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


# Supporting Information

## Silver CatalyzedDirect C-H Functionalization of Quinones with Dialkyl amides

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## 1. General Information:

All the reagents were purchased commercially and used without further purification. $^{1}H$ NMR and $^{13}C$ NMR were recorded with Bruker 400 MHz.$^{1}H$ NMR (400MHz) and $^{13}C$ NMR (100MHz) spectra were recorded in $CDCl_3$ with tetramethylsilane as the internal standard. Multiplicities are reported using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, sep = septet, br = broad resonance. All the NMR spectra were acquired at ambient temperature. Analytical thin layer chromatography (TLC) was performed using Silica Gel 60 Å $F_{254}$ pre-coated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and staining with $I_2$ on silica gel. High resolution mass spectra (HRMS) were recorded on Bruker Compass Data Analysis 4.1, HRMS-ESI Mass Spectrometer with Quadrupole Time of Flight (Q-TOF) Analyzer, Source Type ESI, in positive mode.

## 2. Starting material preparation (1g – 1i)

Arylated naphthoquinones were synthesized by following procedure reported in the literature.[1]To a stirred solution of naphthoquinone (1.0 equiv.) and a suitable phenol or methoxy benzene (2.0 equiv.) in DCM (4 mL), $K_2S_2O_8$ (3.0 equiv.) followed by which $FeCl_3$ (3.0 equiv.) were added in portions at room temperature. The progress of the reaction was monitored by TLC. Upon the complete consumption of starting materials, it was diluted with ethyl acetate and water. The organic phase was separated, extracted two or more times with ethyl acetate, dried over $Na_2SO_4$, filtered and concentrated. The crude product was purified by silica gel column chromatography using hexane/ethyl acetate as eluent to get the product.

**3. General procedure for amidoalkylation of quinones.** To1,4-naphthoquinone(**1a**, 0.25 mmol) taken in a round bottom flask, silver nitrate (10 mol%), TBHP (1.0 mmol) and DMA (1 ml) were added then allowed to stir at 80$^{o}$C, the progress of the reaction was monitored by TLC. After completion, the reaction mixture was allowed to cool down to ambient temperature and quenched with water, extracted with ethyl acetate then the organic layer was separated, dried overanhydrous $Na_2SO_4$ and concentrated under reduced pressure. The crude product was purified through silica gel column chromatography(eluent: Hexane/EtOAc).

**(Of note, because of the electronic nature of amides, most of the products exist as a mixture of two or three rotamers as observed in the NMR charts.)[2]**

**4. Characterization of synthesized compounds**

***N-((1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide(3a):***The reaction was carried out according to the general procedure A using **1a**(40mg, 0.25 mmol), $AgNO_3$(4.2 mg, 10 mol%),TBHP(129mg, 1.0 mmol),Conditions:80$^{o}$C, 6h. The title compound**3a**(46.75mg, 76%)was obtained as a brown gummy liquid after purification through a silica gel column chromatography(Hexane / EtOAc= 6:4); observed as two rotamers of 2:1 ratio in $^{1}$H NMR ($CDCl_3$, 400MHz): δ 8.06-8.12 (m, 2H), 7.74-7.80 (m, 2H), 6.72/6.65 (s, 1H), 4.54/4.49 (s, 2H), 3.10/2.99 (s, 3H), 2.20/2.11 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100MHz): δ 185.1, 184.8, 171.2, 145.5, 134.4, 134.1, 134.0, 133.9, 133.7, 132.2, 132.0, 126.5, 126.5, 126.2,49.4, 46.5, 37.1, 34.2, 21.7, 21.2; HRMS (ESI): exact mass calcd for $C_{14}H_{13}NO_3$ $[M + H]^+$, 244.0973; found: 244.0965.

***N-((3-bromo-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide(3b):***The reaction was carried out according to the general procedure A using **1b**(59 mg, 0.25 mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP(129 mg, 1.0 mmol). Conditions:80°C, 7h. The title compound **3b**(35mg, 44%) was obtained as a brownish yellowgummy liquidafter purification through a silica gel column chromatography(Hexane / EtOAc= 6:4); observed as two rotamers of 1.5:0.5 ratio in$^{1}$H NMR ($CDCl_3$, 400MHz): δ 8.12-8.16 (m, 2H), 7.73-7.80 (m, 2H), 4.75/4.65 (s,2H), 3.12/2.85 (s,3H), 2.43/2.07 (s,3H); $^{13}$C NMR ($CDCl_3$, 100MHz): δ 181.5, 181.3, 177.6, 177.2, 170.9, 146.4, 144.5, 139,7, 134.8, 134.4, 134.9, 134.0, 131.6, 131.0, 127.9, 127.6, 127.4, 127.3, 47.8, 37.5, 21.6; HRMS (ESI): exact mass calcd for $C_{14}H_{12}BrNO_3$:321.0001; found: 321.0005.

***N-((3-methoxy-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide(3c):***The reaction was carried out according to the general procedure A using **1c**(47mg, 0.25 mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP(129 mg, 1.0 mmol). Conditions:80°C, 11 h. The title compound **3c**(28mg, 41%) was obtained as a yellow gummy liquidafter purification through silica gel column chromatography(hexane / EtOAc= 6:4); observed as two rotamers of 1:1 ratio in$^1$H NMR ($CDCl_3$, 400MHz): δ 8.02-8.08 (m, 2H), 7.67-7.77 (m, 2H), 4.52/4.49 (s,2H), 4.23/4.20 (s,3H), 3.06/2.82 (s,3H), 2.35/2.06 (s,3H); $^{13}$C NMR ($CDCl_3$, 100MHz): δ185, 1, 184.5, 181.5, 181.2, 171.5, 170.8, 159.1, 158.9, 134.4, 134.0, 133.6, 133.4, 131.8, 131.5, 131.4, 128.9, 126.7, 126.5, 126.4, 126.3, 126.2, 96.1, 61.7, 61.5, 42.9, 41.2, 37.1, 32.4, 21.9,21.5. HRMS (ESI): exact mass calcd for $C_{15}H_{15}NO_4$ $[M + H]^+$, 274.1079; found: 274.1069.

***N-methyl-N-((3-((methyl-d3)thio)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)acetamide(3d):***The reaction was carried out according to the general procedure A using **1d**(73mg, 0.25 mmol), $AgNO_3$(4.2mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80°C, 15 h. The title compound **3d**(40.3 mg, 55%) was obtained as a yellow gummy liquidafter purification through a silica gel column chromatography (Hexane / EtOAc= 6:4); observed as two rotamers of 1.5/0.5 ratio in$^1$HNMR ($CDCl_3$, 400MHz): δ 8.03-8.08 (m, 2H), 7.68-7.76 (m, 2H), 4.71 (s,2H), 3.00/2.79 (s,3H), 2.38/2.05 (s,3H); $^{13}$C NMR ($CDCl_3$, 100MHz): 181.6, 181.3, 180.9, 171.5, 170.8, 150.8, 142.9, 141.3, 134.3, 133.9, 133.8, 133.5, 133.0, 132.0, 131.8, 127.0, 126.8, 126.7, 46.8, 44.7, 36.5, 31.9, 21.9, 21.6.

***N-methyl-N-((3-methyl-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)acetamide(3e):***The reaction was carried out according to the general procedure A using **1e**(43mg, 0.25 mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80°C, 6.5 h. The title compound **3e**(31 mg, 48%) was obtained as a yellow gummy liquidafter purification through a silica gel column chromatography (hexane / EtOAc= 6:4); observed as two rotamers of 9:1 ratio in $^1$H NMR ($CDCl_3$, 400MHz): δ 8.07-8.10 (m, 2H), 7.71-7.76 (m, 2H), 4.62/4.56 (s,2H), 3.03/2.79 (s,3H), 2.42/2.30 (s,3H), 2.09/1.96 (s,3H); $^{13}$C NMR($CDCl_3$, 100MHz):δ 185.2, 185.0, 170.9, 147.4, 141.0, 134.8, 133.9, 133.6, 132.1, 131.9, 127.7, 126.6, 126.4, 126.4, 126.1 42.6, 36.7, 21.8, 13.0.HRMS (ESI): exact mass calcd for $C_{15}H_{15}NO_3$[M + Na]$^+$, 280.0950; found: 280.0945.

***N-((8-hydroxy-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide and N-((5-hydroxy-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide(3fa and 3fb):***The reaction was carried out according to the general procedure A using **1f**(43 mg, 0.25mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80°C, 8.5 h. The title compound **3f**(42mg, 66%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (hexane / EtOAc= 4:6); in $^1$H NMR ($CDCl_3$, 400MHz): δ 11.9-11.8 (s, 1H), 7.61-7.67 (m, 2H), 7.28-7.32 (m,1H), 6.63-6.69 (m,1H),4.48-4.52 (s,2H), 3.10 (s, 2.6H), 3.11 (s, 0.8H), 2.20 (s, 2.2H), 2.10 (s, 0.8H); $^{13}$C NMR($CDCl_3$, 100MHz):δ 190.3, 190.1, 171.3, 161.6, 161.4, 146.9, 145.5, 136.7, 136.4, 134.7, 133.8, 132.0, 124.6, 124.4, 119.4, 119.3, 119.0, 46.6, 46.1, 37.2, 21.7. The regio isomeric ratio of two products of ***3fa*** and ***3fb*** was found to be in 3.3 : 1 ratio.

***N-methyl-N-((3-(methylthio)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)acetamide(3g):***The reaction was carried out according to the general procedure A using **1g**(51 mg, 0.25mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80°C, 8 h. The title compound **3g**(37 mg, 51%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (hexane / EtOAc= 6:4); observed as two rotamers of 1.5:0.5 ratio in $^1$H NMR ($CDCl_3$, 400MHz): δ 8.04-8.09 (m, 2H), 7.69-7.75 (m, 2H), 4.72 (s,2H), 3.01/2.81 (s,3H), 2.72/2.69 (s,3H), 2.07 (s,3H); $^{13}$C NMR($CDCl_3$, 100MHz):δ 182.2, 180.9, 170.9, 158.7, 152.3, 150.8, 142.9, 141.3, 134.3, 133.9, 133.8, 133.5, 133.0, 132.0, 131.8, 130.8, 128.2, 127.8, 127.0, 126.8, 124.6, 123.4, 46.8, 44.7, 36.6, 31.9, 21.6, 18.1. HRMS (ESI): exact mass calcd for $C_{15}H_{15}NO_3S[M + H]^+$: 290.0851; found: 290.0842.

***N-((3-(4-hydroxyphenyl)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide(3h):***The reaction was carried out according to the general procedure A using **1h**(63mg, 0.25 mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80°C, 20 h. The title compound **3h** (57mg, 67%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (hexane / EtOAc= 6:4); observed as two rotamers of 1.8:1.7 ratio in $^1$H NMR ($CDCl_3$, 400MHz):δ 8.10-8.12 (m, 2H), 7.74-7.80 (m, 2H), 7.00-7.08 (m, 2H), 6.80-6.90 (m, 2H), 4.52/4.49 (s,2H), 2.98/2.69 (s,3H), 1.94/1.89 (s,3H); $^{13}$C

NMR (DMSO, 100MHz): δ184.8, 184.4, 184.0, 183.9, 169.8, 169.2, 157.9, 157.6, 148.5, 146.1, 141.8, 139.6, 134.2, 134.1, 131.8, 131.7, 131.7, 131.1, 126.0, 125.8, 123.4, 122.7, 114.7, 114.5, 46.3, 43.88, 38.8, 37.40, 21.4, 21.2.

***N-((3-(4-hydroxy-3-methylphenyl)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide(3i):***The reaction was carried out according to the general procedure A using **1i**(66 mg, 0.25 mmol), $AgNO_3$ (4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions: 80°C, 18 h. The title compound **3i**(54 mg, 62%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc= 1:1);observed as two rotamers of 2.1/1.0 ratio in $^1$H NMR ($CDCl_3$/DMSO, 400MHz): δ 8.09-8.14 (m, 2H), 7.73 -7.80 (m, 2H), 6.91-6.95 (m, 2H), 6.69-6.82 (m, 1H), 4.52/4.48 (s, 2H), 2.95/2.69 (s, 3H), 2.26/2.22 (s,3H), 2.01/1.90 (s, 3H); $^{13}$C NMR ($CDCl_3$/DMSO, 100MHz): 185.6, 184.9, 184.9, 184.6, 171.4, 155.9, 155.7, 148.0, 140.9, 140.0, 134.1, 133.8, 132.1, 132.1, 128.4, 128.1, 126.9, 126.8, 126.6, 126.2, 124.8, 123.6, 123.7, 123.1, 115.1, 115.0, 46.1, 45.9, 37.6, 21.4, 21.2, 16.0.

***N-((3-(3-allyl-4-hydroxyphenyl)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide (3j):***The reaction was carried out according to the general procedure A using **1j**(73mg, 0.25 mmol), $AgNO_3$ (4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions: 80 °C, 15 h. The title compound **3j**(54 mg, 57%) was obtained as aorangecolour gummy liquid after purification through a silica gel column chromatography (hexane / EtOAc= 1:1); observed as three rotamers in $^1$H NMR ($CDCl_3$, 400MHz): δ8.10-8.12 (m, 2H), 7.73-7.79 (m, 2H), 6.71 - 7.05 (m, 3H), 6.00-6.06 (m, 1H), 5.05-5.09 (m, 1H), 4.58/4.52/4.48 (s, 2H), 3.73-3.83 (m, 1H), 3.38-3.43 (m, 2H), 3.03/2.96/2.90 (s, 3H), 2.10/2.04/1.89 (s, 3H), $^{13}$C NMR ($CDCl_3$, 100MHz): δ 185.7, 185.5, 184.8, 184.5, 172.0, 171.6, 156.2, 155.6, 148.0, 147.8, 140.8, 140.6, 136.6, 136.1, 134.1, 133.8, 132.6, 132.1 130.8, 130.1, 128.8, 126.8, 126.2, 123.7, 123.3, 116.1, 115.6, 60.2, 49.0, 45.7, 37.9, 37.0, 34.4, 31.9, 31.6, 22.7, 21.4, 14.1.; HRMS (ESI): exact mass calcd for $C_{23}H_{21}NO_4[M + H]^+$: 376.1549; found: 376.1540.

***N-((3-(4-methoxyphenyl)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-Nmethylacetamide(3k):***The reaction was carried out according to the general procedure A using **1k**(66 mg, 0.25 mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80

°C, 15 h. The title compound **3k**(54 mg, 62%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc = 6:4); observed as two rotamers of 1.7:1 ratio in $^{1}$H NMR ($CDCl_3$, 400MHz): δ 8.08-8.13 (m, 2H), 7.72-7.79 (m, 2H), 7.11-7.19 (m,2H), 6.98-7.03 (m,2H), 4.46 (s,2H), 3.87/3.85 (s,3H), 2.81/2.65 (s,3H), 2.12/1.86 (m,3H); $^{13}$C NMR ($CDCl_3$, 100MHz): 184.6, 184.3, 171.2, 170. 8, 160.5, 160.0, 146.9, 141.7, 140.2, 134.2, 134.1, 133.8, 132.5, 132.2, 132.0, 131.9, 131.0, 130.7, 128.9, 126.9, 126.7, 126.5, 126.3, 124.8, 123.8, 115.2, 114.0, 113.6, 55.3, 46.6, 45.6, 37.3, 32.8, 21.6; HRMS (ESI): exact mass calcd for $C_{21}H_{19}NO_4$[M + H]$^+$, 350.1392; found: 350.1380.

***N-((3-(3,4-dimethoxyphenyl)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide (3l):***The reaction was carried out according to the general procedure A using **1l**(74 mg 0.25 mmol), $AgNO_3$ (4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions: 80°C, 12.5 h. The title compound **3l**(68 mg, 71%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc = 1:1); observed as two rotamers of 1.9:1 ratio in $^{1}$H NMR ($CDCl_3$, 400MHz): δ 8.08-8.12 (m, 2H), 7.72-7.79 (m, 2H), 6.94-6.98 (m, 1H), 6.69-6.81 (m, 2H), 4.47/4.46 (s,2H), 3.93/3.91/3.88 (s, 6H), 2.80/2.67 (s, 3H), 2.11/1.85 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100MHz): 184.6, 184.3, 171.3, 170.6, 150.0, 149.5, 149.0, 148.5, 146.6, 142.1, 140.5, 134.2, 134.2, 133.8, 132.0, 132.0, 131.9, 126.9, 126.7, 126.6, 126.3,125.1, 124.1, 122.3, 122.0, 112.6, 111.0, 110.7, 56.0, 55.9, 46.6, 45.8, 37.4, 32.1, 21.6, 21.4; HRMS (ESI): exact mass calcd for $C14H_{13}NO_3$ [M + H]$^+$, 243.0895; found-244.0965.

***N-((1,4-dioxo-3-(2,3,4-trimethoxyphenyl)-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide(3m):***The reaction was carried out according to the general procedure Ausing **1m** (81 mg, 0.25 mmol), $AgNO_3$ (4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions: 80°C, 22 h. The title compound **3m**(Trace) was obtained as a yellowcolour gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc = 1:1); observed as two rotamers of 2.3:1.6 ratio in$^{1}$H NMR($CDCl_3$, 400MHz): δ 8.10-8.15 (m, 2H), 7.73-7.79 (m, 2H), 6.84-6.87 (m, 1H), 6.74-6.76 (m, 1H), 4.47/4.39/4.38/4.36 (s, 2H), 3.92/3.90/3.89 (s, 6H), 3.79/3.77 (s, 3H), 2.81/2.66 (s, 3H), 2.11/1.89 (s, 3H).

***N-((3-(4-(dimethylamino)phenyl)-1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylacetamide (3n):*** The reaction was carried out according to the general procedure A using **1n** (70 mg, 0.25mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80 °C, 10.5 h. The title compound **3n**(50.2 mg, 55%) was obtained as a violet colour gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc = 1:1); observed as two rotamers of 2.0:1.9 ratio in $^1$H NMR ($CDCl_3$, 400MHz): δ 8.09 - 8.14 (m, 2H), 7.71-7.77 (m, 2H), 7.06-7.14 (m, 2H), 6.75-6.78 (m, 2H), 4.56/4.52 (s, 2H), 3.04/3.00 (s, 6H), 2.80/2.67 (s, 3H), 2.16/1.87 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100MHz): δ 185.3, 185.1, 184.9, 184.6, 171.4, 170.6, 151.0, 150.8, 149.3, 147.6, 140.5, 139.1, 134.0, 133.9, 133.6, 133.5, 132.3, 132.2, 132.1, 132.1, 131.2, 130.9, 126.8, 126.7, 126.3, 126.1, 119.9, 118.7, 111.5, 111.4, 46.9, 45.3, 40.3, 40.2, 36.9, 32.4, 21.7, 21.5.HRMS (ESI): exact mass calcd for $C_{22}H_{22}N_2O_3$ $[M + H]^+$, 363.1709; found: 363.1700.

***N-((2,4-dimethyl-3,6-dioxocyclohexa-1,4-dien-1-yl)methyl)-N-methylacetamide(3q):***The reaction was carried out according to the general procedure A using **1q**(34mg, mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80°C, 24 h. The title compound **3q**(30 mg, 54%) was obtained as a yellow colour gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc = 1:1); observed as two rotamers of 10:1 ratio in$^1$H NMR ($CDCl_3$, 400MHz): δ6.60/6.58 (s, 1H), 4.43/4.39 (s, 2H), 2.98/2.75 (s, 3H), 2.35/2.15 (s, 3H), 2.07/2.05 (s, 6H); $^{13}$C NMR ($CDCl_3$, 100MHz): δ 188.1, 187.5, 172.9, 170.9, 150.0, 145.8, 144.8, 138.5, 133.1, 125.1, 123.6, 119.1, 116.5, 42.3, 36.7, 21.8, 15.9, 12.5; HRMS (ESI): exact mass calcd for $C_{12}H_{15}NO_3$ $[M + Na]^+$, 244.0950; found: 244.0948.

***N-((4-(tert-butyl)-3,6-dioxocyclohexa-1,4-dien-1-yl)methyl)-N-methylacetamide and N-((5-(tert-butyl)-3,6-dioxocyclohexa-1,4-dien-1-yl)methyl)-N-methylacetamide (3ra and 3rb):*** The reaction was carried out according to the general procedure A using **1r** (41mg, 0.25 mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (104 mg, 1.0 mmol), Conditions:80°C, 4 h. The title compound **3r** (37.5 mg, 60%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc = 1:1); rigioselectivity 3. 1 : 1.$^1$H NMR ($CDCl_3$, 400MHz): δ6.50-6.58 (m, 1H), 6.27-6.35 (m, 1H), 4.32/4.29 (s,2H), 3.01/2.97 (s, 2.1H), 2.90 (s, 0.68H) 2.14/2.13/2.03(s,3H), 1.24/1.23 (s, 9H); $^{13}$C NMR ($CDCl_3$, 100MHz): δ 188.3,

187.5, 171.3, 156.3, 141.6, 133.6, 133.5, 131.6, 131.6, 129.8, 45.5, 37.1, 35.1, 36.7, 21.6:HRMS (ESI): exact mass calcd for $C_{14}H_{19}NO_3$ $[M + H]^+$, 250.1443; found: 250.1434. The regio isomeric ratio of two products of ***3ra*** and ***3rb*** was found to be in 3.1 : 1 ratio

***N-((2,4 -dimethoxy-3,6-dioxocyclohexa-1,4-dien-1-yl)methyl)-N-methylacetamide(3s):***The reaction was carried out according to the general procedure A using **1s**(63 mg, 0.25 mmol), $AgNO_3$ (4.2 mg, 10 mol%), TBHP (104 mg, 1.0 mmol), Conditions: 80°C, 5 h. The title compound **3s**(Trace) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (Hexane/EtOAc= 1:1); observed as two rotamers of 2.4:2.3 ratio in $^1$H NMR ($CDCl_3$, 400MHz): δ 5.87/5.83 (m, 1H), 4.36/4.34 (s, 2H), 4.08/4.06 (s, 3H), 3.82/3.80 (s,3H), 3.04/2.80 (s,3H), 2.29/2.05 (s, 3H); HRMS (ESI): exact mass calcd for $C_{12}H_{15}NO_5$, 253.0950; found: 253.0956.

***N-((1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)-N-methylformamide(3u):***The reaction was carried out according to the general procedure A using **1a** (40mg, 0.25mmol), $AgNO_3$(4.2 mg, 10 mol%), TBHP (104 mg, 1.0 mmol),Conditions:80°C, 6h. The title compound **3t**(27.5mg, 47%) was obtained as a browngummy liquid after purification through a silica gel column chromatography (Hexane/EtOAc = 1:1); observed as three rotamers in $^1$H NMR ($CDCl_3$, 400MHz): δ 8.23 (s, 1H), 8.07-8.11 (m, 2H), 7.75-7.80 (m, 2H), 6.77/6.69 (s, 1H), 4.49/4.40 (s, 2H), 3.15/2.91 (s, 3H), $^{13}$C NMR ($CDCl_3$, 100MHz): δ 184.9, 184.6, 184.3, 163.6, 163.0, 144.7, 144.3, 135.1, 134.8, 134.2, 134.2, 134.0, 134.0, 132.0, 132.0, 126.9, 126.7, 126.5, 126.4, 47.9, 43.1, 38.4, 35.2, 34.7, 30.2.

***N-((1,4-dioxo-1,4-dihydronaphthalen-2-yl)methyl)acetamide (3v):***The reaction was carried out according to the general procedure A using **1a** (40 mg, 0.25 mmol), $AgNO_3$ (4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions:80 °C, 16 h. The title compound **3w** (16.5 mg, 60%) was obtained as a yellow gummy liquid after purification through a silica gel column chromatography (Hexane / EtOAc = 1:1); $^1$H NMR ($CDCl_3$, 400MHz): δ 8.03 – 8.07 (m, 2H), 7.72 – 7.74 (m, 2H), 6.86 (s, 1H), 6.13 (s,1H), 4.36/4.35 (s,2H), 2.04 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100MHz): δ 185.3, 184.9, 170.4, 146.2, 134.7, 134.1, 133.8, 132.0, 131.1, 126. 4, 126.3, 38.7, 26.3, 23.2.

***2-(1-methyl-5-oxopyrrolidin-2-yl)naphthalene-1,4-dione & 2-((2-oxopyrrolidin-1-yl)methyl)naphthalene-1,4-dione (3:1, 3w-3w'):***The reaction was carried out according to the general procedure A using **1a** (40mg, 0.25 mmol), $AgNO_3$ (4.2 mg, 10 mol%), TBHP (129 mg, 1.0 mmol), Conditions: 80°C, 10 h. The title compound **3v-3v'** (43.2 mg, 67%) was obtained as a brown gummy liquid after purification through a silica gel column chromatography. This product could not be isolated in a pure form, being contaminated with 3a'. (Hexane / EtOAc=1:1); $^1$H NMR ($CDCl_3$, 400MHz): δ 8.01 – 8.06 (m, 2H), 7.68 – 7.75 (m, 2H), 6.57 (s, 1H), 4.80- 4.82 (m,1H), 4.37 (s, 1H), 2.79 (s,3H), 2.37 – 2.55 (m,4H), 2.09 (s, 1H), 1.81 – 1.82 (m, 1H),; $^{13}$C NMR ($CDCl_3$, 100MHz): δ 184.9, 184.7, 184.7, 184.5, 175.8, 148.7, 145.0, 134.4, 134.1, 134.0, 133.9, 132.7, 132.1, 132.0, 131.9, 129.5, 126.6, 126.5, 126.4, 126.3, 58.3, 47.9, 41.4, 30.5, 29.0, 28.8, 25.8, 18.1.HRMS (ESI): exact mass calcd for $C_{15}H_{14}NO_3$[M + H]+, 256.0973; found-256.0960.

***N-((10-chloro-5-oxo-5H-benzo[a]phenothiazin-6-yl)methyl)-N-methylacetamide(5):***The reaction was carried out according to the general procedure A using **3b**(30 mg, 0.093 mmol), **4** (15 mg, 0.093 mmol), $Na_2CO_3$(10 mg, 0.093 mmol), Conditions:RT, 40 min. The title compound **6**(27.4 mg,77%) was obtained as a red solid after purification through a silica gel column chromatography (Hexane / EtOAc = 6:4); mp 218-222$^o$C, observed as two rotamers of 1.3:0.2 ratio in$^1$H NMR ($CDCl_3$, 400MHz): δ 8.85-8.86 (m, 1H), 8.31-8.33 (m, 1H), 7.95 (s, 1H), 7.77-7.80 (m, 2H), 7.39-7.50 (m, 2H), 4.84/4.70 (s, 2H), 2.94/2.76 (s,3H), 2.20/2.14(s,3H); $^{13}$C NMR ($CDCl_3$, 100MHz): 179.6, 171.2, 145.3, 139.0, 136.5, 134.3, 133.3, 132.4, 132.0, 131.8, 129.9, 126.3, 126.2, 125.9, 125.8, 122.4, 42.4, 35.4, 21.8.HRMS (ESI): exact mass calcd for $C_{20}H_{15}ClN_2O_2S$ $[M + H]^+$, 383.0621; found: 383.0613.

**5. Molecular docking studies of compound 5b with AChE target (PDB ID: 1B41)**

The docking analyses were performed using Glide tool of Schrödinger-Maestro (ver10.7) package (Schrödinger, LLC, New York, NY, 2013). The compound 5a was sketched by using Chemdraw 12.0 software and converted to .mol file format with the standard settings and processed with default parameters of Ligprep tool from Schrodinger,[3] described by Greenwood et al., 2010. In this study, the compound 5 was docked with active site of AChE (PDB ID-1B41). The 3D structure of proteins were downloaded from the RSCB- Protein Data Bank (PDB)

website and prepared for the docking by removing of all water molecules. For grid generation, the hydrogen's were added, bond orders were assigned and water molecules beyond 5Å from the hetero groups were deleted in the imported protein and sitemap tool was used to locate the binding sites. For protein-ligand docking, the formation of H-bond between the ligand and the residues of the active site and its length along with Glide XP score,[4] were recorded with the help of Glide-Ligand docking tool of Maestro in cluster node of High-Performance Computing Facility (HPC).

**Table-1** Binding interactions of the protein-ligand complexes.

| Molecule | Glide XP Score (Kcal/mol) | Glide emodel score | No.of hydrogen bonds | Hydrogen bond length (Å) | Interacting amino acid residues |
|---|---|---|---|---|---|
| **5** | -8.09 | -59.20 | 2 | 2.10<br>2.24 | PHE-295<br>ARG-296 |

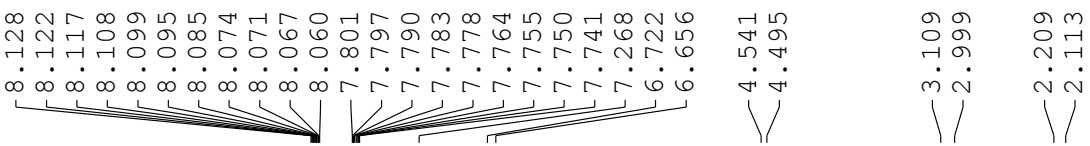
## Reference


(1) T. P. A. Krishna, P. Sakthivel, and A. Ilangovan**,** *Org. Chem. Front.***,**2019, ***6***, 3244-3251.

(2) C. Dai, F. Meschini, J. M. R. Narayanam, C. R. J. Stephenson. *J. Org. Chem.*2012, *77*, 4425−4431.

(3) Greenwood J. R, Calkins D, Sullivan A. P, Shelley J.C., J. Comput. Aided Mol. Des., 2010,24,591-604.

(4) Friesner R. A, Banks, J. L, Murphy R. B, Halgren T. A, Klicic J. J, Mainz D. T, Repasky M. P, Knoll E. H, Shelley M, Perry J. K. J. Med. Chem. 2004, 47, 1739-1749.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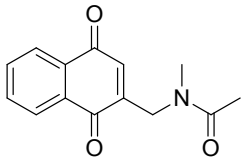
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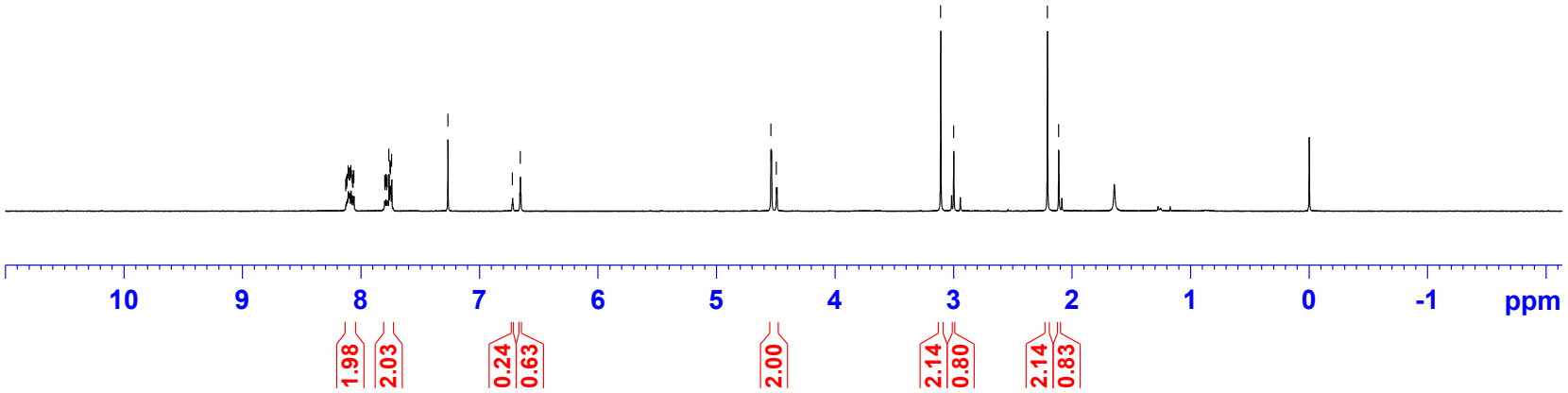
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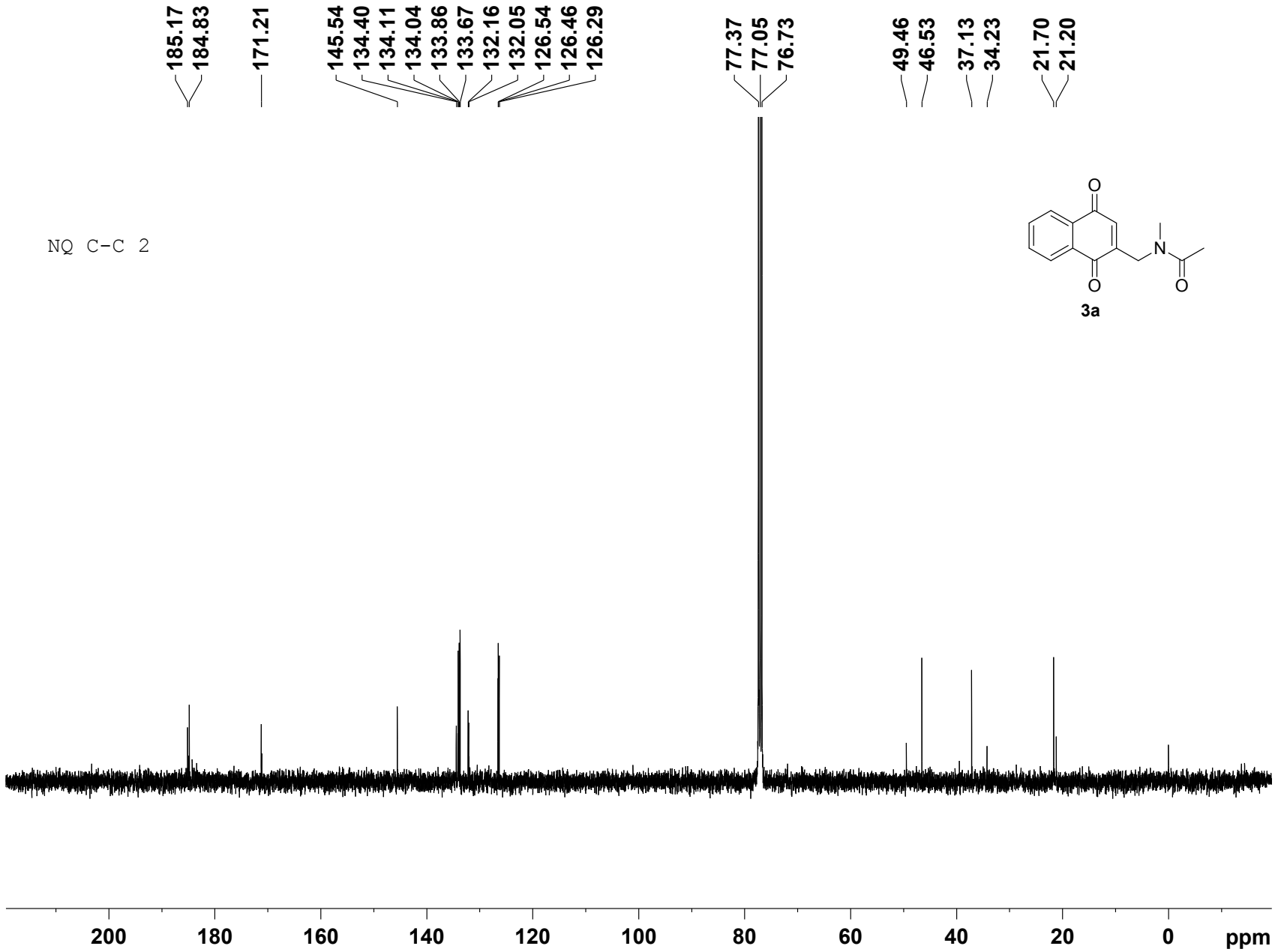
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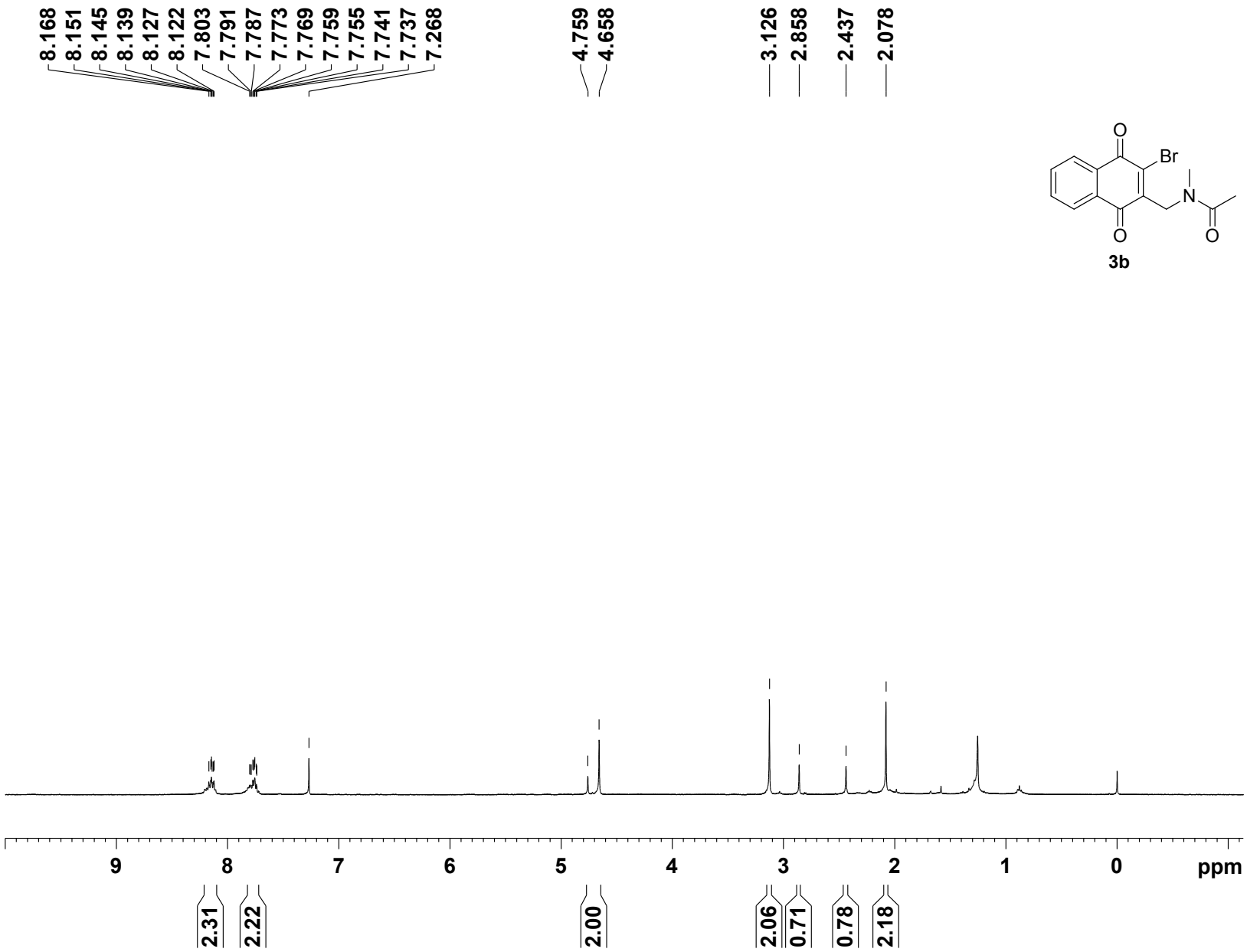
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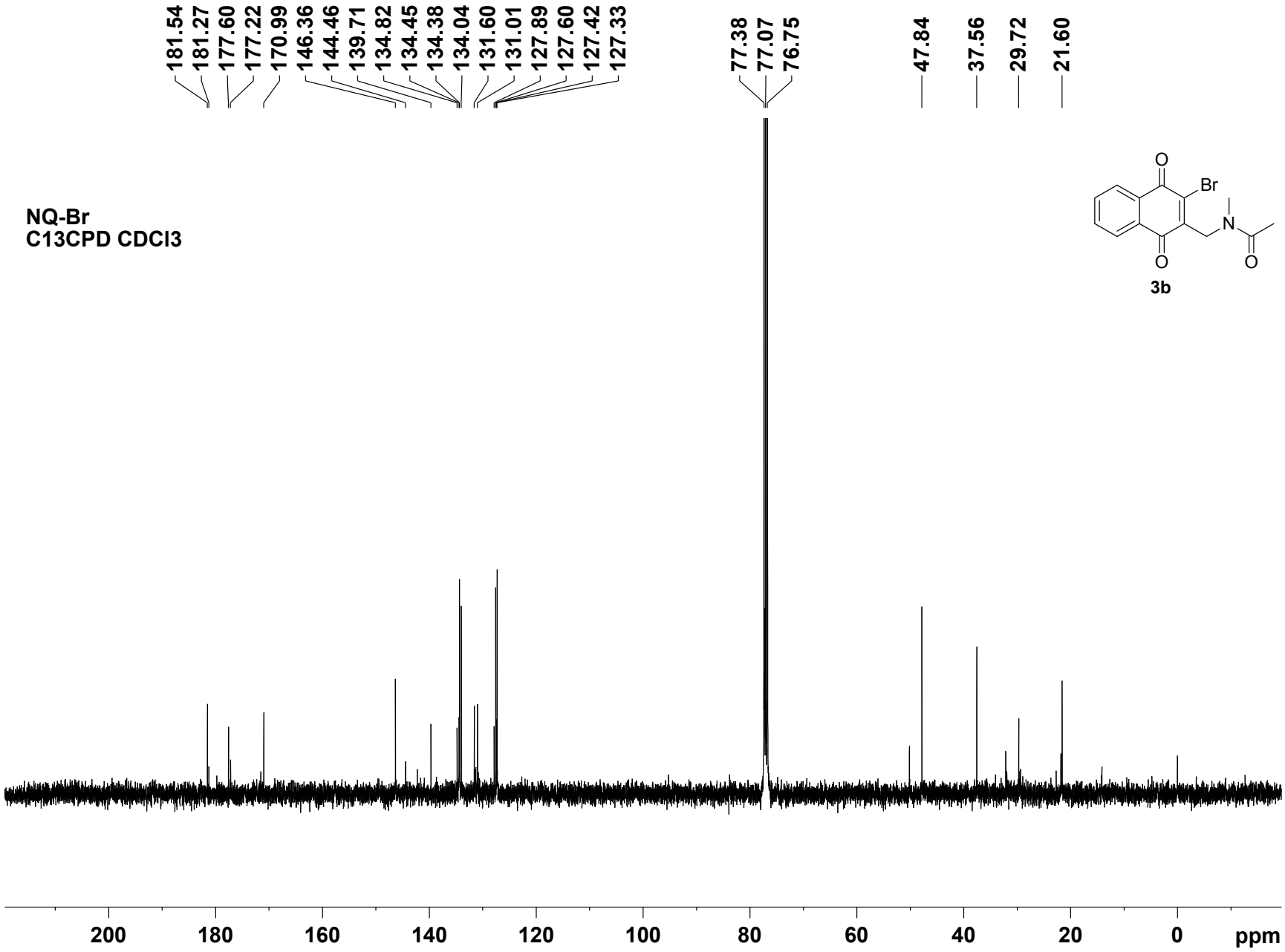
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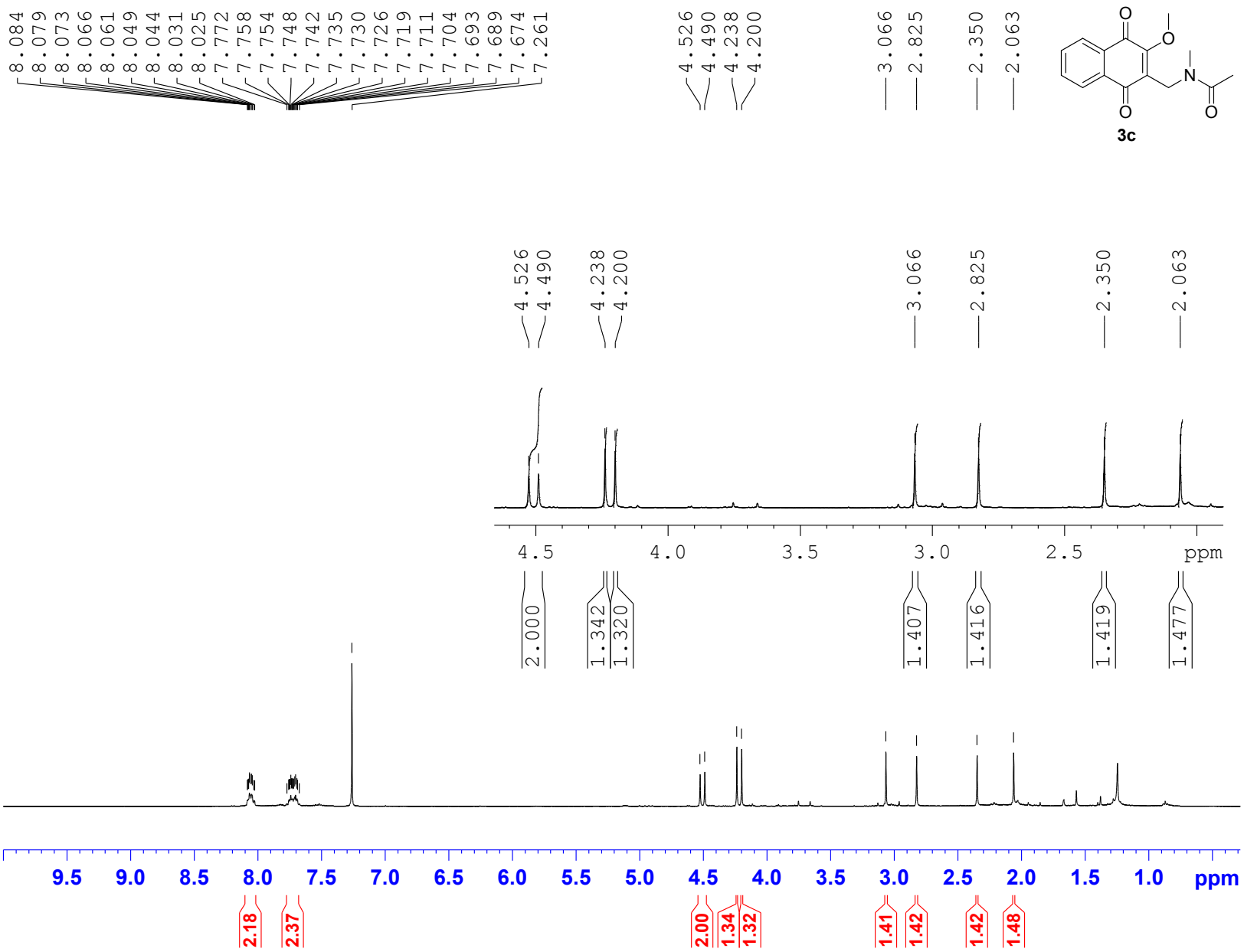
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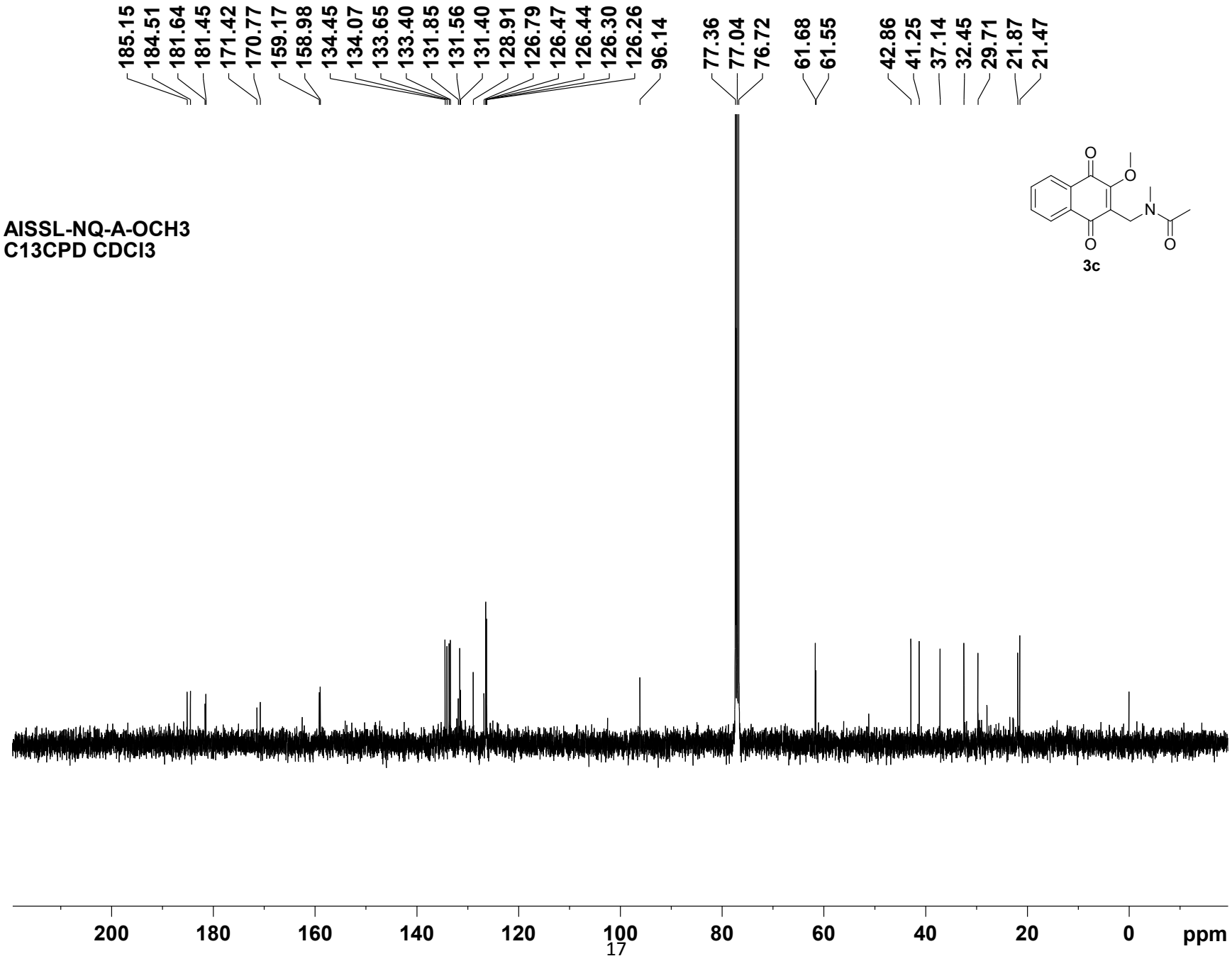
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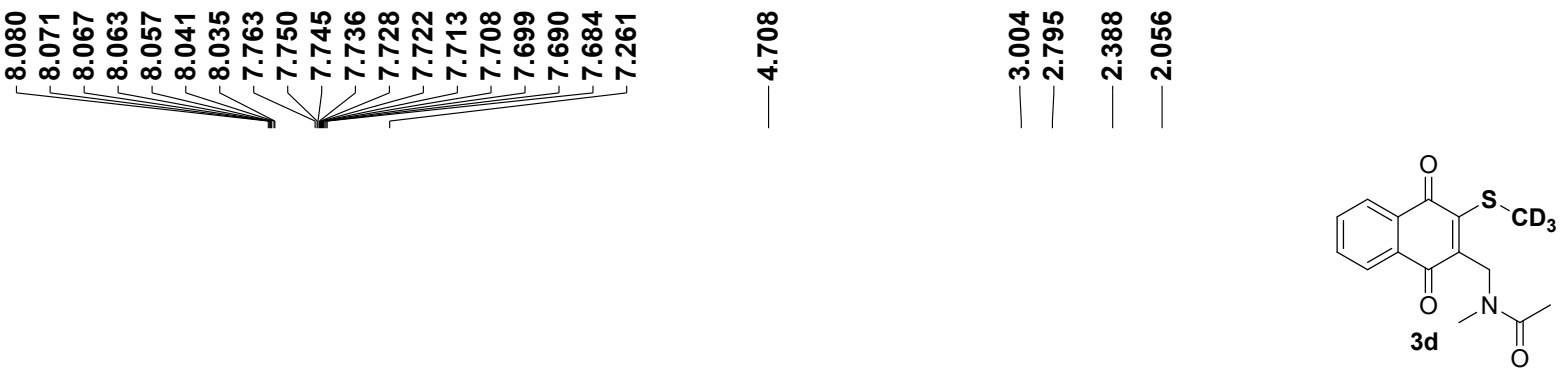
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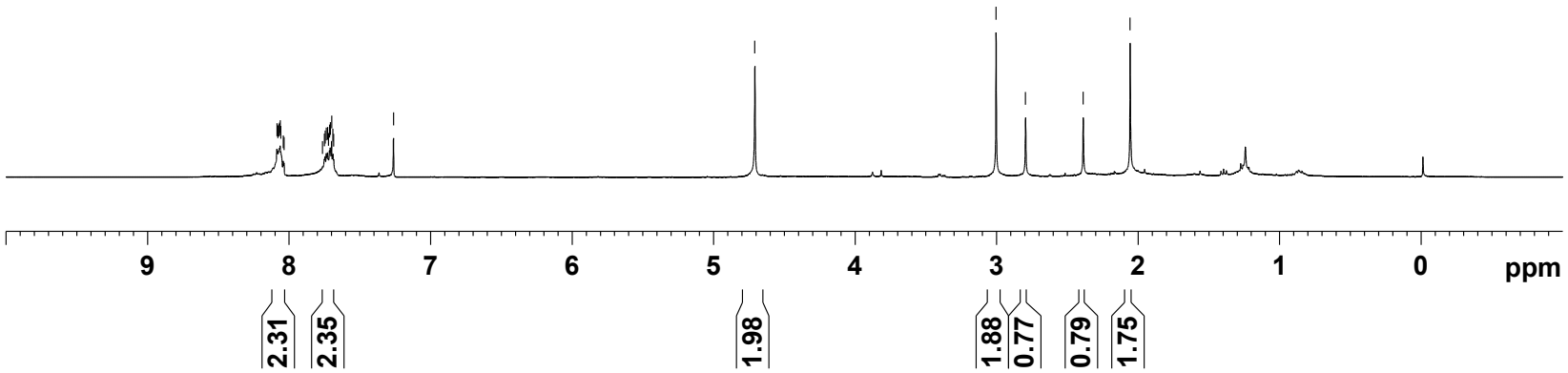
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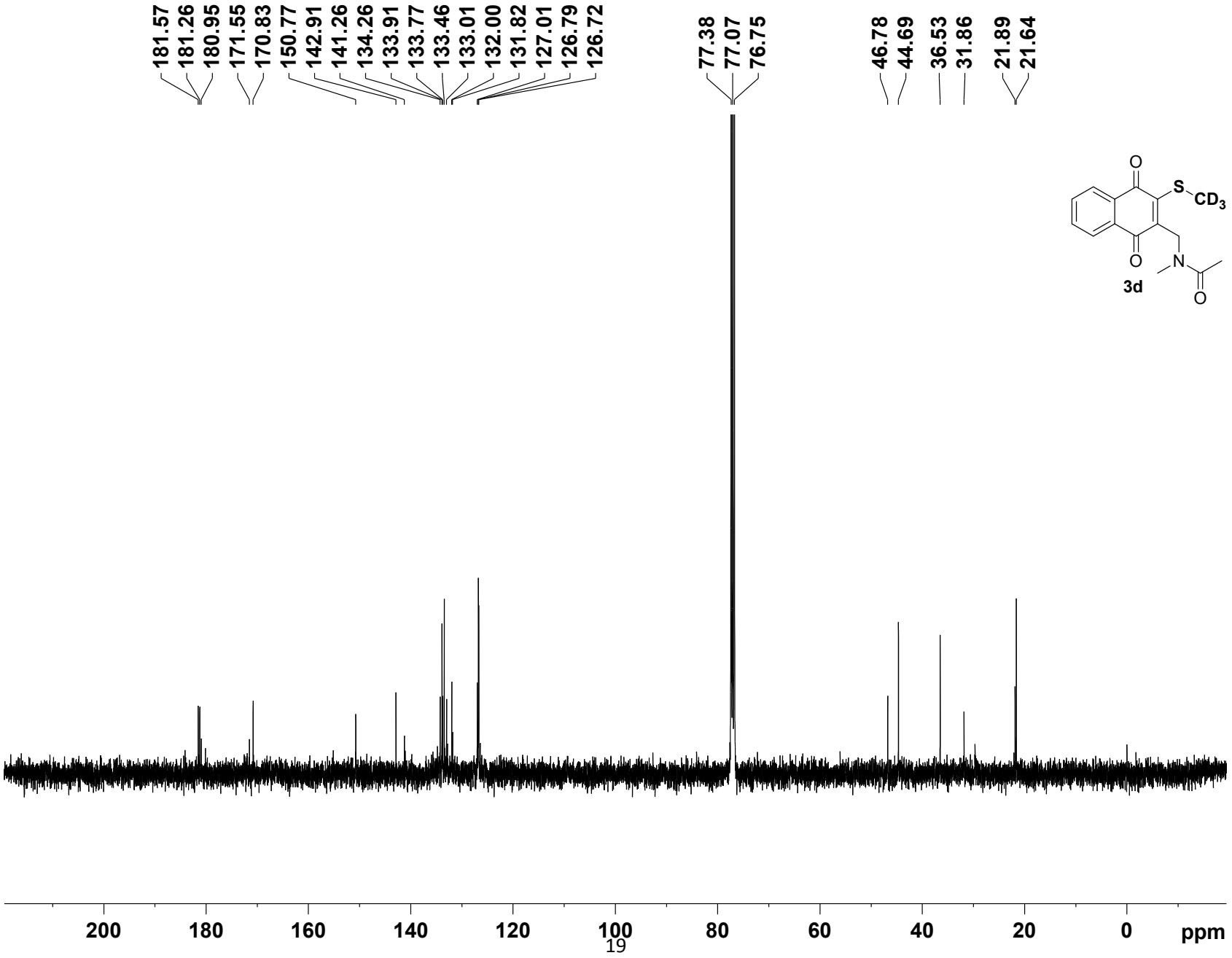
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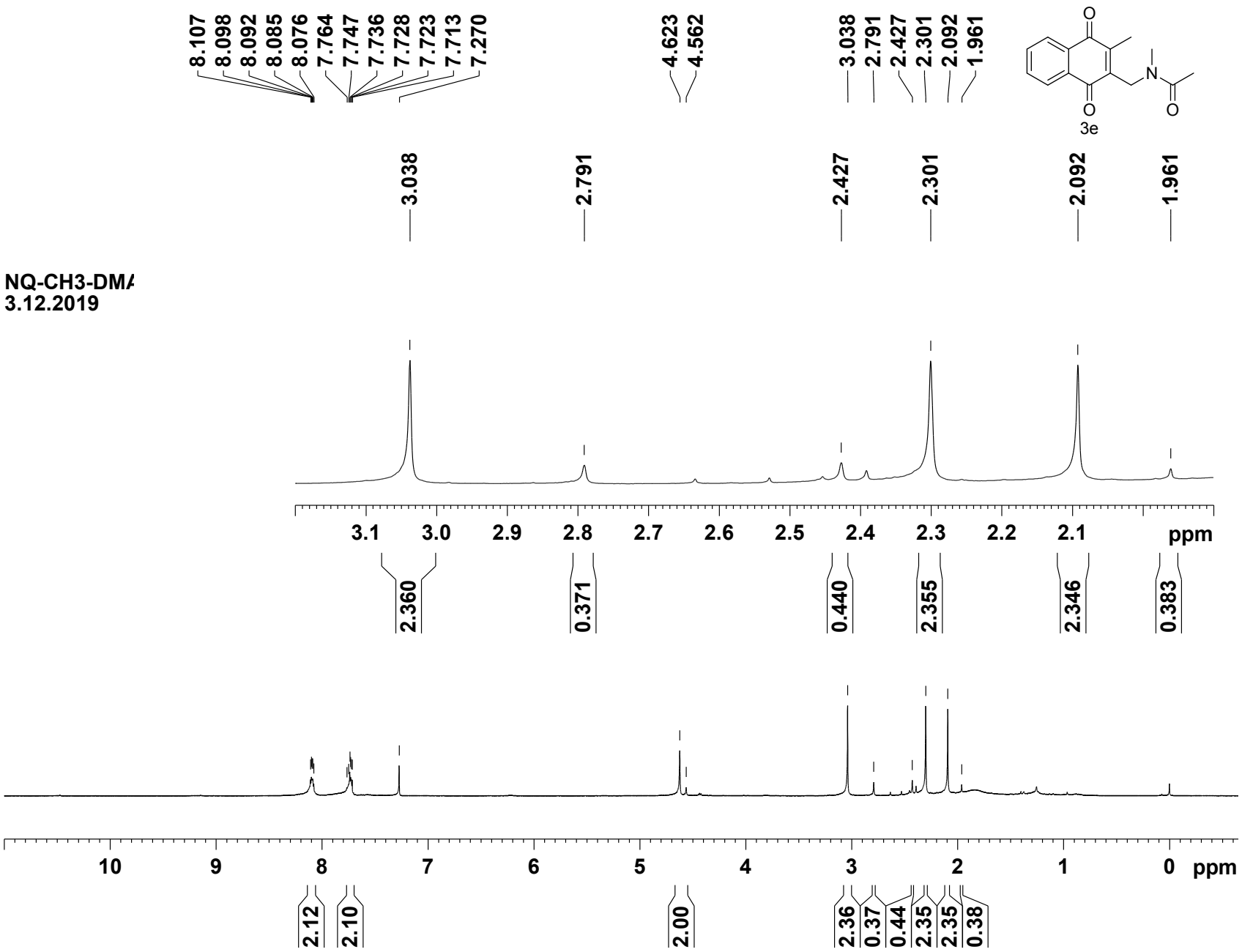
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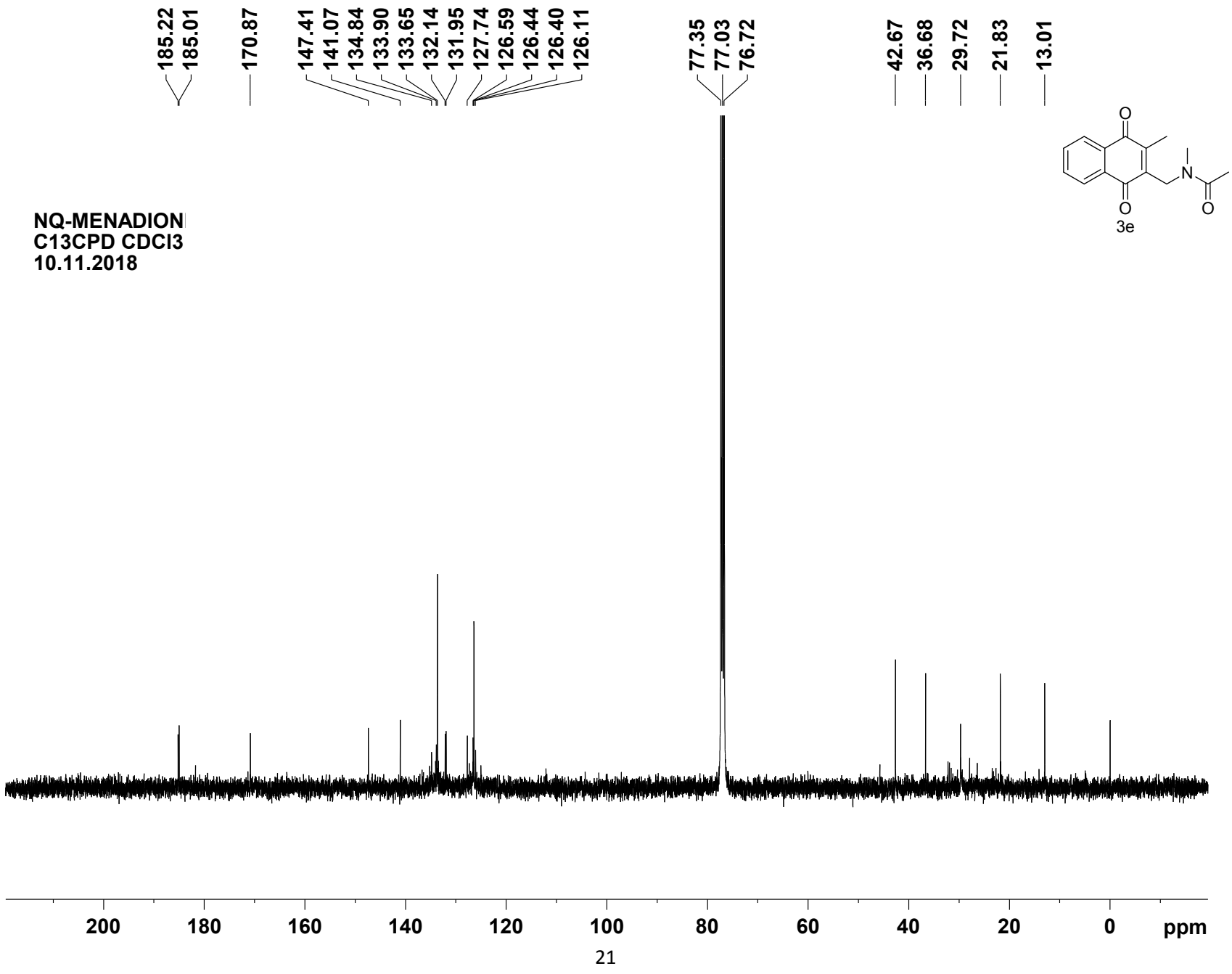
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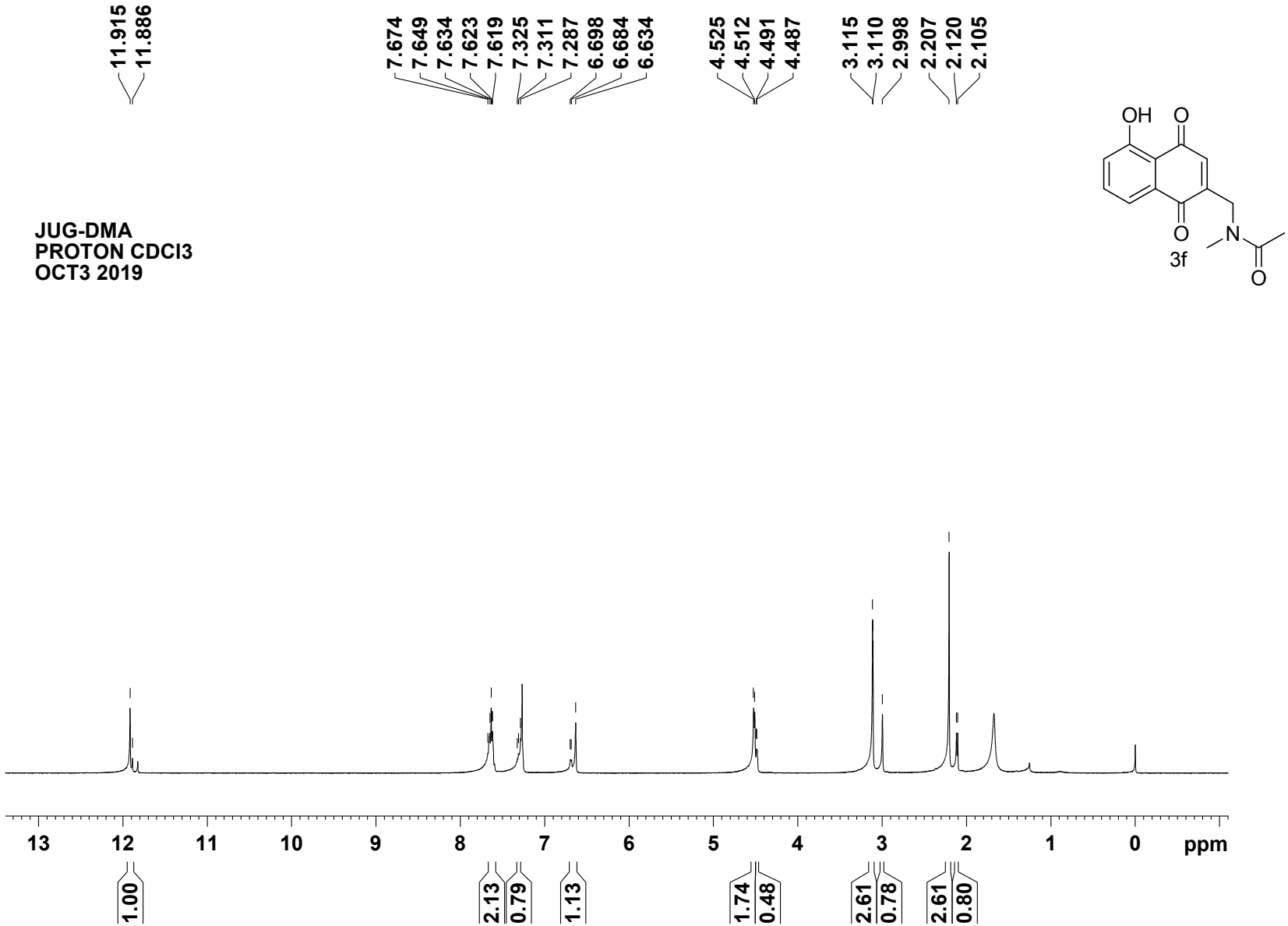
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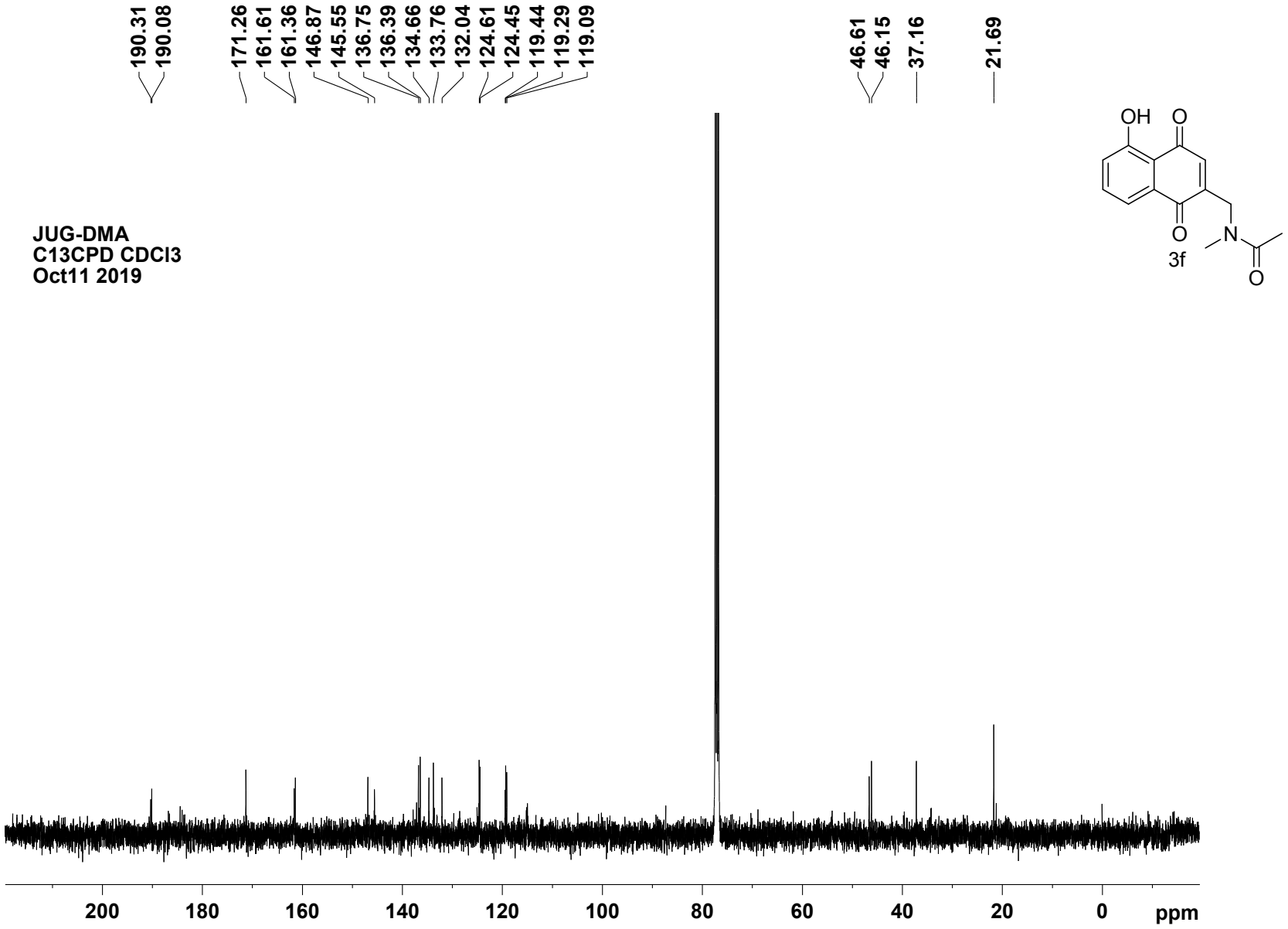
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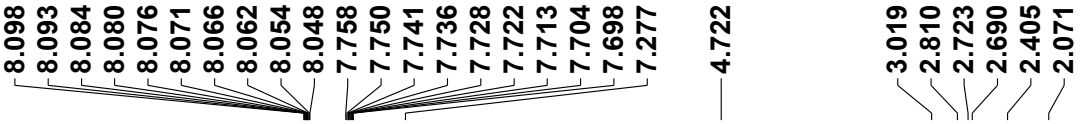
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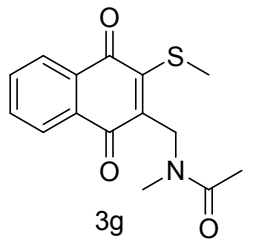
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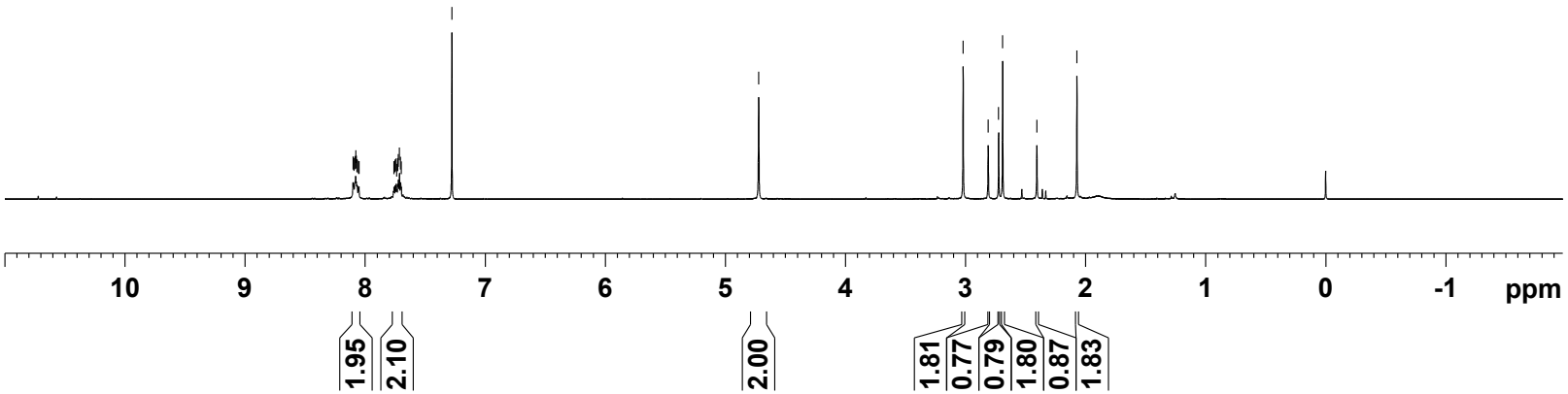
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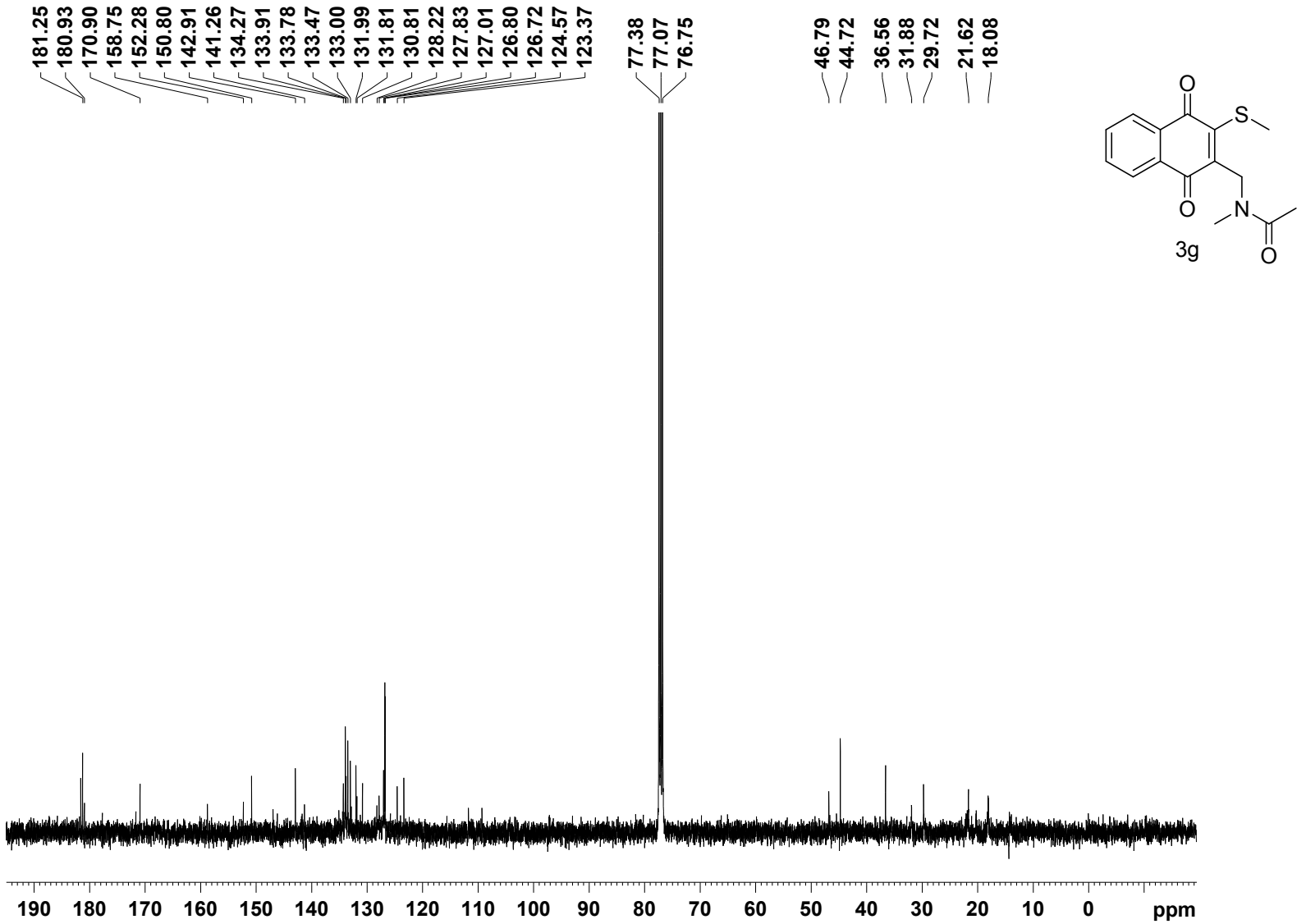
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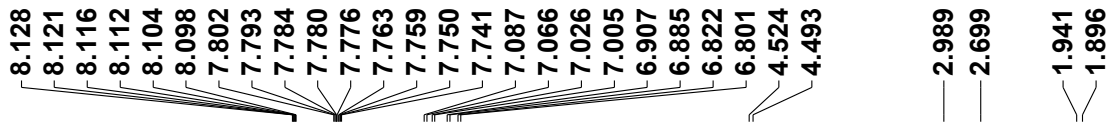
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7.793
7.784
7.780
7.776
7.763
7.759
7.750
7.741
7.087
7.066
7.026
7.005
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6.801
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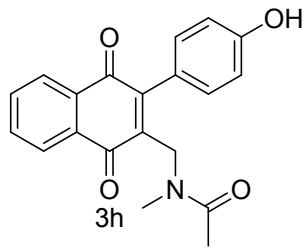
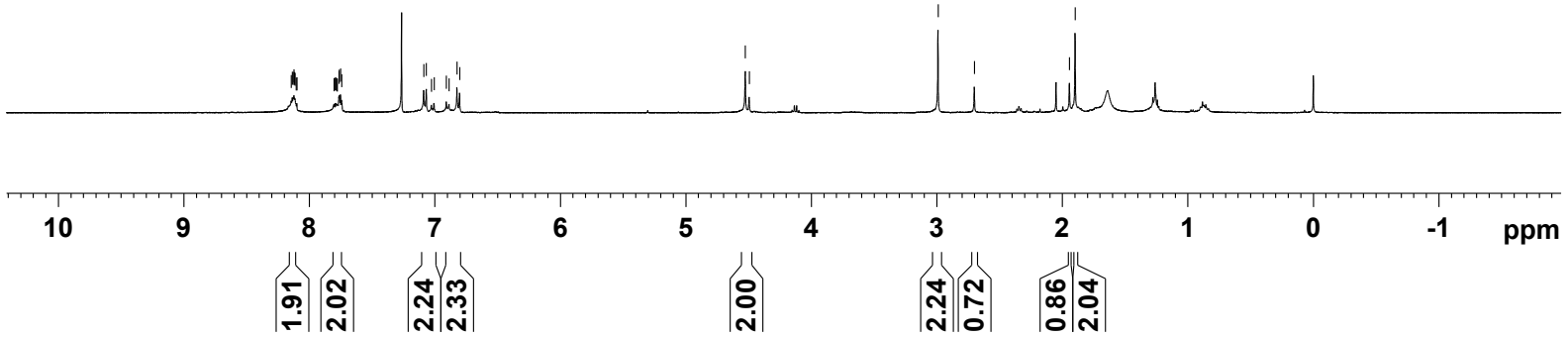
4.493
2.989
2.699
1.941
1.896

NQ-OH-DMA


OH
O
O
O
N
O
3h


10
9
8
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5
4
3
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-1
ppm
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2.33
2.00
2.24
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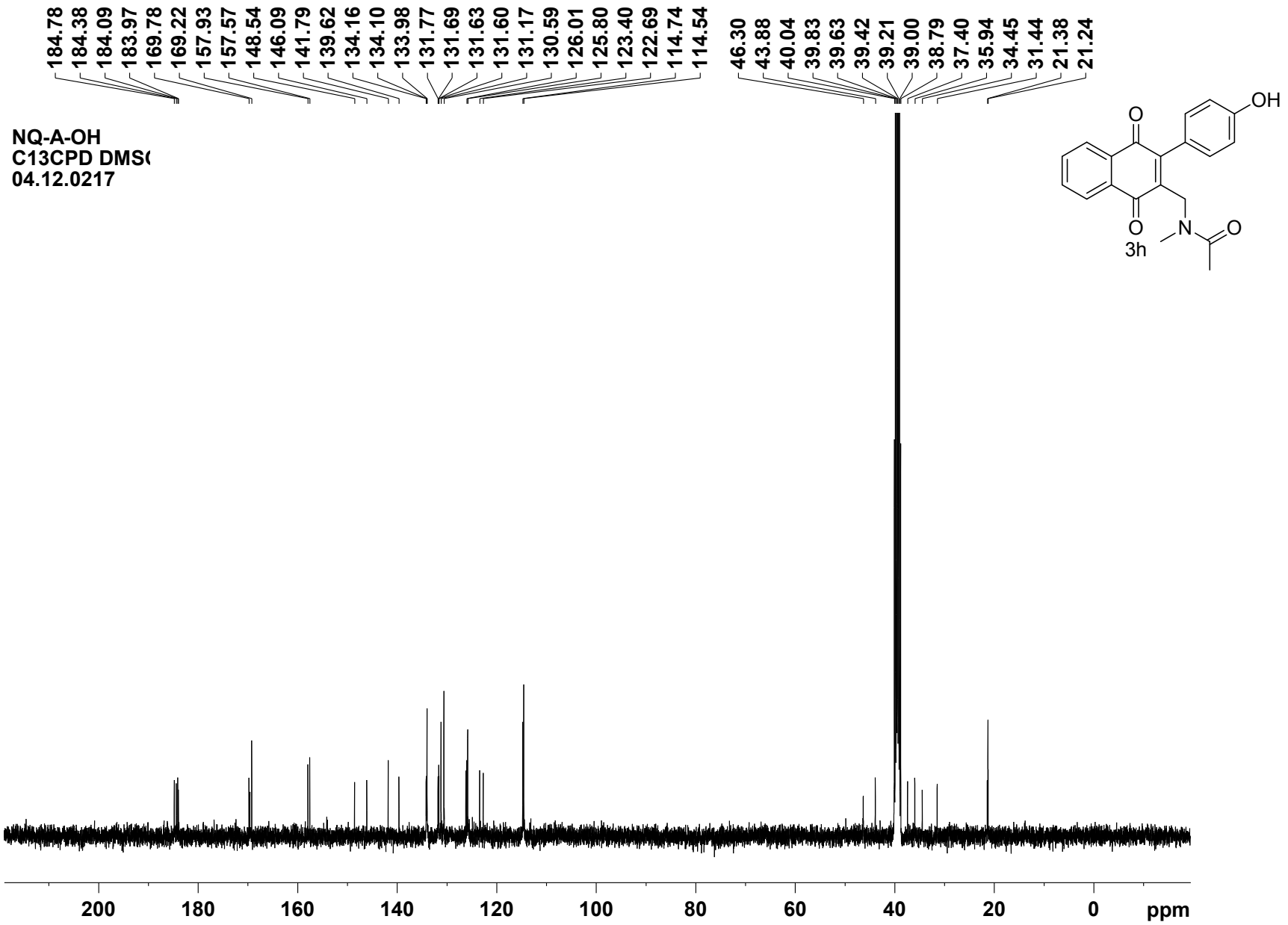
0.86
2.04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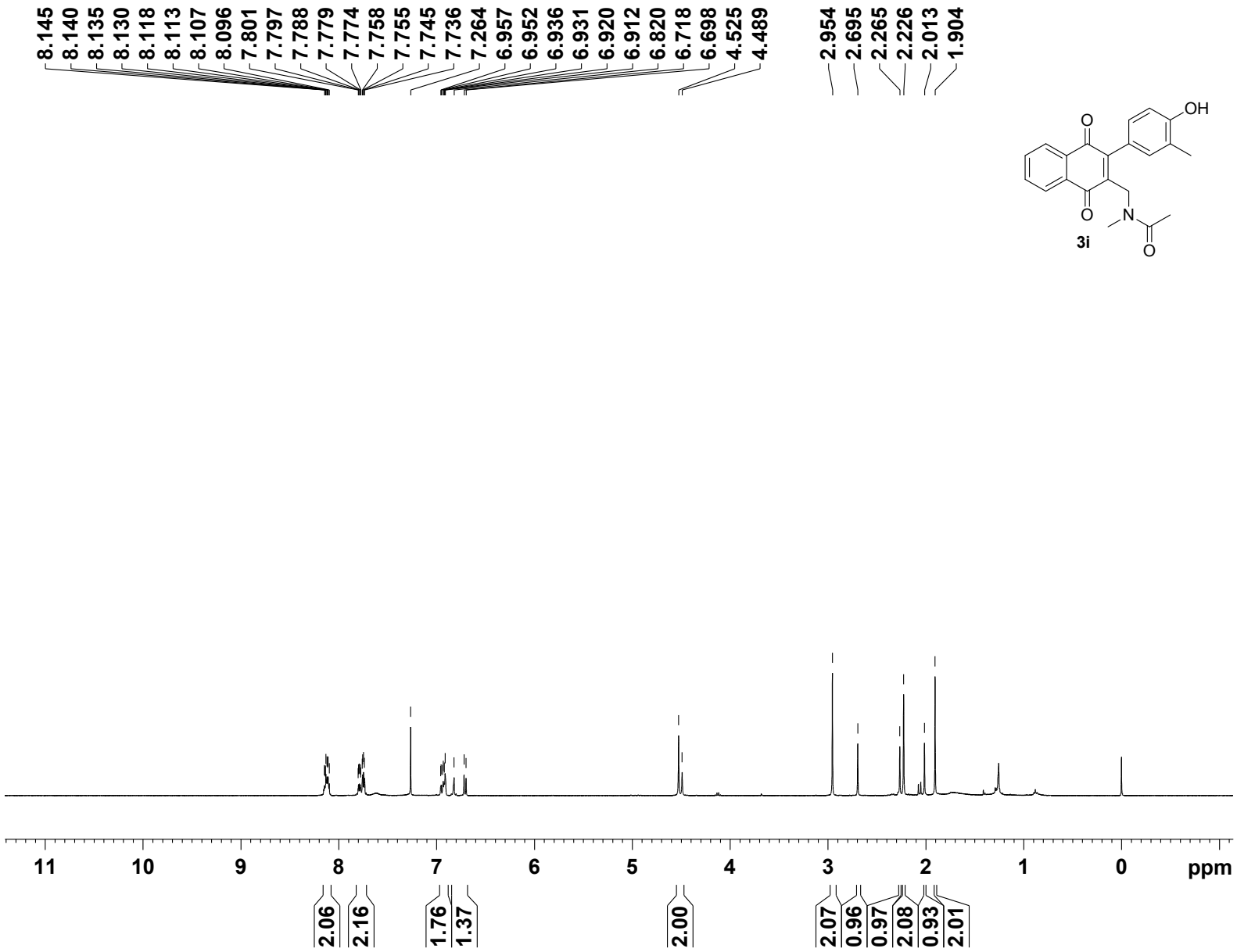
134.16
134.10
133.98
131.77
131.69
131.63
131.60
131.17
130.59
126.01
125.80
123.40
122.69
114.74
114.54
46.30
43.88
40.04
39.83
39.63
39.42
39.21
39.00
38.79
37.40
35.94
34.45
31.44
21.38
21.24
NQ-A-OH
C13CPD DMS(
04.12.0217
OH
O
O
O
N
O
3h
200
180
160
140
120
100
80
60
40
20
0
ppm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8.096
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7.774
7.758
7.755
7.745
7.736
7.264
6.957
6.952
6.936
6.931
6.920
6.912
6.820
6.718
6.698
4.525
4.489
2.954
2.695
2.265
2.226
2.013
1.904
OH
O
O
N
O
3i
11
10
9
8
7
6
5
4
3
2
1
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ppm
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1.76
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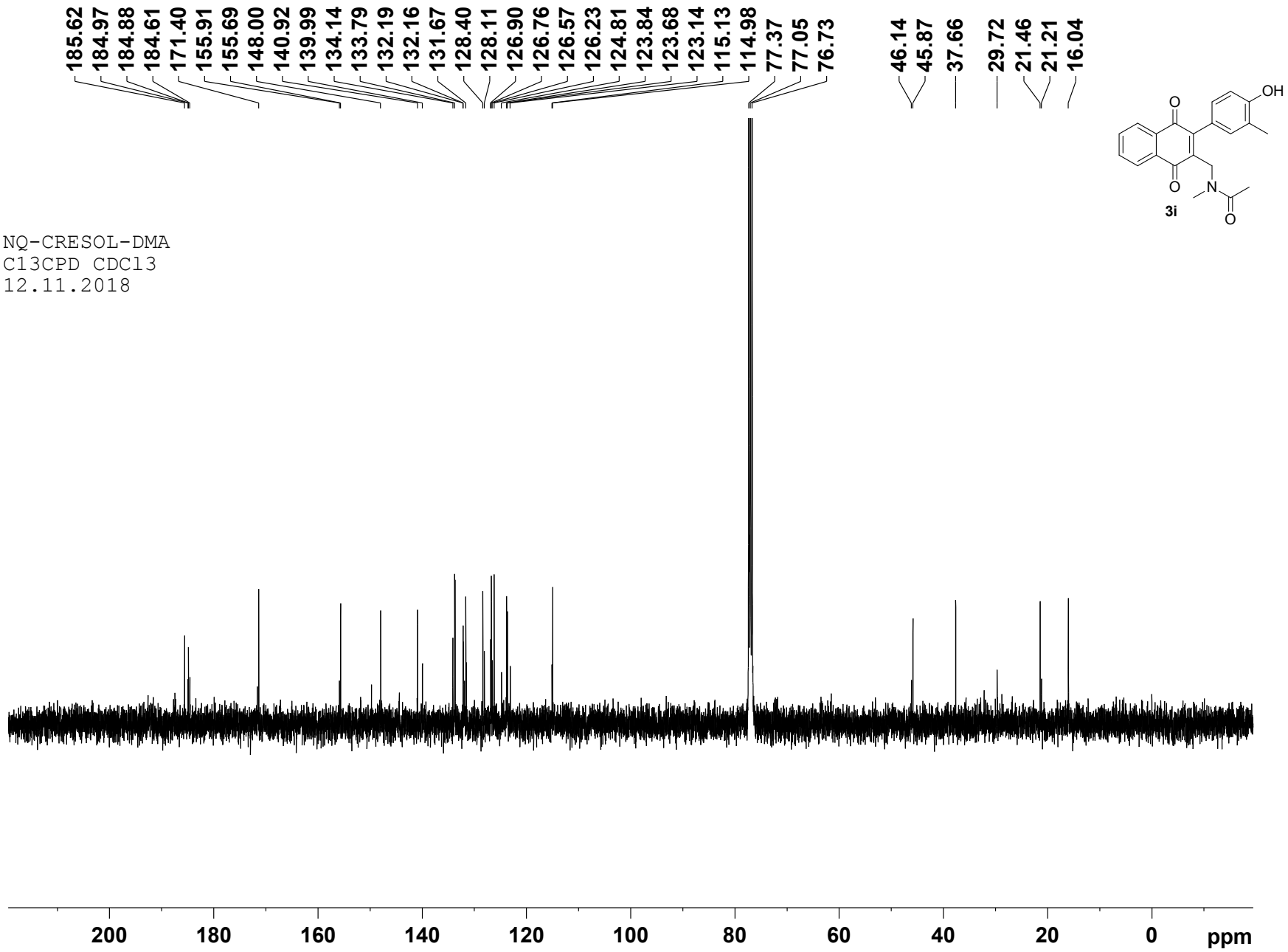
2.08
0.93
2.01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133.79
132.19
132.16
131.67
128.40
128.11
126.90
126.76
126.57
126.23
124.81
123.84
123.68
123.14
115.13
114.98
77.37
77.05
76.73
46.14
45.87
37.66
29.72
21.46
21.21
16.04
OH
O
O
N
O
3i
NQ-CRESOL-DMA
C13CPD CDCl3
12.11.2018
200
180
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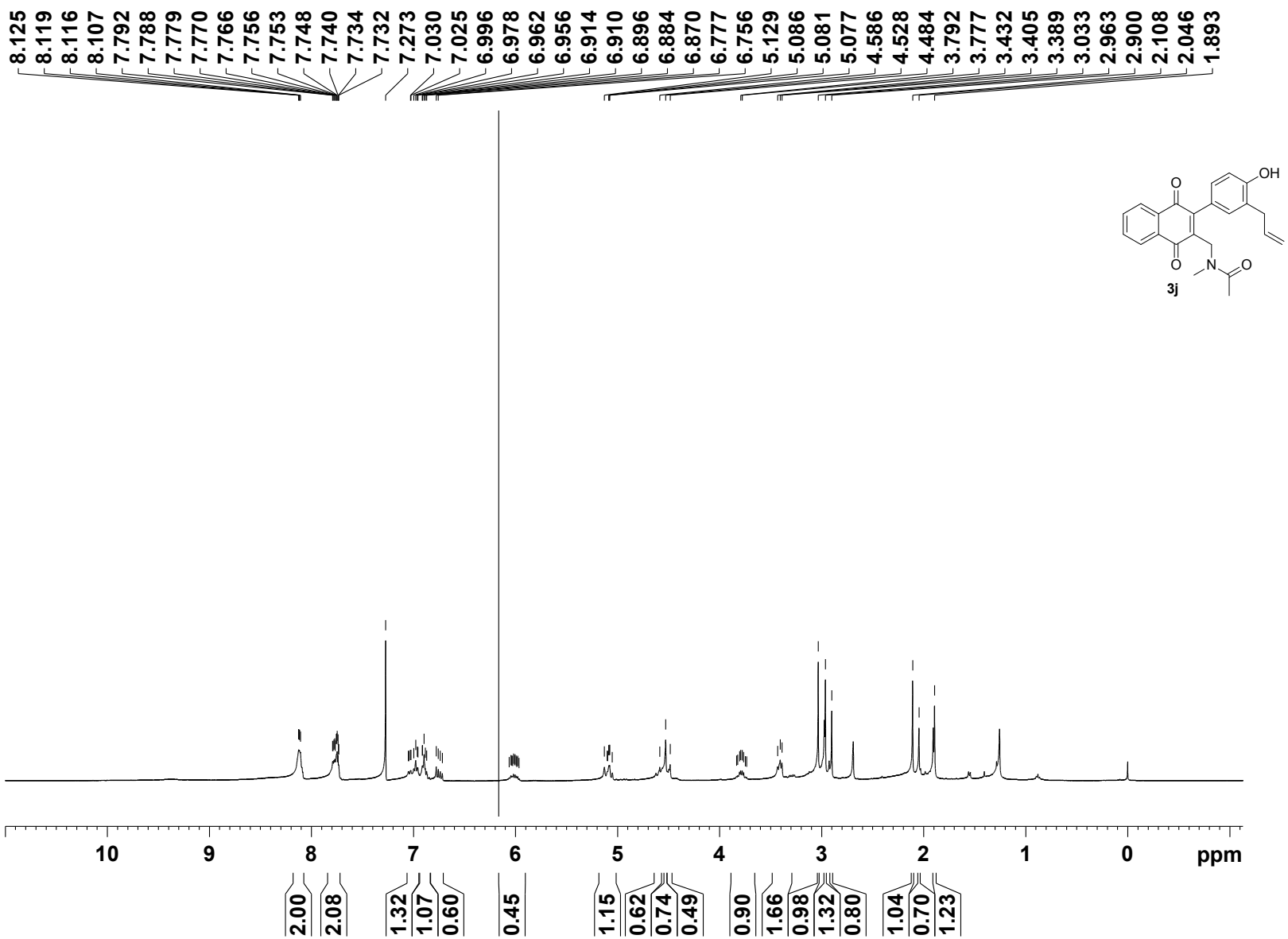
140
120
100
80
60
40
20
0
ppm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8.107
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7.788
7.779
7.770
7.766
7.756
7.753
7.748
7.740
7.734
7.732
7.273
7.030
7.025
6.996
6.978
6.962
6.956
6.914
6.910
6.896
6.884
6.870
6.777
6.756
5.129
5.086
5.081
5.077
4.586
4.528
4.484
3.792
3.777
3.432
3.405
3.389
3.033
2.963
2.900
2.108
2.046
1.893
OH
O
O
N
O
3j
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.08
1.32
1.07
0.60
0.45
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0.49
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1.66
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1.32
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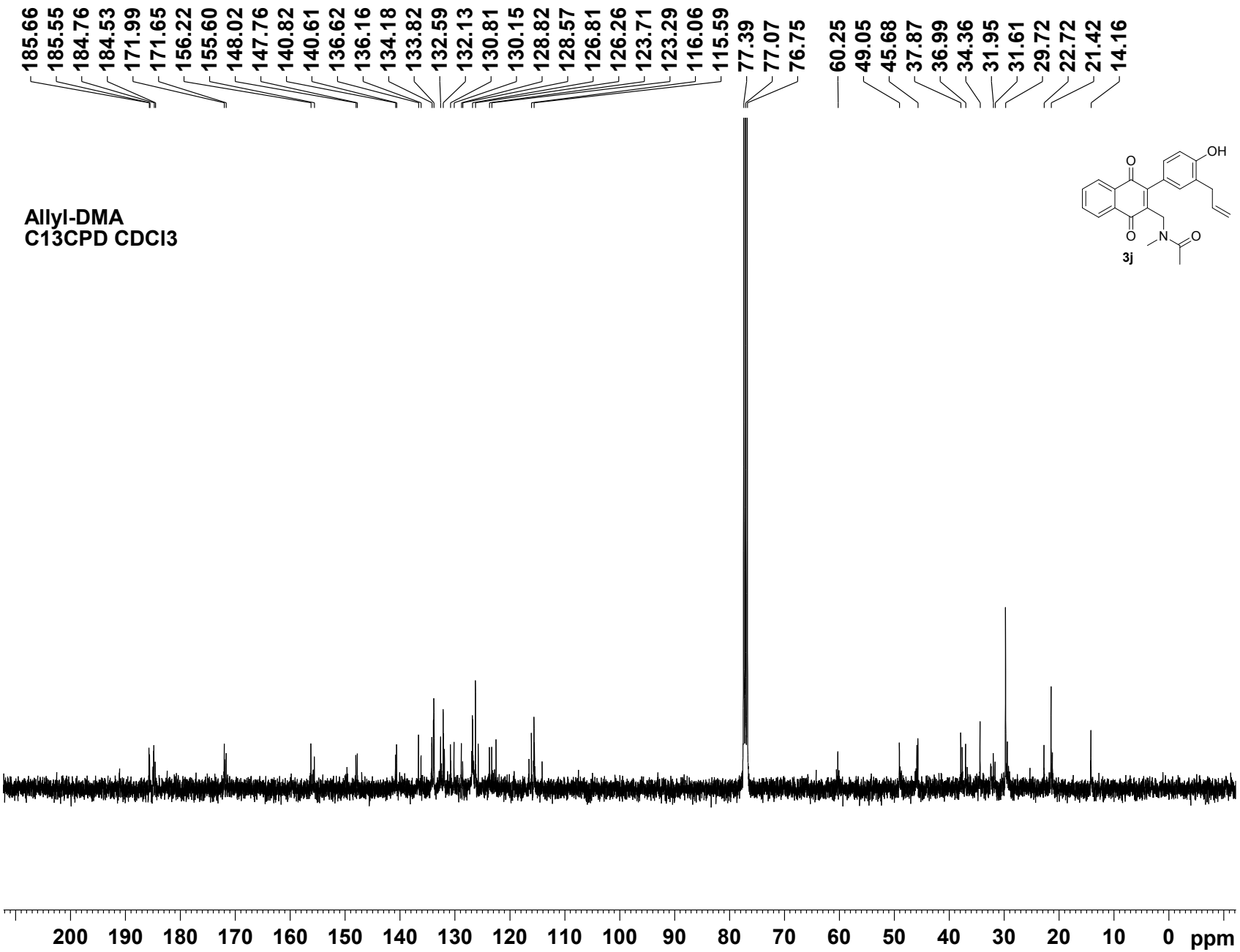
0.70
1.23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155.60
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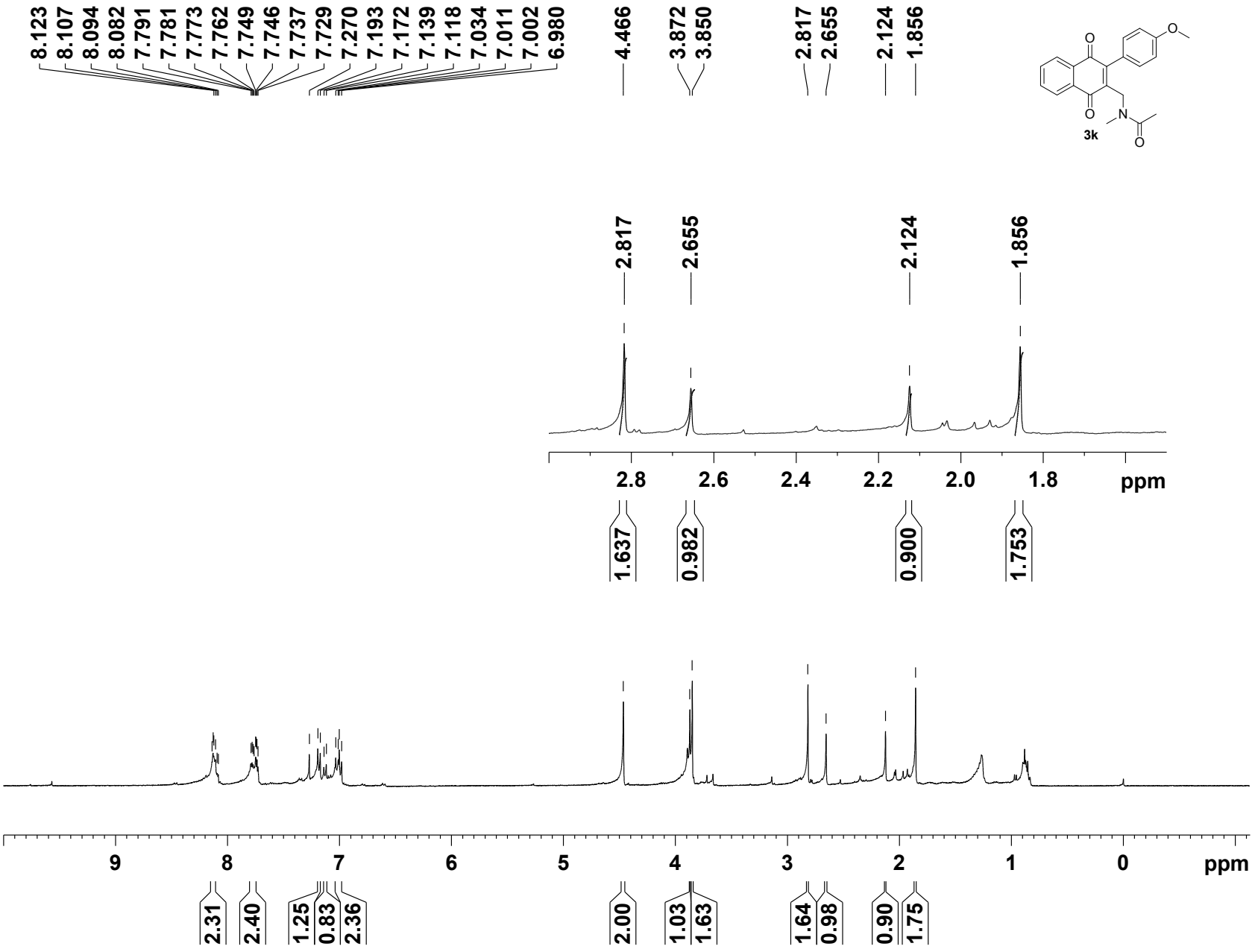
147.76
140.82
140.61
136.62
136.16
134.18
133.82
132.59
132.13
130.81
130.15
128.82
128.57
126.81
126.26
123.71
123.29
116.06
115.59
77.39
77.07
76.75
60.25
49.05
45.68
37.87
36.99
34.36
31.95
31.61
29.72
22.72
21.42
14.16
Allyl-DMA
C13CPD CDCl3
OH
O
O
O
N
O
3j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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8.107
8.094
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7.781
7.773
7.762
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7.746
7.737
7.729
7.270
7.193
7.172
7.139
7.118
7.034
7.011
7.002
6.980
4.466
3.872
3.850
2.817
2.655
2.124
1.856
3k
2.817
2.655
2.124
1.856
2.8
2.6
2.4
2.2
2.0
1.8
ppm
1.637
0.982
0.900
1.753
9
8
7
6
5
4
3
2
1
0
ppm
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1.25
0.83
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2.00
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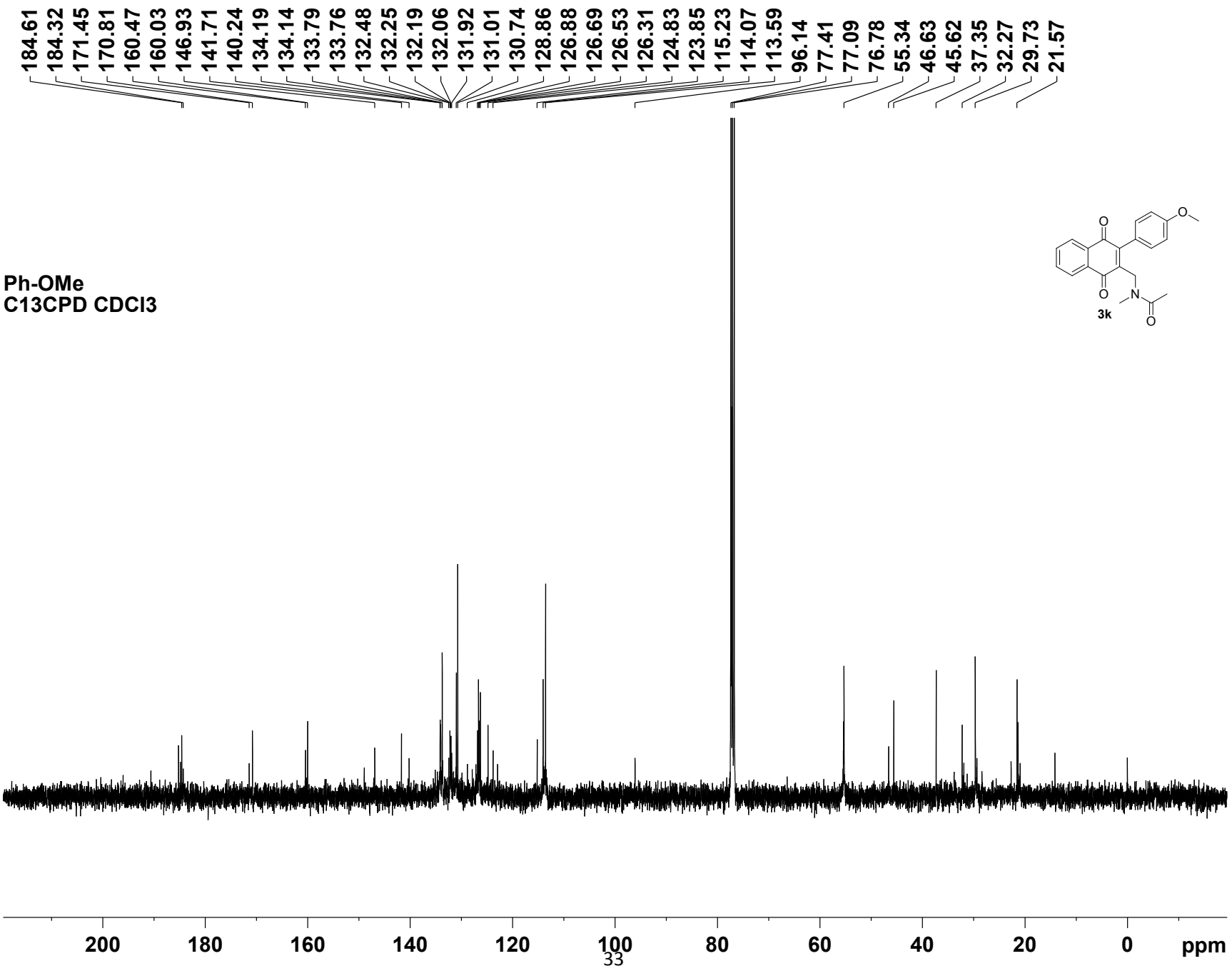
1.63
1.64
0.98
0.90
1.75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184.32
171.45
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160.47
160.03
146.93
141.71
140.24
134.19
134.14
133.79
133.76
132.48
132.25
132.19
132.06
131.92
131.01
130.74
128.86
126.88
126.69
126.53
126.31
124.83
123.85
115.23
114.07
113.59
96.14
77.41
77.09
76.78
55.34
46.63
45.62
37.35
32.27
29.73
21.57
Ph-OMe
C13CPD CDCl3
3k
200
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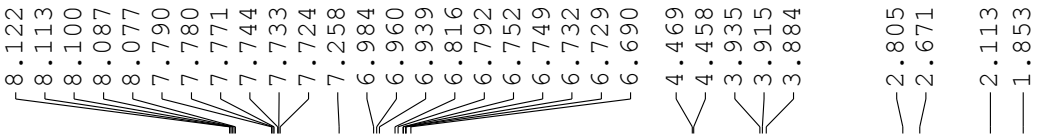
160
140
120
100
80
60
40
20
0
ppm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8.077
7.790
7.780
7.771
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7.733
7.724
7.258
6.984
6.960
6.939
6.816
6.792
6.752
6.749
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6.729
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3.884
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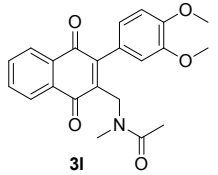
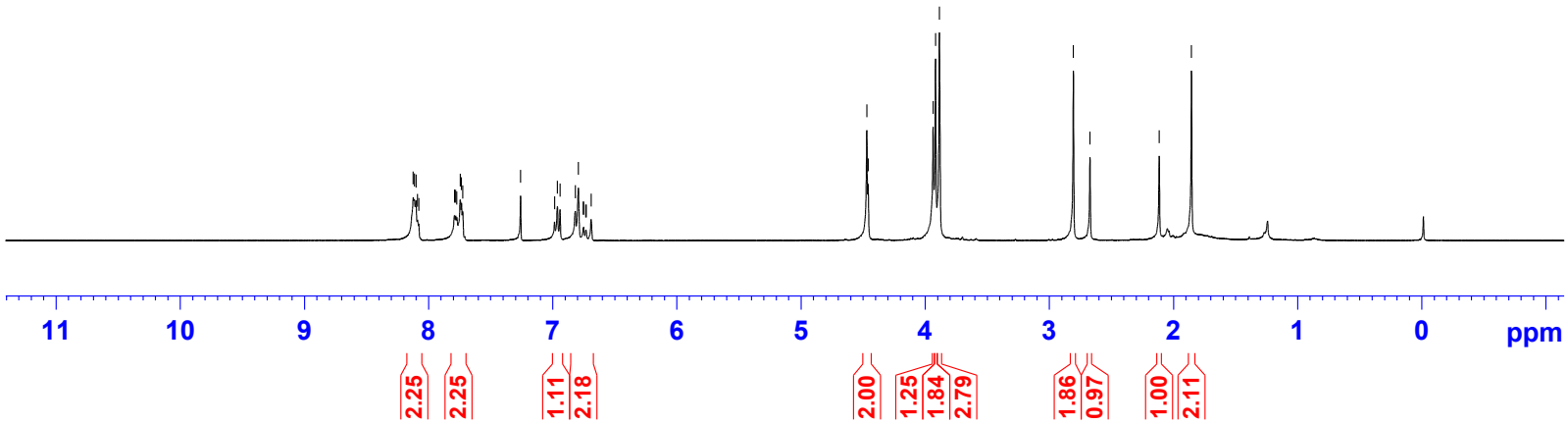
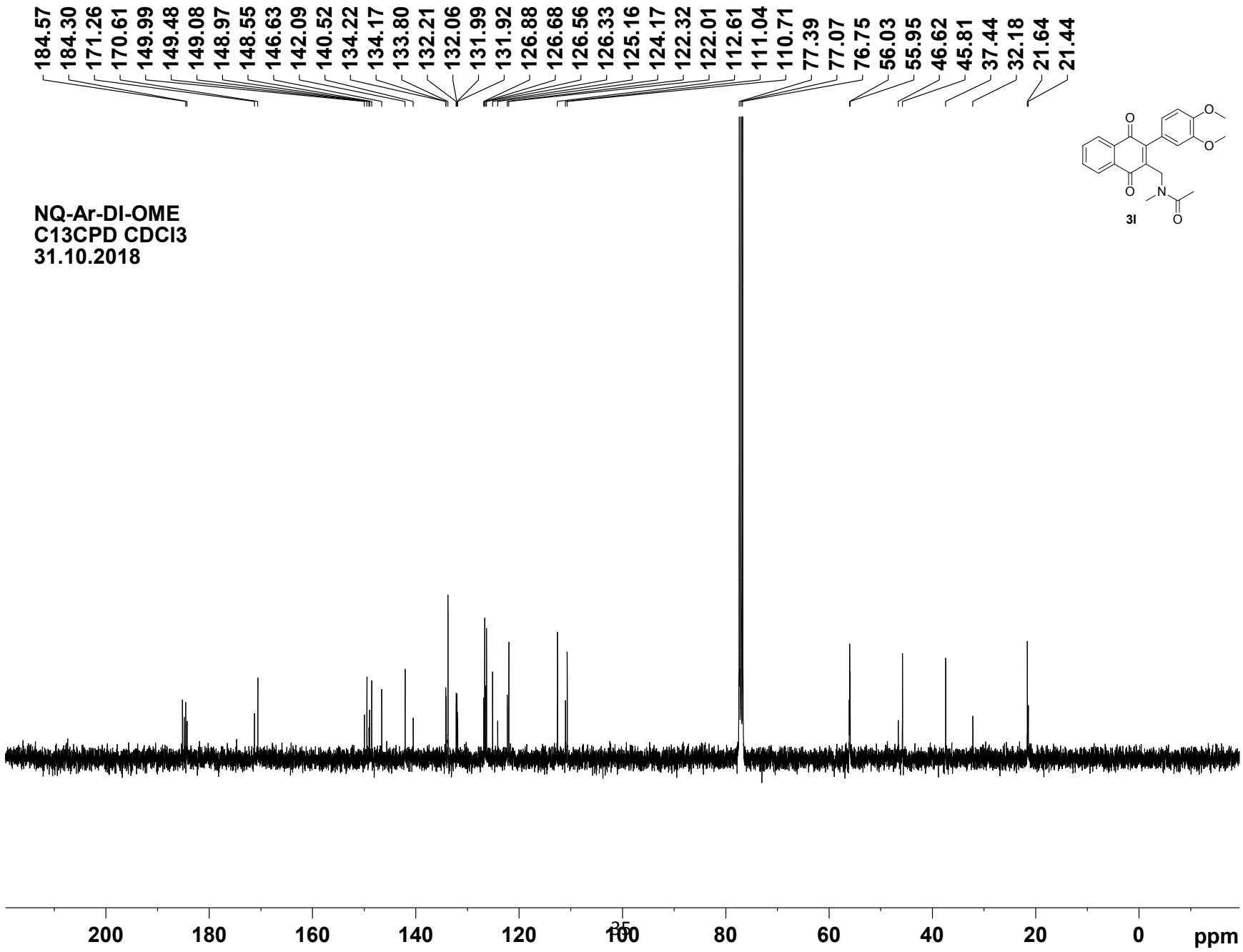
2.113
1.853


O
O
O
O
N
O
3l

NQ-Ar-DI-OME


11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.25
2.25
1.11
2.18
2.00
1.25
1.84
2.79
1.86
0.97
1.00
2.11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170.61
149.99
149.48
149.08
148.97
148.55
146.63
142.09
140.52
134.22
134.17
133.80
132.21
132.06
131.99
131.92
126.88
126.68
126.56
126.33
125.16
124.17
122.32
122.01
112.61
111.04
110.71
77.39
77.07
76.75
56.03
55.95
46.62
45.81
37.44
32.18
21.64
21.44
NQ-Ar-DI-OME
C13CPD CDCl3
31.10.2018
3l
200
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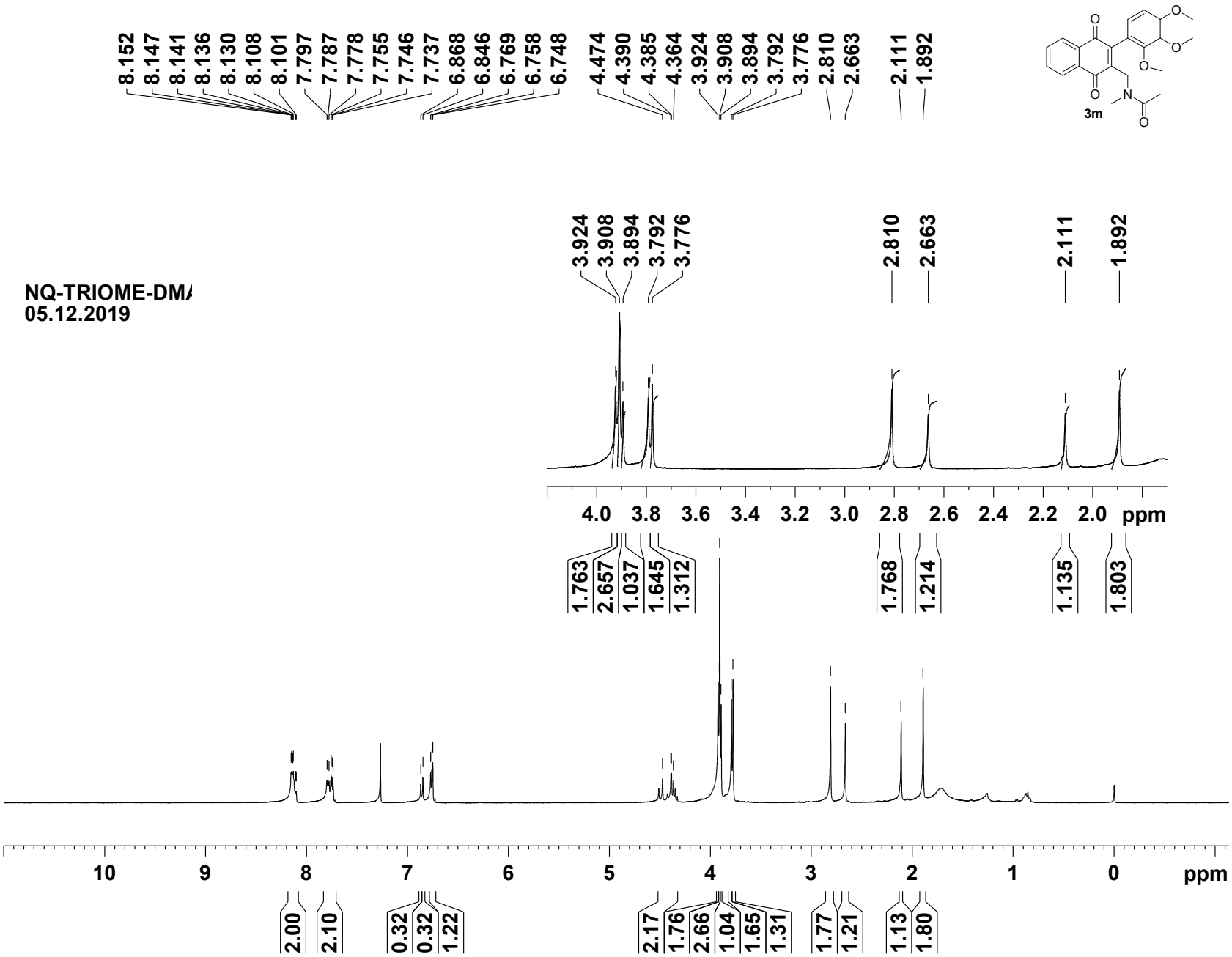
160
140
120
100
80
60
40
20
0
ppm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8.130
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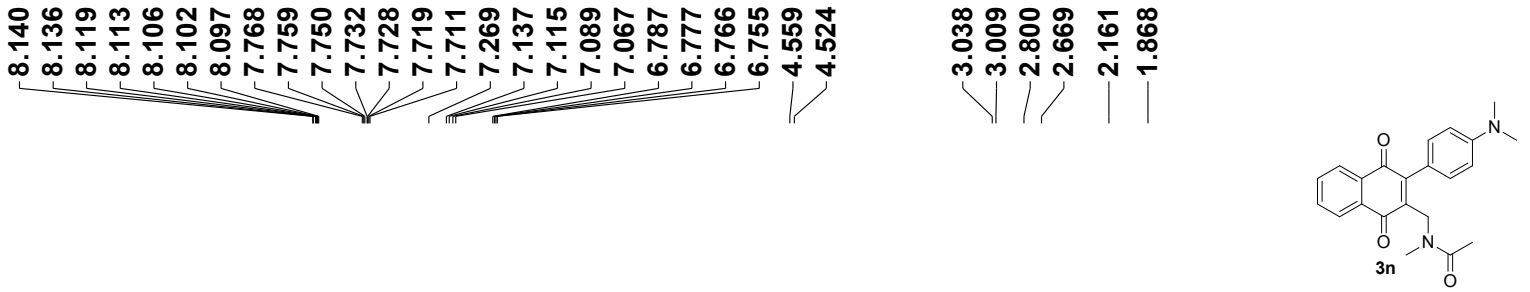
8.101
7.797
7.787
7.778
7.755
7.746
7.737
6.868
6.846
6.769
6.758
6.748
4.474
4.390
4.385
4.364
3.924
3.908
3.894
3.792
3.776
2.810
2.663
2.111
1.892
3m
NQ-TRIOME-DMA
05.12.2019
4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 ppm
1.763
2.657
1.037
1.645
1.312
1.768
1.214
1.135
1.803
10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
2.10
0.32
0.32
1.22
2.17
1.76
2.66
1.04
1.65
1.31
1.77
1.21
1.13
1.80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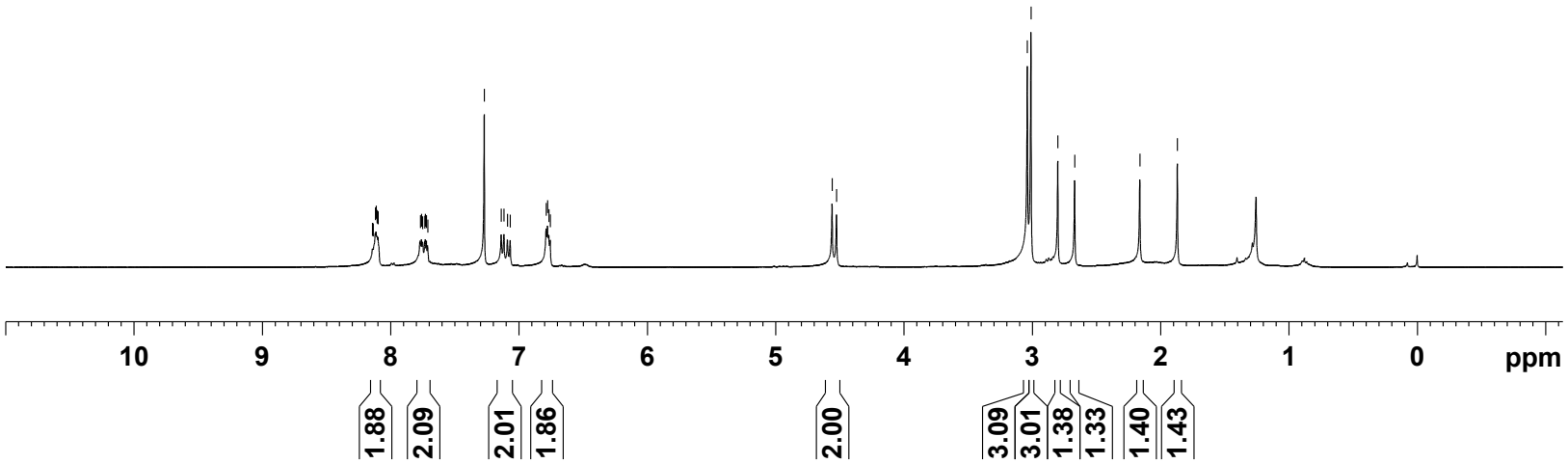
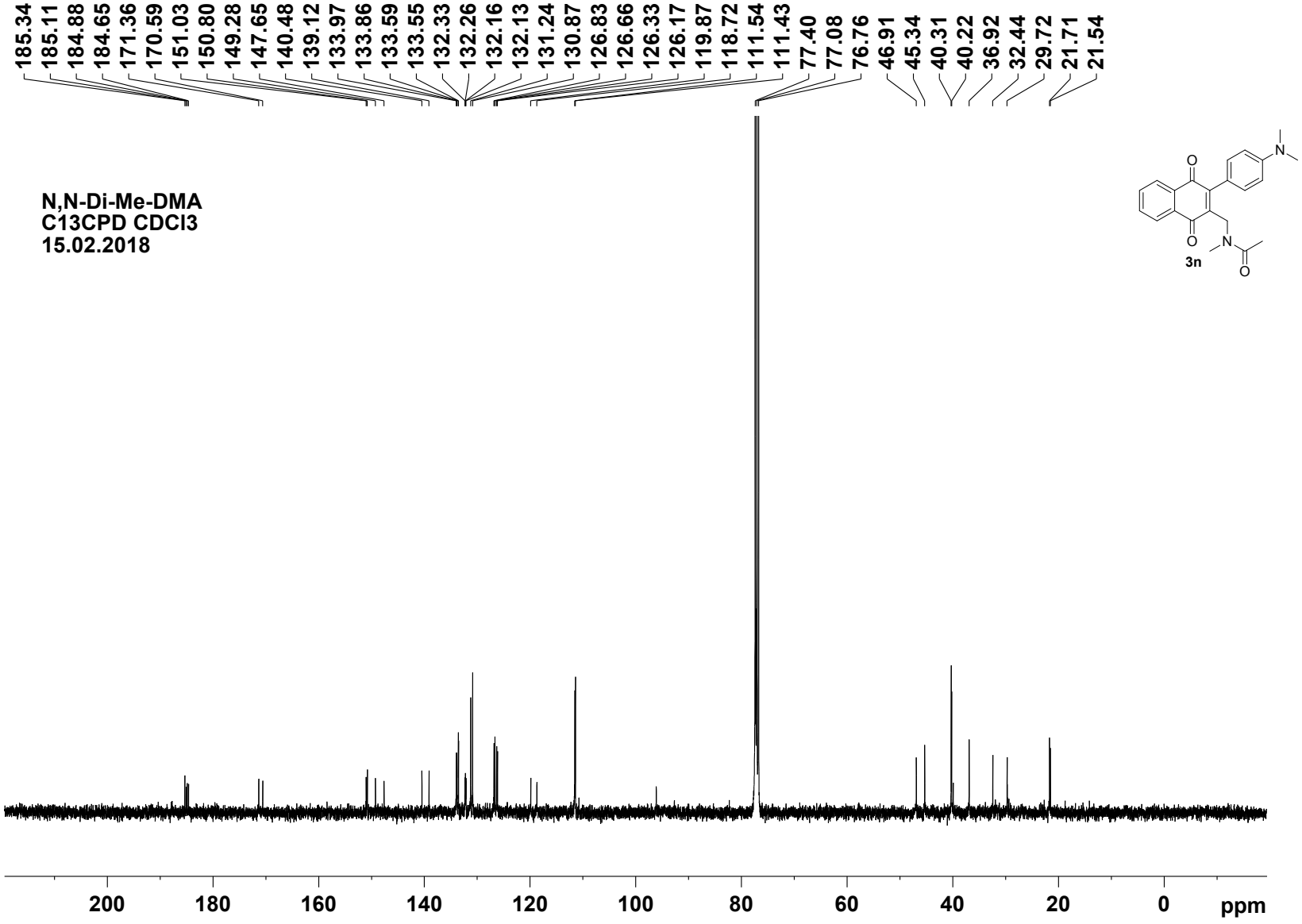
8.136
8.119
8.113
8.106
8.102
8.097
7.768
7.759
7.750
7.732
7.728
7.719
7.711
7.269
7.137
7.115
7.089
7.067
6.787
6.777
6.766
6.755
4.559
4.524
3.038
3.009
2.800
2.669
2.161
1.868
O
N
O
N
O
3n

N,N-Di-Me-DMA


10
9
8
7
6
5
4
3
2
1
0
ppm
1.88
2.09
2.01
1.86
2.00
3.09
3.01
1.38
1.33
1.40
1.43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184.88
184.65
171.36
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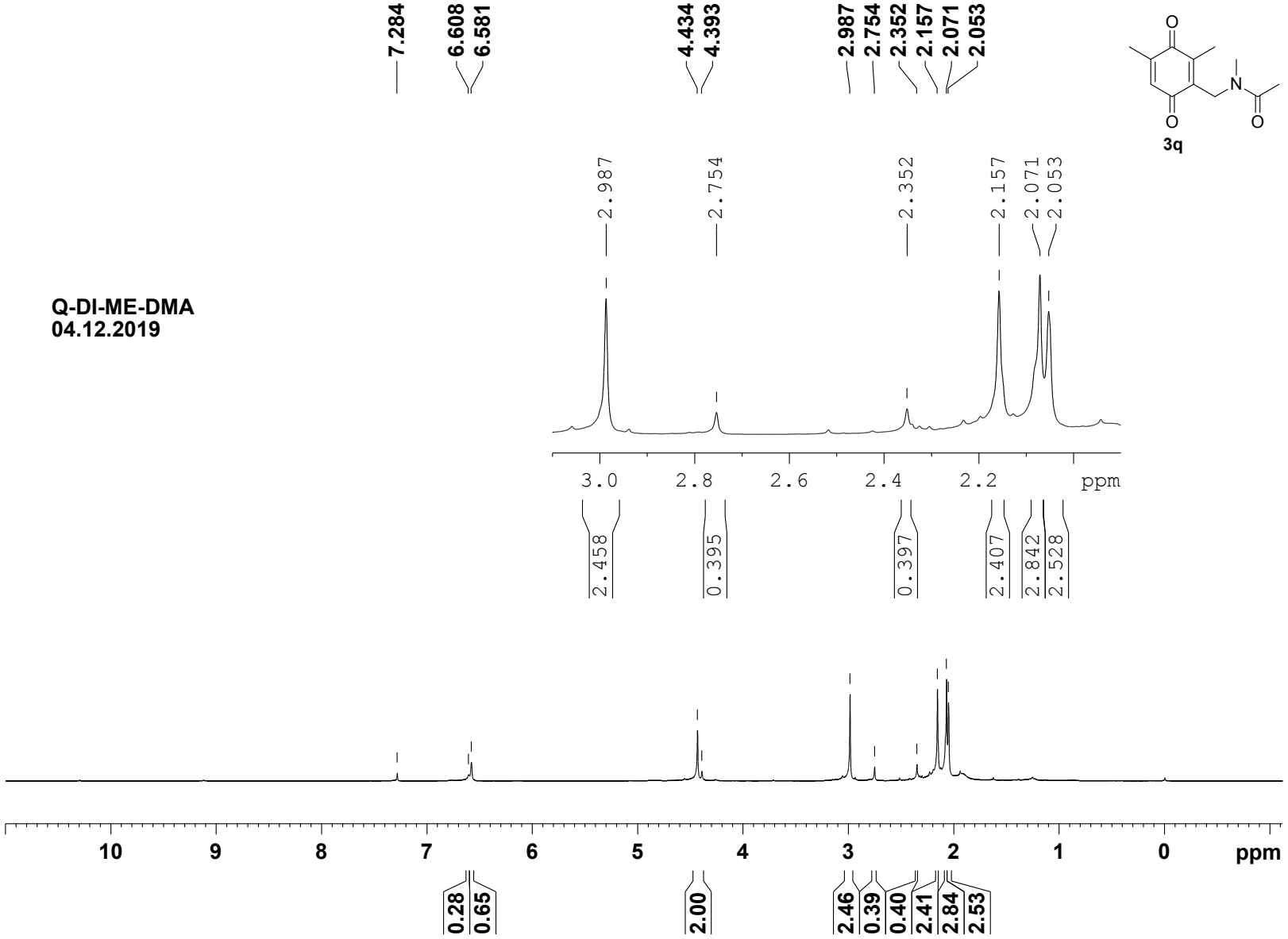
151.03
150.80
149.28
147.65
140.48
139.12
133.97
133.86
133.59
133.55
132.33
132.26
132.16
132.13
131.24
130.87
126.83
126.66
126.33
126.17
119.87
118.72
111.54
111.43
77.40
77.08
76.76
46.91
45.34
40.31
40.22
36.92
32.44
29.72
21.71
21.54
N,N-Di-Me-DMA
C13CPD CDCl3
15.02.2018
3n
200
180
160
140
120
100
80
60
40
20
0
ppm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6.581
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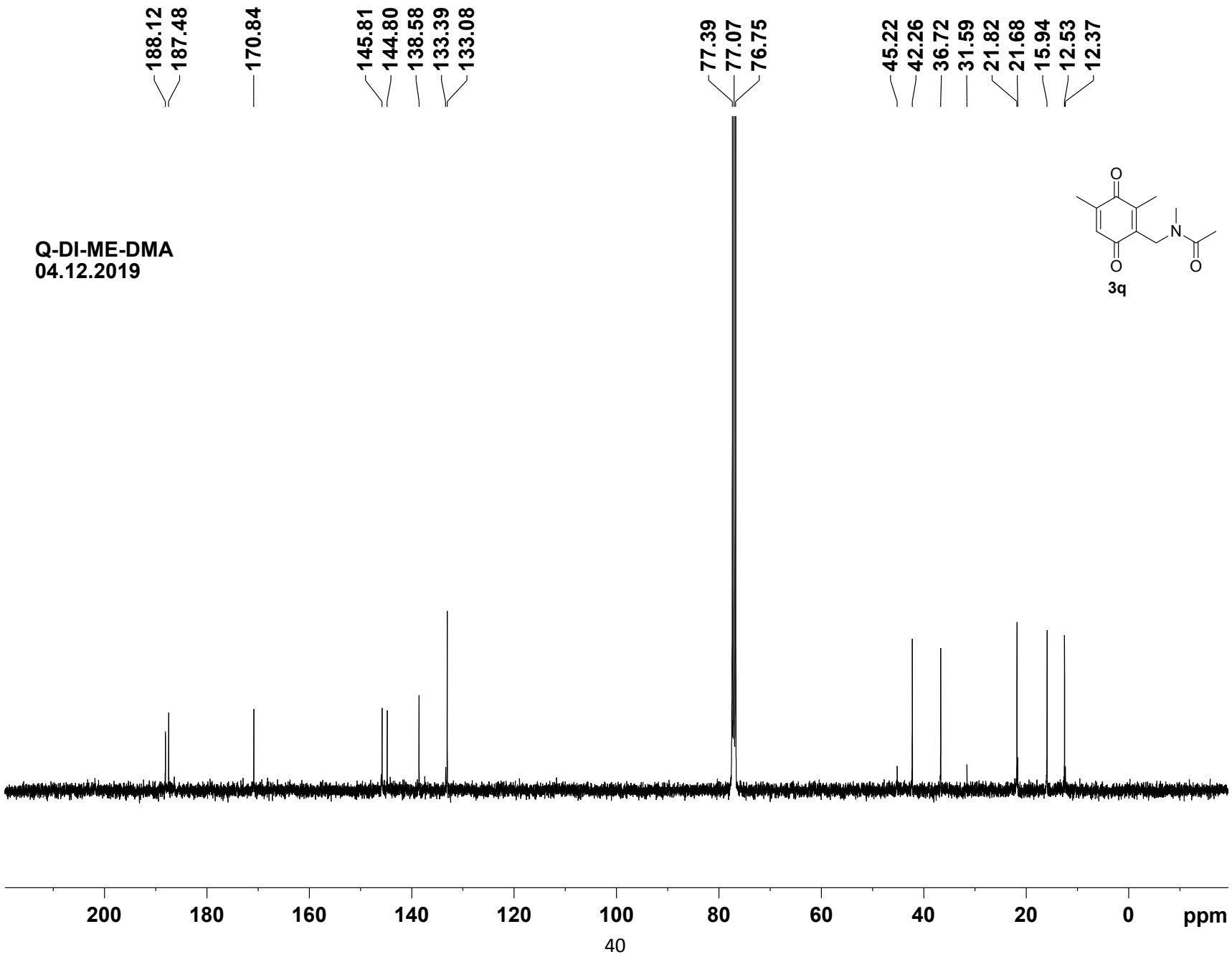
4.393
2.987
2.754
2.352
2.157
2.071
2.053
3q
Q-DI-ME-DMA
04.12.2019
3.0
2.8
2.6
2.4
2.2
ppm
2.458
0.395
0.397
2.407
2.842
2.528
10
9
8
7
6
5
4
3
2
1
0
ppm
0.28
0.65
2.00
2.46
0.39
0.40
2.41
2.84
2.53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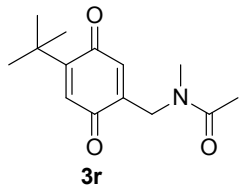
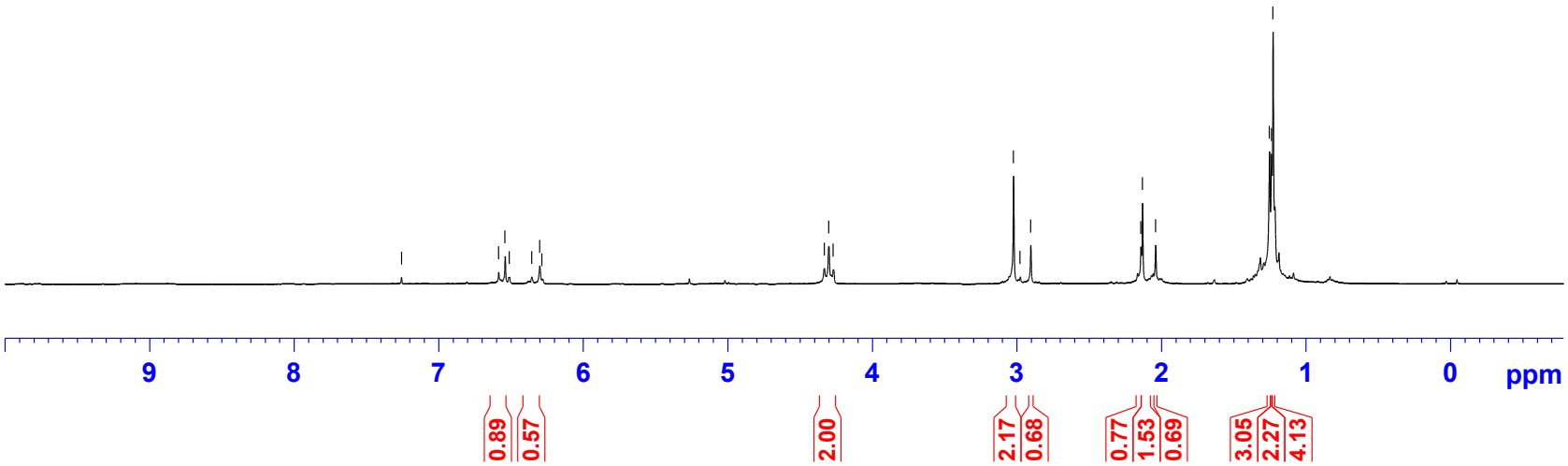
170.84
145.81
144.80
138.58
133.39
133.08
77.39
77.07
76.75
45.22
42.26
36.72
31.59
21.82
21.68
15.94
12.53
12.37
Q-DI-ME-DMA
04.12.2019
O
O
N
O
3q
200
180
160
140
120
100
80
60
40
20
0
ppm

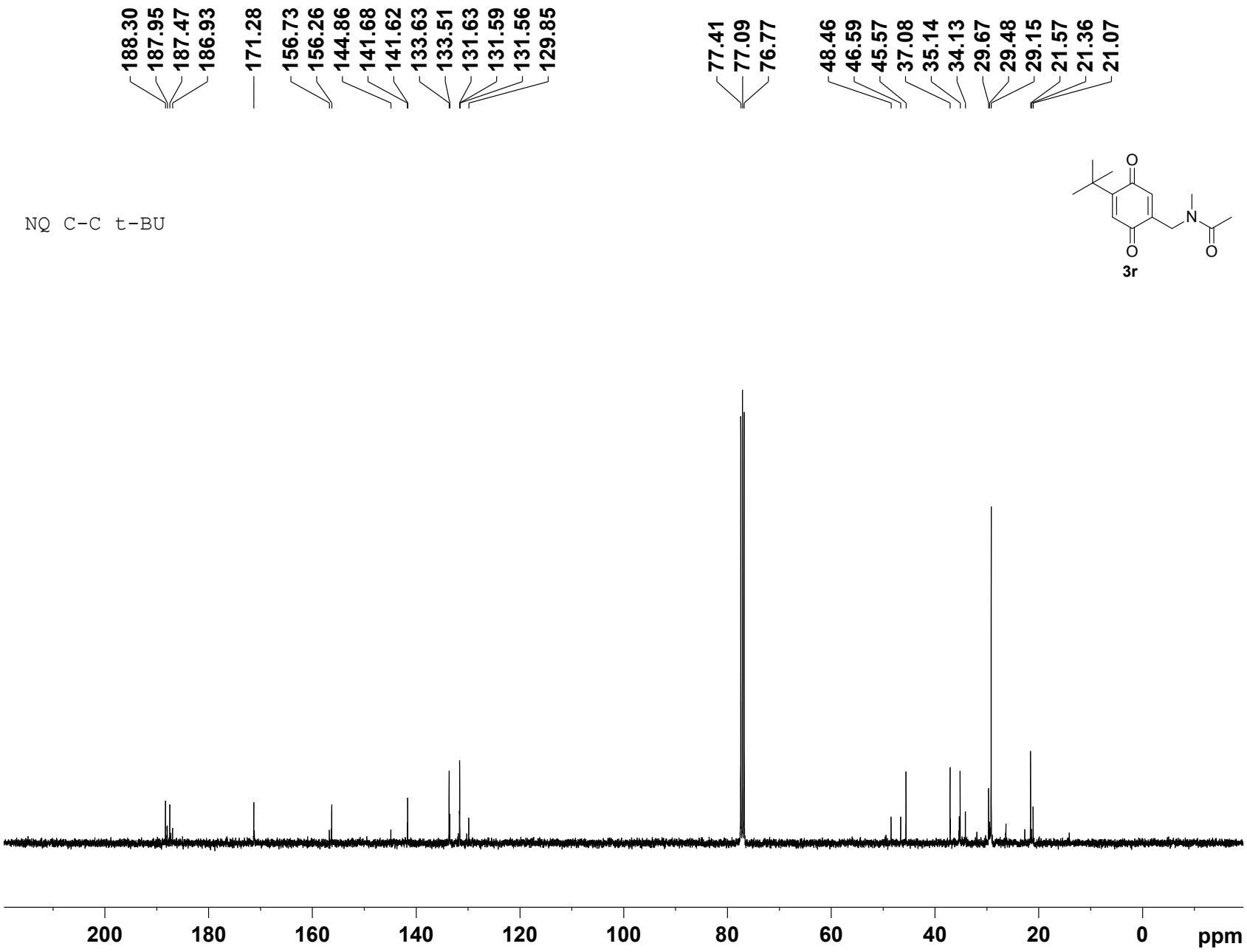
7.257
6.586
6.541
6.511
6.357
6.302
6.285
4.333
4.302
4.272
3.024
2.978
2.905
2.143
2.132
2.041
1.254
1.239
1.229

NQ C-C t-BU



3r

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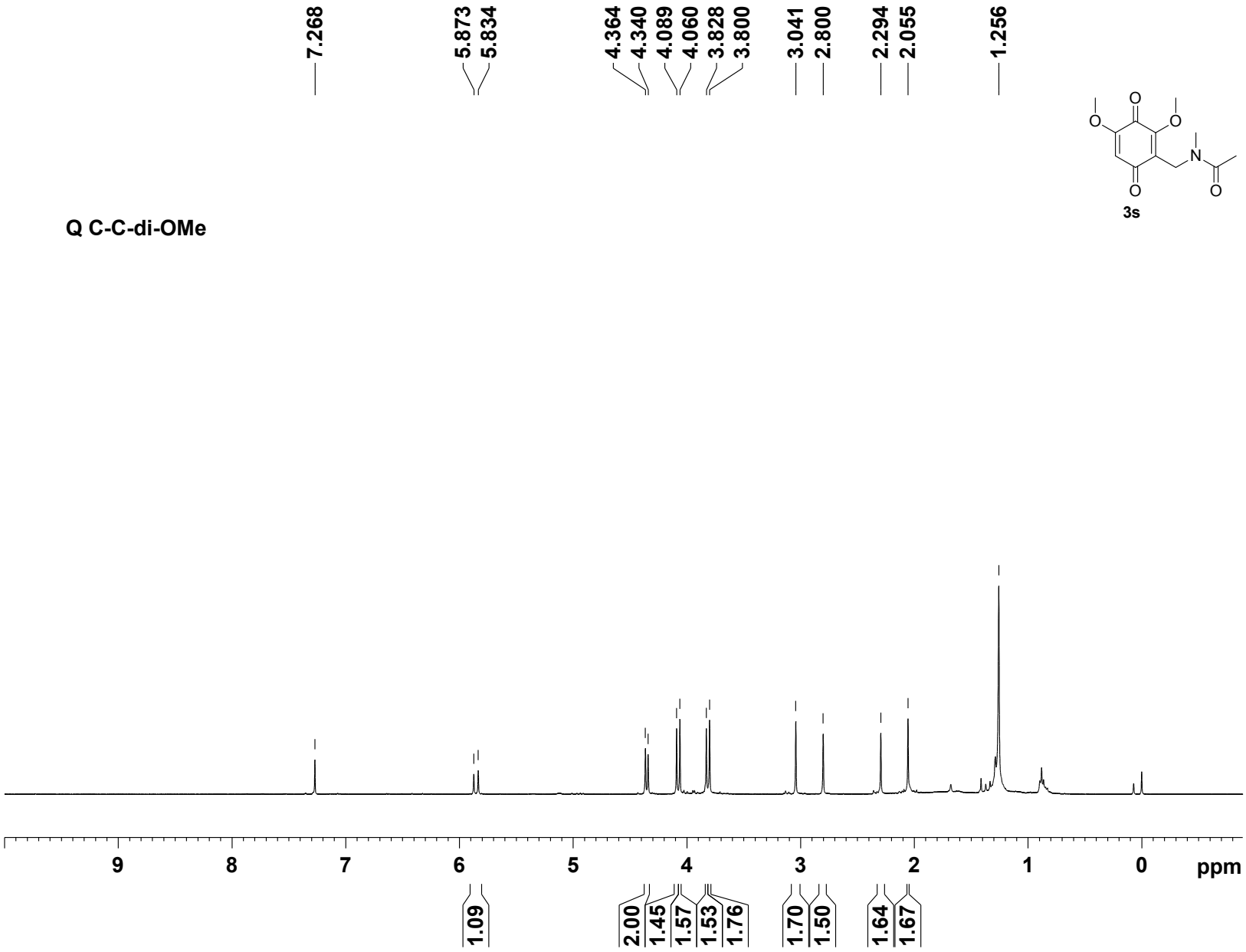
144.86
141.68
141.62
133.63
133.51
131.63
131.59
131.56
129.85
77.41
77.09
76.77
48.46
46.59
45.57
37.08
35.14
34.13
29.67
29.48
29.15
21.57
21.36
21.07
NQ C-C t-BU
O
O
N
O
3r
200
180
160
140
120
100
80
60
40
20
0
ppm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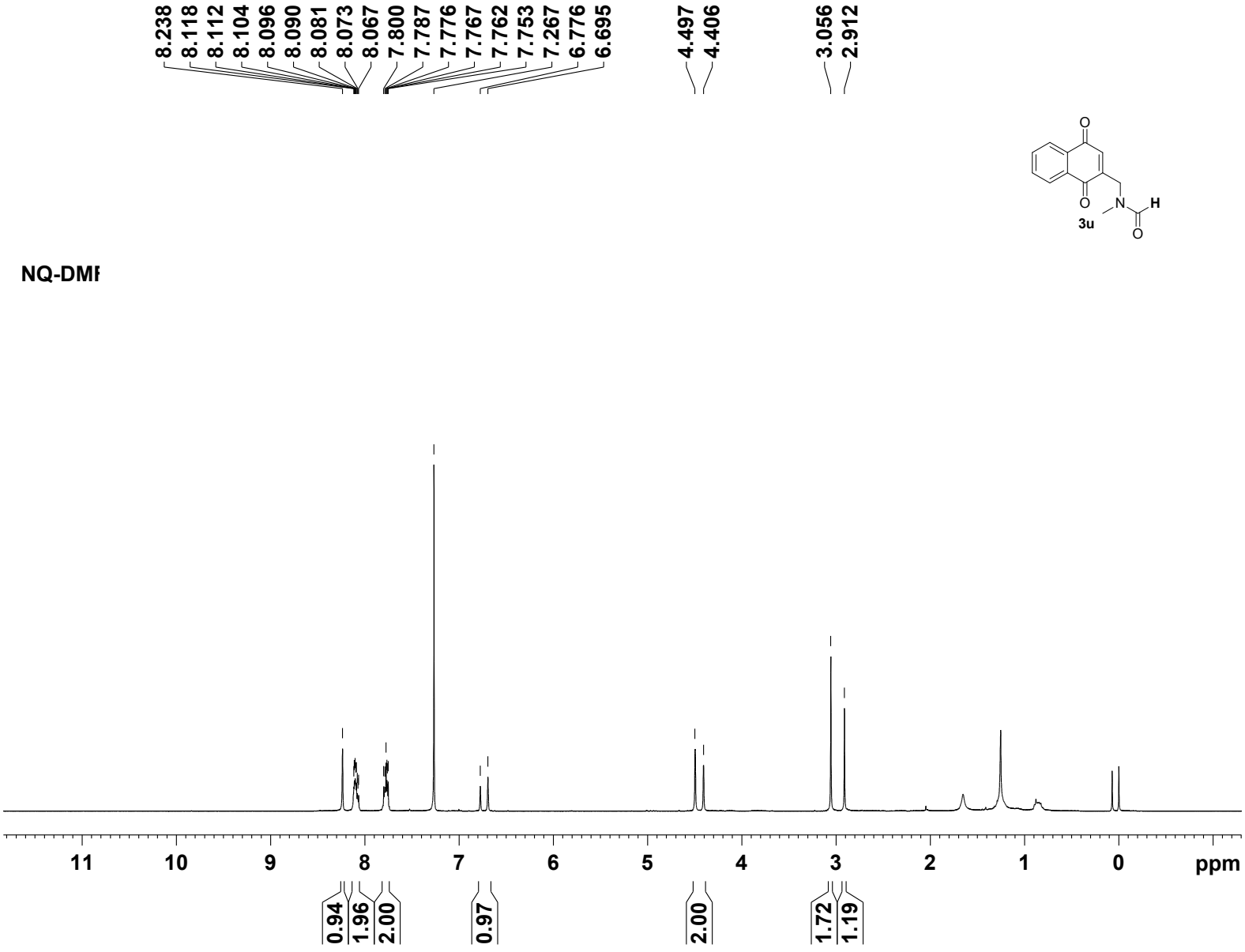
5.873
5.834
4.364
4.340
4.089
4.060
3.828
3.800
3.041
2.800
2.294
2.055
1.256
3s
Q C-C-di-OMe
9
8
7
6
5
4
3
2
1
0
ppm
1.09
2.00
1.45
1.57
1.53
1.76
1.70
1.50
1.64
1.67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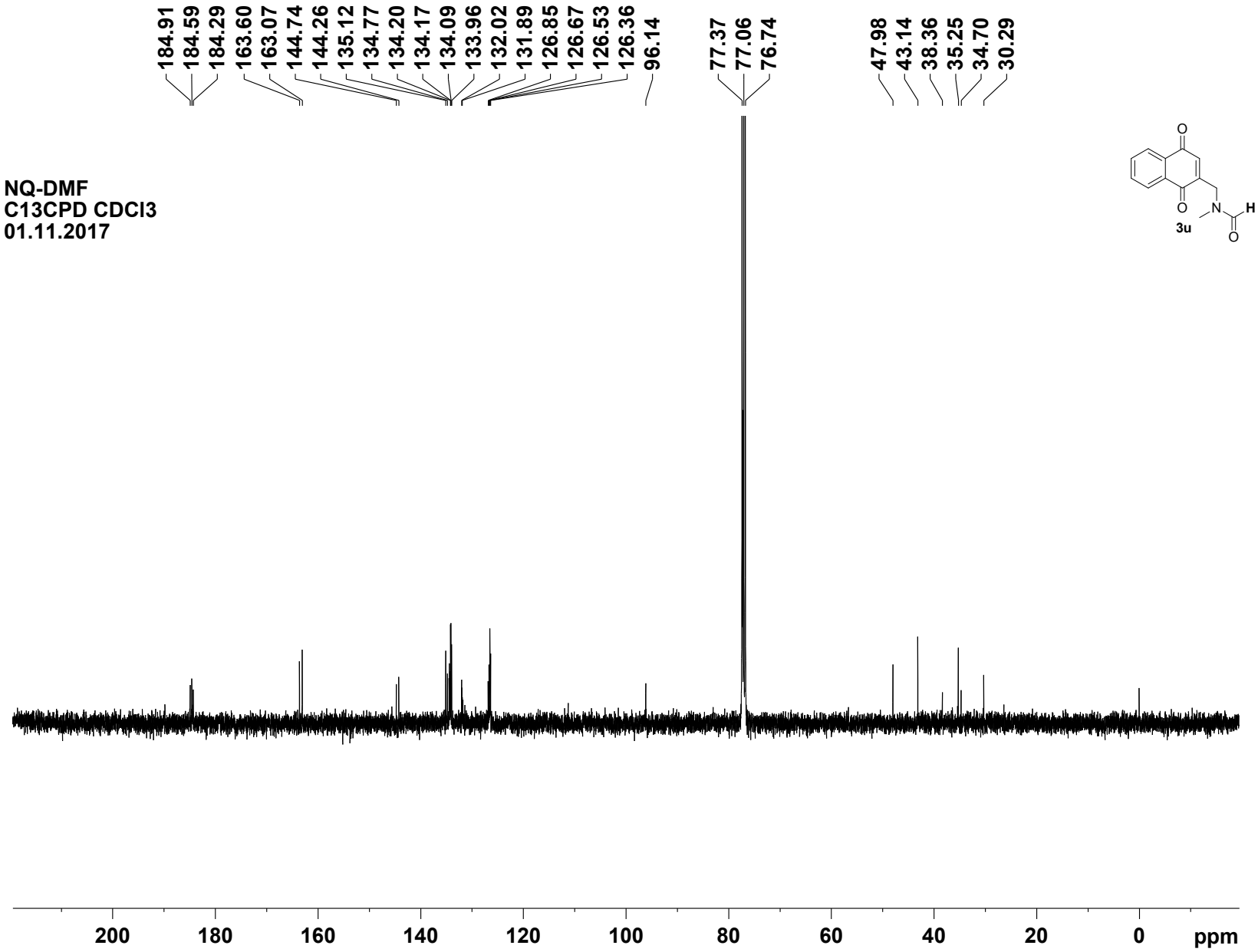
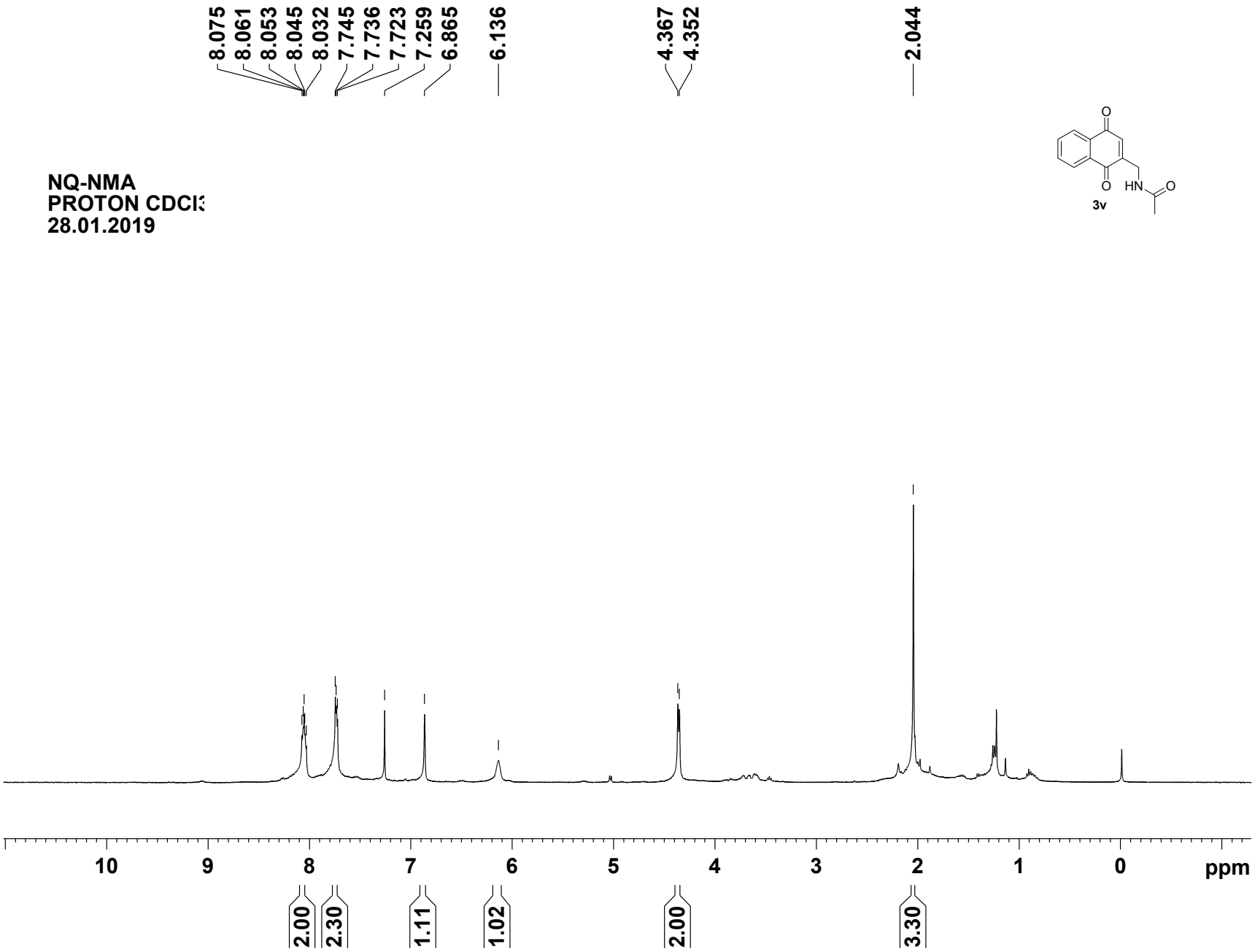
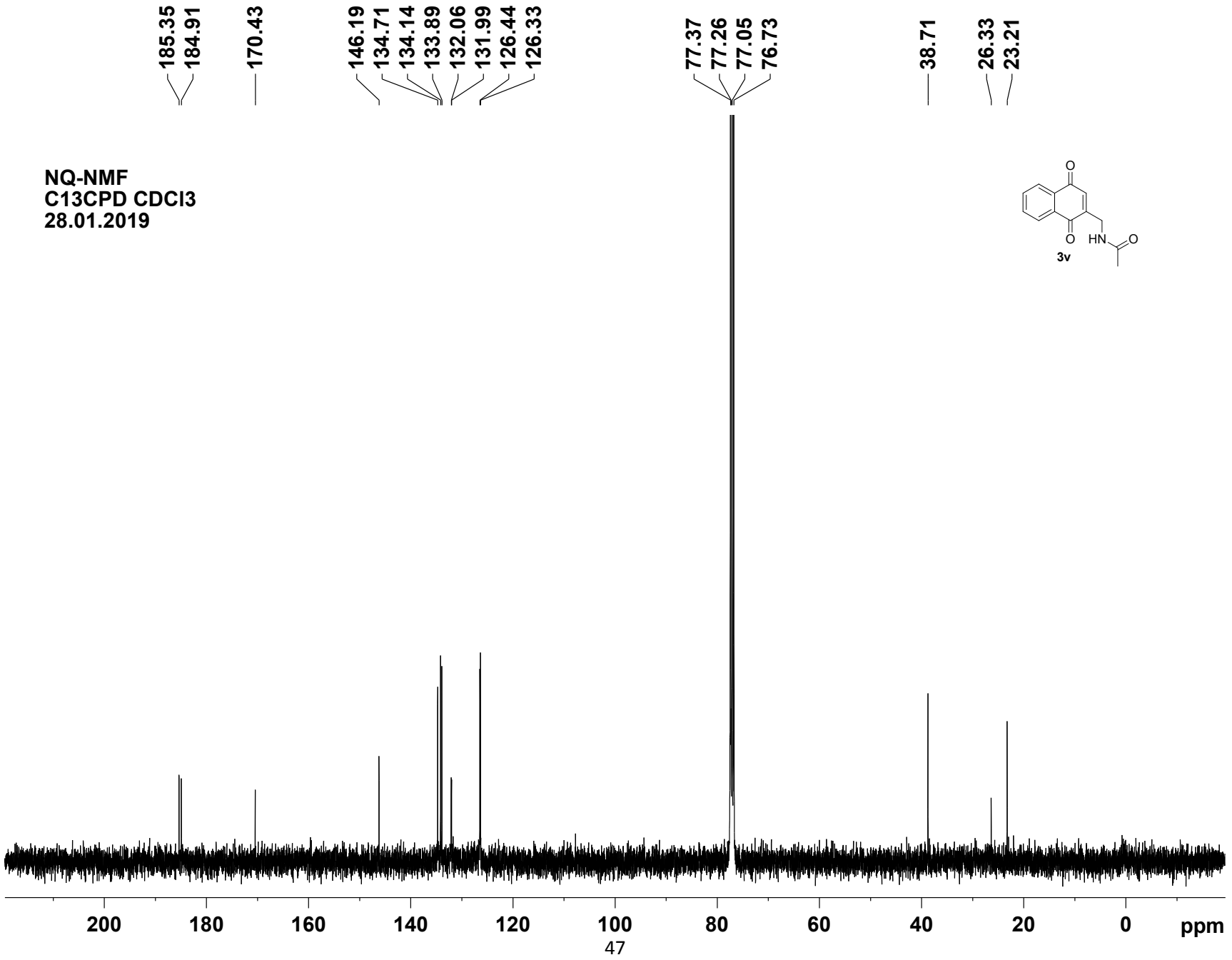
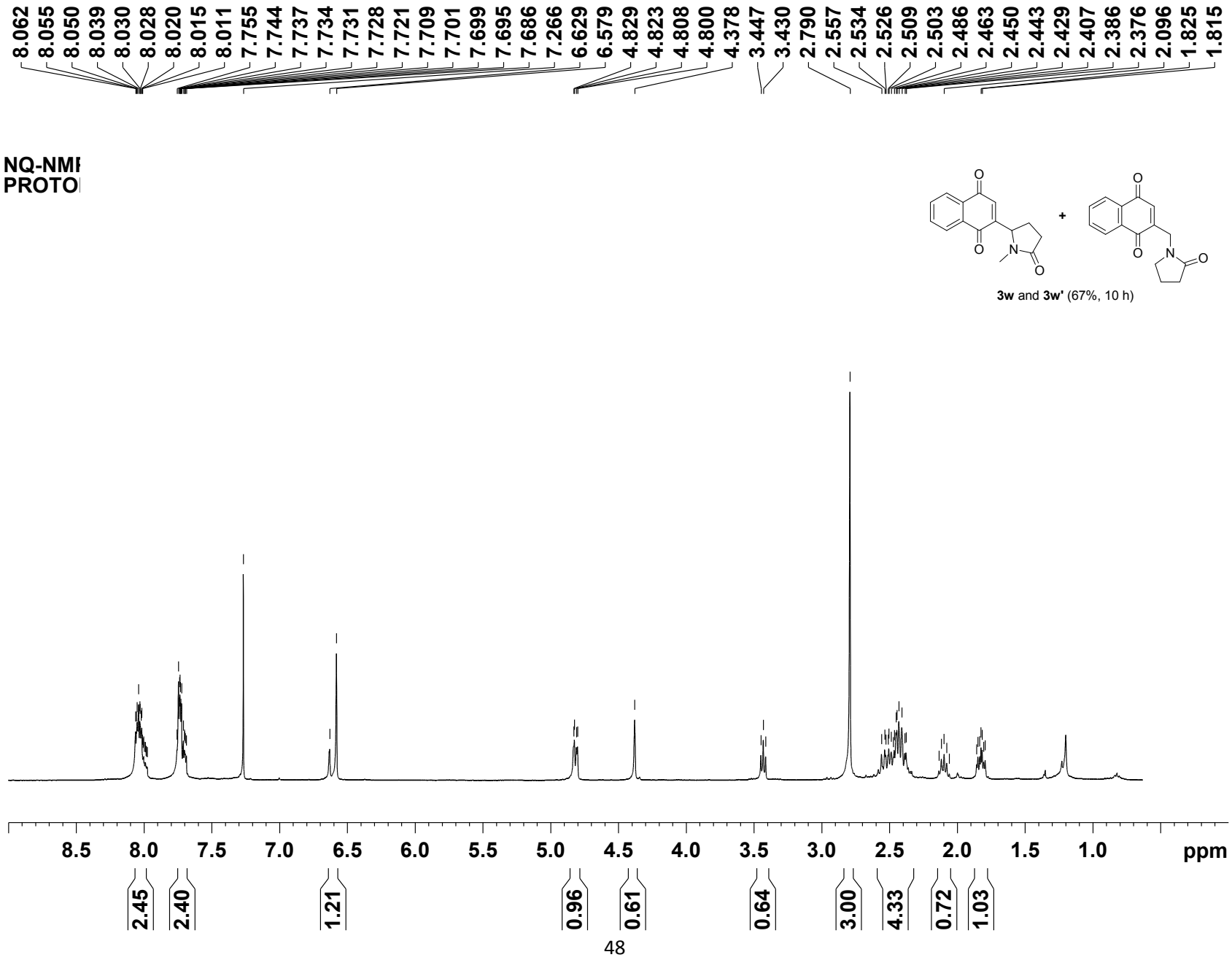
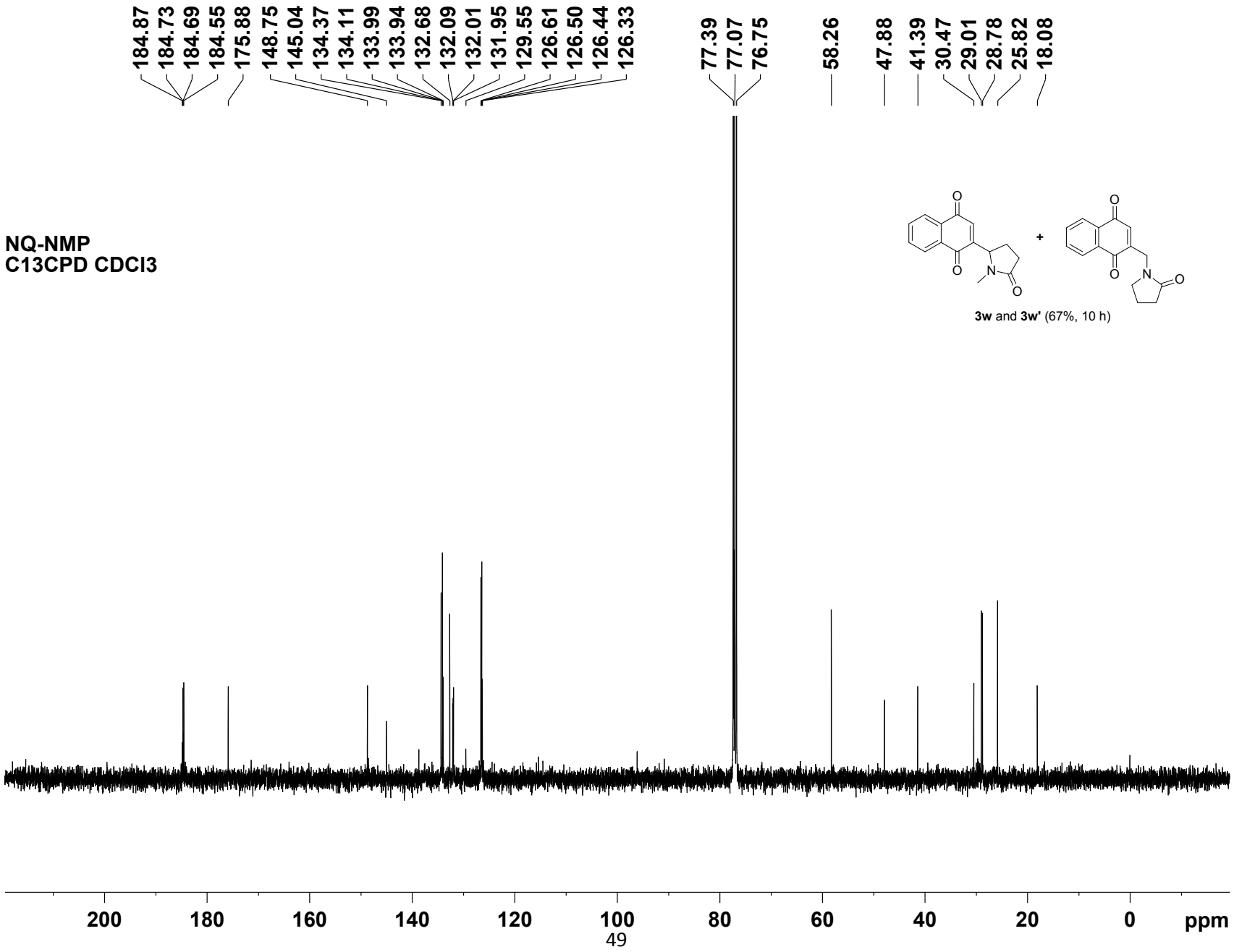
8.112
8.104
8.096
8.090
8.081
8.073
8.067
7.800
7.787
7.776
7.767
7.762
7.753
7.267
6.776
6.695
4.497
4.406
3.056
2.912
O
O
N
H
O
3u
NQ-DMF
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
1.96
2.00
0.97
2.00
1.72
1.19

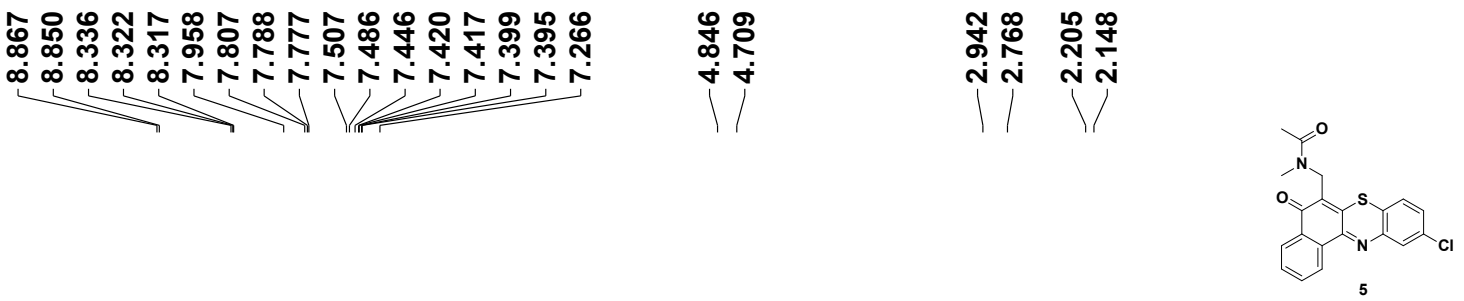

184.91
184.59
184.29
163.60
163.07
144.74
144.26
135.12
134.77
134.20
134.17
134.09
133.96
132.02
131.89
126.85
126.67
126.53
126.36
96.14
77.37
77.06
76.74
47.98
43.14
38.36
35.25
34.70
30.29
NQ-DMF
C13CPD CDCl3
01.11.2017
3u
200
180
160
140
120
100
80
60
40
20
0
ppm


8.075
8.061
8.053
8.045
8.032
7.745
7.736
7.723
7.259
6.865
6.136
4.367
4.352
2.044
NQ-NMA
PROTON CDCl3
28.01.2019
O
O
HN
O
3v
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.30
1.11
1.02
2.00
3.30

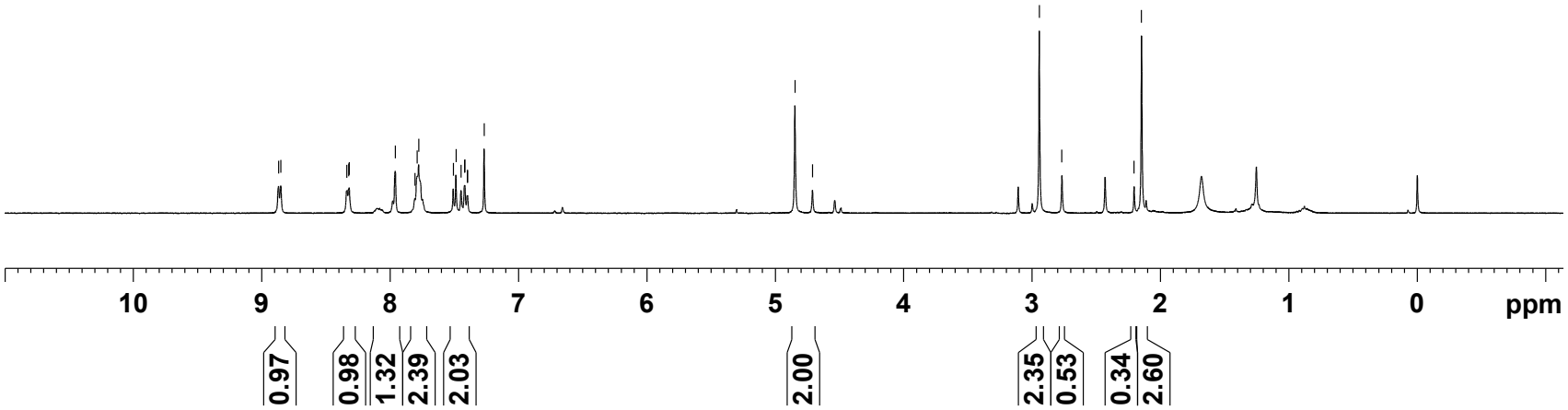

185.35
184.91
170.43
146.19
134.71
134.14
133.89
132.06
131.99
126.44
126.33
77.37
77.26
77.05
76.73
38.71
26.33
23.21
NQ-NMF
C13CPD CDCl3
28.01.2019
O
O
O
HN
O
3v
200
180
160
140
120
100
80
60
40
20
0
ppm

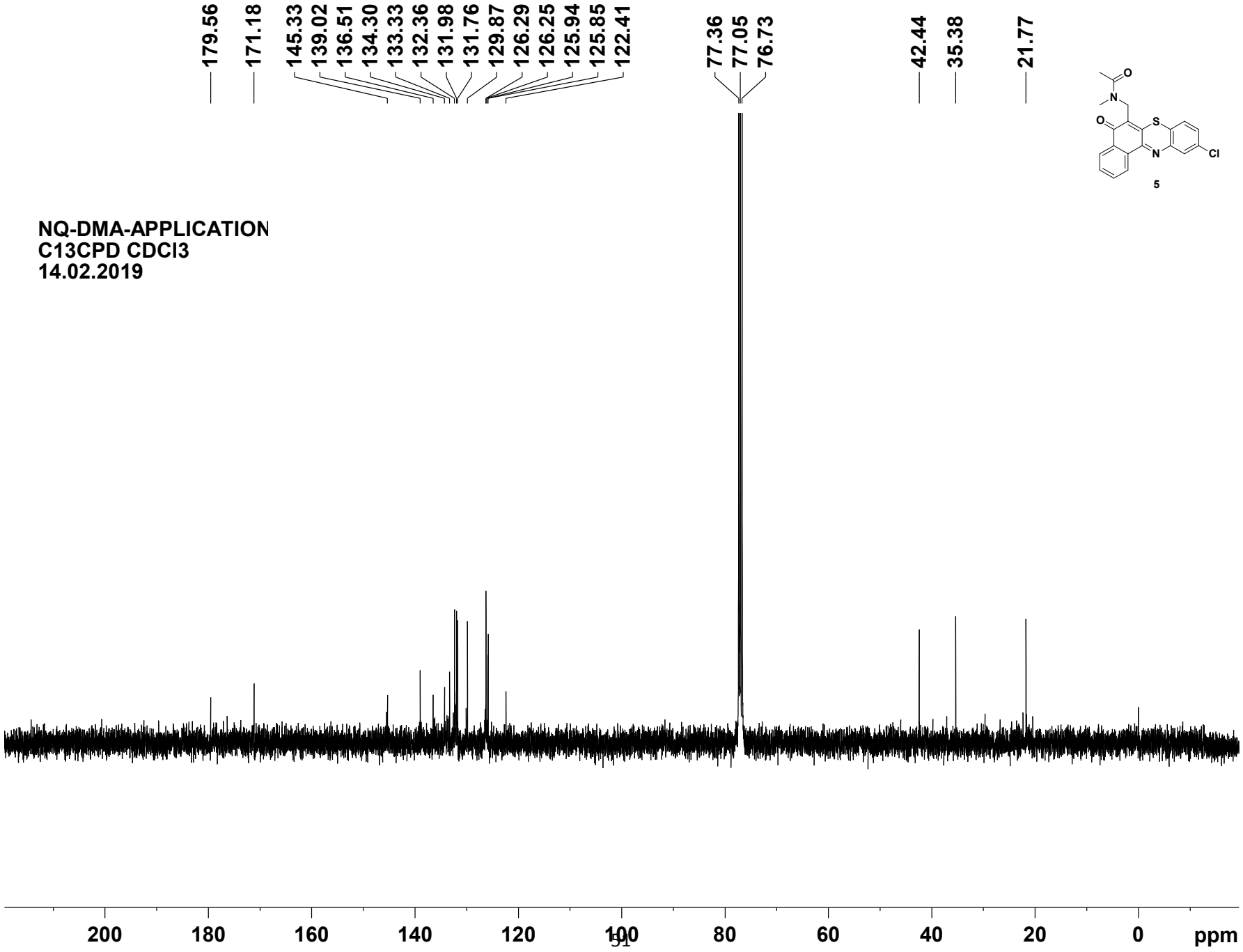

8.062
8.055
8.050
8.039
8.030
8.028
8.020
8.015
8.011
7.755
7.744
7.737
7.734
7.731
7.728
7.721
7.709
7.701
7.699
7.695
7.686
7.266
6.629
6.579
4.829
4.823
4.808
4.800
4.378
3.447
3.430
2.790
2.557
2.534
2.526
2.509
2.503
2.486
2.463
2.450
2.443
2.429
2.407
2.386
2.376
2.096
1.825
1.815
NQ-NMI
PROTO
3w and 3w' (67%, 10 h)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.45
2.40
1.21
0.96
0.61
0.64
3.00
4.33
0.72
1.03


184.87
184.73
184.69
184.55
175.88
148.75
145.04
134.37
134.11
133.99
133.94
132.68
132.09
132.01
131.95
129.55
126.61
126.50
126.44
126.33
77.39
77.07
76.75
58.26
47.88
41.39
30.47
29.01
28.78
25.82
18.08
NQ-NMP
C13CPD CDCl3
3w and 3w' (67%, 10 h)
200
180
160
140
120
100
80
60
40
20
0
ppm




**NQ-DMA-APPLICATION**




179.56
171.18
145.33
139.02
136.51
134.30
133.33
132.36
131.98
131.76
129.87
126.29
126.25
125.94
125.85
122.41
77.36
77.05
76.73
42.44
35.38
21.77
O
N
O
S
N
Cl
5
NQ-DMA-APPLICATION
C13CPD CDCl3
14.02.2019
200
180
160
140
120
100
80
60
40
20
0
ppm