Electronic Supplementary Material (ESI) for ChemComm.

A Palladium-Catalyzed Tandem Reaction of 2-(2-Bromobenzylidene)cyclobutanone with 2-Alkynylphenol

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Supporting Information

General experimental methods:

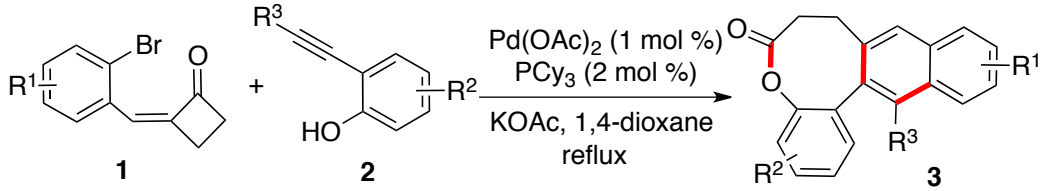
Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

*General experimental procedure for the synthesis of 2-alkylenecyclobutanone **1***

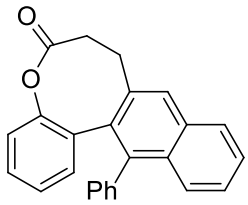
$Ca(OH)_2$ (10 mol %) was added to a solution of aldehyde (1.0 equiv) in ethanol. Then cyclobutanone (3.0 equiv) was injected via a syringe at N_2 atmosphere. The mixture was stirred at 80 ^{o}C for about 24 hours. After completion of conversion indicated by TLC, the solvent was evaporated under vacuum and the residue was purified through flash column chromatogram (petroleum ether: EtOAc 12:1) to give the desired 2-alkylenecyclobutanone. A typical example: (*E*)-2-(2-bromobenzylidene)cyclobutanone **(1a)**, ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 7.8 Hz, 2H), 7.46-7.42 (m, 1H), 7.40 (t, *J* =7.8 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 3.16 (t, *J* = 6.4 Hz, 2H), 2.97 (t, *J* = 7.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 148.2, 133.9, 133.6, 130.9, 129.1, 127.5, 127.1,124.9, 45.7, 23.3. (ref: L. Yu, Y. Wu, H. Cao, X. Zhang, X. Shi, J. Luan, T. Chen, Y. Pan and Q. Xu, *Green Chem.* 2014, **16**, 287.)

General experimental procedure for the palladium-catalyzed tandem reaction of

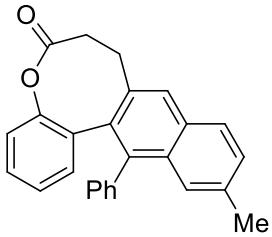
*2-(2-bromobenzylidene)cyclobutanone **1** with 2-alkynylphenol **2**:*



2-(2-bromobenzylidene)cyclobutanone **1** (1.0 mmol) was added to a mixture of 2-alkynylphenol **2** (1.2 mmol), $Pd(OAc)_2$ (1 mol %), PCy_3 (2 mol %) and KOAc (2.0 equiv) in 1,4-dioxane (10 mL) at N_2 atmosphere, and the suspension was heated to 100 °C. After the conversion was completed as indicated by TLC, the solvent was evaporated under reduced pressure and the residue was purified directly by flash column chromatograph (*n*-hexane/ethyl acetate = 15:1) to give the corresponding product **3**.

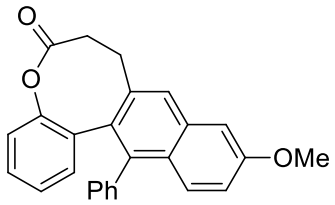


14-Phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3a**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 7.9 Hz, 1H), 7.81 (s, 1H), 7.57-7.49 (m, 2H), 7.39 (m, 3H), 7.26-7.20 (m, 2H), 7.15-7.07 (m, 2H), 7.01-6.97 (m, 1H), 6.88-6.86 (m, 2H), 3.07 (t, *J* = 9.6 Hz, 1H), 2.90 (t, *J* = 10.9 Hz, 1H), 2.80-2.78 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 150.2, 138.7, 137.8, 135.4, 133.6, 132.8, 132.7, 131.4, 131.2, 130.1, 129.3, 128.4, 127.7, 127.3, 127.1, 126.6, 126.3, 126.1, 125.8, 125.6, 125.4, 119.9, 33.4, 28.7. HRMS (ESI) calcd for $C_{25}H_{19}O_2^+$: 351.1380 ($M + H^+$), found: 351.1385.

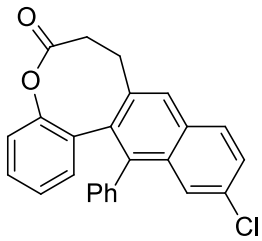


12-Methyl-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3b**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76-7.73 (m, 2H), 7.37-7.30 (m, 4H), 7.22-7.17 (m, 2H), 7.12-7.06 (m, 2H), 6.97-6.90 (m, 2H), 6.86-6.83 (m, 1H), 3.05-2.99 (m, 1H), 2.92-2.87

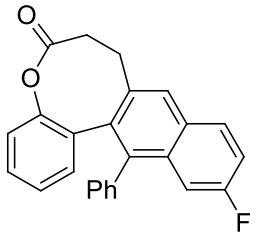
(m, 1H), 2.80-2.75 (m, 2H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.3, 150.9, 138.7, 138.6, 135.8, 134.7, 134.5, 133.4, 132.2, 131.8, 131.6, 130.7, 130.2, 128.8, 128.6, 128.2, 127.7, 127.5, 127.0, 126.7, 125.7, 125.6, 120.4, 34.0, 29.3, 21.9. HRMS (ESI) calcd for $C_{26}H_{21}O_2^+$: 365.1536 (M + H^+), found: 365.1544.



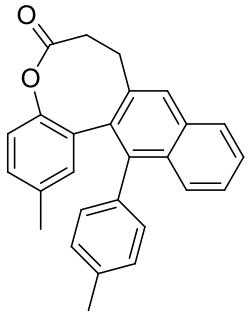
12-Methoxy-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3c**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (s, 1H), 7.47 (d, *J* = 9.2 Hz, 1H), 7.37-7.31 (m, 2H), 7.19-7.00 (m, 6H), 6.96-6.84 (m, 3H), 3.89 (s, 3H), 3.00 (t, *J* = 9.2 Hz, 1H), 2.90 (t, *J* = 10.8 Hz, 1H), 2.80-2.75 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.3, 157.9, 151.0, 139.3, 138.6, 136.4, 134.7, 134.3, 131.9, 131.0, 130.6, 130.1, 128.7, 128.4, 128.2, 127.4, 127.0, 126.8, 126.8, 125.7, 120.4, 118.6, 105.7, 55.3, 33.9, 29.4. HRMS (ESI) calcd for $C_{26}H_{21}O_3^+$: 381.1485 (M + H^+), found: 381.1478.



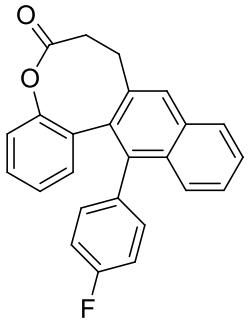
12-Chloro-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3d**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.76 (m, 2H), 7.55 (s, 1H), 7.44-7.41 (m, 1H), 7.38-7.36 (m, 2H), 7.24-7.21 (m, 2H), 7.14-7.08 (m, 2H), 6.97 (t, *J* = 7.4 Hz, 1H), 6.90-6.84 (m, 2H), 3.04 (t, *J* = 9.7 Hz, 1H), 2.91 (t, *J* = 10.4 Hz, 1H), 2.82-2.76 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 150.8, 138.8, 137.7, 136.2, 134.5, 133.9, 132.8, 132.1, 131.6, 130.5, 130.1, 129.3, 129.1, 128.5, 127.8, 127.3, 127.3, 127.2, 125.9, 125.6, 120.6, 33.9, 29.3. HRMS (ESI) calcd for $C_{25}H_{18}ClO_2^+$: 385.0990 (M + H^+), found: 385.0970.



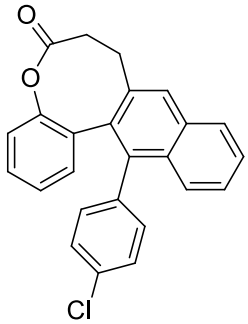
12-Fluoro-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3e**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (s, 1H), 7.58-7.54 (m, 1H), 7.47-7.44 (m, 1H), 7.36-7.35 (m, 2H), 7.25-7.21 (m, 2H), 7.14-7.07 (m, 3H), 6.99-6.95 (m, 1H), 6.91-6.84 (m, 2H), 3.07-3.02 (m, 1H), 2.94-2.89 (m, 1H), 2.82-2.74 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 161.0 (d, J_{CF} = 246.0 Hz), 151.0, 139.7, 138.2, 137.2, 134.4 (d, $^{3}J_{CF}$ = 9.5 Hz), 134.0, 132.7 (d, $^{4}J_{CF}$ = 1.9 Hz), 131.8, 130.6, 130.1, 129.6 (d, $^{3}J_{CF}$ = 8.8 Hz) 129.2, 129.0, 128.4, 127.3, 127.2, 127.1, 125.9, 120.5, 116.3 (d, $^{2}J_{CF}$ = 24.8 Hz), 110.6 (d, $^{2}J_{CF}$ = 20.4 Hz), 33.9, 29.4.HRMS (ESI) calcd for $C_{25}H_{18}FO_2^+$: 369.1285 ($M + H^+$), found: 369.1279.



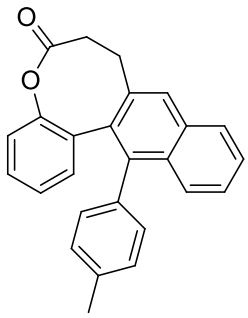
2-Methyl-14-(*p*-tolyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3f**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.0 Hz, 1H), 7.76 (s, 1H), 7.59 (d, *J* = 8.3 Hz, 1H), 7.47 (t, *J* = 7.1 Hz, 1H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.28-7.24 (m, 2H), 7.17-7.16 (m, 1H), 7.00 (s, 1H), 6.92-6.90 (m, 1H), 6.81-6.79 (m, 1H), 6.67 (s, 1H), 3.07-3.02 (m, 1H), 2.92-2.88 (m, 1H), 2.84-2.79 (m, 2H), 2.30 (s, 3H), 2.11 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 148.8, 139.4, 136.3, 135.8, 135.5, 134.0, 133.5, 133.3, 132.3, 132.3, 130.6, 130.0, 129.3, 129.0, 127.7, 127.6, 126.9, 126.2, 125.9, 120.4, 33.9, 29.4, 21.1, 20.6. HRMS (ESI) calcd for $C_{27}H_{23}O_2^+$: 379.1693 ($M + H^+$), found: 379.1691.



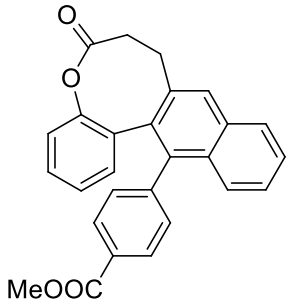
14-(4-Fluorophenyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3g**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 8.1 Hz, 1H), 7.79 (s, 1H), 7.54 (d, J = 8.4 Hz, 1H), 7.47 (t, J = 7.1 Hz, 1H), 7.38-7.33 (m, 2H), 7.25-7.21 (m, 1H), 7.14-7.12 (m, 1H), 7.07-6.97 (m, 2H), 6.90-6.83 (m, 2H), 6.80-6.75 (m, 1H), 3.07-2.99 (m, 1H), 2.93-2.88 (m, 1H), 2.81-2.73 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 161.0 (d, J_{CF} = 246.0 Hz), 151.0, 139.7, 138.2, 137.2, 134.4 (d, $^{3}J_{CF}$ = 9.5 Hz), 134.0, 132.7 (d, $^{4}J_{CF}$ = 1.9 Hz), 131.8, 130.6, 130.1, 129.6 (d, $^{3}J_{CF}$ = 8.8 Hz), 129.2, 129.0, 128.4, 127.3, 127.2, 127.1, 125.9, 120.5, 116.3 (d, $^{2}J_{CF}$ = 24.8 Hz), 110.6 (d, $^{2}J_{CF}$ = 20.4 Hz), 33.9, 29.4. HRMS (ESI) calcd for $C_{25}H_{18}FO_2^+$: 369.1285 ($M + H^+$), found: 369.1282.



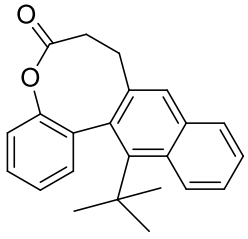
14-(4-Chlorophenyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3h**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J = 8.1 Hz, 1H), 7.80 (s, 1H), 7.53-7.47 (m, 2H), 7.40-7.34 (m, 3H), 7.28-7.24 (m, 1H), 7.15-7.13 (m, 1H), 7.08-7.00 (m, 2H), 6.86-6.84 (m, 2H), 3.06 (t, J = 10.2 Hz, 1H), 2.92 (t, J = 8.5 Hz, 1H), 2.82-2.74 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.6, 150.9, 138.0, 137.0, 135.8, 134.0, 133.5, 133.4, 132.9, 132.0, 131.9, 131.7, 131.6, 129.2, 128.6, 128.3, 127.8, 127.4, 126.5, 126.4, 126.3, 126.1, 120.6, 33.9, 29.3. HRMS (ESI) calcd for $C_{25}H_{18}ClO_2^+$: 385.0990 ($M + H^+$), found: 385.0981.



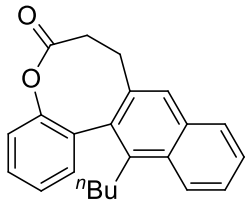
14-(*p*-tolyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3i**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.1 Hz, 1H), 7.76 (s, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.36-7.32 (m, 1H), 7.28-7.11 (m, 4H), 6.97 (t, *J* = 7.4 Hz, 1H), 6.90-6.86 (m, 2H), 6.81-6.79 (m, 1H), 3.06-3.00 (m, 1H), 2.93-2.86 (m, 1H), 2.81-2.72 (m, 2H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.3, 150.9, 139.4, 136.3, 135.7, 135.3, 134.4, 133.3, 133.3, 132.2, 131.8, 130.5, 130.0, 129.0, 128.8, 127.8, 127.6, 126.8, 126.2, 126.0, 125.9, 120.4, 34.0, 29.3, 21.1. HRMS (ESI) calcd for $C_{26}H_{21}O_2^+$: 365.1536 (M + H^+), found: 365.1530.



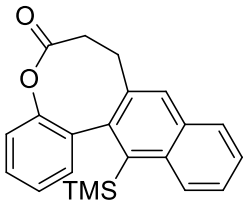
Methyl 4-(6-oxo-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-14-yl)benzoate (**3j**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08-8.05 (m, 1H), 7.87-7.76 (m, 3H), 7.50-7.48 (m, 3H), 7.39-7.35 (m, 1H), 7.25-7.21 (m, 1H), 7.15-7.13 (m, 1H), 7.02-6.95 (m, 2H), 6.85-6.83 (m, 1H), 3.88 (s, 3H), 3.10-3.04 (m, 1H), 2.95-2.90 (m, 1H), 2.80-2.77 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 166.8, 150.8, 143.6, 138.2, 135.8, 133.8, 133.3, 131.6, 131.5, 130.8, 130.3, 129.5, 129.2, 128.6, 128.4, 127.8, 126.5, 126.3, 126.3, 126.0, 120.6, 52.0, 33.8, 29.2. HRMS (ESI) calcd for $C_{27}H_{21}O_4^+$: 409.1434 (M + H^+), found: 409.1432.



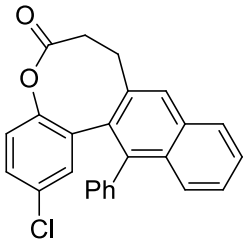
14-(Tert-butyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3k**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.52-8.49 (m, 1H), 7.78-7.75 (m, 1H), 7.59 (s, 1H), 7.44-7.41 (m, 3H), 7.28-7.23 (m, 2H), 7.08-7.06 (m, 1H), 2.97-2.76 (m, 3H), 2.40-2.32 (m, 1H), 1.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.0, 149.7, 145.0 138.5, 136.8, 134.6, 133.0, 132.4, 131.4, 129.2, 128.8, 128.2, 127.5, 125.9, 125.1, 124.0, 121.0, 38.5, 36.2, 34.1, 29.7. HRMS (ESI) calcd for $C_{23}H_{23}O_2^+$: 331.1693 ($M + H^+$), found: 331.1686.



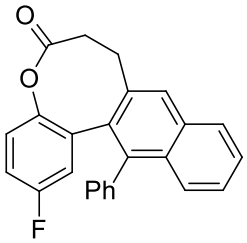
14-Butyl-7,8-dihydro-6H-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3l**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, *J* = 8.0 Hz, 1H), 7.79-7.77 (m, 1H), 7.60 (s, 1H), 7.51-7.42 (m, 3H), 7.36-7.35 (m, 2H), 7.25 (d, *J* = 8.0 Hz, 1H), 2.95-2.87 (m, 2H), 2.84-2.79 (m, 2H), 2.72-2.64 (m, 2H), 1.57-1.51 (m, 2H), 1.28-1.23 (m, 2H), 0.77 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 150.7, 137.8, 135.9, 134.4, 133.6, 133.0, 131.4, 131.0, 129.4, 128.4, 126.5, 126.2, 125.9, 125.9, 124.5, 121.3, 34.2, 32.9, 29.5, 29.4, 22.8, 13.6. HRMS (ESI) calcd for $C_{23}H_{23}O_2^+$: 331.1693 ($M + H^+$), found: 331.1685.



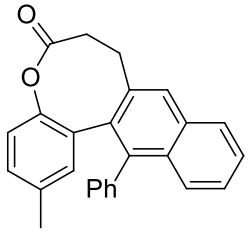
14-(Trimethylsilyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3m**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.29-8.27 (m, 1H), 7.87-7.85 (m, 1H), 7.79 (s, 1H), 7.55-7.48 (m, 3H), 7.39-7.35 (m, 1H), 7.30-7.23 (m, 2H), 3.00-2.84 (m, 3H), 2.66-2.57 (m, 1H), 0.17 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 150.5, 142.2, 137.4, 137.2, 137.0, 135.8, 133.1, 131.9, 129.9, 129.8, 128.6, 128.5, 126.2, 125.6, 125.2, 121.0, 35.0, 29.1, 2.22. HRMS (ESI) calcd for $C_{26}H_{19}ClFN_2O_2^+$: 445.1114 ($M + H^+$), found: 445.1115.



2-Chloro-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3n**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.0 Hz, 1H), 7.79 (s, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.50 (t, *J* = 7.1 Hz, 1H), 7.41-7.39 (m, 3H), 7.26-7.24 (m, 1H), 7.19-7.11 (m, 2H), 7.08-7.06 (m, 1H), 6.91-6.86 (m, 2H), 3.11-3.06 (m, 1H), 2.96-2.90 (m, 1H), 2.86-2.74 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 149.5, 139.6, 138.0, 136.0, 135.2, 133.5, 132.0, 132.0, 131.7, 131.0, 130.6, 129.8, 128.9, 128.5, 128.2, 127.7, 127.3, 127.2, 126.9, 126.6, 126.3, 33.8, 29.2. HRMS (ESI) calcd for $C_{25}H_{18}ClO_2^+$: 385.0990 (M + H^+), found: 385.0972.

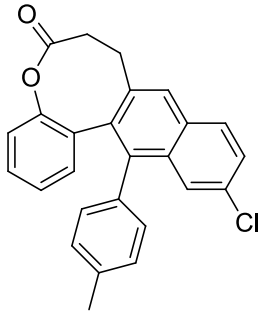


2-Fluoro-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3o**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.1 Hz, 1H), 7.79 (s, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.40-7.38 (m, 3H), 7.24 (s, 1H), 7.15-7.08 (m, 2H), 6.93-6.91 (m, 2H), 6.60-6.58 (m, 1H), 3.11-3.02 (m, 1H), 2.99-2.90 (m, 1H), 2.84-2.74 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 159.7 (d, J_{CF} = 245.0 Hz), 147.0, 139.5, 138.0, 136.2 (d, $^{3}J_{CF}$ = 8.5 Hz), 135.3, 132.1 (d, $^{2}J_{CF}$ = 28.0 Hz), 130.6, 130.0, 128.6 128.5, 128.2, 127.7, 127.3, 127.2, 127.1, 126.7 (d, $^{2}J_{CF}$ = 21.7 Hz), 124.8, 121.9 (d, $^{3}J_{CF}$ = 8.9 Hz), 118.3 (d, $^{2}J_{CF}$ = 23.4 Hz), 115.6 (d, $^{2}J_{CF}$ = 23.3 Hz), 33.8, 29.2. HRMS (ESI) calcd for $C_{25}H_{18}FO_2^+$: 369.1285 (M + H^+), found: 369.1285.

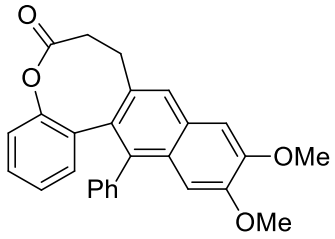


2-Methyl-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3p**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.1 Hz, 1H), 7.77 (s, 1H), 7.57 (d, *J* = 8.1 Hz, 1H),

7.48-7.44 (m, 1H), 7.39-7.33 (m, 3H), 7.22-7.17 (m, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.99 (s, 2H), 6.92 (d, *J* = 7.6 Hz, 1H), 6.65 (s, 1H), 3.07-3.03 (m, 1H), 2.91-2.86 (m, 1H), 2.85-2.78 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.6, 148.8, 139.4, 138.5, 135.7, 135.4, 133.8, 133.5, 133.3, 132.3, 132.0, 130.7, 130.1, 129.3, 128.2, 127.9, 127.6, 126.9, 126.8, 126.8, 126.2, 126.0, 120.0, 33.8, 29.4, 20.5. HRMS (ESI) calcd for $C_{26}H_{21}O_2^+$: 365.1536 ($M + H^+$), found: 365.1530.

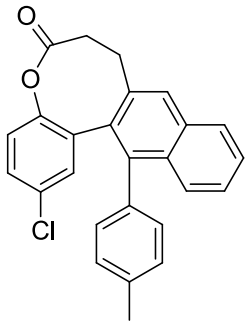


12-Chloro-14-(*p*-tolyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3q**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78-7.74 (m, 2H), 7.57 (s, 1H), 7.43-7.40 (m, 1H), 7.25-7.23 (m, 2H), 7.19-7.13 (m, 2H), 6.99 (t, *J* = 7.5 Hz, 1H), 6.91-6.84 (m, 2H), 6.78-6.76 (m, 1H), 3.06-3.00 (m, 1H), 2.94-2.86 (m, 1H), 2.82-2.75 (m, 2H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 150.8, 138.9, 136.7, 136.2, 134.6, 134.1, 133.0, 132.0, 131.7, 130.4, 130.0, 129.3, 129.2, 129.2, 129.0, 128.3, 128.1, 127.6, 127.2, 126.0, 125.7, 120.5, 33.9, 29.3, 21.2. HRMS (ESI) calcd for $C_{26}H_{20}ClO_2^+$: 399.1146 ($M + H^+$), found: 399.1137.

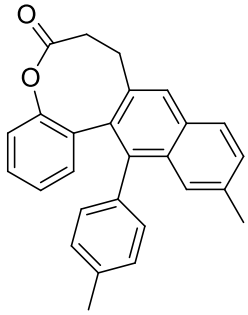


11,12-Dimethoxy-14-phenyl-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3r**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (s, 1H), 7.41-7.34 (m, 2H), 7.23-7.17 (m, 2H), 7.14-7.07 (m, 3H), 6.98-6.84 (m, 4H), 4.01 (s, 3H), 3.71 (s, 3H), 3.03-2.98 (m, 1H), 2.93-2.88 (m, 1H), 2.81-2.76 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.5, 151.0, 149.8, 149.4, 138.8, 138.0, 134.6, 134.1, 131.9, 131.6, 130.3, 130.2, 129.4, 128.7, 128.4, 127.6, 127.1, 126.8, 126.4, 125.7, 120.4 , 106.1, 105.6, 55.9, 55.6, 34.0, 29.3. HRMS

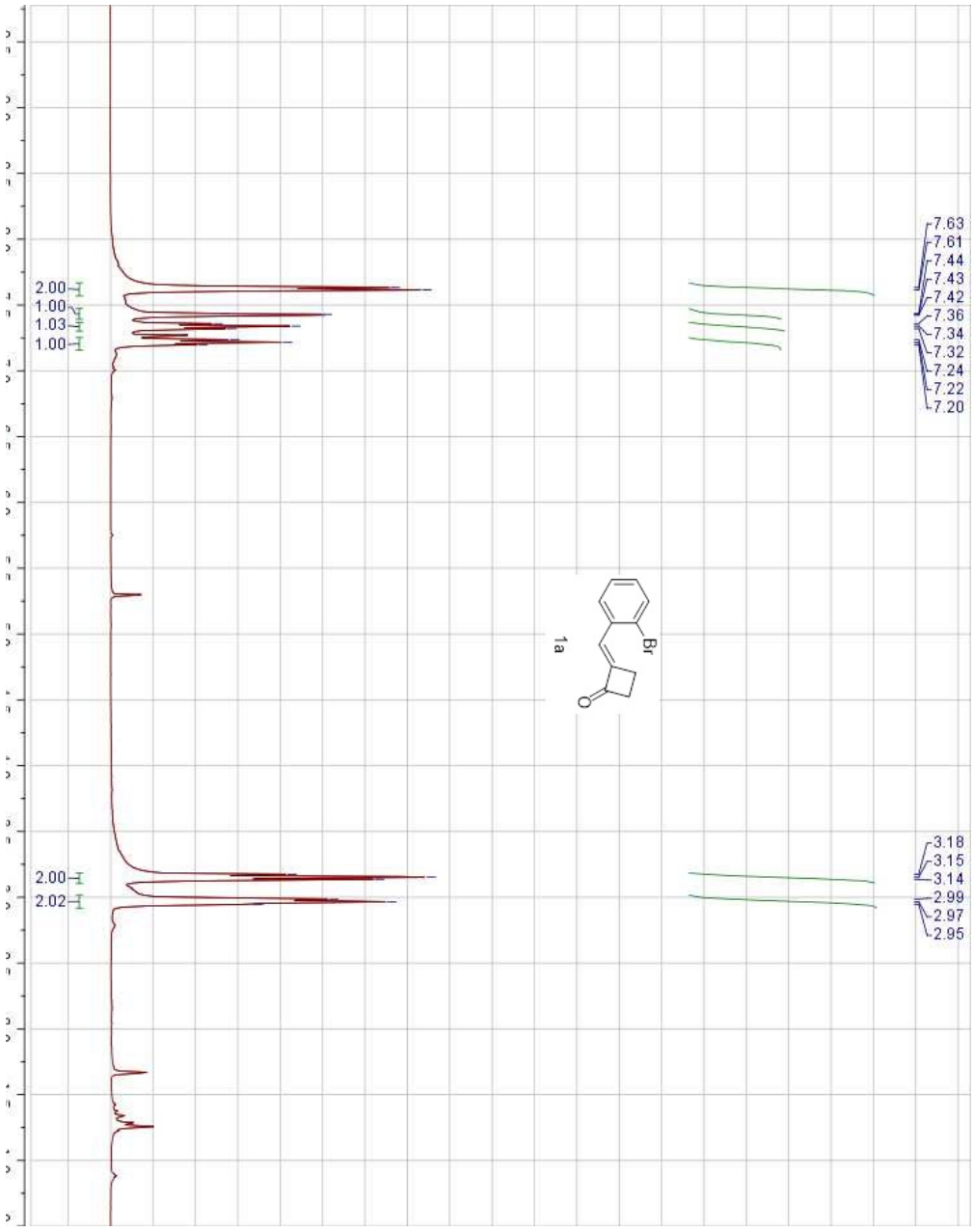
(ESI) calcd for $C_{27}H_{23}O_4^+$: 411.1591 ($M + H^+$), found: 411.1585.



2-Chloro-14-(*p*-tolyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3s**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.1 Hz, 1H), 7.78 (s, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 7.52-7.48 (m, 1H), 7.40-7.35 (m, 1H), 7.28-7.25 (m, 1H), 7.22-7.19 (m, 2H), 7.09-7.08 (m, 1H), 6.95-6.93 (m, 1H), 6.88-6.87 (m, 1H), 6.79-6.77 (m, 1H), 3.10-3.05 (m, 1H), 2.97-2.91 (m, 1H), 2.85-2.74 (m, 2H), 2.33 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 149.5, 139.7, 136.8, 136.2, 135.2, 134.9, 133.5, 132.2, 132.0, 131.7, 131.1, 130.5, 129.7, 129.2, 128.8, 128.1, 128.0, 127.7, 127.0, 126.6, 126.2, 121.8, 33.9, 29.3, 21.2. HRMS (ESI) calcd for $C_{26}H_{20}ClO_2^+$: 399.1146 ($M + H^+$), found: 399.1135.



12-Methyl-14-(*p*-tolyl)-7,8-dihydro-6*H*-benzo[*b*]naphtho[2,3-*d*]oxocin-6-one (**3t**). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73-7.70 (m, 2H), 7.36 (s, 1H), 7.30-7.25 (m, 2H), 7.20-7.09 (m, 3H), 6.97 (t, *J* = 7.4 Hz, 1H), 6.89-6.84 (m, 2H), 6.80-6.78 (m, 1H), 3.02-3.96 (m, 1H), 2.92-2.83 (m, 1H), 2.79-2.69 (m, 2H), 2.35 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.5, 151.0, 138.8, 136.3, 135.8, 135.6, 134.9, 134.7, 133.5, 132.4, 131.9, 131.7, 130.7, 130.1, 129.1, 128.8, 128.6, 127.9, 127.6, 126.0, 125.8, 120.5, 34.2, 29.4, 22.0, 21.3. HRMS (ESI) calcd for $C_{27}H_{23}O_2^+$: 379.1693 ($M + H^+$), found: 379.1684.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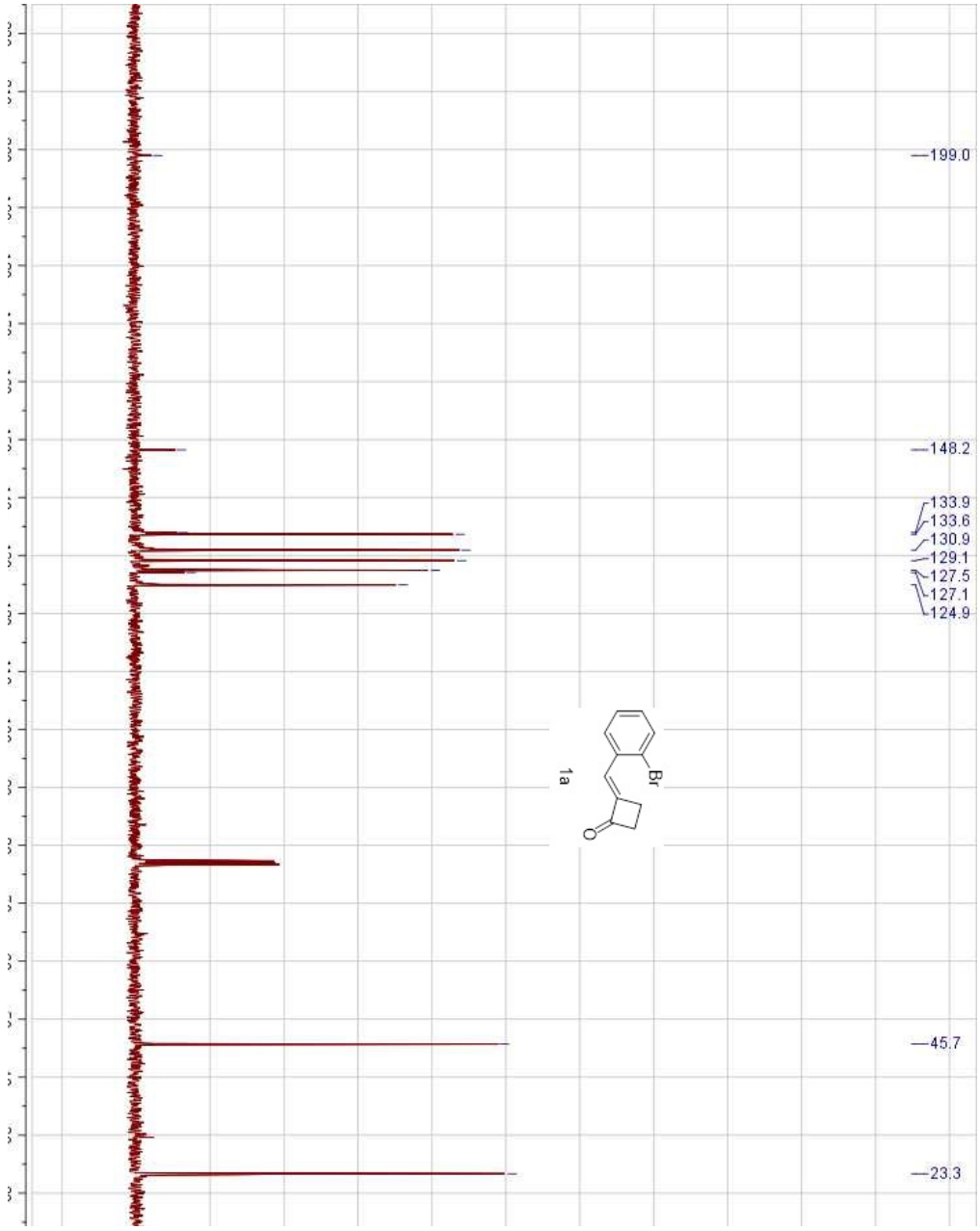
2.02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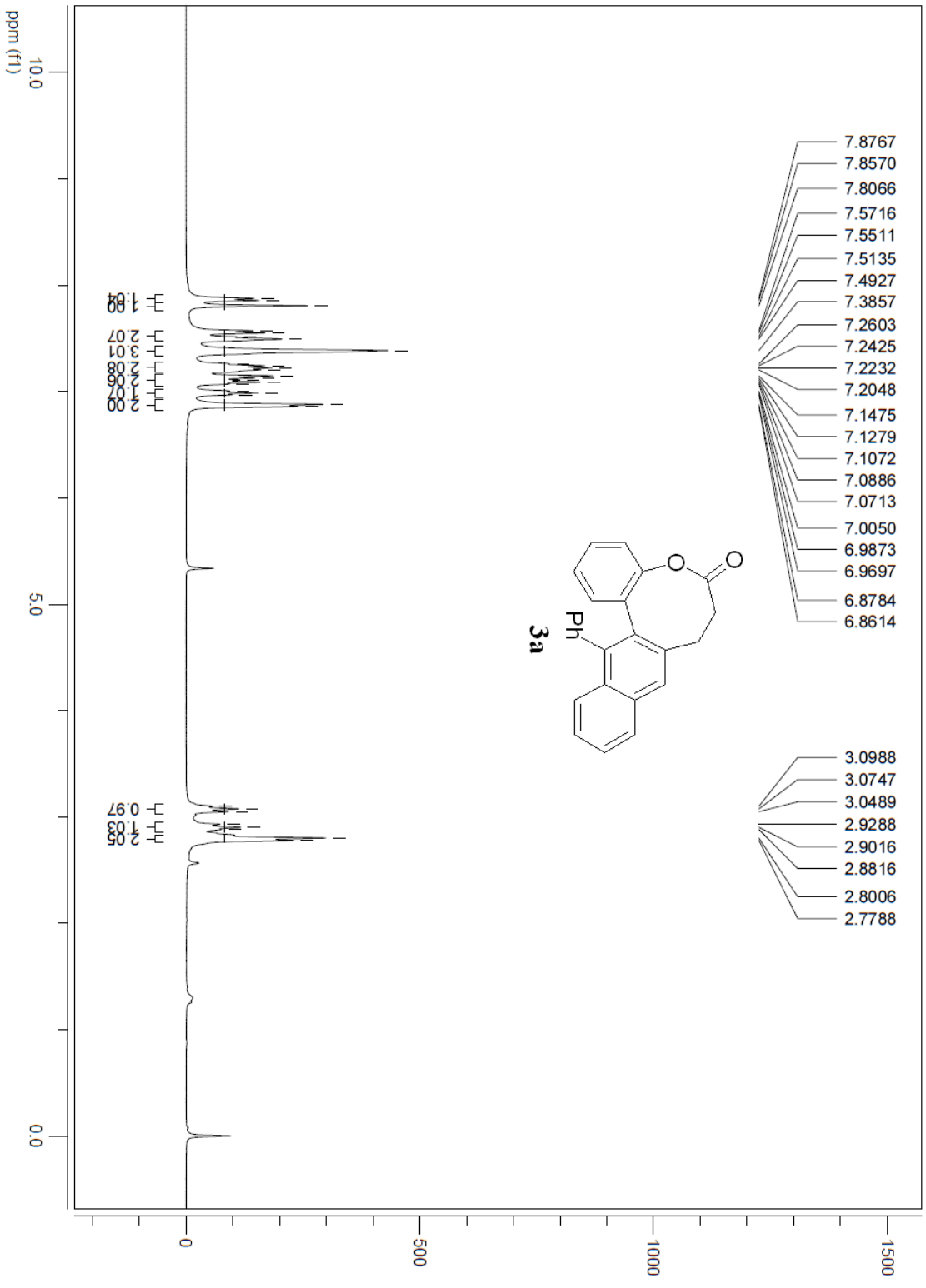
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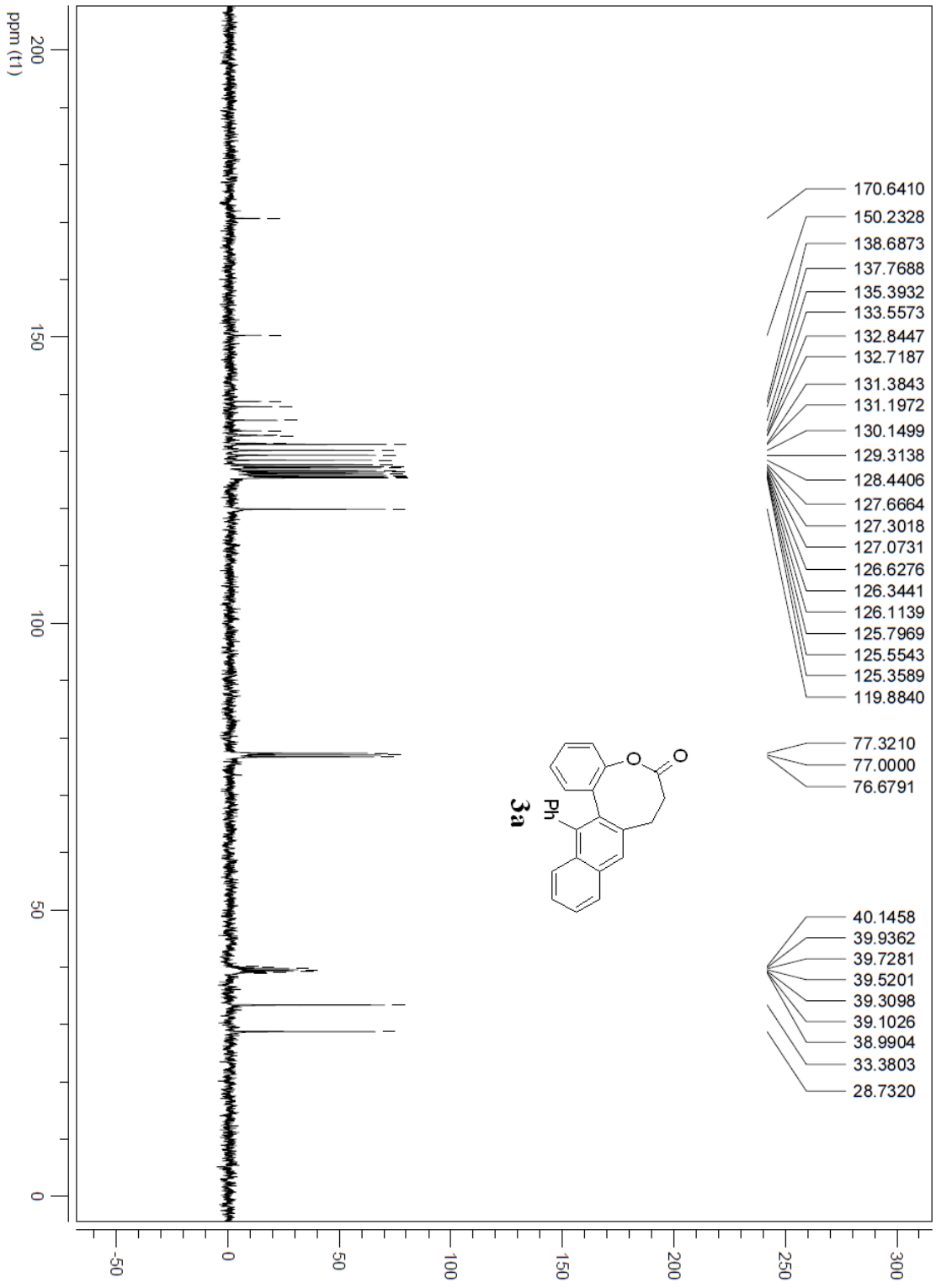
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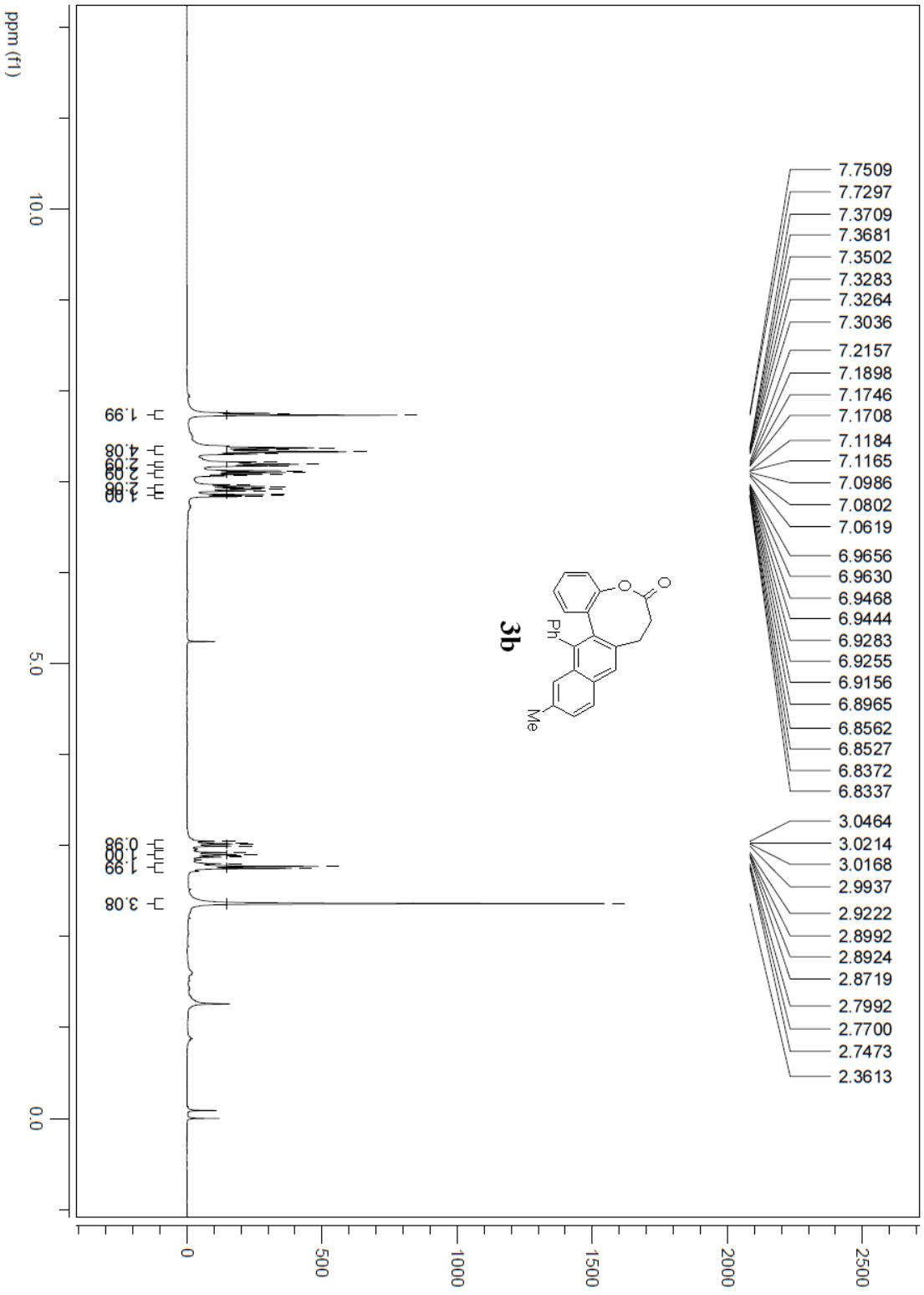
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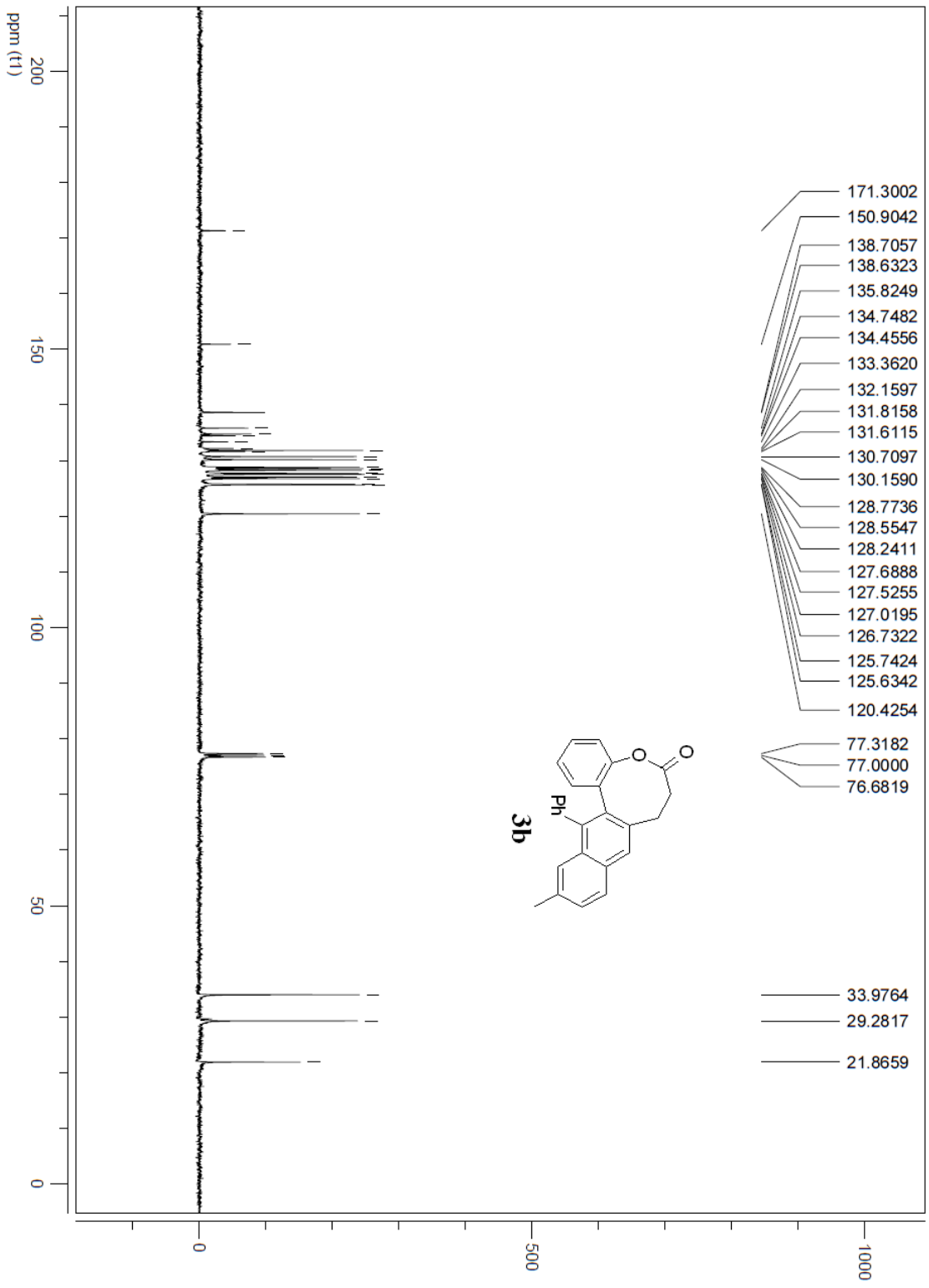
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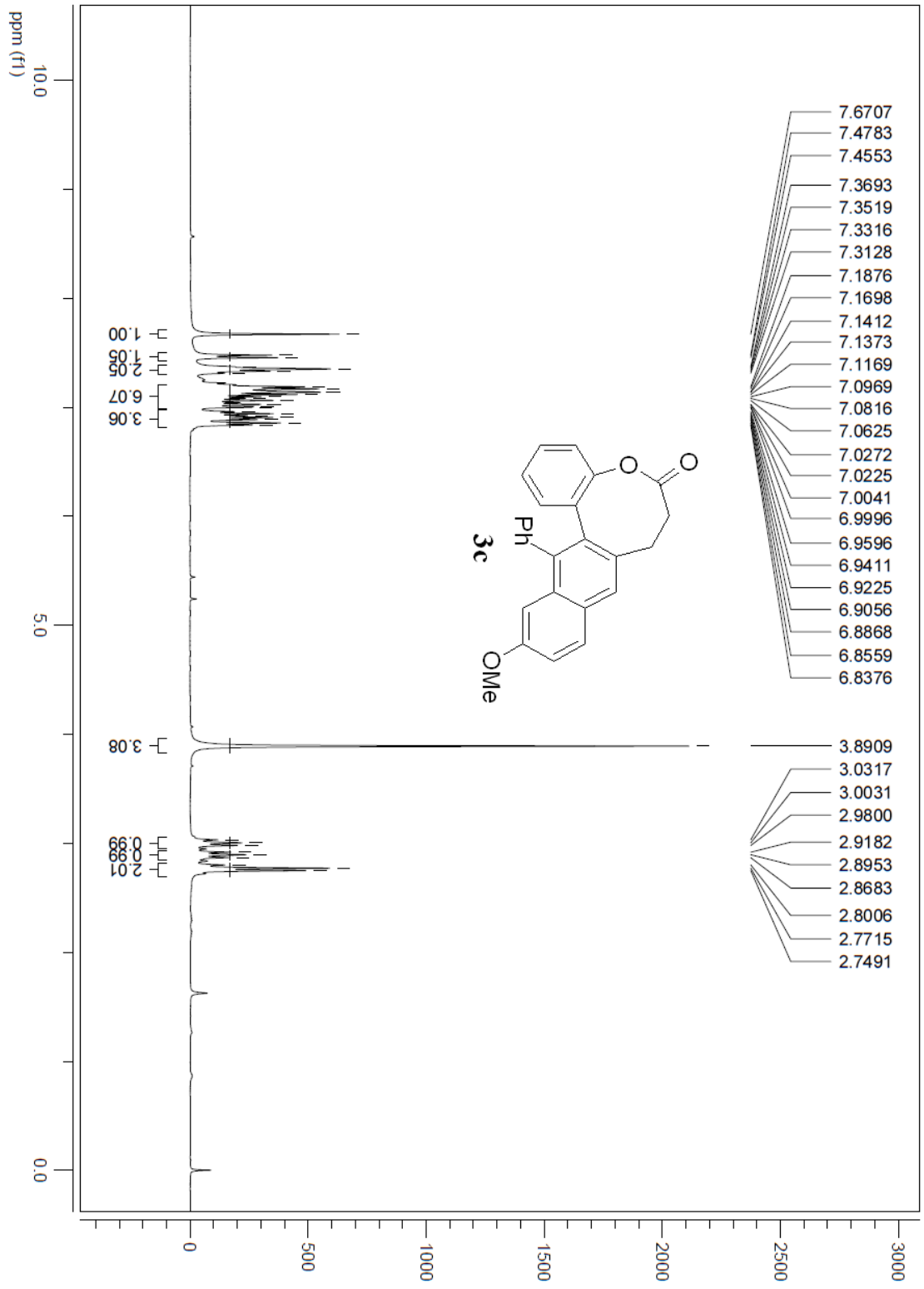
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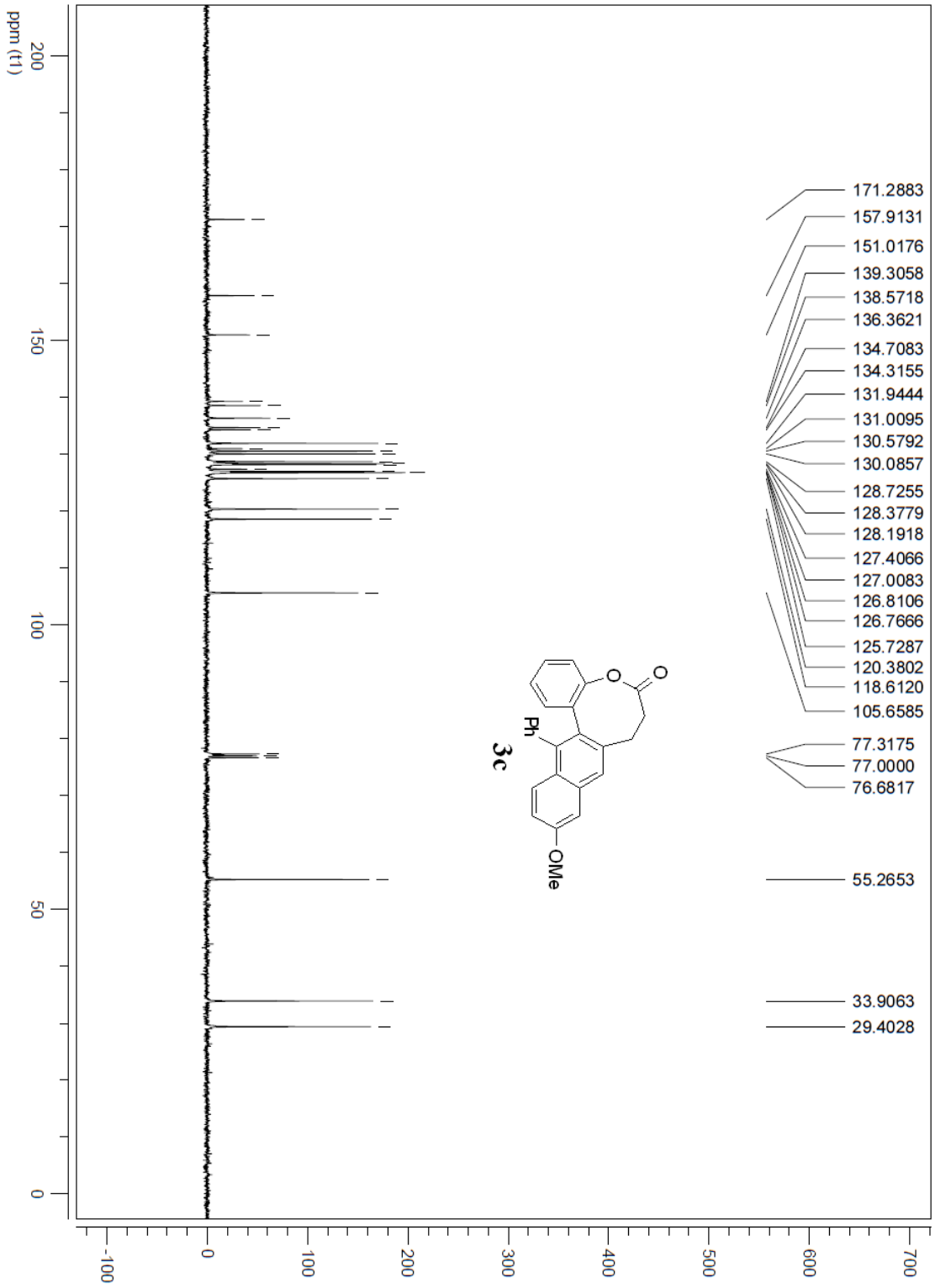
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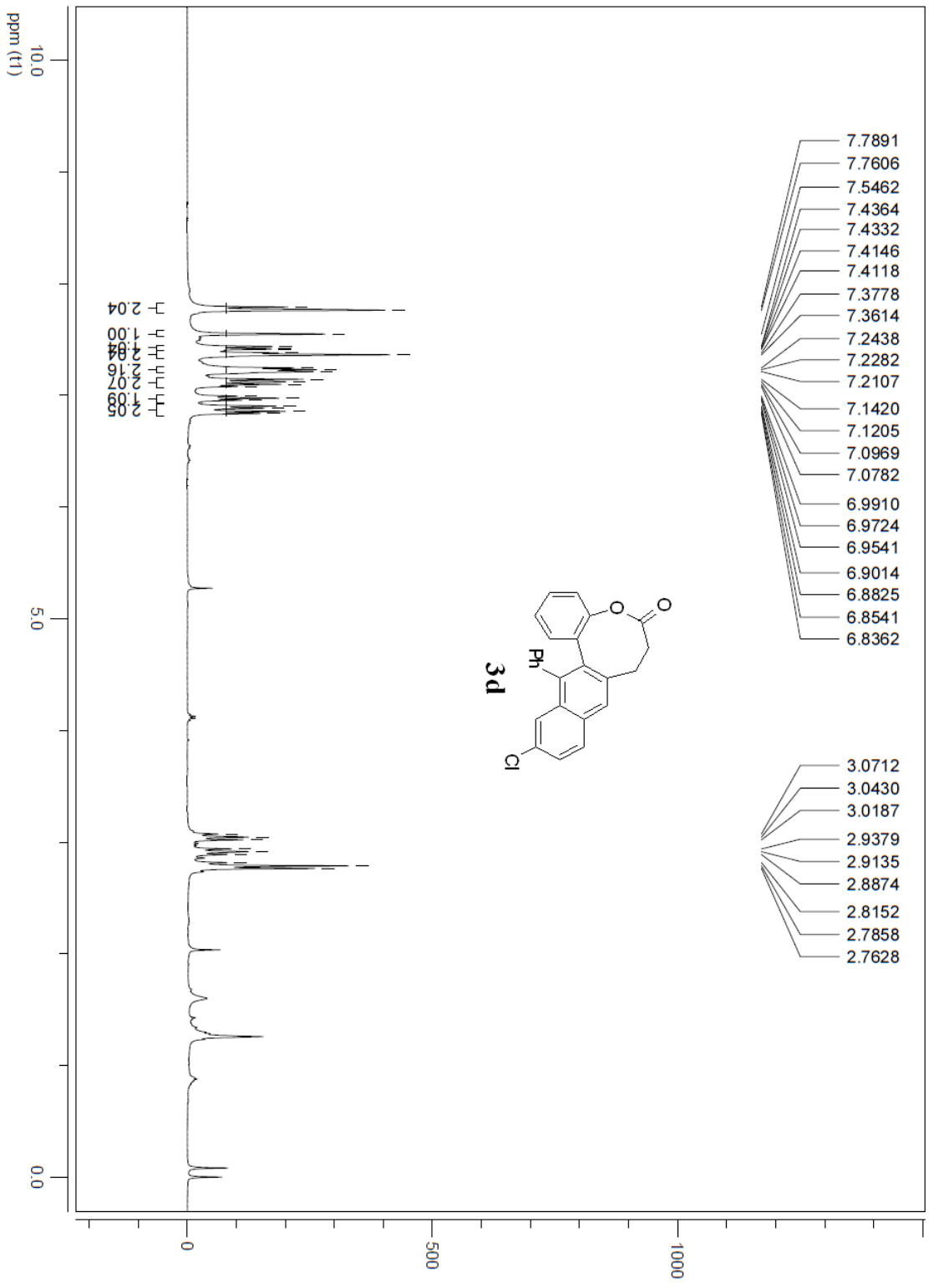
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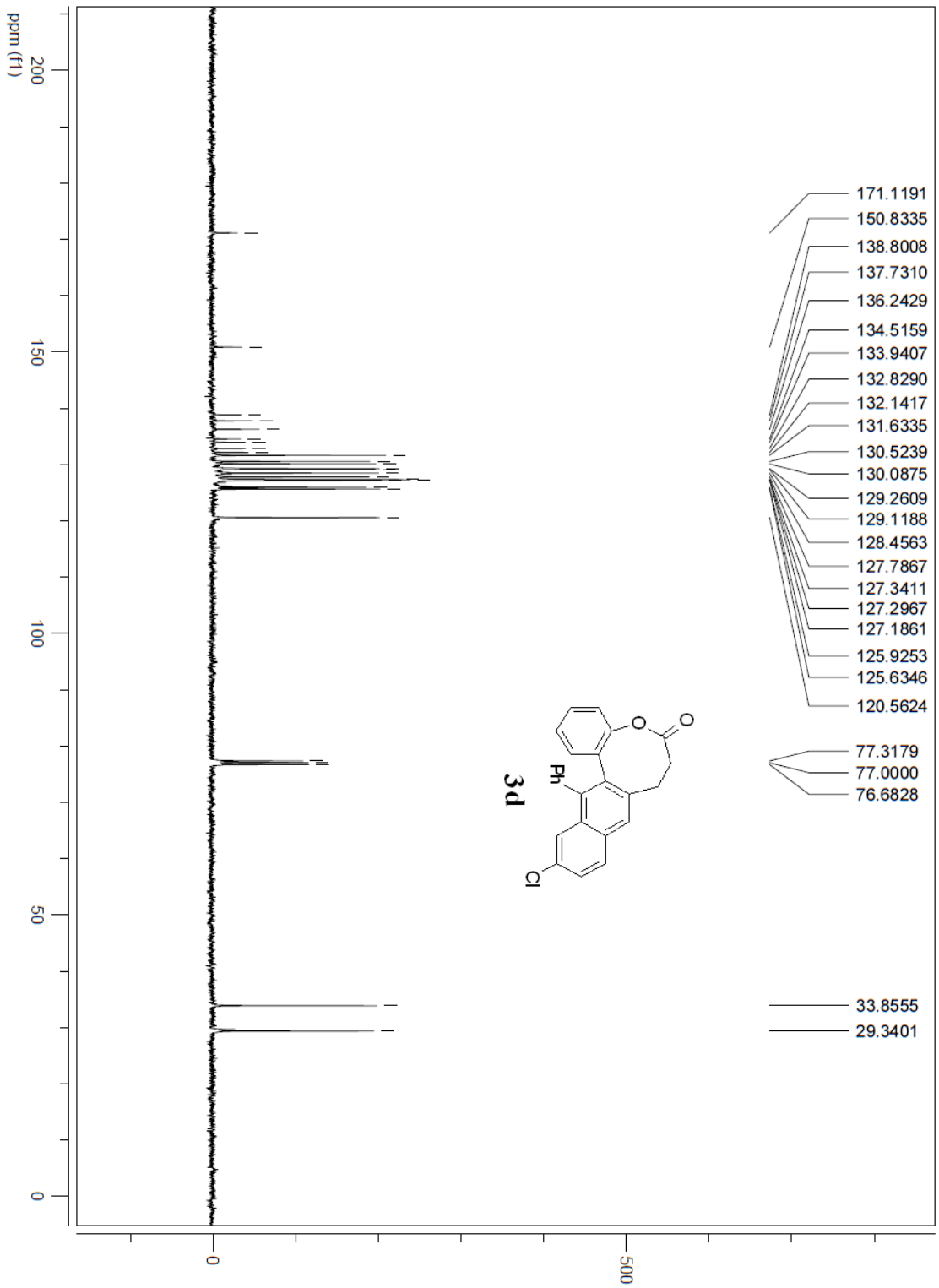
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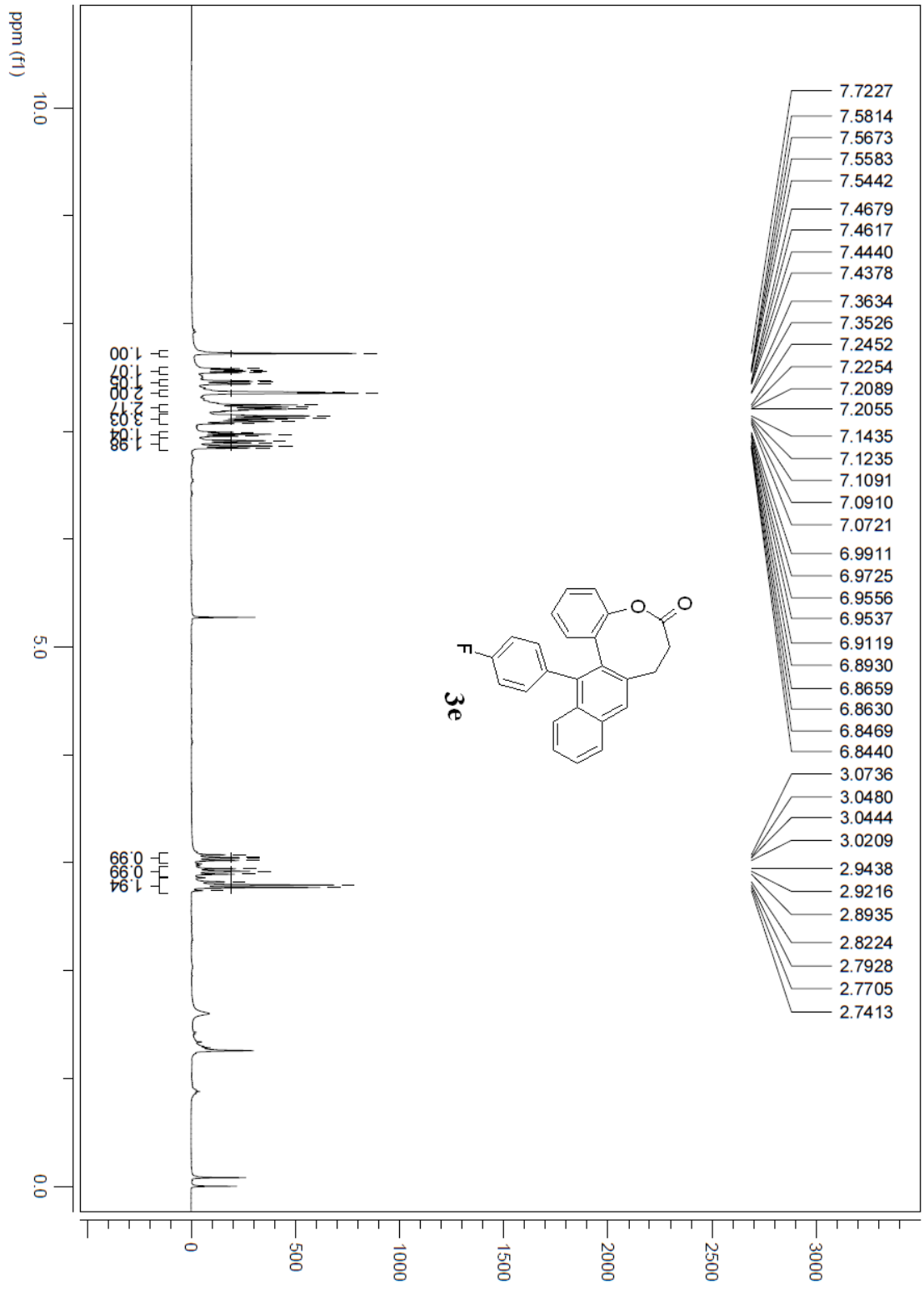
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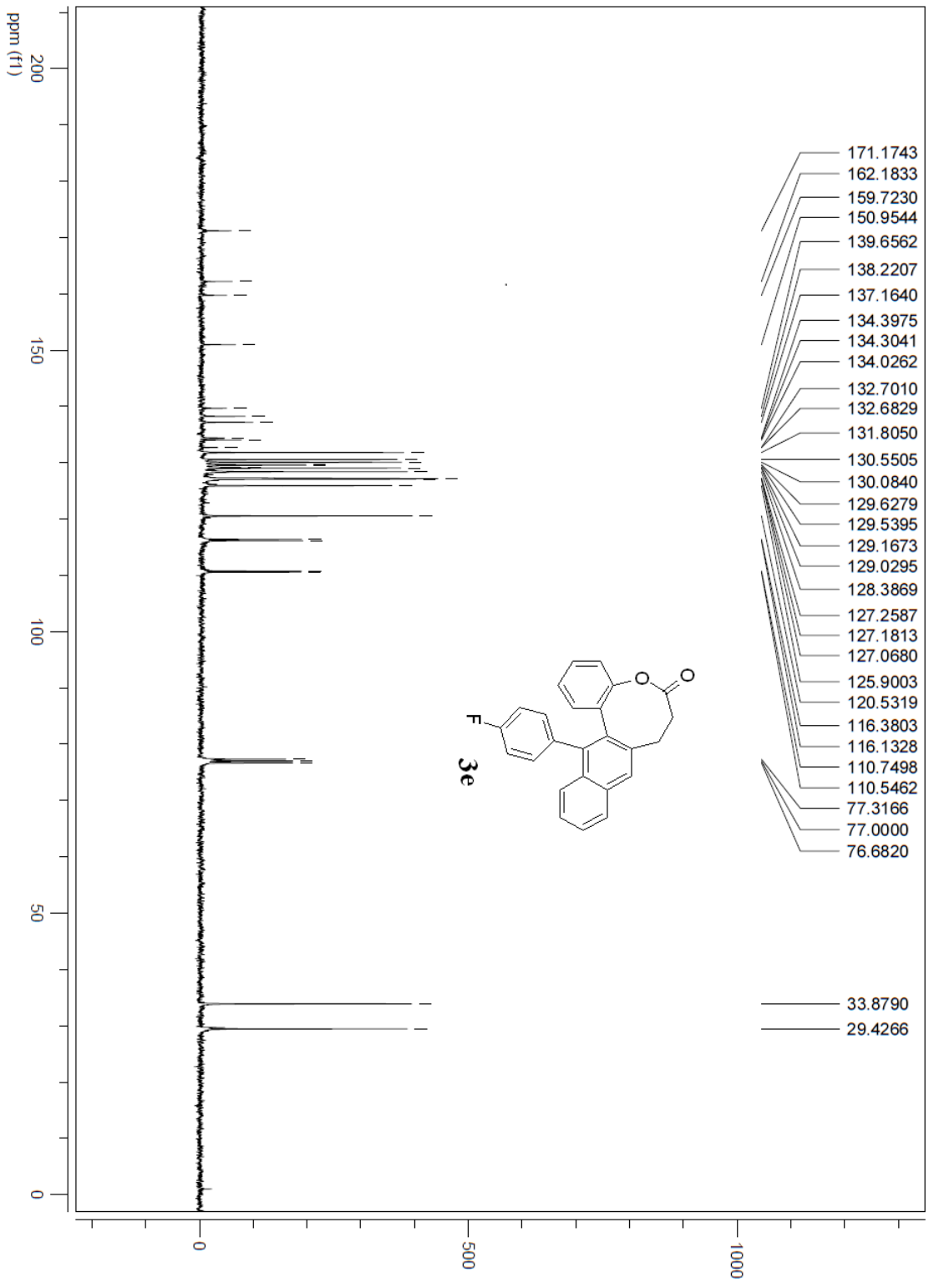
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134.3975
134.3041
134.0262
132.7010
132.6829
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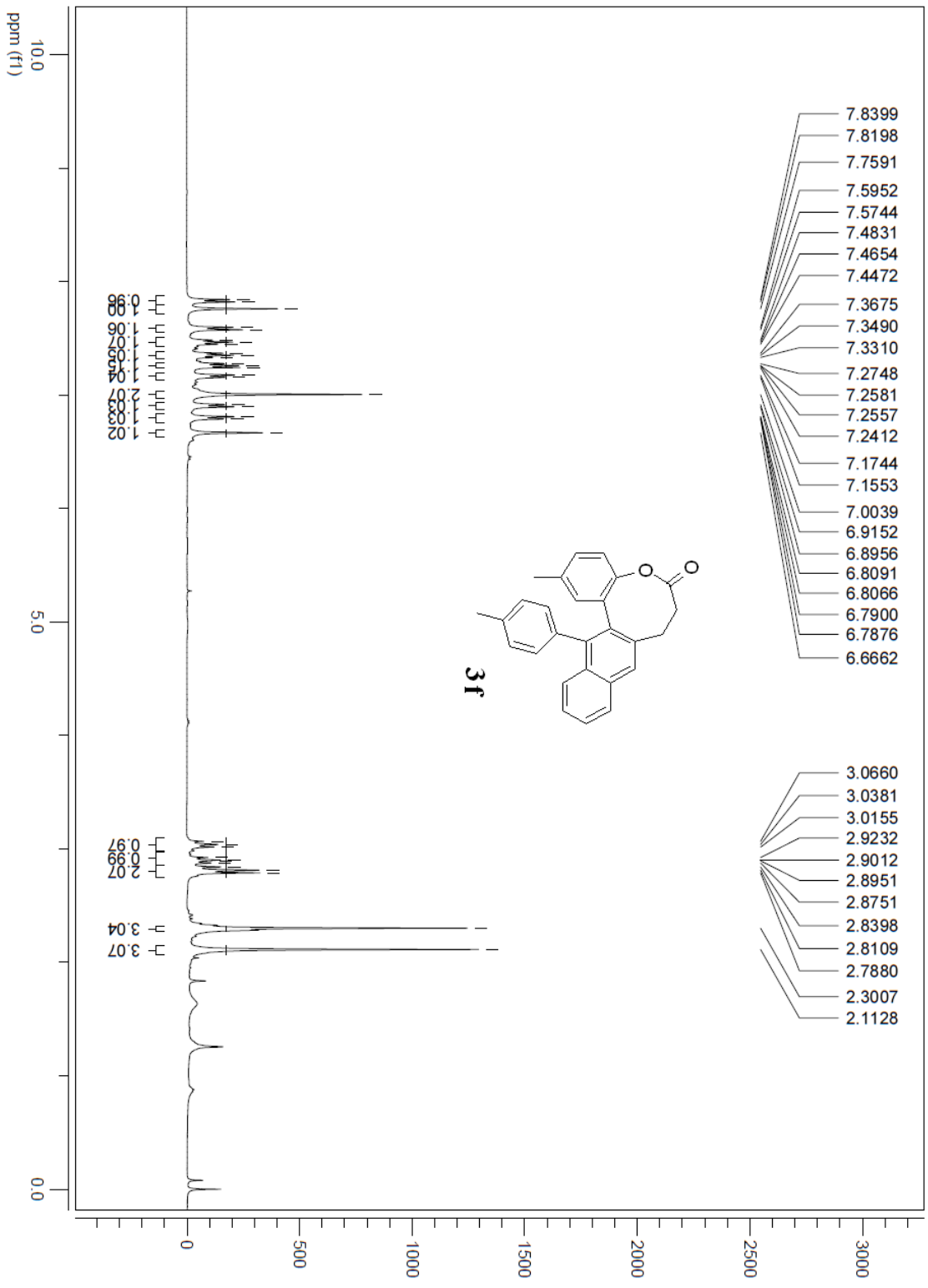
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7.1553
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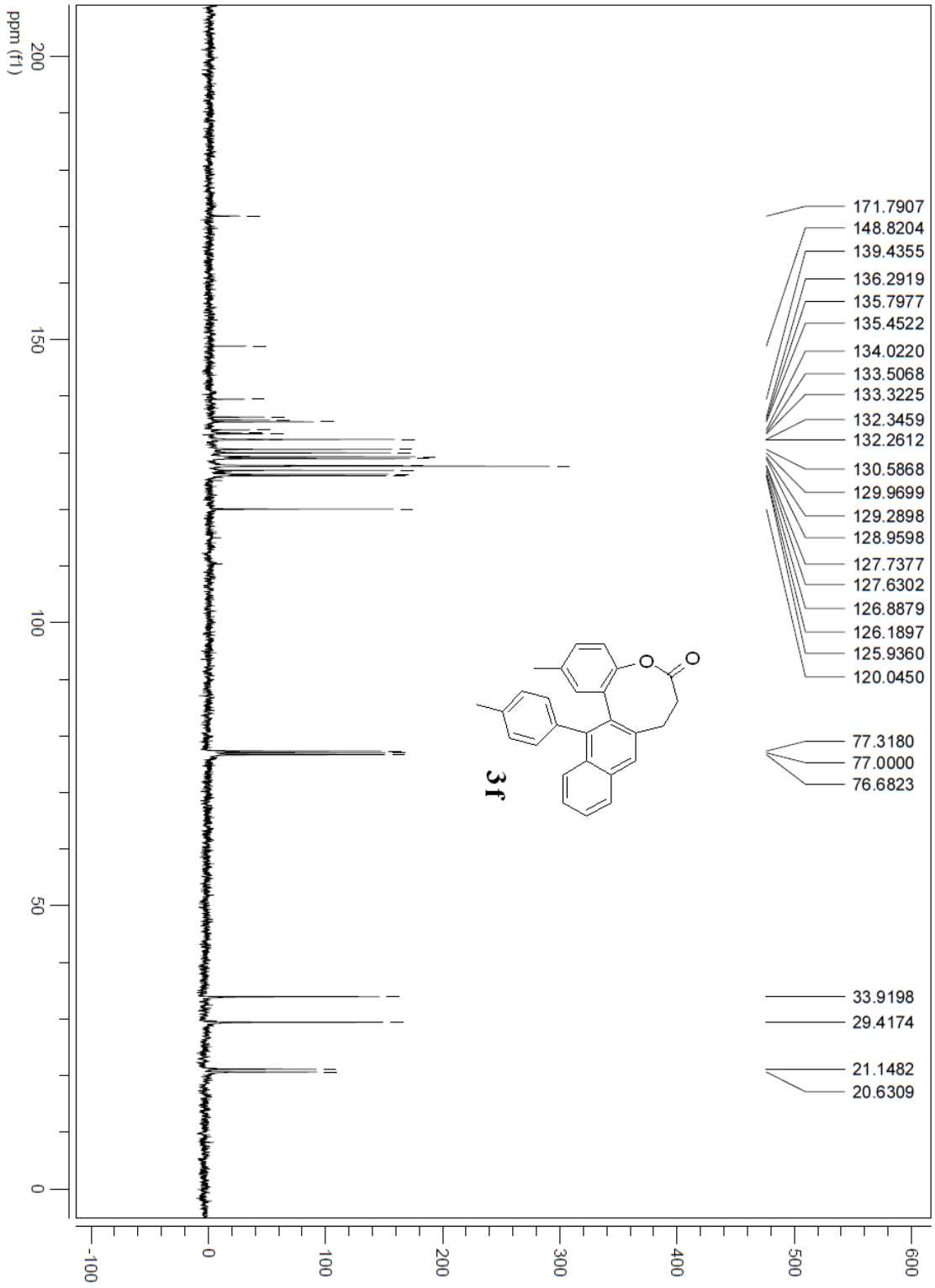
ppm (f1)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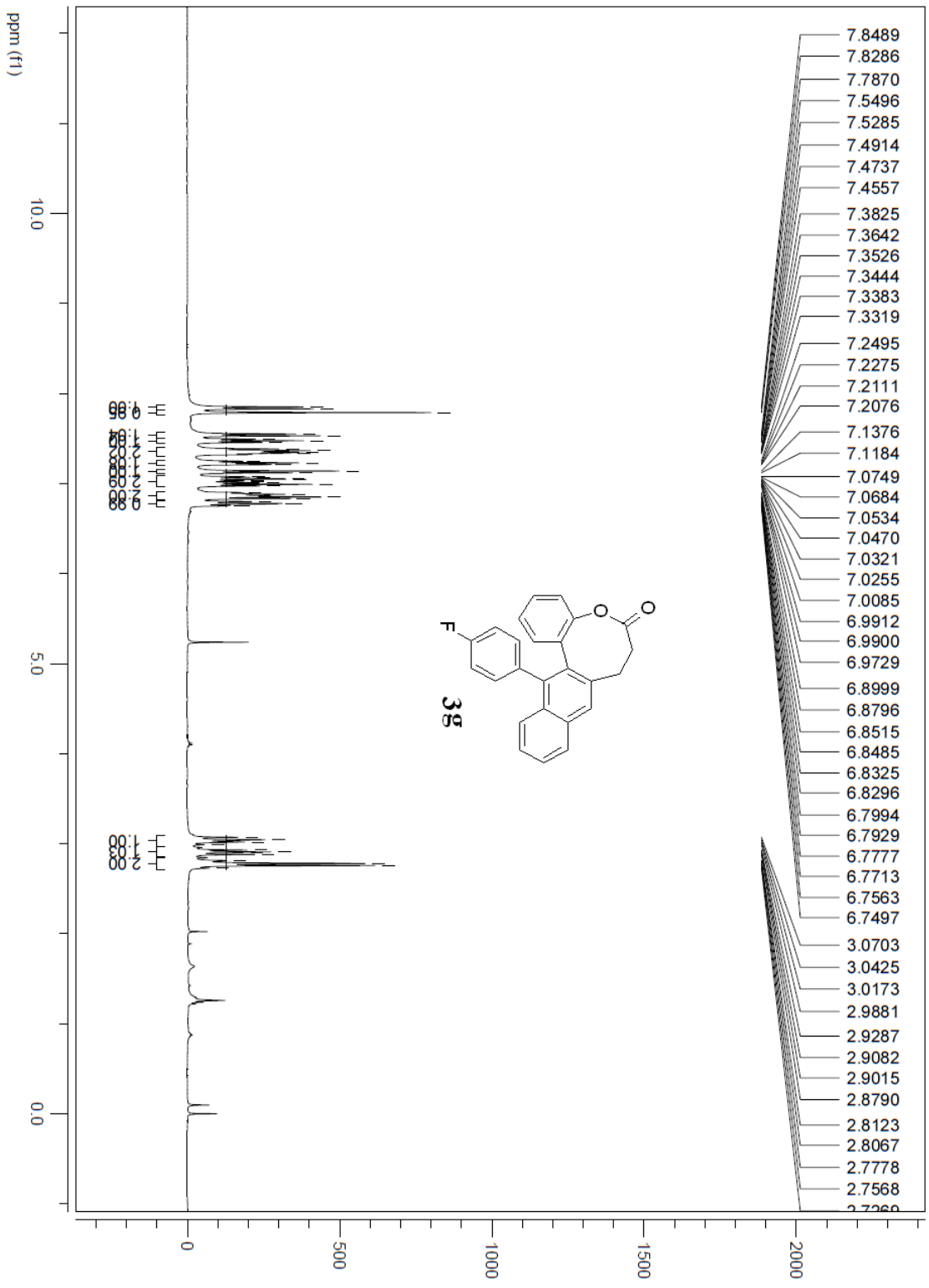
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7.0470
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7.0255
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3.0173
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F
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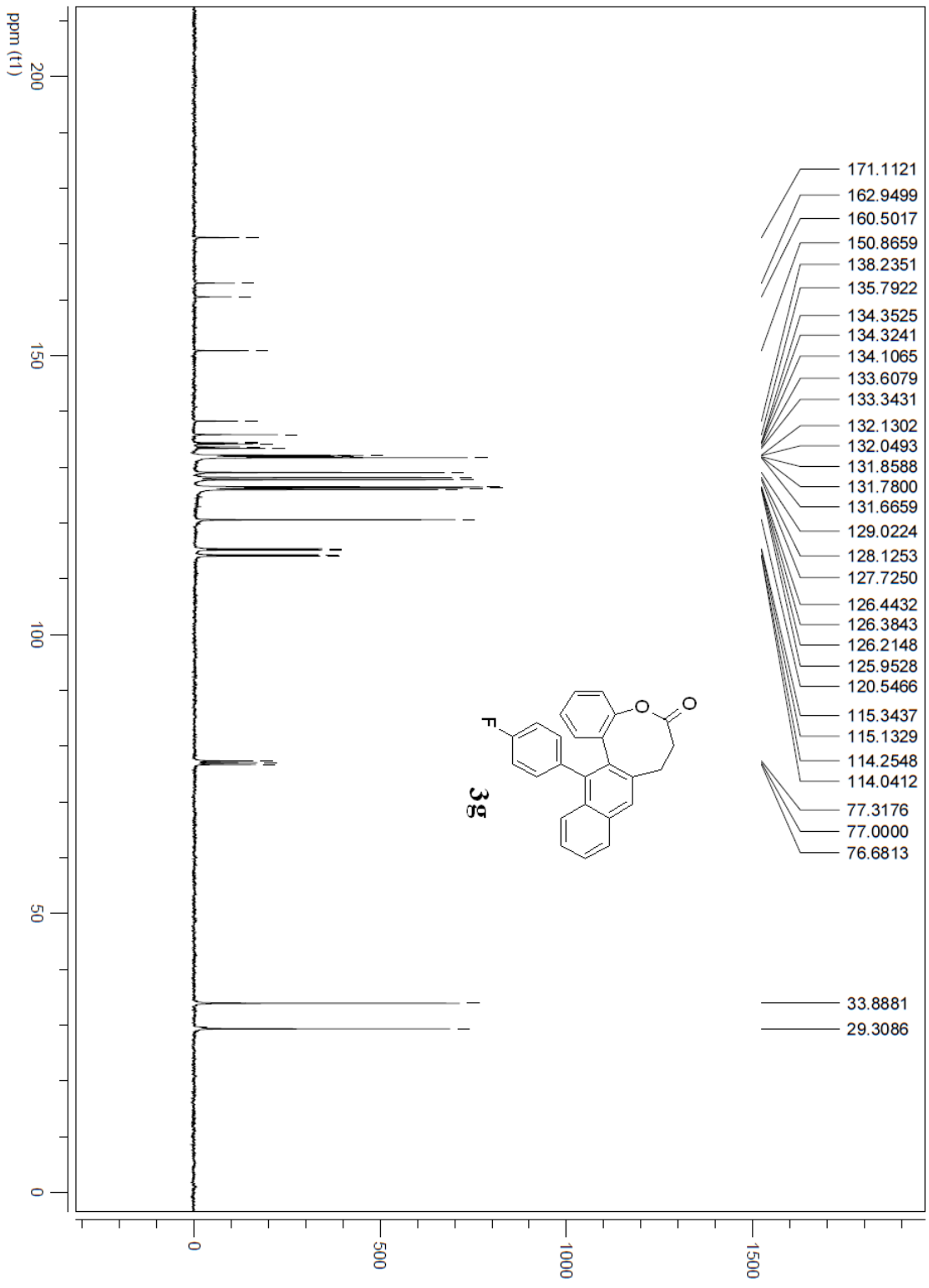
O
3g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120.5466
115.3437
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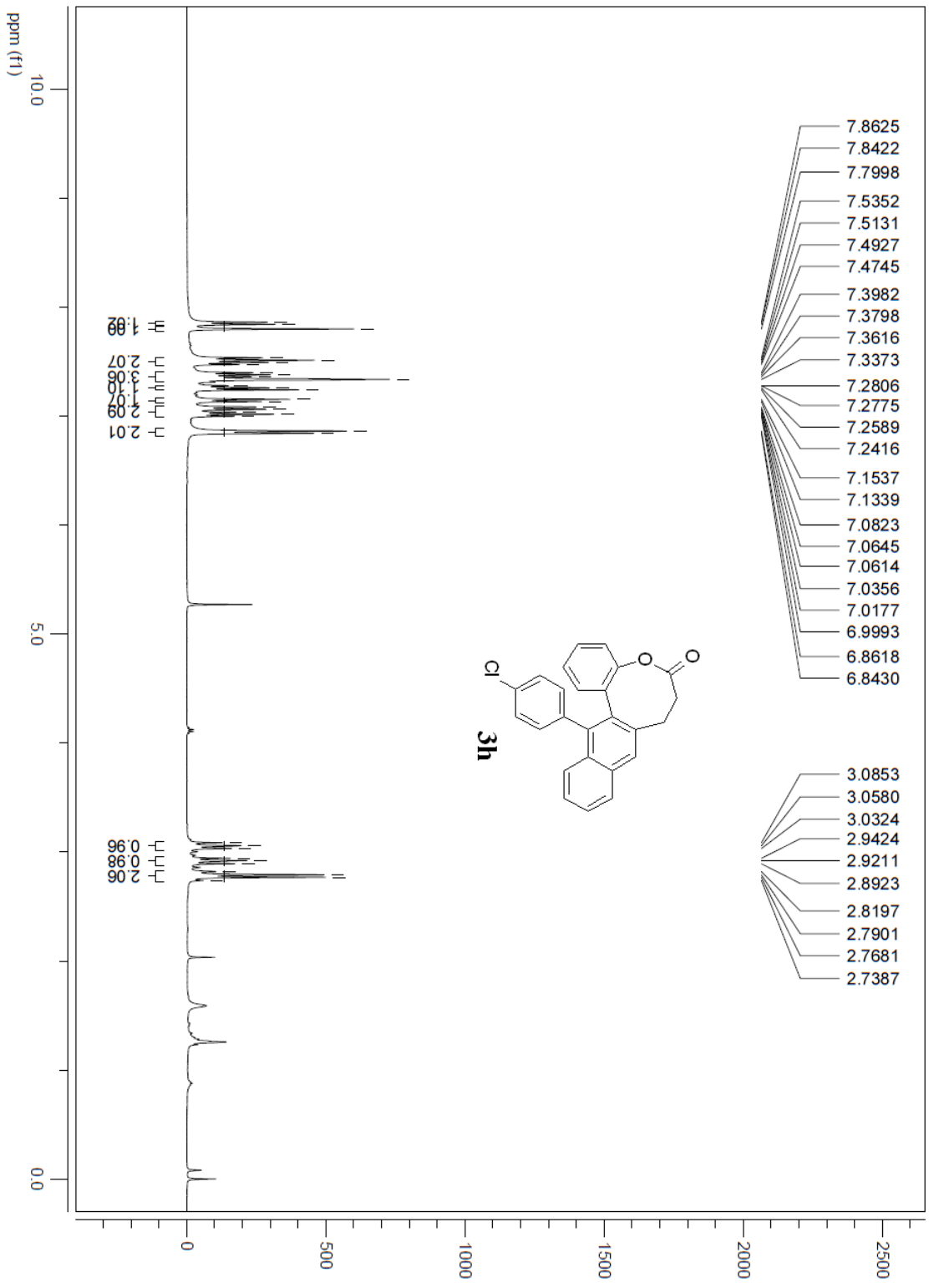
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7.2775
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7.2416
7.1537
7.1339
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7.0645
7.0614
7.0356
7.0177
6.9993
6.8618
6.8430
3.0853
3.0580
3.0324
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2.9211
2.8923
2.8197
2.7901
2.7681
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O
O
3h
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2000
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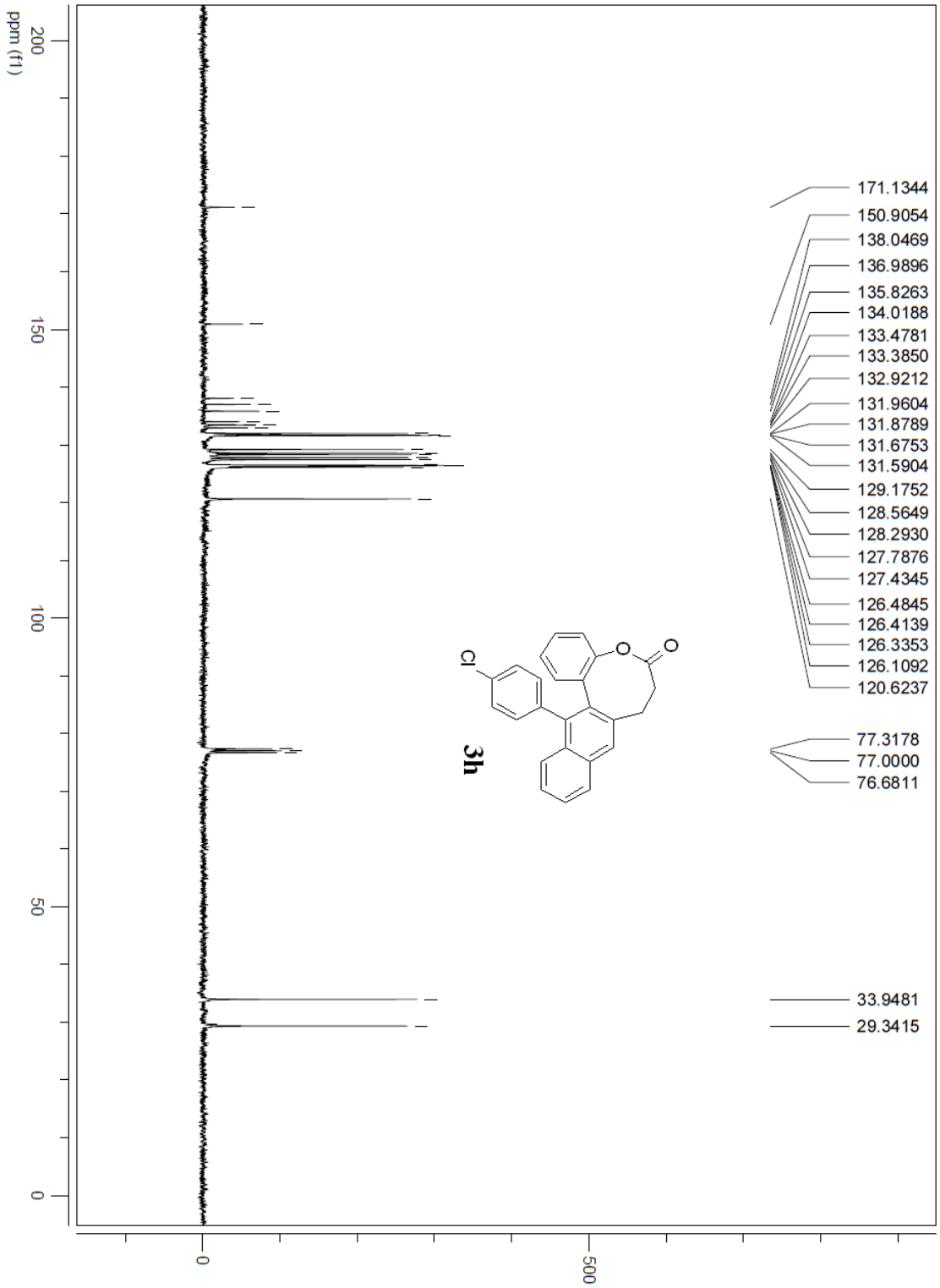
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128.2930
127.7876
127.4345
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126.4139
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Cl
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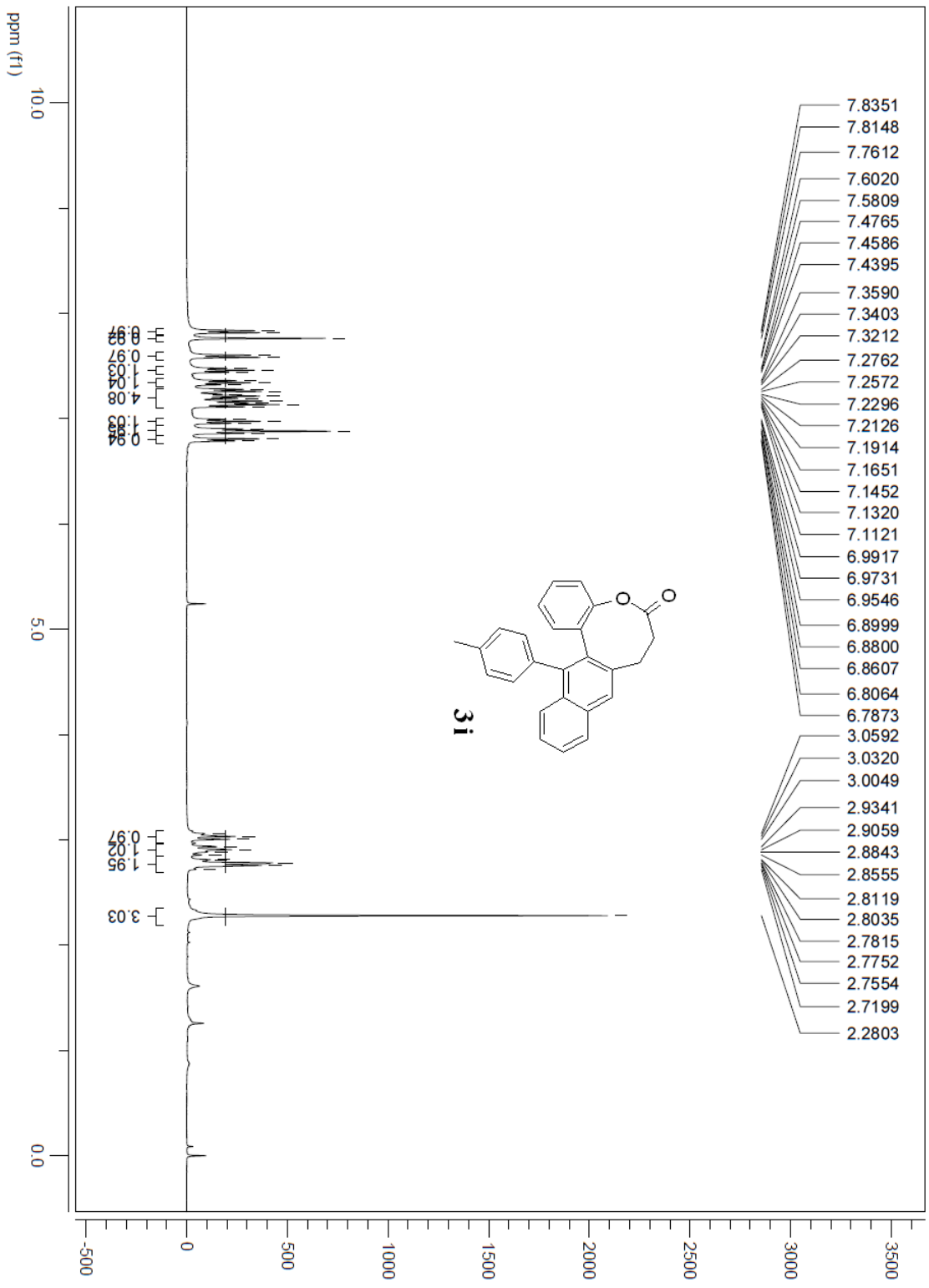
O
3h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7.2762
7.2572
7.2296
7.2126
7.1914
7.1651
7.1452
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6.8999
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6.8607
6.8064
6.7873
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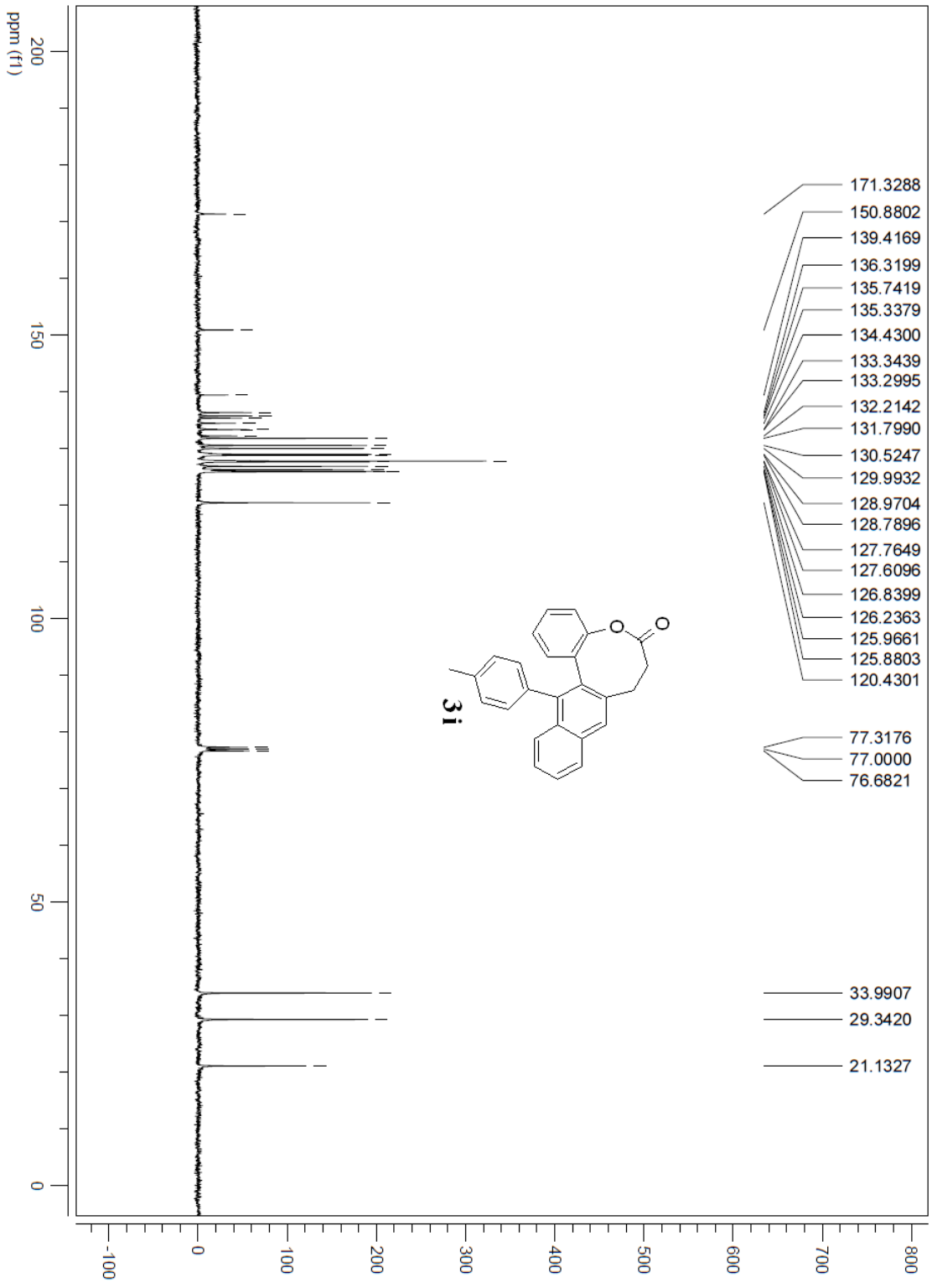
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600
700
800
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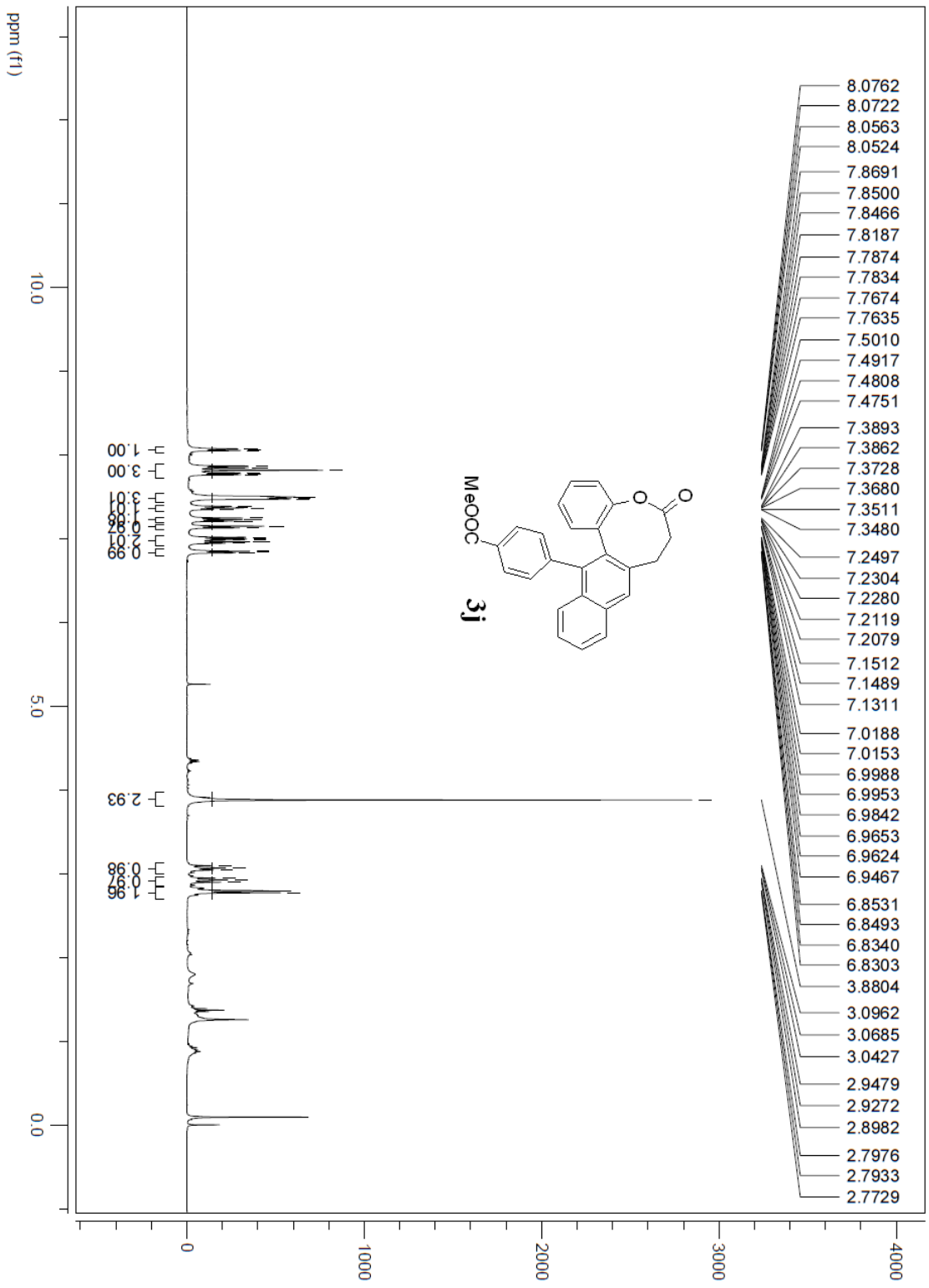
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0.92
2.01
0.99
2.93
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0.97
1.96
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O
O
3j
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7.1311
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6.9953
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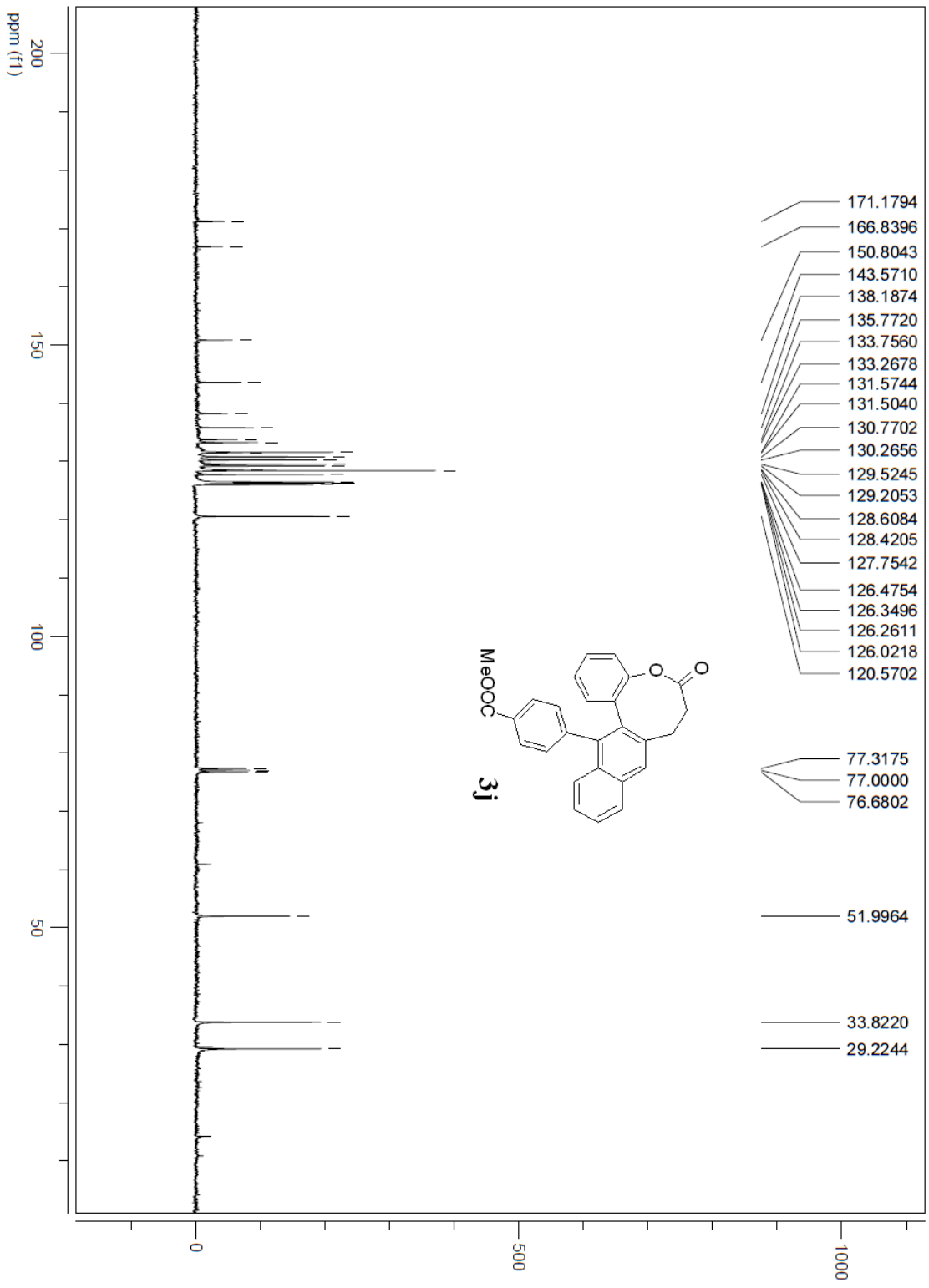
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127.7542
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126.3496
126.2611
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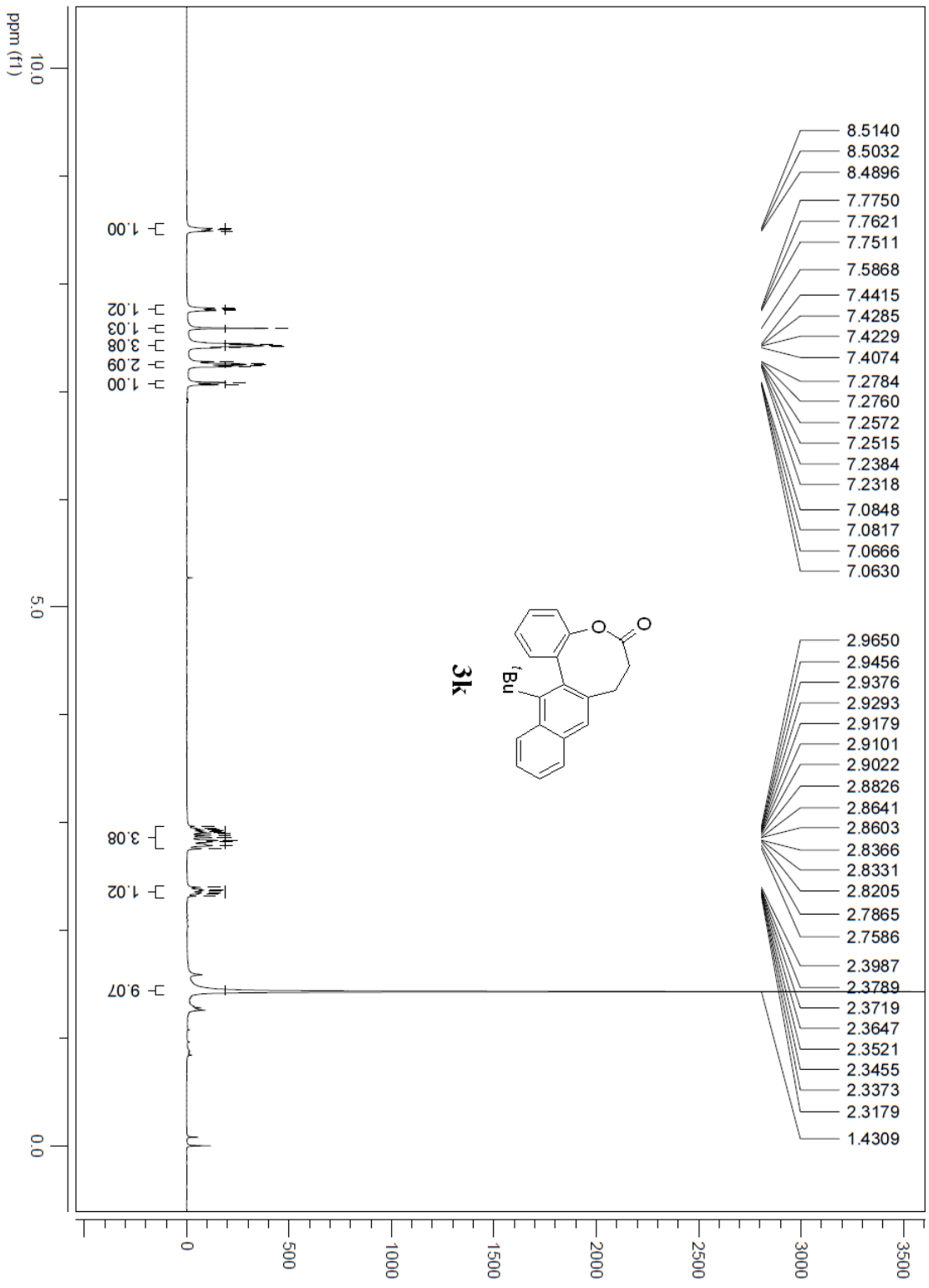
O
O
3j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7.2760
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7.2384
7.2318
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1.02
1.03
3.08
2.09
1.00
3.08
1.02
9.07
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tBu
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O
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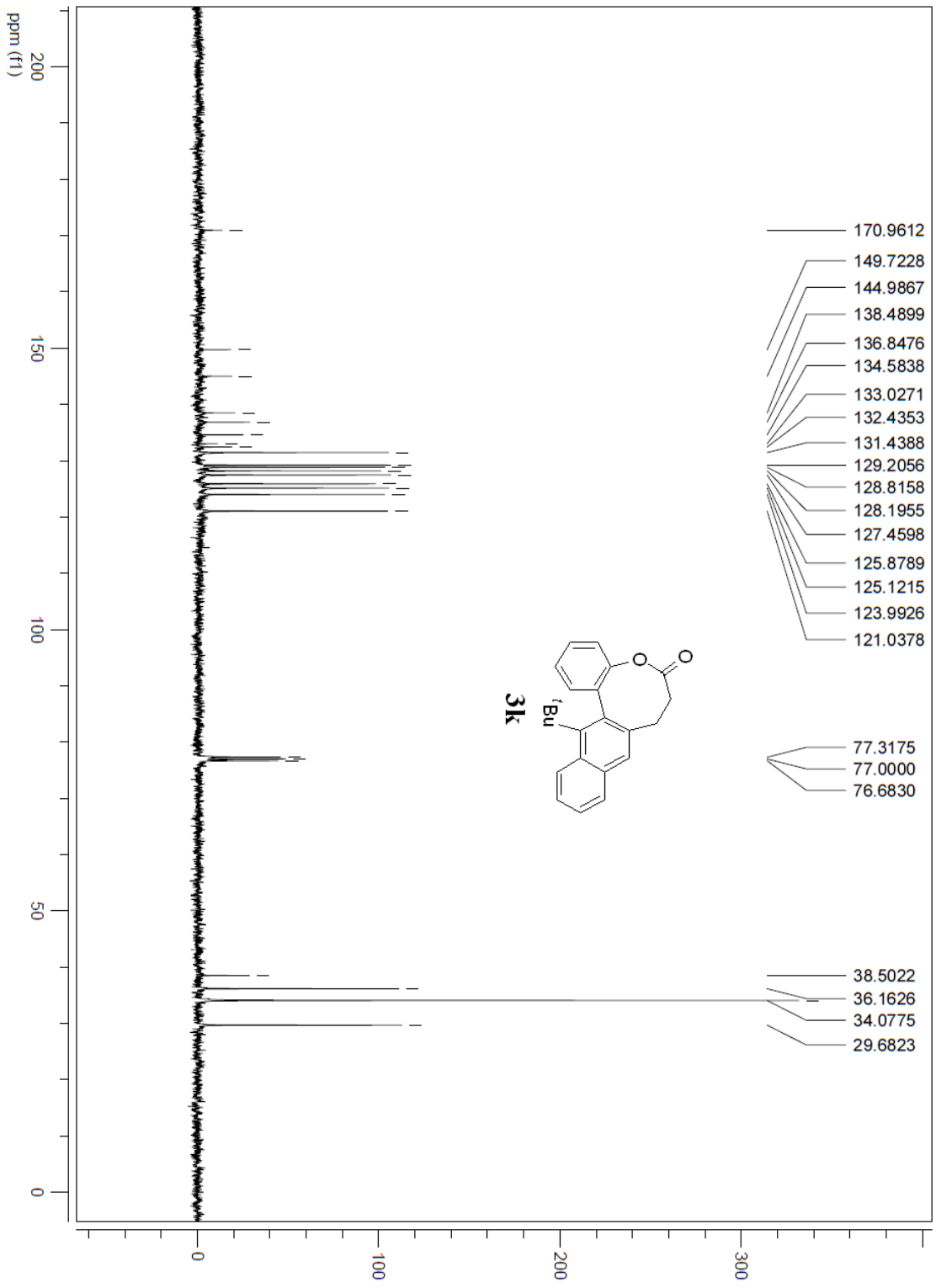
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O
tBu
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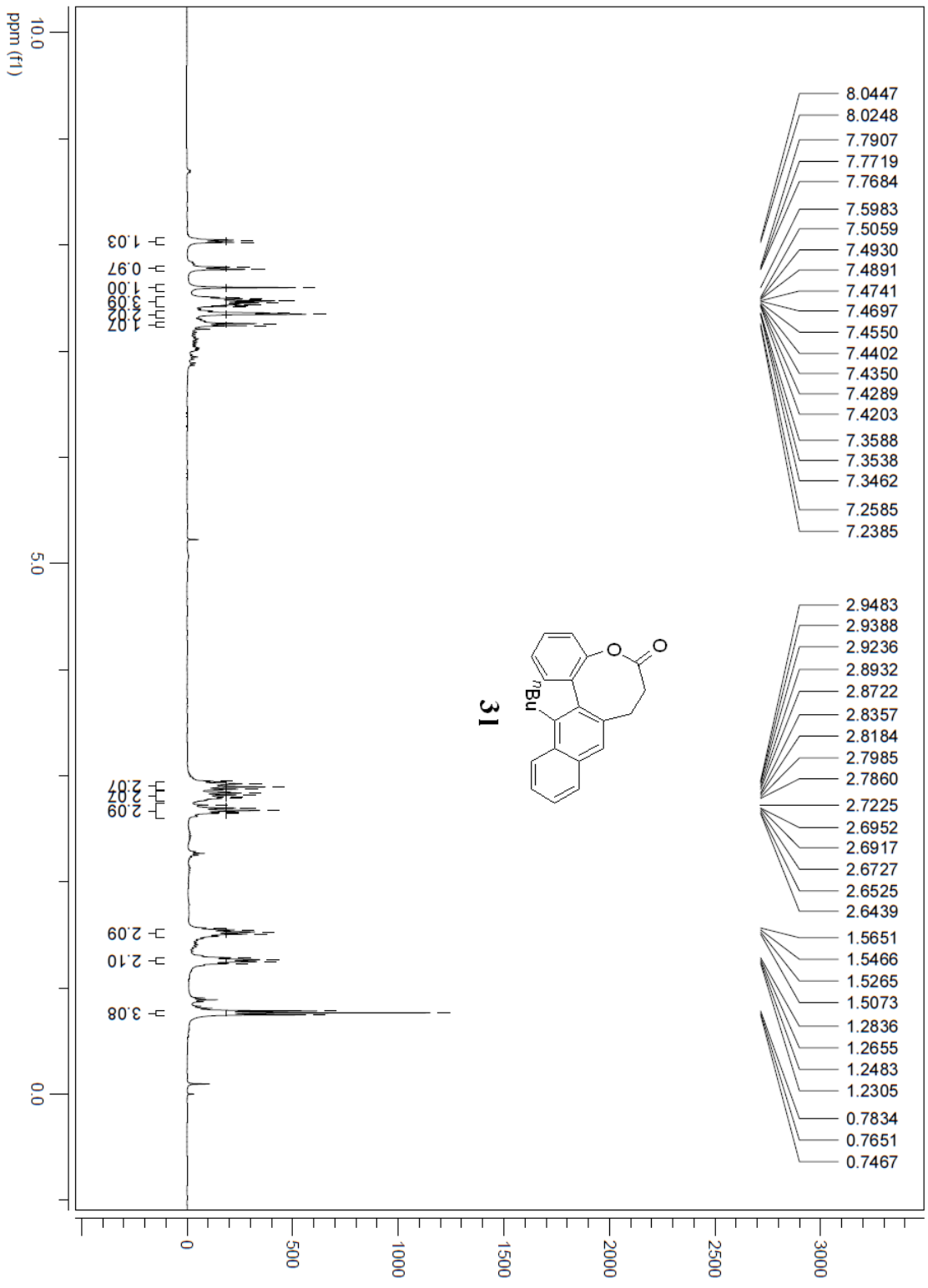
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2.07
2.09
2.09
2.10
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O
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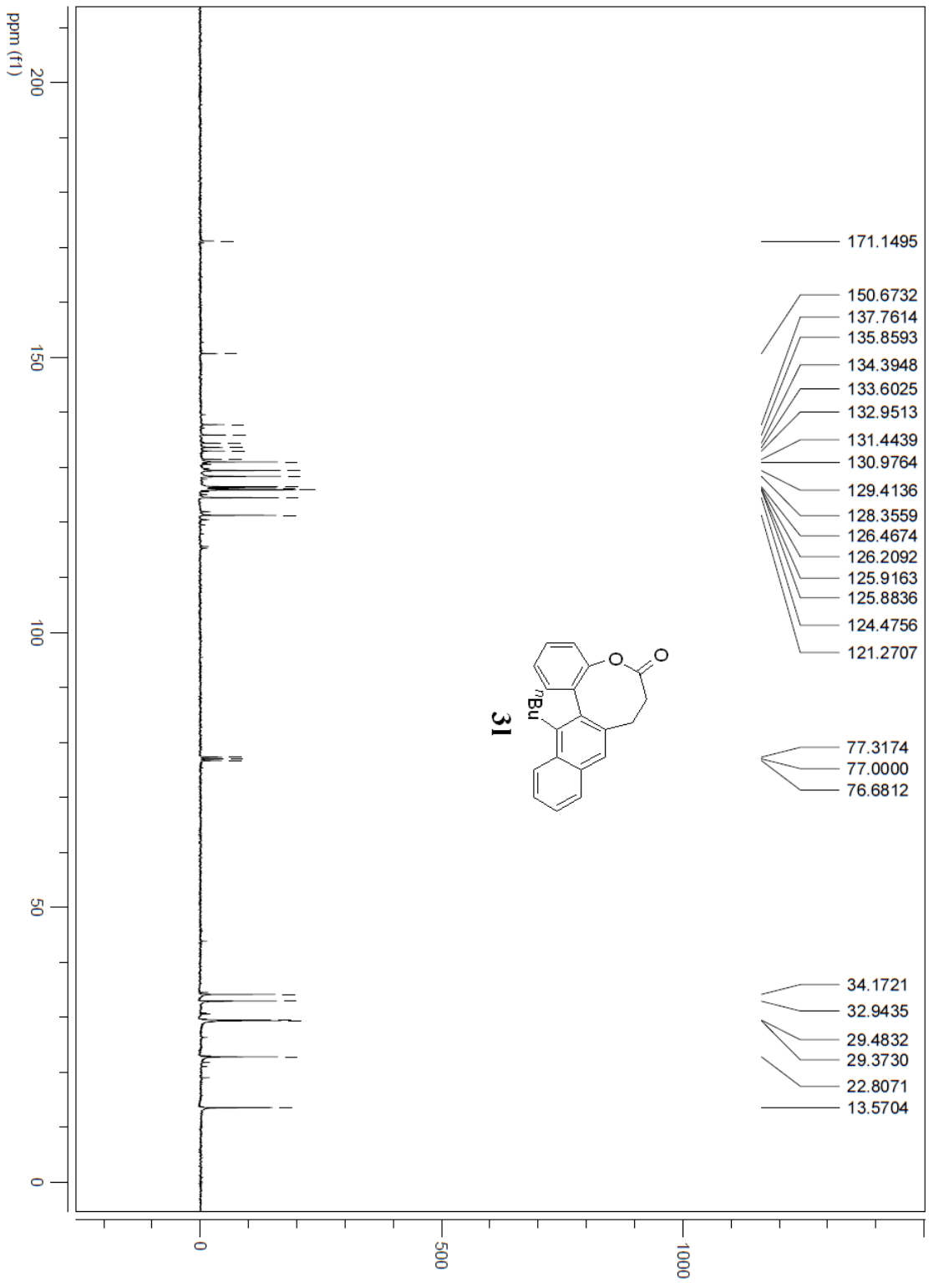
3000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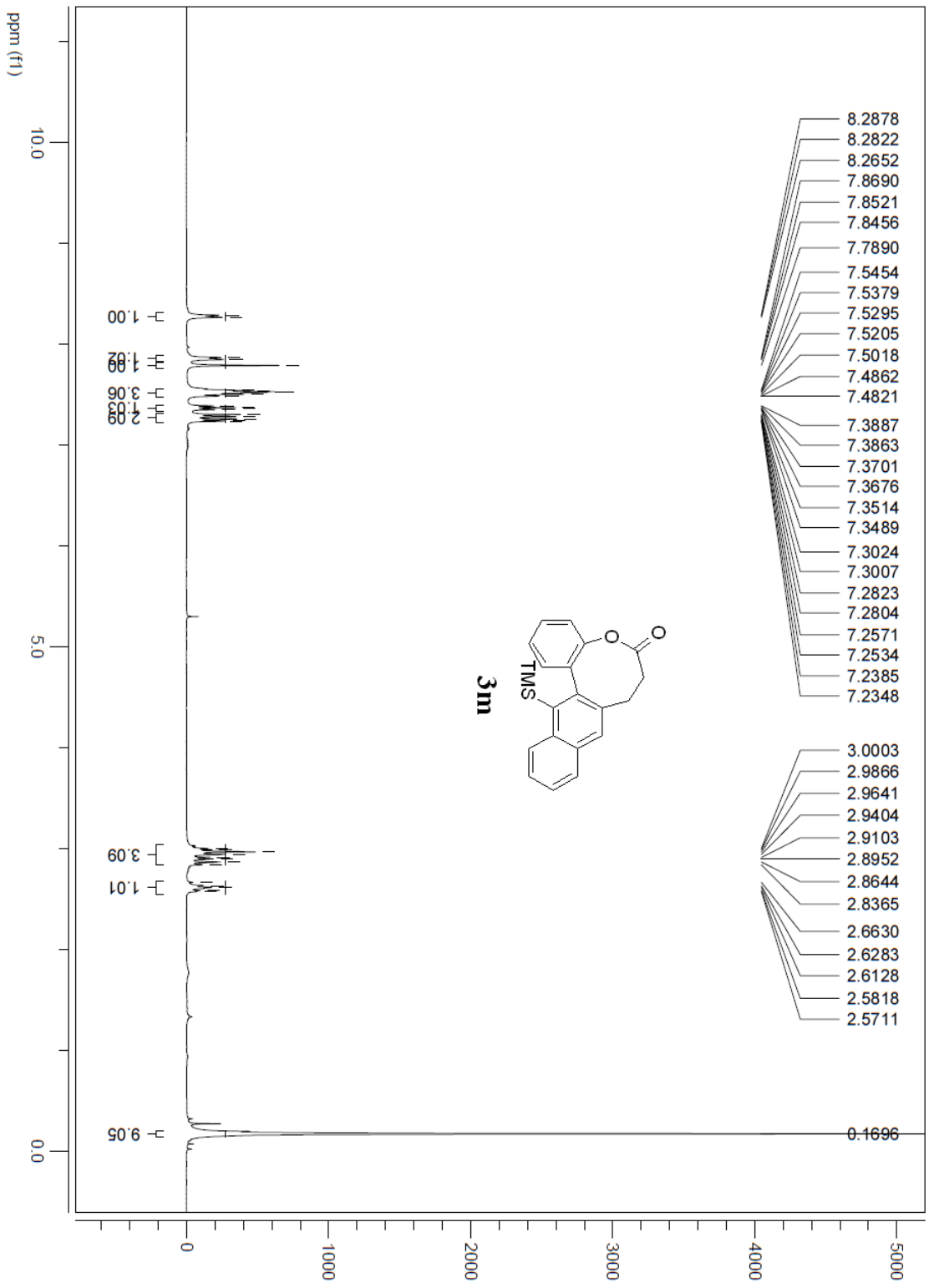
O
nBu
31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3.09
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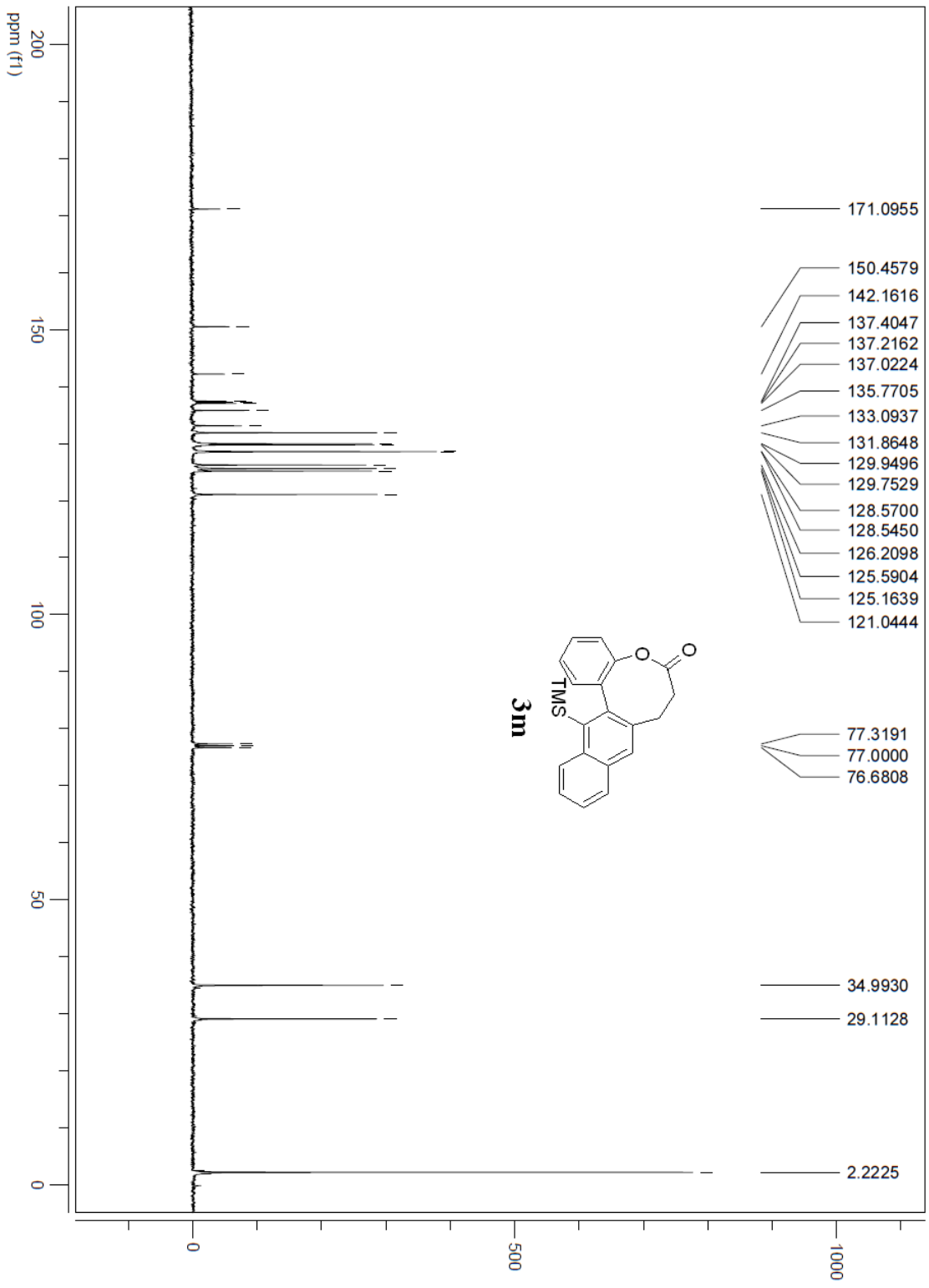
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TMS
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O
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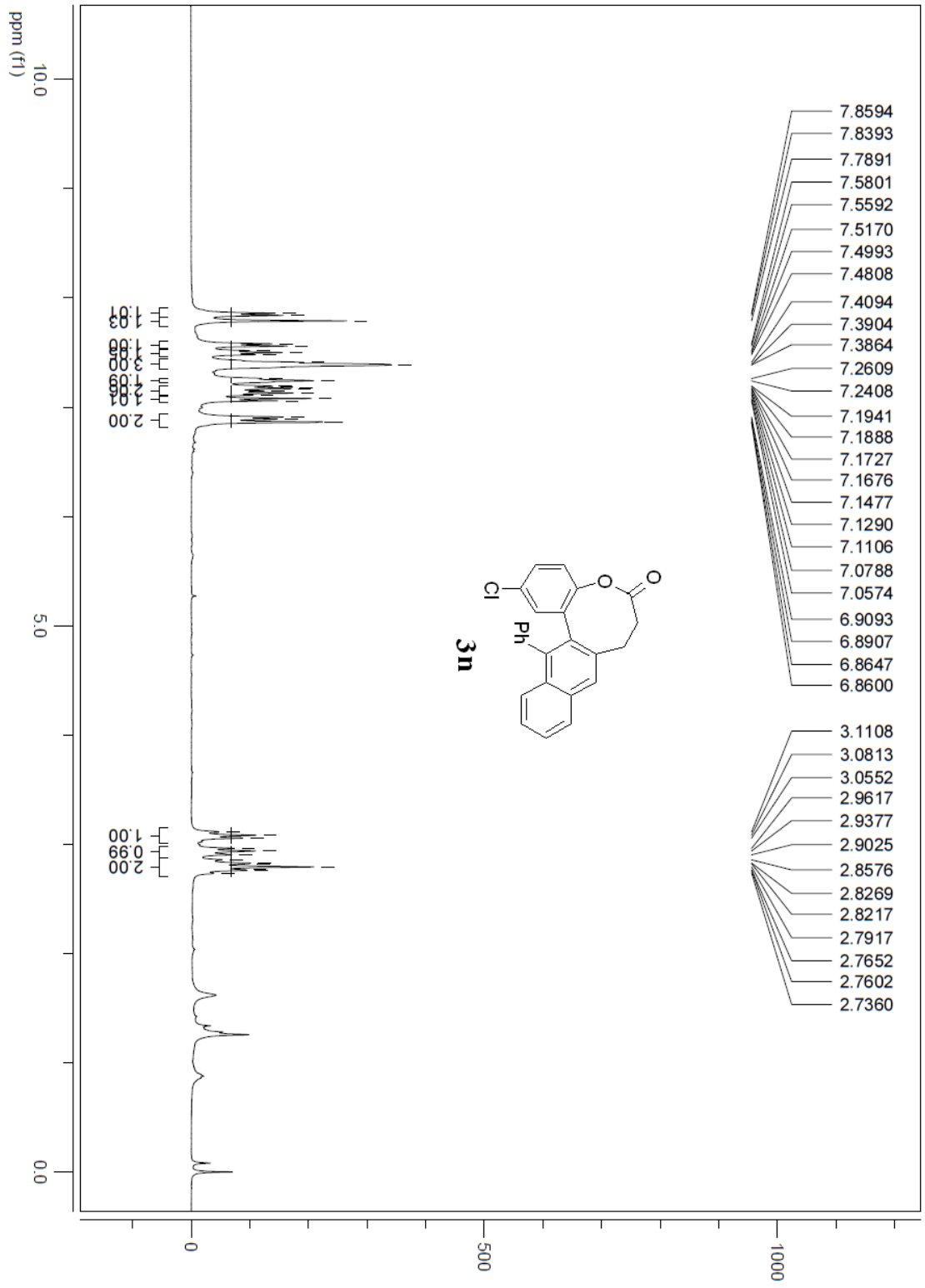
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7.1477
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7.0574
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1.03
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Ph
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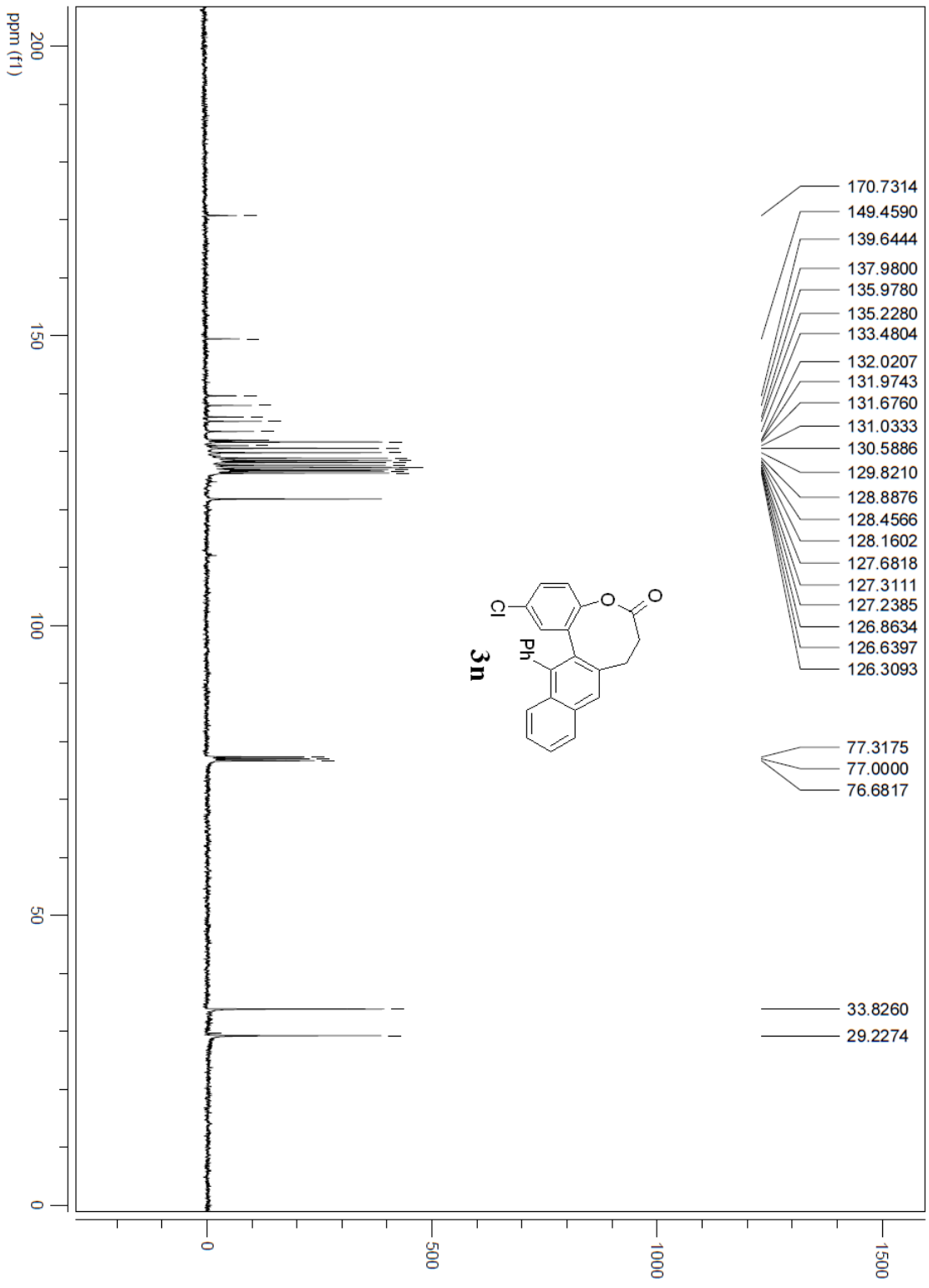
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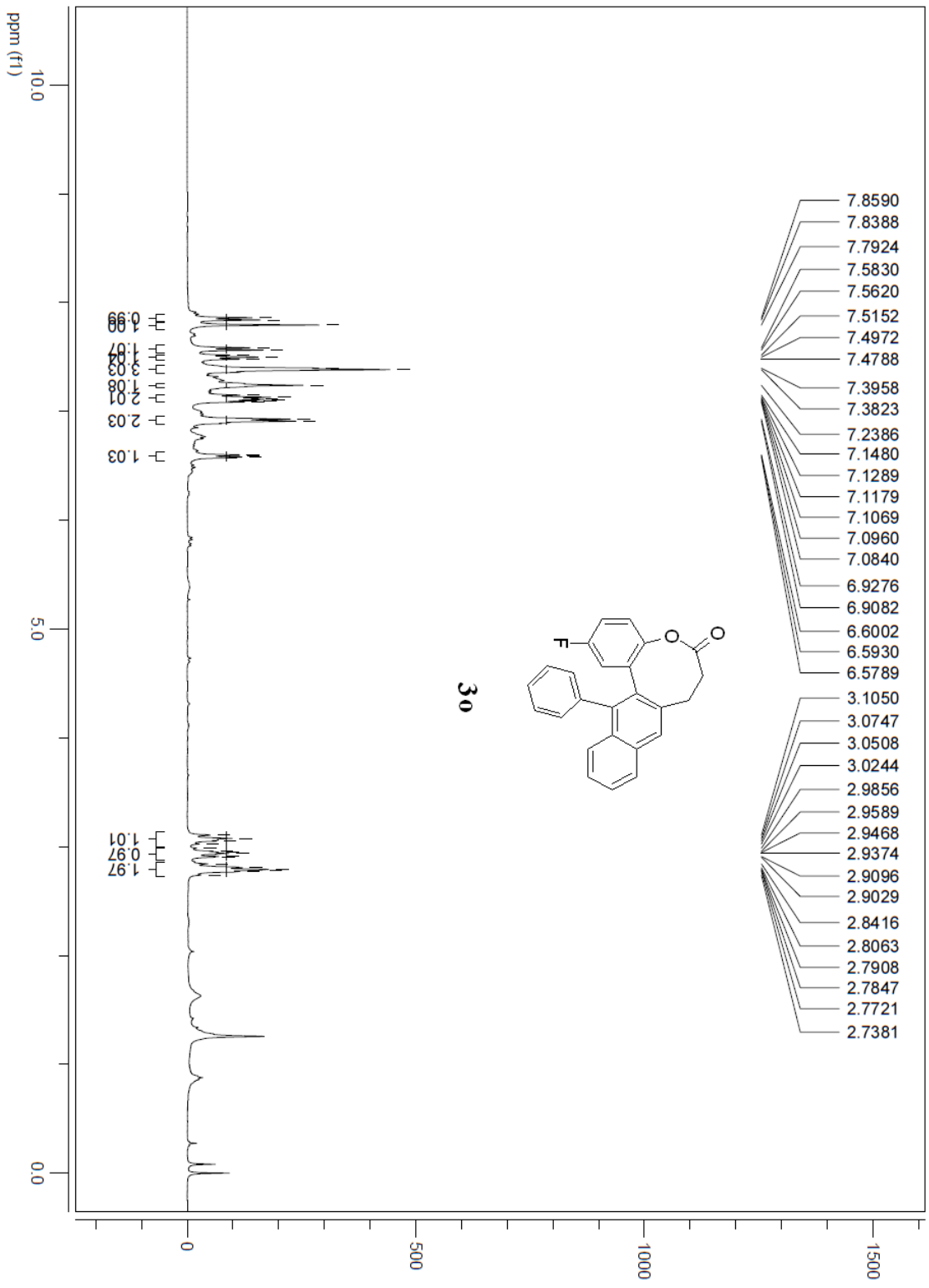
O
Ph
3n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2.01
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3.03
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1.07
1.00
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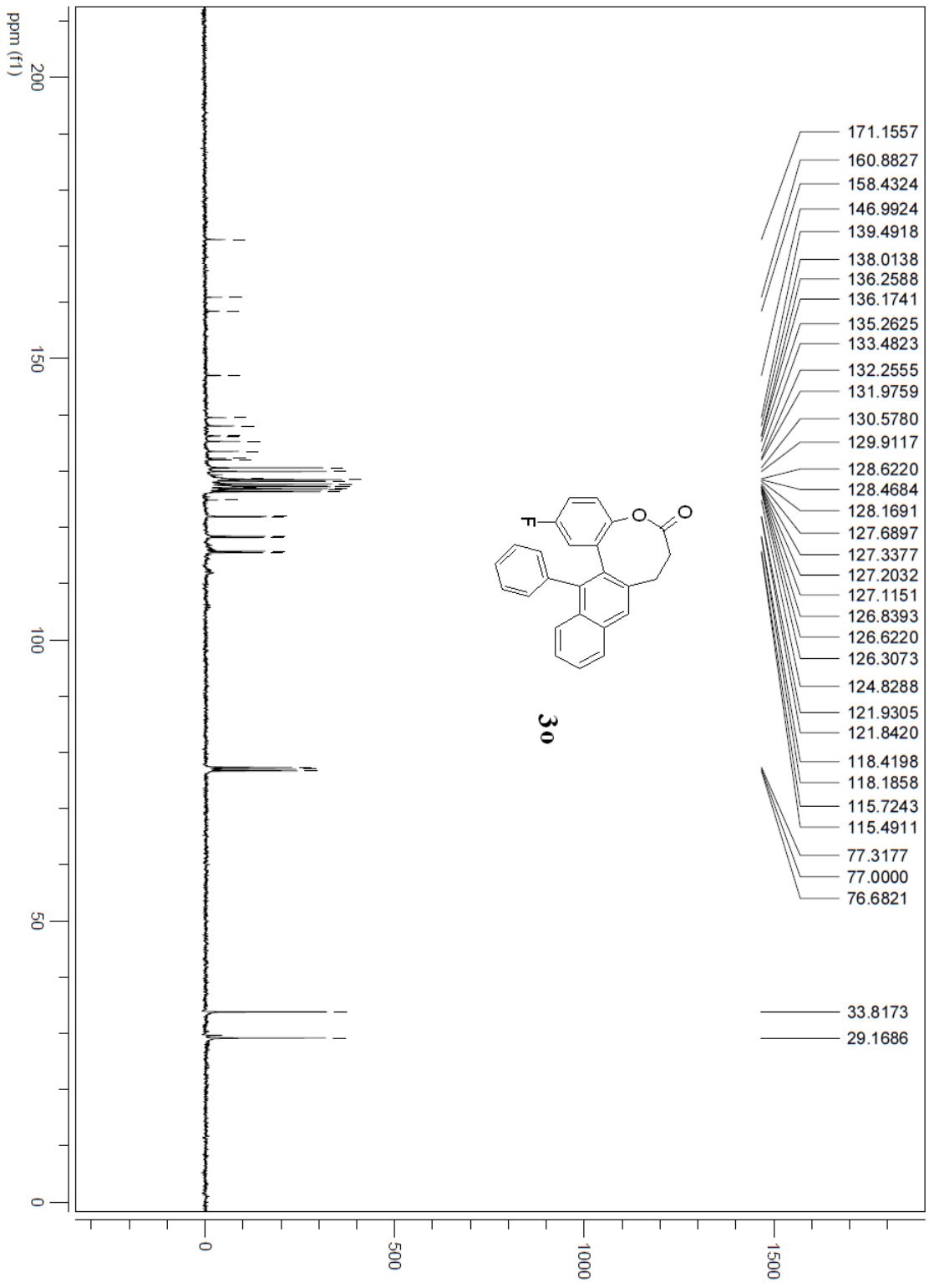
3o
F
O
O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136.2588
136.1741
135.2625
133.4823
132.2555
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130.5780
129.9117
128.6220
128.4684
128.1691
127.6897
127.3377
127.2032
127.1151
126.8393
126.6220
126.3073
124.8288
121.9305
121.8420
118.4198
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77.0000
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F
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O
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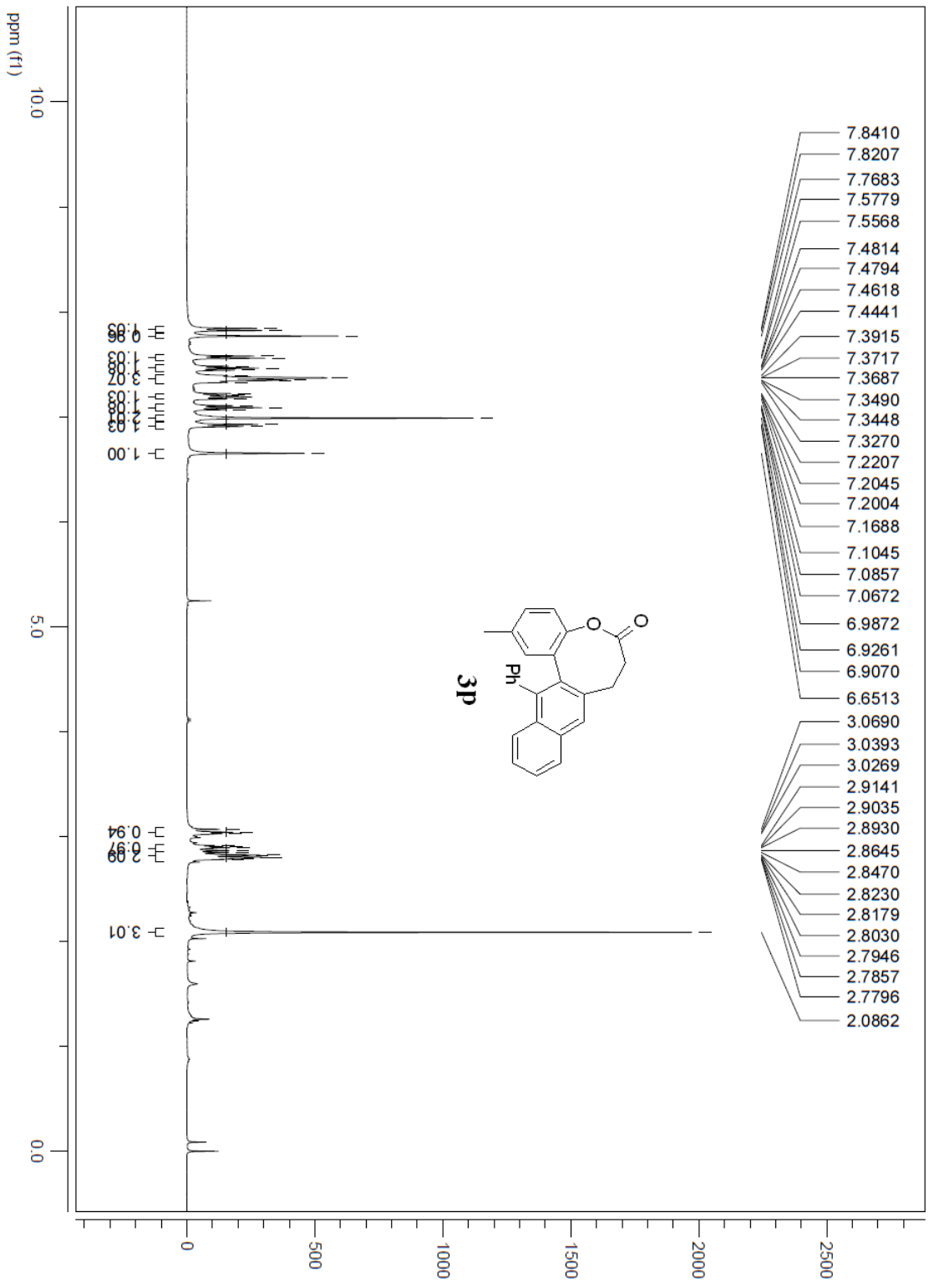
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7.4814
7.4794
7.4618
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7.3915
7.3717
7.3687
7.3490
7.3448
7.3270
7.2207
7.2045
7.2004
7.1688
7.1045
7.0857
7.0672
6.9872
6.9261
6.9070
6.6513
3.0690
3.0393
3.0269
2.9141
2.9035
2.8930
2.8645
2.8470
2.8230
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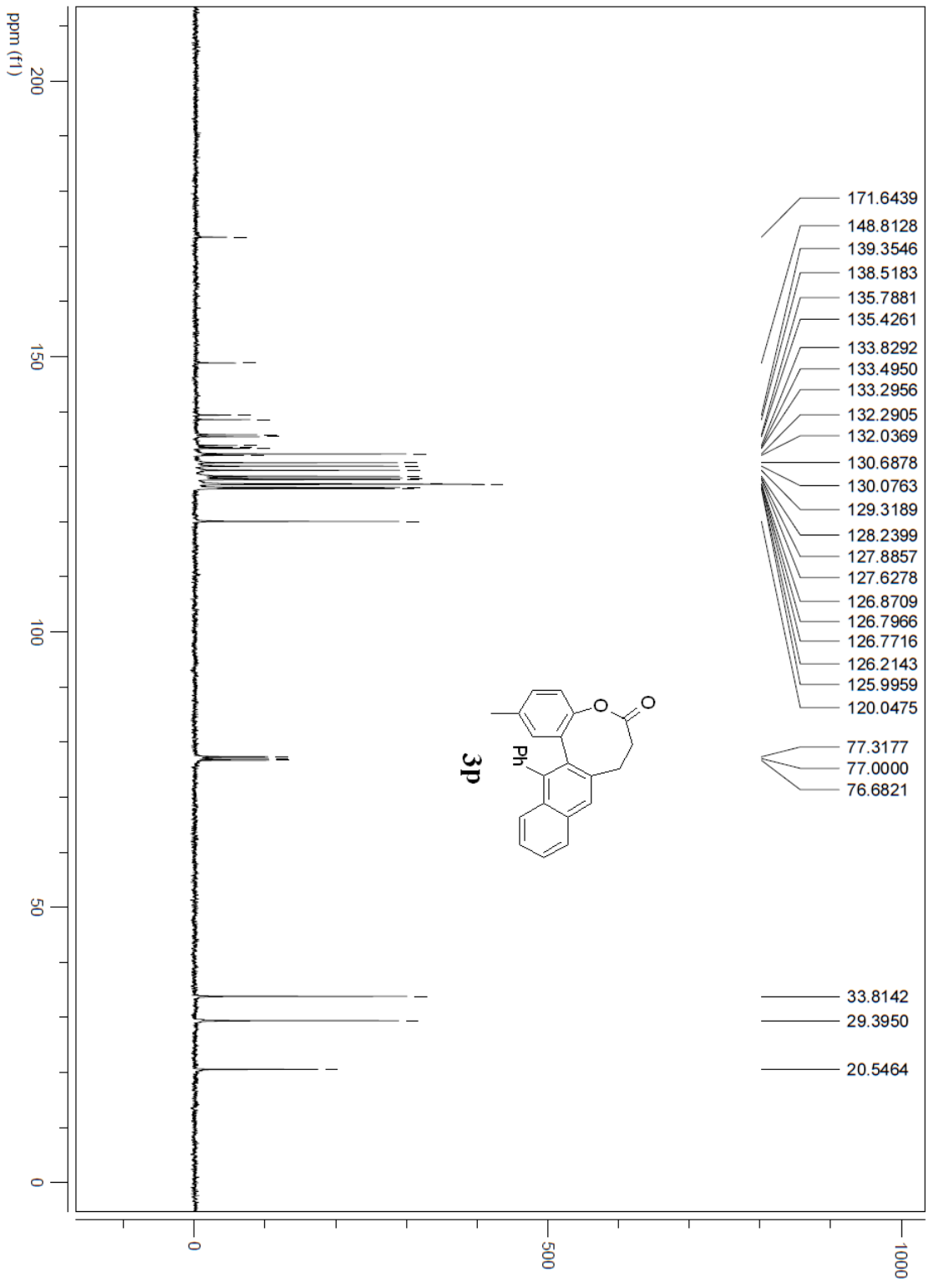
2.7946
2.7857
2.7796
2.0862
1.00
3.01
3p
Ph
O
O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133.8292
133.4950
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132.0369
130.6878
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129.3189
128.2399
127.8857
127.6278
126.8709
126.7966
126.7716
126.2143
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77.0000
76.6821
33.8142
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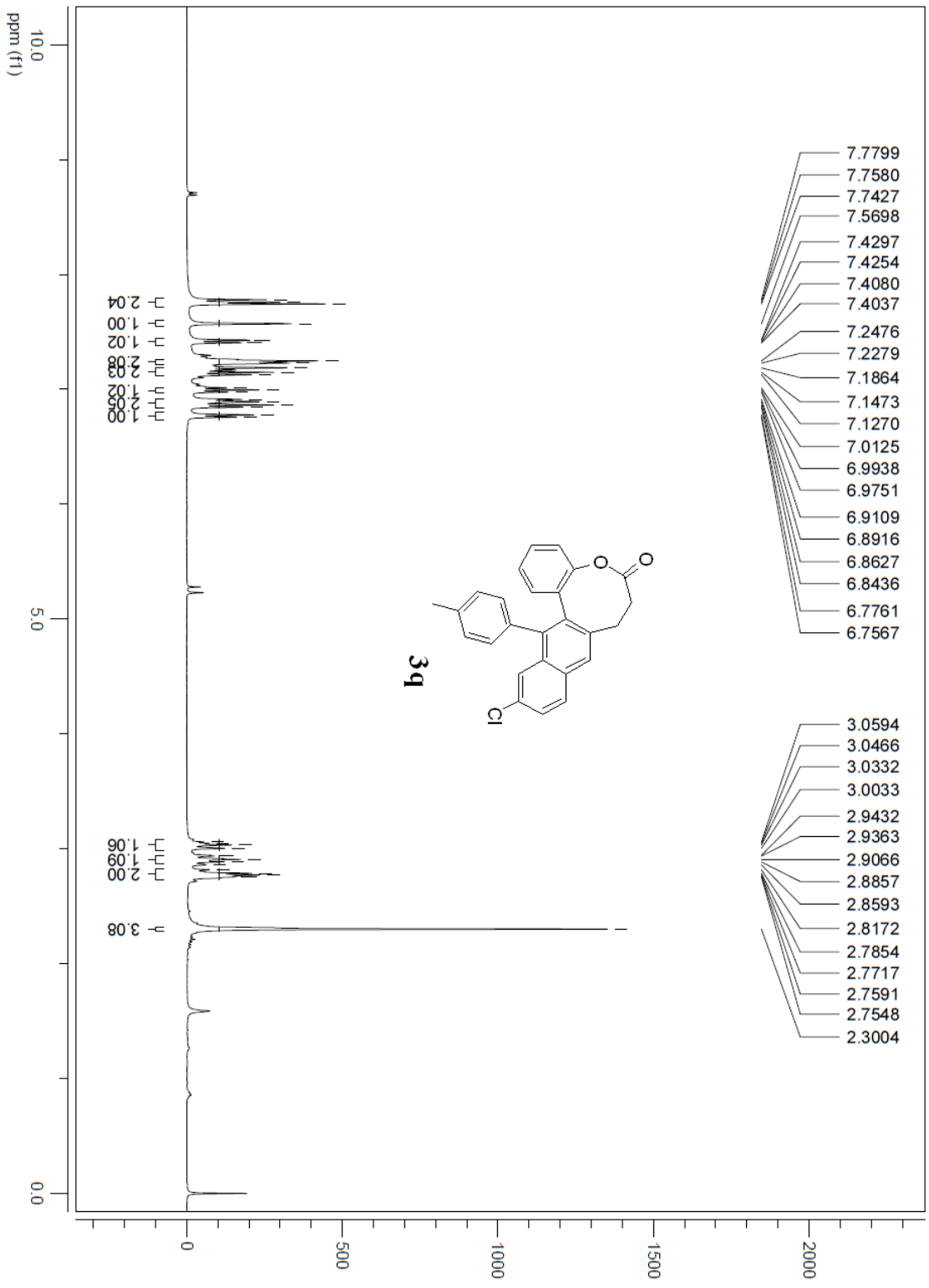
O
O
Ph
3p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7.2279
7.1864
7.1473
7.1270
7.0125
6.9938
6.9751
6.9109
6.8916
6.8627
6.8436
6.7761
6.7567
3.0594
3.0466
3.0332
3.0033
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2.9066
2.8857
2.8593
2.8172
2.7854
2.7717
2.7591
2.7548
2.3004
3g
Cl
O
O
2.04
1.00
1.02
2.08
2.03
1.02
2.05
1.00
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3.08
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1500
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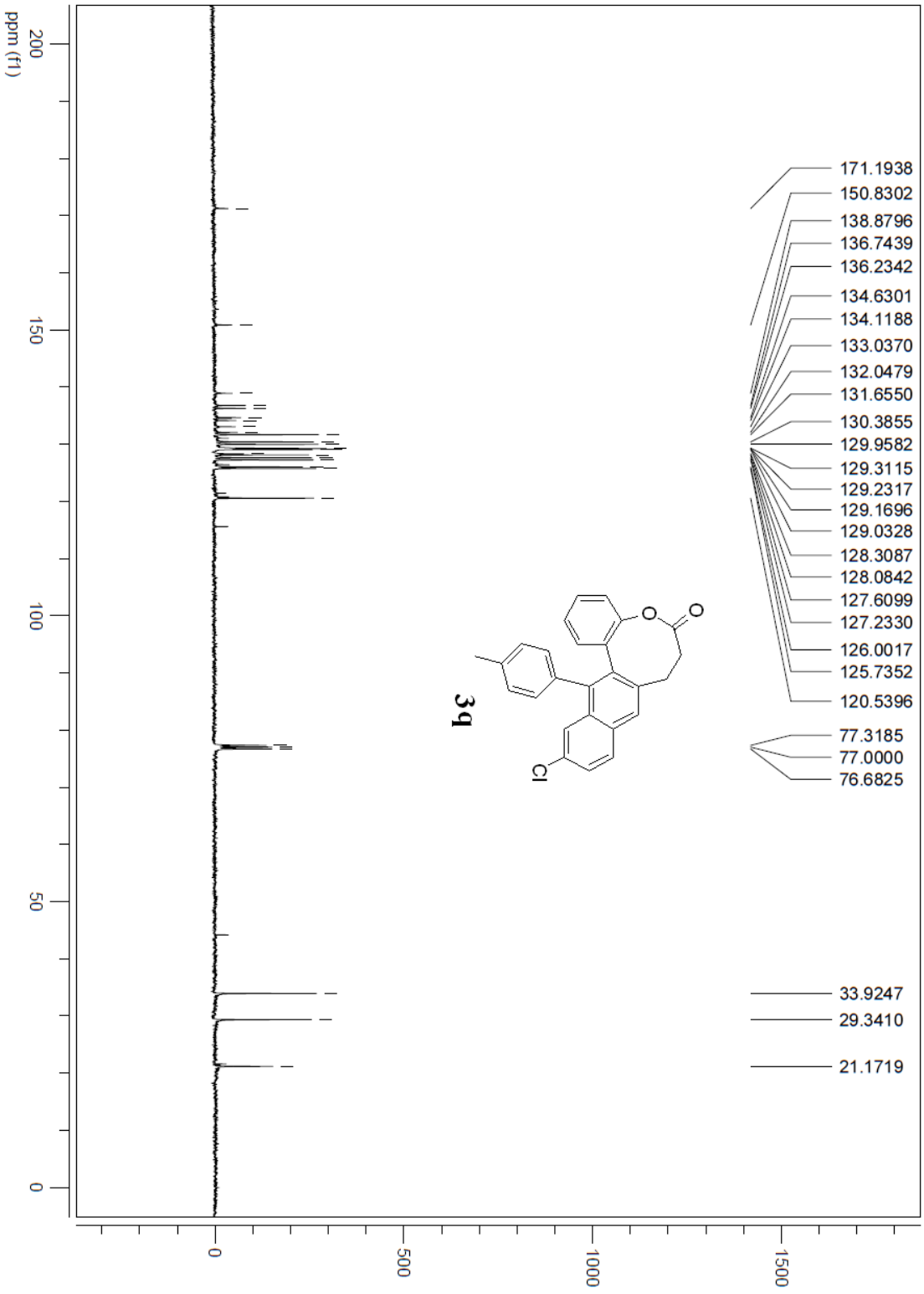
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133.0370
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131.6550
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129.9582
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129.2317
129.1696
129.0328
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128.0842
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127.2330
126.0017
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77.0000
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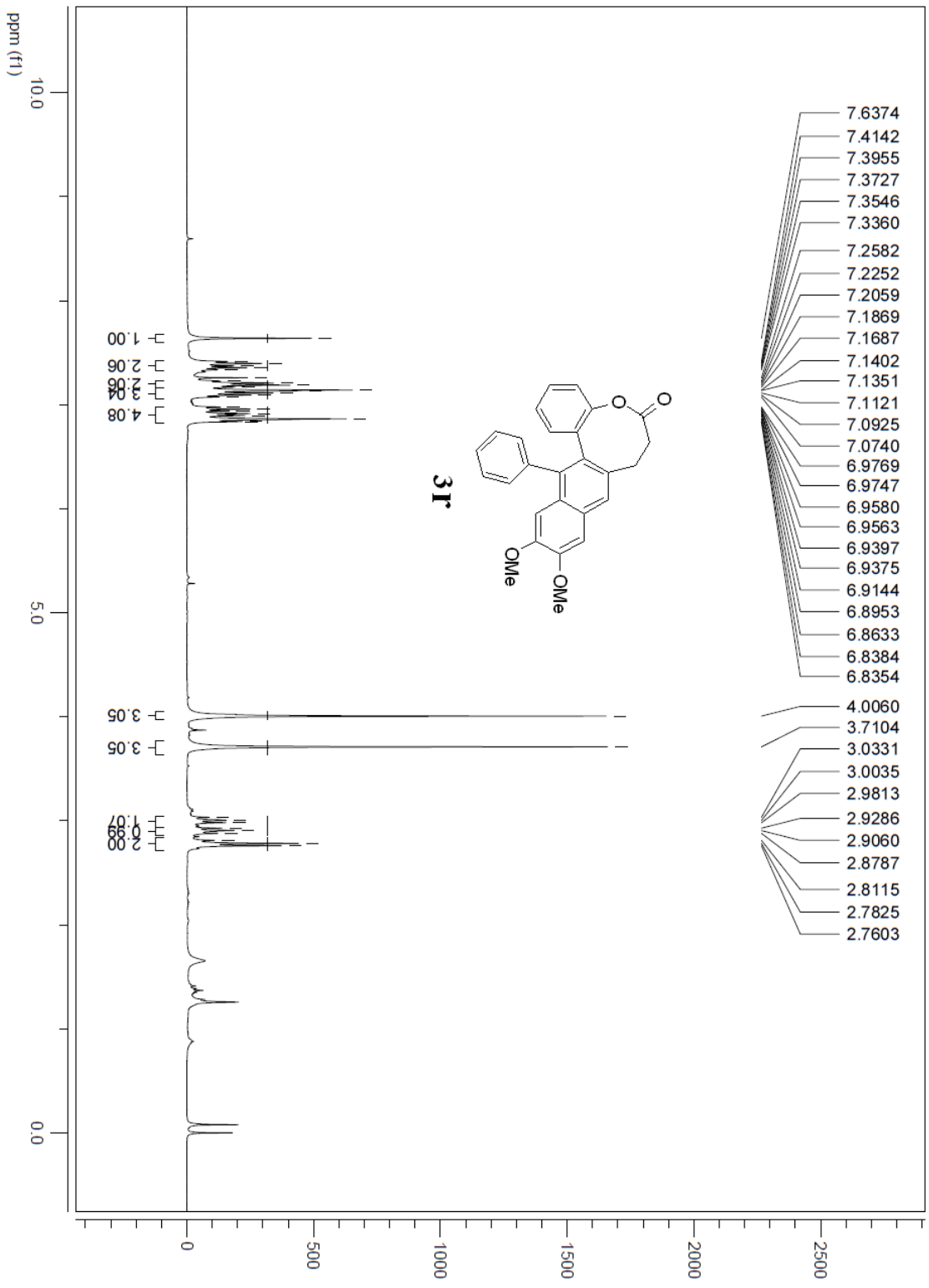
O
Cl
3g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1000
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2000
2500
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2.04
4.08
3.05
3.05
1.07
0.99
2.00
3r
OMe
OMe
O
O
7.6374
7.4142
7.3955
7.3727
7.3546
7.3360
7.2582
7.2252
7.2059
7.1869
7.1687
7.1402
7.1351
7.1121
7.0925
7.0740
6.9769
6.9747
6.9580
6.9563
6.9397
6.9375
6.9144
6.8953
6.8633
6.8384
6.8354
4.0060
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2.8787
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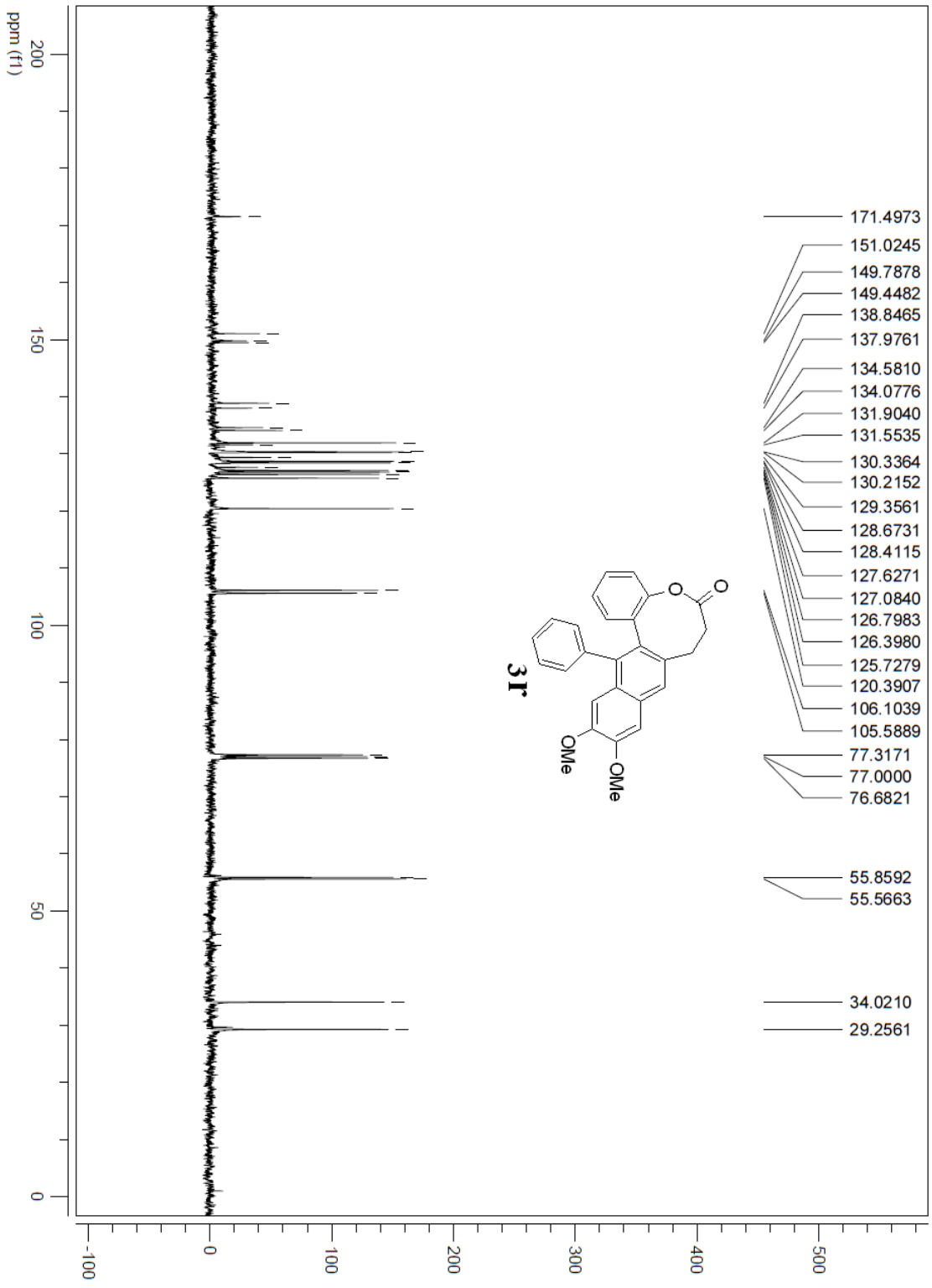
2.7825
2.7603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131.5535
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126.7983
126.3980
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105.5889
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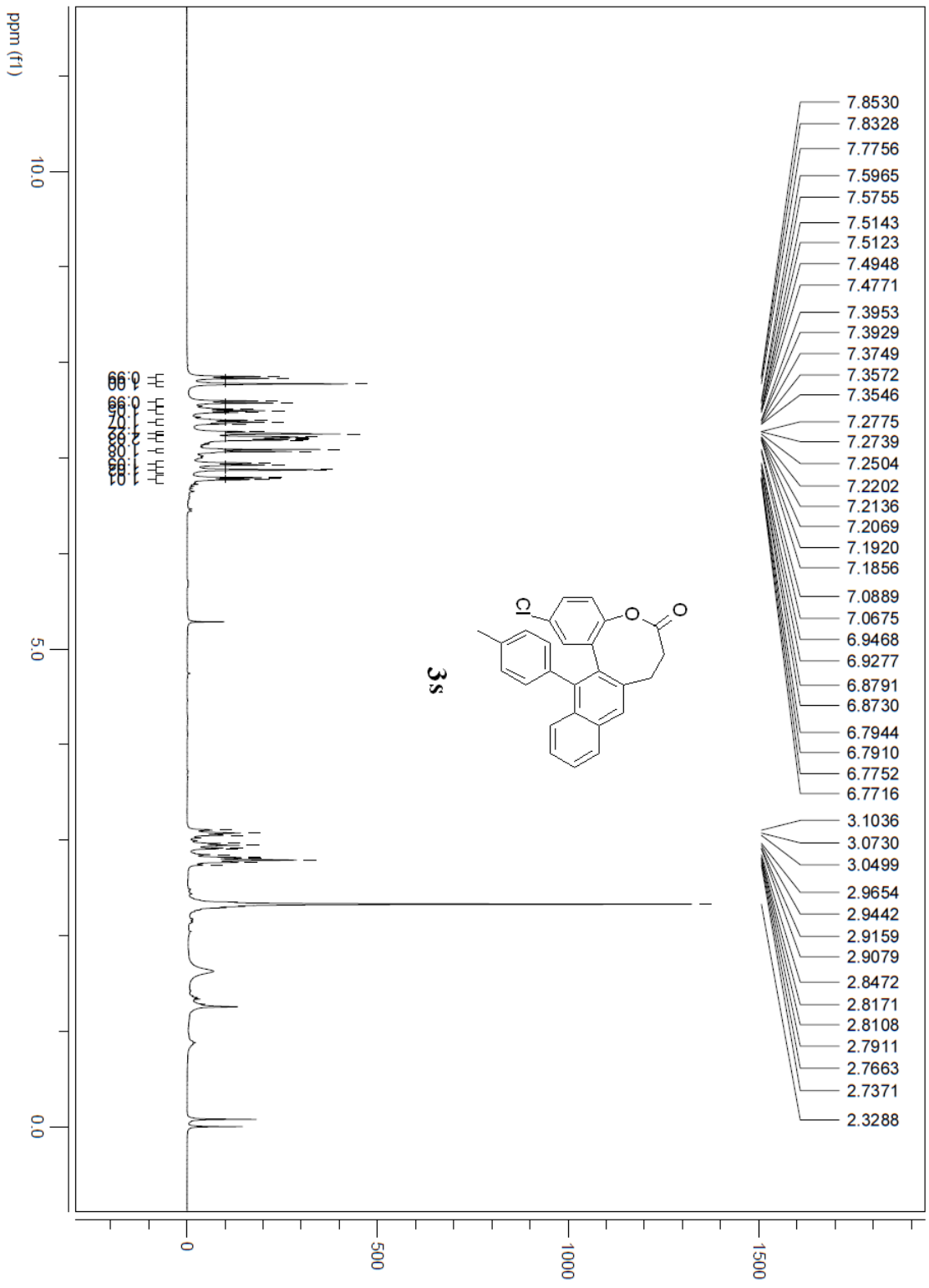
O
O
OMe
OMe
3r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7.5123
7.4948
7.4771
7.3953
7.3929
7.3749
7.3572
7.3546
7.2775
7.2739
7.2504
7.2202
7.2136
7.2069
7.1920
7.1856
7.0889
7.0675
6.9468
6.9277
6.8791
6.8730
6.7944
6.7910
6.7752
6.7716
3.1036
3.0730
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2.9654
2.9442
2.9159
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2.7911
2.7663
2.7371
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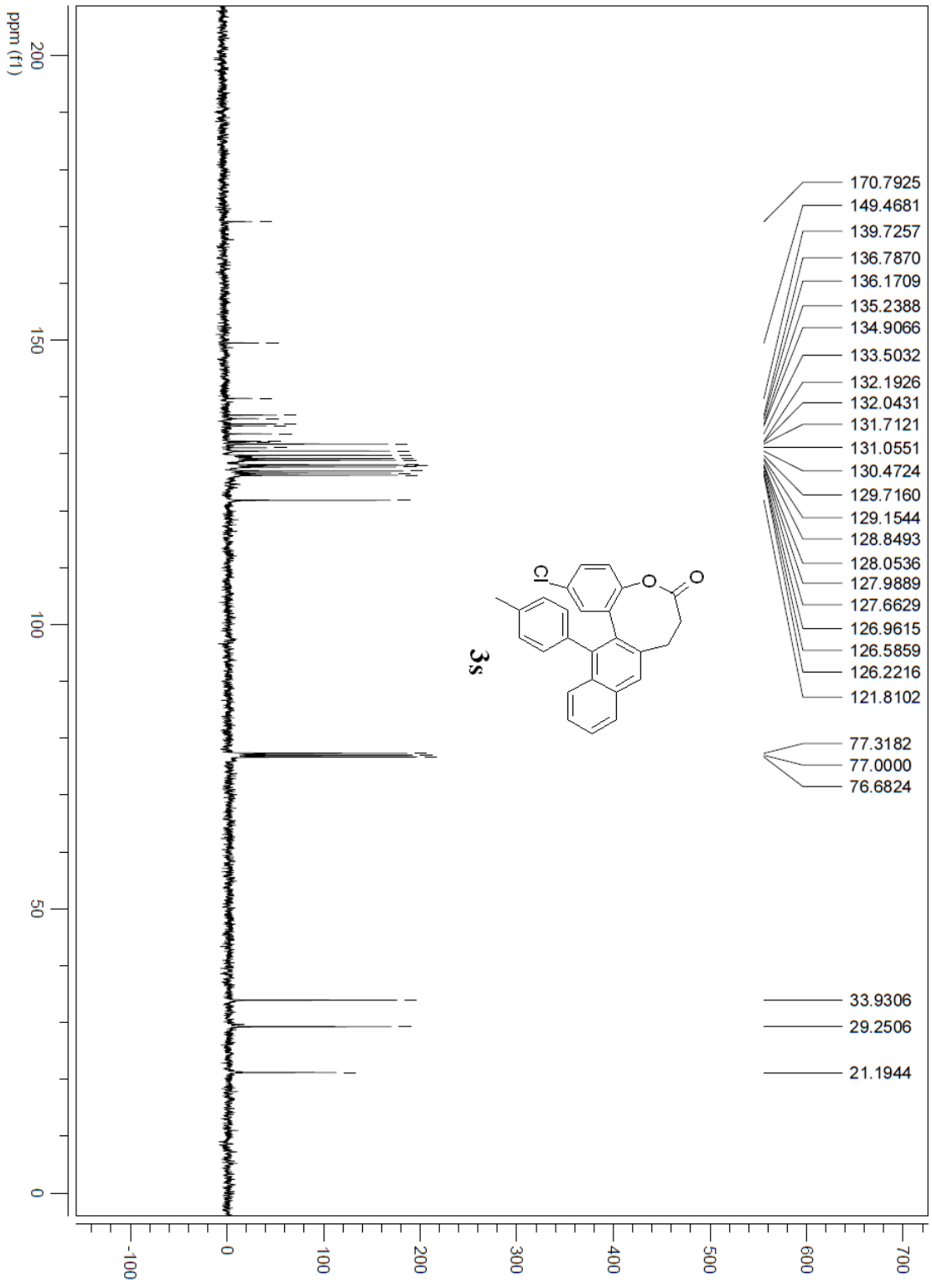
Cl
O
O
3s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600
700
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136.1709
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131.7121
131.0551
130.4724
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21.1944
Cl
O
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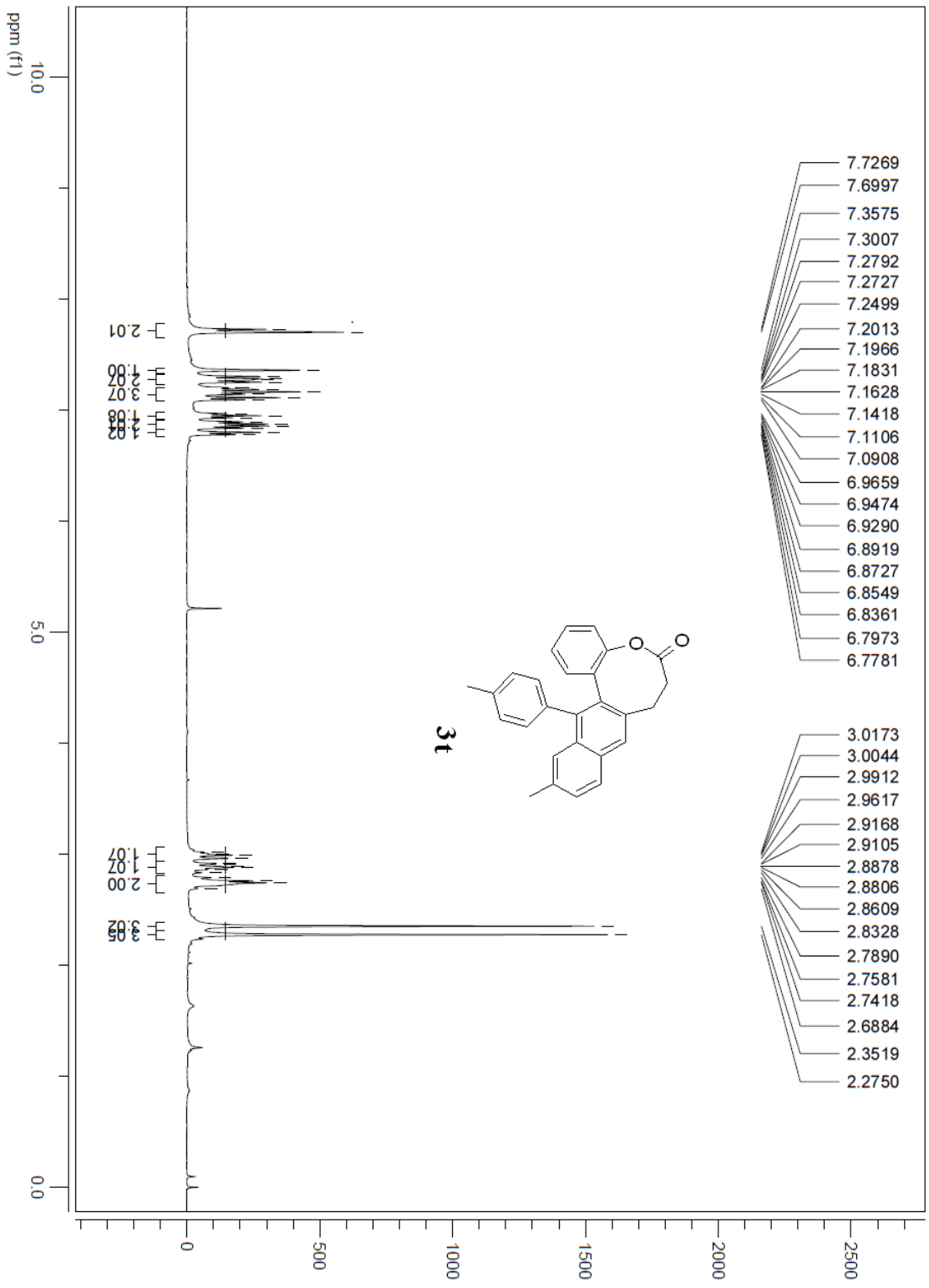
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6.9290
6.8919
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6.7973
6.7781
3.0173
3.0044
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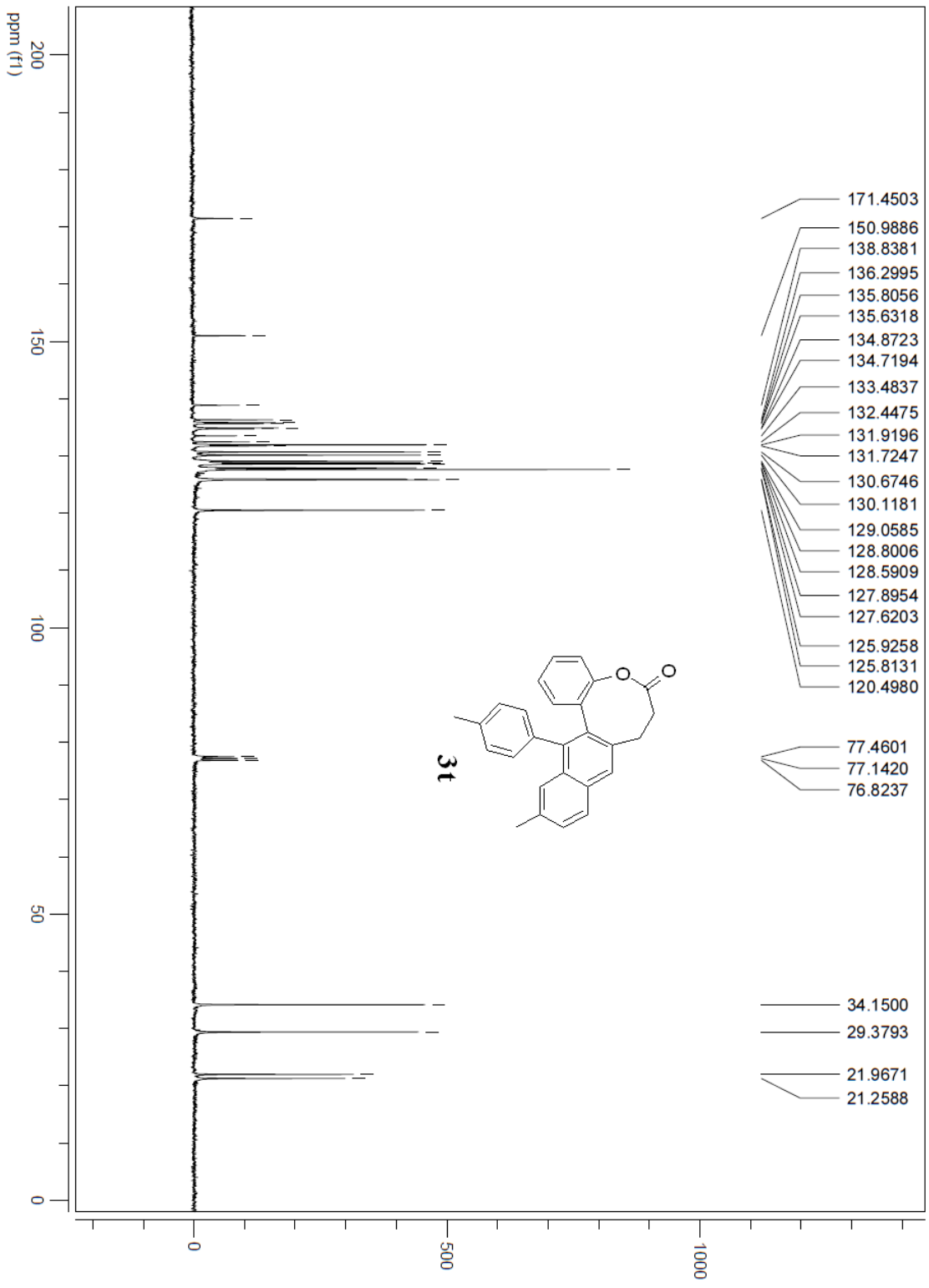
ppm (f1)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131.9196
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130.1181
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O
O
3t