Electronic Supplementary Material (ESI) for ChemComm.
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Formation of amides, their intramolecular reactions to the synthesis of *N*-heterocycles, and preparation of a marketed drug, Sildenafil: A comprehensive coverage

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Supporting Information

I. General Considerations

Unless noted otherwise, all reagents and solvents were purchased from commercial sources and used as received. All reactions were performed in a screw-cap sealed tube. The ^{1}H and ^{13}C NMR spectra were obtained in $CDCl_3$, MeOD or DMSO-d_6 as solvent using a 400 MHz and 100 MHz spectrometer respectively with Me_4Si as an internal standard. Coupling constants (*J* values) are reported in Hz. Column chromatography was performed using silica gel (60-120 mesh, 100-200 mesh, and 230-400 mesh).

II. Experimental section

1. Preparation of substituted α-oxocarboxylic acids:

All substituted α-oxocarboxylic acids were prepared from oxidation of corresponding methyl ketones with SeO_2 according to the reported procedure.[1]

2. General procedure for the synthesis of potassium salts of the α-oxocarboxylic acids

Following a literature procedure, a solution of potassium *tert*-butoxide (1 mmol) in ethanol (1 mL) was added drop by drop to a solution of α-oxocarboxylic acids (1 mmol) in ethanol (1 mL) over 30 minutes. After complete addition, the reaction mixture was stirred for another 2 h at room temperature. A gradual formation of a precipitate was observed. The resulting solid was collected by filtration, washed with ethanol (2 x 10.0 mL), diethyl ether (10.0 mL) respectively, transferred to a round-bottomed flask and dried it under vacuum to give corresponding potassium salts of α-oxocarboxylic acids.[2]

3. Typical procedure for the synthesis of 2-aryl/heteroarylquinazolin-4(3H)-ones (15-25)

In an oven-dried screw cap vial equipped with a magnetic stir bar was charged with 2-amino benzamide derivative (0.3 mmol), α-oxocarboxylate (1.2 equiv), and $K_2S_2O_8$ (3 equiv). The tube was evacuated and backfilled with nitrogen. CH_3CN (2 mL) was added by syringe under flow of nitrogen. The tube was sealed and the mixture was allowed to stir at 80 ^{o}C for 8 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 3:7] to give the desired cyclized product.

4. Typical procedure for the synthesis of 2-arylbenzo[d]thiazoles (30-34)

In an oven-dried screw cap vial equipped with a magnetic stir bar was charged with α-oxocarboxylate (0.3 mmol), 2-amino thiophenol derivative (1.5 equiv), and $K_2S_2O_8$ (2 equiv). The tube was evacuated and backfilled with nitrogen. CH_3CN (2 mL) was added by syringe under flow of nitrogen. The tube was sealed and the mixture was allowed to stir at 80 ^oC for 8 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 0.5:9.5 ~ 1:9] to give the desired cyclized product.

5. General procedure for the synthesis of 2-arylbenzazoles (35-37)

In an oven-dried screw cap vial equipped with a magnetic stir bar was charged with substrates (0.3 mmol), α-oxocarboxylate (1.2 equiv), and $K_2S_2O_8$ (2 equiv). The tube was evacuated and backfilled with nitrogen. CH_3CN (2 mL) was added by syringe under flow of nitrogen. The tube was sealed and the mixture was allowed to stir at 80 ^oC for 8 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 0.5:9.5 ~ 1:9] to give the desired cyclized product.

6. General procedure for the synthesis of nitrogen heterocycles from benzaldehyde (3, 30, 35-37)

In an oven-dried screw cap vial equipped with a magnetic stir bar, substrate (0.3 mmol), benzaldehyde (1.2 equiv), $K_2S_2O_8$ (3 equiv), and CH_3CN (2 mL) as solvent was heated at 80 ^oC for 8 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 0.5:9.5 ~ 3:7] to give desired cyclized product.

7. Typical procedure for the synthesis of 5-(2-ethoxyphenyl)-1-methyl-3-propyl-1H pyrazolo[4,3-d]pyrimidin-7(6H)-one (51)

In an oven-dried screw cap vial equipped with a magnetic stir bar was charged with 4-amino-1-methyl-3-propyl-1H-pyrazole-5-carboxamide (0.5 mmol), 2-ethoxy phenylglyoxylate (1.2 equiv), and $K_2S_2O_8$ (3 equiv). The tube was evacuated and backfilled with nitrogen. CH_3CN (3.3 mL) was added by syringe under flow of nitrogen. The tube was sealed and the mixture was allowed to stir at 80 ^oC for 8 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 3:7] to give the desired cyclized product.

8. Typical procedure for the synthesis of 4-ethoxy-3-(1-methyl-7-oxo-3-propyl-6,7-dihydro 1H-pyrazolo[4,3-d]pyrimidin-5-yl)benzene-1-sulfonyl chloride (51a)

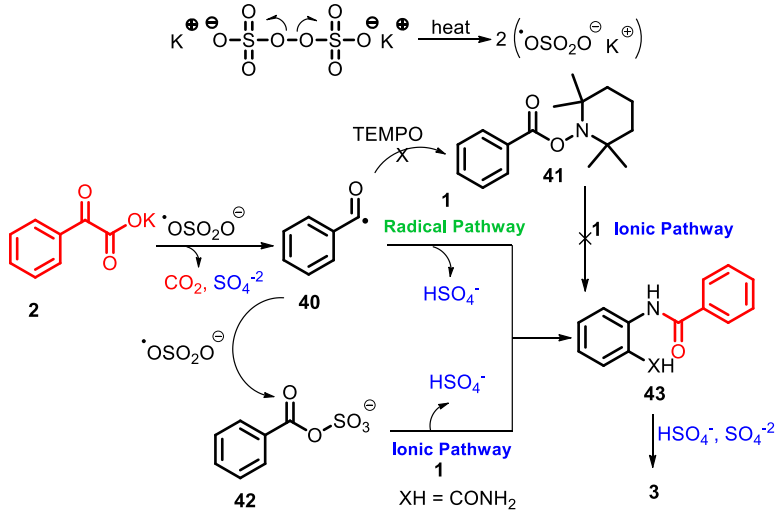
Following a literature procedure,[3] 5-(2-ethoxyphenyl)-1-methyl-3-propyl-1H pyrazolo[4,3-d]pyrimidin-7(6H)-one (0.3 mmol) was added portionwise to chlorosulphonic acid (0.25 mL) at 0 ^oC. The reaction mixture was allowed to stir at room temperature overnight. The reaction mixture was poured to crushed ice. A white precipitate formed which was filtered, washed with cold water and dried it under pressure to give a title compound as a white solid. (112 mg, 91%)

9. Typical procedure for the synthesis of 5-(2-ethoxy-5-((4-methylpiperazin-1 yl)sulfonyl)phenyl)-1-methyl-3-propyl-1H-pyrazolo[4,3-d]pyrimidin-7(6H)-one (Sildenafil) (52)

To the solution of sulfonyl chloride derivative (0.25 mmol) in THF (2 mL), excess of *N*-methyl piperazine was added. The resulting mixture was stirred at room temperature for 3 h. After the completion of reaction, THF was concentrated under reduced pressure and the residue was washed with cold water. The resulting precipitate was filtered, washed with cold water, dried under reduced pressure and recrystallized from ethanol to give white color solid. (123 mg, 89%).

III. Peripheral discussion on mechanism

The possible reaction pathways of the tandem reaction are shown in Scheme 1. Initially, homolytic cleavage of $K_2S_2O_8$ under thermal conditions could generate a sulfate radical anion ($SO_4^{\cdot-}$),[4] which upon reaction with phenylglyoxylate could produce acyl radical **36** via decarboxylation. The formation of amide **38** could occur via two pathways. The acyl radical **36** could react with 2-aminobenzamide **1** to form amide **38** via a radical pathway.[5a] Upon capture of a sulfate radical anion ($SO_4^{\cdot-}$), the acyl radical **36** could also form a benzoyl sulfate **37**, which could undergo nucleophilic substitution with **1** to give **38** via an ionic pathway. Furthermore, intramolecular nucleophilic addition of the primary amide group to the secondary amide in amide **38** in the presence of bases (HSO_4^-, SO_4^{-2}) generated *in situ* could give quinazolinone **3**.

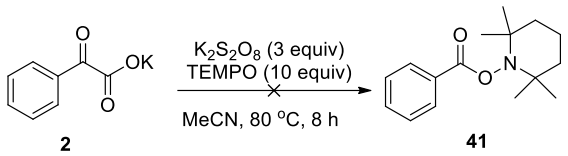


Scheme 1. Possible reaction pathways

Effect of TEMPO or BHT in the tandem reaction

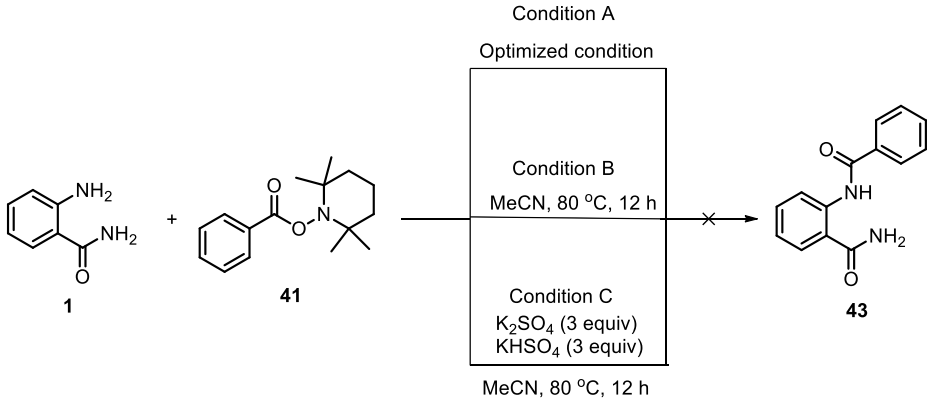
A free radical quencher, such as BHT or TEMPO did not affect the yield of **3** significantly. Apparently, this fact could be explained as follows. When the reaction of 2-aminobenzamide **1** and phenylglyoxylate **2** is carried out in the presence of TEMPO, acyl radical **40** could react with TEMPO to form TEMPO-acyl adduct **41**, which could further undergo nucleophilic substitution with **1** to give the intermediate **43** via an ionic mechanism (Scheme 1). To prove the hypothesis, we investigated the formation of TEMPO-acyl adduct **43** in this reaction and its reaction, prepared independently, with 2-aminobenzamide 1. Reaction of phenylglyoxylate **2** and TEMPO under the

standard conditions did not form the TEMPO-acyl adduct **41** (scheme 2). Thus, the formation of TEMPO-acyl adduct is not warranted in tandem reaction.



Scheme 2. Acyl radical to TEMPO-acyl adduct formation

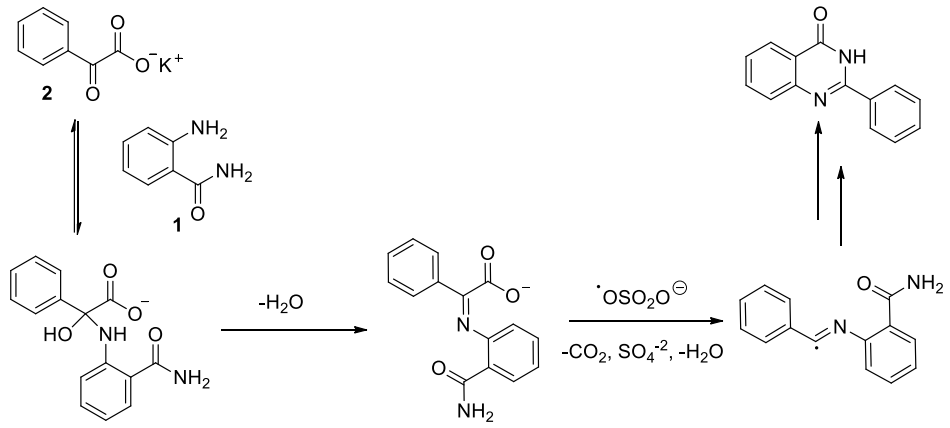
Next, we performed three different reactions of TEMPO-acyl adduct **41** and 2-aminobenzamide **1** under the conditions shown in Scheme 3. When the pure TEMPO-acyl adduct **41** was reacted with **1** under the optimized conditions, the reaction did not give amide **43**. Similarly, the reaction **1** and **41** also did not give the amide **43** in the presence or absence of any base. Therefore, our hypothesis is not supported by these experiments. Probably, the trapping of acyl radical **40** with a sulfate radical anion could be faster than trapping with TEMPO resulting in the formation of acyl sulfate **42**, which could undergo nucleophilic substitution with **1** to give **43** via ionic pathway. This could explain why TEMPO does not inhibit the tandem reaction.



Scheme 3. Reaction of TEMPO-acyl adduct **41** with **1**

The condensation of aliphatic α-oxocarboxylic acid and hydroxylamine,[5b] is reported to occur at temperature below 50 °C in the absence of any reagent. Based on the literature, another possible pathway could be the condensation of **2** and **1** followed by dehydration to generate imine, which could undergo oxidative decarboxylation to form imine radical. Intramolecular cyclization and subsequent oxidation in the presence of $K_2S_2O_8$ could give quinazolinone **3** (Scheme 4). To prove this pathway, we carried out a control experiment, which involved heating arylamine **1** and

phenylglyoxylate **2** in acetonitrile at 40 °C for 8 h. However, the reaction did not form any significant product. Thus, the reaction pathway involving condensation of **1** and **2**, and subsequent oxidative decarboxylation to give the quinazolinone **3** is unlikely. The other pathways (not shown) that reasonably explain the formation of quinazolinone **3** from *ortho*-substituted arylamine **1** and α- phenylglyoxylate **2** are not ruled out.



Scheme 4. Condensation pathway

Preparation of 2,2,6,6-tetramethylpiperidin-1-yl benzoate (41)[6]

According to literature procedure,[6] an oven-dried screw cap vial equipped with a magnetic stir bar, quinoline (0.5 mmol), benzaldehyde (4 equiv), NCS (0.3 equiv), TEMPO (1.1 equiv) and TBHP in decane (5.5M, 3 equiv), in DCE (2 mL) was heated at 105 °C for 16 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 0.2/0.8] to give 2,2,6,6-tetramethylpiperidin-1-yl benzoate (118 mg, 69%).

IV. Control experiments

1. Typical procedure for the synthesis of *N*-phenylbenzamide (39)

In an oven-dried screw cap vial equipped with a magnetic stir bar was charged with aniline (0.3 mmol), α-oxocarboxylate (1.5 equiv), and $K_2S_2O_8$ (2 equiv). The tube was evacuated and backfilled with nitrogen. CH_3CN (2 mL) was added by syringe under flow of nitrogen. The tube

was sealed and the mixture was allowed to stir at 80 ^{o}C for 24 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 1:9] to give the desired cyclized product.

2. Typical procedure for the preparation of 2-(benzoylamino)benzamide (43)

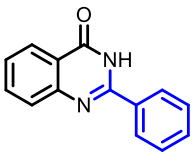
Following a literature procedure,[7] a dried round bottom flask equipped with magnetic stirrer bar charged with 2-amino benzamide (5 mmol), triethyl amine (5 mmol), and acetone (20 mL). The reaction mixture was cool down to 0 ^{o}C and solution of benzoyl chloride (5 mmol) in acetone (5 mL) was added dropwise over 5 minutes. The reaction mixture was stirred for 2 h at room temperature. The product together with a part of separated triethylammonium chloride was separated by filtration through a sintered glass filter, and salt was removed by washing with cold water to obtain the first portion of product. The mother liquor was concentrated under reduced pressure, and the residue was recrystallized from toluene to provide the second portion of product.

3. Typical procedure for the synthesis of 2-phenylquinazolin-4(3H)-one from 2-(benzoylamino)benzamide (3)

In an oven-dried screw cap vial equipped with a magnetic stir bar, 2-(benzoylamino)benzamide (0.3 mmol), $KHSO_4$ (3 equiv), K_2SO_4 (3 equiv), and CH_3CN (2 mL) as solvent was heated at 80 ^{o}C for 8 h. The reaction mixture was allowed to cool to room temperature and neutralized with saturated solution of Na_2CO_3 (10 mL) by constant stirring. It was then extracted with EtOAc (20 mL x 2). The organic layer was dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography [silica, EtOAc - hexanes = 3:7] to give 2-phenylquinazolin-4(3H)-one.

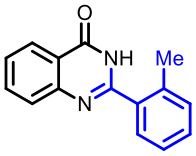
V. Characterization data

2-Phenylquinazolin-4(3H)-one (3)[8]



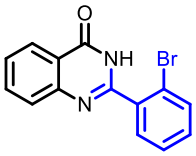
White solid (63 mg, 95%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.81 (s, 1H), 8.35 (d, *J* = 7.6 Hz, 1H), 8.31-8.28 (m, 2H), 7.87-7.80 (m, 2H), 7.62-7.60 (m, 3H), 7.53 (dt, *J* = 7.9, 1.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.4, 151.5, 149.4, 134.9, 132.8, 131.7, 129.1, 128.0, 127.2, 126.9, 126.4, 120.9.

2-(o-Tolyl)quinazolin-4(3H)-one (15)[10]



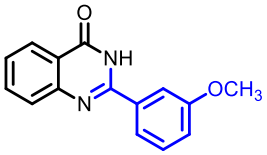
White solid (58 mg, 82%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.93 (s, 1H), 8.32 (d, *J* = 7.8 Hz, 1H), 7.83-7.82 (m, 2H), 7.59-7.52 (m, 2H), 7.47-7.42 (m, 1H), 7.38-7.35 (m, 2H), 2.55 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.8, 153.3, 149.0, 136.8, 134.8, 133.6, 131.4, 130.6, 128.6, 127.8, 127.0, 126.4, 126.3, 120.7, 20.1.

2-(2-Bromophenyl)quinazolin-4(3H)-one (16)[9]



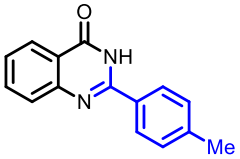
Light brown solid (76 mg, 85%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.82 (s, 1H), 8.31 (d, *J* = 7.9 Hz, 1H), 7.82-7.81 (m, 2H), 7.75 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.72 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.57-7.52 (m, 1H), 7.49 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.40 (dt, *J* = 7.8, 1.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.1, 151.9, 148.9, 136.4 134.9, 134.8, 133.8, 132.1, 131.3, 128.0, 127.4, 126.5, 121.1, 120.8.

2-(3-Methoxyphenyl)quinazolin-4(3H)-one (17)[9]



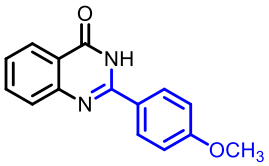
White solid (65 mg, 86%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.87 (s, 1H), 8.33 (d, *J* = 8.0 Hz, 1H), 7.88-7.81 (m, 2H), 7.77-7.73 (m, 2H), 7.55-7.48 (m, 2H), 7.15 (dd, *J* = 8.2, 2.5 Hz, 1H), 3.98 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.9, 160.1, 151.6, 149.4, 134.9, 134.1, 130.1, 128.0, 126.8, 126.3, 120.9, 119.7, 118.2, 112.1, 55.6.

2-(p-Tolyl)quinazolin-4(3H)-one (18)[9]



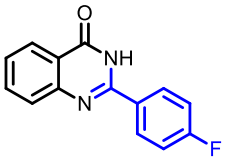
White solid (59 mg, 84%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.47 (s, 1H), 8.34 (d, *J* = 8.1 Hz, 1H), 8.04 (d, *J* = 8.2 Hz, 2H), 7.83-7.81 (m, 2H), 7.52 (dt, *J* = 8.0, 1.9 Hz, 1H), 7.40 (d, *J* = 7.9 Hz, 2H) 2.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.6, 151.6, 149.5, 142.2, 134.8, 129.9, 129.8, 127.9, 127.2, 126.6, 126.3, 120.8, 21.5.

2-(4-Methoxyphenyl)quinazolin-4(3H)-one (19)[8]



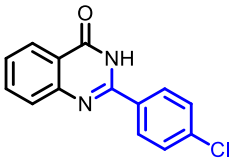
White solid (67 mg, 89%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.15 (s, 1H), 8.33 (d, *J* = 7.6 Hz, 1H), 8.21 (d, *J* = 8.8 Hz, 2H), 7.82-7.80 (m, 2H), 7.52-7.48 (m, 1H), 7.11 (d, *J* = 8.8 Hz, 1H), 3.93(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.5, 151.2, 149.7, 134.9, 132.2, 129.0, 127.7, 126.4, 126.3, 125.0, 120.4, 114.4, 113.6, 55.5.

2-(4-Fluorophenyl)quinazolin-4(3H)-one (20)[11]



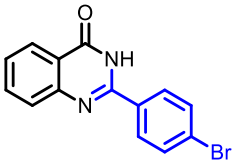
White solid (62 mg, 87%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.5 (s, 1H), 8.24-8.21 (m, 2H), 8.15 (d, J = 7.9 Hz, 1H), 7.84 (dt, J = 8.2, 1.4 Hz, 1H), 7.73 (d, J = 7.9 Hz, 1H), 7.53 (t, J = 7.8 Hz, 1H), 7.41-7.36 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.4 (d, J = 250 Hz), 162.8, 151.9, 149.0, 135.1, 130.8 (d, J = 9 Hz), 129.6 (d, J = 12 Hz), 127.7, 127.1, 126.3, 121.2, 116.2 (d, J = 22 Hz).

2-(4-Chlorophenyl)quinazolin-4(3H)-one (21)[9]



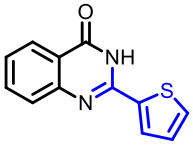
White solid (60 mg, 78%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.60 (s, 1H), 8.19-8.14 (m, 3H), 7.85 (dt, J = 8.3, 1.4 Hz, 1H), 7.75 (d, J = 7.8 Hz, 1H), 7.63 (d, J = 8.6 Hz, 2H), 7.54 (dt, J = 8.0, 1.0 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.6, 151.8, 149.0, 136.7, 135.2, 131.9, 130.0, 129.1, 127.9, 127.3, 126.3, 121.3.

2-(4-Bromophenyl)quinazolin-4(3H)-one (22)[9]



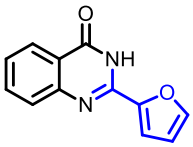
White solid (73 mg, 81%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.15 (dd, J = 7.9, 1.2 Hz, 1H), 8.0 (d, J = 8.6, 2H), 7.85 (dt, J = 8.3, 1.5 Hz, 1H), 7.7-7.3 (m, 3H), 7.54 (dt, J = 8.0, 1.0 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.6, 151.9, 149.0, 135.1, 132.3, 132.1, 130.2, 127.9, 127.3, 126.3, 125.7, 121.4.

2-(Thiophen-2-yl)quinazolin-4(3H)-one (23)[8]



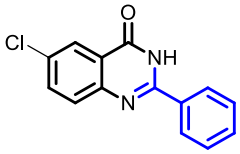
White solid (53 mg, 78%); ^{1}H NMR (400 MHz, DMSO-d_6); δ 12.64 (s, 1H), 8.24 (dd, J = 3.7, 0.9 Hz, 1H), 8.12 (dd, J = 7.8, 1.2 Hz, 1H), 7.87 (dd, J = 5.4, 0.8 Hz, 1H), 7.80 (dt, J = 8.4, 3.9 Hz, 1H), 7.65 (d, J = 7.9 Hz, 1H), 7.49 (t, J = 7.5 Hz, 1H), 7.24 (t, J = 4.4 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.3, 148.3, 137.8, 135.1, 132.6, 129.8, 128.9, 127.3, 126.7, 126.4, 121.3.

2-(furan-2-yl)quinazolin-4(3H)-one (24)[11]



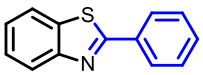
White solid (53 mg, 82%); ^{1}H NMR (400 MHz, $CDCl_3$); δ 11.16 (s, 1H), 8.32 (d, J = 7.8, Hz, 1H), 7.80-7.78 (m, 2H), 7.68 (d, J = 1.1 Hz, 1H), 7.68 (d, J = 3.4 Hz, 1H), 7.51-7.47 (m, 1H), 6.67 (dd, J = 3.5, 1.7 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.7, 149.3, 146.2, 145.6, 143.5, 135.0, 127.7, 126.7, 126.5, 121.0, 114.0. 112.9.

6-Chloro-2-phenylquinazolin-4(3H)-one (25)[8]



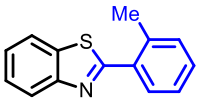
White solid (58 mg, 76%); ^{1}H NMR (400 MHz, DMSO-d_6); δ 8.16 (dd, J = 7.9, 1.0 Hz, 2H), 8.08 (d, J = 2.4 Hz, 1H), 7.87 (dd, J = 8.7, 2.5 Hz, 1H), 7.77 (d, J = 8.7 Hz, 1H), 7.62-7.53 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 161.8, 153.4, 147.8, 135.1, 134.9, 132.9, 131.2, 130.1, 129.1, 128.3, 125.3, 122.6.

2-Phenylbenzo[d]thiazole (30)[13]



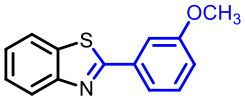
White solid (47 mg, 75%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14-8.09 (m, 3H), 7.93 (d, J = 8.0 Hz, 1H), 7.54-7.50 (m, 4H), 7.41 (dt, J = 8.2, 1.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 154.1, 135.0, 133.6, 131.0, 129.0, 127.5, 126.3, 125.2, 123.2, 121.6.

2-(o-Tolyl)benzo[d]thiazole (31)[14]



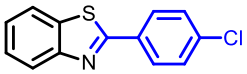
Colorless solid (38 mg, 56%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, J = 8.2 Hz, 1H), 7.96 (d, J = 7.7 Hz, 1H), 7.78 (d, J = 7.2 Hz, 1H), 7.54 (dt, J = 7.7, 1.2 Hz, 1H), 7.46-7.32 (m, 4H), 2.69 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.0, 153.8, 137.2, 135.6, 131.5, 130.5, 130.0, 126.1, 126.1, 125.1, 123.4, 121.3, 21.3.

2-(3-Methoxyphenyl)benzo[d]thiazole (32)[13]



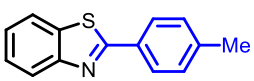
Colorless solid (49 mg, 68%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, J = 8.0 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.71-7.65 (m, 2H), 7.52 (dt, J = 7.3, 1.2 Hz, 1H), 7.44-7.28 (m, 2H), 7.06 (dd, J = 8.2, 2.5 Hz, 1H) 3.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.9, 160.0, 154.0, 135.1, 134.9, 130.0, 126.3, 125.2, 123.2, 121.6, 120.2, 117.3, 112.0, 55.5.

2-(4-Chlorophenyl)benzo[d]thiazole (33)[13]



White solid (53 mg, 72%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (d, J = 8.0 Hz, 1H), 8.04-8.01 (m, 2H), 7.90 (d, J = 7.8 Hz, 1H), 7.52-7.45 (m, 3H), 7.40 (dt, J = 7.3, 1.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6, 154.0, 137.0, 135.0, 132.1, 129.2, 128.7, 126.5, 125.4, 123.3, 121.6.

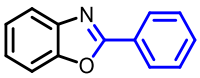
2-(p-Tolyl)benzo[d]thiazole (34)[14]



Colorless solid (50 mg, 74%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (d, J = 8.0 Hz, 1H), 7.98 (d, J = 8.2 Hz, 2H), 7.88 (d, J = 7.6 Hz, 1H), 7.47 (dt, J = 7.2, 1.2 Hz, 1H), 7.36 (dt, J = 8.0, 1.1 Hz, 1H), 7.29 (d, J = 7.9 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR

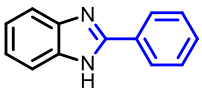
(100 MHz, $CDCl_3$): δ 168.2, 154.1, 141.4, 134.9, 130.9, 129.7, 127.4, 126.2, 125.0, 123.0, 121.5, 21.5.

2-Phenylbenzo[d]oxazole (35)[9]



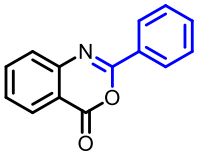
Colorless solid (29 mg, 50%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30-8.28 (m, 2H), 7.82-7.79 (m, 1H), 7.63-7.60 (m, 1H), 7.57-7.55 (m, 3H), 7.39-7.37 (m, 2H).

2-Phenyl-1H-benzo[d]imidazole (36)[12]



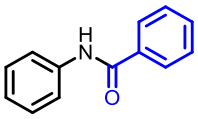
White solid (23 mg, 40%); ^{1}H NMR (400 MHz, MeOD): δ 7.98 (dd, *J* = 8.3, 1.7 Hz, 2H), 7.51-7.49 (m, 2H), 7.45-7.39 (m, 3H), 7.16-7.14 (m, 2H); ^{13}C NMR (100 MHz, MeOD): δ 151.9, 129.9, 129.6, 128.7, 126.4, 122.5.

2-Phenyl-4H-benzo[d][1,3]oxazin-4-one (37)[9]

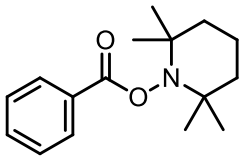


White solid (20 mg, 30%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.34 (dd *J* = 8.0, 0.9 Hz, 2H), 8.27 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.86 (dt, *J* = 8.2, 1.5 Hz, 1H), 7.72 (d, *J* = 7.7 Hz, 1H), 7.60 (tt, *J* = 7.2, 1.3 Hz, 1H), 7.57-7.53 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.6, 157.1, 147.0, 136.6, 132.6, 130.2, 128.7, 128.6, 128.3, 128.2, 127.2, 117.0.

***N*-phenylbenzamide (39)[15]**

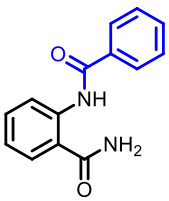


White solid (28 mg, 48%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.8 (dd *J* = 7.9, 0.9 Hz, 2H), 7.83 (brs, 1H), 7.64 (dd, *J* = 8.6, 1.0 Hz, 2H), 7.56 (tt, *J* = 7.3, 1.3 Hz, 1H), 7.50-7.46 (m, 2H), 7.40-7.35 (m, 2H), 7.15 (tt, *J* = 7.4, 1.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.7, 137.9, 135.0, 131.8, 129.1, 128.8, 127.0, 124.6, 120.1. **2,2,6,6-tetramethylpiperidin-1-yl benzoate (41)[6]**



White solid (118 mg, 69%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10-8.08 (m, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 1.84-1.76 (m, 2H), 1.72-1.69 (m, 1H), 1.63-1.59 (m, 2H), 1.50-1.46 (m, 1H), 1.29 (s, 6H), 1.14 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.4, 132.8, 129.7, 129.5, 128.4, 60.4, 39.0, 31.9, 20.8, 17.0.

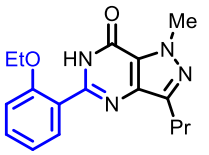
N-(2-carbamoylphenyl)benzamide (43)[3]



Colorless solid (1g, 86%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.9 (s, 1H), 8.68 (d, *J* = 8.2 Hz, 1H), 8.44 (s, 1H), 7.94 (d, *J* = 7.1 Hz, 2H), 7.89 (d, *J* = 7.8 Hz, 1H), 7.85 (s, 1H), 7.63-7.56 (m, 4H), 7.18 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz,

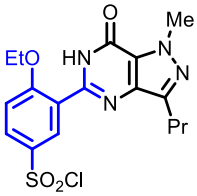
DMSO-d_6): δ 171.6, 164.9, 140.4, 135.0, 133.1, 132.5, 129.4, 129.2, 127.3, 123.2, 120.5, 119.6.

5-(2-ethoxyphenyl)-1-methyl-3-propyl-1H-pyrazolo[4,3-d]pyrimidin-7(6H)-one (51)[17]



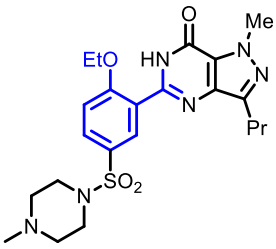
White solid (126 mg, 81%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.13 (s, 1H), 8.48 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.47 (dt, *J* = 8.7, 1.7 Hz, 1H), 7.16 (t, *J* = 7.2 Hz, 1H), 7.05 (d, *J* = 8.3 Hz, 1H). 4.33-4.29 (m, 5H), 2.9 (t, *J* = 7.4 Hz, 2H), 1.8 (sx, *J* = 7.6 Hz, 2H), 1.62 (t, *J* = 7.0 Hz, 3H), 1.05 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 153.9, 148.3, 146.6, 138.7, 132.3, 131.0, 124.4, 121.8, 120.2, 112.9, 65.2, 38.1, 27.8, 22.3, 14.7, 14.0.

4-ethoxy-3-(1-methyl-7-oxo-3-propyl-6,7-dihydro-1H pyrazolo[4,3-d]pyrimidin-5yl)benzene -1-sulfonyl chloride (51a)[16]



White solid (112 mg, 91%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.80 (s, 1H), 9.09 (d, *J* = 2.6 Hz, 1H), 8.10 (dd, *J* = 8.9, 2.6 Hz, 1H), 7.23 (d, *J* = 9 Hz, 1H), 4.42 (q, *J* = 8.5 Hz), 4.27 (s, 3H), 2.95 (t, *J* = 7.4 Hz, 2H), 1.86 (sx, *J* = 7.4 Hz, 2H), 1.66 (t, *J* = 7.0 Hz, 3H), 1.04 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.8, 153.5, 147.2, 145.6, 138.2, 137.5, 131.0, 130.9, 124.4, 121.8, 113.5, 66.6, 38.2, 27.6, 22.3, 14.4, 14.0.

5-(2-ethoxy-5-((4-methylpiperazin-1-yl)sulfonyl)phenyl)-1-methyl-3-propyl-1H- pyrazolo[4, 3-d]pyrimidin-7(6H)-one [Sildenafil] (52)[17]



White solid (123 mg, 89%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.81 (s, 1H), 8.82 (d, *J* = 2.2 Hz, 1H), 7.83 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.14 (d, *J* = 8.8 Hz, 1H), 4.36 (q, *J* = 6.9 Hz, 2H), 4.27 (s, 3H), 3.10 (brs, 4H), 2.92 (t, *J* = 7.4 Hz, 2H), 2.50 (brs, 4H), 2.27 (s, 3H), 1.86 (sx, *J* = 7.4 Hz, 2H), 1.64 (t, *J* = 6.9 Hz, 3H), 1.02 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.2, 153.6, 147.0, 146.4, 138.3, 131.6, 131.2, 128.9, 124.5, 121.0, 113.0, 66.1, 54.0, 45.9, 45.7, 38.2, 27.7, 22.2, 14.5, 14.0.

VI. References

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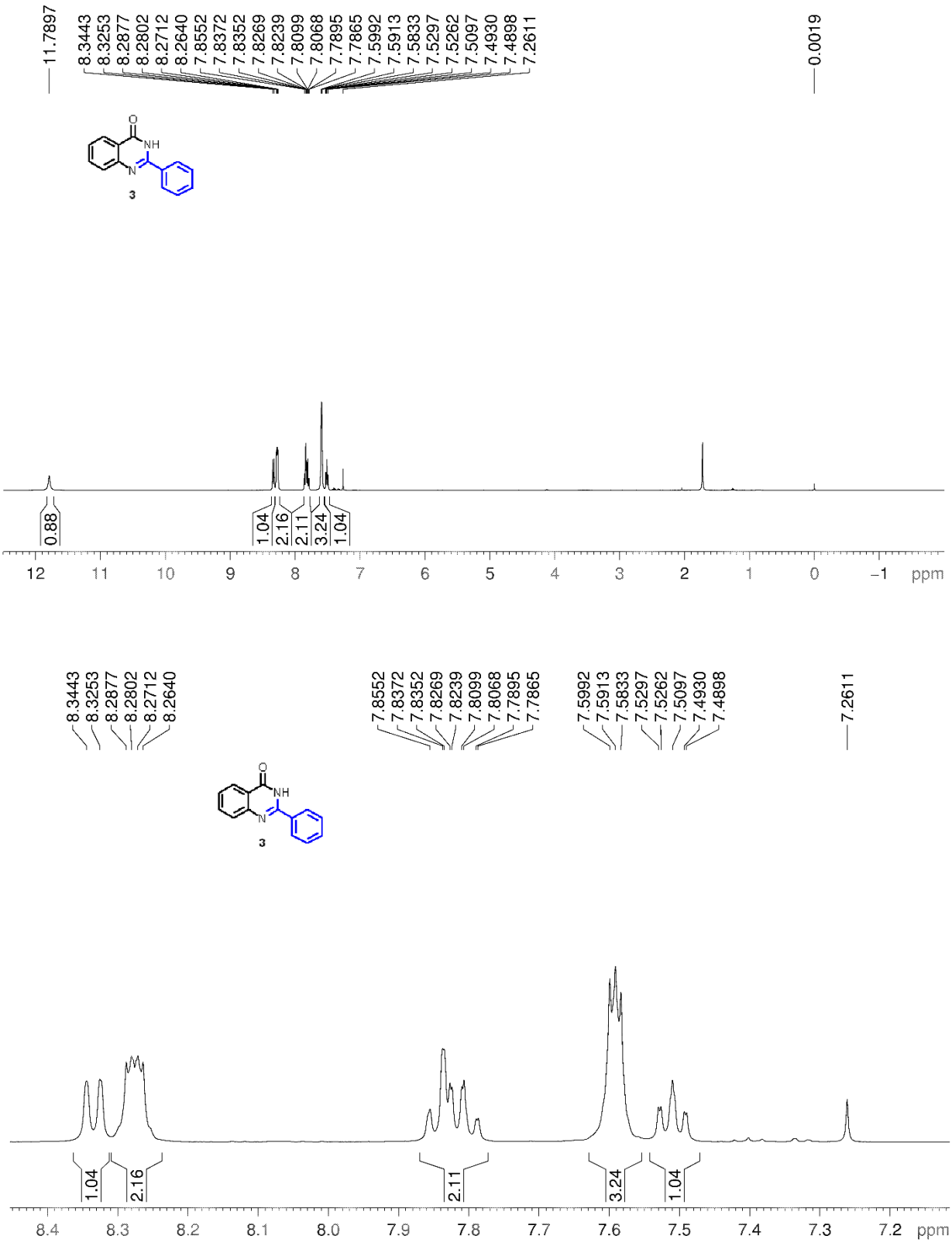
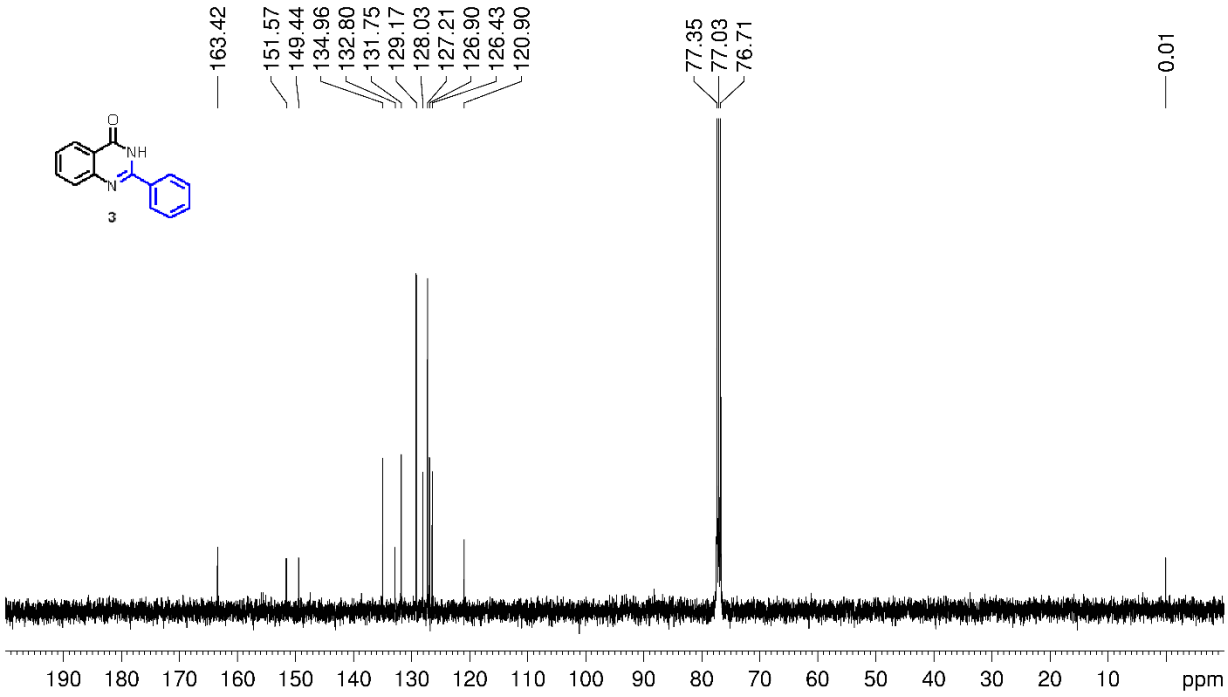
VII. NMR spectra

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134.96
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131.75
129.17
128.03
127.21
126.90
126.43
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77.03
76.71
0.01
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N
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3
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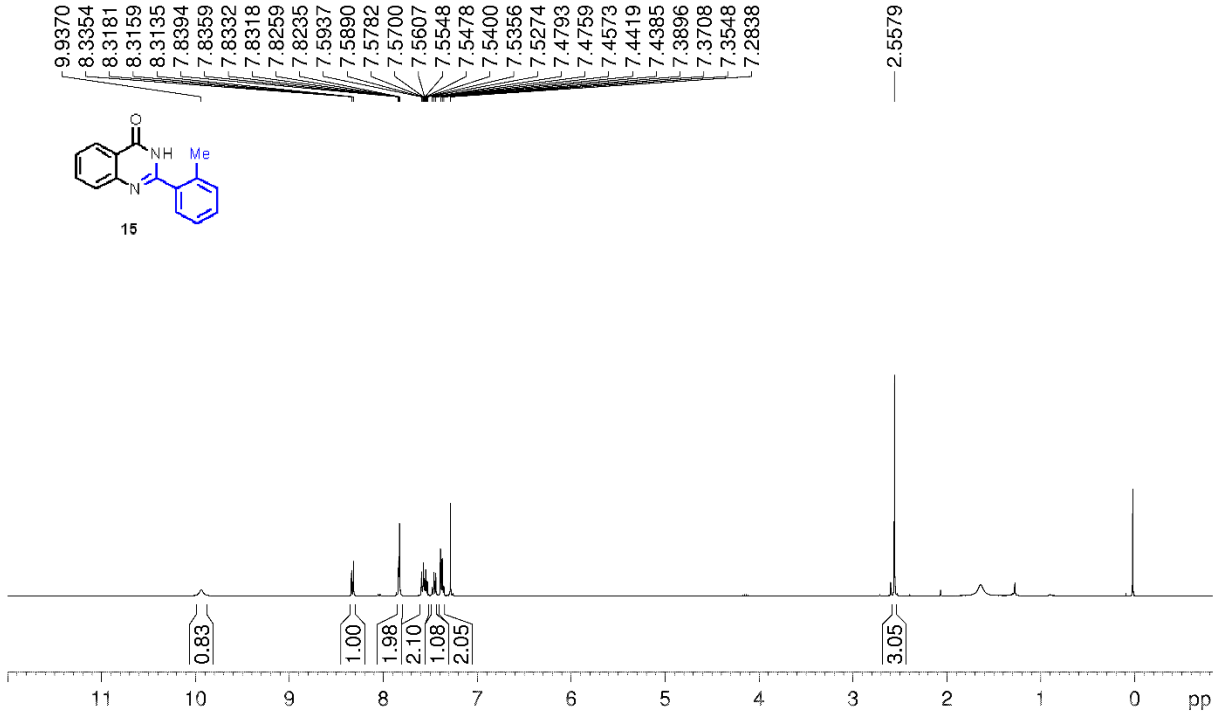
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2.05
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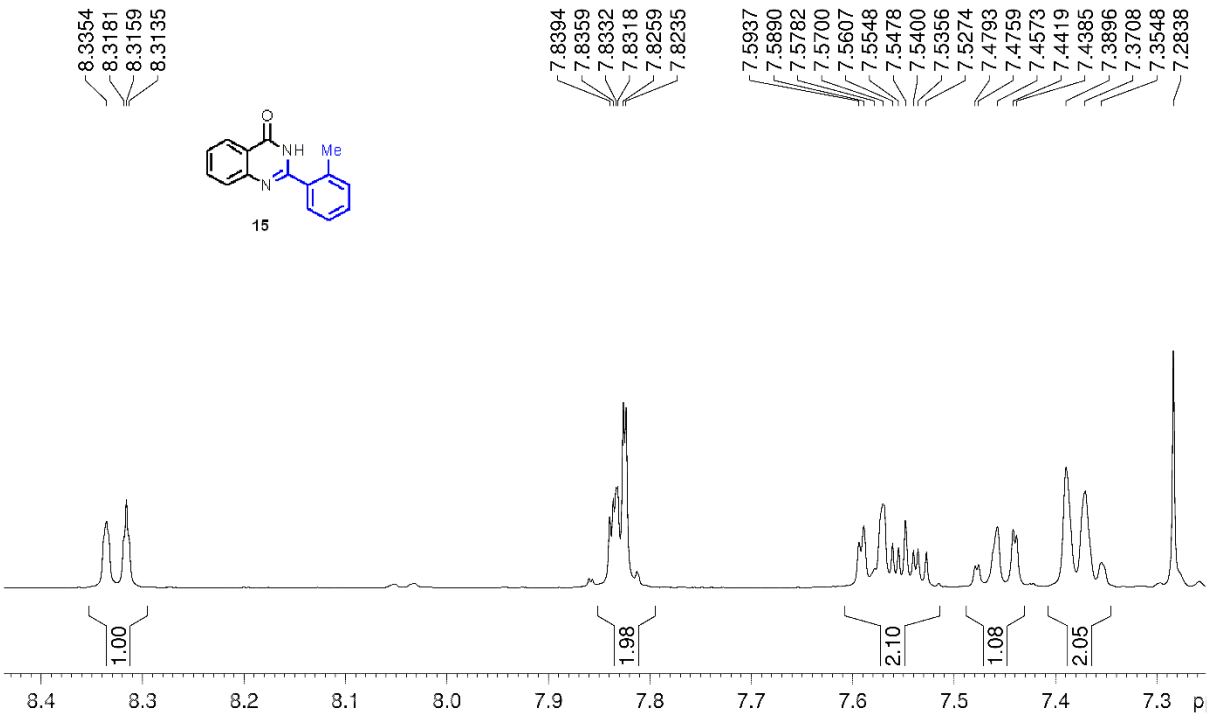
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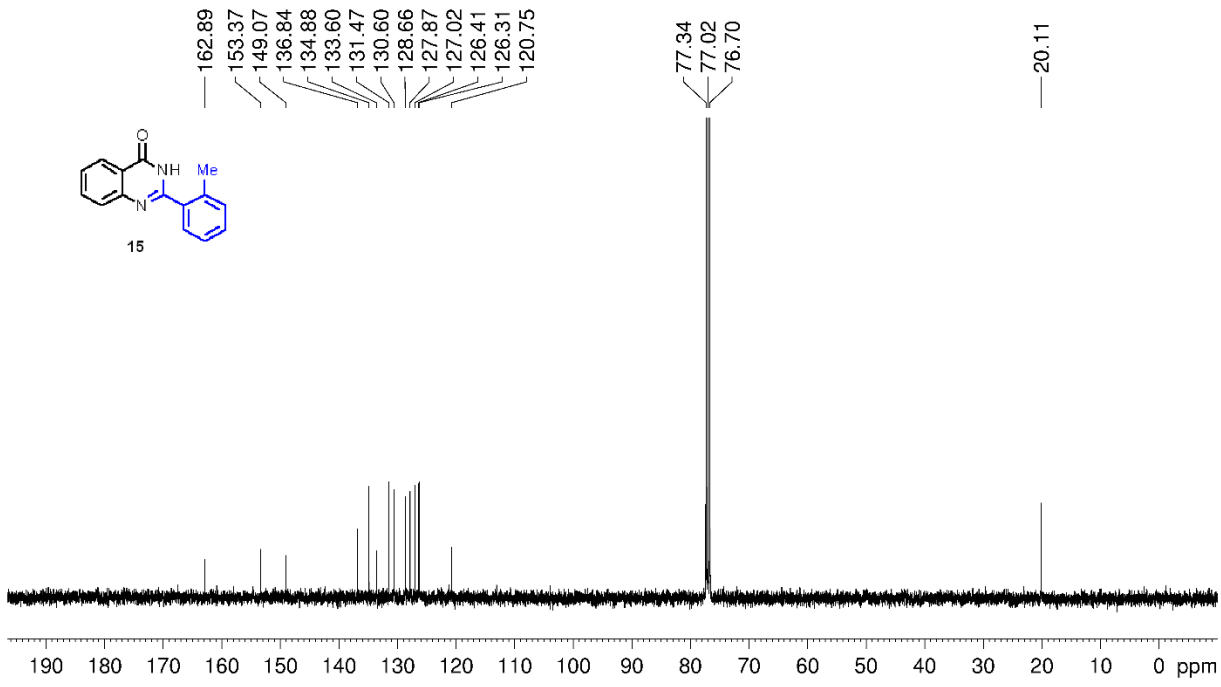
p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126.31
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77.02
76.70
20.11
NH
Me
N
O
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120
110
100
90
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20
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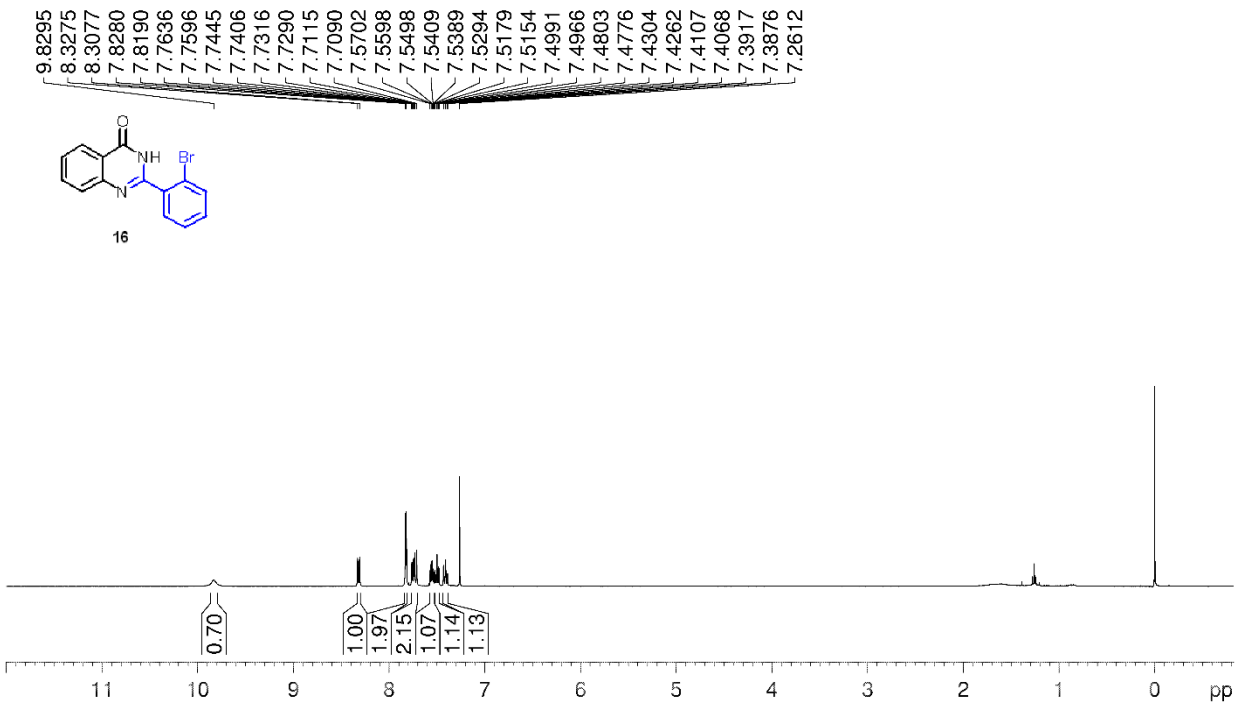
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7.7290
7.7115
7.7090
7.5702
7.5598
7.5498
7.5409
7.5389
7.5294
7.5179
7.5154
7.4991
7.4966
7.4803
7.4776
7.4304
7.4262
7.4107
7.4068
7.3917
7.3876
7.2612
O
NH
Br
N
16
0.70
1.00
1.97
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1.07
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1.13
11
10
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6
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2
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0
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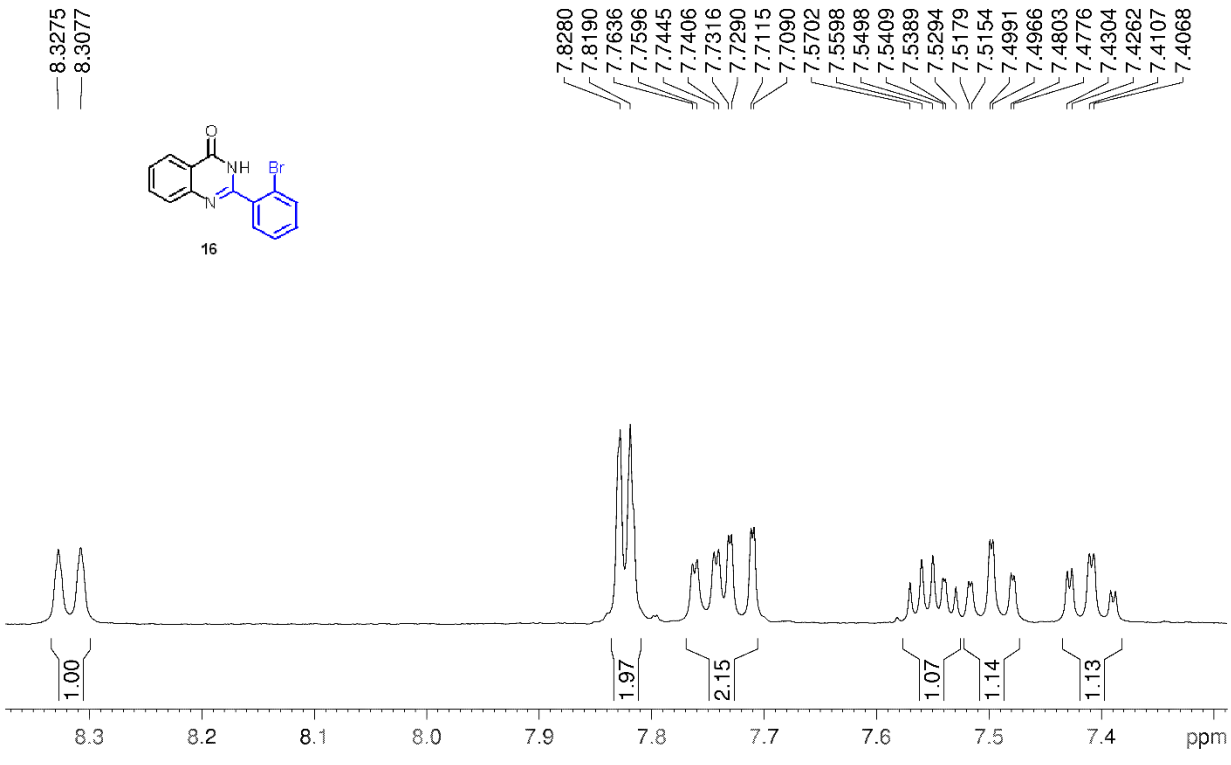
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7.7090
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7.5409
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7.5179
7.5154
7.4991
7.4966
7.4803
7.4776
7.4304
7.4262
7.4107
7.4068
O
NH
Br
N
16
1.00
1.97
2.15
1.07
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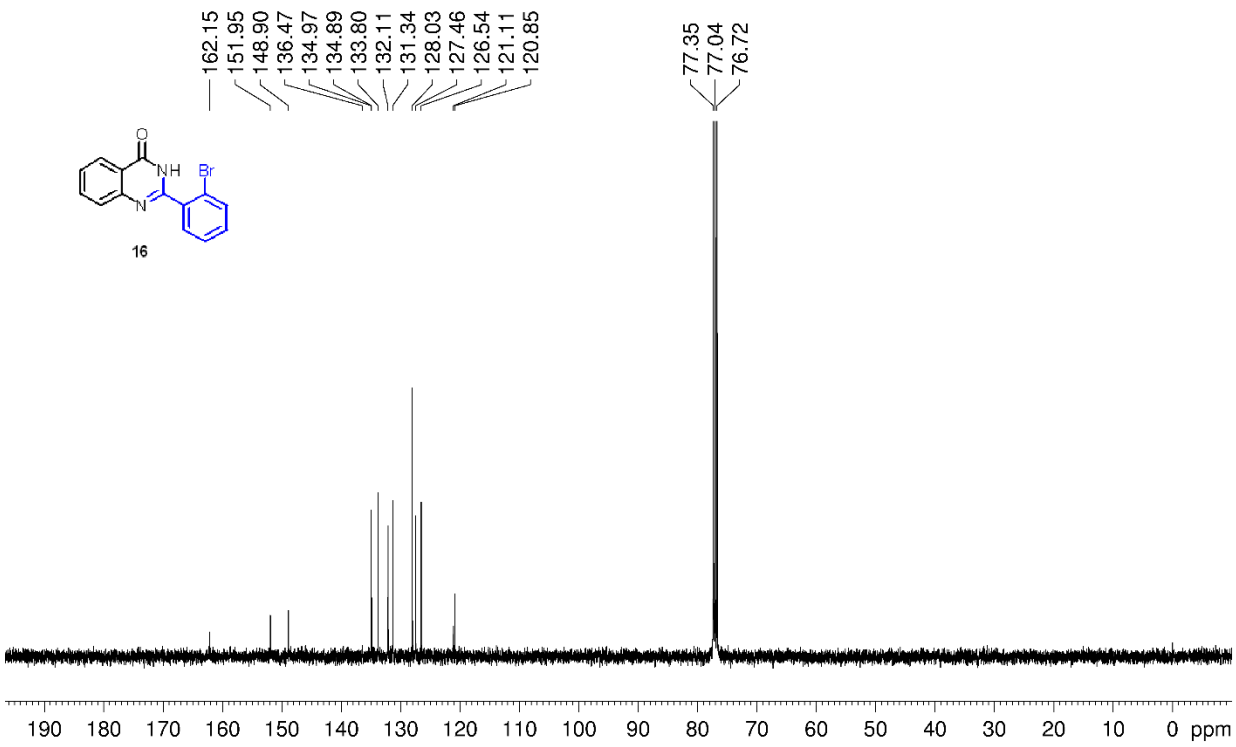
7.4
ppm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132.11
131.34
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126.54
121.11
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77.04
76.72
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NH
Br
N
16
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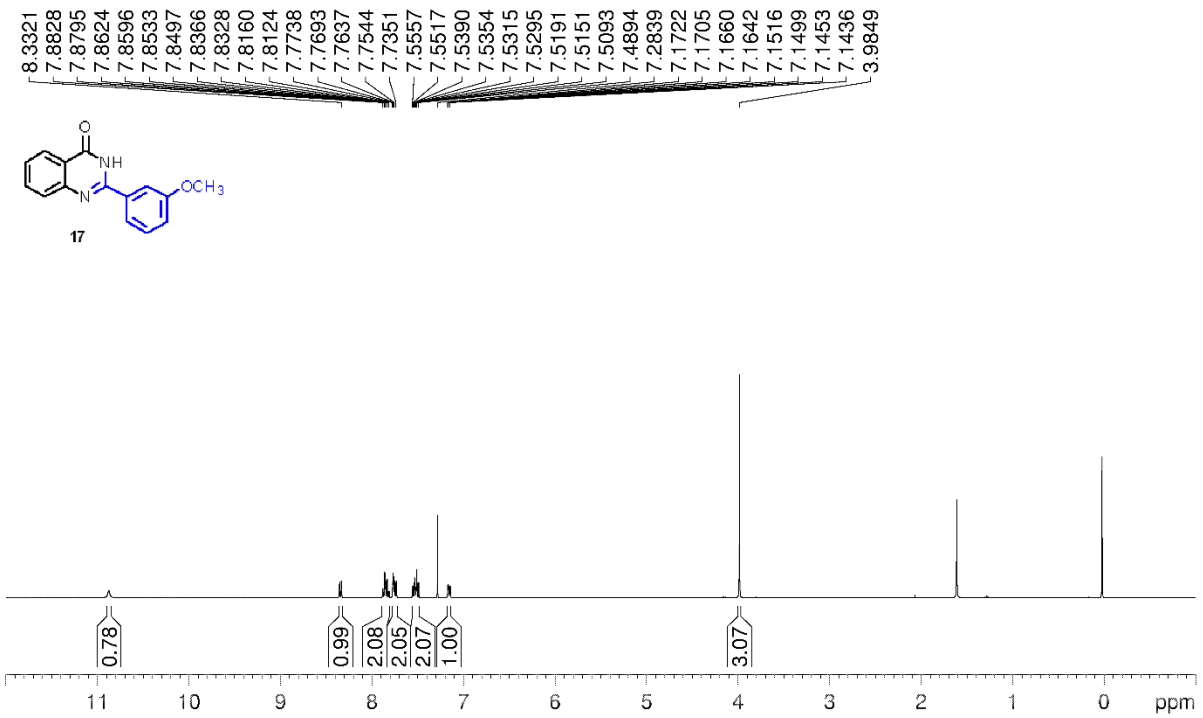
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7.7738
7.7693
7.7637
7.7544
7.7351
7.5557
7.5517
7.5390
7.5354
7.5315
7.5295
7.5191
7.5151
7.5093
7.4894
7.2839
7.1722
7.1705
7.1660
7.1642
7.1516
7.1499
7.1453
7.1436
3.9849
O
NH
OCH3
N
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0.78
0.99
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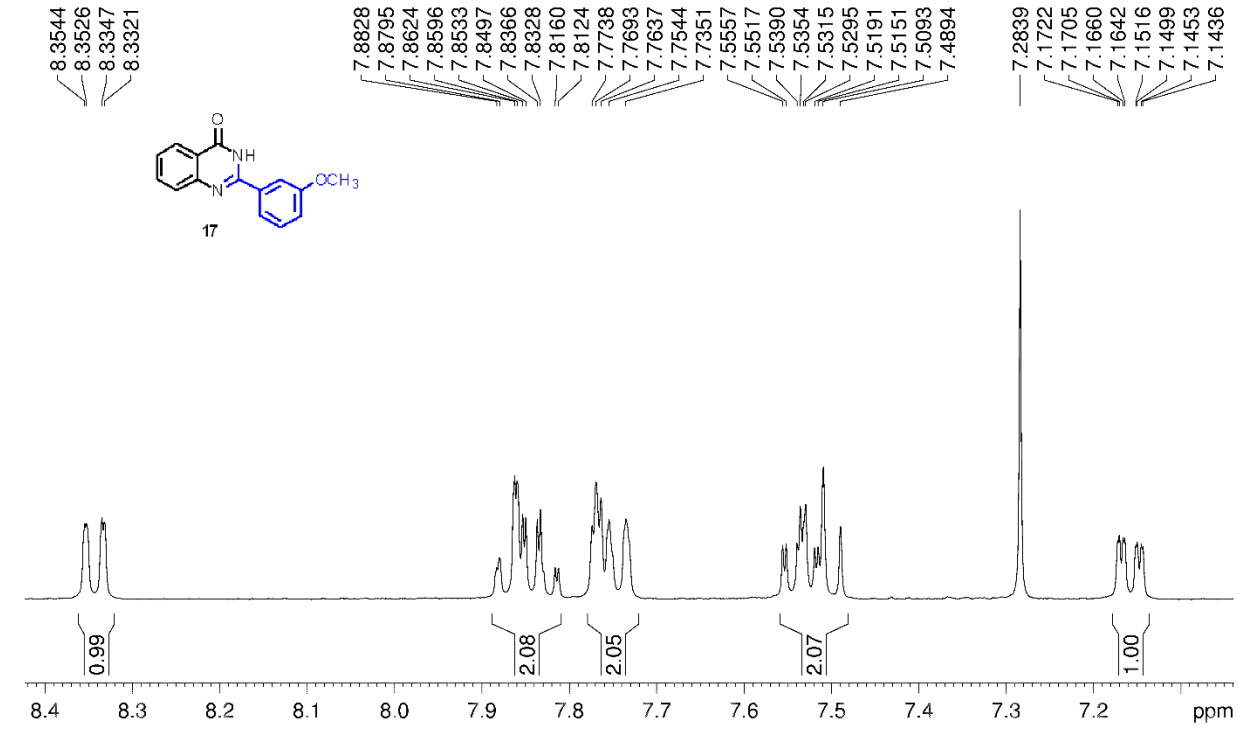
2.05
2.07
1.00
3.07
11 10 9 8 7 6 5 4 3 2 1 0 ppm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7.8328
7.8160
7.8124
7.7738
7.7693
7.7637
7.7544
7.7351
7.5557
7.5517
7.5390
7.5354
7.5315
7.5295
7.5191
7.5151
7.5093
7.4894
7.2839
7.1722
7.1705
7.1660
7.1642
7.1516
7.1499
7.1453
7.1436
NH
N
O
OCH3
17
0.99
2.08
2.05
2.07
1.00
8.4
8.3
8.2
8.1
8.0
7.9
7.8
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7.5
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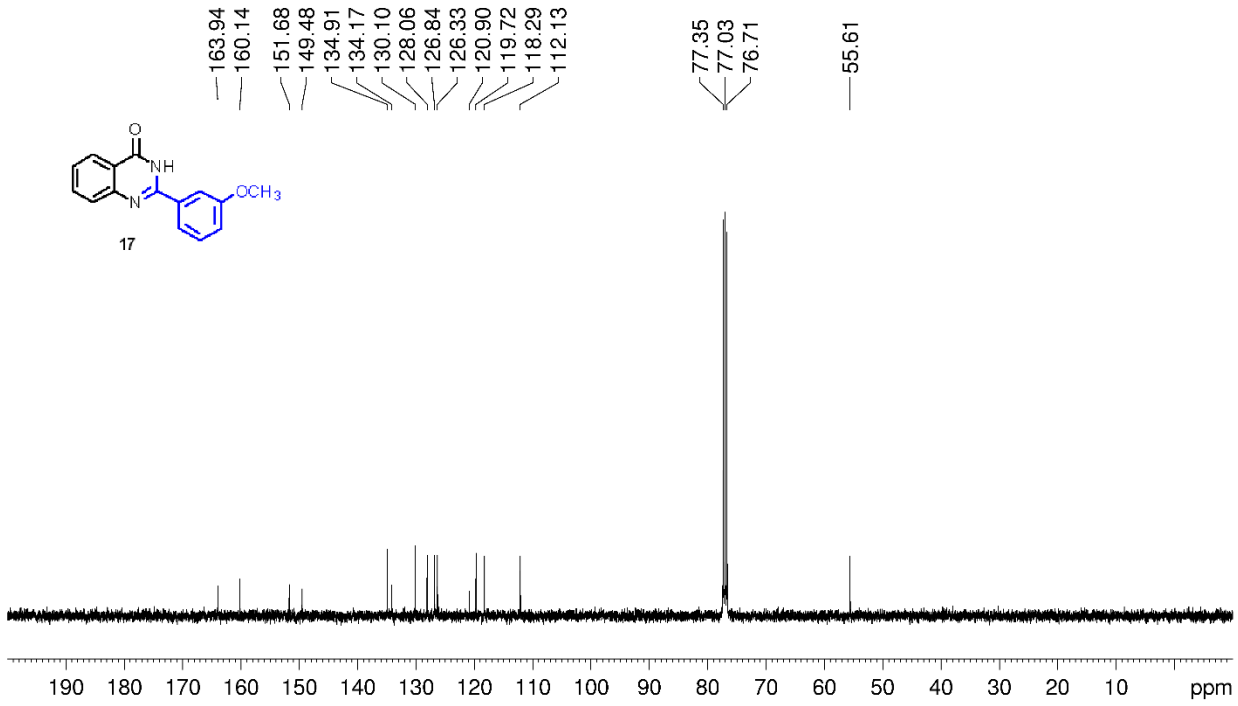
7.3
7.2
ppm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134.17
130.10
128.06
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126.33
120.90
119.72
118.29
112.13
77.35
77.03
76.71
55.61
NH
N
O
OCH3
17
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160
150
140
130
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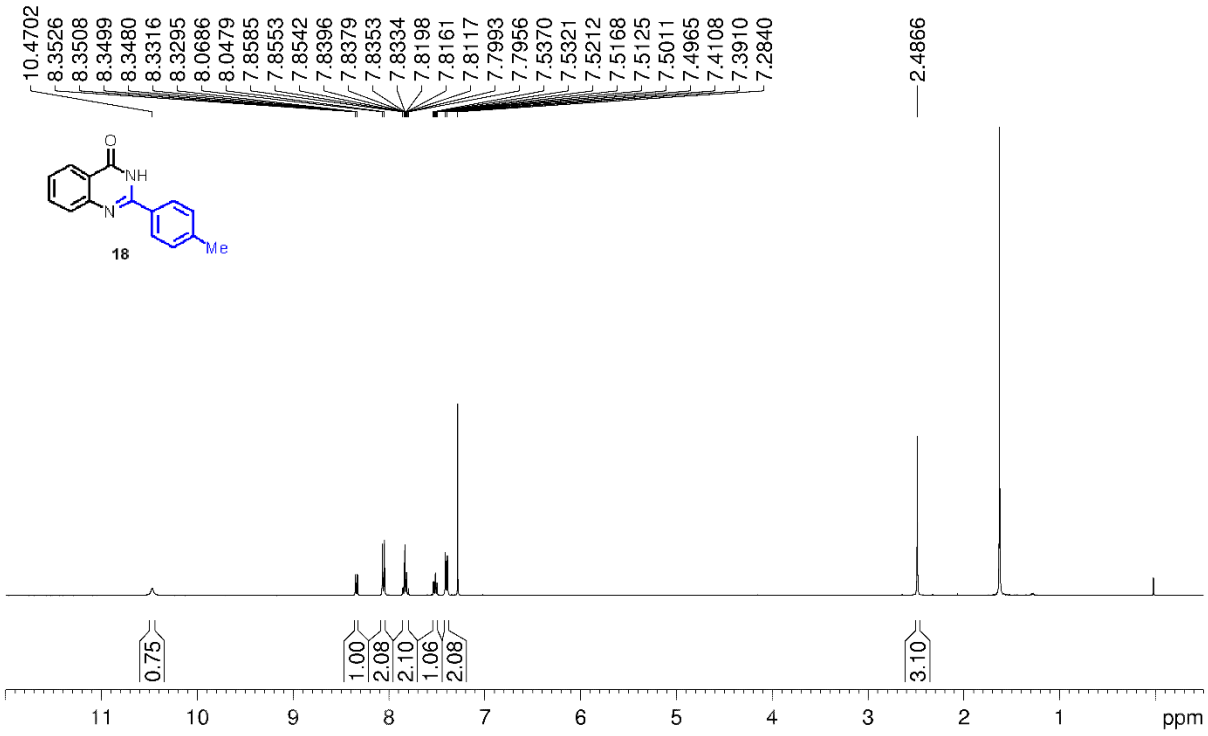
90
80
70
60
50
40
30
20
10
ppm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8.3295
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8.0479
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7.8553
7.8542
7.8396
7.8379
7.8353
7.8334
7.8198
7.8161
7.8117
7.7993
7.7956
7.5370
7.5321
7.5212
7.5168
7.5125
7.5011
7.4965
7.4108
7.3910
7.2840
2.4866
NH
N
O
Me
18
0.75
1.00
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2.10
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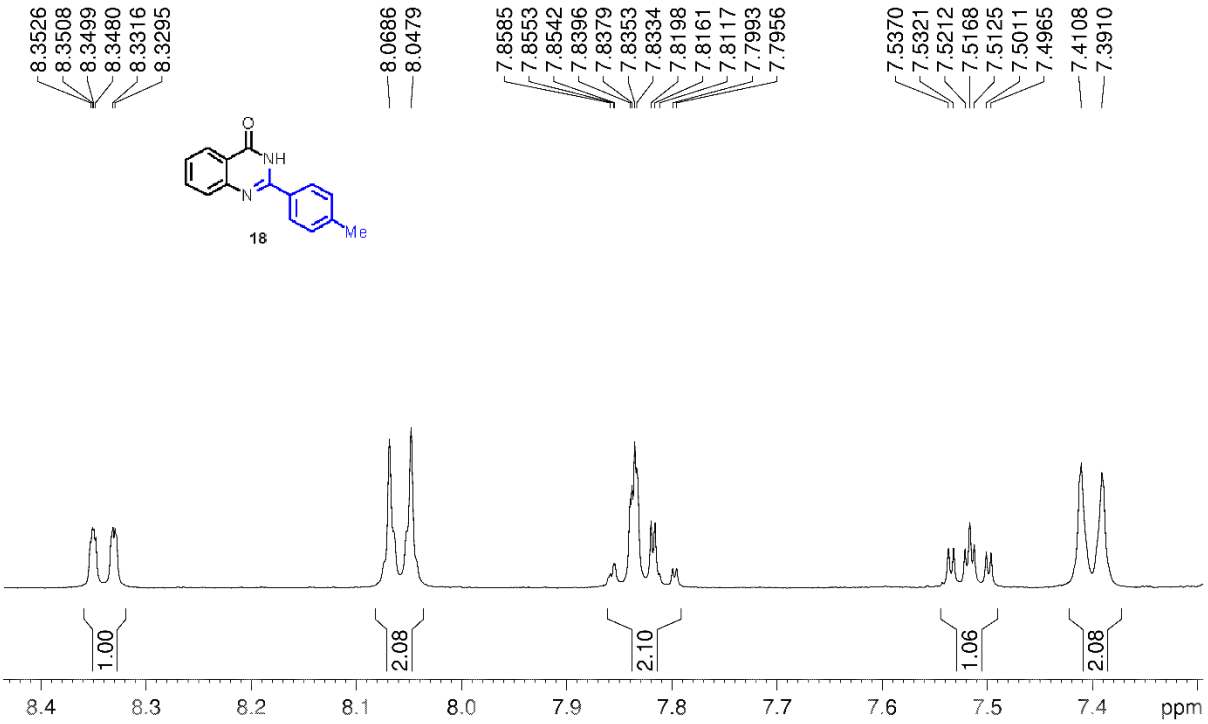
2.08
3.10
11
10
9
8
7
6
5
4
3
2
1
ppm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7.8396
7.8379
7.8353
7.8334
7.8198
7.8161
7.8117
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7.5370
7.5321
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7.5125
7.5011
7.4965
7.4108
7.3910
NH
N
O
Me
18
1.00
2.08
2.10
1.06
2.08
8.4
8.3
8.2
8.1
8.0
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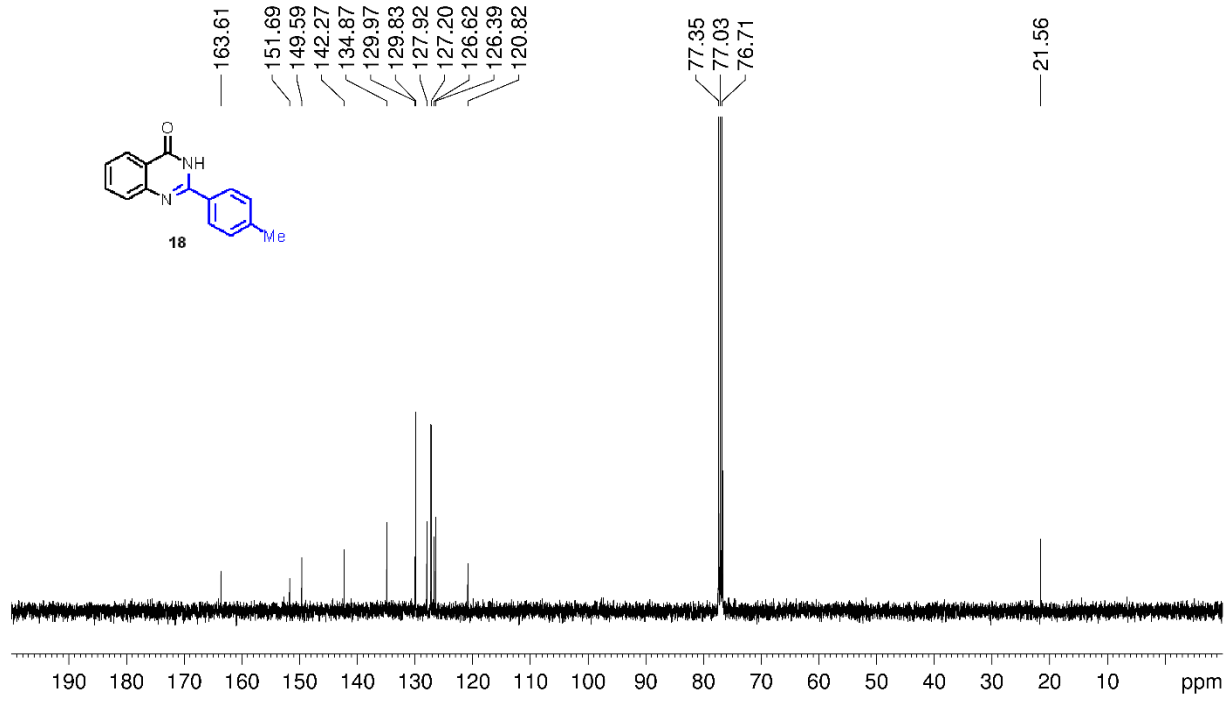
7.7
7.6
7.5
7.4
ppm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127.92
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126.62
126.39
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76.71
21.56
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N
O
Me
18
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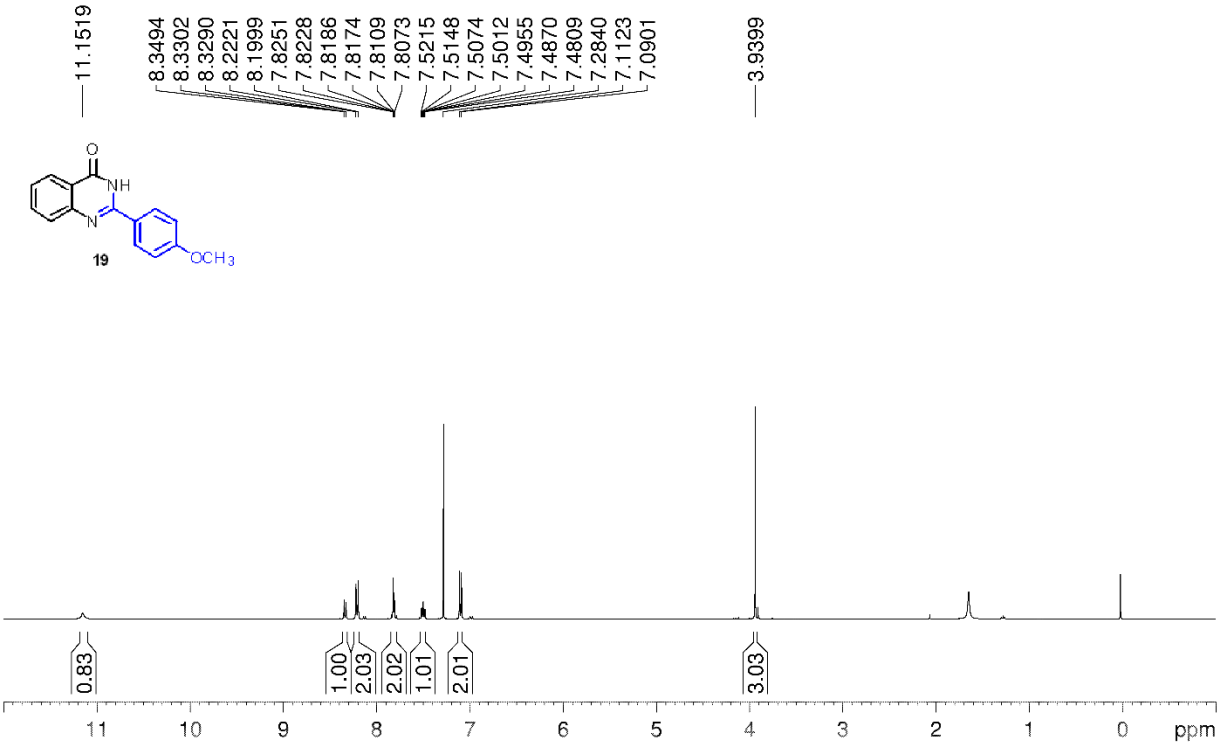
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8.3290
8.2221
8.1999
7.8251
7.8228
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7.8174
7.8109
7.8073
7.5215
7.5148
7.5074
7.5012
7.4955
7.4870
7.4809
7.2840
7.1123
7.0901
3.9399
NH
N
O
OCH3
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1.00
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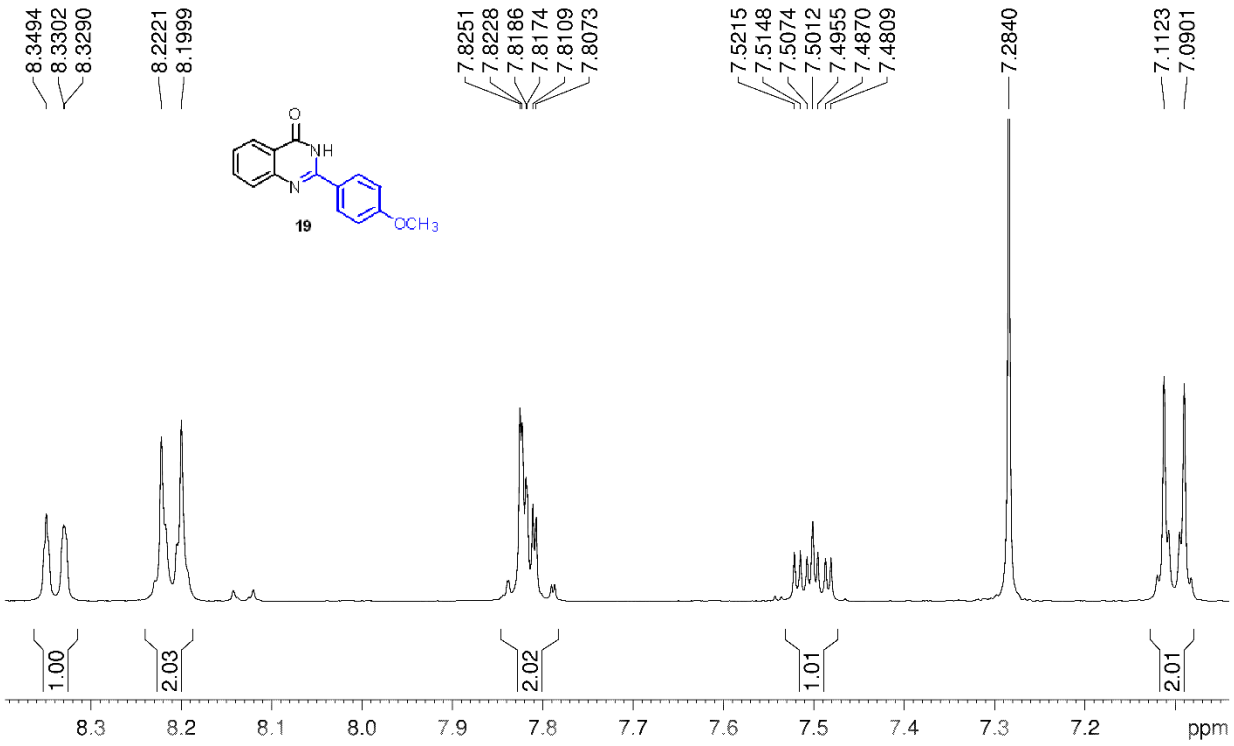
2.02
1.01
2.01
3.03
11 10 9 8 7 6 5 4 3 2 1 0 ppm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8.3302
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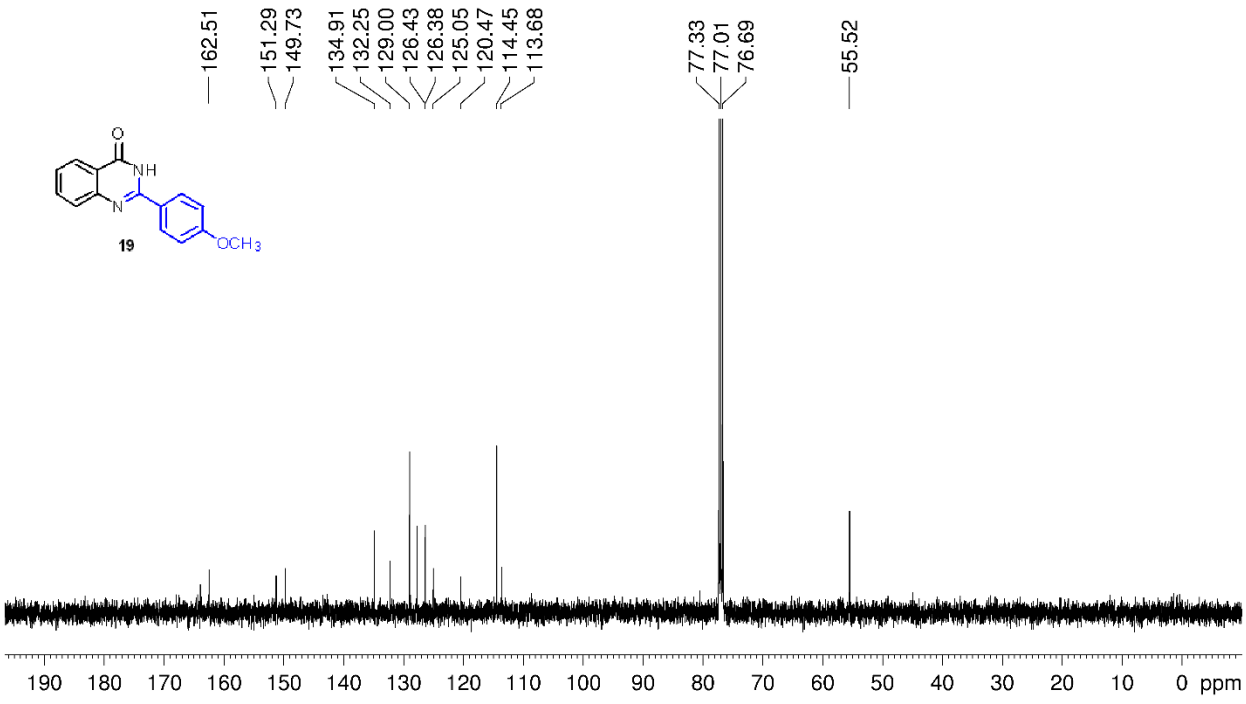
7.5215
7.5148
7.5074
7.5012
7.4955
7.4870
7.4809
7.2840
7.1123
7.0901
19
OCH3
1.00
2.03
2.02
1.01
2.01
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
ppm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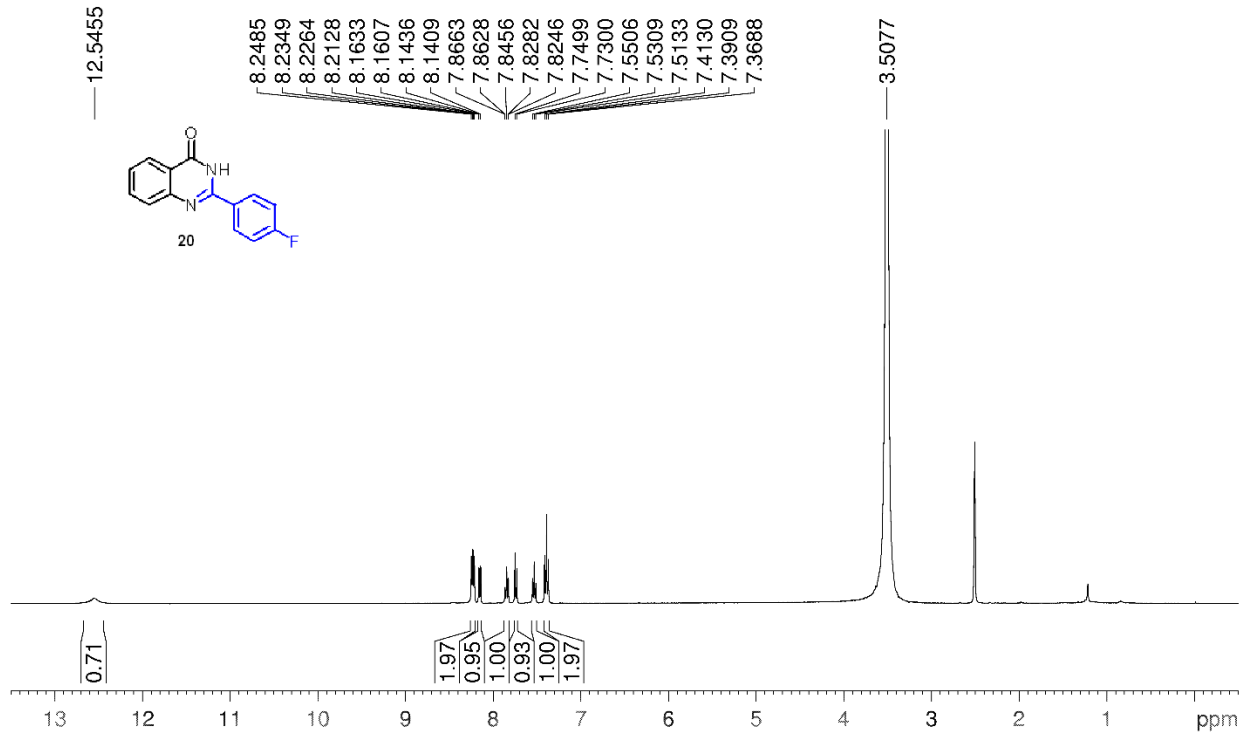
120.47
114.45
113.68
77.33
77.01
76.69
55.52
19
OCH3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm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8.1607
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8.1409
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7.8456
7.8282
7.8246
7.7499
7.7300
7.5506
7.5309
7.5133
7.4130
7.3909
7.3688
3.5077
NH
N
O
F
20
0.71
1.97
0.95
1.00
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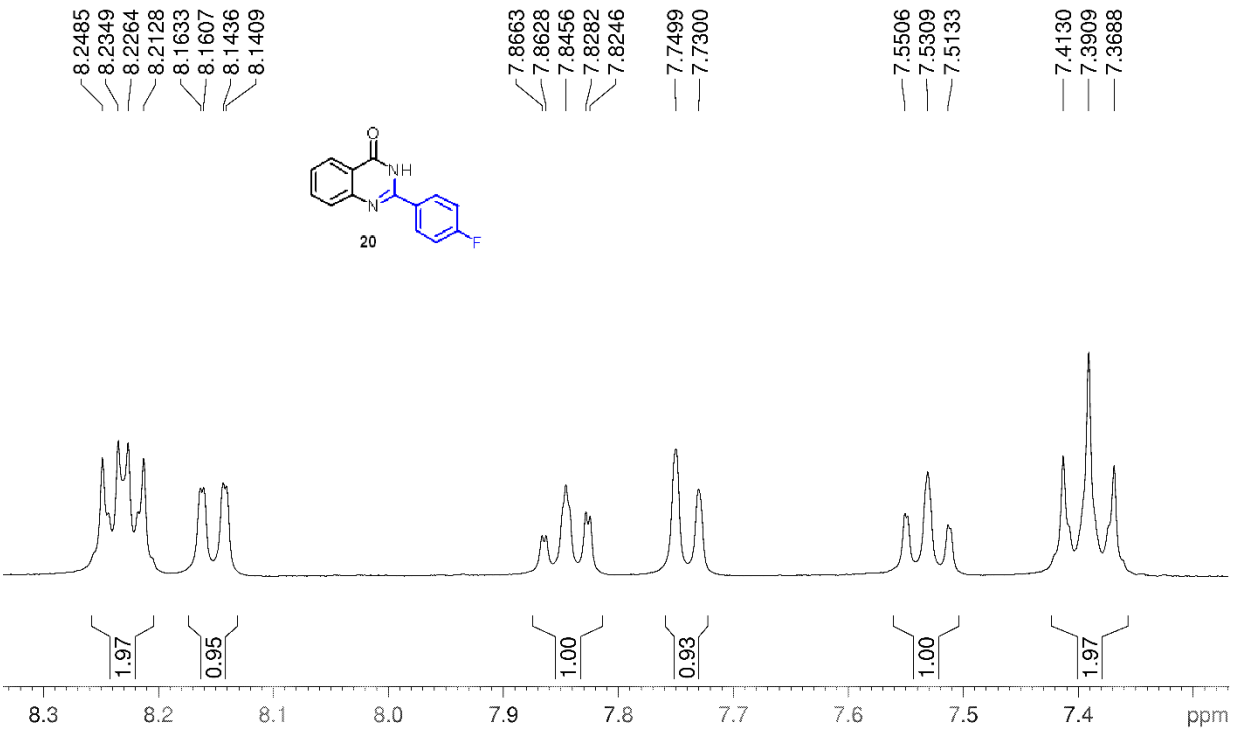
1.97
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm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7.7300
7.5506
7.5309
7.5133
7.4130
7.3909
7.3688
NH
N
O
F
20
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0.95
1.00
0.93
1.00
1.97
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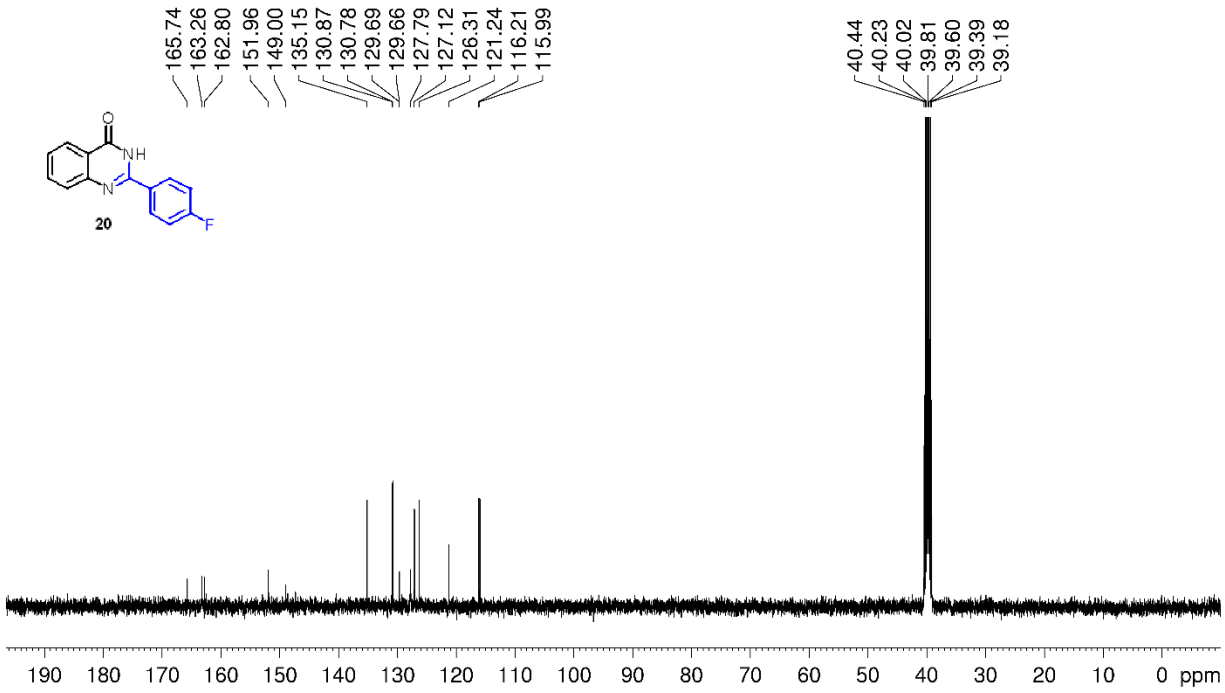
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
ppm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126.31
121.24
116.21
115.99
40.44
40.23
40.02
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39.39
39.18
O
NH
N
F
20
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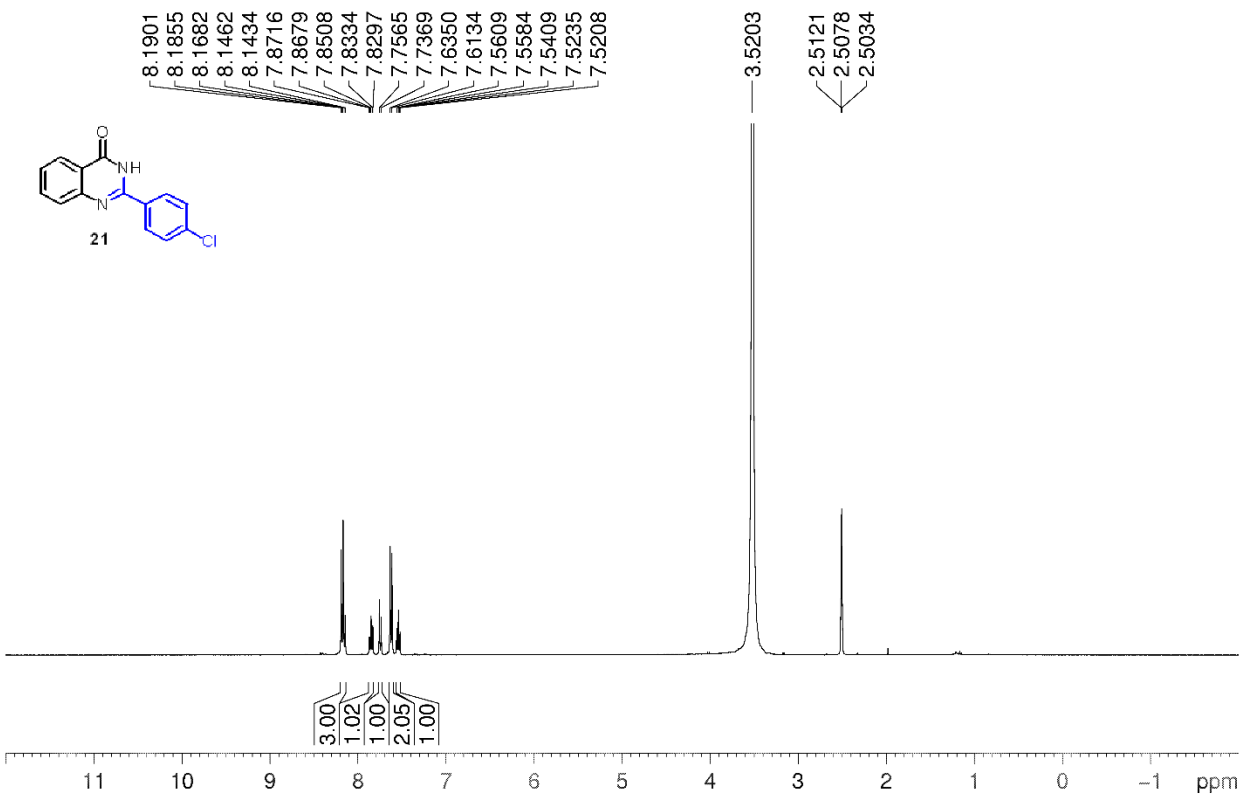
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7.8297
7.7565
7.7369
7.6350
7.6134
7.5609
7.5584
7.5409
7.5235
7.5208
3.5203
2.5121
2.5078
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NH
N
Cl
21
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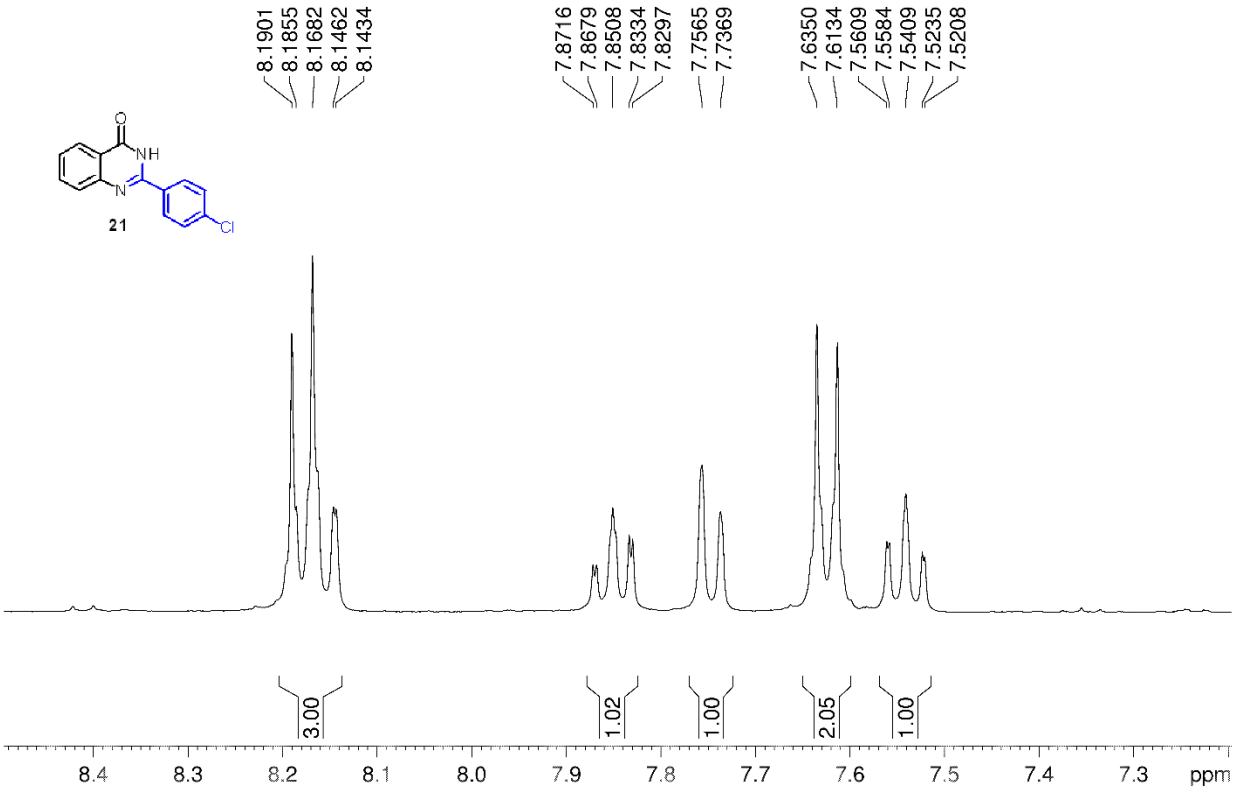
1.00
2.05
1.00
11 10 9 8 7 6 5 4 3 2 1 0 −1 ppm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7.8679
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7.8334
7.8297
7.7565
7.7369
7.6350
7.6134
7.5609
7.5584
7.5409
7.5235
7.5208
NH
N
O
Cl
21
3.00
1.02
1.00
2.05
1.00
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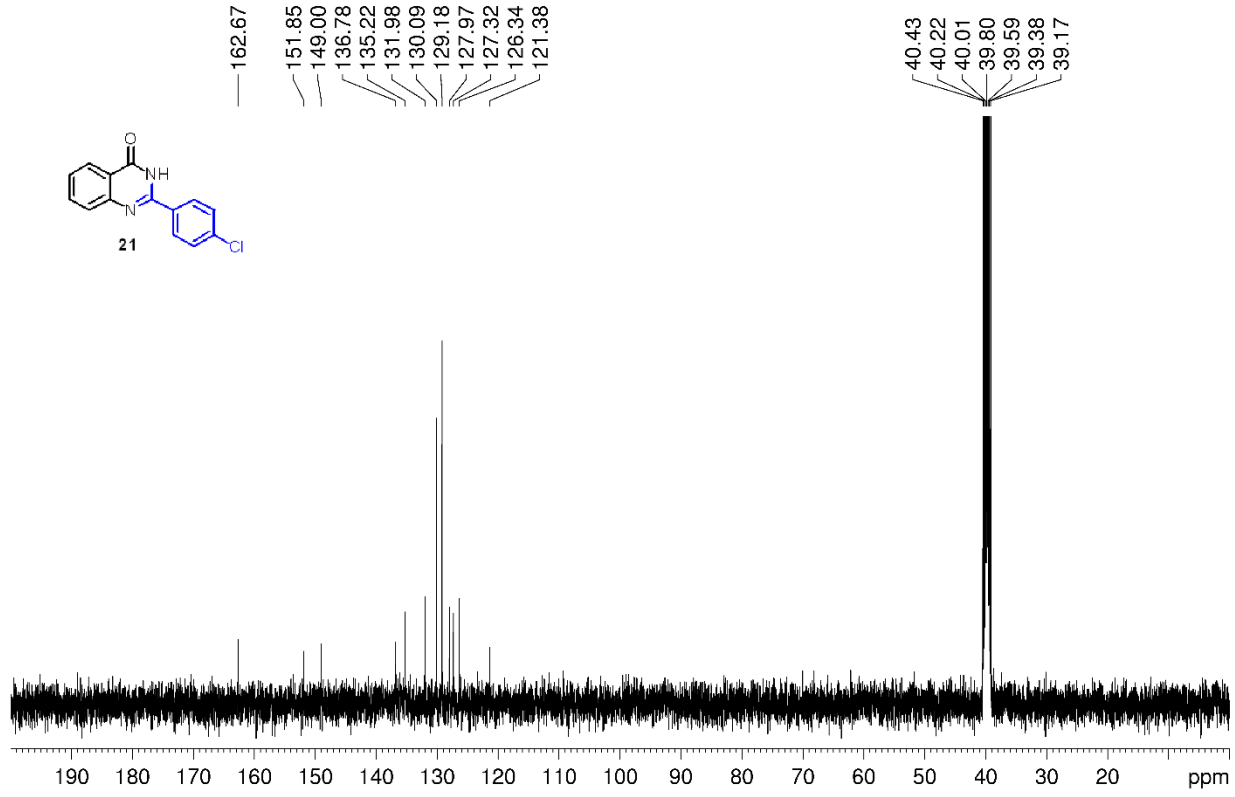
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
ppm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127.32
126.34
121.38
40.43
40.22
40.01
39.80
39.59
39.38
39.17
NH
N
O
Cl
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160
150
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80
70
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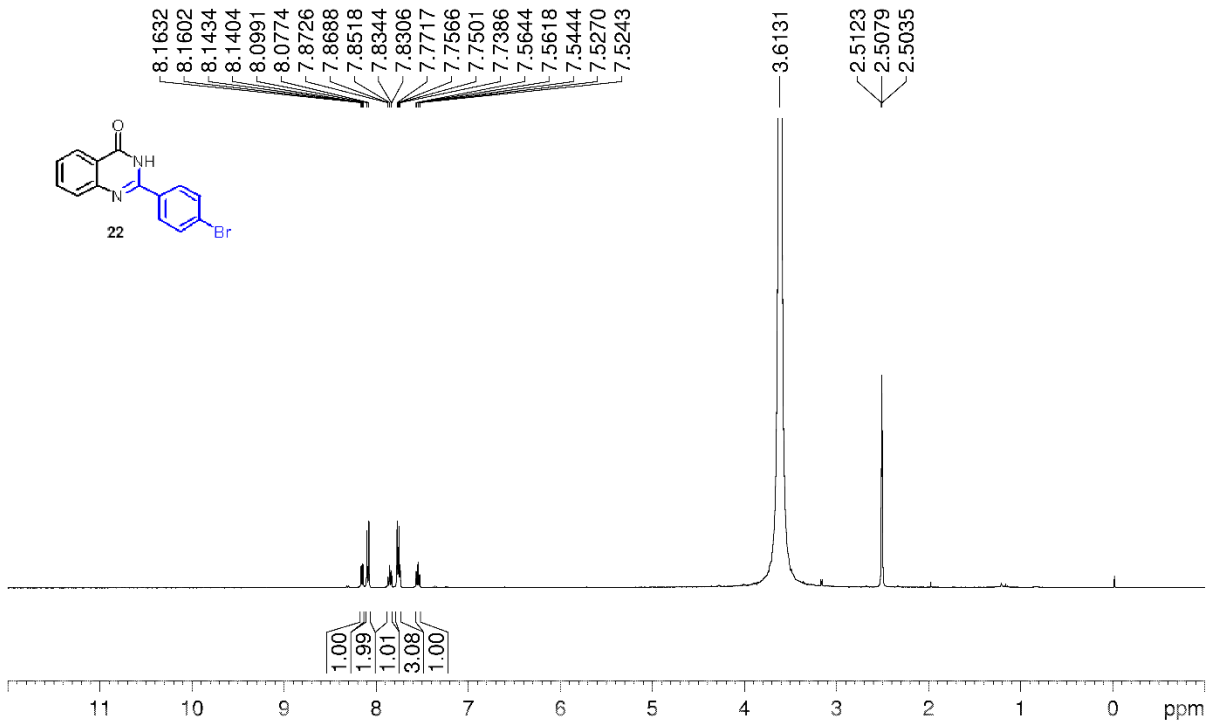
40
30
20
ppm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7.7501
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7.5618
7.5444
7.5270
7.5243
3.6131
2.5123
2.5079
2.5035
O
NH
N
Br
22
1.00
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3.08
1.00
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10
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7
6
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0
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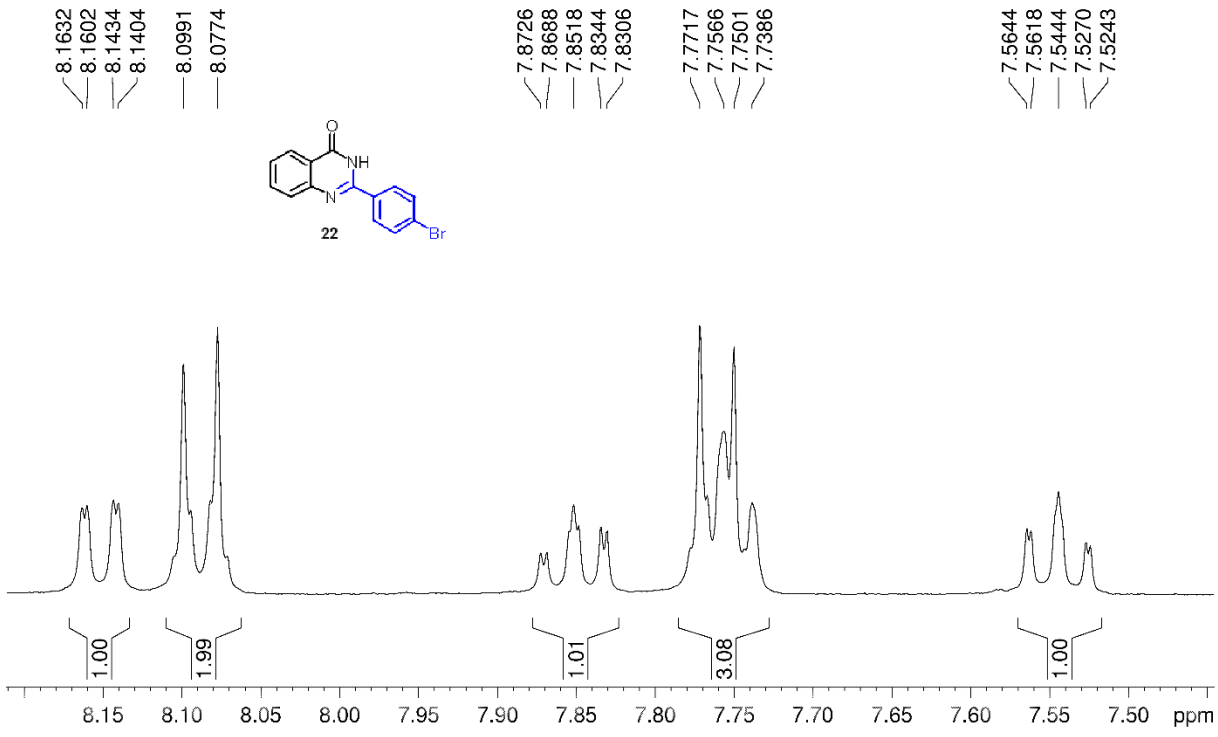
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8.1602
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7.7717
7.7566
7.7501
7.7386
7.5644
7.5618
7.5444
7.5270
7.5243
O
NH
N
Br
22
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1.99
1.01
3.08
1.00
8.15
8.10
8.05
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7.55
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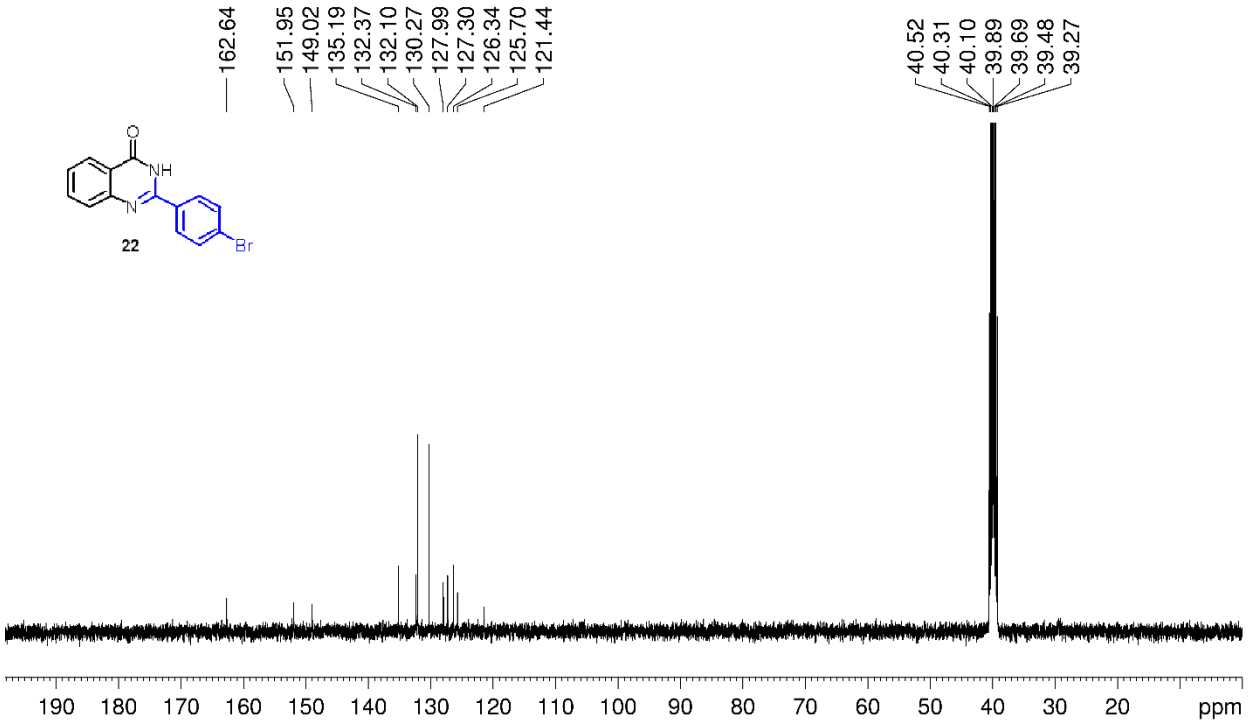
ppm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40.52
40.31
40.10
39.89
39.69
39.48
39.27
O
NH
N
Br
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180
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160
150
140
130
120
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80
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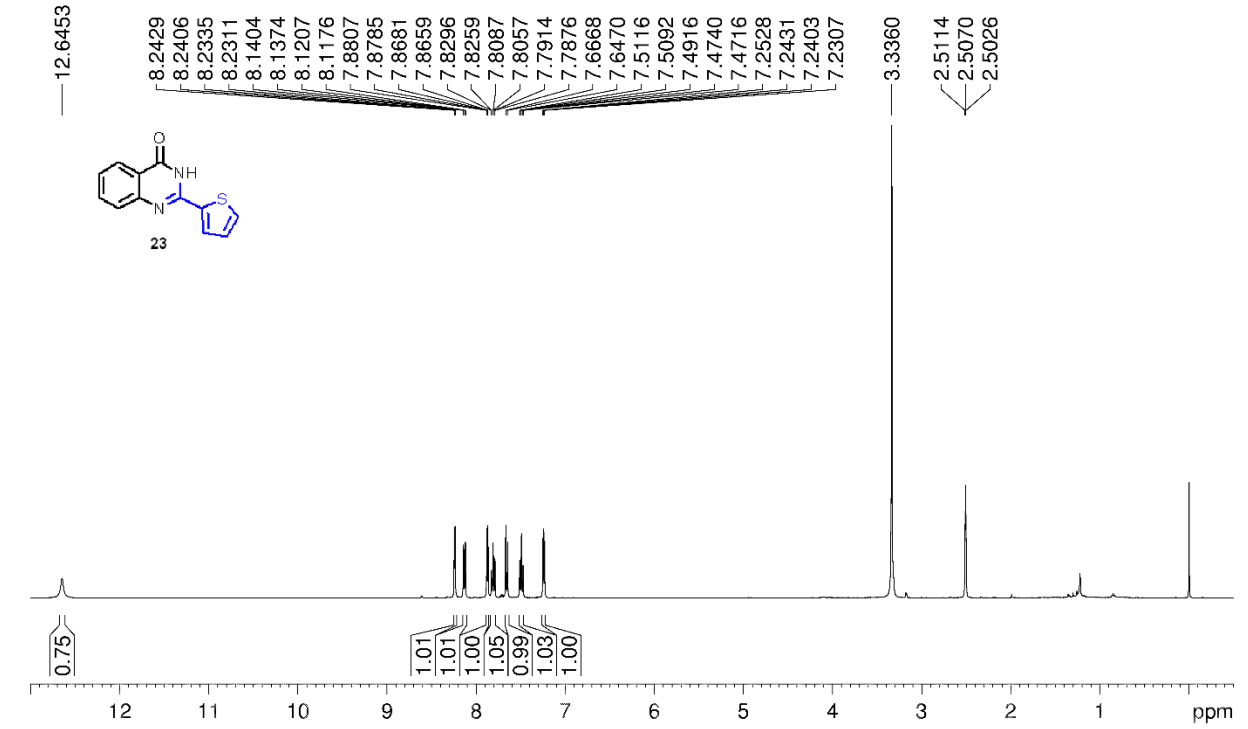
60
50
40
30
20
ppm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8.1374
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8.1176
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7.8785
7.8681
7.8659
7.8296
7.8259
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7.8057
7.7914
7.7876
7.6668
7.6470
7.5116
7.5092
7.4916
7.4740
7.4716
7.2528
7.2431
7.2403
7.2307
3.3360
2.5114
2.5070
2.5026
O
NH
N
S
23
0.75
1.01
1.01
1.00
1.05
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1.03
1.00
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11
10
9
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7
6
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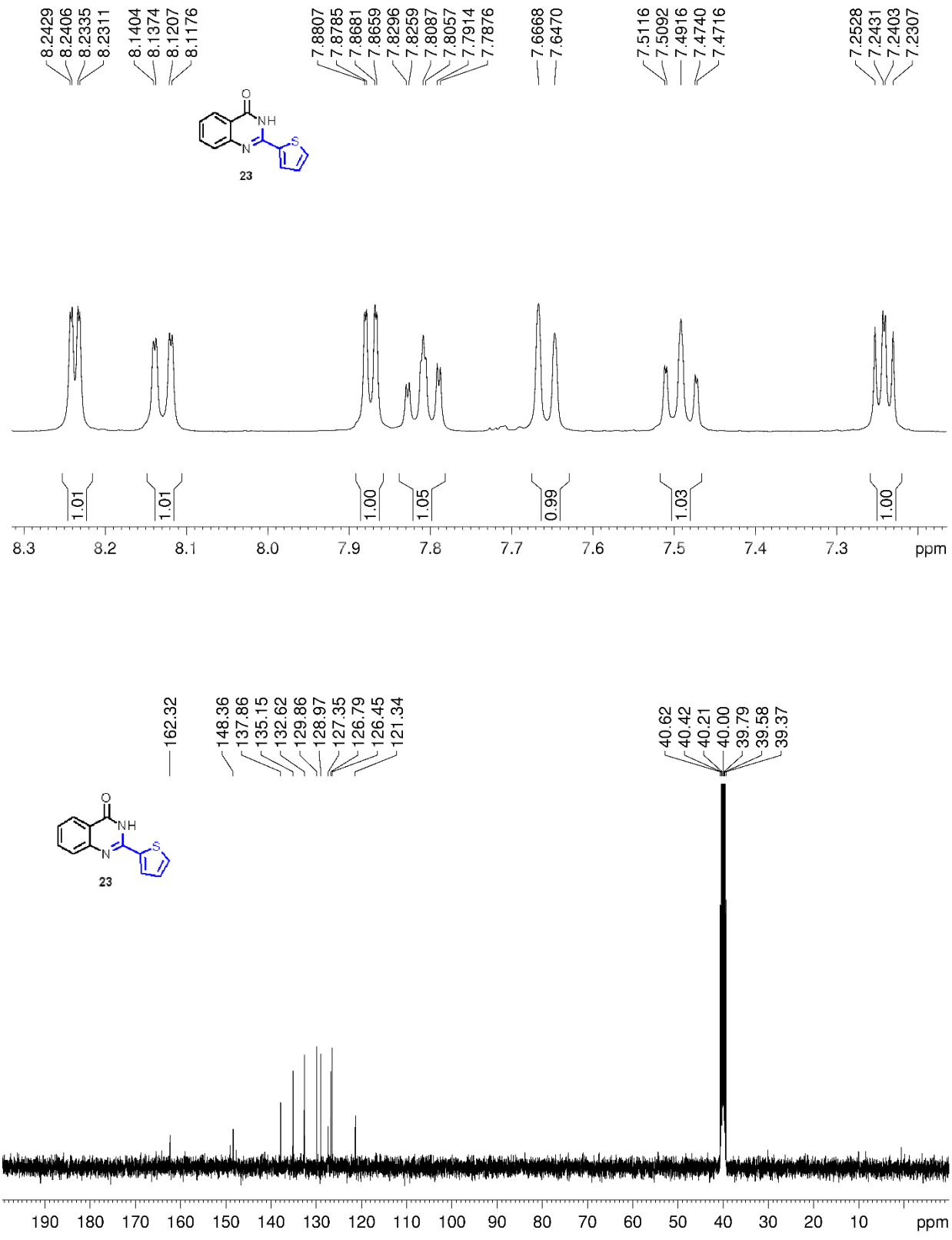
4
3
2
1
ppm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7.8681
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7.6470
7.5116
7.5092
7.4916
7.4740
7.4716
7.2528
7.2431
7.2403
7.2307
O
NH
N
S
23
1.01
1.01
1.00
1.05
0.99
1.03
1.00
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
ppm
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135.15
132.62
129.86
128.97
127.35
126.79
126.45
121.34
40.62
40.42
40.21
40.00
39.79
39.58
39.37
O
NH
N
S
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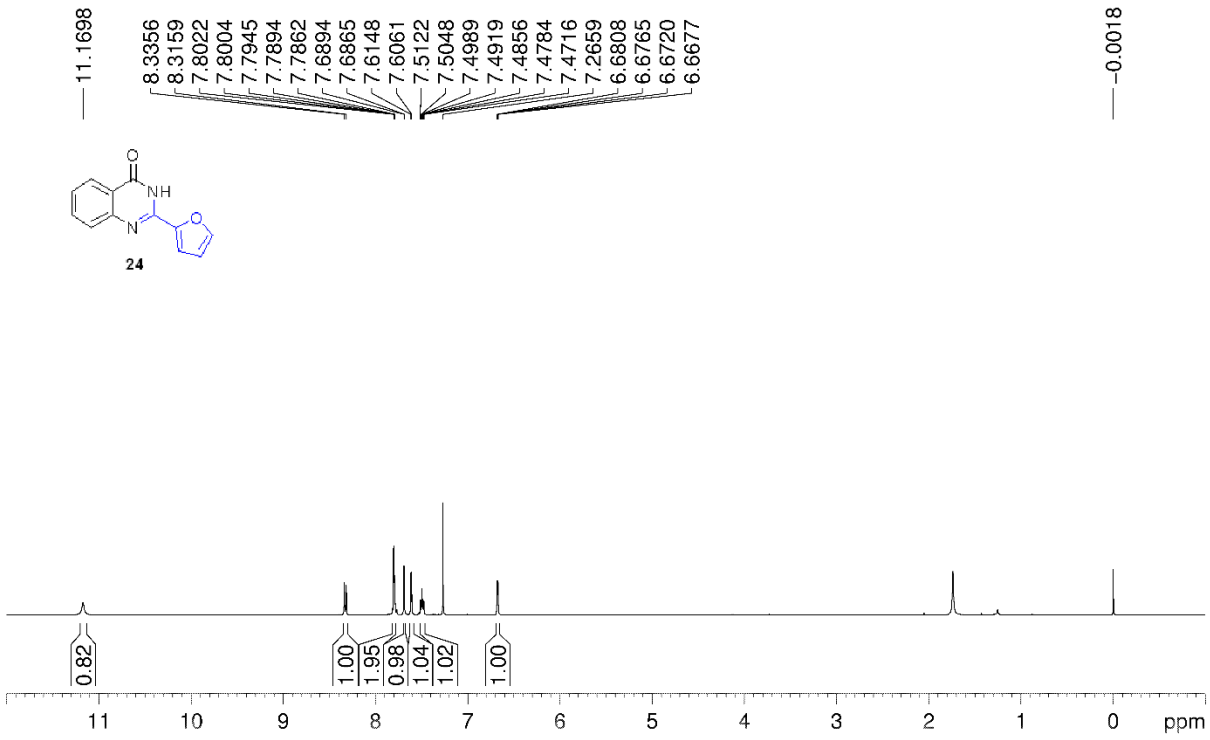
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6.6720
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N
O
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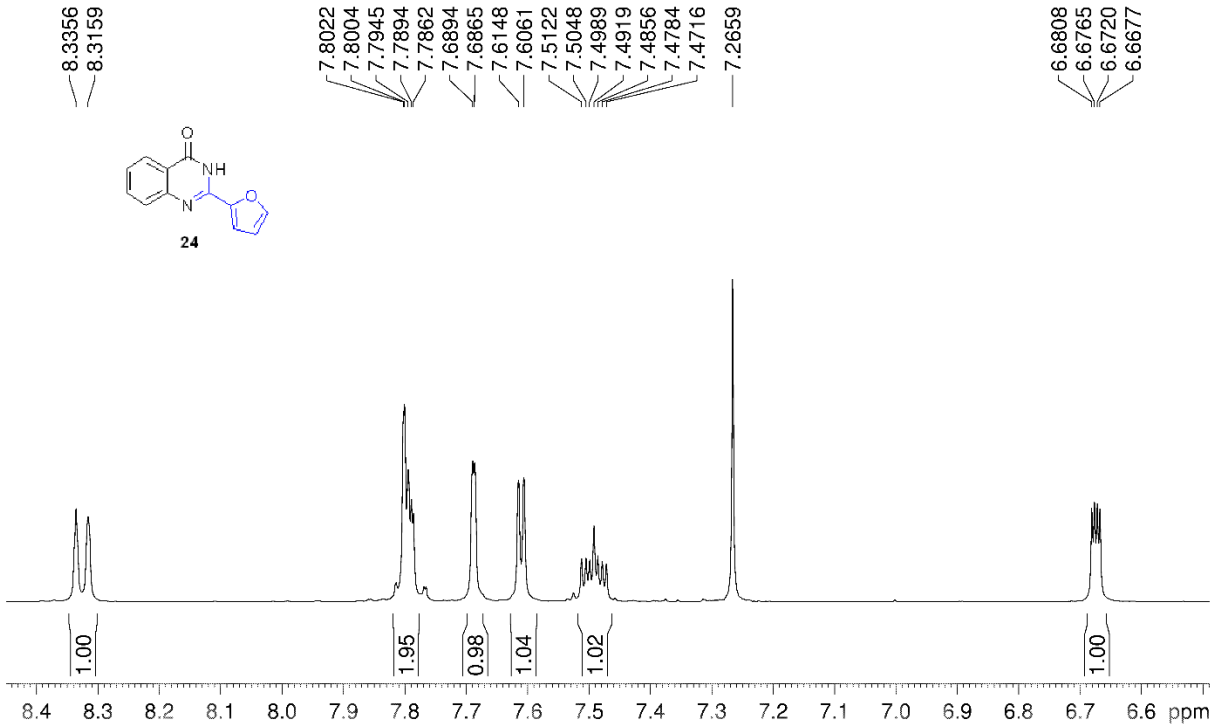
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6.6808
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6.6720
6.6677
NH
N
O
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1.95
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1.02
1.00
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7.9
7.8
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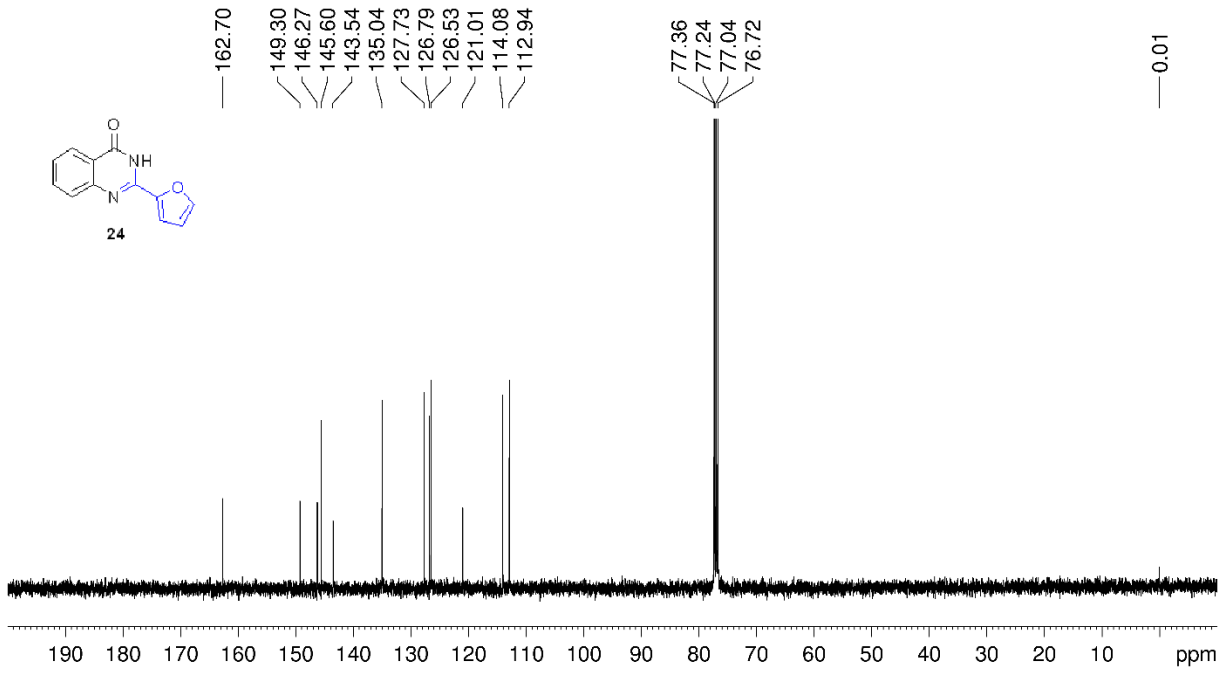
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126.79
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114.08
112.94
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O
NH
N
O
24
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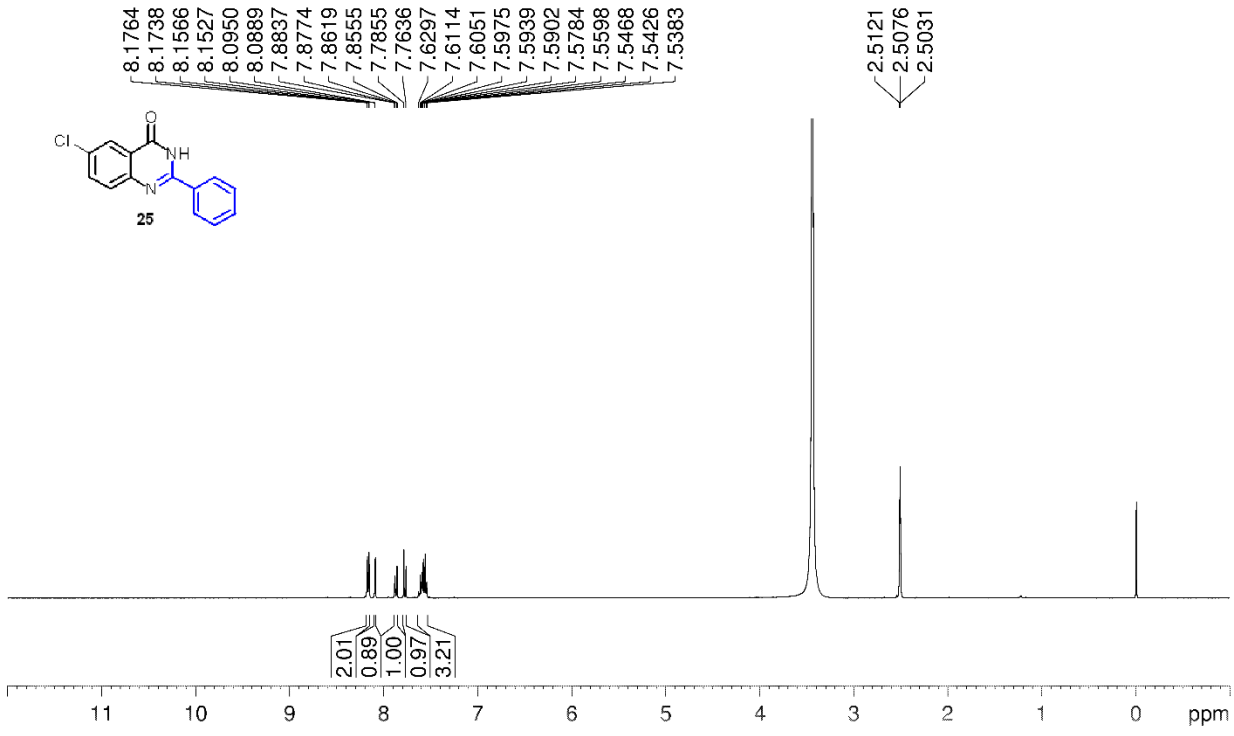
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7.7636
7.6297
7.6114
7.6051
7.5975
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7.5784
7.5598
7.5468
7.5426
7.5383
2.5121
2.5076
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N
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0.97
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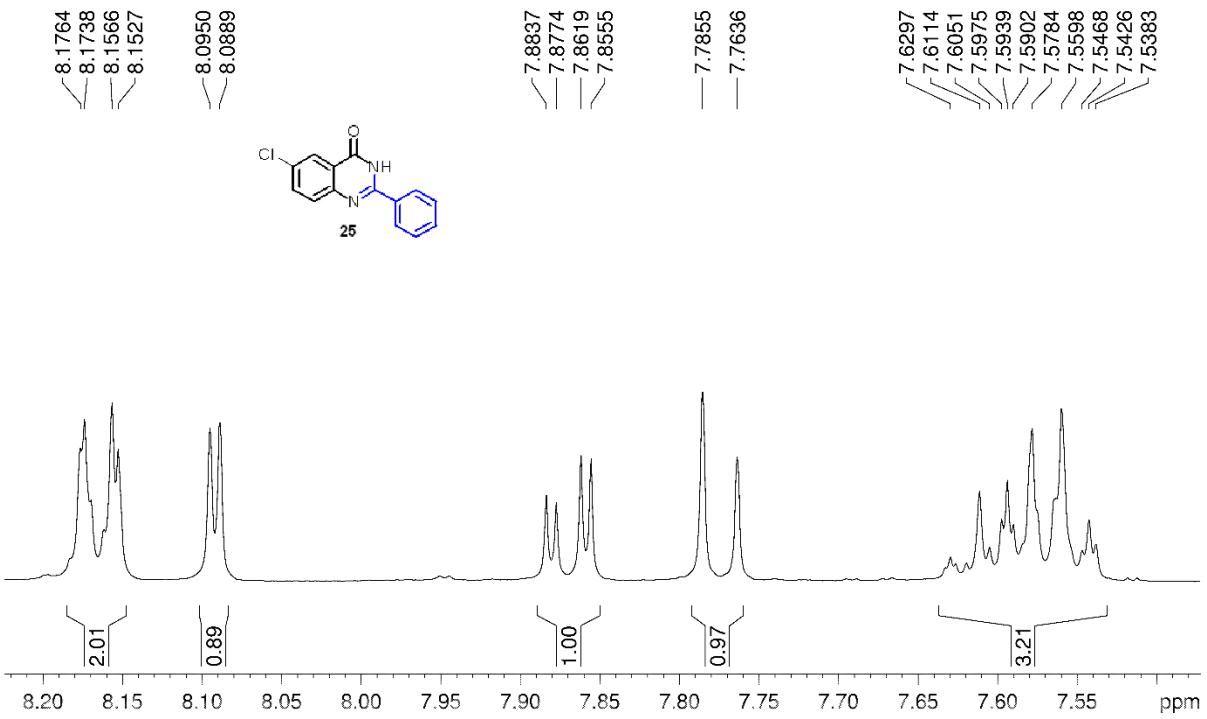
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7.6051
7.5975
7.5939
7.5902
7.5784
7.5598
7.5468
7.5426
7.5383
Cl
NH
N
O
25
2.01
0.89
1.00
0.97
3.21
8.20
8.15
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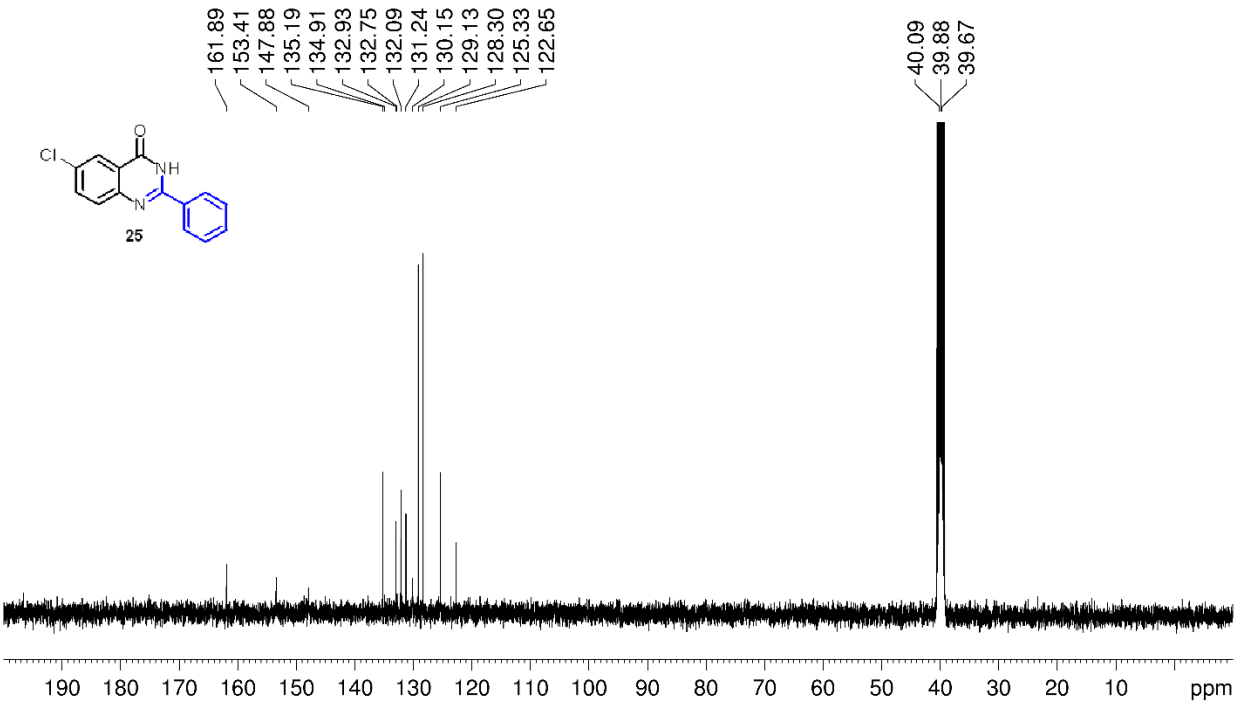
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125.33
122.65
40.09
39.88
39.67
Cl
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N
O
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150
140
130
120
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70
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30
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10
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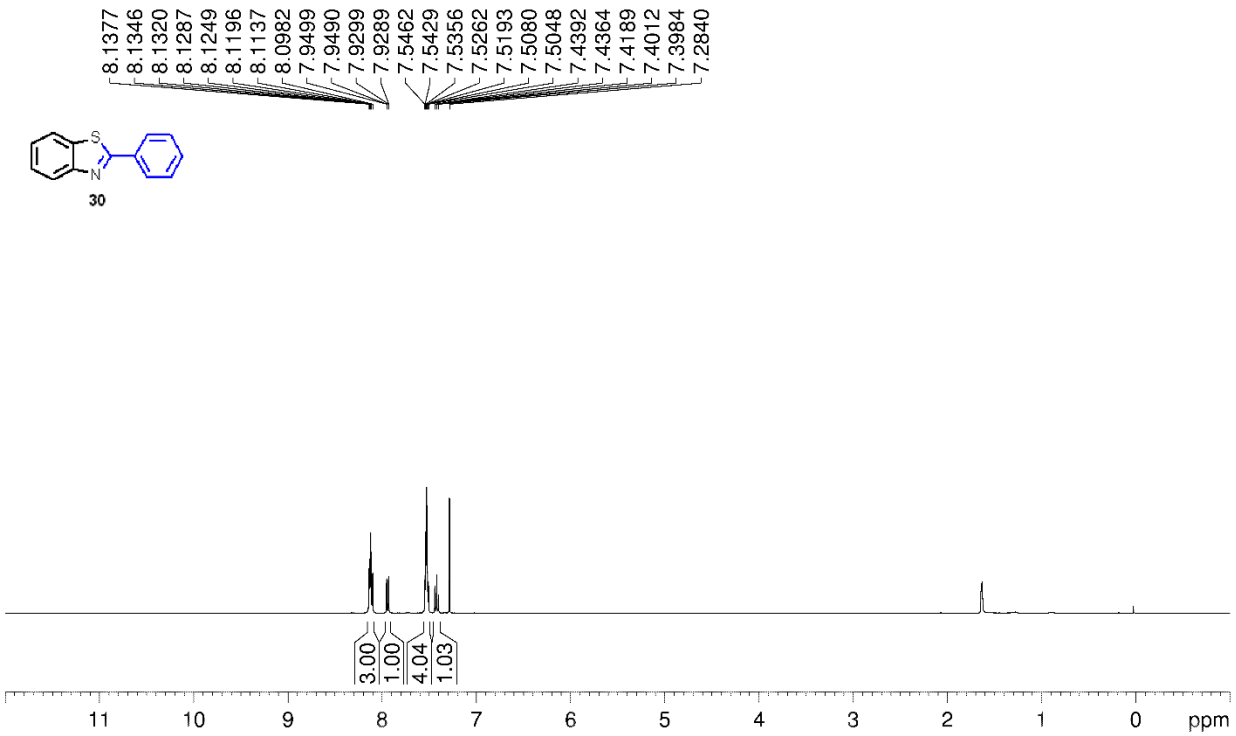
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8.1320
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8.1249
8.1196
8.1137
8.0982
7.9499
7.9490
7.9299
7.9289
7.5462
7.5429
7.5356
7.5262
7.5193
7.5080
7.5048
7.4392
7.4364
7.4189
7.4012
7.3984
7.2840
S
N
30
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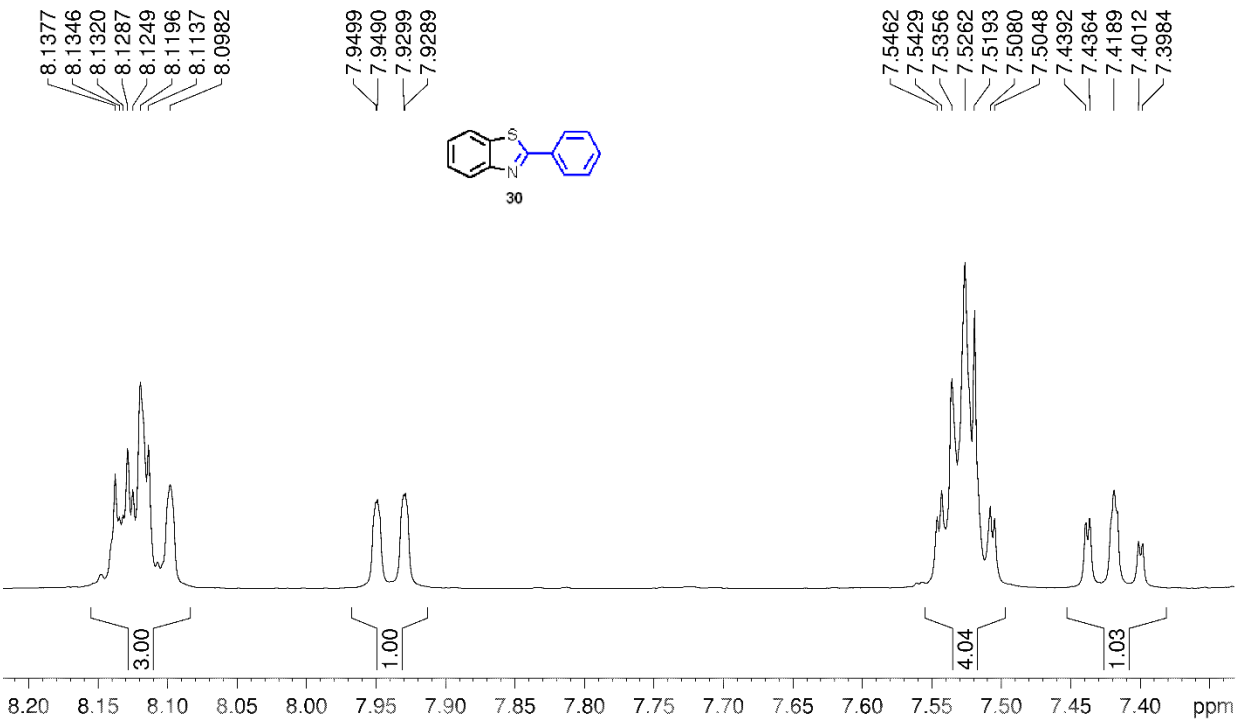
1.00
4.04
1.03
11
10
9
8
7
6
5
4
3
2
1
0
ppm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7.9299
7.9289
7.5462
7.5429
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7.5262
7.5193
7.5080
7.5048
7.4392
7.4364
7.4189
7.4012
7.3984
S
N
30
3.00
1.00
4.04
1.03
8.20
8.15
8.10
8.05
8.00
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7.75
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7.65
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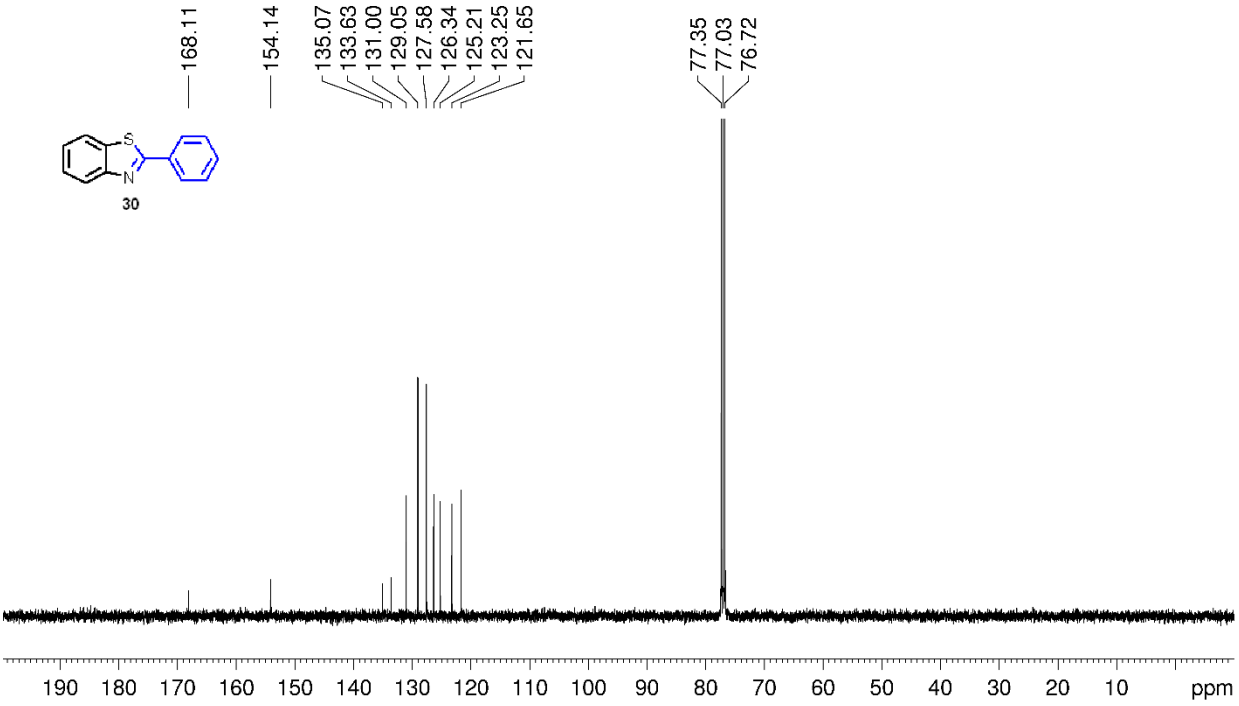
7.40
ppm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129.05
127.58
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121.65
77.35
77.03
76.72
30
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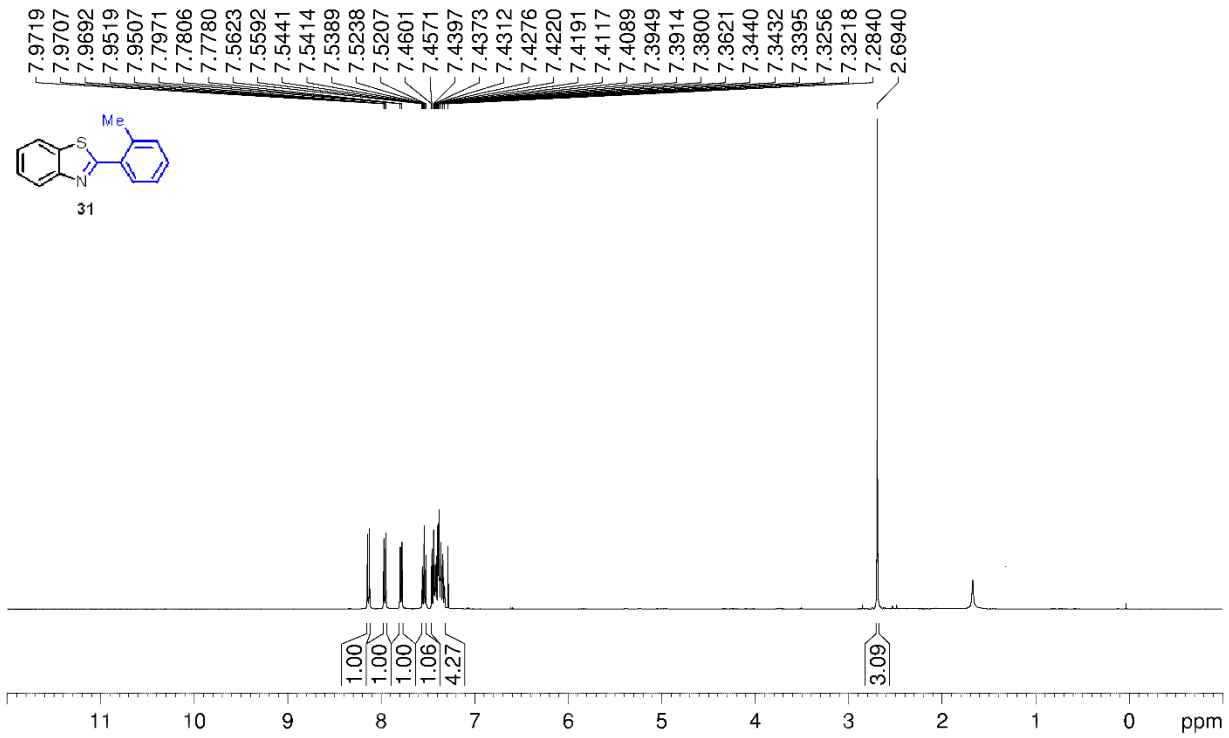
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7.5414
7.5389
7.5238
7.5207
7.4601
7.4571
7.4397
7.4373
7.4312
7.4276
7.4220
7.4191
7.4117
7.4089
7.3949
7.3914
7.3800
7.3621
7.3440
7.3432
7.3395
7.3256
7.3218
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4.27
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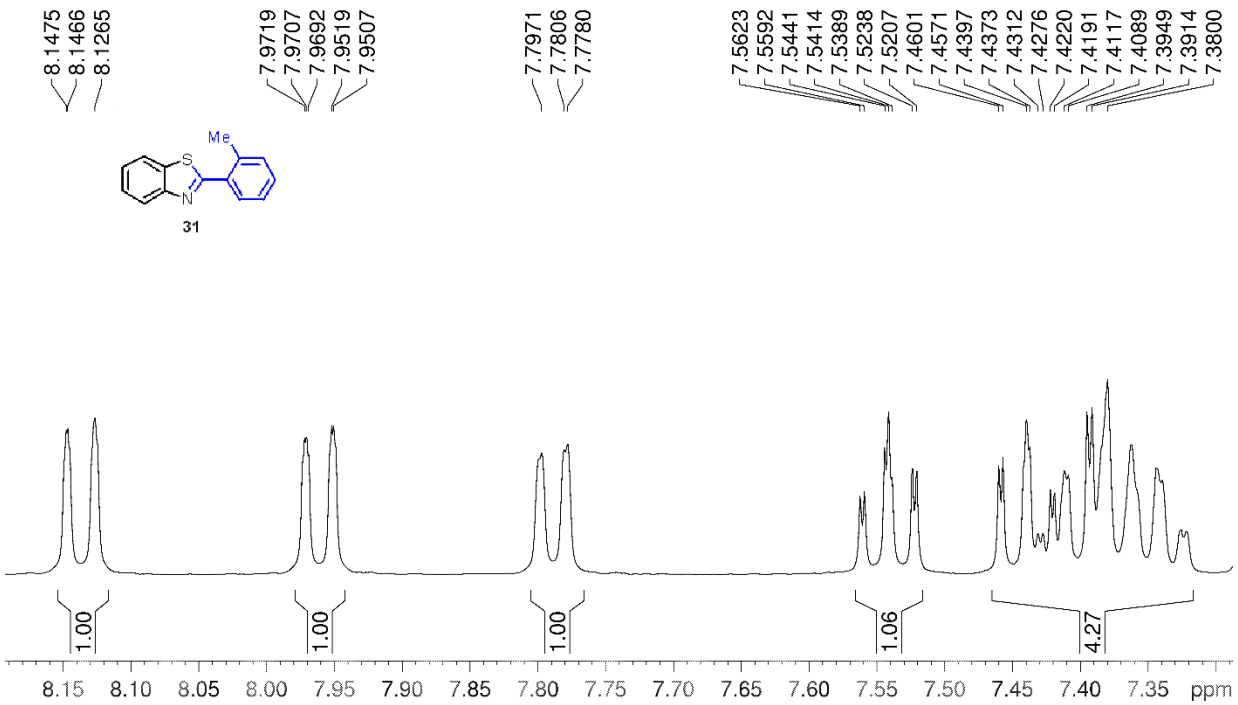
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7.5623
7.5592
7.5441
7.5414
7.5389
7.5238
7.5207
7.4601
7.4571
7.4397
7.4373
7.4312
7.4276
7.4220
7.4191
7.4117
7.4089
7.3949
7.3914
7.3800
Me
S
N
31
1.00
1.00
1.00
1.06
4.27
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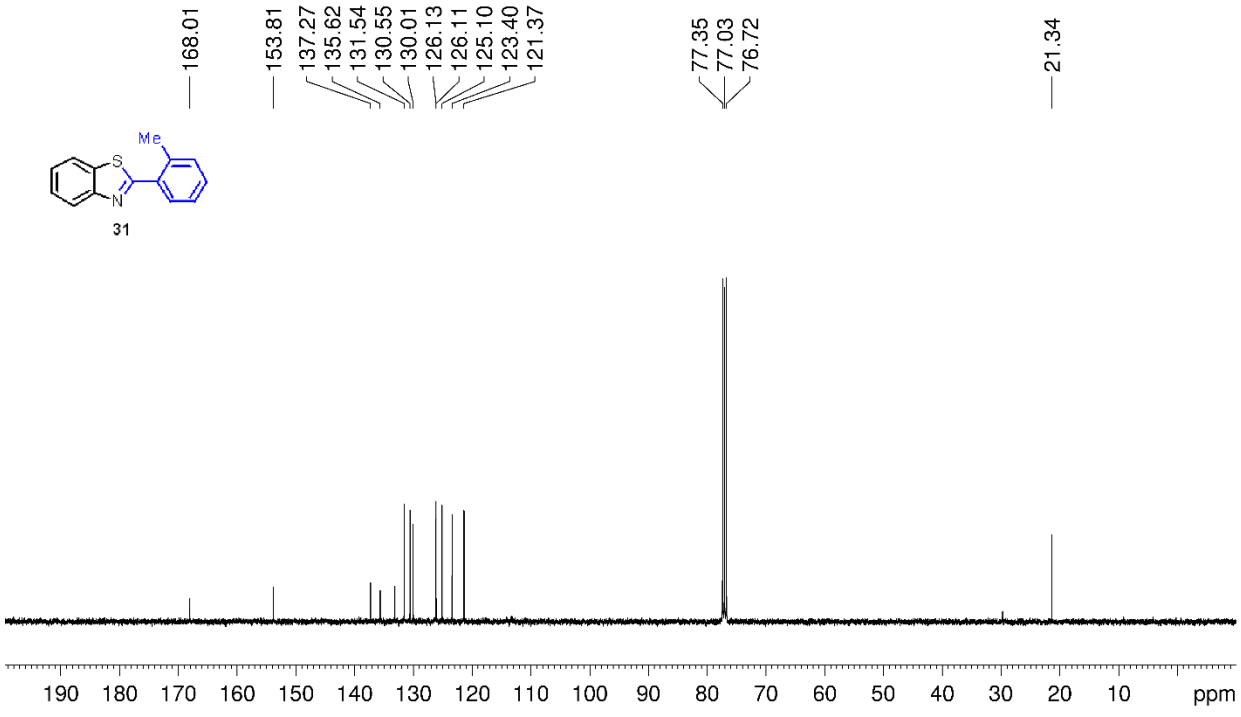
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126.13
126.11
125.10
123.40
121.37
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77.03
76.72
21.34
Me
S
N
31
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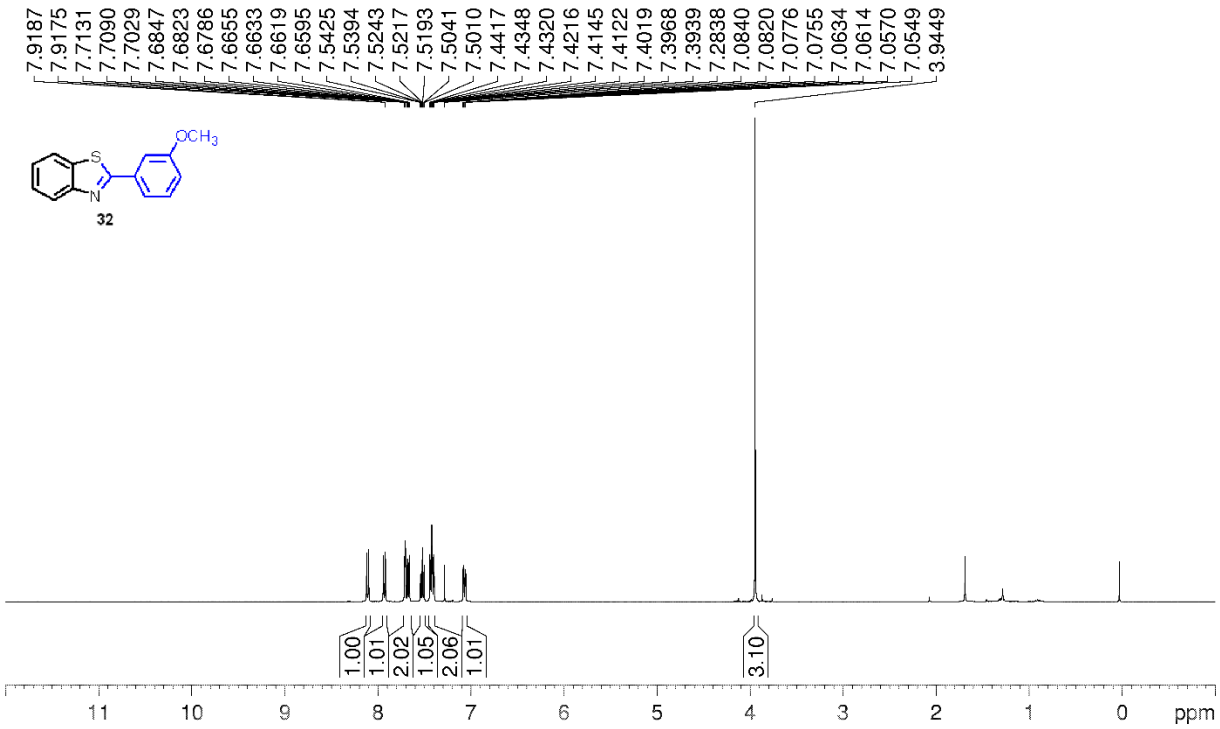
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7.3939
7.2838
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7.0820
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7.0614
7.0570
7.0549
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OCH3
S
N
32
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1.01
2.02
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10
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6
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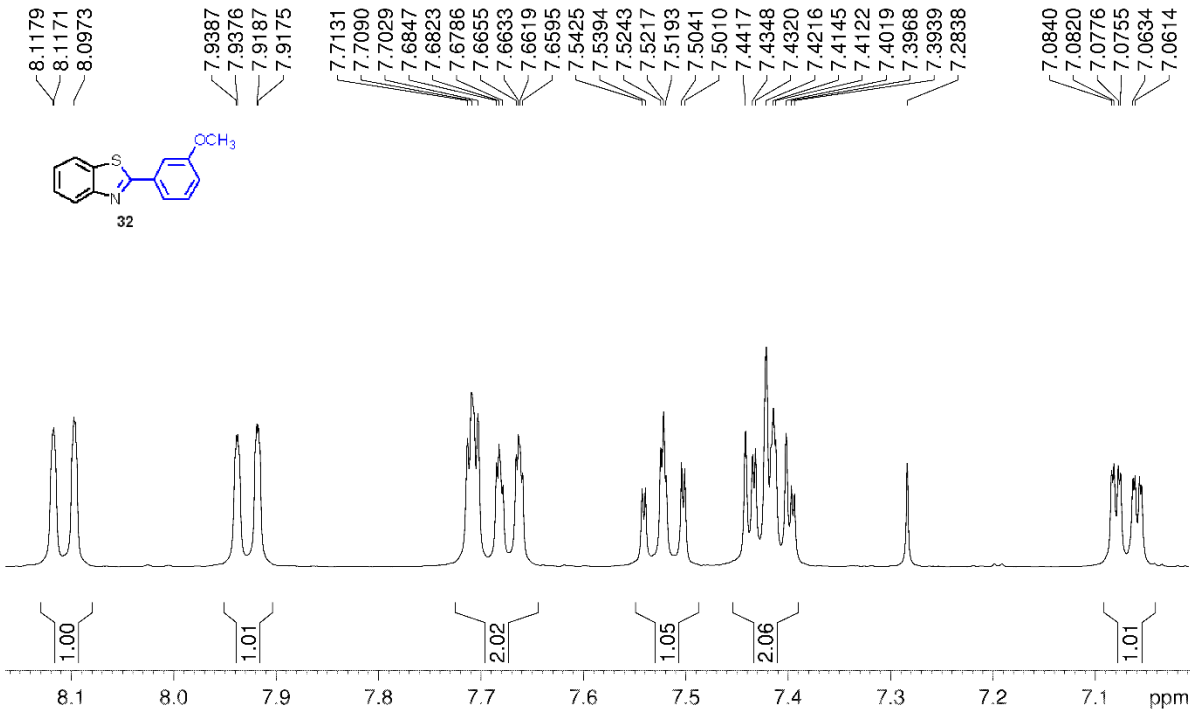
0
ppm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7.6847
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7.6619
7.6595
7.5425
7.5394
7.5243
7.5217
7.5193
7.5041
7.5010
7.4417
7.4348
7.4320
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7.3968
7.3939
7.2838
7.0840
7.0820
7.0776
7.0755
7.0634
7.0614
OCH3
S
N
32
1.00
1.01
2.02
1.05
2.06
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7.8
7.7
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7.4
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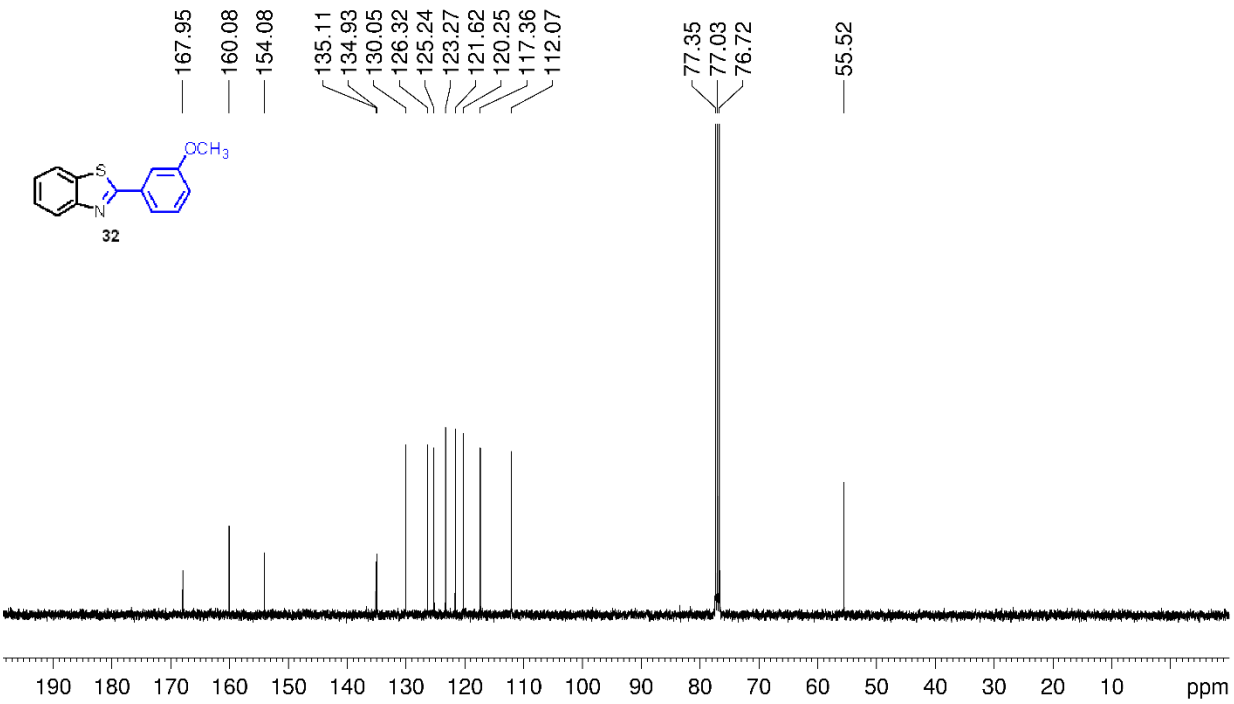
7.2
7.1
ppm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120.25
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76.72
55.52
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S
N
32
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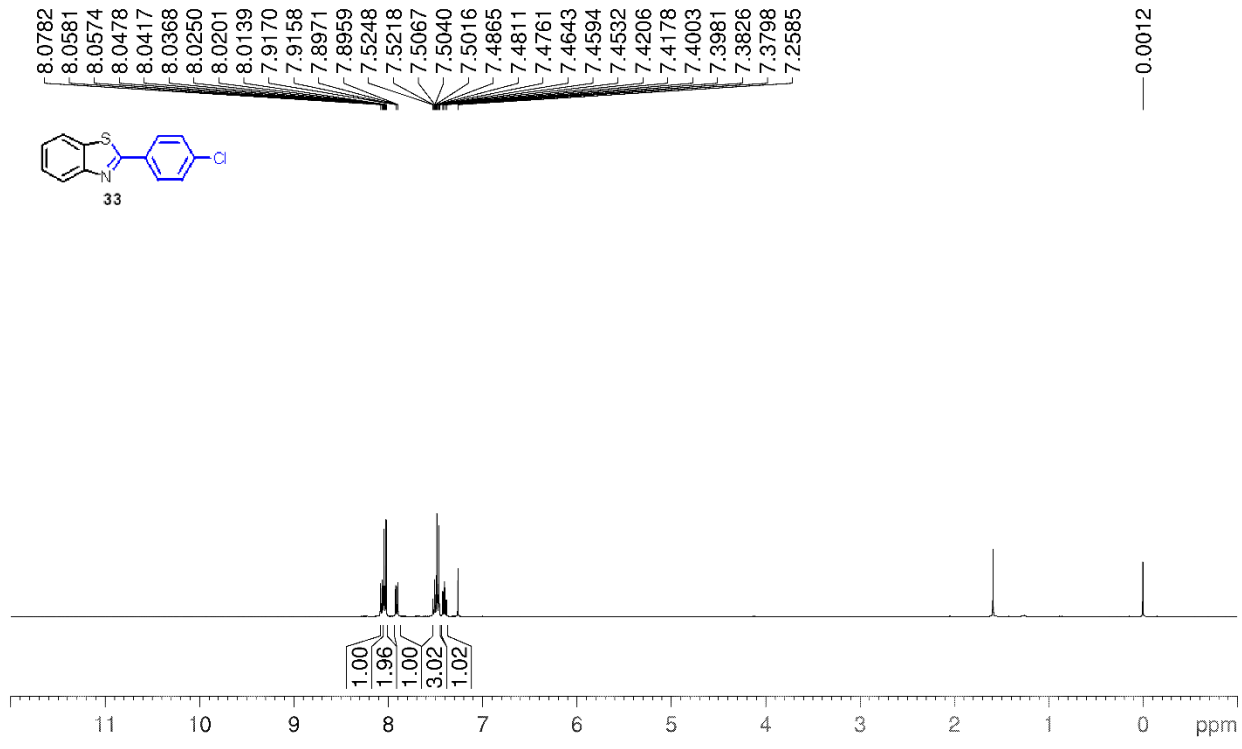
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7.4594
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7.4178
7.4003
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7.3798
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1.02
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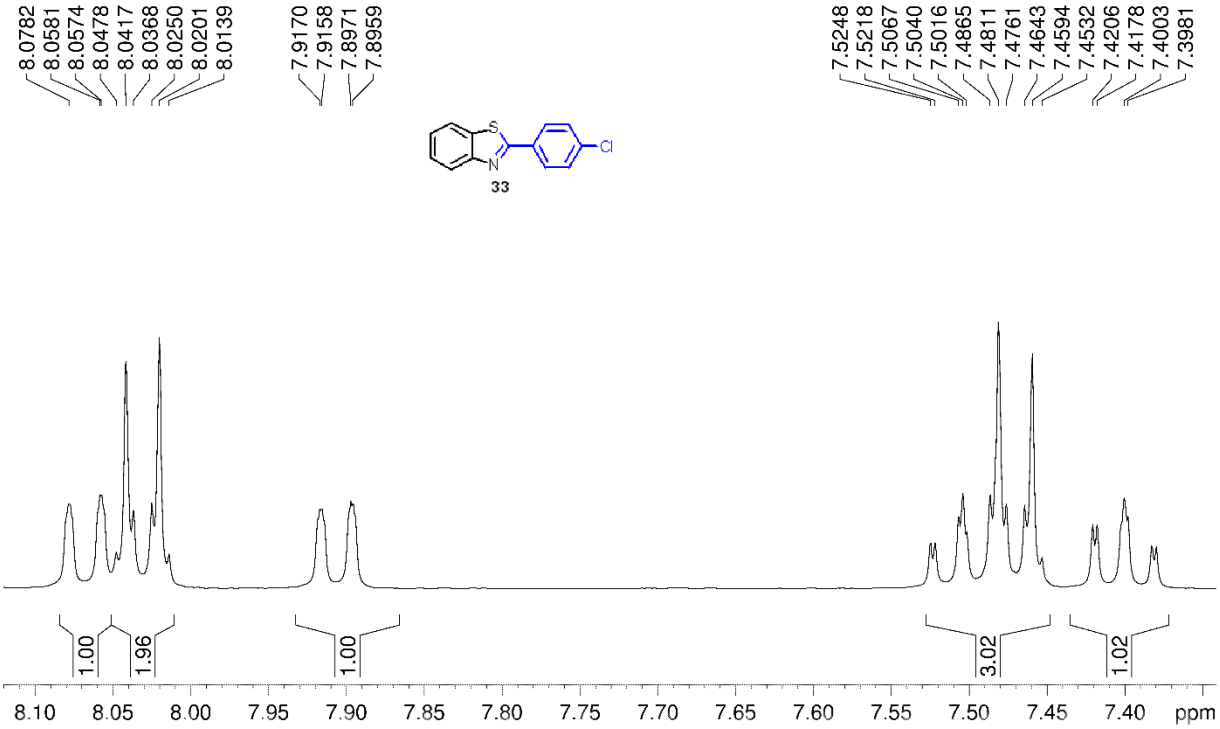
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S
N
Cl
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1.96
1.00
3.02
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8.10
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8.00
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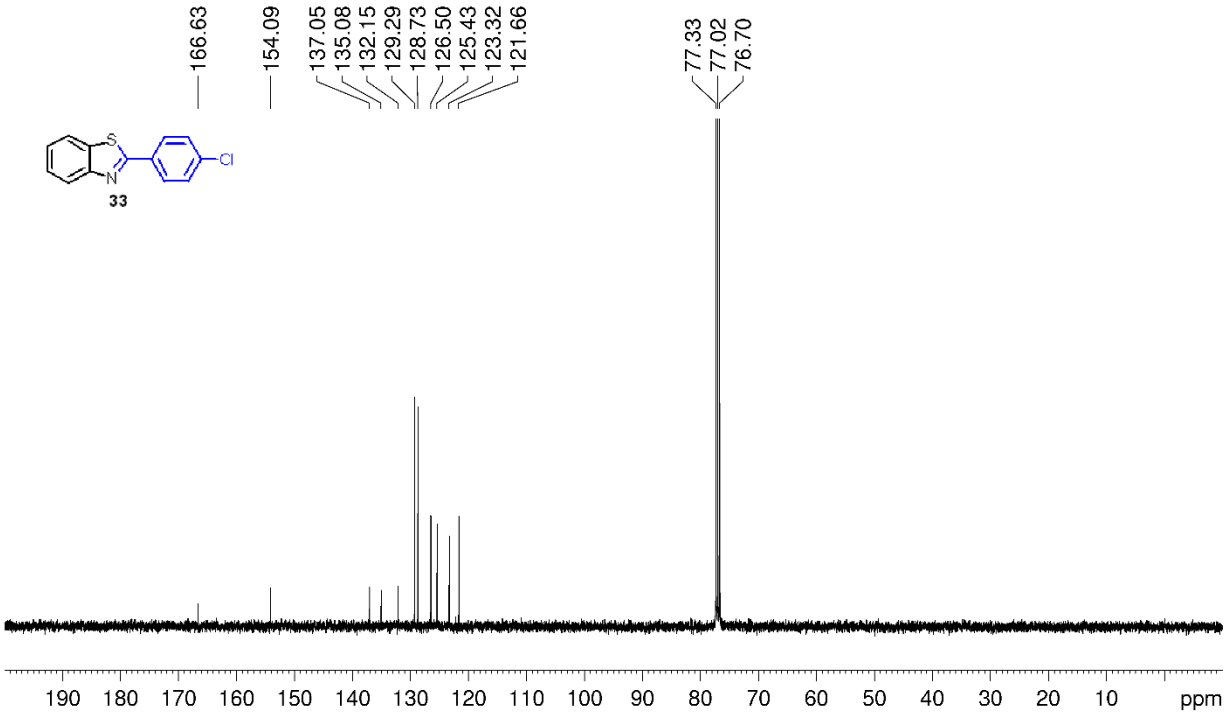
ppm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77.33
77.02
76.70
S
N
Cl
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30
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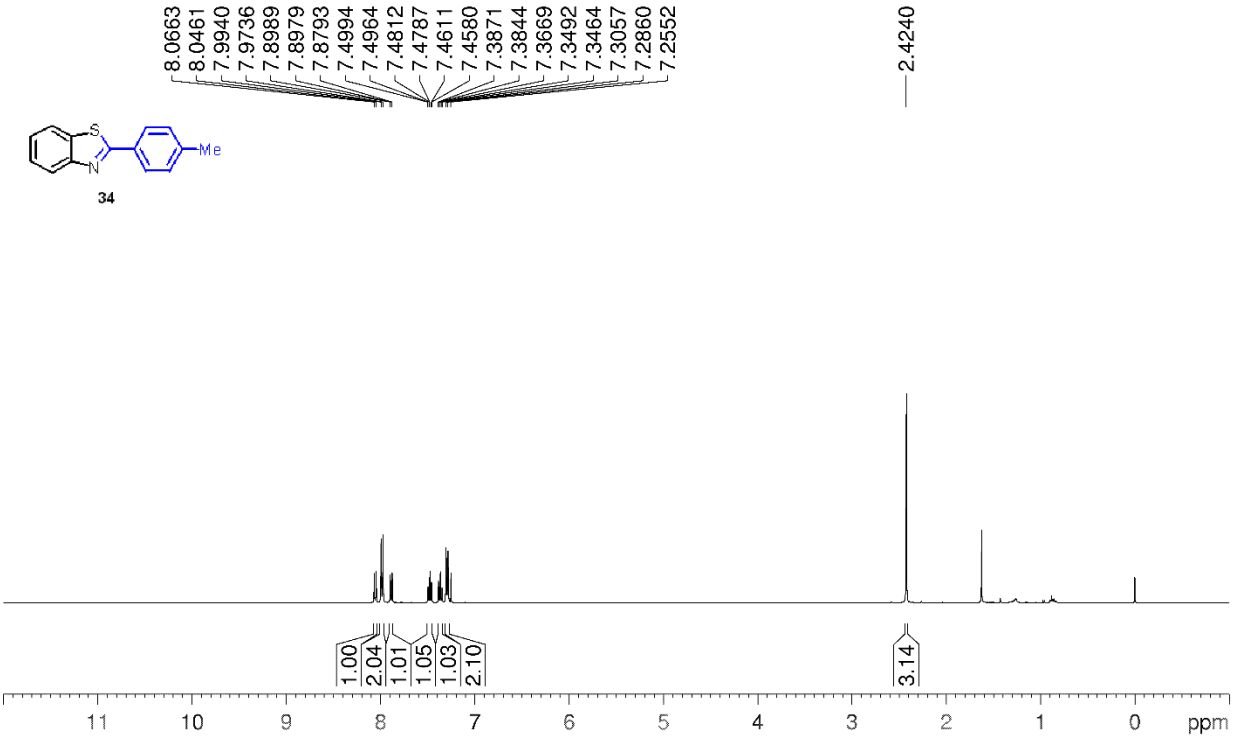
10
ppm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S
N
Me
34
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2.04
1.01
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6
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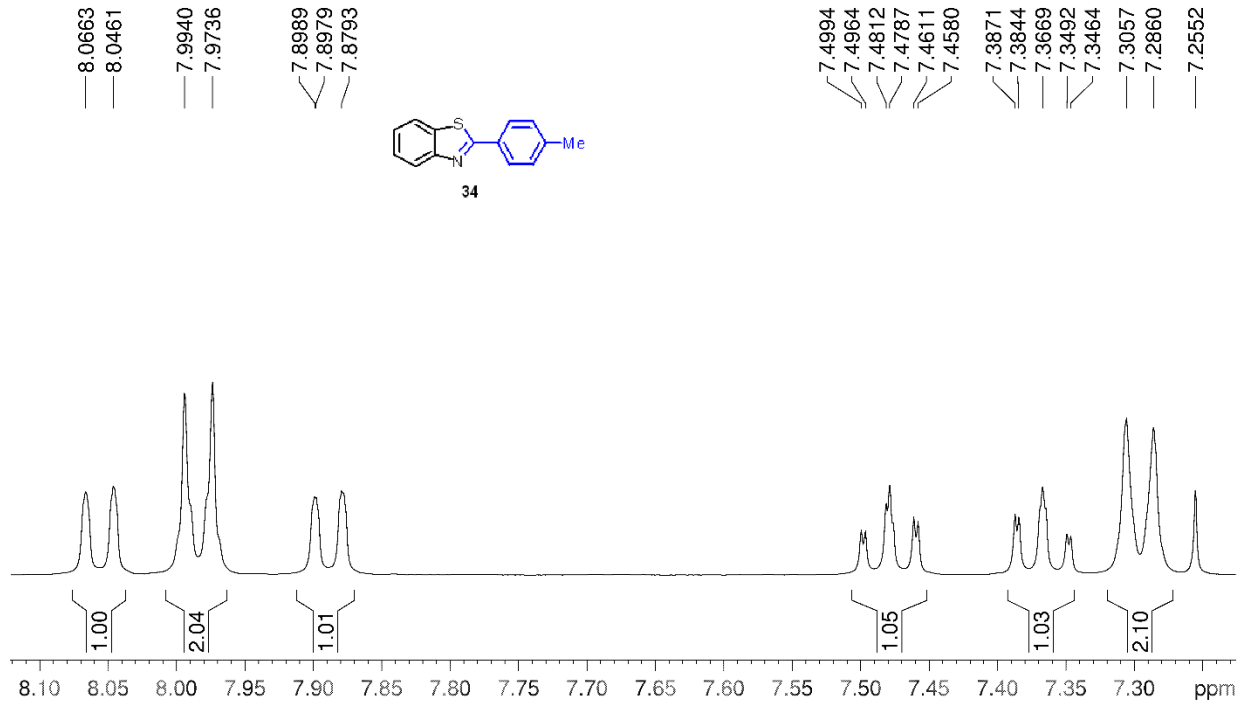
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S
N
Me
34
1.00
2.04
1.01
1.05
1.03
2.10
8.10
8.05
8.00
7.95
7.90
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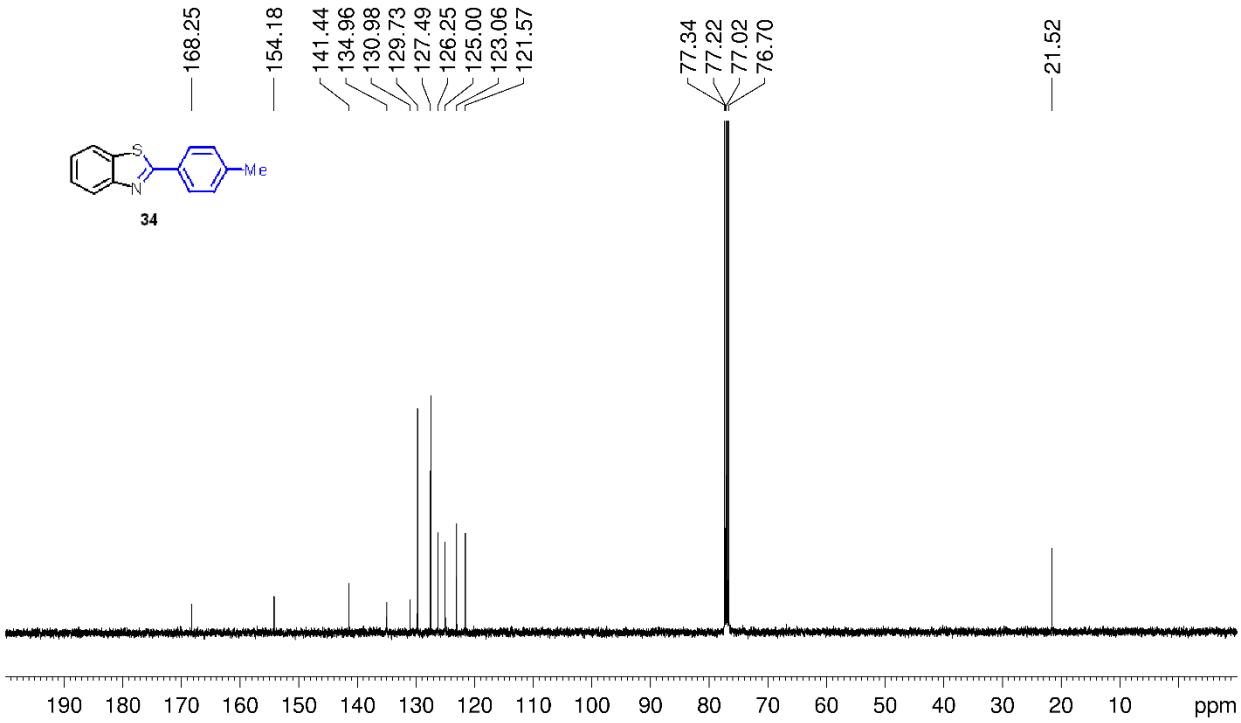
ppm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126.25
125.00
123.06
121.57
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77.02
76.70
21.52
S
N
Me
34
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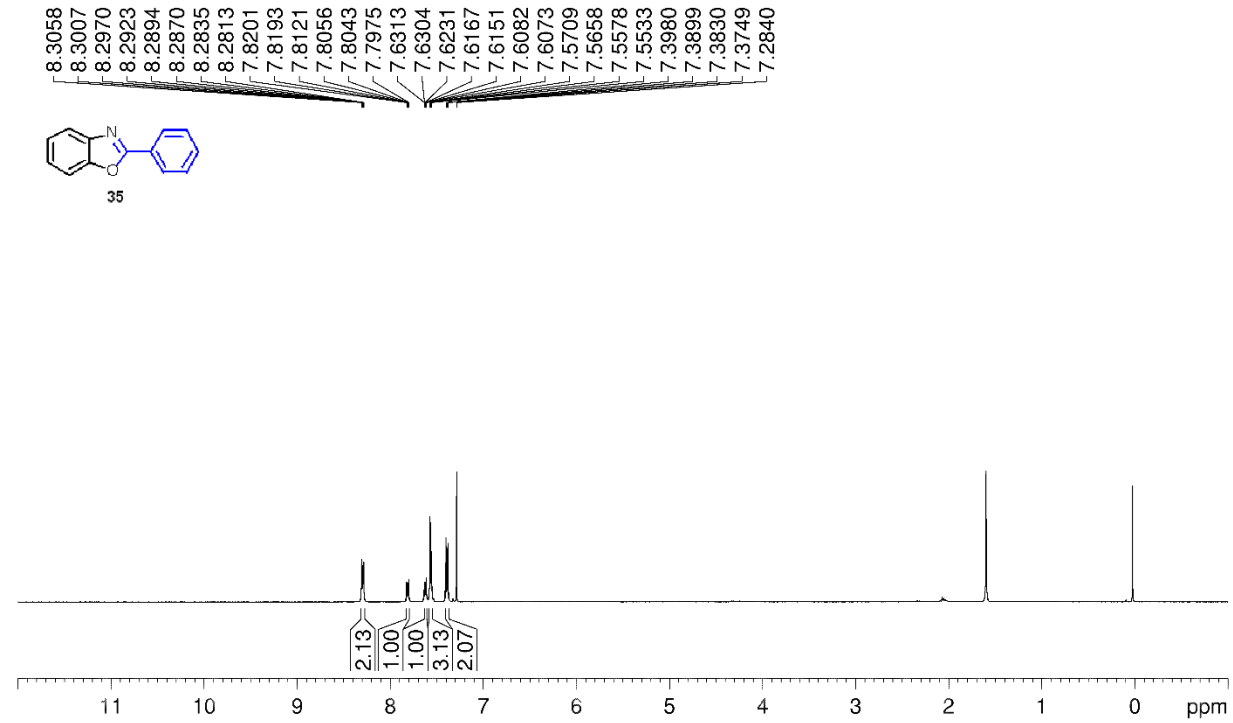
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O
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1.00
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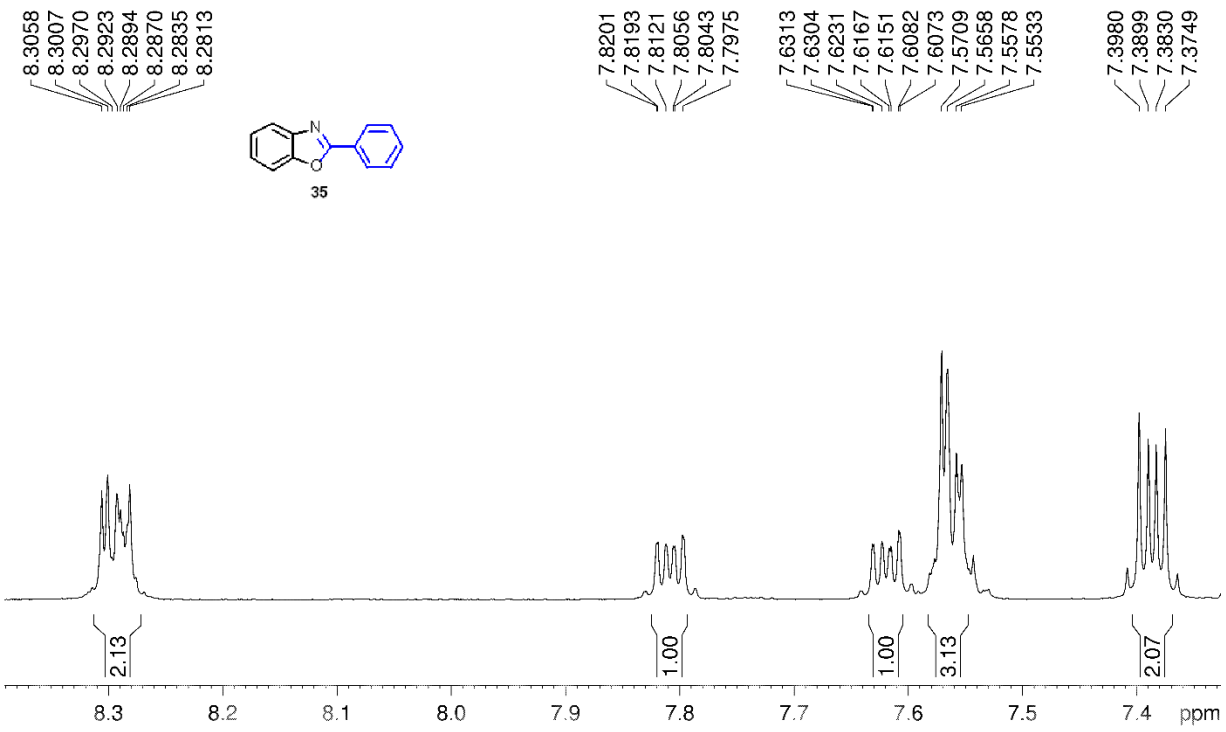
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7.3980
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7.3749
35
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1.00
1.00
3.13
2.07
8.3
8.2
8.1
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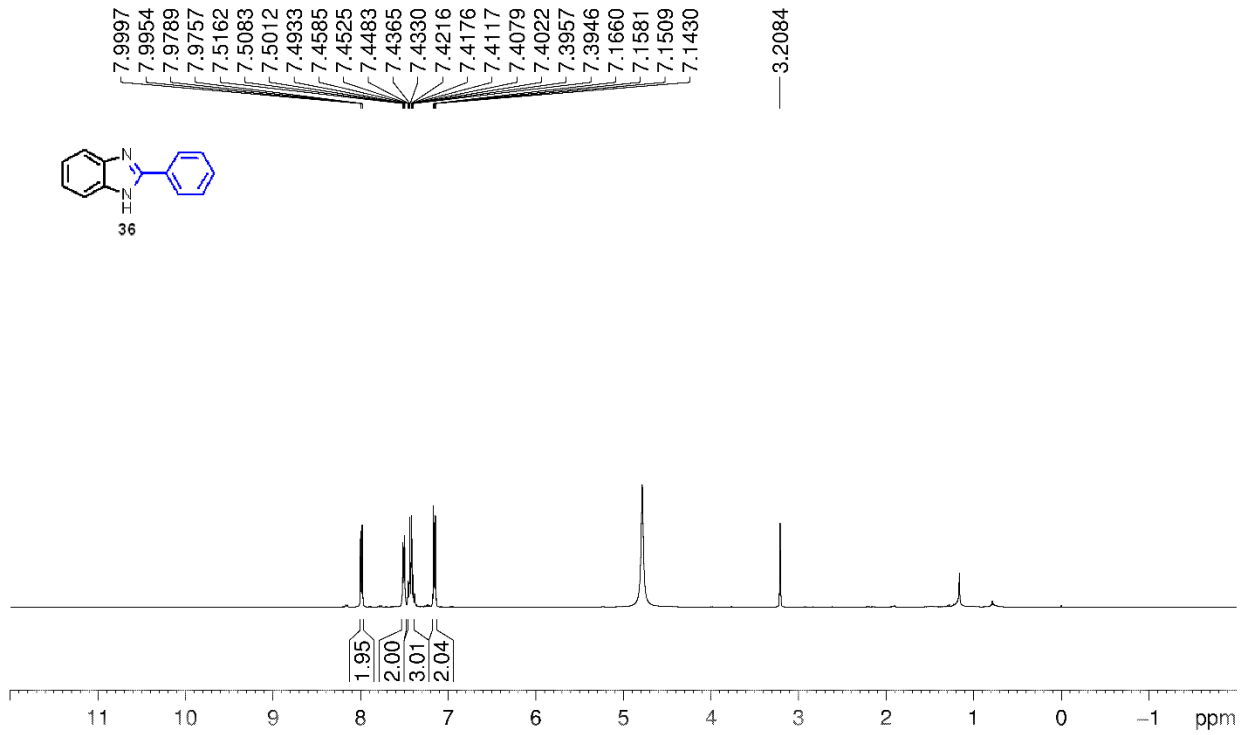
7.8
7.7
7.6
7.5
7.4
ppm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7.5012
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7.4483
7.4365
7.4330
7.4216
7.4176
7.4117
7.4079
7.4022
7.3957
7.3946
7.1660
7.1581
7.1509
7.1430
3.2084
36
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2.04
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4
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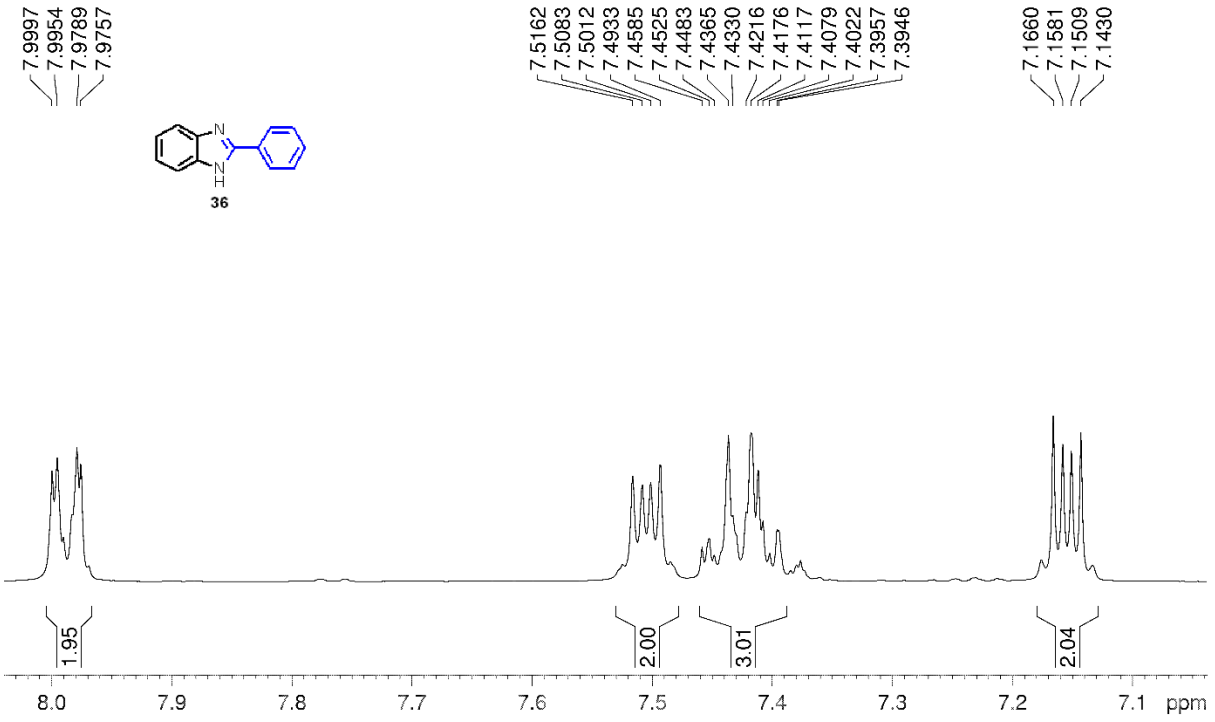
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7.4022
7.3957
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7.1581
7.1509
7.1430
36
1.95
2.00
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7.5
7.4
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7.2
7.1
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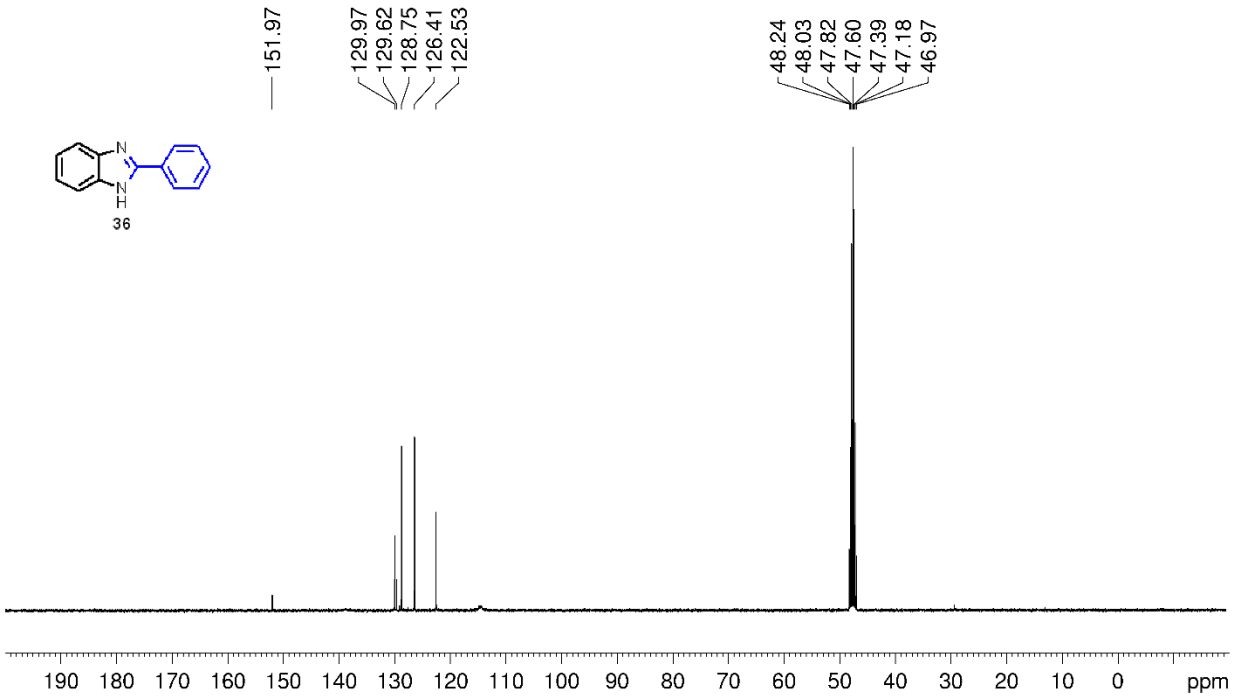
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48.03
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47.60
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47.18
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36
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140
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60
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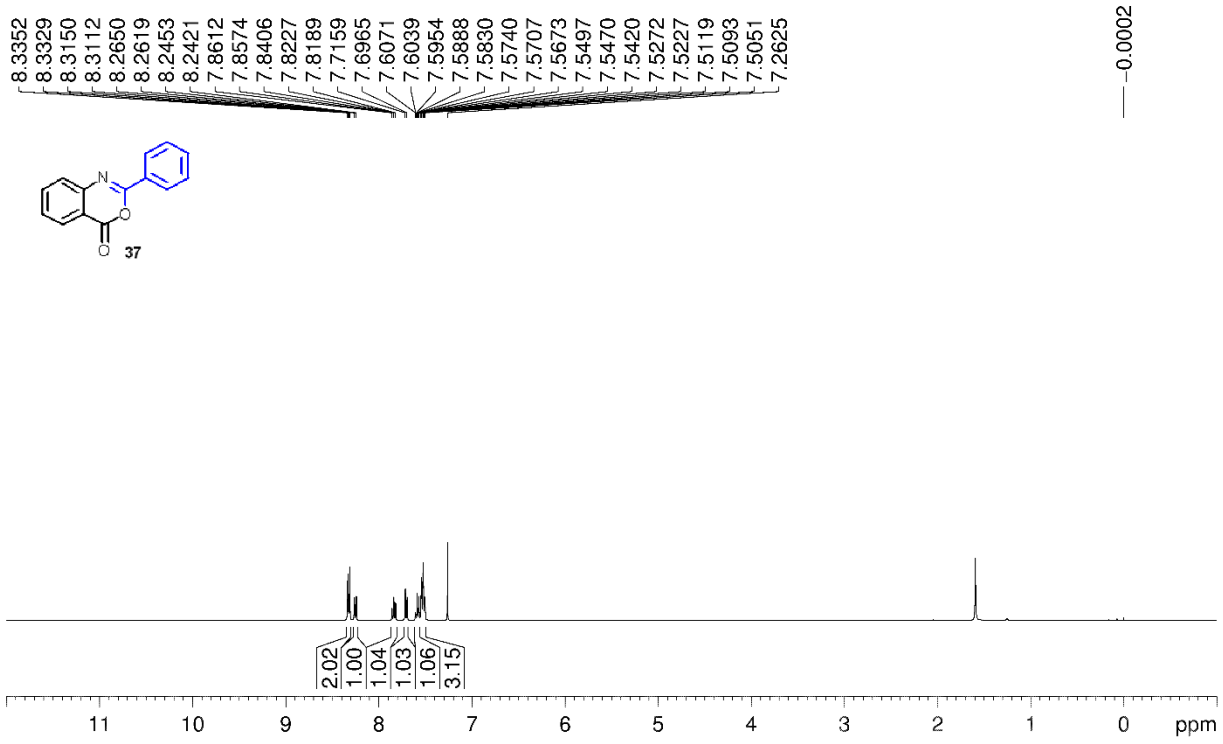
ppm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8.2650
8.2619
8.2453
8.2421
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7.8406
7.8227
7.8189
7.7159
7.6965
7.6071
7.6039
7.5954
7.5888
7.5830
7.5740
7.5707
7.5673
7.5497
7.5470
7.5420
7.5272
7.5227
7.5119
7.5093
7.5051
7.2625
0.0002
37
2.02
1.00
1.04
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1.06
3.15
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10
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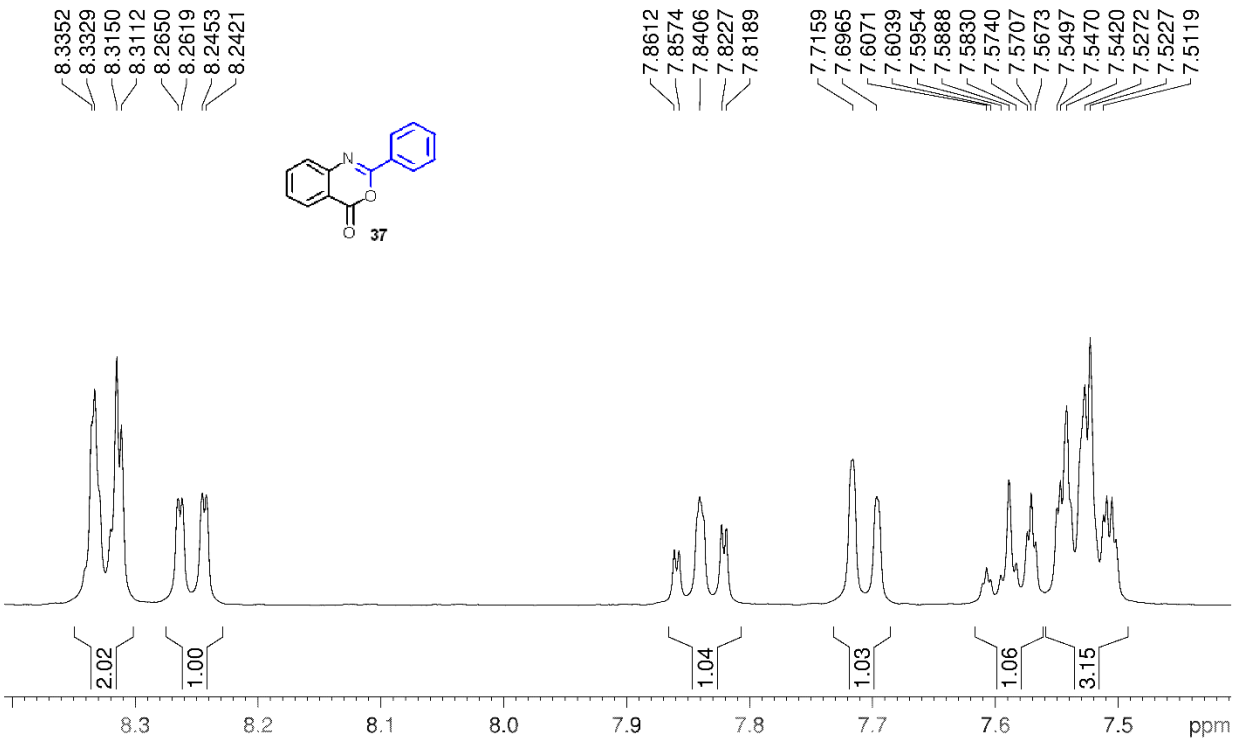
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7.5420
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7.5227
7.5119
37
2.02
1.00
1.04
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8.2
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7.8
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7.6
7.5
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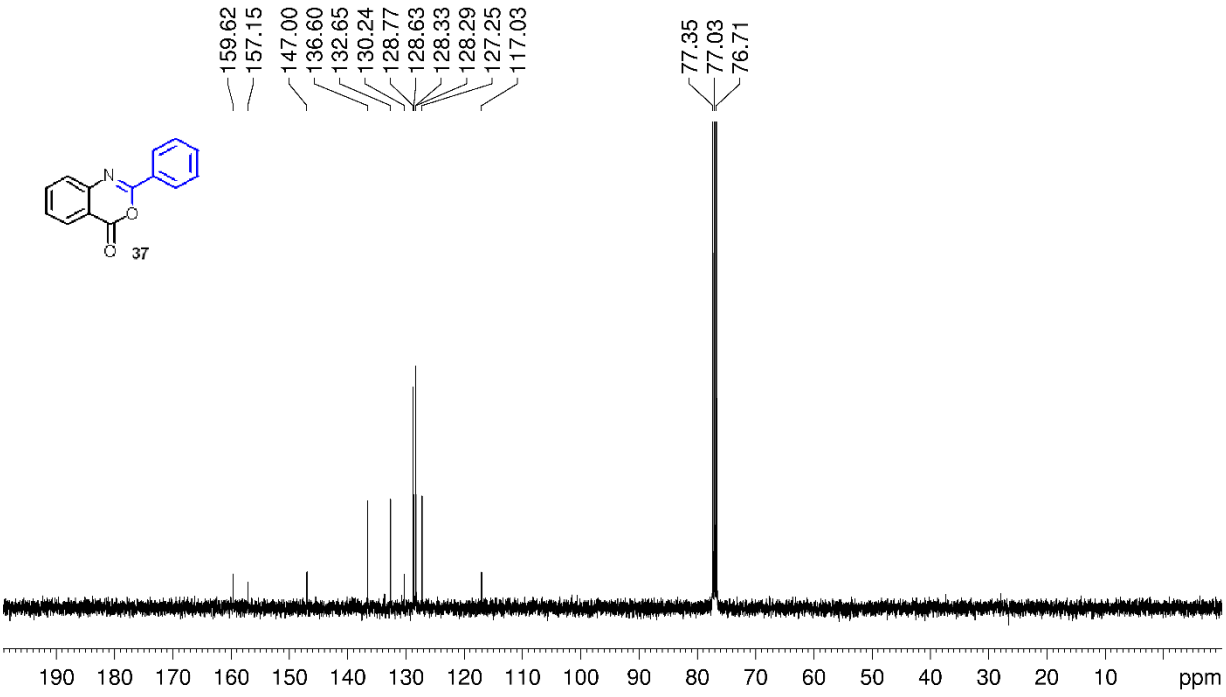
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157.15
147.00
136.60
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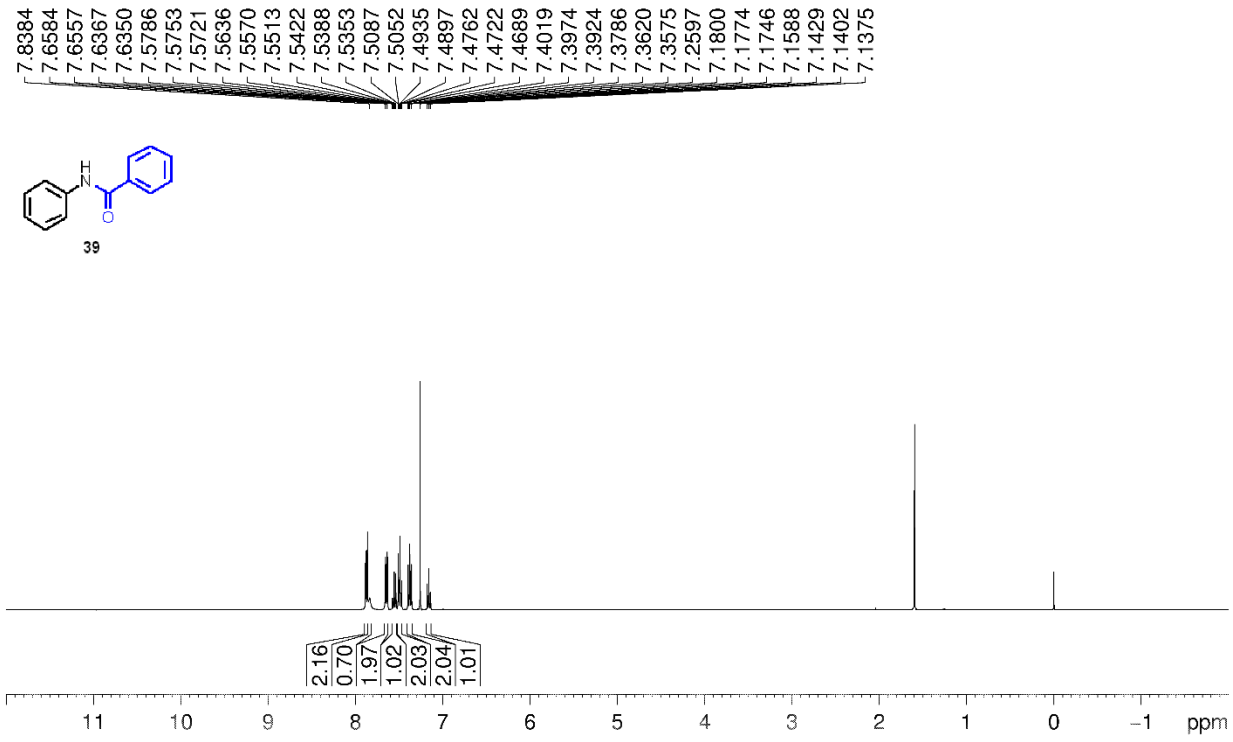
128.77
128.63
128.33
128.29
127.25
117.03
77.35
77.03
76.71
37
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.6557
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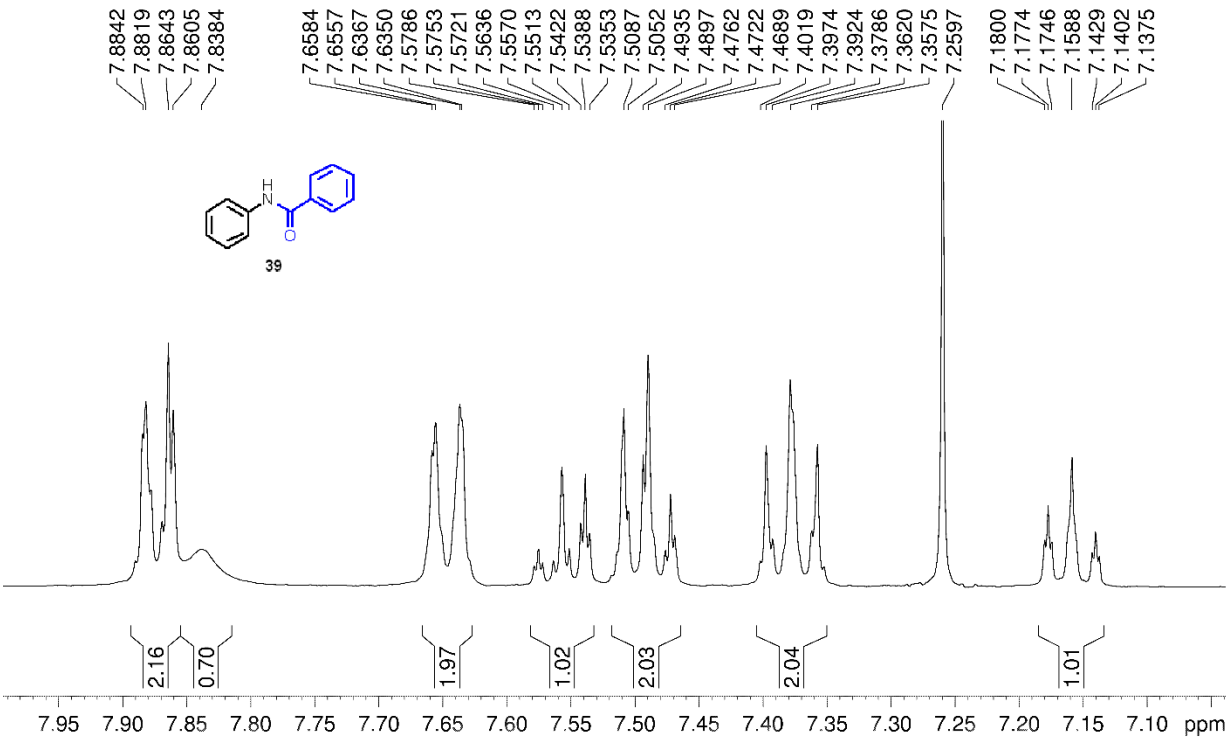
7.6350
7.5786
7.5753
7.5721
7.5636
7.5570
7.5513
7.5422
7.5388
7.5353
7.5087
7.5052
7.4935
7.4897
7.4762
7.4722
7.4689
7.4019
7.3974
7.3924
7.3786
7.3620
7.3575
7.2597
7.1800
7.1774
7.1746
7.1588
7.1429
7.1402
7.1375
39
2.16
0.70
1.97
1.02
2.03
2.04
1.01
11 10 9 8 7 6 5 4 3 2 1 0 −1 ppm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7.6557
7.6367
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7.5753
7.5721
7.5636
7.5570
7.5513
7.5422
7.5388
7.5353
7.5087
7.5052
7.4935
7.4897
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7.4722
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7.3924
7.3786
7.3620
7.3575
7.2597
7.1800
7.1774
7.1746
7.1588
7.1429
7.1402
7.1375
39
2.16
0.70
1.97
1.02
2.03
2.04
1.01
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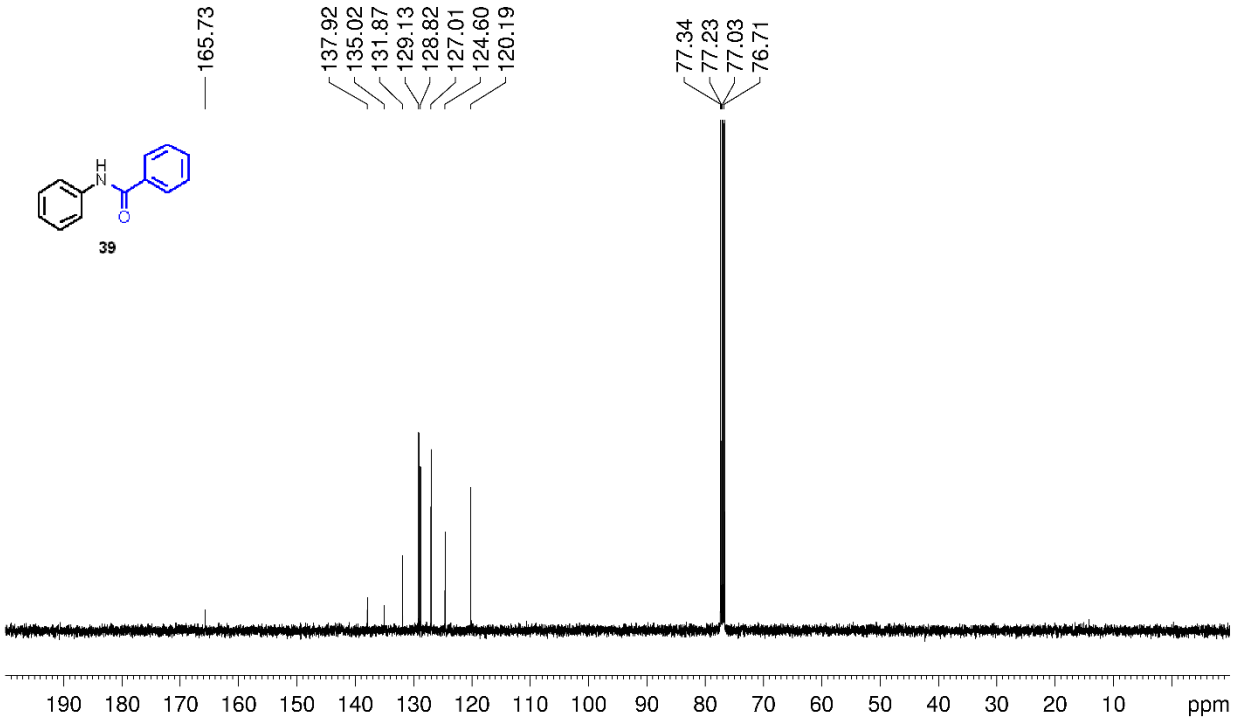
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131.87
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128.82
127.01
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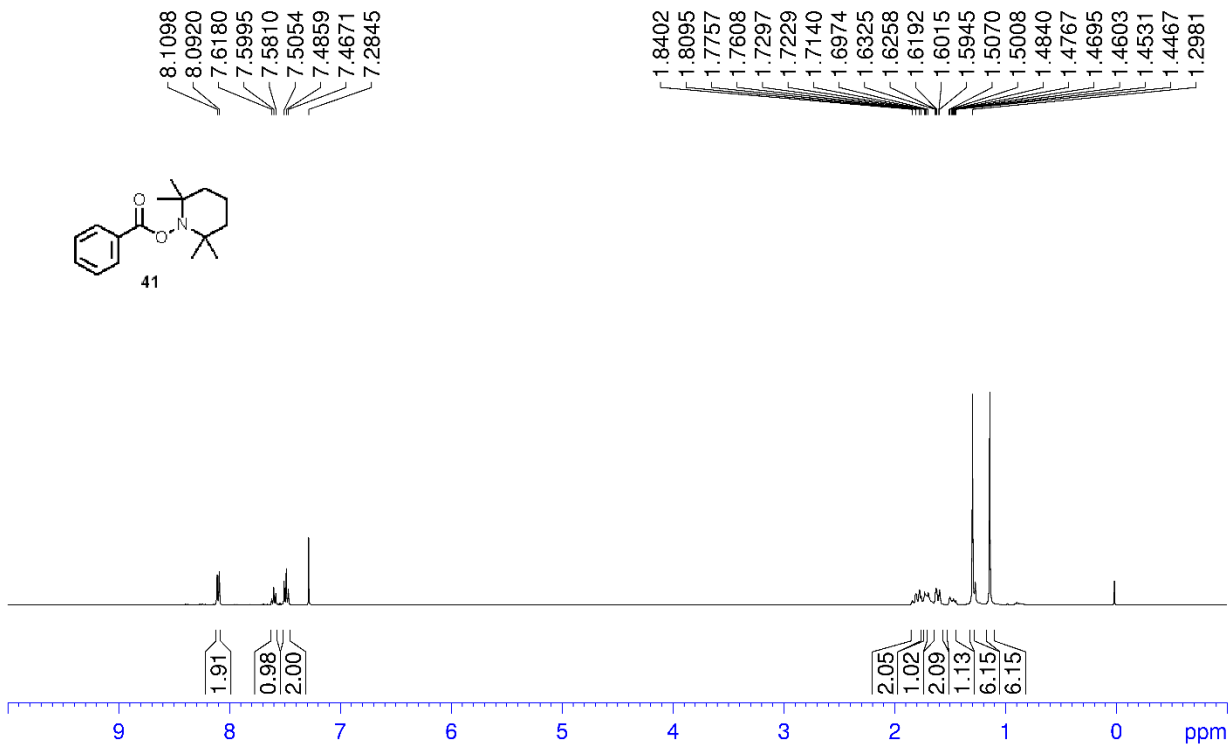
120.19
77.34
77.23
77.03
76.71
39
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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1.8095
1.7757
1.7608
1.7297
1.7229
1.7140
1.6974
1.6325
1.6258
1.6192
1.6015
1.5945
1.5070
1.5008
1.4840
1.4767
1.4695
1.4603
1.4531
1.4467
1.2981
41
1.91
0.98
2.00
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1.13
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6.15
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8
7
6
5
4
3
2
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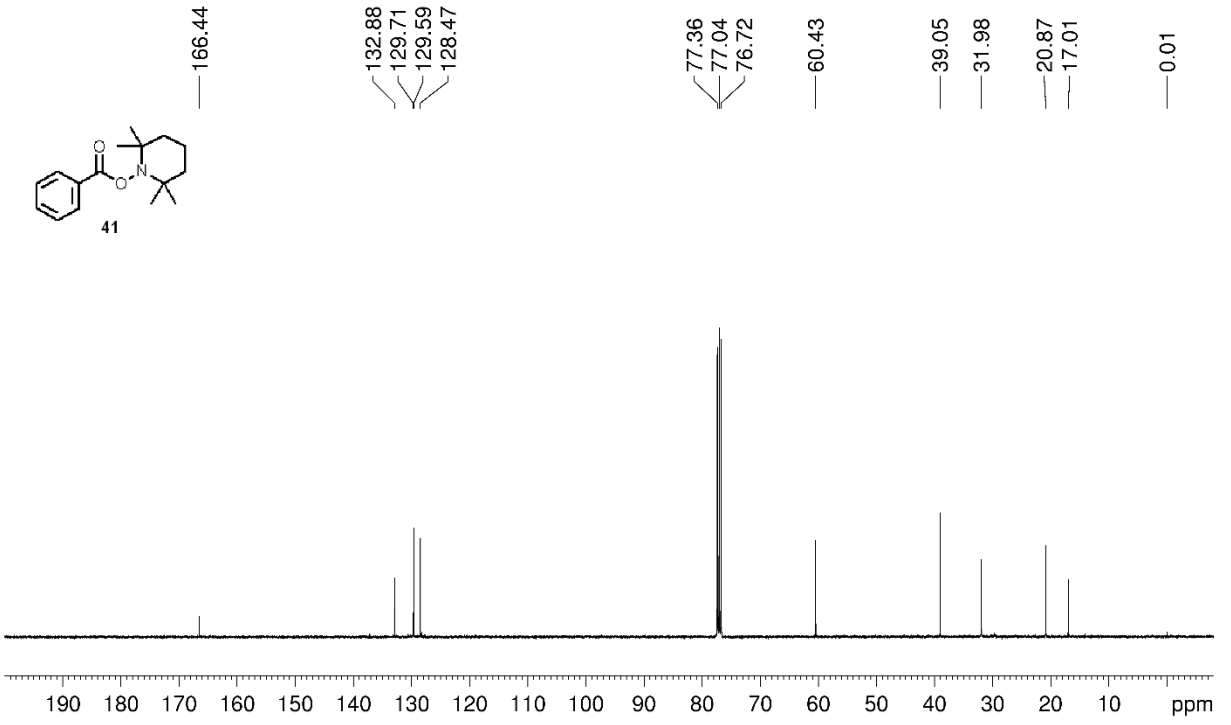
0
ppm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129.71
129.59
128.47
77.36
77.04
76.72
60.43
39.05
31.98
20.87
17.01
0.01
41
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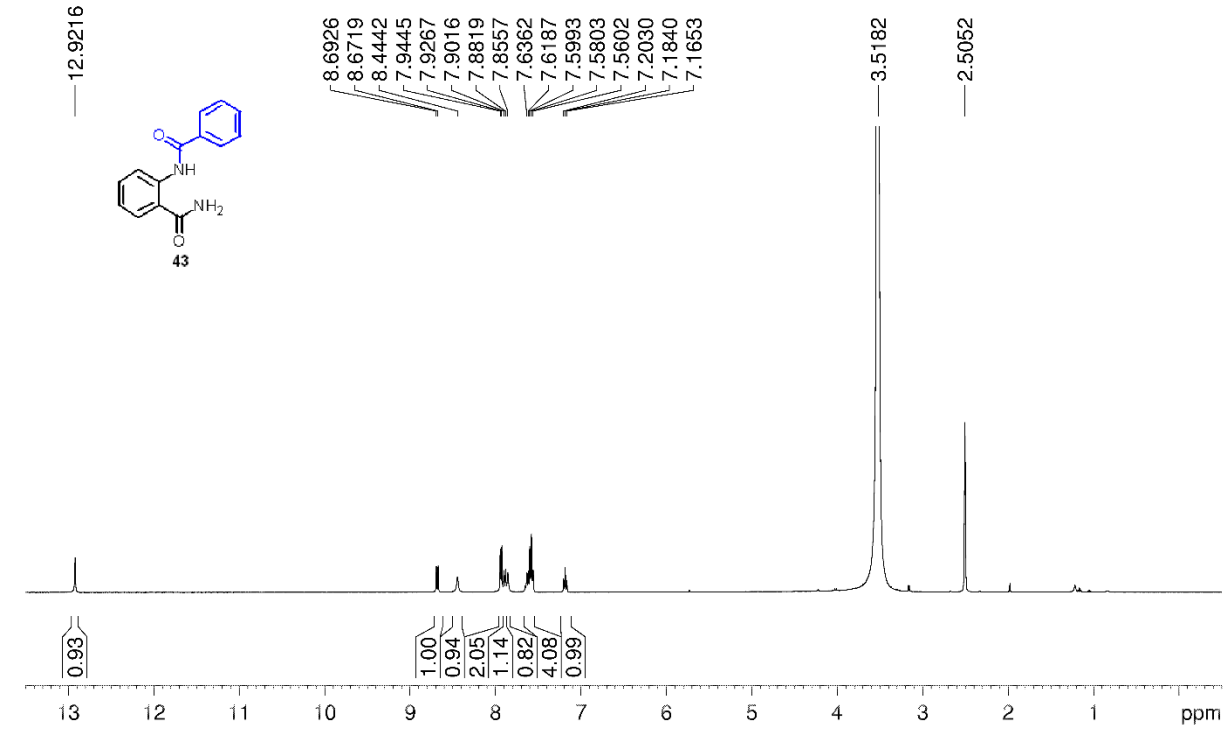
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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8.6719
8.4442
7.9445
7.9267
7.9016
7.8819
7.8557
7.6362
7.6187
7.5993
7.5803
7.5602
7.2030
7.1840
7.1653
3.5182
2.5052
NH
NH2
O
43
0.93
1.00
0.94
2.05
1.14
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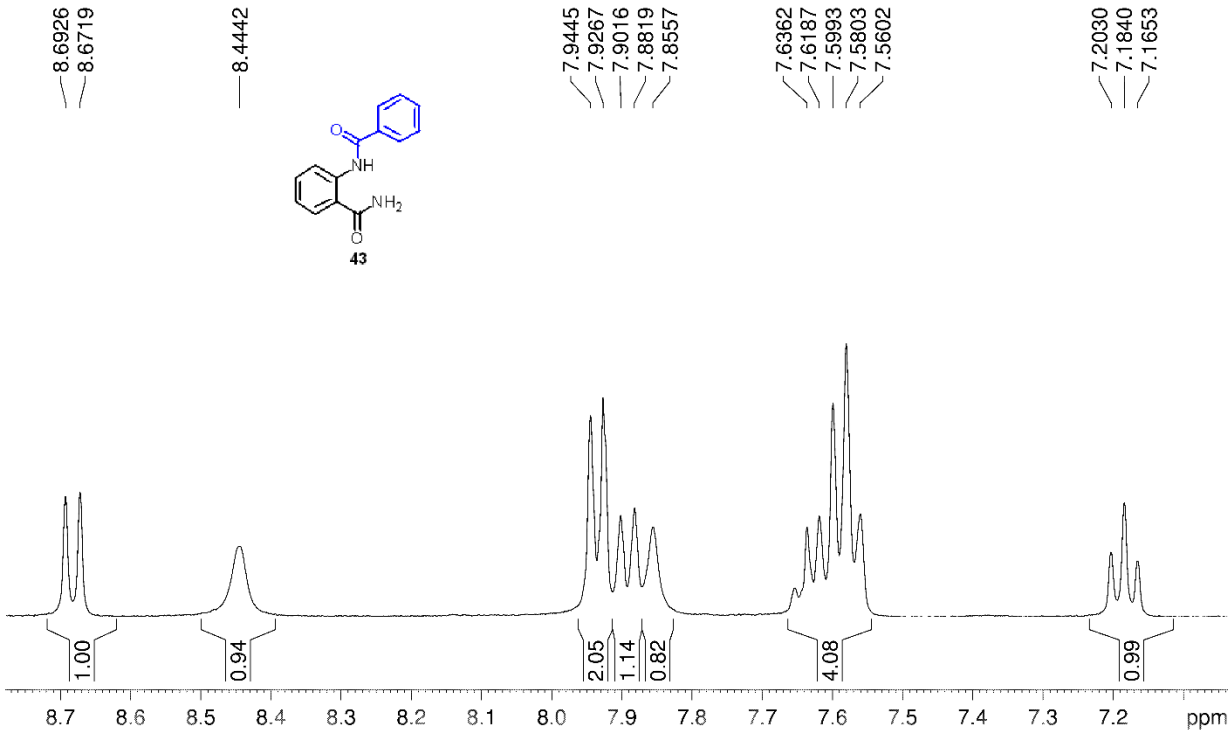
4.08
0.99
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm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8.6719
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7.8819
7.8557
7.6362
7.6187
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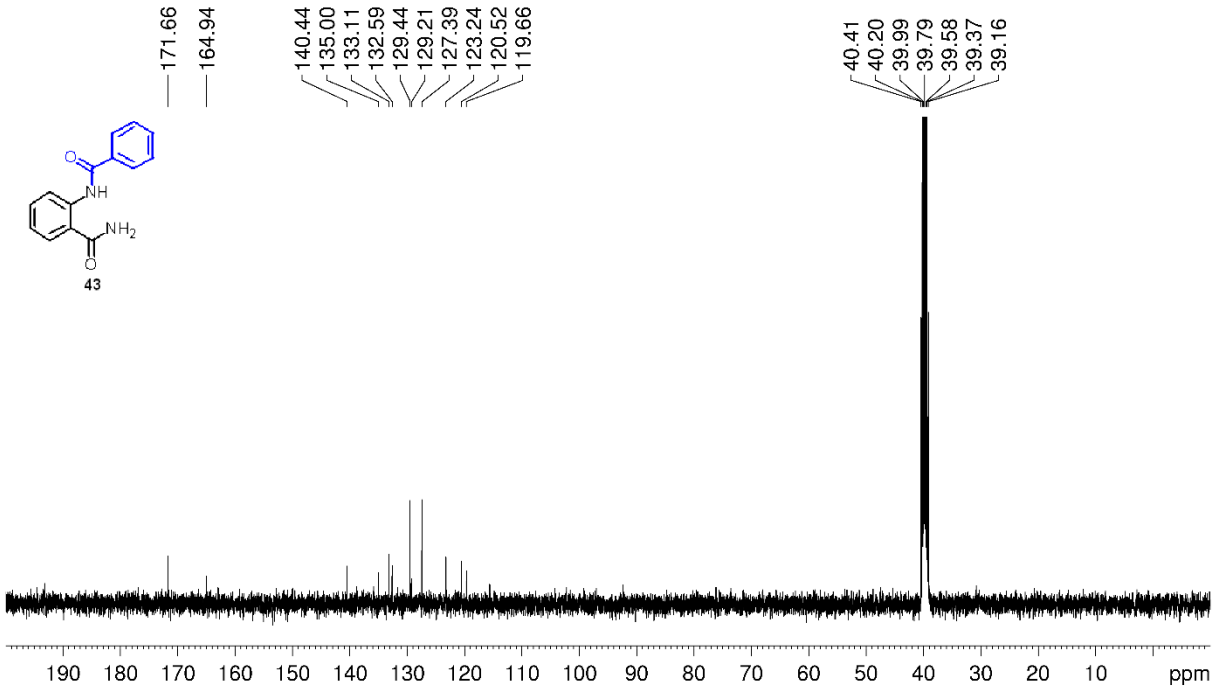
7.5803
7.5602
7.2030
7.1840
7.1653
NH
NH2
O
43
1.00
0.94
2.05
1.14
0.82
4.08
0.99
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
ppm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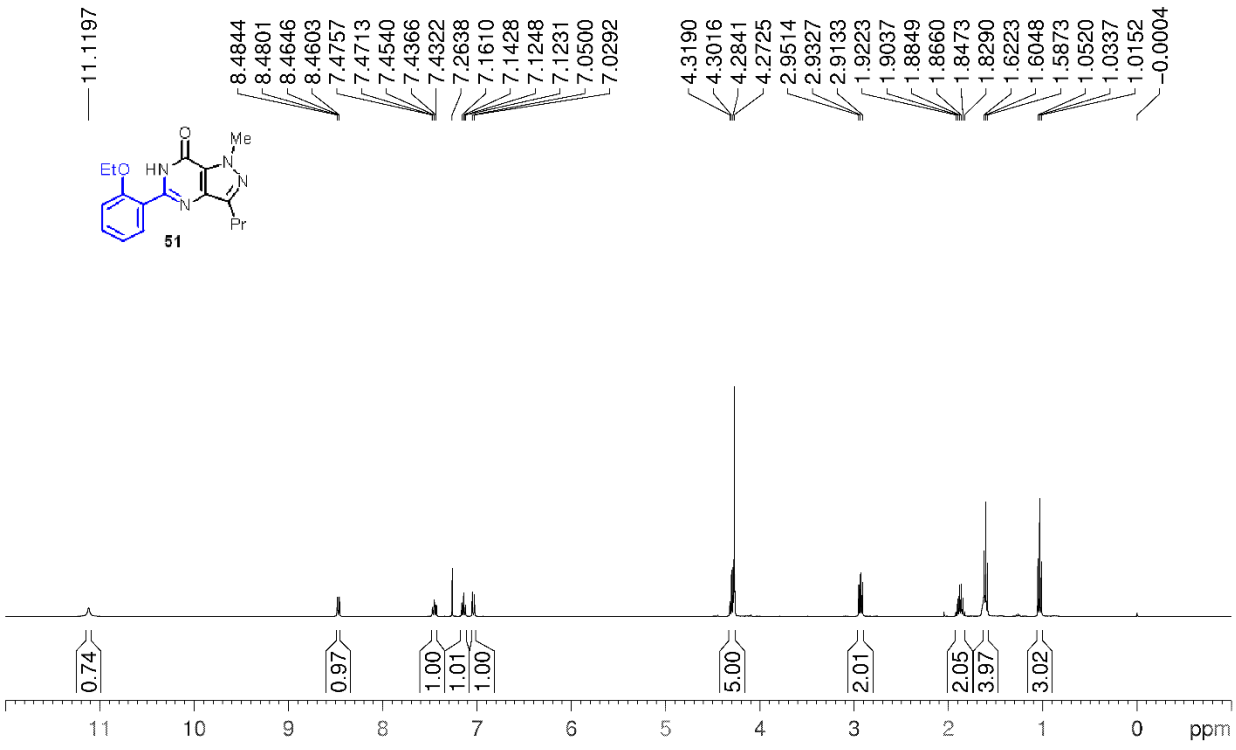
140.44
135.00
133.11
132.59
129.44
129.21
127.39
123.24
120.52
119.66
40.41
40.20
39.99
39.79
39.58
39.37
39.16
43
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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8.4603
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7.4713
7.4540
7.4366
7.4322
7.2638
7.1610
7.1428
7.1248
7.1231
7.0500
7.0292
4.3190
4.3016
4.2841
4.2725
2.9514
2.9327
2.9133
1.9223
1.9037
1.8849
1.8660
1.8473
1.8290
1.6223
1.6048
1.5873
1.0520
1.0337
1.0152
-0.0004
51
0.74
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1.01
1.00
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2.05
3.97
3.02
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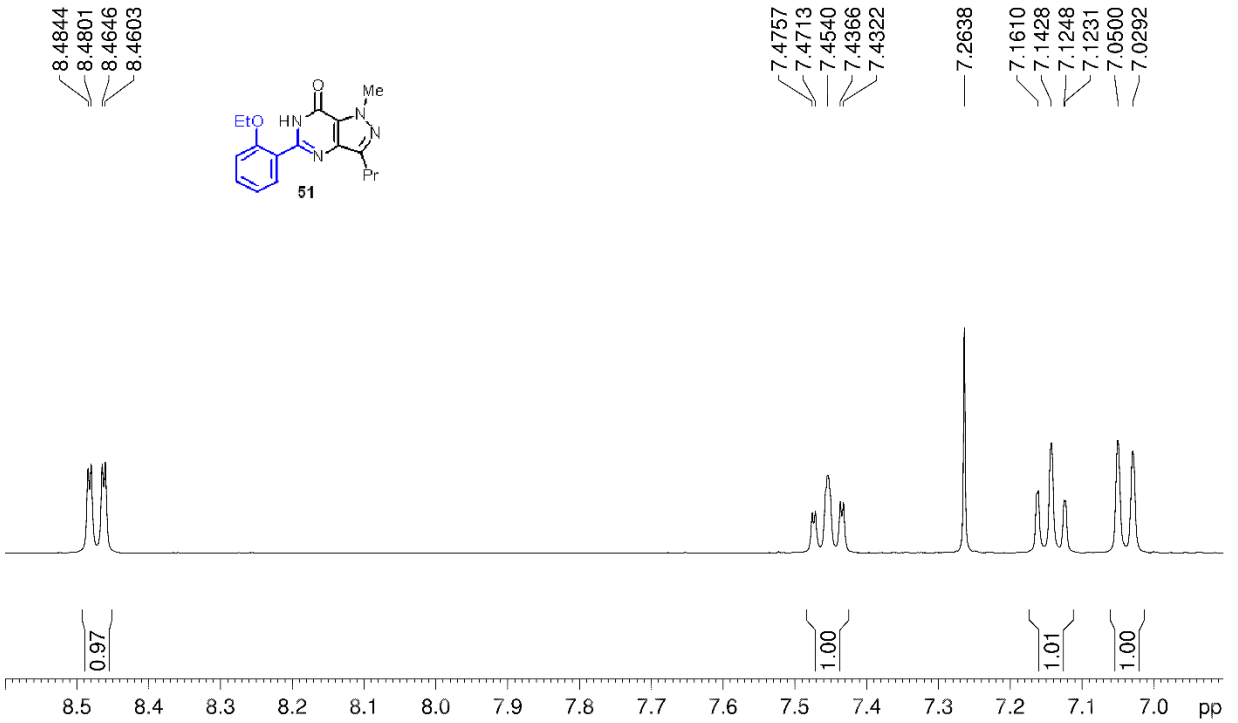
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8.4801
8.4646
8.4603
7.4757
7.4713
7.4540
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7.4322
7.2638
7.1610
7.1428
7.1248
7.1231
7.0500
7.0292
EtO
HN
N
Me
Pr
O
51
0.97
1.00
1.01
1.00
8.5
8.4
8.3
8.2
8.1
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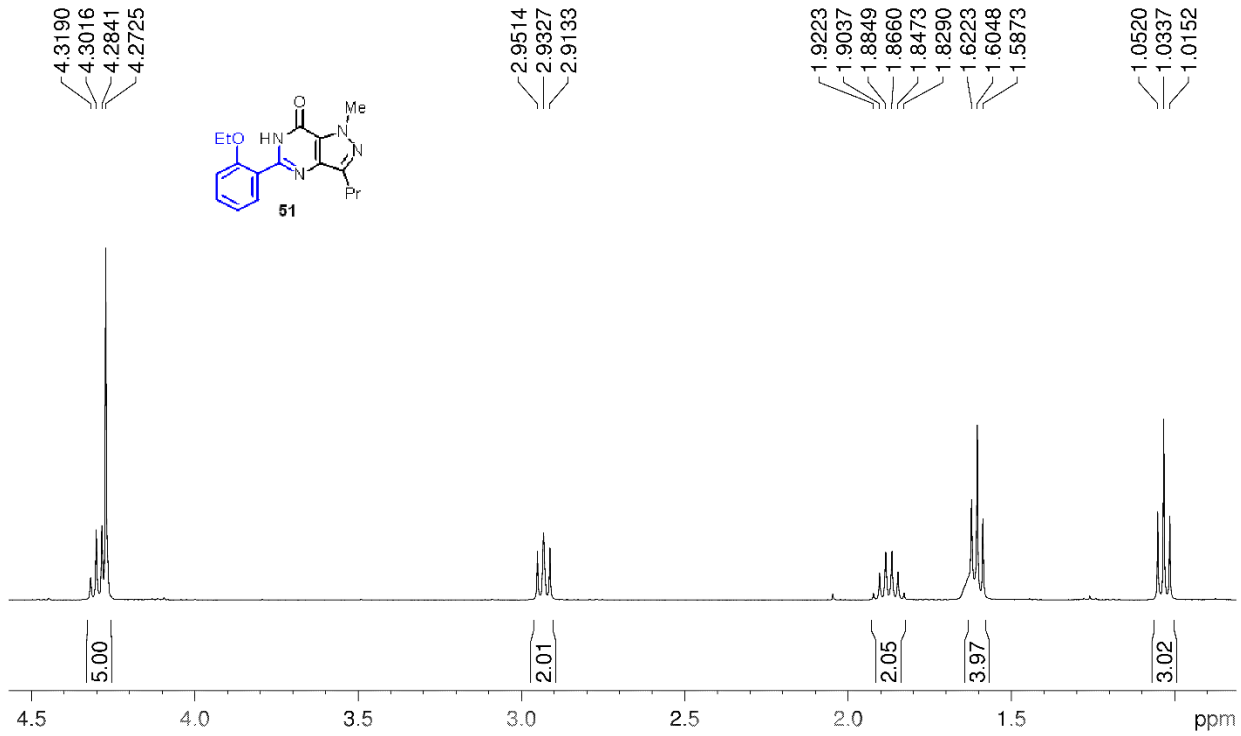
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
pp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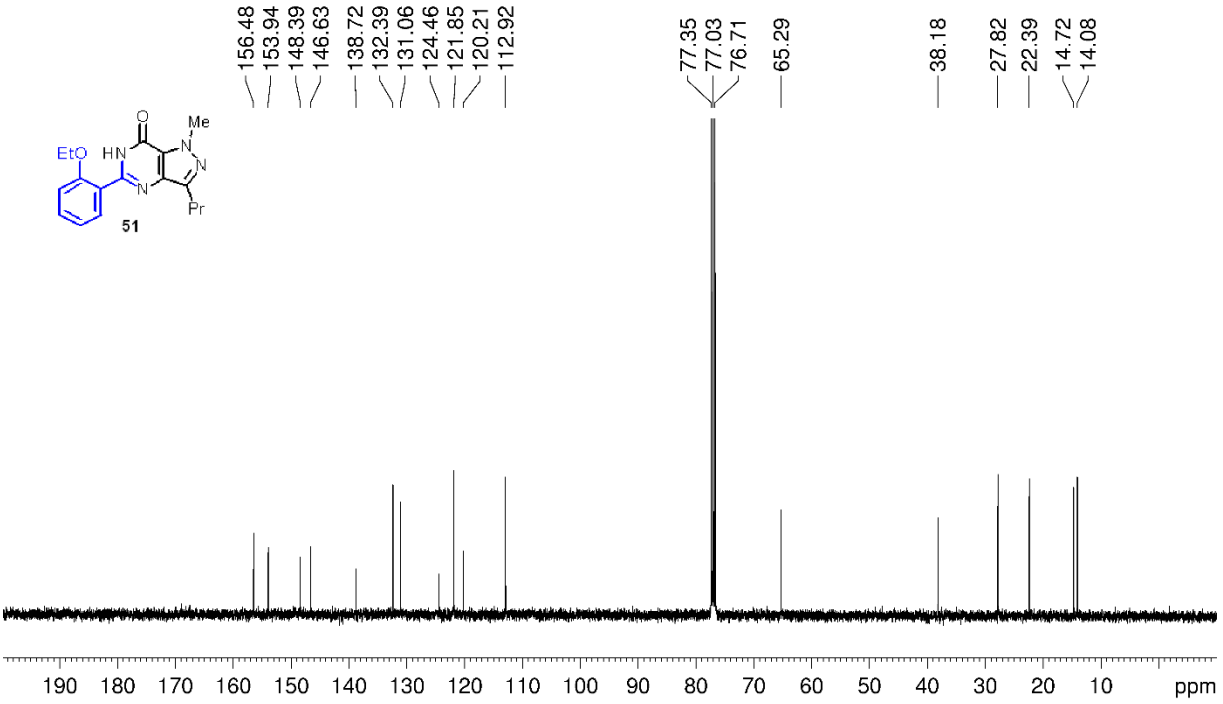
4.3016
4.2841
4.2725
2.9514
2.9327
2.9133
1.9223
1.9037
1.8849
1.8660
1.8473
1.8290
1.6223
1.6048
1.5873
1.0520
1.0337
1.0152
EtO
HN
N
Me
Pr
O
51
5.00
2.01
2.05
3.97
3.02
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm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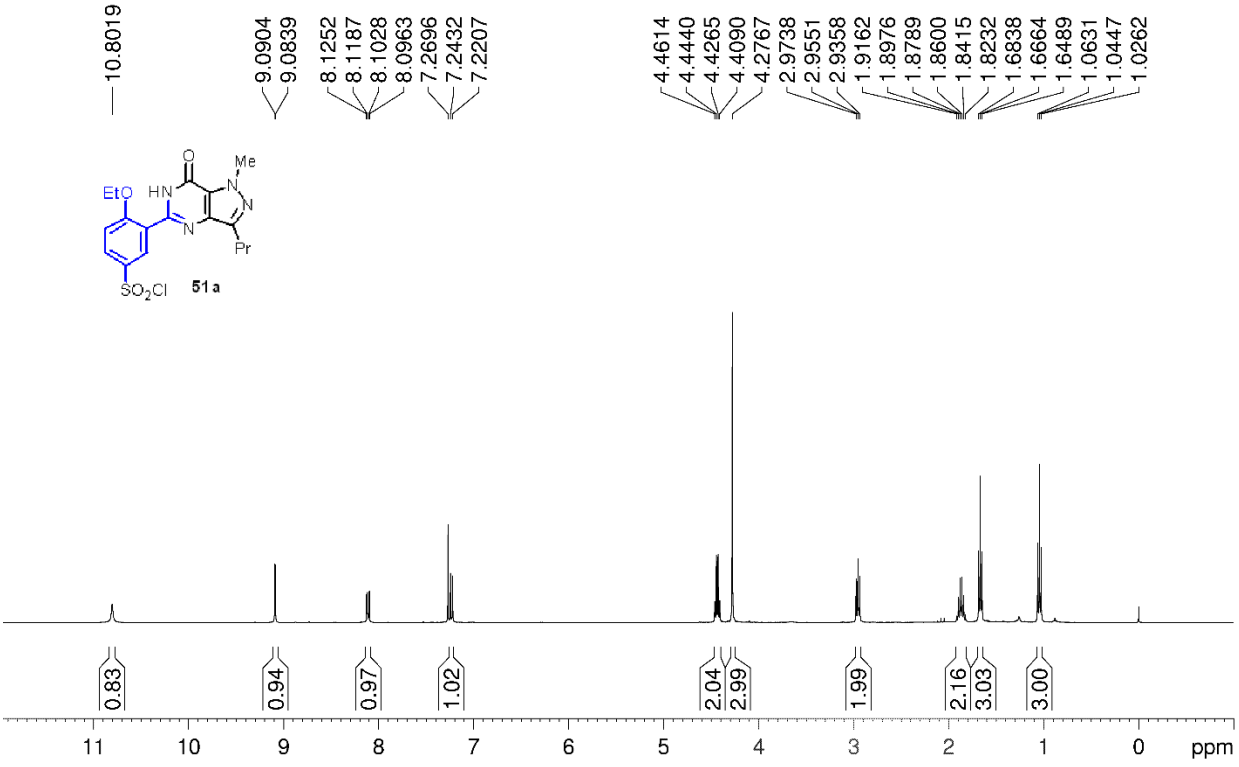
153.94
148.39
146.63
138.72
132.39
131.06
124.46
121.85
120.21
112.92
77.35
77.03
76.71
65.29
38.18
27.82
22.39
14.72
14.08
EtO
HN
N
Me
N
Pr
51
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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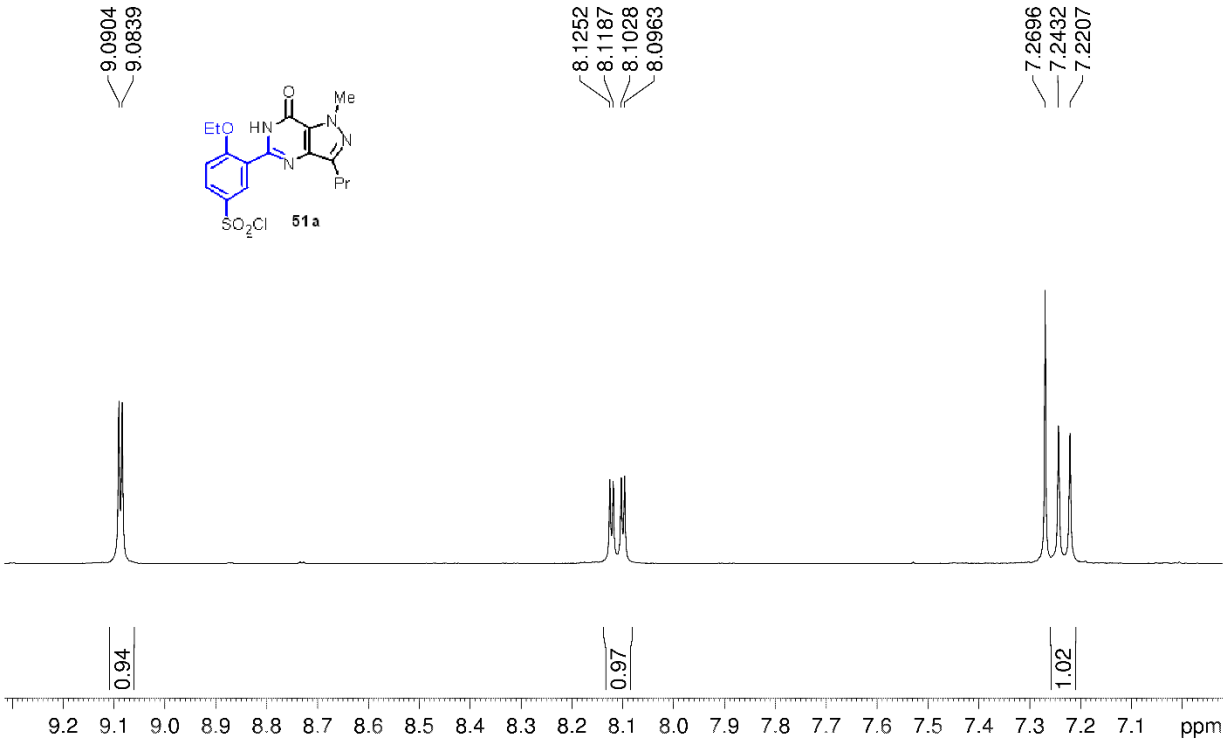
8.1187
8.1028
8.0963
7.2696
7.2432
7.2207
4.4614
4.4440
4.4265
4.4090
4.2767
2.9738
2.9551
2.9358
1.9162
1.8976
1.8789
1.8600
1.8415
1.8232
1.6838
1.6664
1.6489
1.0631
1.0447
1.0262
EtO
HN
N
Me
N
Pr
SO2Cl
51a
0.83
0.94
0.97
1.02
2.04
2.99
1.99
2.16
3.03
3.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm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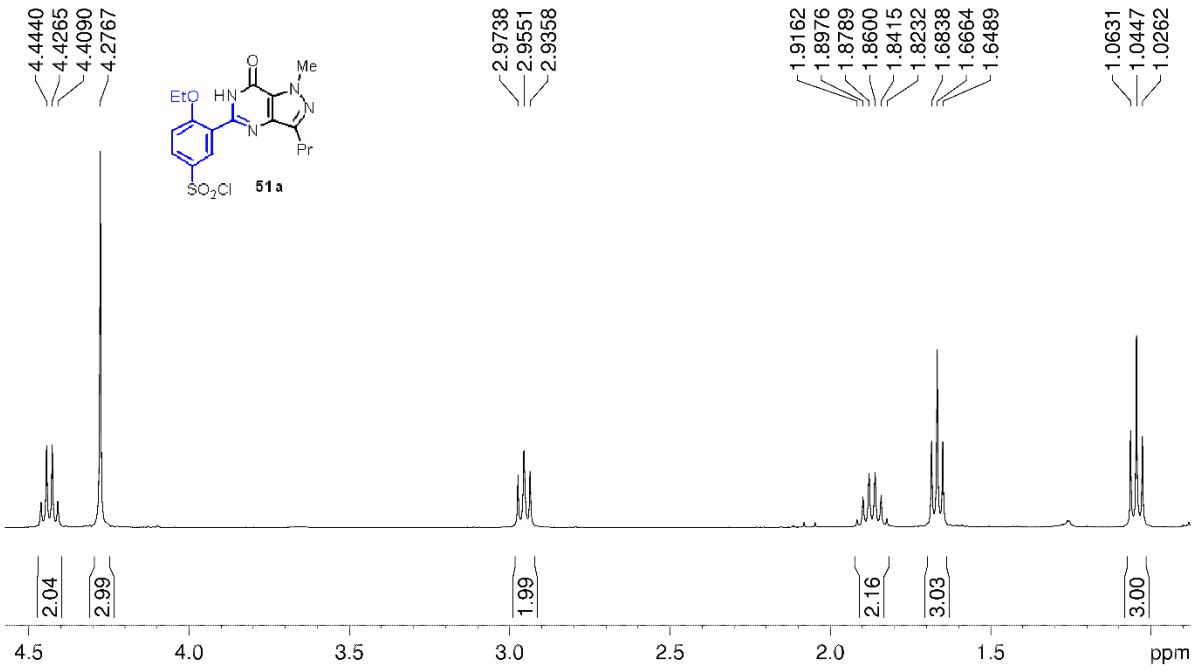
9.0839
8.1252
8.1187
8.1028
8.0963
7.2696
7.2432
7.2207
EtO
HN
N
Me
Pr
SO2Cl
51a
0.94
0.97
1.02
9.2 9.1 9.0 8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm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4.4265
4.4090
4.2767
2.9738
2.9551
2.9358
1.9162
1.8976
1.8789
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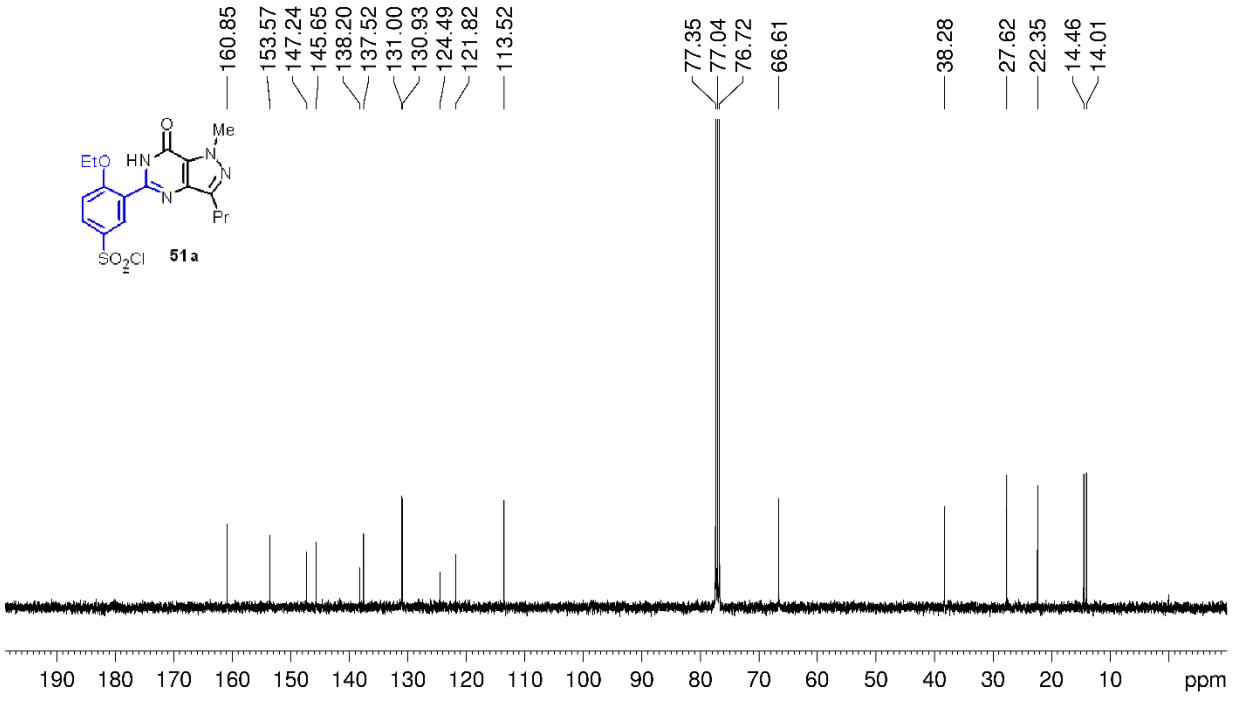
1.8415
1.8232
1.6838
1.6664
1.6489
1.0631
1.0447
1.0262
EtO
HN
N
Me
Pr
SO2Cl
51a
2.04
2.99
1.99
2.16
3.03
3.00
4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm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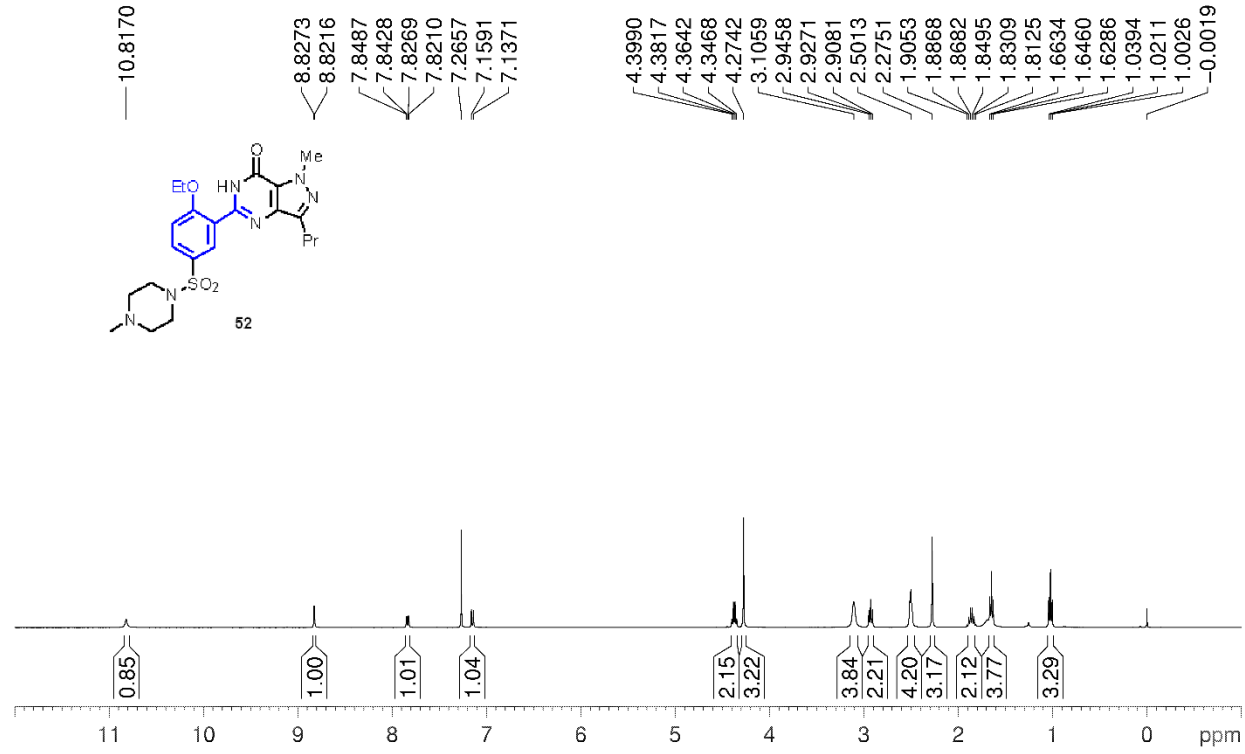
147.24
145.65
138.20
137.52
131.00
130.93
124.49
121.82
113.52
77.35
77.04
76.72
66.61
38.28
27.62
22.35
14.46
14.01
EtO
HN
Me
Pr
SO2Cl
51a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.8210
7.2657
7.1591
7.1371
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4.3817
4.3642
4.3468
4.2742
3.1059
2.9458
2.9271
2.9081
2.5013
2.2751
1.9053
1.8868
1.8682
1.8495
1.8309
1.8125
1.6634
1.6460
1.6286
1.0394
1.0211
1.0026
-0.0019
EtO
HN
Me
Pr
SO2
52
0.85
1.00
1.01
1.04
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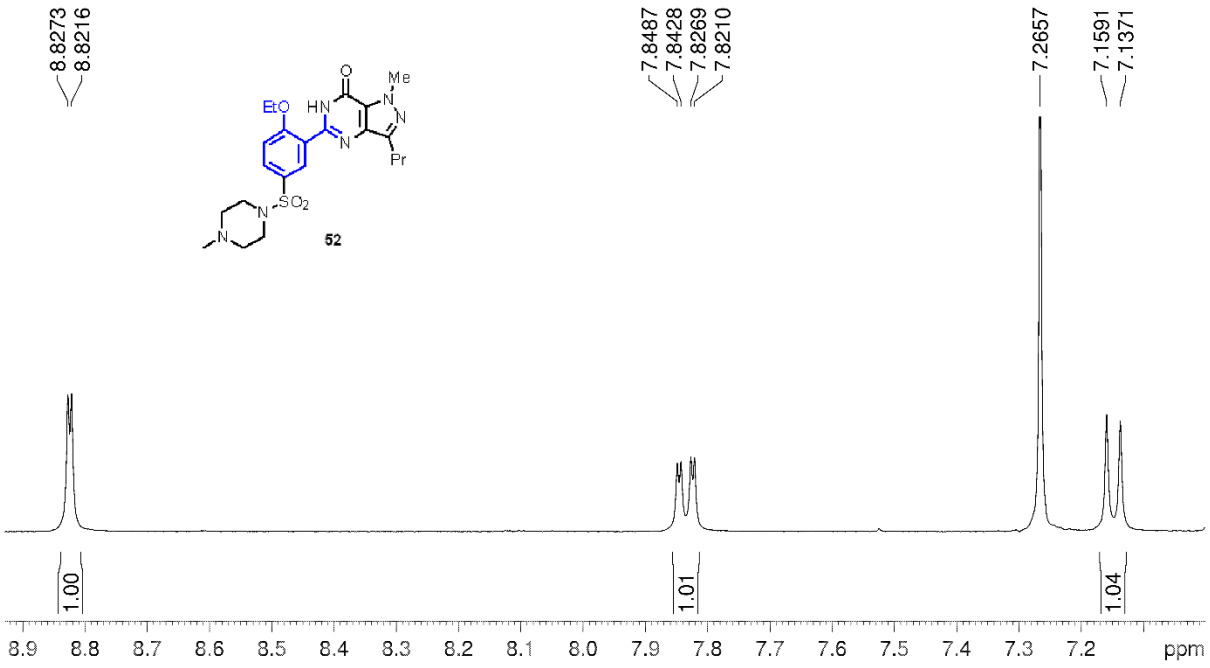
2.21
4.20
3.17
2.12
3.77
3.29
11 10 9 8 7 6 5 4 3 2 1 0 ppm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8.8216
7.8487
7.8428
7.8269
7.8210
7.2657
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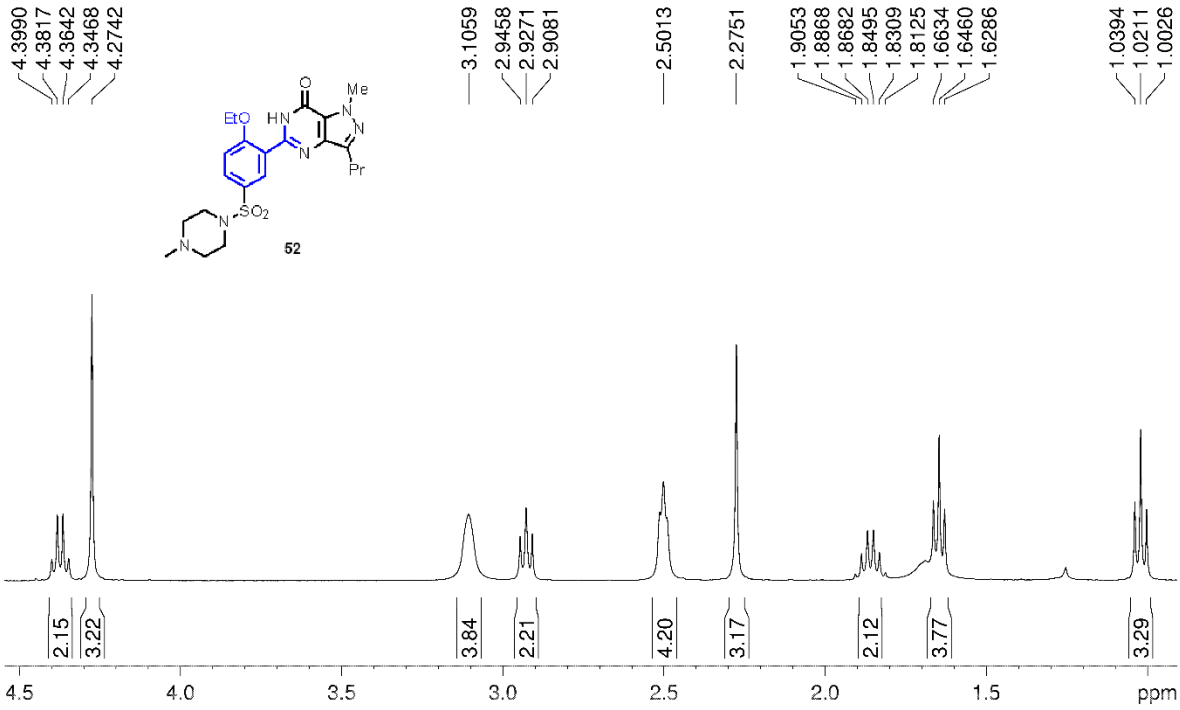
7.1371
EtO
HN
Me
Pr
SO2
52
1.00
1.01
1.04
8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm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4.2742
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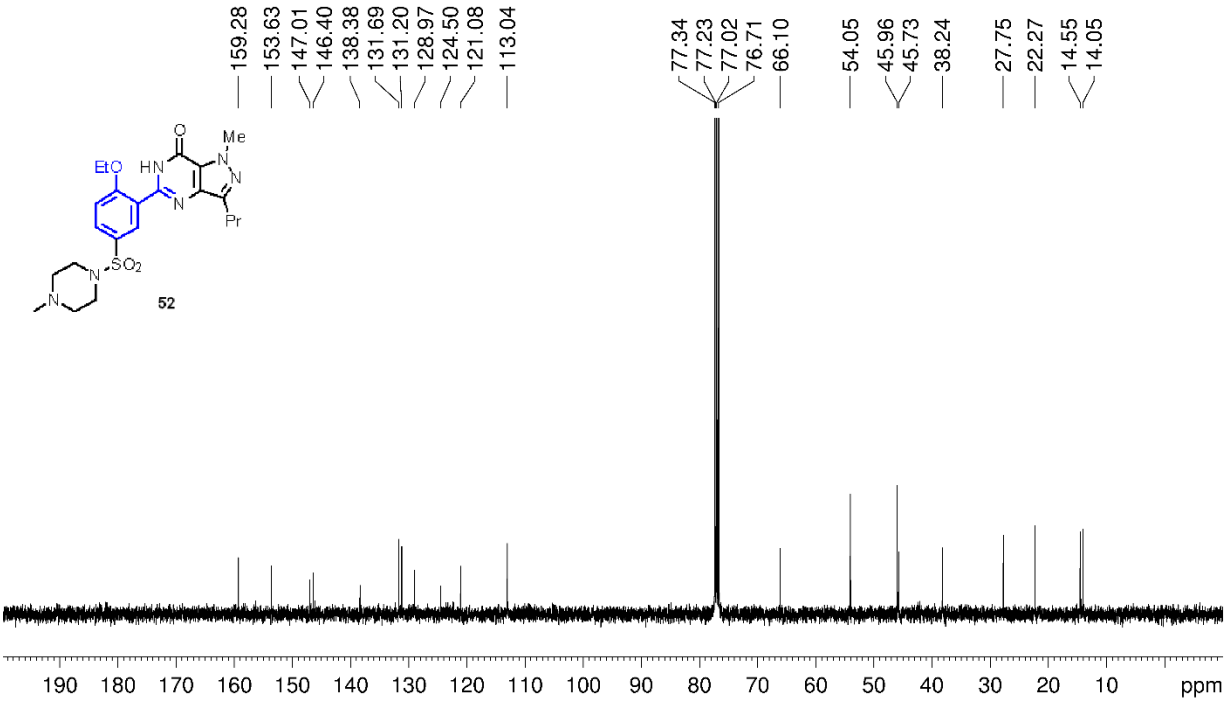
2.9458
2.9271
2.9081
2.5013
2.2751
1.9053
1.8868
1.8682
1.8495
1.8309
1.8125
1.6634
1.6460
1.6286
1.0394
1.0211
1.0026
EtO
HN
Me
Pr
SO2
52
2.15
3.22
3.84
2.21
4.20
3.17
2.12
3.77
3.29
4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm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147.01
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121.08
113.04
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77.23
77.02
76.71
66.10
54.05
45.96
45.73
38.24
27.75
22.27
14.55
14.05
EtO
HN
O
Me
N
N
Pr
SO2
N
N
52
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm