Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information:

Direct *N*-Cyclopropylation of Secondary Acyclic Amides Promoted by Copper

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I - General experimental methods

All reactions were carried out in Carousel Thermo-Fischer tubes (40 mL). Sand was added in the holes where tubes are inserted until the top of the hole. The temperature was checked by inserting the thermometer in sand. The reaction was stirred at 800 rpm.

Pyridine was distilled under CaH_2 and kept over KOH under nitrogen. Toluene was distilled over Na^0/benzophenone then kept over Na^0 under nitrogen. Commercial amides and cesium carbonate (Aldrich, 99.9%) were used without purification. They were crushed, dried at 150 °C for 2 hours then stored in the presence of P_4O_{10} in a bench-top dessicator under vacuum at room temperature. Pinacolic ester of cyclopropylboronic acid (Borochem, 95+%) and copper (II) acetate (Alfa Aesar, 99.999% trace metal basis) were used without further purification. Dry air was obtained by passing air through silica gel (200 mL), then molecular sieves (200 mL) and silica gel (200 mL). All the reagents were weighed in the air.

Column chromatography was performed with SDS 60 Å C.C silica gel (35-70 μm). Thin layer chromatography was carried out using Merck silica gel 60 F_{254} plates.

All products were characterized by their NMR, GC/MS spectra. NMR spectra were recorded at 20°C on a Brüker AC 400 MHz working respectively at 400 MHz for ^1H and 100 MHz for ^{13}C. ^1H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ (unless otherwise stated). Chemical shifts for ^1H spectra are values from $CDCl_3$ (δ 7.26) or DMSO (δ 2.50). Chemical shifts for ^{13}C spectra are values from $CDCl_3$ (δ 77.16) or DMSO (δ 39.52). ^1H NMR spectra are reported as follows: chemical shift (ppm), multiplicity (br: broad; s: singlet; d: doublet; t: triplet; q: quadruplet; m: multiplet), coupling constants (Hz) and integration. Gas chromatography - mass spectra (GC/MS) were recorded on an Agilent Technologies 6890 N instrument with an Agilent 5973 N mass detector (EI) and a HP5-MS 30 m x 0.25 mm capillary apolar column (Stationary phase: 5 % diphenyldimethylpolysiloxane film, 0.25 μm). GC/MS method: Initial temperature: 45°C; Initial time: 2 min; Ramp: 2°C/min until 50°C then 10 °C/min; Final temperature: 250°C; Final time: 10 min. HPLC were recorded on a Shimadzu LC-20AD instrument with Shimadzu SPD-M20A detector and a Supelco analytical apolar column (15 cmx4.6 mm, 5 μm). HPLC method: Gradient: 20% MeCN + 1% TFA, 80% water + 1% TFA to 90% MeCN + 1% TFA, 10% water + 1% TFA in 20 min. HRMS were recorded on a JEOL JMS-DX300 spectrometer (3 keV, xenon) in a *m*-nitrobenzylalcohol matrix. Melting points were obtained on a Büchi B-540 melting point apparatus and are uncorrected. All the compounds, unless specified, are new and fully described in the characterization section.

II – Typical procedures

II-1 Typical procedure for the preparation of compounds 1b, 1d, 1g-j and 4c (Procedure A)

Benzoyl chloride (2.4 mmol, 1.2 equiv.) was added to a solution of aniline (2.0 mmol, 1.0 equiv.) and triethylamine (3.0 mmol, 1.5 equiv.) in dichloromethane (2 mL, 1M) at 0 °C under nitrogen. The reaction was run at 40 °C until all the aniline was consumed (TLC). Dichloromethane was added then a saturated aqueous solution of $NaHCO_3$. The phases were separated and the organic layer was extracted three times with dichloromethane. Organic layers were gathered, washed with brine and the organic phases were separated. Organic phase was dried over $MgSO_4$, filtered then concentrated. The crude mixture obtained was crystallized using heptanes/ethyl acetate. Solids obtained were filtered, rinsed with heptane and dried under vacuum.

II-2 Typical procedure for the preparation of compound 1c[1]

4-methoxybenzoyl chloride (2.2 mmol, 1.1 equiv.) was added to a suspension of aniline (2.0 mmol, 1.0 equiv.), and sodium dicarbonate (6.0 mmol, 3.0 equiv.) in dichloromethane (12 mL, 0.16 M) at 0 °C under nitrogen. The reaction was run at 40 °C during 1 h. Dichloromethane was added then a saturated aqueous solution of $NaHCO_3$. The phases were separated and the organic layer was extracted three times with dichloromethane. Organic layers were gathered, washed with brine and the organic phases were separated. Organic phase was dried over $MgSO_4$, filtered then concentrated. The crude mixture obtained was crystallized using heptanes/ethyl acetate. Solids obtained were filtered, rinsed with heptane and dried under vacuum to furnish pure **1c** as white crystals (309 mg, 68%).

II-3 Typical procedure for the preparation of compound 1e[2]

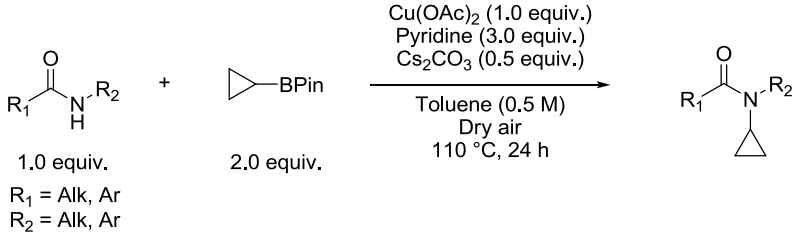
2-chlorobenzoyl chloride (2.2 mmol, 1.1 equiv.) was added to a solution of aniline (2.0 mmol, 1.0 equiv.), pyridine (2.2 mmol, 1.1 equiv.) and DMAP (2.2 mmol, 1.1 equiv.) in dichloromethane (5 mL, 0.4 M) at 0 °C under nitrogen. The reaction was run at 40 °C during 15 h. Dichloromethane was added then a saturated aqueous solution of $NaHCO_3$. The phases were separated and the organic layer was extracted three times with dichloromethane. Organic layers were gathered, washed with brine and the organic phases were separated. Organic phase was dried over $MgSO_4$, filtered then concentrated. The crude mixture obtained was crystallized using heptanes/ethyl acetate. Solids obtained were filtered, rinsed with heptane and dried under vacuum to furnish pure **1e** as white crystals (374 mg, 81%).

[1] Dong, Q.-L.; Liu, G.-S.; Zhou, H.-B.; Chen, L.; Yao, Z. J. *Tetrahedron Lett.* **2008**, *49*, 1636.
[2] Gabbutt, C. D.; Heron, B. M.; Instone, A. C. *Heterocycles* **2003**, *60*, 843.

II-4 Typical procedure for the preparation of compound 1f[3]

Benzoyl chloride (2.1 mmol, 1.05 equiv.) was added to a solution of 4-fluoroaniline (2.0 mmol, 1.0 equiv.) and triethylamine (2.1 mmol, 1.05 equiv.) in ethylacetate (5 mL, 0.4 M) at 0 °C under nitrogen. The reaction was run at 40 °C during 4 h. Ethylacetate was added then a saturated aqueous solution of $NaHCO_3$. The phases were separated and the organic layer was extracted three times with ethylacetate. Organic layers were gathered, washed with brine and the organic phases were separated. Organic phase was dried over $MgSO_4$, filtered then concentrated. The crude mixture obtained was crystallized using heptanes/ethyl acetate. Solids obtained were filtered, rinsed with heptane and dried under vacuum to furnish pure **1f** as beige crystals (228 mg, 53%).

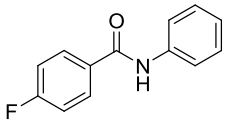
II-5 Typical procedure for the preparation of compounds 3a-j and 5a-j (Procedure B)



An oven-dried (120 °C) Thermo-Fischer tube (Carousel) equipped with a magnetic stirring bar was cool-down under vacuum then back-filled with dry air. Tube was charged with amide (0.2 mmol, 1.0 equiv.), $Cu(OAc)_2$ (0.2 mmol, 1.0 equiv.) and Cs_2CO_3 (0.1 mmol, 0.5 equiv.). The tube was evacuated then back-filled with dry air three times with stirring (400 rpm). Pyridine (0.6 mmol, 3.0 equiv.) the pinacolic ester of cyclopropylboronic acid (0.4 mmol, 2.0 equiv.) and toluene (0.4 mL, 0.5M) were added (no stirring). The tube was sealed, stirred (800 rpm) and heated to 110 °C for 24 h. After cooling to room temperature, 20 ml of dichloromethane were added. The organic phase was washed twice with water (20 mL). Gathered aqueous phases were extracted with dichloromethane (20 mL) for three times. Organic layers were gathered, dried over $MgSO_4$, filtered and concentrated under vacuum to furnish the crude product (a small sample of the crude was analyzed by HPLC).The obtained crude was purified by silica gel chromatography using heptanes/ethyl acetate as eluent.

[3] Ueda, S.; Nagasawa, H. *J. Org. Chem.* **2009**, *74*, 4272.

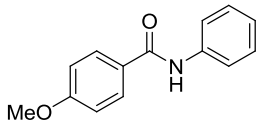
II- Characterization of compounds 1b-j, 3a-j, 4c and 5a-h



1b

4-fluoro-*N*-phenylbenzamide (1b) was obtained by procedure A from 4-fluorobenzoyl chloride (2.4 mmol, 1.2 equiv.) and aniline (2.0 mmol, 1.0 equiv.) as white crystals (86 mg, 20%).

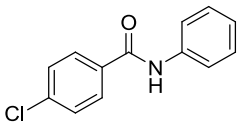
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15-7.19 (m, 3H, CH_{ar}), 7.36-7.40 (m, 2H, CH_{ar}), 7.61-7.63 (m, 2H, CH_{ar}), 7.74 (bs, 1H, NH), 7.87-7.91 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, DMSO) 115.2, 115.4, 120.4, 123.7, 128.6, 130.3 and 130.4 (CH_{ar}), 131.4 (d), 139.1, 162.8 and 164.4 ($C_{q\ ar}$), 165.3 (C=O). m.p. 185-187 °C (Lit. 180-181 °C)[4]; ***GC/MS***: r.t. = 22.1 min., M/Z = 215; ***HRMS*** calculated for $C_{13}H_{11}NOF$ (M+H+) 216.0825, found 216.0828.



1c

4-methoxy-*N*-phenylbenzamide (1c) (white crystals, 309 mg, 68%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.87 (s, 3H, OMe), 6.96-6.98 (m, 2H, CH_{ar}), 7.12-7.16 (m, 1H, CH_{ar}), 7.34-7.38 (m, 2H, CH_{ar}), 7.62-7.64 (m, 2H, CHar), 7.76 (bs, 1H, NH), 7.83-7.85 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 55.6 (CH_3), 114.1, 120.3, 124.5, 129.0 and 129.2 (CH_{ar}), 127.3, 138.2 and 162.6 ($C_{q\ ar}$), 165.3 (C=O). m.p. 172-173 °C (Lit. 173-174 °C)[4]; ***GC/MS***: r.t. = 25.5 min., M/Z = 227; ***HRMS*** calculated for $C_{14}H_{14}NO_2$ (M+H+) 228.1025, found 228.1015.

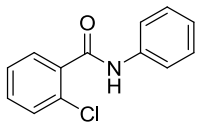


1d

4-chloro-*N*-phenylbenzamide (1d) was obtained by procedure A from 4-chlorobenzoyl chloride (2.4 mmol, 1.2 equiv.) and aniline (2.0 mmol, 1.0 equiv.) as beige crystals (139 mg, 30%).

^{1}H NMR (400 MHz, DMSO) δ: 7.11 (t, *J* = 8 Hz, 1H, CH_{ar}), 7.36 (t, *J* = 8 Hz, 1H, CH_{ar}), 7.61 (d, *J* = 8 Hz, 2H, CH_{ar}), 7.76 (d, *J* = 8 Hz, 2H, CHar), 7.98 (d, *J* = 8 Hz, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, DMSO) δ: 120.4, 123.9, 128.5, 128.7 and 129.7 (CH_{ar}), 133.7, 136.4 and 139.0 ($C_{q\ ar}$), 164.5 (C=O). m.p. 197-198 °C (Lit. 199-200 °C)4; ***GC/MS***: r.t. = 25.5 min., M/Z = 231; ***HRMS*** calculated for $C_{13}H_{11}NOCl$ (M+H+) 232.0529, found 232.0521.

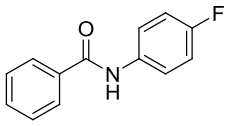
[4] Lijun, Z.; Shunpeng, S.; Hongping, W.; Shaowu, W. *Tetrahedron* **2009**, *65*, 10022.



1e

2-chloro-*N*-phenylbenzamide (1e) was obtained by procedure A from 2-chlorobenzoyl chloride (2.4 mmol, 1.2 equiv.) and aniline (2.0 mmol, 1.0 equiv.) as white crystals (374 mg, 81%).

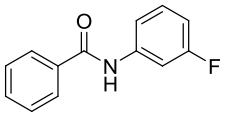
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.16-7.20 (m, 1H, CH_{ar}), 7.36-7.47 (m, 5H, CH_{ar}), 7.64-7.66 (m, 2H, CH_{ar}), 7.77 (dd, *J* = 1.8 and 7.4 Hz, 1H, CH_{ar}), 7.88 (bs, 1H, NH); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 120.3, 124.9, 129.1, 130.2, 130.4 and 131.6 (CH_{ar}), 130.7, 135.4 and 137.7 ($C_{q\ ar}$), 164.8 (C=O). m.p. 115-117 °C; ***GC/MS***: r.t. = 22.9 min., M/Z = 231; ***HRMS*** calculated for $C_{13}H_{11}NOCl$ (M+H+) 232.0529, found 232.0521.



1f

***N*-(4-fluorophenyl)benzamide (1f)** (beige crystals, 228 mg, 53%).

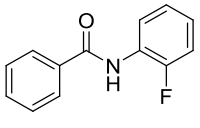
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.05-7.09 (m, 2H, CH_{ar}), 7.48-7.51 (m, 2H, CH_{ar}), 7.54-7.62 (m, 3H, CH_{ar}), 7.82 (bs, 1H, NH), 7.85-7.88 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 115.8, 116.0, 122.2, 122.3, 127.1, 129.0 and 132.1 (CH_{ar}), 134.0 and 134.9 ($C_{q\ ar}$), 160.7 (C=O). m.p. 181-183 °C (Lit. 184-185 °C)[5]; ***GC/MS***: r.t. = 22.2 min., M/Z = 215; ***HRMS*** calculated for $C_{13}H_{11}NOF$ (M+H+) 216.0825, found 216.0817.



1g

***N*-(3-fluorophenyl)benzamide (1g)** was obtained by procedure A from benzoyl chloride (2.4 mmol, 1.2 equiv.) and 3-fluoroaniline (2.0 mmol, 1.0 equiv.) as beige oil (344 mg, 80%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.80-7.60 (m, 3H, CH_{ar} and NH), 7.48-7.51 (m, 2H, CH_{ar}), 7.58-7.40 (m, 4H, CH_{ar}), 7.25-7.19 (m, 2H, CH_{ar}), 6.81-6.76 (m, 1H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 107.6, 111.2, 111.4, 115.3, 127.0, 128.9, 130.2, 132.1, 134.6, 139.4, 161.8, 164.3, 165.7.

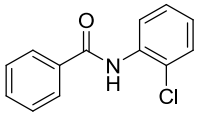


1h

***N*-(2-fluorophenyl)benzamide (1h)** was obtained by procedure A from benzoyl chloride (2.4 mmol, 1.2 equiv.) and 2-fluoroaniline (2.0 mmol, 1.0 equiv.) as white crystals (357 mg, 83%).

[5] Ueda, S.; Nagasawa, H. *J. Org. Chem.* **2009**, *74*, 4272.

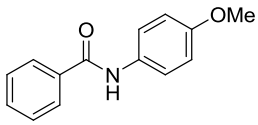
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.07-7.22 (m, 3H, CH_{ar}), 7.50-7.60 (m, 3H, CH_{ar}), 7.89-7.91 (m, 2H, CH_{ar}), 8.07 (bs, 1H, NH), 8.48 (td, *J* = 1.6 and 8.4 Hz, 1H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 114.9, 115.0, 121.9, 124.6, 124.8, 124.9, 127.2, 129.0 and 132.3 (CH_{ar}), 126.6, 126.7, 134.7, 151.7 and 154.0 ($C_{q\ ar}$), 165.6 (C=O). m.p. 112-113 °C (Lit. 108-109 °C)[6]; ***GC/MS***: r.t. = 20.9 min., M/Z = 215; ***HRMS*** calculated for $C_{13}H_{11}NOF$ (M+H+) 216.0825, found 216.0824.



1i

***N*-(2-chlorophenyl)benzamide (1i)** was obtained by procedure A from benzoyl chloride (2.4 mmol, 1.2 equiv.) and 2-chloroaniline (2.0 mmol, 1.0 equiv.) as white crystals (305 mg, 66%).

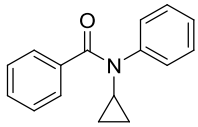
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.09 (td, *J* = 1.2 and 7.8 Hz, 1H, CH_{ar}), 7.32-7.36 (m, 1H, CH_{ar}), 7.42 (dd, *J* = 1.6 and 8.0 Hz, 1H, CH_{ar}), 7.51-7.55 (m, 2H, CH_{ar}), 7.57-7.61 (m, 1H, CH_{ar}), 7.92-7.94 (m, 2H, CH_{ar}), 8.46 (bs, 1H, NH), 8.48 (dd, *J* = 1.6 and 8.2 Hz, 1H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 121.6, 124.9, 127.2, 128.0, 129.1, 129.2 and 132.3 (CH_{ar}), 123.1, 134.8 and 134.9 ($C_{q\ ar}$), 165.4 (C=O). m.p. 103-104 °C; ***GC/MS***: r.t. = 22.1 min., M/Z = 231; ***HRMS*** calculated for $C_{13}H_{11}NOCl$ (M+H+) 232.0529, found 232.0531.



1j

***N*-(4-methoxyphenyl)benzamide (1j)** was obtained by procedure A from benzoyl chloride (2.4 mmol, 1.2 equiv.) and 4-methoxyaniline (2.0 mmol, 1.0 equiv.) as white crystals (313 mg, 69%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.82 (s, 3H, OCH_3), 6.90-6.92 (m, 2H, CH_{ar}), 7.46-7.50 (m, 2H, CH_{ar}), 7.53-7.55 (m, 3H, CH_{ar}), 7.74 (bs, 1H, NH), 7.85-7.87 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 55.7 (OCH_3), 114.4, 122.2, 127.1, 128.9 and 131.8 (CH_{ar}), 131.2 and 135.2 ($C_{q\ ar}$), 156.8 (C=O). m.p. 157-158 °C (Lit. 154-155 °C)6; ***GC/MS***: r.t. = 25.8 min., M/Z = 227; ***HRMS*** calculated for $C_{14}H_{14}NO_2$ (M+H+) 228.1025, found 228.1015.

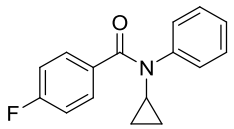


3a

***N*-cyclopropyl-*N*-phenylbenzamide (3a)** was obtained by procedure B from commercial benzanilide (0.2 mmol, 1.0 equiv.) as beige oil (44 mg, 93%).

[6] Tambade, P. J.; Patil, Y. P.; Bhanushali, M. J.; Bhanage, B. M. *Synthesis* **2008**, 2347.

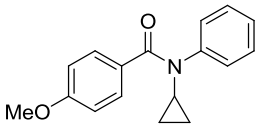
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.49-0.51 (m, 2H, $CH_{2c\text{-propyl}}$), 0.77-0.79 (m, 2H, $CH_{2c\text{-propyl}}$), 3.19-3.23 (m, 1H, $CH_{c\text{-propyl}}$), 6.94-6.97 (m, 2H, CH_{ar}), 7.08-7.11 (m, 8H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 8.1 ($CH_{2c\text{-propyl}}$), 32.4 ($CH_{c\text{-propyl}}$), 126.7, 127.7, 128.1, 128.2, 128.8, 129.4 (CH_{ar}), 136.9, 142.2 ($C_{q\ ar}$), 172.1 (C=O); ***HRMS*** calculated for $C_{16}H_{15}NO$ (M+H+) 238.1232, found 238.1230.



3b

***N*-cyclopropyl-4-fluoro-*N*-phenylbenzamide (3b)** was obtained by procedure B from amide **1b** (0.2 mmol, 1.0 equiv.) as beige crystals (37 mg, 72%).

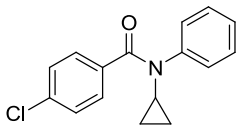
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.49-0.53 (m, 2H, $CH_{2c\text{-propyl}}$), 0.78-0.83 (m, 2H, $CH_{2c\text{-propyl}}$), 3.17-3.23 (m, 1H, $CH_{c\text{-propyl}}$), 6.77-6.82 (m, 2H, CH_{ar}), 6.91-6.96 (m, 2H, CH_{ar}), 7.09-7.13 (m, 1H, CH_{ar}), 7.15-7.20 (m, 2H, CH_{ar}), 7.22-7.28 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 8.1 ($CH_{2c\text{-propyl}}$), 32.6 ($CH_{c\text{-propyl}}$), 114.8, 115.0, 127.0, 128.4, 129.1, 130.7 and 130.8 (CH_{ar}), 133.0 (d, C-F), 133.1, 142.3 ($C_{q\ ar}$), 171.1 (C=O); m.p. 48-49 °C; ***GC/MS***: r.t. = 17.7 min., M/Z = 255; ***HRMS*** calculated for $C_{16}H_{14}NOF$ (M+H+) 256.1138, found 256.1129.



3c

***N*-cyclopropyl-4-methoxy-*N*-phenylbenzamide (3c)** was obtained by procedure B from amide **1c** (0.2 mmol, 1.0 equiv.) as beige oil (47 mg, 90%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.49-0.53 (m, 2H, $CH_{2c\text{-propyl}}$), 0.77-0.83 (m, 2H, $CH_{2c\text{-propyl}}$), 3.15-3.21 (m, 1H, $CH_{c\text{-propyl}}$), 3.68 (s, 3H, OCH_3), 6.60-6.62 (m, 2H, CH_{ar}), 6.95-6.97 (m, 2H, CH_{ar}), 7.07-7.12 (m, 1H, CH_{ar}), 7.16-7.24 (m, 4H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 8.2 ($CH_{2c\text{-propyl}}$), 32.7 ($CH_{c\text{-propyl}}$), 55.3 (OCH_3), 113.1, 126.7, 128.4, 129.0, and 130.6 (CH_{ar}), 129.0, 142.9 and 160.6 ($C_{q\ ar}$), 171.8 (C=O); ***GC/MS***: r.t. = 13.8 min., M/Z = 267; ***HRMS*** calculated for $C_{17}H_{17}NO_2$ (M+H+) 268.1338, found 268.1335.

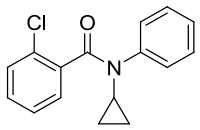


3d

4-chloro-*N*-cyclopropyl-*N*-phenylbenzamide (3d) was obtained by procedure B from amide **1d** (0.2 mmol, 1.0 equiv.) as beige oil (51 mg, 94%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.48-0.52 (m, 2H, $CH_{2c\text{-propyl}}$), 0.78-0.83 (m, 2H, $CH_{2c\text{-propyl}}$), 3.17-3.23 (m, 1H, $CH_{c\text{-propyl}}$), 6.91-6.96 (m, 2H, CH_{ar}), 7.06-7.13 (m, 3H, CH_{ar}), 7.16-7.21 (m, 4H, CH_{ar}); ***^{13}C NMR*** (100 MHz,

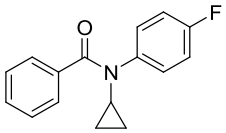
$CDCl_3$) δ: 8.1 ($CH_{2c\text{-propyl}}$), 32.5 ($CH_{c\text{-propyl}}$), 127.1, 128.1, 128.3, 129.1 and 129.9 (CH_{ar}), 135.5, 135.6 and 142.1 ($C_{q\ ar}$), 171.0 (C=O); ***GC/MS***: r.t. = 14.3 min., M/Z = 271; ***HRMS*** calculated for $C_{16}H_{14}NOCl$ (M+H+) 272.0842, found 272.0836.



3e

2-chloro-*N*-cyclopropyl-*N*-phenylbenzamide (3e) was obtained by procedure B from amide **1e** (0.2 mmol, 1.0 equiv.) as beige oil (52 mg, 95%).

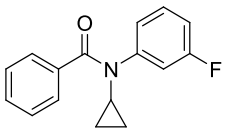
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.23-1.08 (m, 4H, $CH_{2c\text{-propyl}}$), 2.87-3.54 (m, 1H, $CH_{c\text{-propyl}}$), 6.78-7.72 (m, 9H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 7.4 ($CH_{2c\text{-propyl}}$), 31.2 ($CH_{c\text{-propyl}}$), 126.5, 127.1, 127.9, 128.2, 128.9, 129.1, 129.4 and 129.7 (CH_{ar}), 130.3 and 141.1 ($C_{q\ ar}$), 169.6 (C=O); ***GC/MS***: r.t. = 13.9 min., M/Z = 271; ***HRMS*** calculated for $C_{16}H_{14}NOCl$ (M+H+) 272.0833, found 272.0836.



3f

***N*-cyclopropyl-*N*-(4-fluorophenyl)benzamide (3f)** was obtained by procedure B from amide **1f** (0.2 mmol, 1.0 equiv.) as beige oil (21 mg, 41%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.52-0.57 (m, 2H, $CH_{2c\text{-propyl}}$), 0.82-0.87 (m, 2H, $CH_{2c\text{-propyl}}$), 3.22-3.28 (m, 1H, $CH_{c\text{-propyl}}$), 6.91-6.96 (m, 2H, CH_{ar}), 7.00-7.03 (m, 2H, CH_{ar}), 7.20-7.27 (m, 3H, CH_{ar}), 7.29-7.36 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 8.4 ($CH_{2c\text{-propyl}}$), 32.7 ($CH_{c\text{-propyl}}$), 127.9, 128.1, 129.5, 129.6 and 129.7 (CH_{ar}), 138.4 (C-F), 136.9, 159.9 and 162.3 ($C_{q\ ar}$), 172.3 (C=O); ***GC/MS***: r.t. = 18.6 min., M/Z = 255; ***HRMS*** calculated for $C_{16}H_{14}NOF$ (M+H+) 256.1138, found 256.1127.

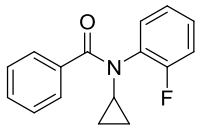


3g

***N*-cyclopropyl-*N*-(3-fluorophenyl)benzamide (3g)** was obtained by procedure B from amide **1g** (0.2 mmol, 1.0 equiv.) as beige oil (22 mg, 42%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.48-0.52 (m, 2H, $CH_{2c\text{-propyl}}$), 0.78-0.83 (m, 2H, $CH_{2c\text{-propyl}}$), 3.12-3.17 (m, 1H, $CH_{c\text{-propyl}}$), 6.75-6.84 (m, 3H, CH_{ar}), 7.10-7.24 (m, 4H, CH_{ar}), 7.27-7.29 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 8.9 ($CH_{2c\text{-propyl}}$), 32.7 ($CH_{c\text{-propyl}}$), 113.6, 113.8, 115.1, 115.3, 123.7, 123.8, 128.0, 128.1, 129.8, 129.9

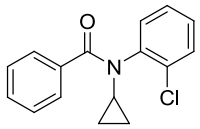
and 130.0 (CH_{ar}), 142.1 (C-F), 136.8, 161.4 and 163.9 ($C_{q\ ar}$), 172.1 (C=O); ***GC/MS***: r.t. = 17.1 min., M/Z = 255; ***HRMS*** calculated for $C_{16}H_{14}NOF$ (M+H+) 256.1138, found 256.1124.



3h

***N*-cyclopropyl-*N*-(2-fluorophenyl)benzamide (3h)** was obtained by procedure B from amide **1h** (0.2 mmol, 1.0 equiv.) as beige cristals (48 mg, 95%).

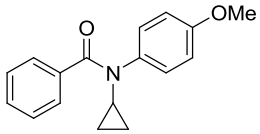
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.56-0.60 (m, 2H, $CH_{2c\text{-propyl}}$), 0.78-0.84 (m, 2H, $CH_{2c\text{-propyl}}$), 3.25-3.31 (m, 1H, $CH_{c\text{-propyl}}$), 6.99-7.07 (m, 3H, CH_{ar}), 7.15-7.28 (m, 4H, CH_{ar}), 7.37 (br d, J = 7.2 Hz, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 7.7 ($CH_{2c\text{-propyl}}$), 32.3 ($CH_{c\text{-propyl}}$), 116.3, 116.5, 124.4, 124.5, 127.7, 127.9, 129.2, 129.3, 129.8 and 130.8 (CH_{ar}), 130.2 (C-F), 136.6, 156.9 and 159.4 ($C_{q\ ar}$), 172.7 (C=O); m.p. 79-80 °C; ***GC/MS***: r.t. = 17.0 min., M/Z = 255; ***HRMS*** calculated for $C_{16}H_{14}NOF$ (M+H+) 256.1138, found 256.1143.



3i

***N*-(2-chlorophenyl)-*N*-cyclopropyl-benzamide (3i)** was obtained by procedure B from amide **1i** (0.2 mmol, 1.0 equiv.) as beige crystals (37 mg, 69%).

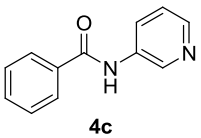
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.33-1.13 (m, 4H, $CH_{2c\text{-propyl}}$), 3.35 (br s, 1H, $CH_{c\text{-propyl}}$), 6.66-8.11 (m, 9H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 127.4, 127.8, 128.9, 129.8 and 130.4 (CH_{ar}), 139.7 ($C_{q\ ar}$), 172.4 (C=O); ***GC/MS***: r.t. = 18.4 min., M/Z = 271; ***HRMS*** calculated for $C_{16}H_{14}NOCl$ (M+H+) 272.0842, found 272.0836.



3j

***N*-cyclopropyl-*N*-(4-methoxyphenyl)benzamide (3j)** was obtained by procedure B from amide **1j** (0.2 mmol, 1.0 equiv.) as white oil (36 mg, 68%).

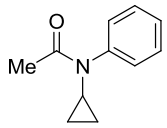
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.53-0.57 (m, 2H, $CH_{2c\text{-propyl}}$), 0.80-0.85 (m, 2H, $CH_{2c\text{-propyl}}$), 3.26-3.32 (m, 1H, $CH_{c\text{-propyl}}$), 3.75 (s, 3H, OCH_3), 6.73-6.76 (m, 2H, CH_{ar}), 6.92-6.94 (m, 2H, CH_{ar}), 7.16-7.24 (m, 3H, CH_{ar}), 7.30-7.32 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 7.7 ($CH_{2c\text{-propyl}}$), 32.6 ($CH_{c\text{-propyl}}$), 55.5 (OCH_3), 114.2, 127.8, 128.3, 129.4 and 129.6 (CH_{ar}), 135.0, 137.2 and 158.2 ($C_{q\ ar}$), 172.4 (C=O); ***GC/MS***: r.t. = 12.6 min., M/Z = 267; ***HRMS*** calculated for $C_{17}H_{17}NO_2$ (M+H+) 268.1338, found 268.1320.



4c

***N*-(pyridine-3-yl)benzamide (4c)** was obtained by procedure A from benzoyl chloride (3.6 mmol, 1.2 equiv.) and pyridine-3-amins (3.0 mmol, 1.0 equiv.) as beige powder (339 mg, 57%).

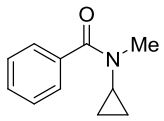
^{1}H NMR (400 MHz, DMSO-d_6) δ: 7.39 (dd, *J* = 4.0 and 8.0 Hz, 1H, CH_{ar}), 7.54-7.57 (m, 2H, CH_{ar}), 7.60-7.64 (m, 1H, CH_{ar}), 7.97-7.99 (m, 2H, CH_{ar}), 8.17-8.22 (m, 1H, CH_{ar}), 8.31 (dd, *J* = 1.2 and 4.0 Hz, 1H, CH_{ar}), 8.92-8.95 (m, 1H, NH); ***^{13}C NMR*** (100 MHz, DMSO-d_6) δ: 19.4 ($\underline{C}H_3$), 42.3 ($\underline{C}H_2Ph$), 123.5, 127.3, 127.7, 128.5, 131.9, 142.0 and 144.6 (CH_{ar}), 134.4 and 135.8 ($C_{q\ ar}$), 166.0 (C=O). m.p. 119-120 °C; ***GC/MS***: r.t. = 15.2 min., M/Z = 198; ***HRMS*** calculated for $C_{12}H_{10}N_2O$ (M+H+) 199.0871, found 199.0864.



5a

***N*-cyclopropyl-*N*-phenylacetamide (5a)** was obtained by procedure B from commercial *N*-phenylbenzamide (0.2 mmol, 1.0 equiv.) as beige oil (35 mg, 78%).

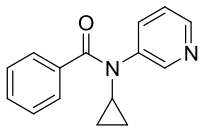
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.49 (br s, 2H, $CH_{2c\text{-propyl}}$), 0.81 (br s, 2H, $CH_{2c\text{-propyl}}$), 2.17 (s, 3H, CH_3), 3.16 (br s, 1H, $CH_{c\text{-propyl}}$), 7.07-7.13 (m, 1H, CH_{ar}), 7.28-7.35 (m, 2H, CH_{ar}), 7.33-7.42 (m, 1H, CH_{ar}), 7.47-7.53 (m, 1H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 23.6 (CH_3), 24.6 ($CH_{c\text{-propyl}}$), 29.8 ($CH_{2c\text{-propyl}}$), 129.0, 124.2 and 119.9 (CH_{ar}), 142.2 ($C_{q\ ar}$), 168.7 (C=O); ***GC/MS***: r.t. = 12.2 min., M/Z = 175; ***HRMS*** calculated for $C_{11}H_{13}NO$ (M+H+) 176.1075, found 176.1072.



5b

***N*-cyclopropyl-*N*-methylbenzamide (5b)** was obtained by procedure B from commercial *N*-methylbenzamide (0.2 mmol, 1.0 equiv.) as beige oil (12 mg, 37%).

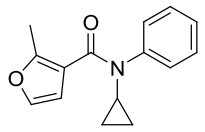
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.30-0.59 (m, 4H, $CH_{2c\text{-propyl}}$), 2.71-2.77 (m, 1H, $CH_{c\text{-propyl}}$), 3.02 (br s, 3H, CH_3), 7.28-7.44 (m, 5H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 9.7 (CH_3), 33.2 ($CH_{c\text{-propyl}}$), 35.1 ($CH_{2c\text{-propyl}}$), 127.3, 128.0 and 129.5(CH_{ar}), 137.3 ($C_{q\ ar}$), 172.6 (C=O); ***GC/MS***: r.t. = 12.2 min., M/Z = 175; ***HRMS*** calculated for $C_{11}H_{13}NO$ (M+H+) 176.1075, found 176.1084.



5c

***N*-cyclopropyl-*N*-(pyridine-3-yl)benzamide (5c)** was obtained by procedure B from amide **4c** (0.2 mmol, 1.0 equiv.) as beige oil (41 mg, 86%).

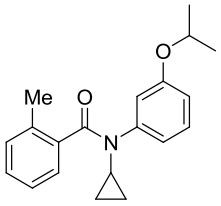
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.48-0.52 (m, 2H, $CH_{2c\text{-propyl}}$), 0.79-0.84 (m, 2H, $CH_{2c\text{-propyl}}$), 3.15-3.21 (m, 1H, $CH_{c\text{-propyl}}$), 7.17-7.30 (m, 6H, CH_{ar}), 7.41 (d, J = 8.0 Hz, 1H, CH_{ar}), 8.37 (br s, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 9.2 ($CH_{2c\text{-propyl}}$), 32.7 ($CH_{c\text{-propyl}}$), 128.1, 128.2, 130.0, 147.4 and 148.7 (CH_{ar}), 136.4 ($C_{q\ ar}$), 172.2 (C=O); ***GC/MS***: r.t. = 14.6 min., M/Z = 238; ***HRMS*** calculated for $C_{15}H_{14}N_2O$ (M+H+) 239.1184, found 239.1183.



5d

***N*-cyclopropyl-2-methyl-*N*-phenylfuran-3-carboxamide (*N*-cyclopropyl Fenfuram) (5d)** was obtained by procedure B from commercial Fenfuram (0.2 mmol, 1.0 equiv.) as beige oil (34 mg, 70%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.55-0.59 (m, 2H, $CH_{2c\text{-propyl}}$), 0.84-0.89 (m, 2H, $CH_{2c\text{-propyl}}$), 2.49 (s, 3H, Me), 3.19-3.26 (m, 1H, $CH_{c\text{-propyl}}$), 5.43 (d, J = 2.0 Hz, CH_{furan}), 6.89 (d, J = 2.0 Hz, CH_{furan}), 7.17-7.30 (m, 6H, CH_{ar}), 7.06-7.08 (m, 2H, CH_{ar}), 7.24-7.35 (m, 3H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 7.7 ($CH_{2c\text{-propyl}}$), 13.9 (Me), 32.1 ($CH_{c\text{-propyl}}$), 110.4 and 139.0 (CH_{furan}), 127.3, 128.7 and 129.2 (CH_{ar}), 116.6, 142.2 and 157.5 ($C_{q\ ar}$), 166.9 (C=O); ***GC/MS***: r.t. = 16.9 min., M/Z = 241; ***HRMS*** calculated for $C_{15}H_{15}NO_2$ (M+H+) 242.1181, found 242.1186.

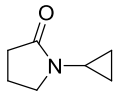


5e

***N*-cyclopropyl-*N*-(3-isopropoxyphenyl)-2-methylbenzamide (*N*-cyclopropyl Mepronil) (5e)** was obtained by procedure B from commercial Mepronil (0.2 mmol, 1.0 equiv.) as beige oil (44 mg, 70%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.54-0.56 (m, 2H, $CH_{2c\text{-propyl}}$), 0.80-0.82 (m, 2H, $CH_{2c\text{-propyl}}$), 1.22 (d, J = 6.0 Hz, 6H, $CH(\underline{CH_3})_2$), 2.34 (s, 3H, Me), 3.20 (br s, 1H, $CH_{c\text{-propyl}}$), 4.34-4.40 (m, 1H, $\underline{CH}(CH_3)_2$), 6.53-6.67 (m, 3H, CH_{ar}), 6.99-7.13 (m, 5H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 8.5 ($CH_{2c\text{-propyl}}$), 19.5 ($\underline{CH}(CH_3)_2$), 22.0 ($CH(\underline{CH_3})_2$), 31.6 ($CH_{c\text{-propyl}}$), 70.4 (Me), 114.7, 115.7, 119.9, 125.2, 126.7, 128.6, 129.3 and 130.2 (CH_{ar}),

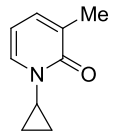
134.3, 137.7, 142.7 and 158.1 ($C_{q\ ar}$), 172.7 (C=O); ***GC/MS***: r.t. = 14.7 min., M/Z = 309; ***HRMS*** calculated for $C_{20}H_{23}NO_2$ (M+H+) 310.1807, found 310.1794.



5f

1-cyclopropylpyrrolidin-2-one (5f) was obtained by procedure B from commercial pyrrolidin-2-one (0.2 mmol, 1.0 equiv.) as beige oil (23 mg, 93%).

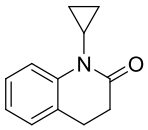
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.66-0.70 (m, 2H, $CH_{2c\text{-propyl}}$), 0.73-0.79 (m, 2H, $CH_{2c\text{-propyl}}$), 1.93-2.00 (m, 2H, $CH_{2pyrrol}$), 2.38 (t, *J* = 8.4 Hz, 2H, $CH_{2pyrrol}$), 2.61-2.66 (m, 1H, $CH_{c\text{-propyl}}$), 3.30 (t, *J* = 6.8 Hz, 2H, $CH_{2pyrrol}$); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 5.1 ($CH_{2c\text{-propyl}}$), 25.3 ($CH_{c\text{-propyl}}$), 18.2, 32.0 and 47.7 ($CH_{2pyrrol}$); ***GC/MS***: r.t. = 14.8 min., M/Z = 125; ***HRMS*** calculated for $C_7H_{11}NO$ (M+H+) 126.0841, found 126.0852.



5g

1-cyclopropyl-3-methylpyridin-2(*1H*)-one (5g) was obtained by procedure B from commercial 3-methylpyridin-2(*1H*)-one (0.2 mmol, 1.0 equiv.) as beige oil (27 mg, 90%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.81-0.85 (m, 2H, $CH_{2c\text{-propyl}}$), 1.07-1.13 (m, 2H, $CH_{2c\text{-propyl}}$), 1.93-2.00 (m, 2H, $CH_{2pyrrol}$), 2.13 (s, 3H, Me), 3.29-3.36 (m, 1H, $CH_{c\text{-propyl}}$), 6.03 (t, *J* = 6.8 Hz, 1H, CH_{ar}), 7.15 (d, *J* = 7.6 Hz, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 6.9 ($CH_{2c\text{-propyl}}$), 17.2 (Me), 32.4 ($CH_{c\text{-propyl}}$), 105.3, 134.2 and 136.2 (CH_{ar}), 129.7 (C_{qar}), 164.7 (C=O); ***GC/MS***: r.t. = 15.3 min., M/Z = 149; ***HRMS*** calculated for $C_9H_{11}NO$ (M+H+) 150.0841, found 150.0834.

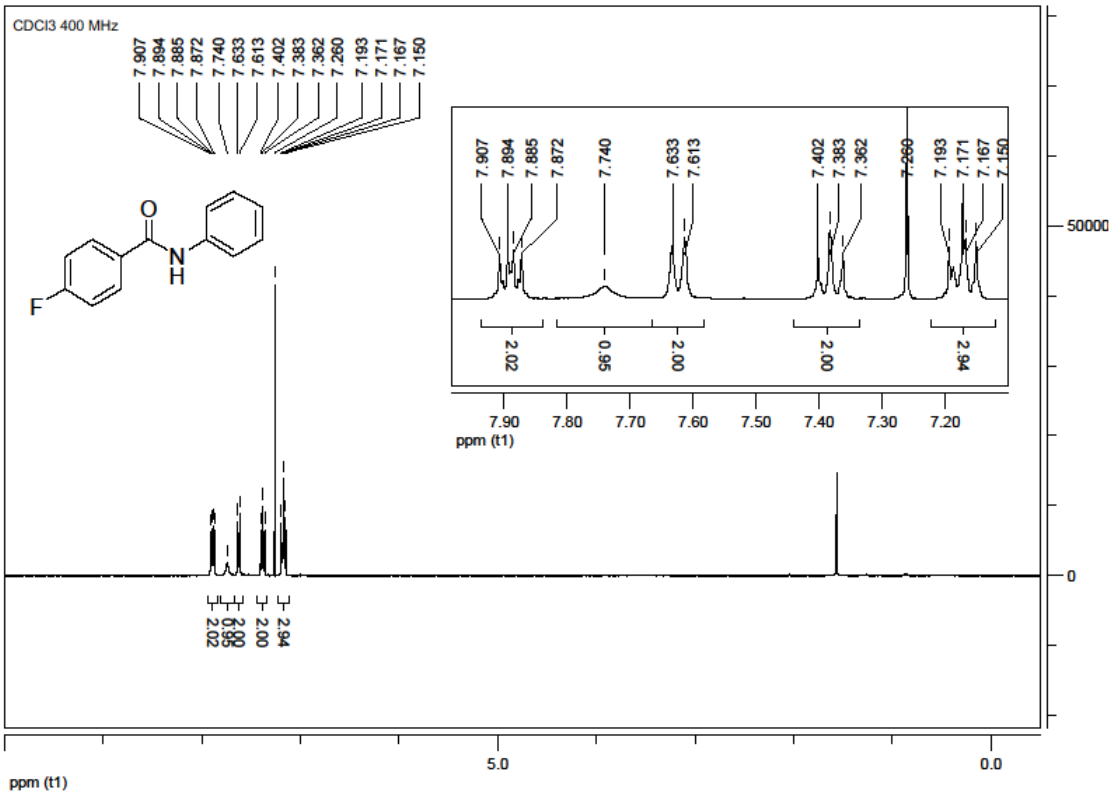


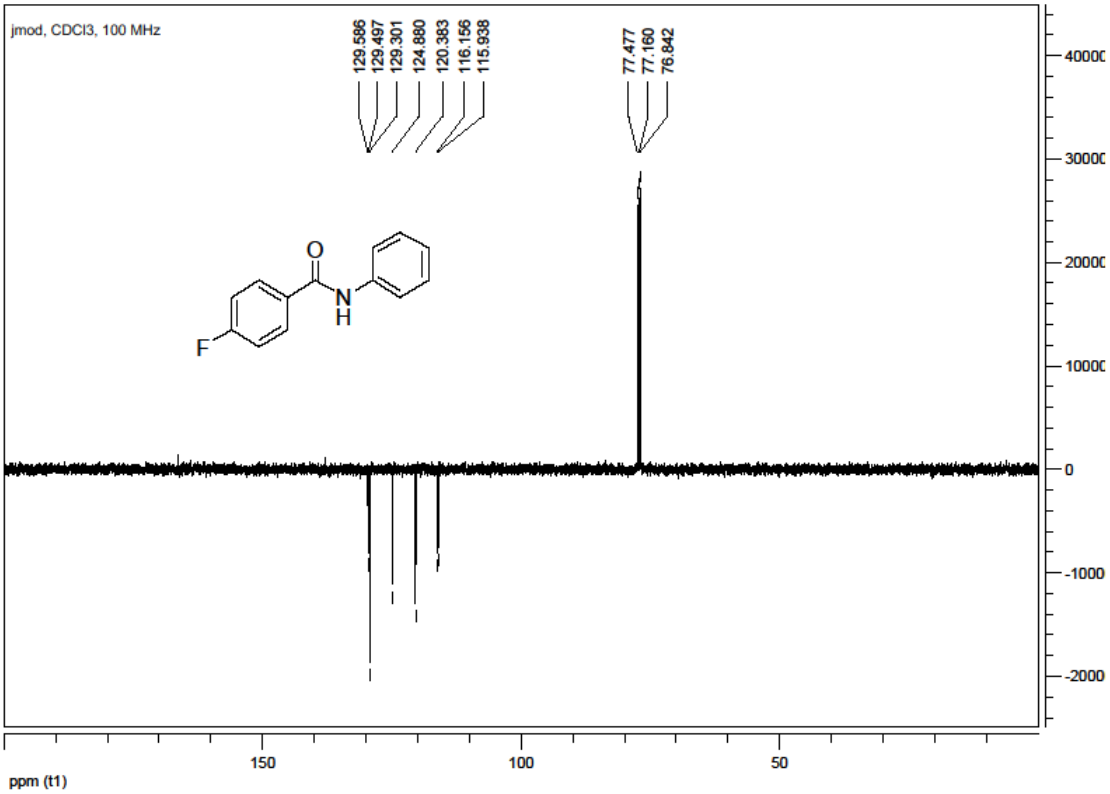
5h

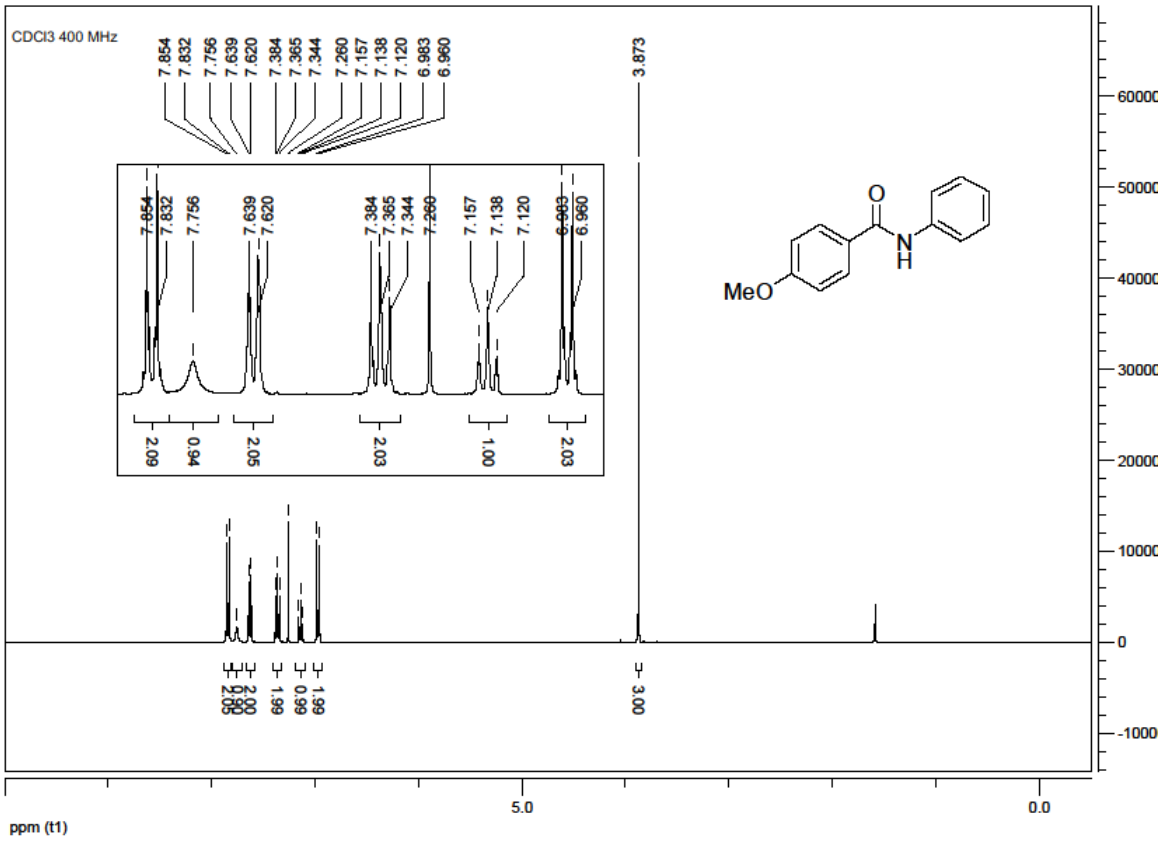
1-cyclopropyl-3,4-dihydroquinolin-2(*1H*)-one (5h) was obtained by procedure B from commercial 3,4-dihydroquinolin-2(*1H*)-one (0.2 mmol, 1.0 equiv.) as beige crystals (34 mg, 92%).

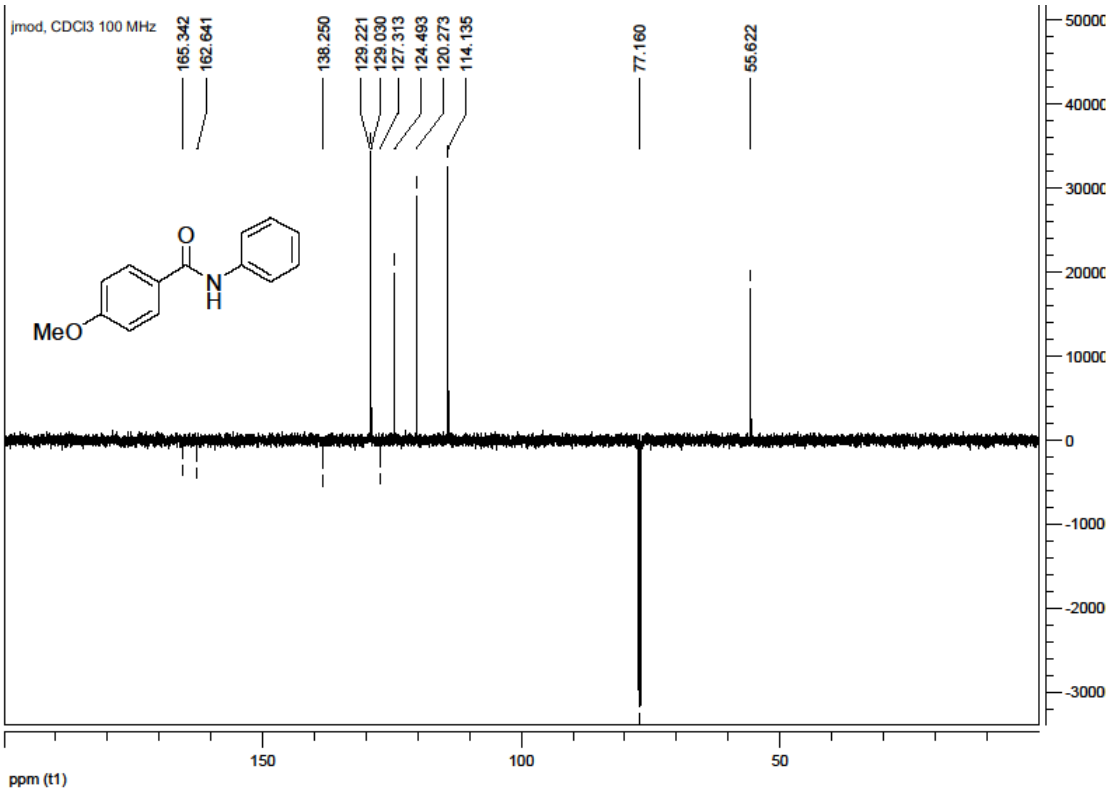
^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.56-0.61 (m, 2H, $CH_{2c\text{-propyl}}$), 1.04-1.09 (m, 2H, $CH_{2c\text{-propyl}}$), 2.52-2.56 (m, 2H, $CH_{2quinoline}$), 2.65-2.75 (m, 3H, $CH_{2quinoline}$ and $CH_{c\text{-propyl}}$), 6.93 (td, *J* = 1.2 and 7.2 Hz, 1H, CH_{ar}), 7.06 (br d, *J* = 7.2 Hz, 1H, CH_{ar}), 7.16-7.24 (m, 2H, CH_{ar}); ***^{13}C NMR*** (100 MHz, $CDCl_3$) δ: 9.9 ($CH_{2c\text{-propyl}}$), 25.3 ($CH_{c\text{-propyl}}$),25.4 and 32.9 ($CH_{2quinoline}$), 116.5, 122.9, 127.2 and 127.4 (CH_{ar}), 126.9 and 140.9 ($C_{q\ ar}$), 172.8 (C=O); m.p. 79-80 °C, ***GC/MS***: r.t. = 14.8 min., M/Z = 187; ***HRMS*** calculated for $C_{20}H_{23}NO_2$ (M+H+) 188.0997, found 188.0989.

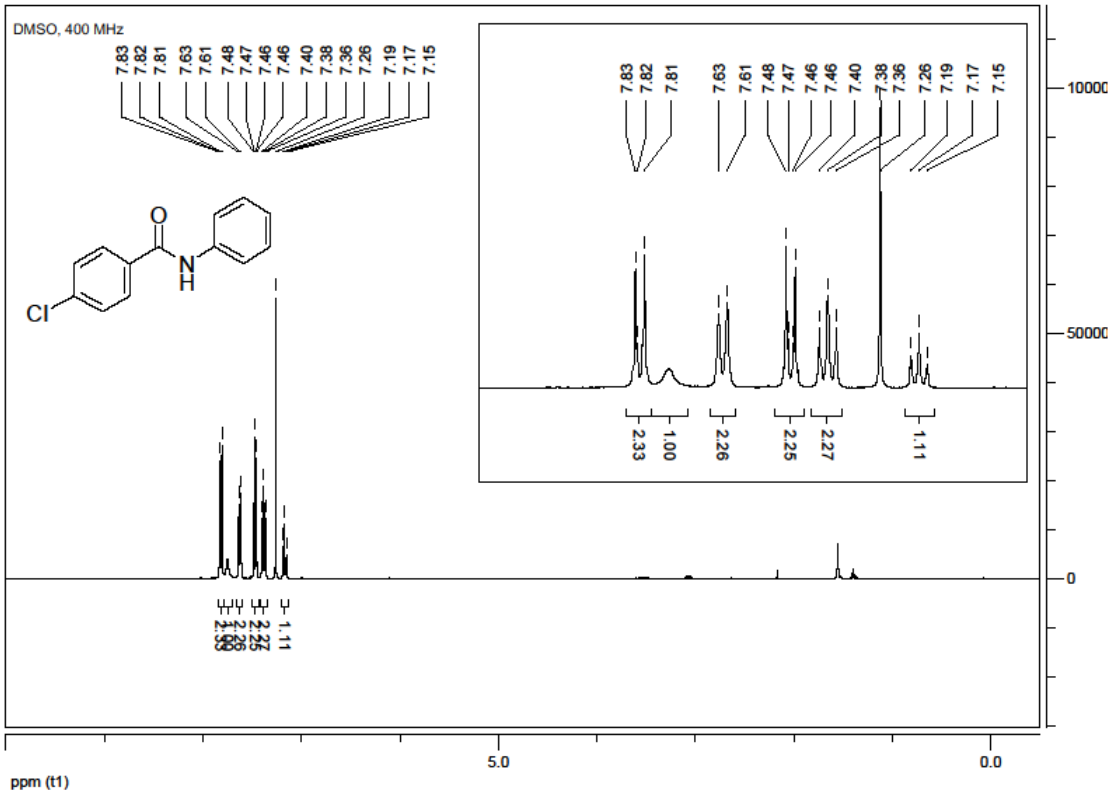
IV- ^{1}H and ^{13}C NMR spectra

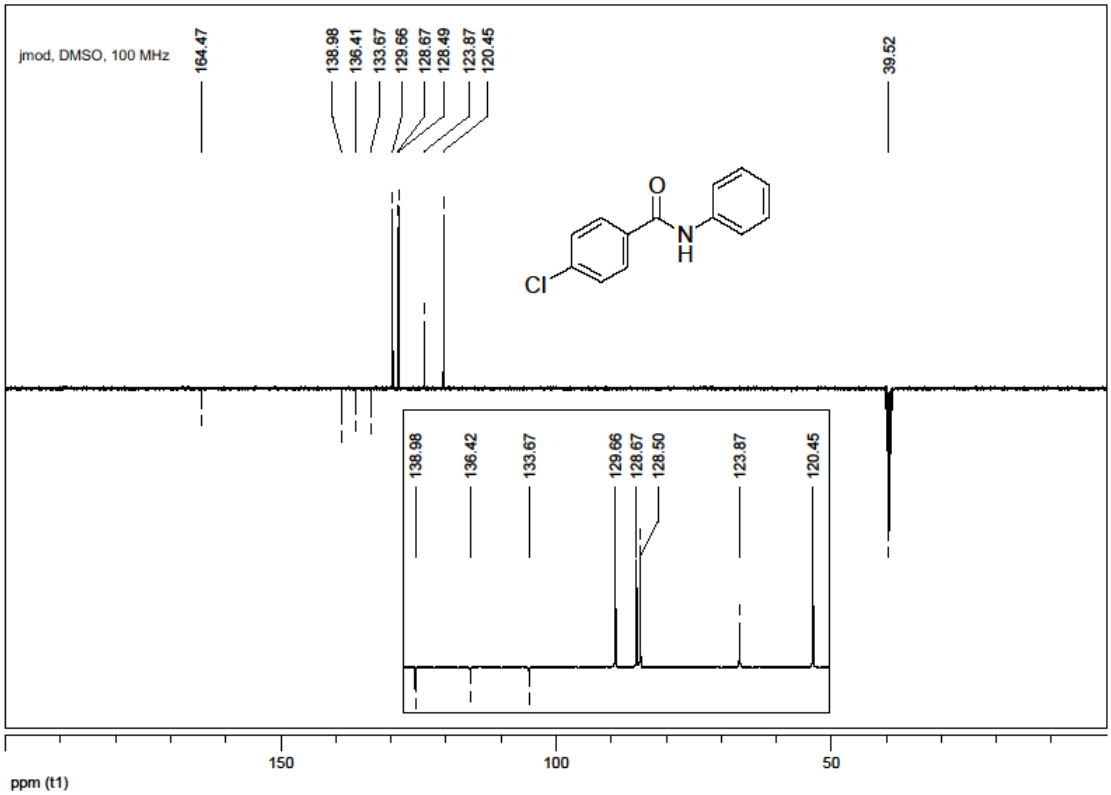


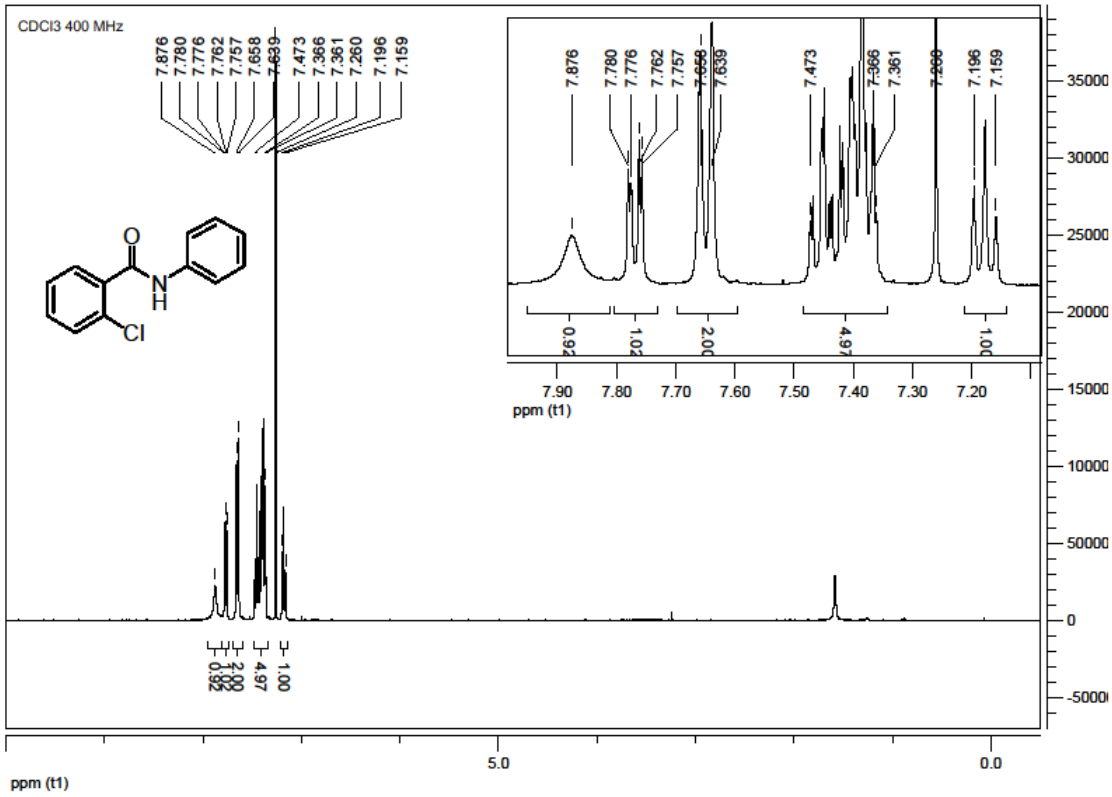


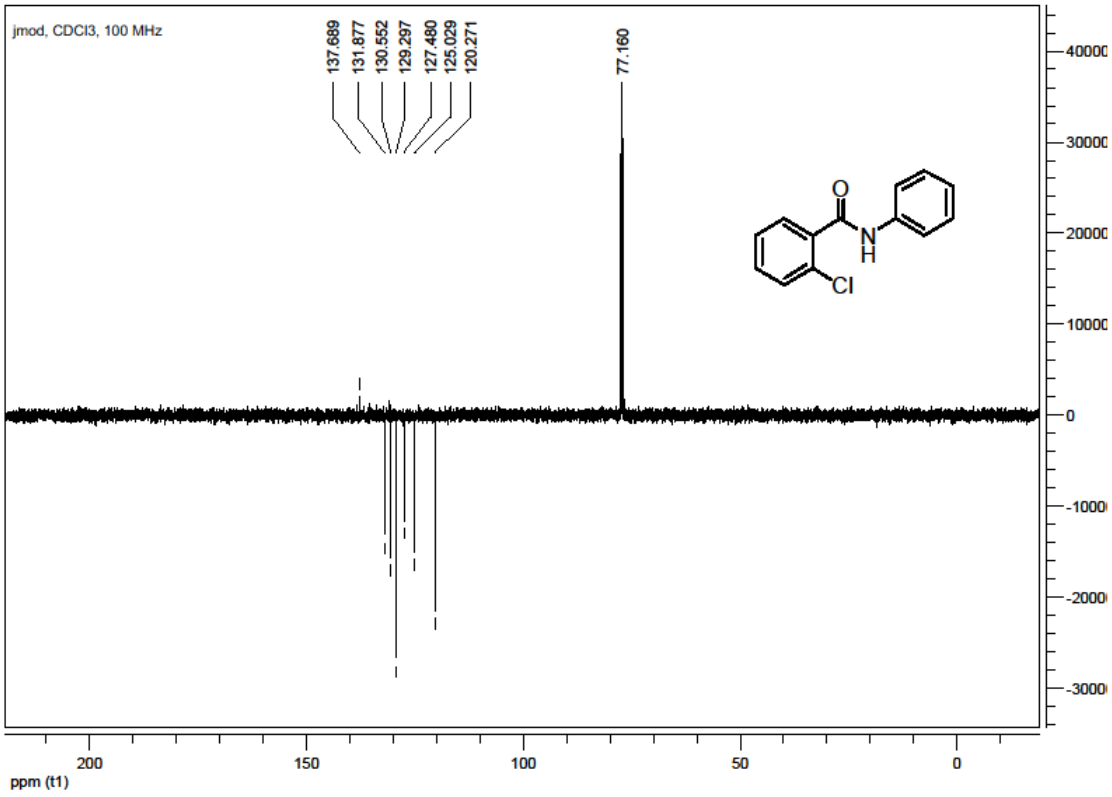

CDCl3 400 MHz
7.854
7.832
7.756
7.639
7.620
7.384
7.365
7.344
7.260
7.157
7.138
7.120
6.983
6.960
3.873
2.09
0.94
2.06
2.03
1.00
2.03
1.99
0.99
1.99
3.00
MeO
O
N
H
ppm (t1)

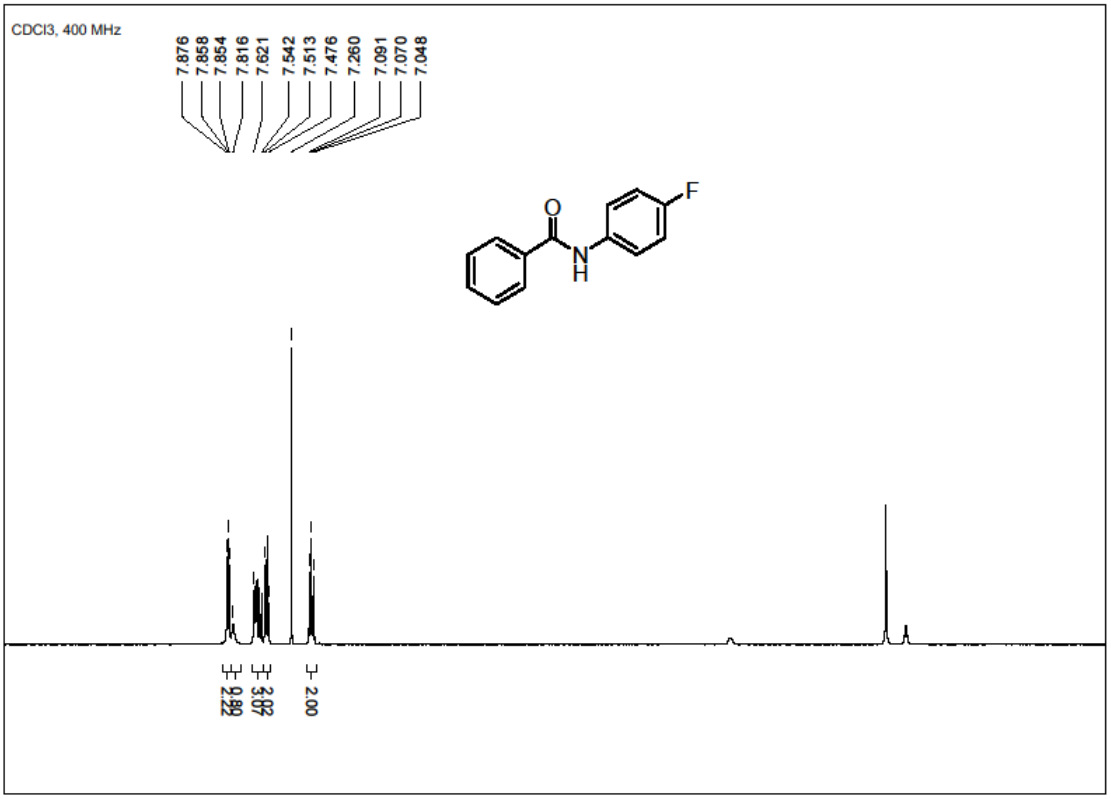

jmod, CDCl3 100 MHz
165.342
162.641
138.250
129.221
129.030
127.313
124.493
120.273
114.135
77.160
55.622
MeO
O
N
H
ppm (t1)

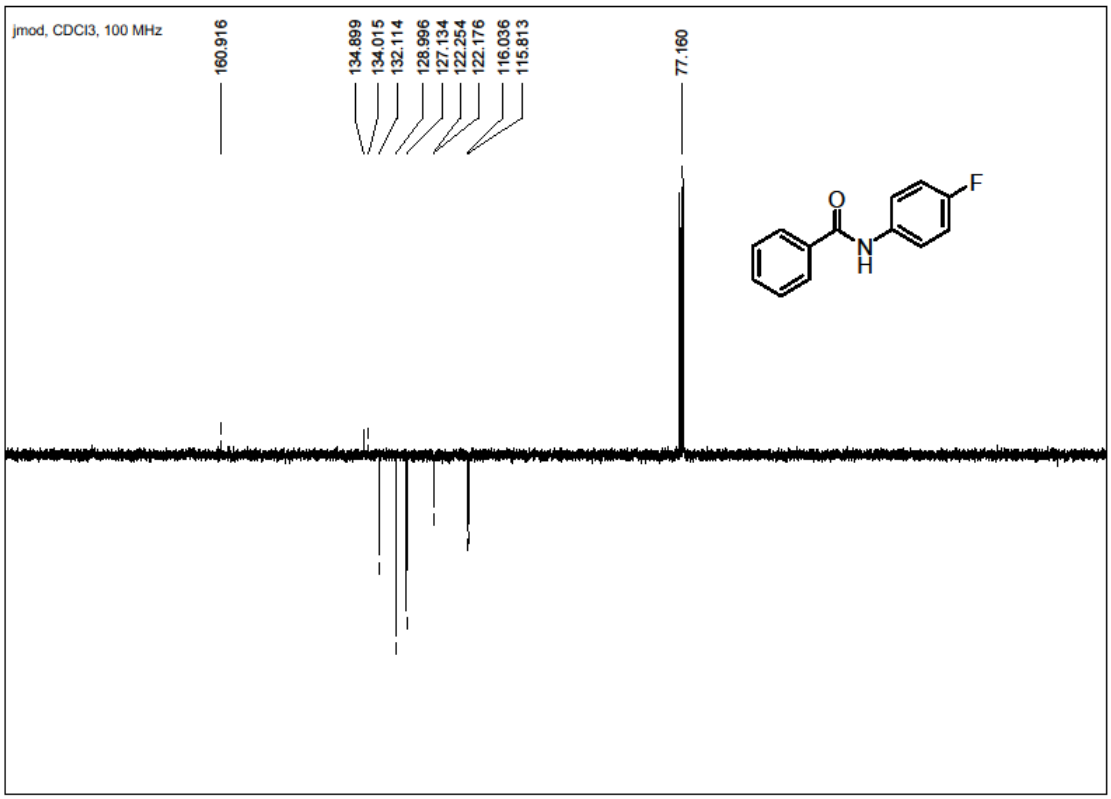

DMSO, 400 MHz
7.83
7.82
7.81
7.63
7.61
7.48
7.47
7.46
7.46
7.40
7.38
7.36
7.26
7.19
7.17
7.15
O
N
H
Cl
2.33
1.00
2.26
2.25
2.27
1.11
10000
50000
0
5.0
0.0
ppm (t1)

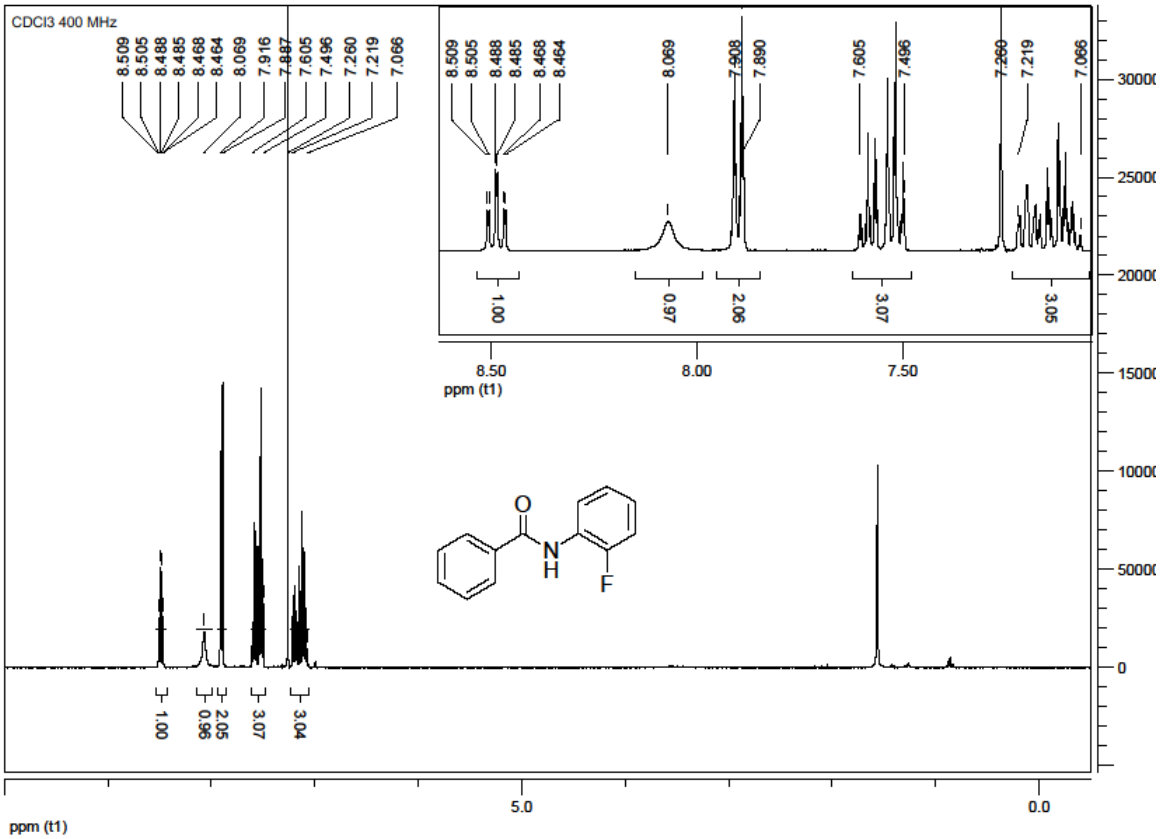

jmod, DMSO, 100 MHz
164.47
138.98
136.41
133.67
129.66
128.67
128.49
123.87
120.45
39.52
O
N
H
Cl
138.98
136.42
133.67
129.66
128.67
128.50
123.87
120.45
150
100
50
ppm (t1)

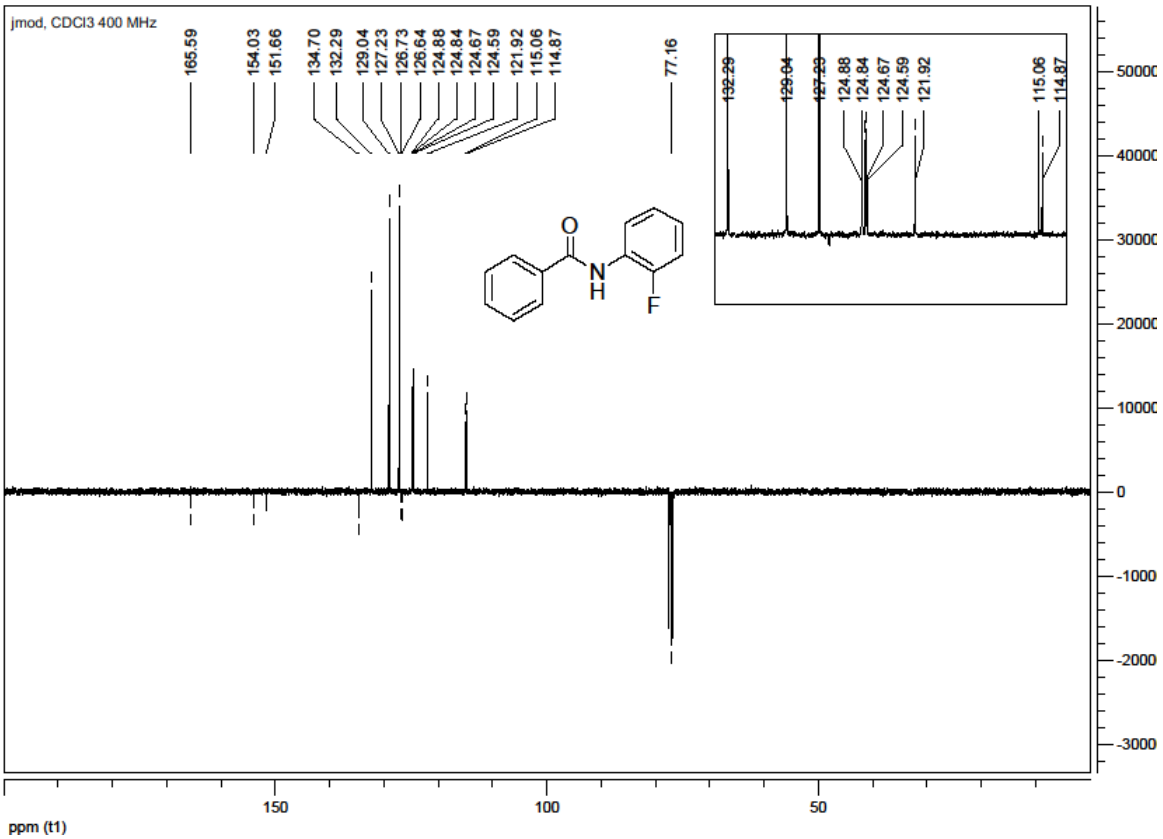

CDCl3 400 MHz
7.876
7.780
7.776
7.762
7.757
7.658
7.639
7.473
7.366
7.361
7.260
7.196
7.159
O
N
H
Cl
0.92
1.04
2.00
4.97
1.00
7.90
7.80
7.70
7.60
7.50
7.40
7.30
7.20
ppm (t1)
35000
30000
25000
20000
15000
10000
50000
0
-5000
5.0
0.0
ppm (t1)

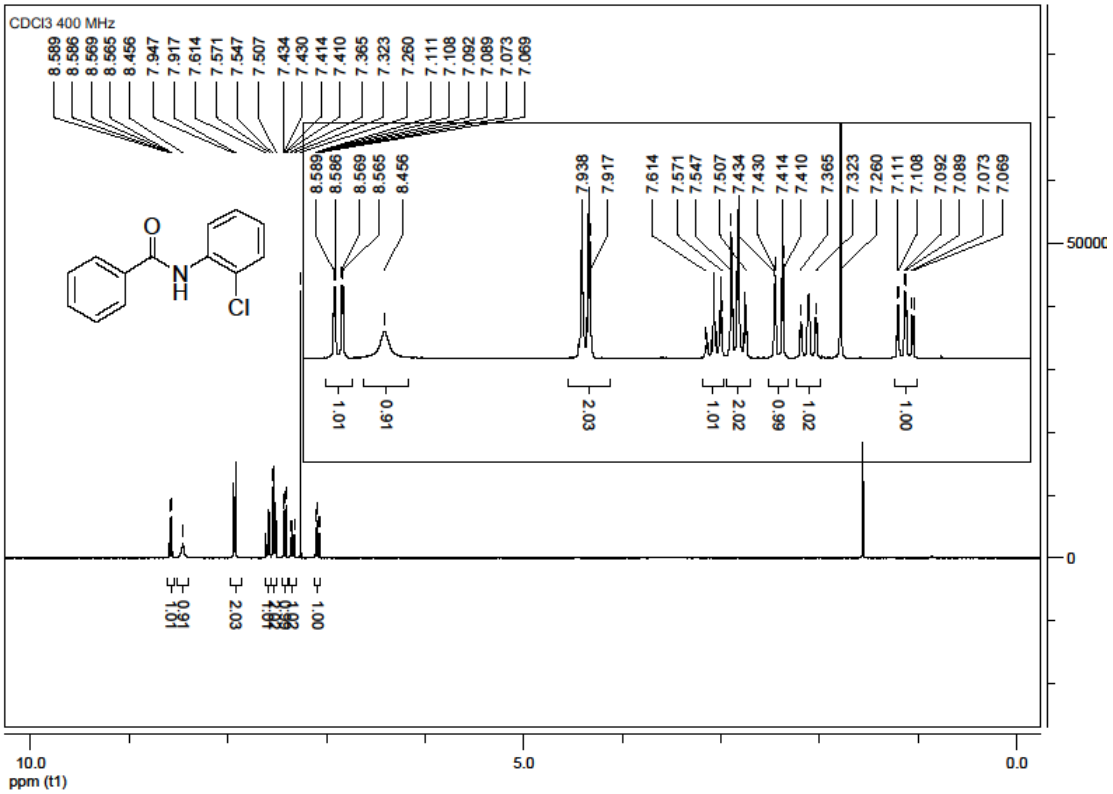

jmod, CDCl3, 100 MHz
137.689
131.877
130.552
129.297
127.480
125.029
120.271
77.160
O
N
H
Cl
40000
30000
20000
10000
0
-10000
-20000
-30000
200
150
100
50
0
ppm (t1)

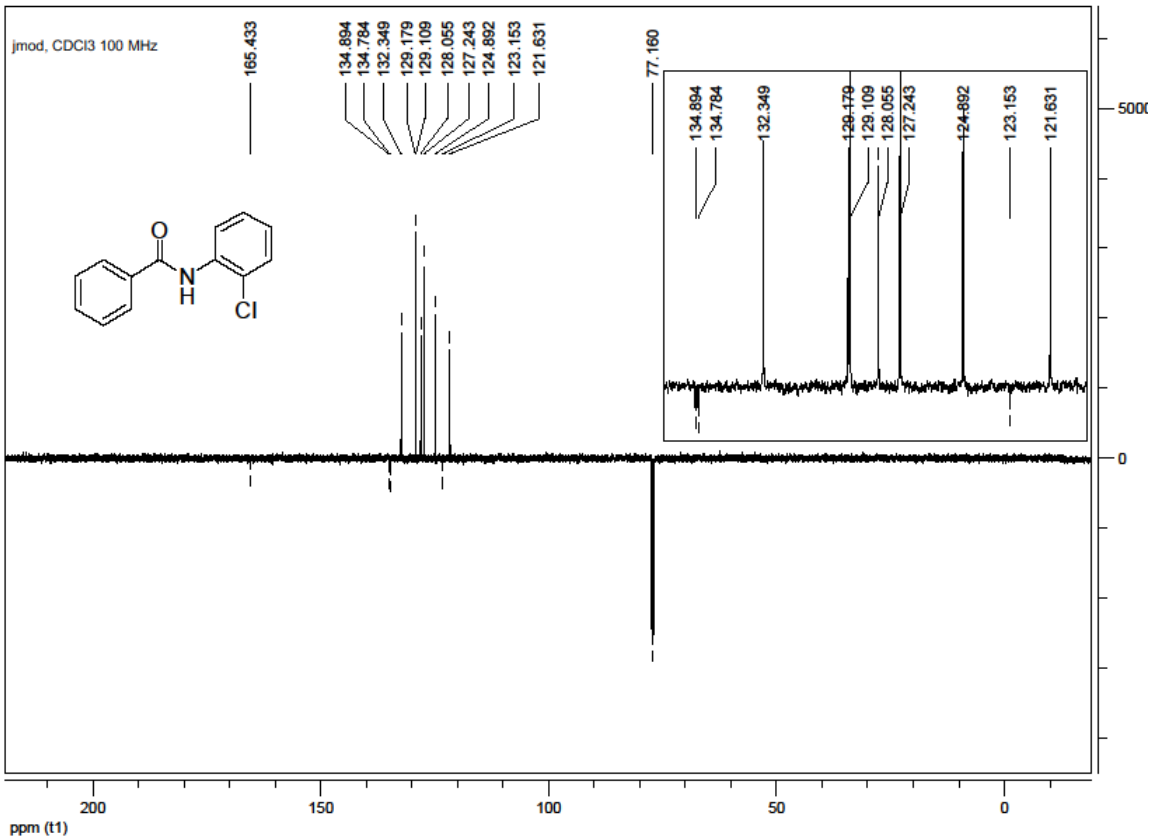

CDCl3, 400 MHz
7.876
7.858
7.854
7.816
7.621
7.542
7.513
7.476
7.260
7.091
7.070
7.048
O
N
H
F
2.00

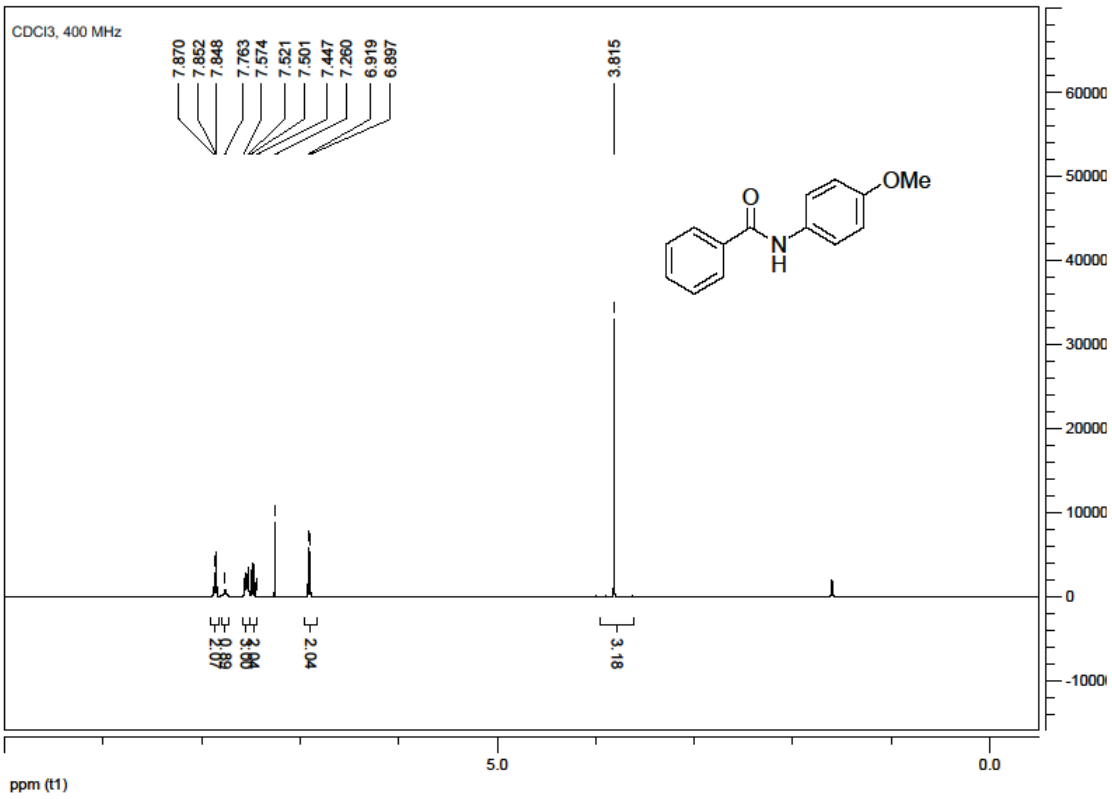

jmod, CDCl3, 100 MHz
160.916
134.899
134.015
132.114
128.996
127.134
122.254
122.176
116.036
115.813
77.160
O
N
H
F

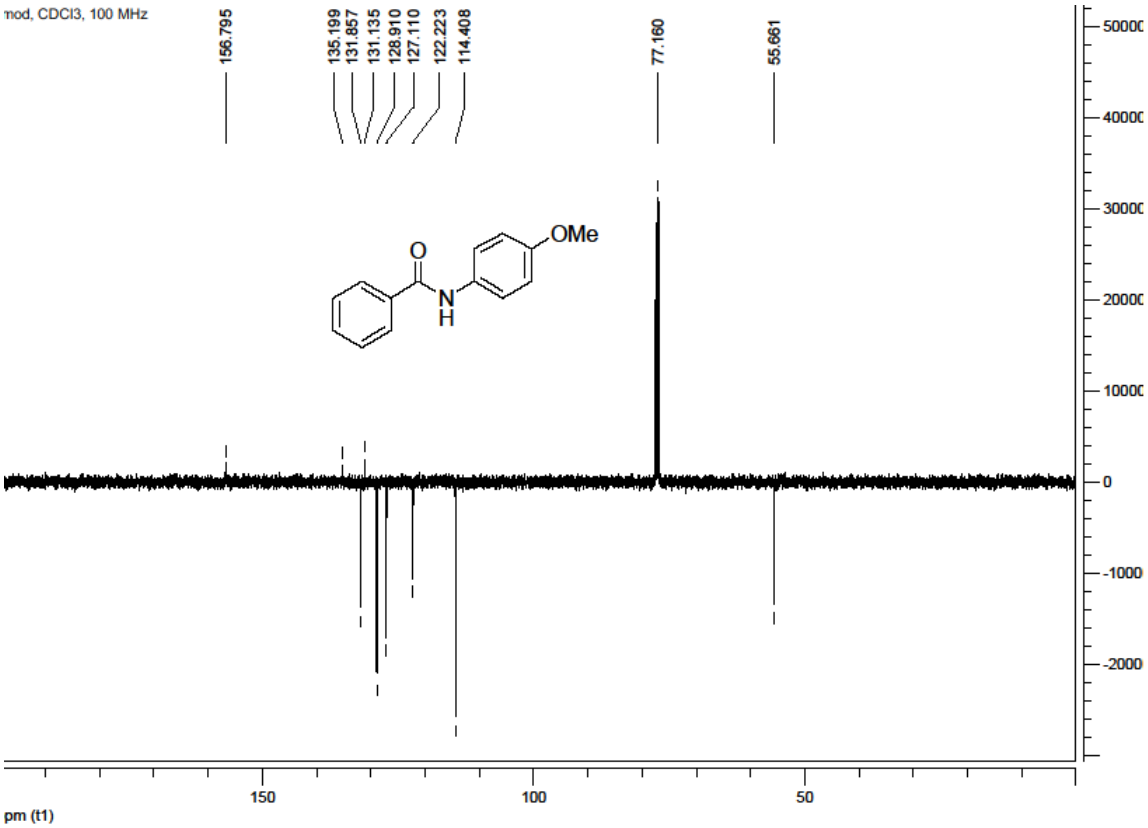

CDCl3 400 MHz
8.509
8.505
8.488
8.485
8.468
8.464
8.069
7.916
7.887
7.605
7.496
7.260
7.219
7.066
1.00
0.96
2.05
3.07
3.04
ppm (t1)

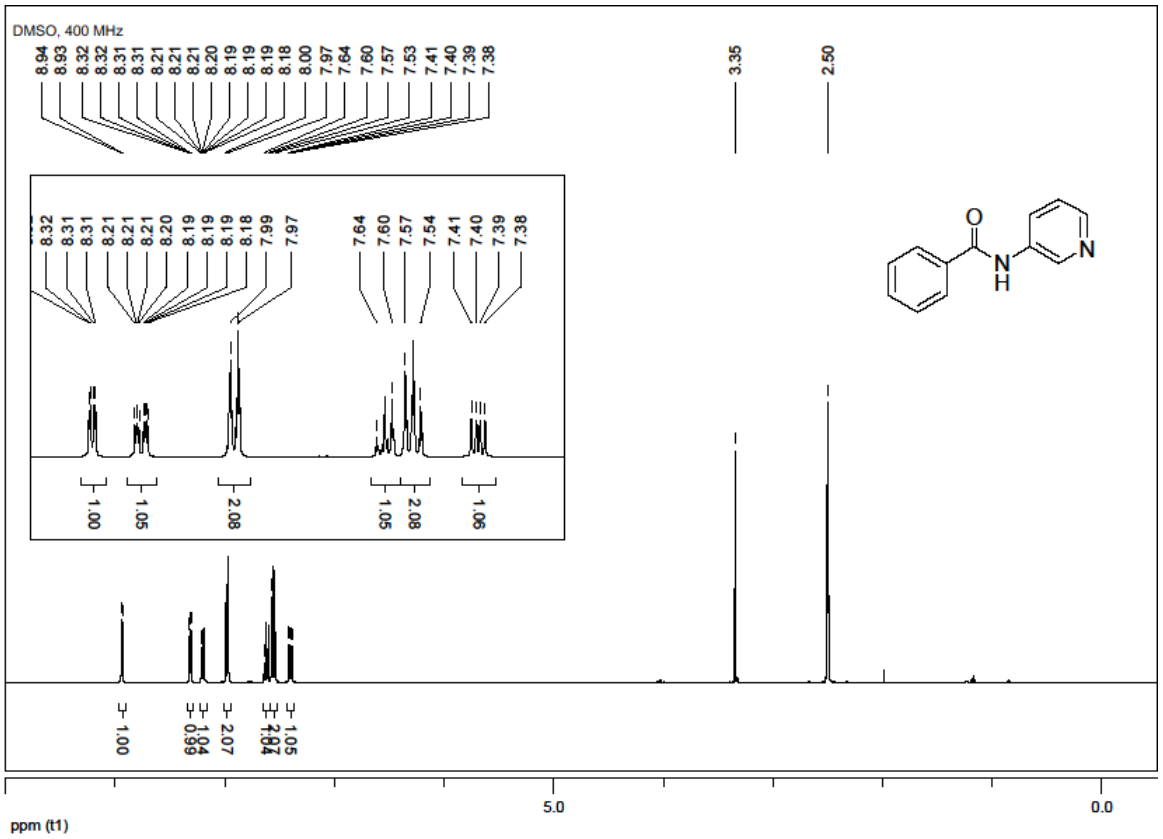

jmod, CDCl3 400 MHz
165.59
154.03
151.66
134.70
132.29
129.04
127.23
126.73
126.64
124.88
124.84
124.67
124.59
121.92
115.06
114.87
77.16
ppm (t1)

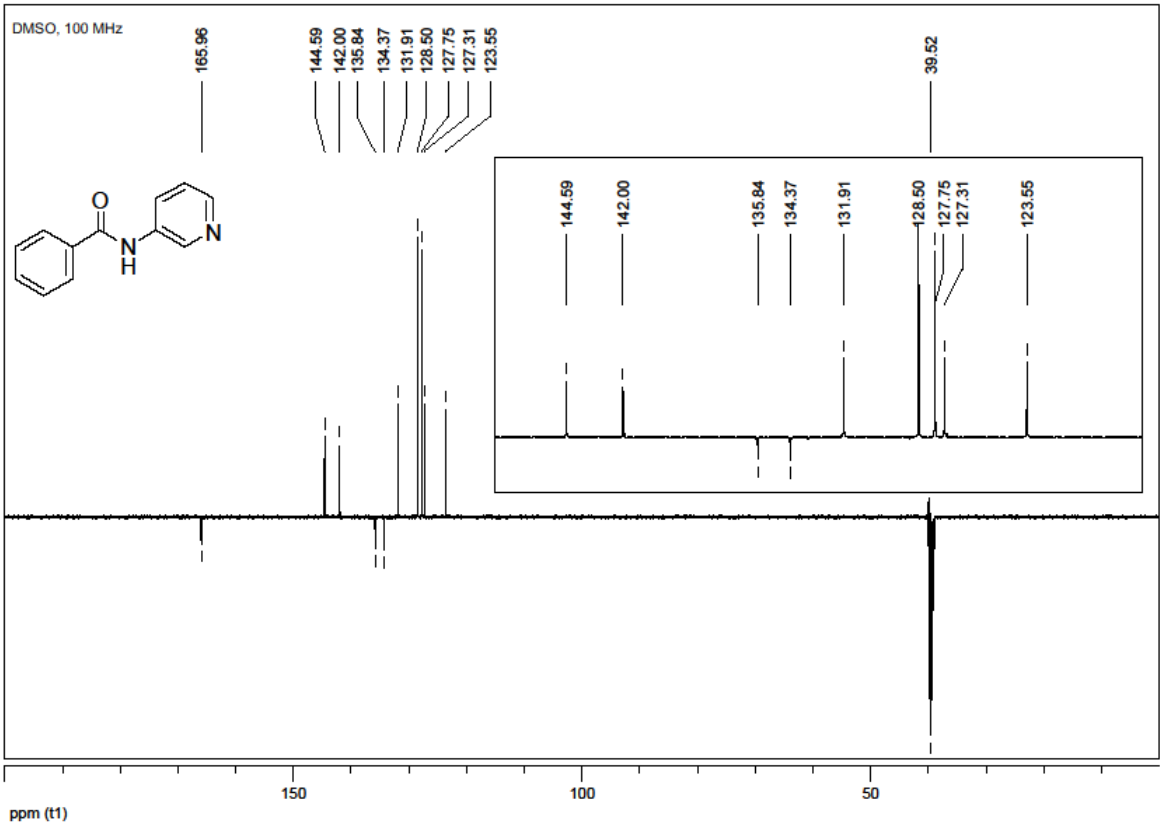

CDCl3 400 MHz
8.589
8.586
8.569
8.565
8.456
7.947
7.917
7.614
7.571
7.547
7.507
7.434
7.430
7.414
7.410
7.365
7.323
7.260
7.111
7.108
7.092
7.089
7.073
7.069
7.938
7.917
7.614
7.571
7.547
7.507
7.434
7.430
7.414
7.410
7.365
7.323
7.260
7.111
7.108
7.092
7.089
7.073
7.069
1.01
0.91
2.03
1.01
2.02
0.99
1.02
1.00
50000
0
10.0
5.0
0.0
ppm (t1)

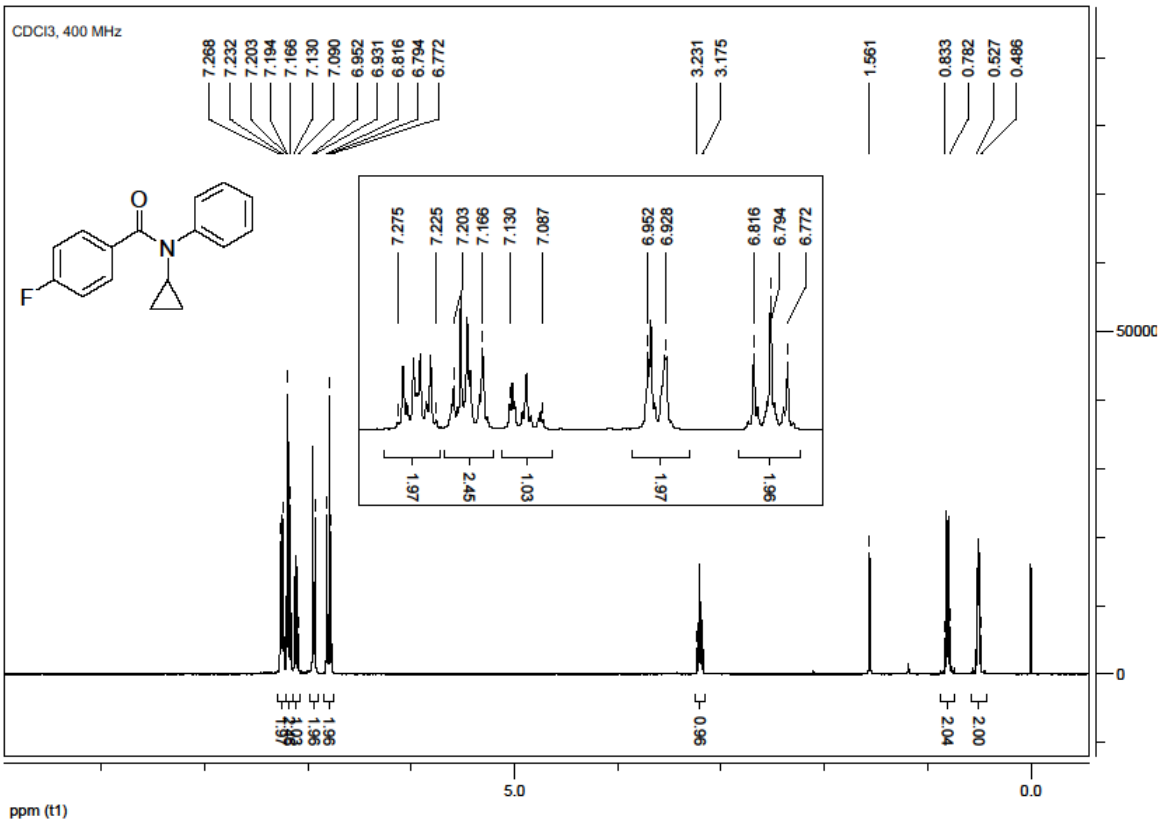

jmod, CDCl3 100 MHz
165.433
134.894
134.784
132.349
129.179
129.109
128.055
127.243
124.892
123.153
121.631
77.160
134.894
134.784
132.349
129.179
129.109
128.055
127.243
124.892
123.153
121.631
500
0
200
150
100
50
0
ppm (t1)

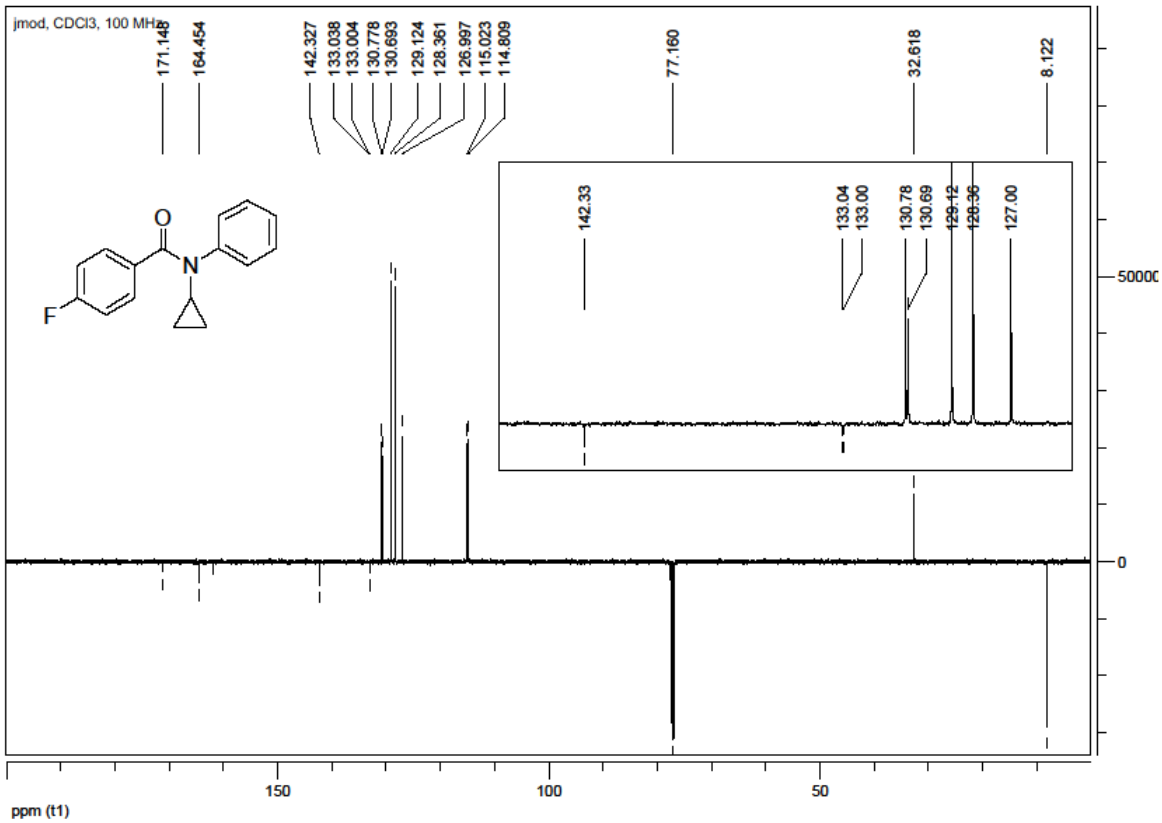

CDCl3, 400 MHz
7.870
7.852
7.848
7.763
7.574
7.521
7.501
7.447
7.260
6.919
6.897
3.815
OMe
O
N
H
2.04
3.18
5.0
0.0
ppm (t1)


mod, CDCl3, 100 MHz
156.795
135.199
131.857
131.135
128.910
127.110
122.223
114.408
77.160
55.661
OMe
O
N
H
150
100
50
pm (t1)


DMSO, 400 MHz
8.94
8.93
8.32
8.32
8.31
8.31
8.21
8.21
8.21
8.20
8.19
8.19
8.19
8.18
8.00
7.97
7.64
7.60
7.57
7.53
7.41
7.40
7.39
7.38
3.35
2.50
1.00
1.05
2.08
1.05
2.08
1.06
1.00
0.99
2.07
2.04
1.05
5.0
0.0
ppm (t1)


DMSO, 100 MHz
165.96
144.59
142.00
135.84
134.37
131.91
128.50
127.75
127.31
123.55
39.52
150
100
50
ppm (t1)


CDCl3, 400 MHz
7.268
7.232
7.203
7.194
7.166
7.130
7.090
6.952
6.931
6.816
6.794
6.772
3.231
3.175
1.561
0.833
0.782
0.527
0.486
7.275
7.225
7.203
7.166
7.130
7.087
6.952
6.928
6.816
6.794
6.772
1.97
2.45
1.03
1.97
1.96
0.96
2.04
2.00
5.0
0.0
ppm (t1)


jmod, CDCl3, 100 MHz
171.148
164.454
142.327
133.038
133.004
130.778
130.693
129.124
128.361
126.997
115.023
114.809
77.160
32.618
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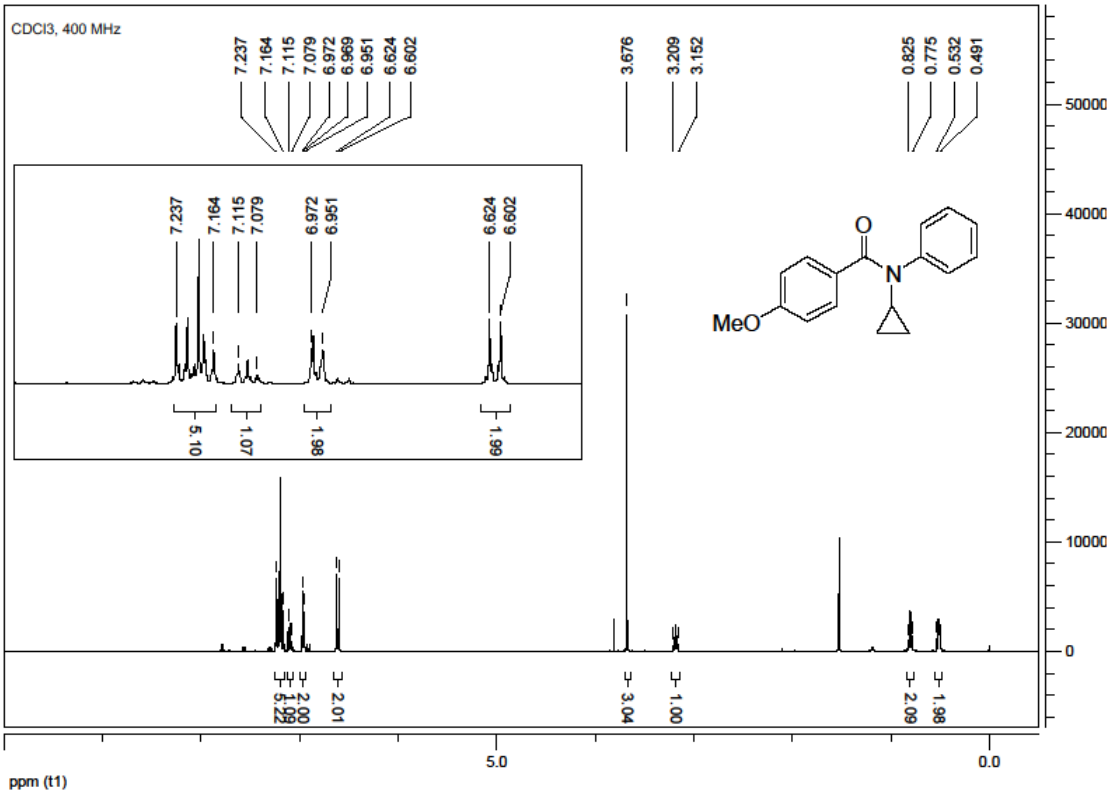
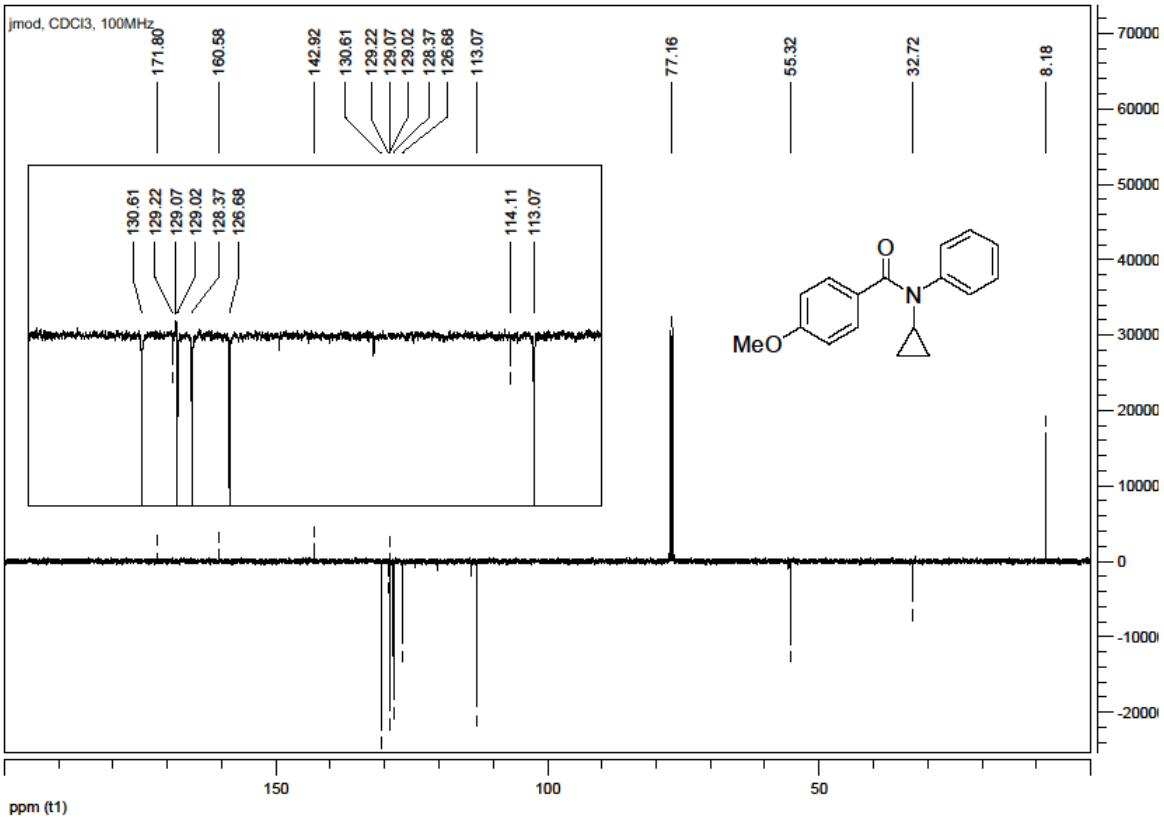
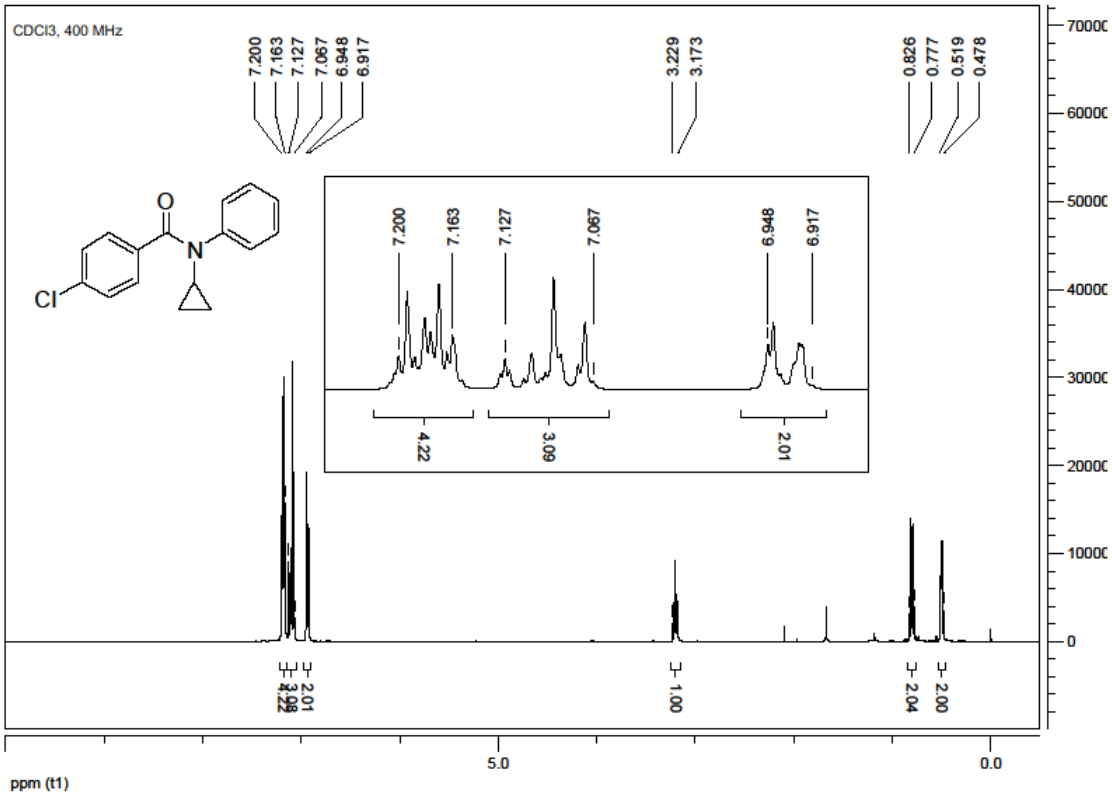
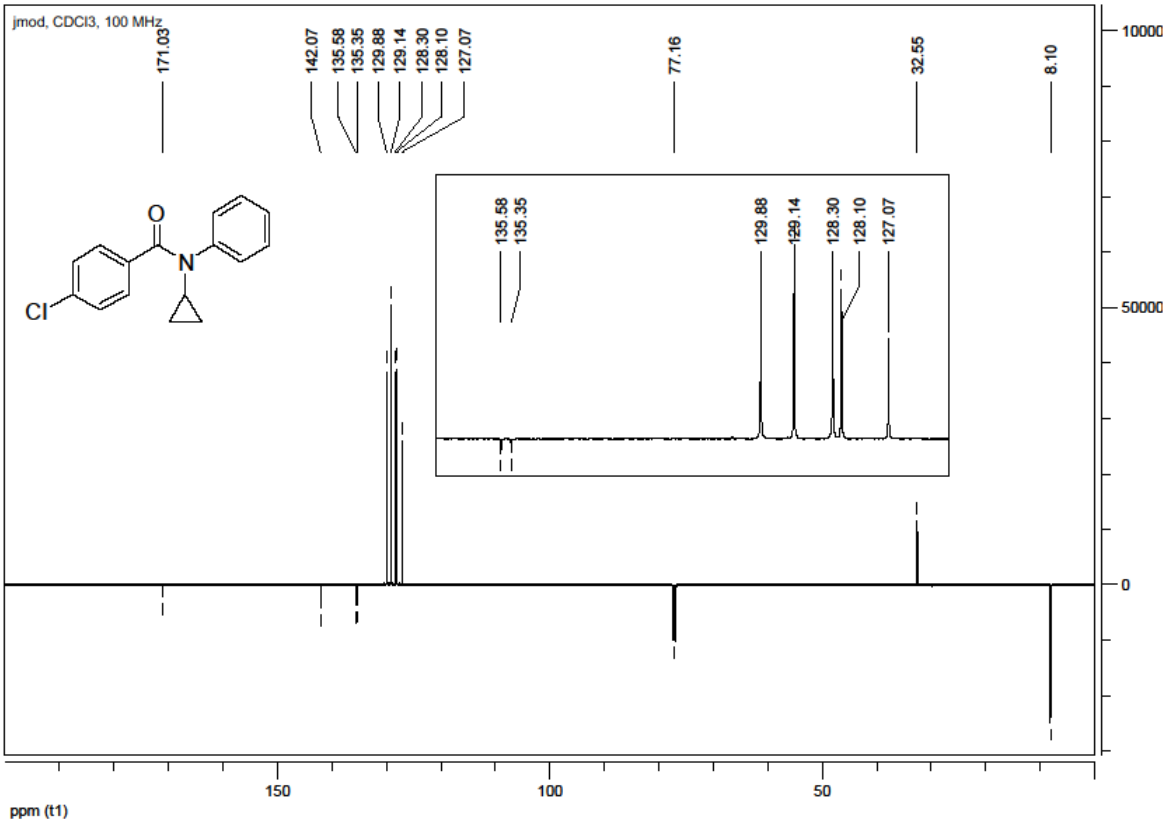
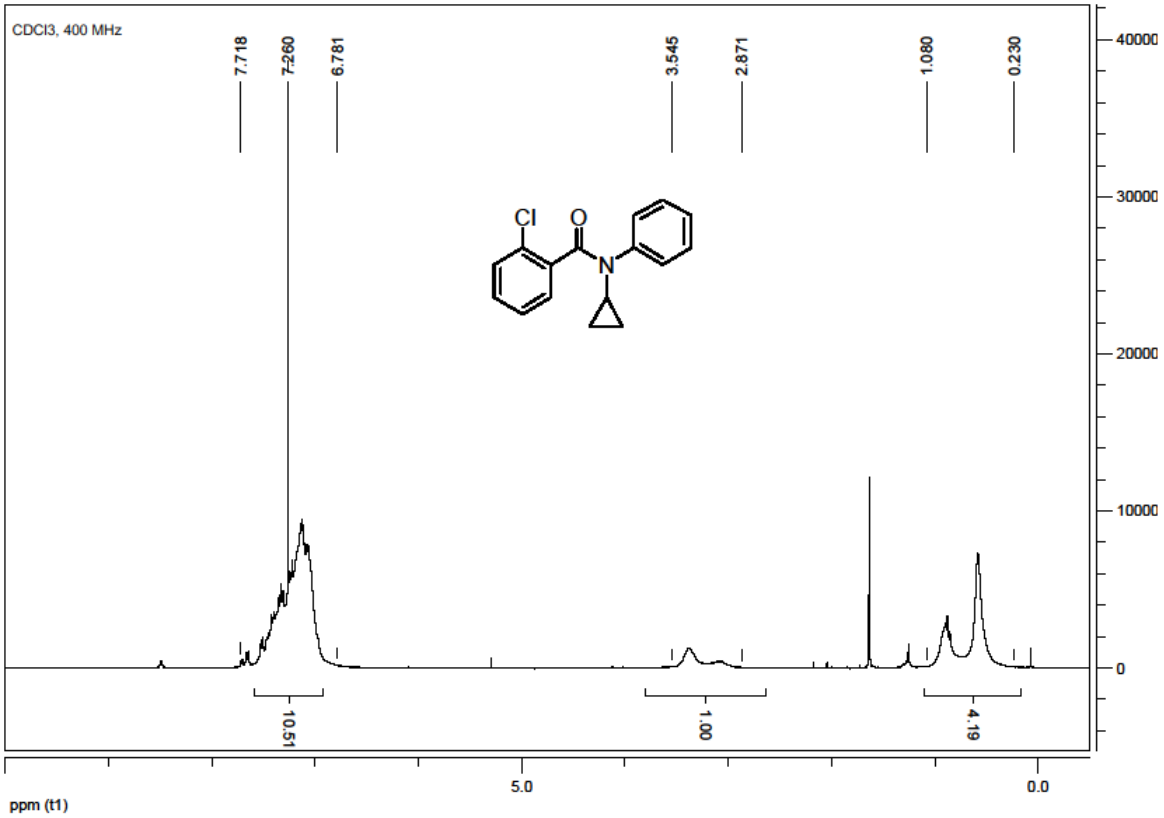
128.36
127.00
150
100
50
ppm (t1)

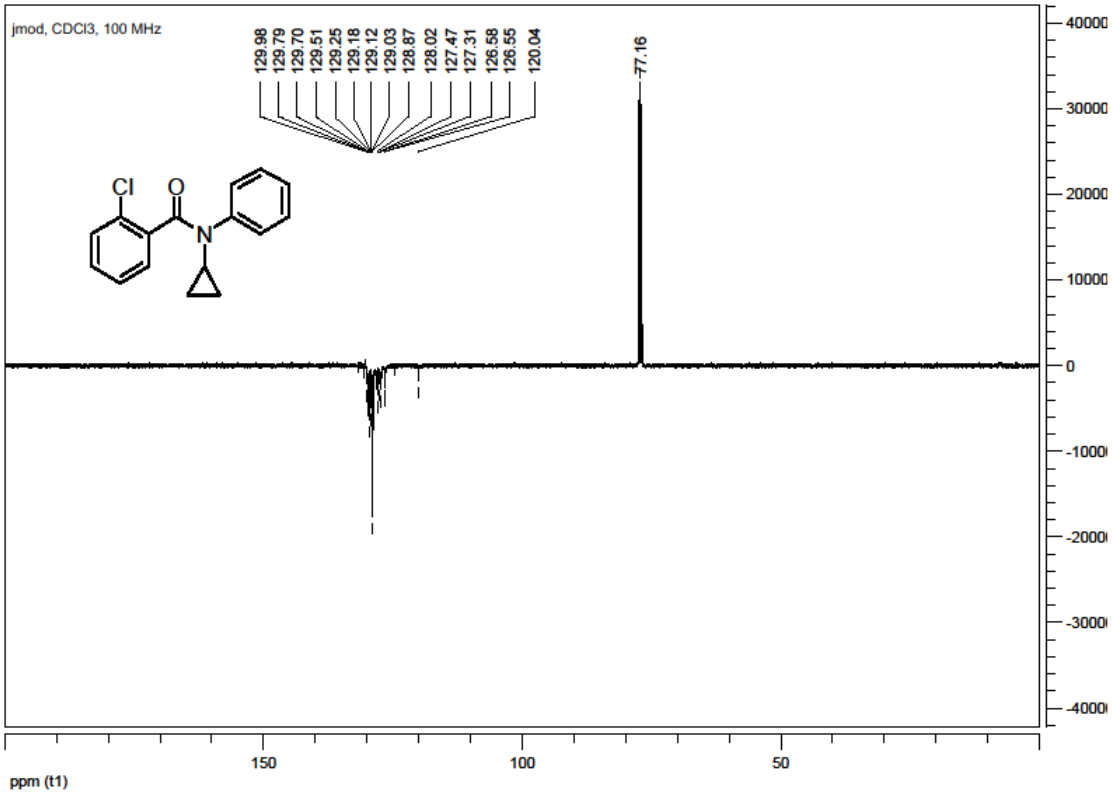

CDCl3, 400 MHz
7.237
7.164
7.115
7.079
6.972
6.969
6.951
6.624
6.602
3.676
3.209
3.152
0.825
0.775
0.532
0.491
5.10
1.07
1.98
1.99
2.01
3.04
1.00
2.09
1.98
MeO
O
N
ppm (t1)

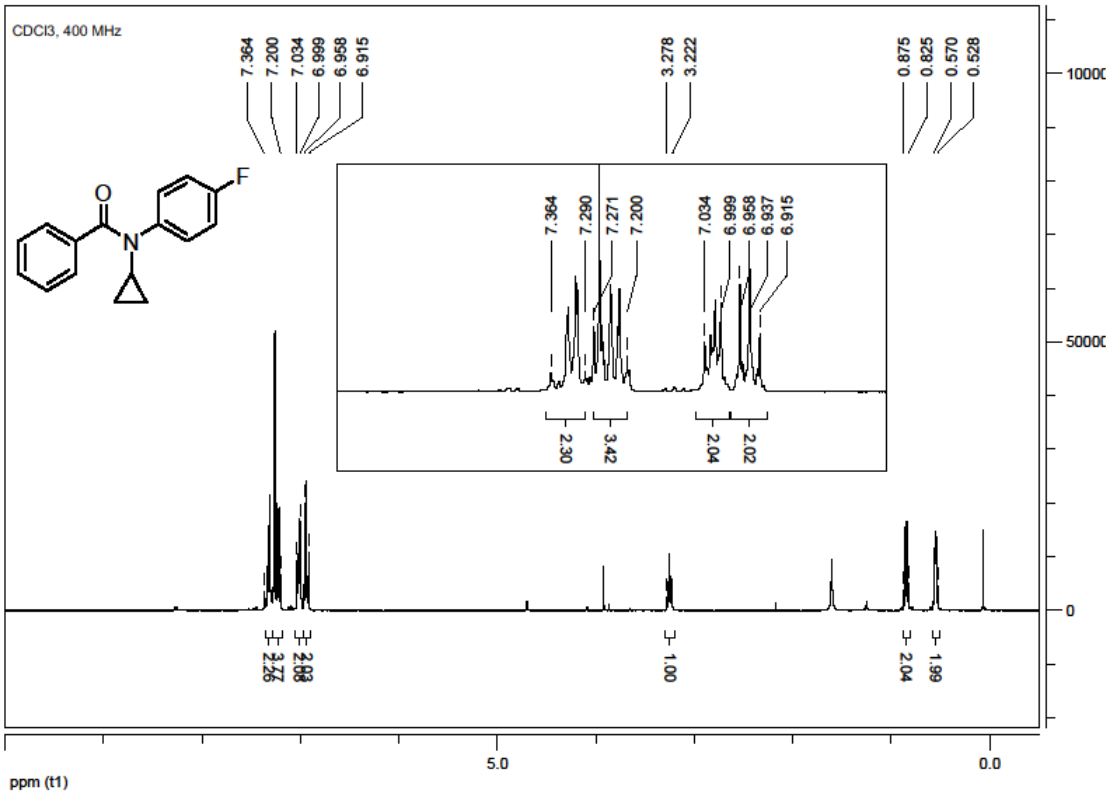

jmod, CDCl3, 100MHz
171.80
160.58
142.92
130.61
129.22
129.07
129.02
128.37
126.68
113.07
77.16
55.32
32.72
8.18
114.11
MeO
O
N
ppm (t1)

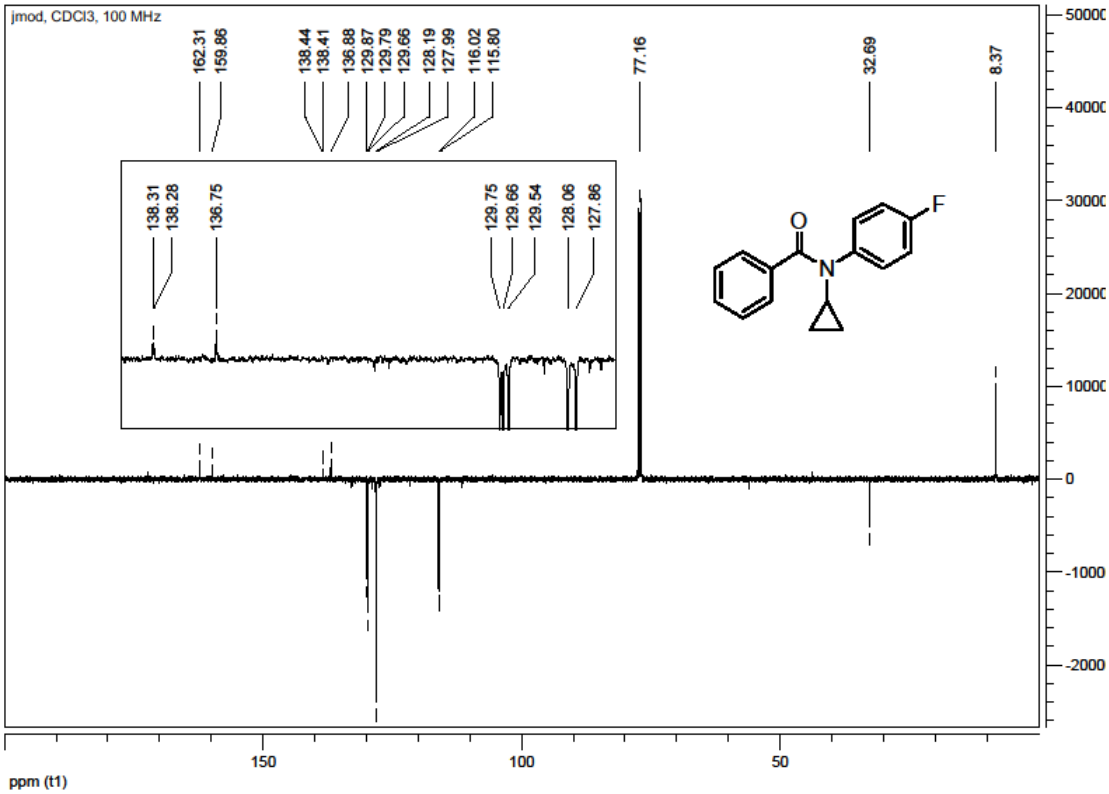

CDCl3, 400 MHz
7.200
7.163
7.127
7.067
6.948
6.917
3.229
3.173
0.826
0.777
0.519
0.478
4.22
3.09
2.01
2.01
1.00
2.04
2.00
ppm (t1)

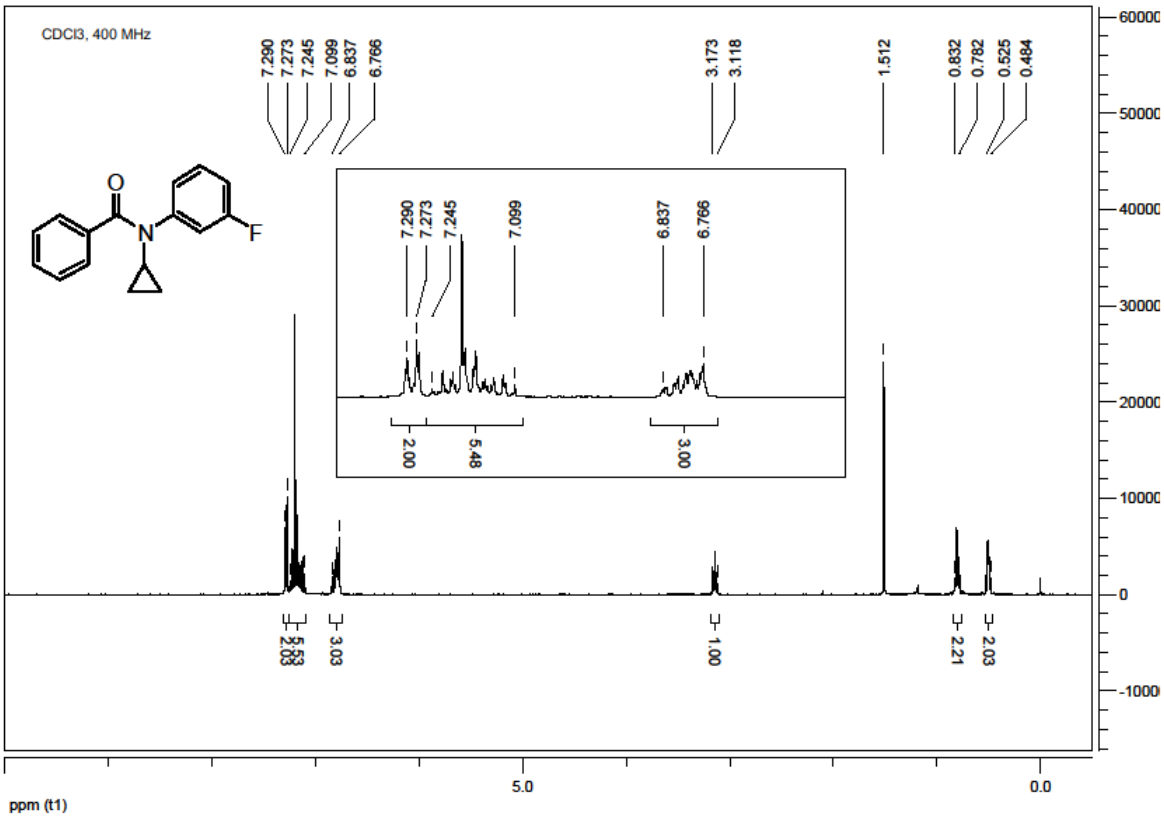

jmod, CDCl3, 100 MHz
171.03
142.07
135.58
135.35
129.88
129.14
128.30
128.10
127.07
77.16
32.55
8.10
ppm (t1)

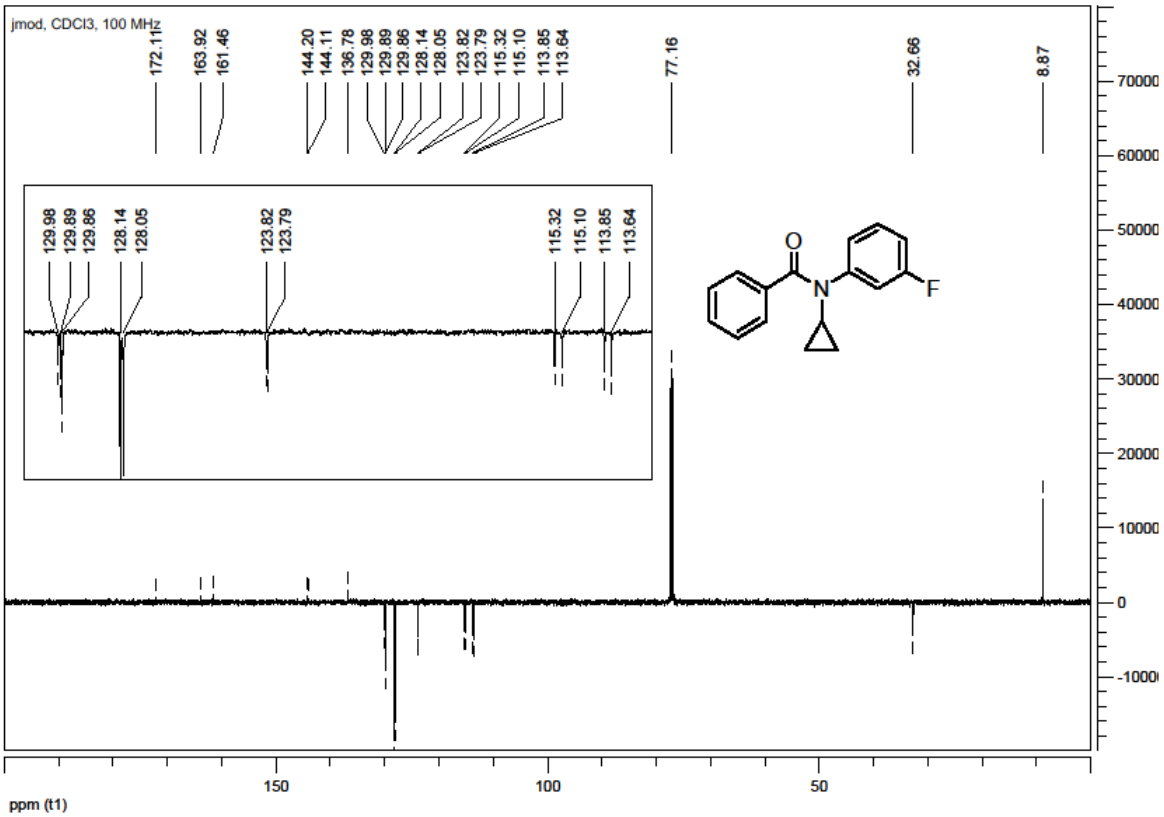

CDCl3, 400 MHz
7.718
7.260
6.781
3.545
2.871
1.080
0.230
Cl
O
N
40000
30000
20000
10000
0
10.51
1.00
4.19
5.0
0.0
ppm (t1)

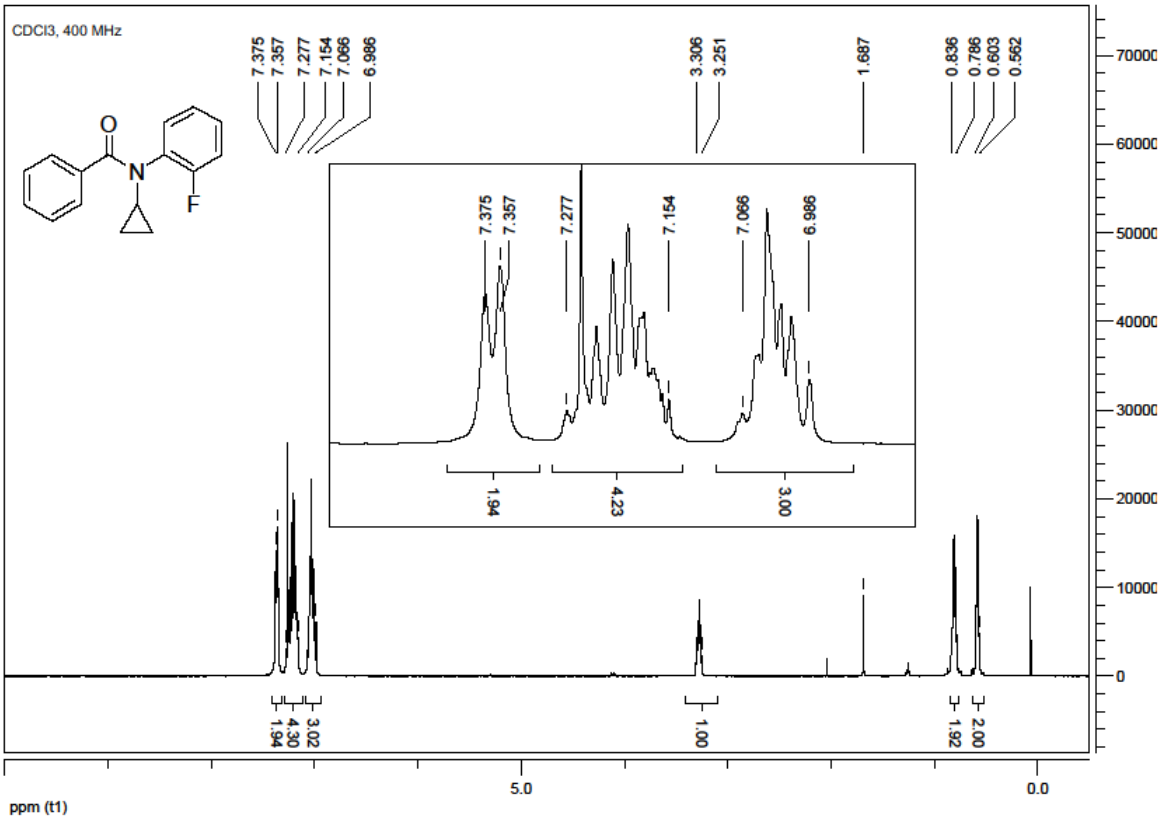

jmod, CDCl3, 100 MHz
129.98
129.79
129.70
129.51
129.25
129.18
129.12
129.03
128.87
128.02
127.47
127.31
126.58
126.55
120.04
77.16
Cl
O
N
40000
30000
20000
10000
0
-10000
-20000
-30000
-40000
150
100
50
ppm (t1)

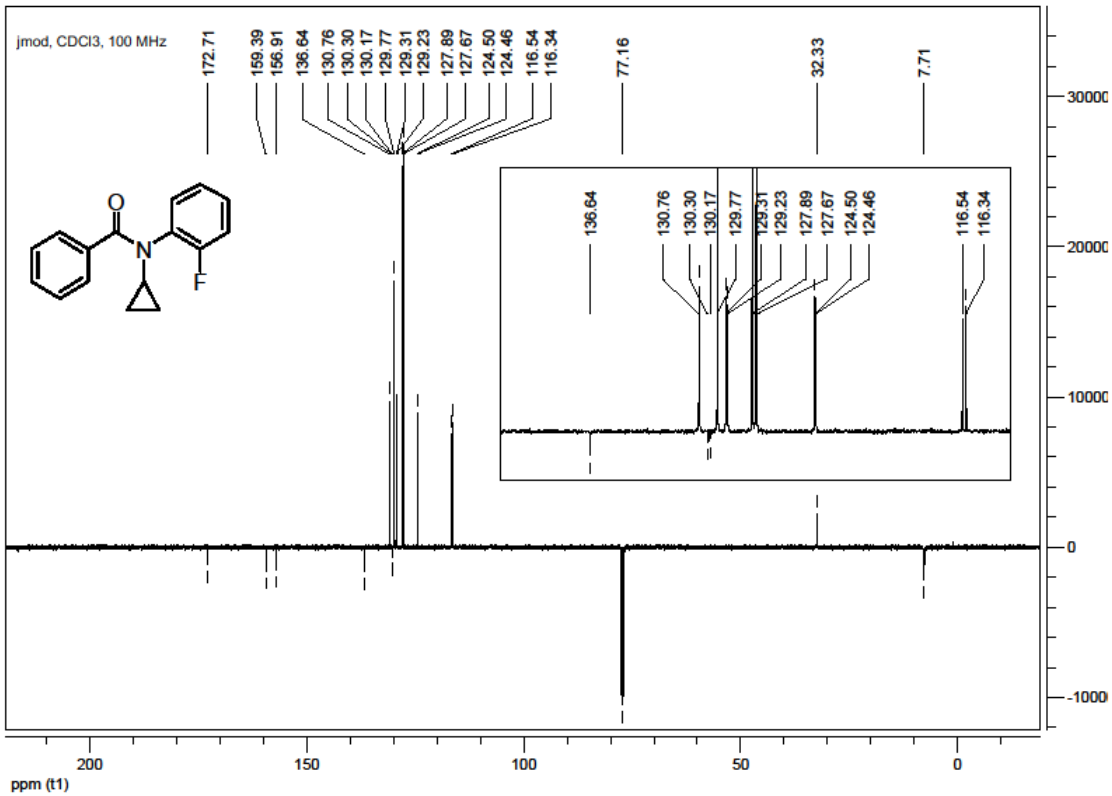

CDCl3, 400 MHz
7.364
7.200
7.034
6.999
6.958
6.915
3.278
3.222
0.875
0.825
0.570
0.528
7.364
7.290
7.271
7.200
7.034
6.999
6.958
6.937
6.915
2.30
3.42
2.04
2.02
2.26
2.08
2.00
2.04
1.99
O
N
F
5.0
0.0
ppm (t1)

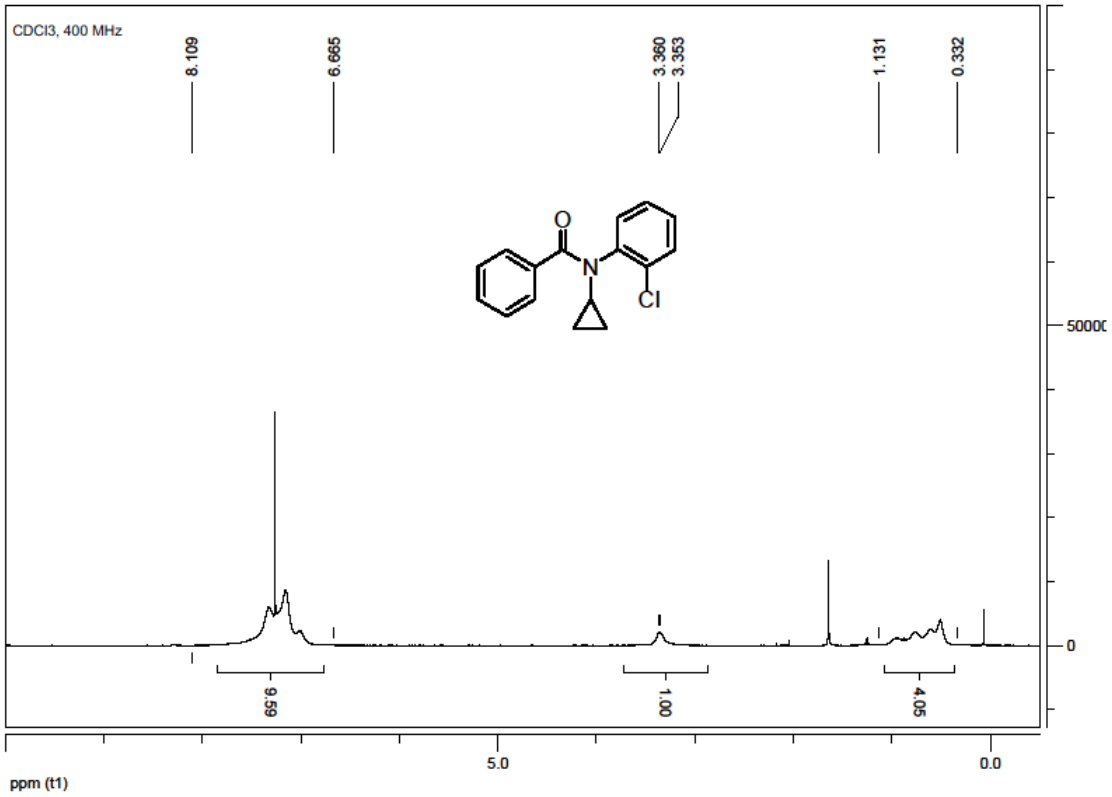

jmod, CDCl3, 100 MHz
162.31
159.86
138.44
138.41
136.88
129.87
129.79
129.66
128.19
127.99
116.02
115.80
77.16
32.69
8.37
138.31
138.28
136.75
129.75
129.66
129.54
128.06
127.86
O
N
F
150
100
50
ppm (t1)

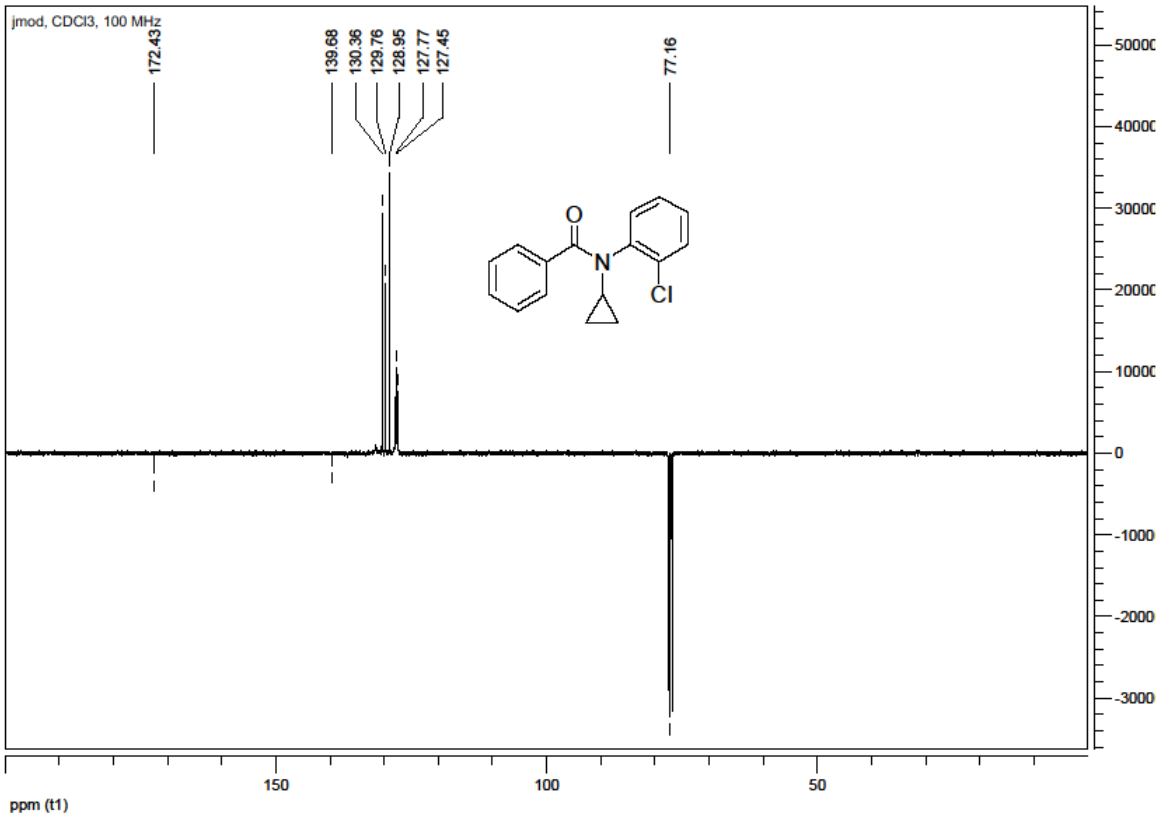

CDCl3, 400 MHz
7.290
7.273
7.245
7.099
6.837
6.766
3.173
3.118
1.512
0.832
0.782
0.525
0.484
O
N
F
2.00
5.48
3.00
2.03
3.03
1.00
2.21
2.03
5.0
0.0
ppm (t1)

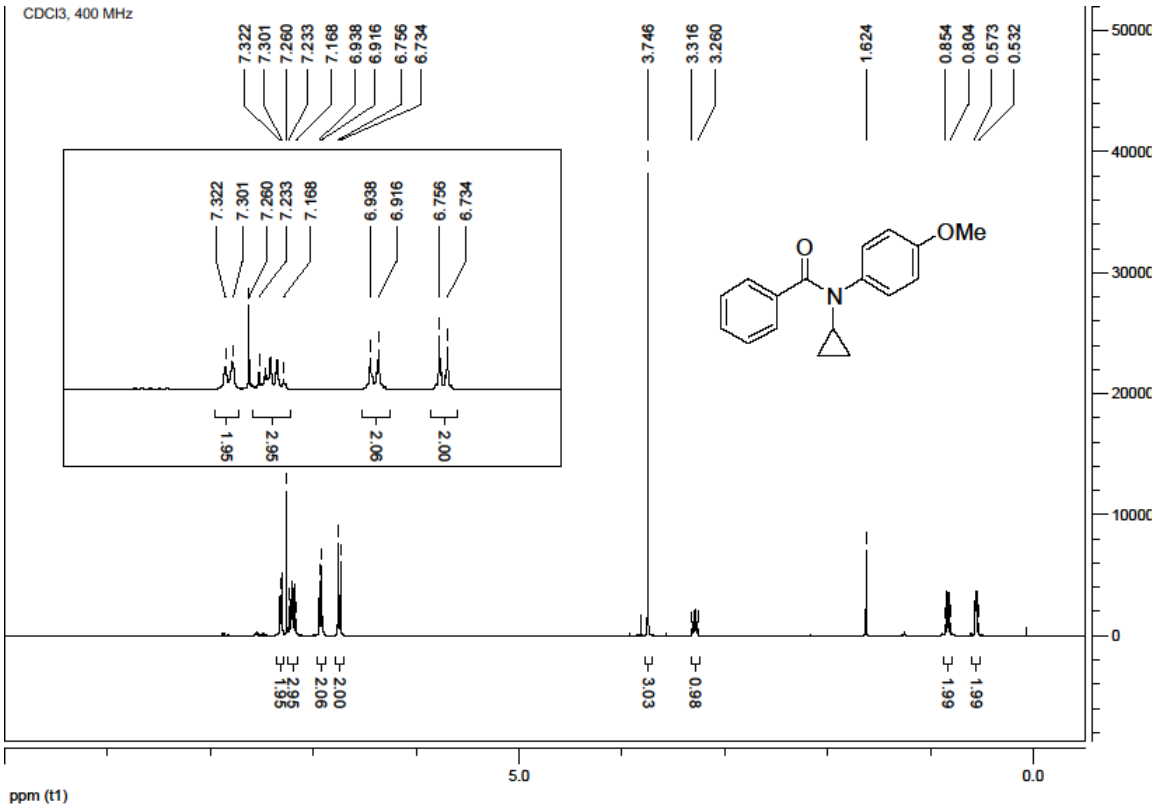

jmod, CDCl3, 100 MHz
172.11
163.92
161.46
144.20
144.11
136.78
129.98
129.89
129.86
128.14
128.05
123.82
123.79
115.32
115.10
113.85
113.64
77.16
32.66
8.87
O
N
F
150
100
50
ppm (t1)

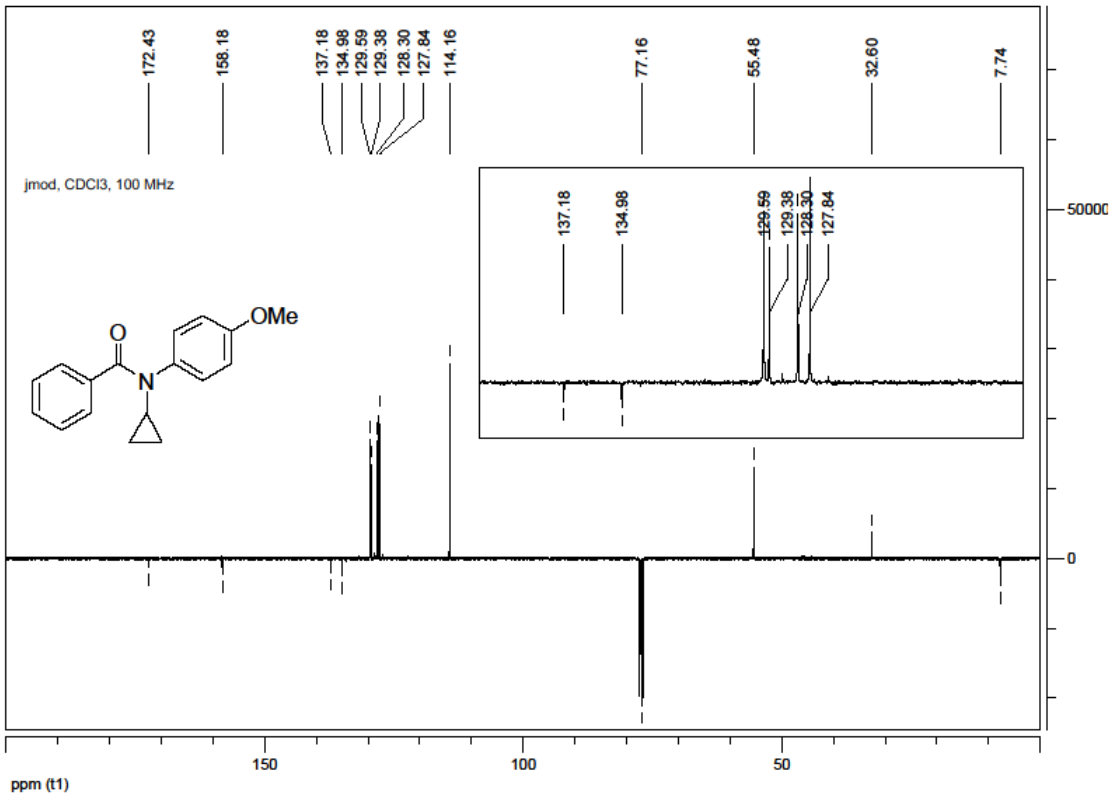

CDCl3, 400 MHz
7.375
7.357
7.277
7.154
7.066
6.986
3.306
3.251
1.687
0.836
0.786
0.603
0.562
1.94
4.23
3.00
1.94
4.30
3.02
1.00
1.92
2.00
ppm (t1)

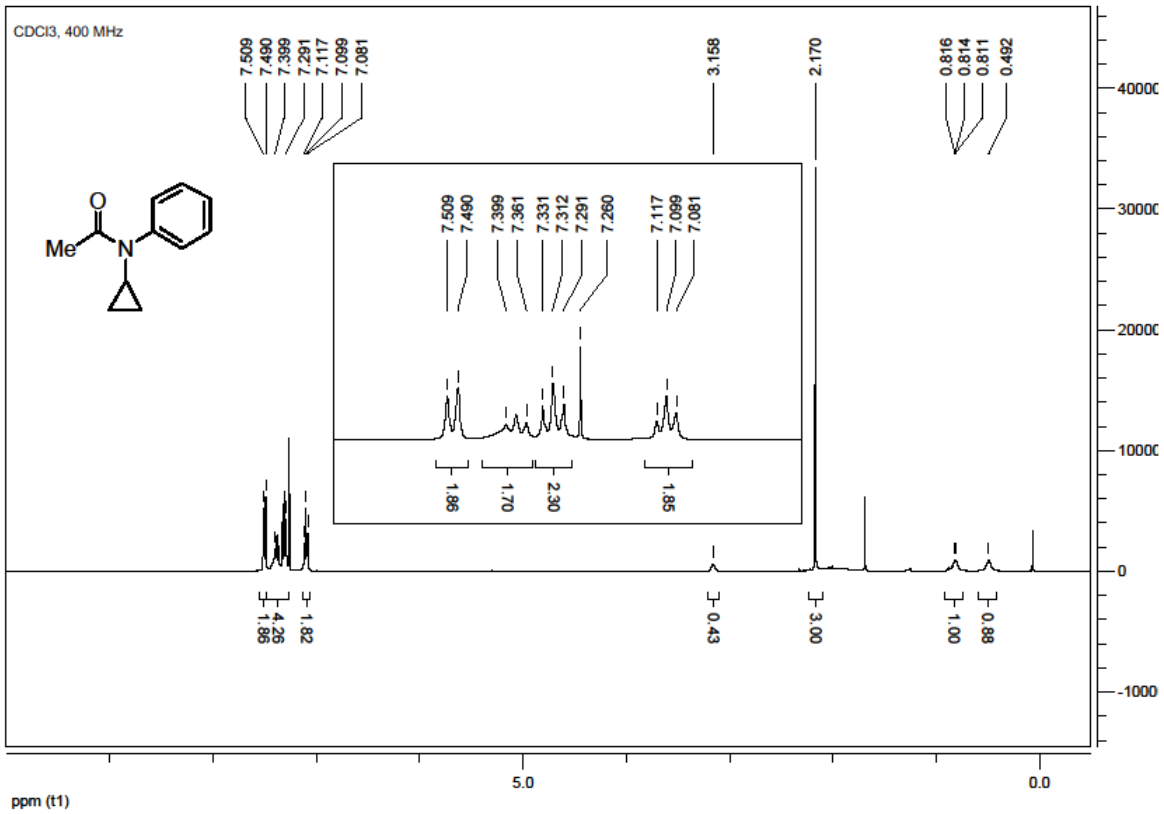

jmod, CDCl3, 100 MHz
172.71
159.39
156.91
136.64
130.76
130.30
130.17
129.77
129.31
129.23
127.89
127.67
124.50
124.46
116.54
116.34
77.16
32.33
7.71
ppm (t1)

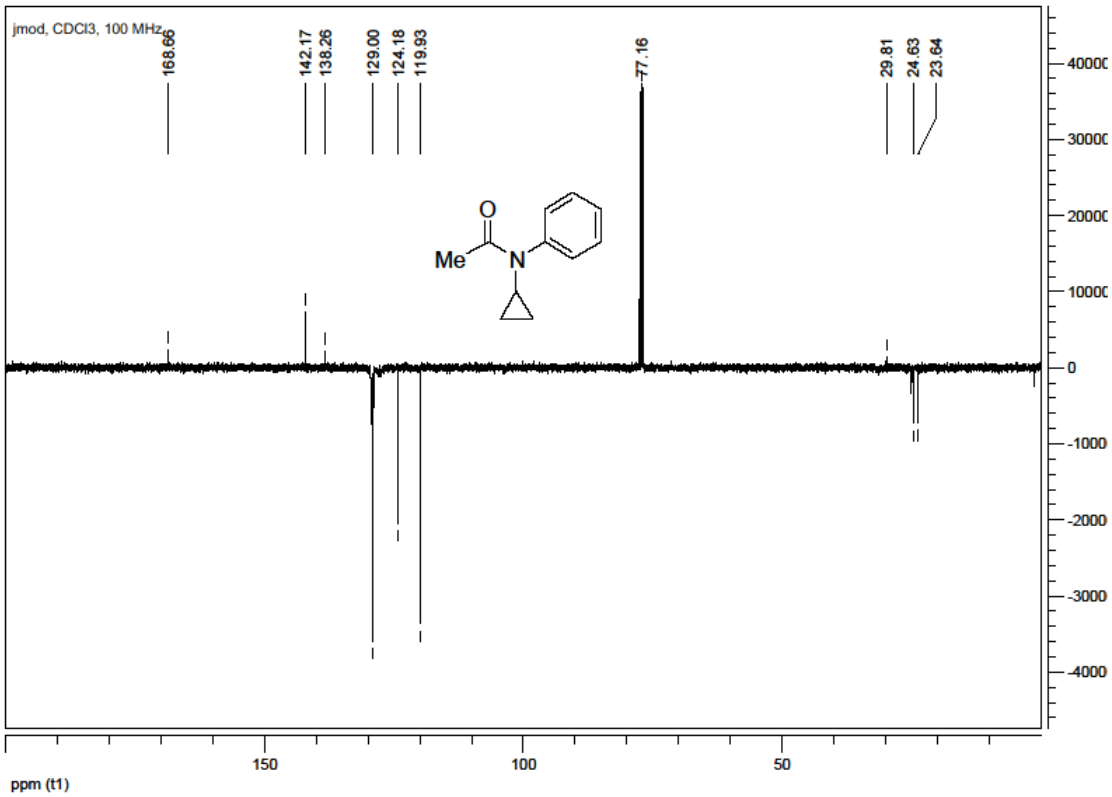

CDCl3, 400 MHz
8.109
6.665
3.360
3.353
1.131
0.332
O
N
Cl
50000
0
9.59
1.00
4.05
5.0
0.0
ppm (t1)

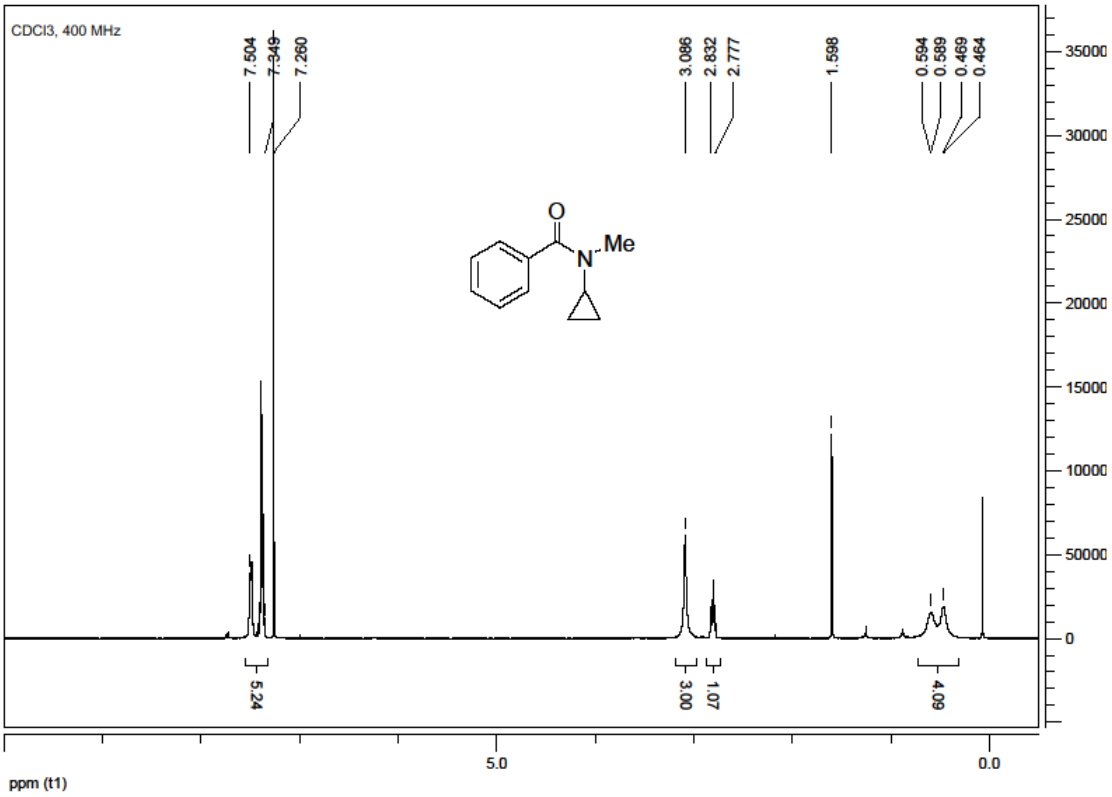

jmod, CDCl3, 100 MHz
172.43
139.68
130.36
129.76
128.95
127.77
127.45
77.16
O
N
Cl
50000
40000
30000
20000
10000
0
-10000
-20000
-30000
150
100
50
ppm (t1)

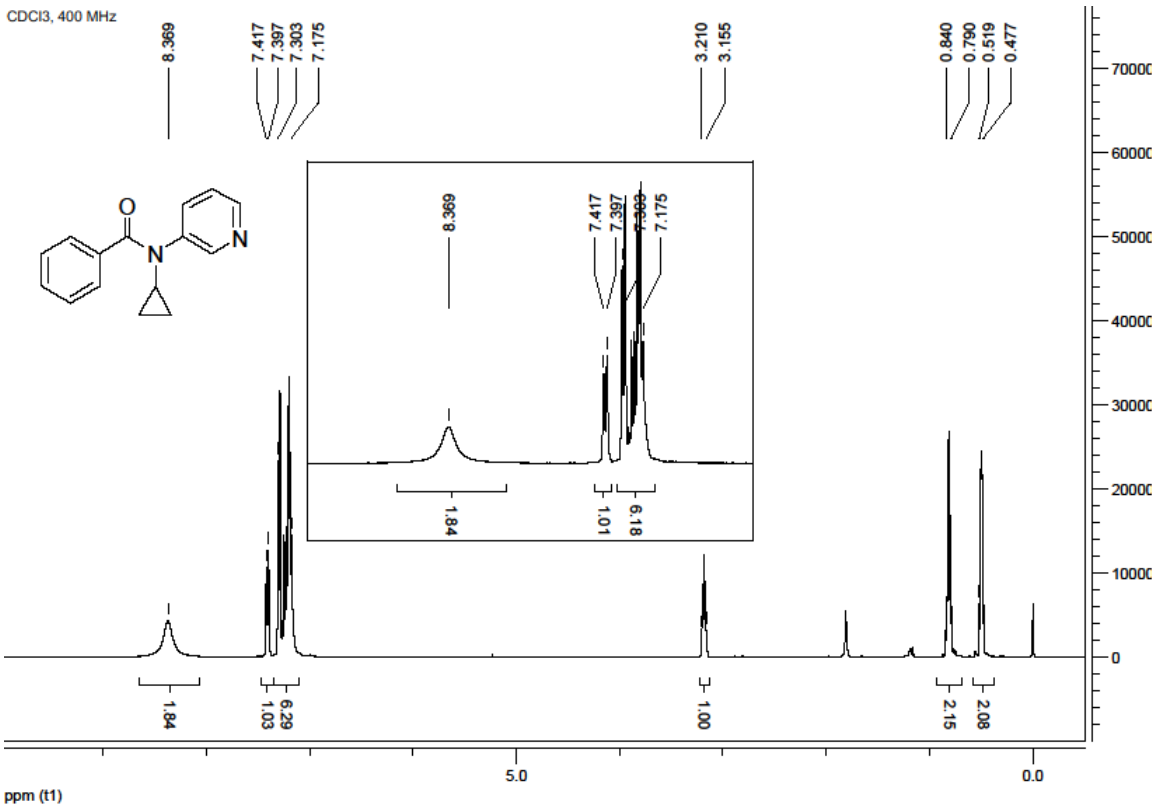

CDCl3, 400 MHz
7.322
7.301
7.260
7.233
7.168
6.938
6.916
6.756
6.734
3.746
3.316
3.260
1.624
0.854
0.804
0.573
0.532
OMe
O
N
1.95
2.95
2.06
2.00
3.03
0.98
1.99
1.99
5.0
0.0
ppm (t1)

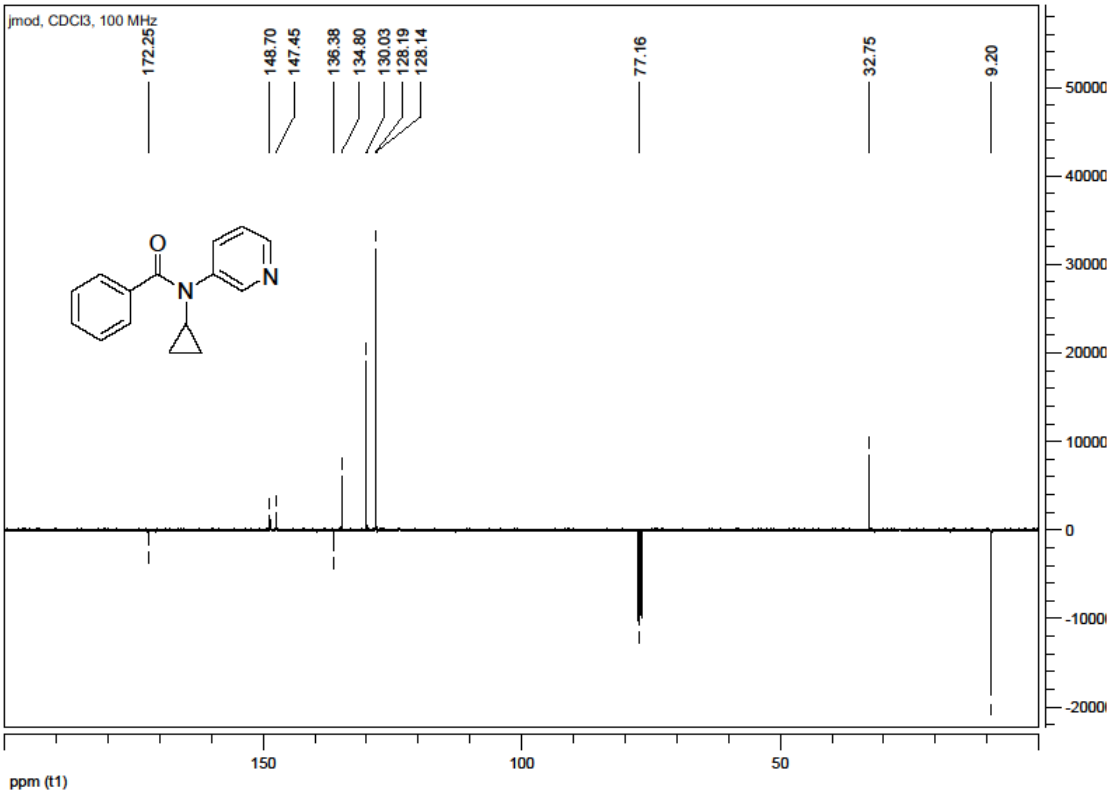

172.43
158.18
137.18
134.98
129.59
129.38
128.30
127.84
114.16
77.16
55.48
32.60
7.74
jmod, CDCl3, 100 MHz
OMe
O
N
150
100
50
ppm (t1)

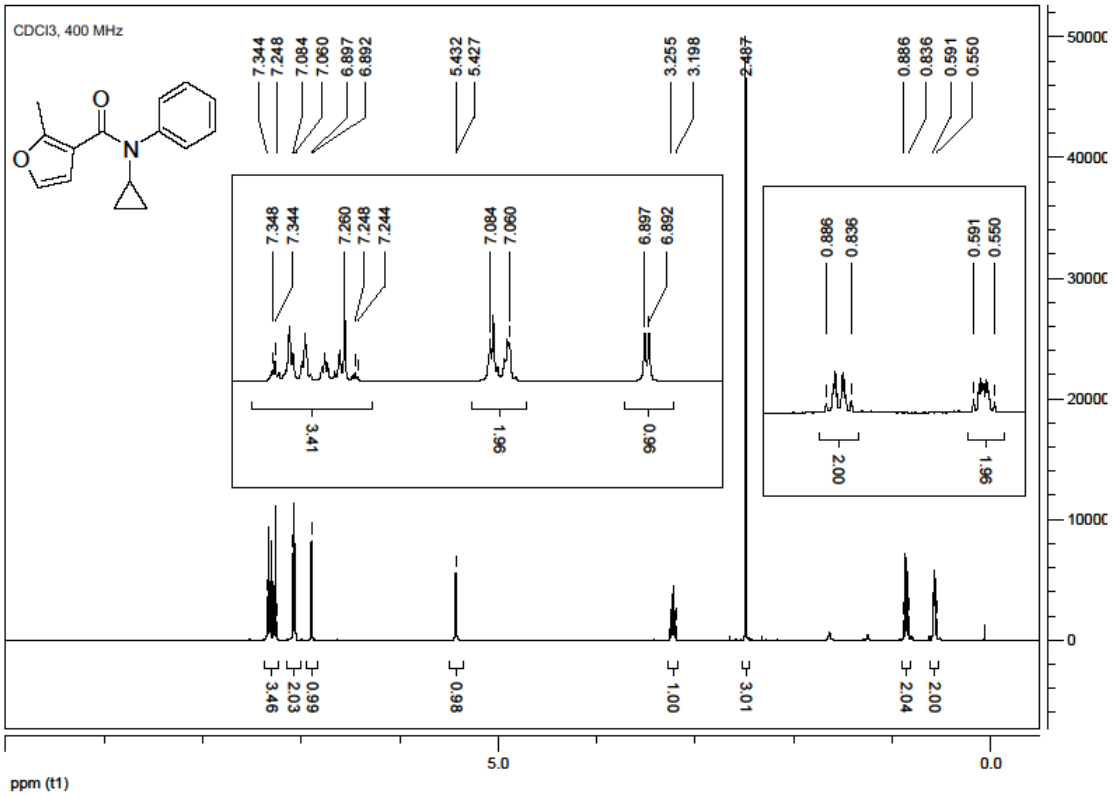

CDCl3, 400 MHz
7.509
7.490
7.399
7.291
7.117
7.099
7.081
3.158
2.170
0.816
0.814
0.811
0.492
Me
N
O
1.86
1.70
2.00
1.85
1.86
4.26
1.82
0.43
3.00
1.00
0.88
5.0
0.0
ppm (t1)

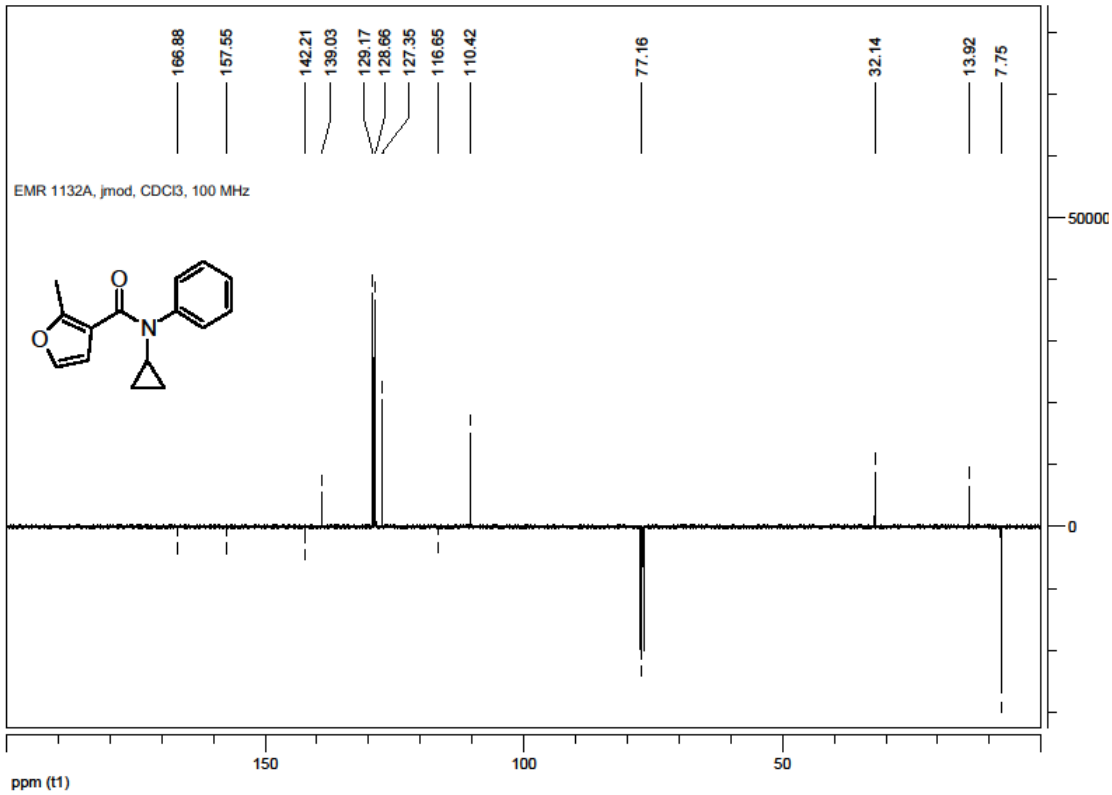

jmod, CDCl3, 100 MHz
168.66
142.17
138.26
129.00
124.18
119.93
77.16
29.81
24.63
23.64
Me
N
O
150
100
50
ppm (t1)

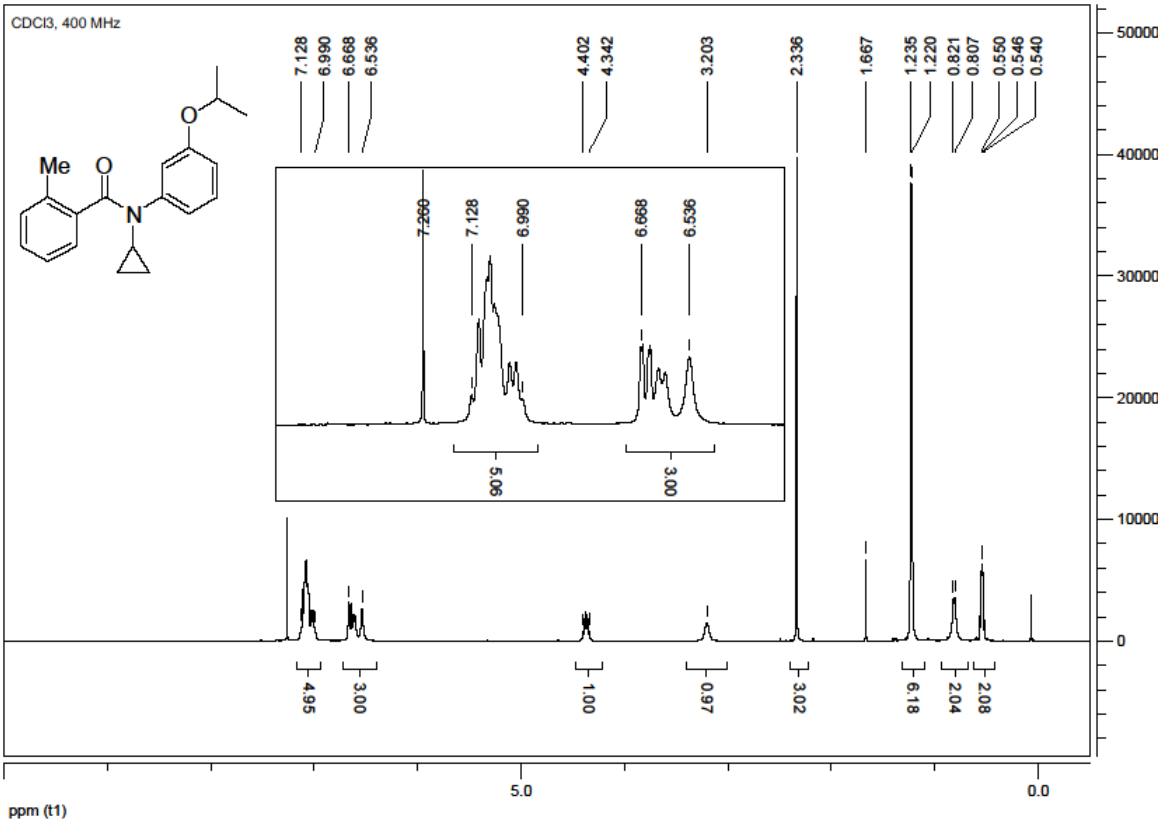

CDCl3, 400 MHz
7.504
7.349
7.260
3.086
2.832
2.777
1.598
0.594
0.589
0.469
0.464
O
N
Me
35000
30000
25000
20000
15000
10000
50000
0
5.24
3.00
1.07
4.09
5.0
0.0
ppm (t1)

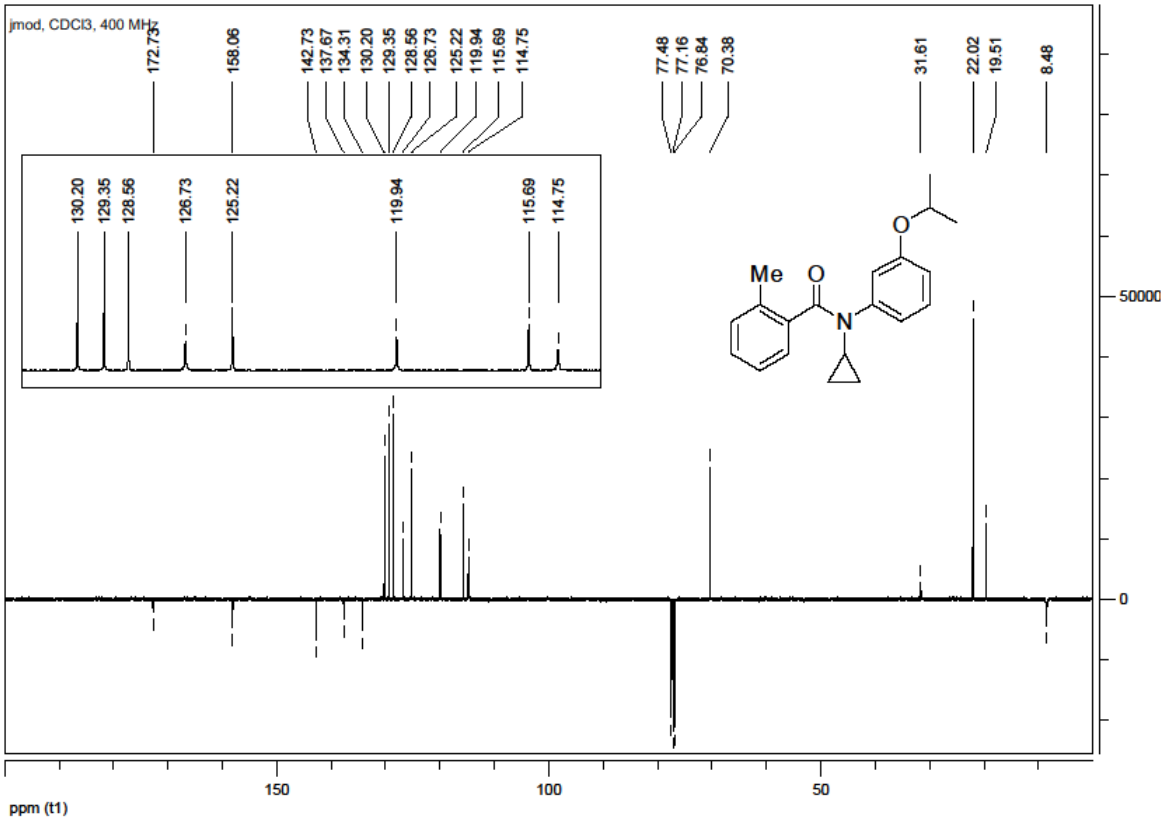

CDCl3, 400 MHz
8.369
7.417
7.397
7.303
7.175
3.210
3.155
0.840
0.790
0.519
0.477
1.84
1.03
6.29
1.00
2.15
2.08
5.0
0.0
ppm (t1)

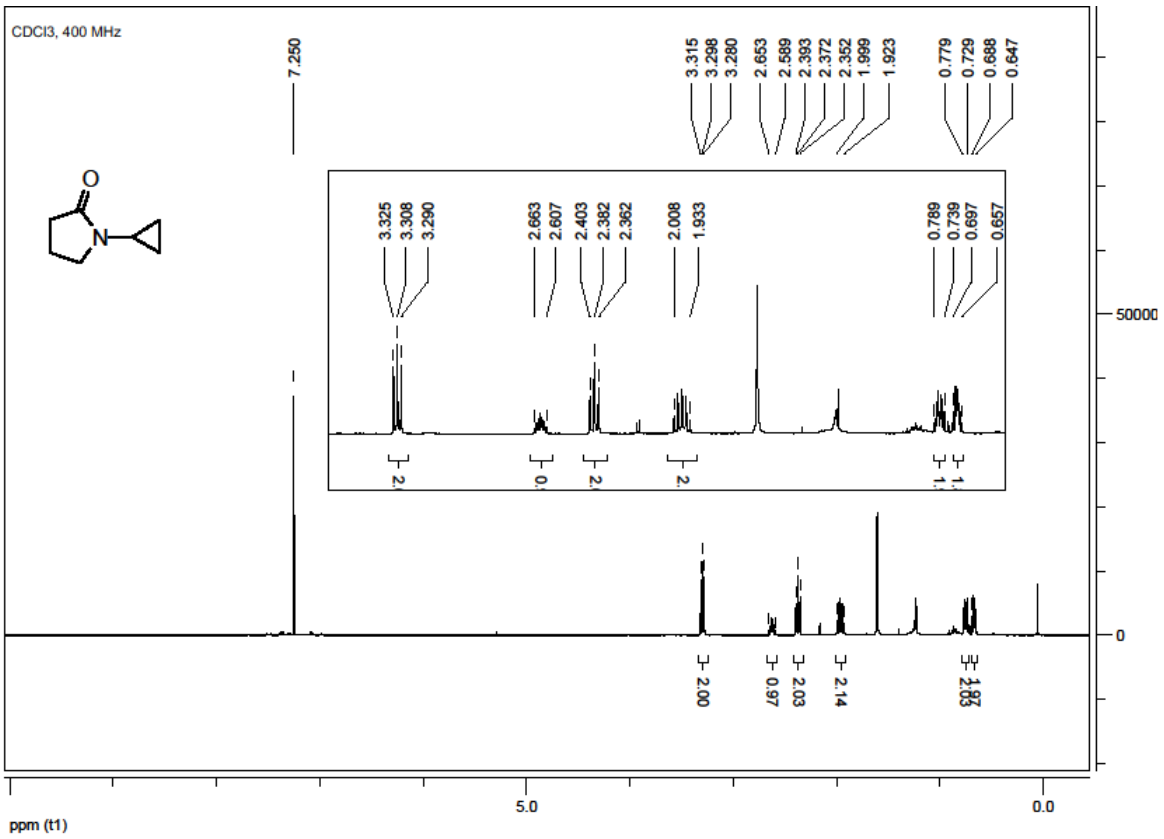

jmod, CDCl3, 100 MHz
172.25
148.70
147.45
136.38
134.80
130.03
128.19
128.14
77.16
32.75
9.20
150
100
50
ppm (t1)

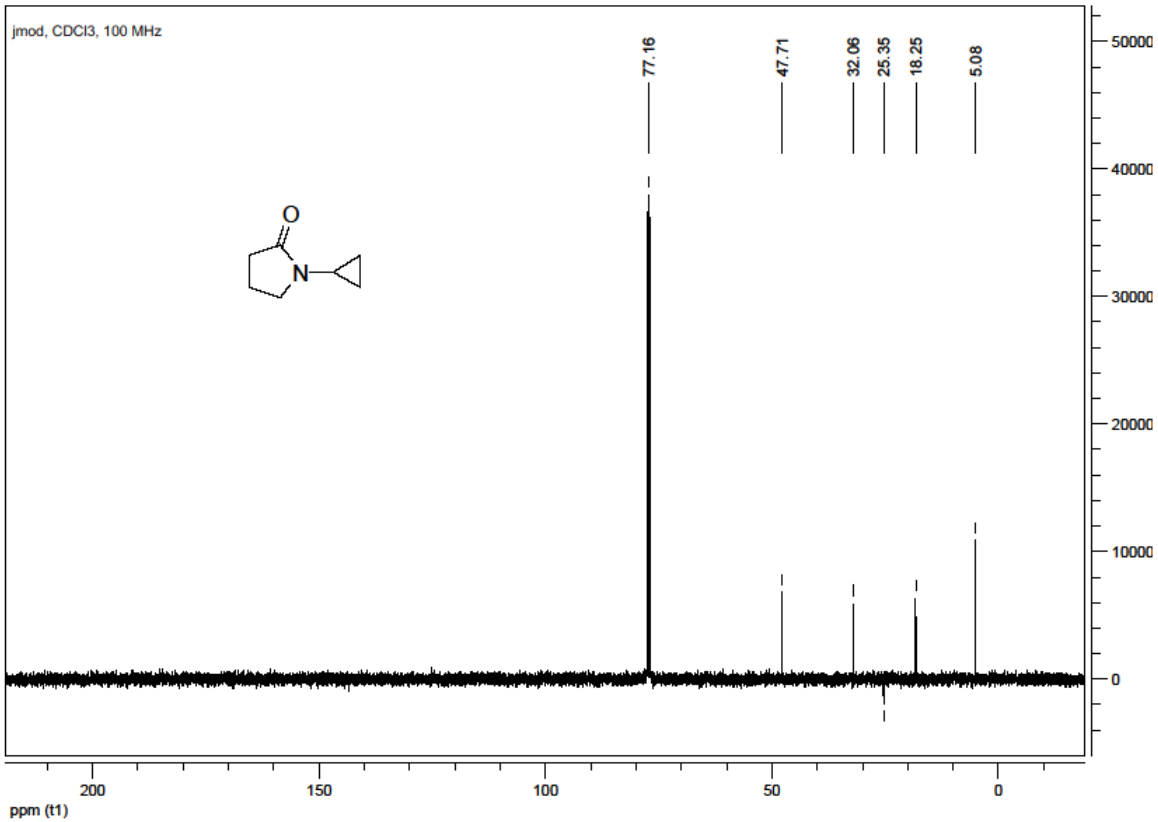

CDCl3, 400 MHz
7.344
7.248
7.084
7.060
6.897
6.892
5.432
5.427
3.255
3.198
2.487
0.886
0.836
0.591
0.550
3.46
2.03
0.99
0.98
1.00
3.01
2.04
2.00
ppm (t1)

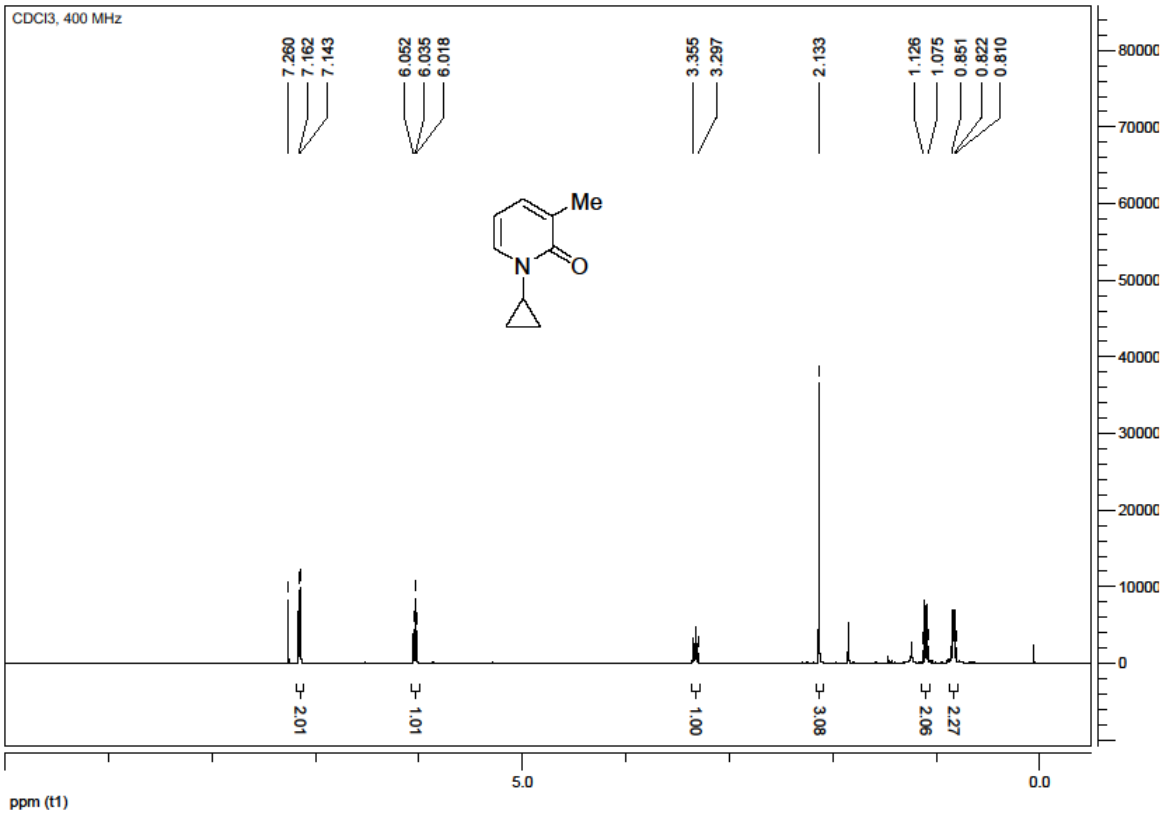

166.88
157.55
142.21
139.03
129.17
128.66
127.35
116.65
110.42
77.16
32.14
13.92
7.75
EMR 1132A, jmod, CDCl3, 100 MHz
ppm (t1)

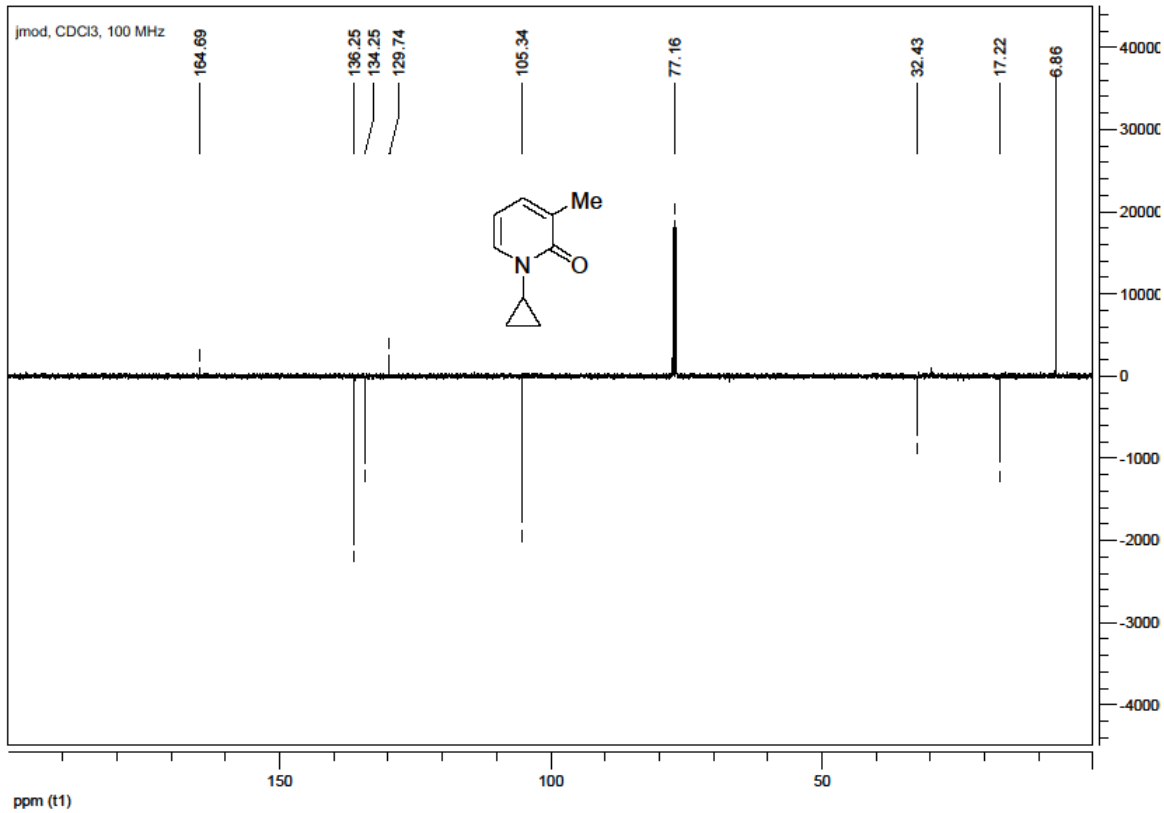

CDCl3, 400 MHz
7.128
6.990
6.668
6.536
4.402
4.342
3.203
2.336
1.667
1.235
1.220
0.821
0.807
0.550
0.546
0.540
Me
O
N
O
7.260
7.128
6.990
6.668
6.536
5.06
3.00
4.95
3.00
1.00
0.97
3.02
6.18
2.04
2.08
ppm (t1)

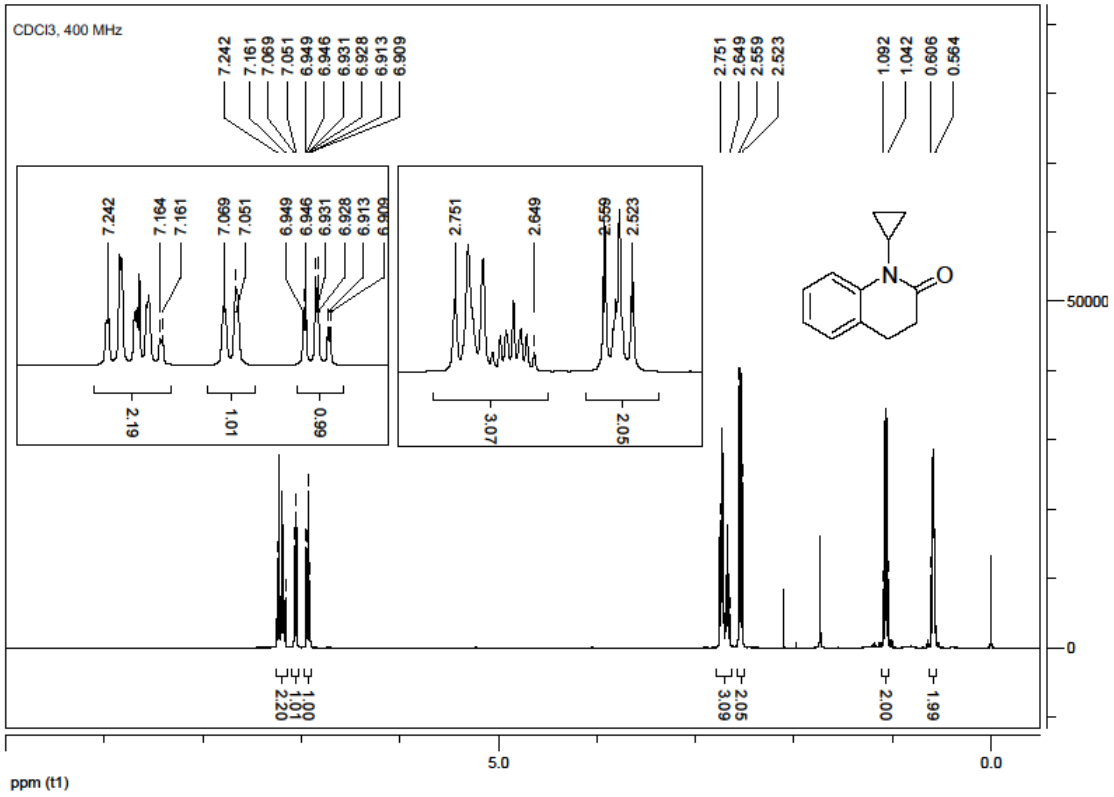

jmod, CDCl3, 400 MHz
172.75
158.06
142.73
137.67
134.31
130.20
129.35
128.56
126.73
125.22
119.94
115.69
114.75
77.48
77.16
76.84
70.38
31.61
22.02
19.51
8.48
Me
O
N
O
ppm (t1)

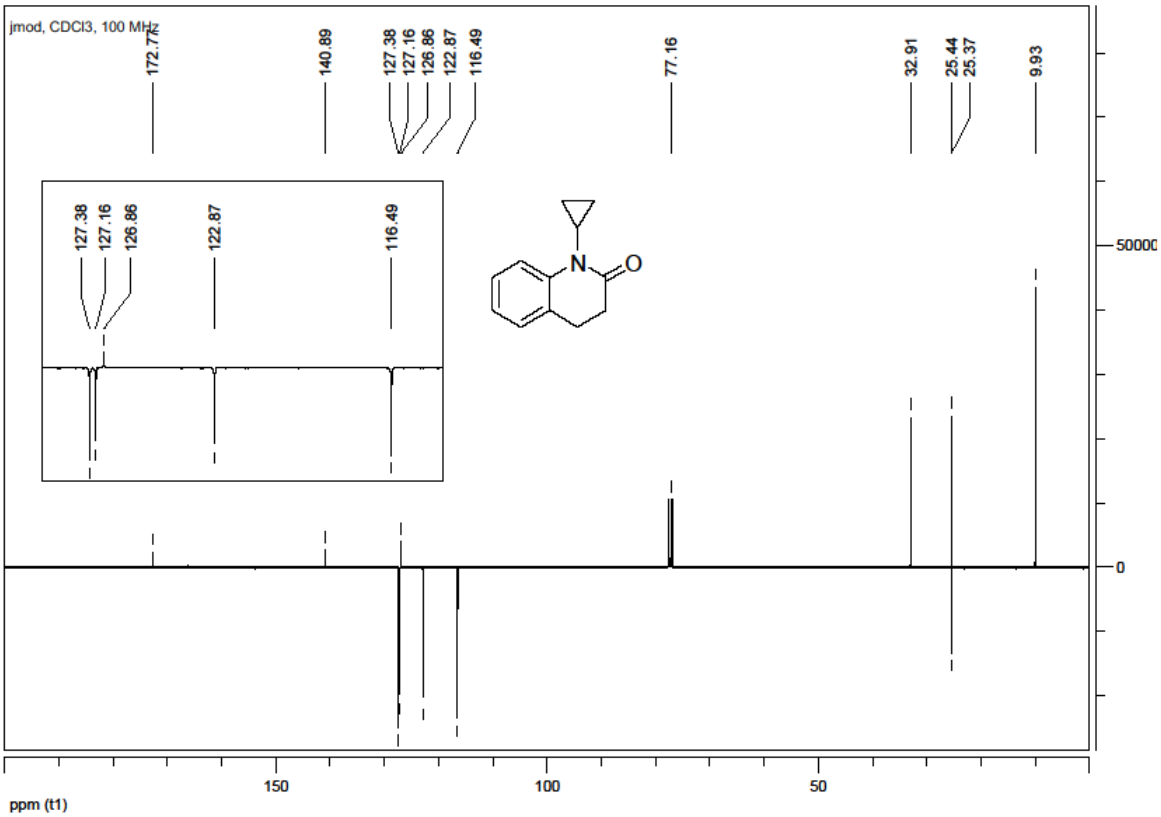

CDCl3, 400 MHz
7.250
3.315
3.298
3.280
2.653
2.589
2.393
2.372
2.352
1.999
1.923
0.779
0.729
0.688
0.647
3.325
3.308
3.290
2.663
2.607
2.403
2.382
2.362
2.008
1.933
0.789
0.739
0.697
0.657
2.00
0.97
2.03
2.14
3.28
50000
0
5.0
0.0
ppm (t1)


jmod, CDCl3, 100 MHz
77.16
47.71
32.06
25.35
18.25
5.08
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)


CDCl3, 400 MHz
7.260
7.162
7.143
6.052
6.035
6.018
3.355
3.297
2.133
1.126
1.075
0.851
0.822
0.810
Me
N
O
2.01
1.01
1.00
3.08
2.06
2.27
5.0
0.0
ppm (t1)


jmod, CDCl3, 100 MHz
164.69
136.25
134.25
129.74
105.34
77.16
32.43
17.22
6.86
Me
N
O
150
100
50
ppm (t1)


CDCl3, 400 MHz
7.242
7.161
7.069
7.051
6.949
6.946
6.931
6.928
6.913
6.909
2.751
2.649
2.559
2.523
1.092
1.042
0.606
0.564
7.242
7.164
7.161
7.069
7.051
6.949
6.946
6.931
6.928
6.913
6.909
2.751
2.649
2.559
2.523
2.19
1.01
0.99
3.07
2.05
N
O
50000
0
2.20
1.01
1.00
3.09
2.05
2.00
1.99
5.0
0.0
ppm (t1)


jmod, CDCl3, 100 MHz
172.77
140.89
127.38
127.16
126.86
122.87
116.49
77.16
32.91
25.44
25.37
9.93
127.38
127.16
126.86
122.87
116.49
N
O
50000
0
150
100
50
ppm (t1)