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# *t*-BuXPhos: a highly efficient ligand for Buchwald-Hartwig coupling in water

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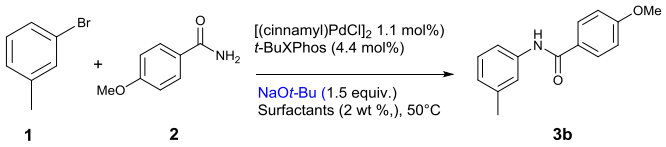
General information:

All reactions were carried out under a nitrogen atmosphere. Chemicals and solvents were purchased from Sigma-Aldrich and were used without further purification. Analytical TLC was performed using silica gel plates Merck 60F254 and plates were visualized by exposure to ultraviolet light. Compounds were purified using Armen spot flash chromatography on silica gel Merck 60 (particle size 0.040-0.063mm). Yields refer to isolated compounds, estimated to be >97% pure as determined by $^{1}$H NMR or HPLC. $^{1}$H and $^{13}$C NMR spectra were recorded on Bruker Avance Spectrometer operating at 300 MHz / 400 MHz and 100 MHz, respectively. All chemical shift values δ and coupling constants J are quoted in ppm and in Hz, respectively, multiplicity (s= singlet, d= doublet, t= triplet, m= multiplet, br. Broad). Analytical RP-HPLC-MS was performed using a LC-MSD 1200SL Agilent with a Thermo Hypersilgold® column (C18, 30 mm × 1 mm; 1.9 μm) using the following parameters : 1) The solvent system: A (acetonitrile) and B (0.05% TFA in $H_2O$); 2) A linear gradient: t = 0 min, 98%B; t = 5 min, 5% B; t = 6 min, 5%B; t = 7 min, 98%B; t = 9 min, 98%B; 3) Flow rate of 0.3 mL/min; 4) Column temperature: 50°C; 5) The ratio of products was determined by integration of spectra recorded at 210 nm or 254 nm; 6) Ionization mode : MM-ES+APCI. HPLC were performed using a Dionex UltiMate 300 using the following parameters: Flow rate of 0.5 mL/min, column temperature: 30°C, solvent system: A (MeOH) and B (0.05% TFA in $H_2O$), t= 0 min to 1 min: 50 to 60% of B then t= 1min to t= 10min: 60 to 100% of B and t= 10min to t= 15min: 100% of B. Infra-Red analyses were performed by FT-IR, on a Nicolet 380 ATR from Thermo and wavenumber were expressed in $cm^{-1}$.

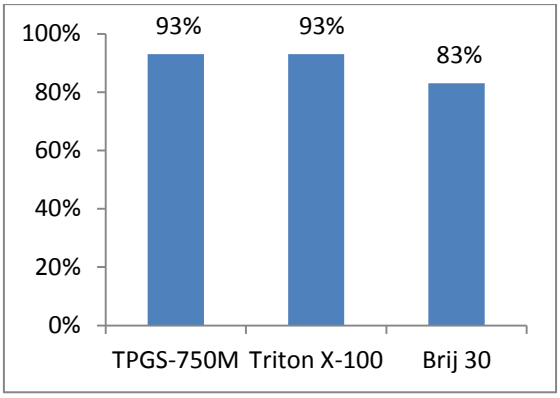
General procedure:

Amine (1.2 equiv.) and aryl or heteroaryl halide (1 equiv.) were added to an aqueous solution of TPGS-750-M (2 wt %, 1mL/mmol). The mixture was degassed by bubbling Argon in through (5 min). NaO*t*-Bu (97% purity, 1.5 equiv.), $[(cinnamyl)PdCl]_2$ (1.1%) and *t*-BuXPhos (4.4%) were added together to the previous solution. The mixture was stirred (at 1200 rpm) at 50°C (2-24h). Volatiles were evaporated and the crude residue was purified by chromatographic column on silica gel using ethyl acetate and *n*-heptane as solvent.

**Figure a:** Impact of some surfactants on the efficiency of the aryl amidation reaction:




1 2 3b




[a] Reaction conditions: [(cinnamyl)PdCl]$_2$ (1.1 mol%), Ligand (4.4 mol %), NaO*t*-Bu (1.5 equiv.), surfactant (2 wt %), 3-bromotoluene (1 equiv.), 4-methoxybenzamide (1.2 equiv.), 50°C, 16h. [b] Average yield of 2 runs, [c] Yields were determined by HPLC/UV using caffeine as an internal standard.

Impact of the variation of the Pd/L ratio for compounds **7a**, **7b**, **8** (Table 2) and for compound **16** (Table 4 )

| Compound | Pd (%mol) | L (%mol) | Yield (%) |
|---|---|---|---|
| **7a** | 2 | 4.4 | 50 |
| **7a** | 5 | 4.4 | 60 |
| **7a** | 5 | 10 | 75 |
| **7a** | 5 | 20 | 69 |
| **7b** | 2 | 4.4 | 54 |
| **7b** | 2 | 8 | 69 |
| **8** | 2 | 4.4 | 40 |
| **8** | 2 | 8 | 77 |
| **16** | 5 | 4.4 | 44 |
| **16** | 5 | 10 | 71 |

Table a: Variation of the Pd/L ratio

## *Atom economy*

$$\text{atom economy} = \frac{\text{molecular mass of desired product}}{\text{molecular mass of all reactants}} \times 100\%$$

| | New process MW | | Abbott process MW |
|---|---|---|---|
| 5-bromo-2-furanoic acid | 138 | 5-bromo-2-furanoic acid | 138 |
| | | iPrOH | 60 |
| HOBT | 152 | $Na_2CO_3$ | 106 |
| EDCI | 192 | 4-chlorobenzylboronic acid (2 eq) | 312 |
| DMF | 73 | $CH_2Cl_2$ | 84 |
| 4-chlorobenzylboronic acid | 156 | Oxalyl chloride | 127 |
| 1-bromo-3,5-dimethoxybenzene | 215 | $CH_2Cl_2$ | 84 |
| | | 3,5-dimethoxyaniline | 153 |
| | | $Et_3N$ | 101 |
| **Total** | **926** | **Total** | **1165** |
| **22** | **357** | **22** | **357** |
| Atom economy | 357/926=38% | Atom economy | 357/1165=30 % |

# *E factors*

**Abbott process** for 5 mmol

| Reactants/Reagents | Mass |
|---|---|
| 5-bromo-2-furanoic acid | 1.00g |
| 4-chlorophenylboronic acid | 0.977g g |
| $PdCl_2(PPh_3)_2$ | 0.112g |
| iPrOH | 37.0g |
| $Na_2CO_3$ | 2.7g |
| Ethyl acetate | 80g |
| Dichloromethane (1) | 50g |
| Oxalyl chloride | 0.760g |
| 3,5-dimethoxyaniline | 0.604g |
| $Et_3N$ | 1.2g |
| Dichloromethane (2) | 16g |
| | |
| **Total** | 190g |
| Compound **22** | 0.924g |
| Total waste for 1 g of 22 | 205g |
| E factor | =190/0.924 = **205** |

**Wagner *et al* process** for 10 mmol

| Reactants/Reagents | Mass |
|---|---|
| 5-bromo-2-furanoic acid | 2.0g |
| HOBT.NH3 | 2.3g |
| EDCI | 2.4g |
| DMF | 9.5g |
| | |
| Catalyst | 0.123 |
| $Et_3N$ | 2.8g |
| 4-chlorophenylboronic acid | 2.8g |
| | |
| *t*BuXPhos | 0.14g |
| $[(cinnamyl)PdCl]_2$ catalyst | 0.04g |
| 1-Bromo-3,5-dimethoxybenzene | 2.0g |
| NaO-*t*Bu | 1.1g |
| | |
| Total for 10 mmol of SM | 25.5 |
| Compound 22 | 1.7 |
| Total waste for 1 g of 22 | 15g |
| E factor | =25.5/1.7 = **15** |

*N*-(3-Methylphenyl)benzamide **3a**:[1]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), benzamide (145 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (95/5 to 8/2), yielded **3a** as white solid (204 mg, 97 %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.34 (s, 3H), 6.95 (d, *J* = 7.6 Hz, 1H), 7.23 (dd, *J* = 7.6 Hz, *J* = 7.9 Hz, 1H), 7.40 (d, *J* = 7.9 Hz, 1H), 7.43-7.54 (m, 4H), 7.83-7.85 (m, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.5, 117.3, 120.9, 125.4, 127.0 128.8, 128.9, 131.8, 135.1, 137.9, 139.0, 165.7.

*N*-(3-Methylphenyl)-4-methoxybenzamide **3b**:[2]

Following the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), *p*-methoxybenzamide (181 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (7/3 to 5/5), yielded **3b** as white solid (123 mg, 93 %). $^1$H NMR (400 MHz, $CDCl_3$) δ 2.34 (s, 3H), 3.87 (s, 3H), 6.95-6.97 (m, 3H), 7.25 (t, J = 7.7 Hz, 1H), 7.41 (d, J = 7.7 Hz, 1H), 7.51 (s, 1H), 7.80 (d, J = 8.9 Hz, 2H), 7.87 (br s, 1H); $^{13}$C NMR (101MHz, $CDCl_3$) δ 21.5, 55.4, 113.9, 117.2, 120.8, 125.1, 127.2, 128.8, 128.9, 138.0, 138.9, 162.4, 165.2.

**Scale up :**
Following the general procedure, using [(cinnamyl)PdCl]$_2$ (48.7 mg, 0.011 mmol), *t*-BuXPhos (146 mg, 0.044 mmol), 3-bromotoluene (0.95 mL, 1.0 mmol), *p*-methoxybenzamide (1.42 g, 1.2 mmol) and NaO*t*-Bu (1.13 g, 1.5 mmol) in aqueous TPGS-750M (2%, 5.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (7/3 to 5/5), yielded **3b** as white solid (1.89 g, 99 %).

*N*-(3-Methylphenyl)-4-(trifluoromethyl)benzamide **3c**:

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (6.2 mg, 0.012 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), 4-(trifluoromethyl)benzamide (227 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (9/1 to 5/5), yielded **3c** as white solid (206 mg, 74 %). $^1$H NMR (400 MHz, $CDCl_3$) δ 2.35 (s, 3H), 6.99 (d, *J* = 7.9 Hz, 1H), 7.25 (t, *J* = 7.9 Hz, 1H), 7.41 (d, *J* = 7.9 Hz, 1H), 7.49 (s, 1H), 7.70 (d, *J* = 8.2 Hz, 2H), 7.94 (d, *J* = 8.2 Hz, 2H), 8.00 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.4, 117.6, 121.1, 122.3, 124.9, 125.7, 125.9, 127.5, 129.0, 133.3, 133.6, 137.4, 138.3, 139.1, 164.5; $^{19}$F NMR (398 MHz, $CDCl_3$) δ -62.98.

3'-Methylacetanilide **3d**:[3]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), acetamide (295 mg, 5.0 mmol) and NaO*t*-Bu

[1] C. K.Lee, J. S. Yu, Y. R. Ji, *J. Heterocycl. Chem.*, 2002, **39**, 1219.
[2] A. Correa, S. Elmore, C. Bolm, *Chem. Eur. J.*, 2008, **14**, 3527.

[3] J. E. Taylor, M. D. Jones, J. M. J. Williams, S. D. Bull, *J. Org. Chem.*, 2012, **77**, 2808.

(144 mg, 1.5 mmol) in aqueous TPGS-750-M (5%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (1/1), yielded **3d** as an oil (81 mg, 54%). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2,15 (s, 3 H), 2,31 (s, 3H), 6.92 (d, *J* = 7.5 Hz, 1H), 7.18 (t, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 8.3 Hz ,3H), 7.36 (s, 1H), 7.78-7.88 (br s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.5, 24.5, 117.2, 120.7, 125.1, 128.7, 137.9, 138.8, 168.8.

3,3-Dimethyl-1-(3-methylphenyl)urea **3f**:[4]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (6.2 mg, 0.012 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), 1,1-dimethylurea (106 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (8/2 to 5/5), yielded **3f** as white solid (125 mg, 70 %). %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.24 (s, 3H), 2.94 (s, 6H), 6.25 (s, 1H), 6.77 (d, J = 6.3 Hz, 1H), 7.05-7.10 (m, 2H), 7.19 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.8, 36.7, 117.1, 120.8, 124.1, 128.9, 139.0, 139.4, 156.1.

*N*-(2-Methoxyphenyl)benzamide **4**:[5]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 2-bromoanisole (125 mg, 1.0 mmol), benzamide (145 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and cyclohexane/ethyl acetate (8/2 to 5/5), yielded **4** as a yellow oil (193 mg, 85%). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 3,96 (s, 3H), 6.94-7.12 (m, 3H), 7.50-7.58 (m, 3H), 7.91-7.94 (m, 2H), 8.55-8.58 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 56.0, 110.1, 121.4, 124.1, 127.3, 128.0, 129.0, 131.9, 135.6, 148.3, 165.5.

*N*-(4-Chlorophenyl)benzamide **5**:[6]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 1-bromo-4-chlorobenzene (190 mg, 1.0 mmol), benzamide (145 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and cyclohexane/ethyl acetate (8/2 to 5/5), yielded **4** as a yellow oil (206 mg, 89%). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.5 Hz, 2H), 7.44-7.61 (m, 5H), 7.84 (d, *J* = 8.5 Hz, 2H), 7.92-8.40 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 121.5, 127.2, 128.8, 129.0, 129.4, 132.2, 134.8, 136.7, 165.9.

*N*-(3,5-Dimethoxyphenyl)furan-2-carboxamide **6**:

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 1-bromo-3,5-dimethoxybenzene (224 mg, 1.0 mmol), furan-2-carboxamide (133

[4] C. E. Houlden, C. D. Bailey, J. Gair Ford, M. R. Gagne, G. C. Lloyd-Jones, K. I. Booker-Milburn, *J. Am. Chem. Soc.*, 2008, **130**, 10066.
[5] C. E. Anderson, Y. Donde, C. J. Douglas, L. E. Overman, *J. Org. Chem.*, 2005, **70**, 648.
[6] L. Zhang, S. Su, H. Wu, S. Wang, *Tetrahedron*, 2009, **65**, 10022.

mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (2/1), yielded **6** as an oil (219 mg, 89%). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 3.79 (s, 6 H), 6.26 (t, *J* = 2.3 Hz, 1H), 6.55 (dd, *J* = 3.4, 1.8 Hz, 1H), 6.88 (d, *J* = 2.3 Hz, 2H), 7.22 (dd, *J* = 3.5, 0.8 Hz, 1H), 7.50 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.95-8.04 (br s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 55.4, 97.1, 98.1, 112.7, 115.3, 139.1, 144.2, 147.8, 156.1, 161.1. HRMS ($M+H^+$) 248.0919 (calcd for $C_{13}H_{13}NO_4H^+$ 248.0917).

*N*-(Pyridine-3-yl)benzamide **7a**:[7] (see Table 5)

Benzamide (145 mg, 1.2 mmol) and TPGS-750-M (20 mg) were heated together until getting an homogenous solution. $H_2O$ (1 mL), 3-bromopyridine (96 µL, 1.0 mmol), NaO*t*-Bu (144 mg, 1.5 mmol), $[(cinnamyl)PdCl]_2$ (25.9 mg, 0.05 mmol), *t*-BuXPhos (42.5mg, 0.10 mmol) were added. The reaction mixture was stirred at 50°C (16h) and directly purified using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (8/2 to 2/8), yielded **6** as yellow solid ( 147.8 mg, 75 %). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.16-7.20 (m, 1H), 7.34 (t, *J* = 7.5 Hz, 2H), 7.44 (td, *J* = 1.5 Hz, *J* = 7.5 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 2H), 8.19-8.22 (m, 2H), 8.61 (d, *J* = 2.4 Hz, 1H), 8.89 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 123.8, 127.3, 128.0, 128.7, 132.1, 134.2, 135.3, 141.6, 145.1, 166.7.

*N*-(Pyridin-3-yl)piperidine-1-carboxamide **7b**:[8] (see Table 5)

Employing the general procedure, using $[(cinnamyl)PdCl]_2$ (10.4 mg, 0.02 mmol), *t*-BuXphos (35.0 mg, 0.08 mmol), 3-bromopyridine (96 µL, 1.0 mmol), piperidine-1-carboxamide (154 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and methanol/dichloromethane (5/95 to 1/9), yielded **7b** as white solid (141 mg, 69 %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 1.46 – 1.59 (m, 6H), 3.38 – 3.41 (m, 4H), 7.13-7.17 (m, 2H), 7.92 (ddd, *J* = 1.4 Hz, *J* = 2.6 Hz, *J* = 8.3 Hz, 1H), 8.13 (dd, *J*= 1.4 Hz, *J* = 4.3 Hz, 1H), 8.39 (d, *J* = 2.6 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 24.3, 25.7, 45.3, 123.5, 127.6, 136.8, 141.2, 143.4, 154.9; HRMS ($M+H^+$) 206.1293 (calcd for $C_{11}H_{15}N_3OH^+$ 206.1288).

*t*-Butyl *N*-(pyridine-5-yl)carbamate **8**:[8] (see Table 5)

Employing the general procedure, using $[(cinnamyl)PdCl]_2$ (10.4 mg, 0.02 mmol), *t*-BuXphos (35.0 mg, 0.08 mmol), 5-bromopyrimidine (159 mg, 1.0 mmol), *t*-butyl carbamate (141 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (7/3), yielded **8** as white solid ( 150 mg, 77 %) $^1$H NMR (400 MHz, $CDCl_3$) δ 1.73 (s, 9H), 7.14 (s, 1H), 9.04 (s, 2H), 9.10 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 28.2, 134.0, 146.5, 152.2, 153.1.

*N*-Benzyl-3-methylaniline **9a**:[9]

Employing the general procedure, using $[(cinnamyl)PdCl]_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), benzylamine (131 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using

[7] C. A. Faler, M. M. Joullie, *Tetrahedron*, 2006, **47**, 7229.
[8] N. A. Isley, S. Dobarco, B. H. Lipshutz, *Green Chem.*, 2014, **16**, 1480.
[9] X. Yu, C. Liu, L. Jiang, Q. Wu, *Org. Lett.*, 2011, **13**, 6184.

column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (9/1), yielded **9a** as a colorless oil (186 mg, 94 %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.30 (s, 3H), 3.90 – 4.06 (br s, 1H), 4.34 (s, 2H), 6.46 – 6.59 (m, 3H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.27 – 7.41 (m, 5H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.7, 48.4, 110.0, 113.6, 118.6, 127.2, 127.6, 128.6, 129.2, 139.1, 148.3.

3-Methyl-*N*-(3-phenylpropyl)aniline **9b**:

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), benzenepropanamine (171 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (95/5), yielded **9b** as an oil (215 mg, 95%). $^1$H NMR (400 MHz, $CDCl_3$) δ 1.92-2.02 (m, 2H), 2.29 (s, 3H), 2.76 (t, *J* = 7.8 Hz, 2H), 3.17 (t, *J* = 7.2 Hz, 2H), 3.52-3.62 (br s, 1H), 6.41- 6.43 (m, 2H), 6.54 (d, *J* = 7.5 Hz, 1H), 7.08 (dd, *J* = 8.7, 7.5 Hz, 1H), 7.20- 7.23 (m, 3H), 7.25- 7.34 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.7, 31.1, 33.4, 43.4, 109.9, 113.5, 118.2, 125.9, 128.4, 129.1, 139.0, 141.7, 148.4; HRMS ($M+H^+$) 226.1597 (calcd for $C_{16}H_{19}NH^+$ 226.1590).

*N*-Butyl-3-methylaniline **9c**:[10]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121µL, 1.0 mmol), butylamine (494µL, 5.0 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (95/5), yielded **9c** as an oil (156 mg, 96%). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 1.10 (t, *J* = 7.2 Hz, 3H), 1.57 (sext, *J* = 7.3 Hz, 2H), 1.71 (quint, *J* = 7.3 Hz, 2H), 2.42 (s, 3H), 3.22 (t, *J* = 7.0 Hz, 2H), 3.60-3.68 (br s, 1 H), 6.53 – 6.56 (m, 2H), 6.65 (d, *J* = 7.5 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.0, 20.4, 21.7, 31.8, 43.8, 109.9, 113.5, 118.1, 129.1, 139.0, 148.7.

*N*-Cyclohexyl-3-methylaniline **9d**:

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), cyclohexanamine (137 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (95/5), yielded **9d** as an oil (136 mg, 72%). 1H NMR (400 MHz, $CDCl_3$) δ 1.14 – 1.46 (m, 6H), 1.75 – 1.82 (m, 2H), 2.05 – 2.10 (m, 2H), 2.29 (s, 3H), 3.23 – 3.31 (m, 1H), 3.42-3.51 ( br s, 1H), 6.42– 6.44 (m, 2H), 6.50 – 6.53 (d, *J* = 7.5Hz, 1H), 7.04 – 7.10 (dd, *J* = 7.5 Hz, 1H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 21.7, 25.1, 26.0, 33.6, 51.7, 110.3, 113.9, 117.8, 129.2, 139.0, 147.5. HRMS ($M+H^+$) 190.1595 (calcd for $C_{13}H_{19}NH^+$ 190.1590).

(R)-3-Methyl-*N*-(1-phenylethyl)aniline **9e**:[11]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121µL, 1.0 mmol), (R)-(+)-1-phenylethylamine (137µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and cyclohexane/ethyl acetate (9/1 to 7/3), yielded **9e** as an orange oil (150 mg, 71%). Chemical and spectral properties were in accordance with the

[10] T. Kubo, C. Katoh, K. Yamada, K. Okano, H. Tokuyama, T. Fukuyama, *Tetrahedron*, 2008, **64**, 11230.
[11] S. F. Zhu, J. B. Xia, Y. Z. Zhang, S. Li, Q. L. Zhou, *J. Am. Chem. Soc.*, 2006, **126**, 12886.

literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 1.53 (d, *J* = 6.8 Hz, 3H), 2.23 (m, 3H), 4.50 (q, *J* = 6.8 Hz, 1H), 6.33 (d, *J* = 6.4 Hz, 1H) 6.39 (s, 1H), 6.50 (d, *J* = 7.2 Hz, 1H), 7.00 (t, *J* = 7.8 Hz, 1H), 7.22-7.28 (m, 1H), 7.31-7.41 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.8, 25.1, 53.7, 110.5, 114.4, 118.5, 126.1, 127.0, 128.8, 129.2, 139.0, 143.7, 147.5.

*N*-Butyl-*N*,3-dimethylaniline **10a**:[12]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (6.2 mg, 0.012 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), *N*-methyl-1-butanamine (148 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (9/1), yielded **10a** as a colorless oil (138 mg, 78 %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 0.91 – 1.00 (t, *J* = 7.2 Hz, 3H), 1.31 – 1.43 (m, 2H), 1.53 – 1.63 (m, 2H), 2.34 (s, 3H), 2.94 (s, 3H), 3.29 – 3.34 (t, *J* = 7.5 Hz, 2H), 6.52 – 6.56 (m, 3H), 7.11 – 7.16 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.0, 20.4, 22.0, 29.0, 38.3, 52.6, 109.4, 112.9, 116.8, 129.0, 138.7, 149.6.

1-Benzyl-4-(*m*-tolyl)piperazine **10b**:[12]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (6.2 mg, 0.012 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), *N*-benzylpiperazine (208 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (9/1 to 7/3), yielded **10b** as a orange oil (195 mg, 73% yield). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.34 (s, 3H), 2.63 (t, *J* = 5.0 Hz, 4H), 3.22 (t, *J* = 5.0 Hz, 4H), 3.60 (s, 2H), 6.69–6.77 (m, 3H), 7.17 (d, *J* = 7.8 Hz, 1H), 7.28-7.40 (m, 5H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.8, 49.3, 53.2, 63.1, 113.6, 116.9, 121.2, 127.1, 128.3, 129.0, 129.2, 138.5, 138.7, 151.5.

6-Methyl-2-(3-phenylpropylamine)pyridine **11**:

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 2-chloro-6-methylpyridine (110 µL, 1.0 mmol), benzenepropanamine (171µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and cyclohexane/ethyl acetate (8/2 to 6/4), yielded **11** as a brown oil (195 mg, 92%). $^1$H NMR (400 MHz, $CDCl_3$) δ 1.97 (quint., *J* = 7.6 Hz, 2H), 2.39 (s, 3 H), 2.76 (t, *J* = 7.6 Hz, 2H), 3.25 (t, *J* = 6.8 Hz, 2H), 4.55-4.62 (br s, 1H), 6.17 (d, *J* = 7.6 Hz, 1H), 6.46 (d, *J* = 7.6 Hz, 1H), 7.20-7.24 (m, 3H), 7.28-7.37 (m, 3H), $^{13}$C NMR (100 MHz, $CDCl_3$) δ 31.3, 33.4, 42.1, 102.5, 112.4, 120.2, 126.1, 128.6, 138.2, 141.7, 157.1, 158.7.

6-Chloro-*N*-(3-phenylpropyl)pyridin-3-amine **12**:

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (11.4 mg, 0.022 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 5-bromo-2-chloropyridine (192 mg, 1.0 mmol), benzenepropanamine (171 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (8/2), yielded **12** as a white solid (199 mg, 81%). $^1$H NMR (400 MHz, $CDCl_3$) δ 1.93-2.01 (m, 2H), 2.74 (t, *J* = 7.5 Hz, 2H), 3.14 (t, *J* = 7.0 Hz, 2H), 6.80 (dd, *J* = 8.5, 3.0 Hz, 1H), 7.07 (dd, *J* = 8.6, 0.6 Hz, 1H), 7.18-7.25 (m, 3H), 7.28-7.34 (m, 2H), 7.72 (d, *J* = 2.76 Hz, 1H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ 30.6, 33.2, 43.2,

[12] C. Salomé, P. Wagner, M. Bollenbach, F. Bihel, J.-J. Bourguignon, M. Schmitt, *Tetrahedron*, 2014, **70**, 3413.

76.7, 77.3, 122.0, 124.0, 126.1, 128.3, 128.5, 134.4, 138.7, 141.1, 143.3. HRMS ($M+H^+$) 247.0995 (calcd for $C_{14}H_{15}ClN_2H^+$ 247.0996).

6-Phenyl-*N*-(3-phenylpropyl)pyridazin-3-amine **13**:

Employing the general procedure with a 0.5M substrate concentration, using $[(cinnamyl)PdCl]_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-chloro-6-phenylpyridazine (190 mg, 1.0 mmol), benzenepropanamine (171 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (5%, 2.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (7/3), yielded **13** as an off-white solid (237 mg, 82%). $^1$H NMR (400 MHz, $CDCl_3$) δ 2.02 (q, *J* = 7.4 Hz, 2H), 2.75 (t, *J* = 7.4 Hz, 2H), 3.48 (q, *J* = 6.8 Hz, 2H), 4.68-4.76 (br s, 1H), 6.62 (d, *J* = 9.3 Hz, 1H), 7.17-7.20 (m, 3H), 7.26- 7.30 (m, 2H), 7.35-7.39 (t, *J* = 7.3 Hz, 1H), 7.42-7.46 (m, 2H), 7.56=7 (d, *J* = 9.3 Hz, 1H), 7.95 (d, *J* = 7.3 Hz, 2H), $^{13}$C NMR (101 MHz, $CDCl_3$) δ 31.0, 33.3, 41.6, 113.5, 125.4, 125.9, 126.0, 128.4, 128.5, 128.6, 128.8, 137.0, 141.5, 151.3, 158.3. HRMS ($M+H^+$) 290.1662 (calcd for $C_{19}H_{19}N_3H^+$ 290.1652).

*N*-(*m*-Tolyl)pyridine-2-amine **14**:[12]

Employing the general procedure, using $[(cinnamyl)PdCl]_2$ (45.8 mg, 0.05 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), 2-pyridinamine (112 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (5%, 2.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (1/0 to 5/5), yielded **14** as a brown oil (163 mg, 89 %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.27 (s, 3H), 6.45 – 6.55 (br s, 1H), 6.64 (t, *J* = 6.2 Hz, t), 6.80 - 6.82 (m, 2H), 7.04 – 7.06 (m, 2H), 7.13 (d, *J* = 8.0 Hz, 1H), 7.38 – 7.43 (m, 1H), 8.12 (d, *J* = 4.4 Hz, 1H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 21.5, 108.2, 115.0, 117.5, 121.1, 123.7, 129.1, 137.6, 139.2, 140.4, 148.5, 155.1.

*N*-(3-Methylphenyl)pyrimidin-2-amine **15**:[12]

Employing the general procedure, using $[(cinnamyl)PdCl]_2$ (45.8 mg, 0.05 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), 2-aminopyrimidine (115 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (5%, 2.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (3/1), yielded **15** as a white solid (151 mg, 82 %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.39 (s, 3H), 6.68 – 6.72 (t, J = 4.8 Hz, 1H), 6.89 – 6.92 (d, J = 7.5 Hz, 1H), 7.23 – 7.29 (t, J = 7.8 Hz, 1H), 7.46 – 7.48 (m, 2H), 8.29 (s, 1H), 8.43 – 8.45 (d, J = 4.8 Hz, 2H); $^{13}$C NMR (101 MHz, $CDCl_3$) δ 21.7, 112.3, 117.1, 120.6, 123.7, 128.8, 138.7, 139.5, 158.0, 160.5.

*N*-(3-methylphenyl)pyridazin-3-amine **16**: (see Table 5)

Employing the general procedure, using $[(cinnamyl)PdCl]_2$ (45.8 mg, 0.05 mmol), *t*-BuXphos (43.8 mg, 0.1 mmol), 3-bromotoluene (121 µL, 1.0 mmol), pyridazin-3-amine (114mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and ethyl acetate/dichloromethane (1/9), yielded **16** as a pale yellow solid (131 mg, 71%). $^1$H NMR (400 MHz, $CDCl_3$) δ 2.34 (s, 3H), 6.93(d, *J* = 7.4 Hz, 1H), 7.09-7.13 (m, 4H), 7.20-7.25 (m, 2H), 8.64-8.65 (d, *J* = 4.0 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.5, 113.1, 118.4, 122.1, 125.0, 127.6, 129.4, 138.9, 139.6, 144.8, 158.4. HRMS ($M+H^+$) 186.1027 (calcd for $C_{11}H_{11}N_3H^+$ 186.1026).

1-(*m*-Tolyl)-*1H*-indole **17**:[13]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (5.7 mg, 0.011 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), indole (115 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and ethyl acetate/cyclohexane (0/1 to 2/8), yielded **17** as a colorless oil (180 mg, 87 %). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.35 (s, 3H), 6.58 (t, J = 3.6 Hz, 1H), 7.07 – 7.32 (m, 7H), 7.46 – 7.50 (m, 1H), 7.58 – 7.61 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.5, 103.6, 110.6, 120.3, 121.1, 121.5, 122.3, 125.1, 127.3, 128.0, 129.3, 129.5, 135.9, 139.7, 139.8.

1-(3-Methylphenyl)-1H-indazole **18**:[12]

Employing the general procedure, using $Pd_2(dba)_3$ (45.8 mg, 0.05 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 3-bromotoluene (121 µL, 1.0 mmol), indazole (142 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (1/0 to 9/1), yielded **18** as a colorless oil (179 mg, 86% yield). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, $CDCl_3$) δ 2.48 (s, 3H), 7.18-7.27 (m, 2H), 7.41–7.47 (m, 2H), 7.53-7.57 (m, 2H), 7.76-7.78 (d, *J* = 8.6 Hz, 1H), 7.80-7.83 (d, *J* = 8.0 Hz, 1H), 8.2 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 21.5, 110.5, 119.7, 121.3, 121.4, 123.6, 125.3, 127.1, 127.5, 129.2, 135.2, 138.8, 139.6, 140.1.

*N*-Phenylthiophen-3-amine **19**:[14]

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (11.4 mg, 0.022 mmol), *t*-BuXphos (18.7 mg, 0.044 mmol), 3-bromothiophene (94 µg, 1.0 mmol), aniline (110 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750-M (2%, 1.0 mL), followed by purification using column chromatography ($SiO_2$) and cyclohexane/ethyl acetate (8/2), yielded **19** as a black oil (166 mg, 95%). $^1$H NMR (400 MHz, $CDCl_3$) δ 5.68-5.75 (br s, 1H), 6.76-6.79 (m, 1H), 6.90 (t, *J* = 7.2 Hz, 1H), 6.95 (dd, *J* = 5.2, 1.6 Hz, 1H), 7.01 (d, *J* = 8.0 Hz, 2H), 7.26-7.30 (m, 3H).$^{13}$C NMR (100 MHz, $CDCl_3$) δ 106.6, 115.8, 119.9, 122.9, 125.2, 129.4, 141.5, 144.7.

Methyl-4-[(4-methoxyphenyl)amino]benzoate **20**:

Employing the general procedure, using [(cinnamyl)PdCl]$_2$ (6.2 mg, 0.012 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), 4-bromoanisole (125 µL, 1.0 mmol), methyl-4-aminobenzoate (181.4 mg, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL) at 30°C (3h), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (8/2 to 7/3), yielded **20** as a orange oil (222 mg, 86% yield). $^1$H NMR (400 MHz, $CDCl_3$) δ 3.62 (s, 3H), 3.86 (s, 3H), 5.87 (s, 1H), 6.81 (d, *J* = 8.5 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 7.13 (d, *J* = 8.5 Hz, 2H), 7.87 (d, *J* = 8.8 Hz, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 51.7, 55.6, 113.2, 114.8, 120.0, 124.4, 131.5, 167.0; HRMS ($M+H^+$) 258.1125 (calcd for $C_{15}H_{15}NO_3H^+$ 258.1125).

Ethyl-3-[methyl(phenyl)amino]benzoate **21**:

[13] Y. Teo, *Adv. Synth. Catal.*, 2009, **351**, 720.
[14] M. W. Hooper, M. Utsunomiya, J. F. Hartwig, *J. Org. Chem.*, 2003, **68**, 2861.

Employing the general procedure, using $[(cinnamyl)PdCl]_2$ (6.2 mg, 0.012 mmol), *t*-BuXPhos (18.7 mg, 0.044 mmol), ethyl-3-bromobenzoate (160 µL, 1.0 mmol), *N*-methylaniline (130 µL, 1.2 mmol) and NaO*t*-Bu (144 mg, 1.5 mmol) in aqueous TPGS-750M (2%, 1.0 mL) at 30°C (3h), followed by purification using column chromatography ($SiO_2$) and *n*-heptane/ethyl acetate (9/1), yielded **21** as a orange oil (247 mg, 97% yield). $^1$H NMR (400 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.2 Hz, 3H), 3.27 (s, 3H), 4.28 (q, *J* = 7.2 Hz, 2H), 6.95 (t, *J* = 7.7 Hz, 1H), 6.99 (d, *J* = 7.7 Hz, 2H), 7.05 (dd, *J* = 2.0 Hz, *J* = 7.7 Hz, 1H), 7.17-7.25 (m, 3H), 7.50 (d, *J* = 7.7 Hz, 1H), 7.58 (t, *J* = 2.0 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.3, 40.3, 60.9, 119.5, 121.5, 121.9, 122.5, 123.5, 129.0, 129.4, 131.5, 148.6, 149.1, 166.8; HRMS ($M+H^+$) 256.1338 (calcd for C16H17NO2H+ 256.1332).

5-Bromofuran-2-carboxamide **24**:

5-Bromofuroic acid (2.0 g, 10.4 mmol) and $HOBt.NH_3$ (2.4 g, 15.6 mmol)[15] were solubilized in DMF (10 mL) followed by the addition of EDCI (2.4 g, 12.4 mmol). The resulting solution was stirred at r.t. for 2h. Solvent was evaporated and the obtained residue was diluted in EtOAc/$H_2O$. The organic layer was washed with a saturated aqueous solution of $NaHCO_3$, an aqueous solution of HCl 1N and brine. The organic layer was dried ($Na_2SO_4$) and concentrated under vacuum, yielding **24** as a white solid (1.88 g, 95%). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (400 MHz, DMSO-$d_6$) δ 6.73 (d, *J* = 3.5 Hz, 1 H), 7.13 (d, *J* = 3.5 Hz, 1 H), 7.41-7.49 (br s, 1 H), 7.48-7.87 (br s, 1 H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 113.9, 116.0, 124.4, 149.9, 158.2.

5-(4-Chlorophenyl)furan-2-carboxamide **25**:[16]

5-Bromofuran-2-carboxamide **24** (189 mg, 2 mmol) was added to an aqueous solution of TPGS-750-M (2%, 2 mL). The mixture was degassed by bubbling Argon through it (5 min). 4-Chlorophenylboronic (625 mg, 4 mmol), $Et_3N$ (834 µL, 6 mmol), and $PdCl_2$(dtbpf) (26mg, 0.04 mmol) were added together to the previous solution. The mixture was stirred (at 1200 rpm) at 50°C (overnight). The reaction mixture was extracted twice with EtOAc. The combined organic layers were dried ($Na_2SO_4$) and concentrated under vacuum. The compound **25** was precipitated in a mixture of *n*-heptane/ethyl acetate (1/1) then filtered and washed with *n*-heptane. 5-(4-Chlorophenyl)furan-2-carboxamide (**25**) was obtained white solid (351 mg, 79%). Chemical and spectral properties were in accordance with the literature. $^1$H NMR (300 MHz, DMSO-$d_6$) δ 7.13-7.17 (m, 2H), 7.42-7.52 (br s, 1H), 7.53 (d, *J* = 8.5 Hz, 2H), 7.93 (d, *J* = 8.5 Hz, 2H), 7.80-7.85 (br s, 1H), $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 38.9, 39.1, 39.3, 39.7, 39.9, 40.1, 108.2, 115.7, 126.0, 128.3, 128.9, 132.9, 147.5, 153.2, 159.1.

5-(4-Chlorophenyl)-*N*-(3,5-dimethoxyphenyl)furan-2-carboxamide **22**:[17]

1-Bromo-3,5-dimethoxybenzene (67 mg, 0.3 mmol) and TPGS-750-M (50 mg) were heated together at 50°C until an homogenous solution. $H_2O$ (0.5 mL), 5-(4-chlorophenyl)furan-2-carboxamide **25** (55 mg, 0.25 mmol), NaO*t*-Bu (37 mg, 0.38 mmol), $[(cinnamyl)PdCl]_2$ (1.4 mg, 0.027 mmol) and *t*-BuXphos (4.7 mg, 0.011 mmol) were added. The reaction mixture was stirred at 50°C (16h) and

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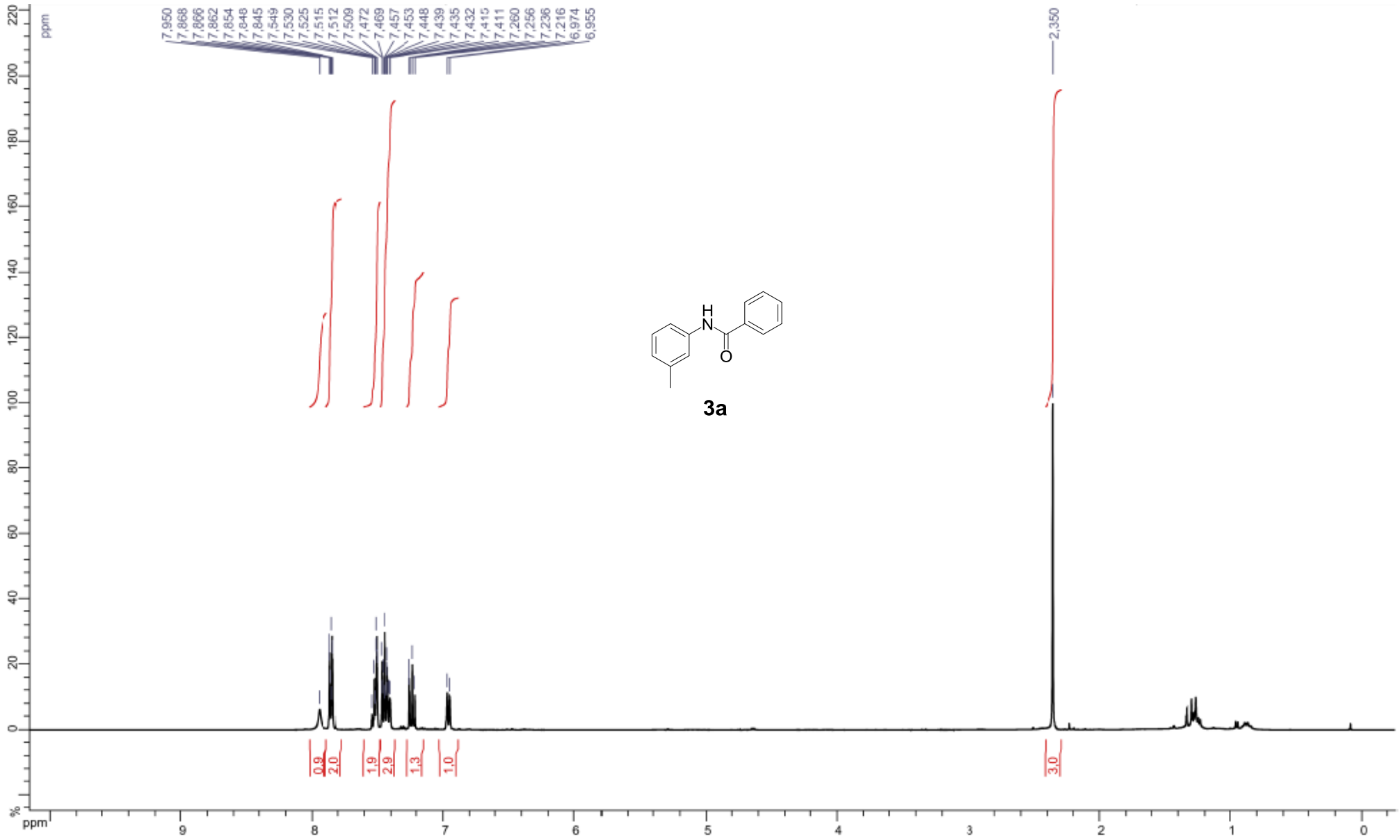
[16] A. Krutošíková and J. Kováč, *Collect. Czech. Chem. Commun.,* 1976, **41**, 2577.

[17] M. E. Kort, I. Drizin, R. J. Gregg, M. J. C. Scanio, L. Shi, M. F. Gross, R. N. Atkinson, M. S. Johnson, G. J. Pacofsky, J. B. Thomas, W. A. Carroll, M. J. Krambis, D. Liu, C.-C. Shieh, X. Zhang, G. Hernandez, J. P. Mikusa, C. Zhong, S. Joshi, P. Honore, R. Roeloffs, K. C. Marsh, B. P. Murray, J. Liu, S. Werness, C. R. Faltynek, D. S. Krafte, M. F. Jarvis, M. L. Chapman, B. E. Marron, *J. Med. Chem*., 2008, **51**, 407.

directly purified by reverse phase column chromatography ($C_{18}$) using AcCN/$H_2O$ (0.05% TFA) as eluent, yielded **22** as a white solid (57 mg, 64%). Chemical and spectral properties were in accordance with the literature. $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 3.75 (s, 6H), 6.29 (t, *J* = 2.3 Hz, 1H), 7.06 (d, *J* = 2.3 Hz, 2H), 7.23 (d, *J* = 3.5 Hz, 1H), 7.40 (d, *J* = 3.5 Hz, 1H), 7.55-7.59 (d, *J* = 8.5 Hz, 2H), 7.97-8.03 (d, *J* = 8.5 Hz, 2H), 10.11 (s, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 55.1, 95.9, 98.8, 108.6, 117.1, 126.3, 128.1, 129.0, 133.2, 140.0, 146.8, 154.1, 155.9, 160.4.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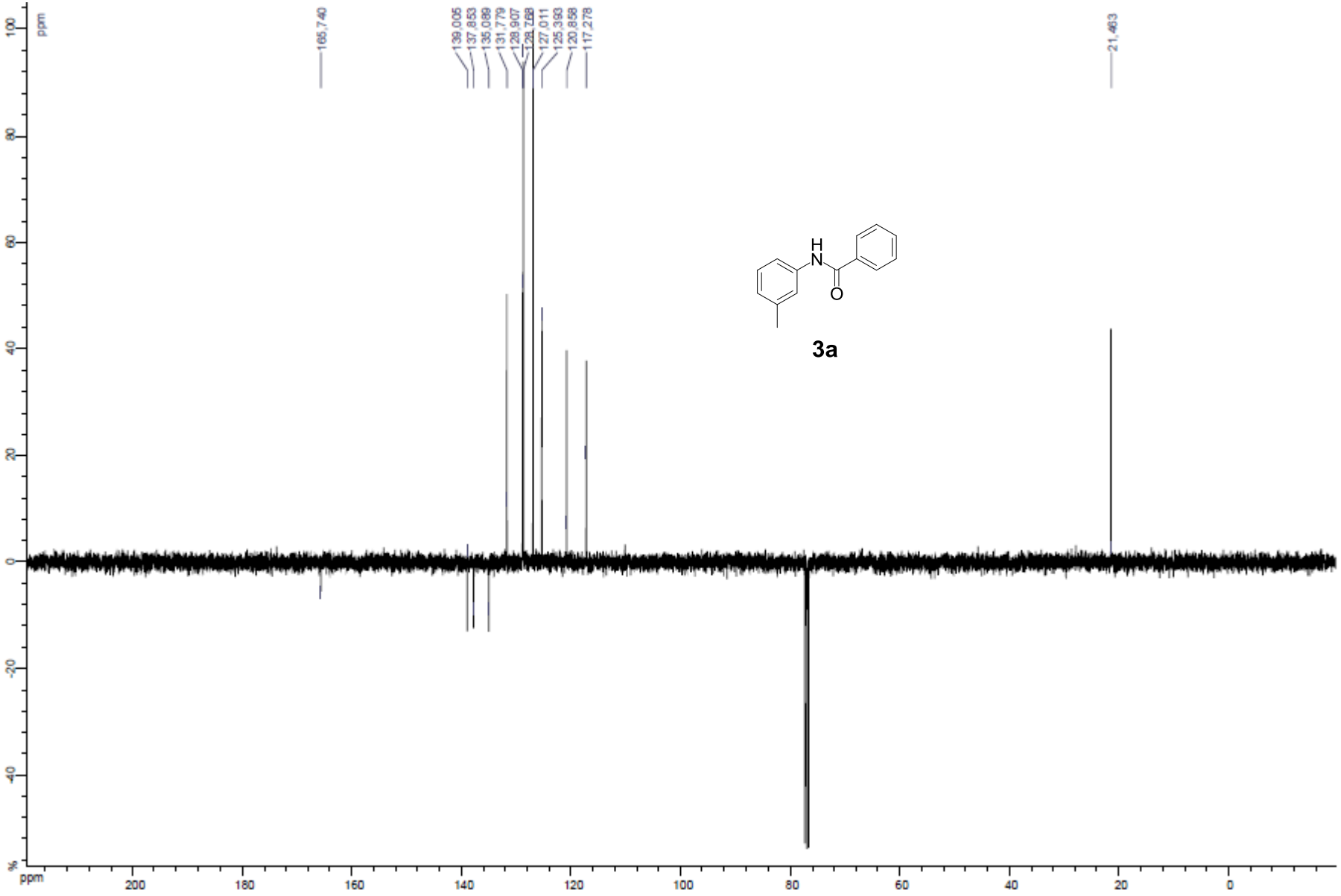
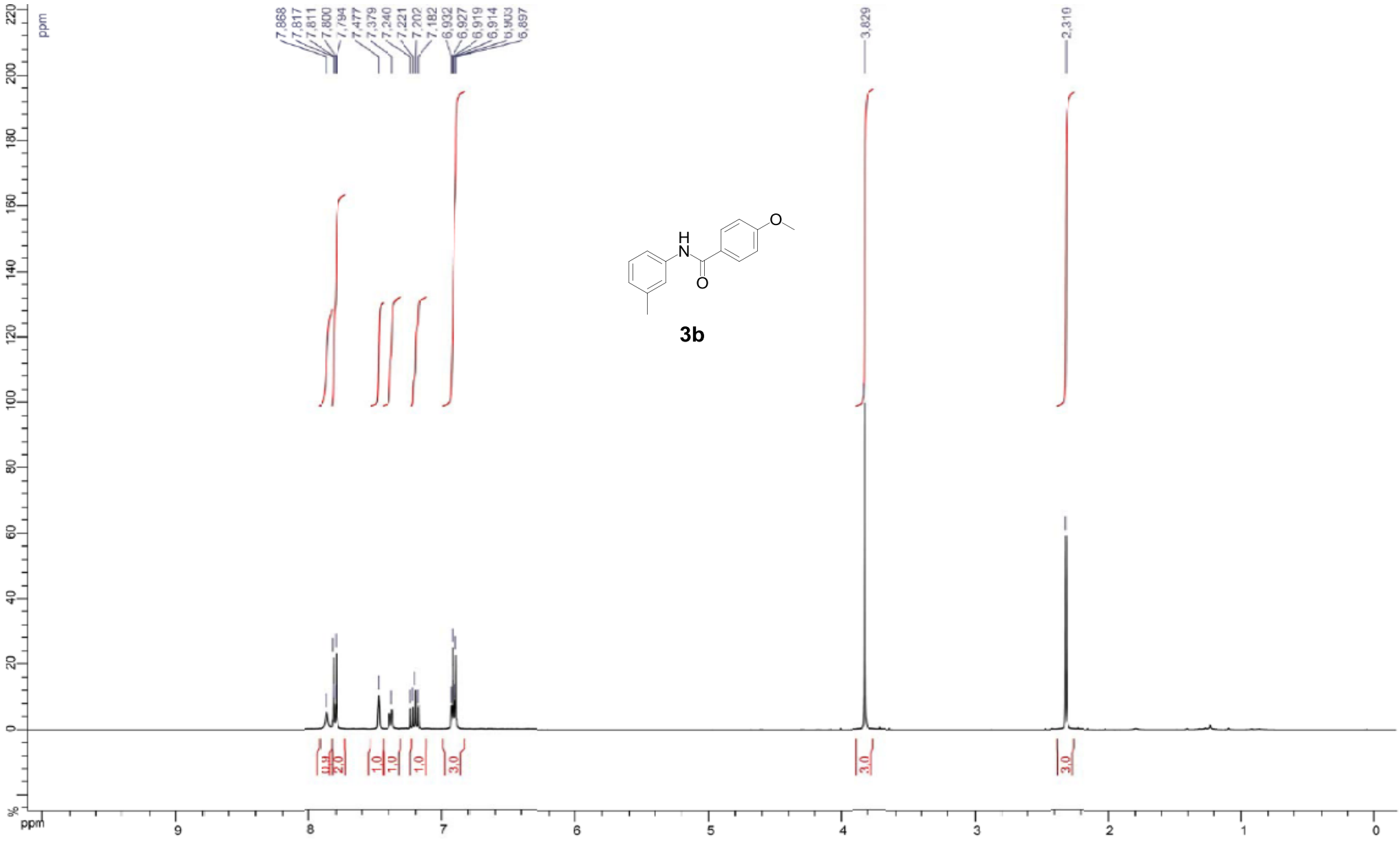
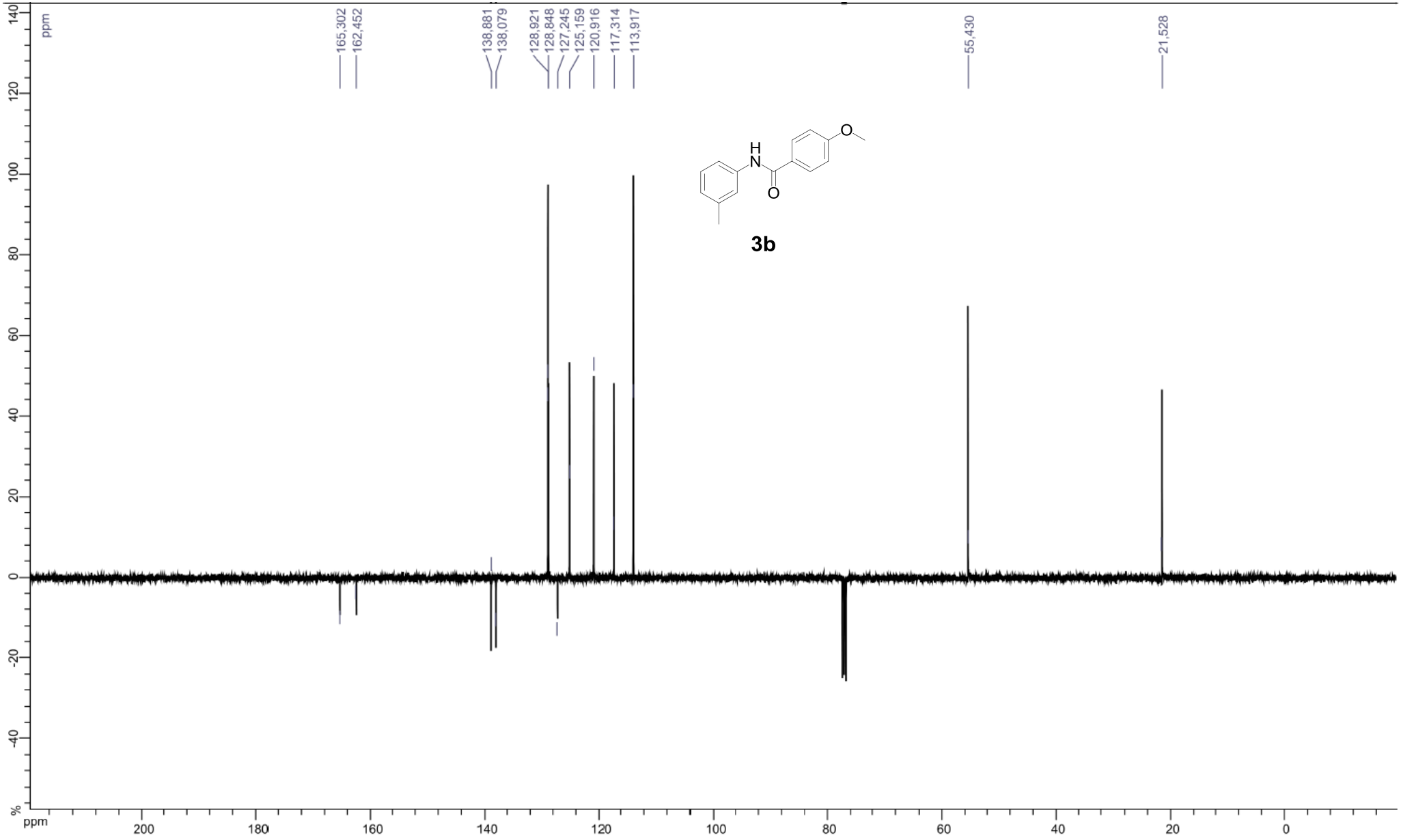
7,868
7,866
7,862
7,854
7,848
7,845
7,549
7,530
7,525
7,515
7,512
7,509
7,472
7,469
7,457
7,453
7,448
7,439
7,435
7,432
7,415
7,411
7,260
7,256
7,236
7,216
6,974
6,955
2,350
0,9
2,0
1,9
2,9
1,3
1,0
3,0
ppm

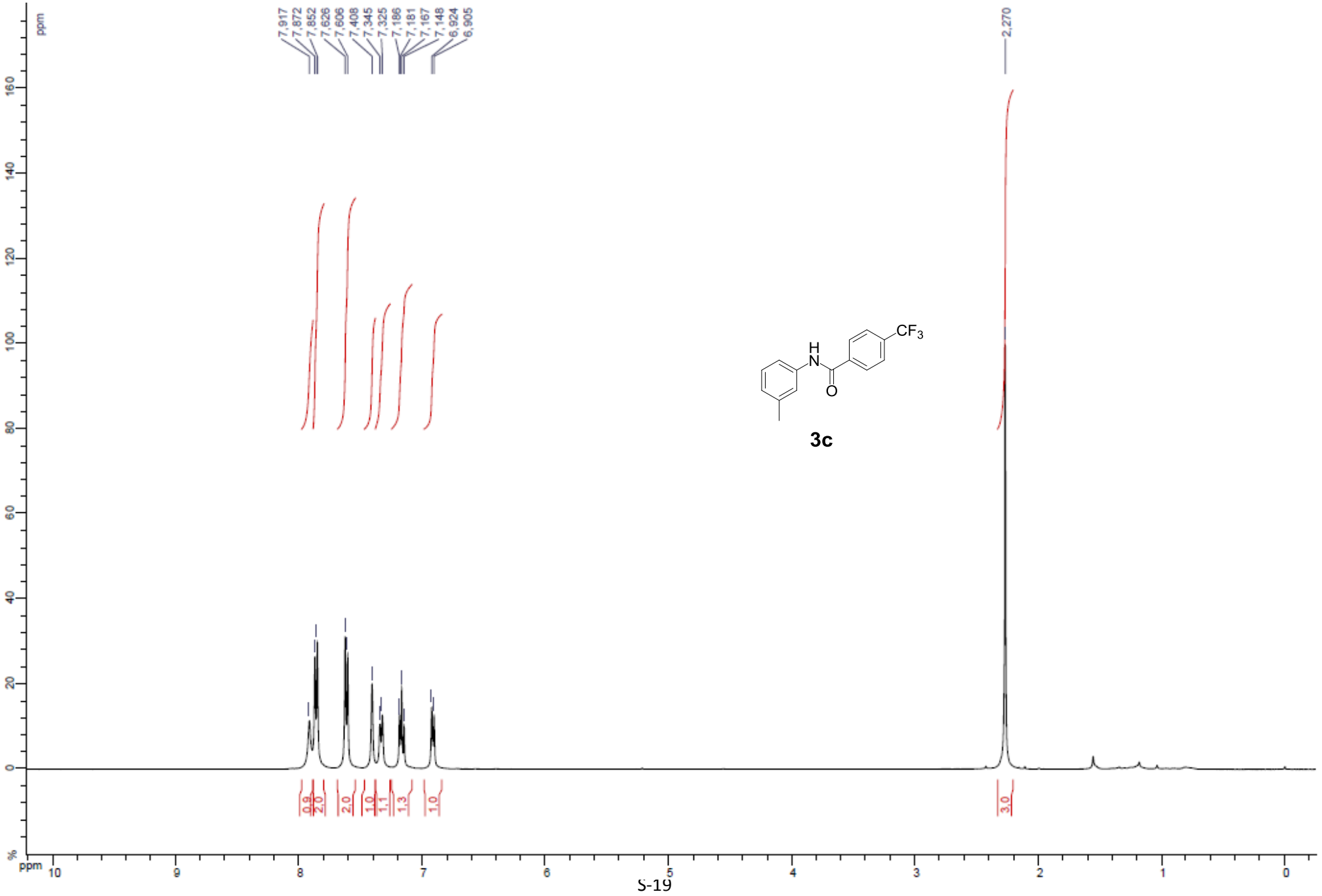
**3a**

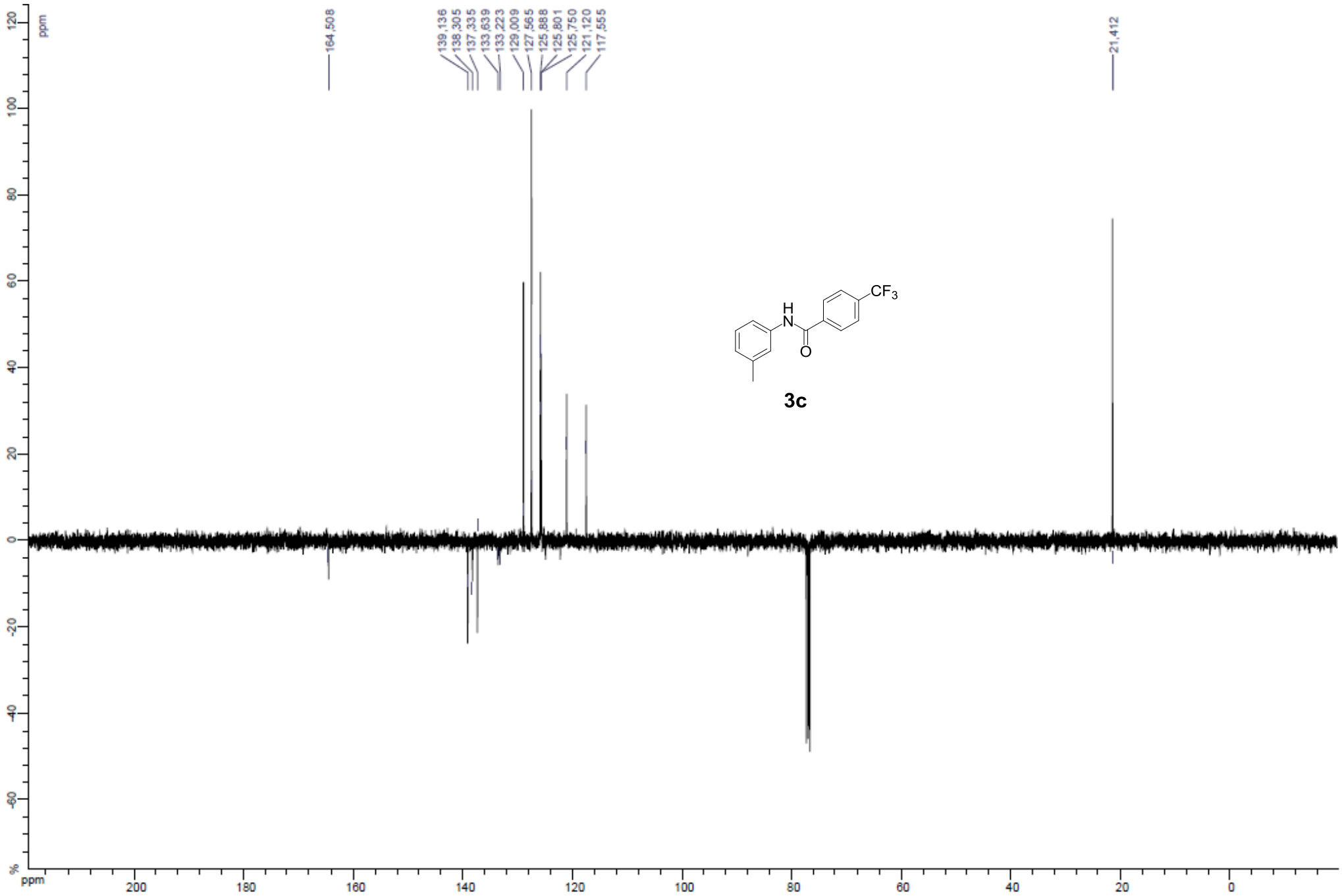

ppm
165.740
139.005
137.853
135.089
131.779
128.907
128.766
127.011
125.393
120.858
117.278
21.463
H
N
O
3a
100
80
60
40
20
0
-20
-40
%
ppm
200
180
160
140
120
100
80
60
40
20
0

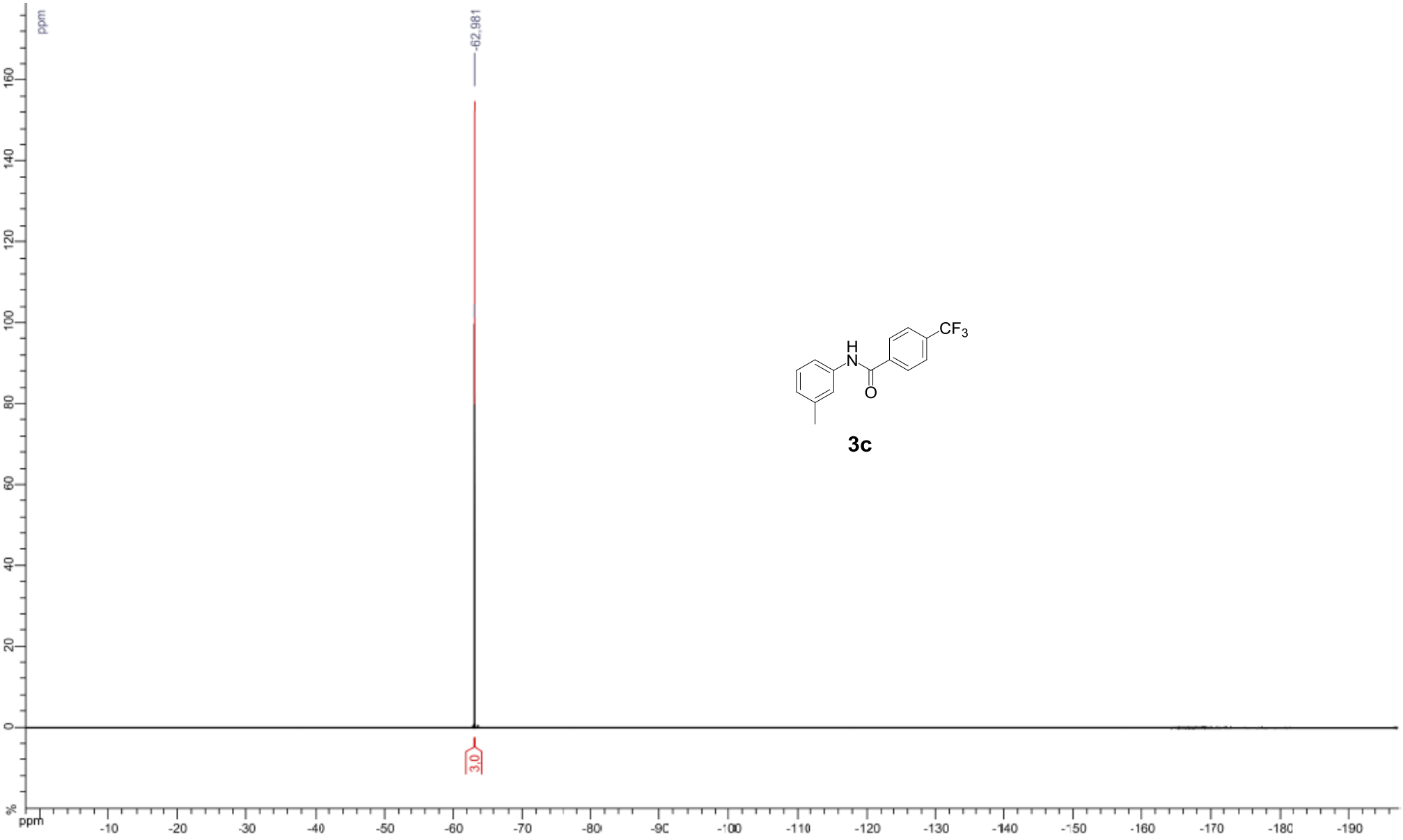

7.868
7.817
7.811
7.800
7.794
7.477
7.379
7.240
7.221
7.202
7.182
6.932
6.927
6.919
6.914
6.897
3.829
2.316
ppm
1.3
2.0
1.0
1.0
1.0
3.0
3.0
3.0
%
ppm
9
8
7
6
5
4
3
2
1
0

**3b**

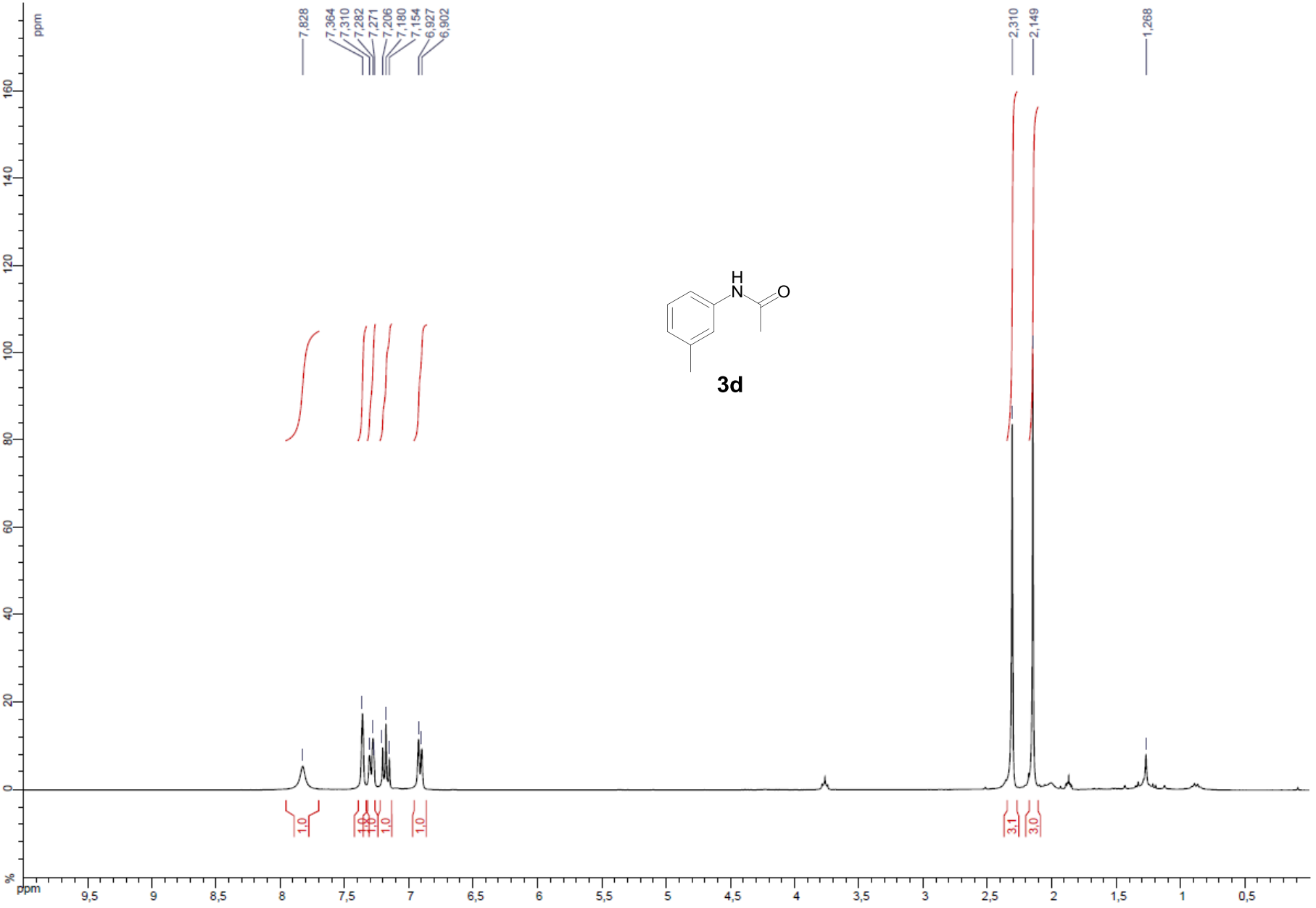

ppm
165.302
162.452
138.881
138.079
128.921
128.848
127.245
125.159
120.916
117.314
113.917
55.430
21.528
H
N
O
O
3b
%
ppm
200
180
160
140
120
100
80
60
40
20
0

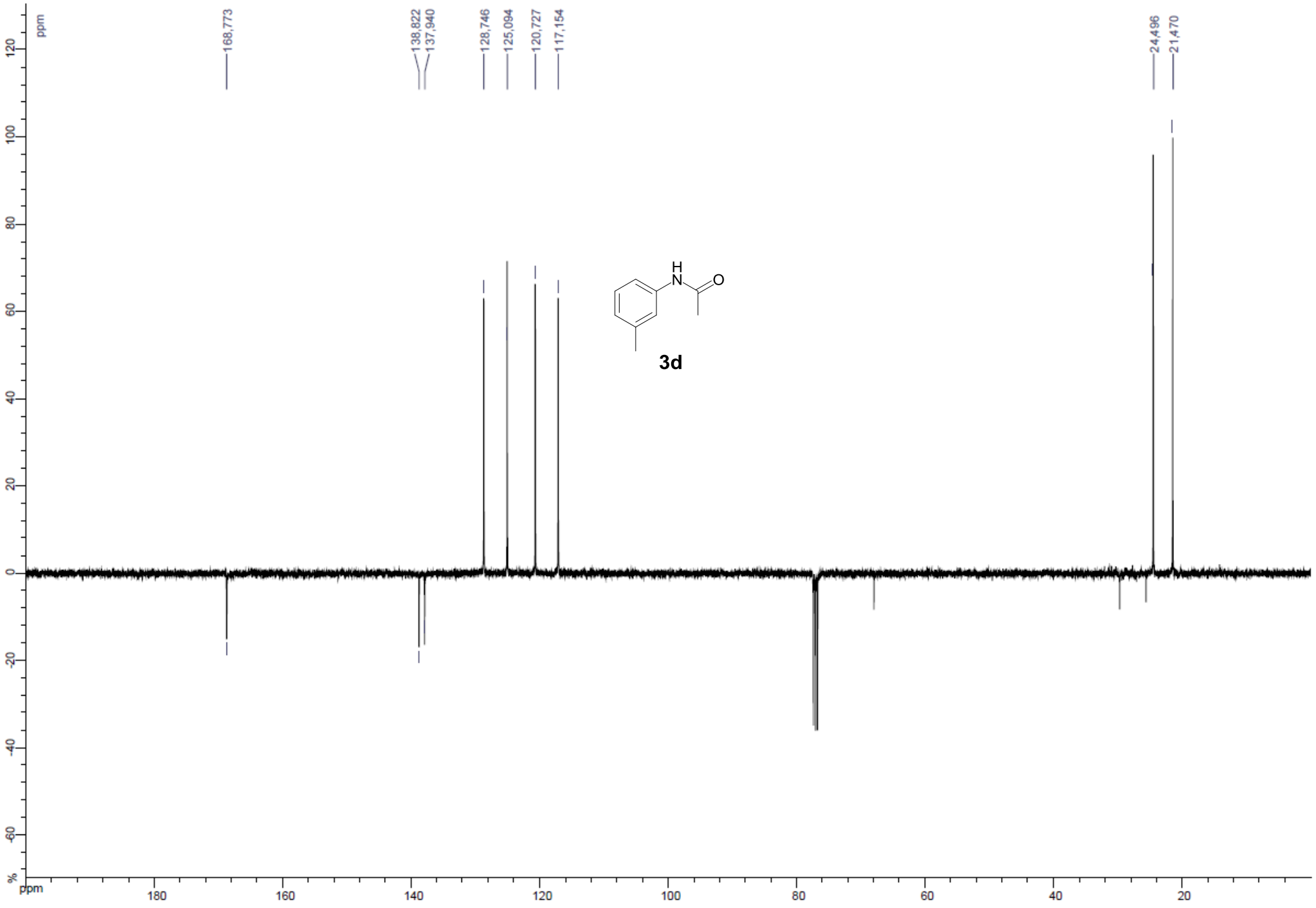

7,917
7,872
7,852
7,626
7,606
7,408
7,345
7,325
7,186
7,181
7,167
7,148
6,924
6,905
2,270
CF3
H
N
O
3c
0.9
2.0
2.0
1.0
1.1
1.3
1.0
3.0
ppm
10
9
8
7
6
5
4
3
2
1
0

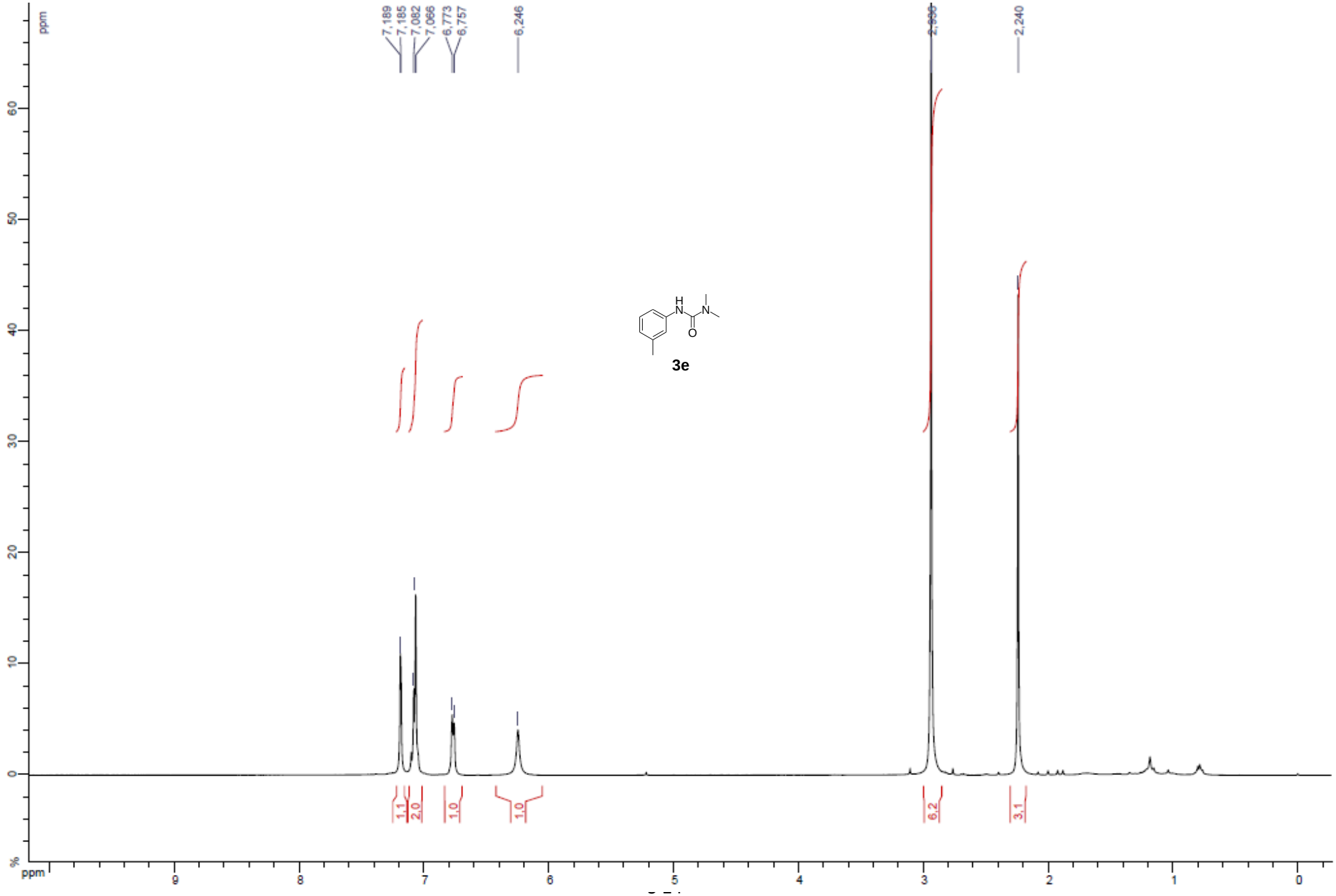

ppm
164,508
139,136
138,305
137,335
133,639
133,223
129,009
127,565
125,888
125,801
125,750
121,120
117,555
21,412
CF3
H
N
O
3c
120
100
80
60
40
20
0
-20
-40
-60
%
ppm
200
180
160
140
120
100
80
60
40
20
0


ppm
-62.981
3.0
%
ppm
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
H
N
O
CF3

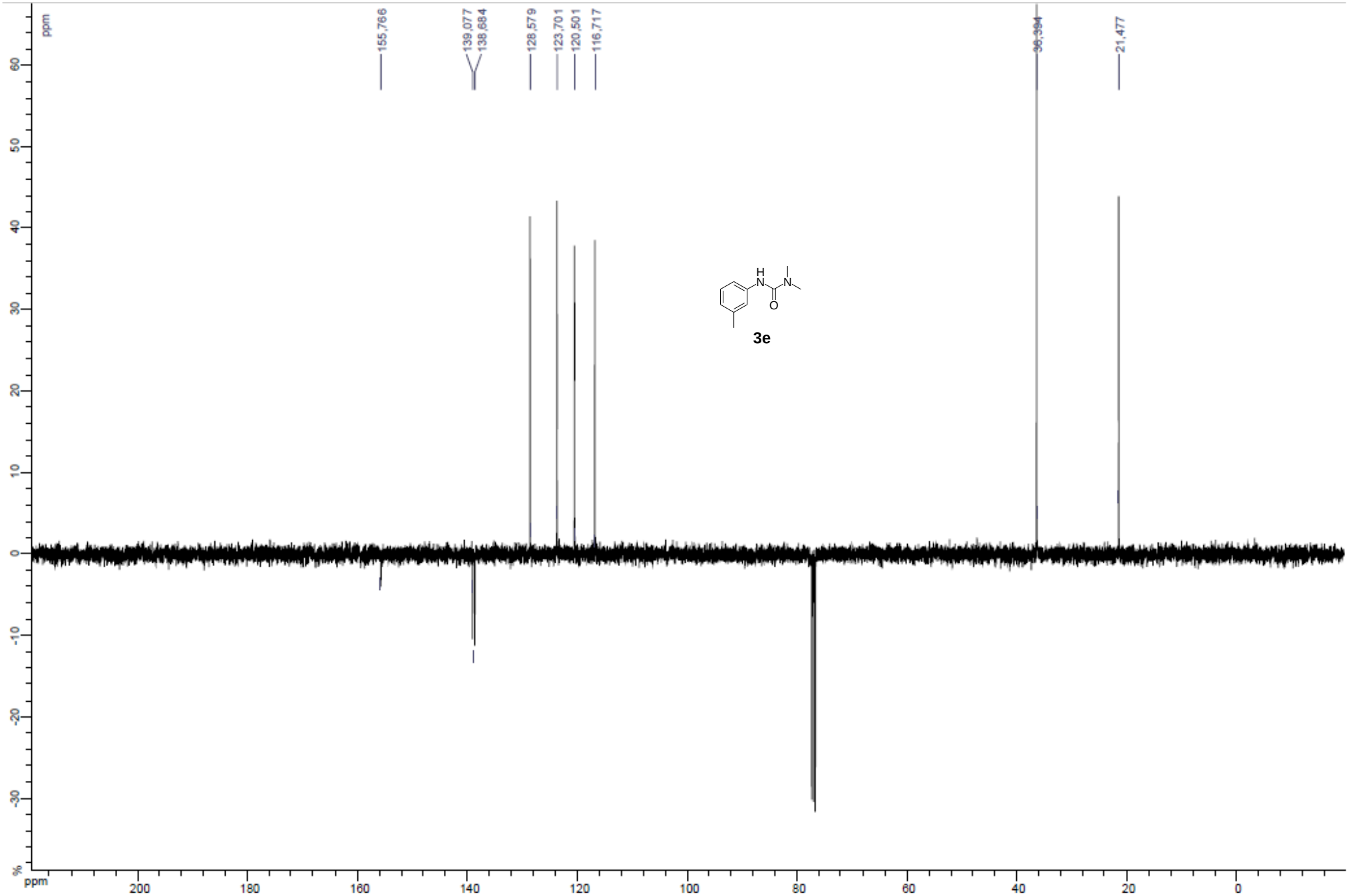
**3c**

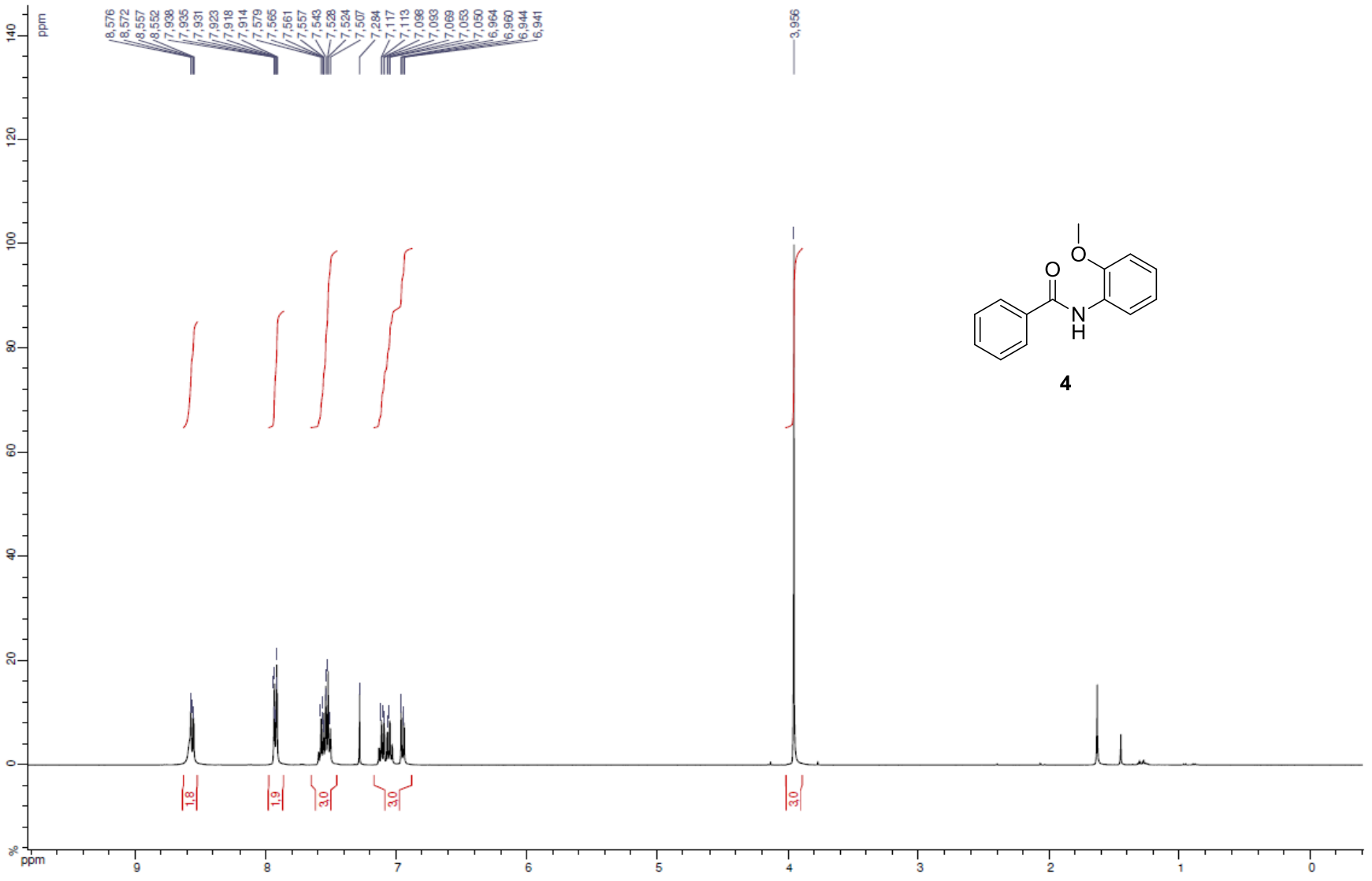

7,828
7,364
7,310
7,282
7,271
7,206
7,180
7,154
6,927
6,902
2,310
2,149
1,268
3d
1,0
1,0
1,0
1,0
3,1
3,0
ppm


ppm
168,773
138,822
137,940
128,746
125,094
120,727
117,154
24,496
21,470
H
N
O
3d
120
100
80
60
40
20
0
-20
-40
-60
%
ppm
180
160
140
120
100
80
60
40
20

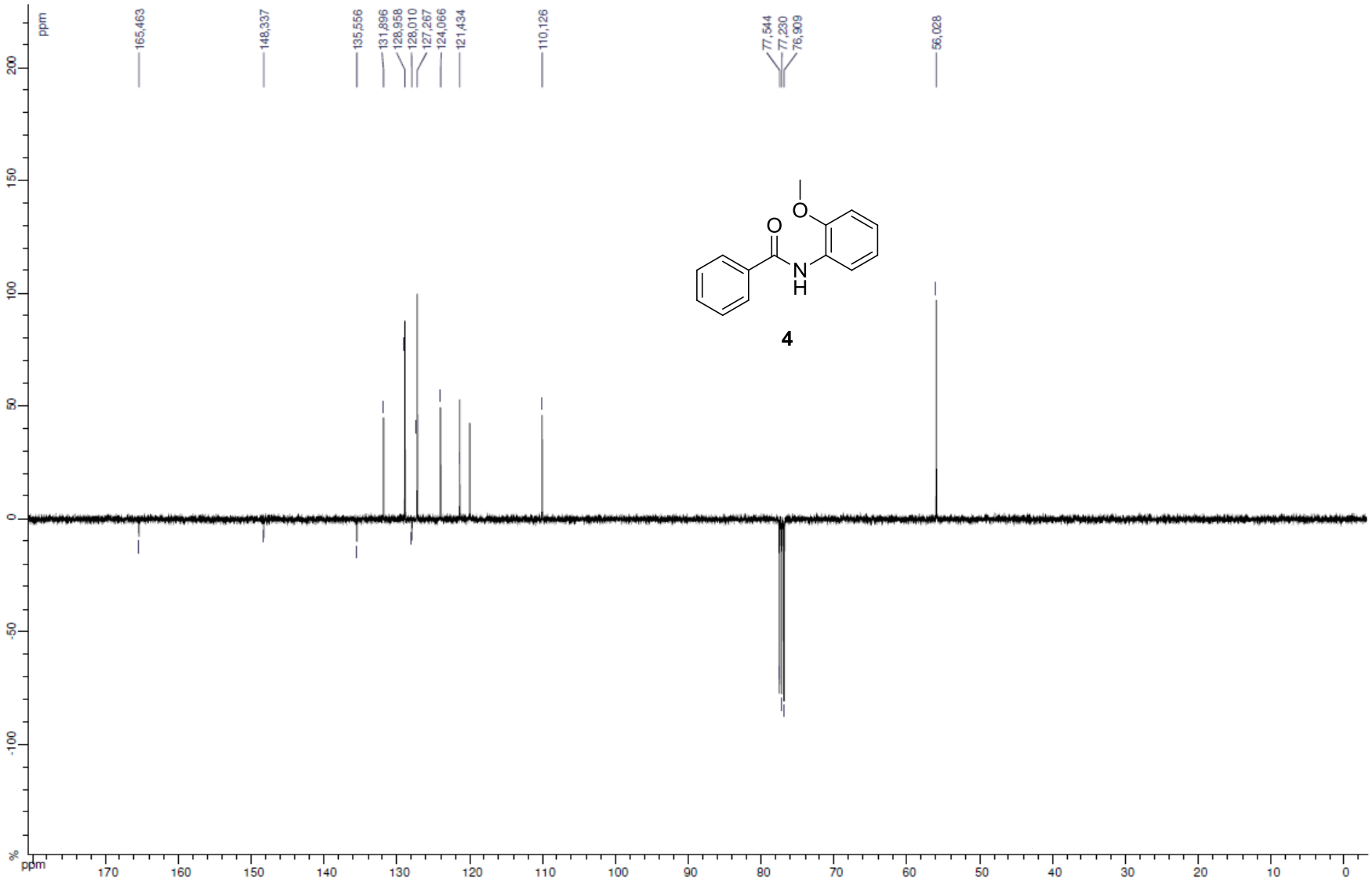

ppm
7,189
7,185
7,082
7,066
6,773
6,757
6,246
2,240
1,1
2,0
1,0
1,0
6,2
3,1
%
ppm
9
8
7
6
5
4
3
2
1
0

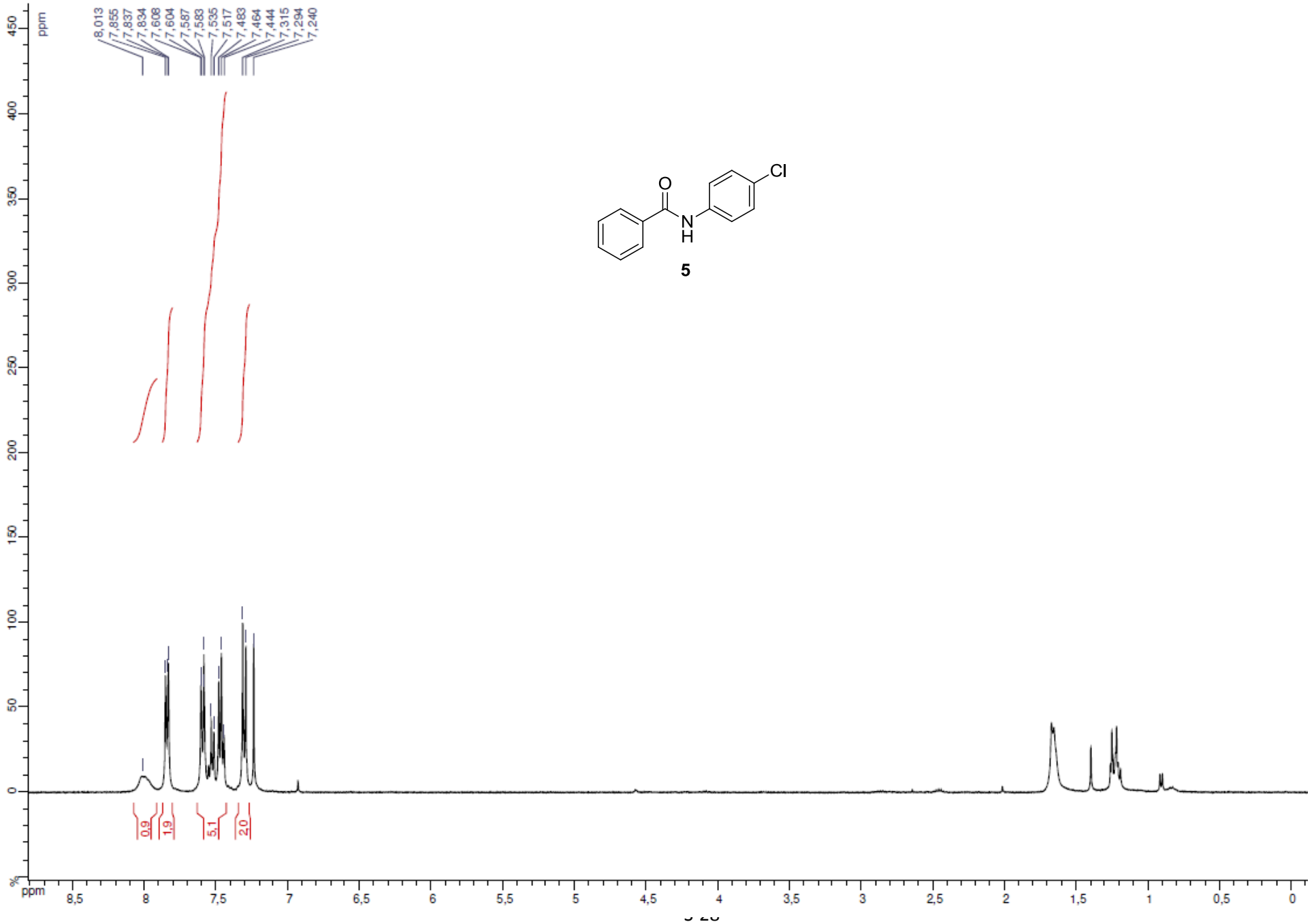
**3e**


ppm
155,766
139,077
138,684
128,579
123,701
120,501
116,717
36,394
21,477
3e
%
ppm
200
180
160
140
120
100
80
60
40
20
0

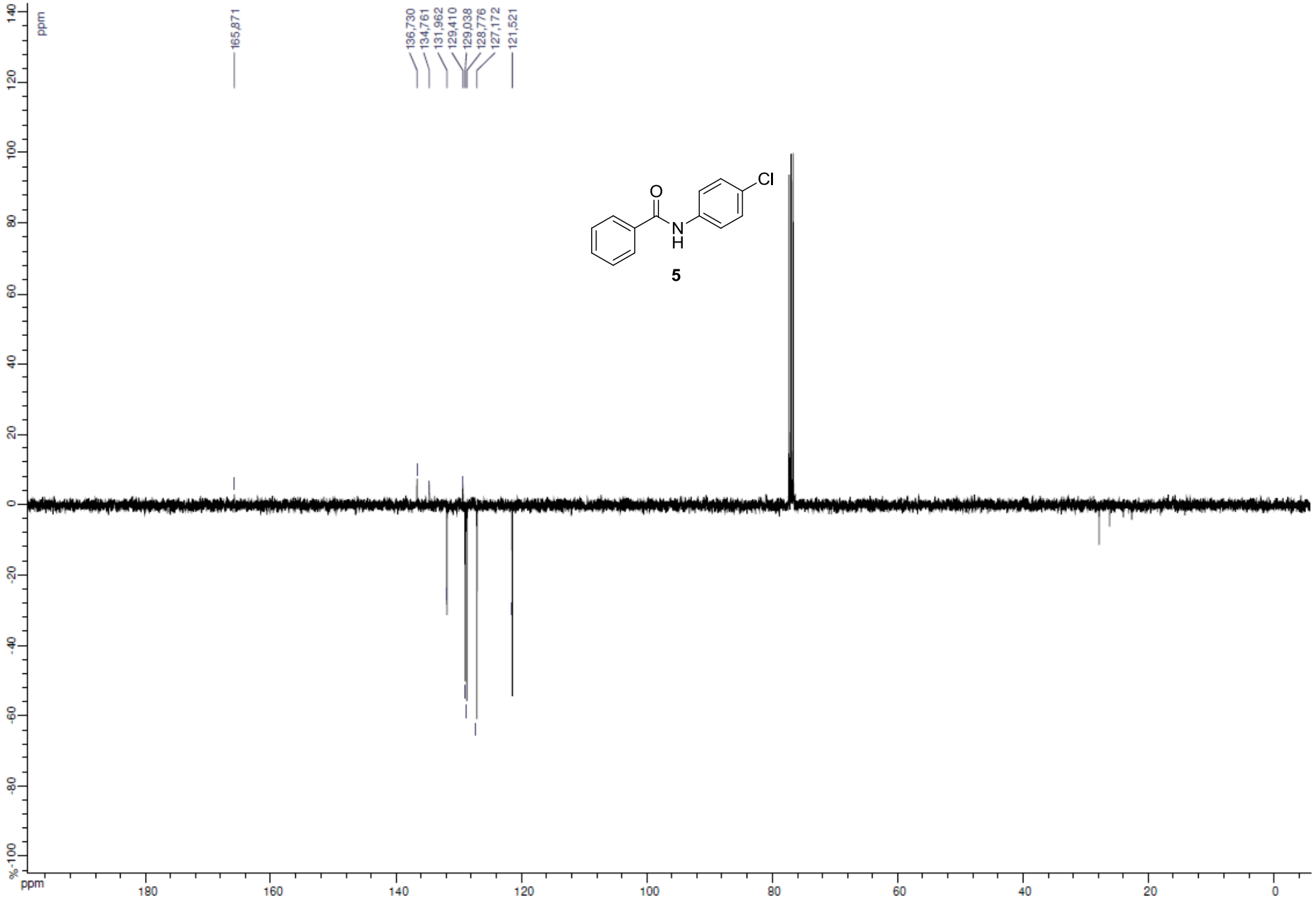

8.576
8.572
8.557
8.552
7.938
7.935
7.931
7.923
7.918
7.914
7.579
7.565
7.561
7.557
7.543
7.528
7.524
7.507
7.284
7.117
7.113
7.098
7.093
7.089
7.053
7.050
6.964
6.960
6.944
6.941
3.956
1.8
1.9
3.0
3.0
3.0
ppm
O
O
N
H

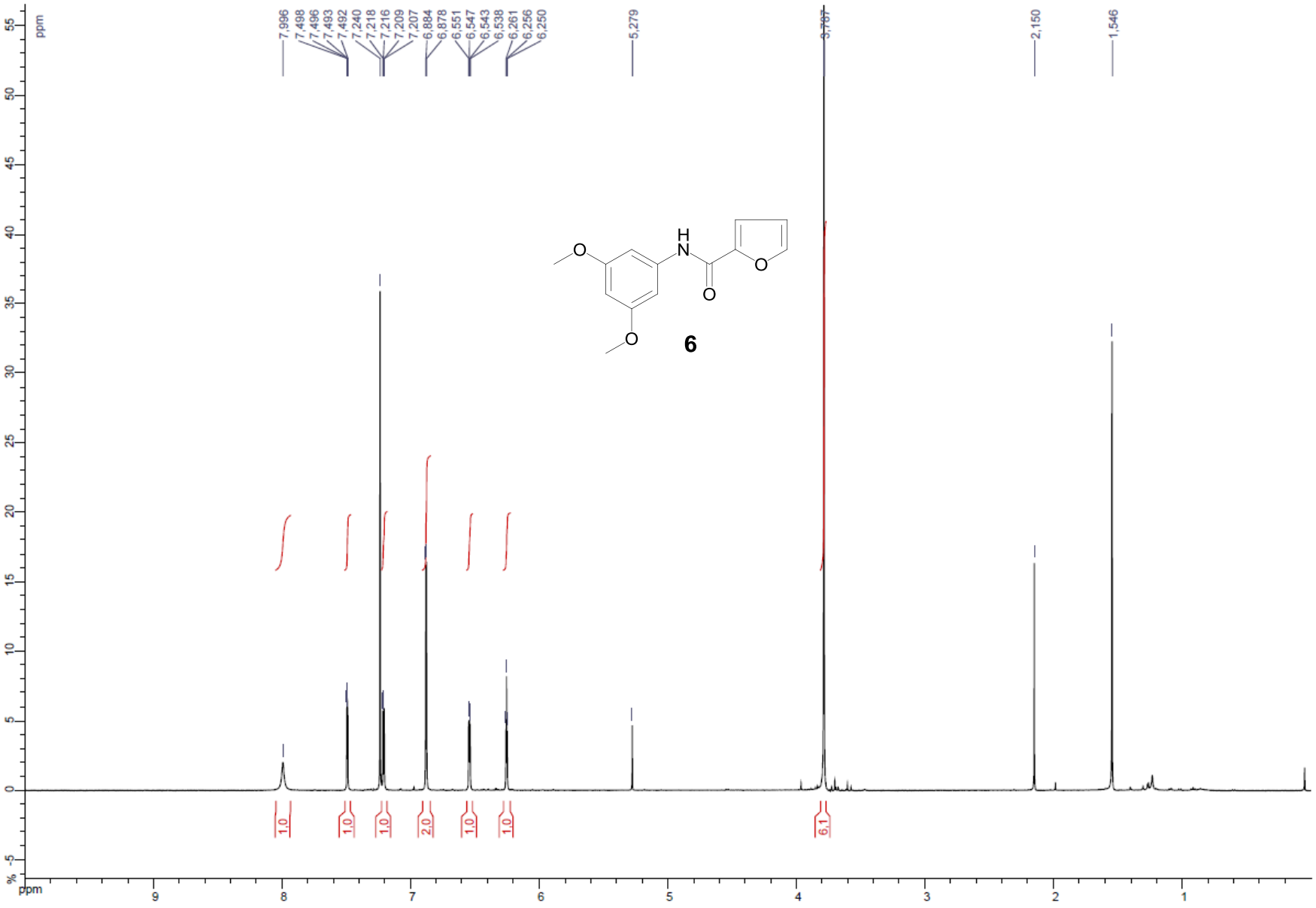
**4**

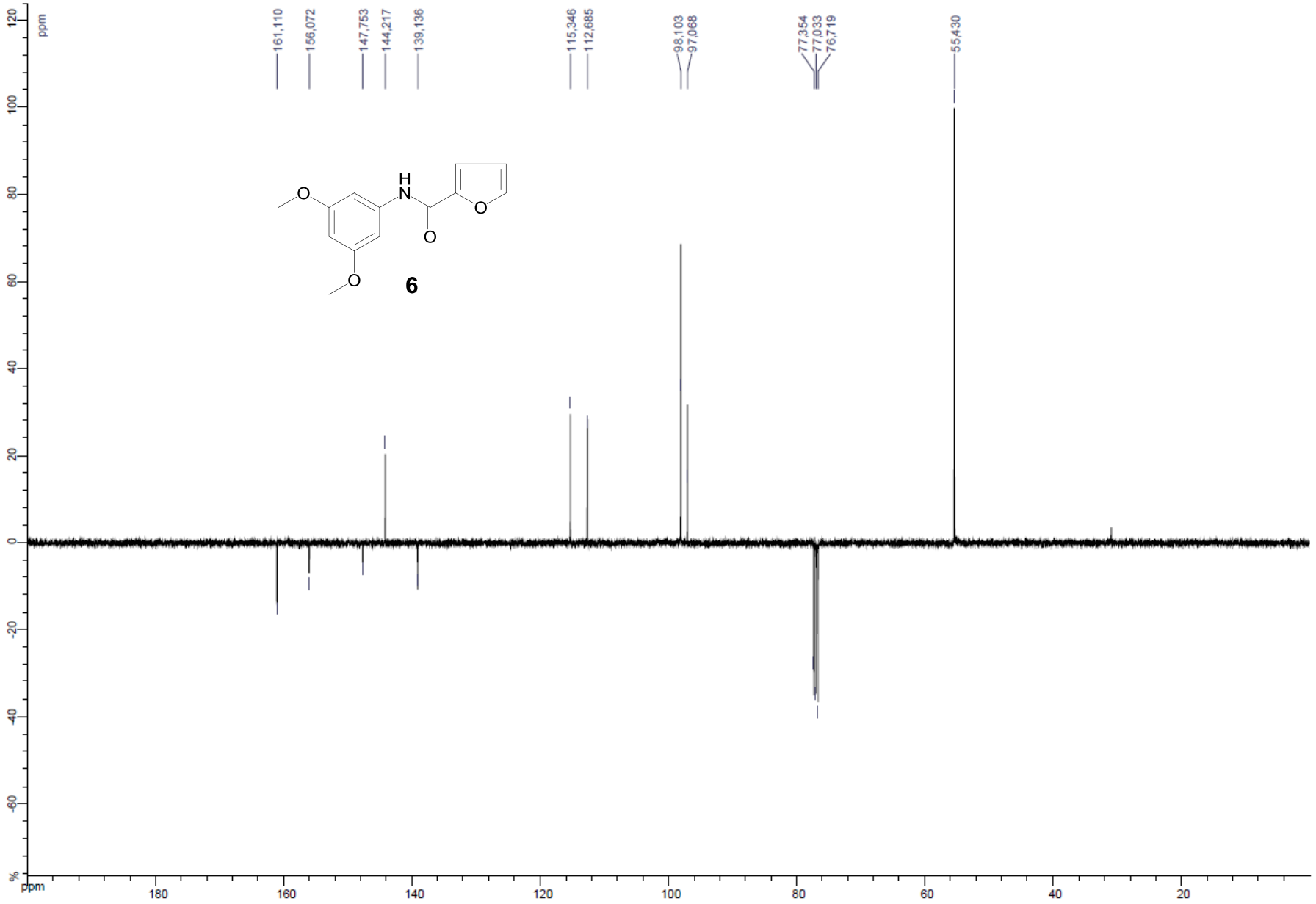

ppm
165,463
148,337
135,556
131,896
128,958
128,010
127,267
124,066
121,434
110,126
77,544
77,230
76,909
56,028
O
O
N
H
4

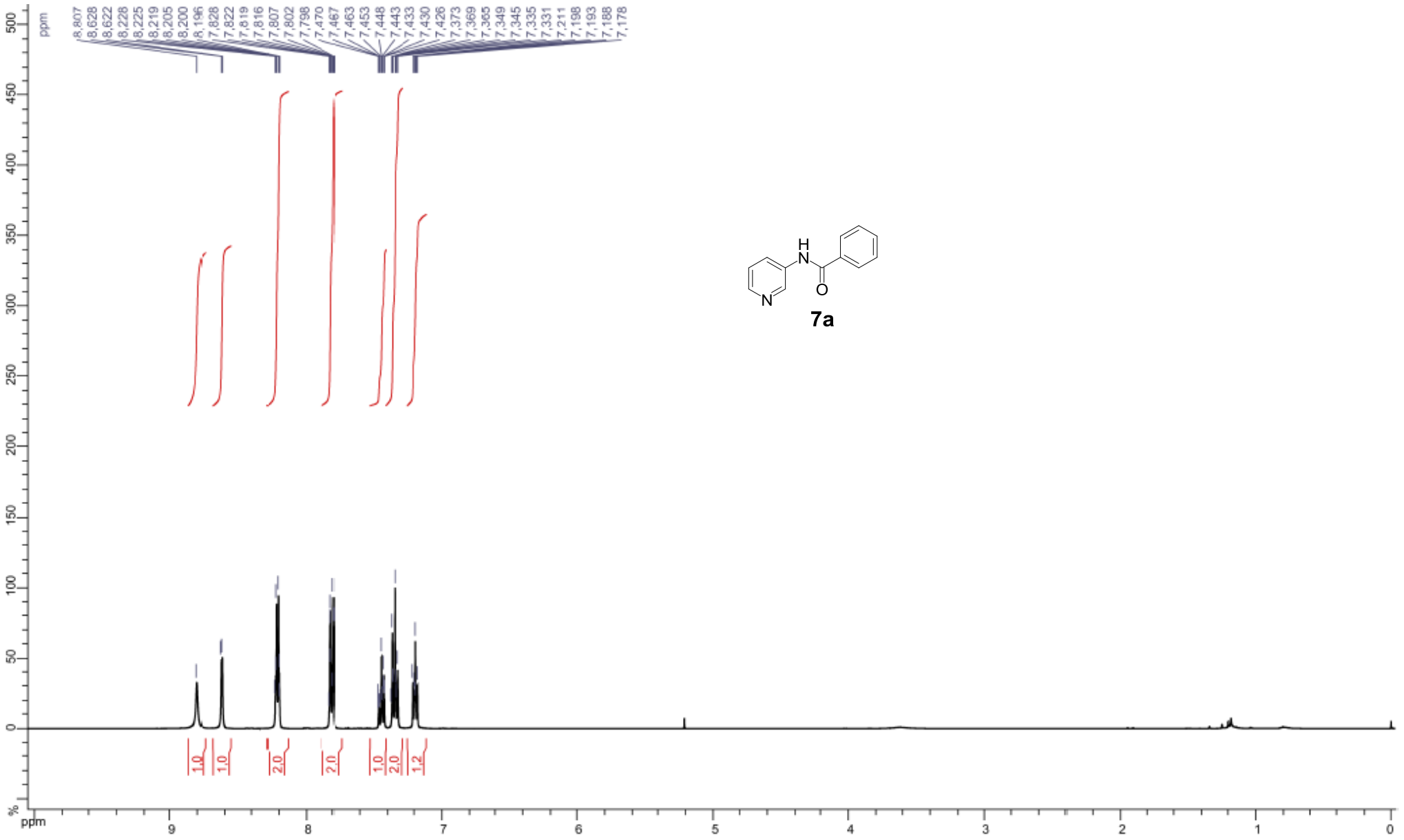

8,013
7,855
7,837
7,834
7,608
7,604
7,587
7,583
7,535
7,517
7,483
7,464
7,444
7,315
7,294
7,240
ppm
0,9
1,9
5,1
2,0
Cl
O
N
H

**5**

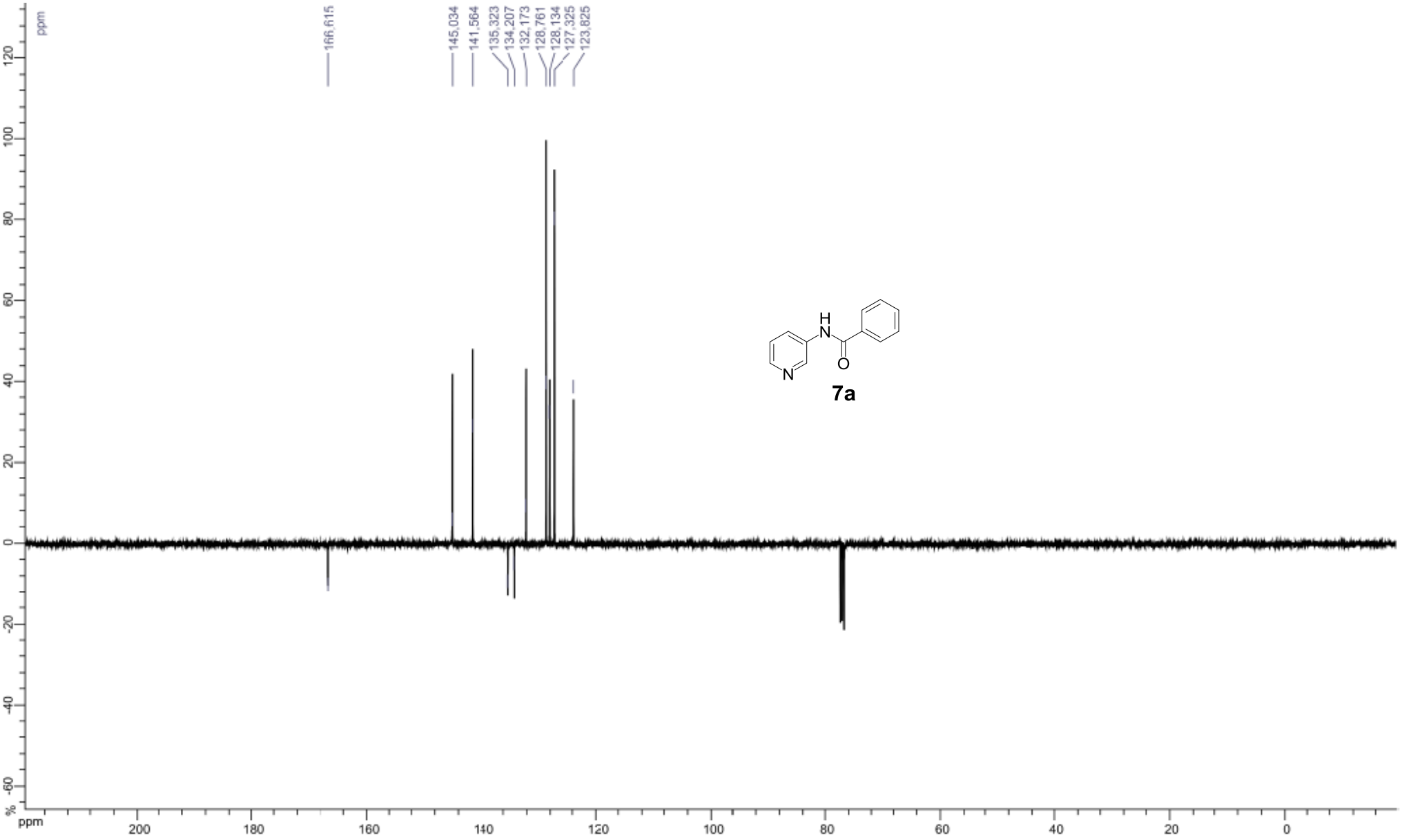

ppm
165,871
136,730
134,761
131,962
129,410
129,038
128,776
127,172
121,521
O
N
H
Cl
5
ppm
180
160
140
120
100
80
60
40
20
0
%
-100
-80
-60
-40
-20
0
20
40
60
80
100
120
140

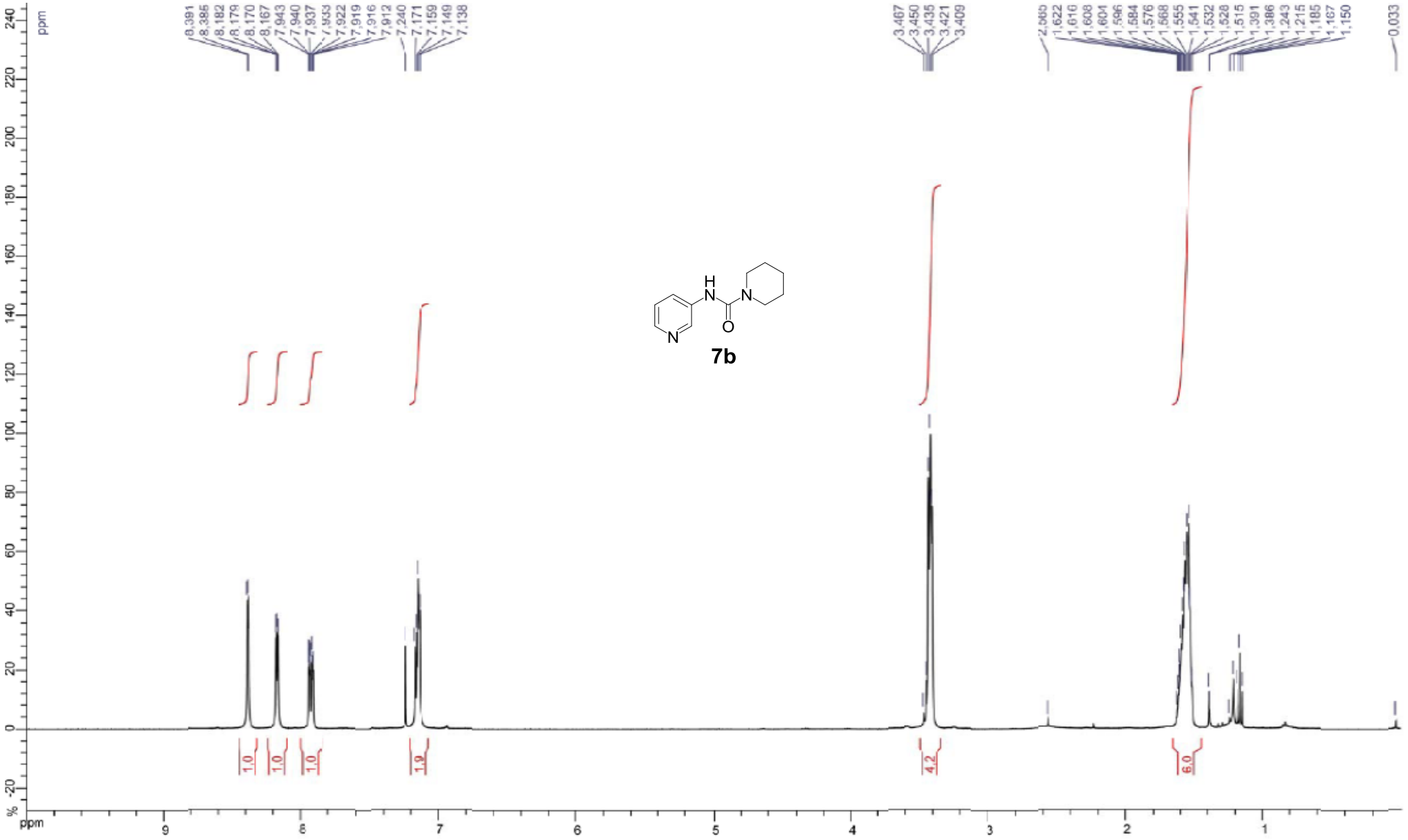

7,996
7,498
7,496
7,493
7,492
7,240
7,218
7,216
7,209
7,207
6,884
6,878
6,551
6,547
6,543
6,538
6,261
6,256
6,250
5,279
3,787
2,150
1,546
ppm
1,0
1,0
1,0
2,0
1,0
1,0
6,1
H
N
O
O
O
O
6


ppm
161,110
156,072
147,753
144,217
139,136
115,346
112,685
98,103
97,068
77,354
77,033
76,719
55,430
H
N
O
O
O
O
6
120
100
80
60
40
20
0
-20
-40
-60
%
ppm
180
160
140
120
100
80
60
40
20

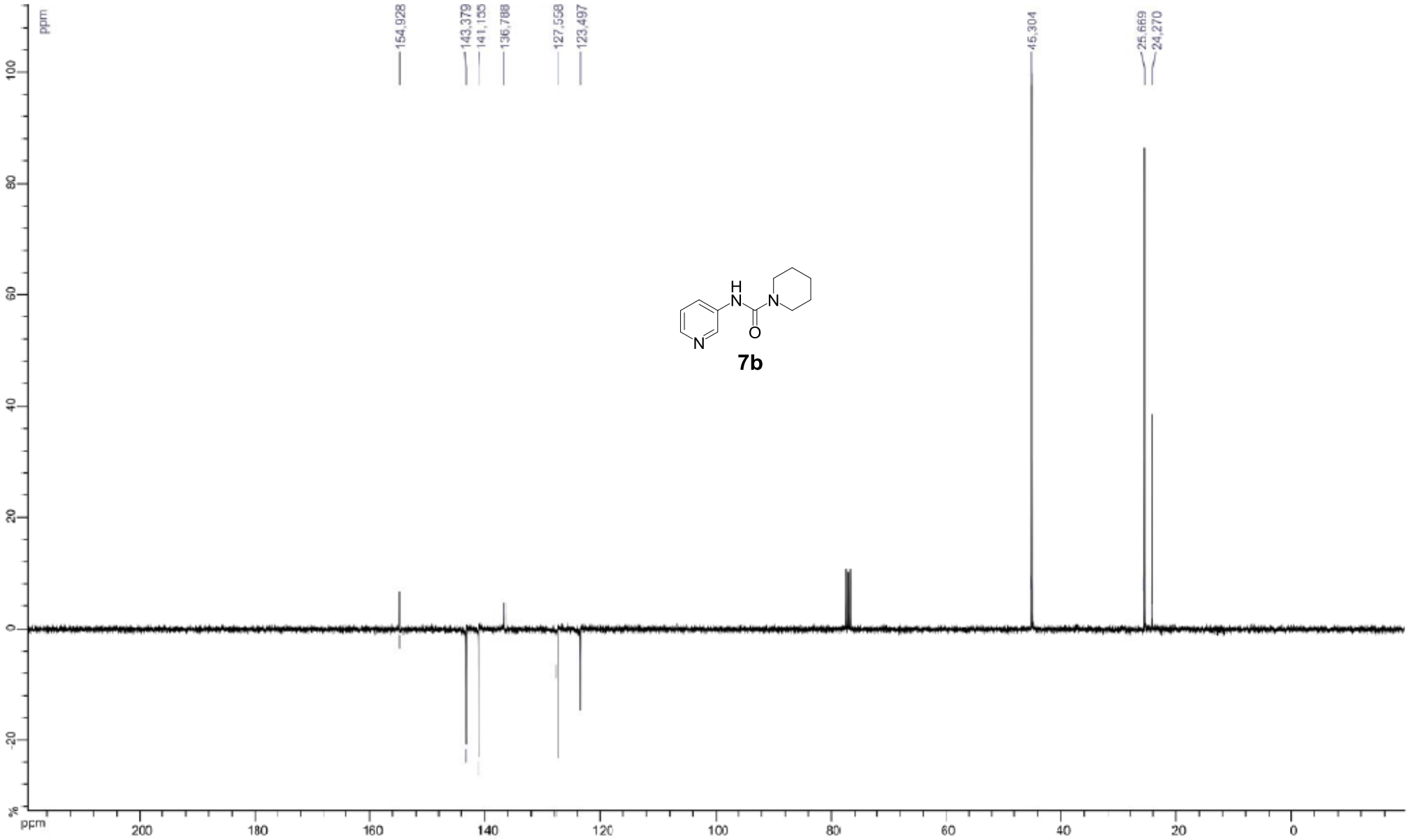

8,807
8,628
8,622
8,228
8,225
8,219
8,205
8,200
8,196
7,828
7,822
7,819
7,816
7,807
7,802
7,798
7,470
7,467
7,463
7,453
7,448
7,443
7,433
7,430
7,426
7,373
7,369
7,365
7,349
7,345
7,335
7,331
7,211
7,198
7,193
7,188
7,178
ppm
1,0
1,0
2,0
2,0
1,0
2,0
1,2
%
ppm

**7a**

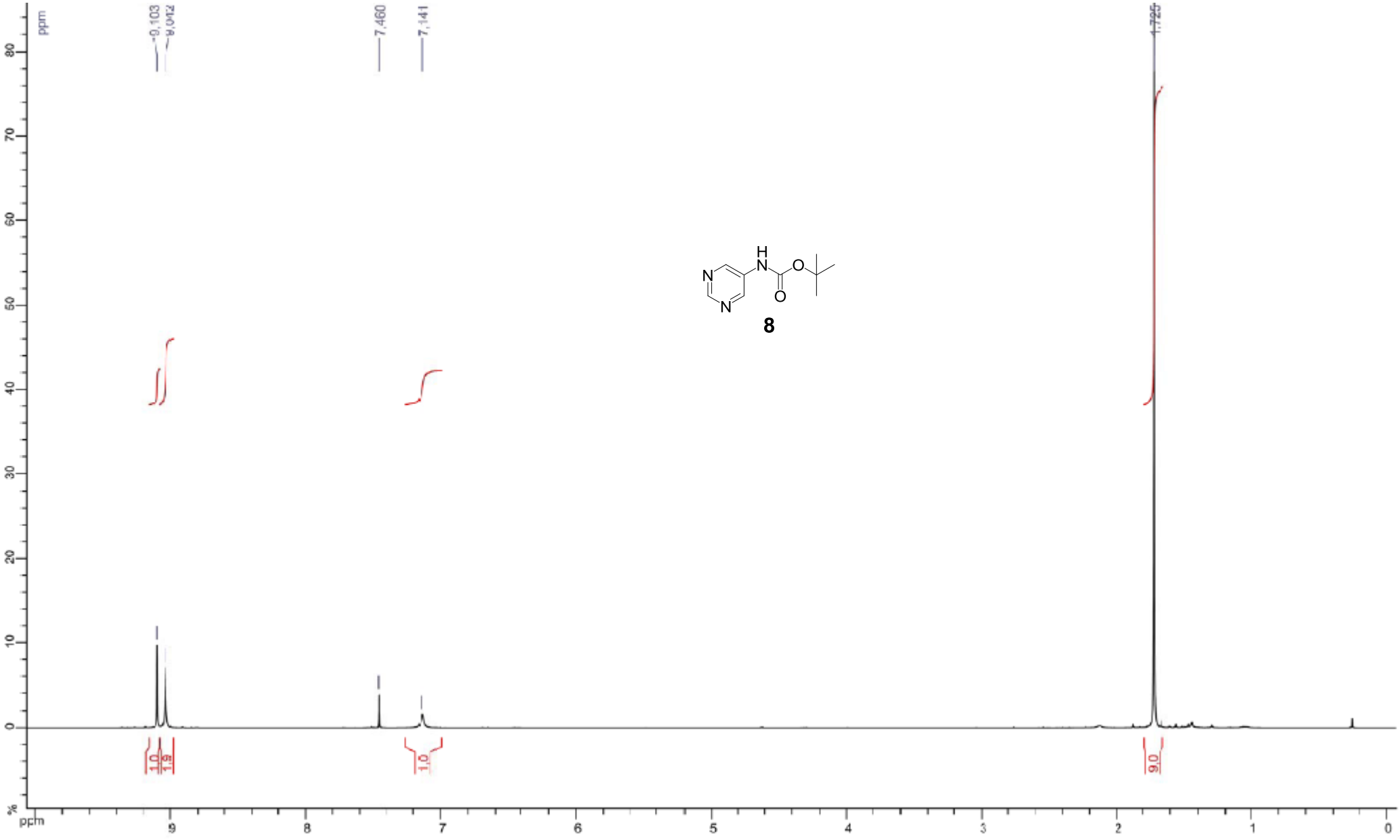

ppm
166,615
145,034
141,564
135,323
134,207
132,173
128,761
128,134
127,325
123,825
120
100
80
60
40
20
0
-20
-40
-60
%
ppm
200
180
160
140
120
100
80
60
40
20
0
H
N
O
N
7a


8.391
8.385
8.182
8.179
8.170
8.167
7.943
7.940
7.937
7.922
7.919
7.916
7.912
7.240
7.171
7.159
7.149
7.138
3.467
3.450
3.435
3.421
3.409
1.622
1.616
1.608
1.604
1.596
1.584
1.576
1.568
1.555
1.541
1.532
1.528
1.515
1.391
1.386
1.243
1.215
1.185
1.167
1.150
0.033
1.0
1.0
1.0
1.9
4.2
6.0
ppm

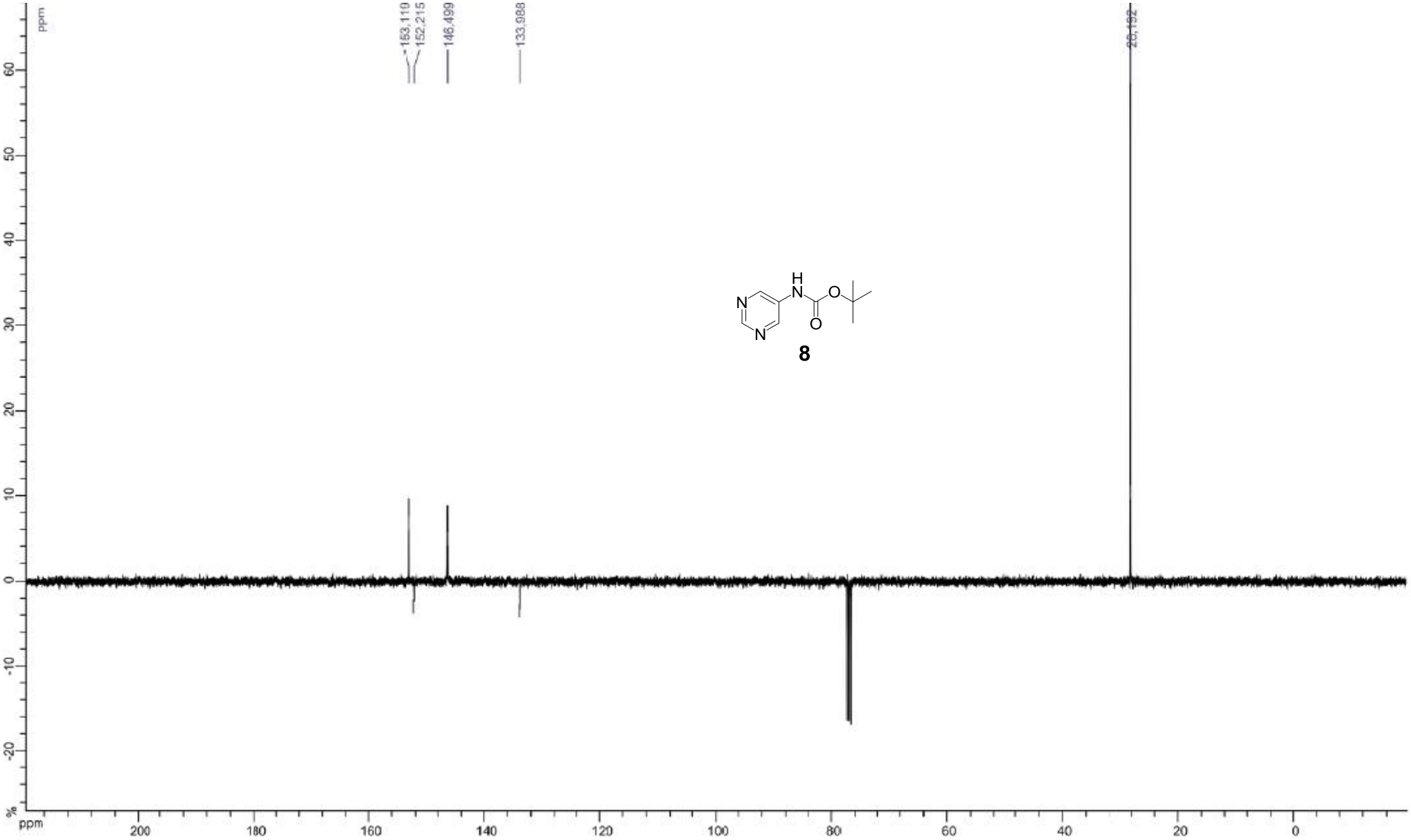
**7b**

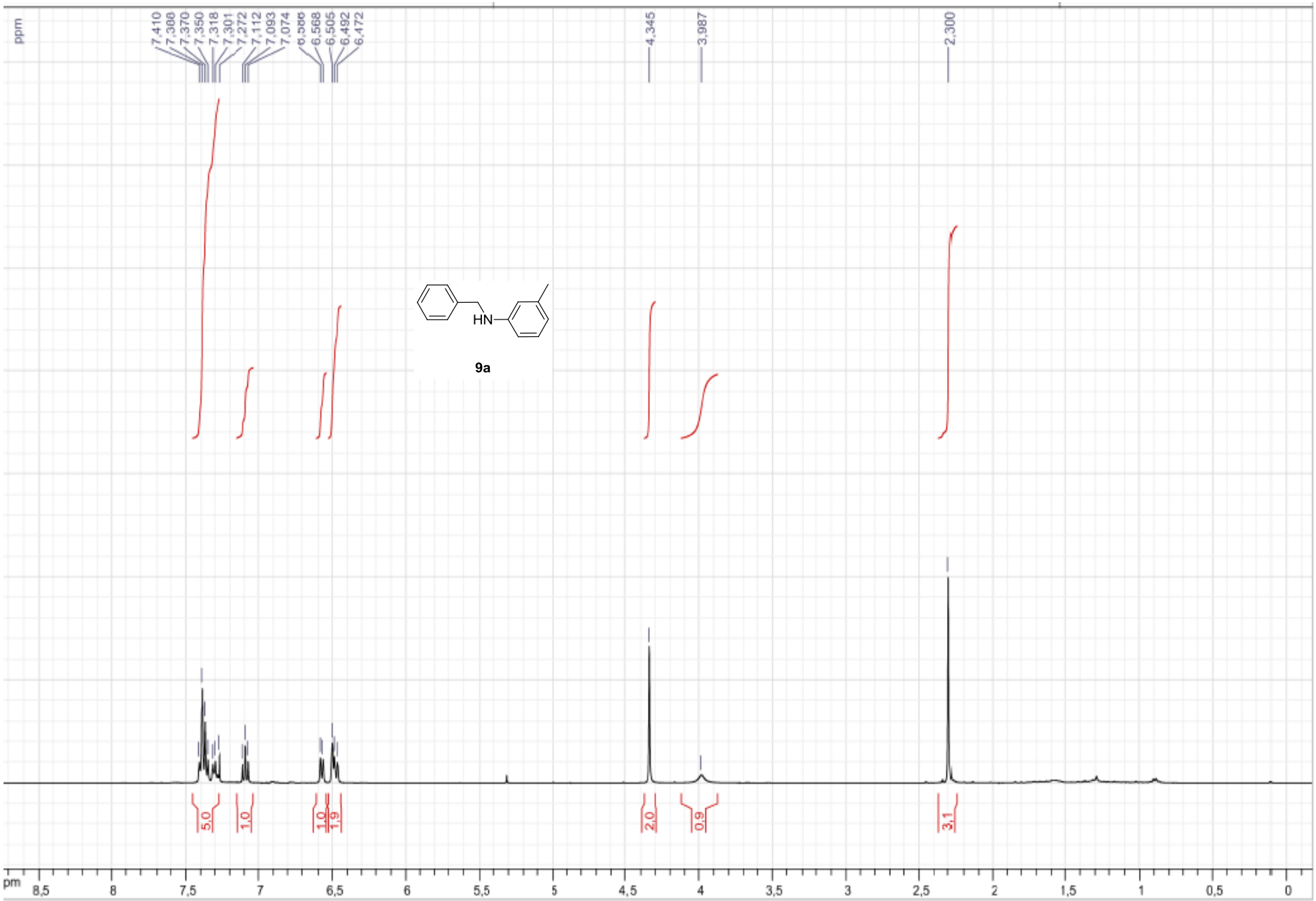

ppm
154,928
143,379
141,155
136,788
127,558
123,497
45,304
25,669
24,270
100
80
60
40
20
0
-20
%
ppm
200
180
160
140
120
100
80
60
40
20
0
H
N
N
O
N

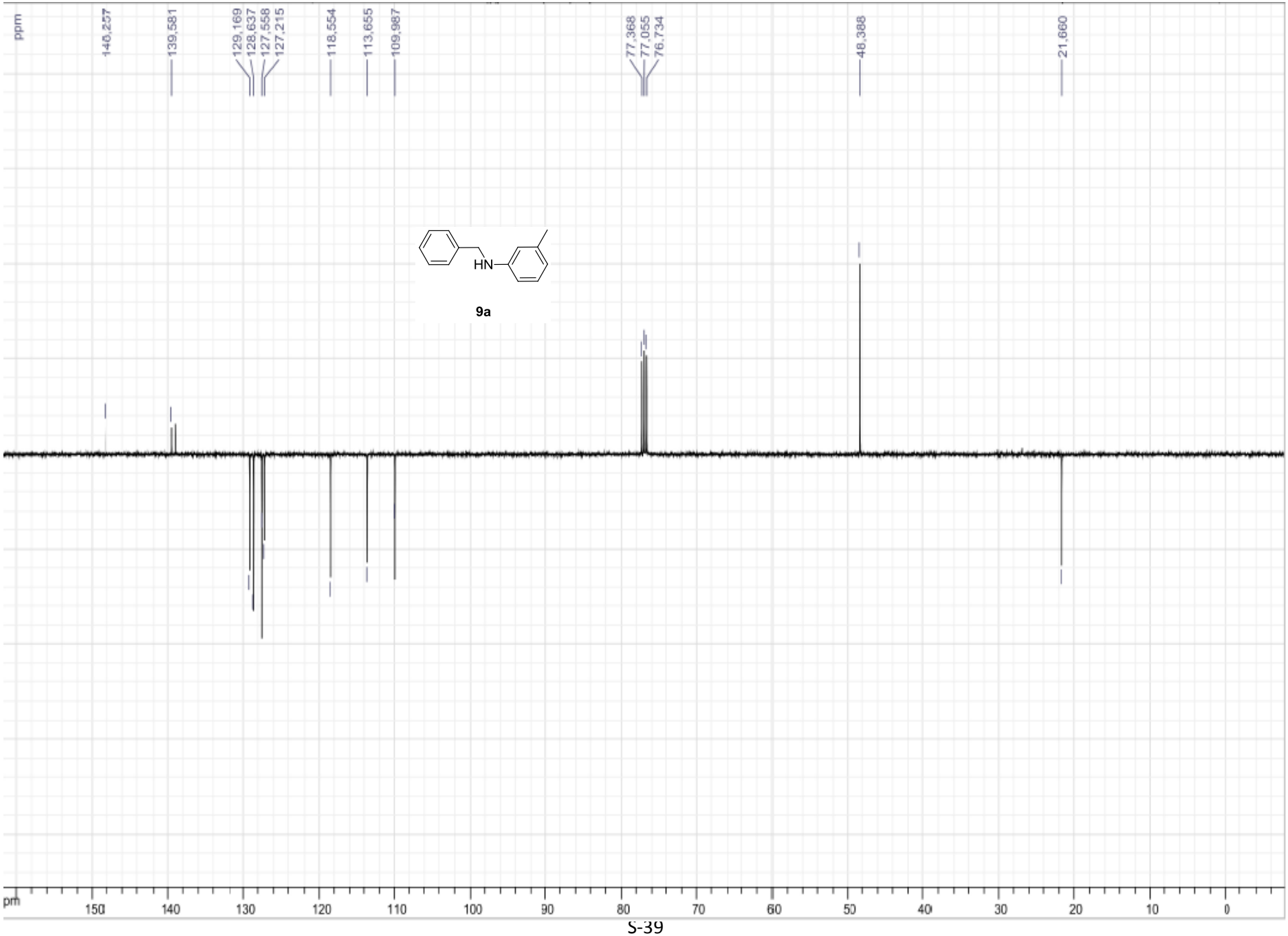
**7b**

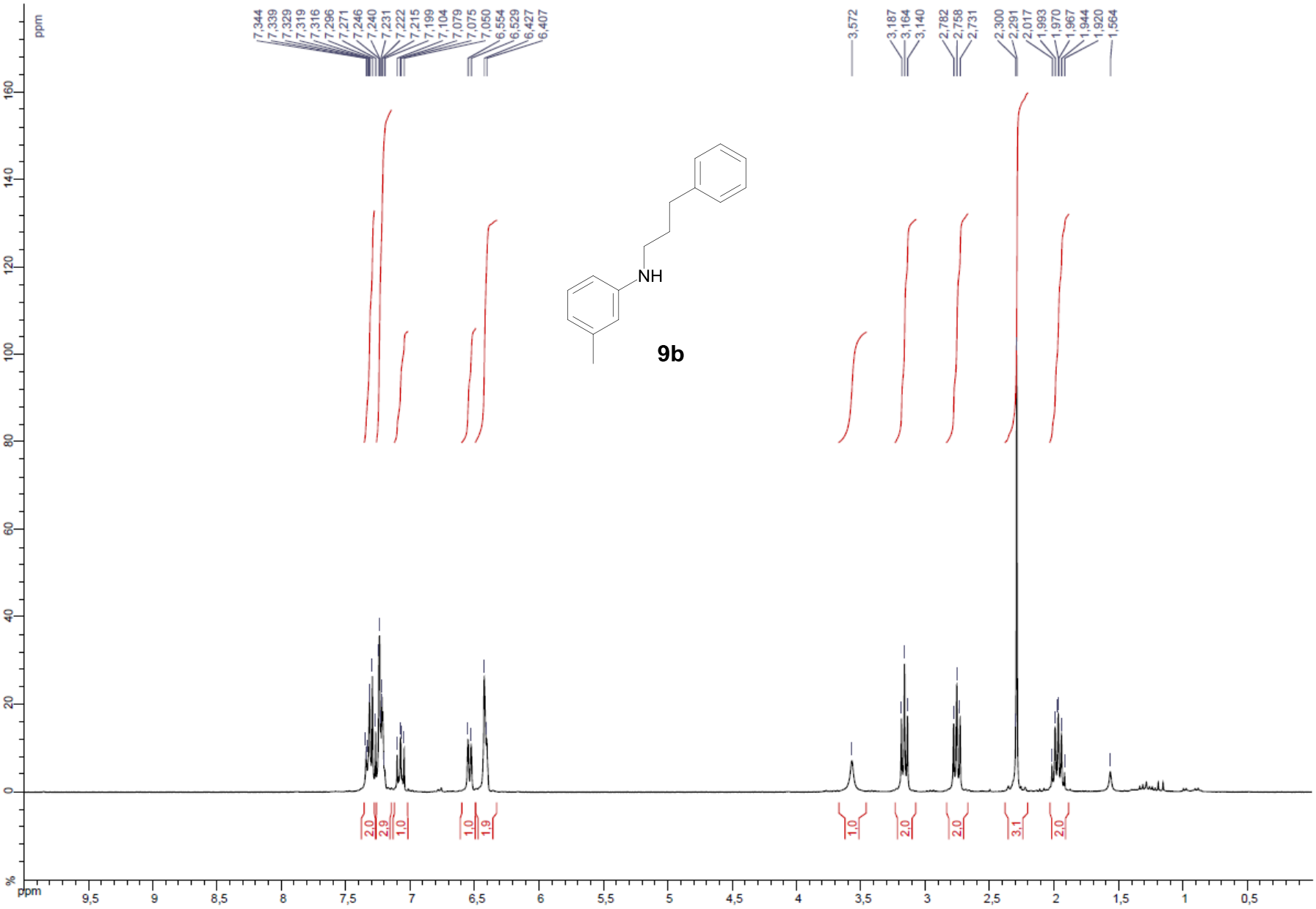


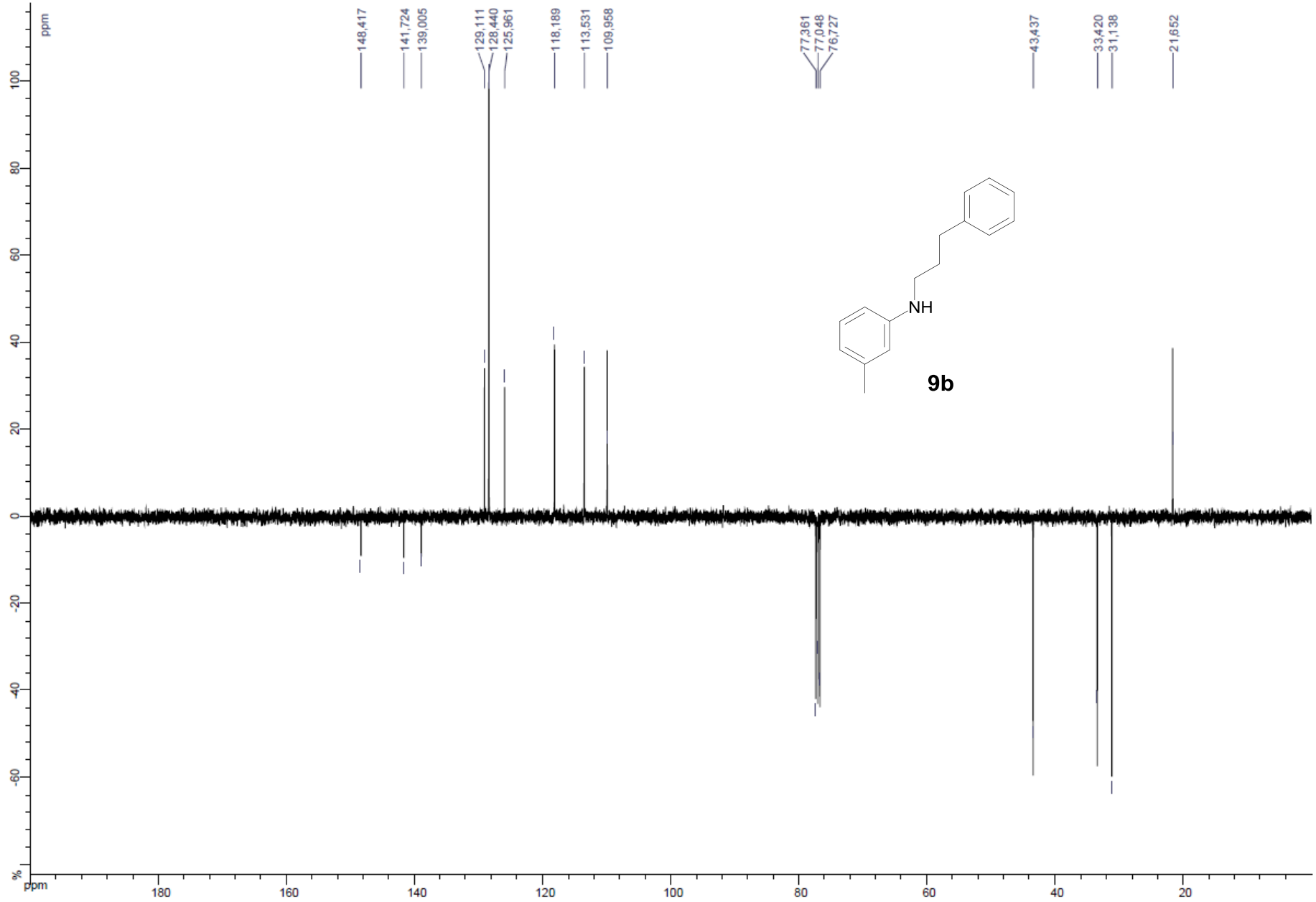

**8**

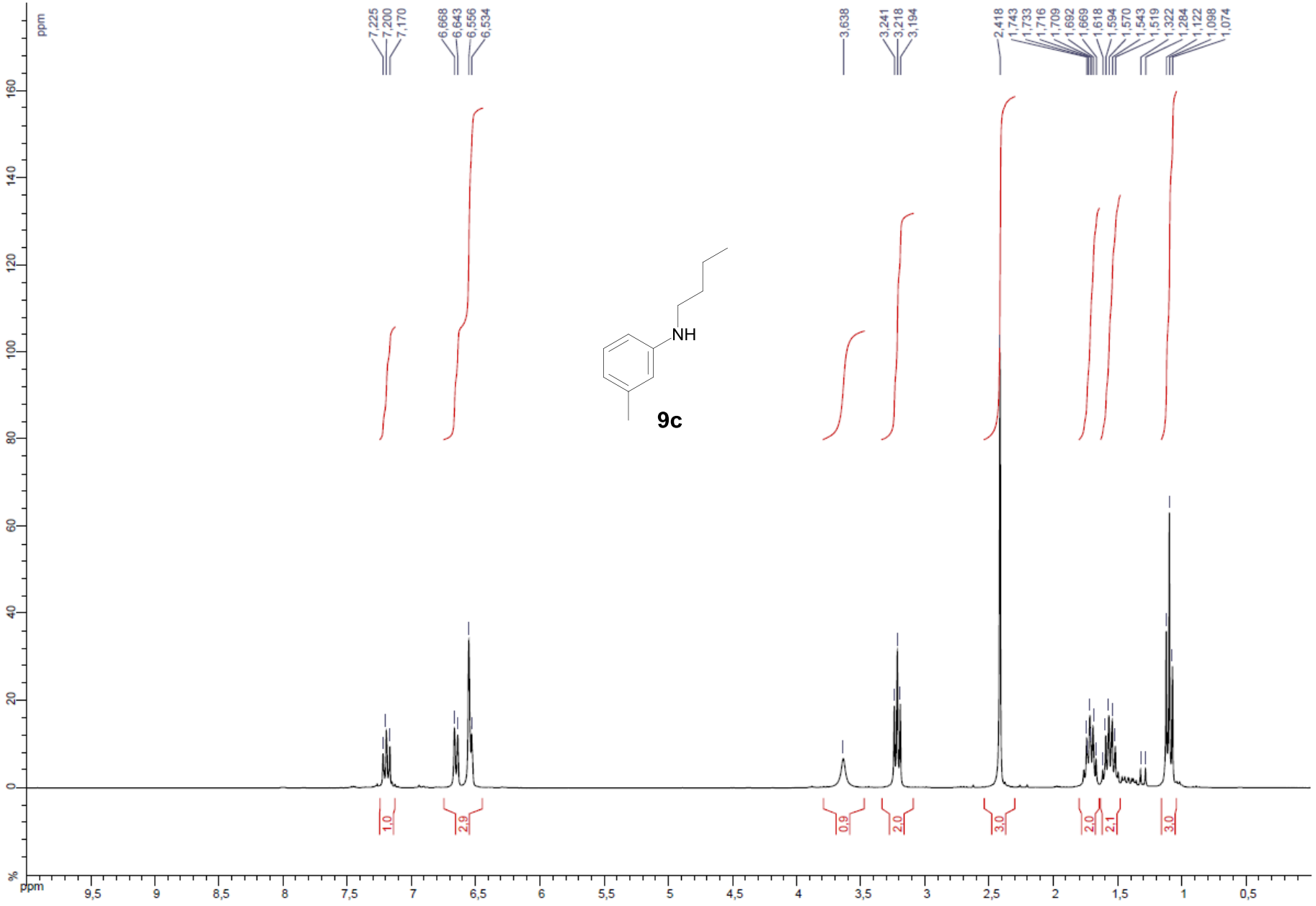

ppm
153,119
152,215
146,499
133,988
28,192
8
ppm
200
180
160
140
120
100
80
60
40
20
0
%
60
50
40
30
20
10
0
-10
-20

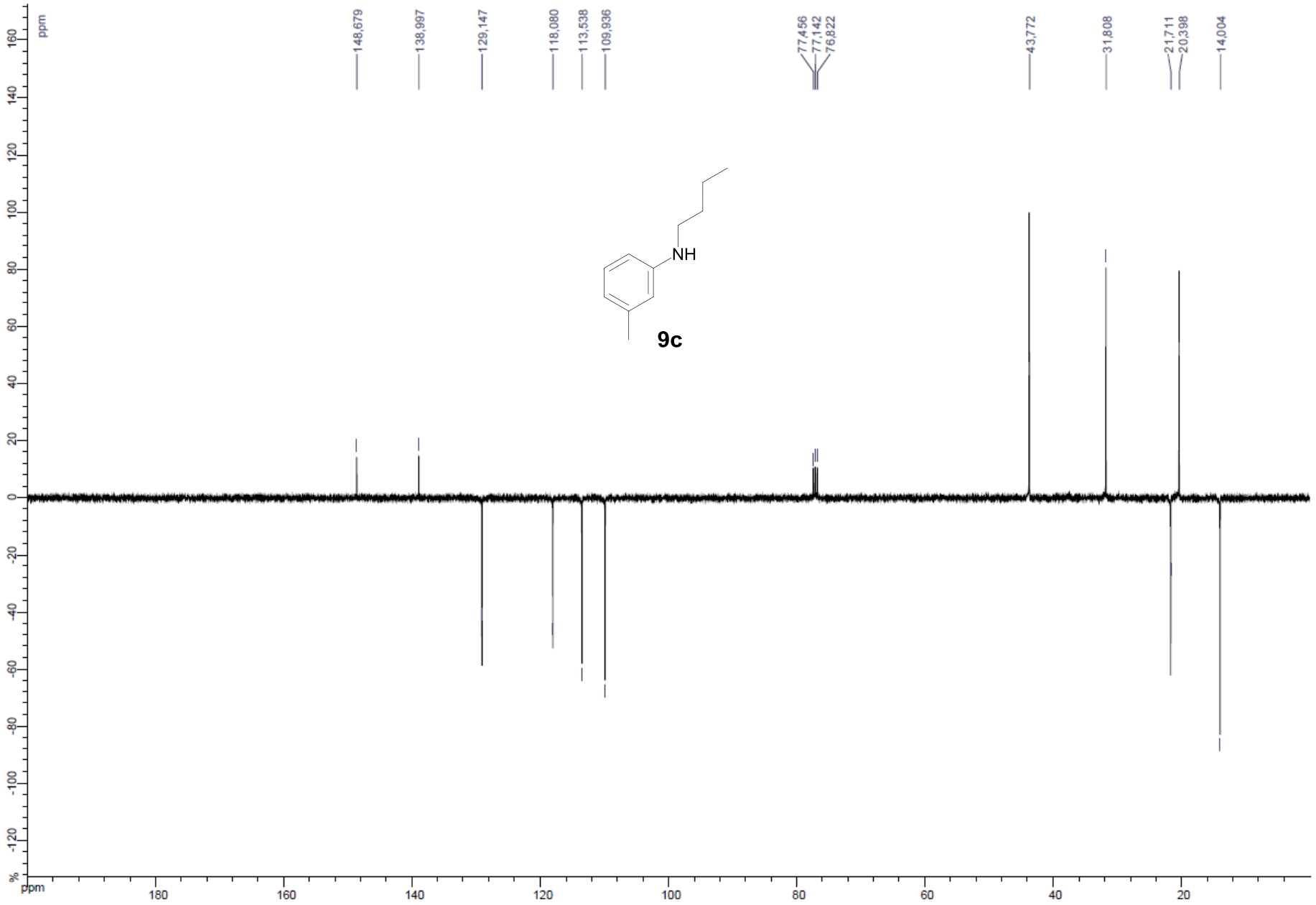

ppm
7,410
7,388
7,370
7,350
7,318
7,301
7,272
7,112
7,093
7,074
6,568
6,505
6,492
6,472
4,345
3,987
2,300
HN
9a
5,0
1,0
1,0
1,9
2,0
0,9
3,1
pm
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0

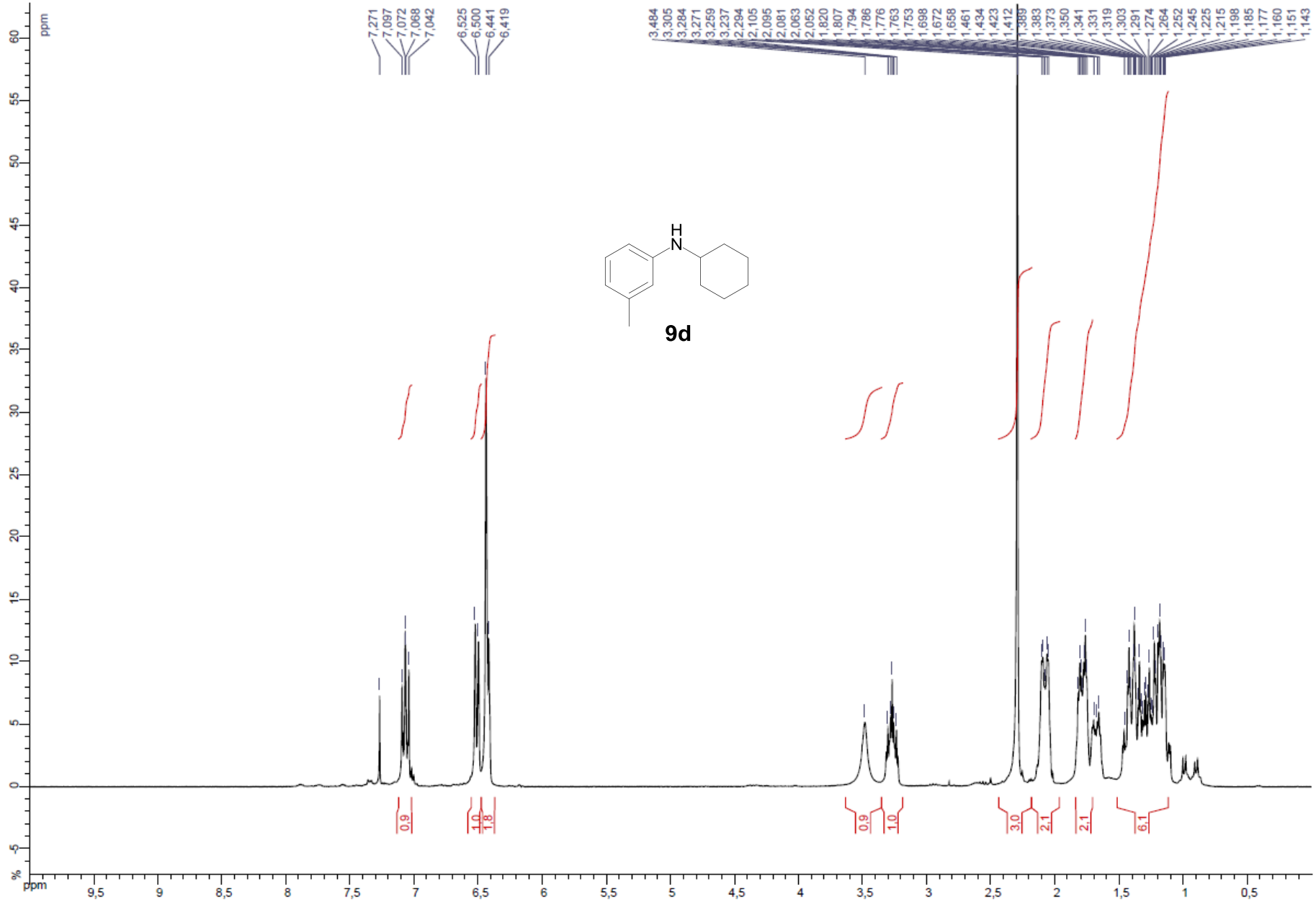

ppm
146,257
139,581
129,169
128,637
127,558
127,215
118,554
113,655
109,987
77,368
77,055
76,734
48,388
21,660
HN
9a
ppm
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

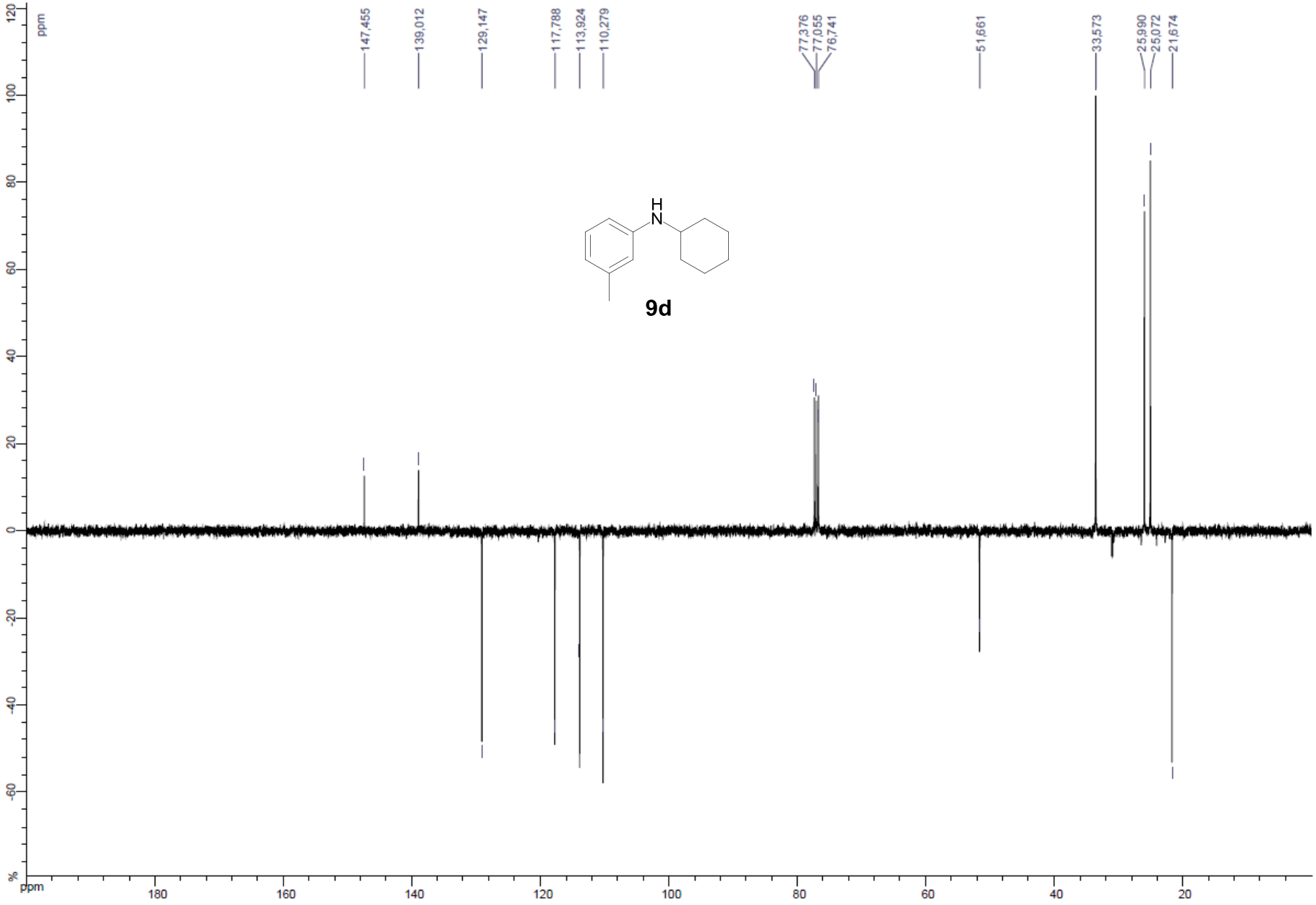

7,344
7,339
7,329
7,319
7,316
7,296
7,271
7,246
7,240
7,231
7,222
7,215
7,199
7,104
7,079
7,075
7,050
6,554
6,529
6,427
6,407
3,572
3,187
3,164
3,140
2,782
2,758
2,731
2,300
2,291
2,017
1,993
1,970
1,967
1,944
1,920
1,564
NH
9b
2,0
2,9
1,0
1,0
1,9
1,0
2,0
2,0
3,1
2,0
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5

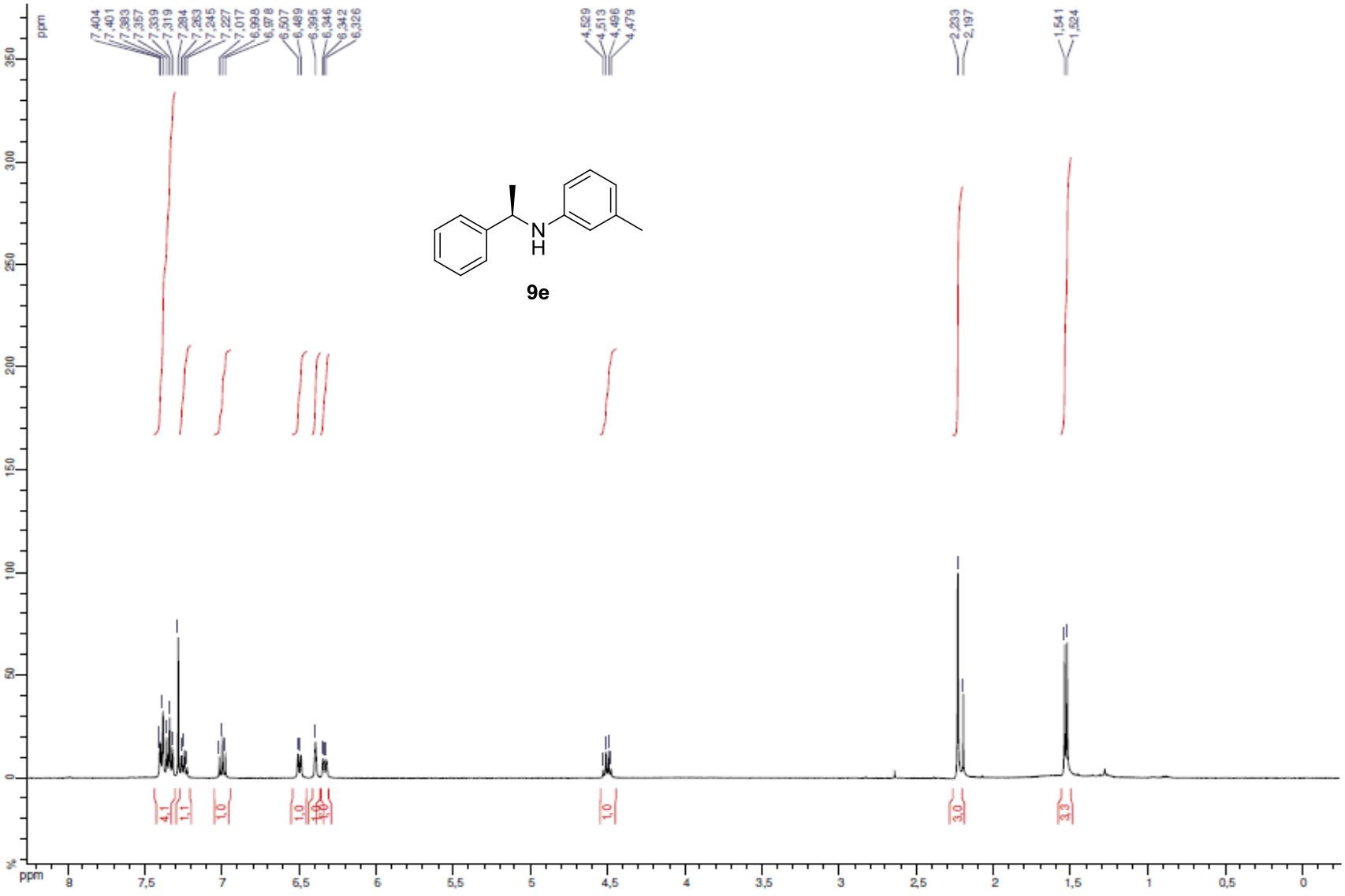

148,417
141,724
139,005
129,111
128,440
125,961
118,189
113,531
109,958
77,361
77,048
76,727
43,437
33,420
31,138
21,652
NH
9b
ppm
%

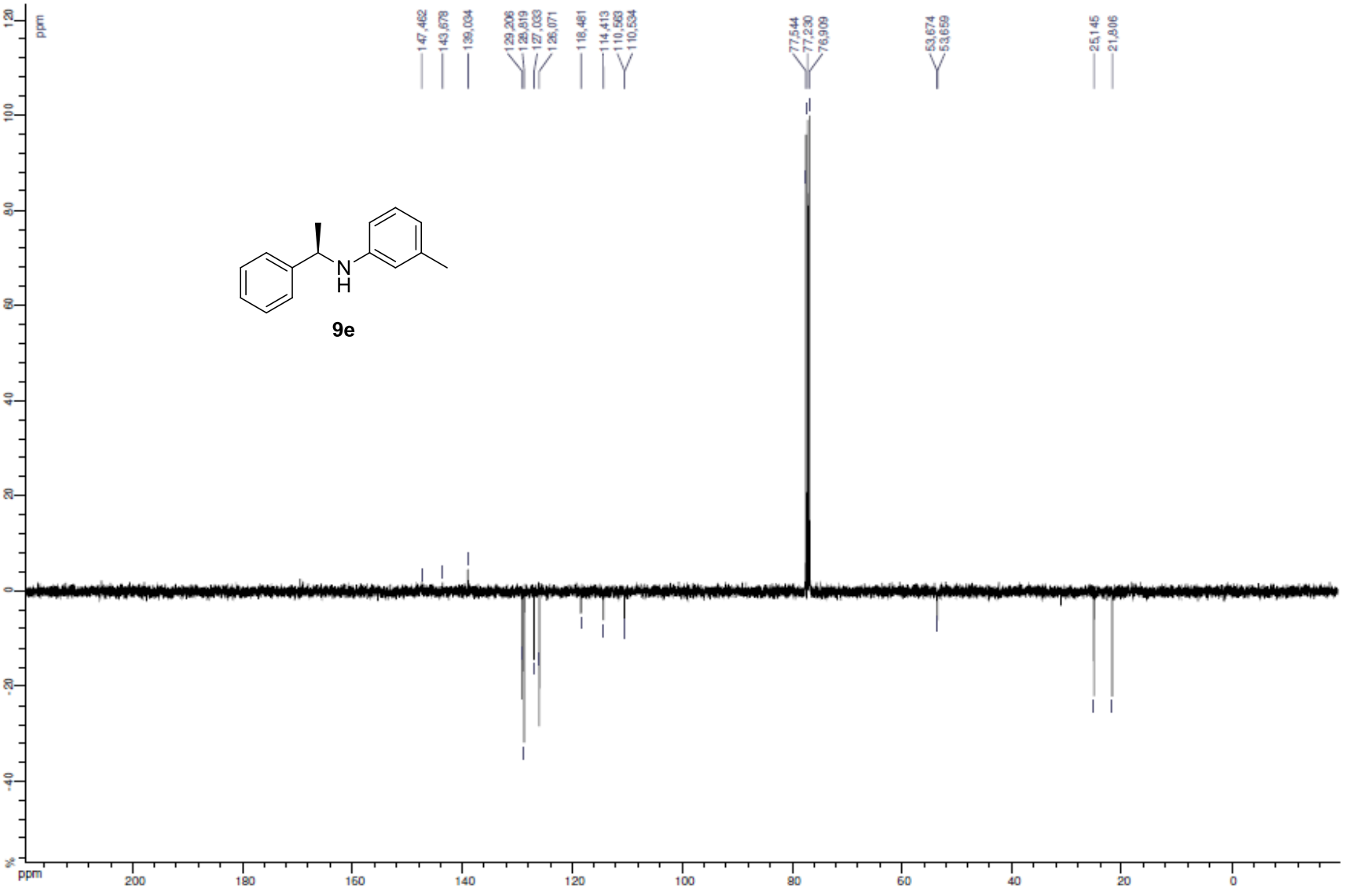

7,225
7,200
7,170
6,668
6,643
6,556
6,534
3,638
3,241
3,218
3,194
2,418
1,743
1,733
1,716
1,709
1,692
1,669
1,618
1,594
1,570
1,543
1,519
1,322
1,284
1,122
1,098
1,074
NH
9c
1,0
2,9
0,9
2,0
3,0
2,0
2,1
3,0
ppm

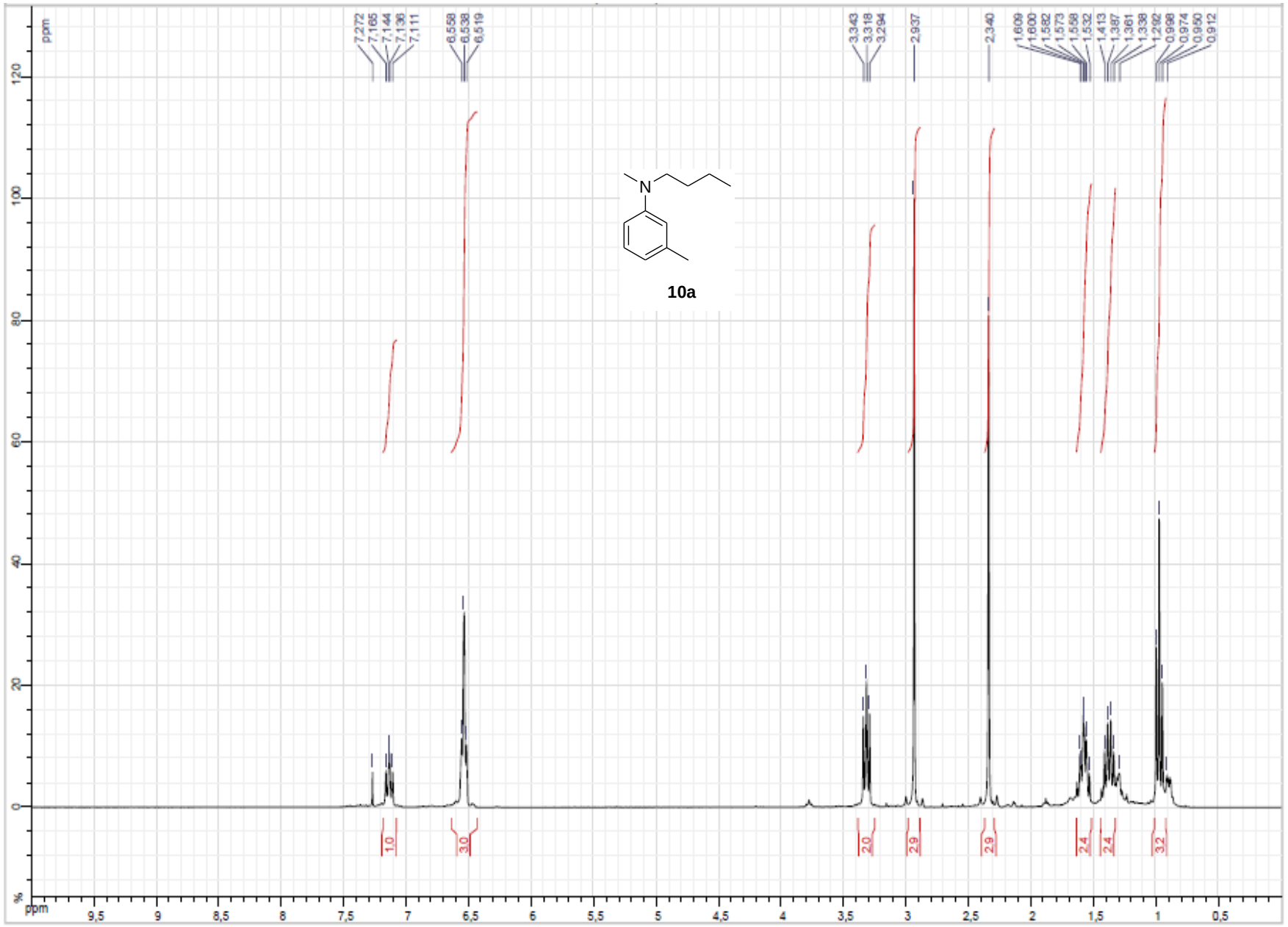

148,679
138,997
129,147
118,080
113,538
109,936
77,456
77,142
76,822
43,772
31,808
21,711
20,398
14,004
NH
9c
ppm
180
160
140
120
100
80
60
40
20

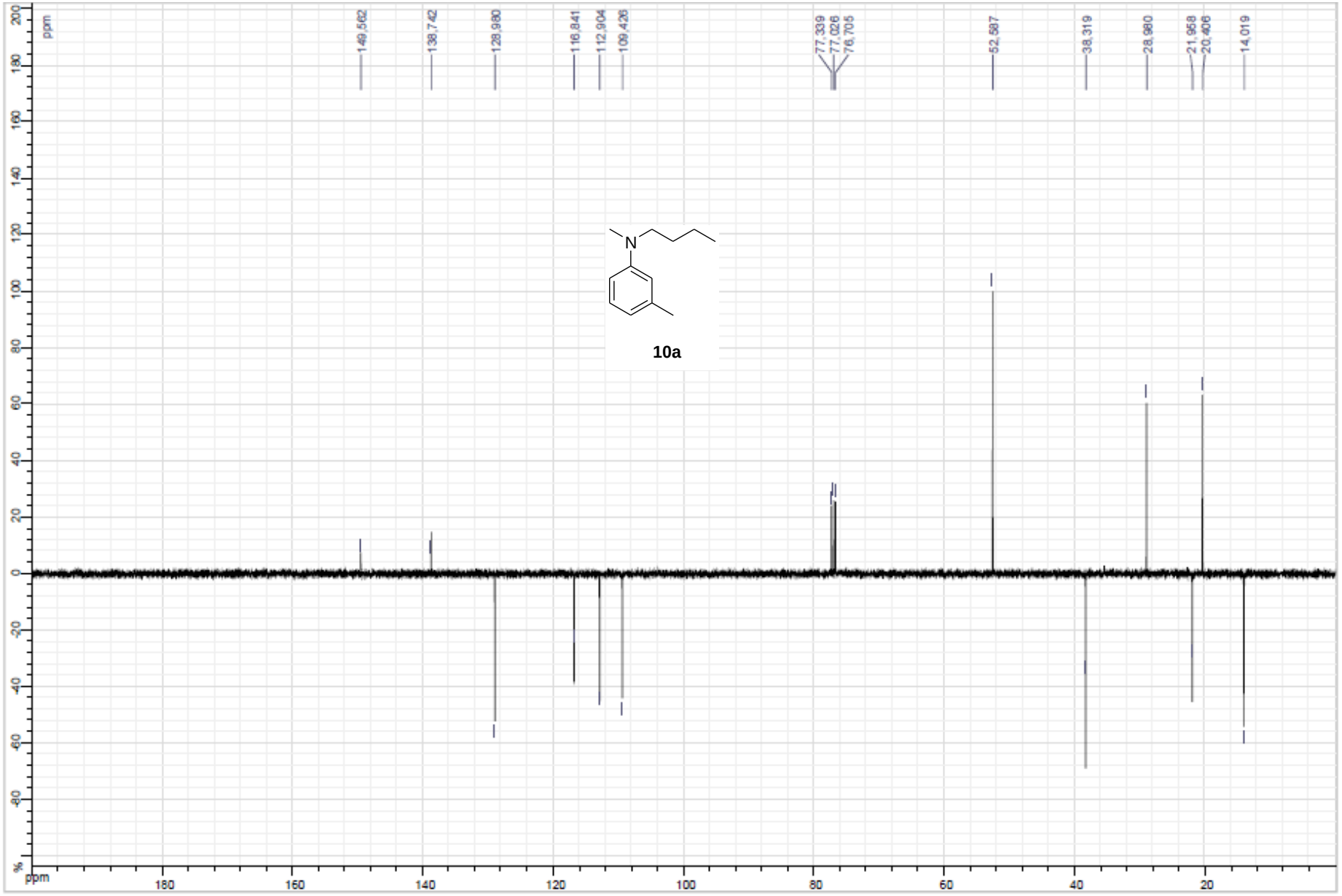

9d
7,271
7,097
7,072
7,068
7,042
6,525
6,500
6,441
6,419
0,9
1,0
1,8
0,9
1,0
3,0
2,1
2,1
6,1
ppm

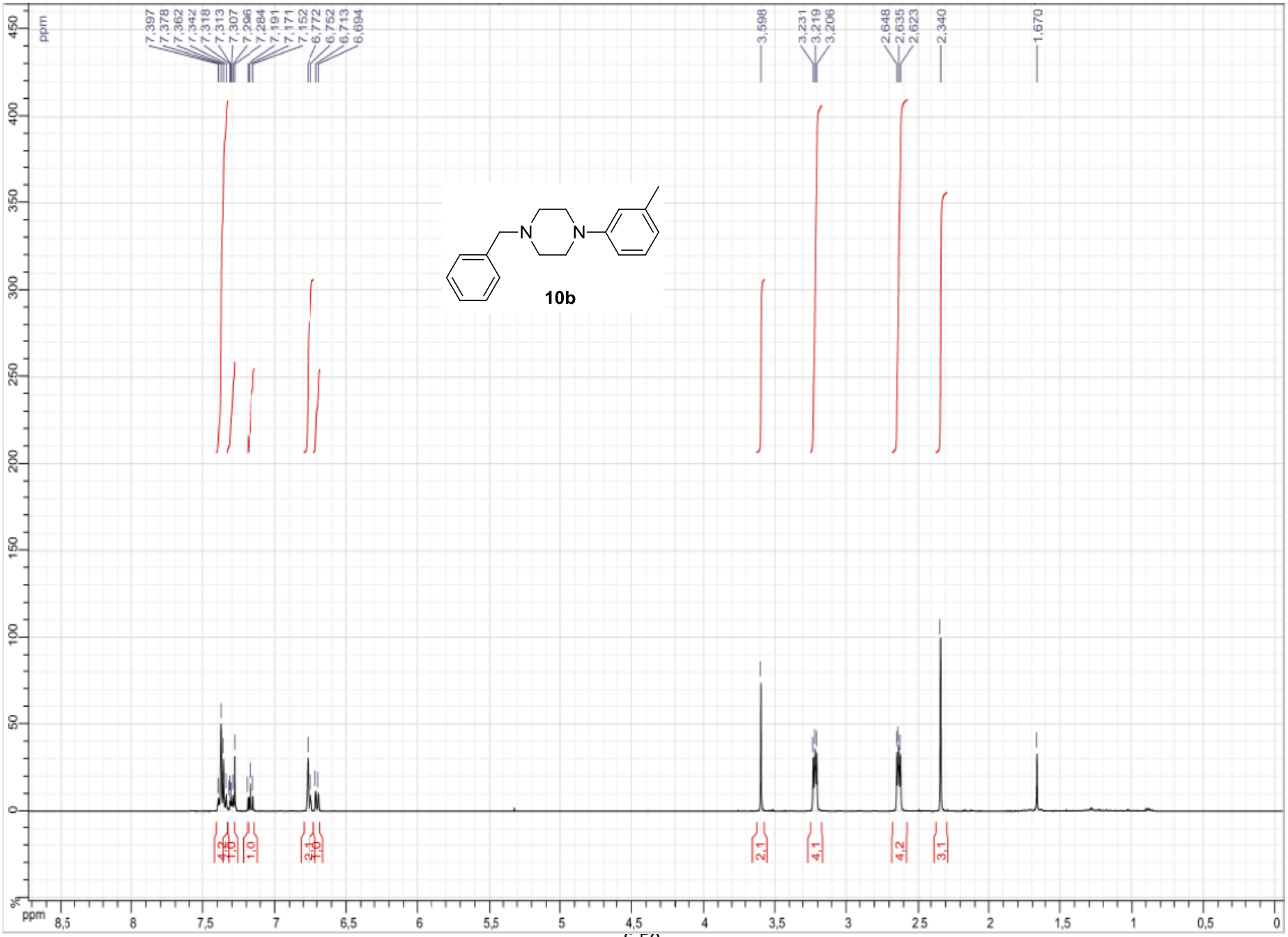

147,455
139,012
129,147
117,788
113,924
110,279
77,376
77,055
76,741
51,661
33,573
25,990
25,072
21,674
ppm
9d

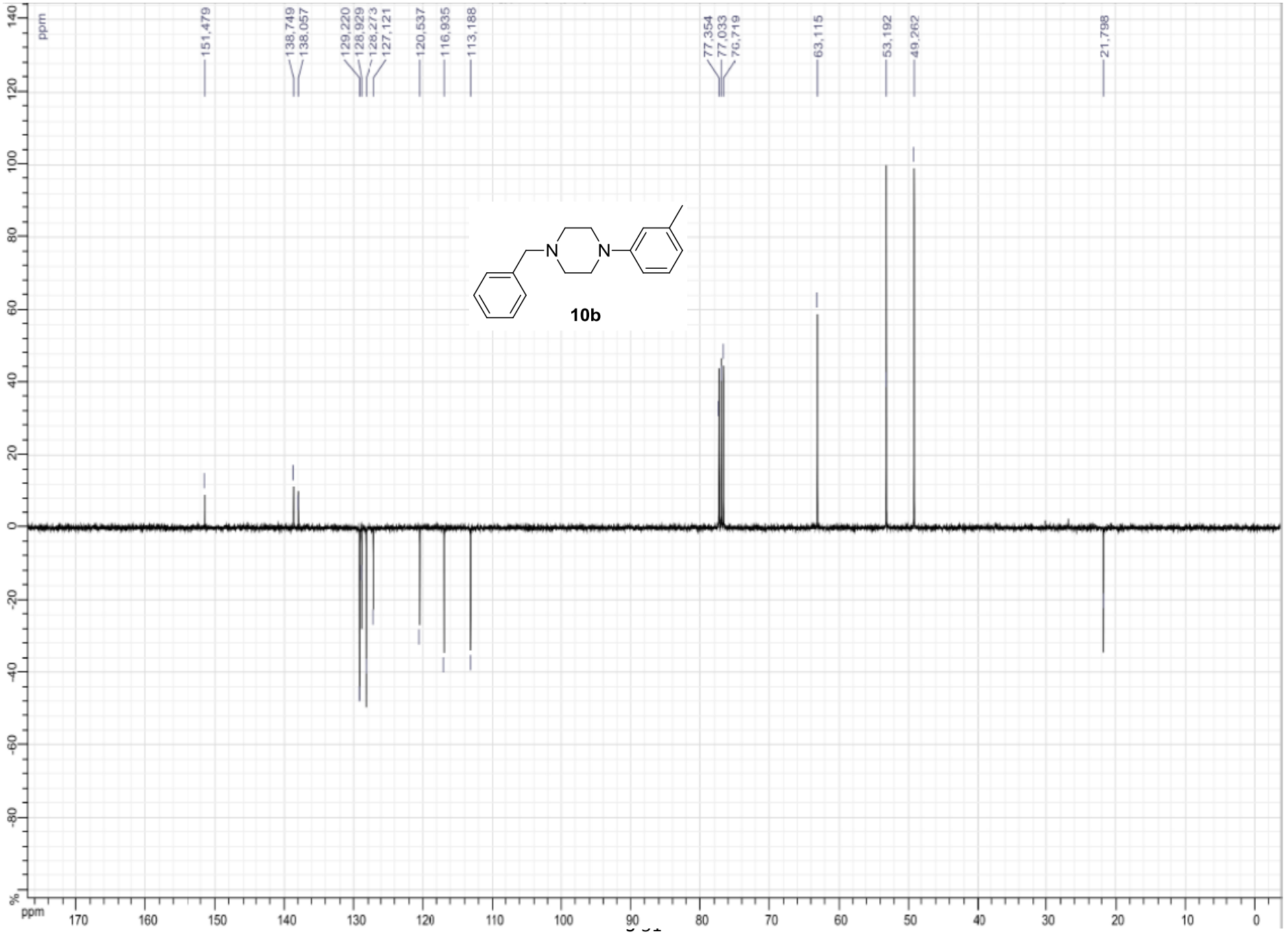

7,404
7,401
7,383
7,357
7,339
7,319
7,284
7,263
7,245
7,227
7,017
6,998
6,978
6,507
6,489
6,395
6,346
6,342
6,326
4,529
4,513
4,496
4,479
2,233
2,197
1,541
1,524
N
H
9e
ppm
4,1
1,1
1,0
1,0
1,0
1,0
3,0
3,3

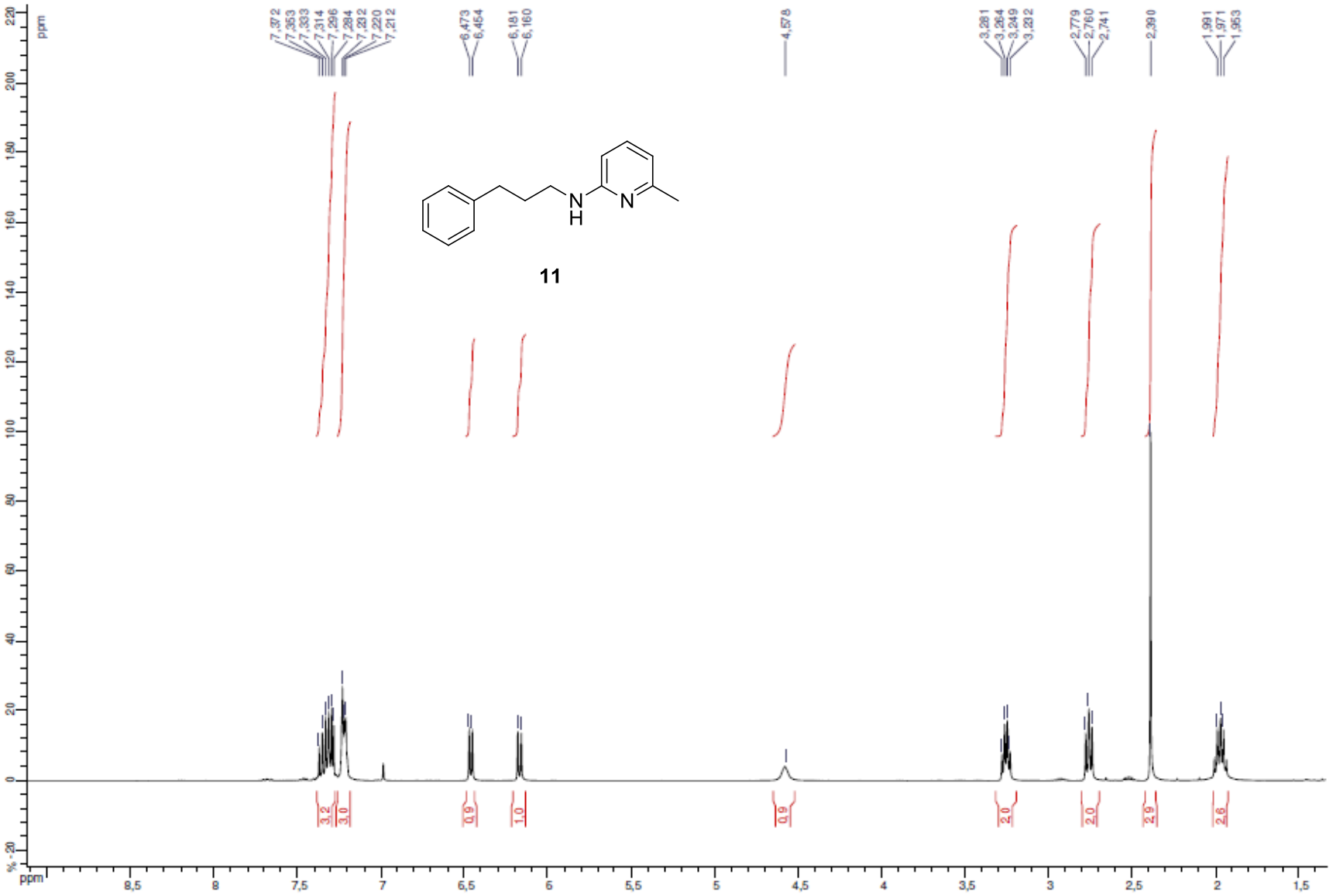

147,462
143,678
139,034
129,206
128,819
127,033
126,071
118,481
114,413
110,569
110,534
77,544
77,230
76,909
53,674
53,659
25,145
21,806
ppm
9e

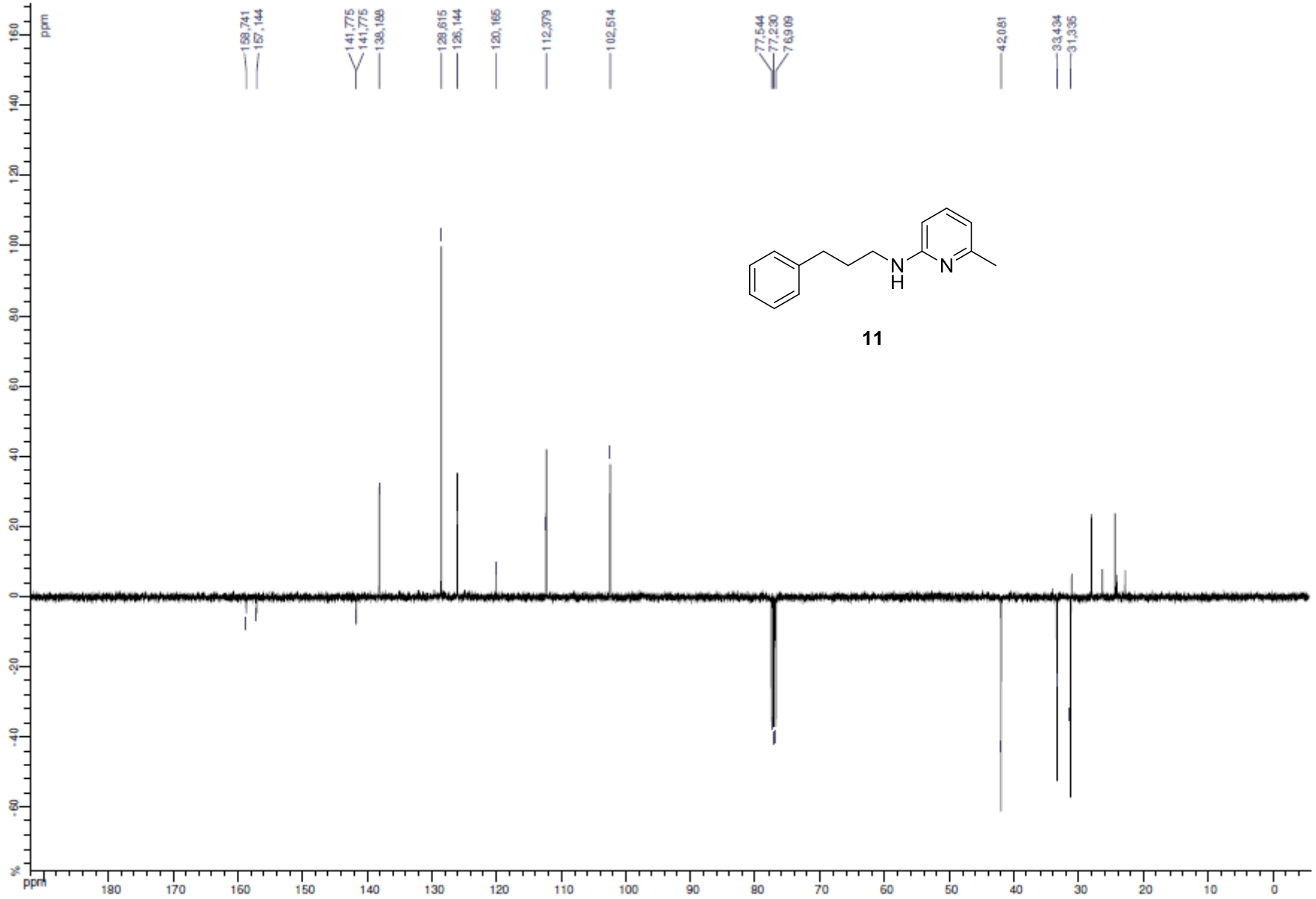

7.272
7.165
7.144
7.136
7.111
6.558
6.538
6.519
3.343
3.318
3.294
2.937
2.340
1.609
1.600
1.582
1.573
1.558
1.532
1.413
1.387
1.361
1.338
1.292
0.998
0.974
0.950
0.912
1.0
3.0
2.0
2.9
2.9
2.4
2.4
3.2
ppm
%
10a


ppm
149,562
138,742
128,980
116,841
112,904
109,426
77,339
77,026
76,705
52,587
38,319
28,980
21,958
20,406
14,019
N
10a
%
ppm
180
160
140
120
100
80
60
40
20

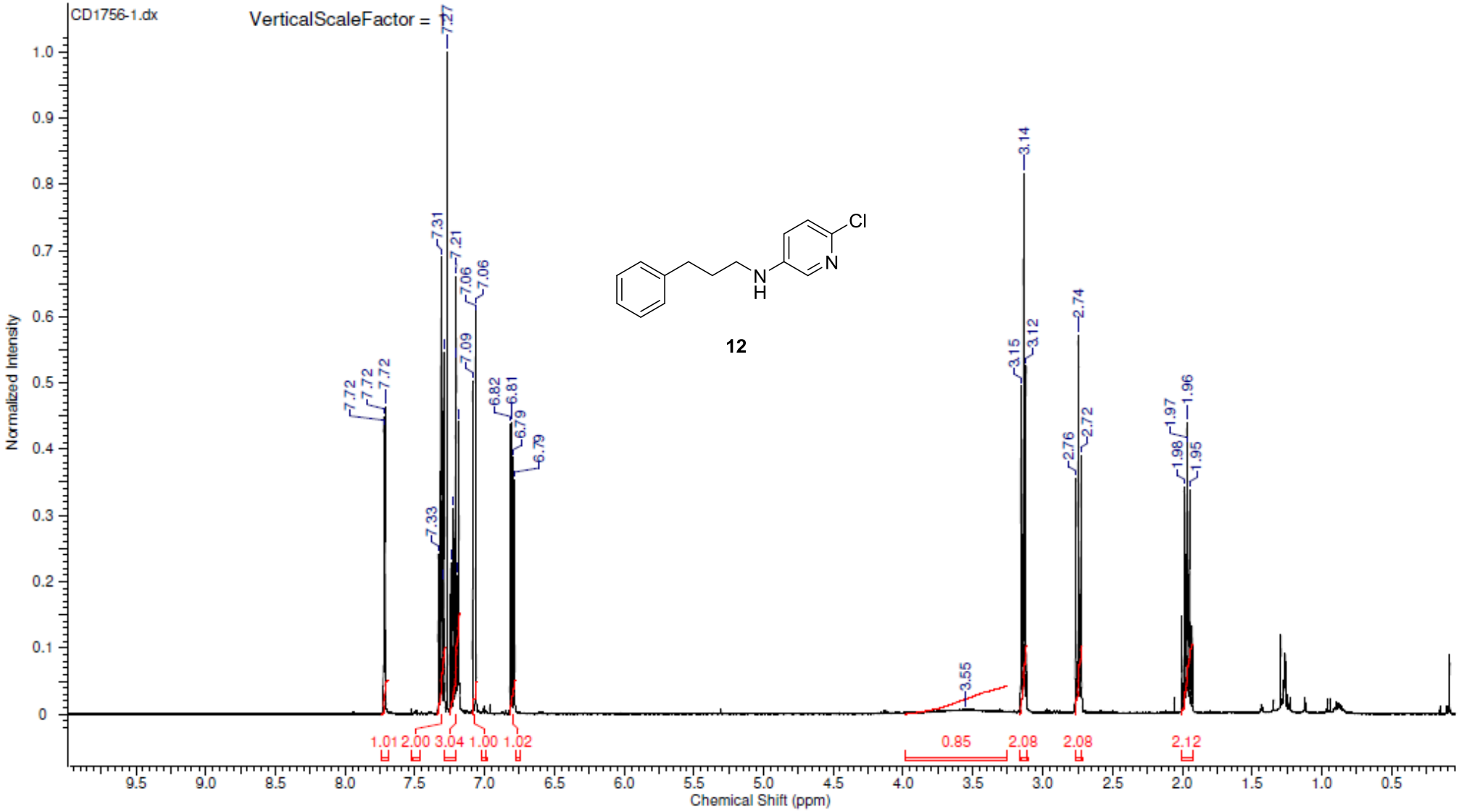

7,397
7,378
7,362
7,342
7,318
7,313
7,307
7,296
7,284
7,191
7,171
7,152
6,772
6,752
6,713
6,694
3,598
3,231
3,219
3,206
2,648
2,635
2,623
2,340
1,670
10b
ppm


151,479
138,749
138,057
129,220
128,929
128,273
127,121
120,537
116,935
113,188
77,354
77,033
76,719
63,115
53,192
49,262
21,798
10b
ppm

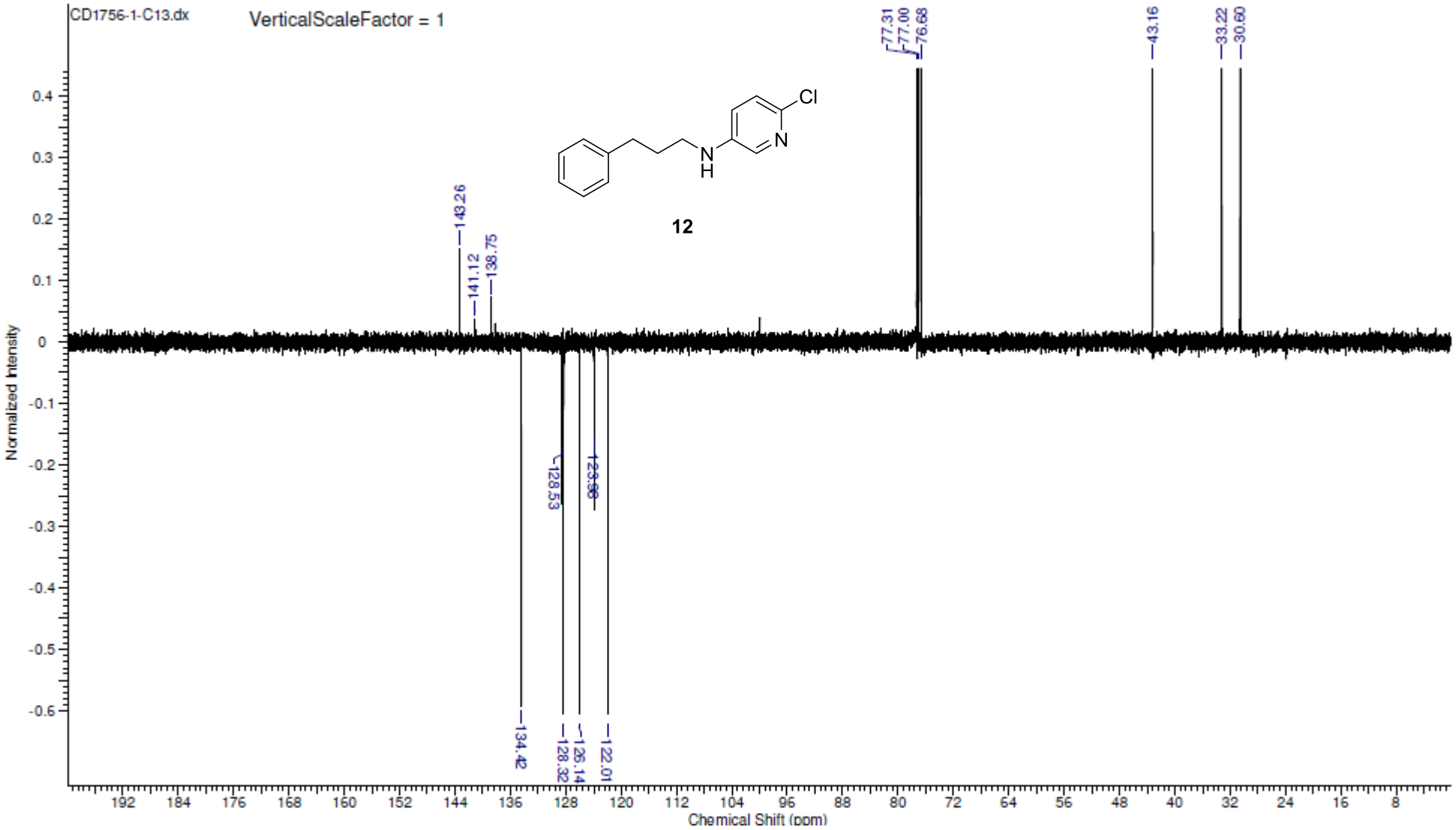

7,372
7,353
7,333
7,314
7,296
7,284
7,232
7,220
7,212
6,473
6,454
6,181
6,160
4,578
3,281
3,264
3,249
3,232
2,779
2,760
2,741
2,390
1,991
1,971
1,953
N
H
N
11
ppm
3,2
3,0
0,9
1,0
0,9
2,0
2,0
2,9
2,6

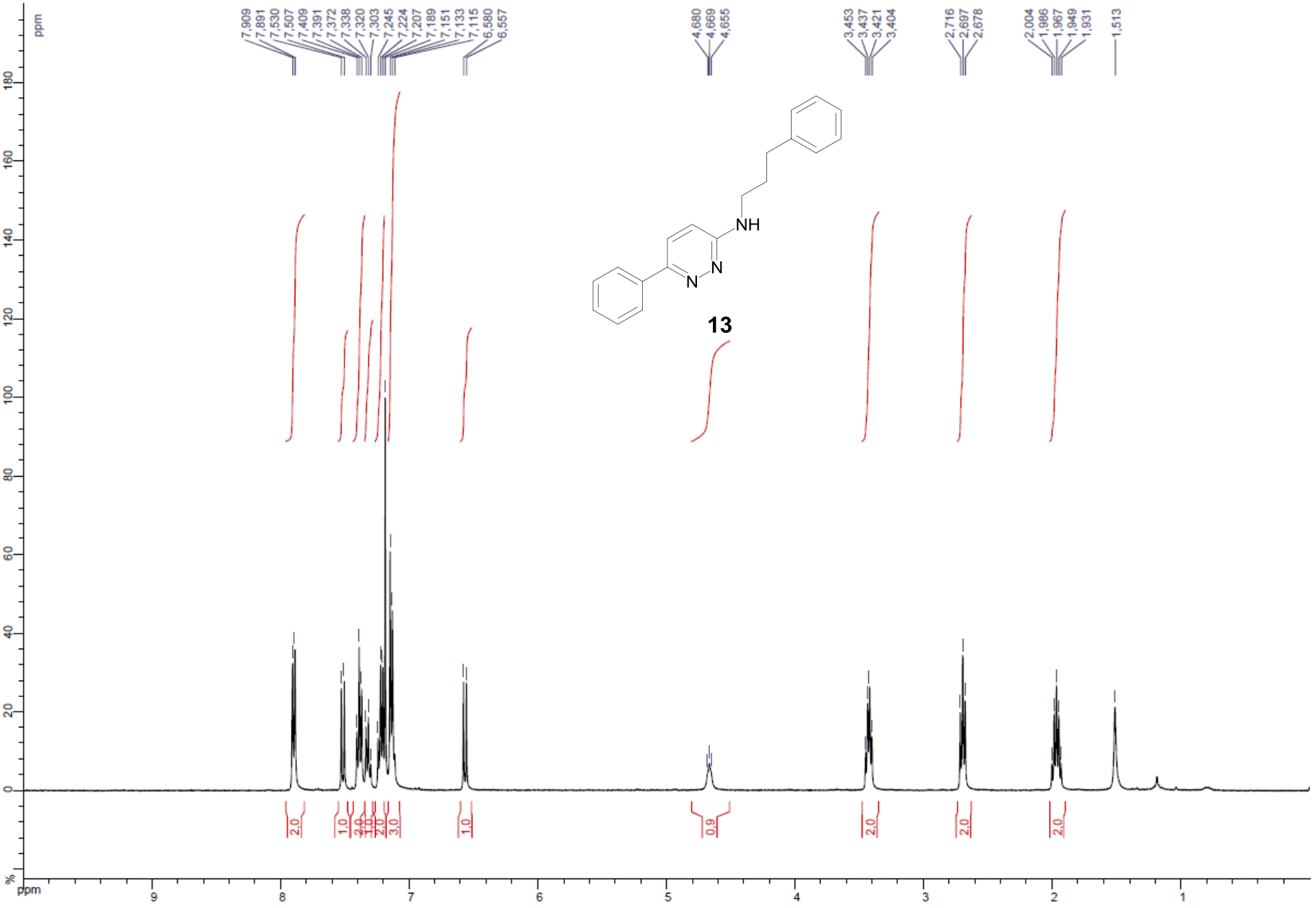

158,741
157,144
141,775
141,775
138,198
129,615
128,144
120,165
112,379
102,514
77,544
77,230
76,909
42,081
33,434
31,335
ppm
N
H
N

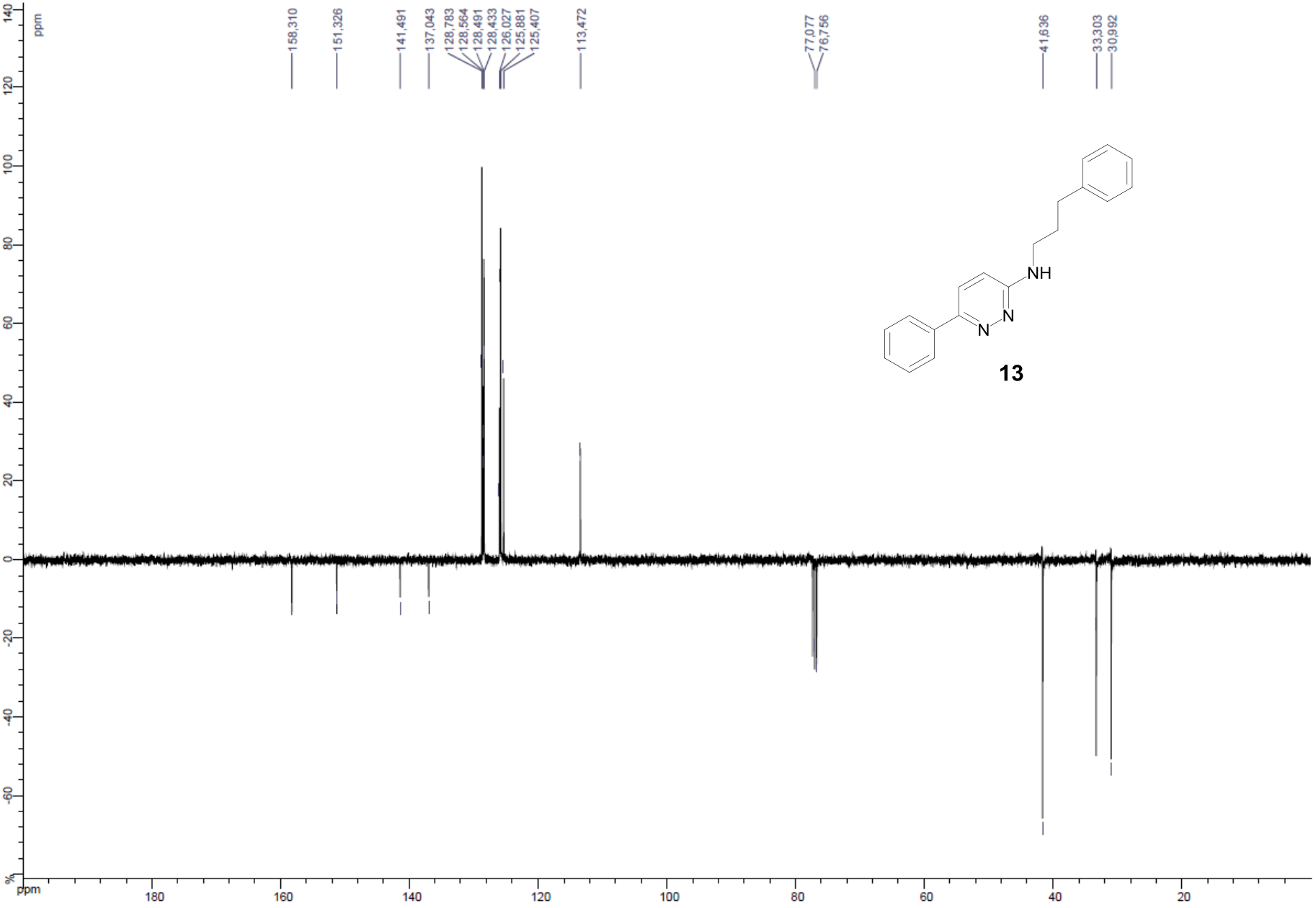
**11**

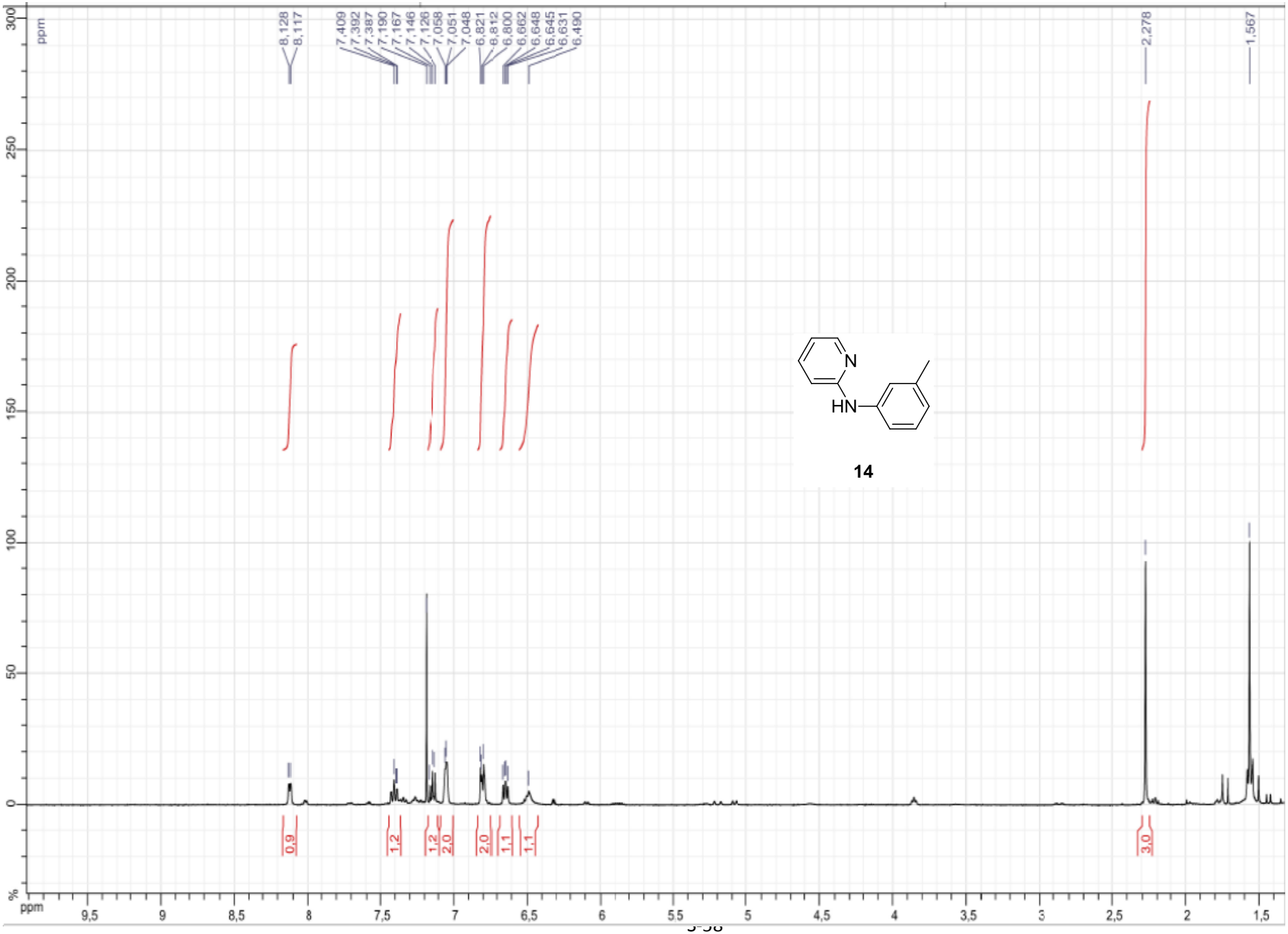

CD1756-1.dx
VerticalScaleFactor = 7.27
Normalized Intensity
Chemical Shift (ppm)
7.72
7.72
7.72
7.33
7.31
7.21
7.06
7.06
7.09
6.82
6.81
6.79
6.79
3.55
3.15
3.14
3.12
2.76
2.74
2.72
1.98
1.97
1.96
1.95
1.01
2.00
3.04
1.00
1.02
0.85
2.08
2.08
2.12
Cl
N
H
N

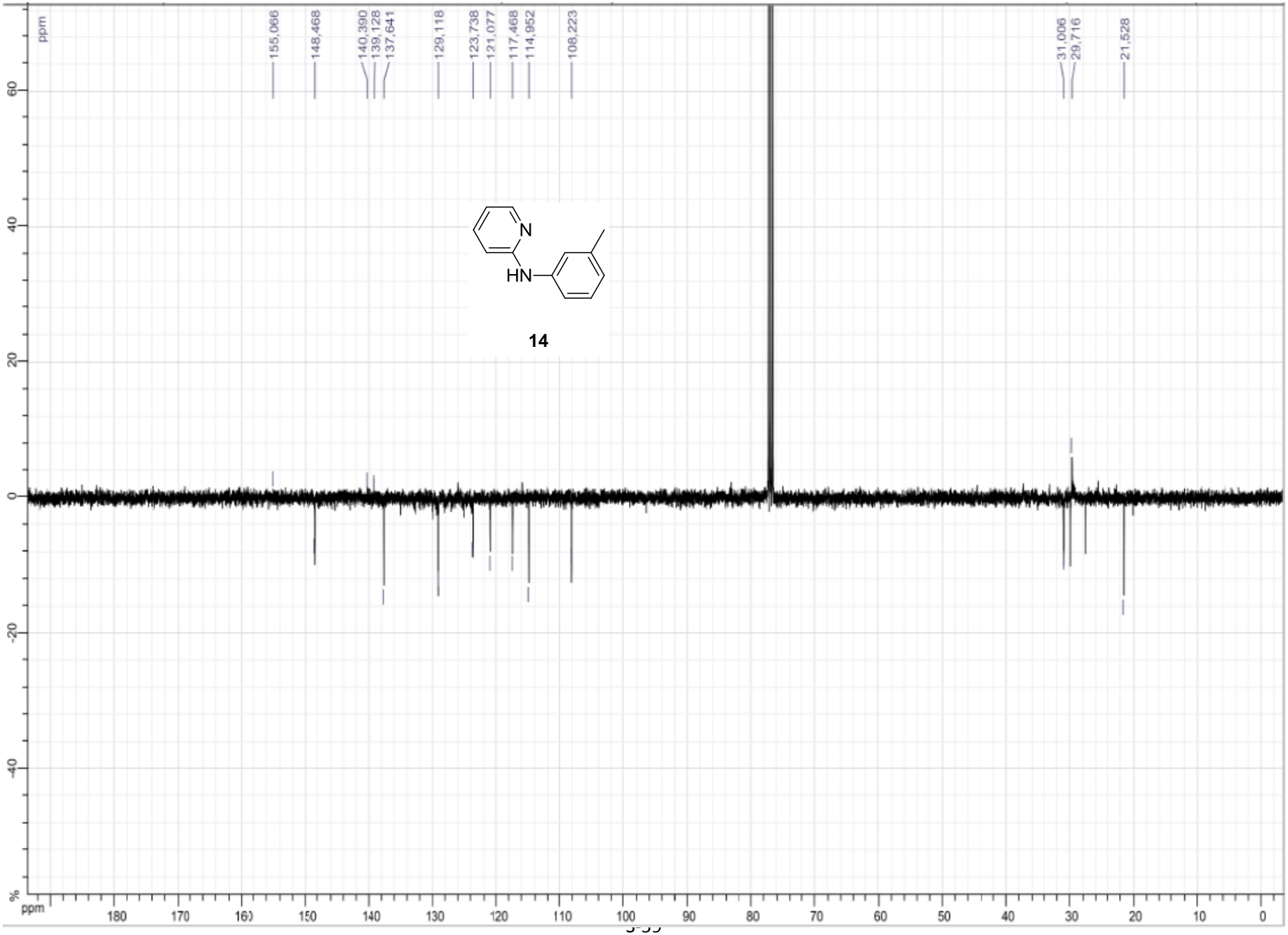
**12**

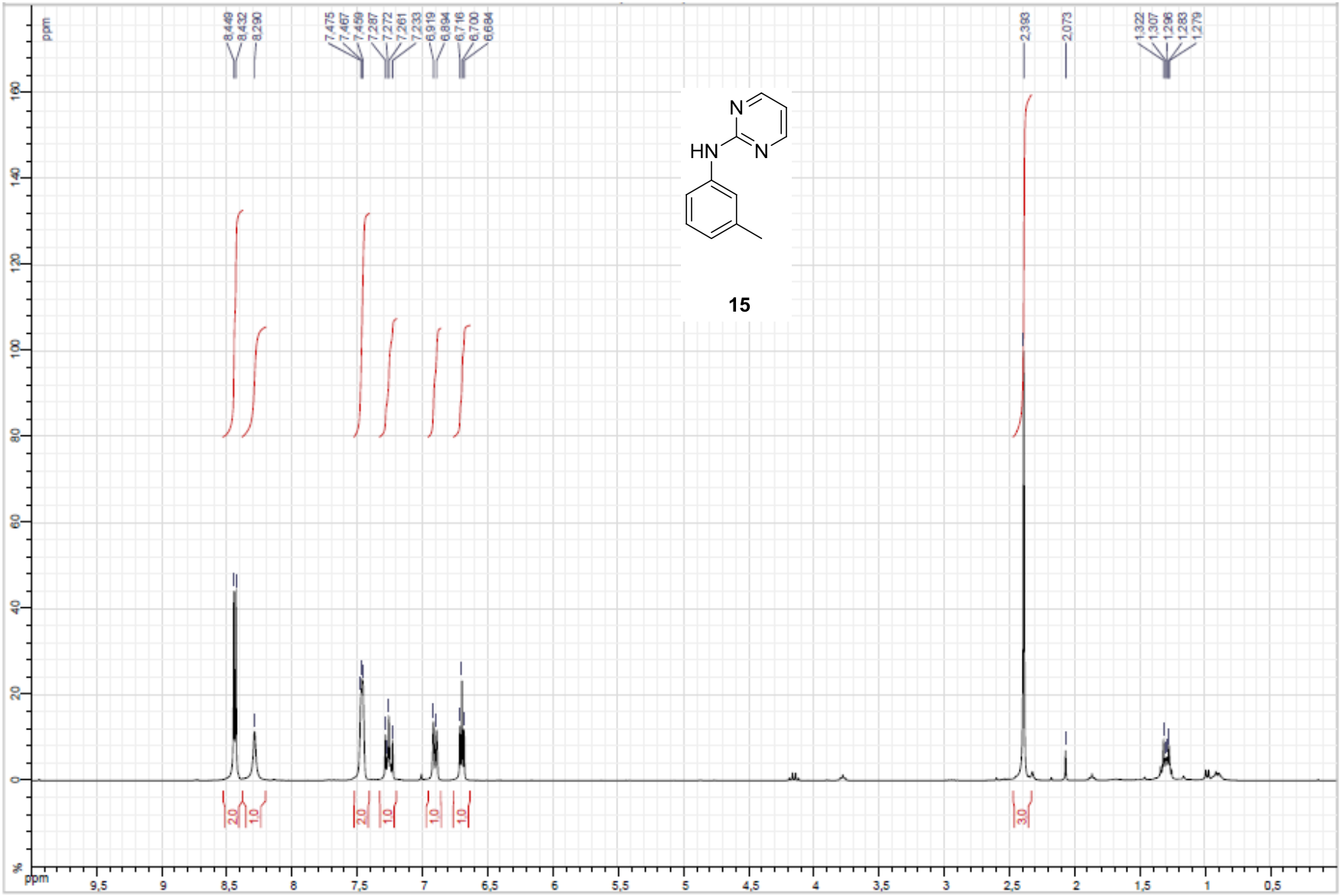

CD1756-1-C13.dx
VerticalScaleFactor = 1
Cl
N
N
H
12
143.26
141.12
138.75
134.42
128.53
128.32
126.14
123.68
122.01
77.31
77.00
76.68
43.16
33.22
30.60
Normalized Intensity
Chemical Shift (ppm)

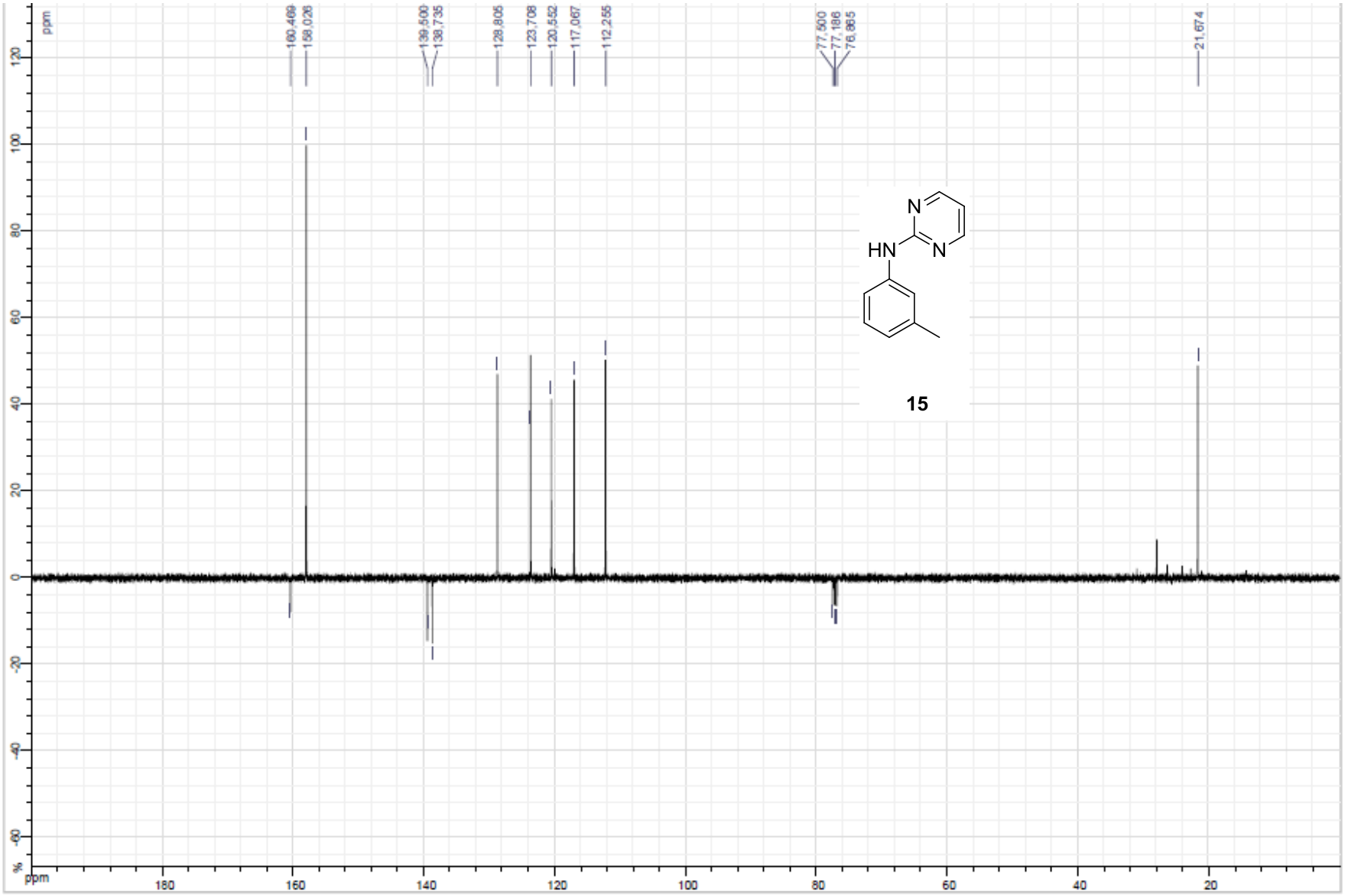

7,909
7,891
7,530
7,507
7,409
7,391
7,372
7,338
7,320
7,303
7,245
7,224
7,207
7,189
7,151
7,133
7,115
6,580
6,557
4,680
4,669
4,655
3,453
3,437
3,421
3,404
2,716
2,697
2,678
2,004
1,986
1,967
1,949
1,931
1,513
NH
N
N
13
ppm
%
2,0
1,0
2,0
1,0
2,0
3,0
1,0
0,9
2,0
2,0
2,0

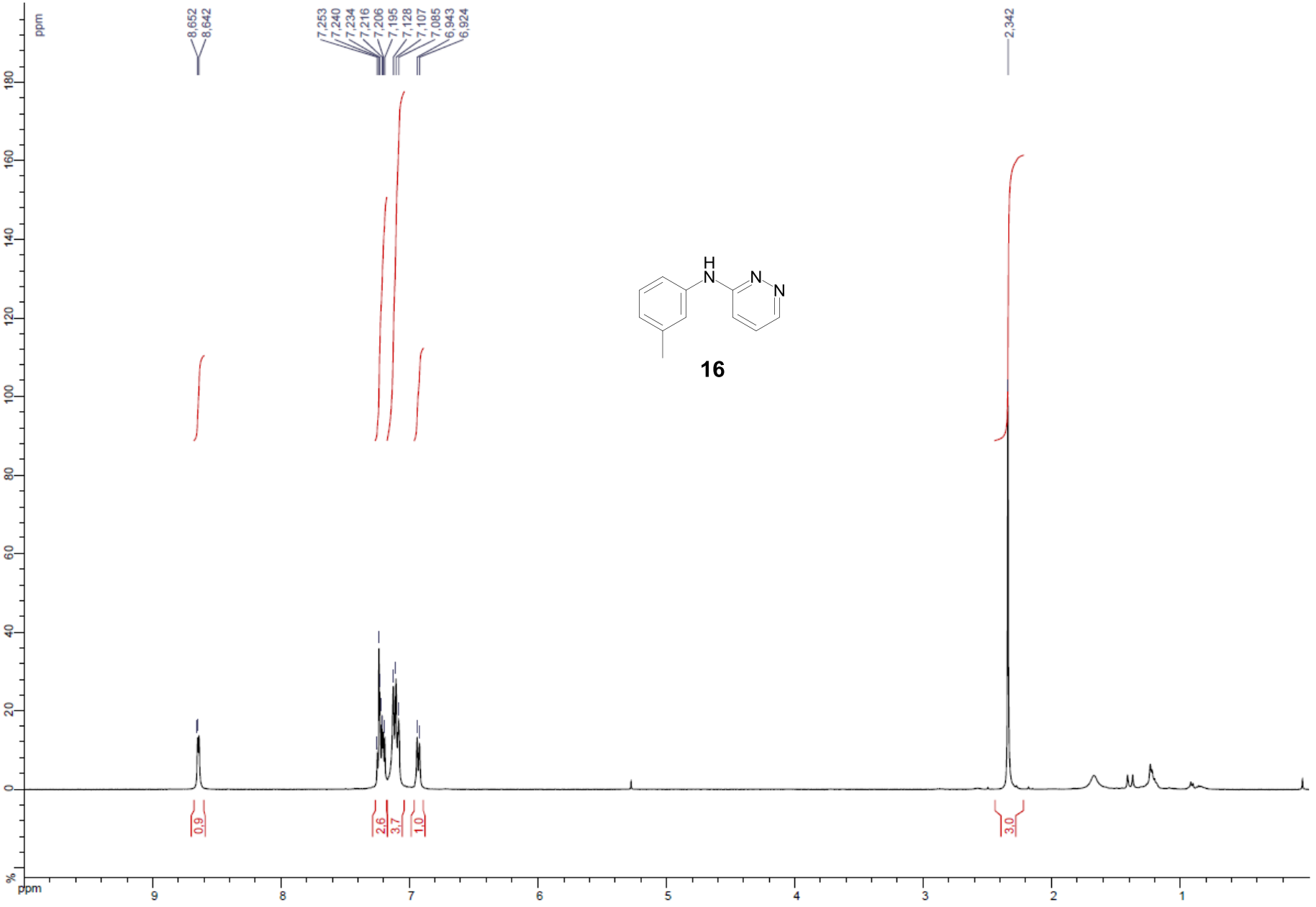

158,310
151,326
141,491
137,043
128,783
128,564
128,491
128,433
126,027
125,881
125,407
113,472
77,077
76,756
41,636
33,303
30,992
NH
N
N
13
ppm
180
160
140
120
100
80
60
40
20

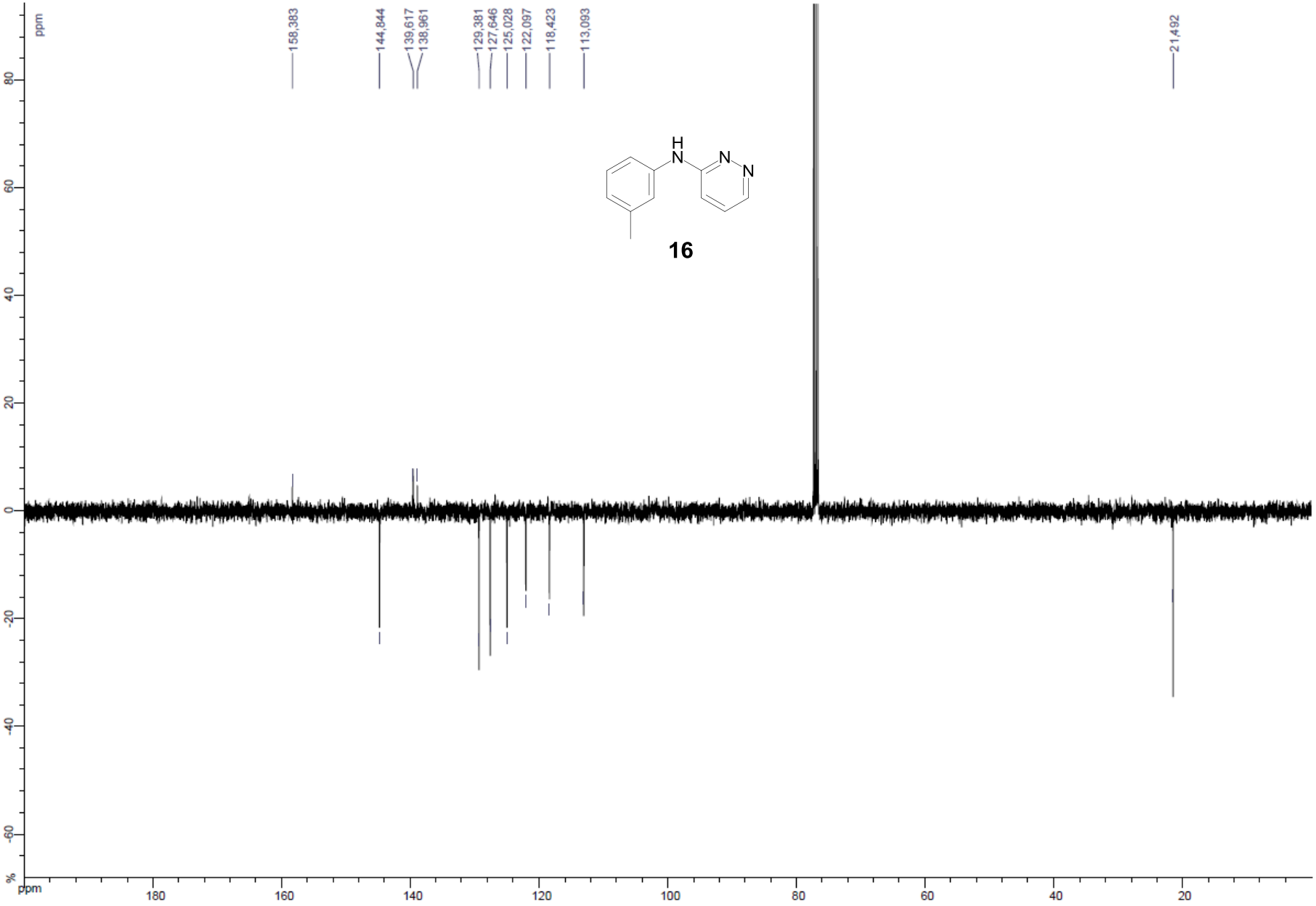

ppm
8,128
8,117
7,409
7,392
7,387
7,190
7,167
7,146
7,126
7,058
7,051
7,048
6,821
6,812
6,800
6,662
6,648
6,645
6,631
6,490
2,278
1,567
N
HN
14
0,9
1,2
1,2
2,0
2,0
1,1
1,1
3,0
%
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5

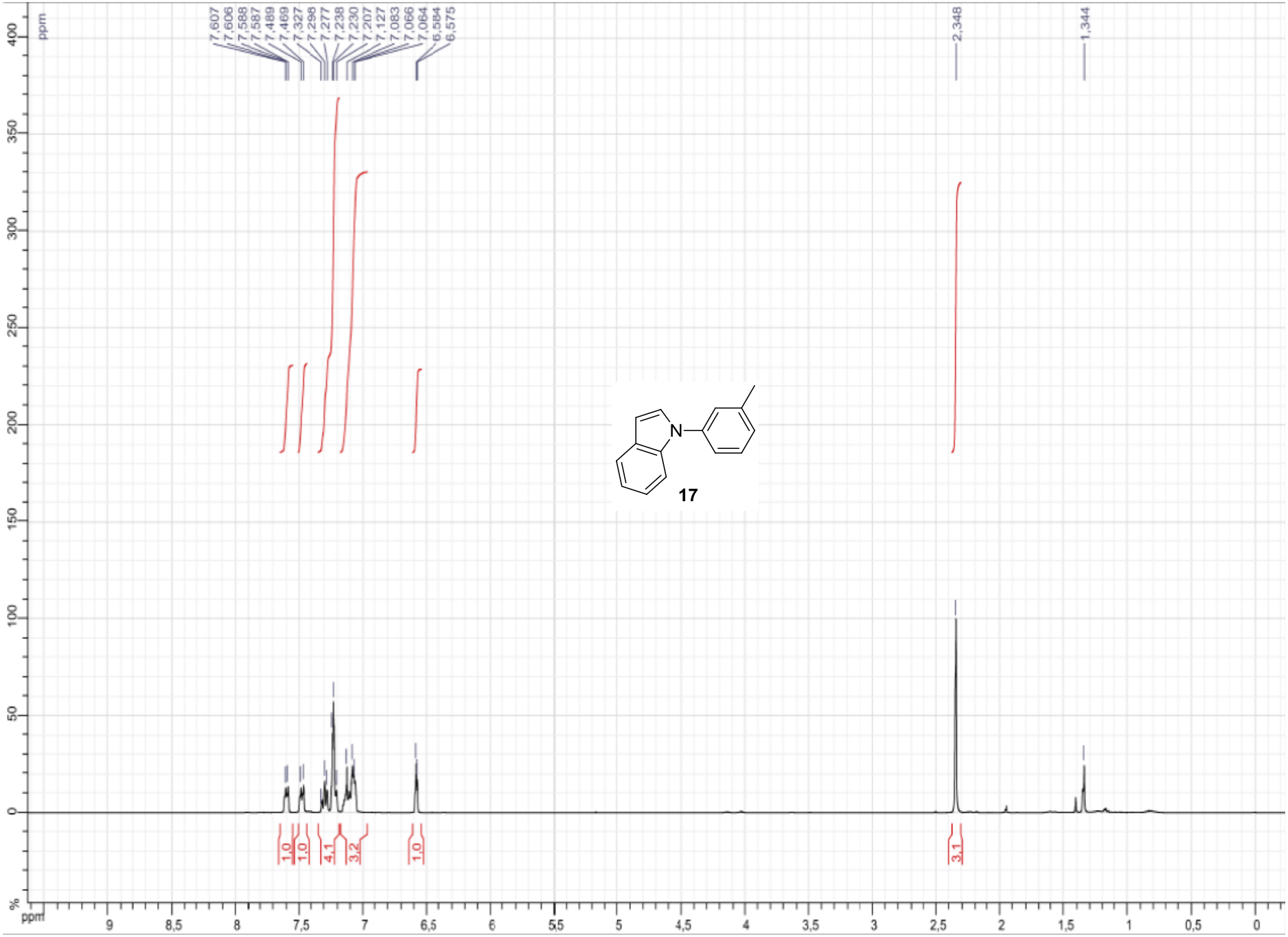

ppm
155,066
148,468
140,390
139,128
137,641
129,118
123,738
121,077
117,468
114,952
108,223
31,006
29,716
21,528
N
HN
14
60
40
20
0
-20
-40
%
ppm
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

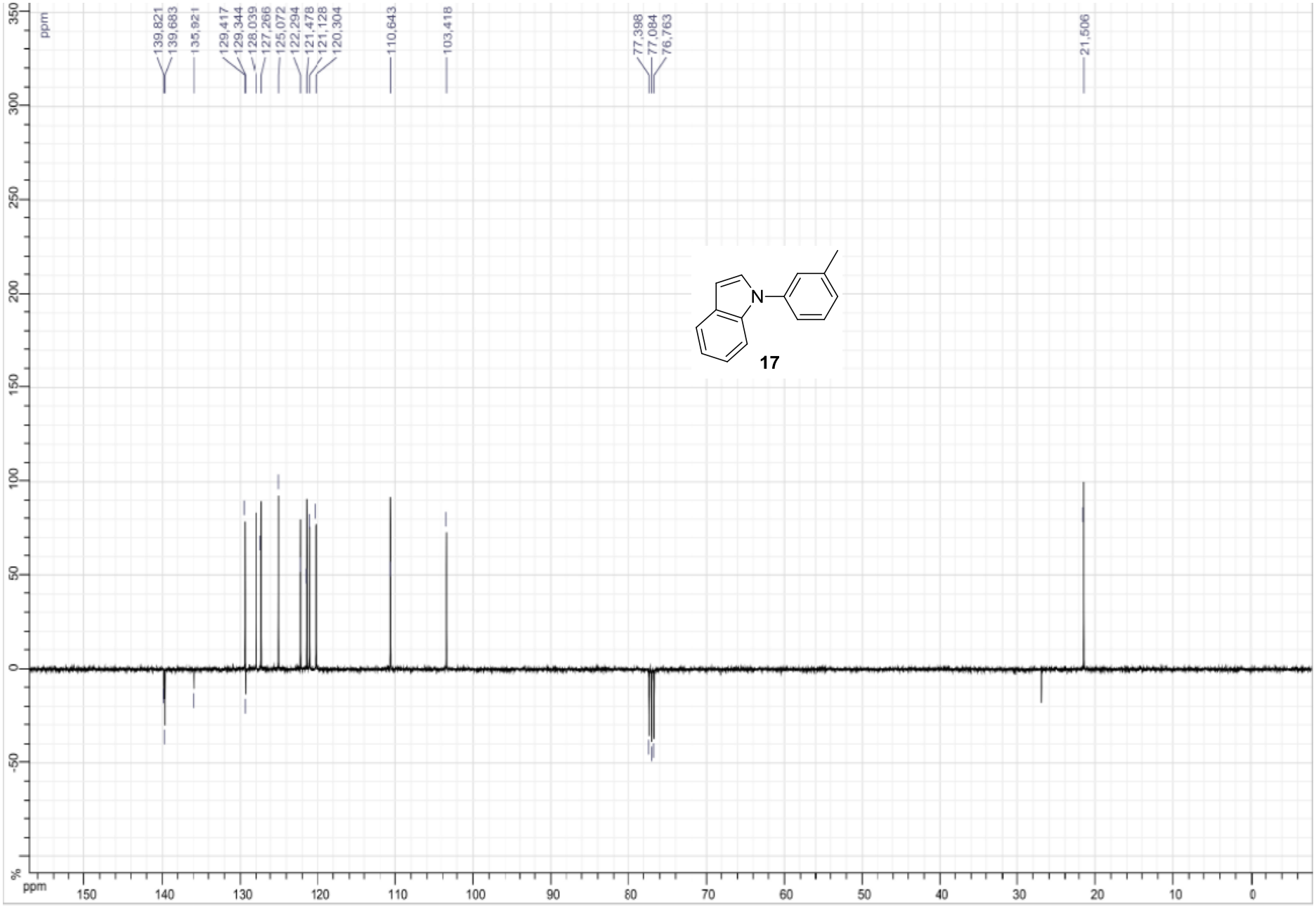

8.449
8.432
8.290
7.475
7.467
7.459
7.287
7.272
7.261
7.233
6.919
6.894
6.716
6.700
6.684
2.393
2.073
1.322
1.307
1.296
1.283
1.279
HN
N
N
15
2.0
1.0
2.0
1.0
1.0
1.0
3.0
ppm

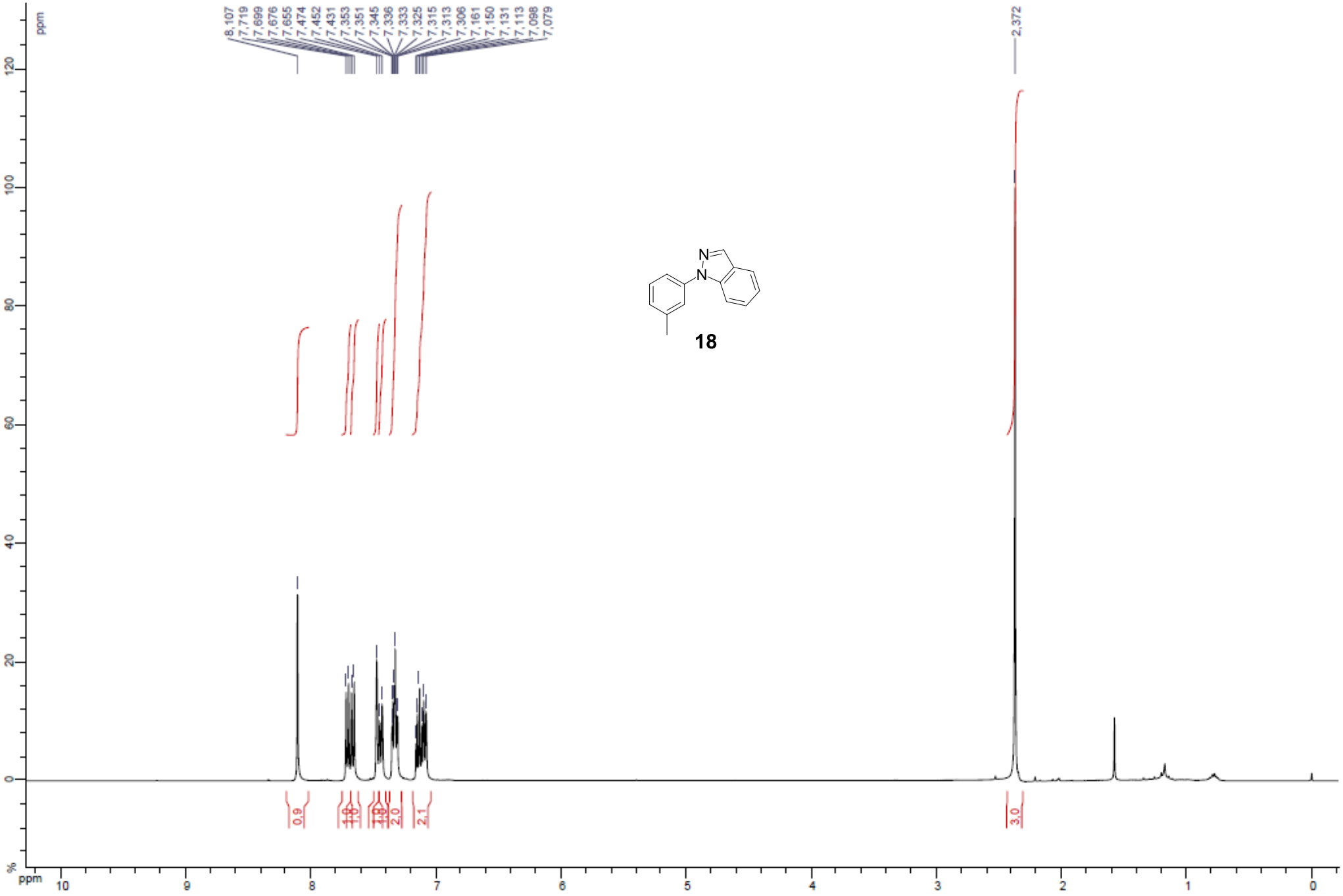

160,469
158,026
139,500
138,735
128,805
123,708
120,552
117,067
112,255
77,500
77,186
76,865
21,674
HN
N
N
15


ppm
8,652
8,642
7,253
7,240
7,234
7,216
7,206
7,195
7,128
7,107
7,085
6,943
6,924
2,342
H
N
N
N
16
0,9
2,6
3,7
1,0
3,0
%
ppm
9
8
7
6
5
4
3
2
1

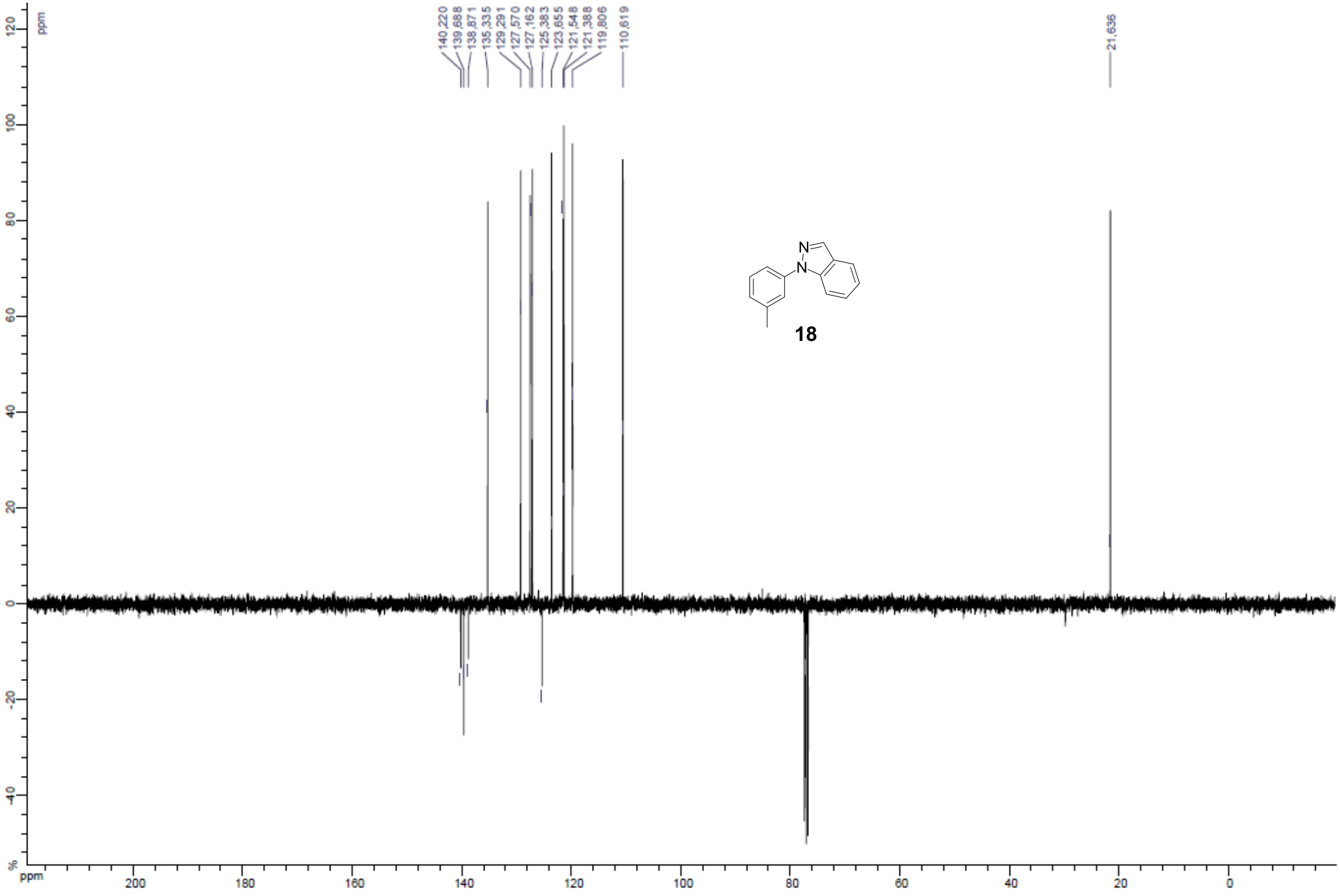

ppm
158,383
144,844
139,617
138,961
129,381
127,646
125,028
122,097
118,423
113,093
21,492
H
N
N
N
16
%
80
60
40
20
0
-20
-40
-60
ppm
180
160
140
120
100
80
60
40
20

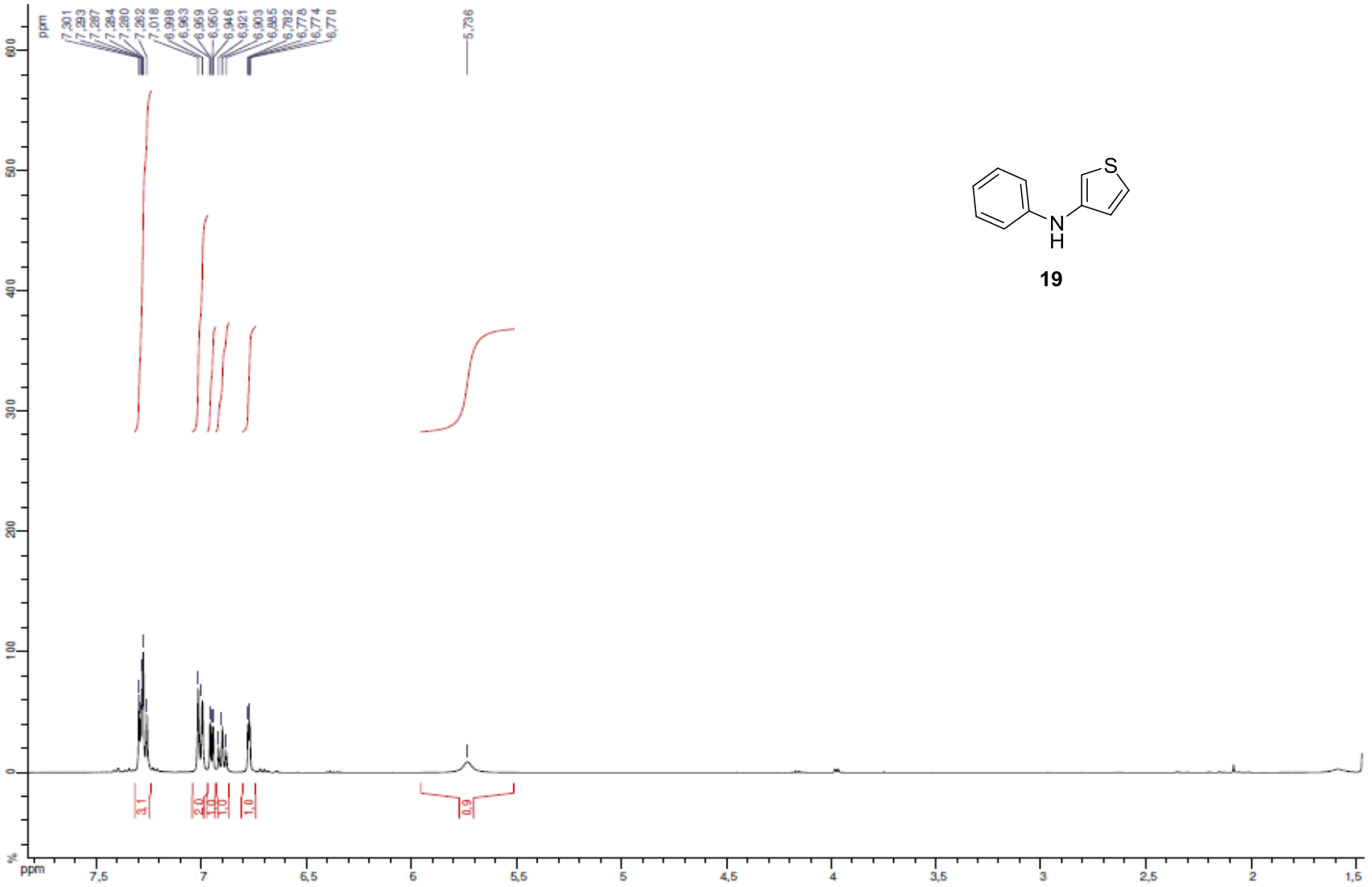

ppm
7,607
7,606
7,588
7,587
7,489
7,469
7,327
7,298
7,277
7,238
7,230
7,207
7,127
7,083
7,066
7,064
6,584
6,575
2,348
1,344
N
17
1,0
1,0
4,1
3,2
1,0
3,1
%
ppm
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5
0
400
350
300
250
200
150
100
50
0


ppm
139,821
139,683
135,921
129,417
129,344
128,039
127,266
125,072
122,294
121,478
121,128
120,304
110,643
103,418
77,398
77,084
76,763
21,506
17
350
300
250
200
150
100
50
0
-50
%
ppm
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


ppm
8,107
7,719
7,699
7,676
7,655
7,474
7,452
7,431
7,353
7,351
7,345
7,336
7,333
7,325
7,315
7,313
7,306
7,161
7,150
7,131
7,113
7,098
7,079
2,372
0.9
1.8
1.8
2.0
2.1
3.0
%
ppm
10
9
8
7
6
5
4
3
2
1
0

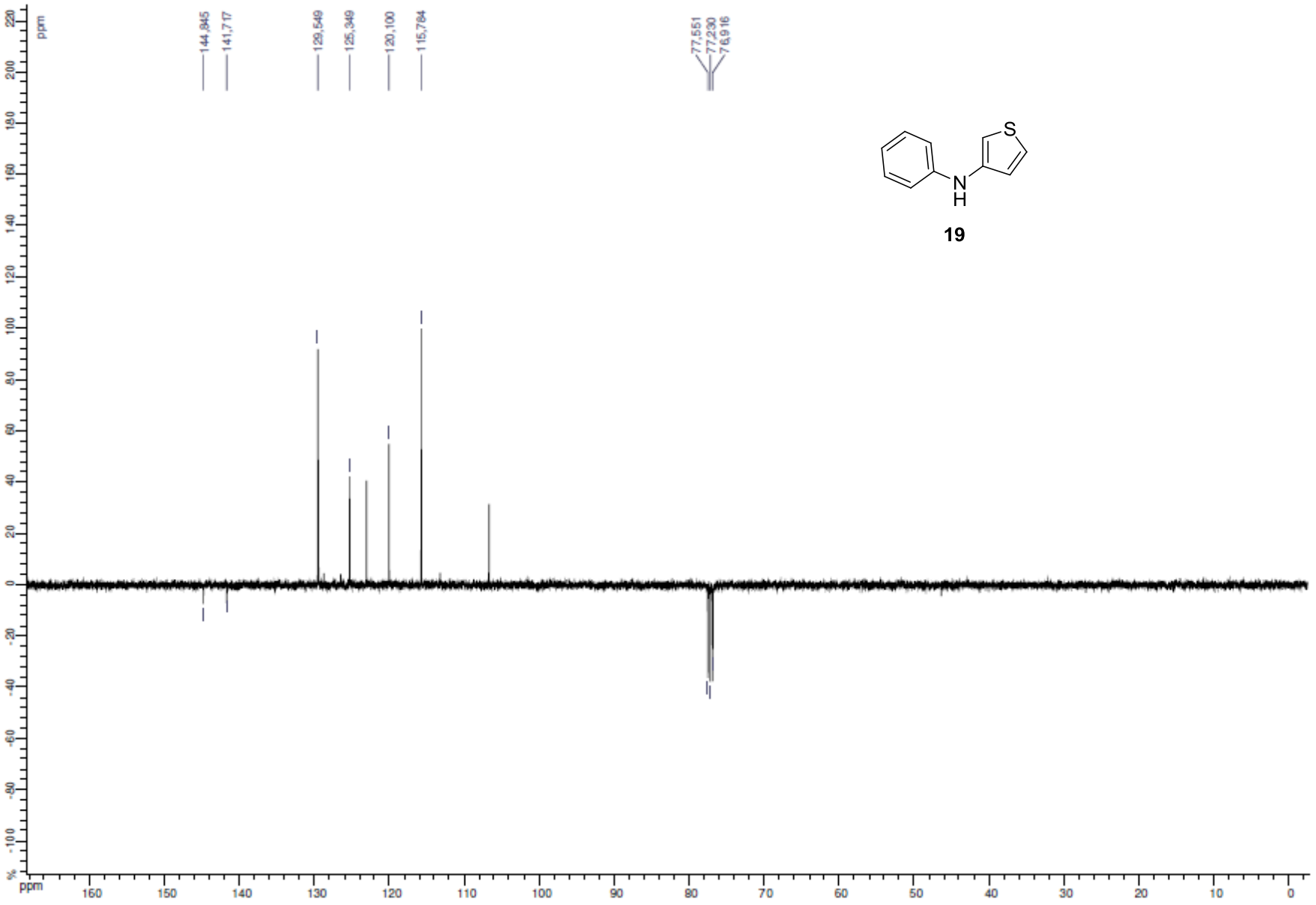
**18**


ppm
140,220
139,688
138,871
135,335
129,291
127,570
127,162
125,383
123,655
121,548
121,388
119,806
110,619
21,536
N
N
18
%
ppm
200
180
160
140
120
100
80
60
40
20
0
-40
-20
0
20
40
60
80
100
120


ppm
7,301
7,293
7,287
7,284
7,280
7,262
7,018
6,998
6,963
6,959
6,950
6,946
6,921
6,903
6,885
6,782
6,778
6,774
6,770
5,736
3,1
2,0
1,0
1,0
0,9
ppm
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
%
600
500
400
300
200
100
0

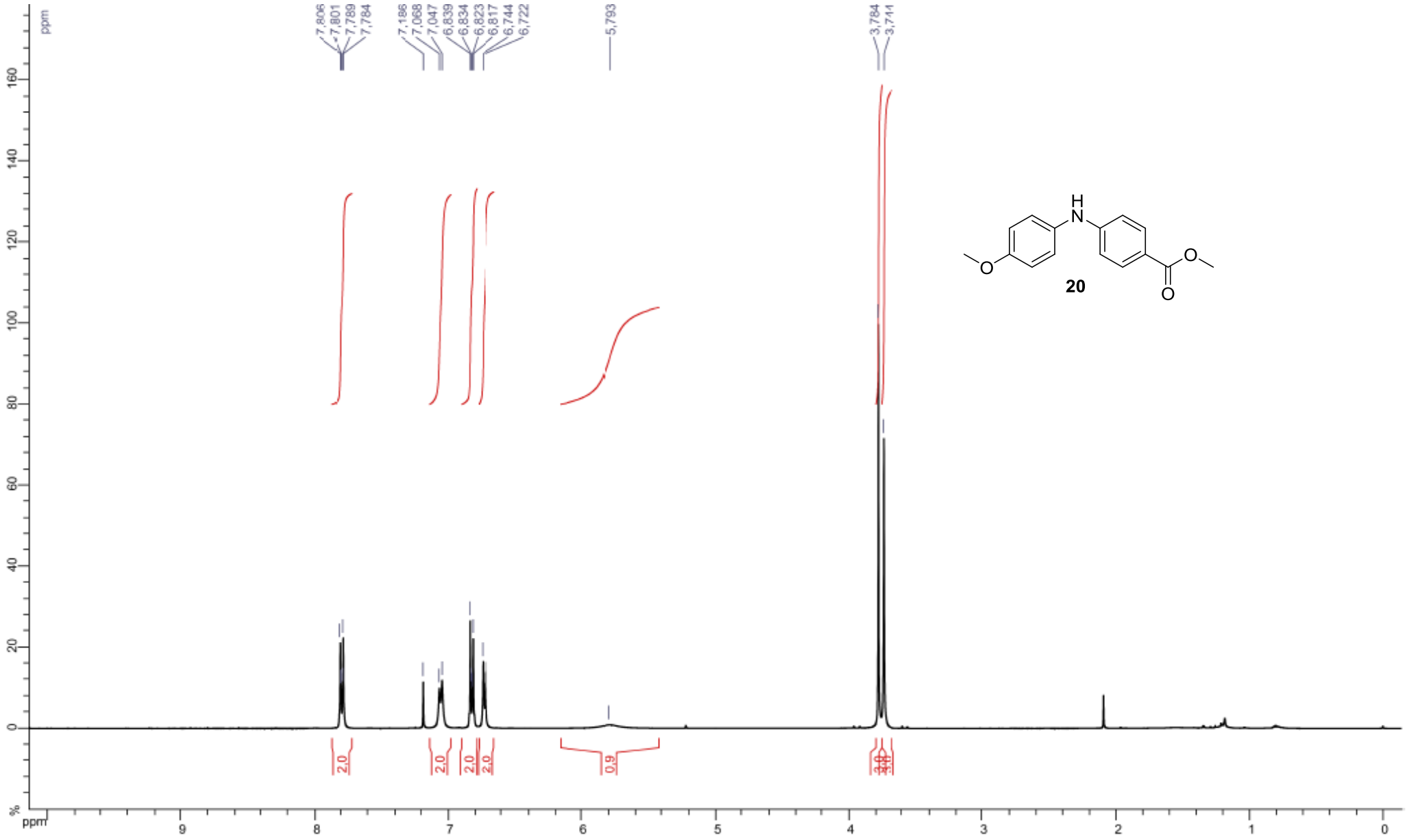

S
N
H

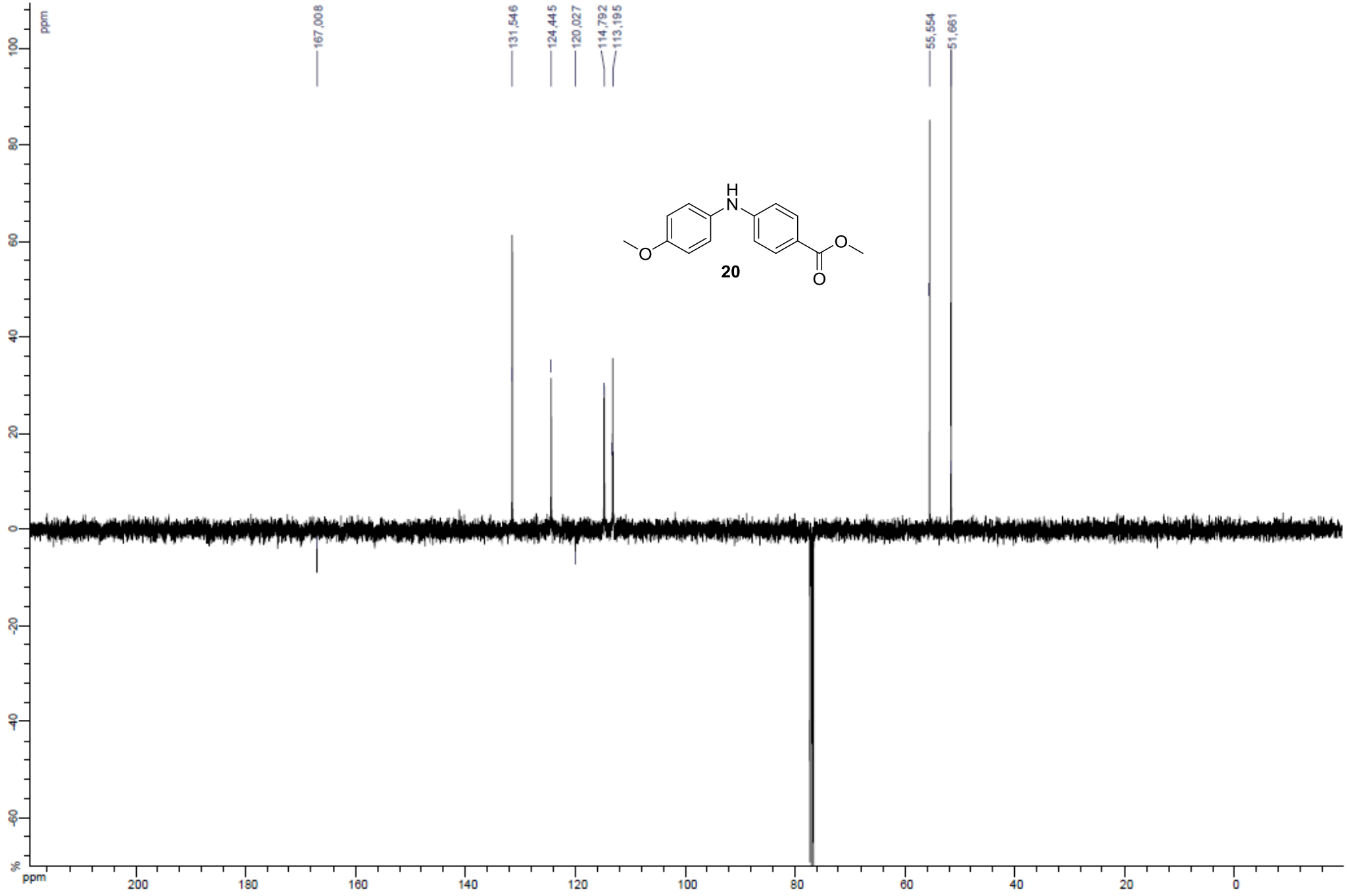
**19**

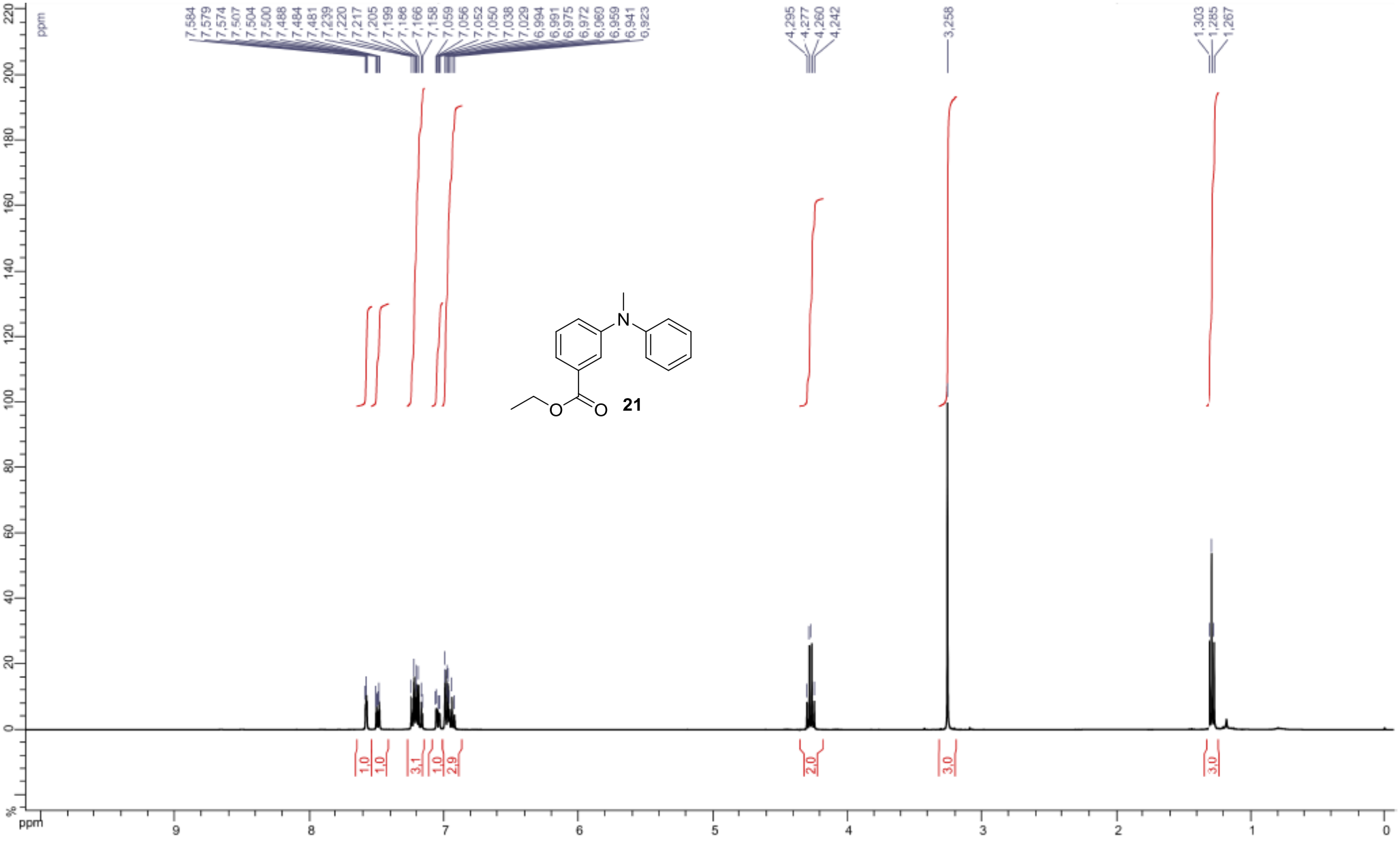

144,845
141,717
129,549
125,349
120,100
115,784
77,551
77,230
76,916
ppm
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

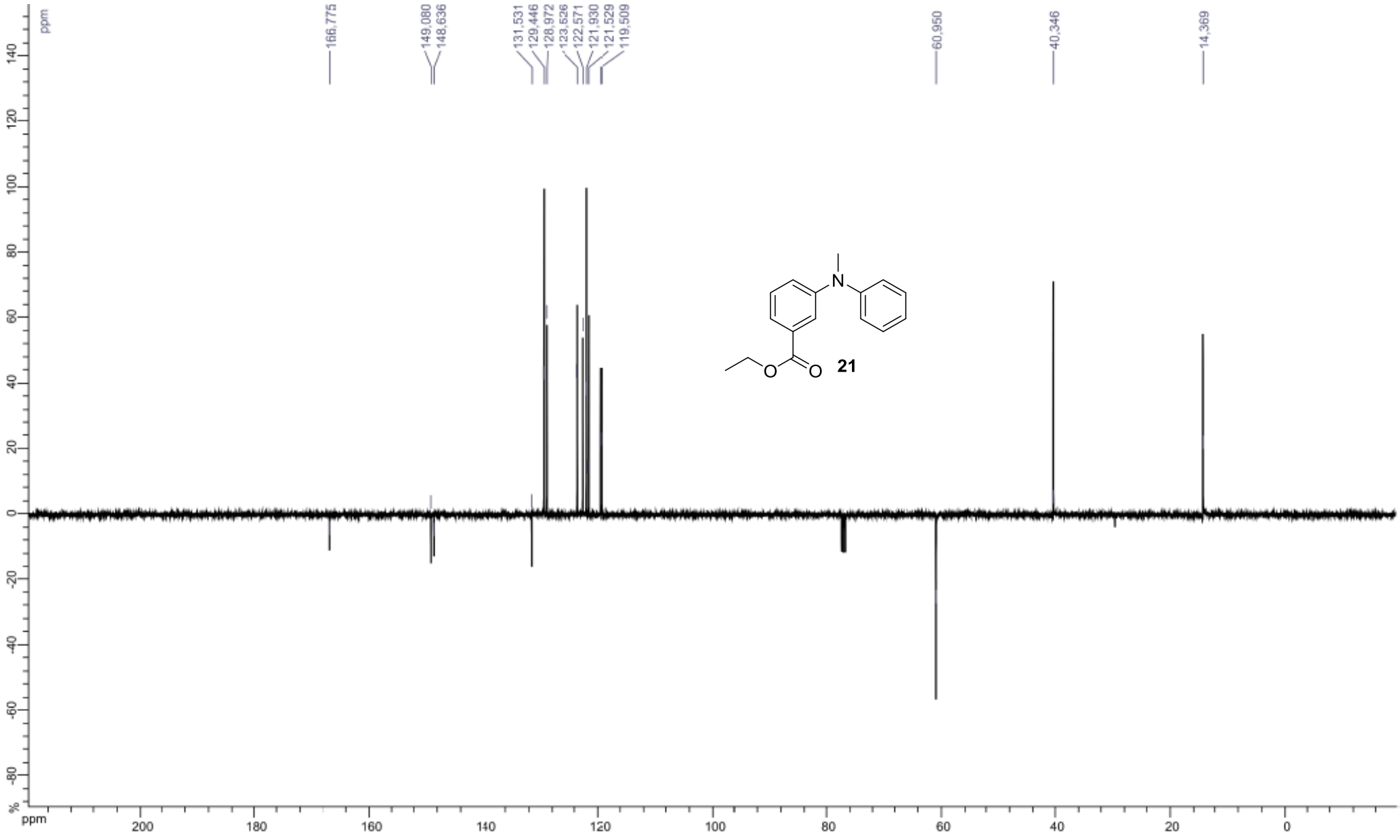

S
N
H

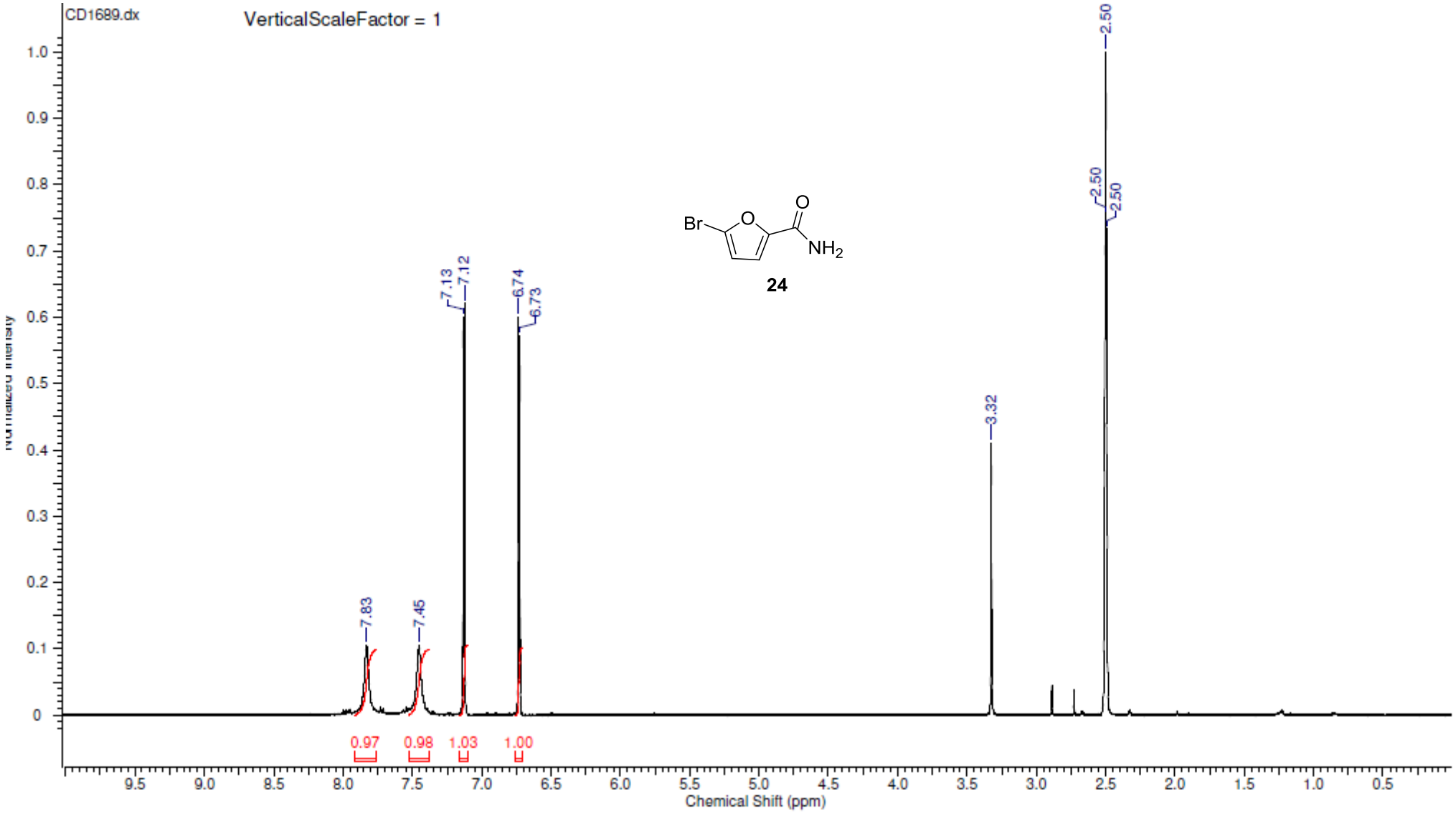
**19**

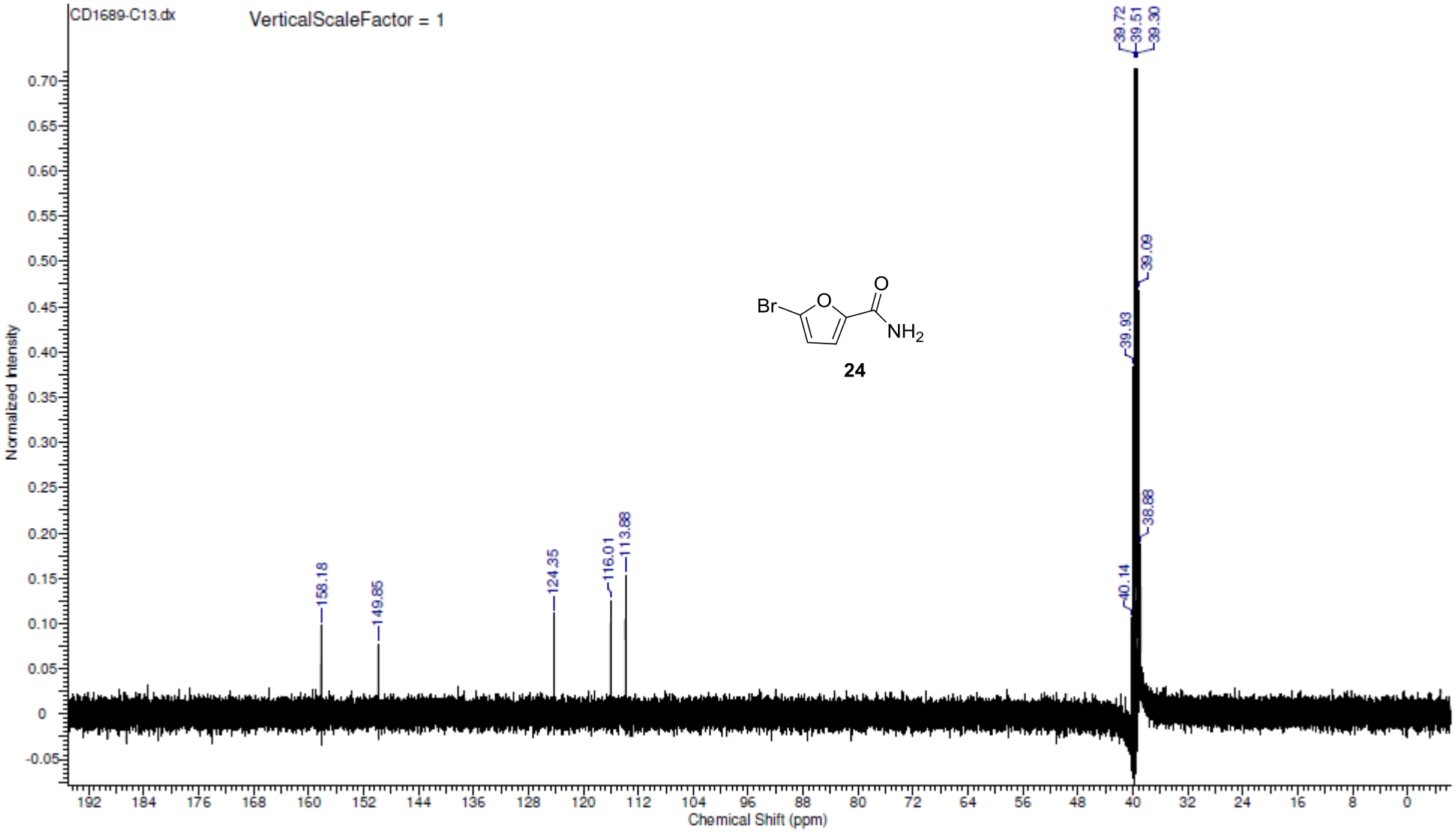

7,806
7,801
7,789
7,784
7,186
7,068
7,047
6,839
6,834
6,823
6,817
6,744
6,722
5,793
3,784
3,711
ppm
2,0
2,0
2,0
2,0
0,9
3,0
3,0
%
ppm
9
8
7
6
5
4
3
2
1
0
H
N
O
O
O
20

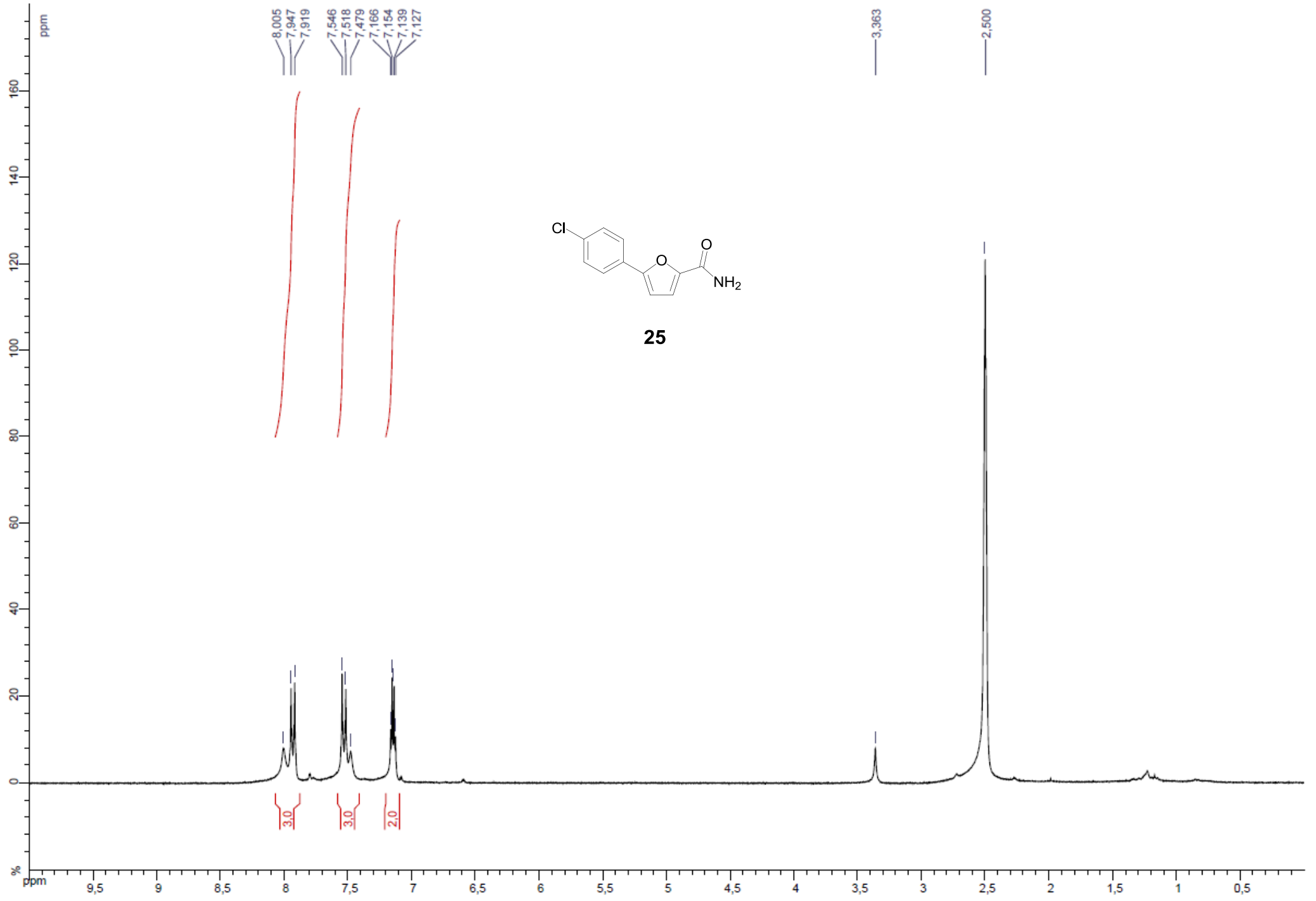

ppm
167,008
131,546
124,445
120,027
114,792
113,195
55,554
51,661
H
N
O
O
O
20
100
80
60
40
20
0
-20
-40
-60
%
ppm
200
180
160
140
120
100
80
60
40
20
0

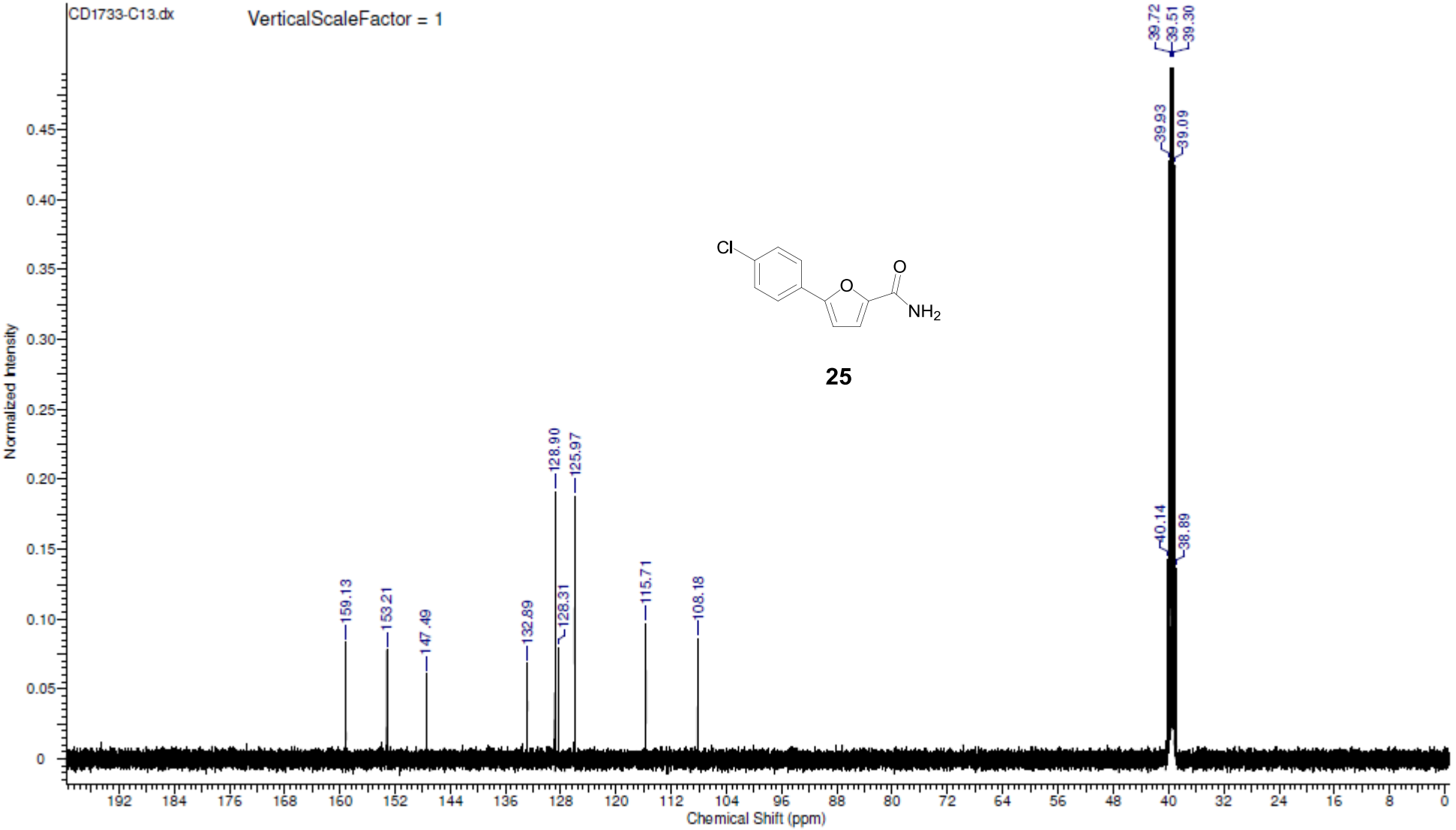

7,584
7,579
7,574
7,507
7,504
7,500
7,488
7,484
7,481
7,239
7,220
7,217
7,205
7,199
7,186
7,166
7,158
7,059
7,056
7,052
7,050
7,038
7,029
6,994
6,991
6,975
6,972
6,960
6,959
6,941
6,923
4,295
4,277
4,260
4,242
3,258
1,303
1,285
1,267
21
1,0
1,0
3,1
1,0
2,9
2,0
3,0
3,0
ppm
9
8
7
6
5
4
3
2
1
0


ppm
166.775
149.080
148.636
131.531
129.446
128.972
123.526
122.571
121.930
121.529
119.509
60.950
40.346
14.369
21
%
ppm
200
180
160
140
120
100
80
60
40
20
0

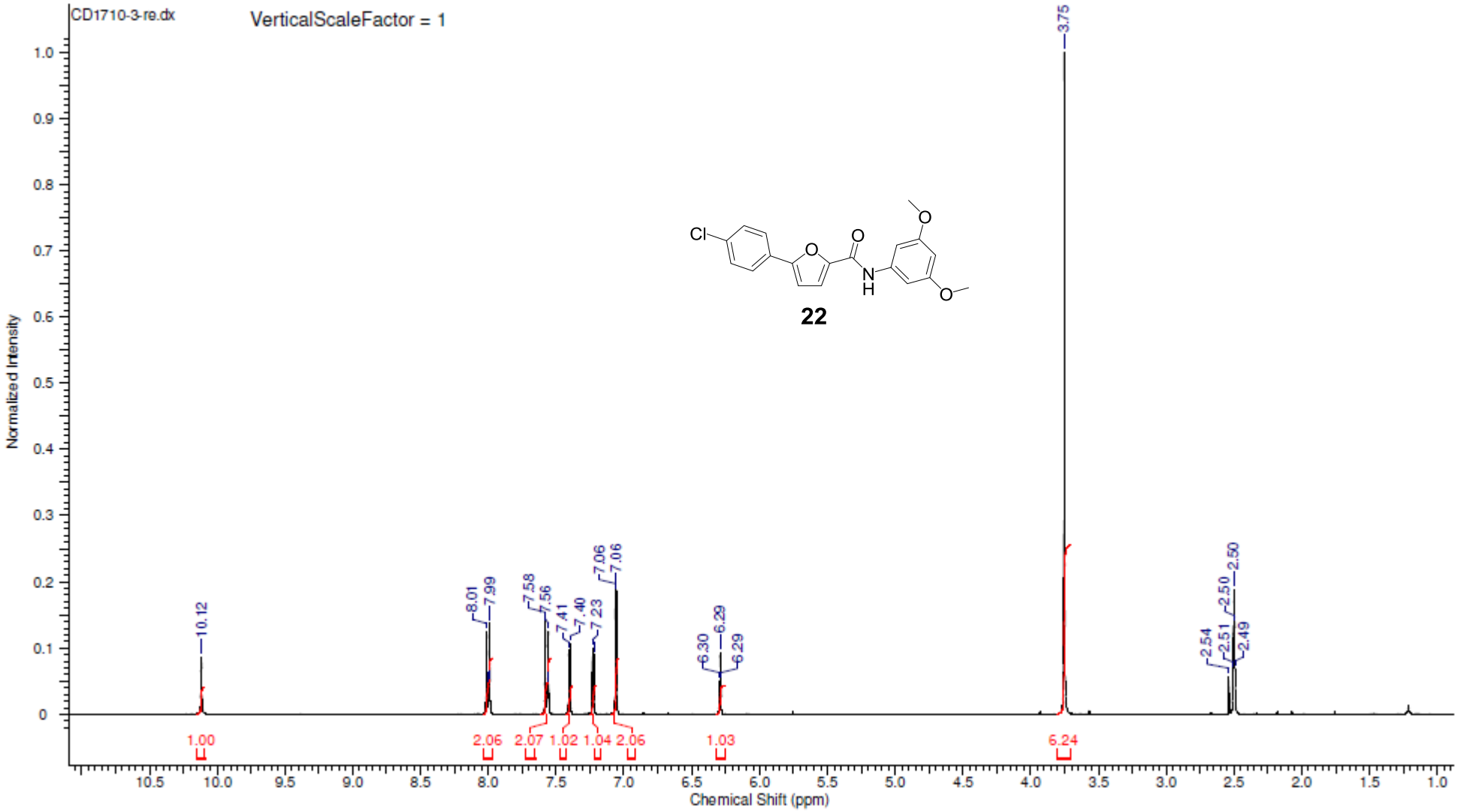

CD1689.dx
VerticalScaleFactor = 1
Br
O
O
NH2
24
7.83
7.45
7.13
7.12
6.74
6.73
3.32
2.50
2.50
2.50
0.97
0.98
1.03
1.00
Chemical Shift (ppm)


CD1689-C13.dx
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
158.18
149.85
124.35
116.01
113.88
39.72
39.51
39.30
39.09
39.93
38.88
40.14
Br
O
O
NH2
24

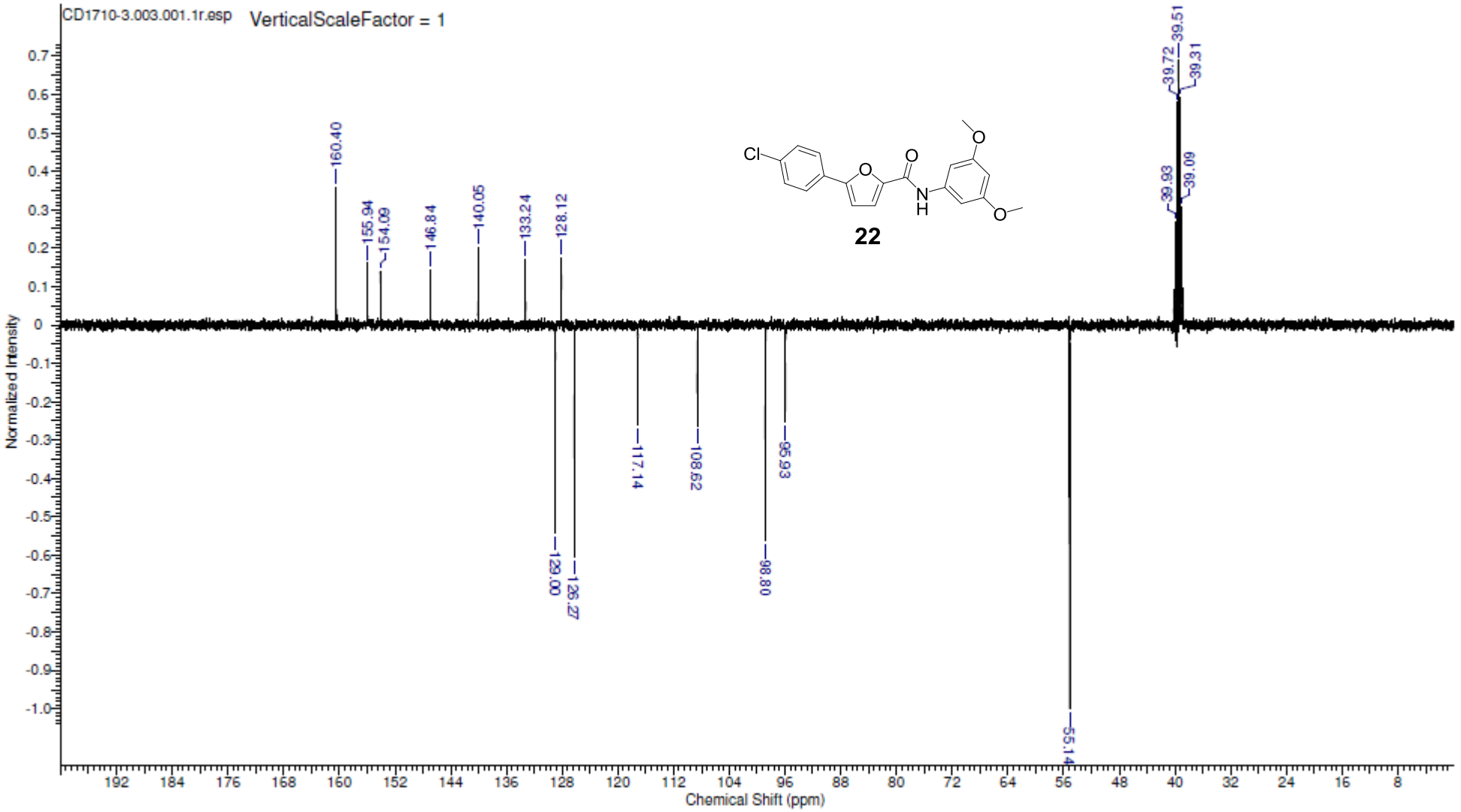

ppm
8,005
7,947
7,919
7,546
7,518
7,479
7,166
7,154
7,139
7,127
3,363
2,500
Cl
O
O
NH2
25
3,0
3,0
2,0
%
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5


CD1733-C13.dx
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
159.13
153.21
147.49
132.89
128.90
128.31
125.97
115.71
108.18
40.14
39.93
39.72
39.51
39.30
39.09
38.89
Cl
O
O
NH2

**25**


CD1710-3-re.dx
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
10.12
8.01
7.99
7.58
7.56
7.41
7.40
7.23
7.06
7.06
6.30
6.29
6.29
3.75
2.54
2.51
2.50
2.50
2.49
1.00
2.06
2.07
1.02
1.04
2.06
1.03
6.24
Cl
O
O
N
H
O
O

**22**


CD1710-3.003.001.1r.esp
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
160.40
155.94
154.09
146.84
140.05
133.24
128.12
129.00
126.27
117.14
108.62
95.93
98.80
55.14
39.72
39.51
39.31
39.93
39.09
Cl
O
N
H
22