Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Photocatalytic xanthate-based radical addition/cyclization reaction sequence toward 2-biphenyl isocyanides: synthesis of 6-alkylated phenanthridines

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Content

1 General Information

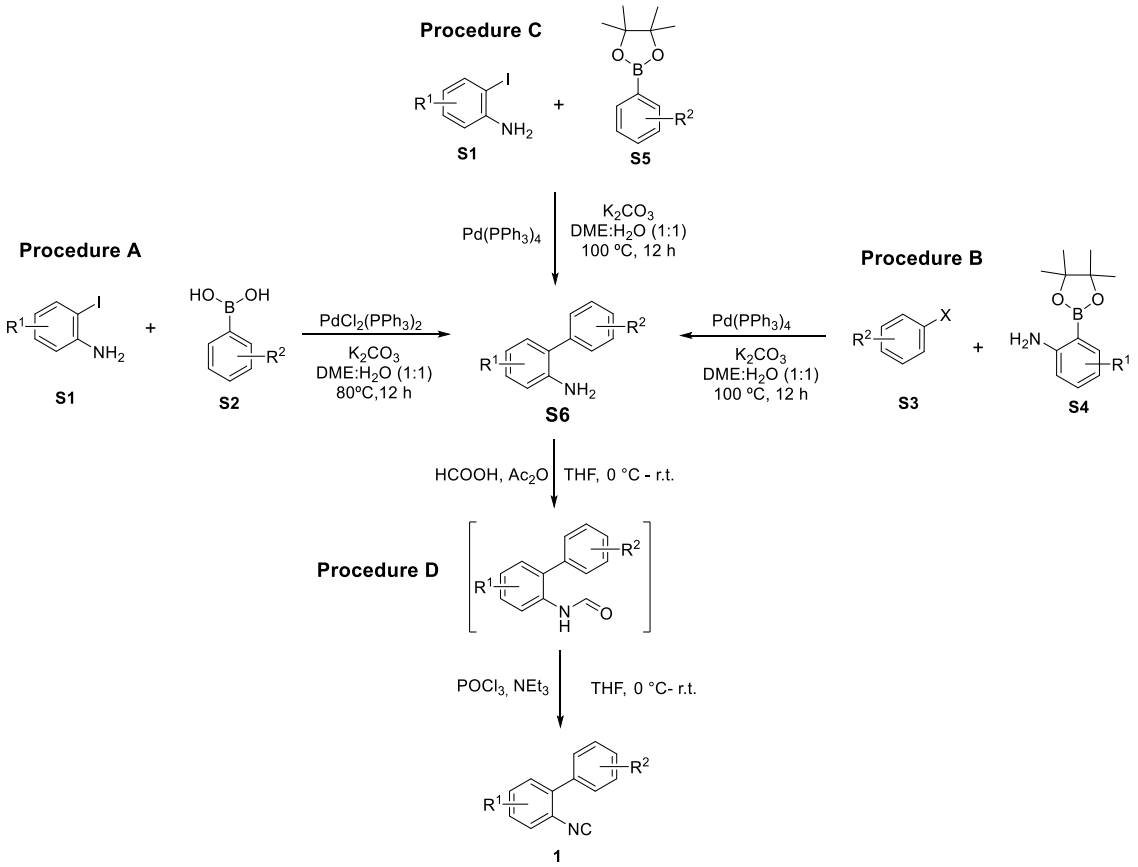
Solvents and reagents were purchased from Sigma-Aldrich and used without further purification. Catalysts ($Ir(ppy)_3$, $Ru(bpy)_3$ and $[Ir(dtbbpy)(ppy)_2]PF_6$) were purchased from Sigma-Aldrich and used without further purification. Reaction progress was monitored by TLC on silica gel plates 60 F254 purchased from Merk and the spots were detected either by short-wave UV light (254 nm) or by using vanillin stain followed by heating. Flash column chromatography was performed using Aldrich silica gel 230−400 mesh with hexane and ethyl acetate as eluents. ^1H and ^{13}C NMR spectra were recorded on Jeol Eclipse-300 MHz, Bruker Avance III 400 MHz and Bruker Fourier 300 MHz model spectrometers using $CDCl_3$ as solvent and reference ($CDCl_3$ δ =7.26 for ^1H, δ =77.16for ^{13}C). Chemical shifts are reported as parts per million. NMR coupling constants are reported in hertz (Hz). Low- and high-resolution $DART^+$ mass spectra were obtained on Jeol JMST100LC spectrometer. FT-IR spectral data were recorded in a Bruker Tensor 27 spectrometer. Fluorescence experiments were measured on a spectrofluorometer FS5 from Edinburgh instruments. Melting points were determined on a Fisher apparatus and are not corrected. Reactions were carried out in a handmade photoreactor equipped with six 4 W LEDs (24 W) and a fan under an argon atmosphere[1].

[1] D. A. Contreras-Cruz, M. Cantú-Reyes, J. M. García-Sanchez, D. Peña-Ortíz, M. A. Sanchez-Carmona, L. D. Miranda, *J. Chem. Educ.* 2019, **96**, 2015-2020.

2 Preparation of starting materials

2.1 Preparation of isocyanides

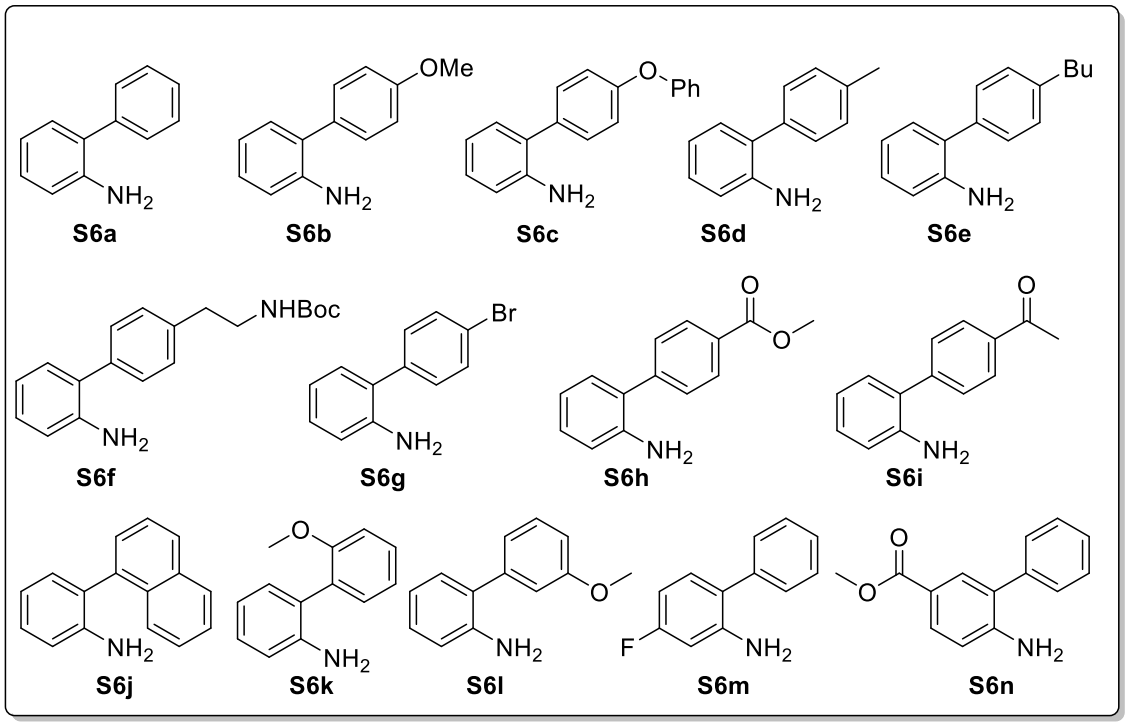
Isocyanides **1a-n** were prepared in three steps according to the next scheme:



2.1.1 Preparation of amines

Amines **S6a**, **S6b**, **S6e**, **S6g**, **S6j**, **S6m**, **S6n** were prepared according to previously reported procedure **A**[2]. Amines **S6d**, **S6h, S6k** and **S6l** were prepared according to previously reported procedure **B**[3]. Amines **S6c**, **S6f** and **S6i** were prepared according to previously reported procedure **C**[3].

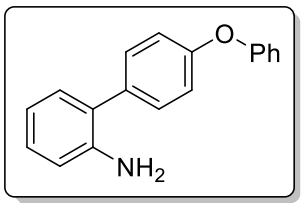
[2] M. Tobisu, K. Koh, T. Furukawa, N. Chatani, *Angew. Chem. Int. Ed*., 2012, **51**, 11363-1136.
[3] T. Chatterjee, G. B. Roh, M. A. Shoaib, C. H. Suhl, J. S. Kim, C. G. Cho, E. J. Cho, *Org. Lett.*, 2017, **19**, 1906-1909.



2.1.2 Analytical data of amines

Analytical data of amines **S6a**[4], **S6b**[4], **S6d**[4], **S6g**[4], **S6h**[5], **S6**i[6], **S6j**[3], **S6k**[7], **S6**l[8], **S6m**[4], **S6n**[9] agree with those previously reported.

4'-phenoxy-[1,1'-biphenyl]-2-amine (S6c):

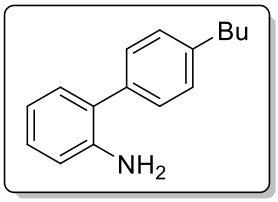


Following the **general procedure C**, **S6c** was obtained as a yellow solid (60% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 – 7.30 (m, 4H), 7.34 – 6.98 (m, 7H), 6.92 – 6.69 (m, 2H), 3.80 (br, 2H). ^{13}C

[4] J. Rong, L. Deng, P. Tan, C. Ni, Y. Gu, J. Hu, *Angew.Chem. Int.Ed.,* 2016, **55**, 2743-2747.
[5] S. Parisien-Collette, C. Cruché, X. Abel-Snape, S. K. Collins, *Green Chem.*, 2017, **19**, 4798-4803.
[6] E. Ullah, J. McNulty, A. Robertson, Eur. J. Org. Chem., 2012, 2127–2131
[7] F. Willig, J. Lang, A. C. Hans, M. R. Ringenberg, D. Pfeffer, W. Frey, R. Peters, *J. Am. Chem. Soc*. 2019, **141**, 12029-12043.
[8] S. M. Elbert, P. Wagner, T. Kanagasundaram, F. Rominger, M. Mastalerz, *Chem. Eur. J.* 2017, **23**, 935-945.
[9] K. J. Kayser-Bricker, M. P. Glenn, S. H. Lee, S. M. Sebti, J. Q. Cheng, A. D. Hamilton, *Bioorg. Med. Chem.*, 2009, **17**, 1764-1771.

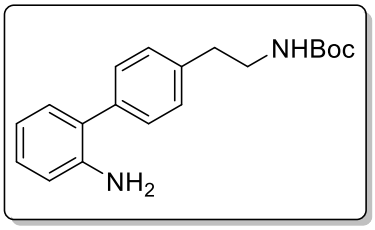
NMR (75 MHz, $CDCl_3$) δ 157.1, 156.7, 143.7, 134.4, 130.6, 129.9, 128.5, 127.1, 123.6, 119.3, 119.0, 118.8, 115.7.

4'-butyl-[1,1'-biphenyl]-2-amine (S6e):



Following the **general procedure B**, **S6e** was obtained as a white solid (80% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$ δ 7.41 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.23 – 7.14 (m, 2H), 6.90 – 6.77 (m, 2H), 3.72 (br, 2H), 2.70 (t, *J* = 7.3 Hz, 2H), 1.79 – 1.25 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.7, 142.0, 136.8, 130.6, 129.0, 128.9, 128.4, 127.8, 118.7, 115.6, 35.5, 33.8, 22.6, 14.1.

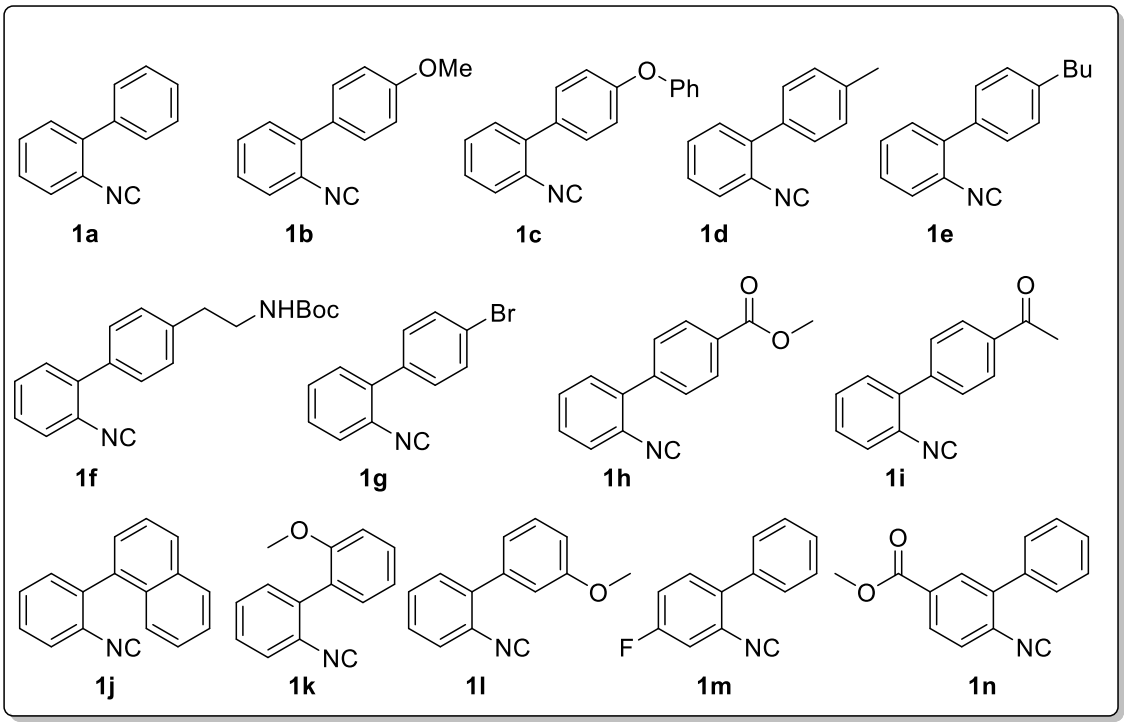
tert-butyl (2-(2'-amino-[1,1'-biphenyl]-4-yl)ethyl)carbamate (S6f):



Following the **general procedure C**, **S6f** was obtained as an orange oil (82% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.26 – 7.15 (m, 2H), 6.94 – 6.78 (m, 2H), 4.94 (br, 1H), 3.86(br, 2H), 3.48 (q, *J* = 7.1 Hz,2H), 2.90 (t, *J* = 7.1 Hz, 2H), 1.53 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.9, 143.6, 137.9, 137.5, 130.3, 129.2, 128.3, 127.2, 118.5, 115.5, 79.1, 41.7, 35.9, 28.4.

2.1.3 Preparation of isocyanides

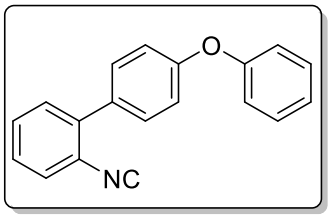
All isocyanides (**1a-m**) were prepared according to procedure **D**[4].



2.1.4 Analytical data of isocyanides

Analytical data of isocyanides **1a**[4], **1b**[4], **1d**[4], **1g**[4], **1h**[10], **1**i[11], **1k**[4], **1**l[4] and **1m**[4] agree with those previously reported.

2-isocyano-4'-phenoxy-1,1'-biphenyl (1c):

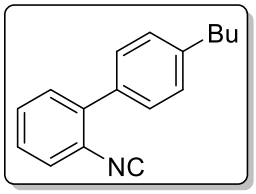


Following the **general procedure D**, **1c** was obtained as a green oil (85% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 – 7.33 (m, 8H), 7.22 – 7.08 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 157.8, 156.6, 138.2, 131.7, 130.5, 130.0, 129.6, 128.1, 127.9, 123.9, 119.6, 118.4. HRMS (DART) calc. for $C_{19}H_{14}NO$ $[M + H]^+$

[10] W. Wang, Y. Guo, K. Sun, S. Wang, S. Zhang, C. Liu, Q.-Y. Chen, *J. Org. Chem.*, 2018, **83**, 14588-14599.
[11] W. Wan, X. Xu, Y. Chen, H. Jiang, Y. Wang, H. Deng, J. Hao, *Eur. J. Org. Chem*, 2017, 3145-3151.

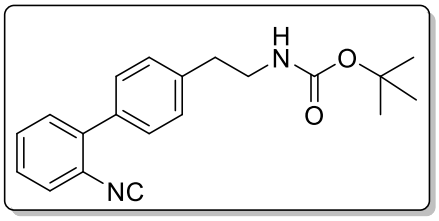
272.10754, found 272.10809. IR (KBr, cm^{-1}): 3060,2924, 2853, 2122, 1586, 1511, 1479, 1233, 850, 760.

4'-butyl-2-isocyano-1,1'-biphenyl (1e):



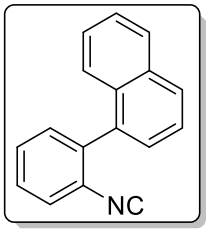
Following the **general procedure D**, **1e** was obtained as a green oil (83% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 – 7.41 (m, 5H), 7.39 – 7.33 (m, 1H), 7.33 (d, *J* = 9.0 Hz, 2H), 2.70 (t, J = 9.0 Hz, 2H), 1.75 – 1.58 (m, 2H), 1.53 – 1.33 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 143.3, 138.9, 134.3, 130.6, 129.6, 128.9, 128.7, 127.9 (2C), 124.6, 35.5, 33.6, 22.5, 14.1. HRMS (DART) calc. for $C_{17}H_{18}N$ $[M + H]^+$ 236.14392, found 236.14293. IR (KBr, cm^{-1}): 3063, 3027, 2957, 2929, 2859, 2120, 1480, 760.

tert-butyl (2-(2'-isocyano-[1,1'-biphenyl]-4-yl)ethyl)carbamate (1f):



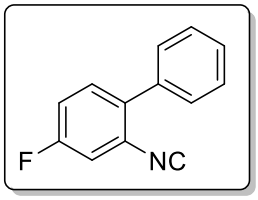
Following the **general procedure D**, **1f** was obtained as brown oil (40% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.50 – 7.34 (m, 6H), 7.30 (d, *J* = 8.1 Hz, 2H), 4.69 (s, 1H), 3.45 – 4.39 (m, 2H), 2.86 (t, *J* = 7.1 Hz, 2H), 1.44 (s. 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.5, 156.0, 139.4, 138.6, 135.1, 130.6, 129.6, 129.2, 129.1, 128.1, 127.9, 124.5, 79.3, 41.7, 36.1, 28.5. HRMS (DART) calc. for $C_{20}H_{23}N_2O_2$ $[M + H]^+$ 323.17595, found 323.17551. IR (KBr, cm^{-1}): 3333, 2975, 2930, 2871, 1694, 1518, 1447, 1276, 1250, 1169, 757.

1-(2-isocyanophenyl)naphthalene (1j):



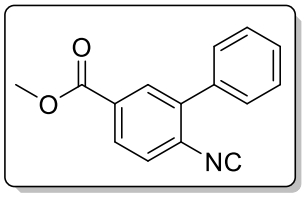
Following the **general procedure D**, **1j** was obtained as a green solid (80% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 – 7.92 (m, 2H), 7.68 – 7.40 (m, 9H). ^{13}C NMR (75 MHz, $CDCl^3$) δ 166.3, 138.1, 134.9, 133.7, 131.9, 131.4, 129.2, 129.1, 128.6, 128.6, 127.5, 127.3, 126.6, 126.2, 125.3 (2C), 121.4. HRMS (DART) calc. for $C_{17}H_{12}N$ $[M + H]^+$ 230.09697, found 230.09638. IR (KBr, cm^{-1}): 3056, 2125, 1509, 1486, 1437, 1392, 807, 777, 763.

4-fluoro-2-isocyano-1,1'-biphenyl (1m):



Following the **general procedure D**, **1m** was obtained as a green solid (85% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 – 7.36 (m, 6H), 7.29 – 7.13 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.96, 161.44 (d, J = 249.6 Hz), 136.12, 135.28 (d, J = 3.7 Hz), 132.08 (d, J = 8.7 Hz), 128.98, 128.69, 128.53, 125.3, 117.18 (d, J = 21.0 Hz), 114.97 (d, J = 25.4 Hz). HRMS (DART) calc. for $C_{13}H_9FN$ $[M + H]^+$ 198.07190, found 198.07190. IR (KBr, cm^{-1}): 3068, 2124, 1607, 1508, 1479, 1267, 1141, 948, 865, 839, 770, 700. Analytical data agree with those previously reported[9].

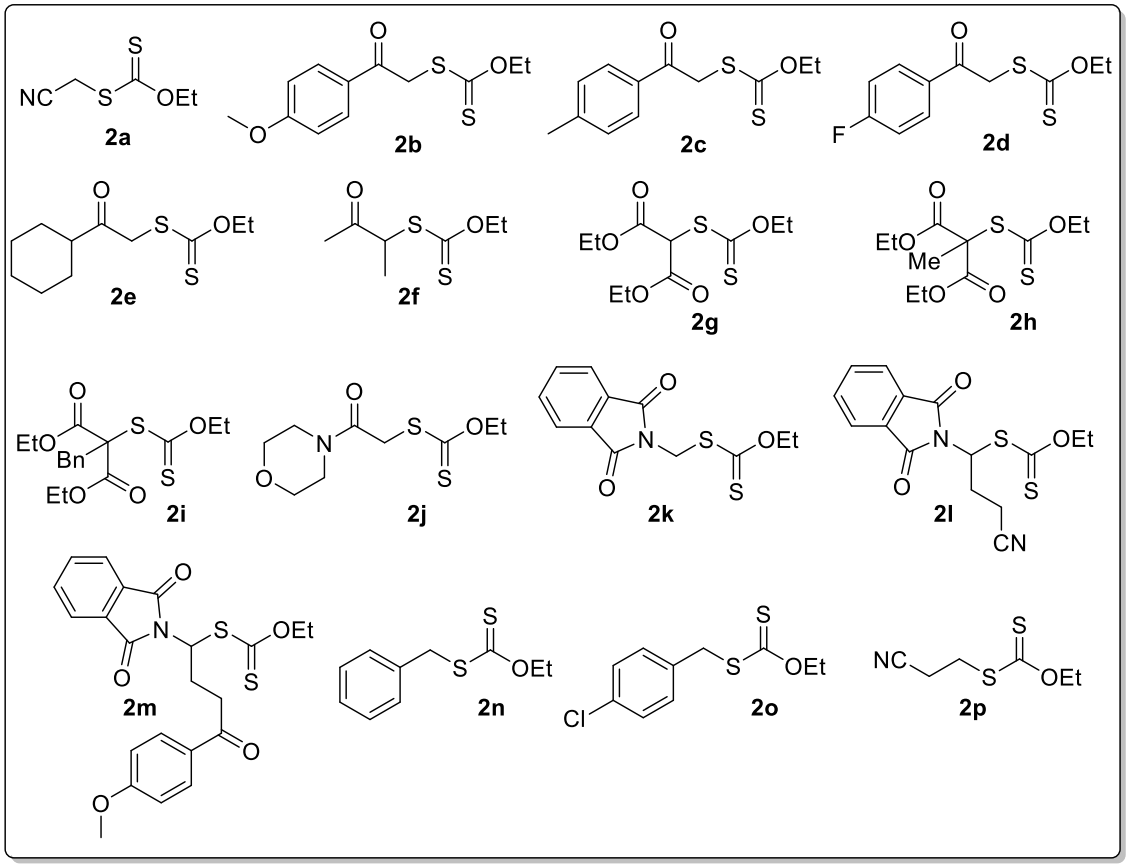
methyl 6-isocyano-[1,1'-biphenyl]-3-carboxylate (1n):



Following the **general procedure D**, **1n** was obtained as a white solid (80% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12 (dd, J = 1.9, 0.5 Hz, 1H), 8.04 (dd, J = 8.3, 1.9 Hz, 1H), 7.61 – 7.41 (m, 6H), 3.94 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.4, 165.6, 140.8, 139.1, 136.1, 132.0, 131.1, 129.3, 129.0, 128.9, 128.8, 128.1, 52.7. HRMS (DART) calc. for $C_{15}H_{12}NO_2$ $[M + H]^+$ 238.08680, found 238.08603. IR (KBr, cm^{-1}): 3080, 3047, 2952, 2120, 1725, 1429, 1313, 1282, 1244, 1126, 1106, 766.

2.2 Preparation of xanthates

Xanthates **2a**[12], **2b**[12], **2c**[12], **2d**[12], **2e**[12], **2f**[12], **2g**[12], **2h**[13], **2i**[13], **2j**[14], **2k**[12], **2l**[15], **2m**[16], **2n**[12], **2o**[12], **2p**[12] were prepared according with previously reported methods.

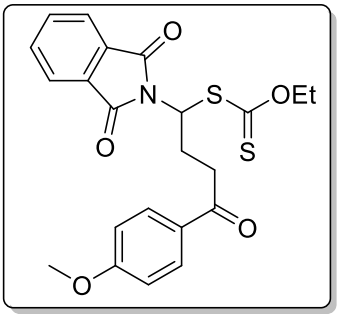


[12] V. Liautard, F. Robert, Y. Landais, *Org. Lett.*, 2011, **13**, 2658–2661.
[13] Contreras-Cruz, D. A. (2018). Síntesis de ciclopropanos funcionalizados utilizando una secuencia de reacciones de Ugi/Corey-Chaykovsky y un estudio sintético para la preparación del alcaloide tronocarpina. (Doctoral dissertation). Retrieved from http://132.248.9.195/ptd2018/junio/0775838/Index.html
[14] L. D. Miranda, E. Icelo-Ávila, Á. Rentería-Gõmez, M. Pila, J. G. Marrero, *Eur. J. Org. Chem.* 2015, 4098–4101.
[15] P. López-Mendoza, J. E. Díaz, A. E. Loaiza, L. D. Miranda, *Tetrahedron,* 2018, **74**, 5494-5502.
[16] B. Quiclet-Sire, G. Revol, S. Z. Zard *Tetrahedron*, 2010, **66**, 6656-6666.

2.2.1 Analytical data of xanthates

Analytical data of xanthates **2a**[12], **2b**[17], **2c**[18], **2d**[18], **2e**[19], **2f**, **2g**[20], **2h**[13], **2i**[13], **2j**[21], **2k**[22], **2l**[16], 2m, **2n**[23], **2o**[24], **2p**[25] agree with those previously reported.

S-(1-(1,3-dioxoisoindolin-2-yl)-4-(4-methoxyphenyl)-4-oxobutyl) O-ethyl carbonodithioate (2m):

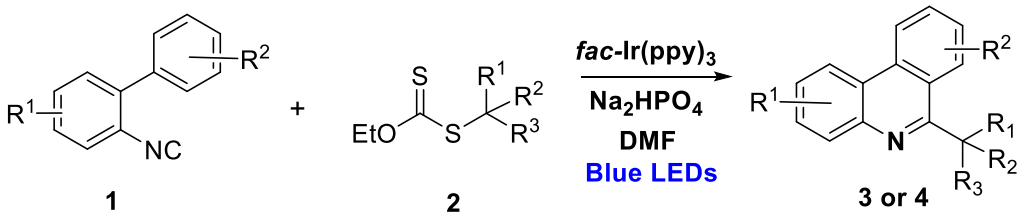


Following the procedure of reference 16, **2m** was obtained as a colorless oil (30% yield) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97 – 7.77 (m, 4H), 7.78 – 7.69 (m, 2H), 6.89 (d, *J* = 8.9 Hz, 2H), 6.38 (t, *J* = 7.9 Hz, 1H), 4.60 (qd, *J* = 7.1, 2.5 Hz, 2H), 3.85 (s, 3H), 3.14 – 3.02 (m, 2H), 2.73 – 2.56 (m, 2H), 1.38 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (76 MHz, $CDCl_3$) δ 211.2, 196.5, 166.9, 163.7, 134.5, 131.7, 130.5, 129.7, 123.8, 113.9, 70.7, 57.6, 55.6, 35.1, 28.3, 13.8.

[17] R. F. Guignard, S. Z. Zard, *Chem. Commun.,* 2011, **47**, 12185-12187.
[18] N. Sawengngen, P. N. Chalikidi, S. Araby, F. Hampel, P. Gmeiner, O. V. Serdyuk *Org. Biomol. Chem.,* 2019, **17**, 4850-4855.
[19] A. Gheorghe, B. Quiclet-Sire, X. Vila, S. Z. Zard, *Org. Lett.*, 2005, **7**, 1653-1656.
[20] S. Fabre, X. Vila, S. Z. Zard, *Chem. Commun.,* 2006, 4964-4966.
[21] A. Lilienkampf, S. Karkola, S. Alho-Richmond, P. Koskimies, N. Johansson, K. Huhtinen, K. Vihko, K. Wahala, *J. Med. Chem.,* 2009, **52**, 6660–6671.
[22] F. Pennetreau, O. Riant, S. Hermans, *Chem. Eur. J.,* 2014, **20**, 15009 -15012.
[23] L. Liu, D. Lu, H. Wang, Q. Dong, P. Wang, R. Bai, *Chem. Commun.,* 2011, **47**, 7839-7841.
[24] A. R. Katritzky, U. Gruntz, N. Mongelli, M. C. Rezende, *J. Chem. Soc., Perkin Trans. 1,* 1979, 1953-1956.
[25] A. W. Erian, D. L. Reid, J. Warketin, *Journal of Sulfur Chemistry*, 2005, **26**, 203-209.

3 Synthesis of 6-alkylated phenanthridines

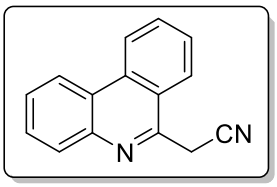
3.1 General procedure H: synthesis of 6-alkylated phenanthridines



In a 4.0 mL glass vial equipped with a stirring bar, were added consecutively, isocyanide **1** (2.0 equiv, 0.4 mmol), xanthate **2** (1.0 equiv, 0.2 mmol) ***fac*-Ir(ppy)$_3$** (0.02 equiv, 0.004 mmol), **Na_2HPO_4** (1.2 equiv, 0.24 mmol) and **DMF** (0.8 mL, 0.25 M). The resulting solution was degassed by three consecutive freeze−pump−thaw cycles and backfilled with pure argon. Finally, the reaction mixture was stirred and irradiated in a Blue LEDs reactor (24 W) at 30 °C. After consumption of the xanthate (as judged by TLC), the mixture was concentrated in *vacuo* and the product was purified by flash column chromatography on silica gel to afford the desired phenanthridine **3** or **4**.

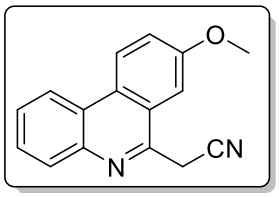
3.1.1 Analytical data of phenanthridines

(phenanthridin-6-yl)acetonitrile (3a):



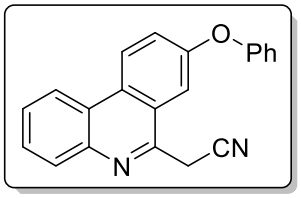
Following the **general procedure H**, **3a** was obtained as a brown solid (75% yield, 31.8 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. m.p 110 – 112 °C**.** ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.60 (dt, *J* = 8.3, 0.6 Hz, 1H), 8.53 – 8.46 (m, 1H), 8.20 – 8.11 (m, 1H), 8.08 – 8.02 (m, 1H), 7.85 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1Hj), 7.77 – 7.63 (m, 3H), 4.40 (s, 2H**).** ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.2, 143.3, 133.2, 131.3, 130.2, 129.2, 128.0, 127.8, 125.1, 124.2, 124.1, 122.9, 122.1, 116.6, 26.1. HRMS (DART) calc. for $C_{15}H_{11}N_2$ $[M + H]^+$ 219.09222, found 219.09276. IR (KBr, cm^{-1}): 3066, 2945, 2921, 2255, 1609, 1583, 1529, 1486, 1447, 1366, 785, 720.

2-(8-methoxyphenanthridin-6-yl)acetonitrile (3b):



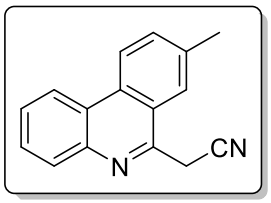
Following the **general procedure H**, **3b** was obtained as a brown solid (76% yield, 37.5 mg) after purification *via* flash column chromatography using silica gel using a mixture of Hex:AcOEt (9:1) as eluent. m. p. 128 - 130 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.55 (d, *J* = 9.1 Hz, 1H), 8.49 – 8.42 (m, 1H), 8.16 – 8.09 (m, 1H), 7.75 – 7.62 (m, 2H), 7.50 (dd, *J* = 9.1, 2.5 Hz, 1H), 7.35 (d, *J* = 2.5 Hz, 1H), 4.39 (s, 2H), 4.00 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.2, 149.2, 142.6, 130.1, 128.2, 127.9, 127.7, 125.5, 124.7, 124.4, 121.8, 121.7, 116.6, 105.3, 55.8, 26.3. HRMS (DART) calc. for $C_{16}H_{13}N_2O$ $[M + H]^+$ 249.10279, found 249.10216. IR KBr, cm^{-1}):3062, 2922, 2848, 2256, 1615, 1581, 1533, 1486, 1460, 1378, 1220, 1034, 831, 748.

2-(8-phenoxyphenanthridin-6-yl)acetonitrile (3c):



Following the **general procedure H**, **3c** was obtained as a red solid (41% yield, 25.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m.p 35 – 37 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.64 (d, *J* = 8.5 Hz, 1H), 8.54 – 8.44 (m, 1H), 8.21 – 8.12 (m, 1H), 7.81 – 7.63 (m, 2H), 7.65 – 7.55 (m, 2H), 7.51 – 7.38 (m, 2H), 7.24 (d, *J* = 8.2 Hz, 1H), 7.21 – 7.07 (m, 2H), 4.31 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.1, 156.2, 149.4, 142.9, 130.3, 130.3, 129.0, 128.7, 128.0, 125.4, 125.1, 124.6, 124.1, 123.5, 121.8, 119.6, 116.3, 112.0, 26.2. HRMS (DART) calc. for $C_{21}H_{15}N_2O$ $[M + H]^+$ 311.11844, found 311.11833. IR (KBr, cm^{-1}): 3064, 2921, 2852, 2186, 1736, 1714, 1591, 1486, 1460, 1245, 1220, 762.

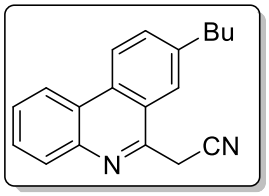
2-(8-methylphenanthridin-6-yl)acetonitrile (3d):



Following the **general procedure H**, **3d** was obtained as a brown solid (37% yield, 16.9 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m.p. 112 – 113 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.64 – 8.47 (m, 1H), 8.20 – 8.10 (m, 1H), 7.86 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.80 – 7.63 (m, 3H), 4.42 (s, 2H), 2.63 (s, 3H). ^{13}C NMR (75 MHz,

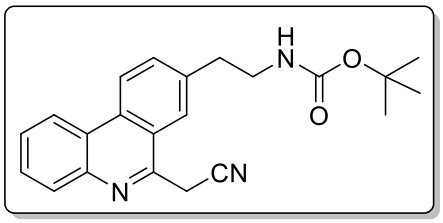
$CDCl_3$) δ 149.9, 143.1, 138.3, 133.2, 131.3, 130.1, 128.8, 127.8, 124.6, 124.4, 122.9, 122.0, 116.7, 26.2, 22.0. HRMS (DART) calc. for $C_{16}H_{13}N_2$ $[M + H]^+$ 233.10787, found 233.10774. IR (KBr, cm^{-1}): 3063, 2922, 2853, 2253, 1752, 1657, 1622, 1580, 1483, 1460, 1369, 1341, 763.

2-(8-butylphenanthridin-6-yl)acetonitrile (3e):



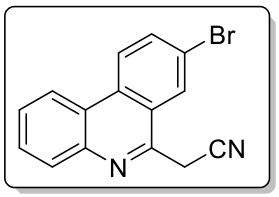
Following the **general procedure H**, **3e** was obtained as a red oil (52% yield, 28.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. ^1H NMR (300 MHz, $CDCl_3$) δ 8.57 (d, *J* = 8.6 Hz, 1H), 8.52 (dd, *J* = 7.7, 2.0 Hz, 1H), 8.15 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.87 – 7.81 (m, 1H), 7.79 – 7.63 (m, 3H), 2.94 – 2.80 (m, 2H), 1.82 – 1.66 (m, 2H), 1.55 – 1.32 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.0, 143.2, 143.1, 132.8, 132.4, 131.4, 128.7, 127.7, 124.4, 124.0, 122.9, 122.0, 116.7, 36.0, 33.7, 26.2, 22.5, 14.1. HRMS (DART) calc. for $C_{19}H_{19}N_2$ $[M + H]^+$ 275.15482, found 275.15554. IR (KBr, cm^{-1}): 2956, 2928, 2859, 2253, 1755, 1622, 1579, 1483, 1461, 1372, 1343, 763.

tert-butyl (2-(6-(cyanomethyl)phenanthridin-8-yl)ethyl)carbamate (3f):



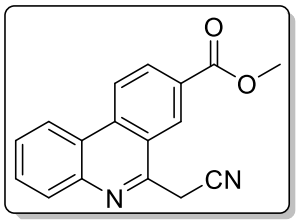
Following the **general procedure H**, **3f** was obtained as a brown oil (66% yield, 47.7 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. ^1H NMR (300 MHz, $CDCl_3$) δ 8.65 – 8.35 (m, 2H), 8.18 – 8.05 (m, 1H), 7.99 – 7.79 (m, 1H), 7.79 – 7.52 (m, 3H), 7.08 (s, 1H), 4.61 (br, 1H), 4.37 (s, 1H), 3.76 – 3.23 (m, 2H), 3.01 (t, *J* = 6.0 Hz, 2H), 1.36 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.1, 150.0, 148.8, 143.2, 142.7, 139.8, 139.4, 132.9, 132.5, 130.5, 130.4, 130.2, 129.4, 129.2, 129.0, 128.5, 128.3, 127.9, 124.8, 124.4, 124.2, 123.9, 123.9, 123.6, 123.3, 122.1, 122.1, 117.2, 116.7, 79.7, 41.8, 41.7, 36.8, 36.7, 36.2, 32.5, 28.5, 28.5, 26.2, 19.8. HRMS (DART) calc. for $C_{22}H_{24}N_3O_2$ $[M + H]^+$ 362.18685, found 362.18571. IR (KBr, cm^{-1}): 3363, 2875, 2929, 2856, 2252, 1702, 1517, 1366, 1250, 1169, 764.

2-(8-bromophenanthridin-6-yl)acetonitrile (3g):



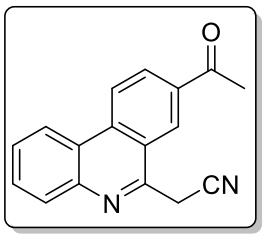
Following the **general procedure H**, **3g** was obtained as a brown solid (35% yield, 20.5 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m.p. 180 – 182 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.57 – 8.46 (m, 2H), 8.21 (d, *J* = 2.0 Hz, 1H), 8.18 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.97 (dd, *J* = 8.9, 2.0 Hz, 1H), 7.79 (ddd, *J* = 8.2, 7.1, 1.6 Hz, 1H), 7.72 (ddd, *J* = 8.5, 7.1, 1.5 Hz, 1H), 4.42 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.0, 143.3, 134.6, 132.1, 130.5, 129.7, 128.4, 127.6, 125.4, 124.9, 123.7, 122.2, 122.0, 116.2, 26.1. HRMS (DART) calc. for $C_{15}H_{10}{}^{81}BrN_2$ $[M + H]^+$ 299.00069, found 299.00116. IR (KBr, cm^{-1}): 3078, 3057, 3033, 2923, 2851, 2253, 1579, 1520, 1476, 1413, 1363, 1195, 1090, 1002, 830, 761.

methyl 6-(cyanomethyl)phenanthridine-8-carboxylate (3h):



Following the **general procedure H**, **3h** was obtained as a yellow solid (65% yield, 35.3 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m.p. 208 – 210 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.81 (s, 1H), 8.74 (d, *J* = 8.7 Hz, 1H), 8.60 (d, *J* = 8.1 Hz, 1H), 8.23 (d, *J* = 8.0 Hz, 1H), 7.85 (t, *J* = 7.6 Hz, 1H), 7.76 (t, *J* = 7.5 Hz, 1H), 4.55 (s, 2H), 4.06 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 150.7, 144.2, 136.3, 131.1, 130.6, 130.4, 129.5, 128.3, 127.3, 127.2, 123.4, 122.8, 122.7, 116.3, 52.8, 26.2. HRMS (DART) calc. for $C_{17}H_{13}N_2O_2$ $[M + H]^+$ 277.09770, found 277.09798. IR (KBr, cm^{-1}): 3074, 3007, 2952, 2924, 2852, 2254, 1714, 1614, 1581, 1439, 1375, 1309, 1269, 1113, 754.

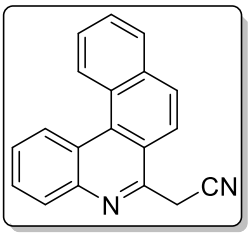
2-(8-acetylphenanthridin-6-yl)acetonitrile (3i):



Following the **general procedure H**, **3i** was obtained as a brown solid (60% yield, 29.9 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.75 (d, *J* = 8.7 Hz, 1H), 8.71 (d, *J* = 1.7 Hz, 1H), 8.59 (dd, *J* = 8.2, 1.5 Hz, 1H), 8.43 (dd, *J* = 8.6, 1.7 Hz, 1H), 8.22 (dd, *J* = 8.2, 1.4 Hz, 1H),

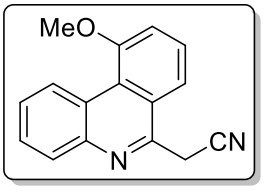
7.84 (ddd, *J* = 8.2, 7.1, 1.6 Hz, 1H), 7.76 (ddd, *J* = 8.3, 7.0, 1.5 Hz, 1H), 4.54 (s, 2H), 2.80 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.0, 150.9, 144.2, 136.4, 136.0, 130.6, 130.5, 130.0, 128.4, 125.9, 123.8, 123.7, 123.6, 122.8, 116.3, 27.0, 26.3. HRMS (DART) calc. for $C_{17}H_{13}N_2O$ $[M + H]^+$ 261.10279, found 261.10231. IR

2-(benzo[k]phenanthridin-6-yl)acetonitrile (3j):



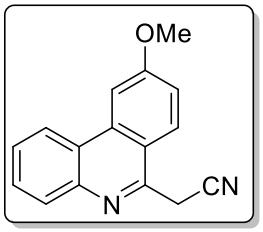
Following the **general procedure H**, **3j** was obtained as a brown solid (34% yield, 18.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m. p. 57 – 60 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.21 – 9.09 (m, 1H), 9.07 – 8.97 (m, 1H), 8.38 – 8.28 (m, 1H), 8.17 – 8.03 (m, 2H), 8.00 (d, *J* = 8.9 Hz, 1H), 7.87 – 7.68 (m, 4H), 7.26 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.3, 145.4, 134.9, 132.8, 130.1, 129.5, 129.2, 129.0, 128.9, 128.7, 128.5, 127.5, 127.5, 127.3, 124.6, 123.0, 121.1, 116.8, 26.6. HRMS (DART) calc. for $C_{19}H_{13}N_2$ $[M + H]^+$ 269.10787, found 269.10760. IR (KBr, cm^{-1}): 3957, 2923, 2852, 2193, 1694, 1615, 1479, 1389, 1233, 771, 755.

2-(10-methoxyphenanthridin-6-yl)acetonitrile (3k):



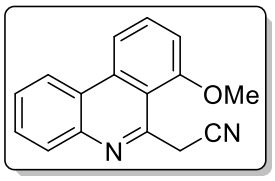
Following the **general procedure H**, **3k** was obtained as a brown solid (28% yield, 13.6 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m. p. 150 – 153 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.49 (dd, *J* = 8.2, 1.8 Hz, 1H), 8.22 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.94 – 7.57 (m, 4H), 7.40 (dd, *J* = 6.3, 2.8 Hz, 1H), 4.48 (s, 2H), 4.17 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 149.9, 129.6, 128.7, 128.5, 128.1, 127.9, 126.2, 124.2, 123.9, 117.4, 116.6, 112.6, 56.1, 26.5. HRMS (DART) calc. for $C_{16}H_{13}N_2O$ $[M + H]^+$ 249.10279, found 249.10190. IR (KBr, cm^{-1}): 2925, 2854, 1699, 1650, 1455, 1234, 1264, 1028, 764, 717.

2-(9-methoxyphenanthridin-6-yl)acetonitrile (3l):



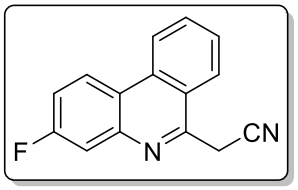
Following the **general procedure H**, **3l** was obtained as a brown oil (31% yield, 15.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (dd, *J* = 8.2, 1.5 Hz, 1H), 8.18 (dd, *J* = 8.1, 1.3 Hz, 1H), 8.07 (d, *J* = 9.0 Hz, 1H), 7.98 (d, *J* = 2.5 Hz, 1H), 7.78 (ddd, *J* = 8.3, 7.0, 1.5 Hz, 1H), 7.70 (ddd, *J* = 8.4, 7.1, 1.5 Hz, 1H), 7.37 (dd, *J* = 9.0, 2.5 Hz, 1H), 4.44 (s, 2H), 4.08 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0, 149.7, 135.7, 130.0, 129.4, 127.4, 127.2, 124.2, 122.2, 119.1, 118.4, 116.7, 103.7, 55.8, 26.0. HRMS (DART) calc. for $C_{16}H_{13}N_2O$ $[M + H]^+$ 249.10279, found 249.10198. IR (KBr, cm^{-1}): 2923, 2853, 2254, 1699, 1616, 1521, 1506, 1459, 1379, 1235, 1021, 762.

2-(7-methoxyphenanthridin-6-yl)acetonitrile (3l´):



Following the **general procedure H**, **3l´** was obtained as a brown solid (35% yield, 17.3 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. m. p. 154 – 157 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.49 (d, *J* = 8.1 Hz, 1H), 8.22 (d, *J* = 8.5 Hz, 1H), 8.12 (d, *J* = 8.1 Hz, 1H), 7.87 – 7.61 (m, 3H), 7.12 (d, *J* = 8.2 Hz, 1H), 4.60 (s, 2H), 4.08 (d, *J* = 1.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.6, 149.4, 143.2, 136.0, 131.9, 129.8, 129.4, 127.7, 123.6, 122.6, 117.9, 116.1, 115.0, 108.5, 56.0, 31.8. HRMS (DART) calc. for $C_{16}H_{13}N_2O$ $[M + H]^+$ 249.10279, found 249.10282. IR (KBr, cm^{-1}): 2959, 2928, 2852, 2251, 1067, 1582, 1458, 1270, 1254, 1094, 1012, 749.

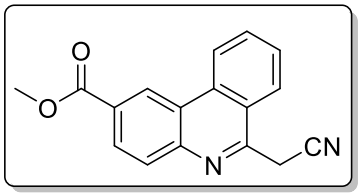
2-(3-fluorophenanthridin-6-yl)acetonitrile (3m):



Following the **general procedure H**, **3m** was obtained as a brown solid (56% yield, 26.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m.p. 145 – 148 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.60 (d, *J* = 8.3 Hz, 1H), 8.53 (dd, *J* = 9.1, 5.7 Hz, 1H), 8.11 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.91 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 1H), 7.82 (dd, *J* = 9.6, 2.8 Hz, 1H), 7.75 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.45 (tdd, *J* =

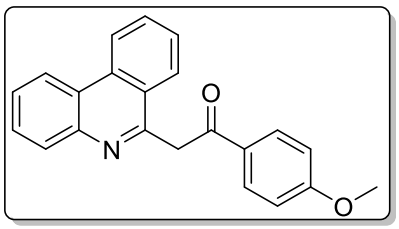
9.0, 2.8, 1.0 Hz, 1H), 4.45 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.89 (d, *J* = 249.3 Hz), 151.63, 144.61 (d, *J* = 14.1 Hz), 133.12, 131.75, 129.03, 127.98, 125.26, 124.14 (d, *J* = 9.5 Hz), 123.73, 122.81, 120.97, 116.95 (d, *J* = 23.8 Hz), 116.37, 114.81 (d, *J* = 20.7 Hz), 26.16. HRMS (DART) calc. for $C_{15}H_{10}FN_2$ $[M + H]^+$ 237.08280, found 237.08282. IR (KBr, cm^{-1}): 3071, 2926, 2853, 2254, 1616, 1584, 1485, 1461, 1407, 1372, 1204, 1154, 974, 848, 820, 762.

methyl 6-(cyanomethyl)phenanthridine-2-carboxylate (3n):



Following the **general procedure H**, **3n** was obtained as a brown solid (57% yield, 31.4 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. m.p. 147 – 150 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.30 (d, *J* = 1.8 Hz, 1H), 8.79 (d, *J* = 8.3 Hz, 1H), 8.36 (dd, *J* = 8.6, 1.8 Hz, 1H), 8.22 (d, *J* = 8.6 Hz, 1H), 8.15 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.97 (ddd, *J* = 8.3, 7.1, 1.3 Hz, 1H), 7.82 (ddd, *J* = 8.3, 7.0, 1.2 Hz, 1H), 4.49 (s, 2H), 4.04 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8, 152.6, 145.6, 133.4, 132.0, 130.4, 129.2, 129.0, 128.7, 125.3, 124.8, 124.3, 123.8, 123.2, 116.3, 52.6, 26.3. HRMS (DART) calc. for $C_{17}H_{13}N_2O_2$ $[M + H]^+$ 277.09770, found 277.09688. IR (KBr, cm^{-1}): 2951, 2923, 2851, 2192, 1716, 1642, 1614, 1594, 1294, 1269, 1246, 1110, 764.

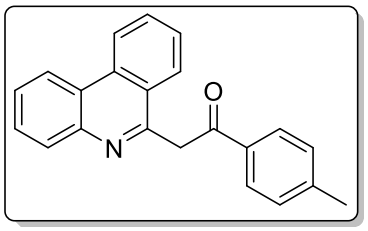
1-(4-methoxyphenyl)-2-(phenanthridin-6-yl)ethan-1-one (4a):



Following the **general procedure H**, **4a** was obtained as a yellow solid (72% yield, 47.1 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m.p.135 – 138 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 15.75 (s, 1H), 8.33 (d, *J* = 8.2 Hz, 1H), 8.25 (d, *J* = 8.2 Hz, 1H), 8.19 (d, *J* = 8.1 Hz, 1H), 8.03 (d, *J* = 9.2 Hz, 2H), 7.75 (t, *J* = 7.7 Hz, 1H), 7.64 – 7.53 (m, 1H), 7.53 – 7.41 (m, 2H), 7.30 (d, *J* = 7.6 Hz, 1H), 6.99 (d, *J* = 8.9 Hz, 2H), 6.78 (s, 1H), 3.89 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.5, 161.8, 152.4, 134.9, 133.4, 132.2, 132.1, 131.9, 129.9, 129.5, 128.8, 128.0, 127.2, 125.1, 124.6, 123.3, 122.6, 122.5, 122.1, 120.1, 118.0, 114.2, 113.6, 84.8, 55.4. HRMS (DART)

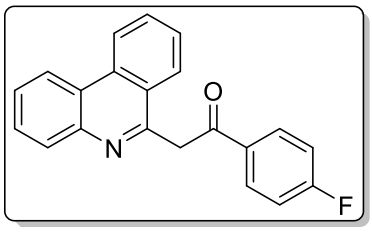
calc. for $C_{22}H_{18}NO_2$ $[M + H]^+$ 328.13375, found 328.13239. IR (KBr, cm^{-1}): 3069, 2955, 2926, 2840, 1594, 1552, 1507, 1345, 1249, 1217, 1172, 757.

2-(phenanthridin-6-yl)-1-(*p*-tolyl)ethan-1-one (4b):



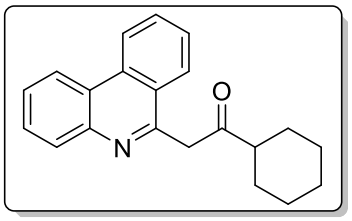
Following the **general procedure H**, **4b** was obtained as a brown solid (70% yield, 43.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (9:1) as eluent. m.p.105 – 108 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.33 (d, *J* = 8.2 Hz, 1H), 8.25 (d, *J* = 8.2 Hz, 1H), 8.19 (d, *J* = 8.1 Hz, 1H), 7.96 (d, *J* = 7.9 Hz, 2H), 7.75 (t, *J* = 7.7 Hz, 1H), 7.59 (d, *J* = 7.7 Hz, 1H), 7.55 – 7.39 (m, 2H), 7.31 – 7.29 (m, 3H), 2.43 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.1, 152.7, 141.2, 138.1, 134.9, 132.3, 132.1, 130.0, 129.2, 128.2, 127.1, 125.3, 124.6, 123.6, 122.8, 122.6, 120.3, 118.2, 85.2, 21.6. HRMS (DART) calc. for $C_{22}H_{18}NO$ $[M + H]^+$ 312.13884, found 312.13861. IR (KBr, cm^{-1}): 2923, 2854, 1599, 1554, 1346, 1204, 1180.

1-(4-fluorophenyl)-2-(phenanthridin-6-yl)ethan-1-one (4c):



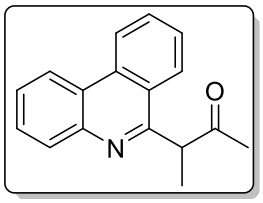
Following the **general procedure H**, **4c** was obtained as a yellow solid (55% yield, 34.4 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. m.p. 93 – 95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 15.78 (s, 1H), 8.32 (d, *J* = 8.2 Hz, 1H), 8.24 – 8.16 (m, 2H), 8.09 – 7.99 (m, 2H), 7.74 (t, *J* = 7.9 Hz, 1H), 7.65 – 7.39 (m, 3H), 7.36 – 7.27 (m, 1H), 7.21 – 7.09 (m, 2H), 6.72 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.75, 164.55 (d, *J* = 250.6 Hz), 152.96, 137.04, 134.69, 132.20, 130.04, 129.23 (d, *J* = 8.3 Hz), 128.23, 125.26, 124.41, 123.78, 122.78, 122.63, 120.41, 118.24, 115.39 (d, *J* = 21.6 Hz), 98.35, 85.00. HRMS (DART) calc. for $C_{21}H_{15}FNO$ $[M + H]^+$ 316.11377, found 316.11341. IR (KBr, cm^{-1}): 2924, 2854, 1596, 1348, 1213, 1153.

1-cyclohexyl-2-(phenanthridin-6-yl)ethan-1-one (4d):



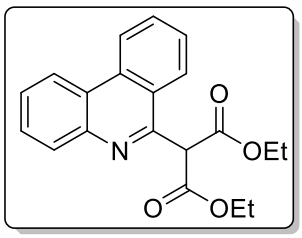
Following the **general procedure H**, **4d** was obtained as a red oil (45 % yield, 27.1 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 15.30 (s, aH), 8.29 (d, J = 8.2 Hz, 1H), 8.16 – 8.11 (m, 2), 7.71 (ddd, J = 8.3, 7.1, 1.3 Hz, 1H), 7.59 – 7.46 (m, 1H), 7.40 – 7.29 (m, 1H), 6.11 (s, 1H), 2.41 (tt, J = 11.7, 3.4 Hz, 1H), 2.08 – 1.26 (m, 10H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.2, 152.1, 135.1, 132.3, 131.9, 129.9, 128.1, 125.2, 124.5, 123.2, 122.7, 122.6, 120.0, 117.9, 86.7, 77.6, 50.8, 30.5, 26.4, 26.3. HRMS (DART) calc. for $C_{21}H_{22}NO$ $[M + H]^+$ 304.17014, found 304.17042. IR (KBr, cm^{-1}): 2926, 2852, 1703, 1601, 1552, 1442, 1337, 1216, 1154, 1111, 750.

3-(phenanthridin-6-yl)butan-2-one (4e):



Following the **general procedure H**, **4e** was obtained as a brown solid (51 % yield, 25.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. m.p.73 – 75 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.68 (d, *J* = 8.2 Hz, 1H), 8.57 (dd, *J* = 8.1, 1.2 Hz, 1H), 8.22 (d, *J* = 8.3 Hz, 1H), 8.15 (d, *J* = 8.0 Hz, 1H), 7.87 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1H), 7.82 – 7.55 (m, 3H), 4.73 (q, *J* = 7.0 Hz, 1H), 2.12 (s, 3H), 1.70 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.2, 160.1, 133.5, 130.8, 130.3, 128.9, 127.8, 127.2, 125.9, 125.0, 123.8, 122.9, 122.0, 53.7, 28.4, 16.0. HRMS (DART) calc. for $C_{17}H_{16}NO$ $[M + H]^+$ 250.12319, found 250.12279. IR (KBr, cm^{-1}): 2975, 2930, 1710, 1612, 1578, 1356, 1175, 1004, 757, 725.

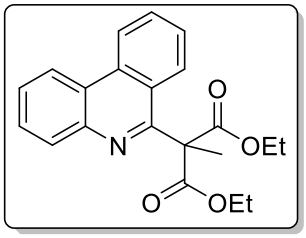
diethyl 2-(phenanthridin-6-yl)malonate (4f):



Following the **general procedure H**, **4f** was obtained as an orange solid (61% yield, 41.1 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. m.p. 103 – 105 ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.67 (d, *J* = 8.3 Hz,

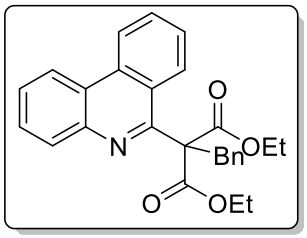
1H), 8.61 – 8.49 (m, 1H), 8.20 – 8.09 (m, 1H), 8.05 – 7.96 (m, 1H), 7.85 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 1H), 7.76 – 7.62 (m, 3H), 5.64 (s, 1H), 4.33 (qq, *J* = 7.1, 3.7 Hz, 4H), 1.28 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.7, 153.7, 143.5, 133.4, 130.8, 130.7, 128.8, 127.7, 127.5, 125.4, 125.2, 124.2, 122.8, 122.0, 62.2, 59.6, 14.2. HRMS (DART) calc. for $C_{20}H_{20}NO_4$ $[M + H]^+$ 338.13923, found 338.13909. IR (KBr, cm^{-1}): 3069, 2983, 2931, 1751, 1446, 1368, 1319, 1198, 1146, 1032.

diethyl 2-methyl-2-(phenanthridin-6-yl)malonate (4g):



Following the **general procedure H**, **4g** was obtained as a yellow pale solid (66% yield, 46.3 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. m. p. 88 - 91 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 – 8.63 (m, 1H), 8.58 – 8.50 (m, 1H), 8.09 – 8.03 (m, 1H), 7.92 (ddd, *J* = 8.4, 1.3, 0.6 Hz, 1H), 7.79 (ddd, *J* = 8.3, 7.0, 1.2 Hz, 1H), 7.73 – 7.57 (m, 3H), 4.32 (q, *J* = 7.1 Hz, 4H), 2.13 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 158.6, 142.8, 133.9, 130.6, 130.1, 128.7, 127.4, 126.8, 126.8, 124.0, 124.0, 122.9, 121.9, 63.8, 62.0, 22.2, 14.1. HRMS (DART) calc. for $C_{21}H_{22}NO_4$ $[M + H]^+$ 352.15488, found 352.15442. IR (KBr, cm^{-1}): 2982, 2929, 2853, 1729, 1577, 1461, 1253, 1111, 1019, 999, 765.

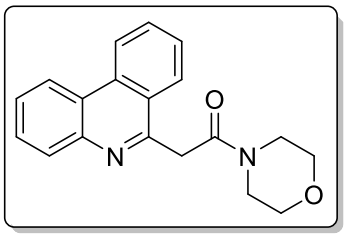
diethyl 2-benzyl-2-(phenanthridin-6-yl)malonate (4h):



Following the **general procedure H**, **4h** was obtained as a yellow pale oil (67% yield, 57.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.62 (d, *J* = 8.3 Hz, 1H), 8.55 (dd, *J* = 8.2, 1.4 Hz, 1H), 8.19 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.97 (d, *J* = 8.2 Hz, 1H), 7.81 – 7.63 (m, 3H), 7.51 (ddd, *J* = 8.3, 7.0, 1.2 Hz, 1H), 7.30 – 7.21 (m, 1H), 4.19 – 4.06 (m, 6H), 1.03 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 156.2, 142.8, 137.3, 133.5, 131.0, 130.6, 130.0, 128.7, 127.8, 127.5, 127.1, 126.7, 126.6, 125.3, 124.1, 122.5, 122.0, 67.6, 61.8, 41.4, 13.9. HRMS (DART) calc. for

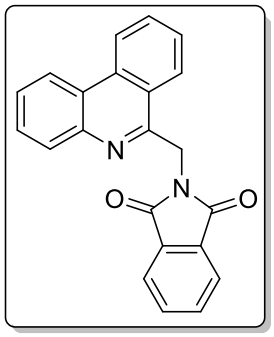
$C_{27}H_{26}NO_4$ $[M + H]^+$ 428.18618, found 428.18746. IR (KBr, cm^{-1}): 2984, 2958, 2937, 2903, 1768, 1579, 1490, 1458, 1264, 1168, 1147, 1080, 1043, 750.

1-morpholino-2-(phenanthridin-6-yl)ethan-1-one (4i):



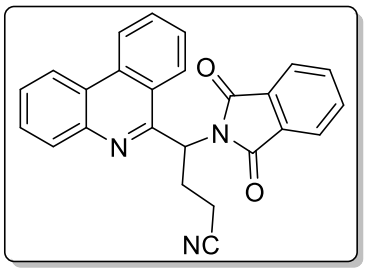
Following the **general procedure H**, **4i** was obtained as an orange oil (68% yield, 41.6 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (3:7) as eluent. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.65 (d, *J* = 8.2 Hz, 1H), 8.61 – 8.53 (m, 1H), 8.43 (d, *J* = 8.2 Hz, 1H), 8.13 – 8.04 (m, 1H), 7.86 (t, *J* = 7.7 Hz, 1H), 7.79 – 7.62 (m, 3H), 4.50 (s, 2H), 4.01 – 3.49 (m, 8H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.4, 156.0, 143.6, 133.2, 130.9, 129.9, 128.8, 127.9, 127.2, 126.8, 125.8, 124.0, 122.5, 122.2, 67.0, 66.9, 47.1, 43.0, 42.6. HRMS (DART) calc. for $C_{19}H_{19}N_2O_2$ $[M + H]^+$ 307.14465 , found 307.14456. IR (KBr, cm^{-1}): 3070, 2960, 2922, 2854, 1645, 1440, 1230, 1114, 756.

2-(phenanthridin-6-ylmethyl)isoindoline-1,3-dione (4j):



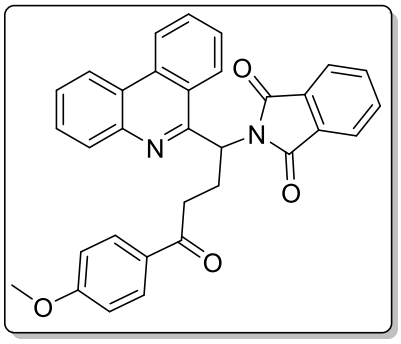
Following the **general procedure H**, **4j** was obtained as a yellow pale solid (46% yield, 31.1 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. m.p. > 200 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.66 (d, *J* = 8.4 Hz, 1H), 8.55 – 8.47 (m, 1H), 8.27 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.96 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.88 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1H), 7.84 – 7.69 (m, 4H), 7.58 (tt, *J* = 5.7, 4.7 Hz, 2H), 5.62 (s, 2H). ^{13}C NMR (75 MHz, SC) δ 168.9, 152.6, 134.1, 133.2, 132.8, 130.8, 130.6, 128.6, 127.7, 127.0, 124.7, 124.3, 124.1, 123.6, 122.9, 122.0, 41.5. HRMS (DART) calc. $C_{22}H_{15}N_2O_2$ $[M + H]^+$ 339.11335, found 339.11313. IR (KBr, cm^{-1}): 3072, 2922, 2852, 1765, 1713, 1425, 1399, 1114, 749, 717.

4-(1,3-dioxoisoindolin-2-yl)-4-(phenanthridin-6-yl)butanenitrile (4k):



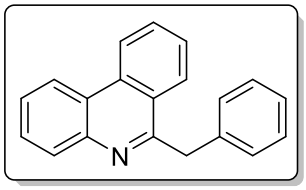
Following the **general procedure H**, **4k** was obtained as a brown solid (35% yield, 27.2 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. m.p. 195 – 198 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.65 (d, *J* = 8.4 Hz, 1H), 8.55 (d, *J* = 7.8 Hz, 1H), 8.26 – 8.02 (m, 2H), 7.86 – 7.52 (m, 8H), 6.33 (dd, *J* = 8.0, 6.5 Hz, 1H), 3.18 (m, 1H), 3.00 (m, 1H), 2.77 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 154.1, 143.0, 134.4, 133.5, 131.8, 130.8, 130.7, 128.9, 128.0, 127.7, 124.4, 124.2, 124.1, 123.7, 123.1, 122.1, 119.4, 52.1, 26.9, 15.2. HRMS (DART) calc. $C_{25}H_{18}N_3O_2$ [M + H]$^+$ 392.13990, found 392.13999. IR (KBr, cm^{-1}): 2960, 2939, 2244, 1775, 1711, 1611, 1580, 1373, 1126, 882, 759, 726.

2-(4-(4-methoxyphenyl)-4-oxo-1-(phenanthridin-6-yl)butyl)isoindoline-1,3-dione (4l):



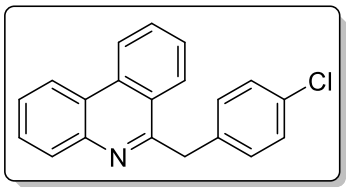
Following the **general procedure H**, **4l** was obtained as a brown solid (37% yield, 36.7 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (8:2) as eluent. m.p. 193 – 195 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (dd, *J* = 8.3, 1.2 Hz, 1H), 8.55 (m, 2H), 8.17 (d, *J* = 7.8 Hz, 1H), 8.02 (d, *J* = 8.9 Hz, 2H), 7.93 – 7.81 (m, 3H), 7.81 – 7.63 (m, 5H), 6.93 (d, *J* = 8.9 Hz, 2H), 6.50 (dd, *J* = 9.2, 5.5 Hz, 1H), 3.88 (s, 3H), 3.50 – 3.33 (m, 1H), 3.32 – 3.07 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 168.9, 163.6, 156.4, 134.1, 133.6, 132.2, 130.8, 130.6, 130.4, 130.1, 128.8, 128.0, 127.4, 125.6, 124.3, 124.2, 123.8, 123.5, 122.8, 122.0, 113.8, 55.6, 54.0, 35.7, 25.7. HRMS (DART) calc. $C_{32}H_{25}N_2O_4$ [M + H]$^+$ 501.18143, found 501.18221. IR (KBr, cm^{-1}): 2925, 2853, 1775, 1713, 1674, 1599, 1381, 1259, 1170, 1085, 762, 721.

6-benzylphenanthridine (4m):



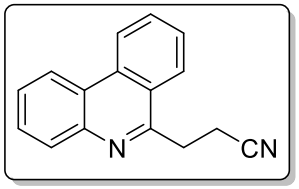
Following the **general procedure H**, **4m** was obtained as a yellow solid (33% yield, 17.8 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. m.p. 84 – 86 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.60 – 8.55 (m, 1H), 8.50 (dd, *J* = 8.1, 1.5 Hz, 1H), 8.19 – 8.10 (m, 2H), 7.79 – 7.63 (m, 2H), 7.60 (ddd, *J* = 8.3, 7.0, 1.5 Hz, 1H), 7.52 (ddd, *J* = 8.3, 7.0, 1.2 Hz, 1H), 7.30 – 7.21 (m, 2H), 7.22 – 7.07 (m, 3H), 4.70 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 139.2, 133.5, 130.6, 129.9, 128.9, 128.7, 127.5, 127.3, 126.9, 126.5, 125.5, 124.1, 122.6, 122.1, 43.1. HRMS (DART) calc. for $C_{20}H_{16}N$ $[M + H]^+$ 270.12827, found 270.12883. IR (KBr, cm^{-1}): 3062, 3028, 2924, 2853, 1694, 1607, 1580, 1521, 1489, 1450, 1361, 757, 727, 701.

6-(4-chlorobenzyl)phenanthridine (4n):



Following the **general procedure H**, **4n** was obtained as a yellow solid (42% yield, 25.3 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. m.p. 120 – 122 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 8.3 Hz, 1H), 8.56 (dd, *J* = 8.2, 1.4 Hz, 1H), 8.21 (d, *J* = 8.2 Hz, 1H), 8.14 (dd, *J* = 8.3, 1.3 Hz, 1H), 7.80 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1H), 7.75 (ddd, *J* = 8.3, 7.0, 1.4 Hz, 1H), 7.67 (ddd, *J* = 8.3, 7.0, 1.4 Hz, 1H), 7.60 (ddd, *J* = 8.2, 7.0, 1.2 Hz, 1H), 7.25 – 7.15 (m, 4H), 4.73 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 137.6, 133.5, 132.3, 130.8, 130.1, 129.8, 129.0, 128.8, 127.6, 127.1, 127.0, 125.3, 124.1, 122.7, 122.1, 42.3. HRMS (DART) calc. for $C_{20}H_{15}ClN$ $[M + H]^+$ 304.08930, found 304.08901. IR (KBr, cm^{-1}): 2961, 2923, 2852, 1575, 1487, 1088, 1015, 848, 807, 761, 728.

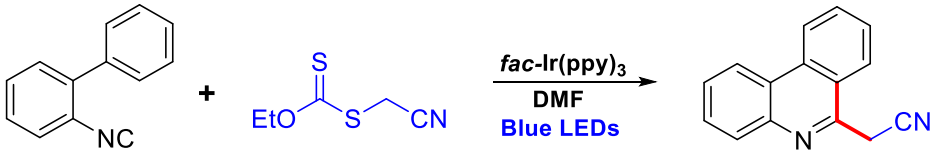
3-(phenanthridin-6-yl)propanenitrile (4o):



Following the **general procedure H**, **4o** was obtained as a brown solid (24% yield, 11.1 mg) after purification *via* flash column chromatography using silica gel and a mixture of Hex:AcOEt (95:5) as eluent. m.p. 115 – 117°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 8.3 Hz, 1H), 8.56 (dd, *J* = 8.2, 1.5 Hz, 1H), 8.24 – 8.08 (m, 2H), 7.88 (ddd, *J* = 8.3, 7.1, 1.3 Hz, 1H), 7.81 – 7.70 (m, 2H), 7.67 (ddd, *J* = 8.2, 7.1, 1.3 Hz, 1H), 3.75 (t, *J* = 7.5 Hz, 2H), 3.20 (t, *J* = 7.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 133.0, 131.0, 129.9, 129.0, 127.2, 125.2, 125.0, 123.9, 122.8, 122.1, 120.4, 30.4, 14.7. HRMS (DART) calc. for $C_{16}H_{13}N_2$ $[M + H]^+$ 233.10787, found 233.10676. IR (KBr, cm^{-1}): 2924, 2853, 2241, 1735, 1613, 1584, 1306, 1241, 1034, 1008, 773, 745, 722.

4 Optimization of stoichiometry

Table 1. Optimization of stoichiometry



1a **2a** **3a**

Entry	Equiv. 1a	Equiv. 2a	% R 3a
1[a]	1.0	1.0	31 %
2	2.0	1.0	57 %
3	1.0	2.0	27 %

[a] **Reaction condition**: **1** (0.2 mmol, 1.0 equiv), **2** (0.2 mmol, 1.0 equiv.), ***fac*-Ir(ppy)$_3$** (0.004 mmol, 0.02 equiv.), **DMF** (0.8 mL) under blue LEDs irradiation at 30 °C.

5 Fluorescent quenching experiments

To a solution 0.0398 mM of *fac*-Ir(ppy)$_3$ in deoxigenated DMF, xanthate **2a** was added to obtain a solution 0.039, 0.059, 0.079, 0.156, 0.195, 0.252, 0.308 and 0.383 mM as a final concentration of **2a**. The corresponding spectra were acquired irradiating at 385 nm with an emission maximum wavelength at 517 nm.

The fluorescence intensity ratio (I_0/I) at 517 nm was plotted against **2a** concentration and a linear variation was observed that is in accordance with Stern-Volmer equation indicating a SET or an energy transfer process. The quenching rate constant obtained to this process was $k_q = 1.0 \times 10^5\ M^{-1}s^{-1}$.

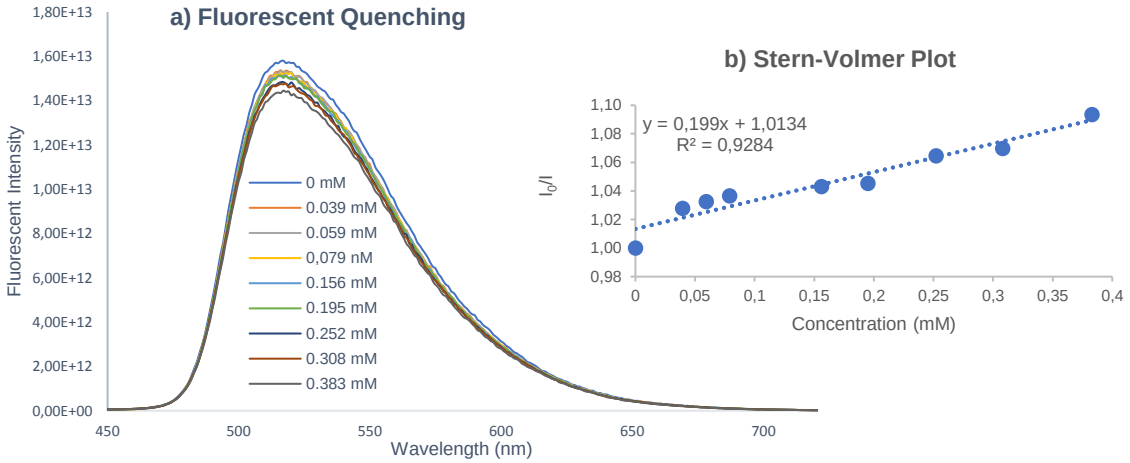
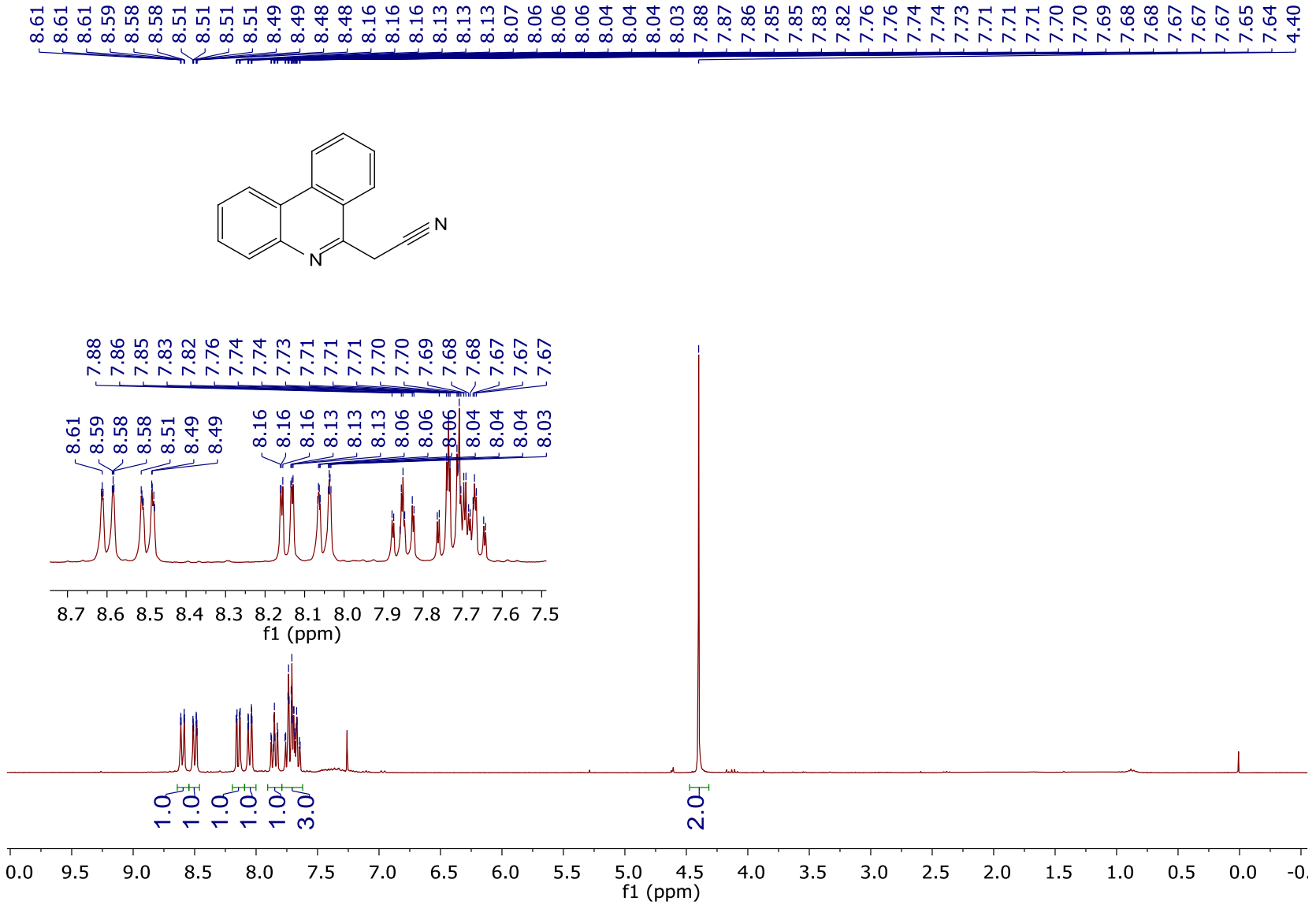
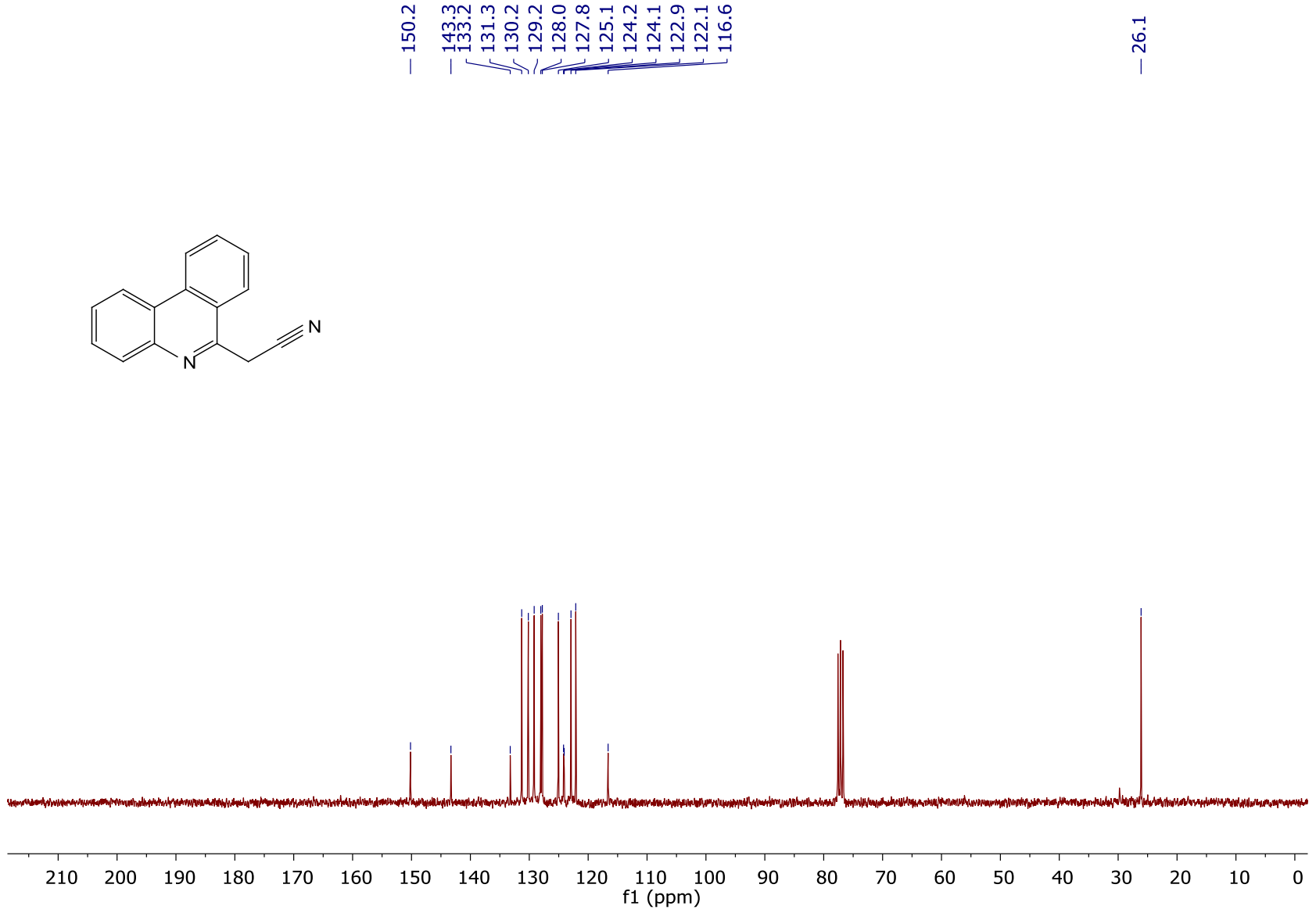


Fig. 1 a) Fluorescence quenching experiment. b) Stern-Volmer.

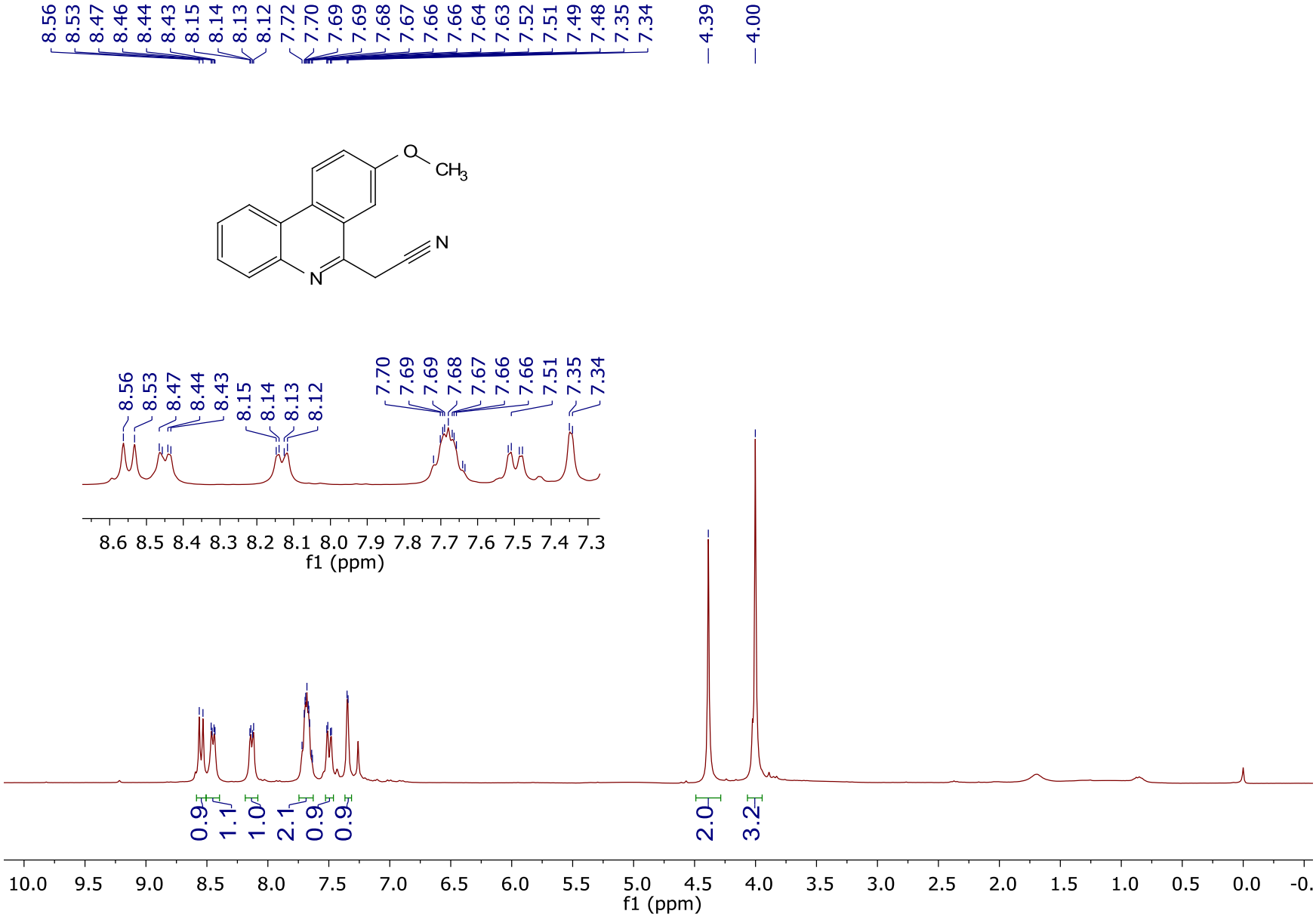
6 ^{1}H and ^{13}C NMR spectra of products

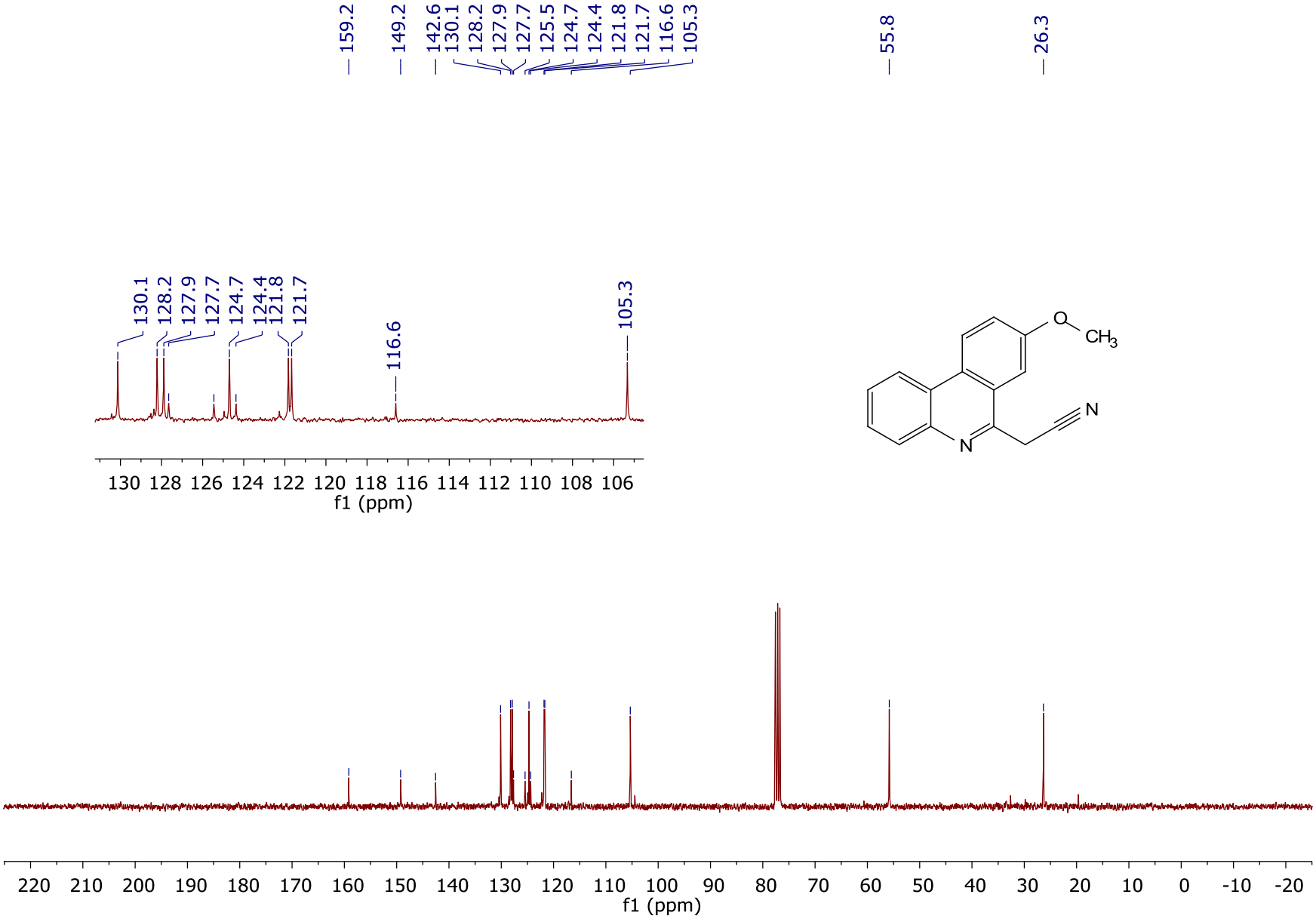
(phenanthridin-6-yl)acetonitrile (3a):



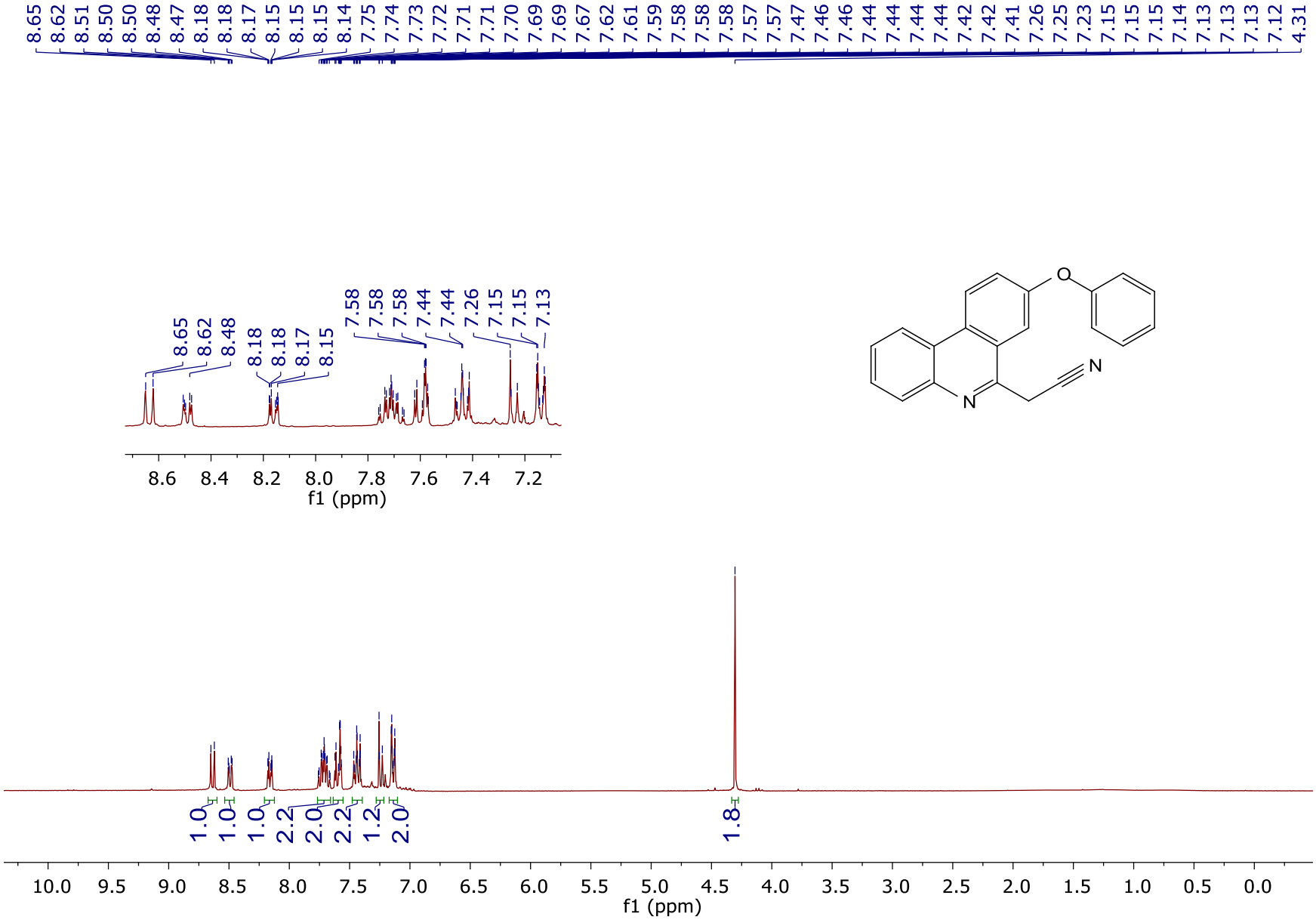

150.2
143.3
133.2
131.3
130.2
129.2
128.0
127.8
125.1
124.2
124.1
122.9
122.1
116.6
26.1
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

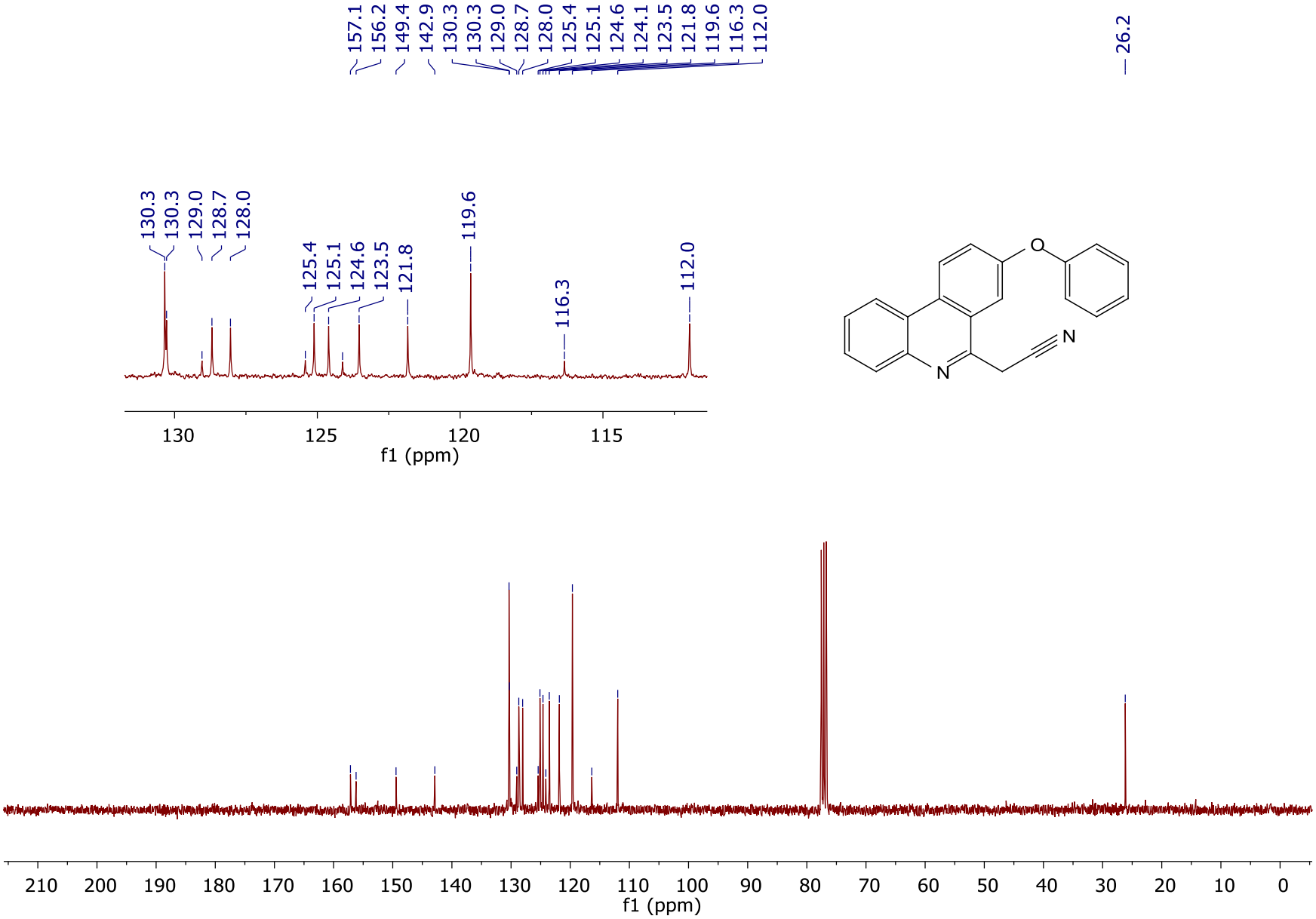
2-(8-methoxyphenanthridin-6-yl)acetonitrile (3b):



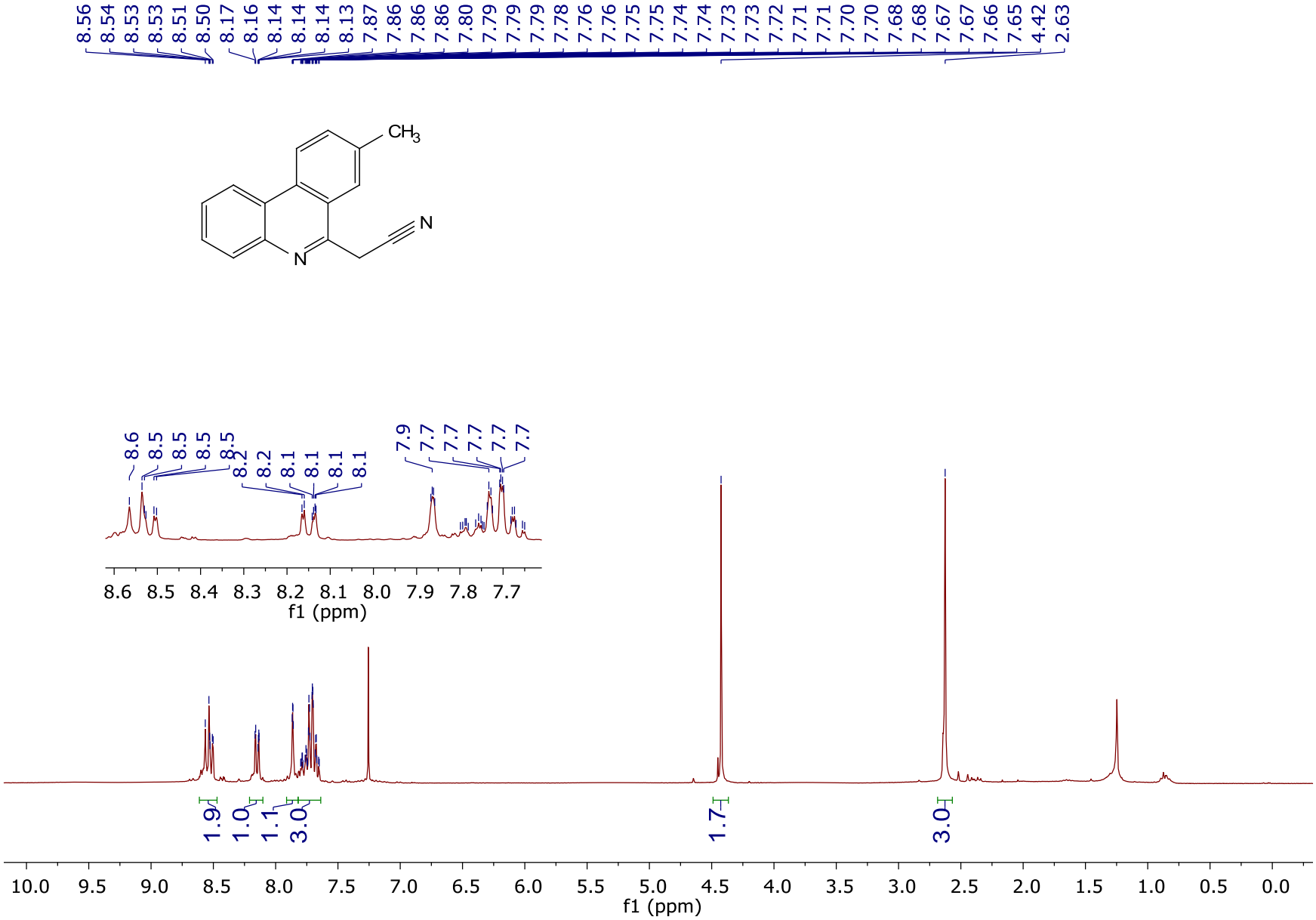

159.2
149.2
142.6
130.1
128.2
127.9
127.7
125.5
124.7
124.4
121.8
121.7
116.6
105.3
55.8
26.3
O
CH3
N
N
130 128 126 124 122 120 118 116 114 112 110 108 106
f1 (ppm)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

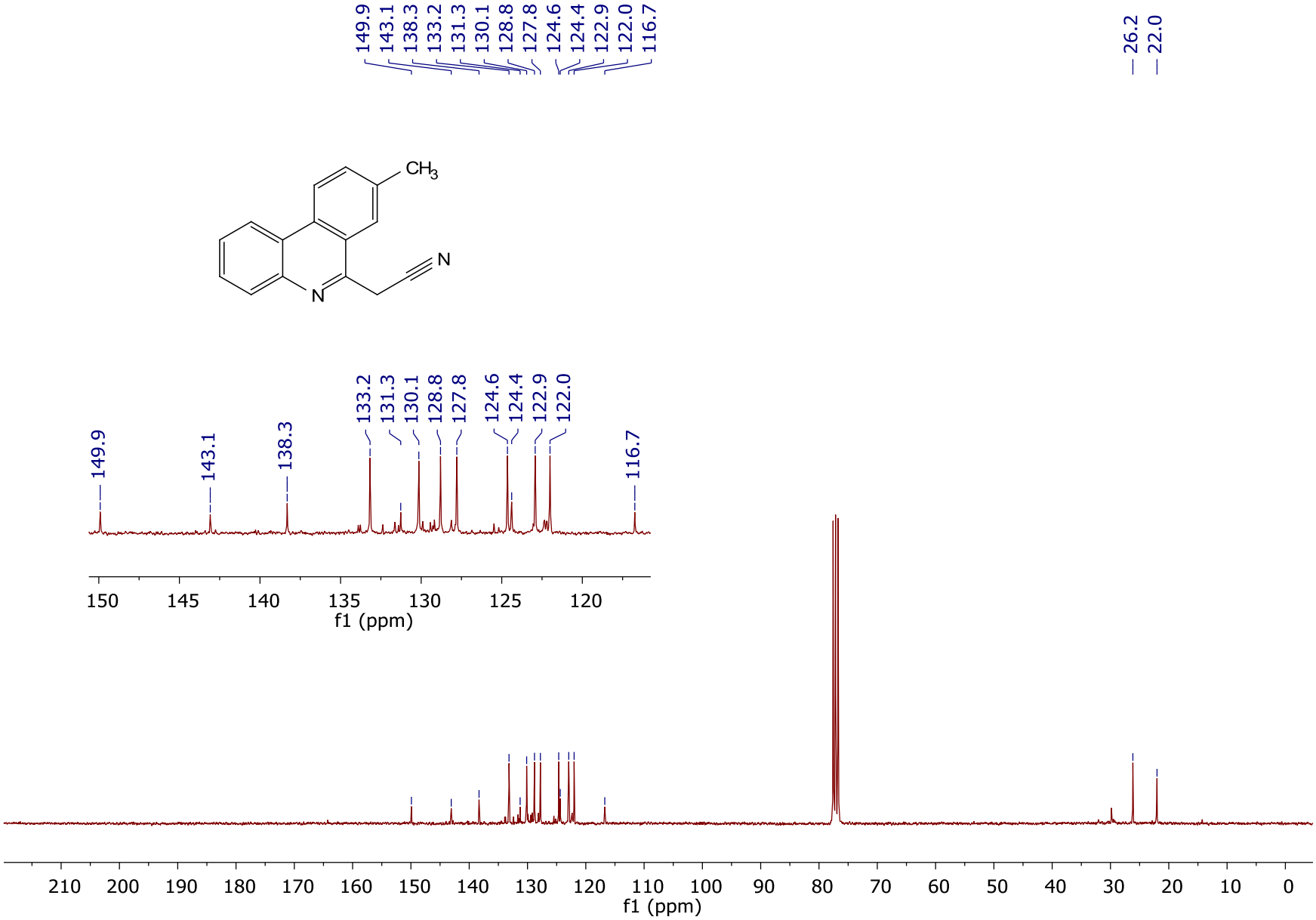
(8-phenoxyphenanthridin-6-yl)acetonitrile (3c):



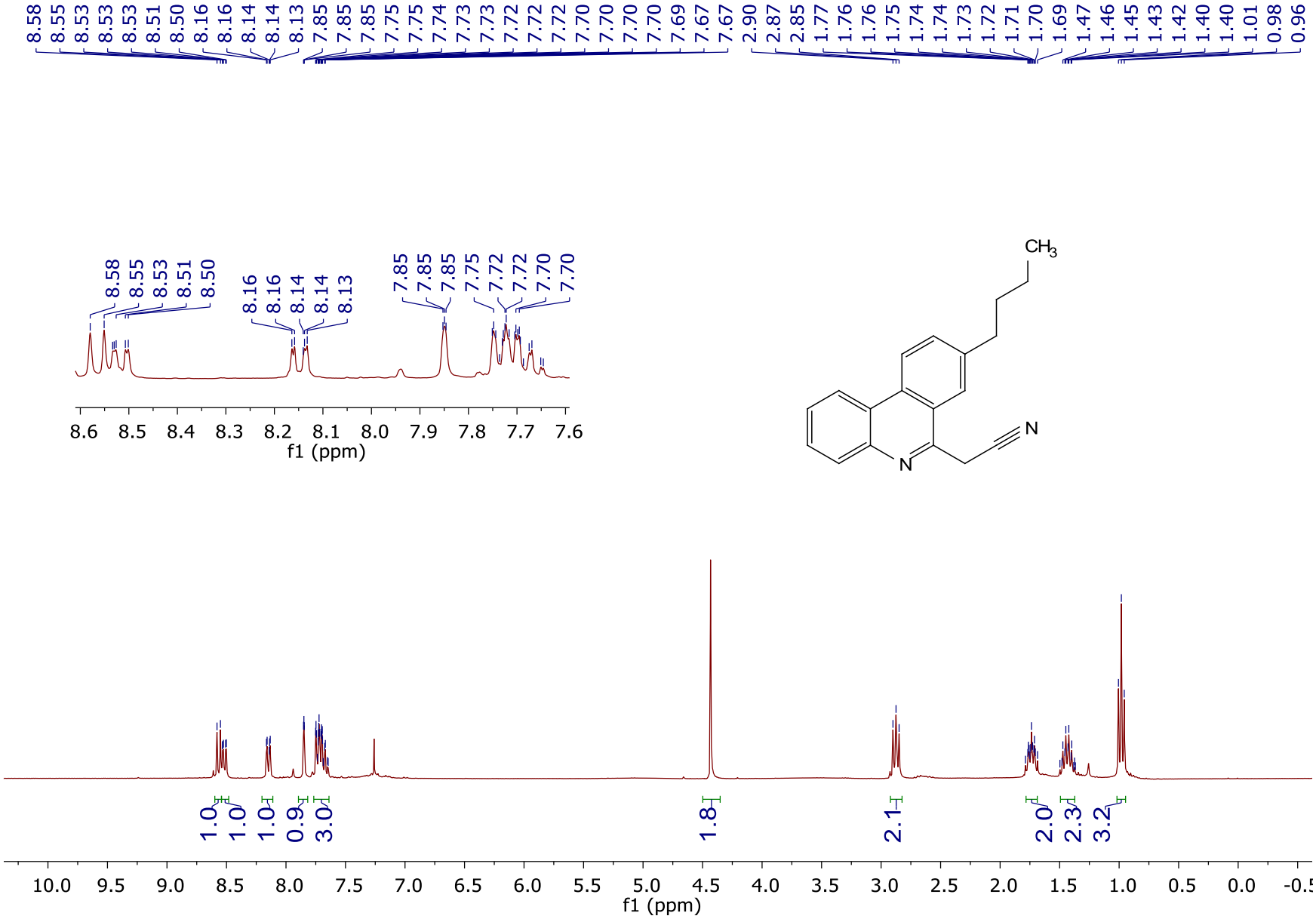

157.1
156.2
149.4
142.9
130.3
130.3
129.0
128.7
128.0
125.4
125.1
124.6
124.1
123.5
121.8
119.6
116.3
112.0
26.2
130.3
130.3
129.0
128.7
128.0
125.4
125.1
124.6
123.5
121.8
119.6
116.3
112.0
130
125
120
115
f1 (ppm)
O
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

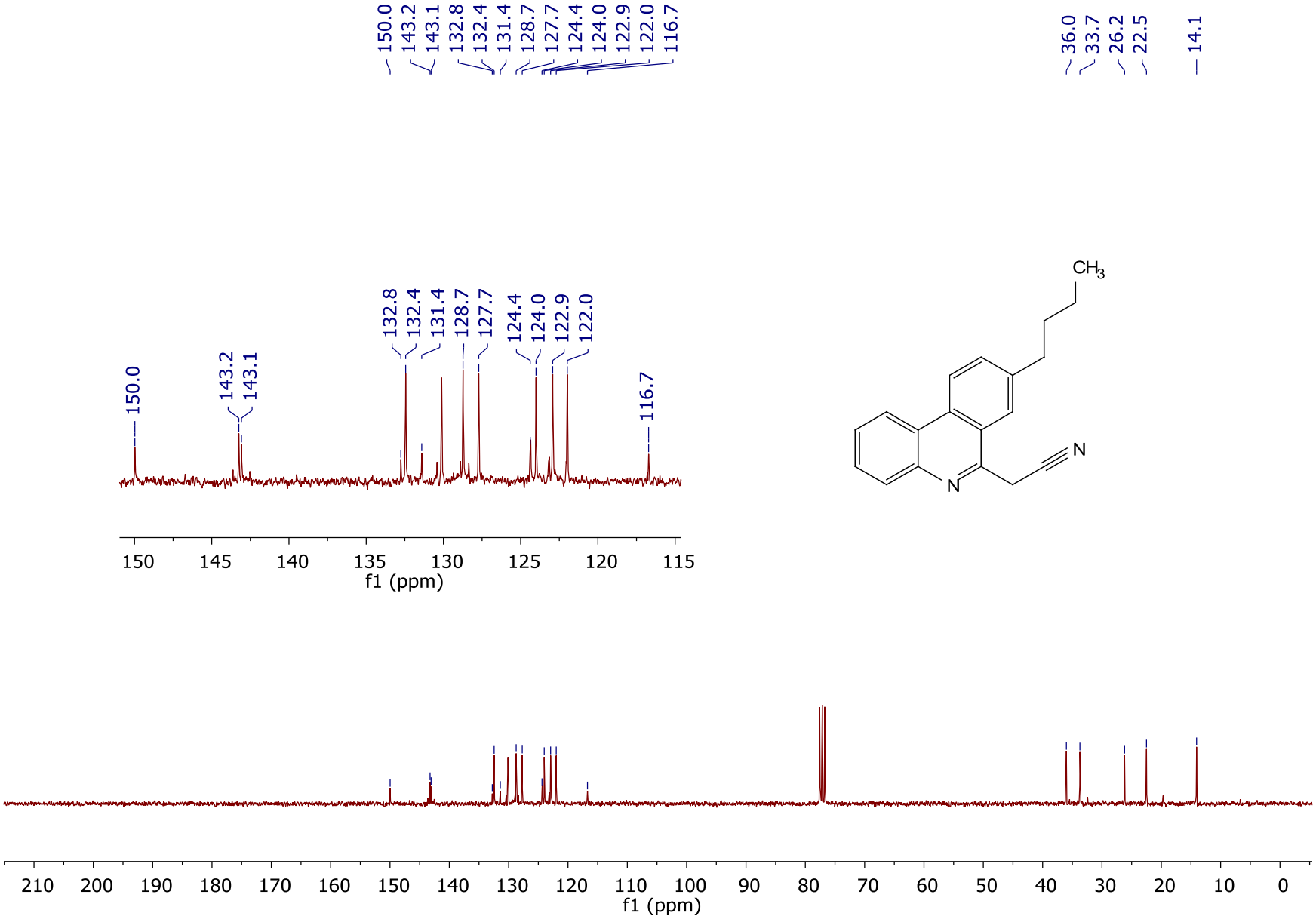
2-(8-methylphenanthridin-6-yl)acetonitrile (3d):



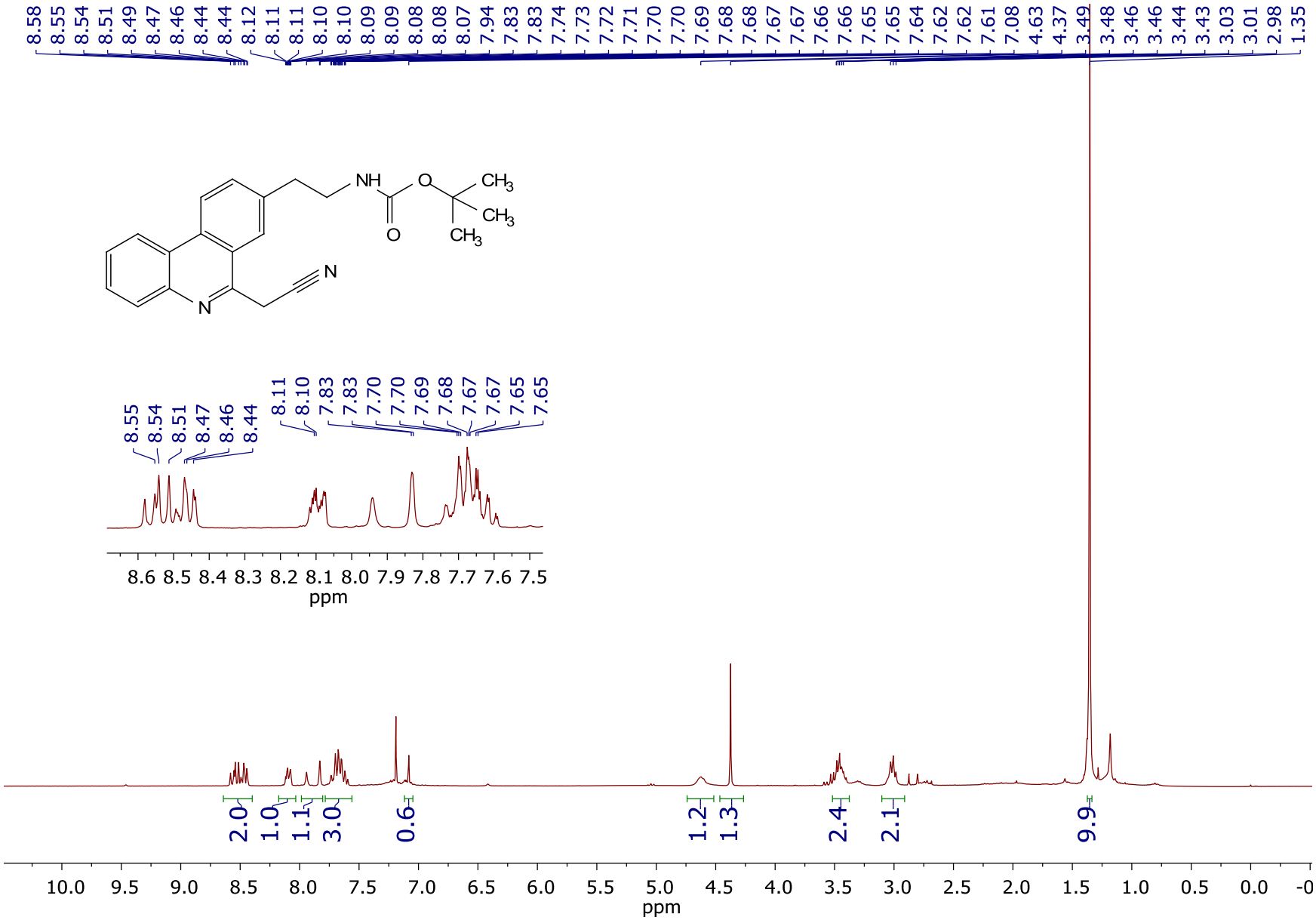

149.9
143.1
138.3
133.2
131.3
130.1
128.8
127.8
124.6
124.4
122.9
122.0
116.7
26.2
22.0
CH3
N
N
150
145
140
135
130
125
120
f1 (ppm)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

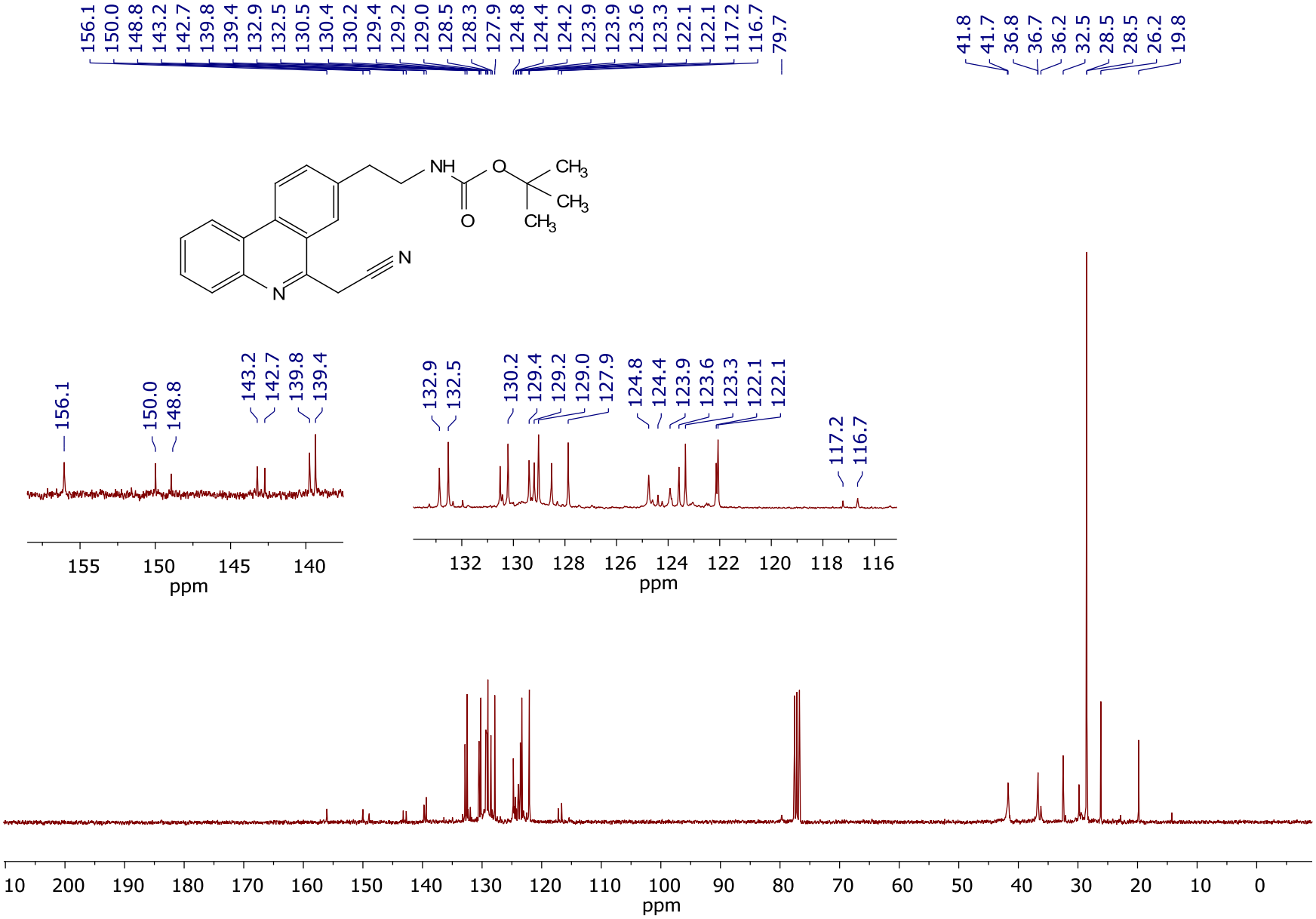
2-(8-butylphenanthridin-6-yl)acetonitrile (3e):



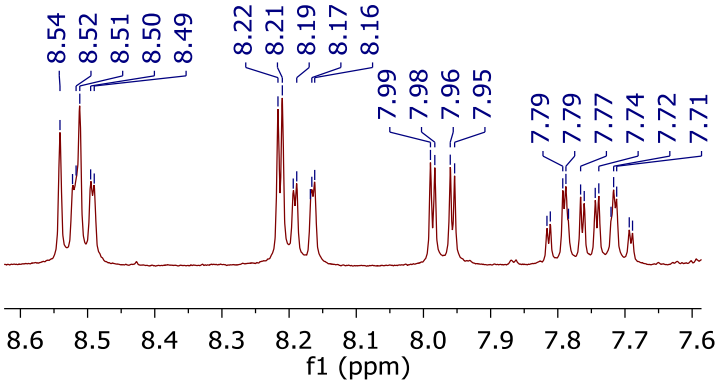

150.0
143.2
143.1
132.8
132.4
131.4
128.7
127.7
124.4
124.0
122.9
122.0
116.7
36.0
33.7
26.2
22.5
14.1
CH3
N
N
f1 (ppm)
f1 (ppm)

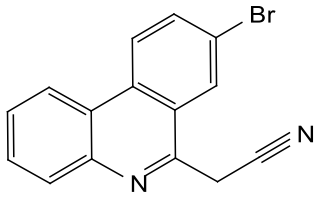
tert-butyl (2-(6-(cyanomethyl)phenanthridin-8-yl)ethyl)carbamate (3f):

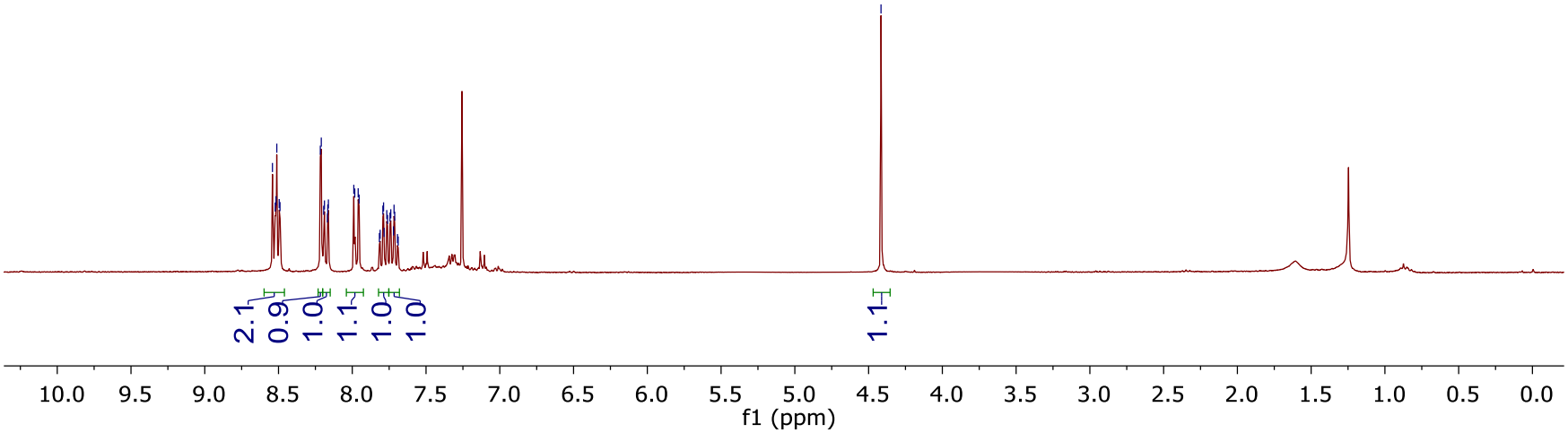


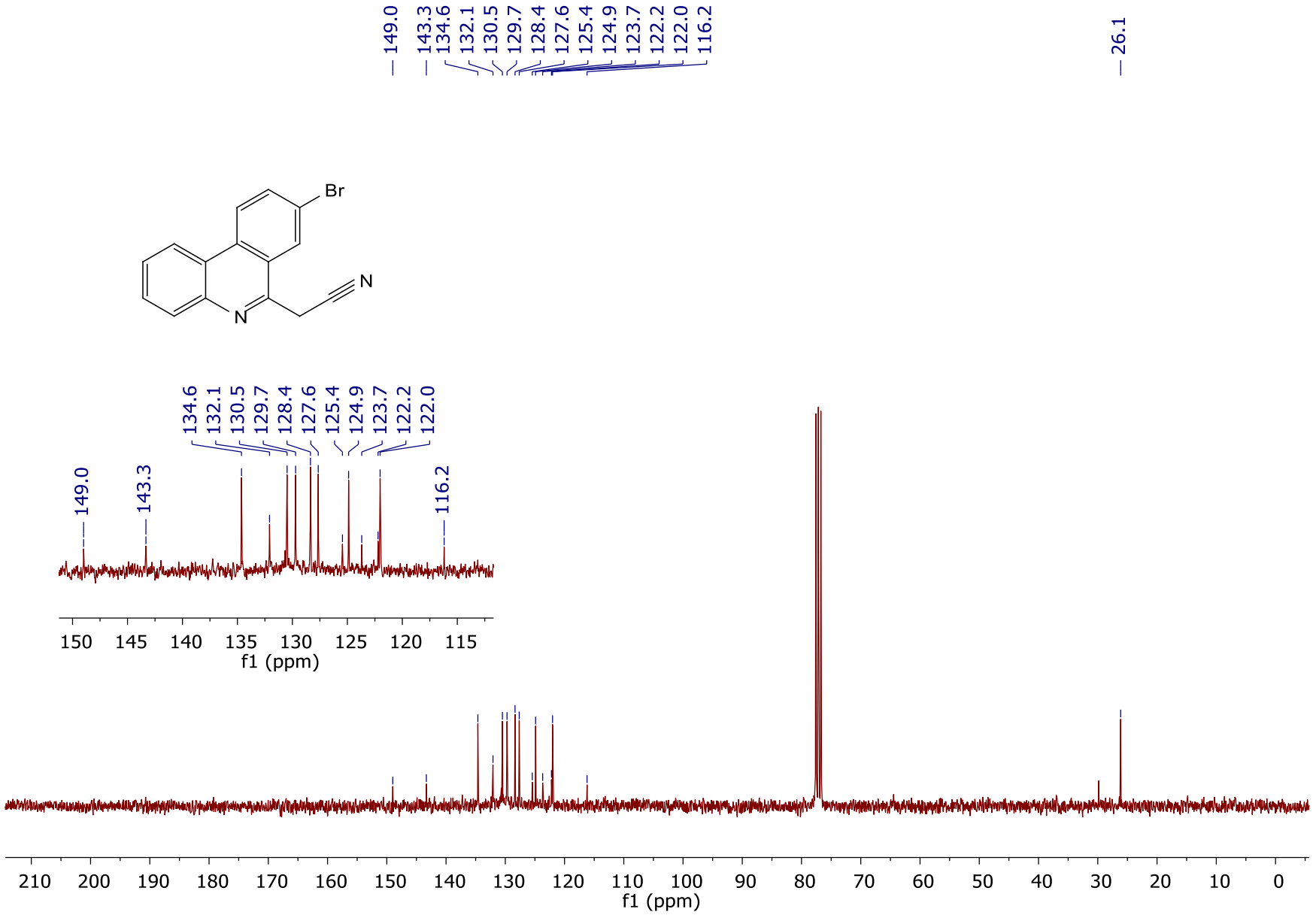

156.1
150.0
148.8
143.2
142.7
139.8
139.4
132.9
132.5
130.2
129.4
129.2
129.0
128.5
128.3
127.9
124.8
124.4
124.2
123.9
123.9
123.6
123.3
122.1
122.1
117.2
116.7
79.7
41.8
41.7
36.8
36.7
36.2
32.5
28.5
28.5
26.2
19.8
NH
O
O
CH3
CH3
CH3
N
N
ppm
ppm
ppm

2-(8-bromophenanthridin-6-yl)acetonitrile (3g):

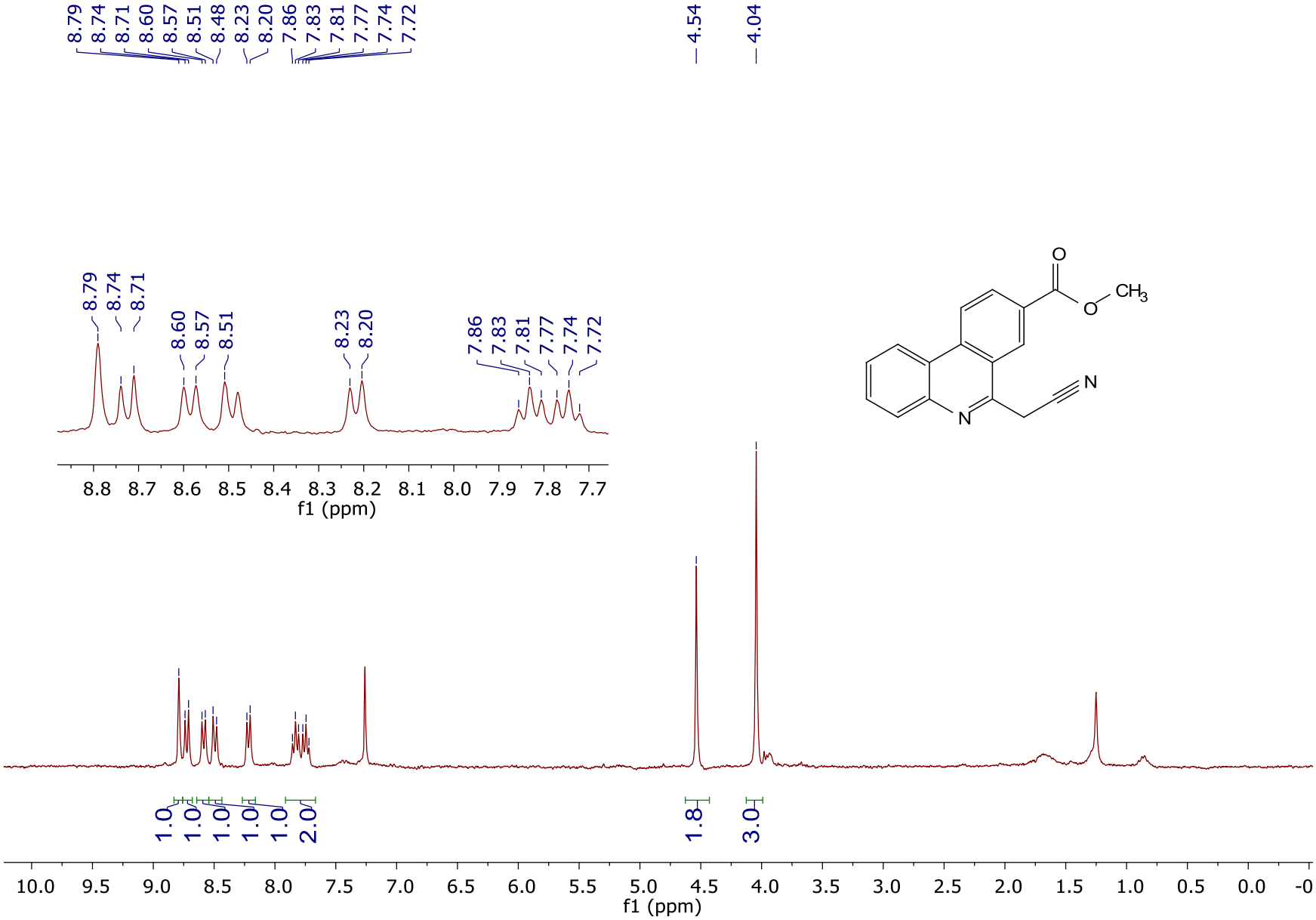


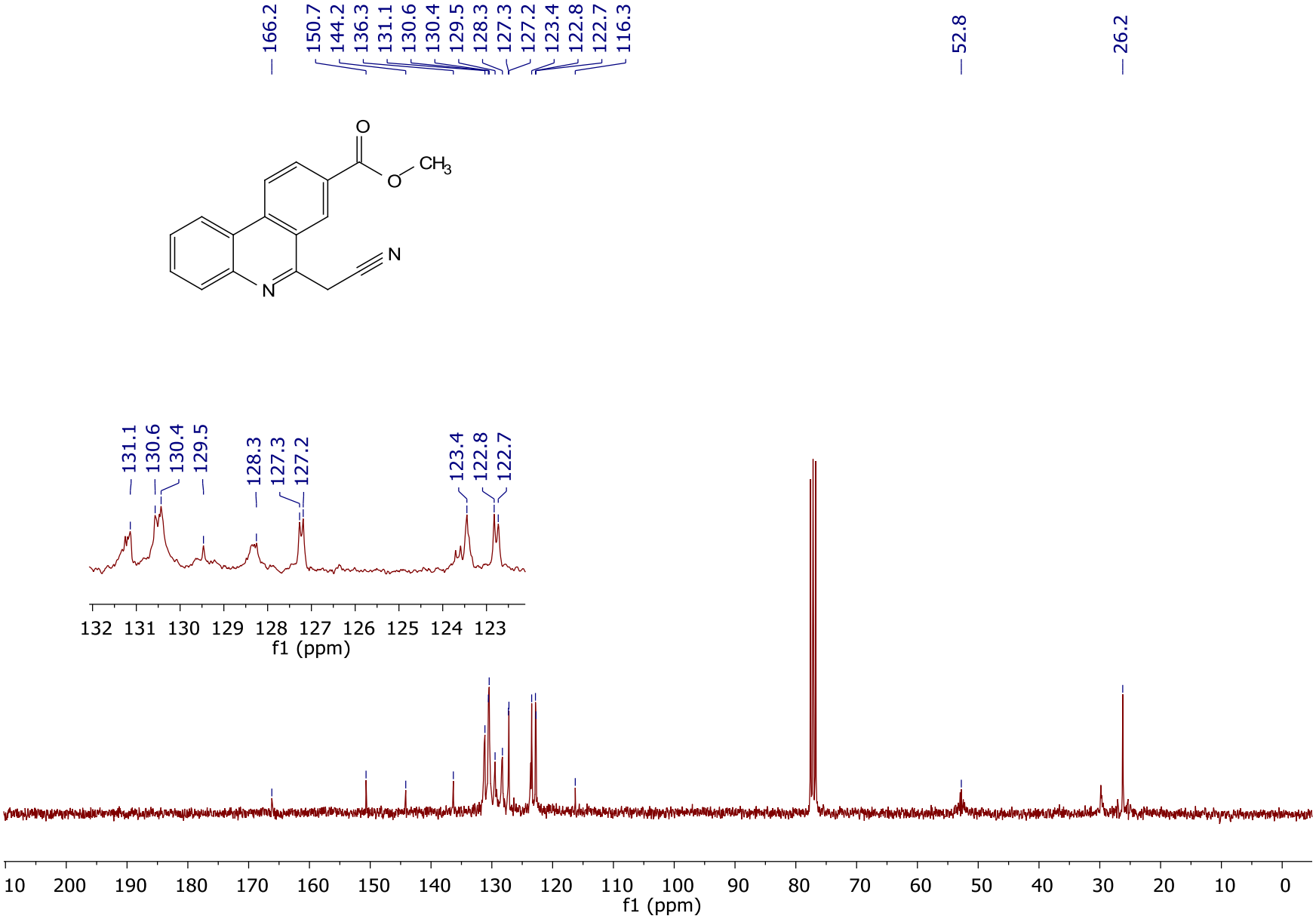




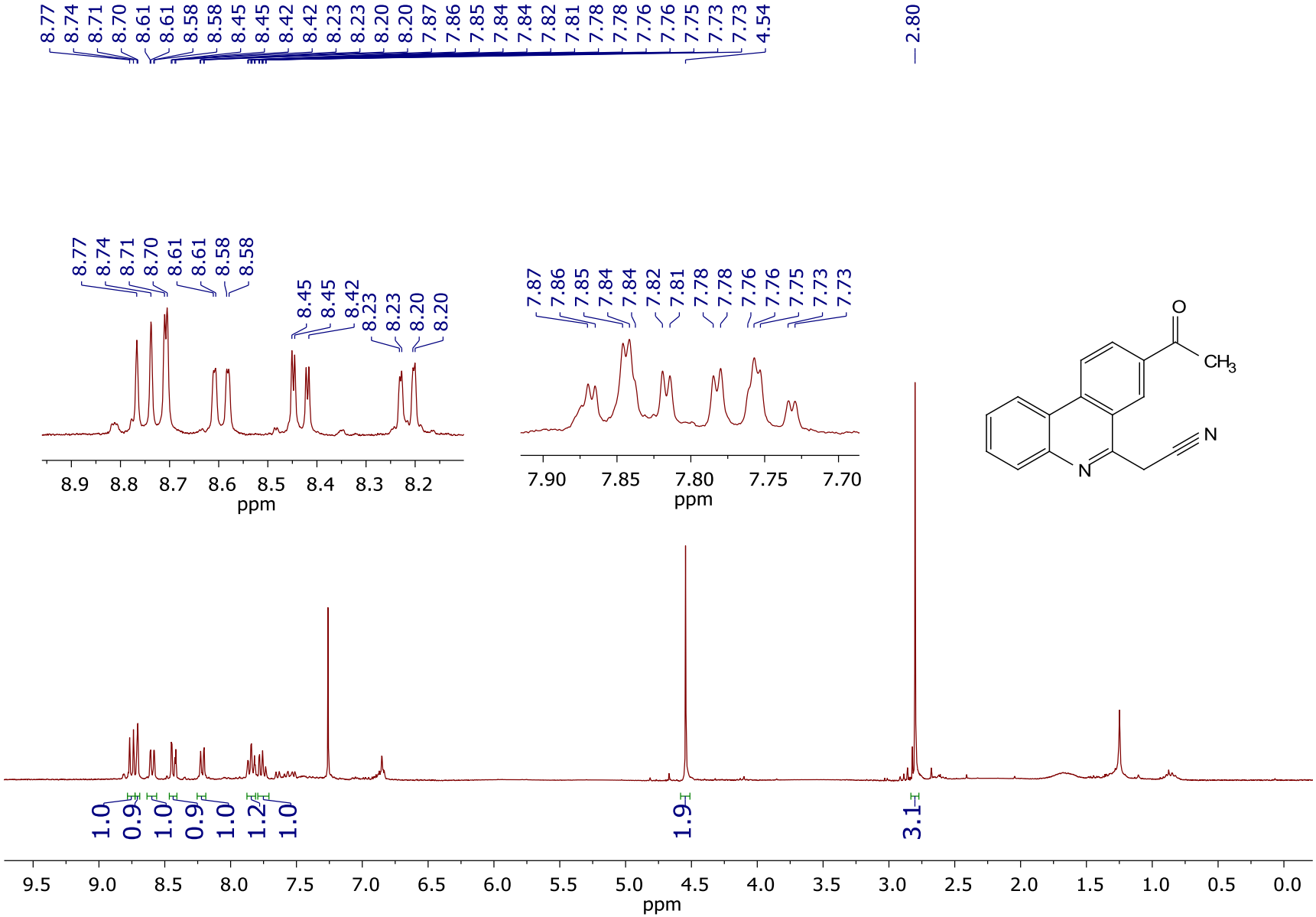

149.0
143.3
134.6
132.1
130.5
129.7
128.4
127.6
125.4
124.9
123.7
122.2
122.0
116.2
26.1
Br
N
N
f1 (ppm)
f1 (ppm)

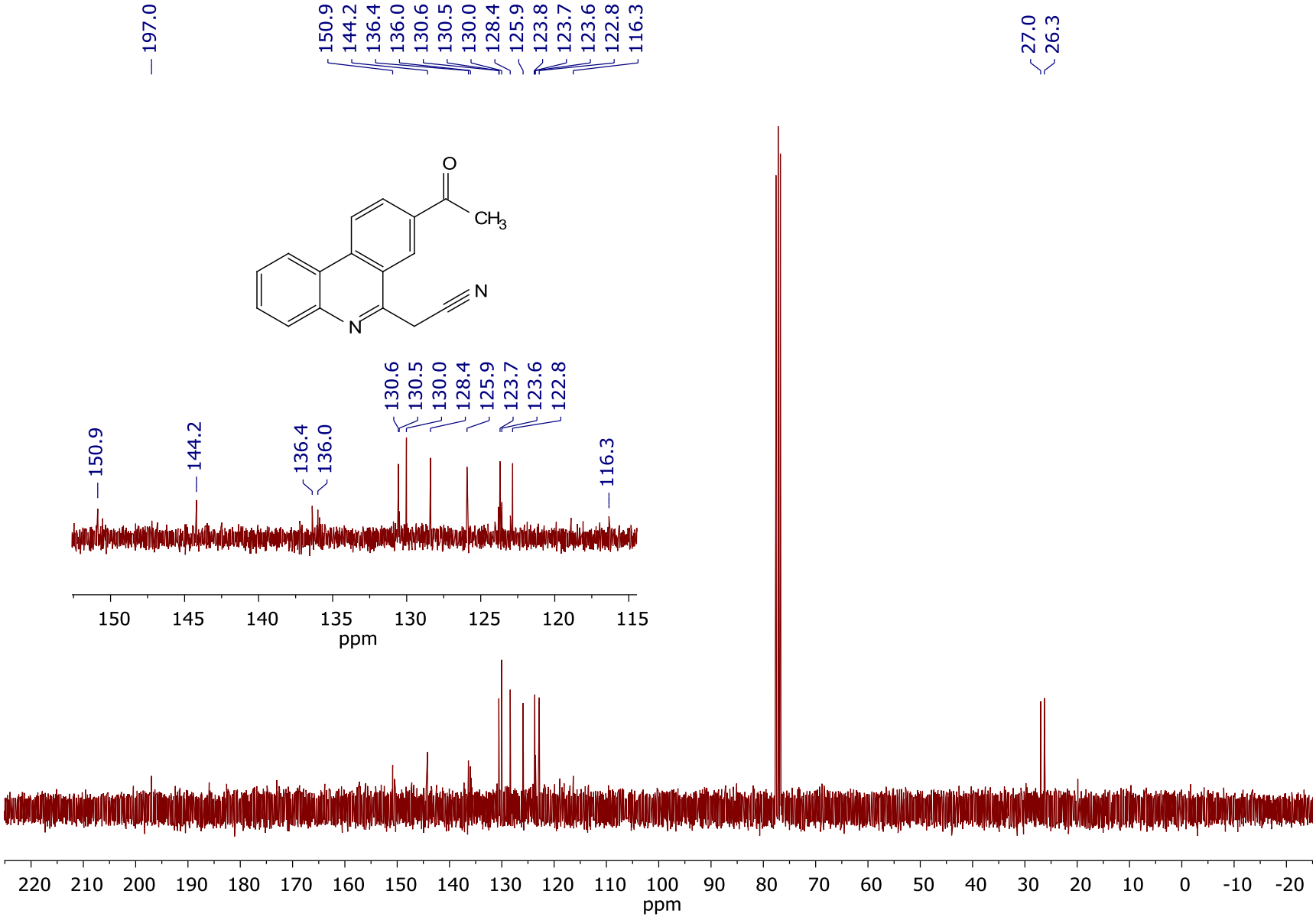
methyl 6-(cyanomethyl)phenanthridine-8-carboxylate (3h):



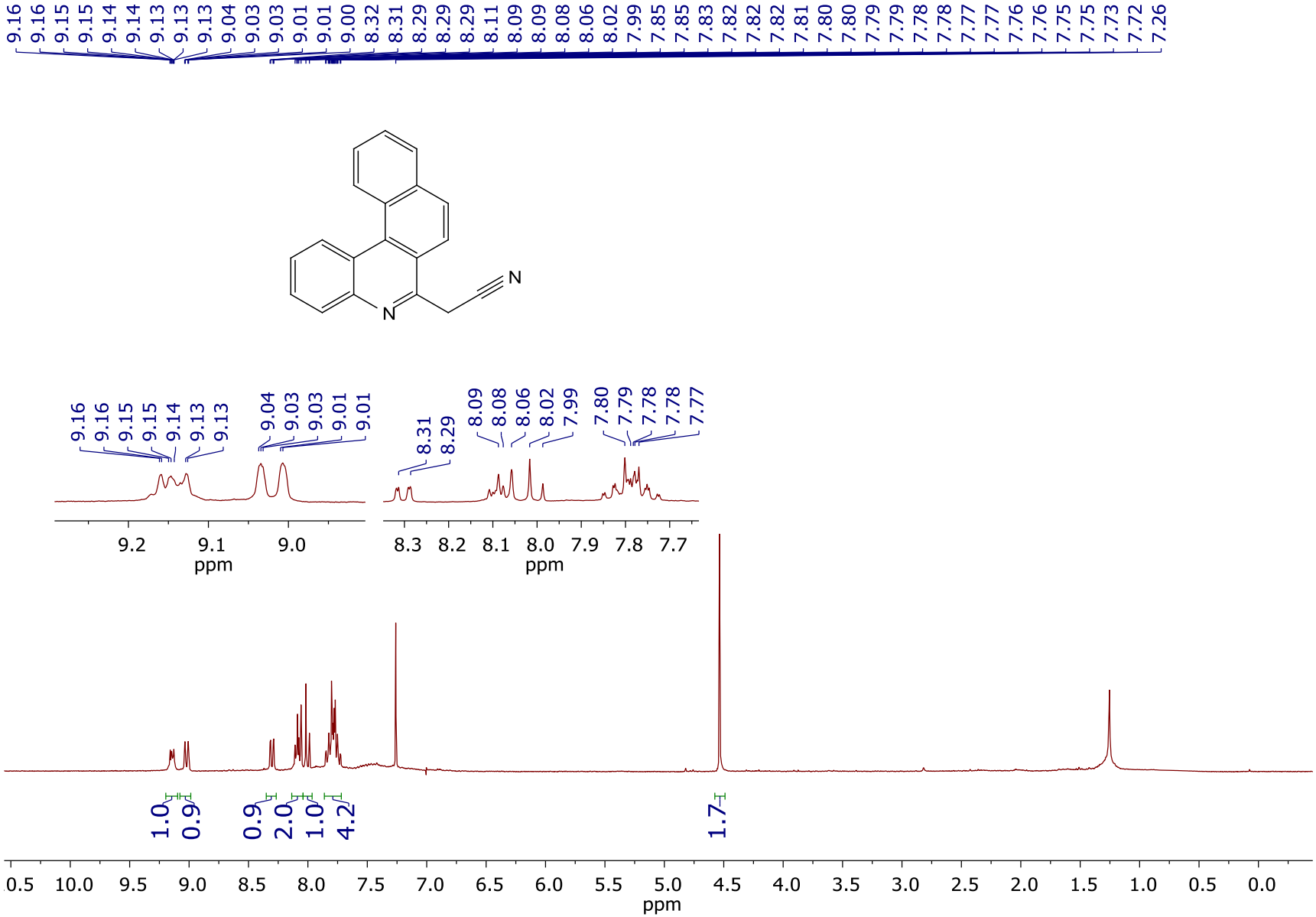

166.2
150.7
144.2
136.3
131.1
130.6
130.4
129.5
128.3
127.3
127.2
123.4
122.8
122.7
116.3
52.8
26.2
O
O
CH3
N
N
131.1
130.6
130.4
129.5
128.3
127.3
127.2
123.4
122.8
122.7
132 131 130 129 128 127 126 125 124 123
f1 (ppm)
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

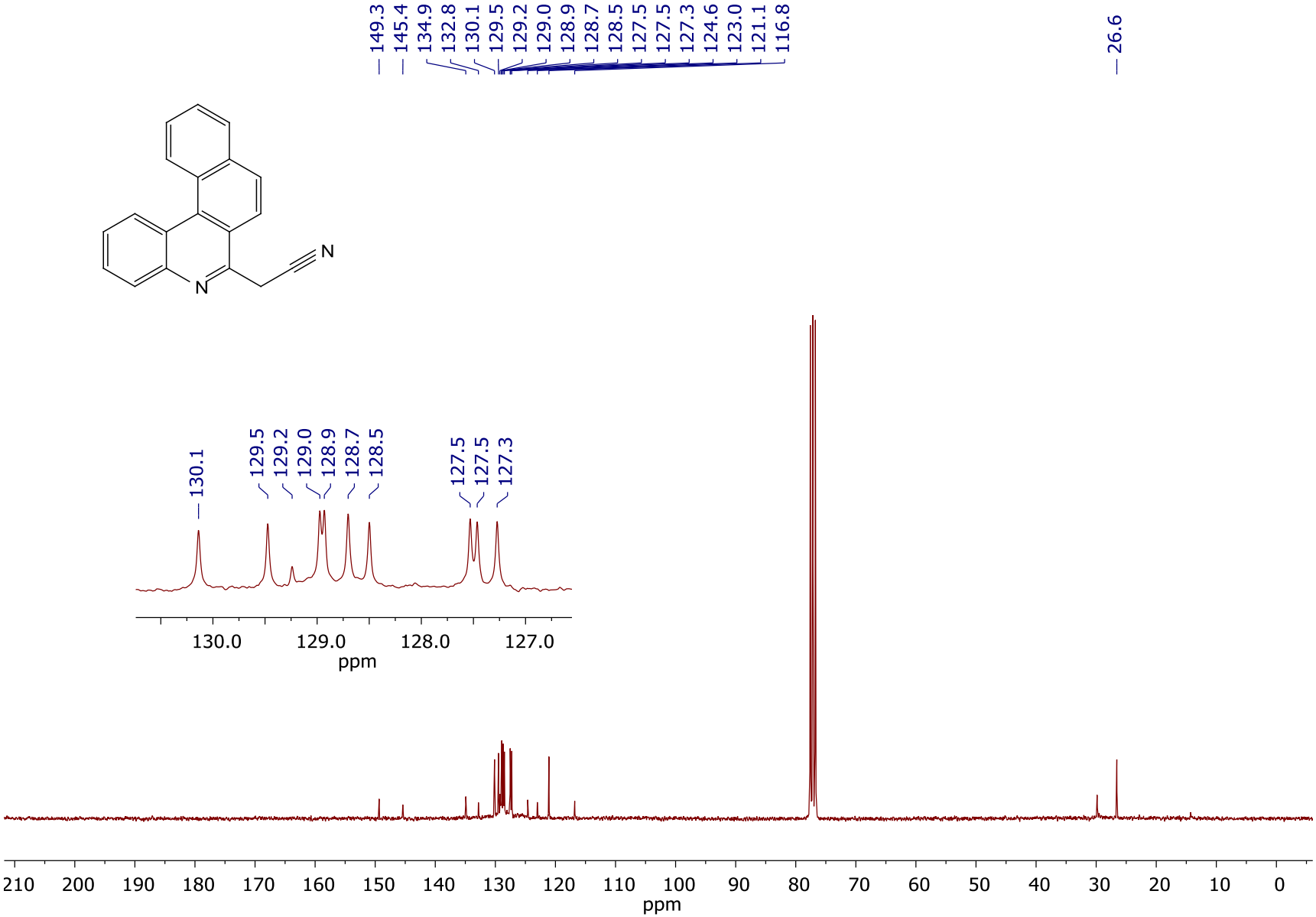
2-(8-acetylphenanthridin-6-yl)acetonitrile (3i):



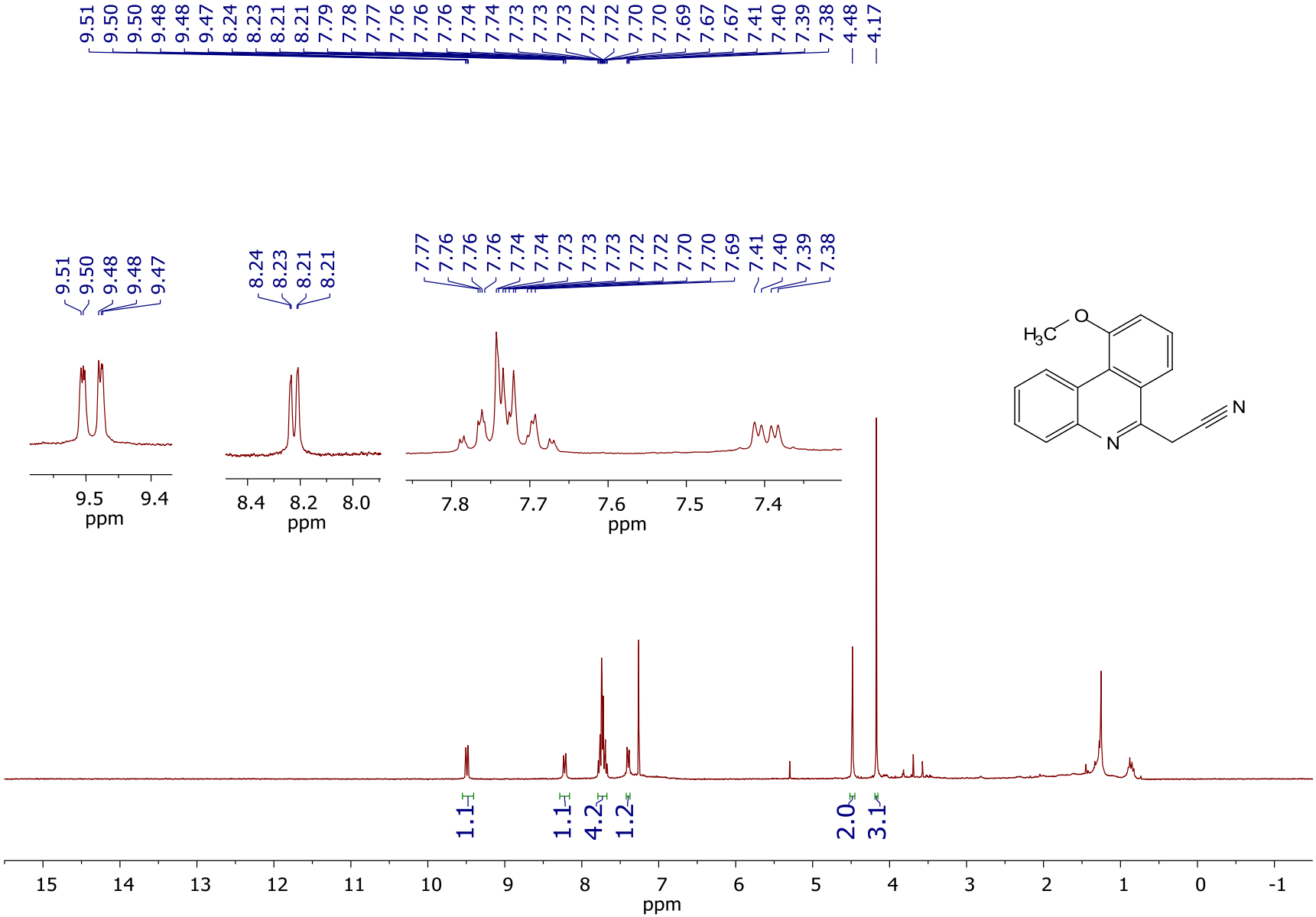

197.0
150.9
144.2
136.4
136.0
130.6
130.5
130.0
128.4
125.9
123.8
123.7
123.6
122.8
116.3
27.0
26.3
O
CH3
N
N
150.9
144.2
136.4
136.0
130.6
130.5
130.0
128.4
125.9
123.7
123.6
122.8
116.3
150 145 140 135 130 125 120 115
ppm
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
ppm

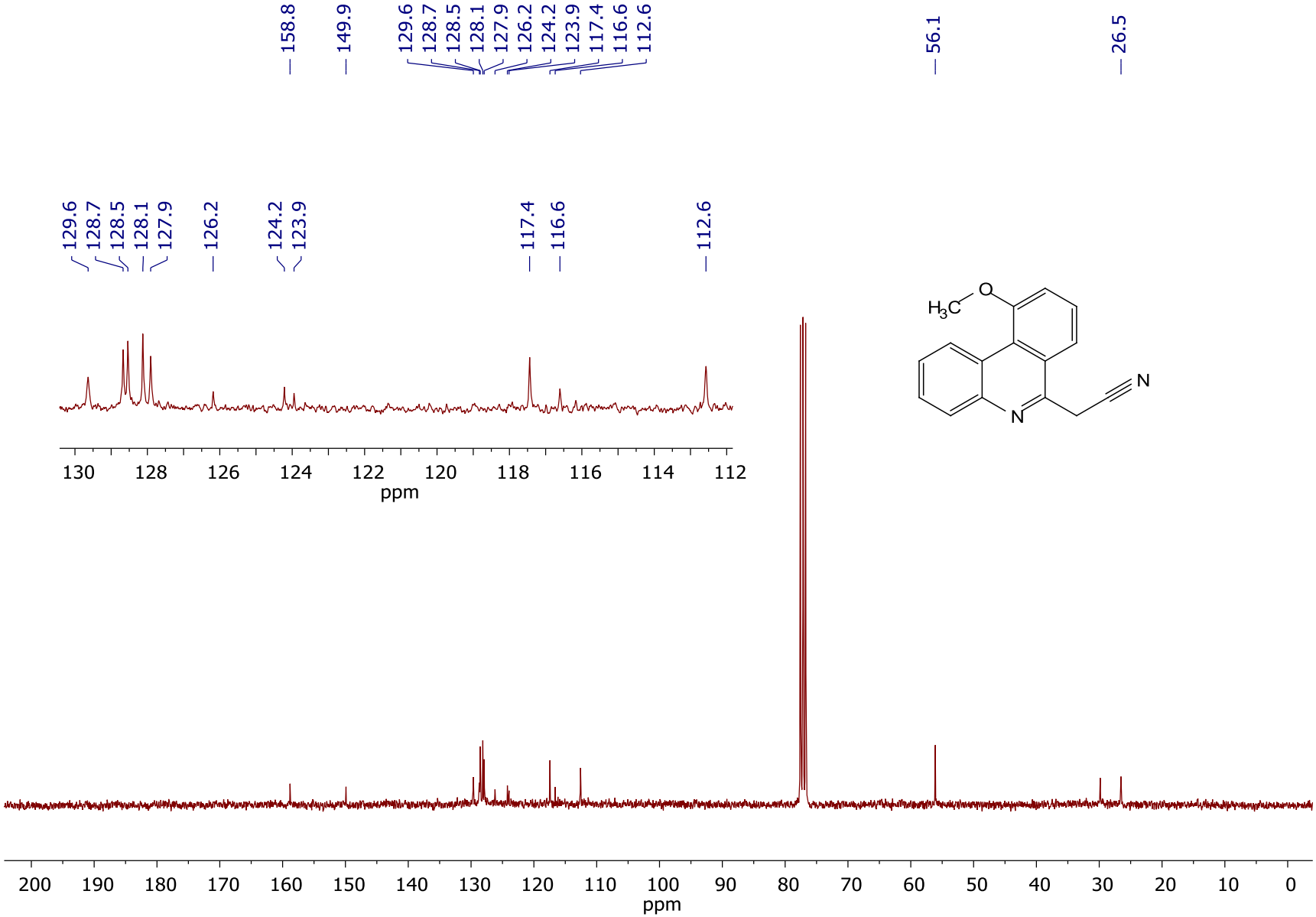
2-(benzo[k]phenanthridin-6-yl)acetonitrile (3j):



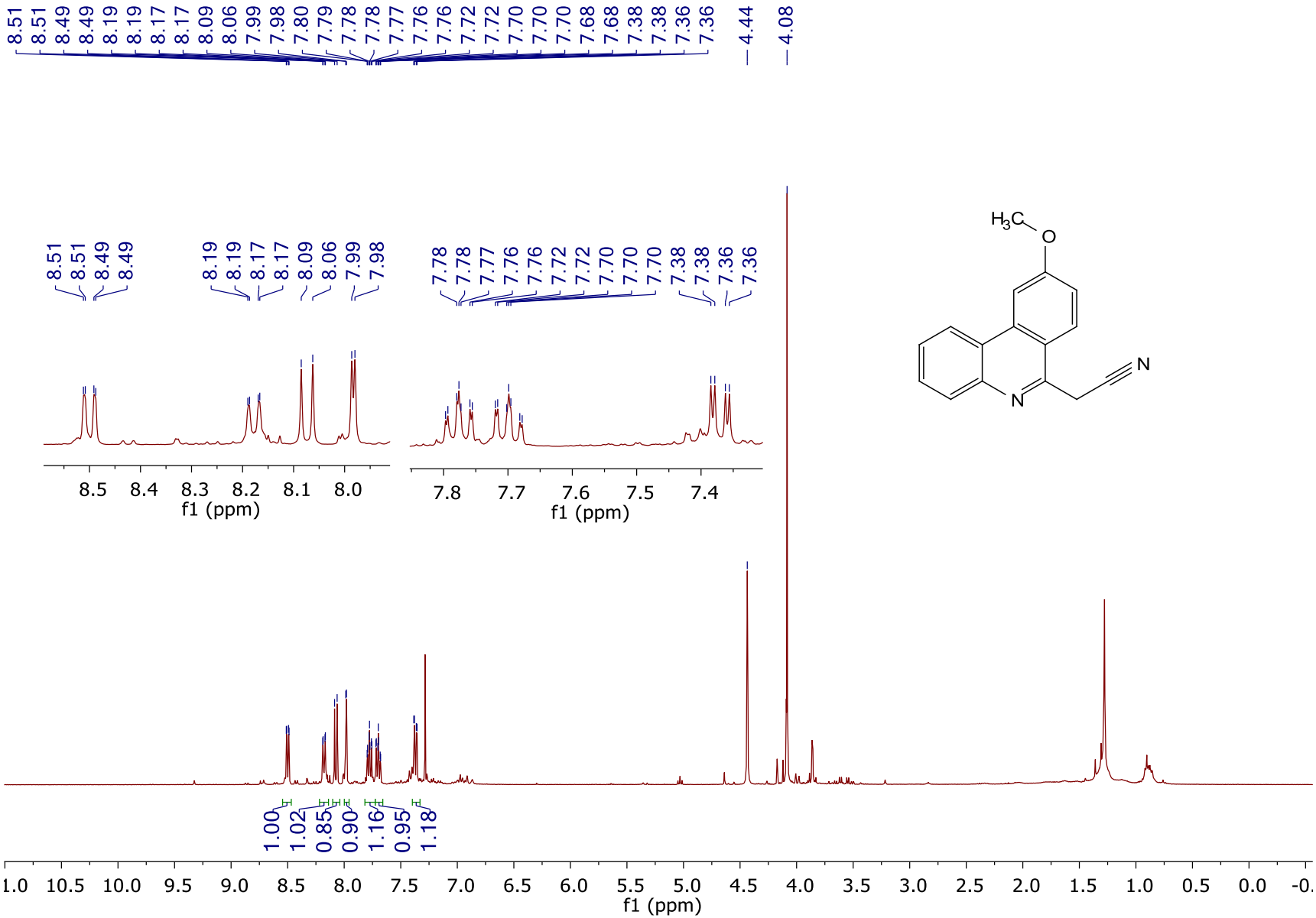

149.3
145.4
134.9
132.8
130.1
129.5
129.2
129.0
128.9
128.7
128.5
127.5
127.5
127.3
124.6
123.0
121.1
116.8
26.6
N
N
130.1
129.5
129.2
129.0
128.9
128.7
128.5
127.5
127.5
127.3
130.0
129.0
128.0
127.0
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

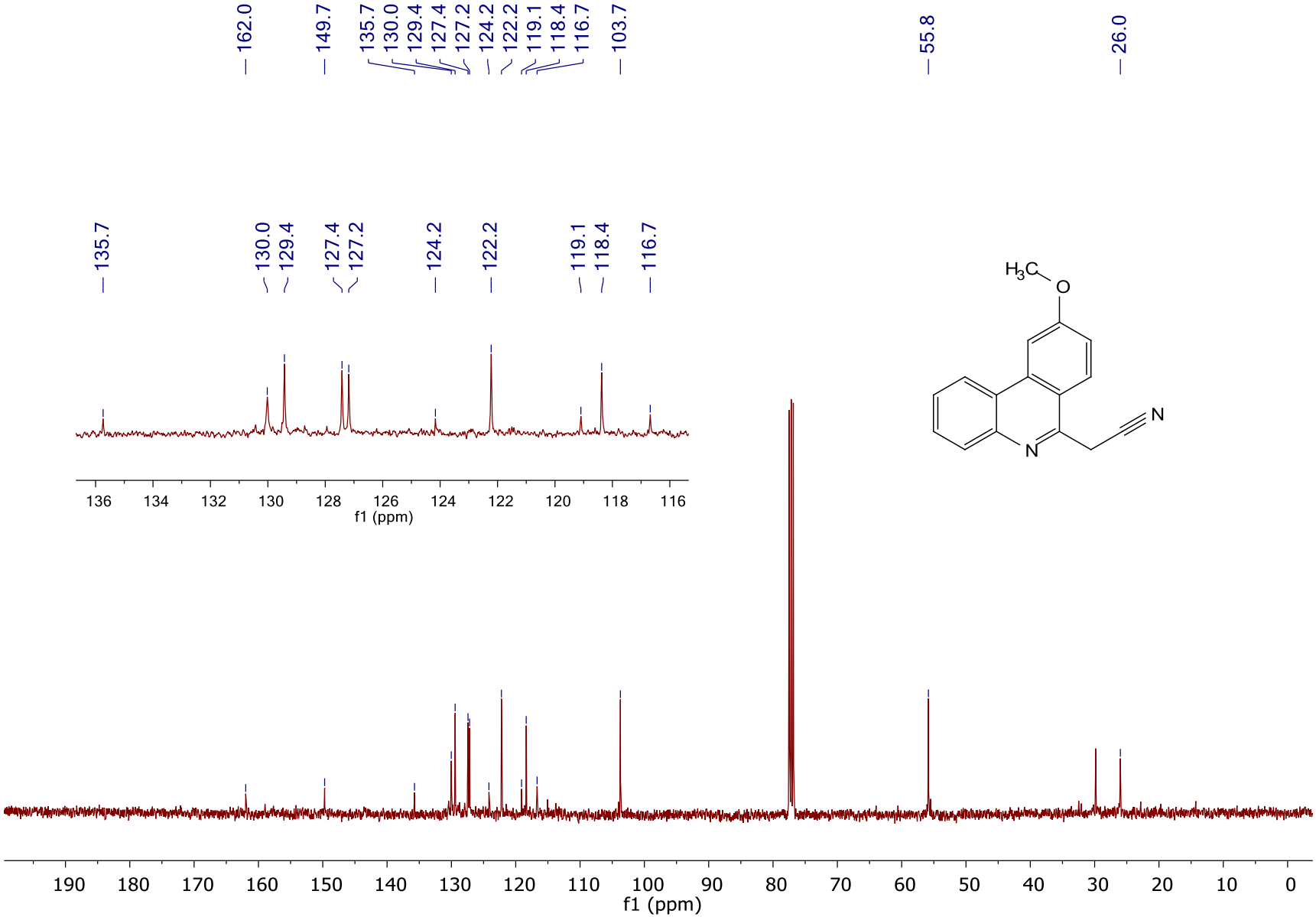
2-(10-methoxyphenanthridin-6-yl)acetonitrile (3k):



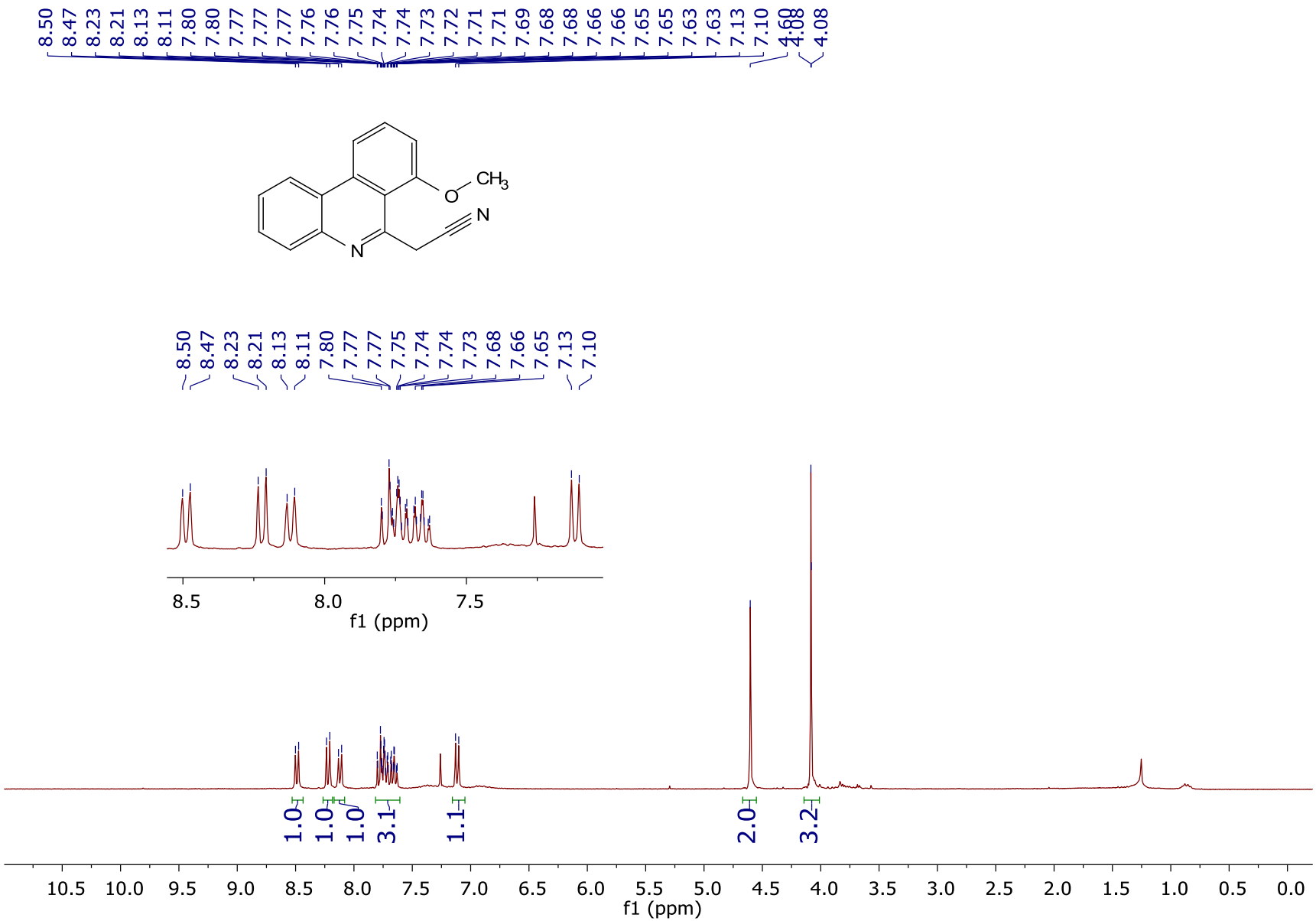

158.8
149.9
129.6
128.7
128.5
128.1
127.9
126.2
124.2
123.9
117.4
116.6
112.6
56.1
26.5
O
H3C
N
N
ppm
ppm

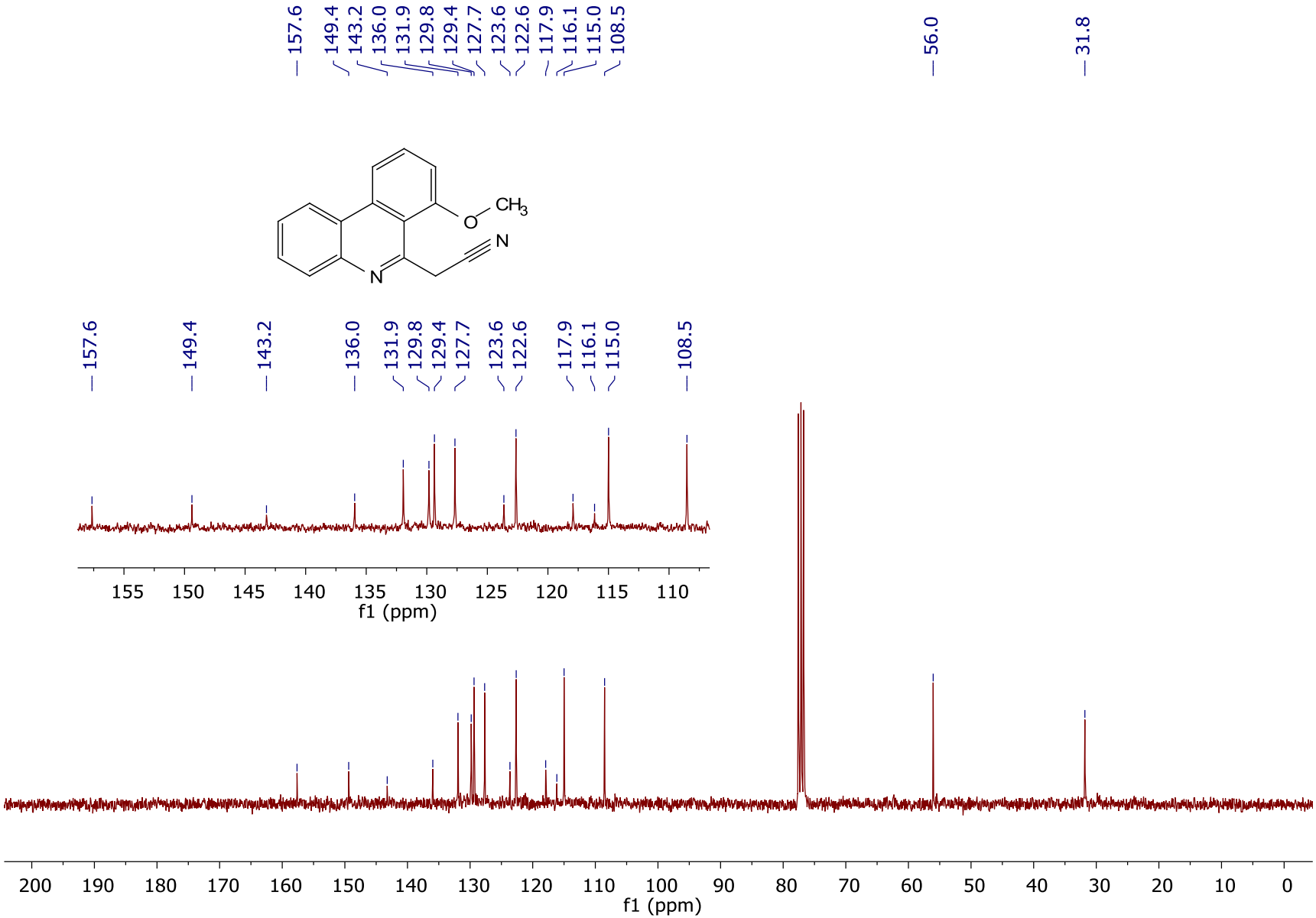
2-(9-methoxyphenanthridin-6-yl)acetonitrile (3l):



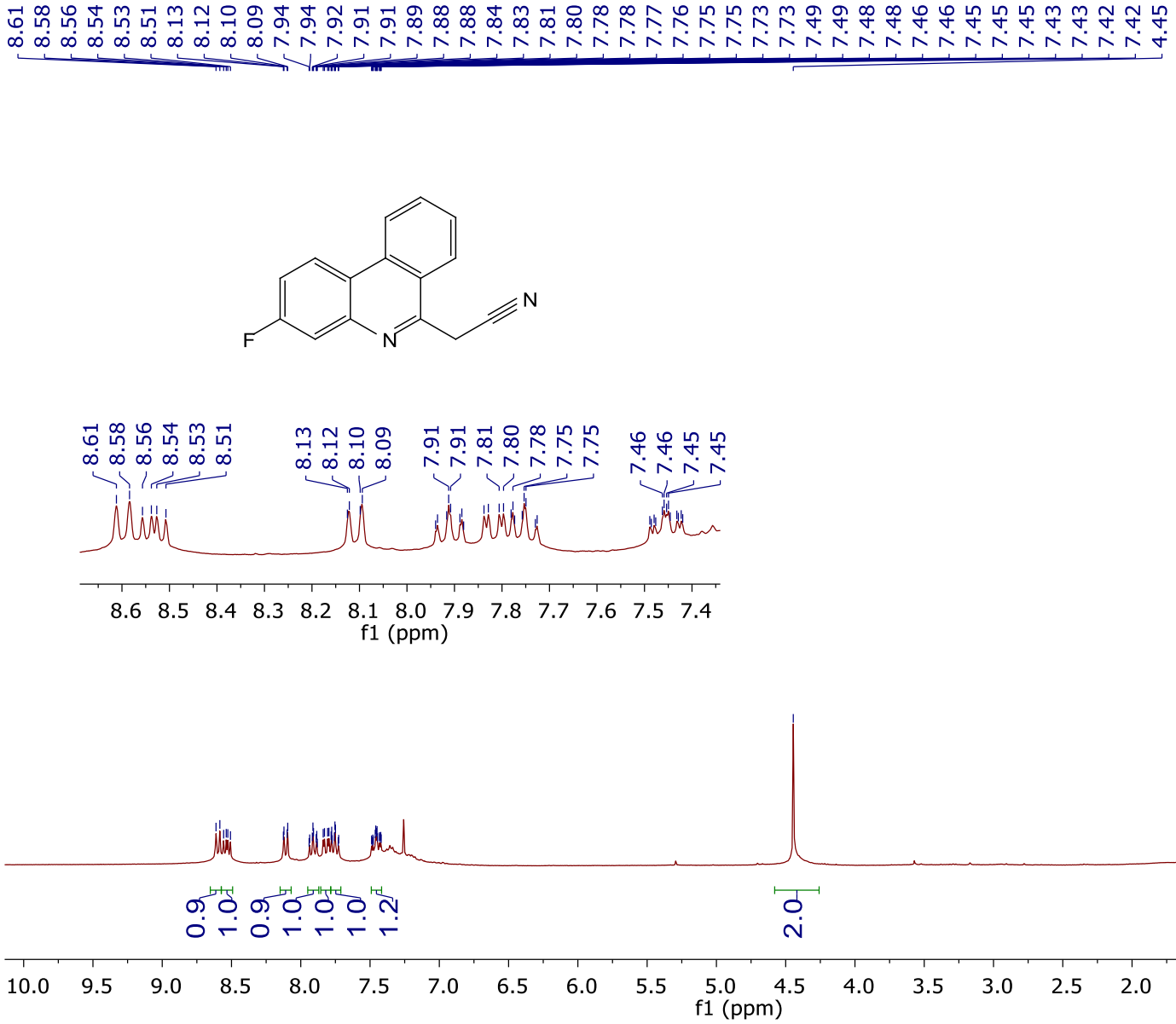

162.0
149.7
135.7
130.0
129.4
127.4
127.2
124.2
122.2
119.1
118.4
116.7
103.7
55.8
26.0
H3C
O
N
N
f1 (ppm)
f1 (ppm)

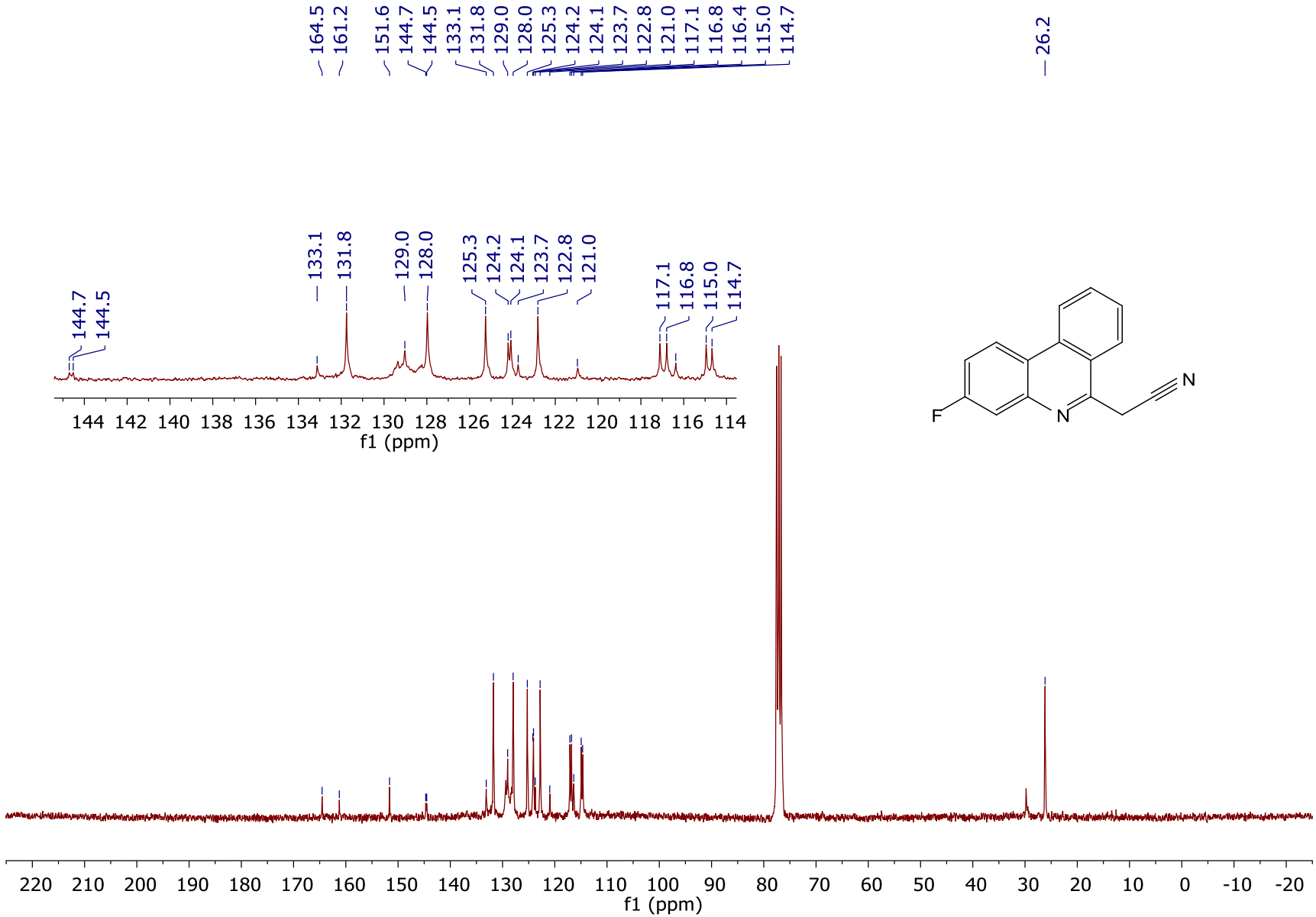
2-(7-methoxyphenanthridin-6-yl)acetonitrile (3l´):



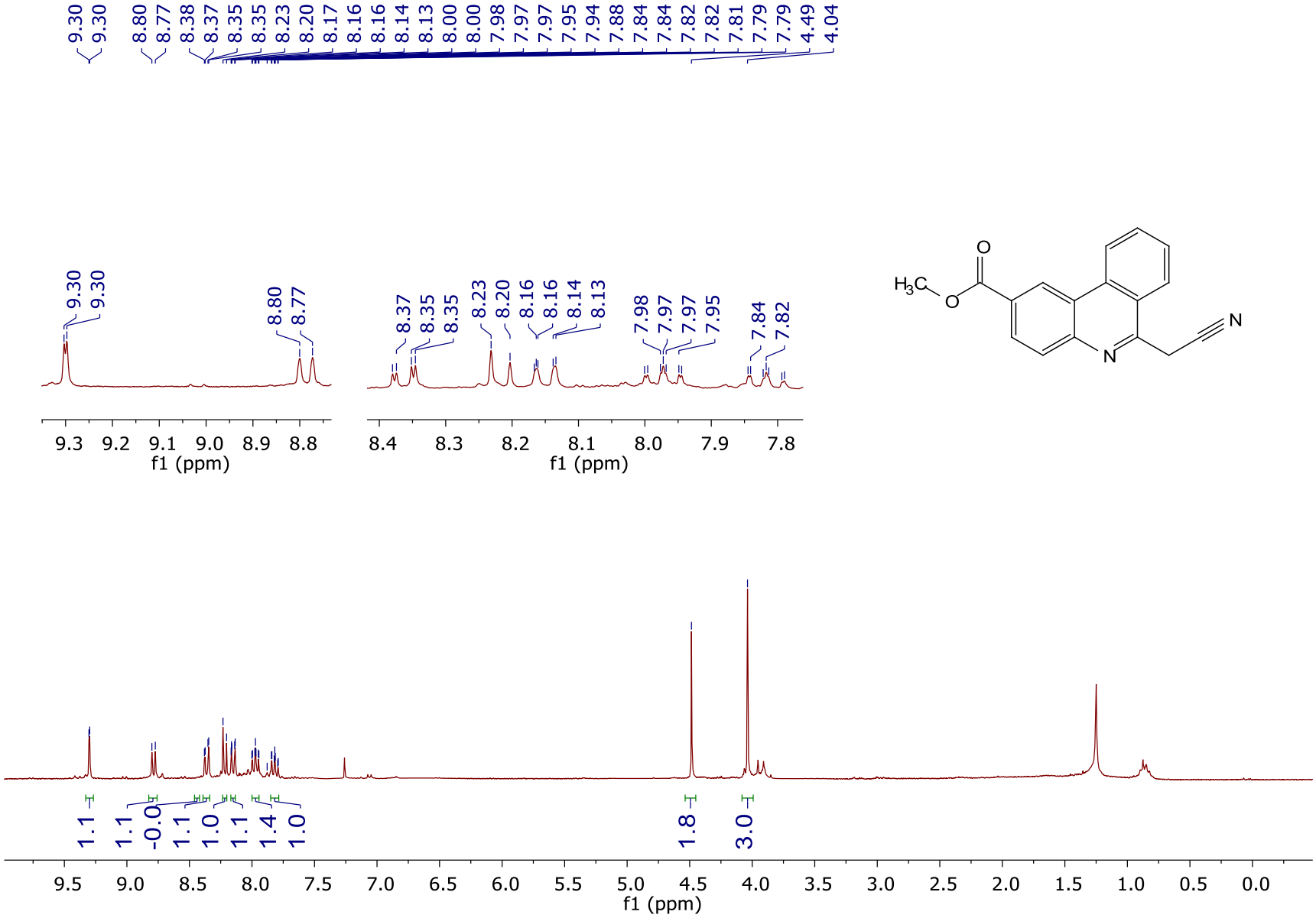

157.6
149.4
143.2
136.0
131.9
129.8
129.4
127.7
123.6
122.6
117.9
116.1
115.0
108.5
56.0
31.8
O
CH3
N
N
f1 (ppm)
f1 (ppm)

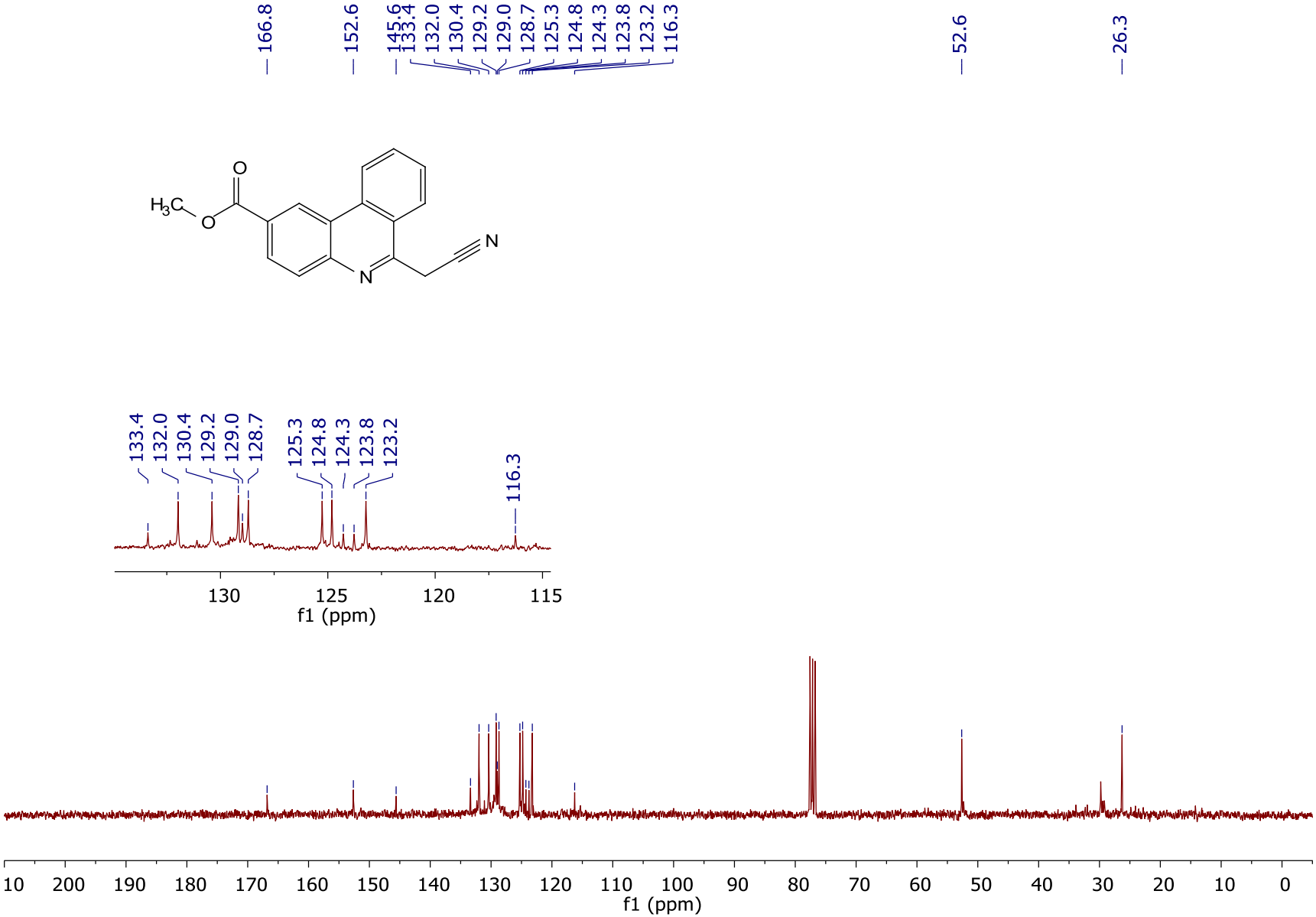
2-(3-fluorophenanthridin-6-yl)acetonitrile (3m):



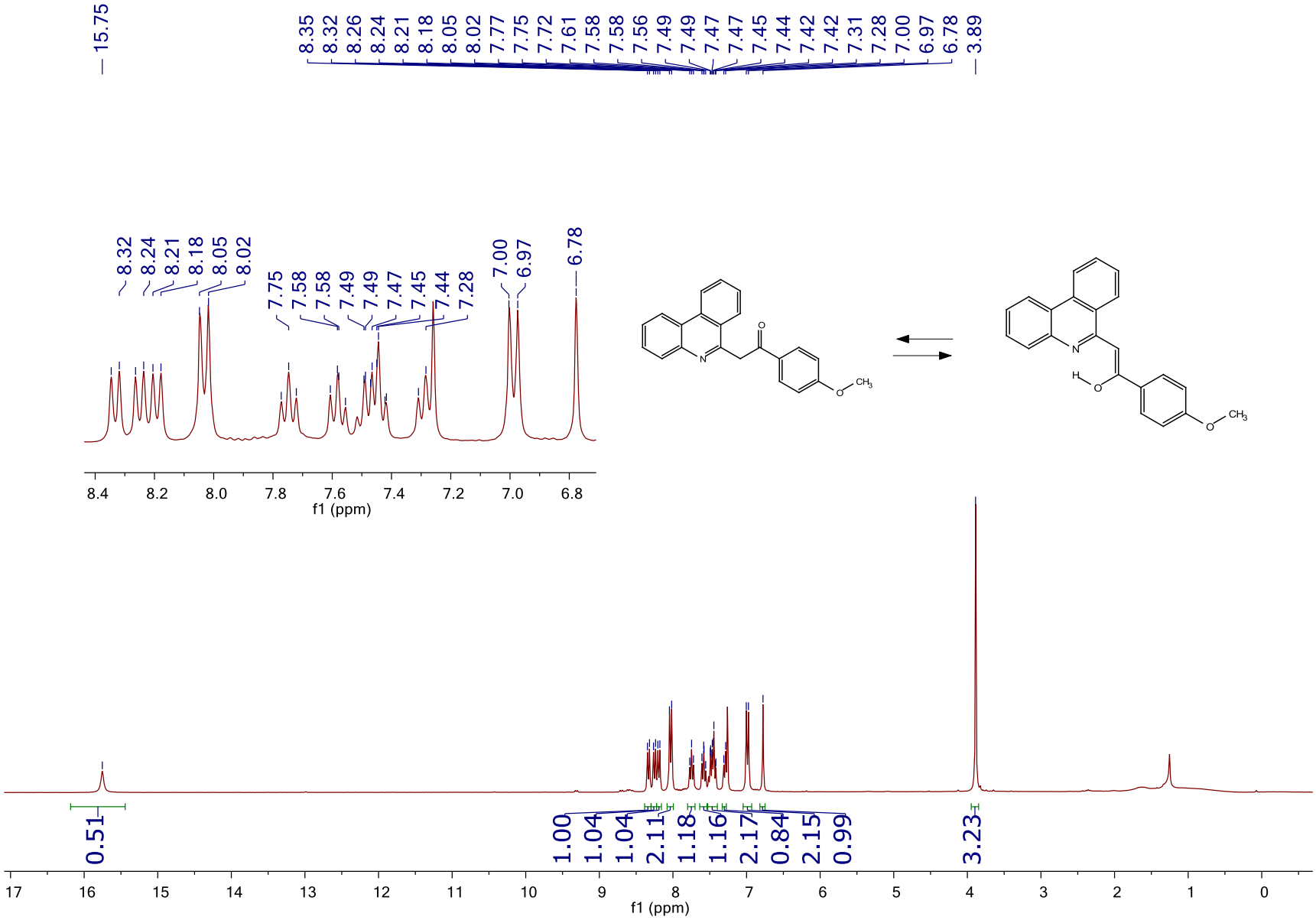

164.5
161.2
151.6
144.7
144.5
133.1
131.8
129.0
128.0
125.3
124.2
124.1
123.7
122.8
121.0
117.1
116.8
116.4
115.0
114.7
26.2
144.7
144.5
133.1
131.8
129.0
128.0
125.3
124.2
124.1
123.7
122.8
121.0
117.1
116.8
115.0
114.7
144 142 140 138 136 134 132 130 128 126 124 122 120 118 116 114
f1 (ppm)
F
N
N
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

methyl 6-(cyanomethyl)phenanthridine-2-carboxylate (3n):

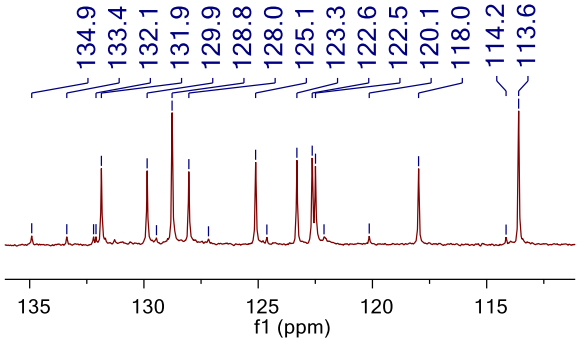


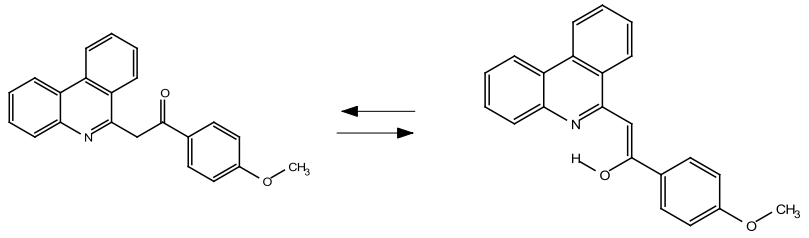

166.8
152.6
145.6
133.4
132.0
130.4
129.2
129.0
128.7
125.3
124.8
124.3
123.8
123.2
116.3
52.6
26.3
O
H3C
O
N
N
133.4
132.0
130.4
129.2
129.0
128.7
125.3
124.8
124.3
123.8
123.2
116.3
130
125
120
115
f1 (ppm)
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

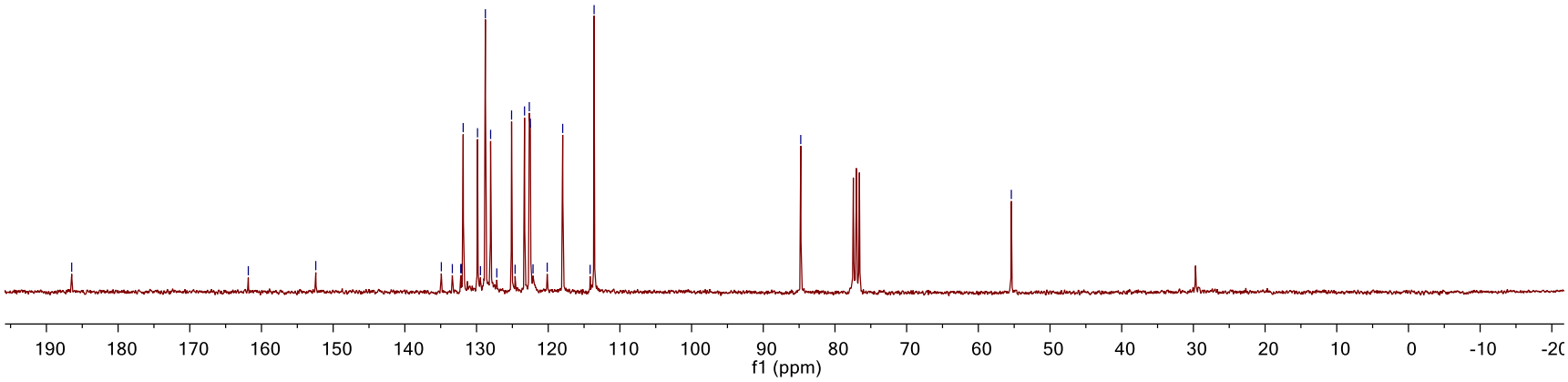
1-(4-methoxyphenyl)-2-(phenanthridin-6-yl)ethan-1-one (4a):



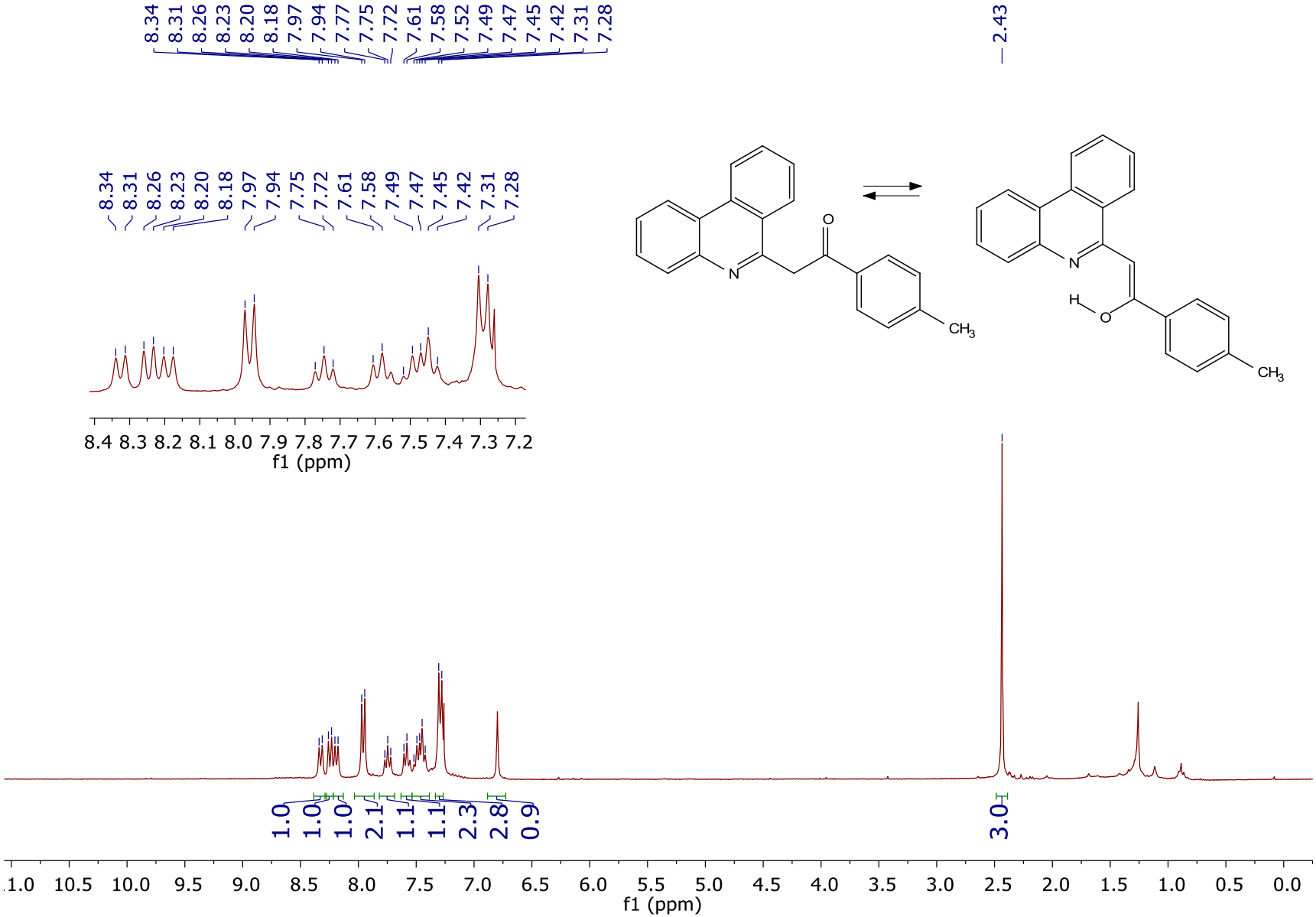

186.5
161.8
152.4
134.9
133.4
132.2
132.1
131.9
129.9
129.5
128.8
128.0
127.2
125.1
124.6
123.3
122.6
122.5
122.1
120.1
118.0
114.2
113.6
84.8
55.4

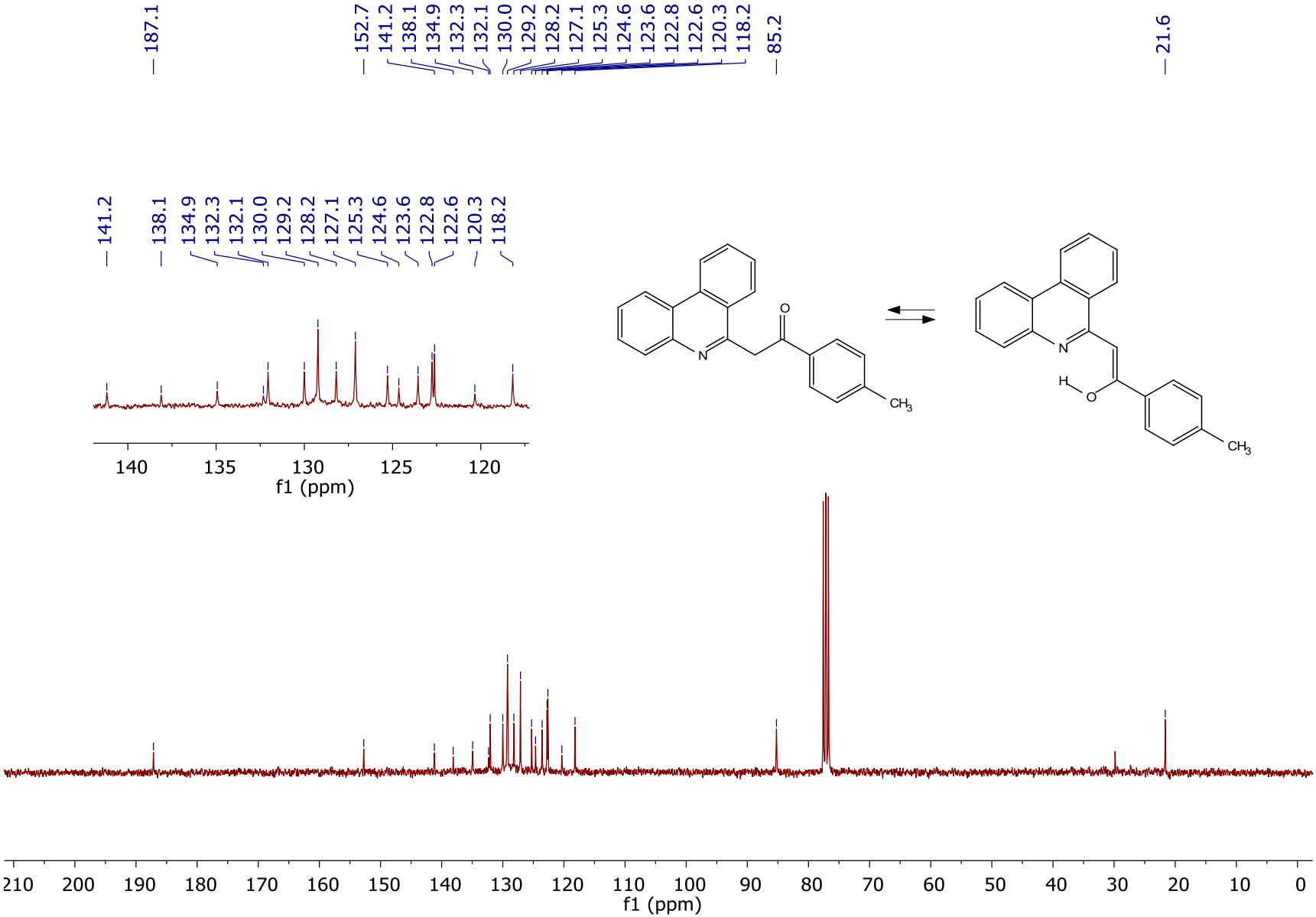

134.9
133.4
132.1
131.9
129.9
128.8
128.0
125.1
123.3
122.6
122.5
120.1
118.0
114.2
113.6
135
130
125
120
115
f1 (ppm)


O
N
O
CH3
N
H
O
O
CH3

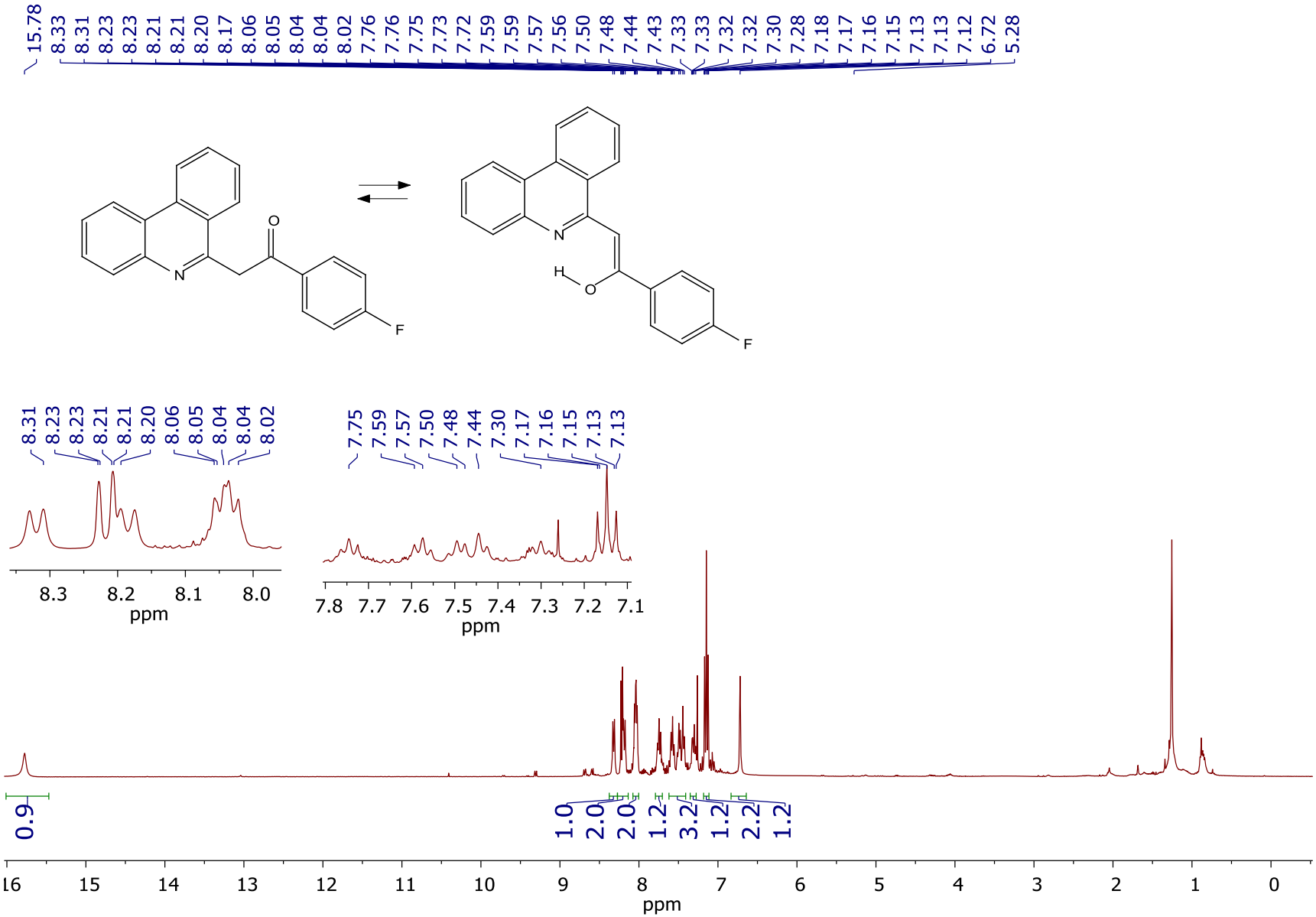

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

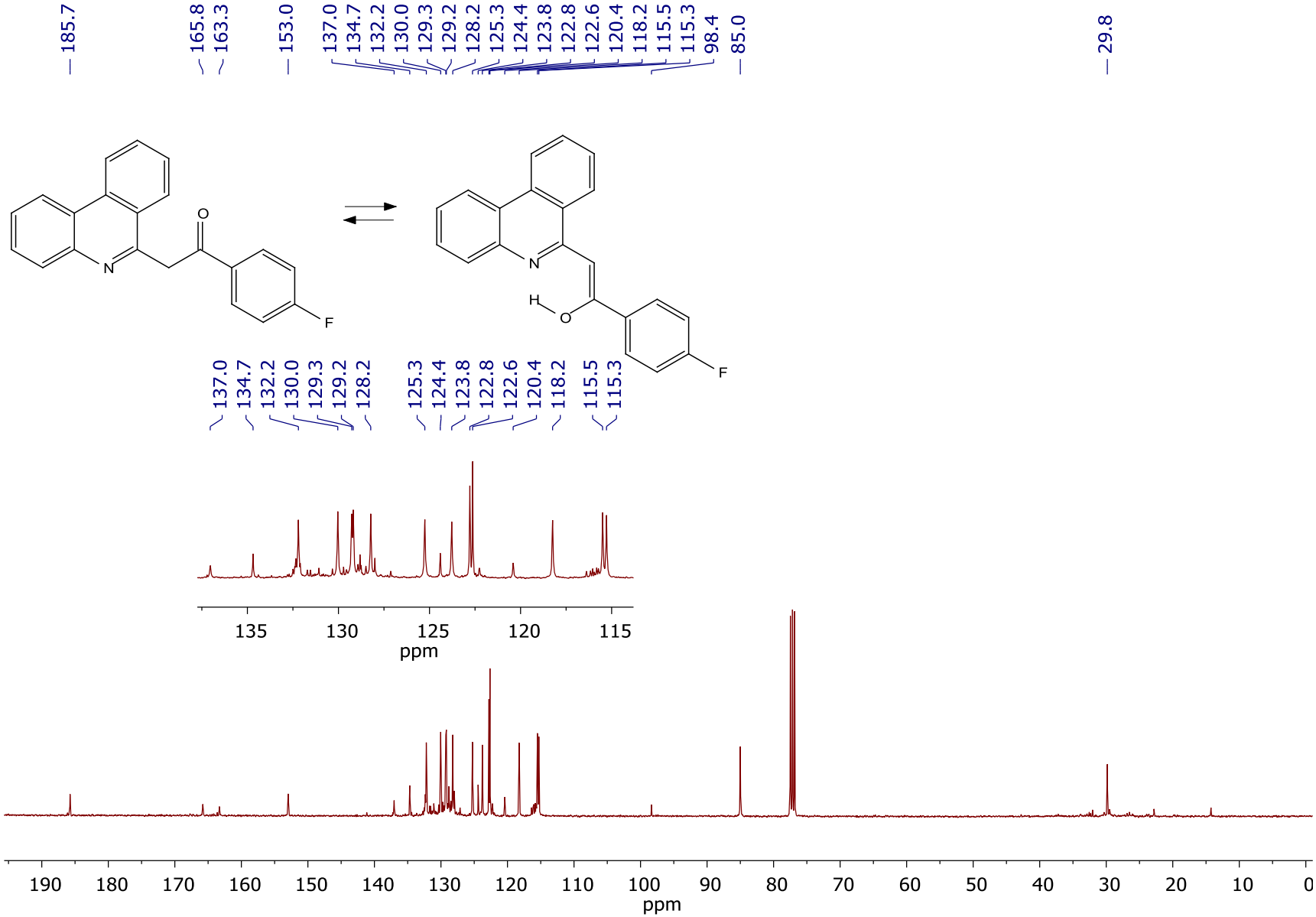
2-(phenanthridin-6-yl)-1-(*p*-tolyl)ethan-1-one (4b):



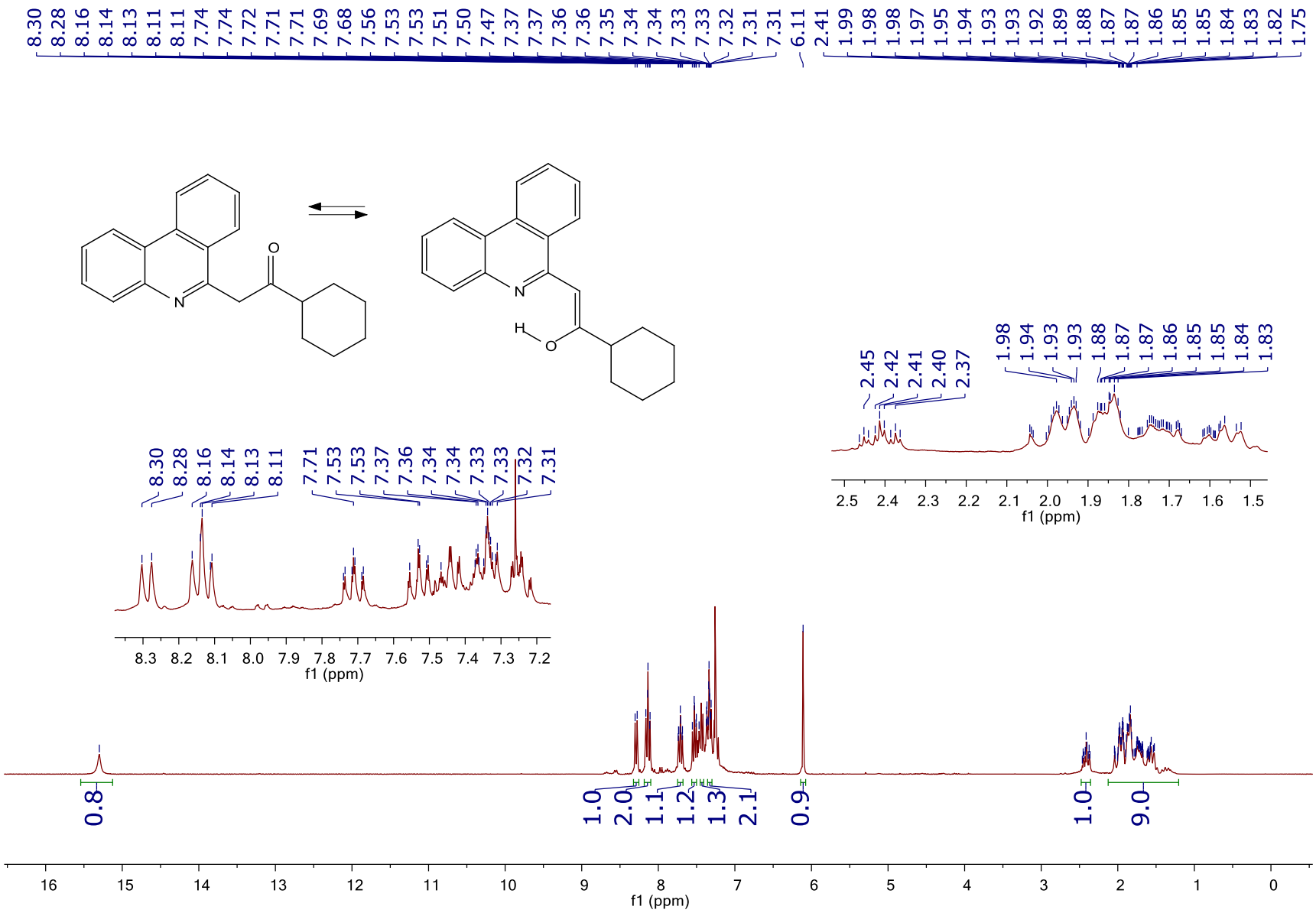

187.1
152.7
141.2
138.1
134.9
132.3
132.1
130.0
129.2
128.2
127.1
125.3
124.6
123.6
122.8
122.6
120.3
118.2
85.2
21.6
141.2
138.1
134.9
132.3
132.1
130.0
129.2
128.2
127.1
125.3
124.6
123.6
122.8
122.6
120.3
118.2
140
135
130
125
120
f1 (ppm)
O
N
CH3
N
H
O
CH3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

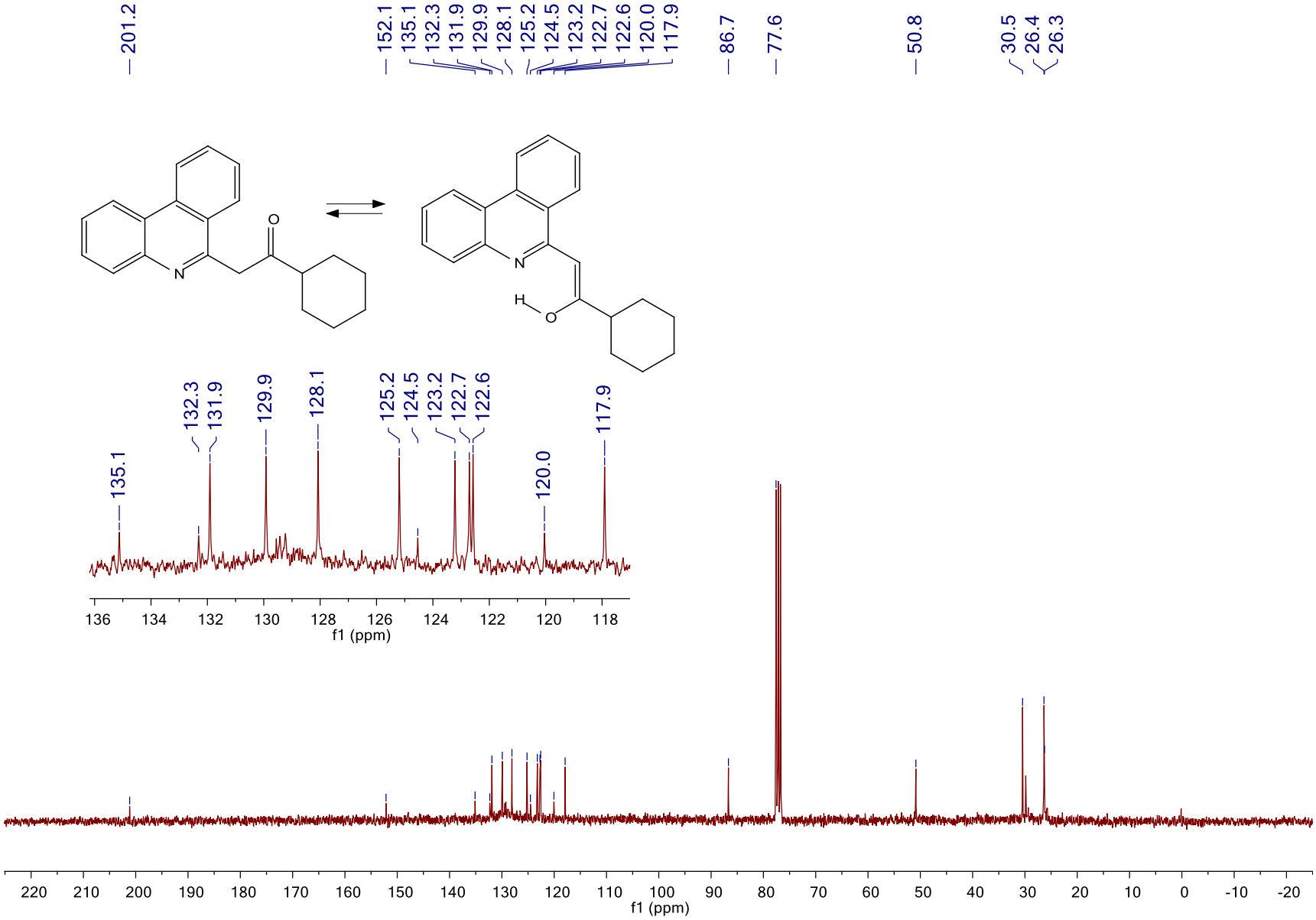
1-(4-fluorophenyl)-2-(phenanthridin-6-yl)ethan-1-one (4c):



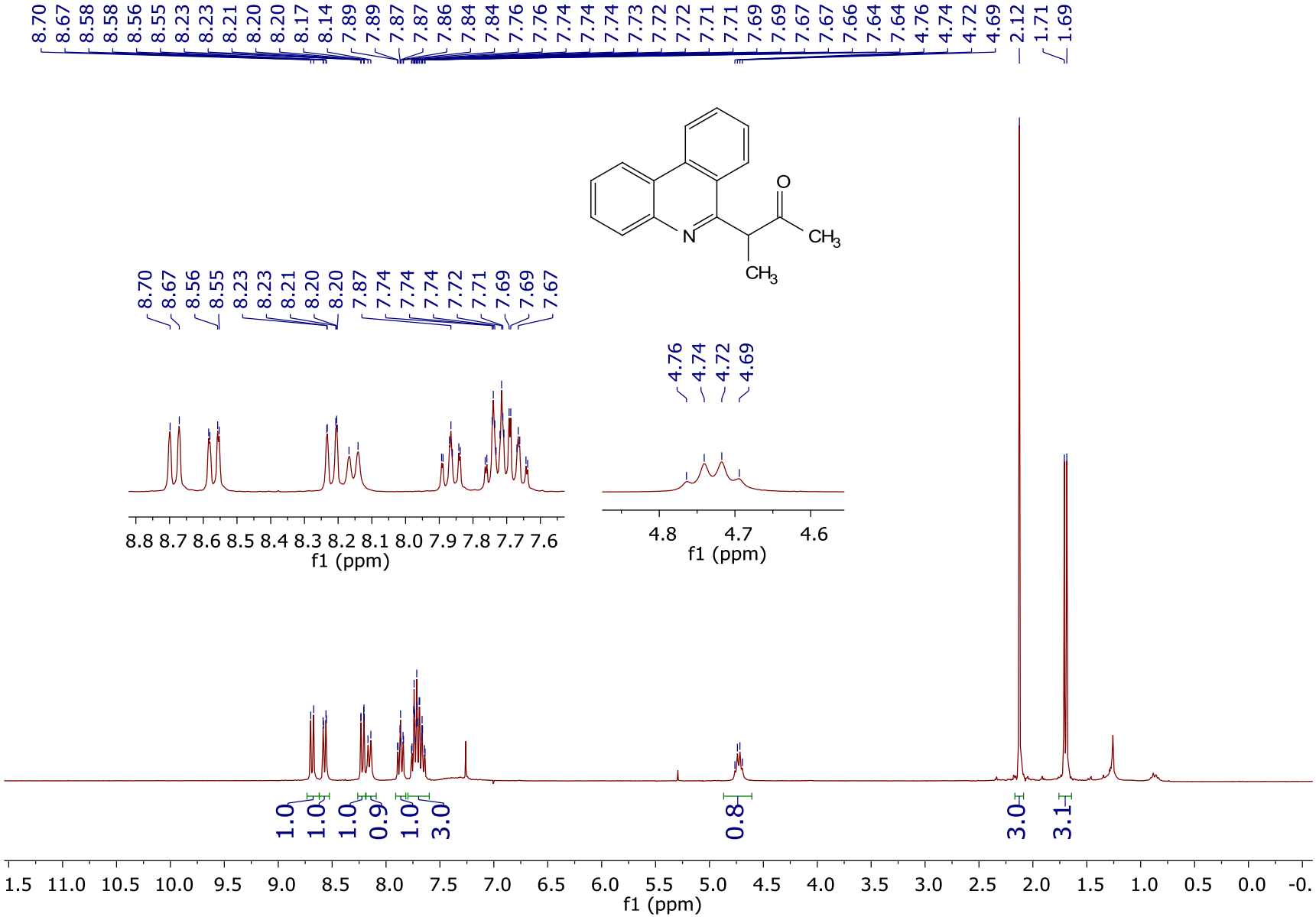

185.7
165.8
163.3
153.0
137.0
134.7
132.2
130.0
129.3
129.2
128.2
125.3
124.4
123.8
122.8
122.6
120.4
118.2
115.5
115.3
98.4
85.0
29.8
O
N
F
H
O
N
F
137.0
134.7
132.2
130.0
129.3
129.2
128.2
125.3
124.4
123.8
122.8
122.6
120.4
118.2
115.5
115.3
135
130
125
120
115
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

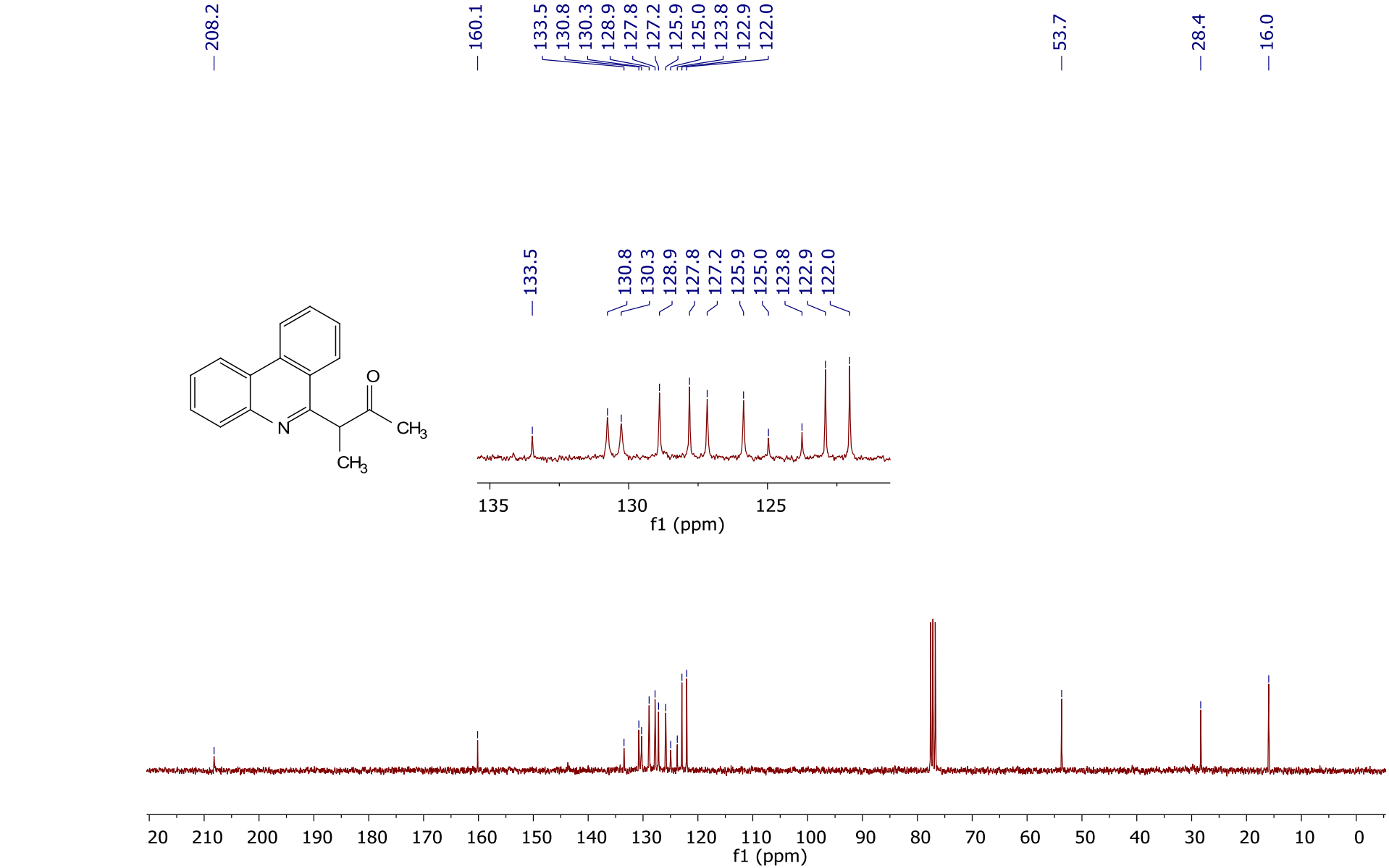
1-cyclohexyl-2-(phenanthridin-6-yl)ethan-1-one (4d):



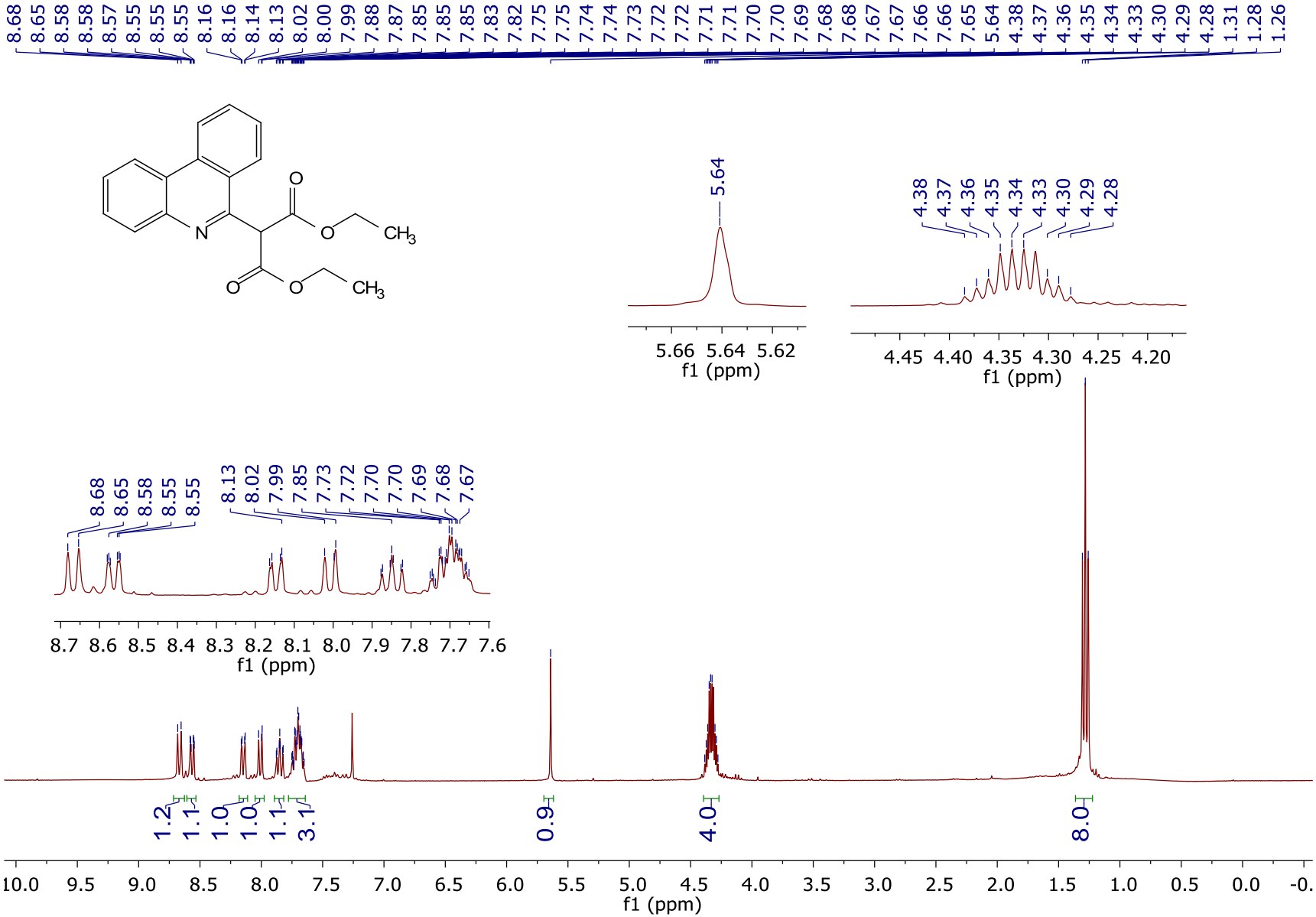

201.2
152.1
135.1
132.3
131.9
129.9
128.1
125.2
124.5
123.2
122.7
122.6
120.0
117.9
86.7
77.6
50.8
30.5
26.4
26.3
O
N
H
O
f1 (ppm)
f1 (ppm)

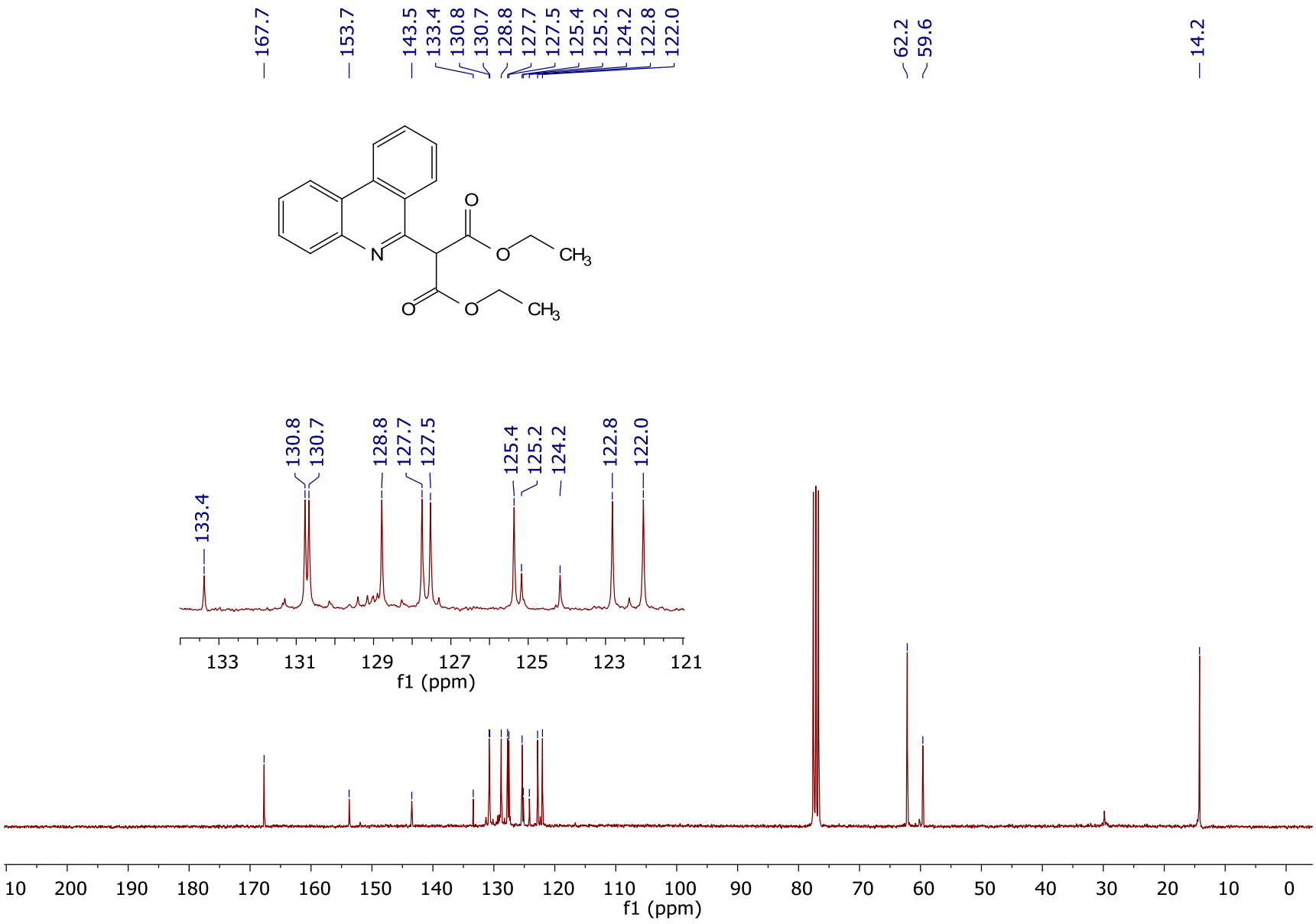
3-(phenanthridin-6-yl)butan-2-one (4e):



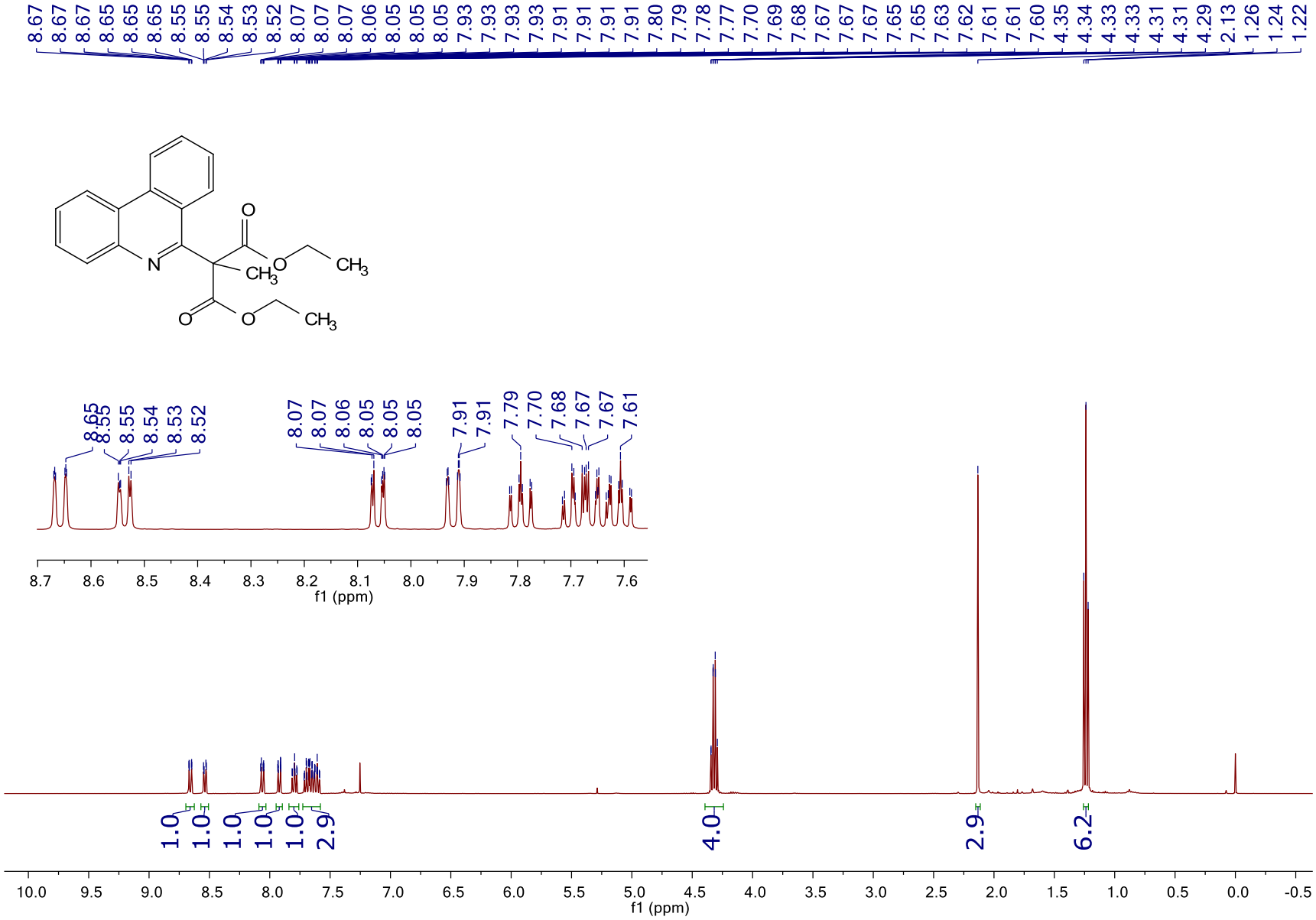

208.2
160.1
133.5
130.8
130.3
128.9
127.8
127.2
125.9
125.0
123.8
122.9
122.0
53.7
28.4
16.0
O
N
CH3
CH3
135
130
125
f1 (ppm)
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

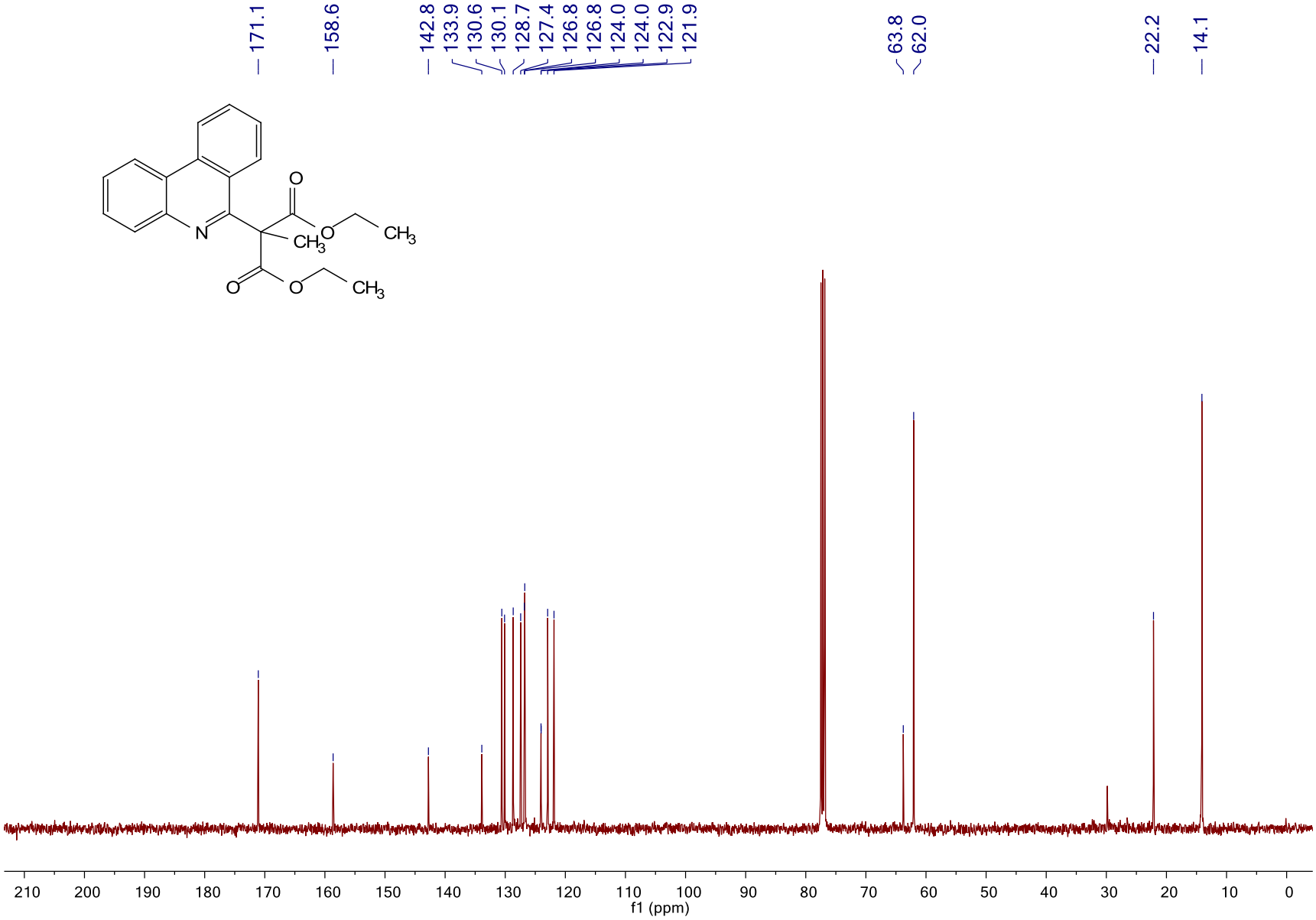
diethyl 2-(phenanthridin-6-yl)malonate (4f):



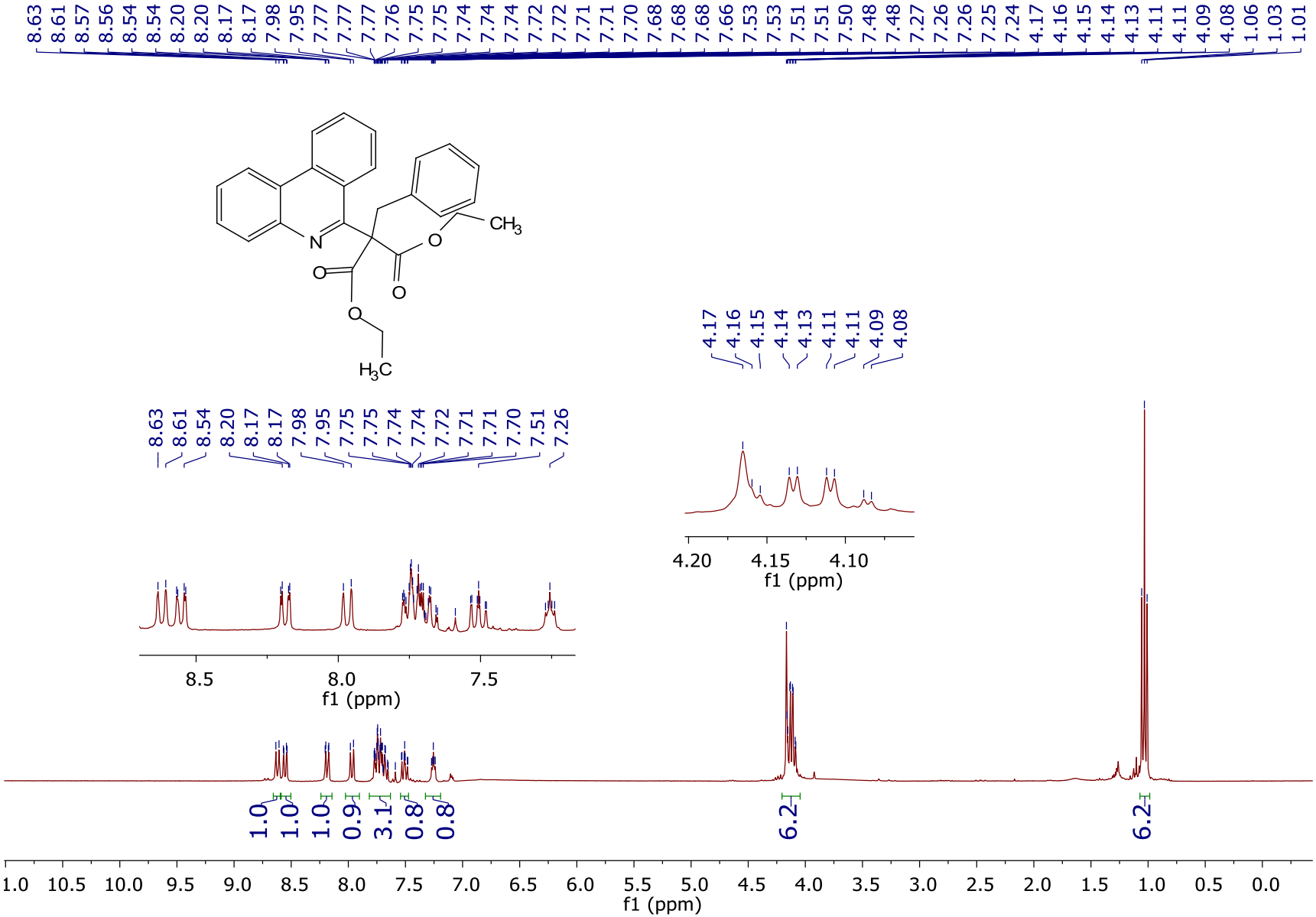

167.7
153.7
143.5
133.4
130.8
130.7
128.8
127.7
127.5
125.4
125.2
124.2
122.8
122.0
62.2
59.6
14.2
O
O
N
O
CH3
O
O
CH3
133.4
130.8
130.7
128.8
127.7
127.5
125.4
125.2
124.2
122.8
122.0
133
131
129
127
125
123
121
f1 (ppm)
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

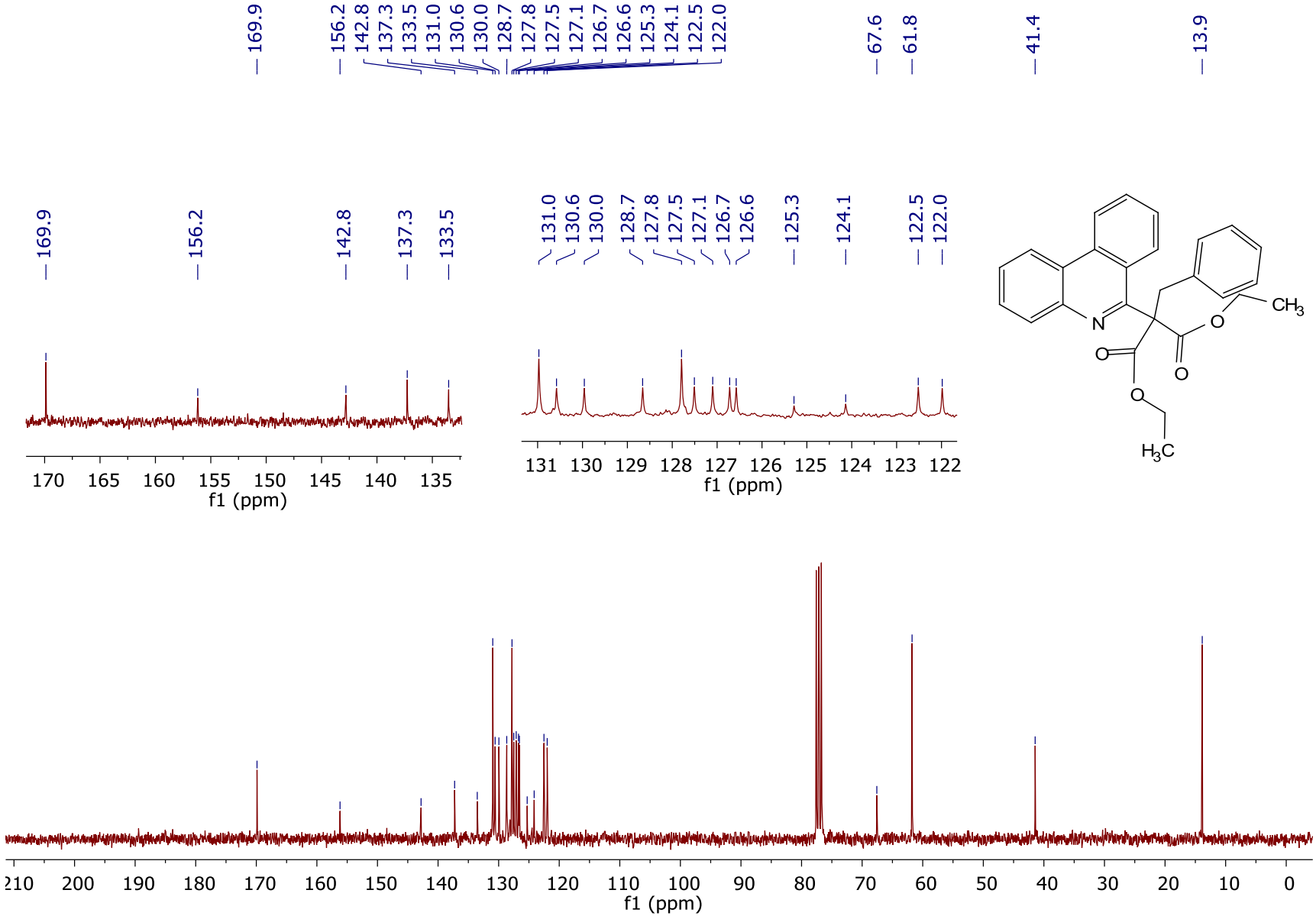
diethyl 2-methyl-2-(phenanthridin-6-yl)malonate (4g):



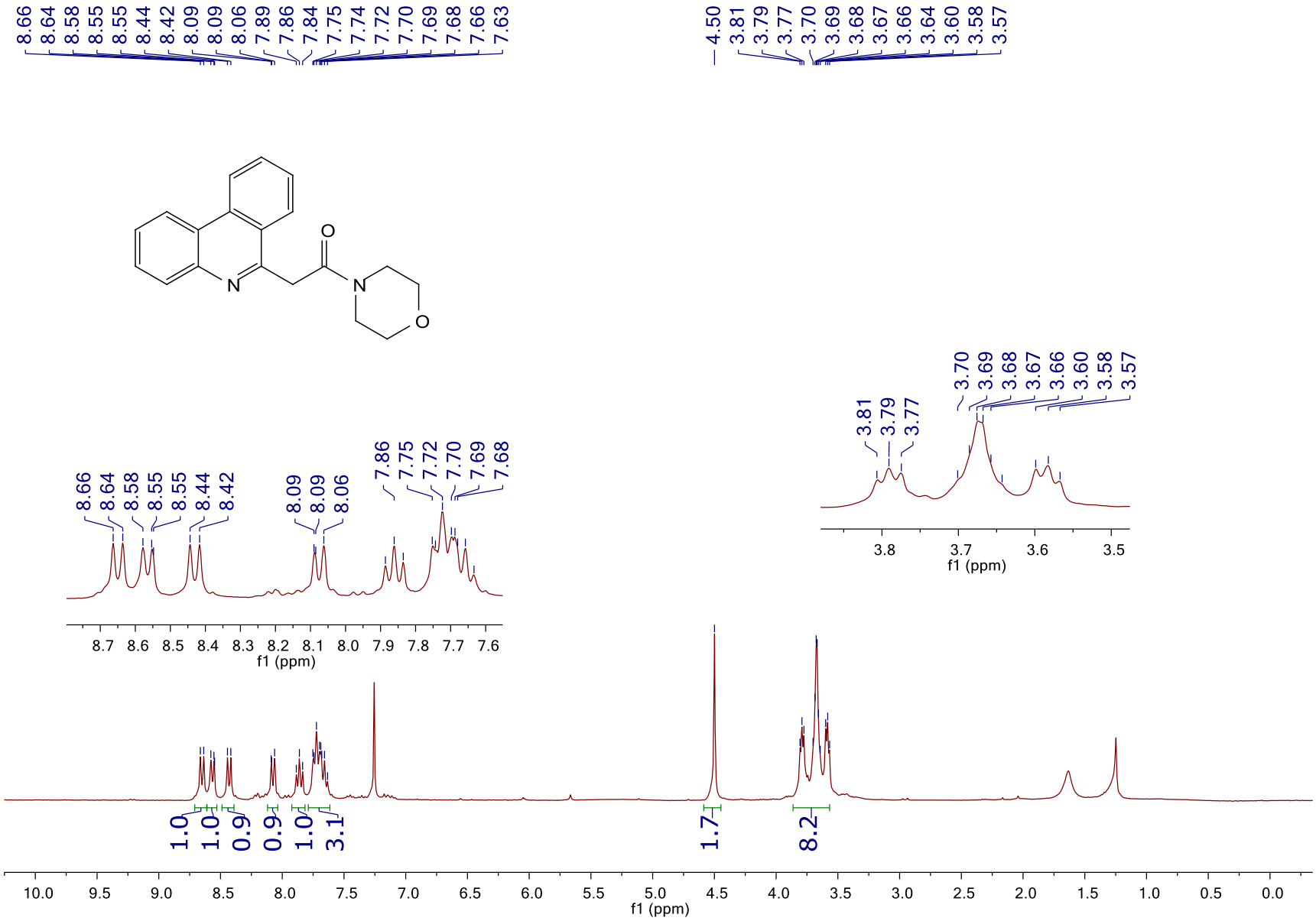

171.1
158.6
142.8
133.9
130.6
130.1
128.7
127.4
126.8
126.8
124.0
124.0
122.9
121.9
63.8
62.0
22.2
14.1
O
O
CH3
N
CH3
O
O
CH3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

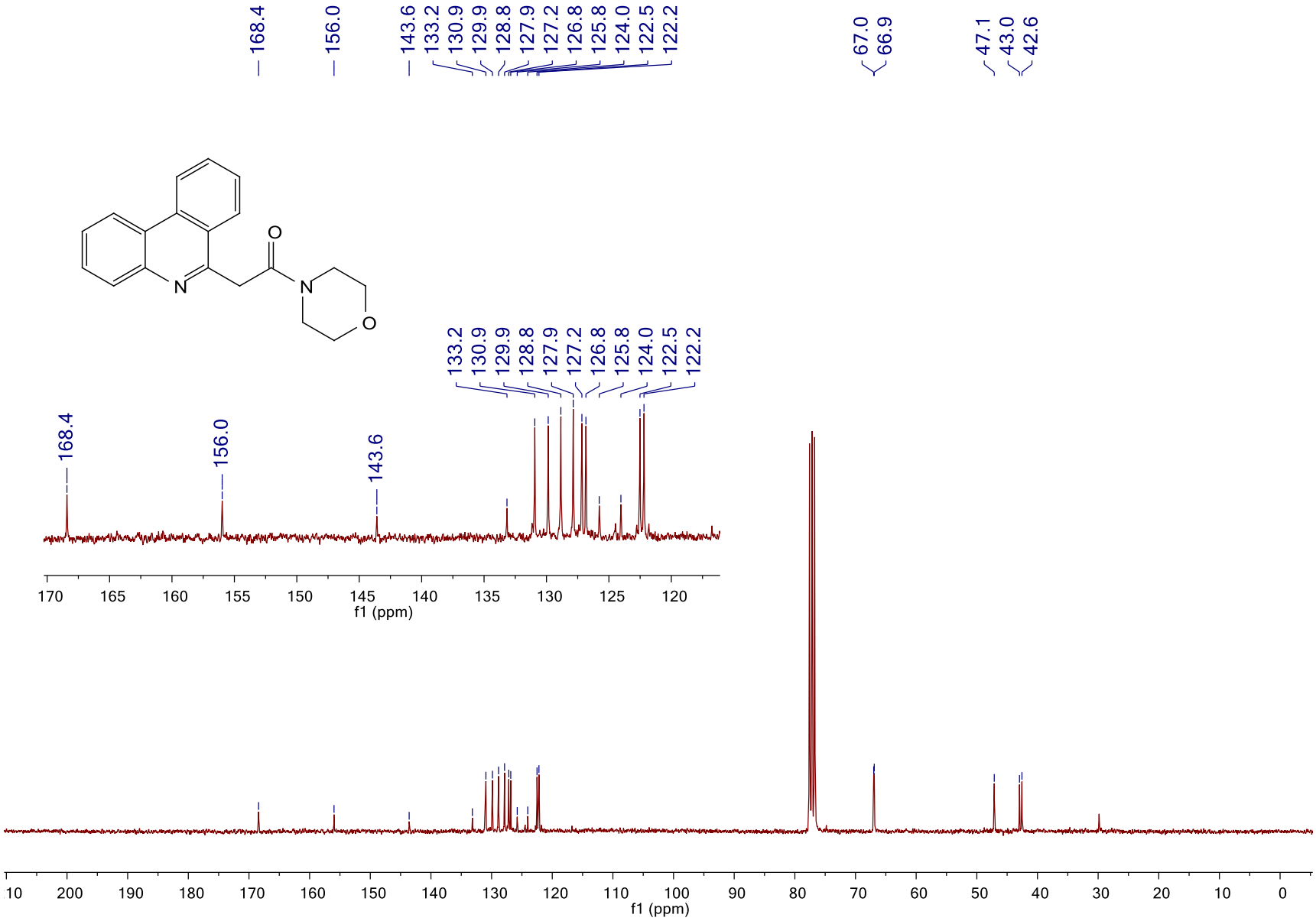
diethyl 2-benzyl-2-(phenanthridin-6-yl)malonate (4h):



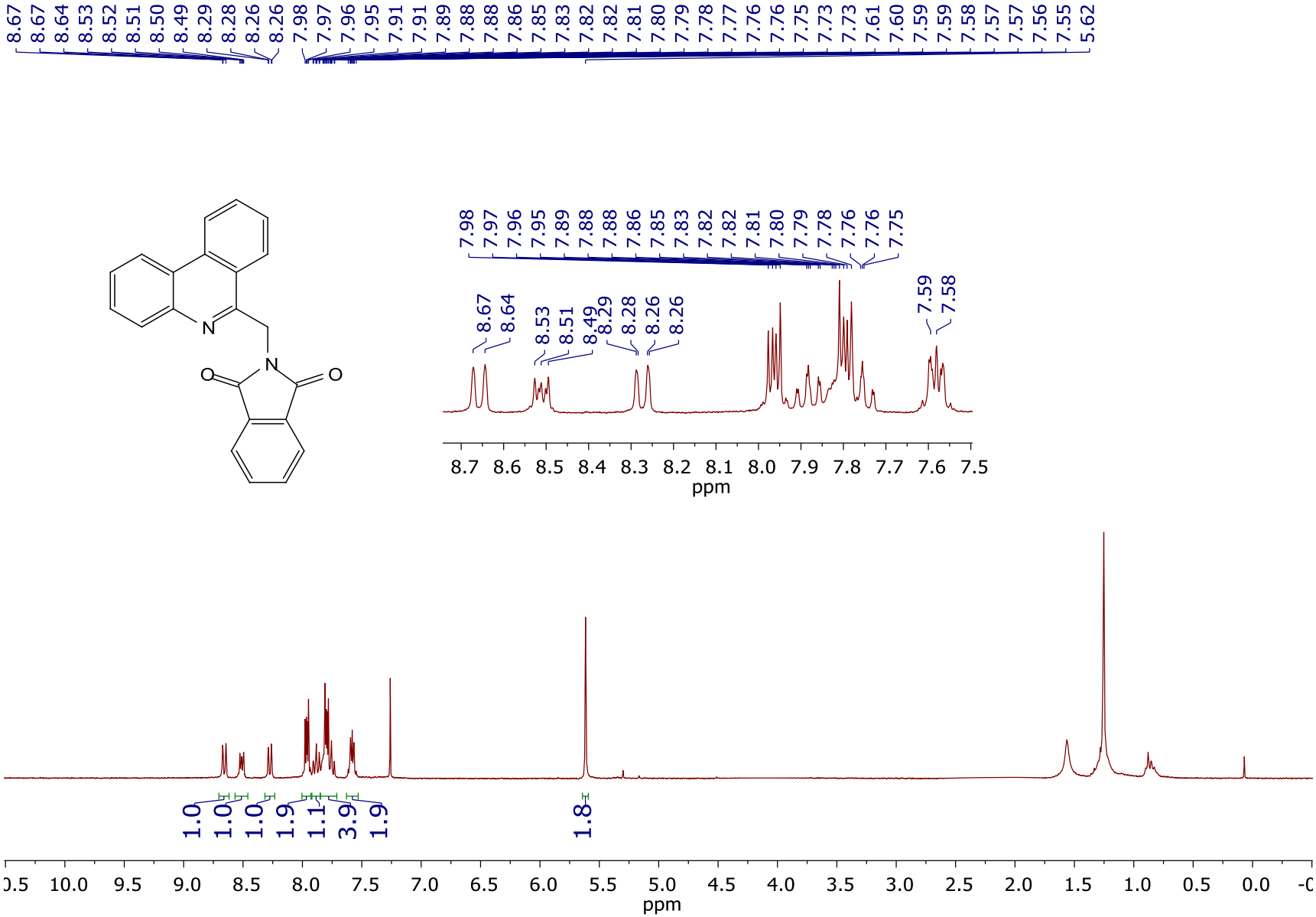

169.9
156.2
142.8
137.3
133.5
131.0
130.6
130.0
128.7
127.8
127.5
127.1
126.7
126.6
125.3
124.1
122.5
122.0
67.6
61.8
41.4
13.9
f1 (ppm)
CH3
N
O
H3C

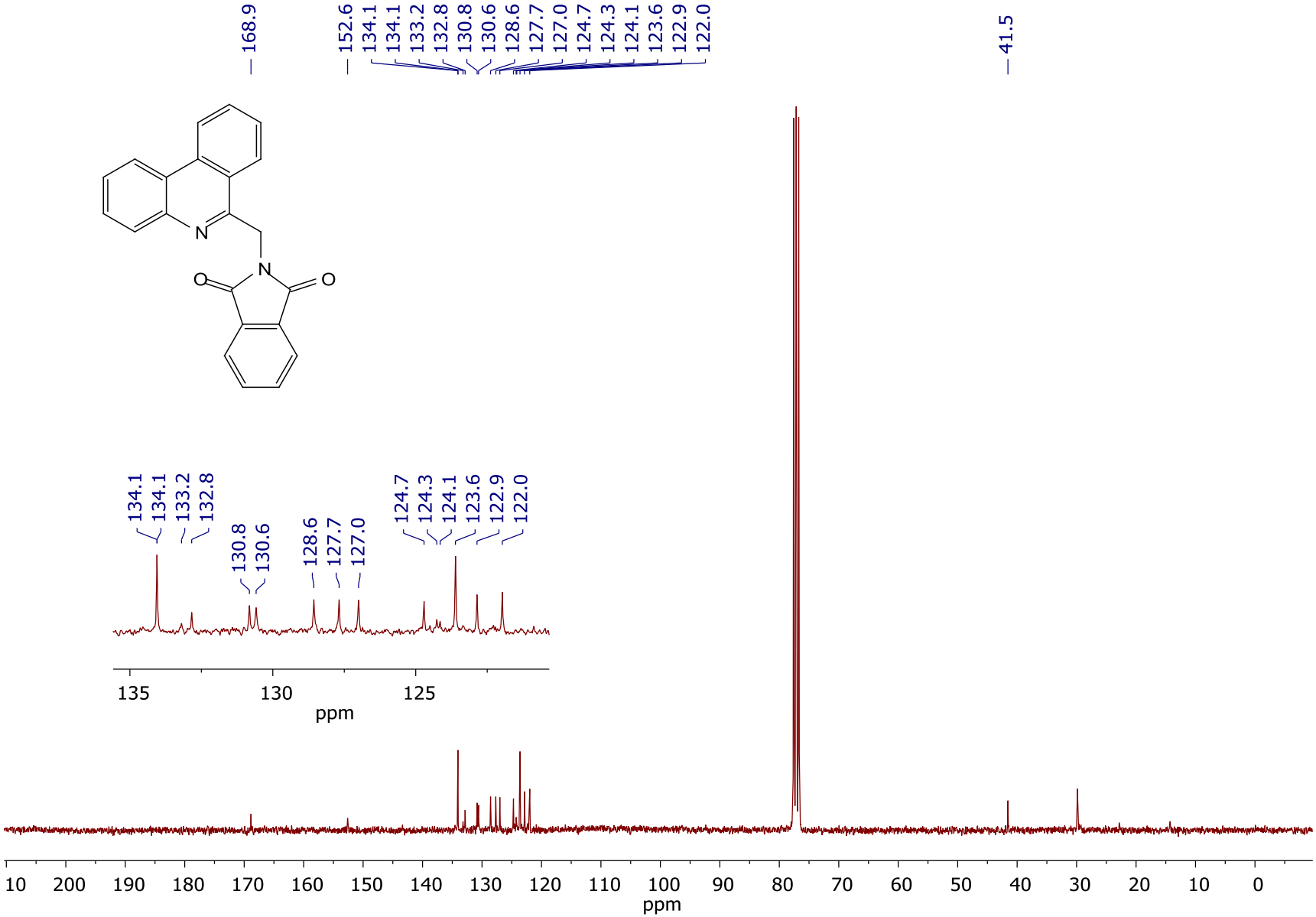
1-morpholino-2-(phenanthridin-6-yl)ethan-1-one (4i):



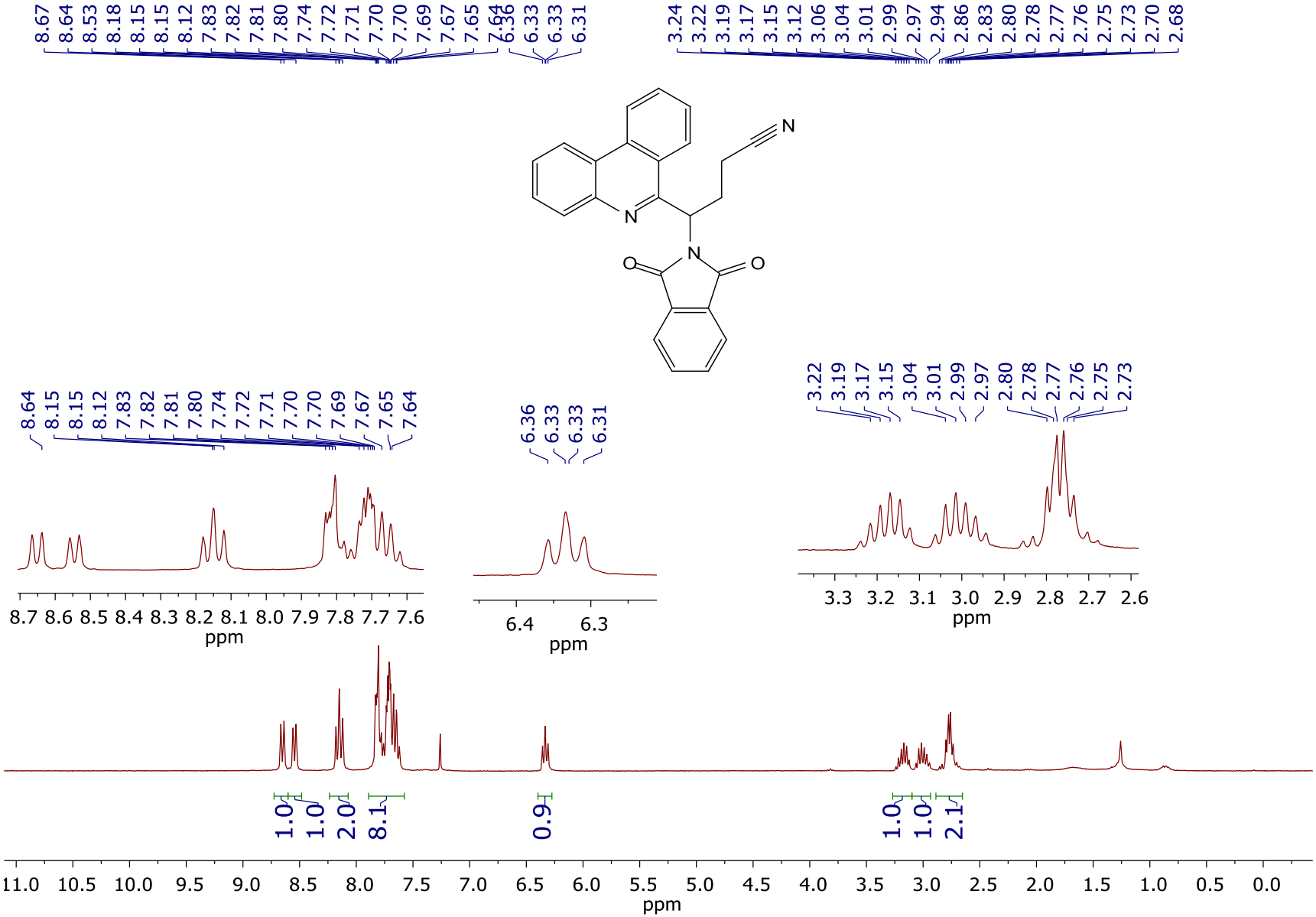

168.4
156.0
143.6
133.2
130.9
129.9
128.8
127.9
127.2
126.8
125.8
124.0
122.5
122.2
67.0
66.9
47.1
43.0
42.6
O
N
N
O
f1 (ppm)
f1 (ppm)

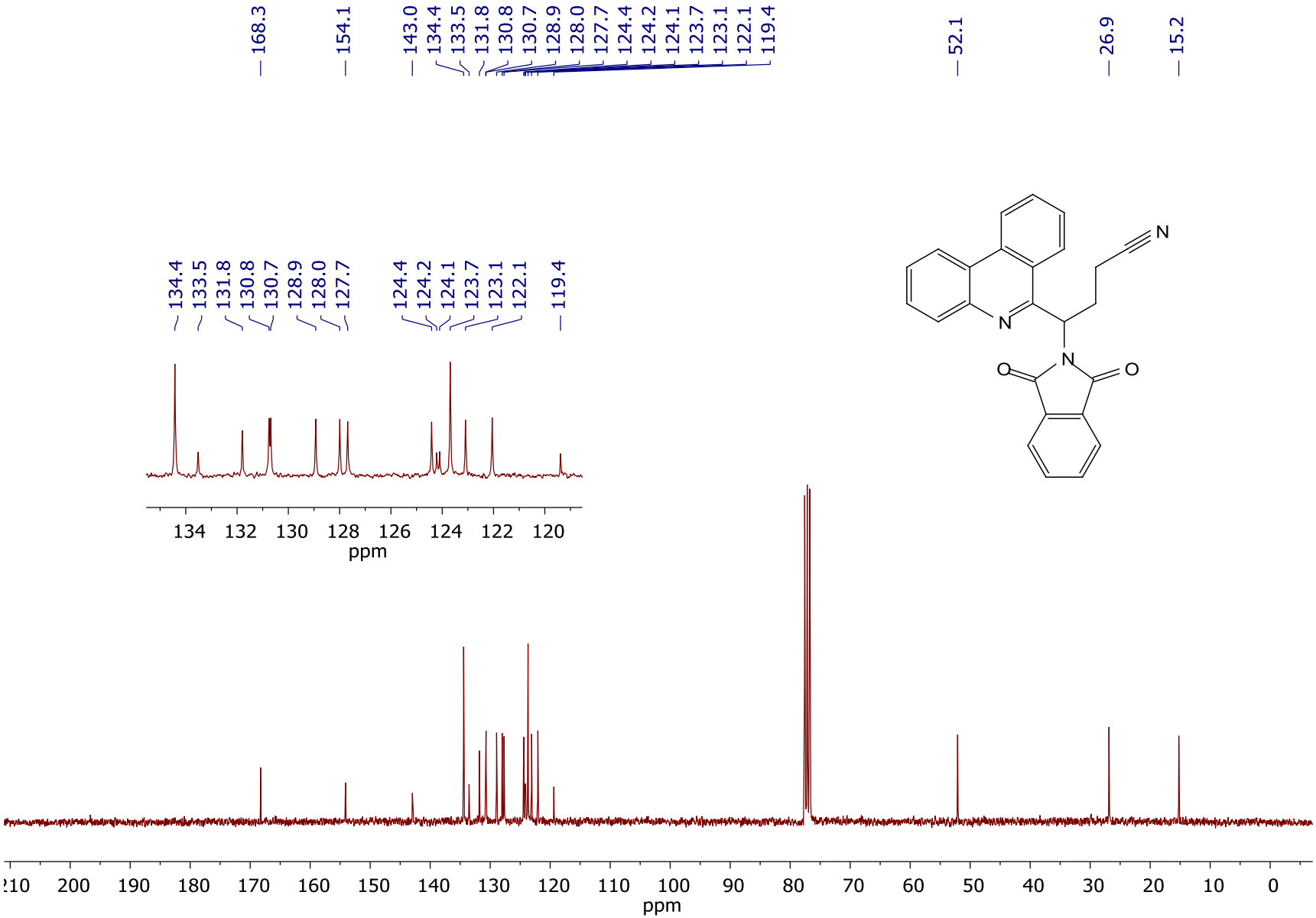
2-(phenanthridin-6-ylmethyl)isoindoline-1,3-dione (4j):



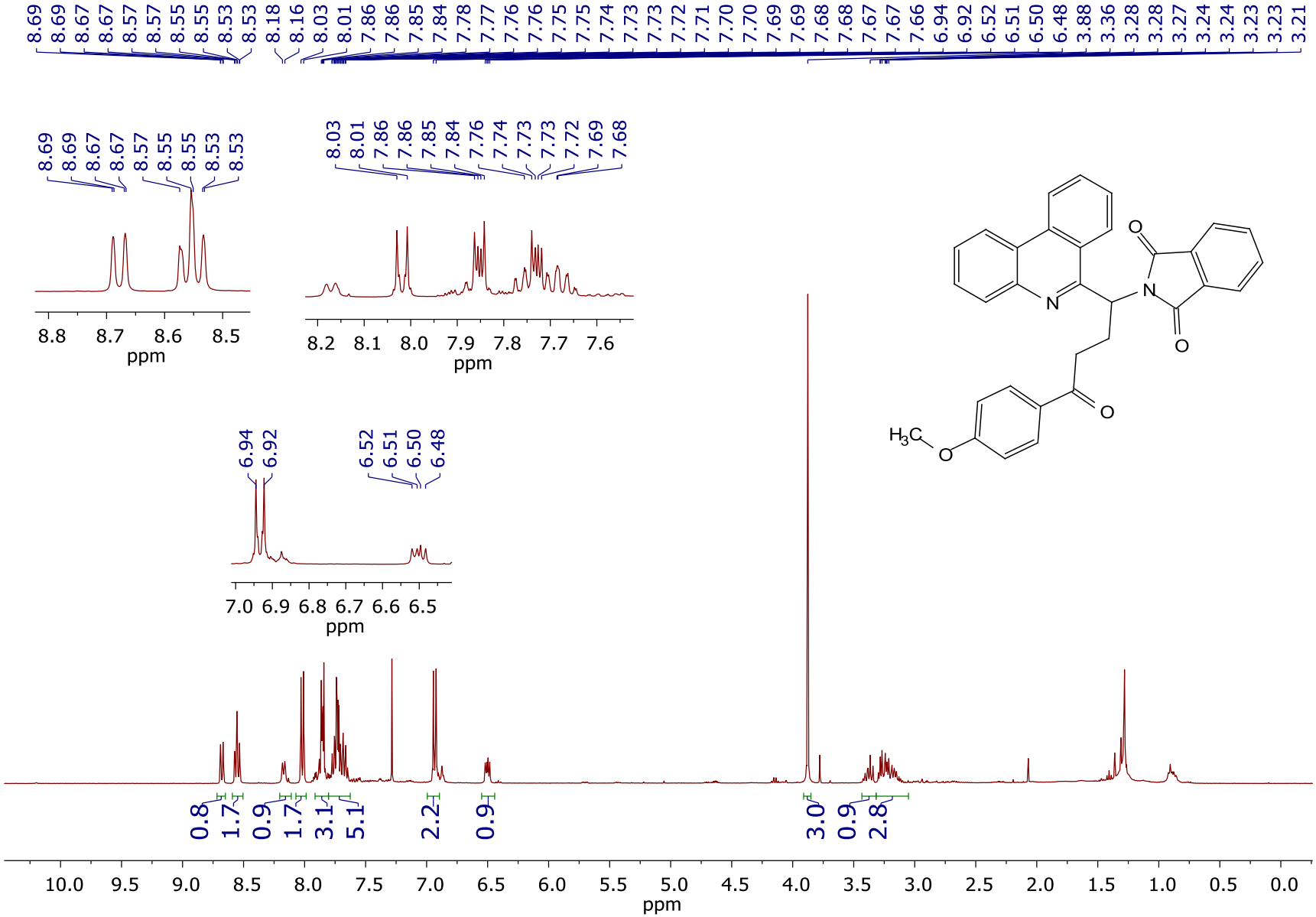

168.9
152.6
134.1
134.1
133.2
132.8
130.8
130.6
128.6
127.7
127.0
124.7
124.3
124.1
123.6
122.9
122.0
41.5
N
N
O
O
ppm
ppm

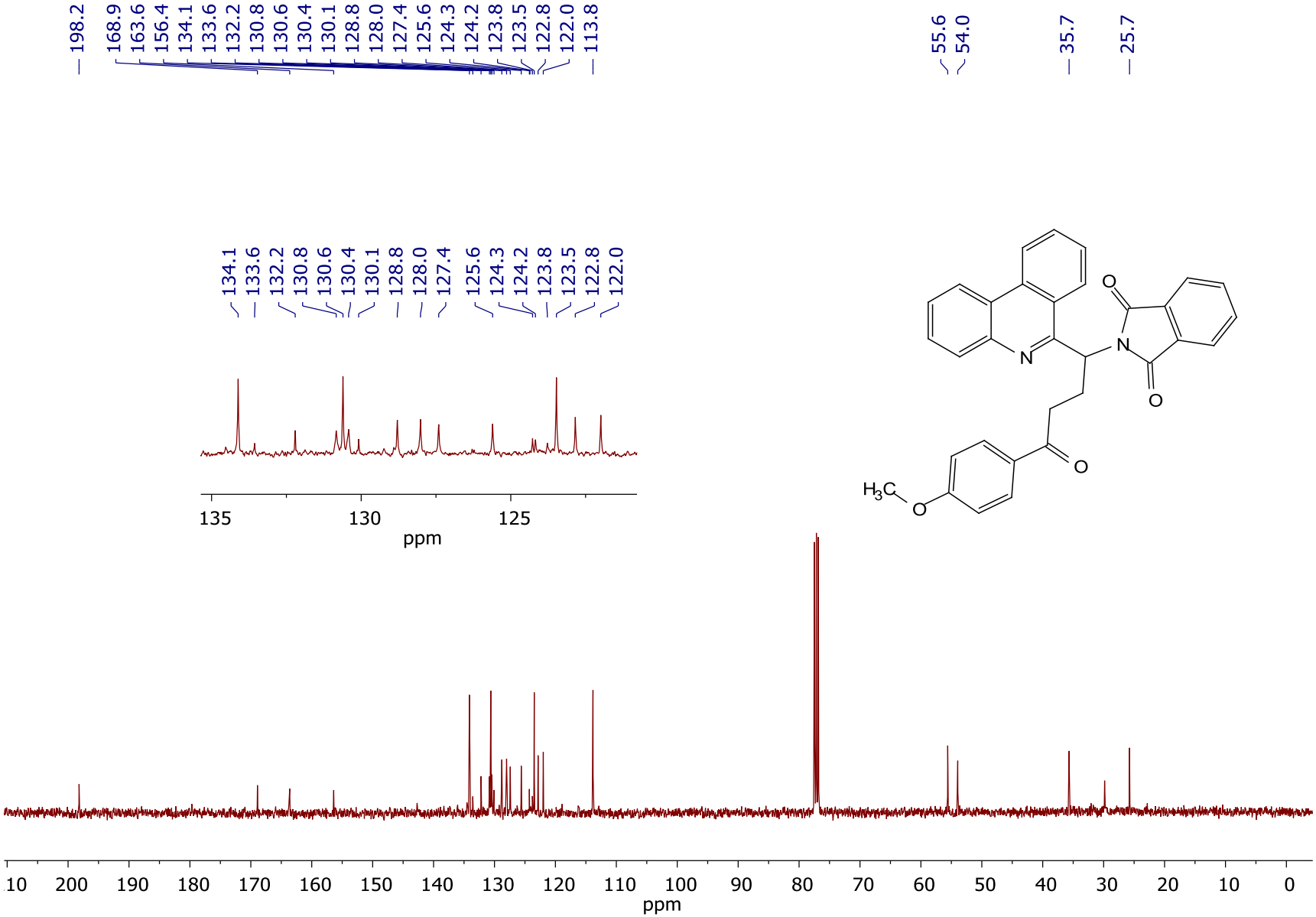
4-(1,3-dioxoisoindolin-2-yl)-4-(phenanthridin-6-yl)butanenitrile (4k):



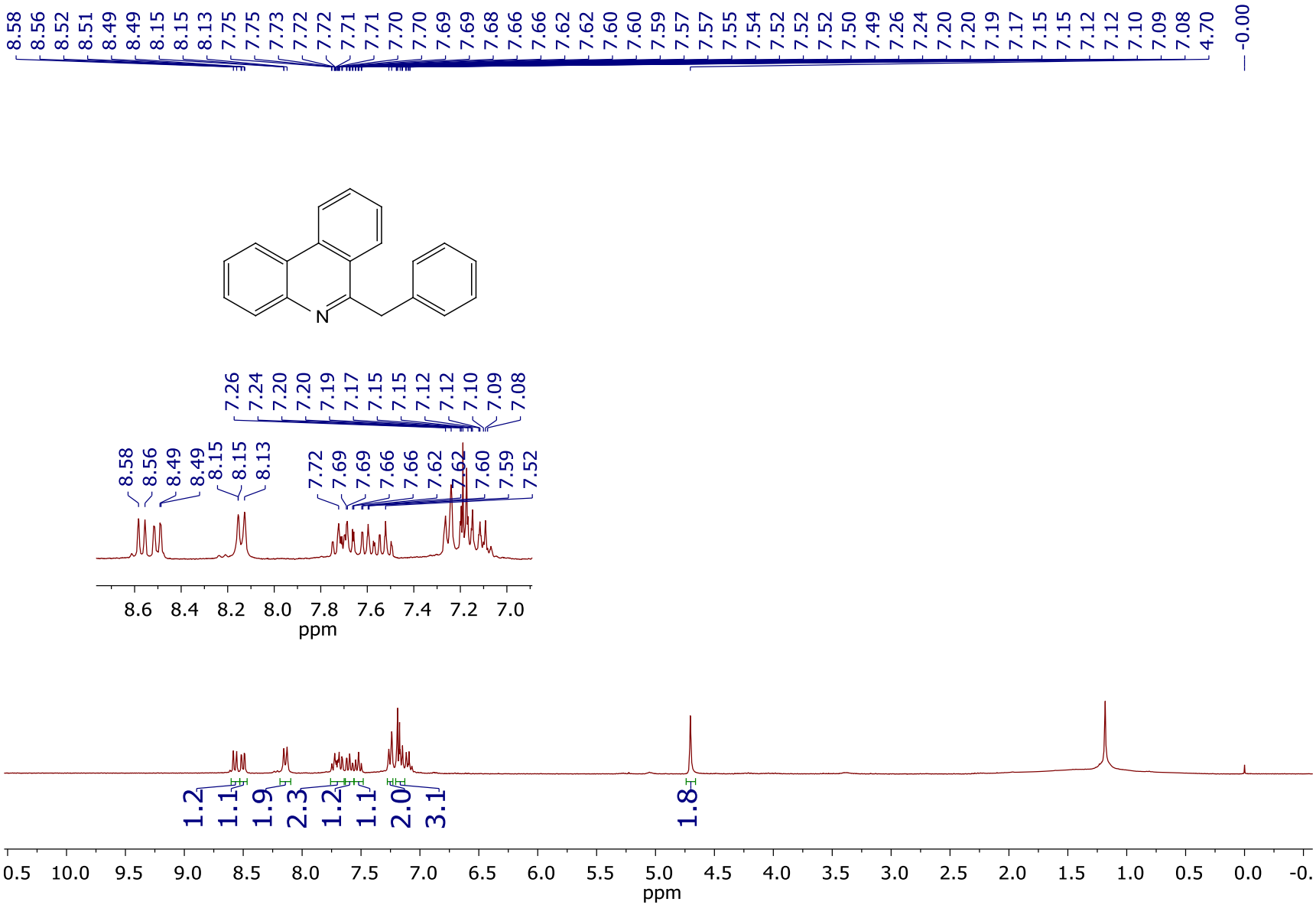

168.3
154.1
143.0
134.4
133.5
131.8
130.8
130.7
128.9
128.0
127.7
124.4
124.2
124.1
123.7
123.1
122.1
119.4
52.1
26.9
15.2
N
N
O
O
ppm
ppm

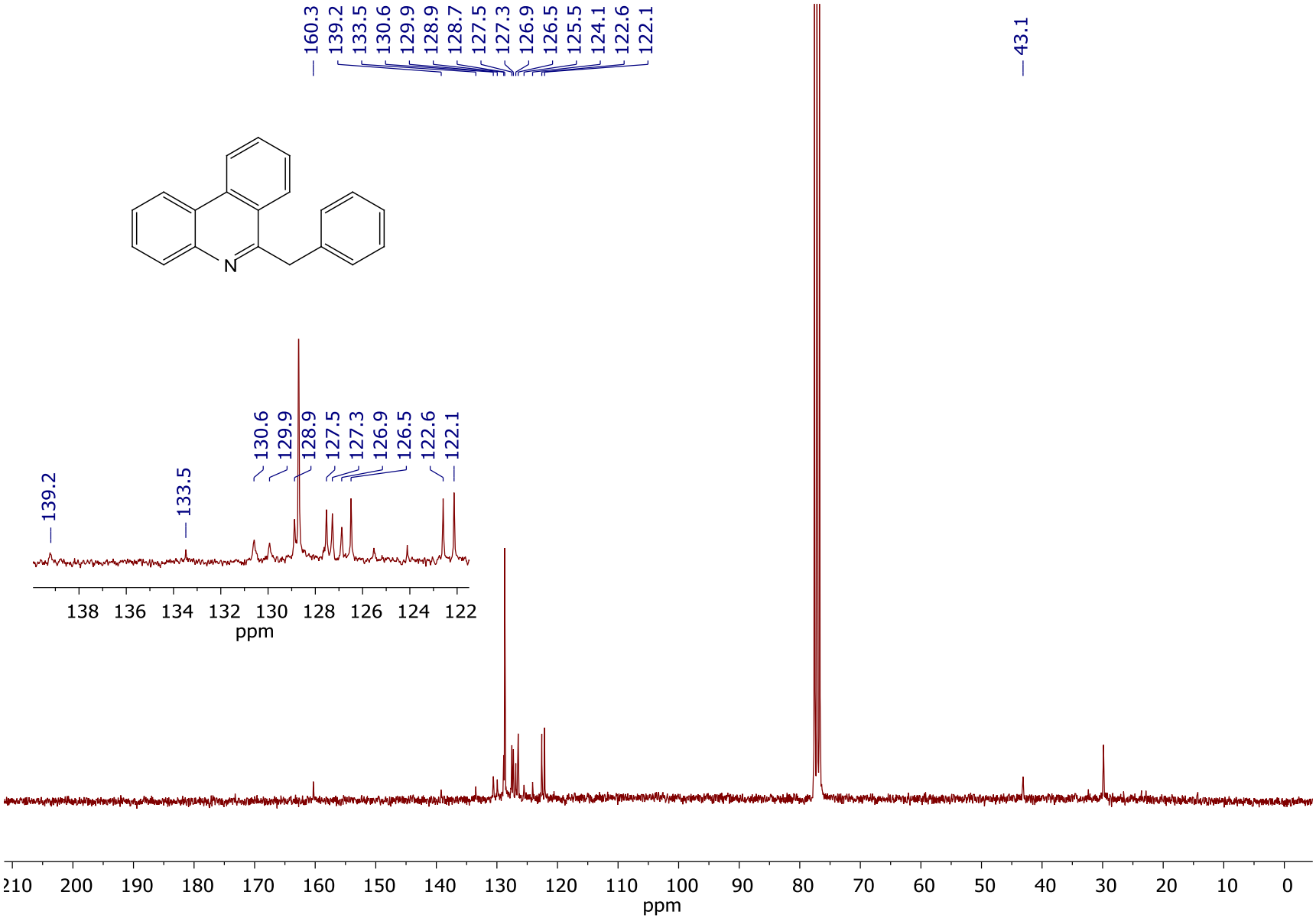
2-(4-(4-methoxyphenyl)-4-oxo-1-(phenanthridin-6-yl)butyl)isoindoline-1,3-dione (4l):



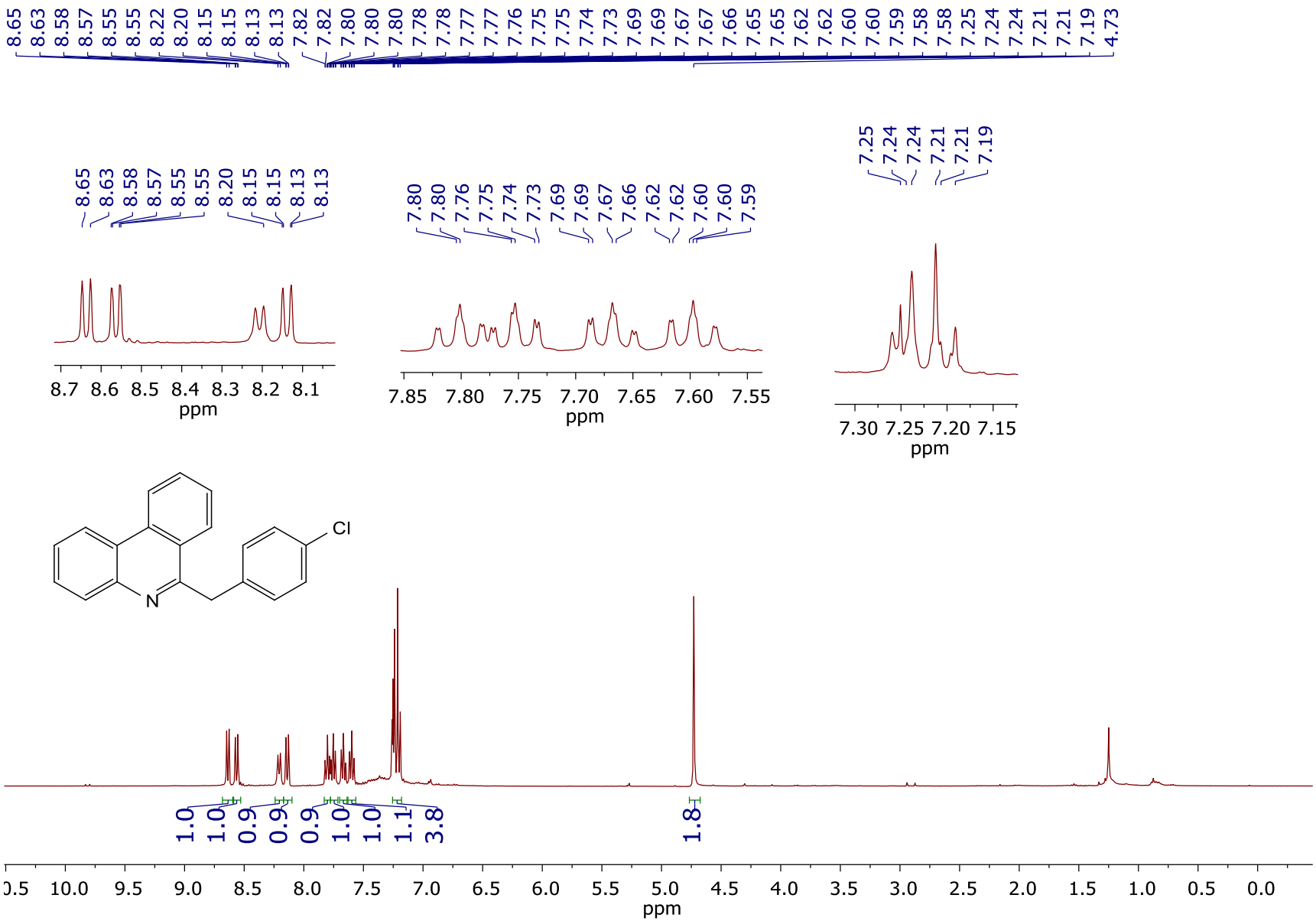

198.2
168.9
163.6
156.4
134.1
133.6
132.2
130.8
130.6
130.4
130.1
128.8
128.0
127.4
125.6
124.3
124.2
123.8
123.5
122.8
122.0
113.8
55.6
54.0
35.7
25.7
134.1
133.6
132.2
130.8
130.6
130.4
130.1
128.8
128.0
127.4
125.6
124.3
124.2
123.8
123.5
122.8
122.0
135
130
125
ppm
O
N
N
O
O
O
H3C
O
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

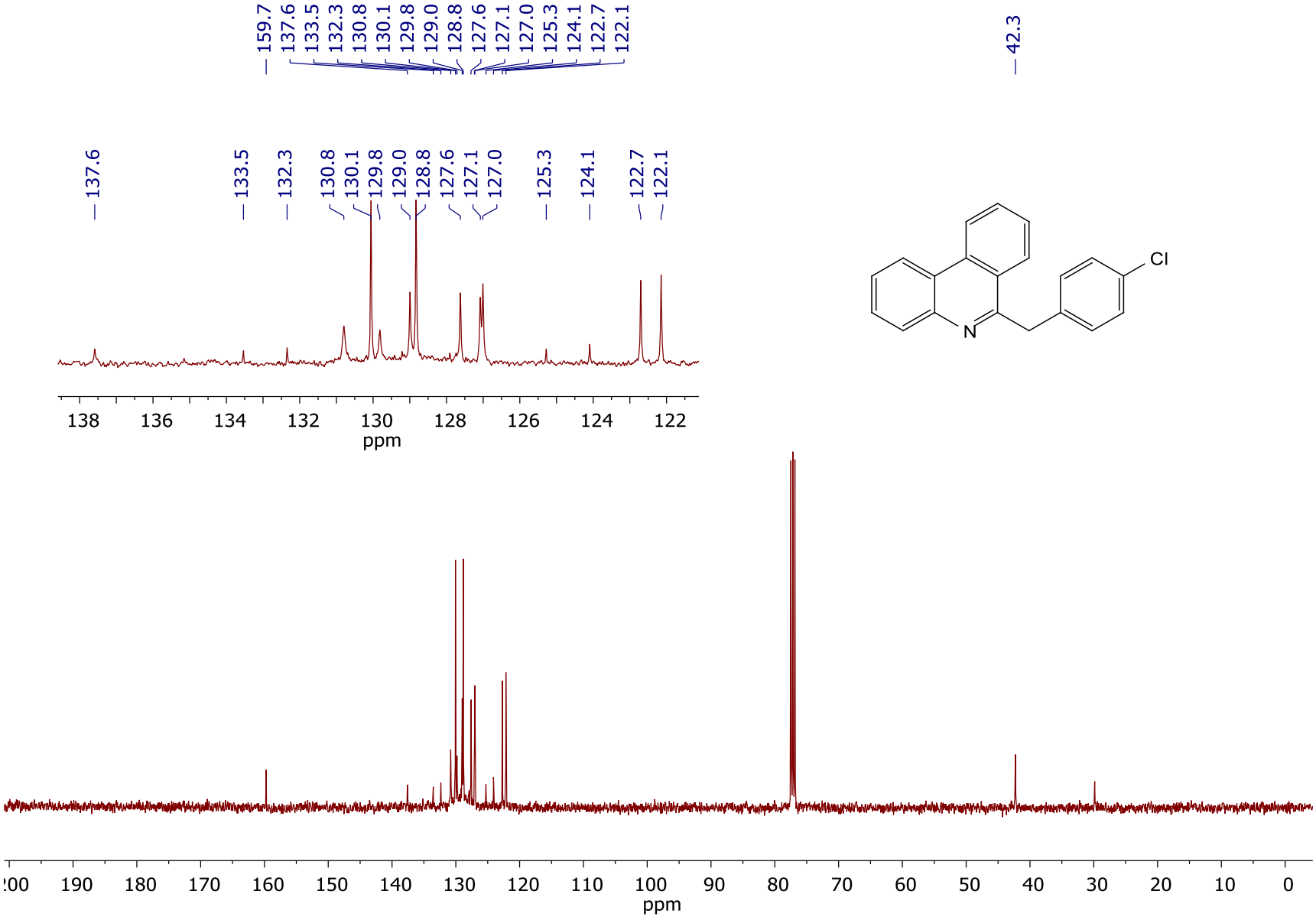
6-benzylphenanthridine (4m):



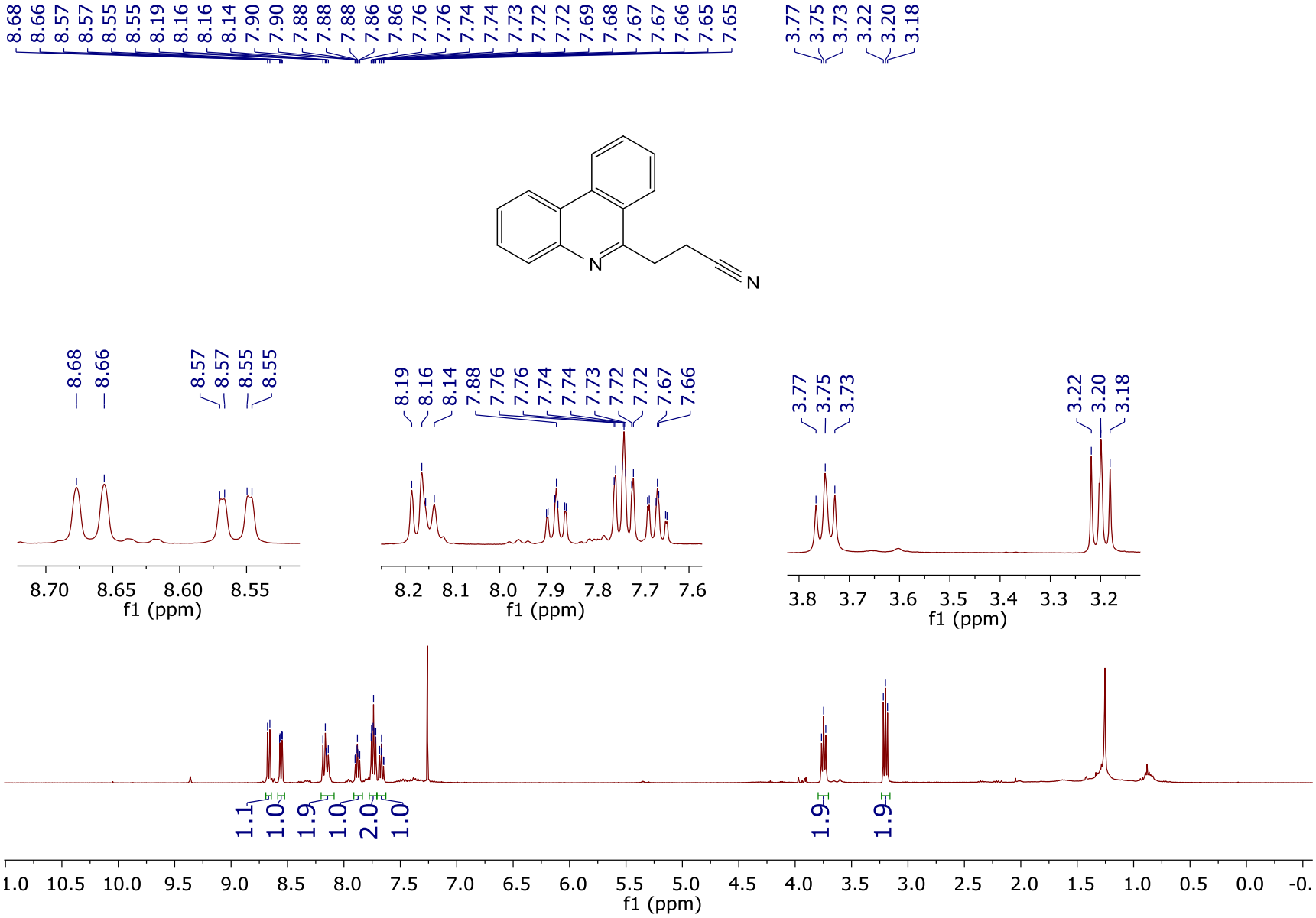

160.3
139.2
133.5
130.6
129.9
128.9
128.7
127.5
127.3
126.9
126.5
125.5
124.1
122.6
122.1
43.1
N
139.2
133.5
130.6
129.9
128.9
127.5
127.3
126.9
126.5
122.6
122.1
138 136 134 132 130 128 126 124 122
ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

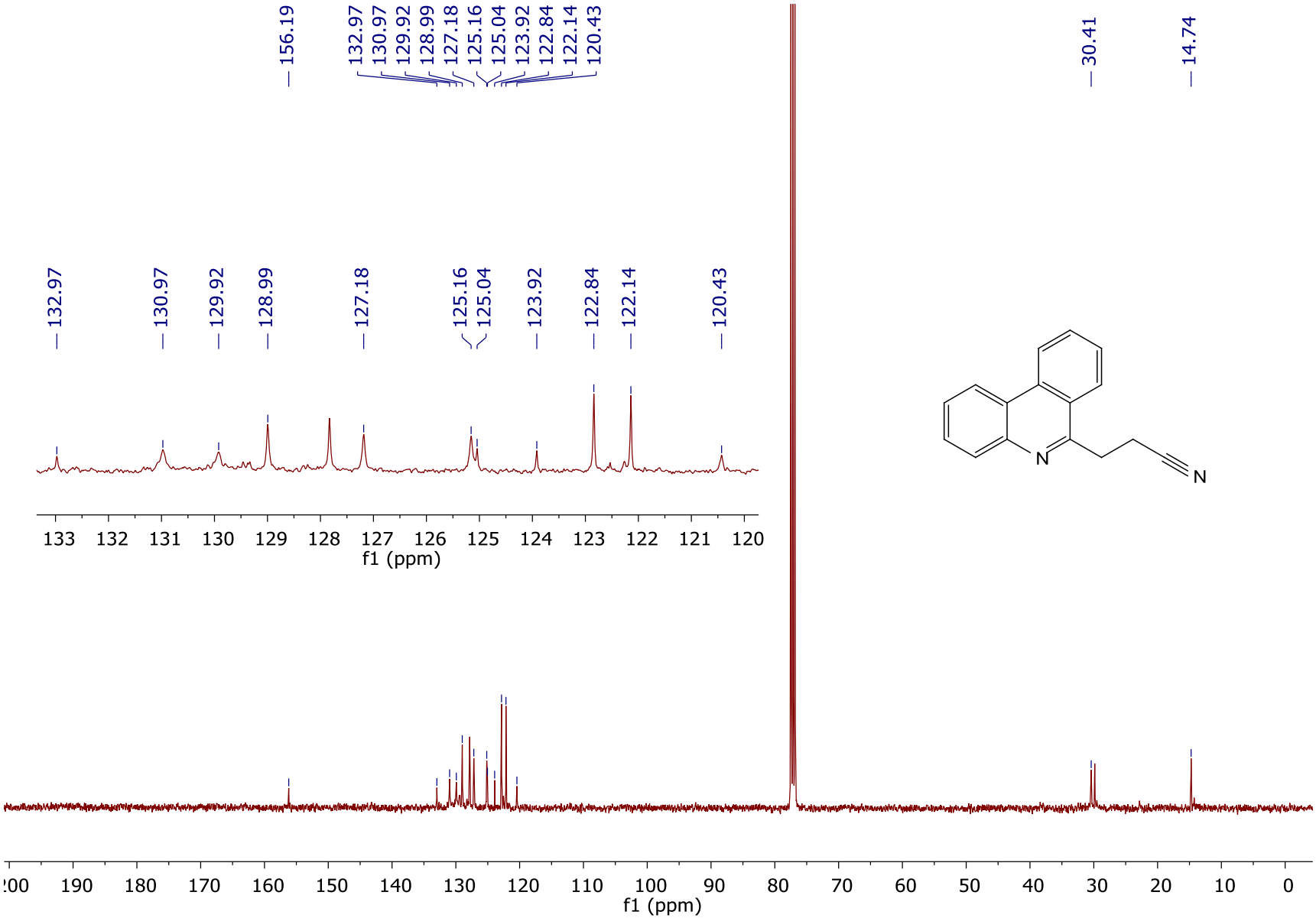
6-(4-chlorobenzyl)phenanthridine (4n):




159.7
137.6
133.5
132.3
130.8
130.1
129.8
129.0
128.8
127.6
127.1
127.0
125.3
124.1
122.7
122.1
42.3
Cl
N
ppm
ppm

3-(phenanthridin-6-yl)propanenitrile (4o):




156.19
132.97
130.97
129.92
128.99
127.18
125.16
125.04
123.92
122.84
122.14
120.43
30.41
14.74
f1 (ppm)
f1 (ppm)
N
N