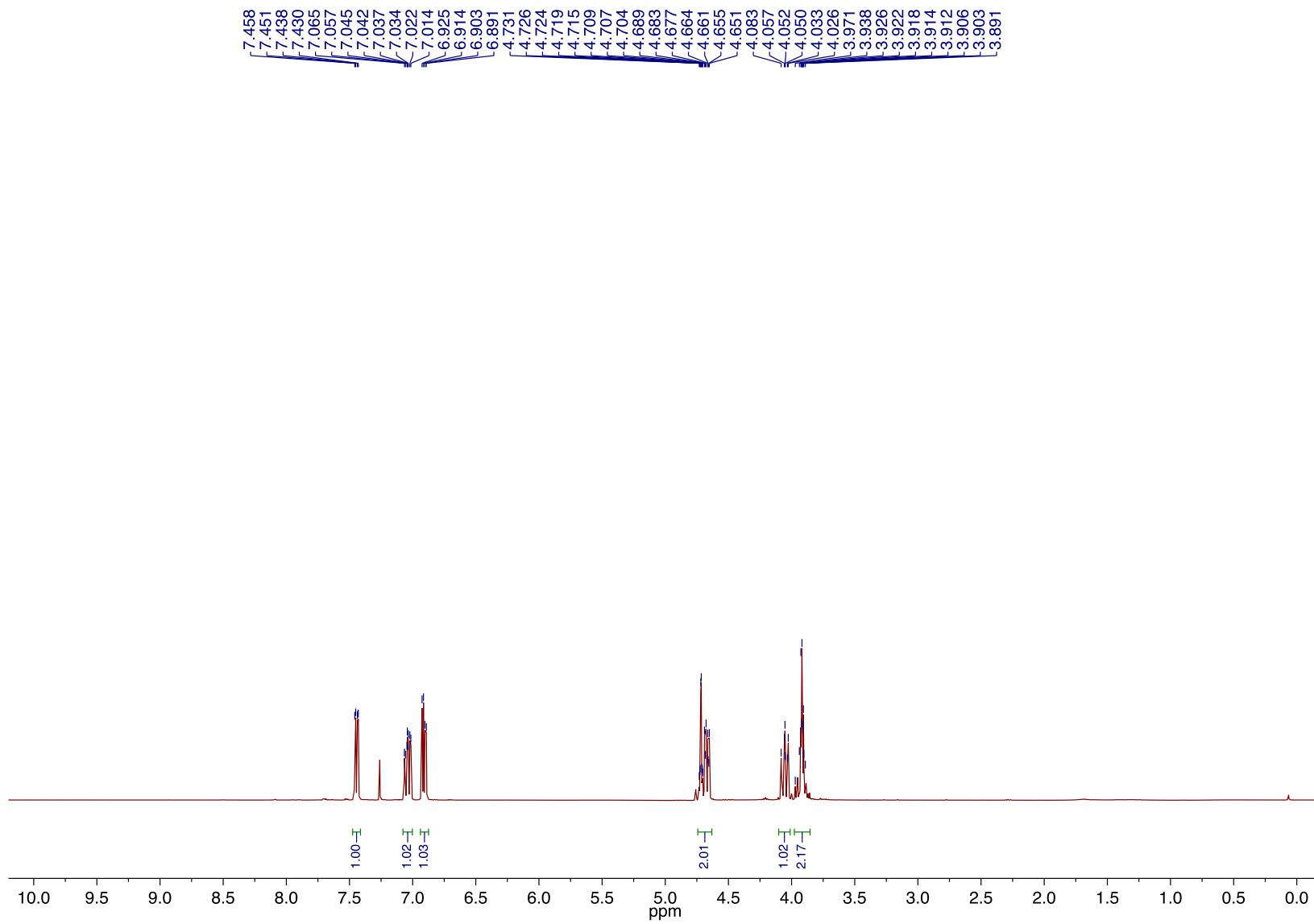
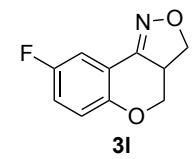
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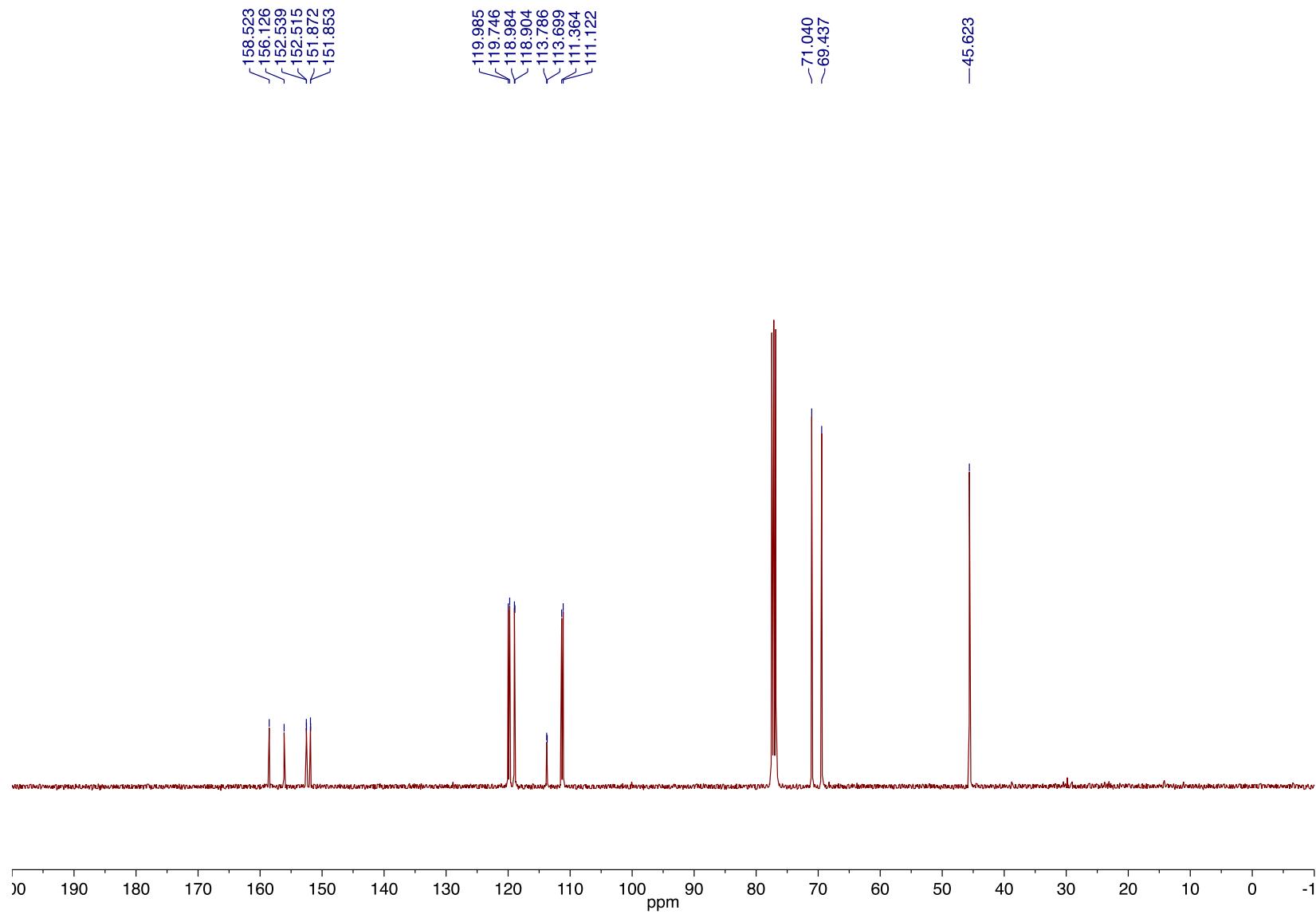
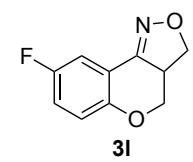
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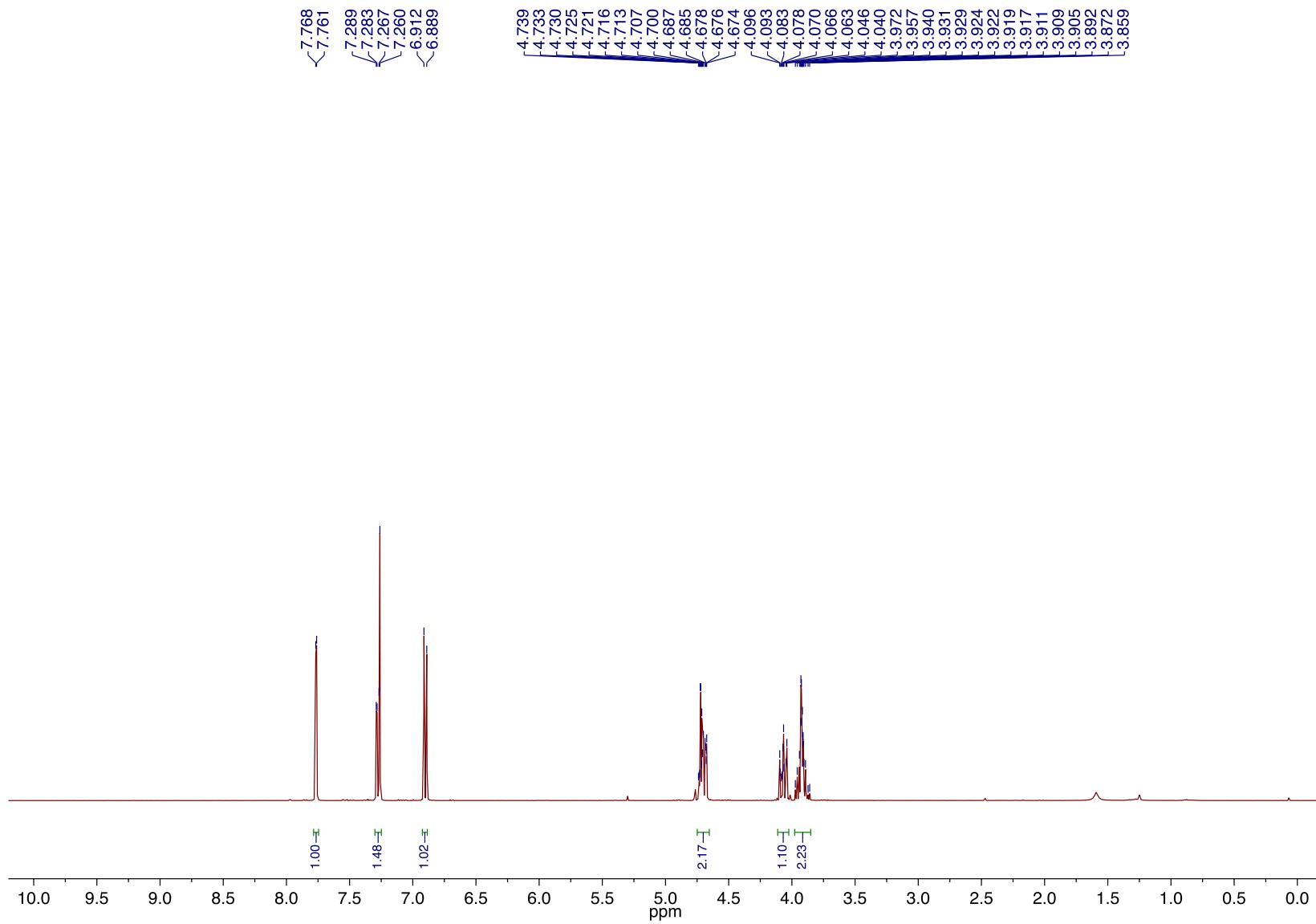
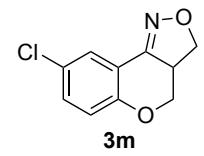
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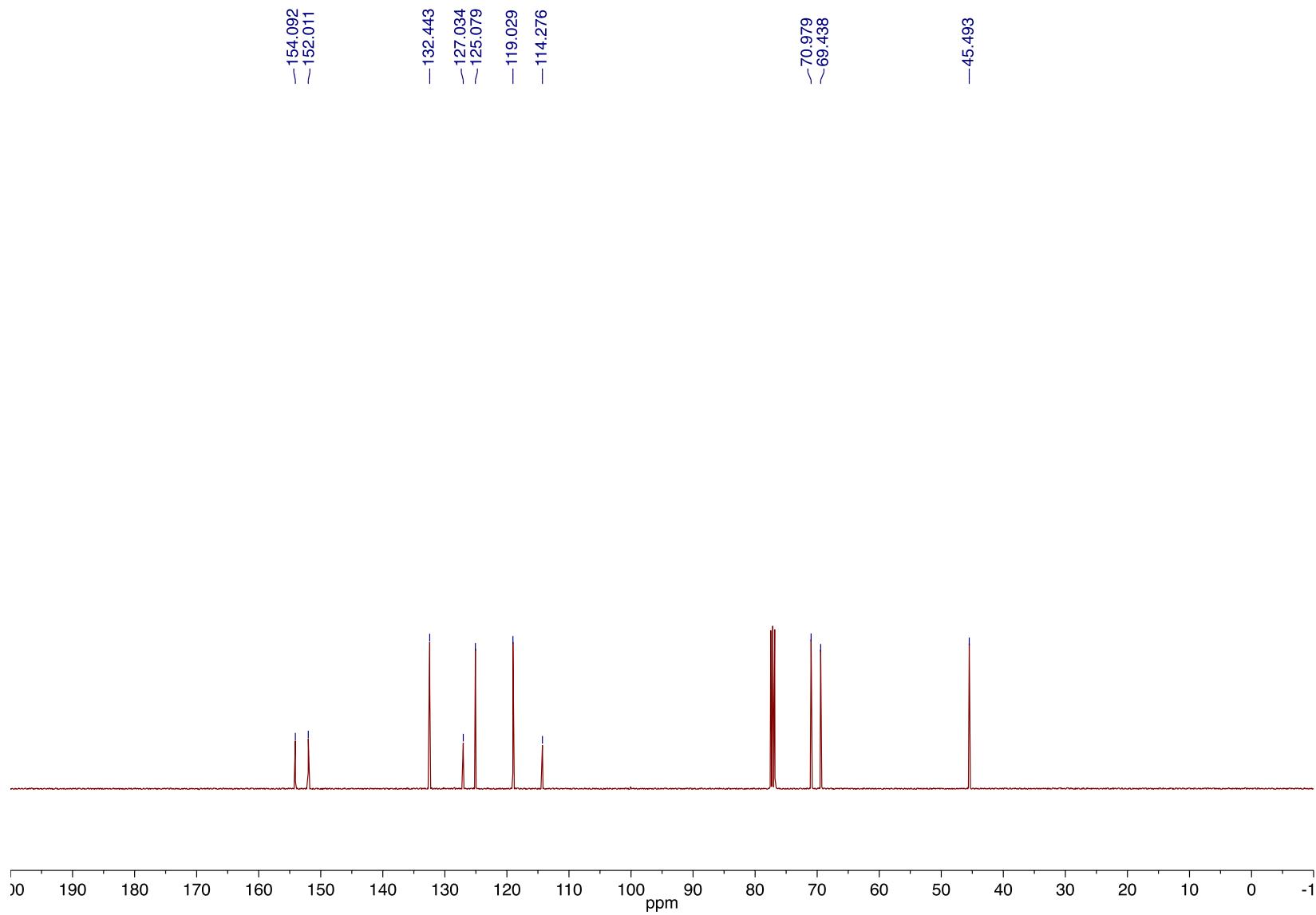
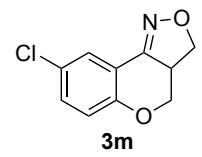
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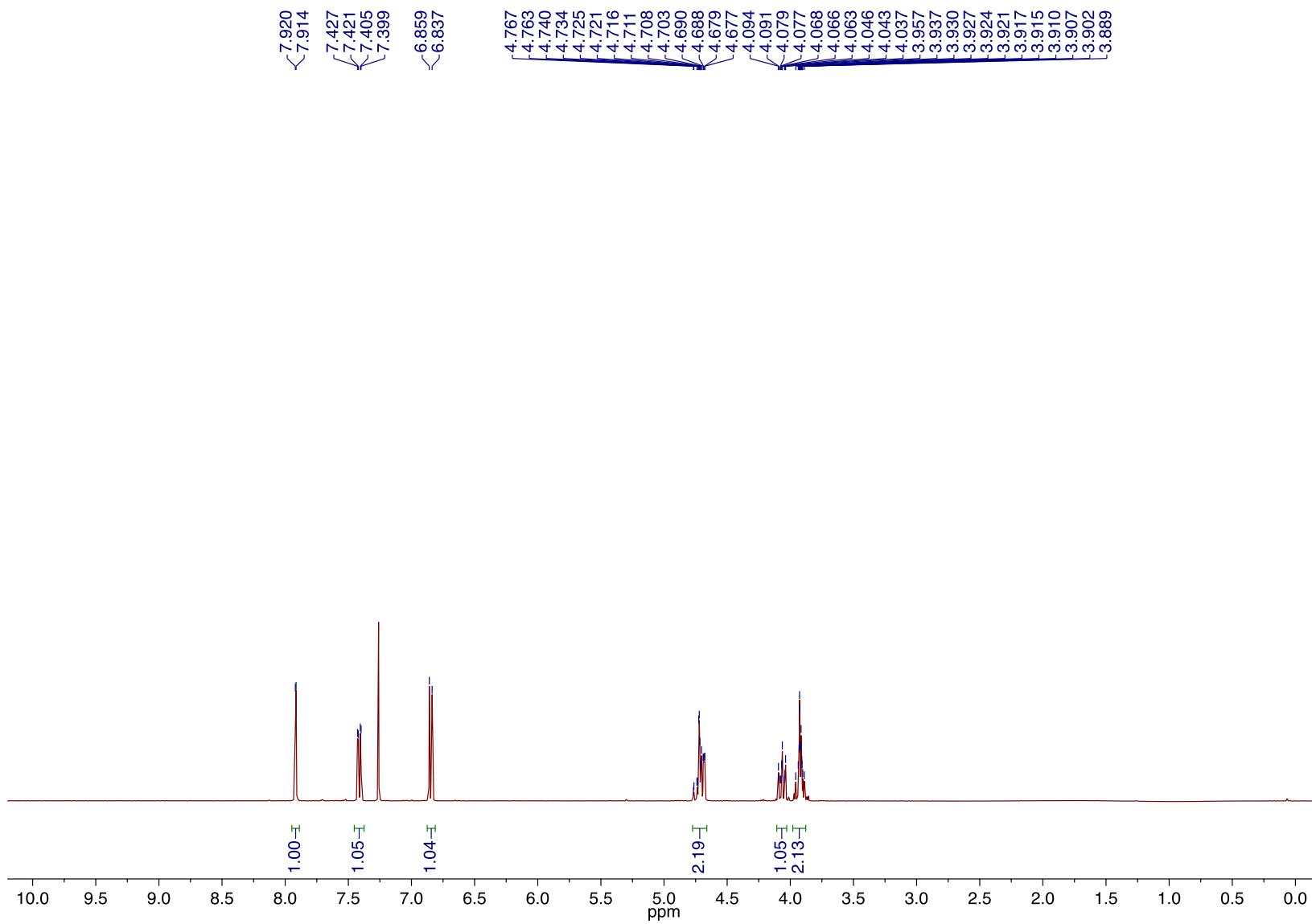
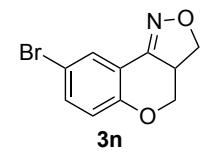
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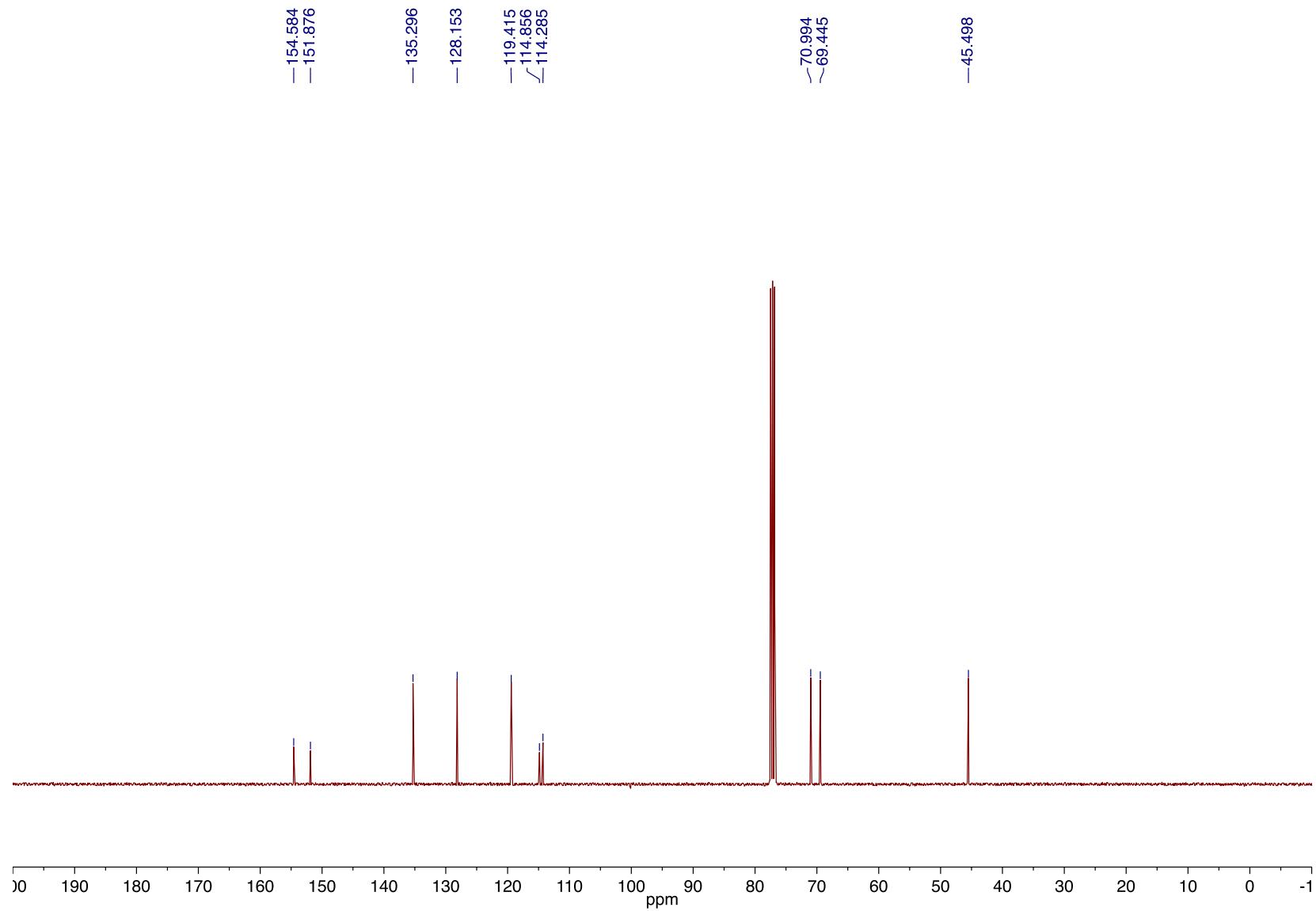
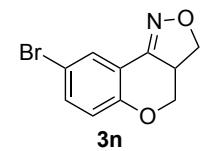
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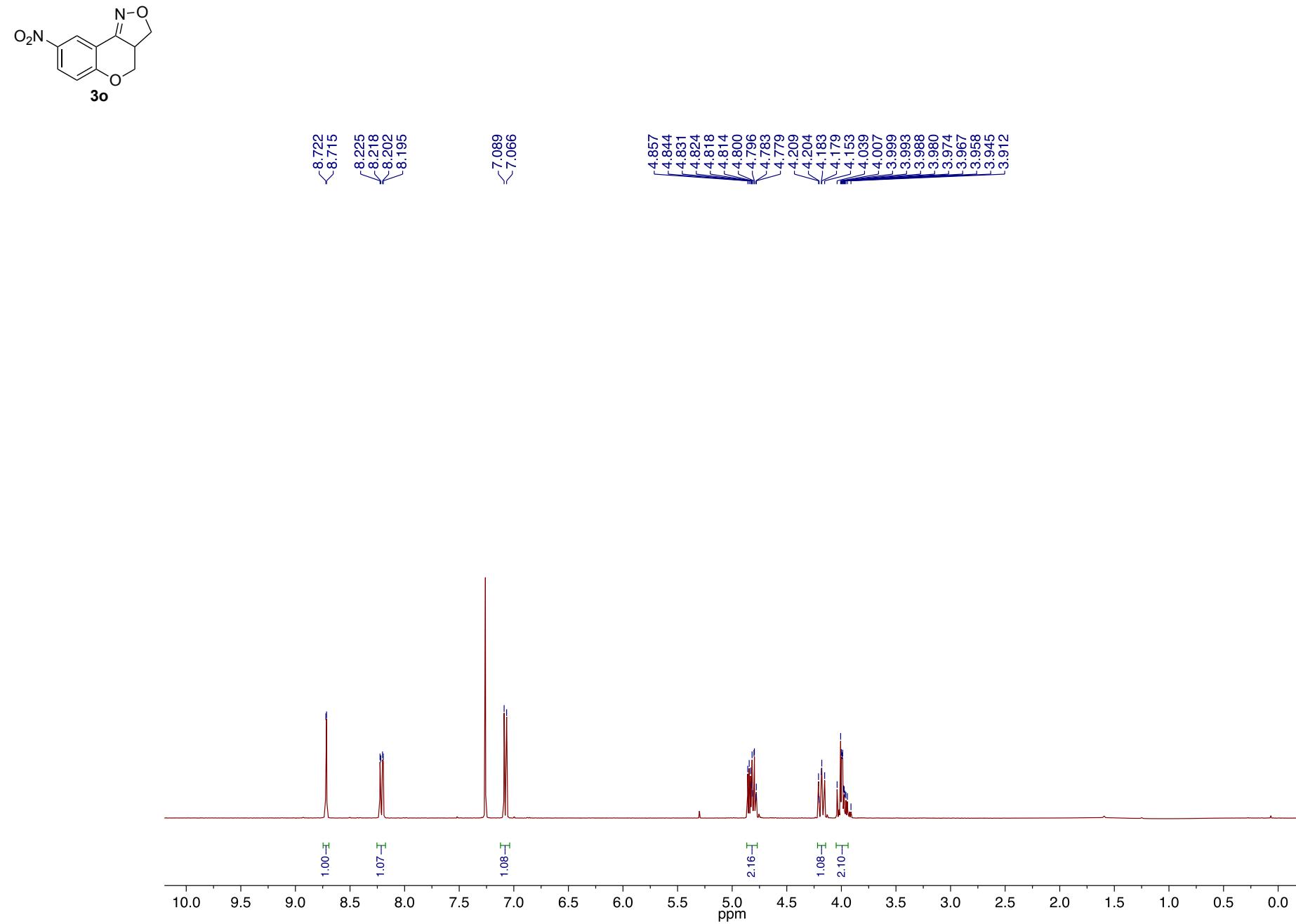
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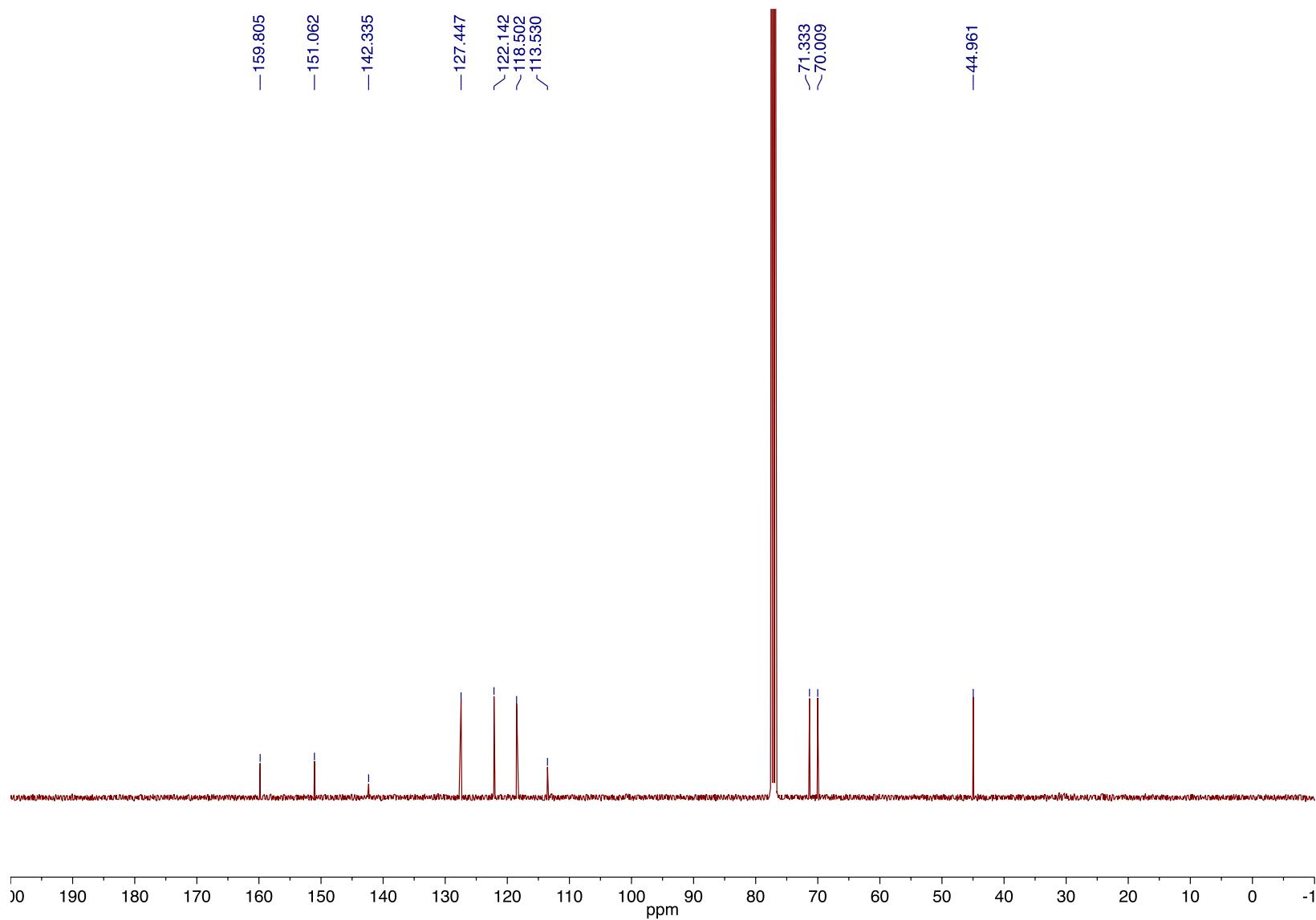
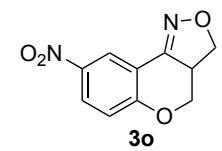
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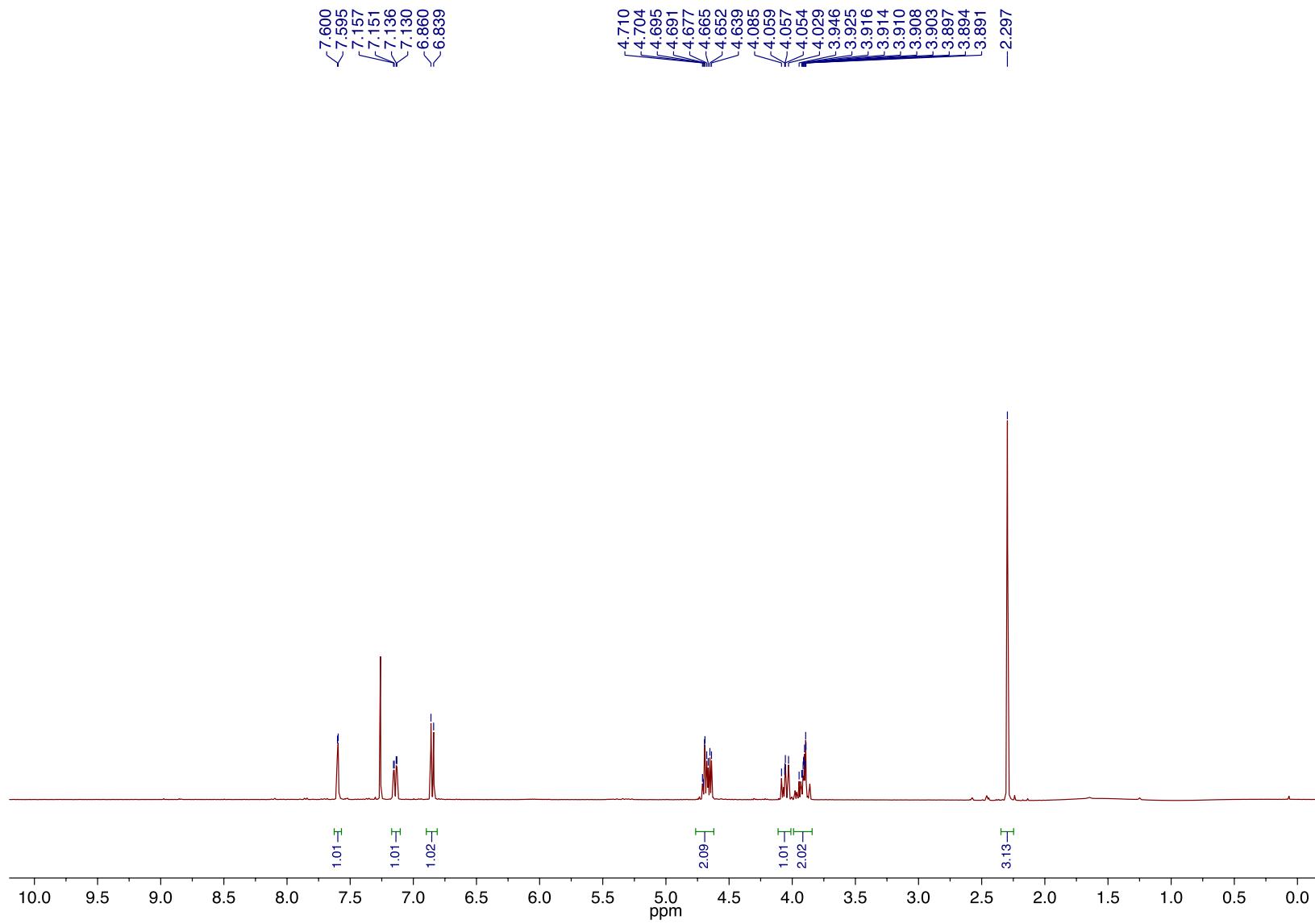
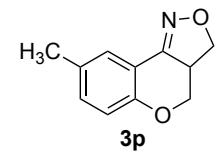
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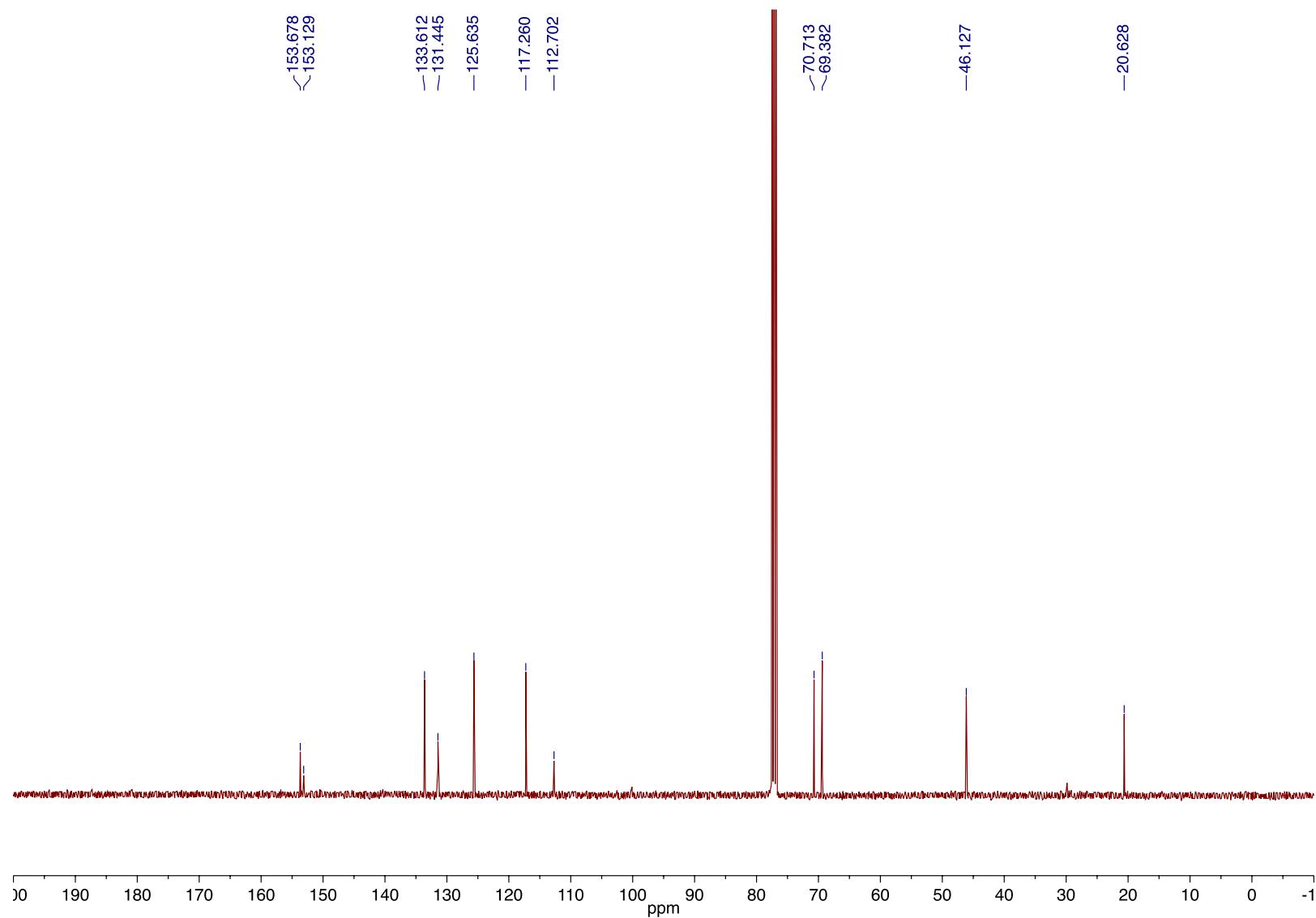
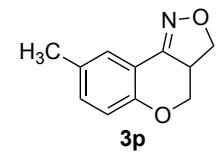
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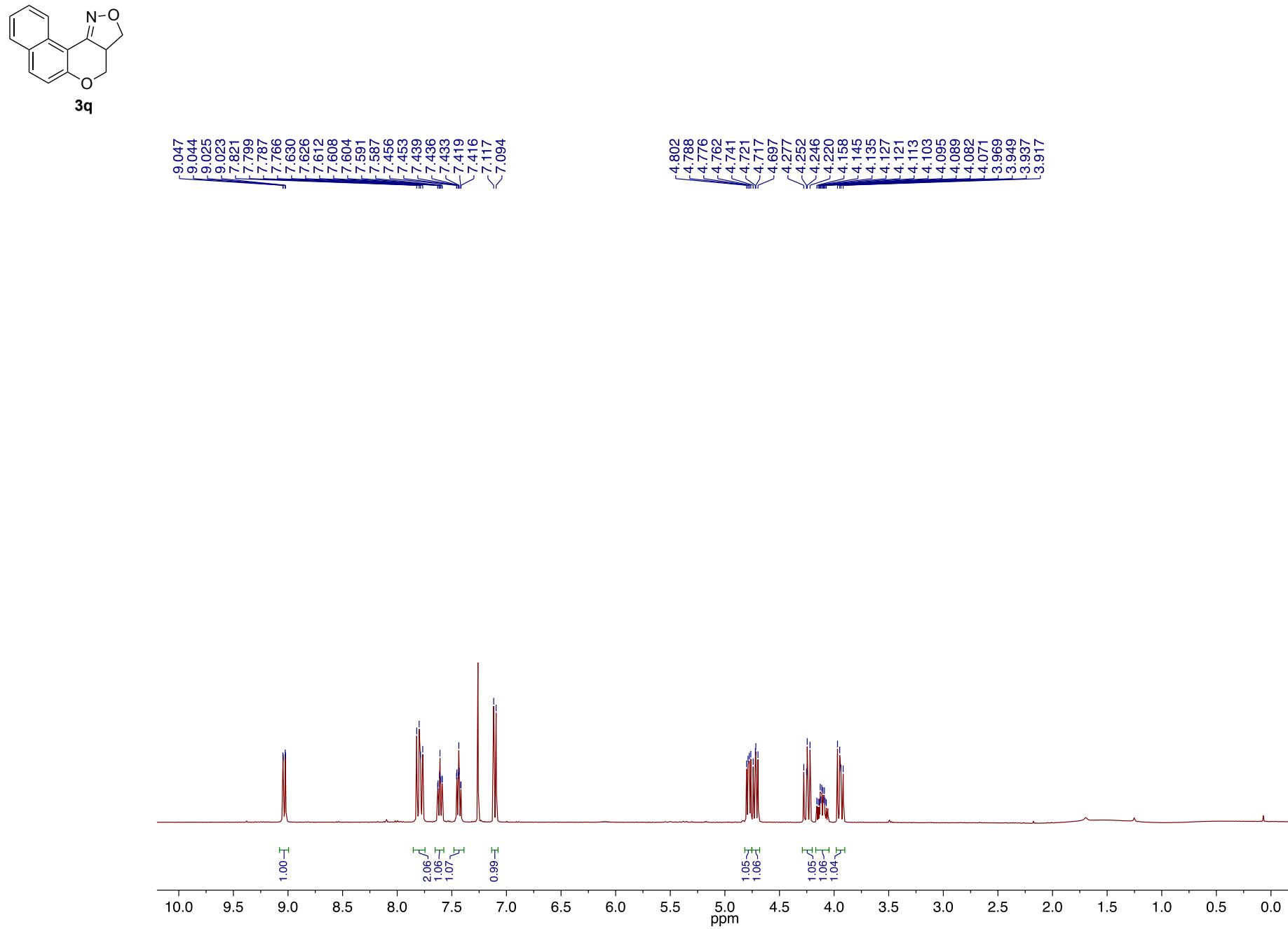
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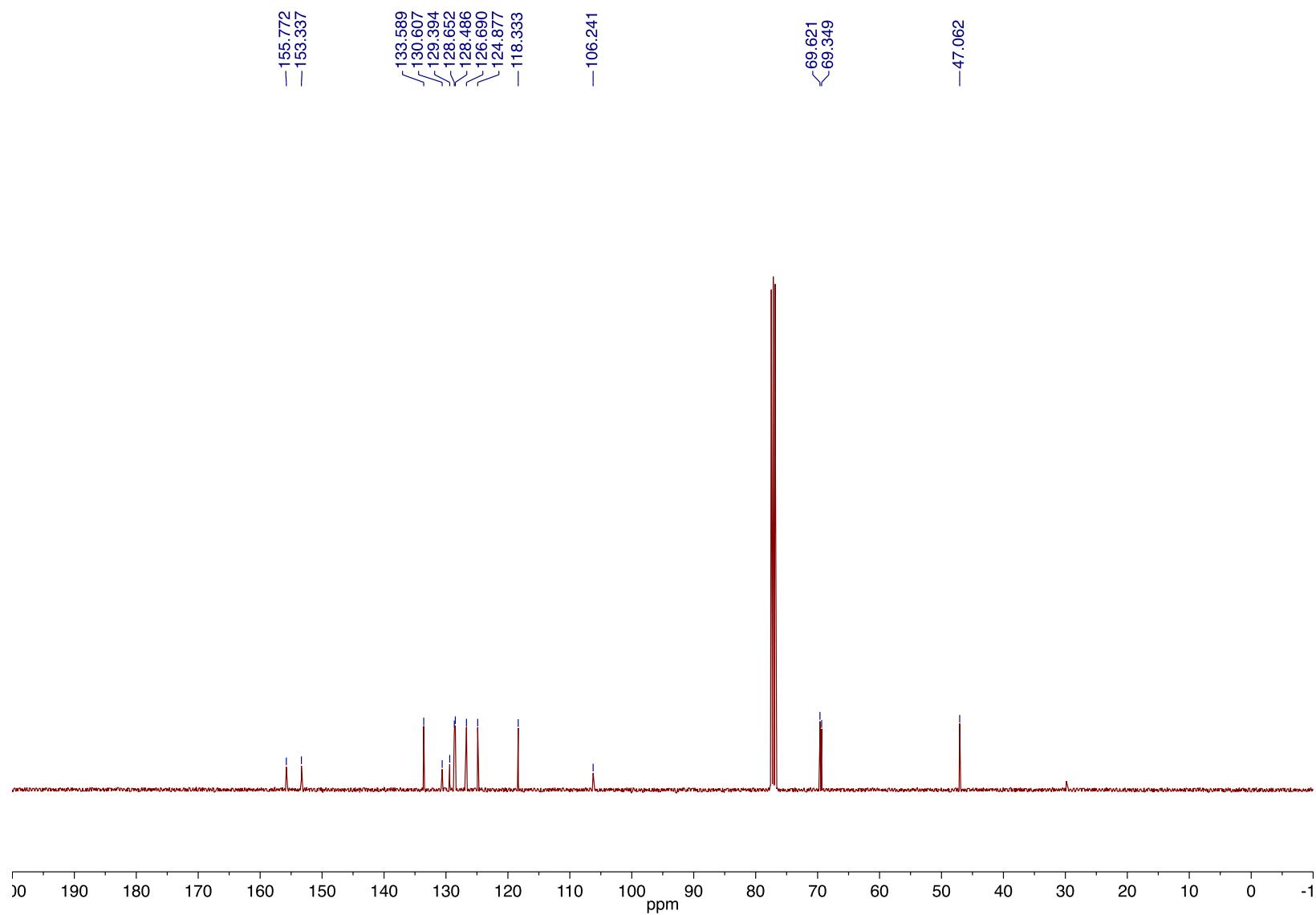
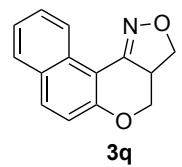
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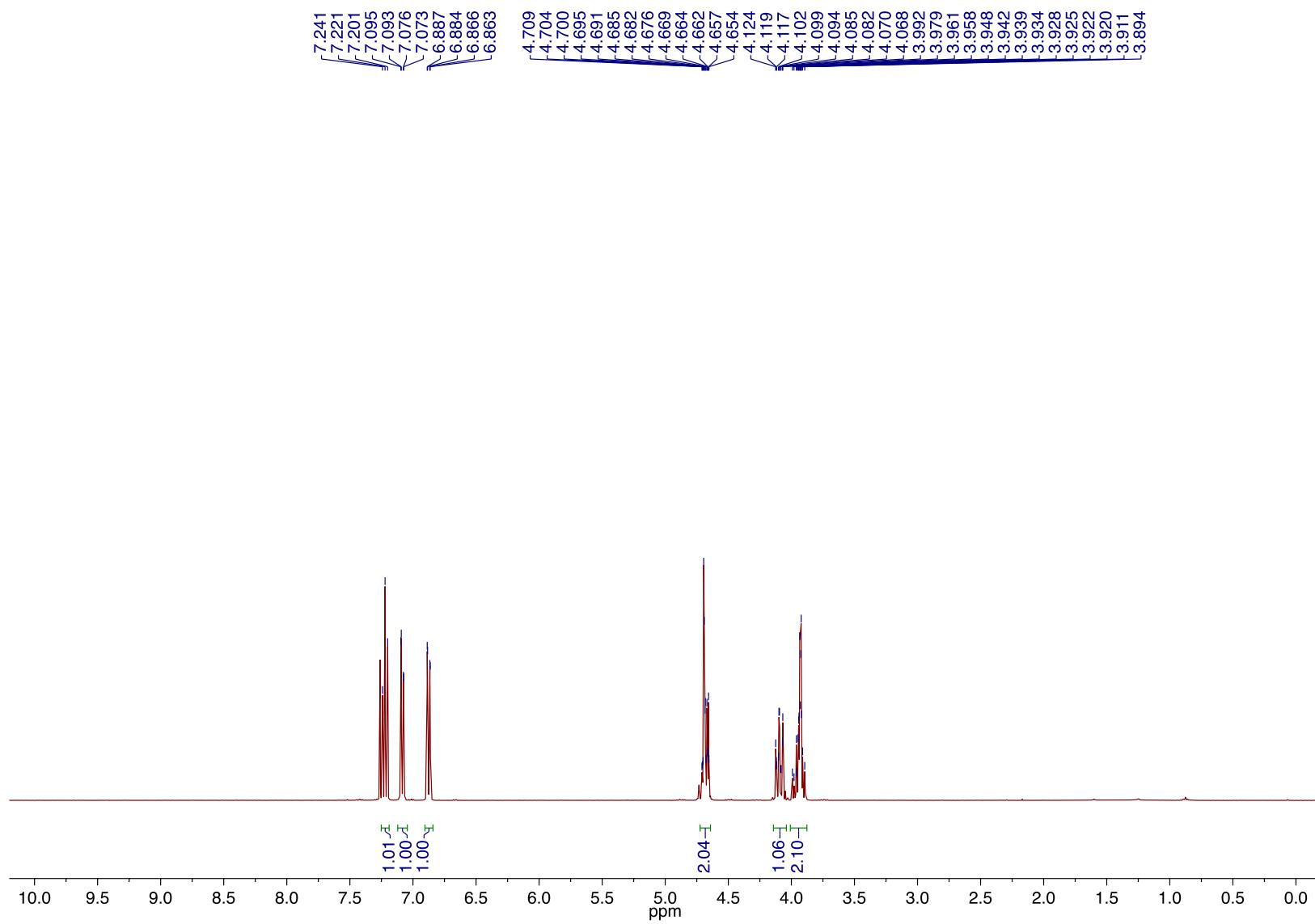
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¹³C NMR (101 MHz, CDCl₃)



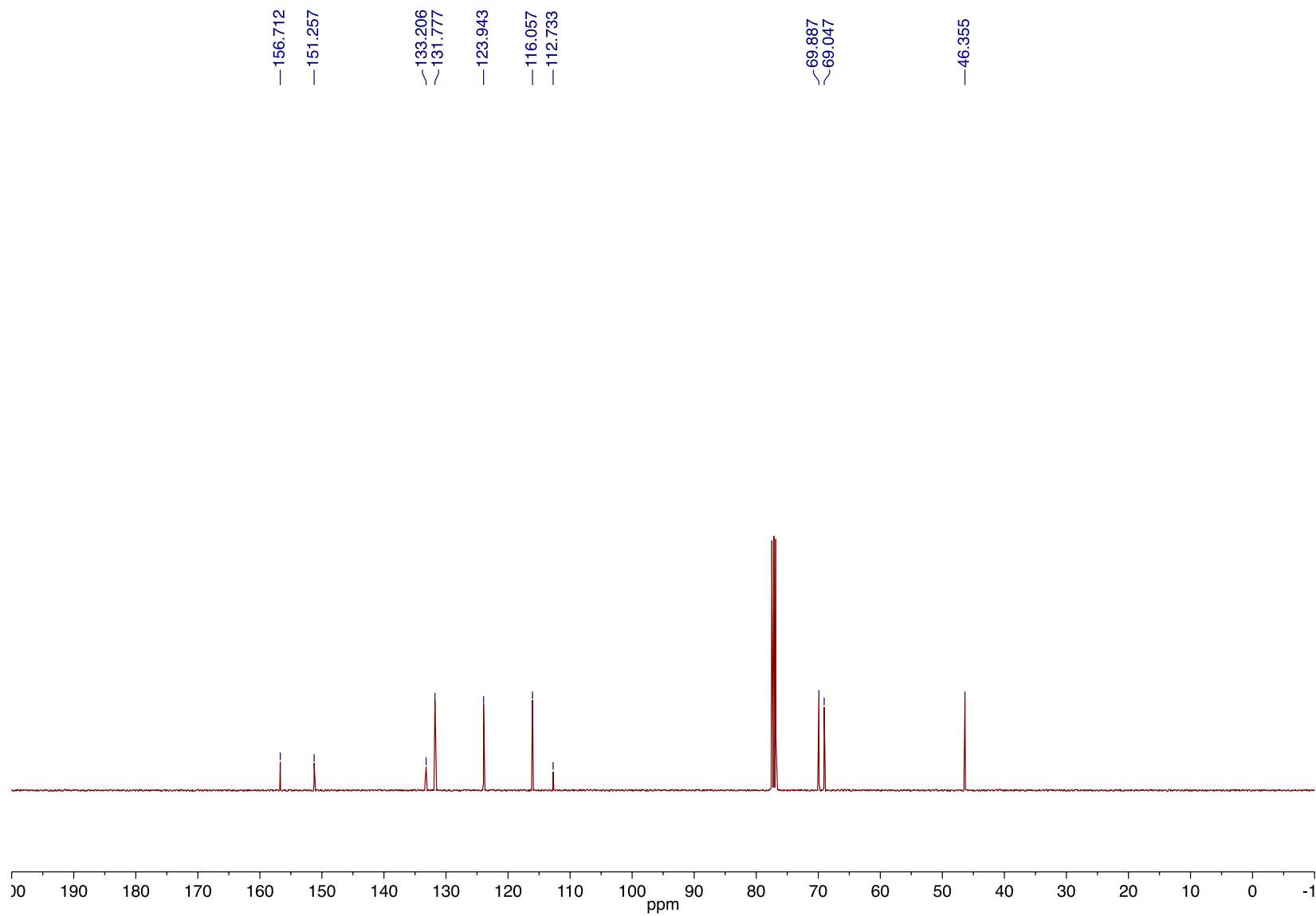
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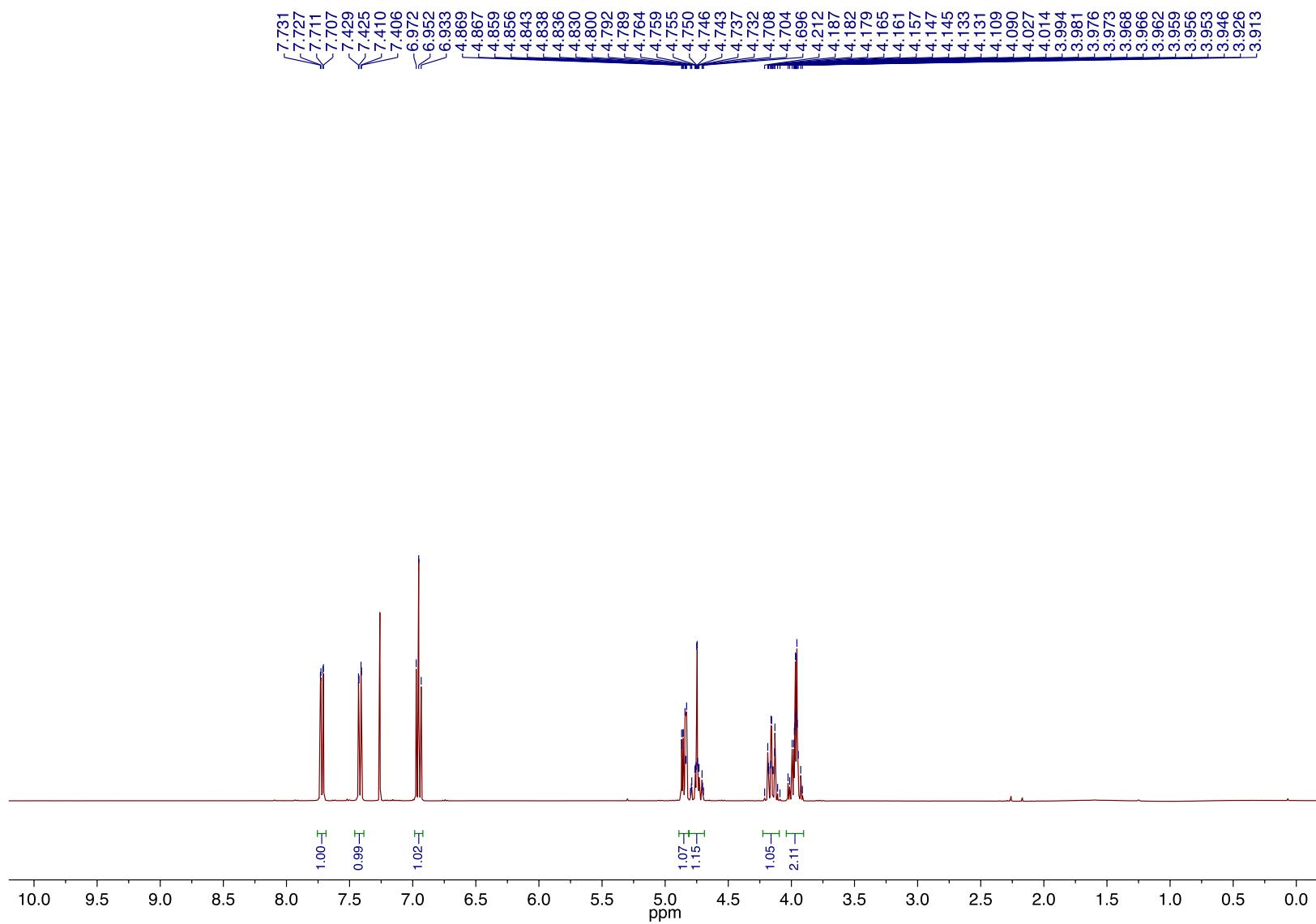
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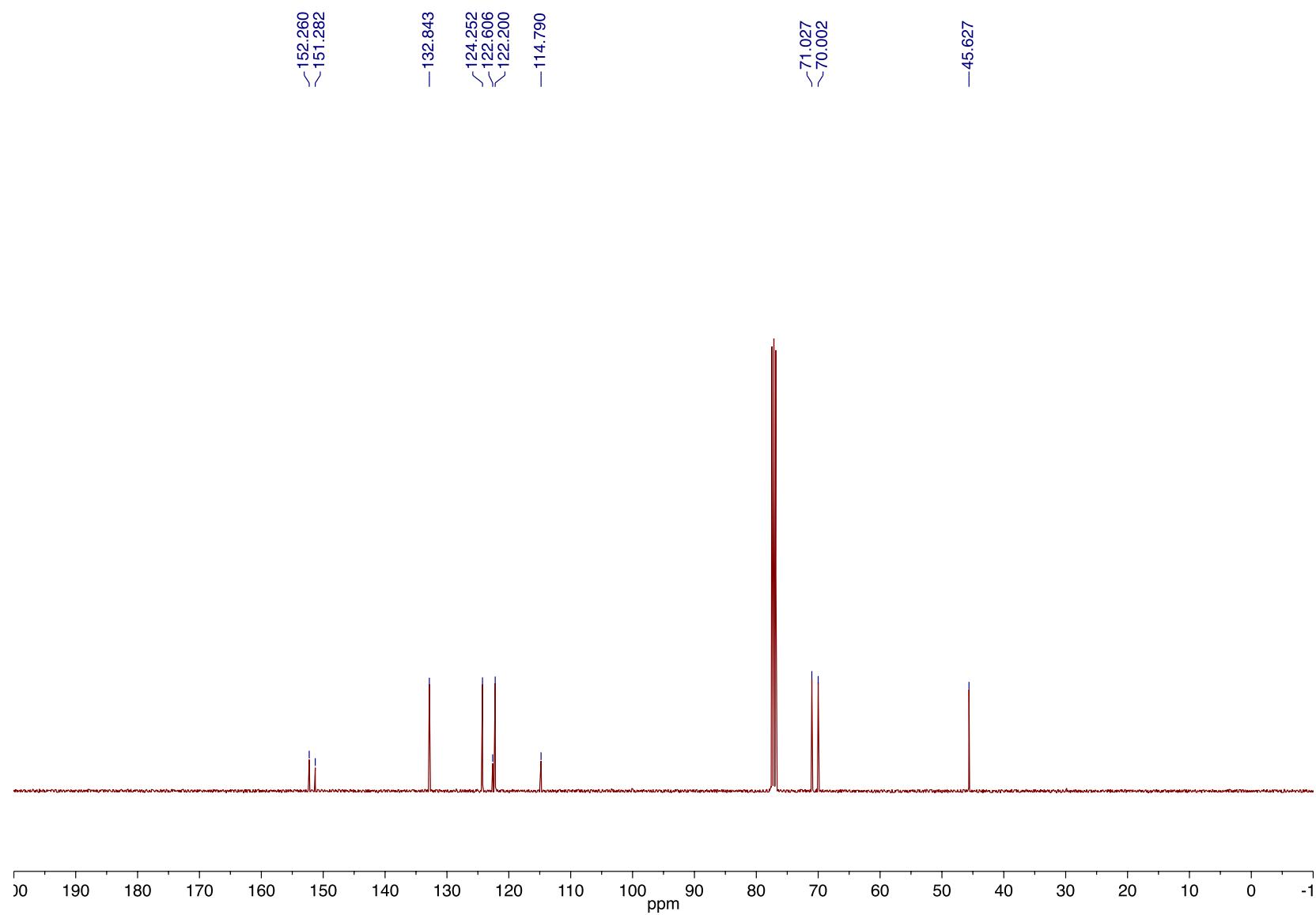
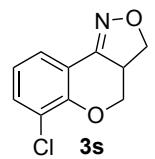
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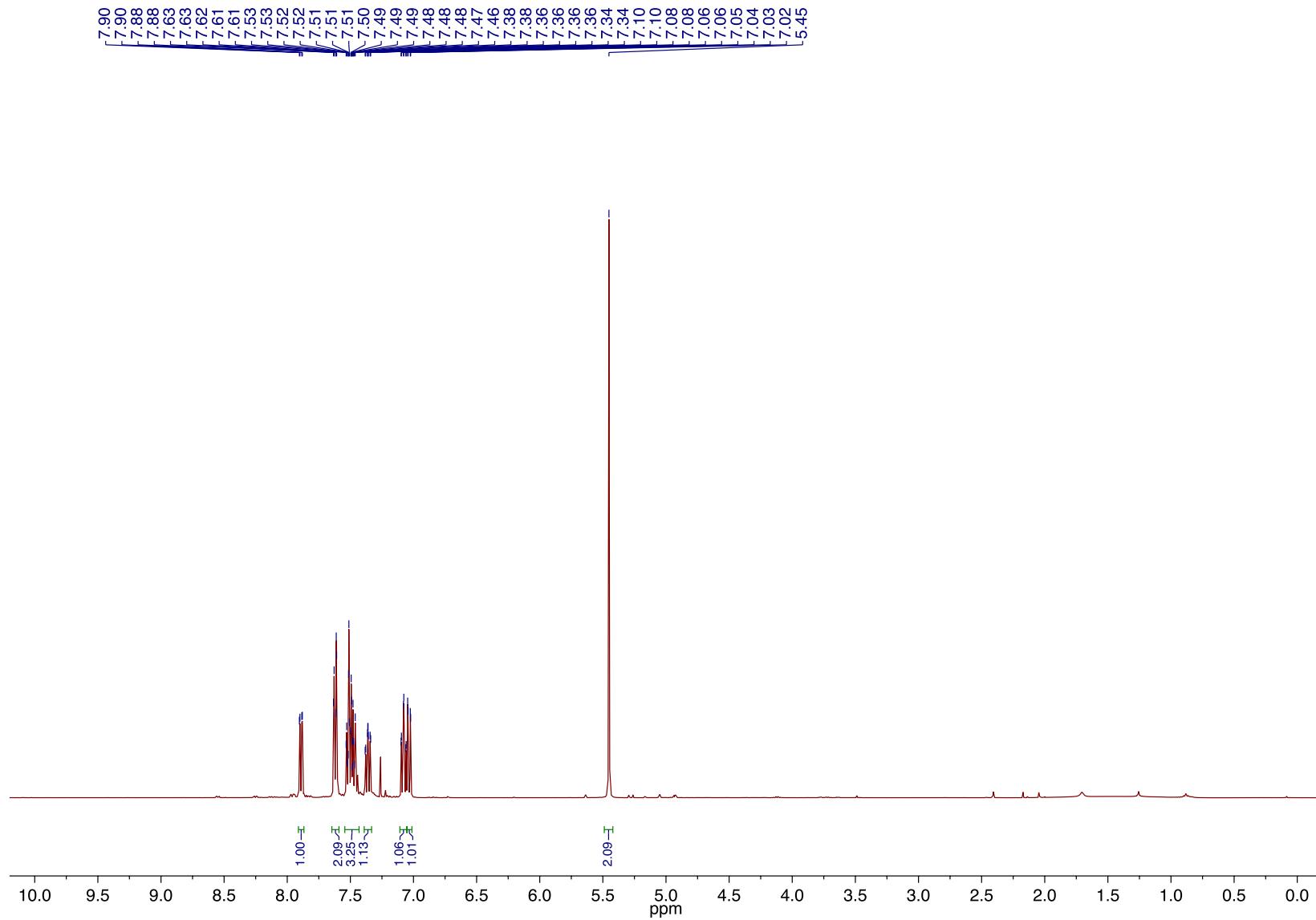
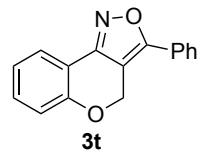
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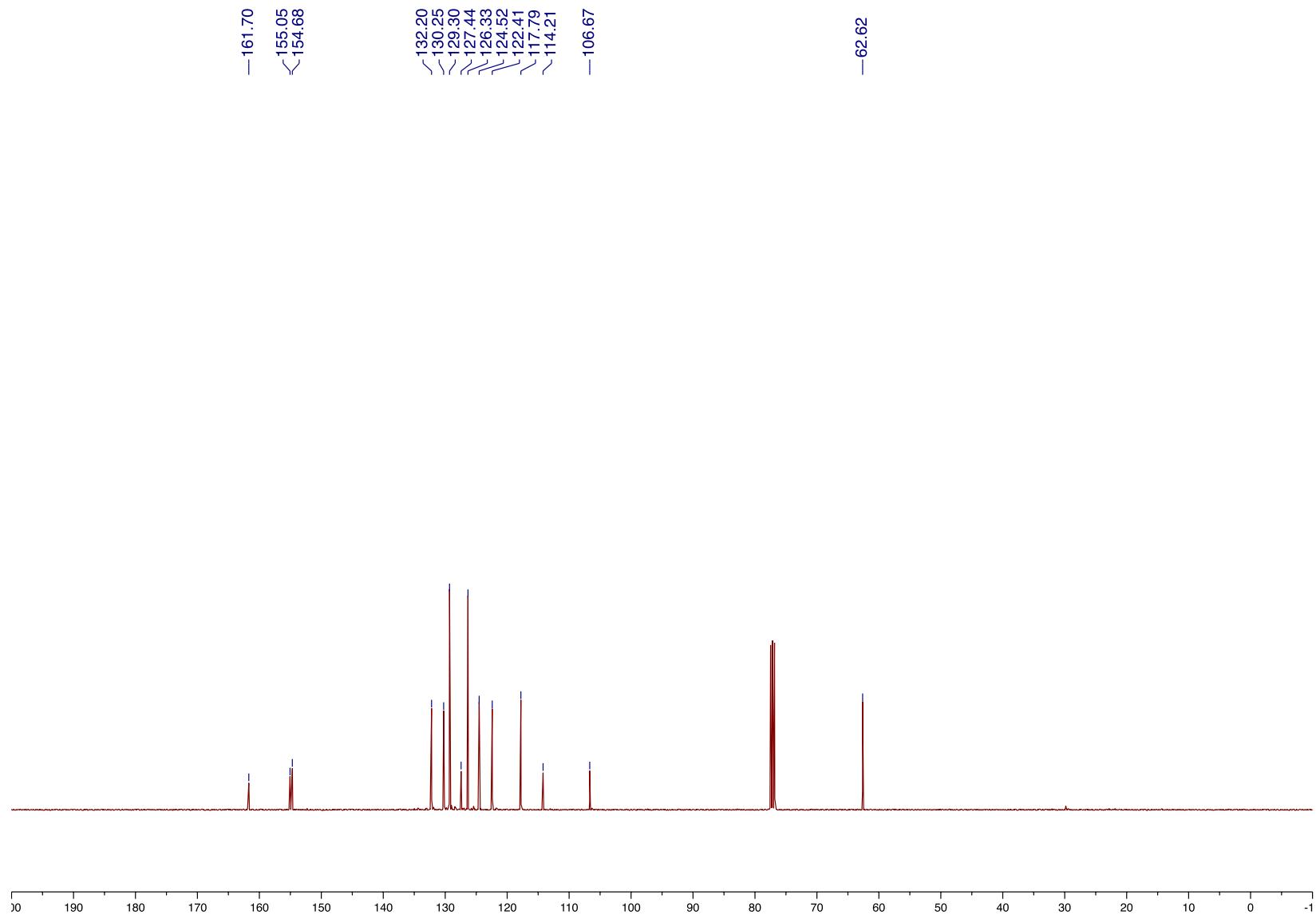
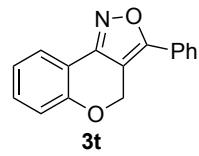
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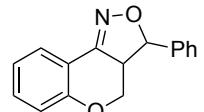
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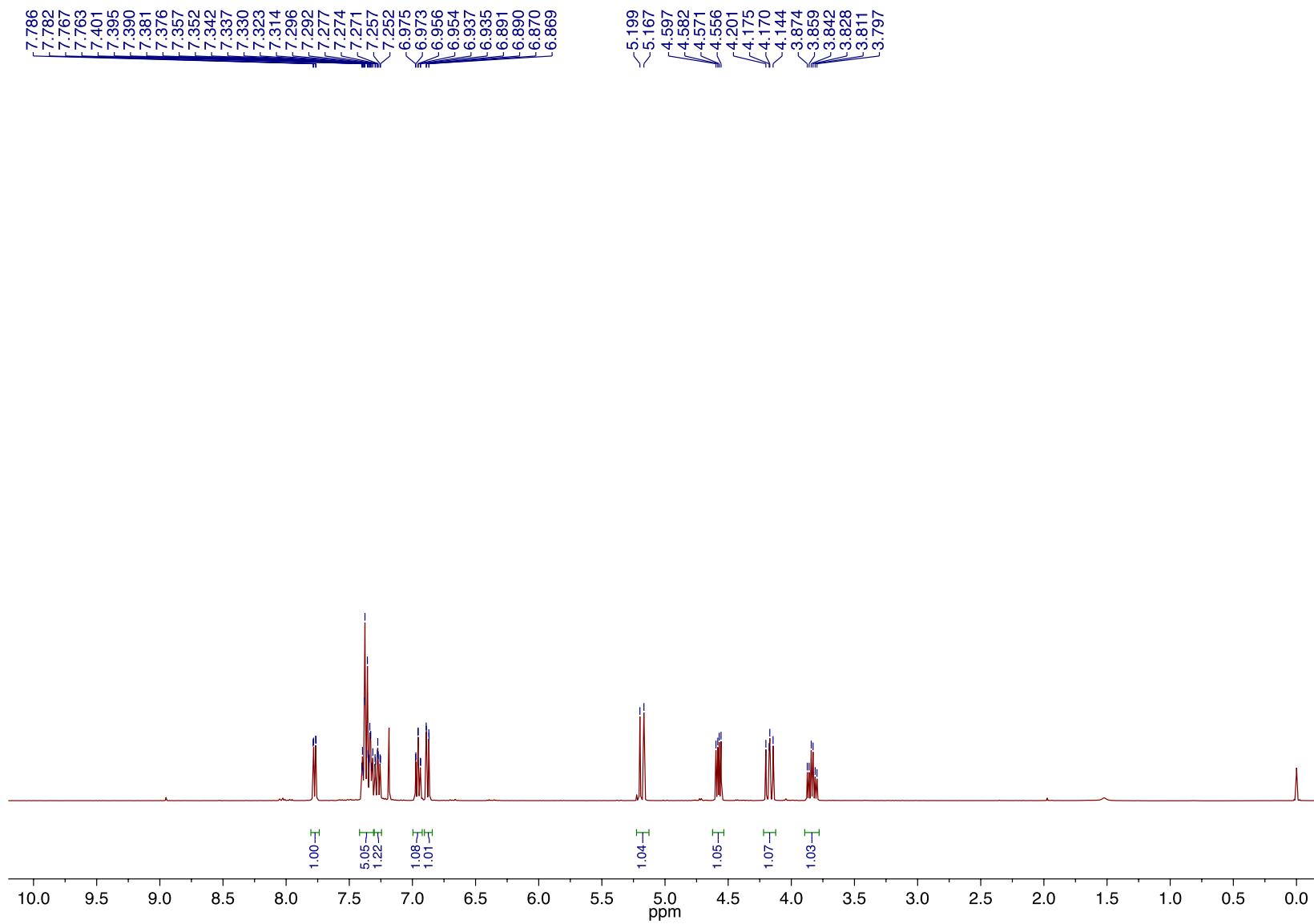
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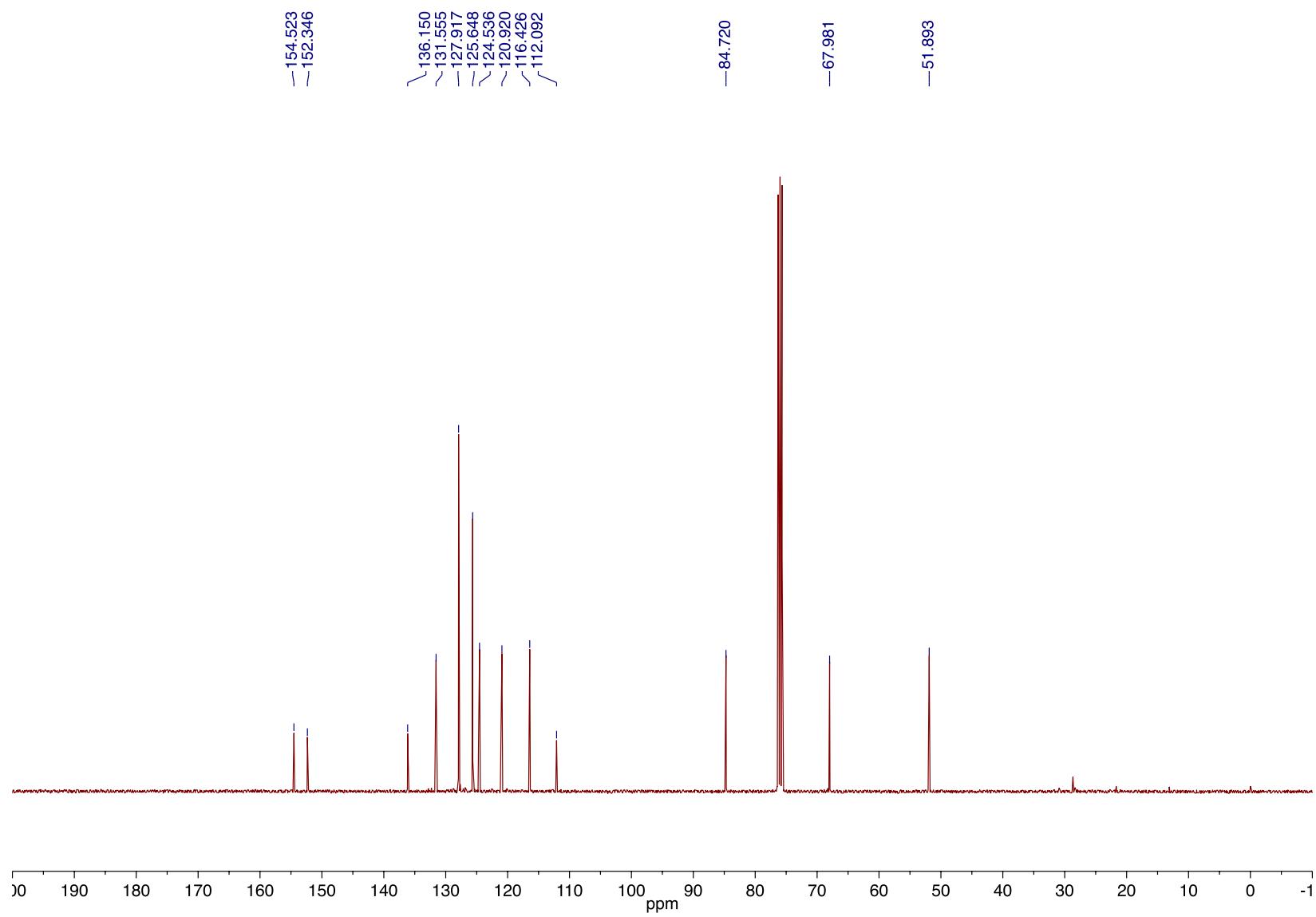
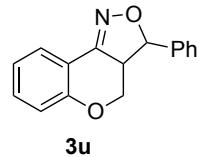
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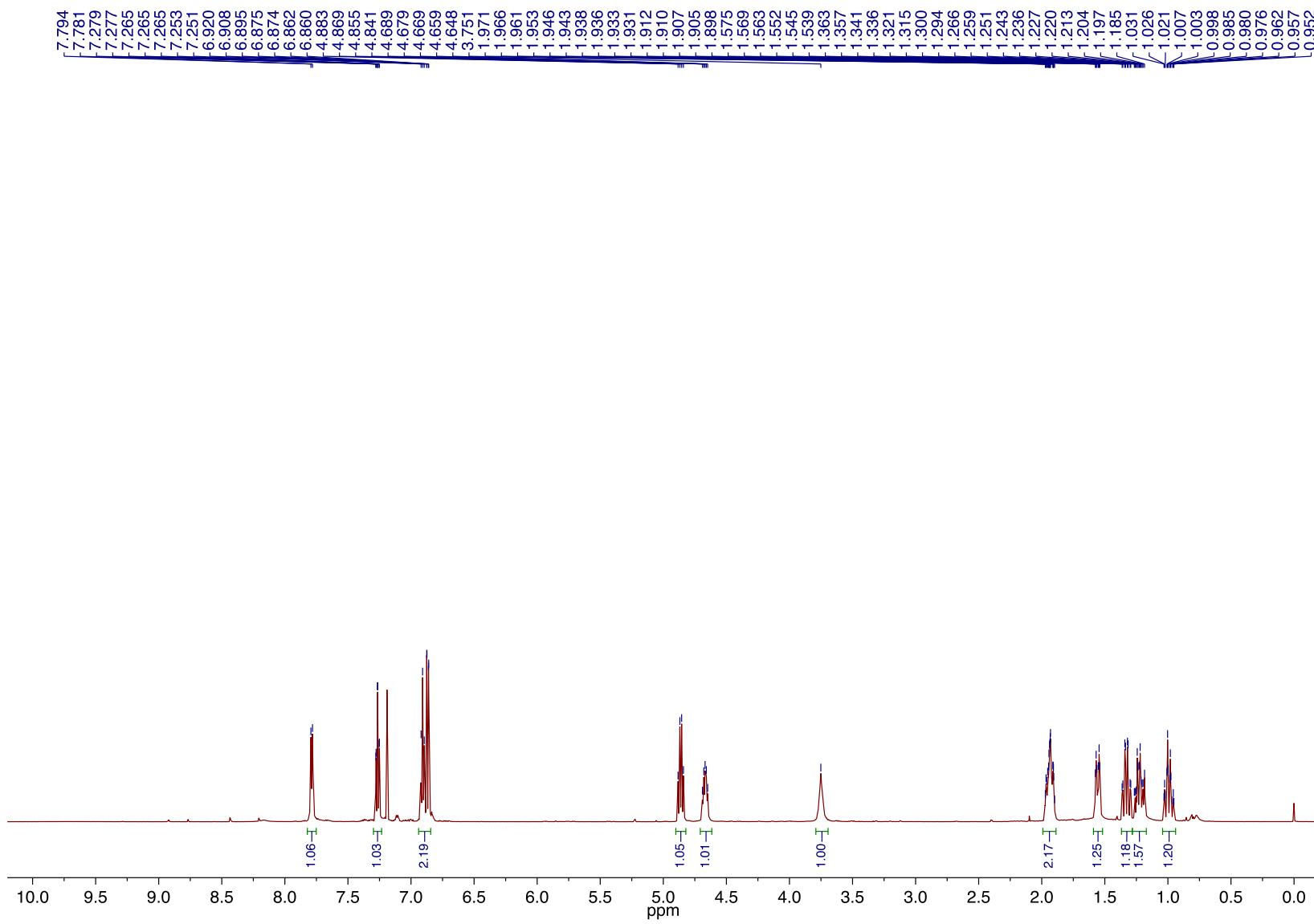
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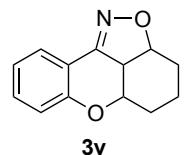
¹³C NMR (101 MHz, CDCl₃)



¹H NMR (600 MHz, CDCl₃)



¹³C NMR (151 MHz, CDCl₃)



3v

