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Supporting Information

Toluene Derivatives as Simple Coupling Precursors for Cascade Palladium-Catalyzed Oxidative C-H bond Acylation of Acetanilides

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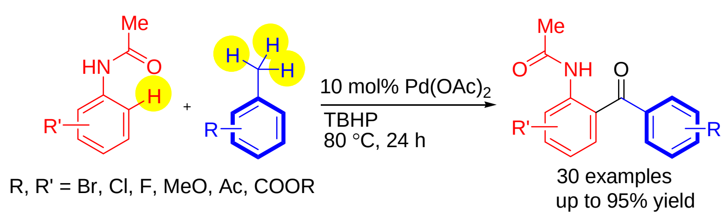

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1. General considerations

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All the reactions were performed in Rotaflo® (England) resealable screw-cap Schlenk flask (approx. 20 mL volume) in the presence of Teflon coated magnetic stirrer bar (4 mm × 10 mm). Dichloroethane (DCE) was distilled under calcium hydride under reduced pressure. Dioxane and toluene were distilled from sodium under nitrogen. Acetonitrile was distilled from calcium hydride under nitrogen prior to use. The concentration of *tert*-butyl hydroperoxide (TBHP) was determined by means of iodometric method.[1] Thin layer chromatography was performed on Merck precoated silica gel 60 F254 plates. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. ^{1}H NMR spectra were recorded on a Bruker (400 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in $CDCl_3$ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^{13}C NMR spectra were referenced to $CDCl_3$ (δ 77.0 ppm, the middle peak). Coupling constants (*J*) were reported in Hertz (Hz). High-resolution mass spectra (HRMS) were obtained on a Brüker APEX 47e FT-ICR mass spectrometer (ESIMS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m × 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP 6890 GC-FID system. Compounds described in the literatures were characterized by comparison of their ^{1}H, and/or ^{13}C NMR spectra to the previously reported data.

2. Preparation of substituted acetanilide substrates

All the substituted acetanilides in Table 3 were prepared from their corresponding precursors with Ac_2O in CH_2Cl_2 according to the literature method without modifications.[2]

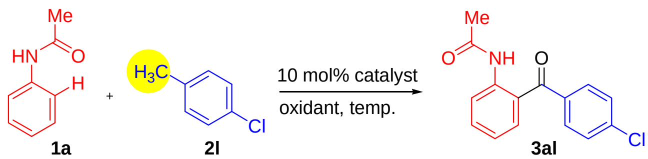
N-phenylbenzamide, *N*-phenylpivalamide and *N*-phenylisobutyramide in Scheme 2 were prepared from phenylamine with their corresponding acyl chlorides in pyridine according to the literature method without modifications.[3]

3. General procedures for reaction condition screenings and coupling reactions

General procedures for screening: Acetanilide (0.135 g, 1.0 mmol) and metal complex (10 mol% or as indicated in Table 1) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar under air. 4-Chlorotoluene (2.0 mL) was added into the tube. The solution was stirred for about 1 to 2 minutes until all solid dissolved. TBHP (12.0 mmol or as indicated in Table 1) were loaded into the tube. The tube was stirred at room temperature for ~5 minutes and then placed into a preheated oil bath (40-120 ^{o}C) for 24 hours. After completion of reaction, the reaction tube was allowed to cool to room temperature. Ethyl acetate (~10 mL), dodecane (227 μL, internal standard) and water (~3 ml) were added. The organic layer was subjected to GC analysis. The GC yield obtained was previously calibrated by authentic sample/dodecane calibration curve.

General procedure for C-H bond coupling of acetanilides and substituted toluene: Substituted acetanilide (1.0 mmol) and $Pd(OAc)_2$ (22.4 mg, 0.10 mmol) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar under air. Substituted toluene (2.0 mL) was added into the tube (Substituted toluene in solid form was added in 80 *equiv.*). TBHP (12.0 mmol) were loaded into the tube. The tube was stirred at room temperature for ~5 minutes and then placed into a preheated oil bath at 80 ^{o}C for 24 hours. After completion of reaction as judged by GC analysis, the reaction tube was allowed to cool to room temperature and quenched with saturated K_2CO_3. The organic layer was separated and the aqueous layer was washed with EtOAc. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired oxidatively coupled product.

Table 1 Screening of reaction conditions[a]

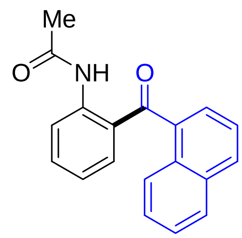


entry	catalyst	oxidant (equiv.)	temp. /°C	%yield[b]
1	$Pd(OAc)_2$	TBHP (6)	100	15
2	$PdCl_2$	TBHP (6)	100	6
3	$Pd(MeCN)_2Cl_2$	TBHP (6)	100	9
4	$Pd(TFA)_2$	TBHP (6)	100	15
5	$Ni(acac)_2$	TBHP (6)	100	0
6	$Rh(PPh_3)_3Cl$	TBHP (6)	100	0
7	$Pd(OAc)_2$	air	100	0
8	$Pd(OAc)_2$	$K_2S_2O_8$ (6)	100	5
9	$Pd(OAc)_2$	$PhI(OAc)_2$ (6)	100	0
10	$Pd(OAc)_2$	$(t\text{-BuO})_2$ (6)	100	6
11	$Pd(OAc)_2$	TBHP (8)	100	39
12	$Pd(OAc)_2$	TBHP (10)	100	76
13	$Pd(OAc)_2$	TBHP (12)	100	88
14	$Pd(OAc)_2$	TBHP (16)	100	89
15	**$Pd(OAc)_2$**	**TBHP (12)**	**80**	**94 (90)**
16	$Pd(OAc)_2$	TBHP (12)	60	66
17	$Pd(OAc)_2$	TBHP (12)	40	58
18[c]	$Pd(OAc)_2$	TBHP (12)	80	11
19[d]	$Pd(OAc)_2$	TBHP (12)	80	0
20[e]	$Pd(OAc)_2$	TBHP (12)	80	6
21[f]	$Pd(OAc)_2$	TBHP (12)	80	60

[a] Reaction conditions: Acetanilide **1a** (1.0 mmol), catalyst (10 mol%), oxidant (as indicated), 4-chlorotoluene **2l** (2 mL) were stirred at indicated reaction temperature for 24 h under air. [b]Calibrated GC yields were reported using dodecane as the internal standard. Isolated yield in parenthesis. [c]DCE as solvent (**2l**, 10 equiv.). [d]Dioxane as solvent (**2l**, 10 equiv.). [e]MeCN as solvent (**2l**, 10 equiv.). [f]5 mol% $Pd(OAc)_2$ was used.

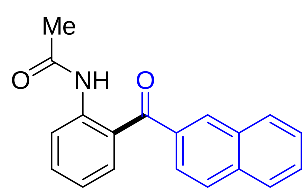
4. Characterization data of coupling products

N-(2-(1-Naphthoyl)phenyl)acetamide (Table 2, product 3ab)[4]



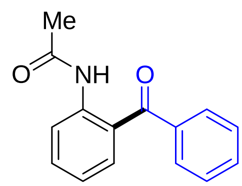
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.49 (s, 1H), 8.69 (d, *J* = 8.5 Hz, 1H), 7.91 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.86-7.79 (m, 2H), 7.50-7.43 (m, 2H), 7.43-7.38 (m, 3H), 7.32 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.92-6.79 (m, 1H), 2.20 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.16, 169.66, 141.54, 137.01, 135.39, 134.82, 133.63, 131.21, 130.63, 128.55, 127.41, 127.18, 126.62, 125.31, 124.43, 123.24, 122.19, 120.87, 25.56.

N-(2-(2-Naphthoyl)phenyl)acetamide (Table 2, product 3ac)[4]



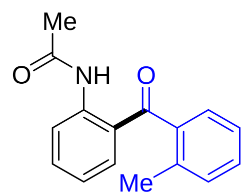
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.79 (s, 1H), 8.66 (d, *J* = 8.3 Hz, 1H), 8.18 (s, 1H), 7.94 (dd, *J* = 12.3, 8.7 Hz, 3H), 7.84 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.68-7.54 (m, 4H), 7.15-7.07 (m, 1H), 2.24 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.38, 172.01, 140.29, 135.94, 135.23, 134.20, 133.49, 132.45, 131.84, 129.55, 128.64, 128.31, 127.83, 127.05, 126.77, 125.42, 122.28, 121.78, 25.25.

N-(2-Benzoylphenyl)acetamide(Table 2, product 3ad) [5]



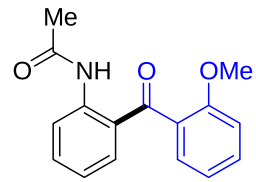
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.81 (s, 1H), 8.60 (d, *J* = 8.2 Hz, 1H), 7.71-7.67 (m, 2H), 7.56 (dd, *J* = 15.8, 7.8 Hz, 3H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.35-7.33 (m, 1H), 7.08 (s, 1H), 2.21 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.75, 169.44, 140.35, 134.28, 133.53, 132.57, 129.92, 128.36, 126.97, 122.18, 121.60, 25.23.

***N*-(2-(2-Methylbenzoyl)phenyl)acetamide (Table 2, product 3ae)** [4]



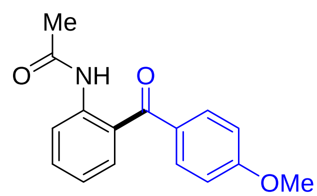
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.56 (s, 1H), 8.75 (d, *J* = 8.5 Hz, 1H), 7.56 (dd, *J* = 11.4, 4.3 Hz, 1H), 7.39 (dd, *J* = 9.4, 2.6 Hz, 1H), 7.29 (d, *J* = 7.7 Hz, 1H), 7.25-7.22 (m, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), 2.29 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.92, 169.57, 141.54, 139.29, 135.90, 135.32, 134.52, 130.94, 130.25, 127.87, 125.38, 122.46, 122.17, 120.74, 25.55, 19.73.

***N*-(2-(2-Methoxybenzoyl)phenyl)acetamide (Table 2, product 3af)**[6]



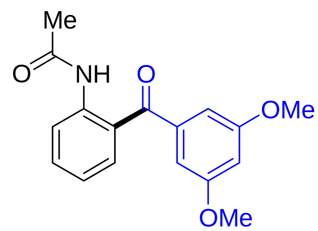
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.61 (s, 1H), 8.75 (d, *J* = 8.5 Hz, 1H), 7.58-7.53 (m, 1H), 7.52-7.44 (m, 2H), 7.32-7.28 (m, 1H), 7.06 (td, *J* = 7.5, 0.7 Hz, 1H), 7.04-6.97 (m, 2H), 3.77 (d, *J* = 3.4 Hz, 3H), 2.26 (d, *J* = 15.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.62, 169.64, 156.81, 141.29, 135.09, 134.55, 131.99, 129.09, 128.50, 126.95, 122.58, 120.49, 120.47, 111.44, 55.64, 25.55.

***N*-(2-(4-Methoxybenzoyl)phenyl)acetamide (Table 2, product 3ag)**[4]



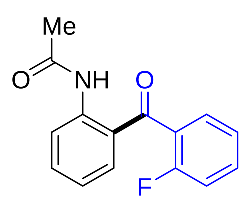
Hexane: EtOAc =1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.44 (s, 1H), 8.47 (d, *J* = 8.6 Hz, 1H), 7.65 (d, *J* = 8.8 Hz, 2H), 7.45 (dd, *J* = 10.1, 3.9 Hz, 2H), 7.01 (dd, *J* = 11.3, 3.8 Hz, 1H), 6.88 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.89, 169.04, 163.45, 139.80, 133.51, 132.72, 132.61, 130.88, 124.27, 122.07, 121.72, 113.64, 55.52, 25.14.

N-(2-(3,5-Dimethoxybenzoyl)phenyl)acetamide (Table 2, product 3ah)[12]



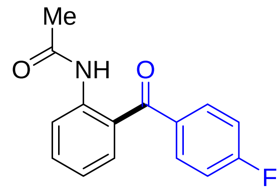
Hexane: EtOAc = 1:5, R_f = 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.69 (s, 1H), 8.53 (d, *J* = 8.3 Hz, 1H), 7.58-7.38 (m, 2H), 7.01 (t, *J* = 7.6 Hz, 1H), 6.73 (d, *J* = 2.2 Hz, 2H), 6.60 (t, *J* = 2.2 Hz, 1H), 3.74 (s, 6H), 2.16 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.40, 169.30, 160.55, 140.45, 140.38, 134.37, 133.51, 122.12, 121.54, 107.65, 104.81, 55.62, 25.26.

N-(2-(2-Fluorobenzoyl)phenyl)acetamide (Table 2, product 3ai) [4]



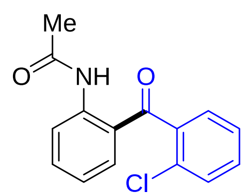
Hexane: EtOAc = 1:5, R_f = 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.36 (s, 1H), 8.75 (d, *J* = 8.5 Hz, 1H), 7.62-7.57 (m, 1H), 7.54 (dd, *J* = 6.7, 1.4 Hz, 1H), 7.52-7.48 (m, 1H), 7.45 (d, *J* = 7.0 Hz, 1H), 7.31 – 7.25 (m, 1H), 7.20 (d, *J* = 9.4 Hz, 1H), 7.09-7.03 (m, 1H), 2.28 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.09, 169.48, 158.4,141.32, 135.57, 134.16, 133.04 (d, J_{CF} = 8.3 Hz), 130.19 (d, J_{CF} = 2.3 Hz), 127.61 (d, J_{CF} = 16.8 Hz), 124.28 (d, J_{CF} = 3.5 Hz), 122.27, 120.79, 116.37 (d, J_{CF} = 21.4 Hz), 25.52.

N-(2-(4-Fluorobenzoyl)phenyl)acetamide (Table 2, product 3aj) [4]



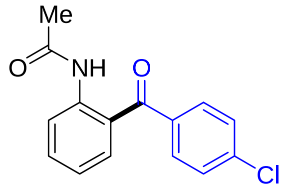
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.65 (s, 1H), 8.61 (d, *J* = 8.4 Hz, 1H), 7.82-7.74 (m, 2H), 7.64-7.49 (m, 2H), 7.19 (t, *J* = 8.6 Hz, 2H), 7.11 (s, 1H), 2.24 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.81, 169.09, 166.53, 164.00, 140.06, 134.56 (d, J_{CF} = 3.1 Hz), 132.90, 132.49 (d, J_{CF} = 9.2 Hz), 127.57 (d, J_{CF} = 155.5 Hz), 123.30, 121.88 (d, J_{CF} = 43.1 Hz), 115.43 (d, J_{CF} = 22.0 Hz), 25.07.

N-(2-(2-Chlorobenzoyl)phenyl)acetamide (Table 2, product 3ak) [4]



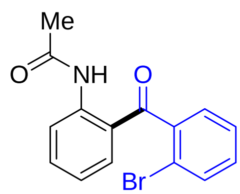
Hexane: EtOAc = 1:5, R_f = 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ11.56 (s, 1H), 8.86-8.74 (m, 1H), 7.60 (s, 1H), 7.51-7.45 (m, 2H), 7.38 (ddd, *J* = 5.5, 3.3, 1.7 Hz, 2H), 7.33 (dd, *J* = 7.5, 1.3 Hz, 1H), 7.06-6.99 (m, 1H), 2.31 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.27, 169.65, 141.92, 138.74, 135.95, 134.60, 131.20, 130.94, 130.14, 128.65, 126.75, 122.28, 121.27, 120.64, 25.63.

***N*-(2-(4-Chlorobenzoyl)phenyl)acetamide (Table 2, product 3al)** [4]



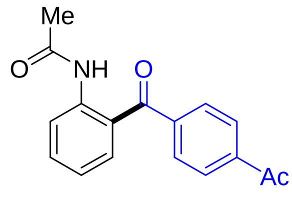
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.70 (s, 1H), 8.62 (d, *J* = 8.4 Hz, 1H), 7.66 (d, *J* = 8.2 Hz, 2H), 7.59 (t, *J* = 7.9 Hz, 1H), 7.49 (dd, *J* = 13.5, 8.1 Hz, 3H), 7.10 (t, *J* = 7.6 Hz, 1H), 2.23 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.92, 168.10, 138.62, 137.81, 135.07, 133.14, 131.21, 130.29, 127.94, 126.01, 123.24, 122.25, 23.99.

***N*-(2-(2-Bromobenzoyl)phenyl)acetamide (Table 2, product 3am)**[4]



Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.54 (s, 1H), 8.80 (d, *J* = 8.5 Hz, 1H), 7.65 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.57 (dd, *J* = 11.4, 4.2 Hz, 1H), 7.39-7.32 (m, 3H), 7.30-7.28 (m, 1H), 7.03-6.98 (m, 1H), 2.29 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.88, 169.63, 142.03, 140.83, 135.94, 134.68, 133.23, 131.23, 128.56, 127.28, 122.27, 120.98, 120.64, 119.25, 25.62.

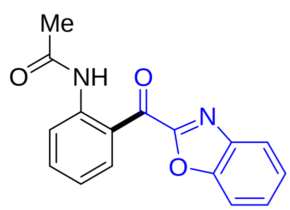
N-(2-(4-Acetylbenzoyl)phenyl)acetamide (Table 2, product 3an)



3an

Hexane: EtOAc = 1:4, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.93 (d, *J* = 8.0 Hz, 1H), 7.82-7.80 (d, *J* = 8.0 Hz, 1H), 7.76-7.74 (d, *J* = 8.0 Hz, 1H), 7.39-7.37 (d, *J* = 8.0 Hz, 1H), 7.25-7.24 (m, 1H), 7.22-7.17 (s, 1H), 2.47 (s, 3H); 2.44 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.64, 197.45, 171.20, 143.99, 140.66, 139.78, 133.58, 130.40, 129.84, 125.91, 122.20, 121.62, 27.00, 24.85. HRMS(H^+): calcd. for $C_{17}H_{16}NO_3$: 282.3218, found 282.3210.

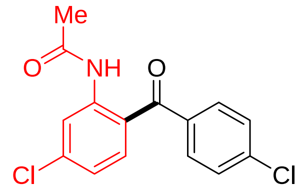
***N*-(2-(benzo*[d]*oxazole-2-carbonyl)phenyl)acetamide (Table 2, product 3ao)**



3ao

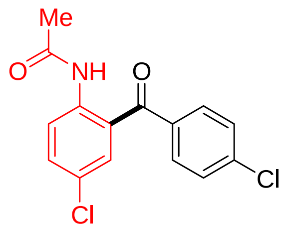
Hexane: EtOAc = 1:2.5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (s, 1H), 8.42 (s, 1H), 7.87-7.85 (d, *J* = 8.0 Hz, 1H), 7.75-7.63 (m, 1H), 7.56-7.52 (t, *J* = 11.4, 4.2 Hz, 1H), 7.43 – 7.41 (m, 2H), 7.39-7.26 (m, 1H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.91, 171.31, 169.39, 160.39, 149.33, 137.38, 132.12, 130.69, 125.91, 124.10, 119.54, 116.59, 114.20, 107.43, 104.17, 24.85. HRMS(H^+): calcd. for $C_{16}H_{12}N_2O_3$: 281.2832, found 282.2829.

N-(5-Chloro-2-(4-chlorobenzoyl)phenyl)acetamide (Table 3, product 3bl)[12]



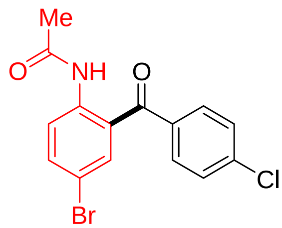
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.87 (s, 1H), 8.75 (d, *J* = 2.0 Hz, 1H), 7.63 (d, *J* = 8.5 Hz, 2H), 7.50-7.44 (m, 3H), 7.07 (dd, *J* = 8.5, 2.0 Hz, 1H), 2.24 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.62, 169.28, 141.63, 141.03, 139.23, 136.65, 134.29, 131.15, 128.83, 122.36, 121.42, 120.79, 25.31.

N-(4-Chloro-2-(4-chlorobenzoyl)phenyl)acetamide (Table 3, product 3cl)[9]



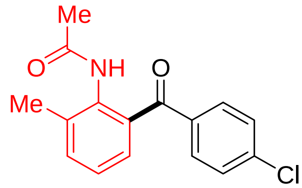
Hexane: EtOAc = 1:3, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.52 (s, 1H), 8.60 (d, *J* = 9.0 Hz, 1H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.56-7.45 (m, 4H), 2.23 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.92, 167.80, 139.06, 137.79, 135.35, 133.14, 131.21, 130.29, 127.94, 126.30, 123.31, 122.25, 24.19.

N-(4-Bromo-2-(4-chlorobenzoyl)phenyl)acetamide (Table 3, product 3dl)[10]



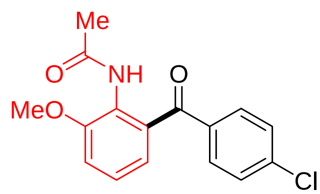
Hexane: EtOAc = 1:3, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.52 (s, 1H), 8.54 (d, *J* = 9.0 Hz, 1H), 7.69-7.63 (m, 3H), 7.61 (d, *J* = 2.3 Hz, 1H), 7.53-7.49 (m, 2H), 2.22 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ196.94, 169.13, 139.65, 139.29, 137.06, 136.08, 135.12, 131.32, 128.97, 124.68, 123.51, 114.68, 25.24.

_N_-(2-(4-Chlorobenzoyl)-6-methylphenyl)acetamide(Table 3, product 3el)[12]



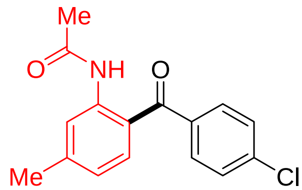
Hexane: EtOAc = 1:3, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.89-7.82 (m, 2H), 7.44-7.37 (m, 2H), 7.16 (t, *J* = 8.0 Hz, 1H), 6.99 (dd, *J* = 8.3, 1.1 Hz, 1H), 6.90 (dd, *J* = 7.7, 1.1 Hz, 1H), 3.84 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.87, 168.20, 151.78, 138.94, 135.41, 133.54, 131.76, 128.38, 124.91, 123.78, 120.92, 112.84, 55.93, 23.58.

_N_-(2-(4-Chlorobenzoyl)-6-methoxyphenyl)acetamide(Table 3, product 3fl)[12]



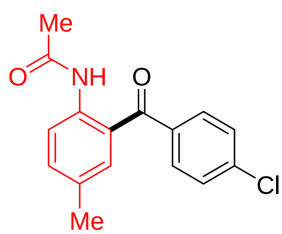
Hexane: EtOAc = 1:3, R_f= 0.4; 1H NMR (400 MHz, $CDCl_3$) δ 8.73 (s, 1H), 7.71 (d, *J* = 8.5 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.24-7.20 (m, 1H), 7.06 (d, *J* = 5.0 Hz, 2H), 2.12 (s, 3H), 1.85 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.39, 168.96, 139.61, 135.52, 135.39, 134.02, 133.89, 131.94, 128.58, 127.34, 125.30, 77.46, 77.14, 76.82, 23.18, 18.47.

N-(2-(4-Chlorobenzoyl)-5-methylphenyl)acetamide (Table 3, product 3gl)[12]



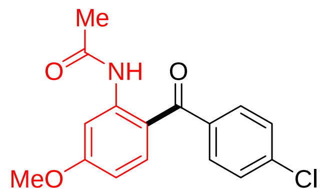
Hexane: EtOAc = 1:2, R_f = 0.3; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.90 (s, 1H), 8.49 (s, 1H), 7.64-7.58 (m, 2H), 7.51-7.43 (m, 2H), 7.40 (d, *J* = 8.1 Hz, 1H), 6.89 (dd, *J* = 8.0, 0.8 Hz, 1H), 2.43 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (101 MHz, d-DMSO) δ 194.06, 168.08, 142.33, 137.12, 136.27, 131.21, 131.10, 130.12, 128.71, 128.19, 124.67, 123.50, 23.13, 21.04.

N-(2-(4-Chlorobenzoyl)-4-methylphenyl)acetamide (3hl)[12]



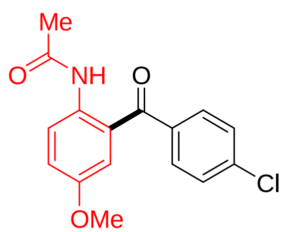
Hexane: EtOAc = 1:3, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.74 (s, 1H), 8.61 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 8.1 Hz, 2H), 7.57 (t, *J* = 7.6 Hz, 2H), 7.31 (s, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 2.46 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (101 MHz, d-DMSO) δ 199.36, 169.22, 143.53, 140.19, 135.82, 133.97, 133.29, 130.23, 129.04, 123.68, 122.05, 121.58, 25.27, 21.67.

***N*-(2-(4-Chlorobenzoyl)-5-methoxyphenyl)acetamide (Table 3, product 3il)**[12]



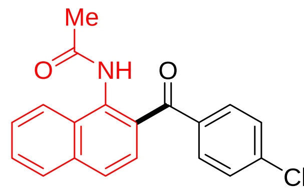
Hexane: EtOAc = 1:3, R_f= 0.3; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.48 (s, 1H), 8.40 (d, *J* = 2.6 Hz, 1H), 7.62-7.56 (m, 2H), 7.51-7.44 (m, 3H), 6.59 (dd, *J* = 8.9, 2.6 Hz, 1H), 3.92 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.62, 169.66, 164.84, 143.91, 138.10, 137.75, 135.93, 130.78, 128.59, 115.21, 109.11, 104.88, 55.71, 25.57.

***N*-(2-(4-Chlorobenzoyl)-4-methoxyphenyl)acetamide (Table 3, product 3jl)**[12]



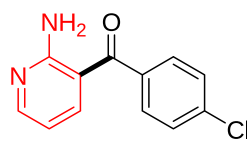
Hexane: EtOAc = 1:3, R_f= 0.3; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.20 (s, 1H), 8.46 (d, *J* = 9.1 Hz, 1H), 7.70 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.15 (dd, *J* = 9.1, 3.0 Hz, 1H), 6.99 (d, *J* = 3.0 Hz, 1H), 3.77 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (101 MHz, d-DMSO) δ193.21, 167.89, 155.79, 137.38, 135.64, 132.73, 131.27, 131.10, 128.22, 125.77, 117.13, 113.84, 55.42, 22.57.

N-(2-(4-Chlorobenzoyl)naphthalen-1-yl)acetamide (Table 3, product 3kl)[12]



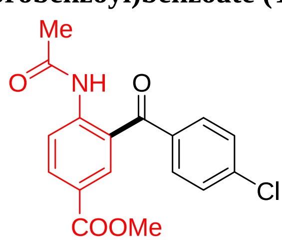
Hexane: EtOAc = 1:3, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.91 (s, 1H), 7.95 (dd, *J* = 17.6, 8.3 Hz, 2H), 7.80 (d, *J* = 8.3 Hz, 3H), 7.59 (dd, *J* = 18.0, 6.1 Hz, 2H), 7.53 (s, 2H), 7.46-7.42 (m, 2H), 2.19 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.86, 169.80, 139.72, 135.35, 134.84, 131.92, 131.36, 130.38, 128.83, 128.65, 128.07, 127.77, 126.88, 125.70, 124.88, 124.18, 23.54.

(2-Aminopyridin-3-yl)(4-chlorophenyl)methanone (Table 3, product 3ll)[12]



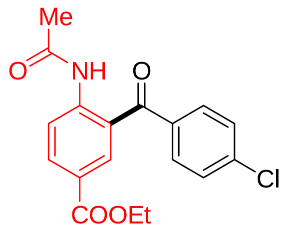
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ9.07 (s, 1H), 8.38 (d, *J* = 8.4 Hz, 1H), 8.19 (s, 1H), 7.91-7.84 (m, 2H), 7.81-7.70 (m, 1H), 7.45 (d, *J = 8.6 Hz,* 2H), 7.06 (dd, *J* = 6.9, 5.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.84, 151.49, 147.82, 138.58, 132.71, 129.07, 128.75 120.09, 114.38.

Methyl 4-acetamido-3-(4-chlorobenzoyl)benzoate (Table 3, product 3ml)[12]



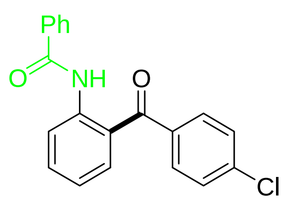
Hexane: EtOAc = 1:5, R_f= 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.86 (s, 1H), 8.69 (d, *J* = 9.4 Hz, 2H), 8.17 – 8.12 (m, 3H), 7.59 (d, *J* = 8.5 Hz, 2H), 7.43 (d, *J* = 8.5 Hz, 2H), 3.81 (s, 2H), 2.19 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ196.86, 168.40, 143.23, 138.54, 135.35, 134.41, 133.70, 130.31, 127.95, 127.65, 122.67, 121.09, 120.00, 51.30, 24.40.

Ethyl 4-acetamido-3-(4-chlorobenzoyl)benzoate (Table 3, product 3nl)[12]



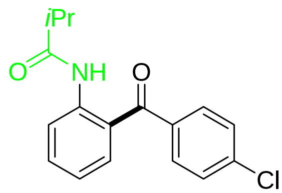
Hexane: EtOAc = 1:5, R_f = 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.93 (s, 1H), 8.76 (d, *J* = 9.4 Hz, 1H), 8.23 (s, 2H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.38 (s, 1H), 4.36 (d, *J* = 7.1 Hz, 2H), 2.27 (s, 3H), 1.37 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.85, 168.36, 143.13, 138.52, 135.36, 134.31, 133.69, 130.32, 127.91, 127.53, 123.05, 121.10, 119.93, 60.26, 24.38, 13.28.

***N*-(2-(4-Chlorobenzoyl)phenyl)benzamide (Scheme 2, product 5)**[11]



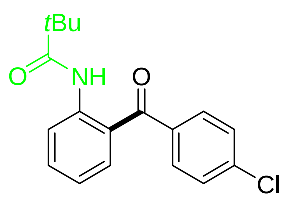
Hexane: EtOAc = 1:5, R_f = 0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.83 (s, 1H), 8.68 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 8.5 Hz, 2H), 7.65 (d, *J* = 8.5 Hz, 2H), 7.57-7.52 (m, 5H), 7.46 (d, *J* = 7.4 Hz, 1H), 7.36 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.30 (d, *J* = 4.4 Hz, 1H), 7.26 (d, *J* = 7.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.97, 190.95, 141.06, 138.99, 137.00, 134.87, 134.49, 133.70, 132.16, 130.92, 129.47, 128.88, 127.40, 122.86, 122.31, 121.64.

N-(2-(4-Chlorobenzoyl)phenyl)isobutyramide (Scheme 2, product 6)[12]



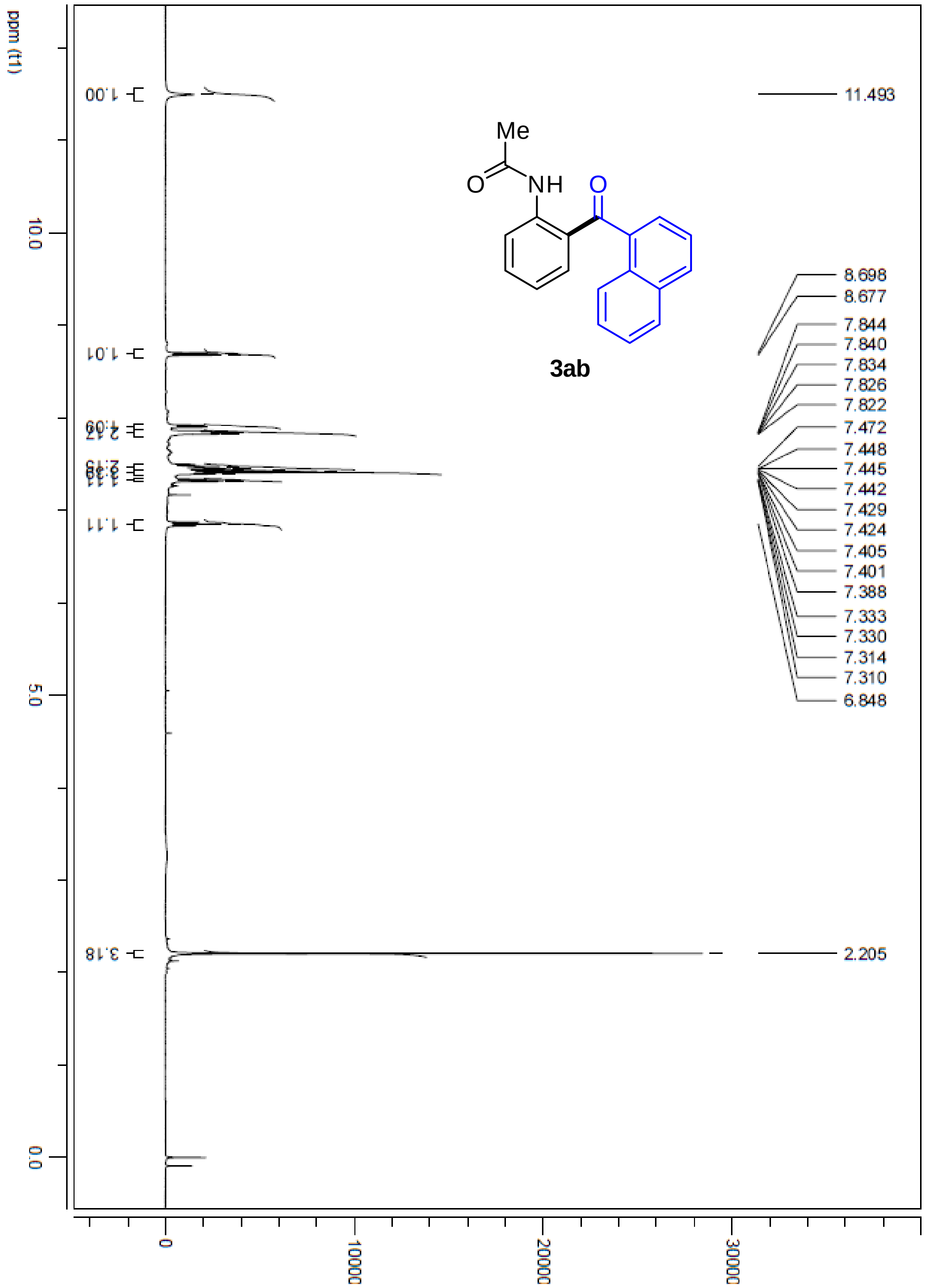
Hexane: EtOAc = 1:10, R_f= 0.3; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.87 (s, 1H), 8.69 (d, *J* = 8.4 Hz, 1H), 7.65 (d, *J* = 8.5 Hz, 2H), 7.58 (s, 1H), 7.55-7.53 (m, 1H), 7.47 (d, *J* = 8.5 Hz, 2H), 7.08 (s, 1H), 1.30 (s, 3H), 1.28 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.54, 176.56, 140.93, 138.94, 137.03, 134.63, 133.39, 131.40, 128.99, 128.92, 121.98, 121.64, 37.09, 19.50.

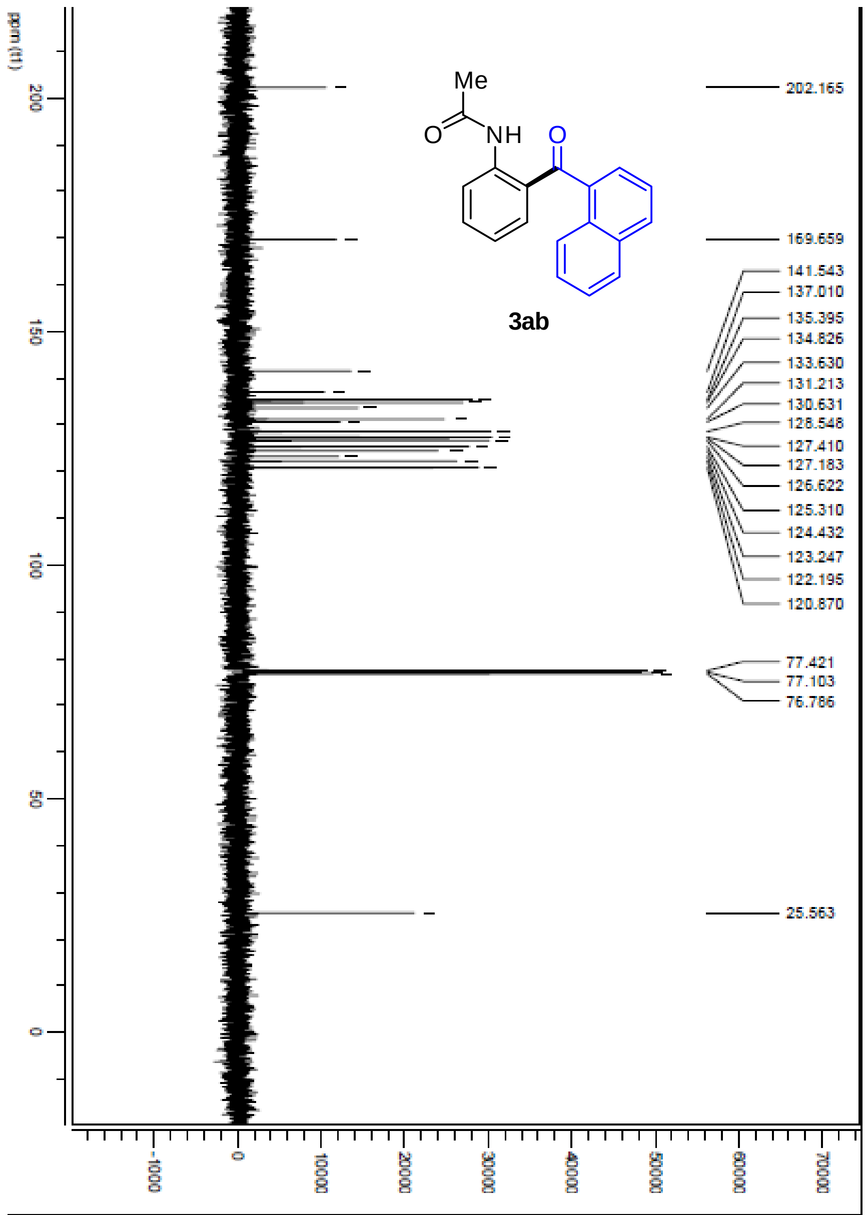
N-(2-(4-Chlorobenzoyl)phenyl)pivalamide (Scheme 2, product 7)[12]



Hexane: EtOAc =1:10, R_f= 0.3; NMR (400 MHz, $CDCl_3$) δ 11.12 (s, 1H), 8.73 (s, 1H), 8.71 (d, *J* = 8.5 Hz, 2H), 7.65 (d, *J* = 8.5 Hz, 1H), 7.60-7.56 (m, 2H), 7.48 (s, 1H), 7.10 – 7.07 (m, 1H), 1.35 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.50, 177.74, 141.19, 138.84, 137.09, 134.65, 133.51, 131.29, 128.90, 128.65, 121.92, 121.55, 40.18, 27.79.

5. ^{1}H and ^{13}C NMR and HRMS spectra

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131.213
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128.548
127.410
127.183
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125.310
124.432
123.247
122.195
120.870
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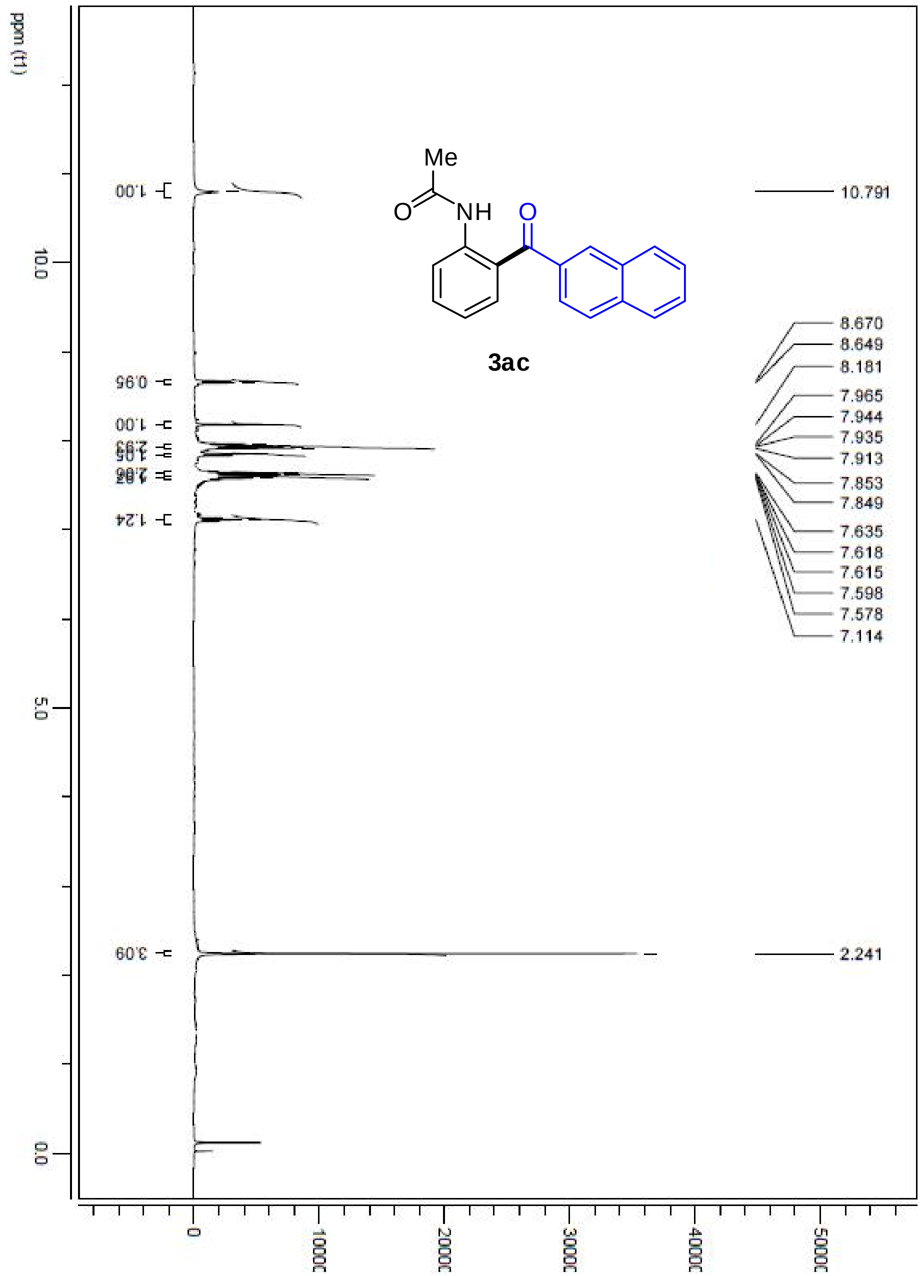
77.103
76.786
25.563
ppm (t1)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7.849
7.635
7.618
7.615
7.598
7.578
7.114
2.241
1.00
0.95
1.00
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1.24
3.09
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0.0
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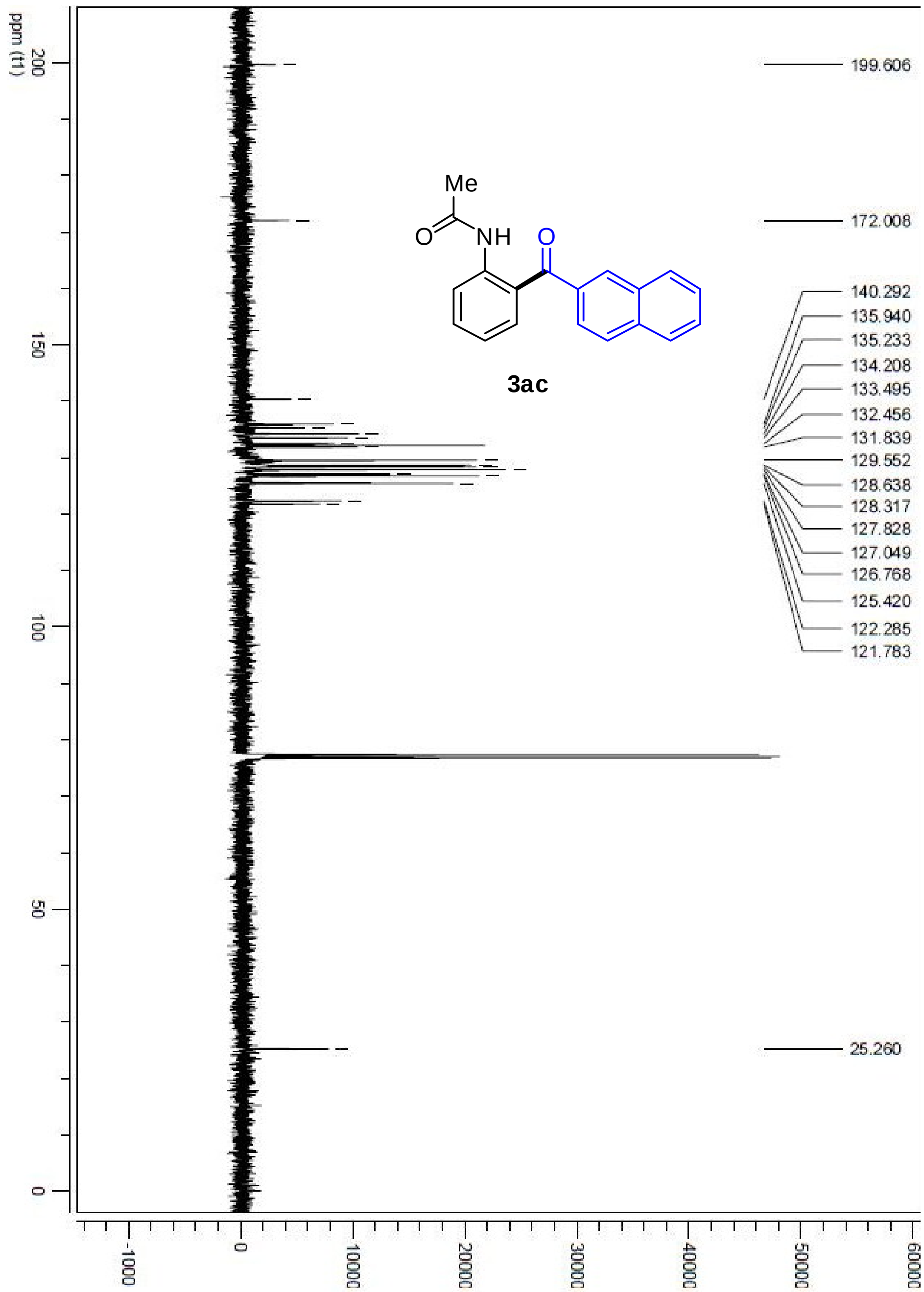
3000
4000
5000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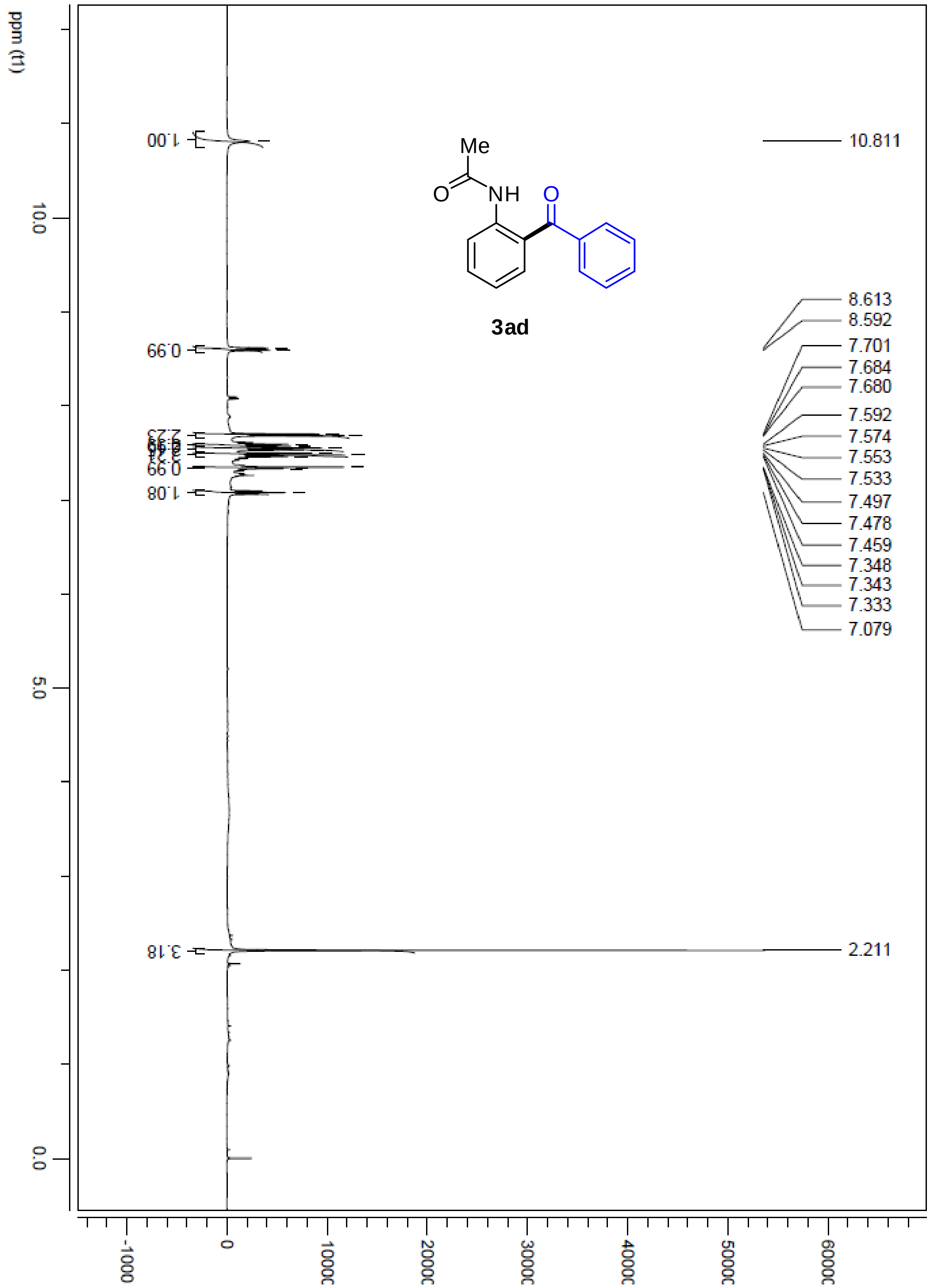

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7.459
7.348
7.343
7.333
7.079
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0.99
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0
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30000
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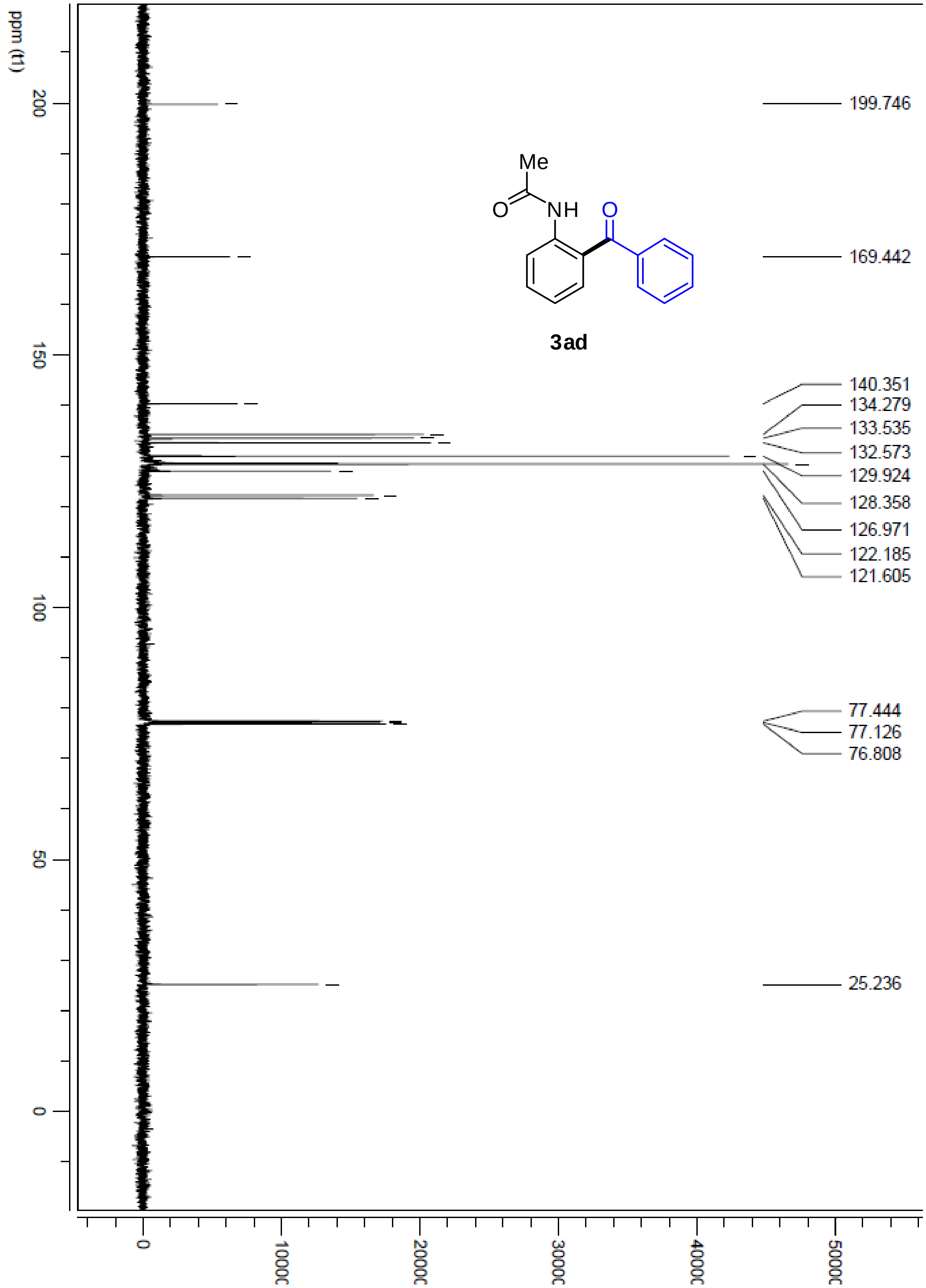
60000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25.236
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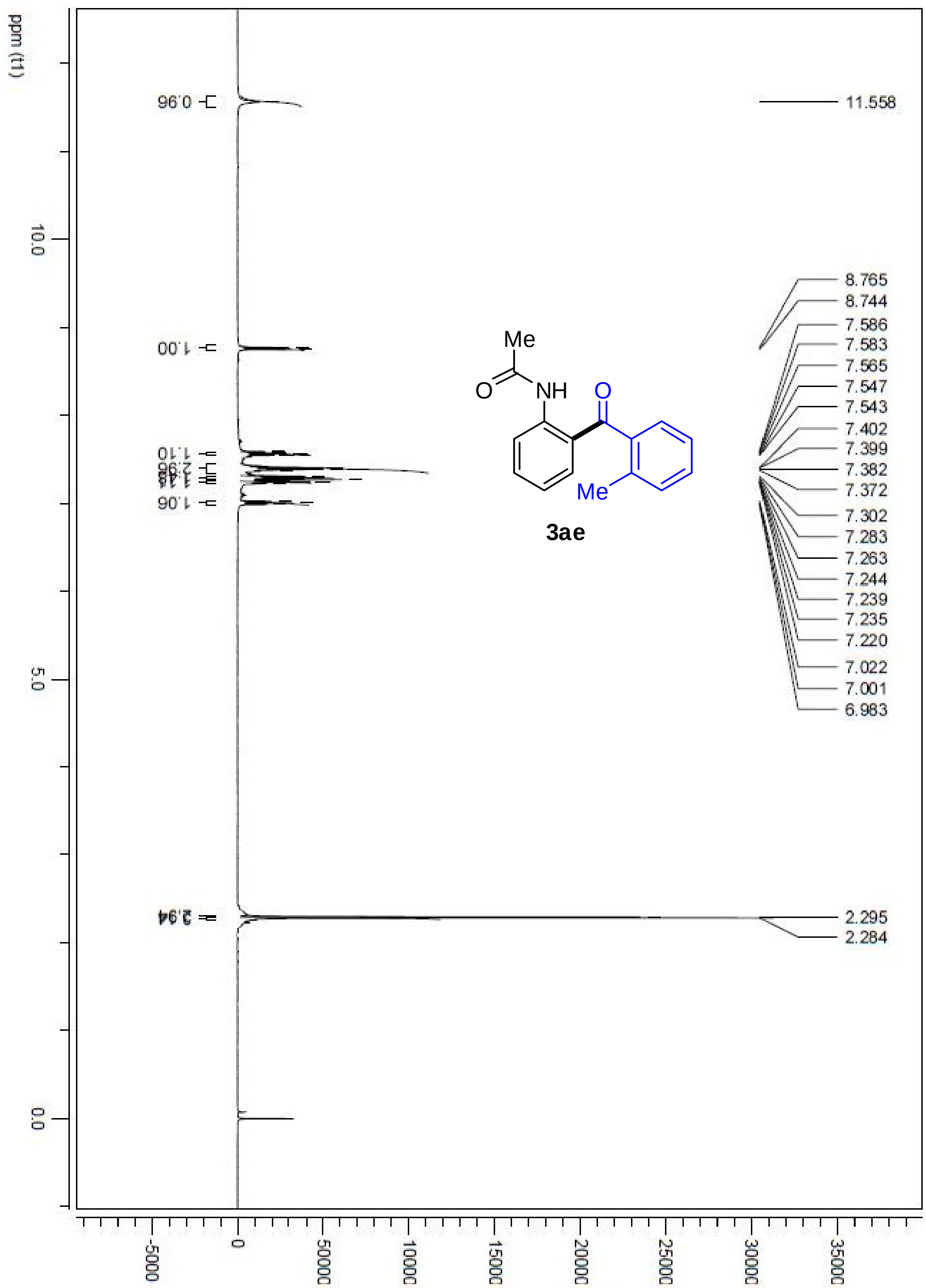
O
NH
O
3ad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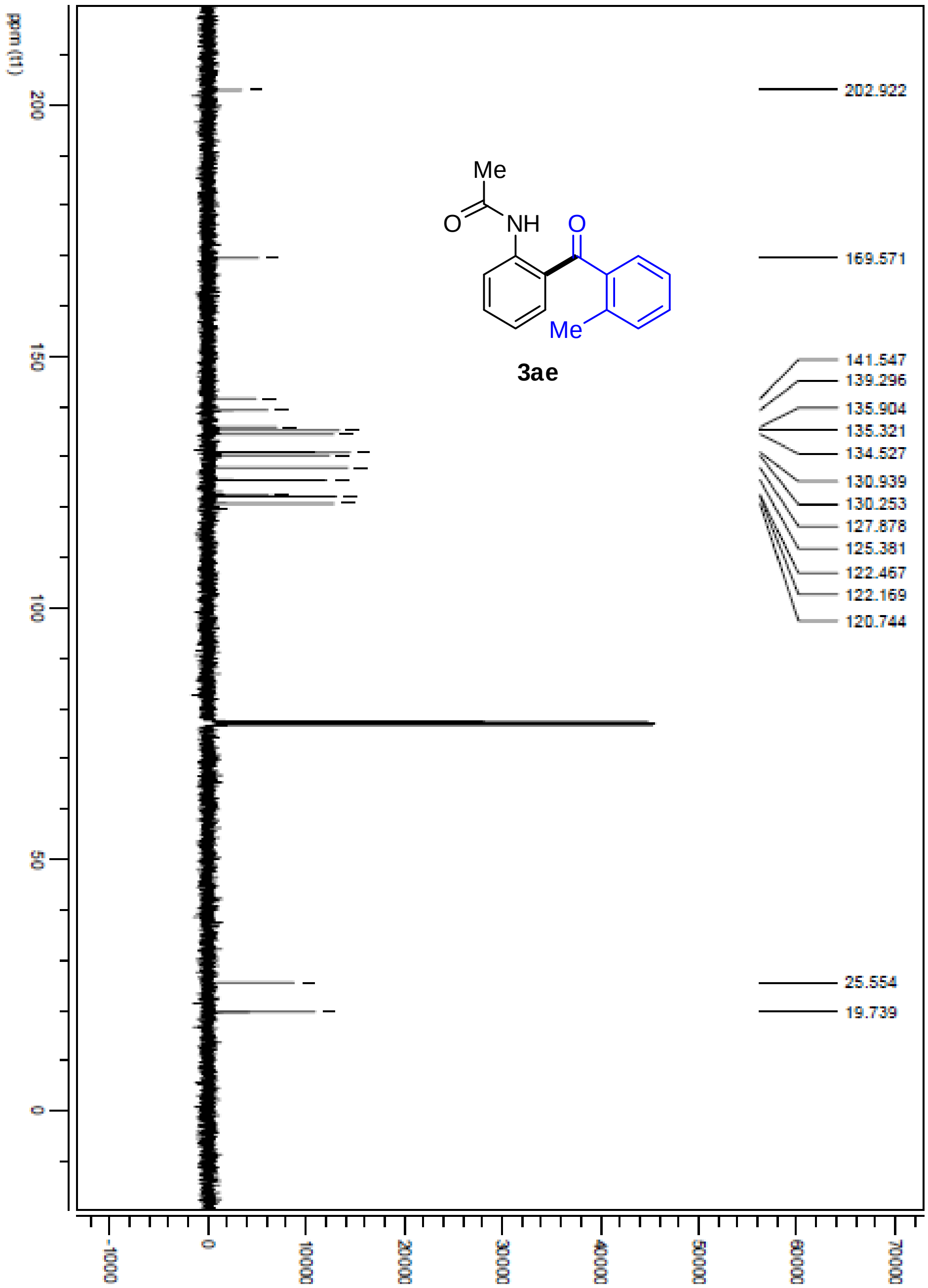
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134.527
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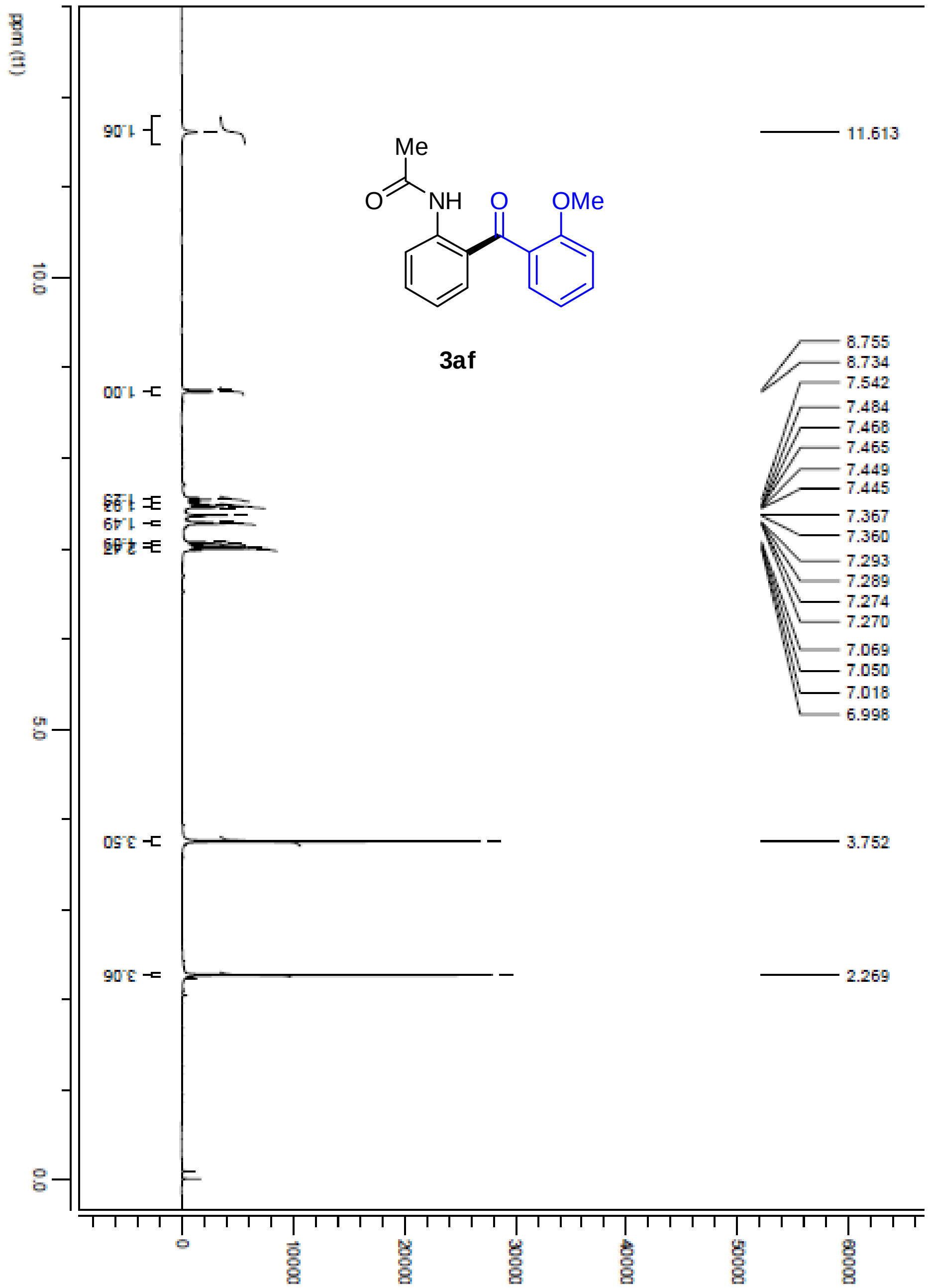
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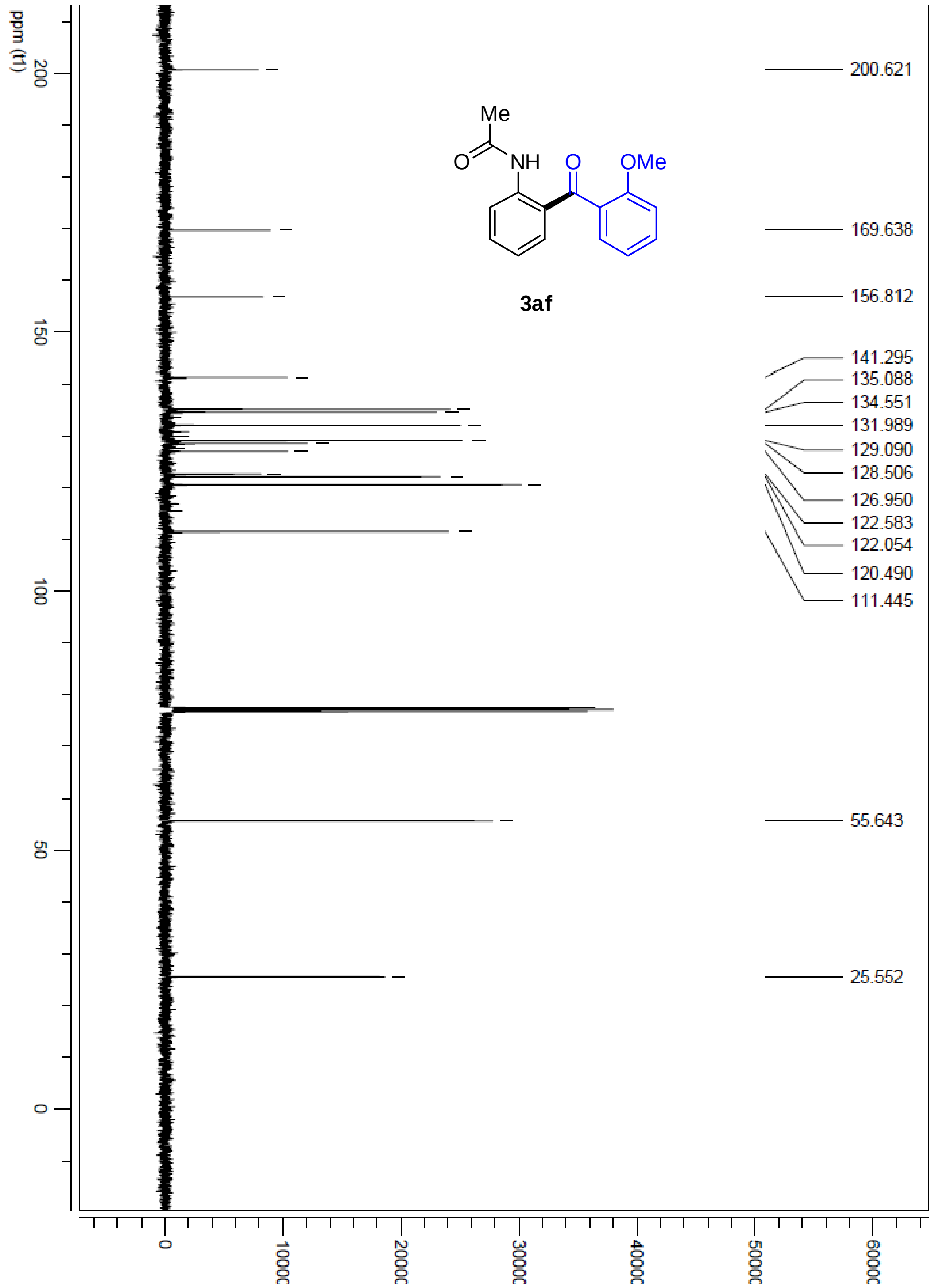
6.998
3.752
2.269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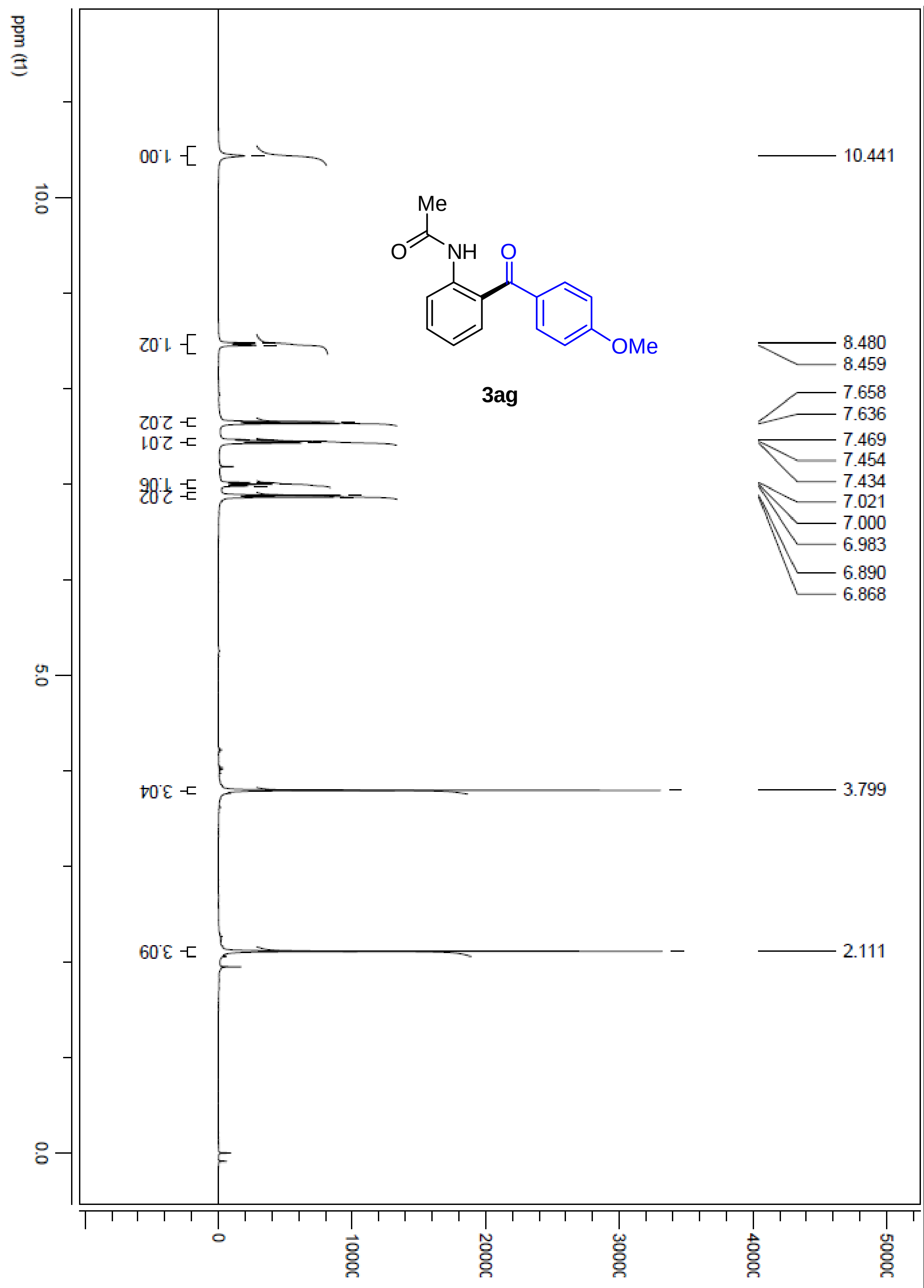
25.552
ppm (t1)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7.434
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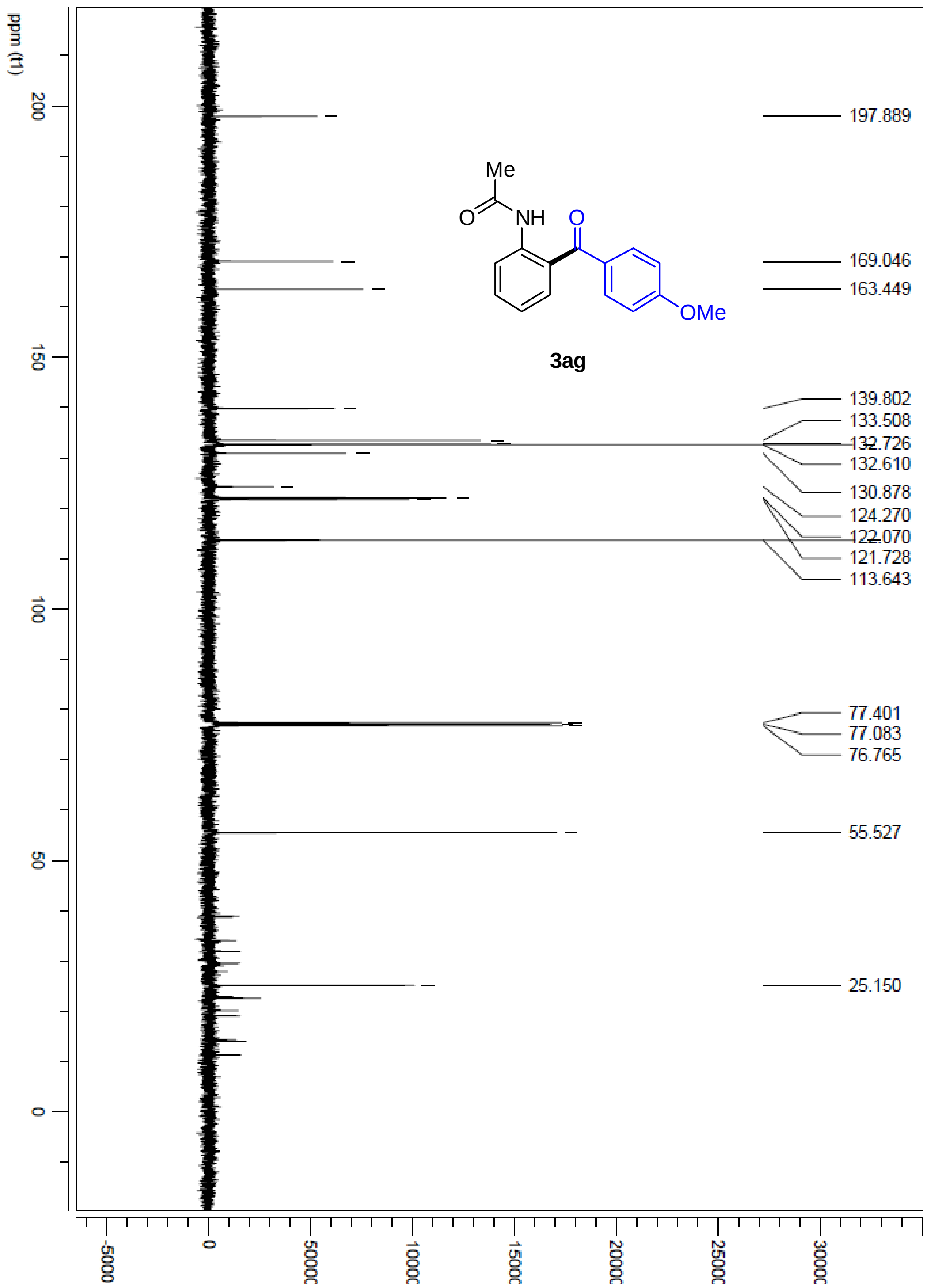
7.000
6.983
6.890
6.868
3.799
2.111

#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130.878
124.270
122.070
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77.401
77.083
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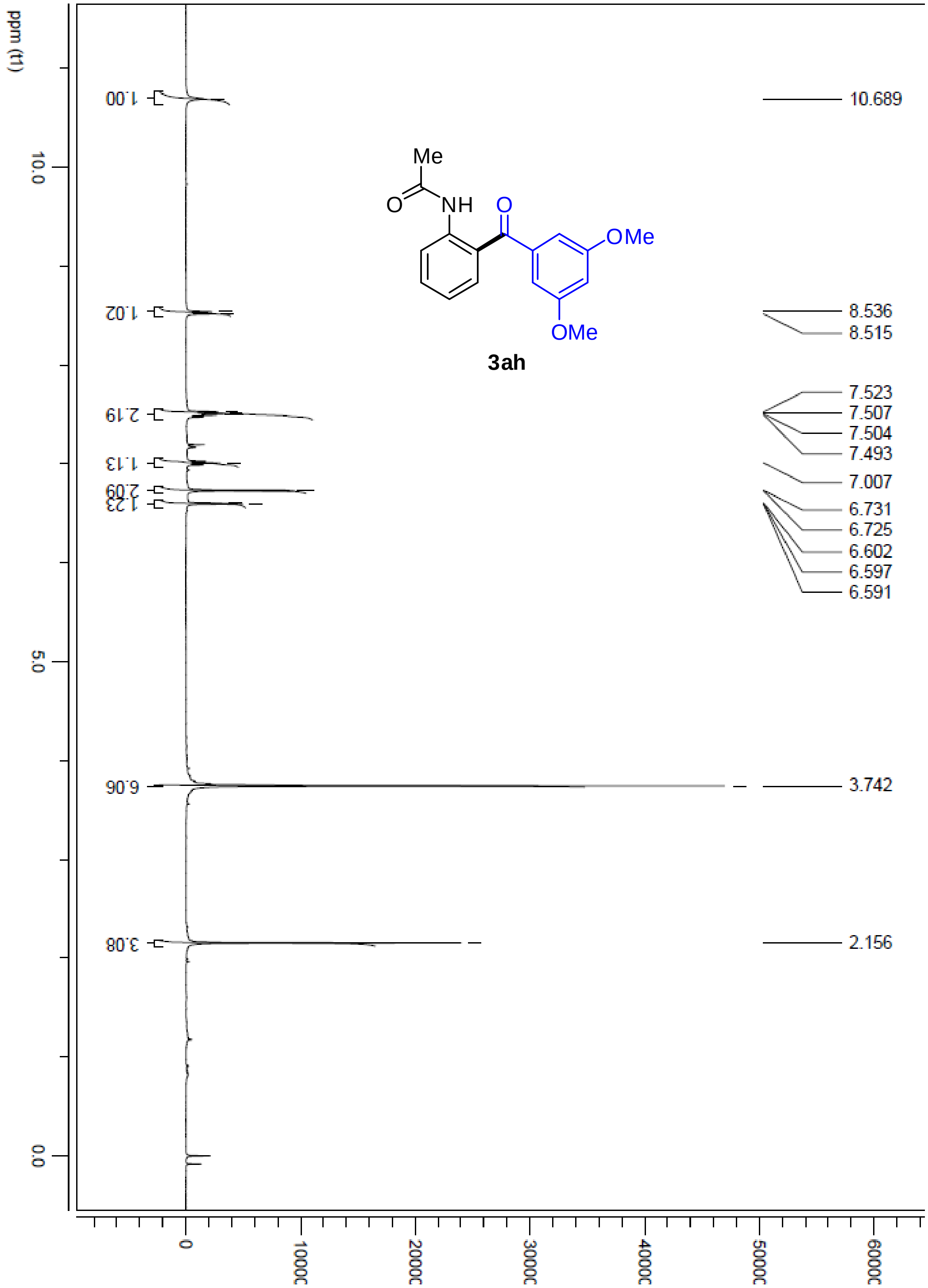
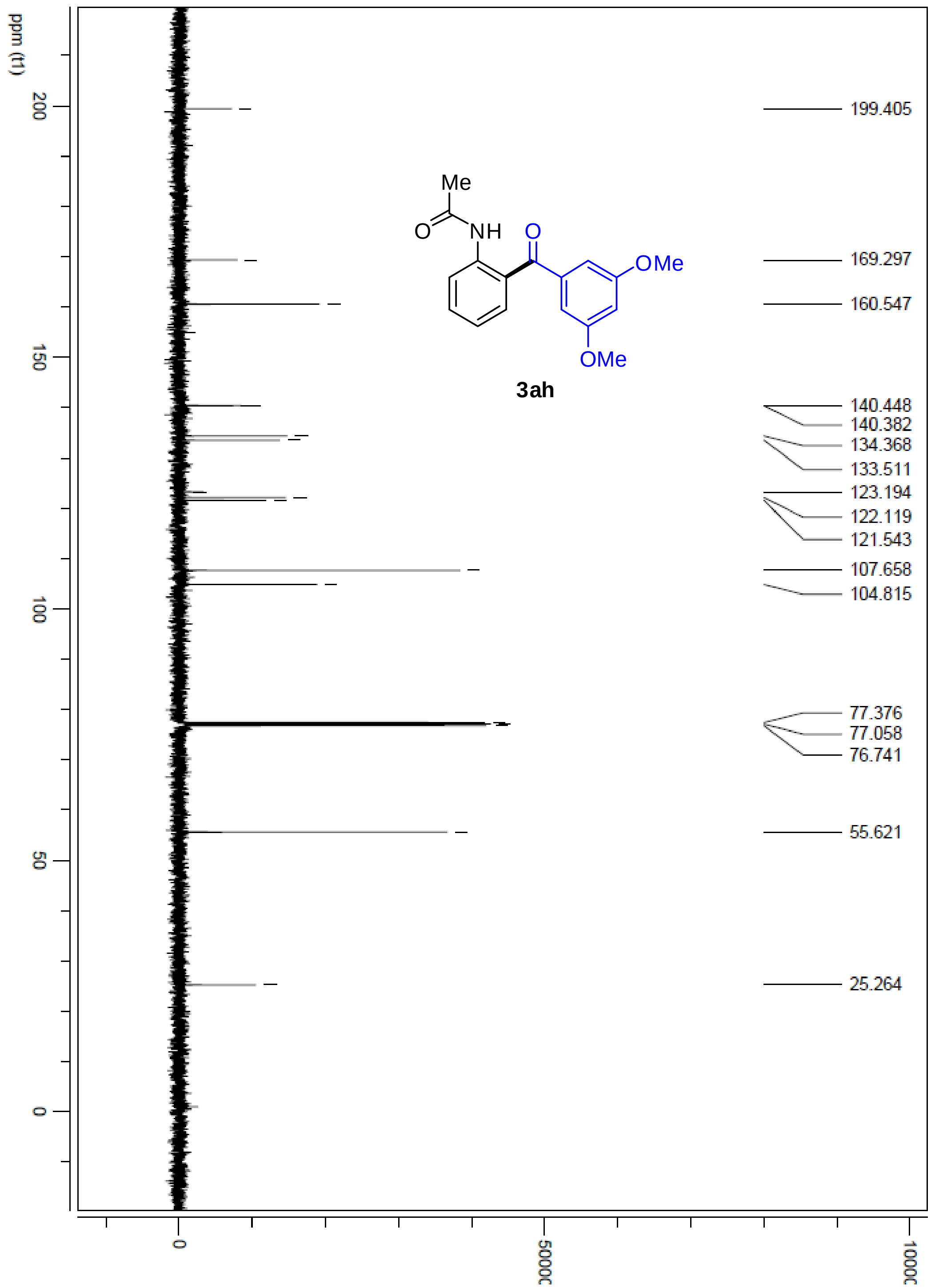
25.150
Me
O
NH
O
OMe
3ag
ppm (t1)

Electronic Supplementary Material (ESI) for Chemical Communications

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121.543
107.658
104.815
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77.058
76.741
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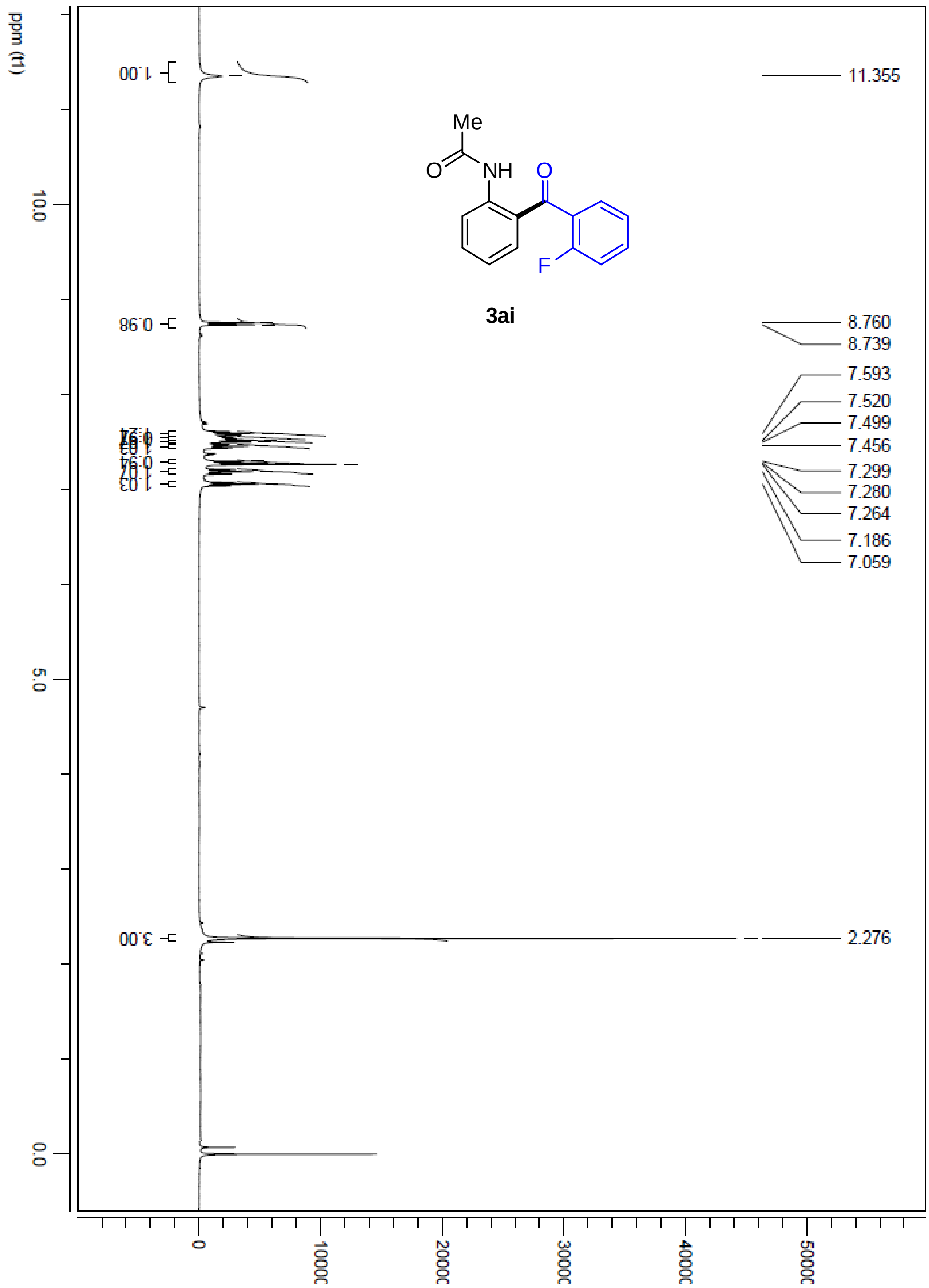
25.264
Me
O
NH
O
OMe
OMe
3ah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7.520
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7.456
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7.264
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7.059
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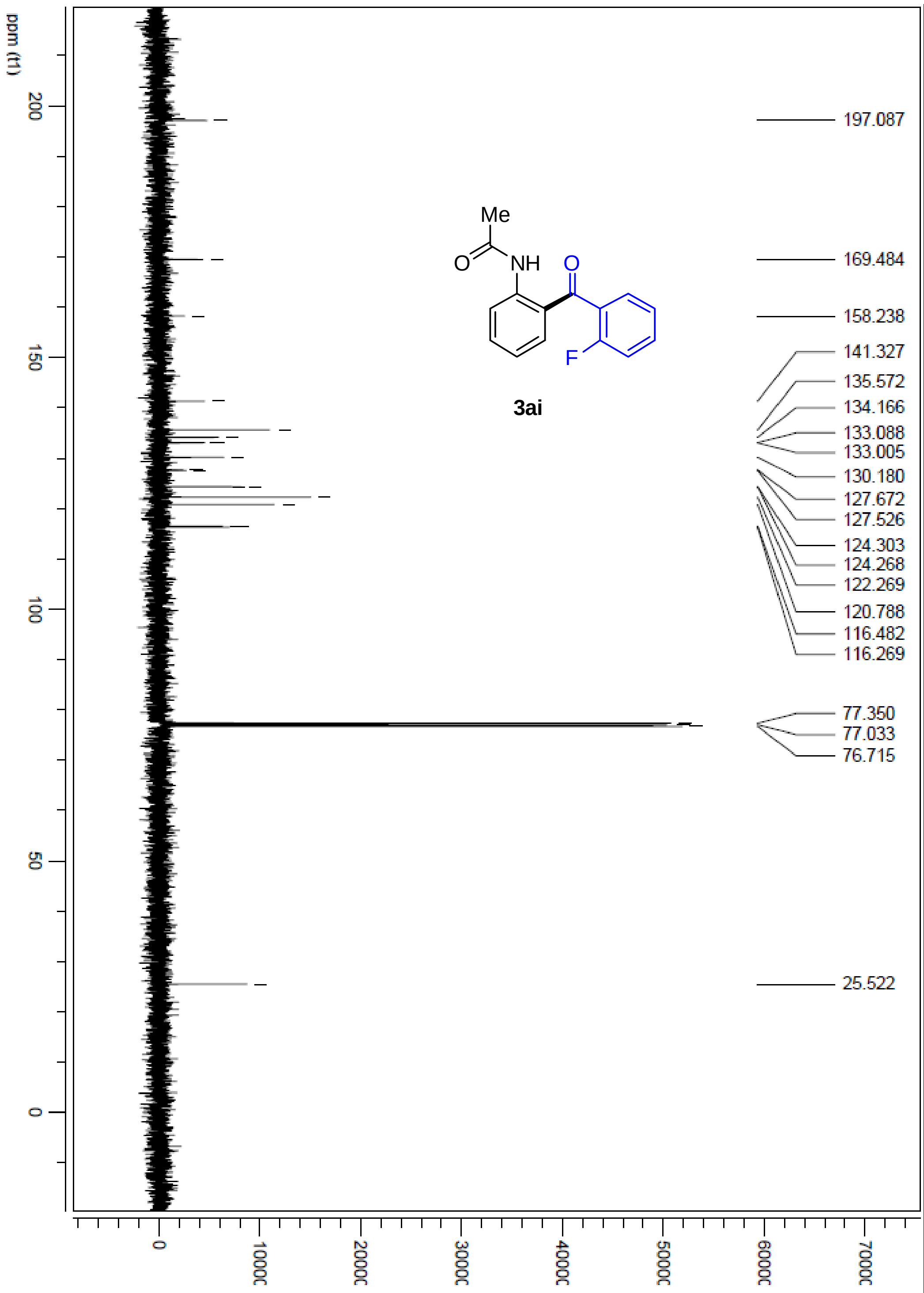
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O
NH
O
F
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134.166
133.088
133.005
130.180
127.672
127.526
124.303
124.268
122.269
120.788
116.482
116.269
77.350
77.033
76.715
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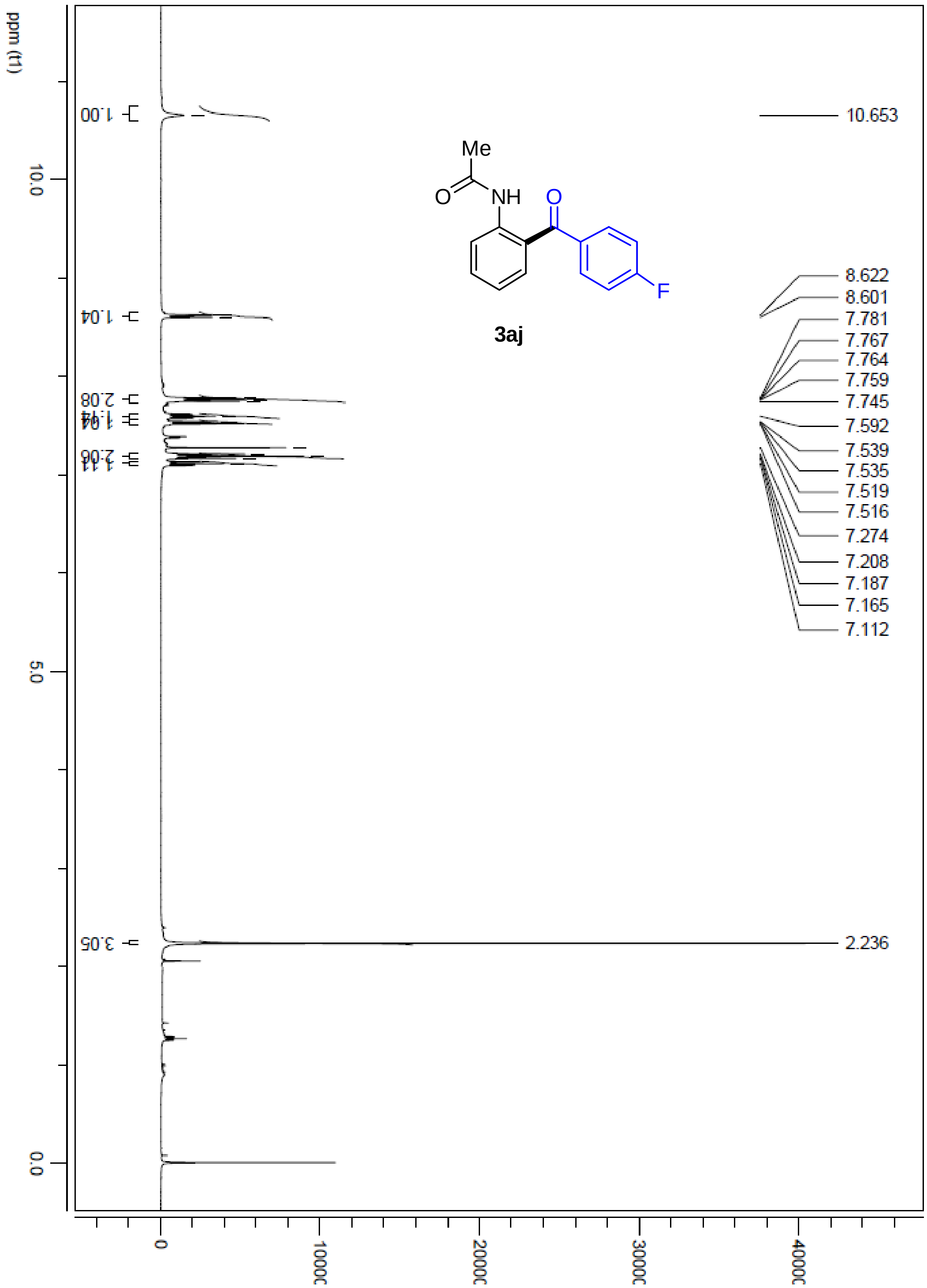
2000C
3000C
4000C
5000C
6000C
7000C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O
F
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7.759
7.745
7.592
7.539
7.535
7.519
7.516
7.274
7.208
7.187
7.165
7.112
2.236
1.00
1.04
2.08
1.14
1.04
2.06
1.11
3.05
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5.0
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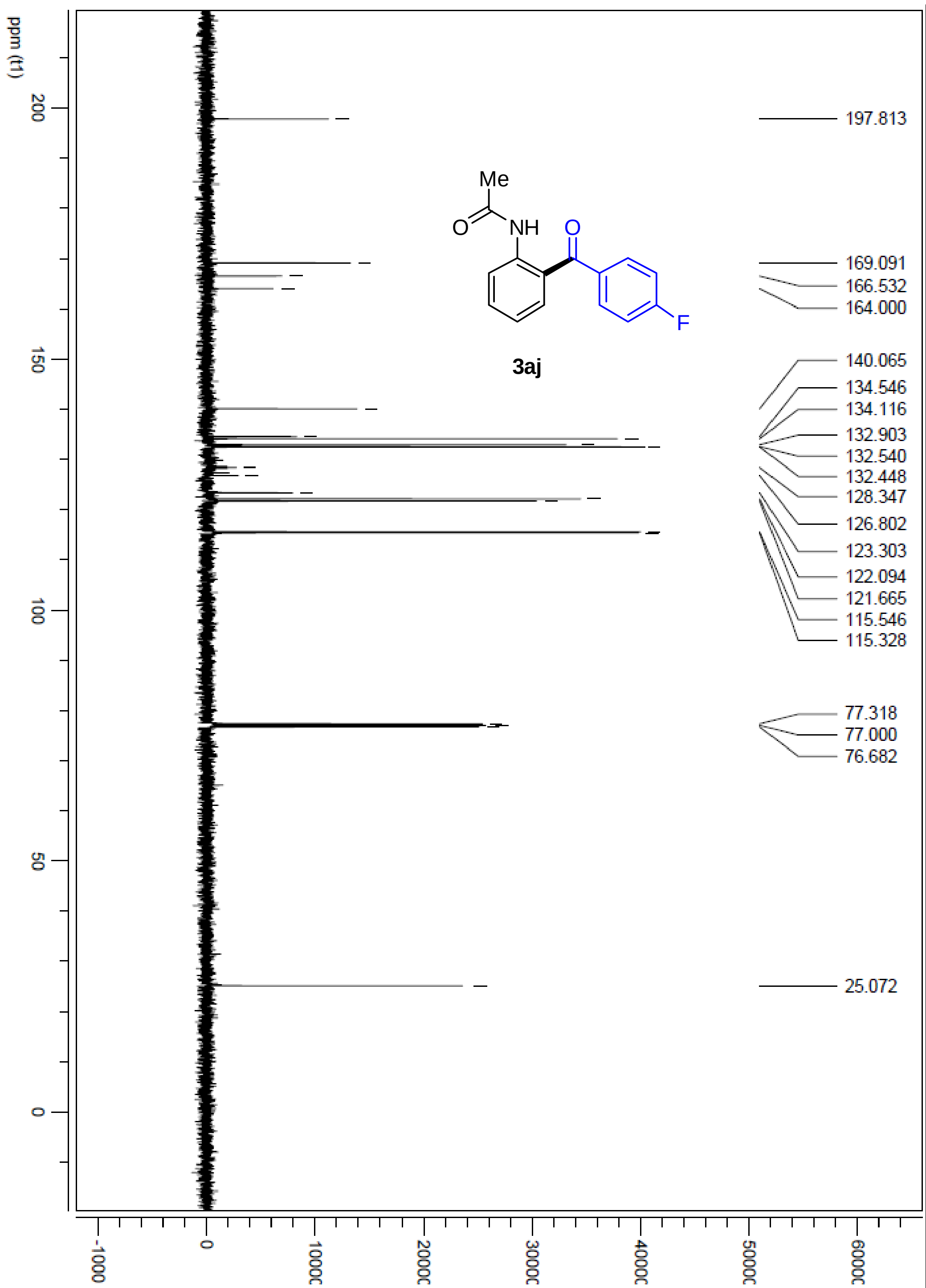
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134.116
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132.540
132.448
128.347
126.802
123.303
122.094
121.665
115.546
115.328
77.318
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5000
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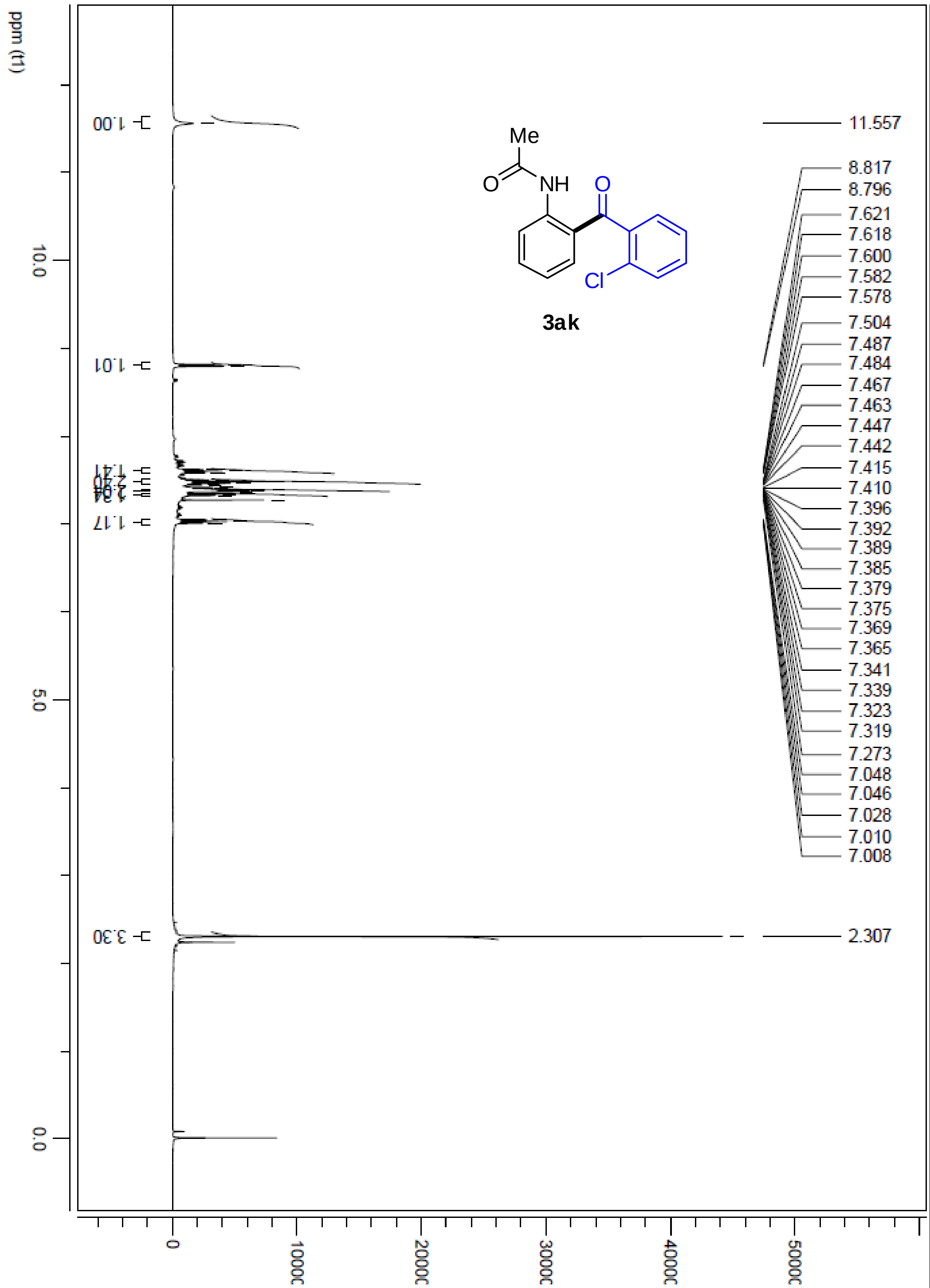
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Cl
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7.582
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7.504
7.487
7.484
7.467
7.463
7.447
7.442
7.415
7.410
7.396
7.392
7.389
7.385
7.379
7.375
7.369
7.365
7.341
7.339
7.323
7.319
7.273
7.048
7.046
7.028
7.010
7.008
2.307
1.00
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3.30
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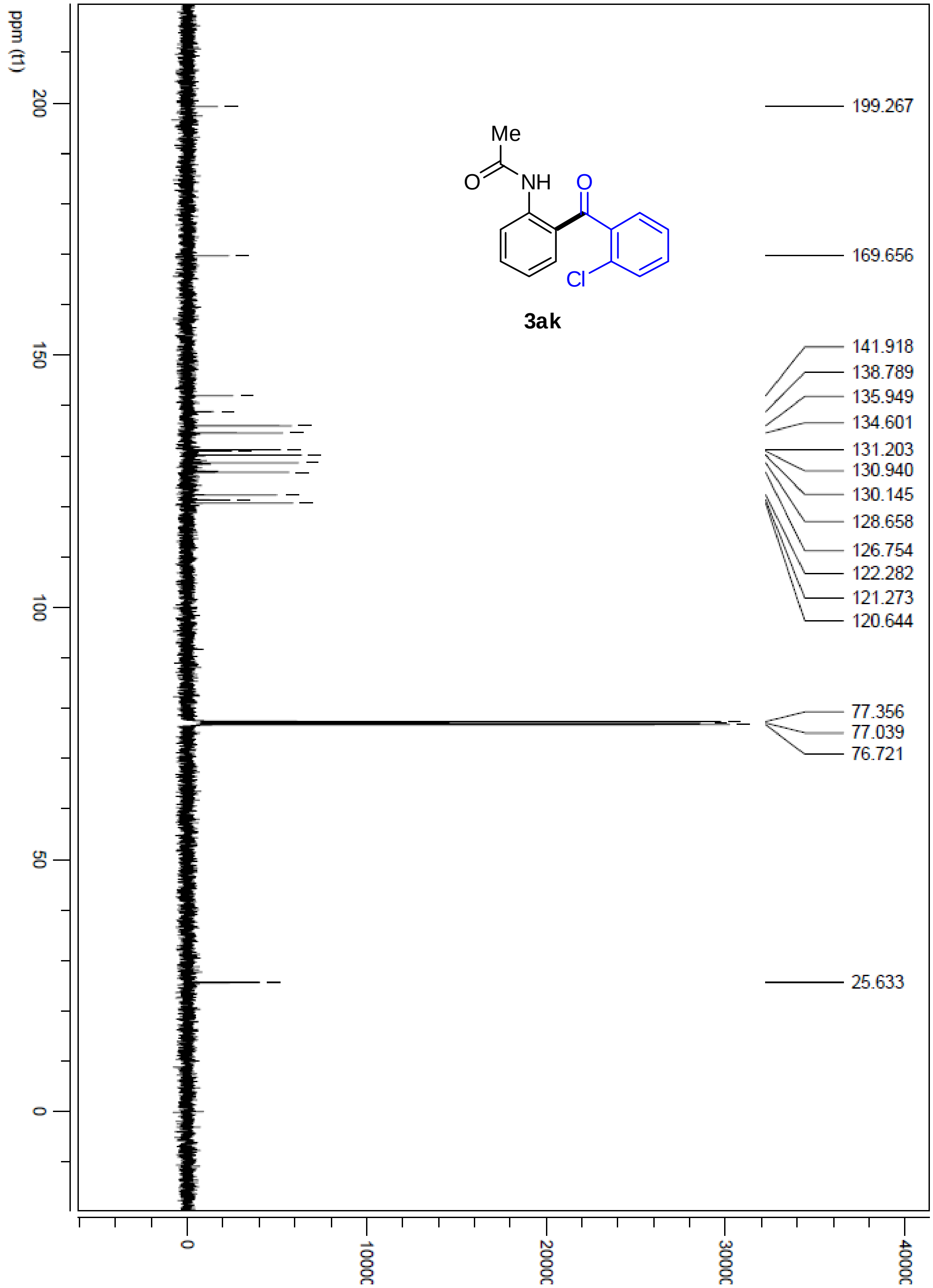
5000C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134.601
131.203
130.940
130.145
128.658
126.754
122.282
121.273
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40000
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O
NH
O
Cl
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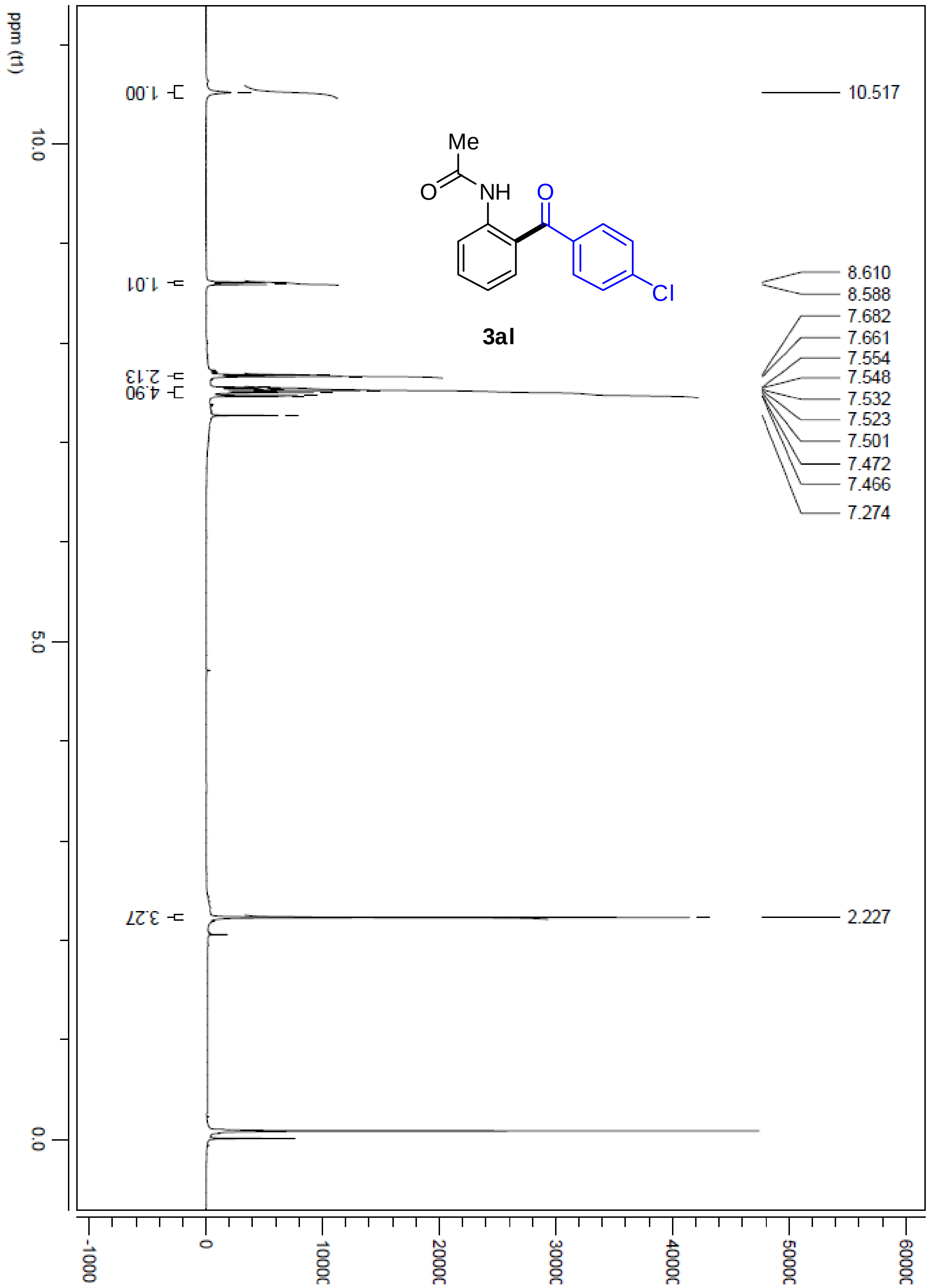
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O
Cl
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7.501
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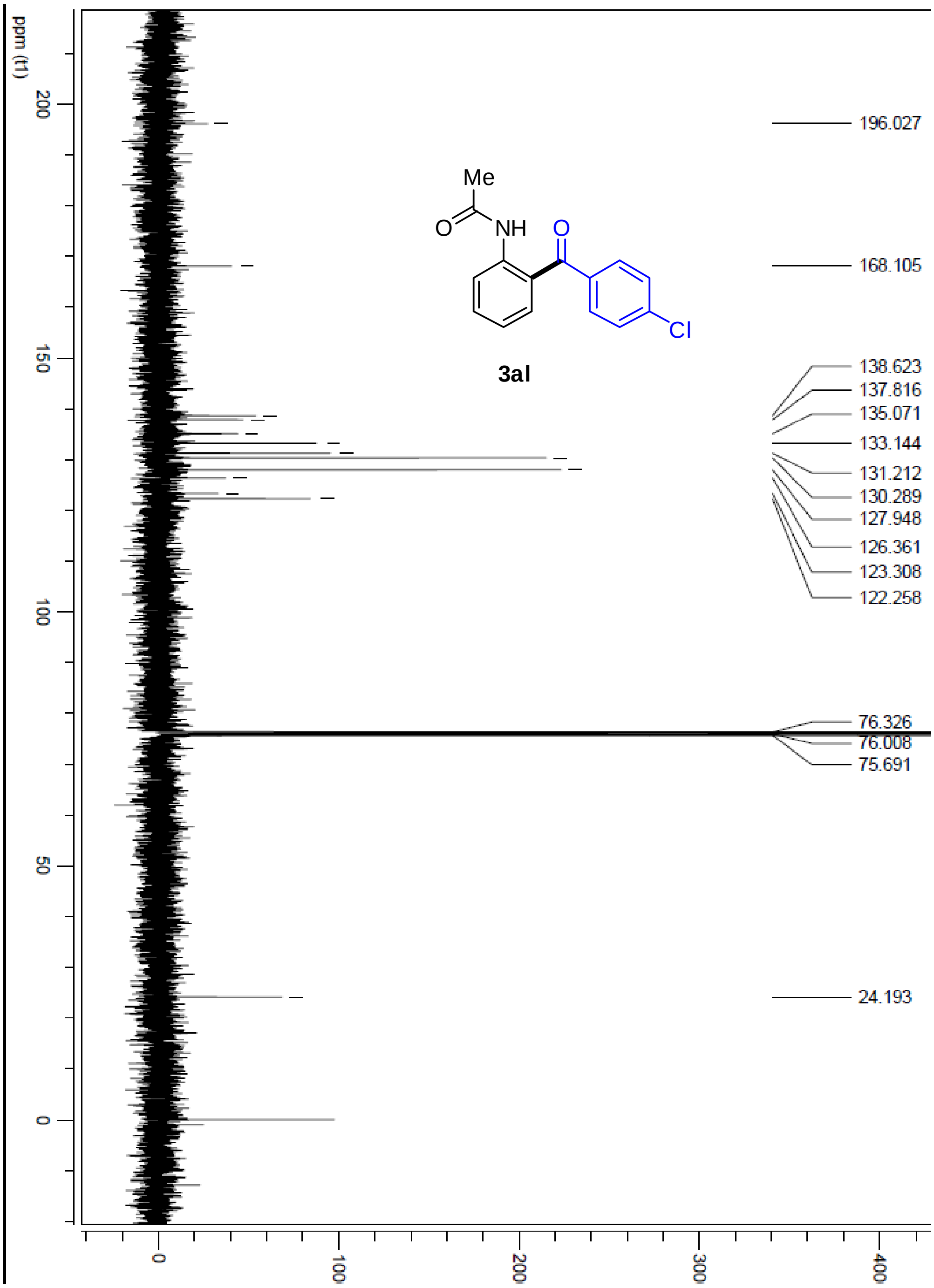
7.466
7.274
2.227


ppm (t1)
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100
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0
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200
300
400
Me
O
NH
O
Cl
3al
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168.105
138.623
137.816
135.071
133.144
131.212
130.289
127.948
126.361
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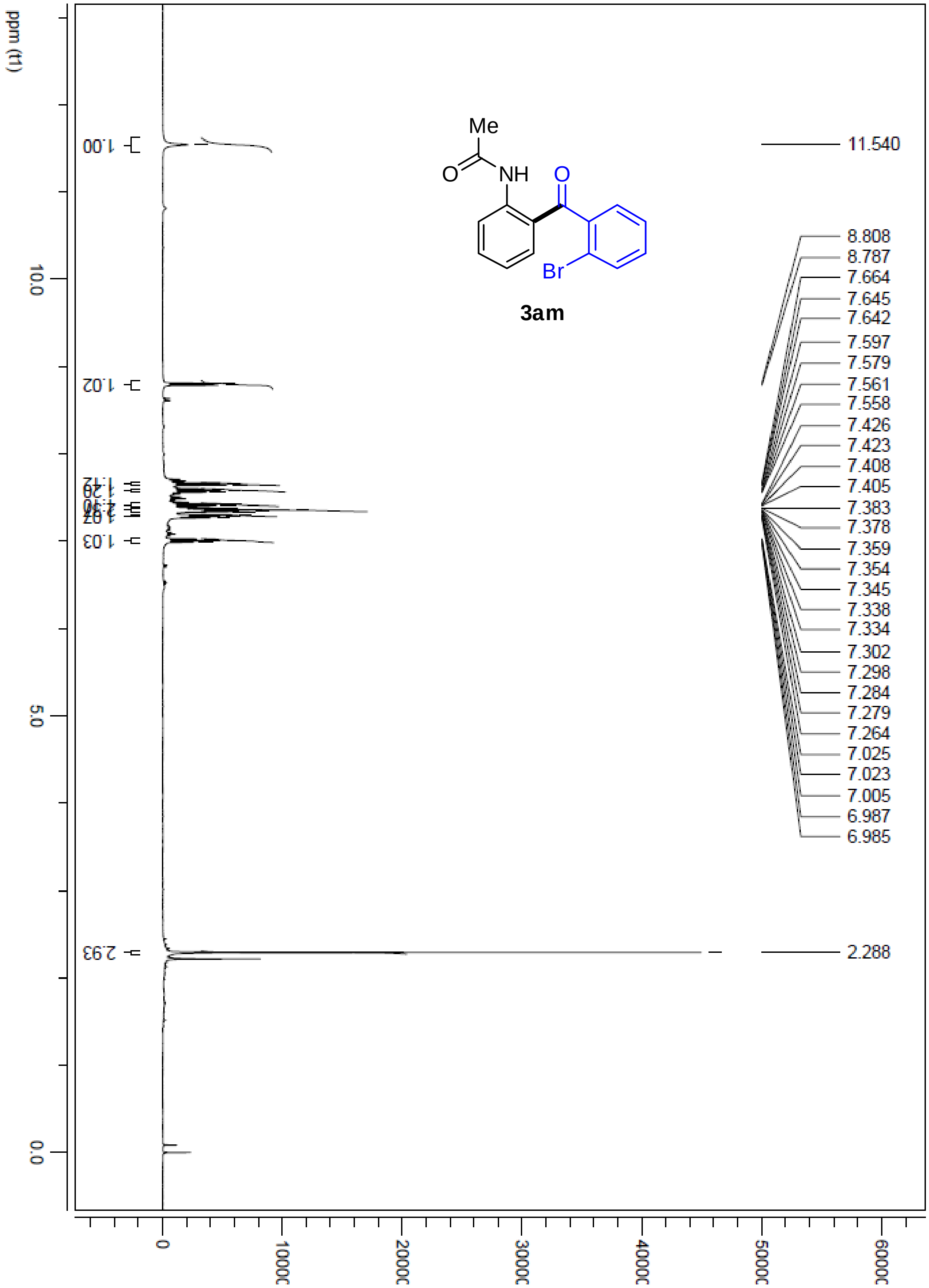
122.258
76.326
76.008
75.691
24.193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NH
O
Br
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7.642
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7.561
7.558
7.426
7.423
7.408
7.405
7.383
7.378
7.359
7.354
7.345
7.338
7.334
7.302
7.298
7.284
7.279
7.264
7.025
7.023
7.005
6.987
6.985
2.288
1.00
1.02
1.12
1.20
2.10
1.07
1.03
2.93
ppm (t1)
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10000
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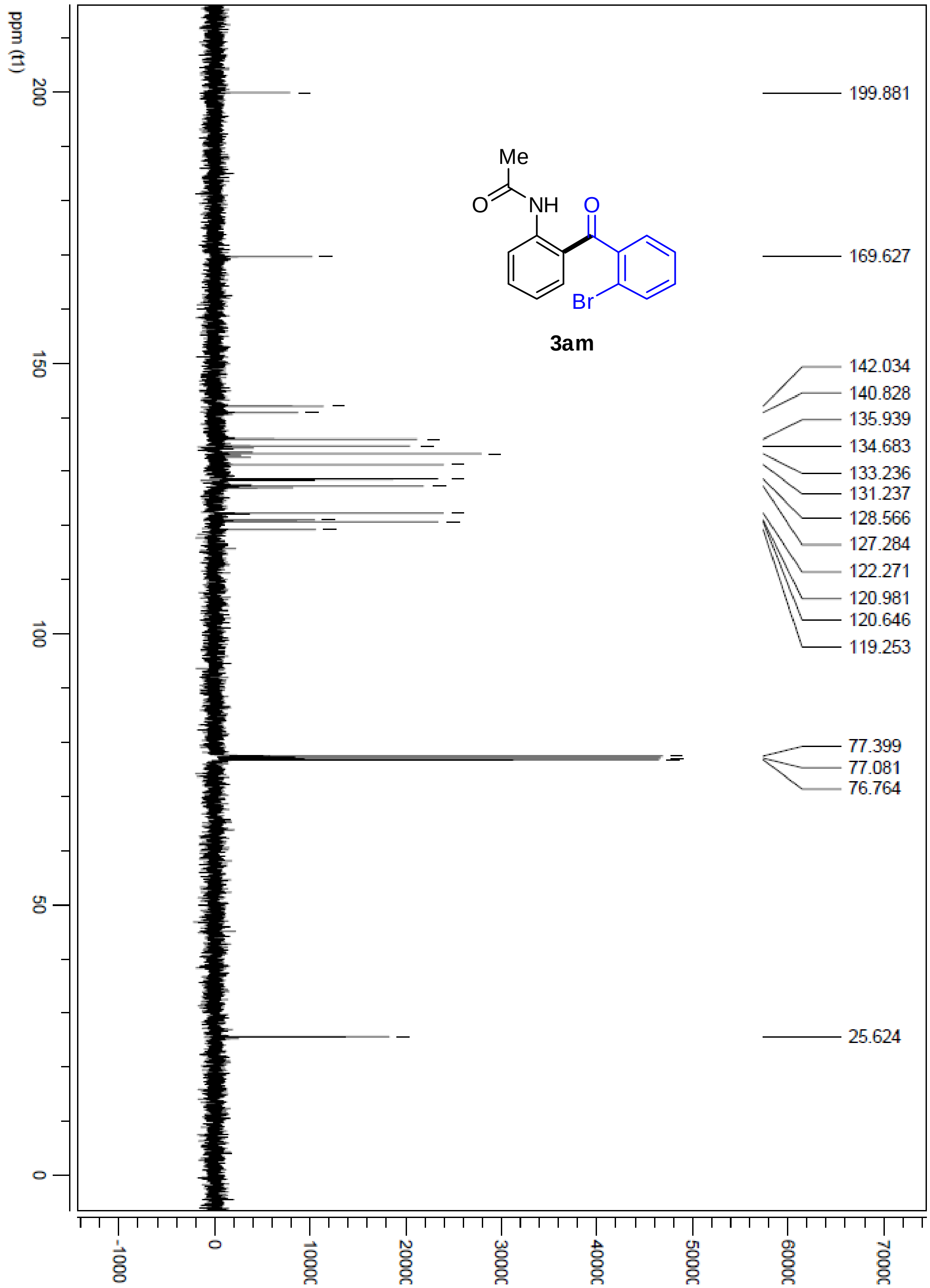
30000
40000
50000
60000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NH
O
Br
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140.828
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134.683
133.236
131.237
128.566
127.284
122.271
120.981
120.646
119.253
77.399
77.081
76.764
25.624
ppm (t1)
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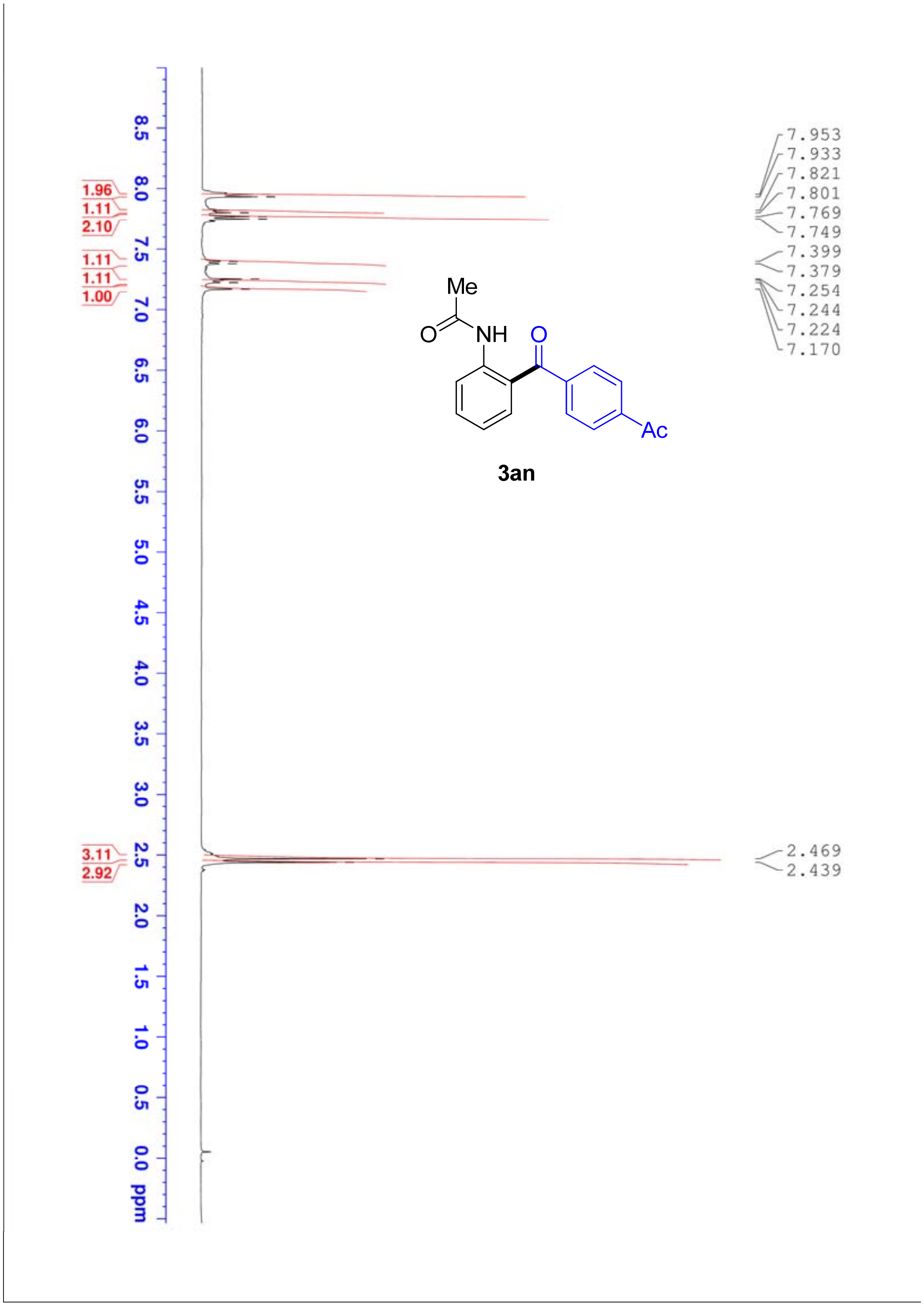
150
100
50
0
-1000
0
1000C
2000C
3000C
4000C
5000C
6000C
7000C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7.821
7.801
7.769
7.749
7.399
7.379
7.254
7.244
7.224
7.170
2.469
2.439
1.96
1.11
2.10
1.11
1.11
1.00
3.11
2.92
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
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O
NH
O
Ac
3an

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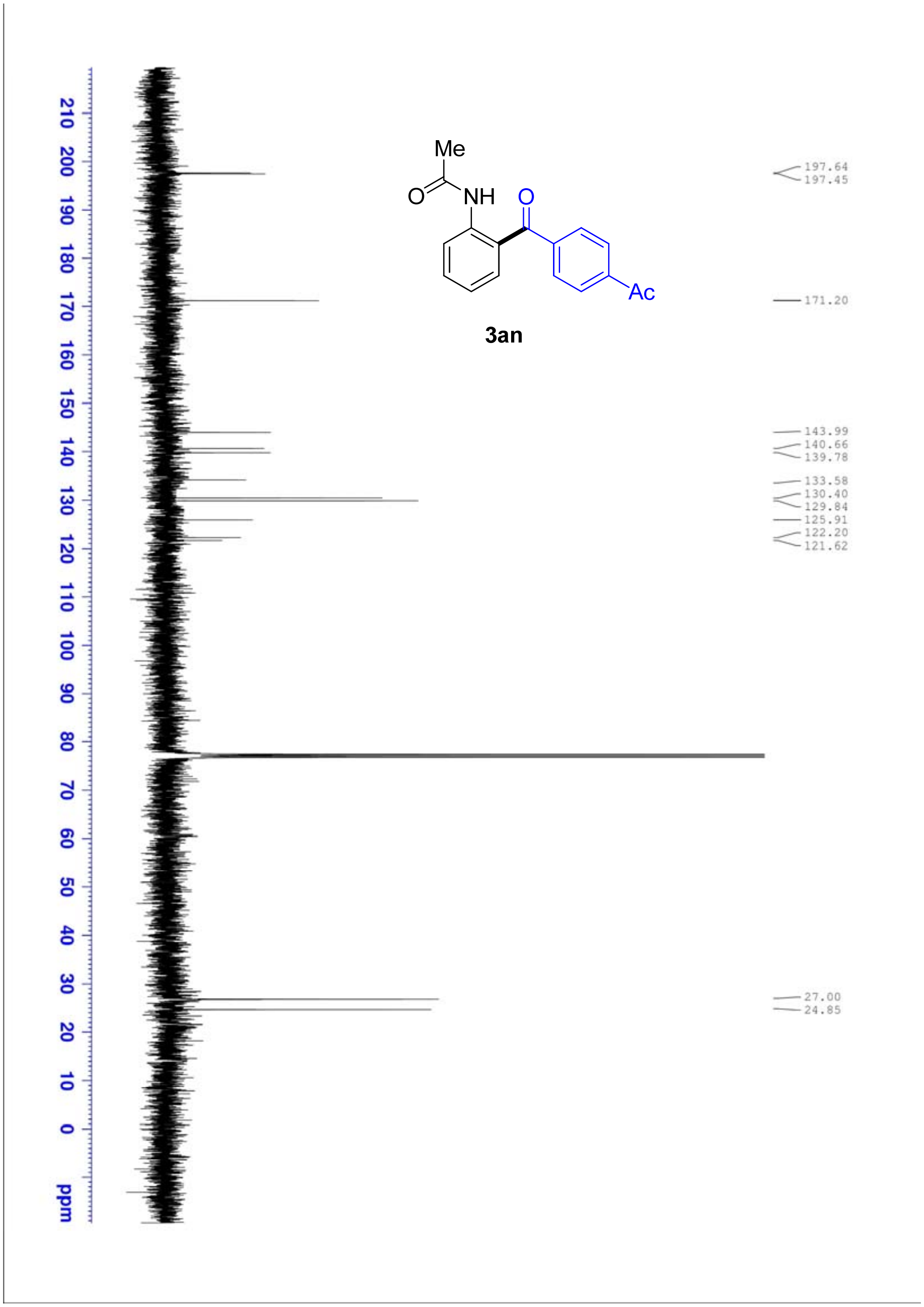

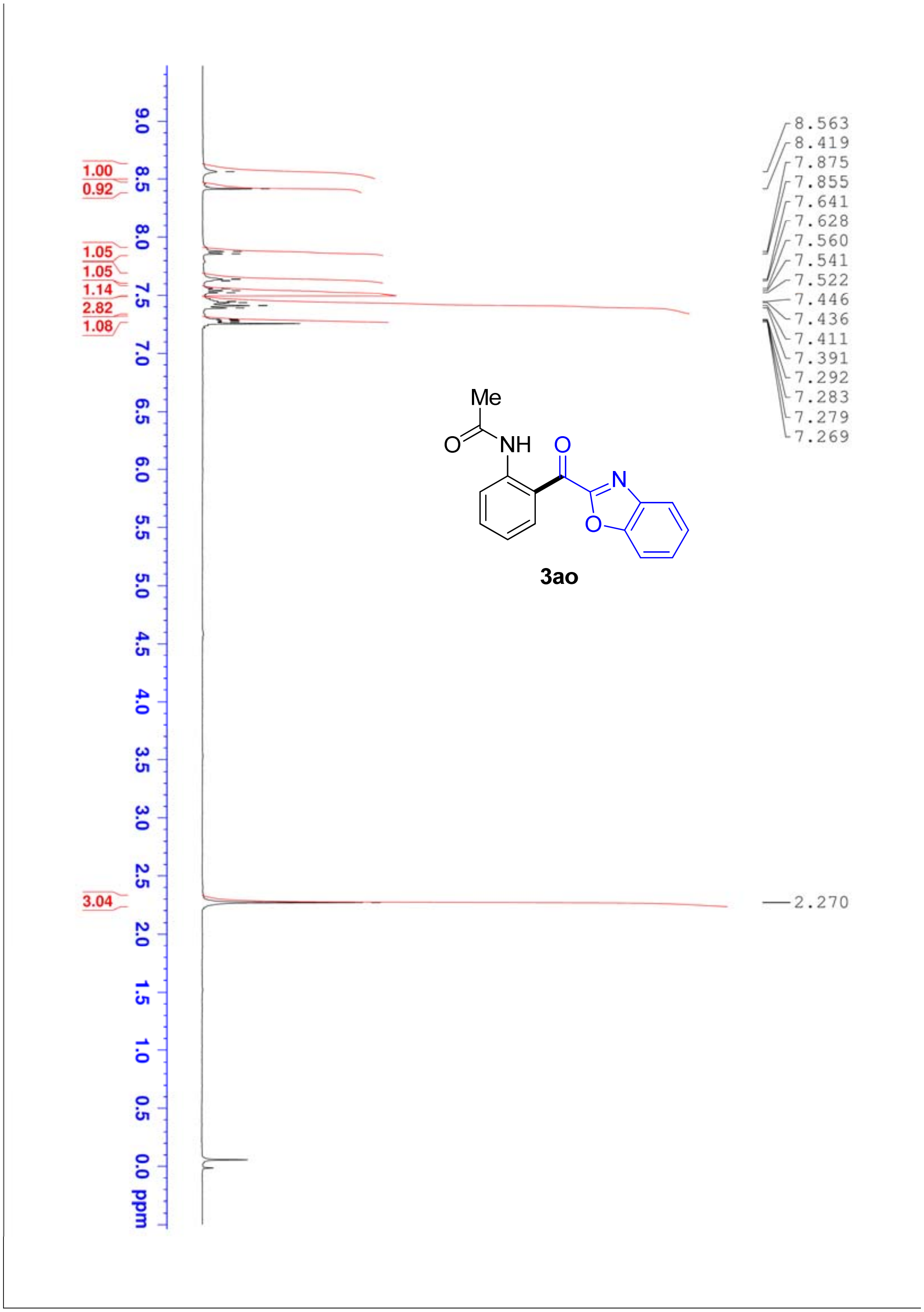
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8.419
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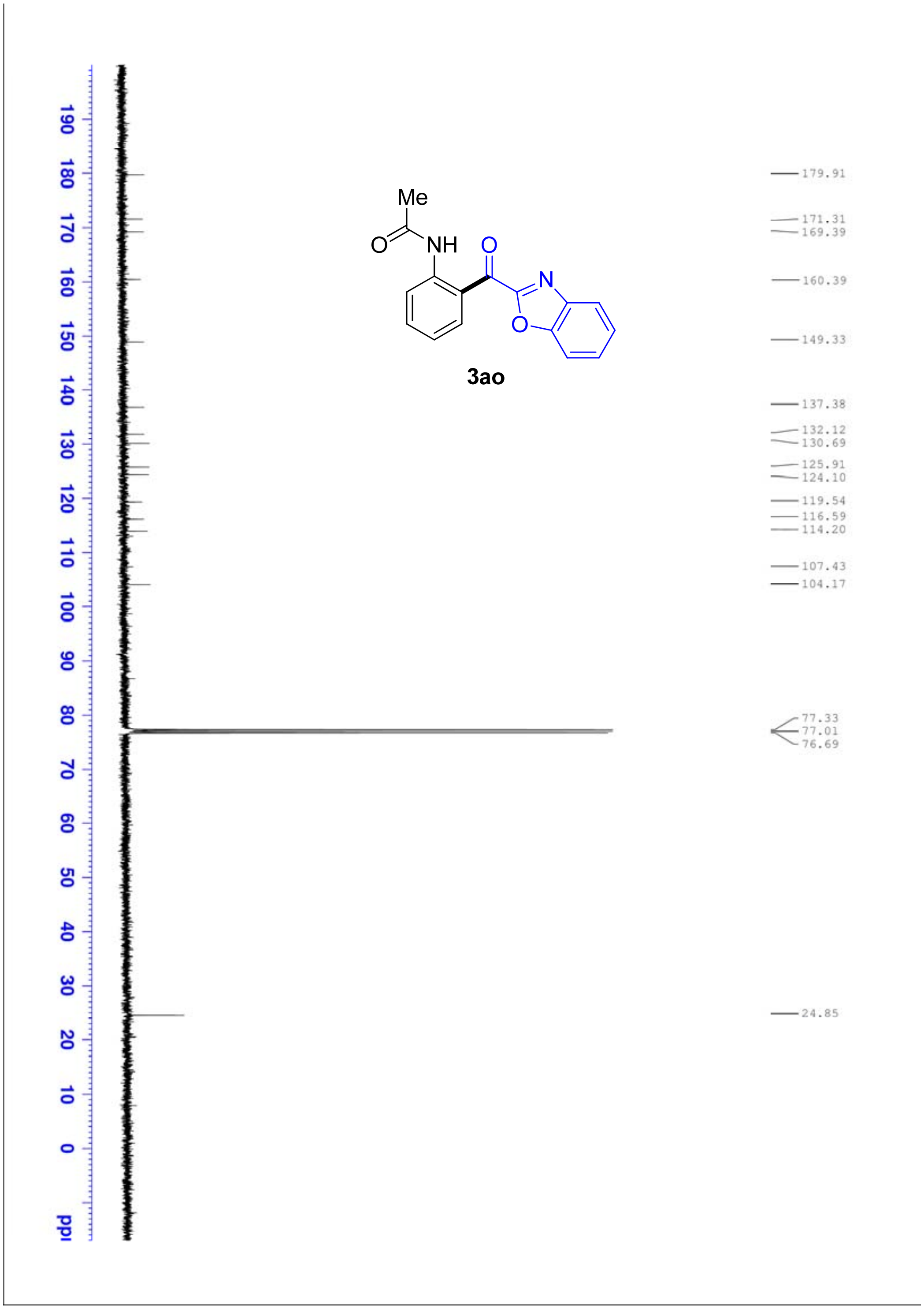
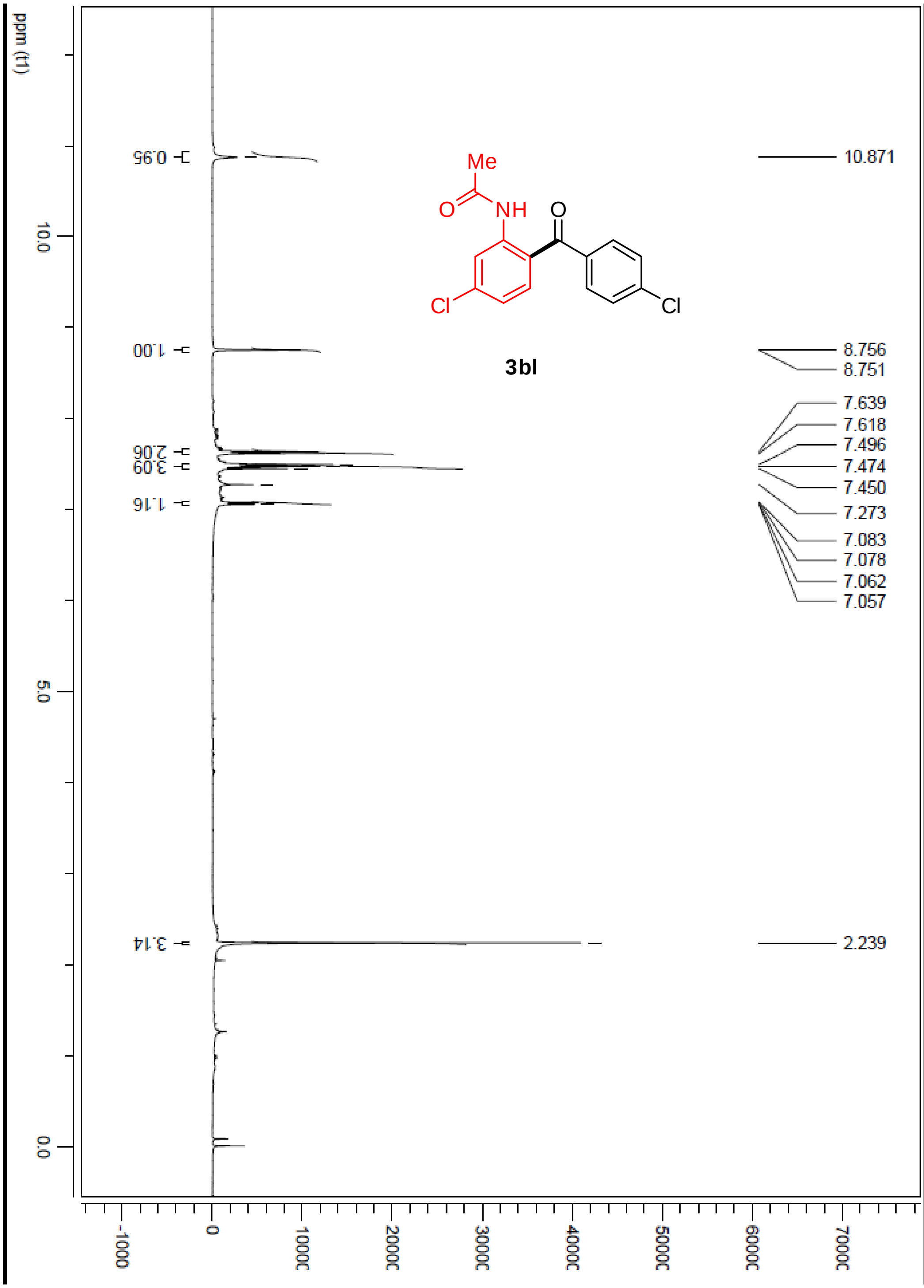
7.641
7.628
7.560
7.541
7.522
7.446
7.436
7.411
7.391
7.292
7.283
7.279
7.269
2.270
1.00
0.92
1.05
1.05
1.14
2.82
1.08
3.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm
Me
O
NH
O
N
O
3ao


Me
NH
O
O
N
O
3ao
179.91
171.31
169.39
160.39
149.33
137.38
132.12
130.69
125.91
124.10
119.54
116.59
114.20
107.43
104.17
77.33
77.01
76.69
24.85
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


3bl
Me
O
NH
O
Cl
Cl
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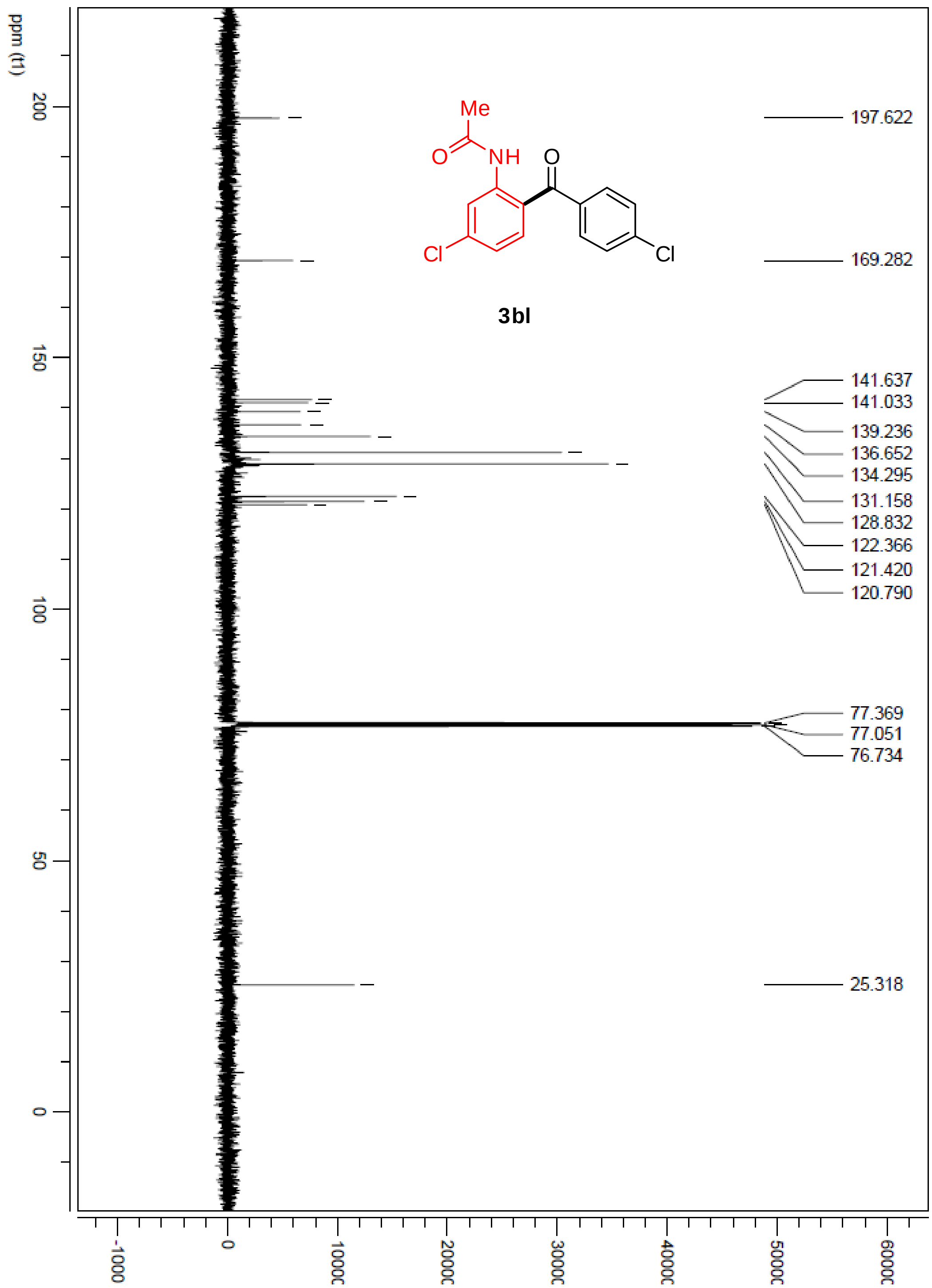
7.496
7.474
7.450
7.273
7.083
7.078
7.062
7.057
2.239
0.95
1.00
2.06
3.09
1.16
3.14
ppm (t1)
10.0
5.0
0.0
-1000
0
1000
2000
3000
4000
5000
6000
7000


ppm (t1)
200
150
100
50
0
Me
O
NH
O
Cl
Cl
3bl
197.622
169.282
141.637
141.033
139.236
136.652
134.295
131.158
128.832
122.366
121.420
120.790
77.369
77.051
76.734
25.318
-1000
0
10000
20000
30000
40000
50000
60000

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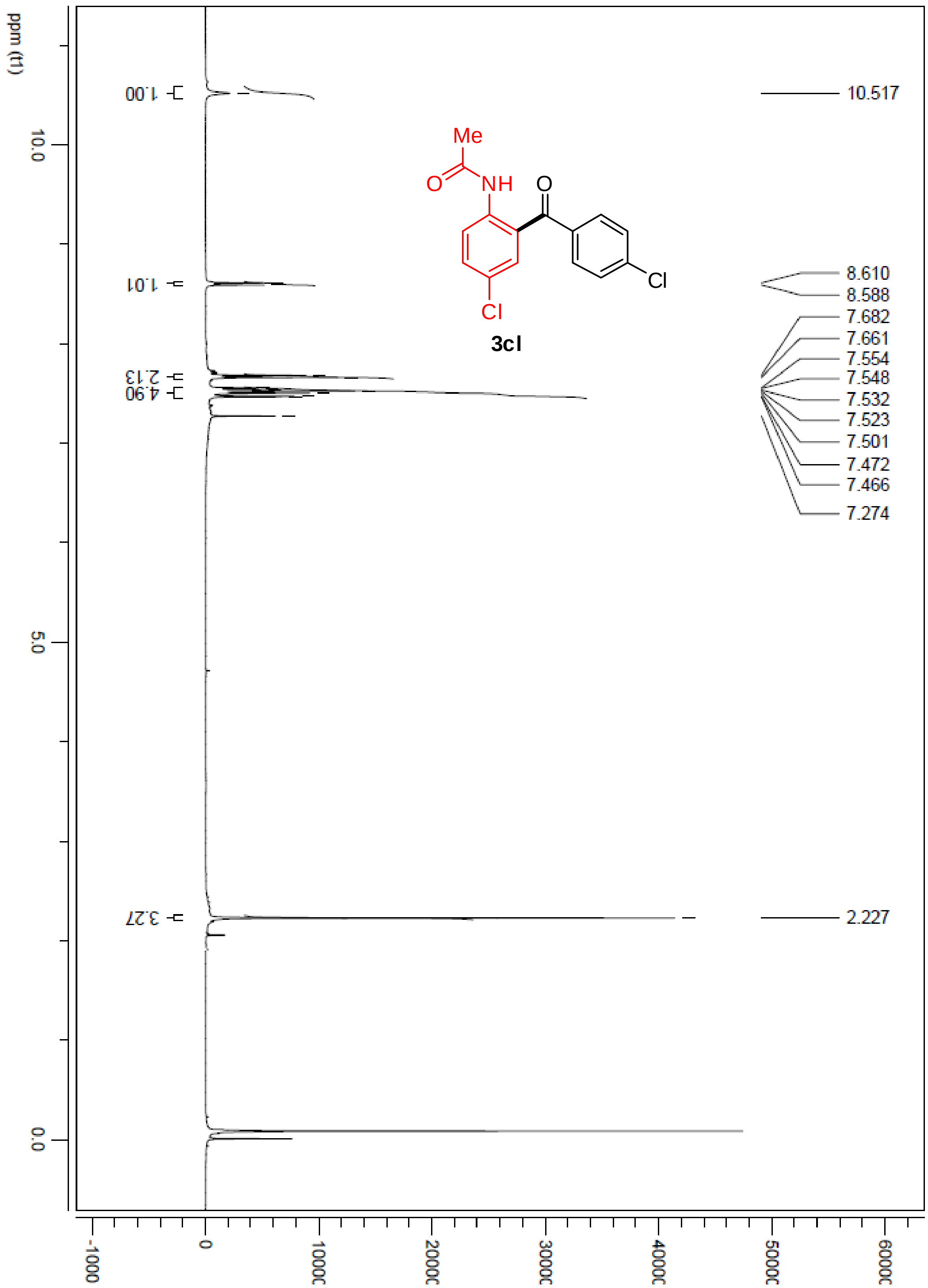

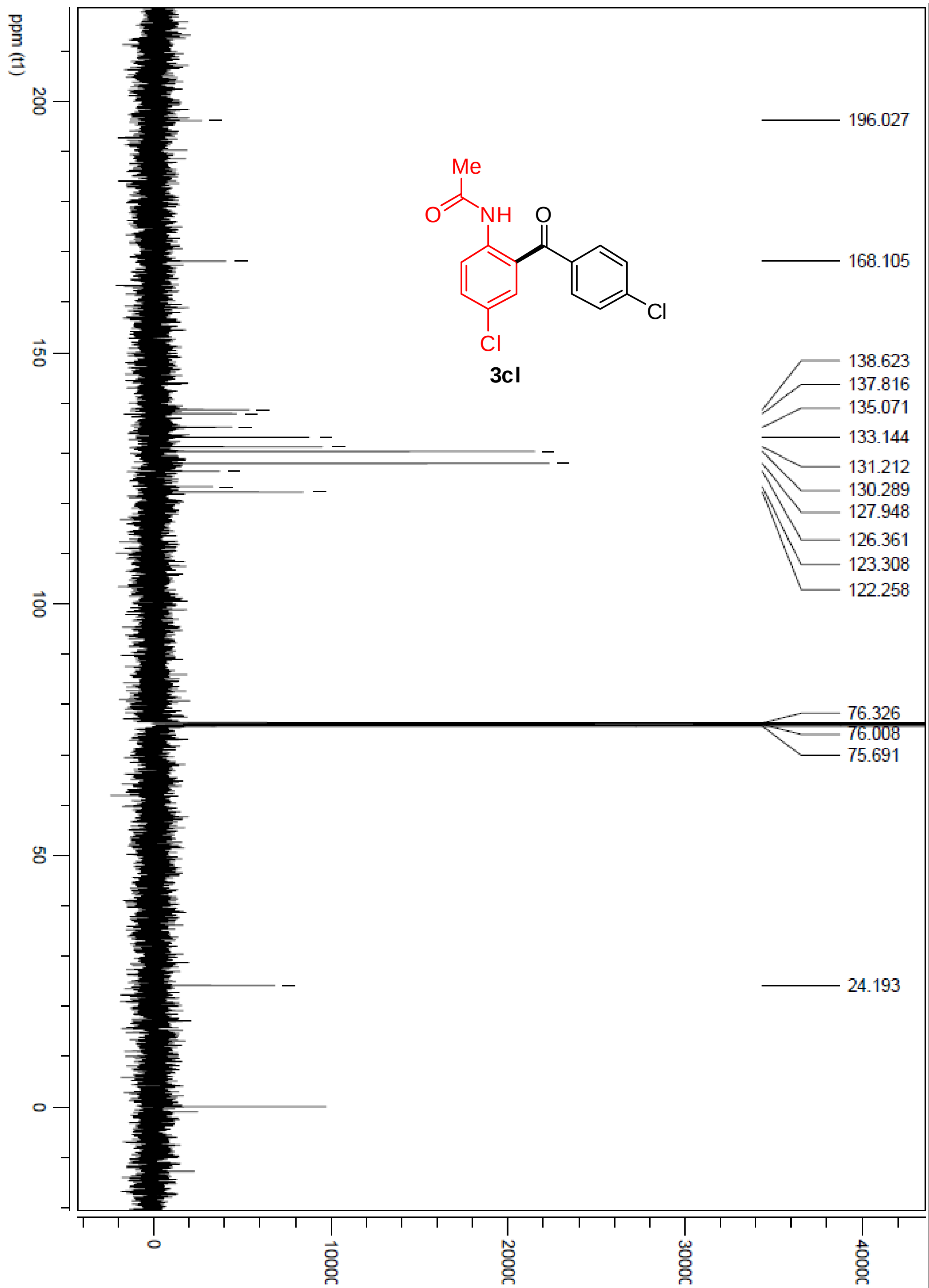
Me
O
NH
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Cl
Cl
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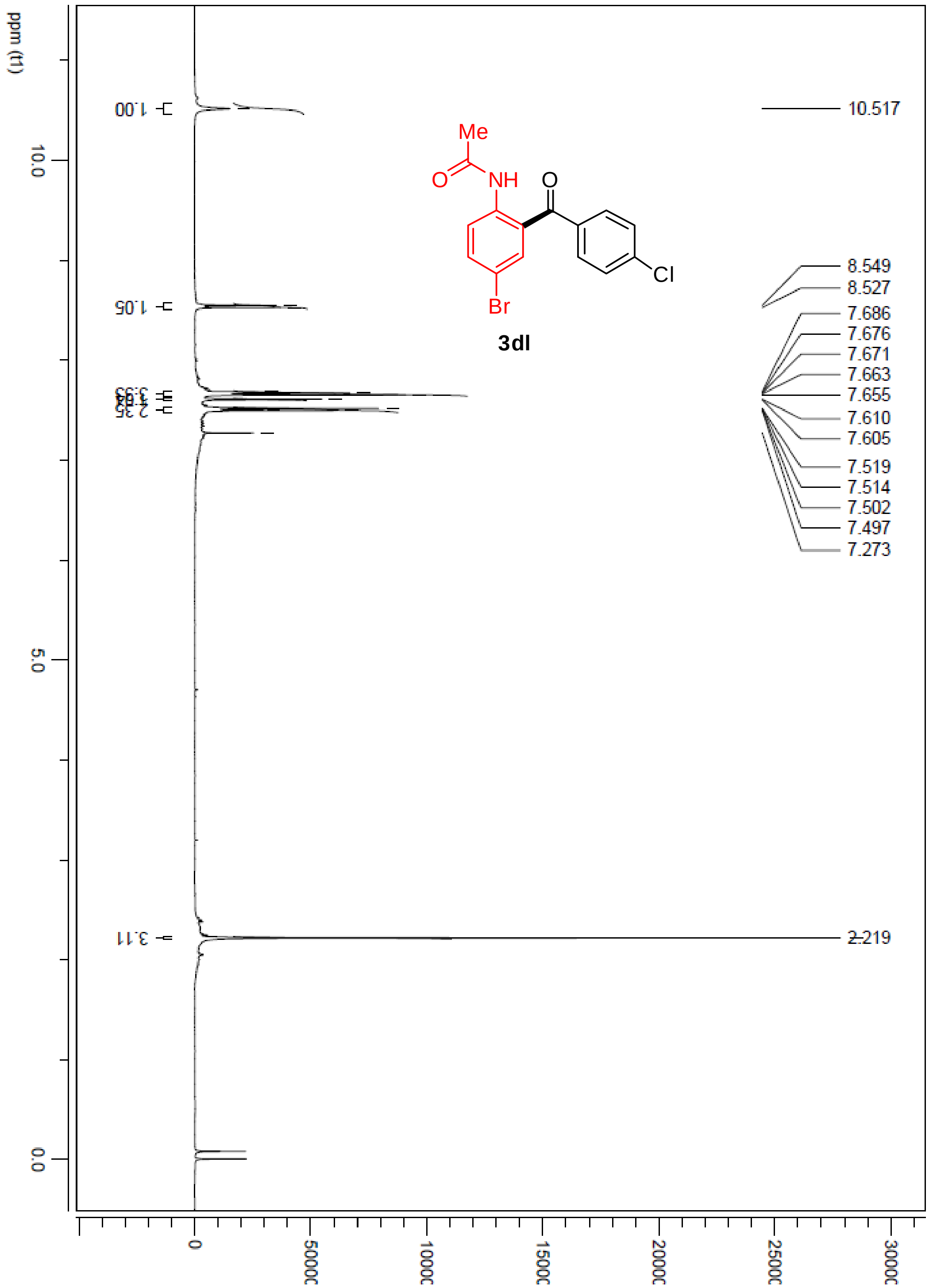
138.623
137.816
135.071
133.144
131.212
130.289
127.948
126.361
123.308
122.258
76.326
76.008
75.691
24.193
ppm (t1)
200
150
100
50
0
10000
20000
30000
40000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8.549
8.527
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7.676
7.671
7.663
7.655
7.610
7.605
7.519
7.514
7.502
7.497
7.273
2.219
Me
O
NH
O
Br
Cl
3dl
ppm (t1)
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1.00
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0
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10000
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20000
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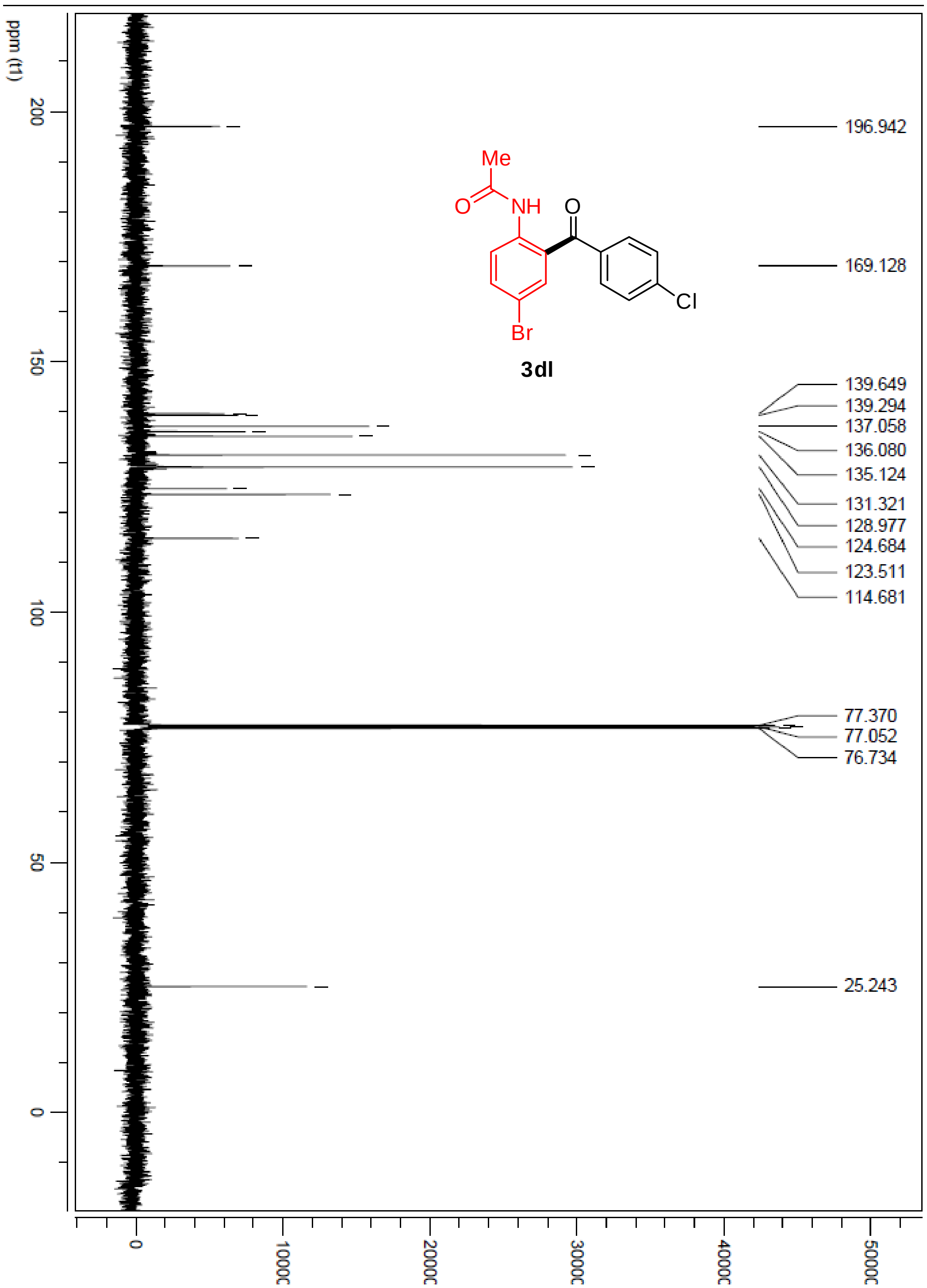
30000


ppm (t1)
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136.080
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131.321
128.977
124.684
123.511
114.681
77.370
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Me
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3dl
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20000
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40000
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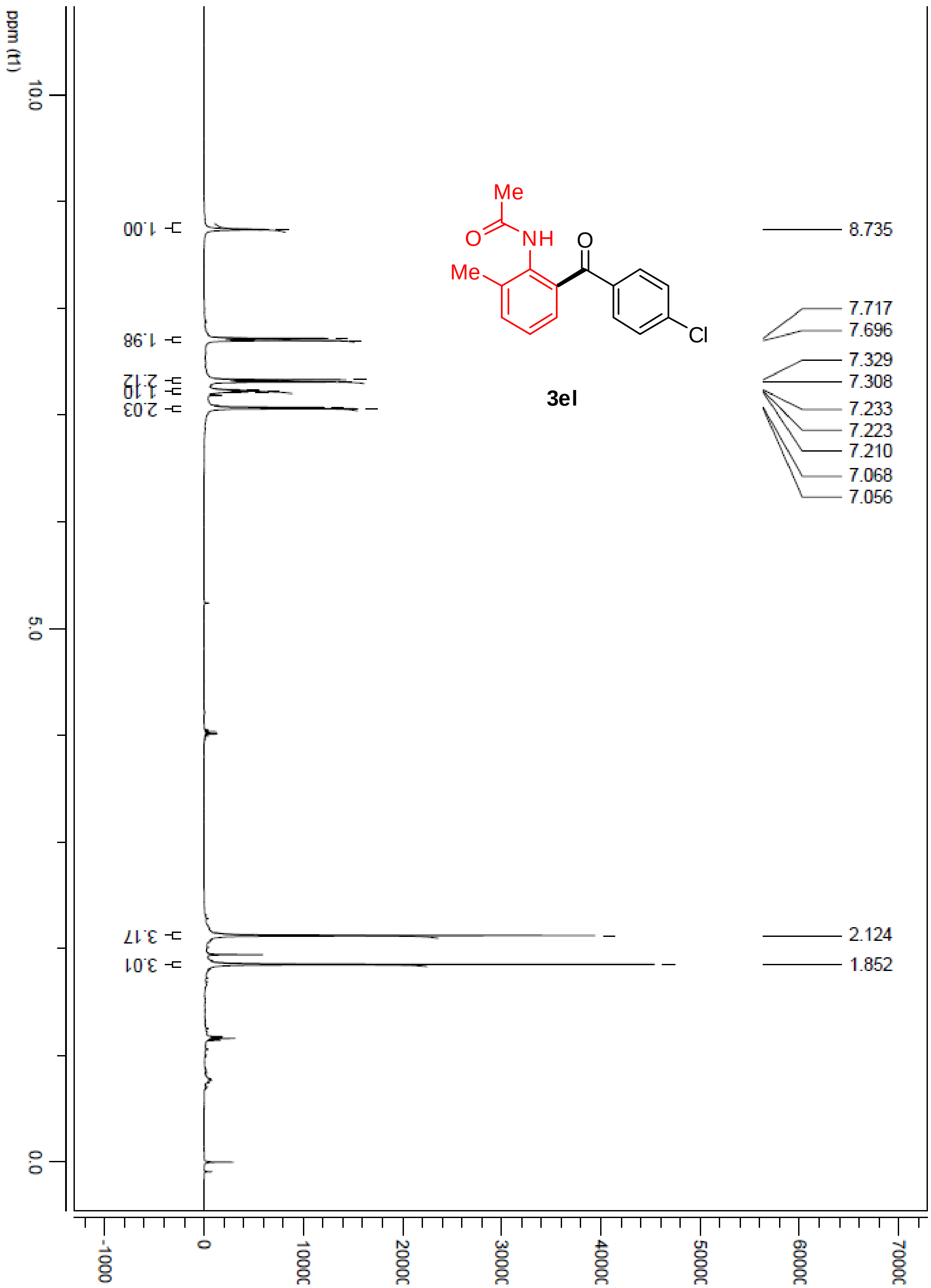
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NH
O
Me
Cl
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7.233
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7.068
7.056
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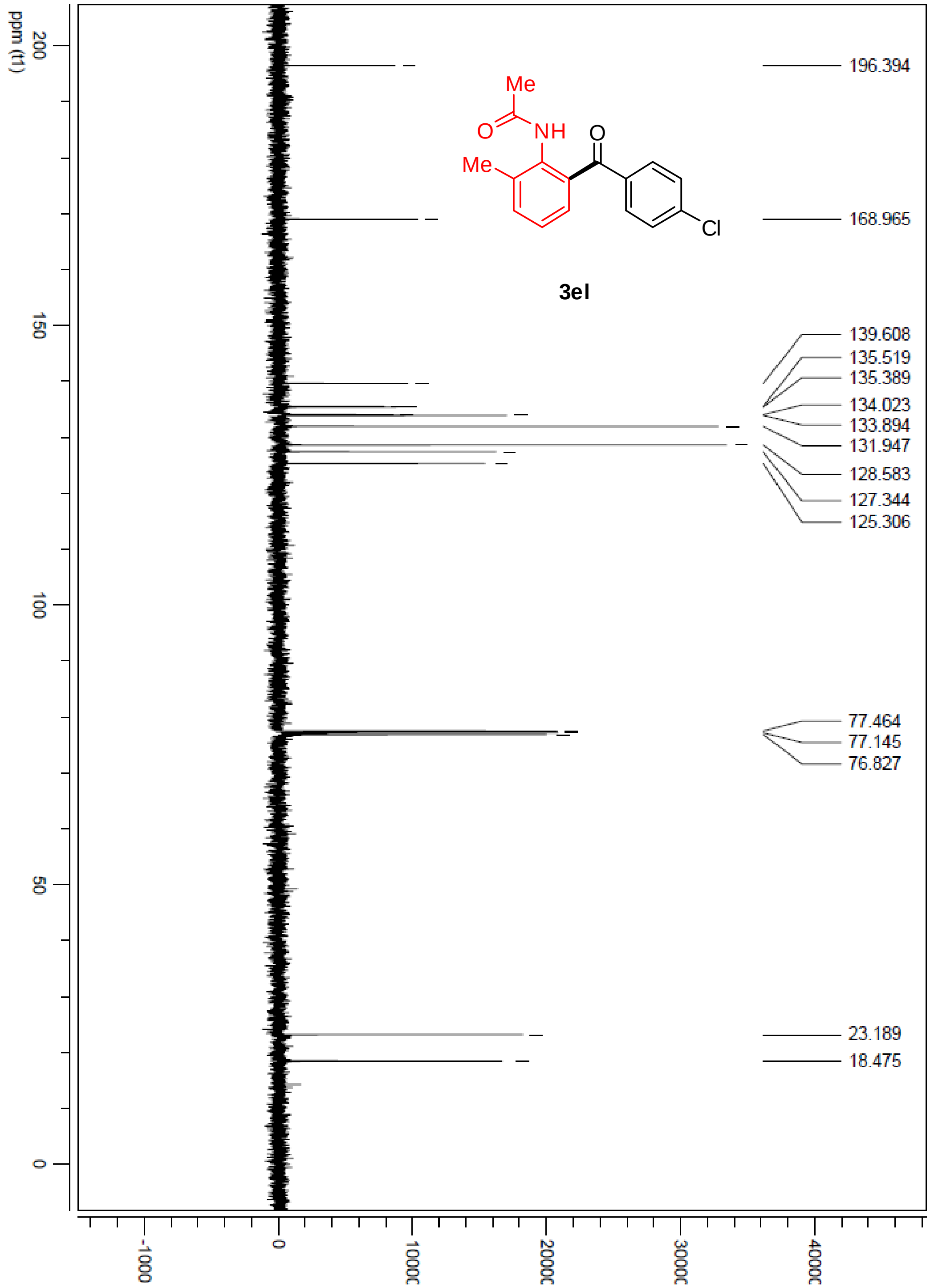
1.852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O
Me
Cl
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139.608
135.519
135.389
134.023
133.894
131.947
128.583
127.344
125.306
77.464
77.145
76.827
23.189
18.475
ppm (t1)
200
150
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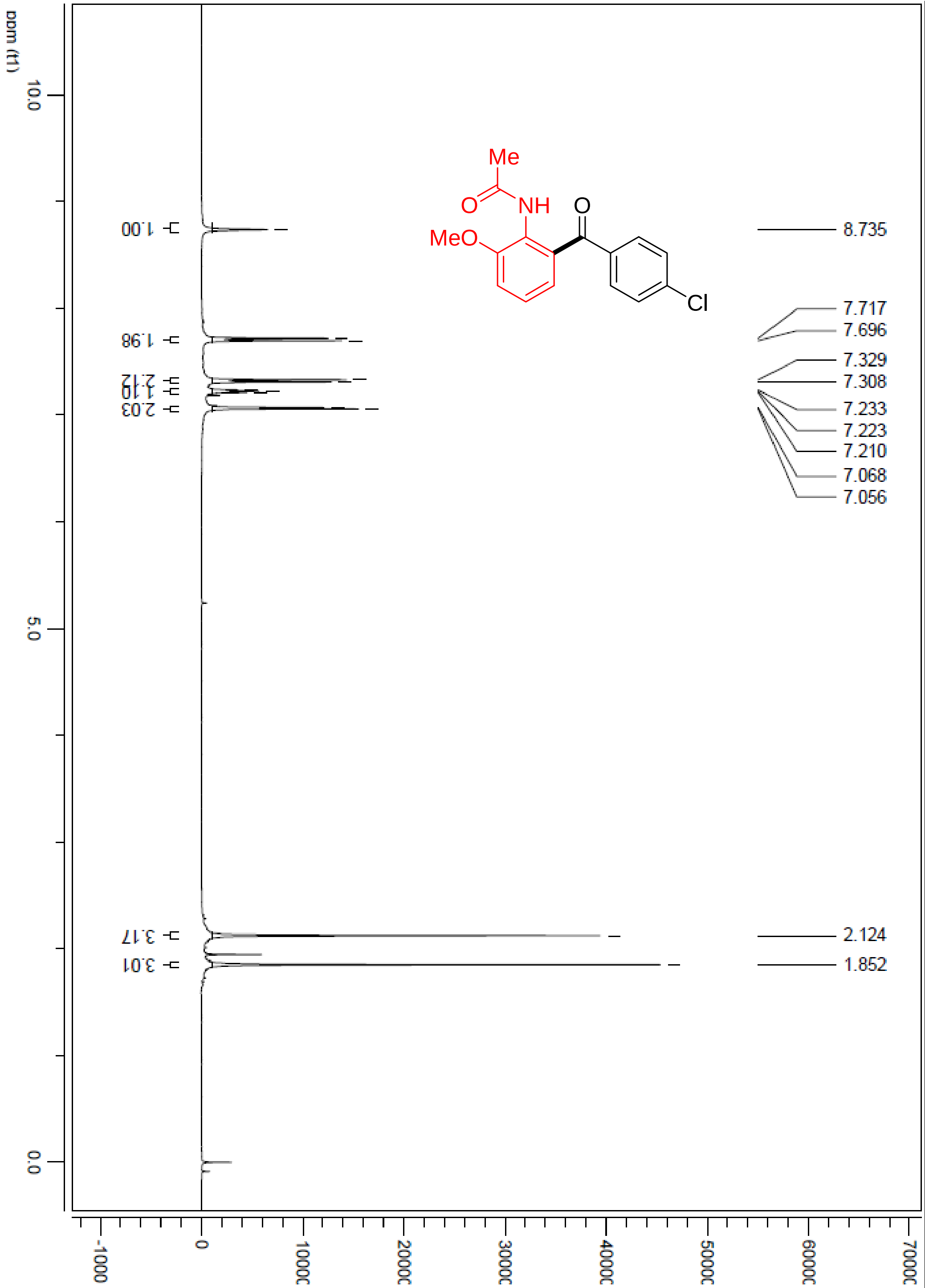
50
0
-1000
0
1000C
2000C
3000C
4000C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NH
O
MeO
Cl
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7.717
7.696
7.329
7.308
7.233
7.223
7.210
7.068
7.056
2.124
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2.12
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3.17
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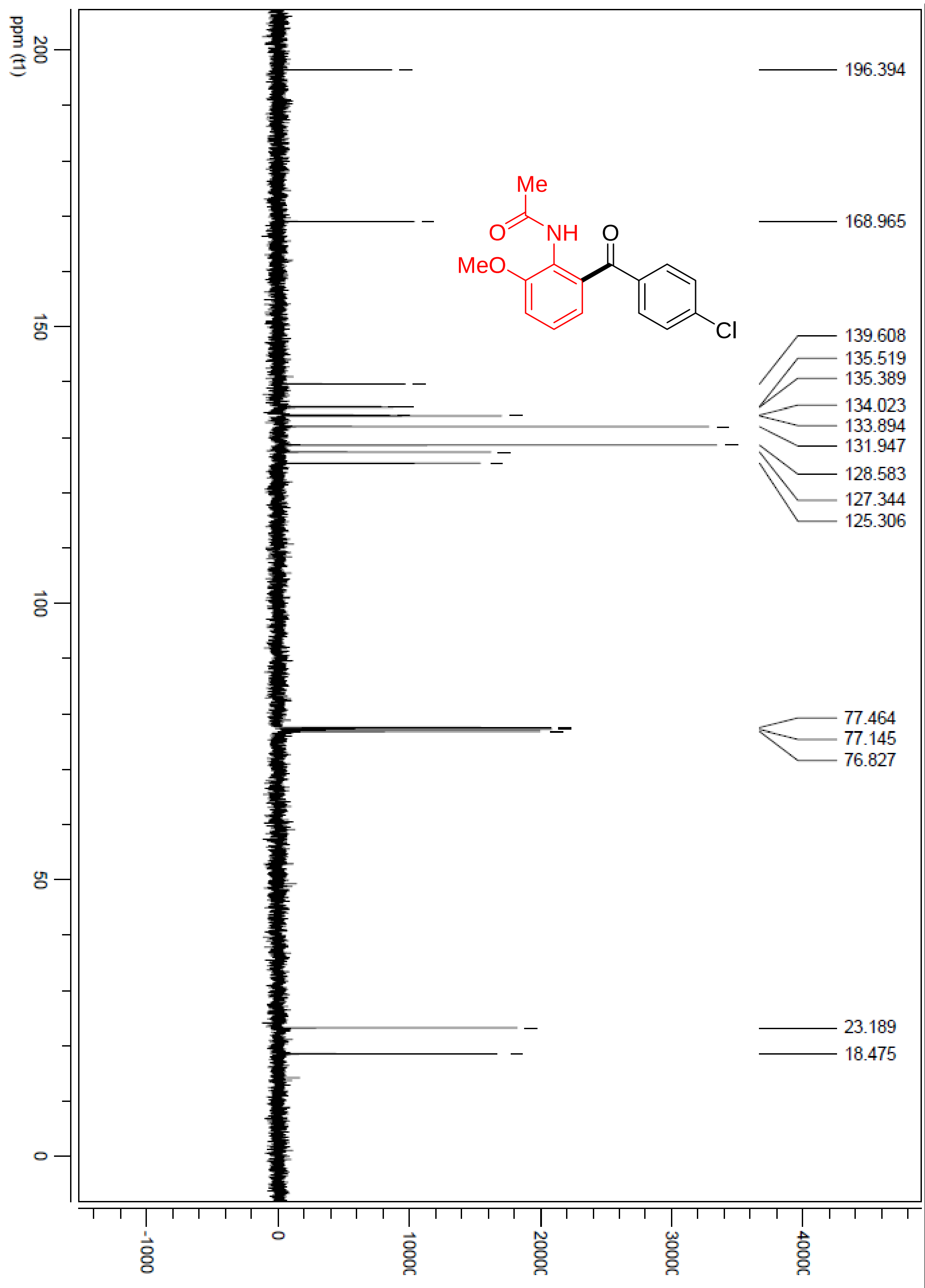
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O
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139.608
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134.023
133.894
131.947
128.583
127.344
125.306
77.464
77.145
76.827
23.189
18.475
ppm (t1)
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150
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50
0
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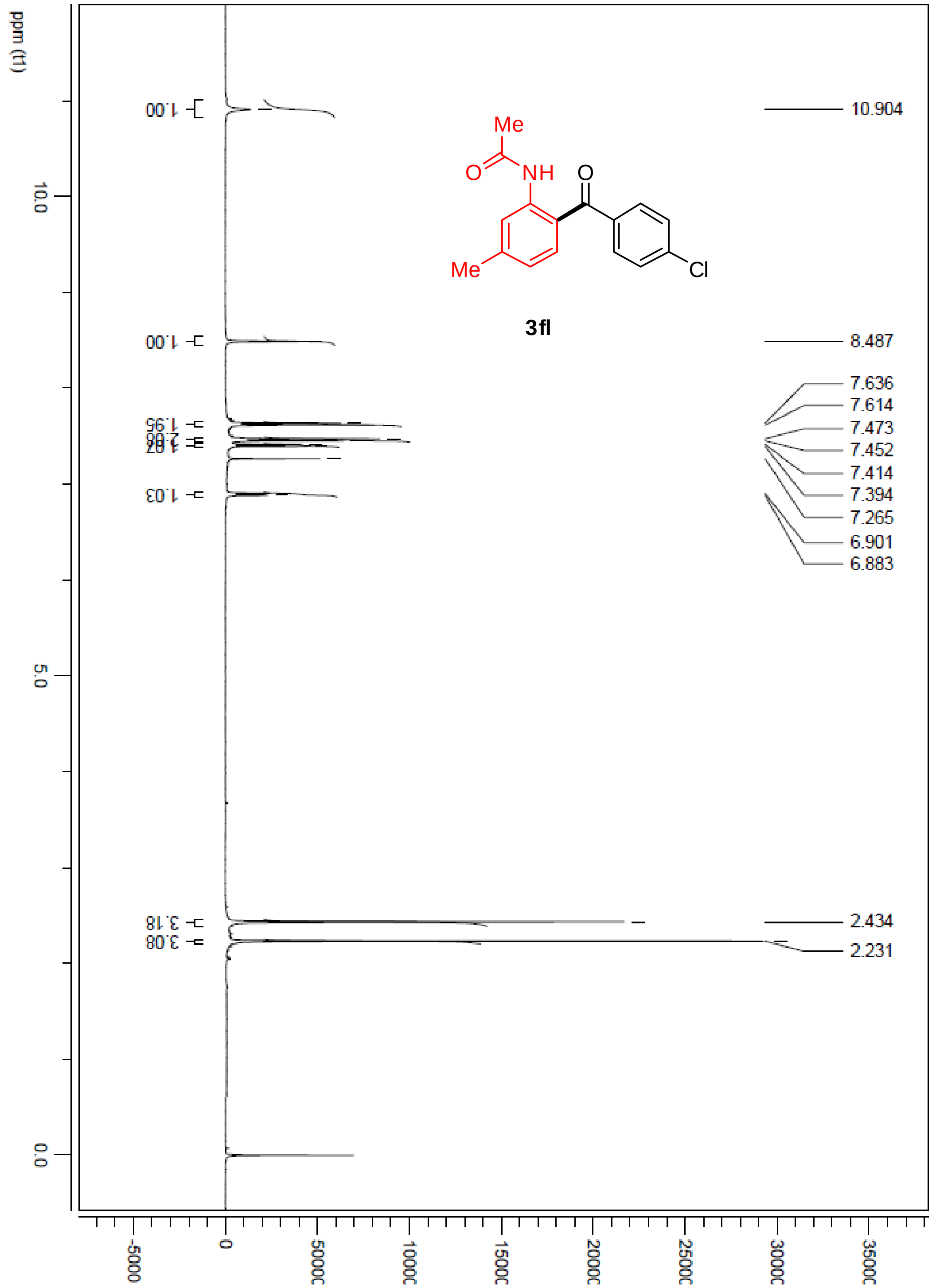
0
1000
2000
3000
4000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7.394
7.265
6.901
6.883
2.434
2.231
1.00
1.00
1.95
2.06
1.02
1.03
3.18
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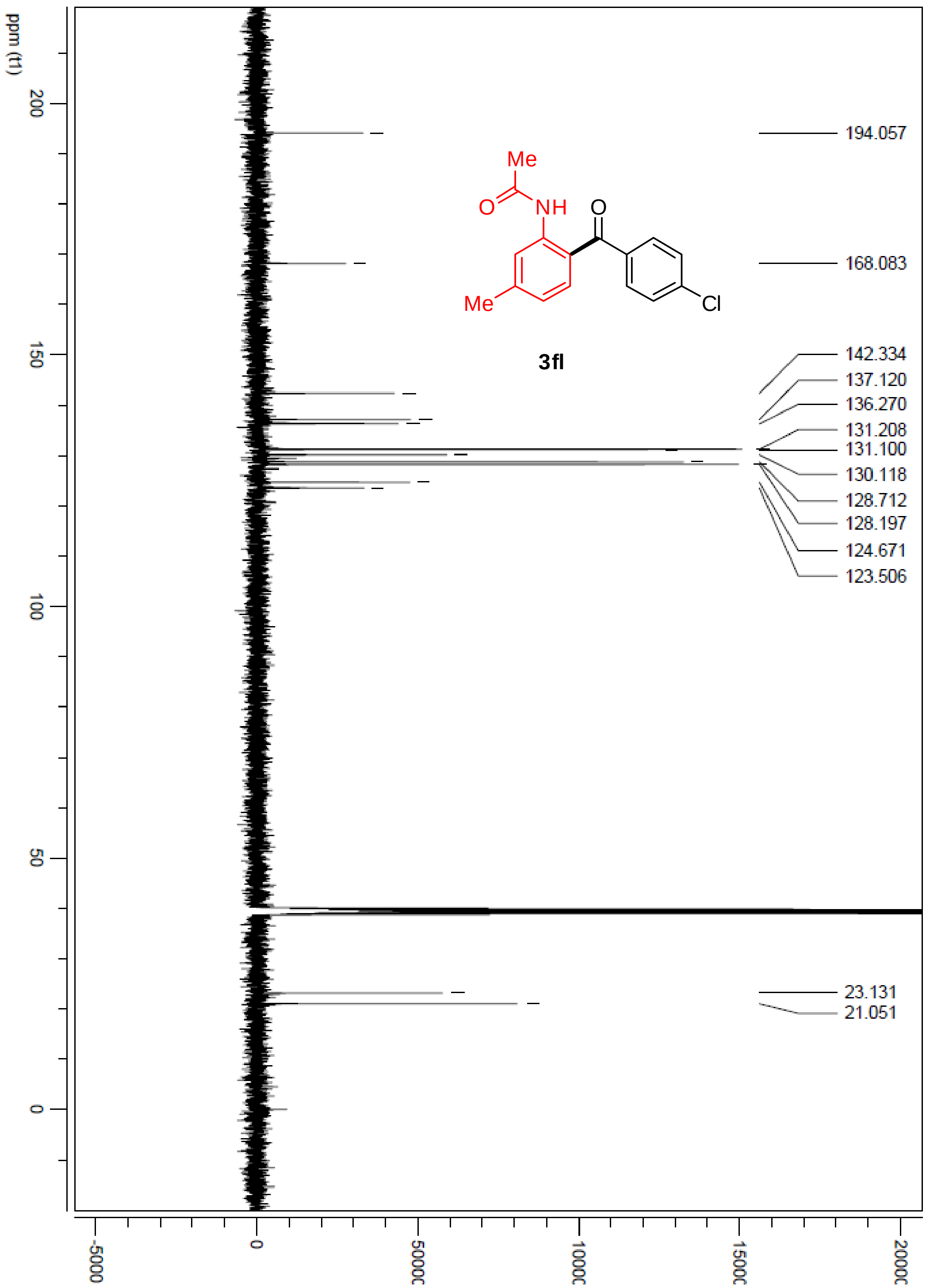
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0
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10000
15000
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136.270
131.208
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128.197
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Cl
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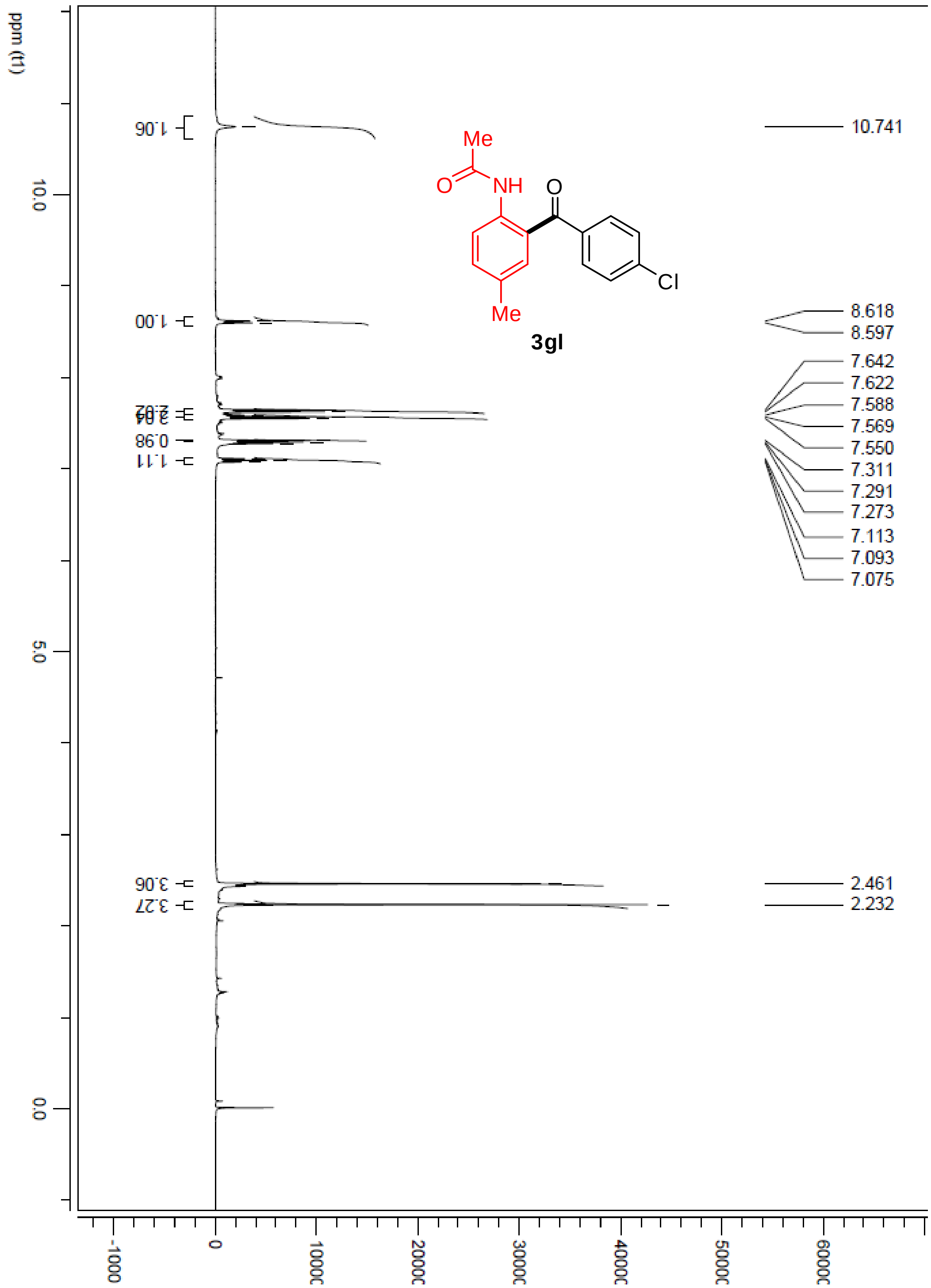
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O
O
Cl
Me
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7.642
7.622
7.588
7.569
7.550
7.311
7.291
7.273
7.113
7.093
7.075
2.461
2.232
1.06
1.00
2.02
2.04
0.98
1.11
3.06
3.27
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10000
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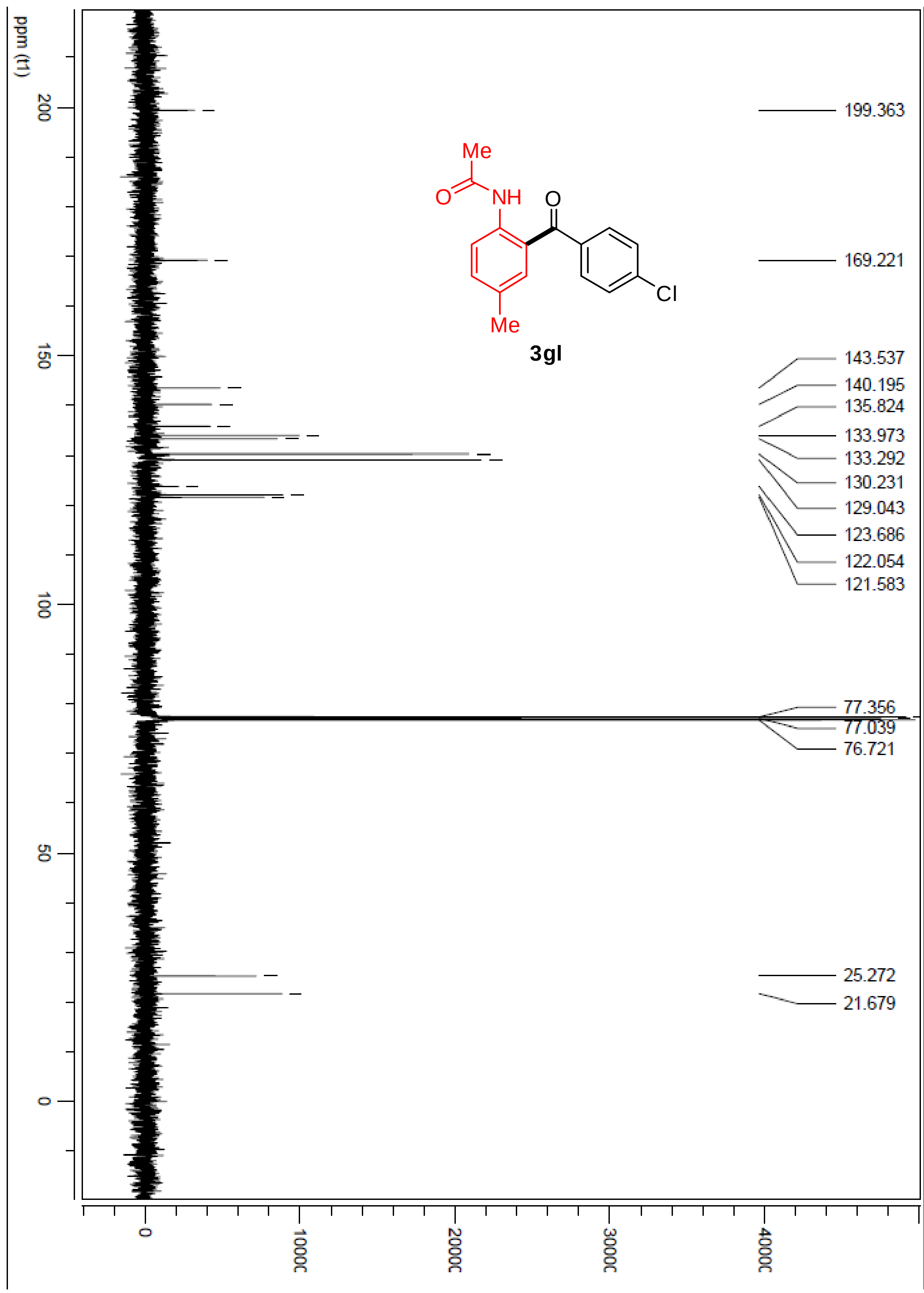
30000
40000
50000
60000


ppm (t1)
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100
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133.973
133.292
130.231
129.043
123.686
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77.039
76.721
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21.679
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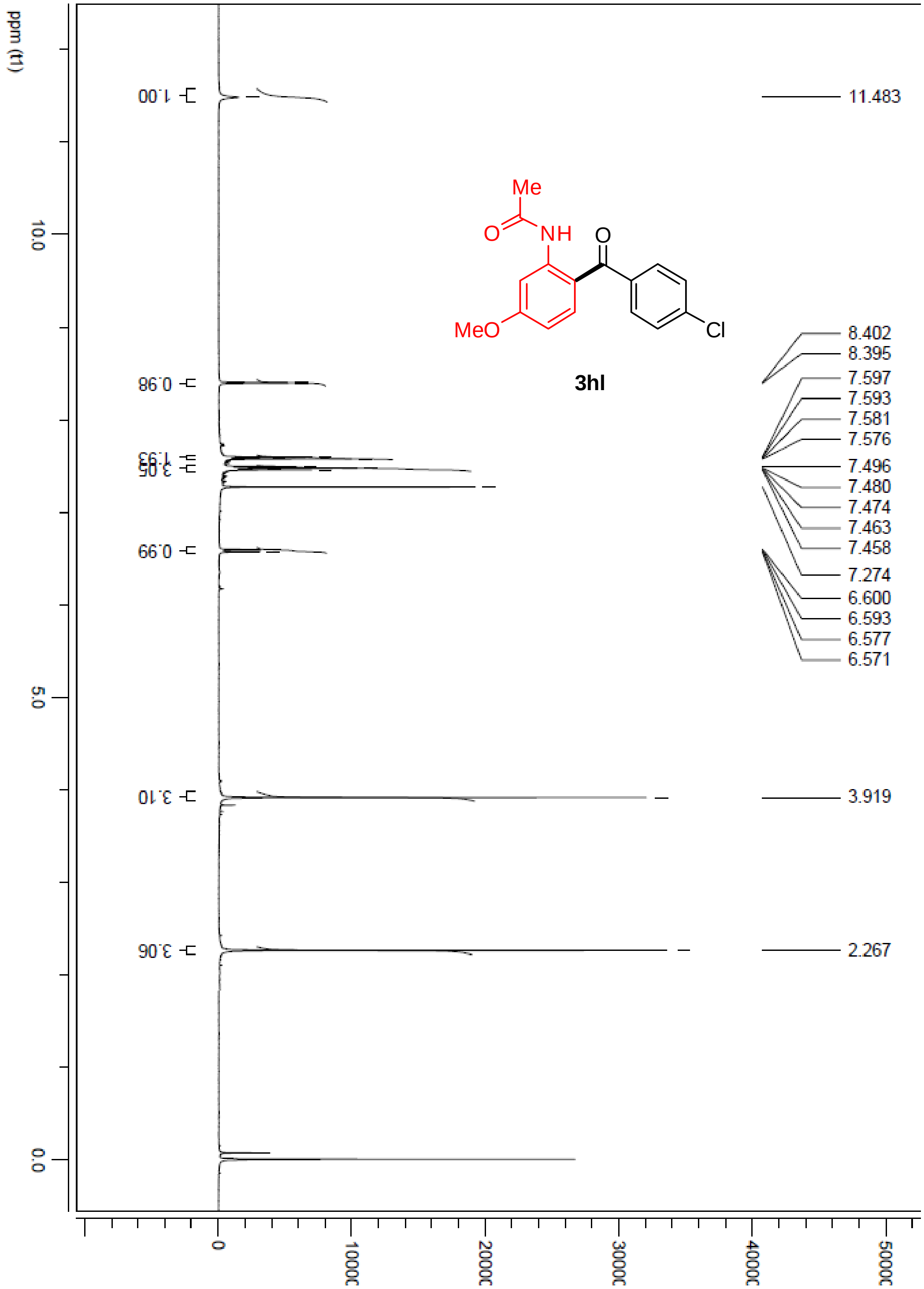
O
Cl
Me
3gl


ppm (t1)
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MeO
Cl
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7.576
7.496
7.480
7.474
7.463
7.458
7.274
6.600
6.593
6.577
6.571
3.919
2.267
1.00
0.98
1.93
3.05
0.99
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30000
40000
50000

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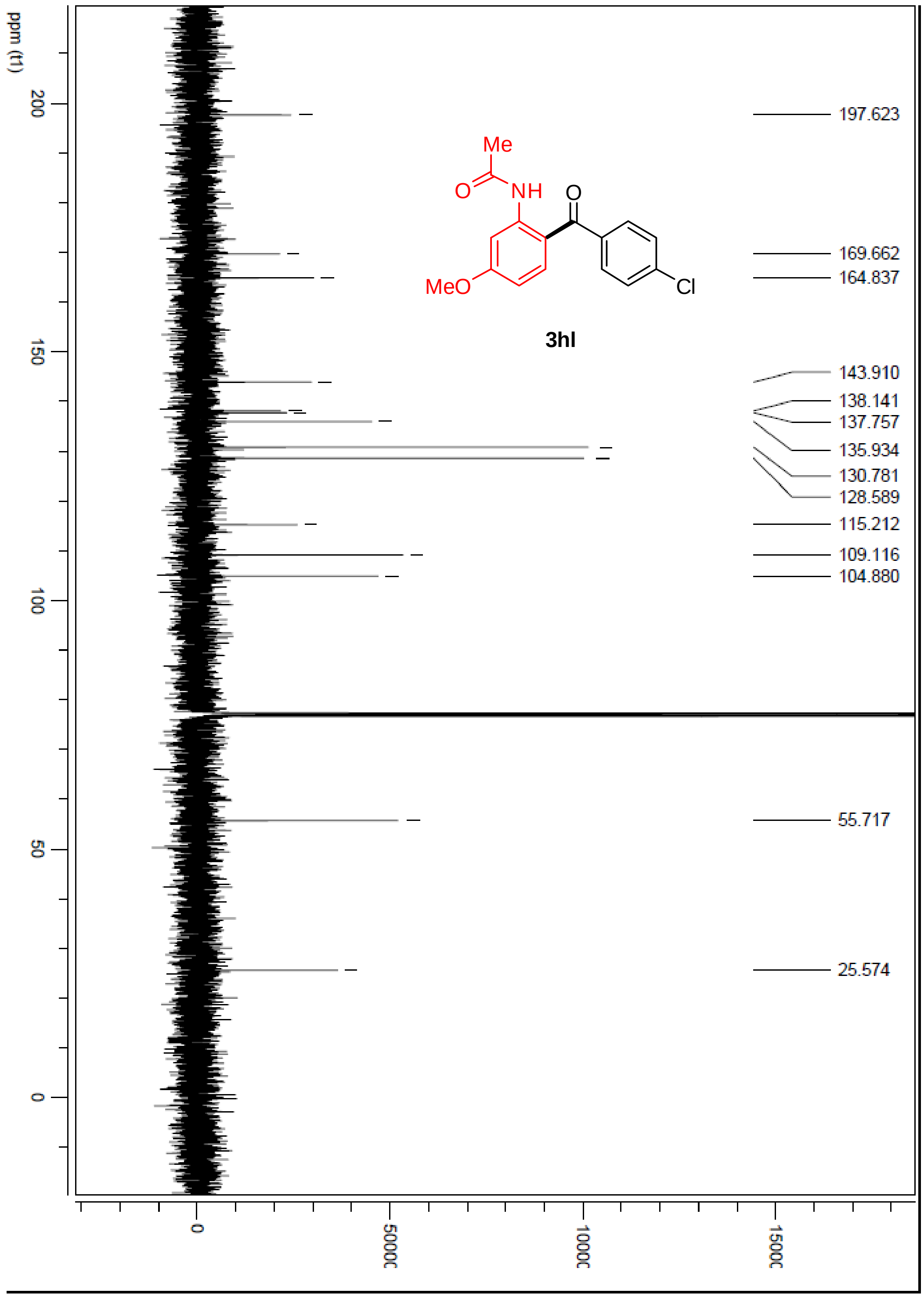

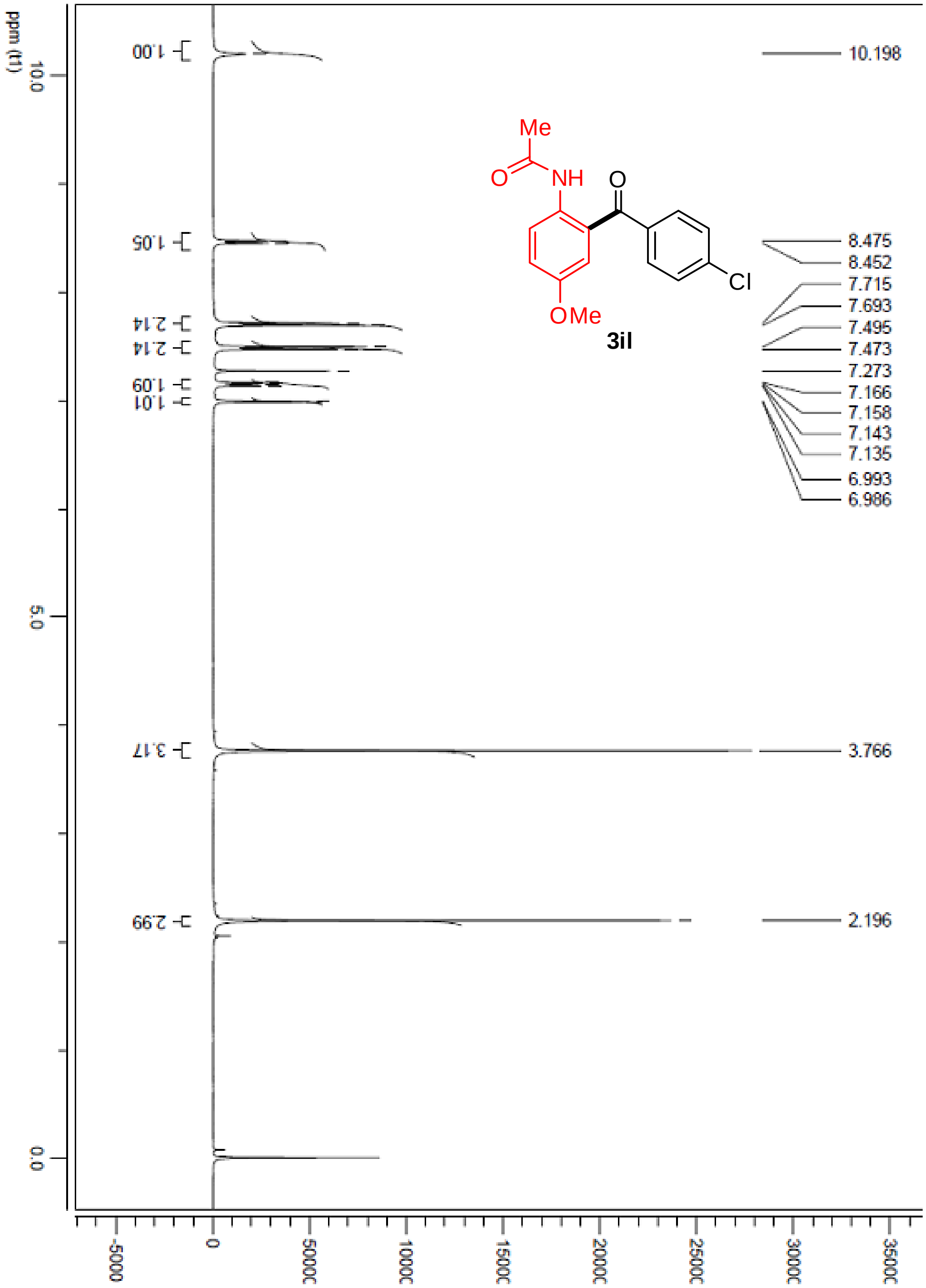
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O
Cl
OMe
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7.273
7.166
7.158
7.143
7.135
6.993
6.986
3.766
2.196
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2.14
1.09
1.01
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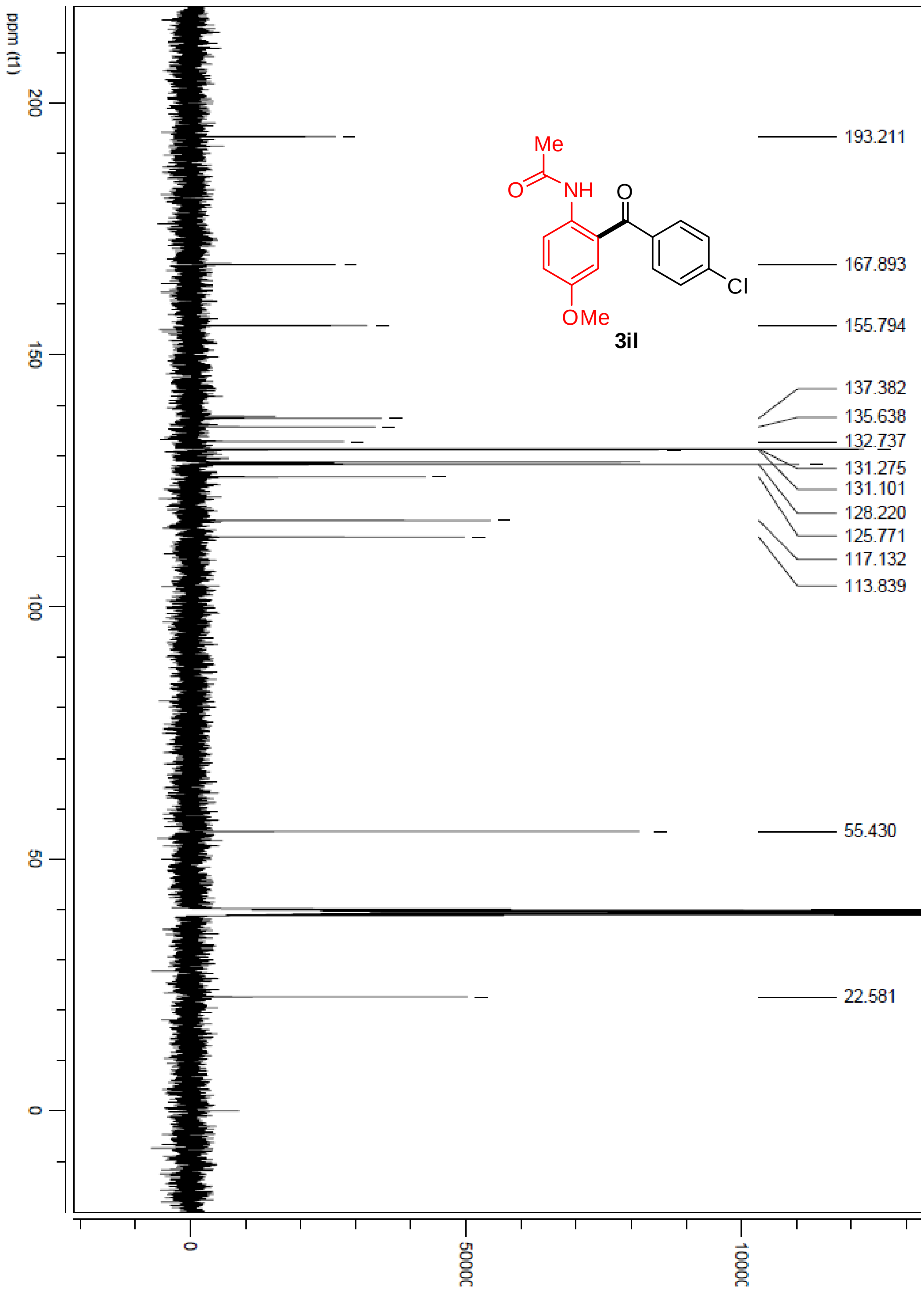
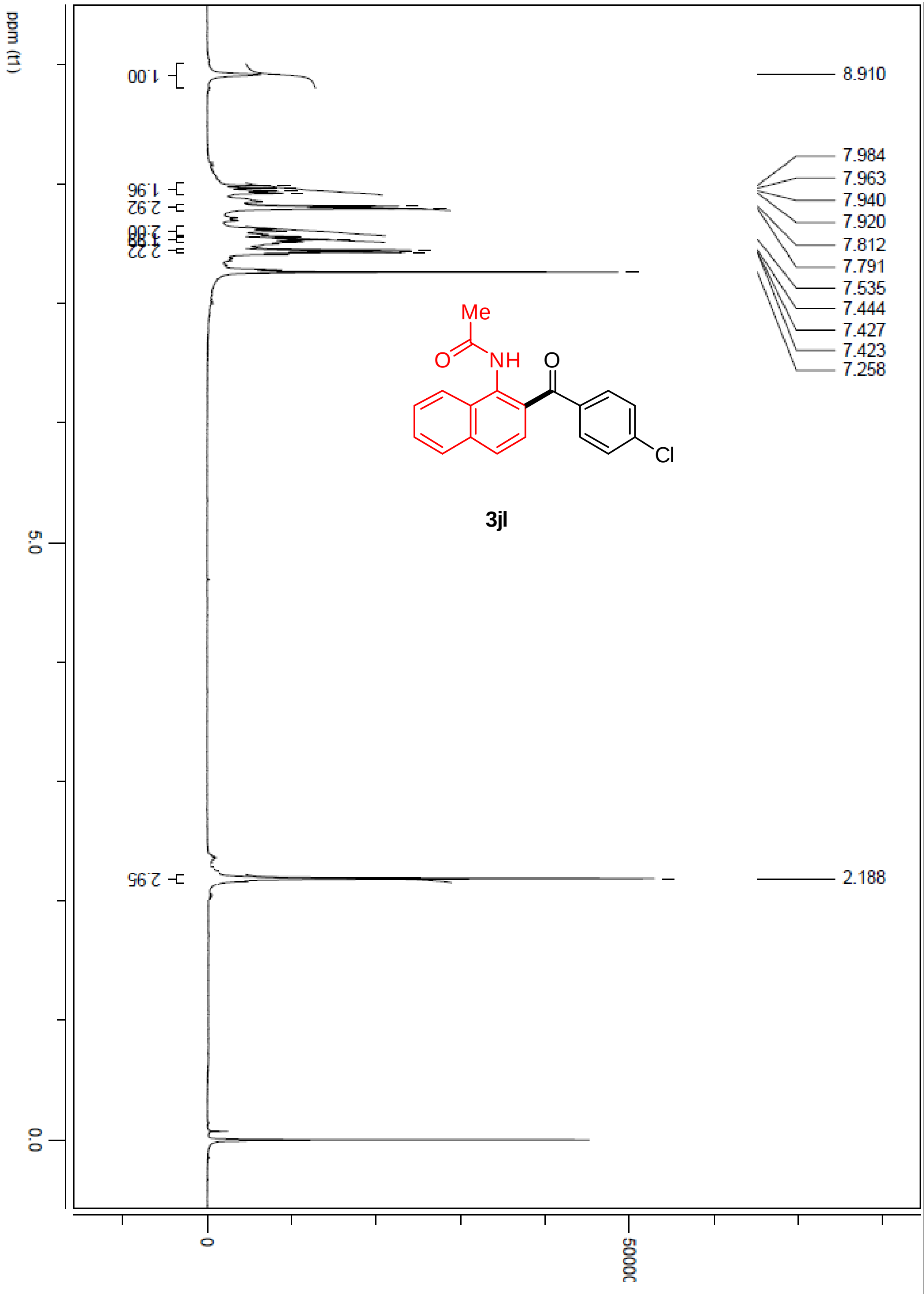
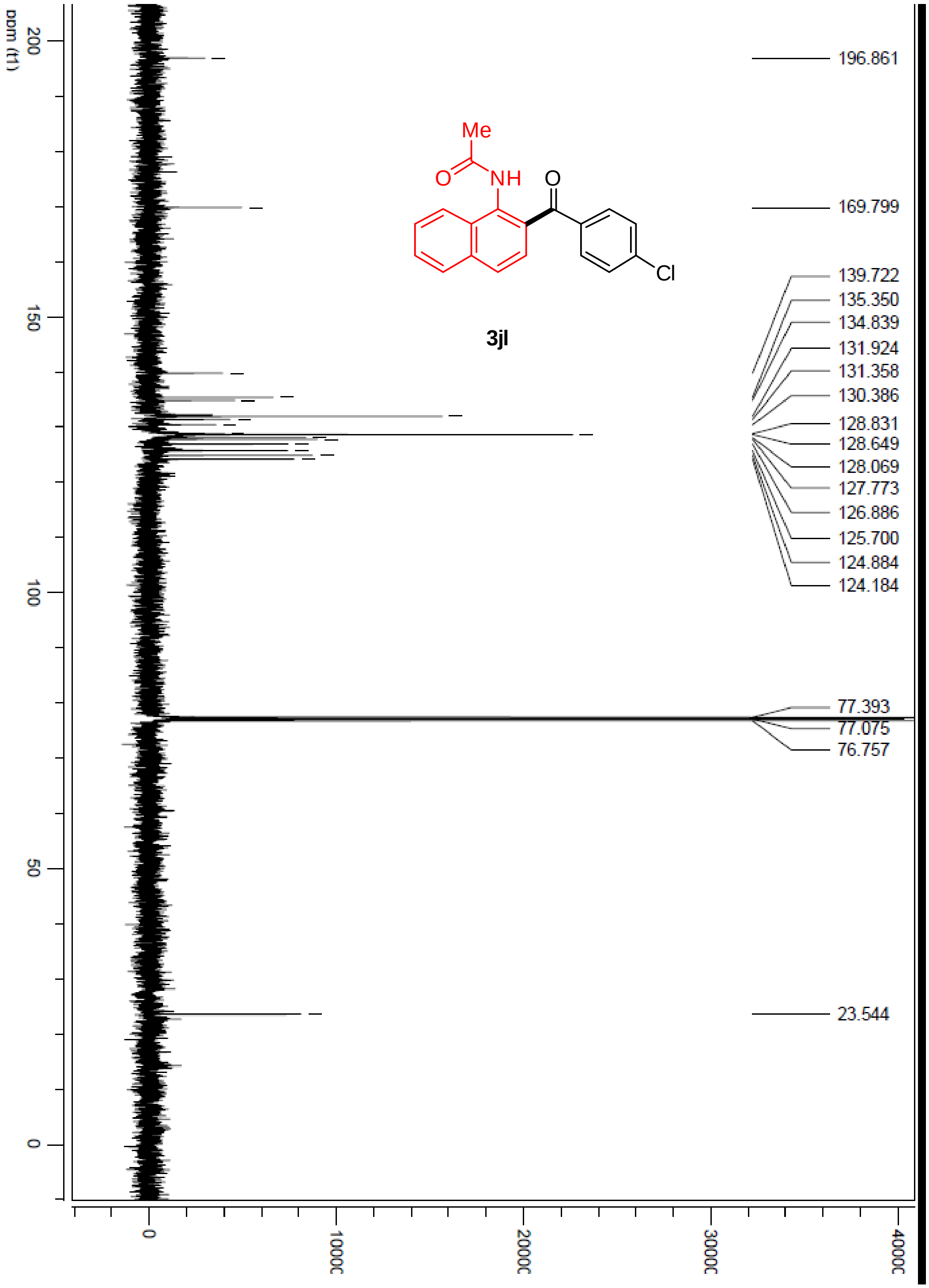
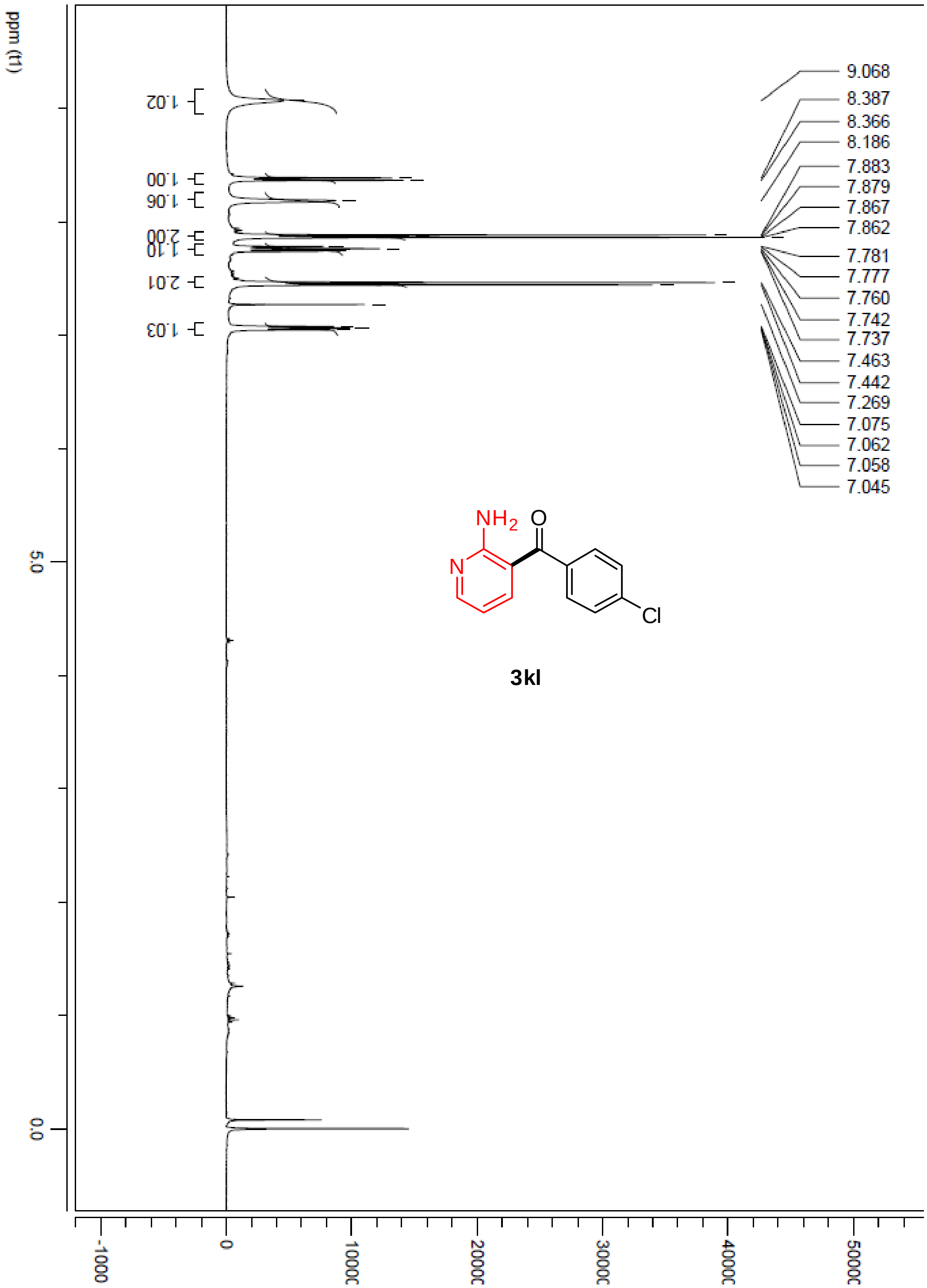
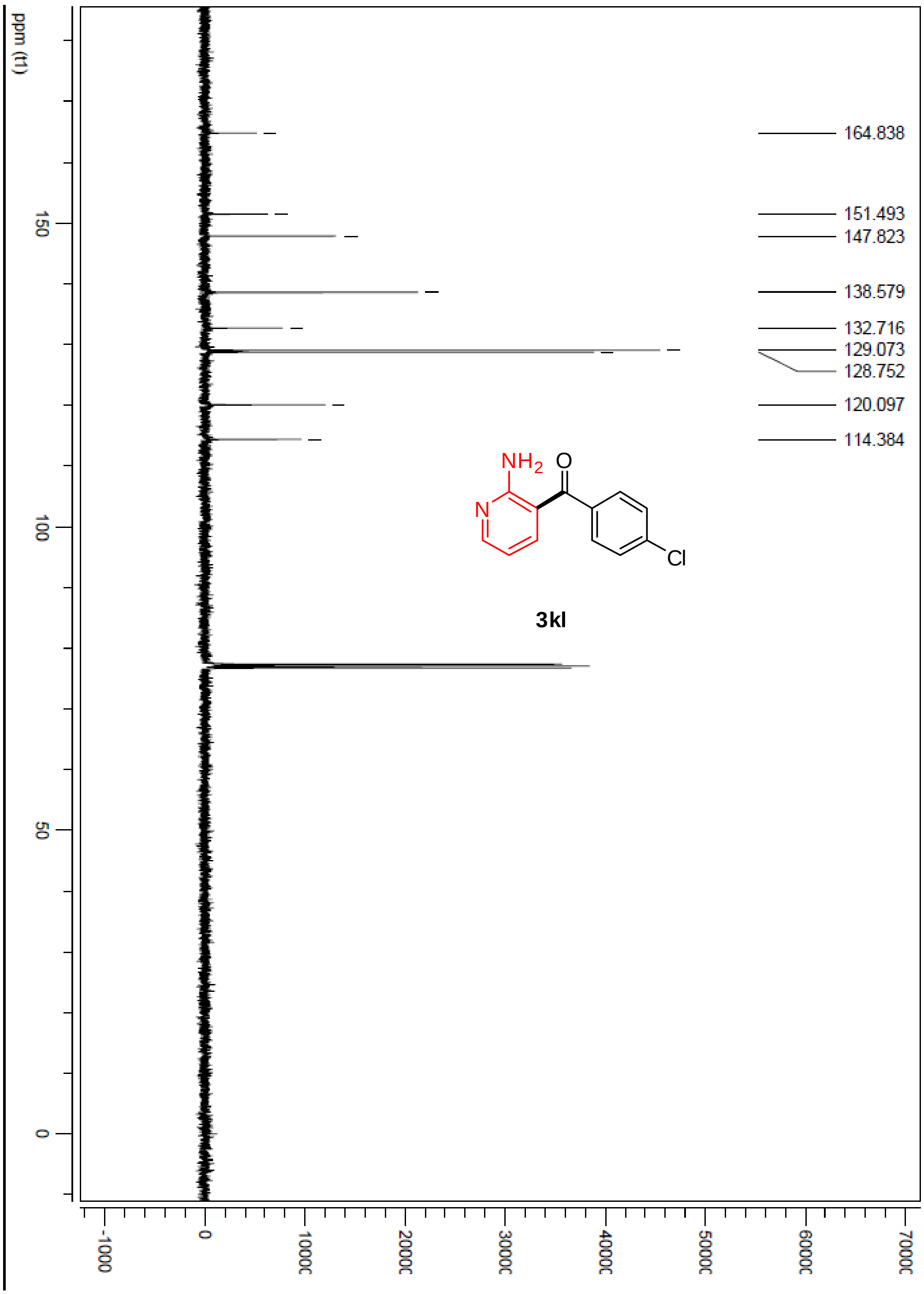

193.211
167.893
155.794
137.382
135.638
132.737
131.275
131.101
128.220
125.771
117.132
113.839
55.430
22.581
Me
O
NH
O
Cl
OMe
3il
ppm (t1)
200
150
100
50
0
5000
10000

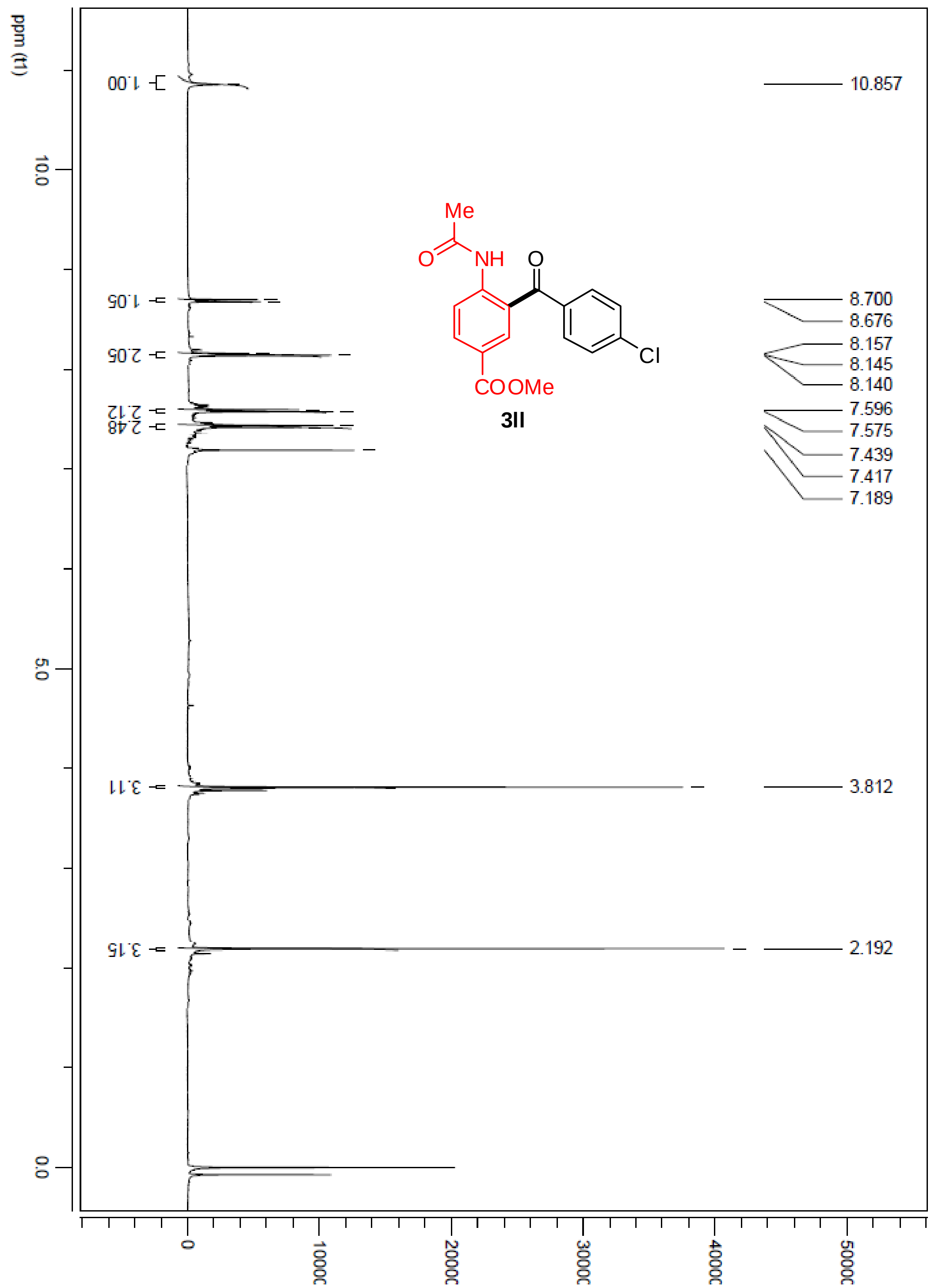

8.910
7.984
7.963
7.940
7.920
7.812
7.791
7.535
7.444
7.427
7.423
7.258
2.188
1.00
1.96
2.92
2.00
2.22
2.95
ppm (t1)
5.0
0.0
0
5000
Me
O
NH
O
Cl
3jl

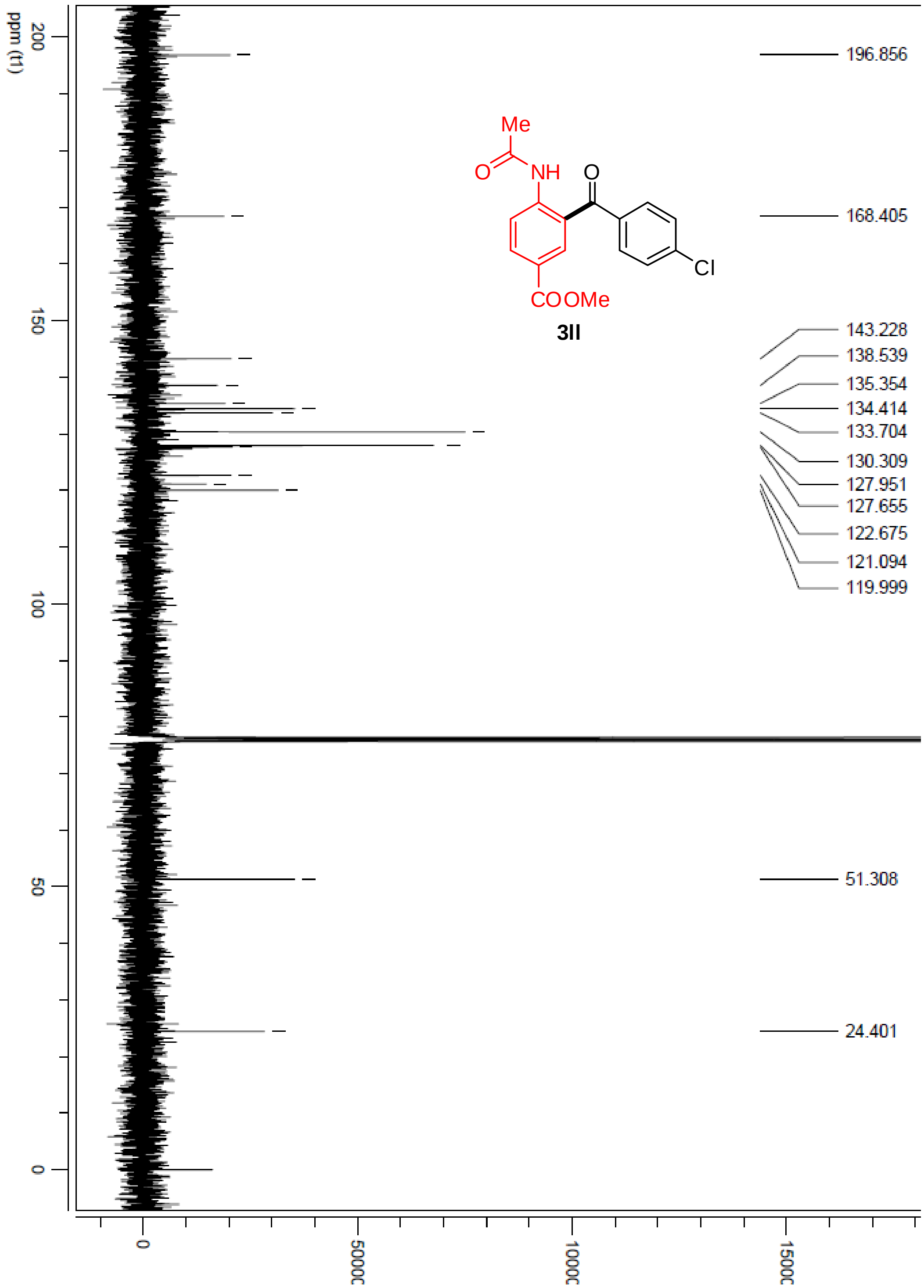
Electronic Supplementary Material (ESI) for Chemical Communications

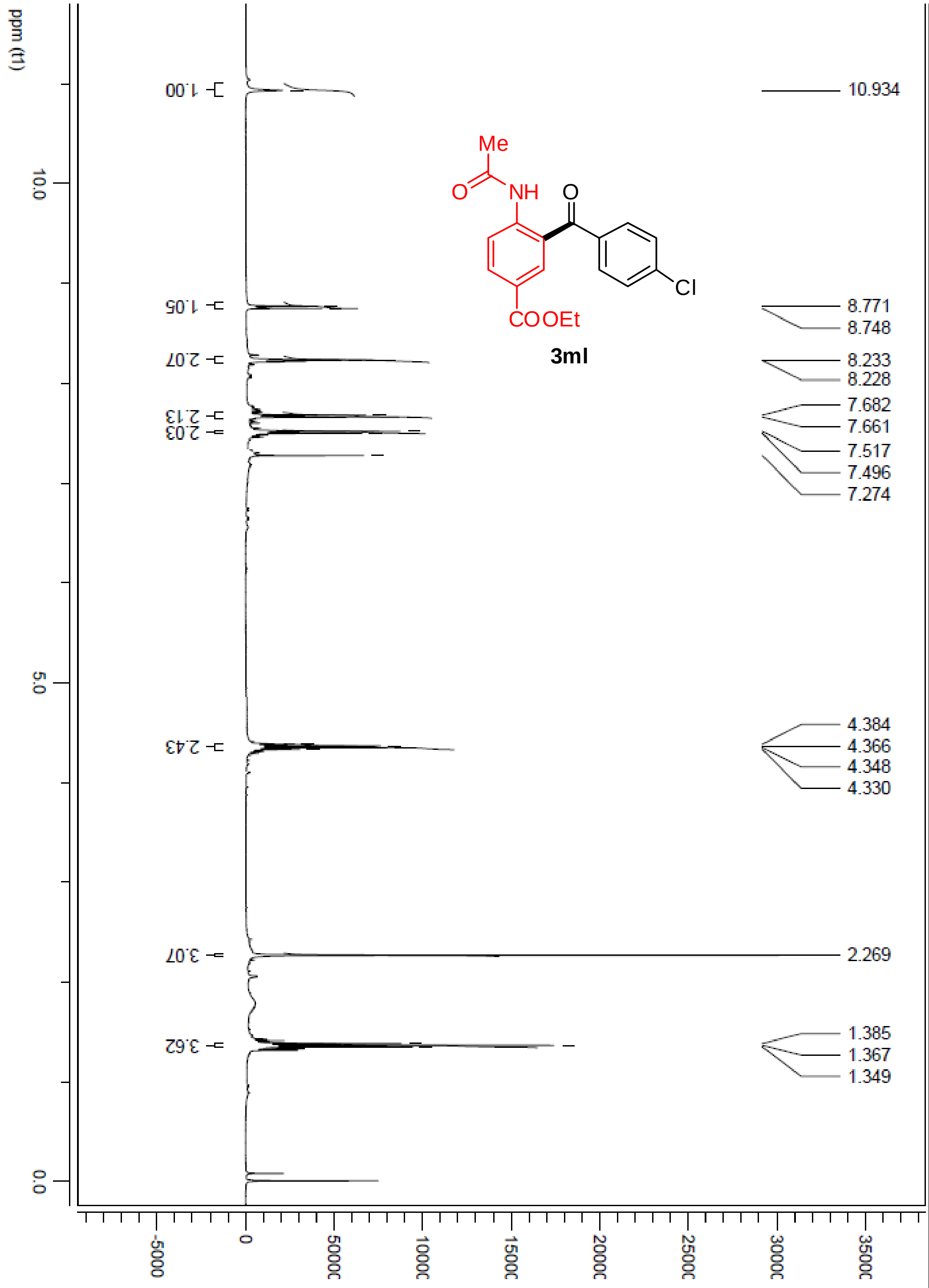


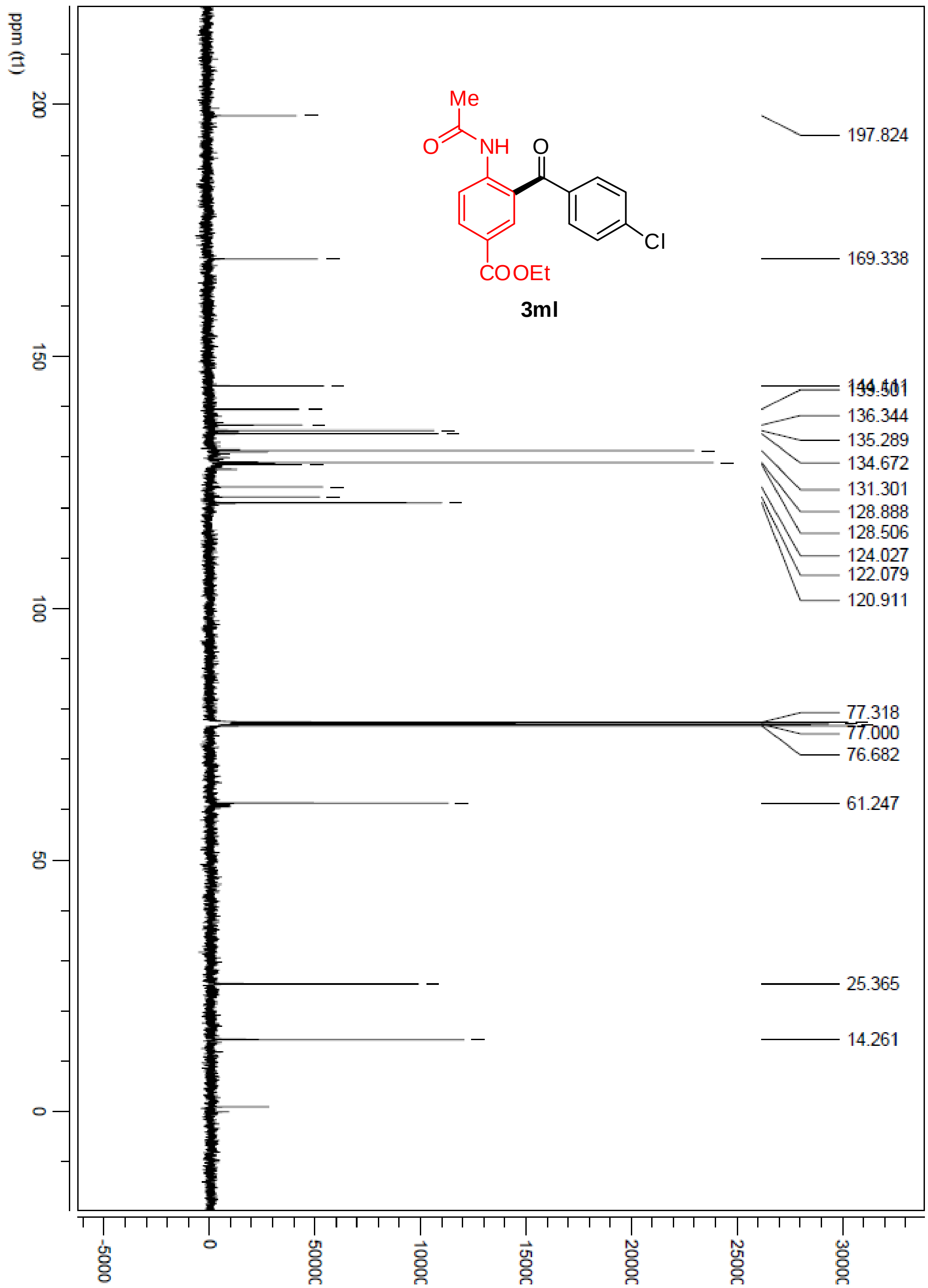

9.068
8.387
8.366
8.186
7.883
7.879
7.867
7.862
7.781
7.777
7.760
7.742
7.737
7.463
7.442
7.269
7.075
7.062
7.058
7.045
NH2
O
N
Cl
3kl
1.02
1.00
1.06
2.00
1.10
2.01
1.03
ppm (t1)
5.0
0.0

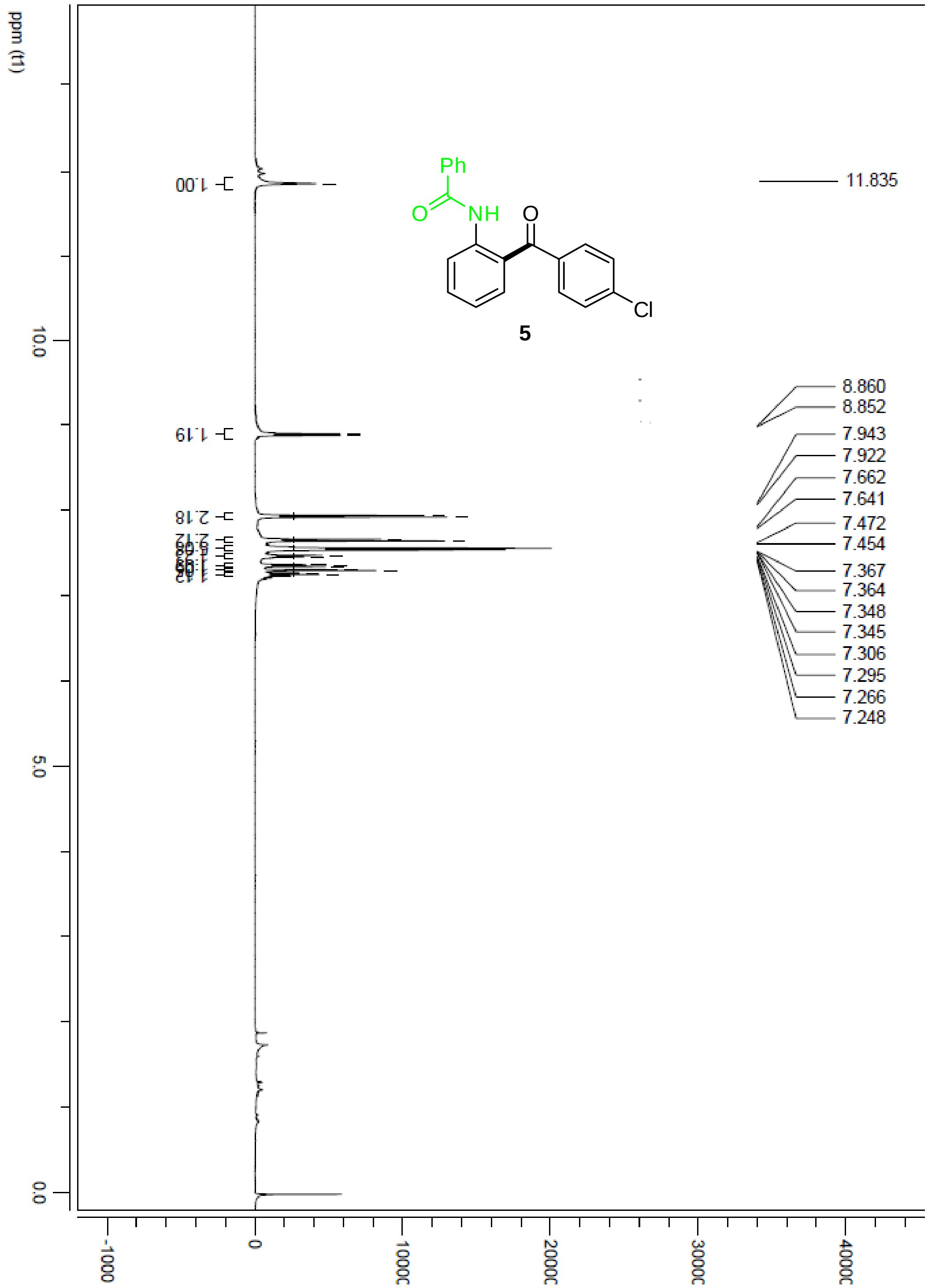

164.838
151.493
147.823
138.579
132.716
129.073
128.752
120.097
114.384
NH2
O
N
Cl
3kl
ppm (t1)
150
100
50
0
-1000
0
10000
20000
30000
40000
50000
60000
70000


Me
O
NH
O
Cl
COOMe
3ll
10.857
8.700
8.676
8.157
8.145
8.140
7.596
7.575
7.439
7.417
7.189
3.812
2.192
1.00
1.05
2.05
2.12
2.48
3.11
3.15
ppm (t1)
10.0
5.0
0.0
0
1000
2000
3000
4000
5000

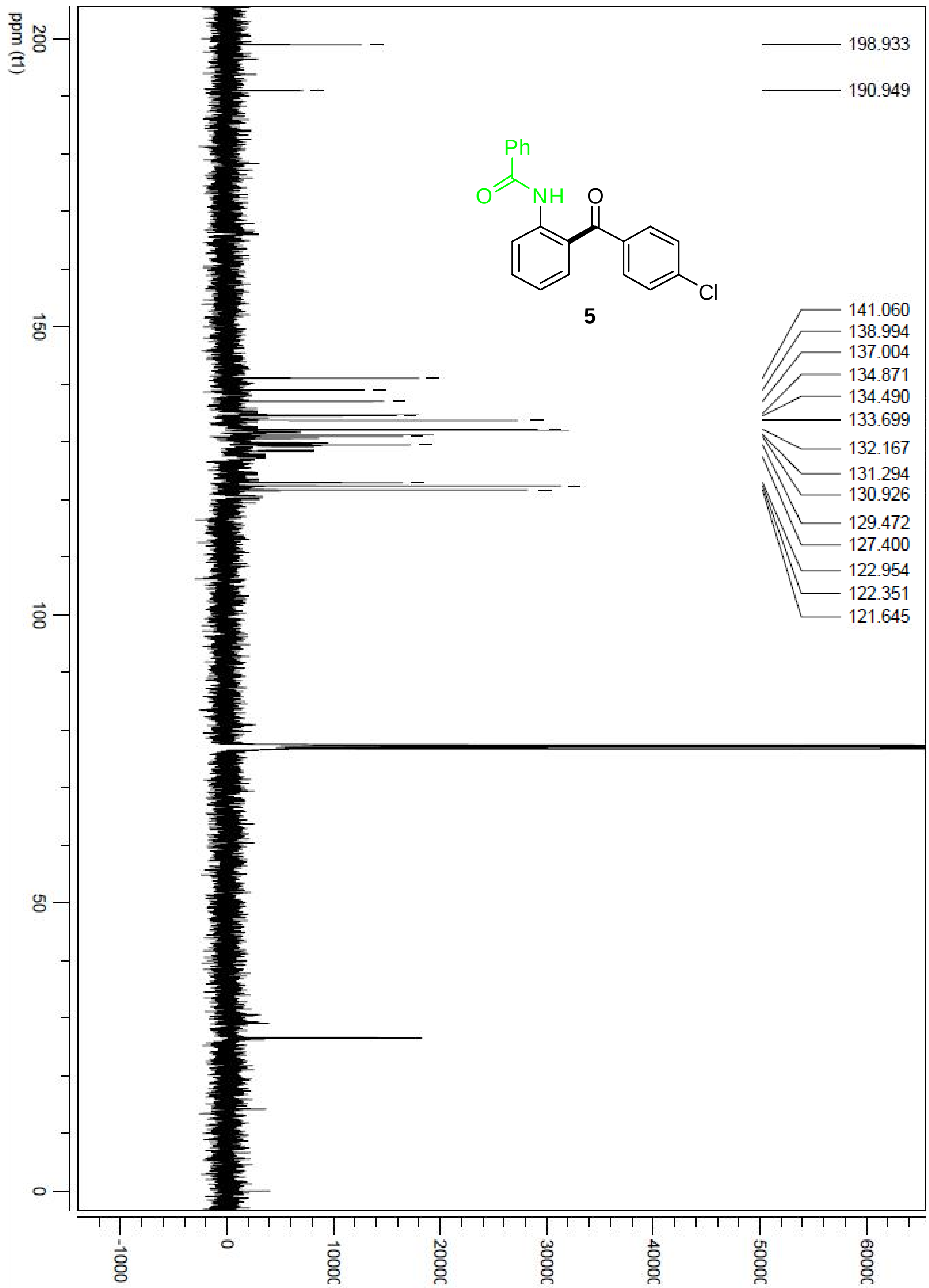

ppm (t1)
200
150
100
50
0
196.856
168.405
143.228
138.539
135.354
134.414
133.704
130.309
127.951
127.655
122.675
121.094
119.999
51.308
24.401
Me
O
NH
O
Cl
COOMe
3ll
0
5000
10000
15000

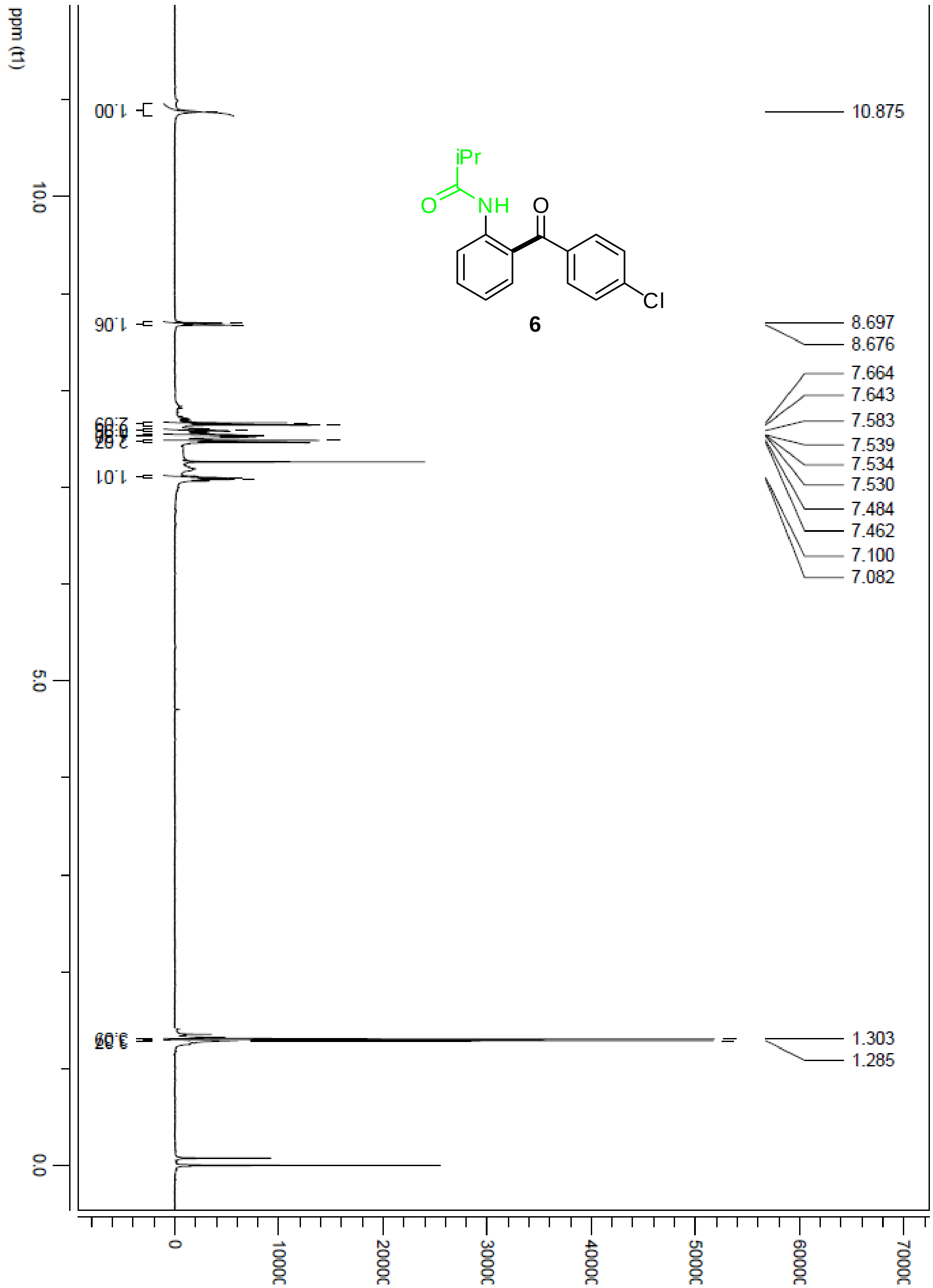

Me
O
NH
O
Cl
COOEt
3ml
10.934
8.771
8.748
8.233
8.228
7.682
7.661
7.517
7.496
7.274
4.384
4.366
4.348
4.330
2.269
1.385
1.367
1.349
1.00
1.05
2.07
2.13
2.03
2.43
3.07
3.62
ppm (t1)
10.0
5.0
0.0
-5000
0
5000
10000
15000
20000
25000
30000
35000

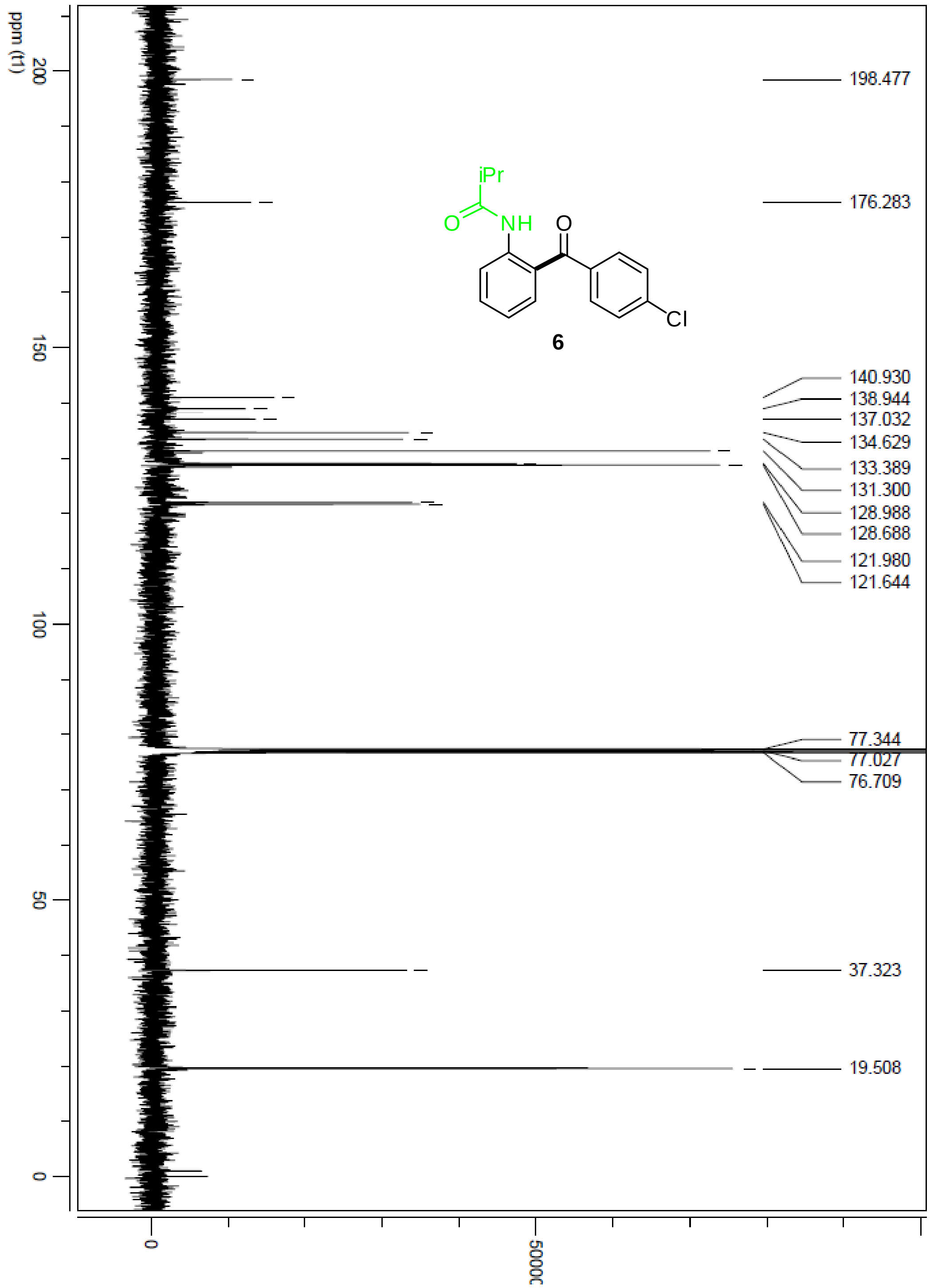

Me
NH
O
O
Cl
COOEt
3ml
197.824
169.338
136.344
135.289
134.672
131.301
128.888
128.506
124.027
122.079
120.911
77.318
77.000
76.682
61.247
25.365
14.261
ppm (t1)

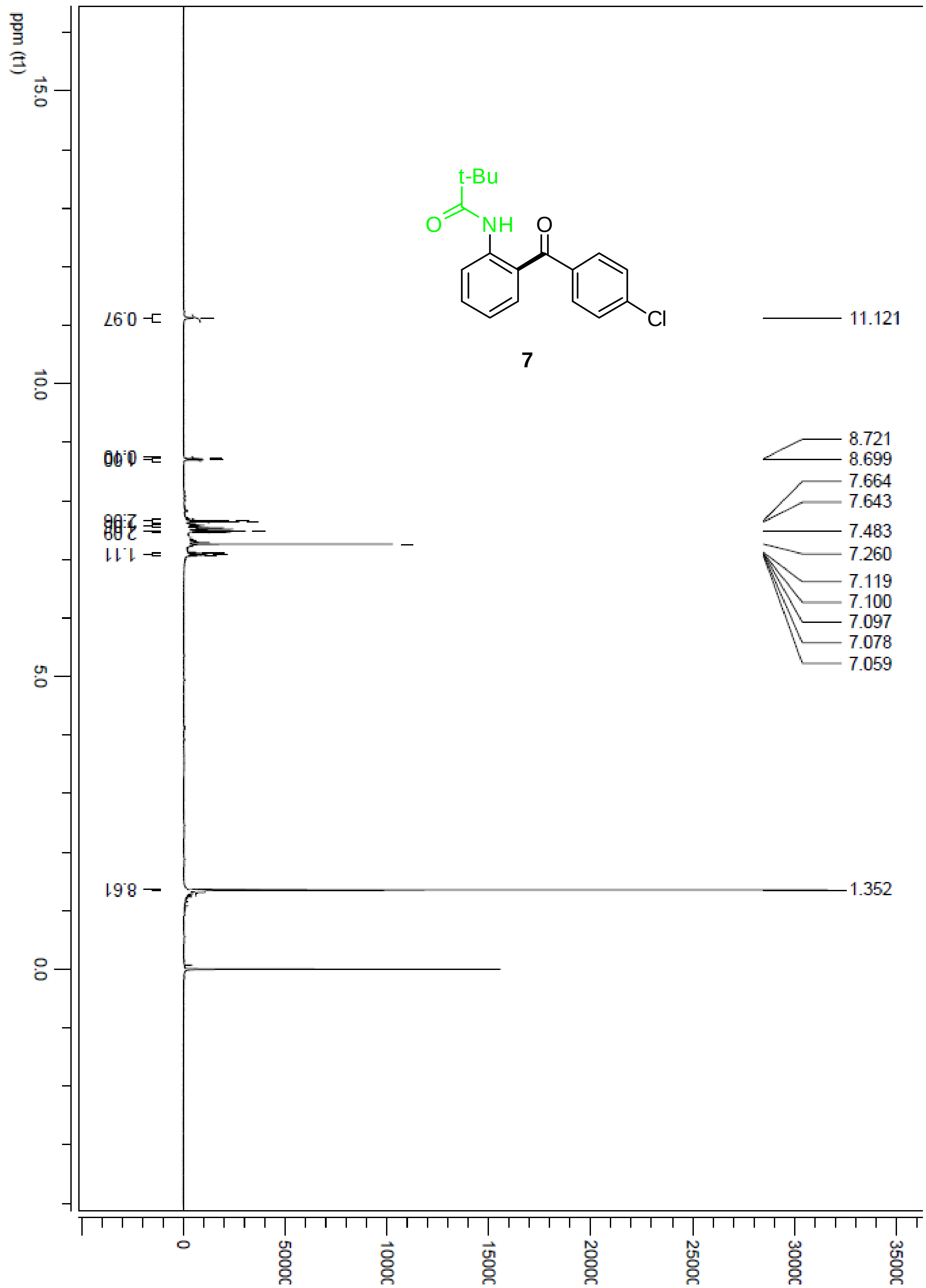

Ph
O
NH
O
Cl
5
11.835
8.860
8.852
7.943
7.922
7.662
7.641
7.472
7.454
7.367
7.364
7.348
7.345
7.306
7.295
7.266
7.248
1.00
1.19
2.18
ppm (t1)
10.0
5.0
0.0
-1000
0
1000C
2000C
3000C
4000C

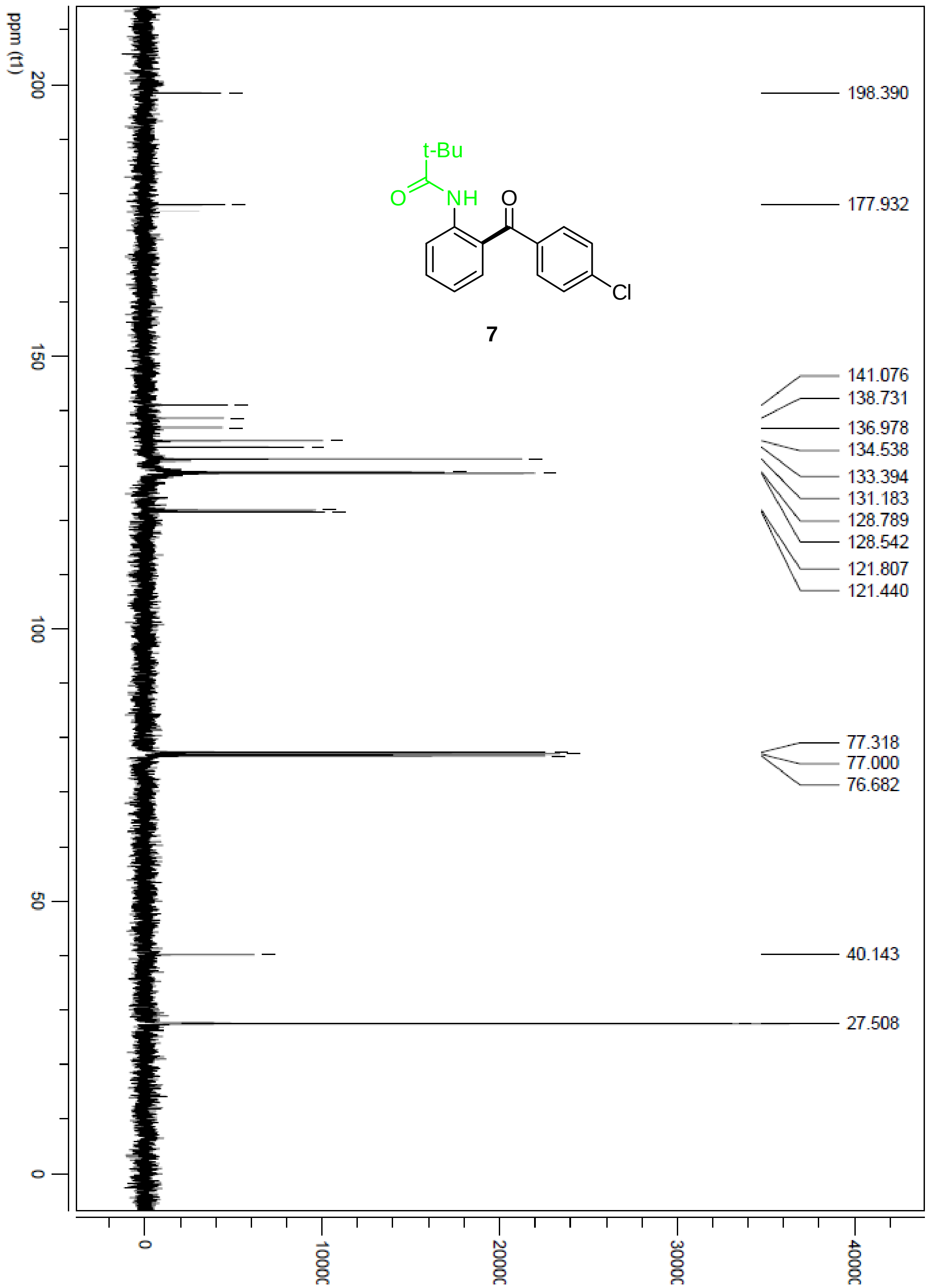
Electronic Supplementary Material (ESI) for Chemical Communications




iPr
O
NH
O
Cl
6
10.875
8.697
8.676
7.664
7.643
7.583
7.539
7.534
7.530
7.484
7.462
7.100
7.082
1.303
1.285
1.00
1.06
1.01
ppm (t1)
10.0
5.0
0.0
0
10000
20000
30000
40000
50000
60000
70000


ppm (t1)
200
150
100
50
0
0
50000
iPr
O
NH
O
Cl
6
198.477
176.283
140.930
138.944
137.032
134.629
133.389
131.300
128.988
128.688
121.980
121.644
77.344
77.027
76.709
37.323
19.508


t-Bu
O
NH
O
Cl
7
11.121
8.721
8.699
7.664
7.643
7.483
7.260
7.119
7.100
7.097
7.078
7.059
1.352
0.97
1.00
2.00
2.08
1.11
8.61
ppm (t1)
15.0
10.0
5.0
0.0


t-Bu
O
NH
O
Cl
7
198.390
177.932
141.076
138.731
136.978
134.538
133.394
131.183
128.789
128.542
121.807
121.440
77.318
77.000
76.682
40.143
27.508
ppm (t1)
200
150
100
50
0
0
10000
20000
30000
40000

6. References

[1] W. L. F. Armarego and D. D. Perrin, *In Purification of Laboratory Chemicals*, 4th Ed., Butterworth-Heinemann: Oxford UK: 1996.

[2] D. M. Tschaen, L. Abramson, D. Cai, R. Desmond, U.-H. Dolling, L. Frey, S. Karady, Y. Shi and T. R. Verhoeven, *J. Org. Chem.* 1995, **60**, 4322.

[3] L. Huang, Z. Luo, F. He, J. Lu and X. Li, *Bioorg. Med. Chem.* 2010, **18**, 4475.

[4] P. Fang, M. Z. Li and H. Ge, *J. Am . Chem. Soc*. 2010, **132**, 11898.

[5] K. K. Park and J. J. Lee, *Tetrahedron* 2004, **60**, 2993.

[6] J. H. Adams, P. Gupta, M. S. Khan and J. R. Lewis, *J. Chem. Soc. Perkin Transactions 1: Organic and Bio-Organic Chemistry* 1977, **19**, 2173.

[7] M. C. Carter, D. G. Alber, R. C. Baxter, S. K. Bithell, J. Budworth, A. Chubb, G. S. Cockerill, V. C. L. Dowdell, E. A. Henderson, S. J. Keegan, R. D. Kelsey, M. J. Lockyer, J. N. Stables, L. J. Wilson and K. L. Powell, *J. Med. Chem.* 2006, **49**, 2311.

[8] K. G. Deshpande, K. S. Naragund and S. N. Kulkarni, *J. Indian Chem. Soc.* 1978, **55**, 813.

[9] D. Lednicer and D. E. Emmert, *J. Heterocycl. Chem.* 1971, **8**, 903.

[10] P. R. Angibaud, M. G. Venet, W. Filliers, R. Broeckx, Y. A. Ligny, P. Muller, V. S. Poncelet and D. W. End, *Eur. J. Org. Chem.* 2004, **3**, 479.

[11] W. Zahler and R. Huisgen, *Chem. Ber.* 1963, **96**, 765.

[12] Y. Wu, B. Li, F. Mao, X. Li and F. Y. Kwong, *Org. Lett.* 2011, **13**, 3258.