Supporting information

Electrochemical Dehydrogenative and Desulfurative Annulation for the Synthesis of Isoxazolines and Pyrazolines

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General information

Unless otherwise noted, all reactions were carried out under atmospheric conditions and all reagents were purchased from commercial suppliers and used without further purification. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker Advance 600 and Bruker Ascend 400 instrument. Multiplicities were reported by use of the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet, br = broad. All chemical shifts in ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.00) and relative to the signal of solvent (7.26 ppm, chloroform-*d*; 2.50 ppm, DMSO-d_6), in ^{13}C NMR are relative to d-solvent peaks (77.16 ppm, chloroform-*d*; 39.60 ppm, DMSO-d_6). High- resolution mass spectra (HRMS) were recorded on Waters XEVO G2 Q-TOF.

Graphical guide for the set-up

As experimental set-up, a graphite electrode (1 cm×1 cm), a carbon paper electrode (13 mm×13 mm×0.2 mm), an undivided three-necked round-bottom flask (10 mL) and a potentiostat (ITECH T66122S) were used.

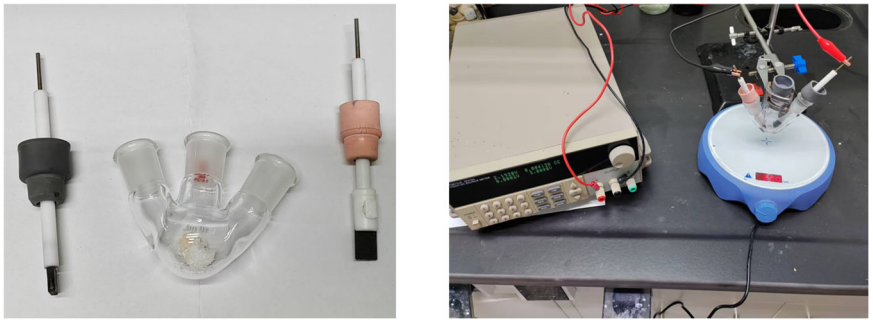
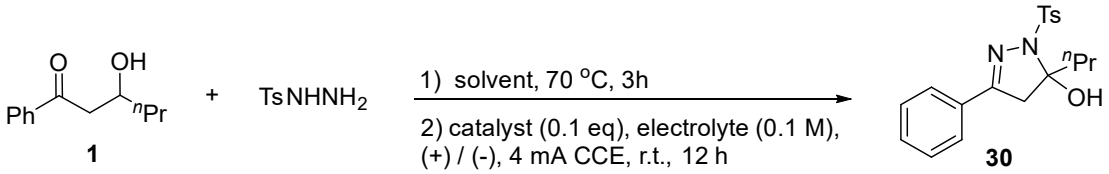


Figure. S1. Reaction setup for the milligram scale reaction

Optimization of reaction conditions

Table S1. Optimization of the conditions for pyrazolines synthesis [a]



Entry	Catalyst	Electrolyte	Anode/cathode	Solvent	Yield (%)[b] **30**
1	-	Bu_4NBr	Pt/Pt	CH_3CN	12

2	-	$LiClO_4$	Pt/Pt	CH_3CN	N.D.
3	TEMPO	Bu_4NBr	Pt/Pt	CH_3CN	41
4	Cat-A	Bu_4NBr	Pt/Pt	CH_3CN	59
5	Cat-B	Bu_4NBr	Pt/Pt	CH_3CN	55
6	Cat-D	Bu_4NBr	Pt/Pt	CH_3CN	N.D.
7	Cat-C	Bu_4NBr	Pt/Pt	CH_3CN	32
8	Cat-A	Bu_4NBr	Pt/Pt	MeOH	N.D.
9	Cat-A	Bu_4NBr	Pt/Pt	TFE	N.D.
10	Cat-A	Bu_4NBr	Pt/Pt	DMF	53
11	Cat-A	Et_4NBF_4	Pt/Pt	CH_3CN	37
12	Cat-A	Bu_4NOTs	Pt/Pt	CH_3CN	N.D.
13	Cat-A	Bu_4NBr	C_g/Pt	CH_3CN	66
14	Cat-A	Bu_4NBr	C_g/Ni	CH_3CN	31
15	Cat-A	Bu_4NBr	C_g/Fe	CH_3CN	27
16	Cat-A	Bu_4NBr	C_g/C_g	CH_3CN	69
17	**Cat-A**	**Bu_4NBr**	**C_g/ C_p**	**CH_3CN**	**77**
18[c]	Cat-A	Bu_4NBr	C_g/ C_p	CH_3CN	36
19[d]	Cat-A	Bu_4NBr	C_g/ C_p	CH_3CN	62
20[e]	Cat-A	Bu_4NBr	C_g/ C_p	CH_3CN	N.D.

[a]Reaction conditions: **1** (38.5 mg, 0.2 mmol), $TsNHNH_2$ (40.9 mg, 0.22 mmol), solvent (3 mL), 70 °C, 3h; then cat. (0.02 mmol), electrolyte (0.3 mmol), 4.0 mA and 12 h, undivided cell. [b]Isolated yields. [c]Omission of the step 1 and reacted at 50 °C. [d]Under N_2. [e]Without electricity. C_g = graphite, C_p = carbon paper

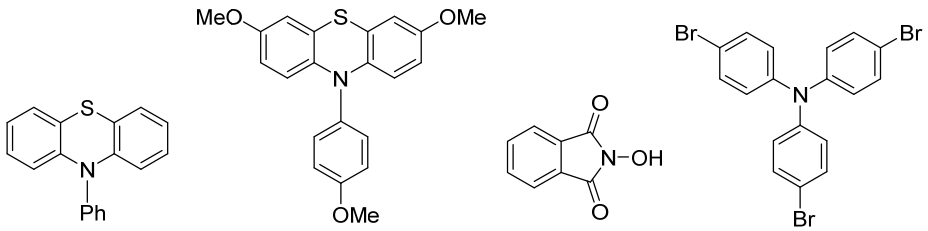
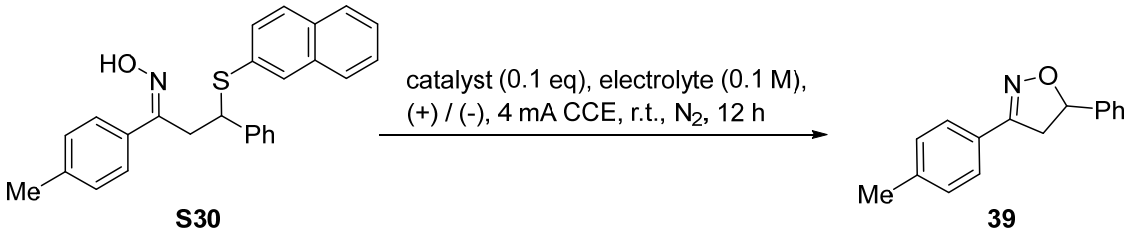


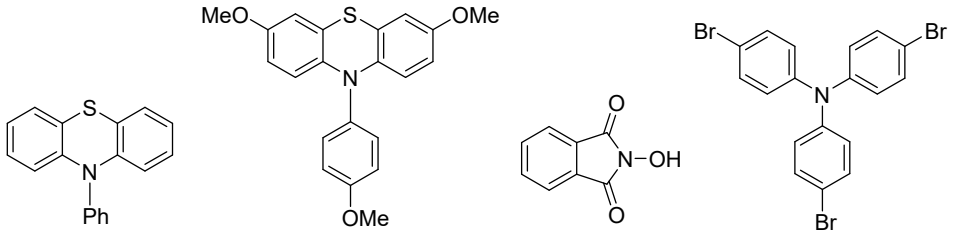
Table S2. Optimization of the conditions for desulfurative annulation towards the synthesis of isoxazolines[a]



Entry	Catalyst	Electrolyte	Anode/cathode	Solvent	Yield (%)[b] **39**
1	**Cat-A**	**Bu_4NBr**	**C_g/ C_p**	**CH_3CN**	**91**
2	Cat-B	Bu_4NBr	C_g/ C_p	CH_3CN	82
3	Cat-C	Bu_4NBr	C_g/ C_p	CH_3CN	65
4	Cat-D	Bu_4NBr	C_g/ C_p	CH_3CN	22
5	TEMPO	Bu_4NBr	C_g/ C_p	CH_3CN	37
6	Cat-A	$LiClO_4$	C_g/ C_p	CH_3CN	13
7	Cat-A	Et_4NCl	C_g/ C_p	CH_3CN	19
8	Cat-A	Bu_4NPF_6	C_g/ C_p	CH_3CN	11
9	Cat-A	NaBr	C_g/ C_p	CH_3CN	16
10	Cat-A	Bu_4NBr	C_g/ C_p	DMF	12
11	Cat-A	Bu_4NBr	C_g/ C_p	DMSO	N.D.

12	Cat-A	Bu_4NBr	C_g/ C_p	MeOH	71
13	Cat-A	Bu_4NBr	C_g/Pt	CH_3CN	66
14	Cat-A	Bu_4NBr	C_g/Ni	CH_3CN	73
15	Cat-A	Bu_4NBr	C_g/Cu	CH_3CN	22
16	Cat-A	Bu_4NBr	Pt/Pt	CH_3CN	37
17	Cat-A	Bu_4NBr	Pt/ C_p	CH_3CN	62
18[c]	Cat-A	Bu_4NBr	C_g/ C_p	CH_3CN	76
19[d]	Cat-A	Bu_4NBr	C_g/ C_p	CH_3CN	N.D.

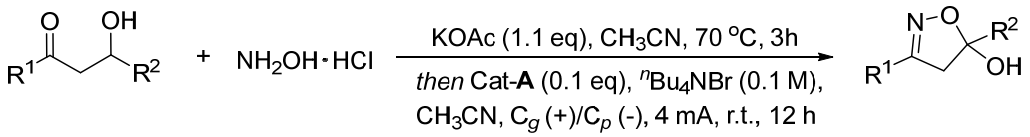
[a]Reaction conditions: **S39** (72.6 mg, 0.2 mmol), cat. (0.02 mmol), electrolyte (0.3 mmol), solvent (3 mL), 4.0 mA, N_2 and 12 h, undivided cell. [b]Isolated yields. [c]Under air. [d]Without electricity. C_g = graphite, C_p = carbon paper



Cat-**A** Cat-**B** Cat-**C** Cat-**D**

General procedure for electrochemical synthesis of isoxazolines

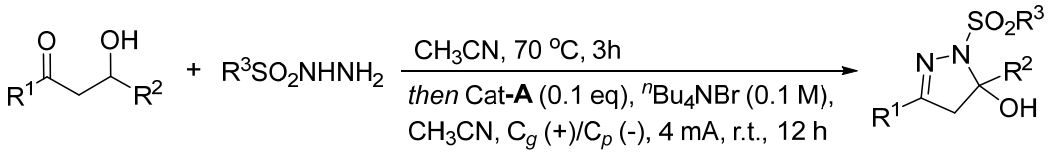
An oven-dried undivided electrochemical cell (10 mL) fitted with a stir bar was charged with β-hydroxyl ketone (0.2 mmol), hydroxylamine hydrochloride (15.3 mg, 0.22 mmol), KOAc (21.6 mg, 0.22 mmol), and CH_3CN (3 mL), the mixture was stirred at 70 °C for 3h; then Cat-**A** (5.5 mg, 0.02 mmol), $^nBu_4NBr$ (96.7 mg, 0.3 mmol), equipped with a graphite anode (1 cm × 1 cm) and a carbon paper cathode (1 cm × 1.3 cm). The system was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography to afford the desired products.



General procedure for electrochemical one-pot synthesis of pyrazolines

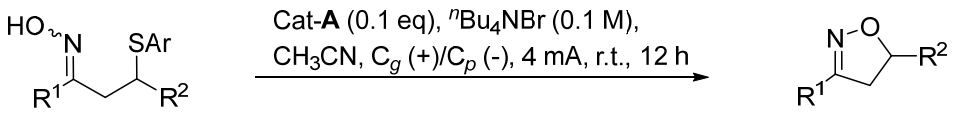
An oven-dried undivided electrochemical cell (10 mL) fitted with a stir bar was

charged with β-hydroxyl ketone (0.2 mmol), sulfonylhydrazine (0.22 mmol) and CH_3CN (3 mL), the mixture was stirred at 70 °C for 3h; then Cat-**A** (5.5 mg, 0.02 mmol), $^nBu_4NBr$ (96.7 mg, 0.3 mmol), equipped with a graphite anode (1 cm × 1 cm) and a carbon paper cathode (1 cm × 1.3 cm). The mixture was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography to afford the desired products.



General procedure for electrochemical desulfurative annulation towards the synthesis of isoxazolines

An oven-dried undivided electrochemical cell (10 mL) fitted with a stir bar was charged with β-arylthioketone ketone (0.2 mmol), Cat-**A** (5.5 mg, 0.02 mmol), $^nBu_4NBr$ (96.7 mg, 0.3 mmol), and CH_3CN (3 mL). The system was equipped with a graphite anode (1 cm × 1 cm) and a carbon paper cathode (1 cm × 1.3 cm). After purging the reaction system with nitrogen for 5 min to displace the air, the mixture was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography to afford the desired products.



Scalability and synthetic utility

Continuous gram-scale production of 2 with flow cell

To a 250 mL round-bottom flask, 3-hydroxy-1-phenylhexan-1-one **1** (1.92 g, 10 mmol), hydroxylamine hydrochloride (0.75 g, 11 mmol), KOAc (1.08 g, 11 mmol), and CH_3CN (150 mL) were added. The mixture was stirred at 70 °C for 4.5 h; then Cat-**A** (137.5 mg, 0.5 mmol), $^nBu_4NBr$ (2.42 g, 7.5 mmol) were added. The solution was pushed by a peristaltic pump to pass through the flow electrolytic cell operated with a flow rate of 0.30 mL min^{-1} and a constant current (50–65 mA). The collected solution was concentrated under reduced pressure on a rotary evaporator. The residue was purified through silica gel chromatography (petroleum: ethyl acetate = 5: 1) to afford the desired products as white solid (1.58 g, 77 %).

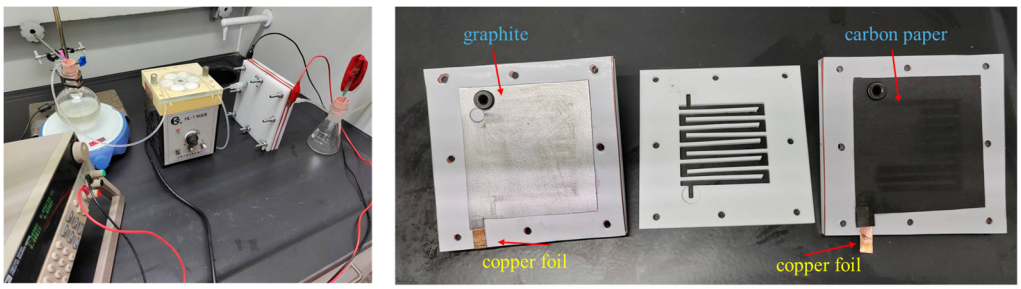
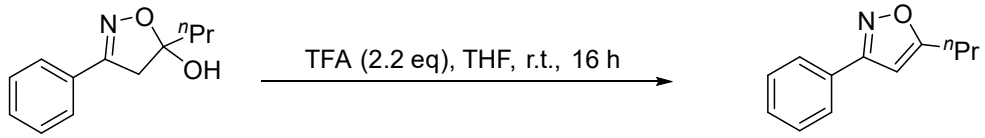


Figure. S2. Reaction setup for the gram scale reaction with flow cell

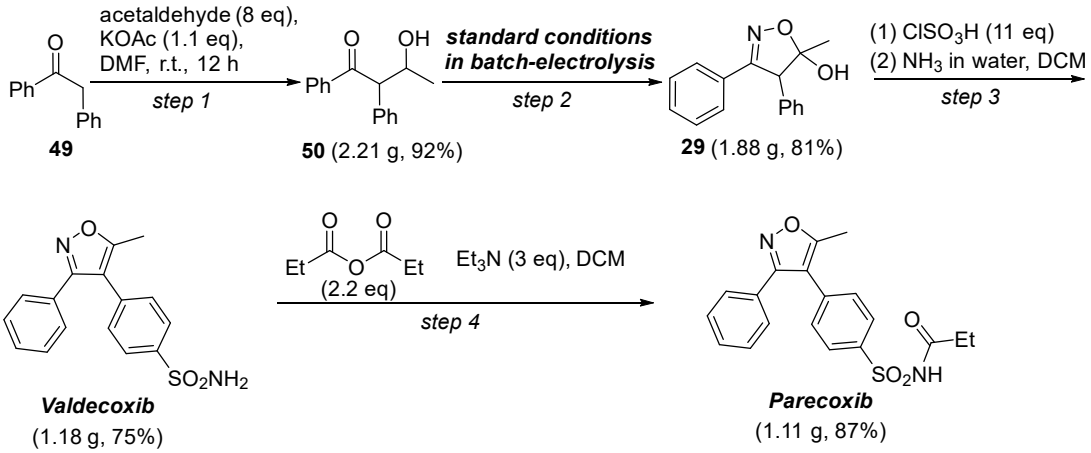
Dehydration of 2 to synthesis isoxazole



2 **48** (0.87 g, 93%)

To a 3-phenyl-5-propyl-4,5-dihydroisoxazol-5-ol **2** (1.03 g, 5.0 mol) solution in THF (15 mL) was added TFA (1.25 g, 11.0 mmol) dropwisely at 0 °C. Then the reaction mixture was warmed to room temperature and stirred for 16 h. The solvent was removed under vacuum and the residue was purified by silica gel chromatography (petroleum: ethyl acetate = 20: 1) to provide the desired product as yellow solid (0.87 g, 93 %).

Gram-scale synthesis of *Parecoxib*



Step 1:

To a solution of 1,2-diphenylethan-1-one **49** (10 mmol) and KOAc (1.08 g, 11 mmol) in anhydrous DMF (15 mL) was added acetaldehyde (3.5 g, 80 mmol). The reaction mixture was stirred at room temperature for 12 h. The residue was diluted with water (100 mL) and filtered. The solid was washed with water and dried under vacuum to get the product as white solid (2.21 g, 92 %). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 (ddd, *J* = 9.3, 8.4, 1.3 Hz, 2H), 7.48 – 7.41 (m, 1H), 7.38 – 7.30 (m, 3H), 7.30 – 7.25 (m, 3H), 7.24 – 7.17 (m, 1H), 4.56 – 4.52 (m, 1H), 4.52 – 4.49 (m, 0.44H), 4.49 (t,

J = 2.8 Hz, 0.56H), 1.20 (d, *J* = 6.2 Hz, 0.88H), 1.10 (d, *J* = 6.1 Hz, 2.21H).

Step 2:

A mixture of 3-hydroxy-1,2-diphenylbutan-1-one **50** (2.1 g, 8.7 mmol), hydroxylamine hydrochloride (0.67 g, 9.6 mmol), and KOAc (1.04 g, 10.6 mmol) in CH_3CN (130 mL) heated at 70 °C until complete consumption of the **50** (monitored by TLC). The reaction mixture was transferred to a 150 mL beaker which was equipped with a graphite anode and a carbon paper cathode. The mixture was electrolyzed with constant current of 60.0 mA at room temperature for 12 h. The solvent was removed under vacuum and the residue was purified by silica gel chromatography (petroleum: ethyl acetate = 5: 1) to provide the desired product **29**[1] as pale-yellow solid (1.79 g, 81 %).

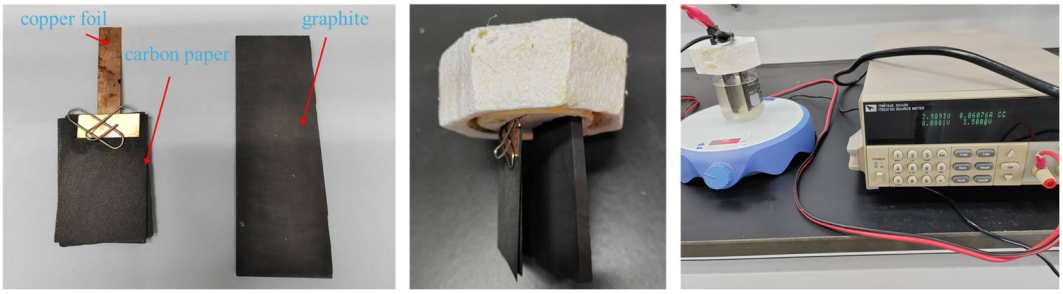


Figure. S3. Reaction setup for the gram scale reaction with batch electrolysis

Step 3:

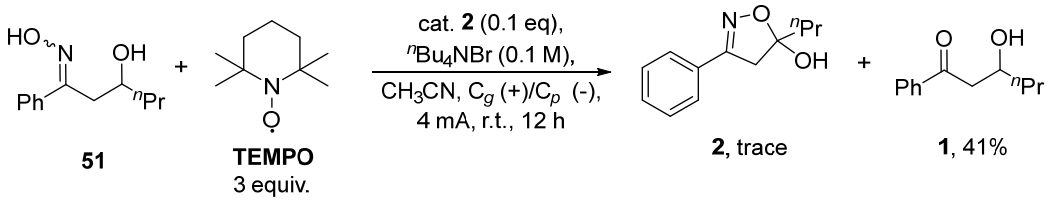
A method modified from the reported literature.[2] The mixture of the isoxazoline (1.27 g, 5.0 mmol) in DCM (5 mL) in a round bottom flask equipped with magnetic stirrer was cooled to 0 °C and then chlorosulfonic acid (6.3 g, 55 mmol) was added. The brown reaction mixture was stirred at 0 °C for 4 h and then dropwise added to a stirring suspension of ice (60mL) and dichloromethane (30 mL). The organic phase was added directly to a solution of ammonium hydroxide (28% NH_3 in water, 50 mL) kept at 0 °C. This biphasic mixture was vigorously stirred at 0 °C for 5 h. The two phases were separated, and the aqueous layer was extracted three times with CH_2Cl_2. The combined organic extracts were dried over anhydrous Na_2SO_4 and then evaporated under vacuum. The solvent was removed under vacuum and the residue was purified by silica gel chromatography (petroleum: ethyl acetate = 3: 1) to provide the desired product as white solid (1.18 g, 75 %).

Step 4:

A solution of *Valdecoxib* (1.09 g, 3.5 mmol) and Et_3N (1.06 g, 10.5 mmol) in CH_2Cl_2 (20 mL) was added propionic anhydride (1.0 g, 7.7 mmol) dropwise over 20 min at 0 °C. The reaction mixture was stirred at room temperature for 10 h, and the resulting mixture was washed with water and brine, the organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel chromatography (petroleum: DCM = 4: 5) to provide the desired product as white solid (1.13 g, 87 %).

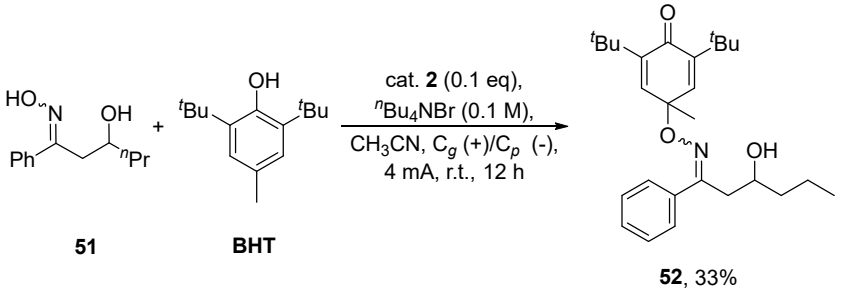
Mechanism studies

Quenching experiment

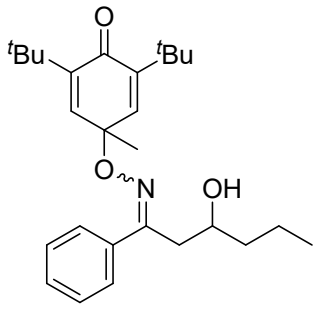


An oven-dried undivided electrochemical cell (10 mL) fitted with a stir bar was charged with β-hydroxyl oxime **51** (41.4 mg, 0.2 mmol), Cat-**A** (5.5 mg, 0.02 mmol), $^{n}Bu_4NBr$ (96.7 mg, 0.3 mmol), TEMPO (93.8 mg, 0.6 mmol) and CH_3CN (3 mL). The system was equipped with a graphite anode (1 cm x 1 cm) and a carbon paper cathode (1 cm x 1.3 cm). The mixture was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. None of the product **2** was detected while β-hydroxyl ketone **1** was isolated in 41% yield.

Experimental Evidence for the oxime radical



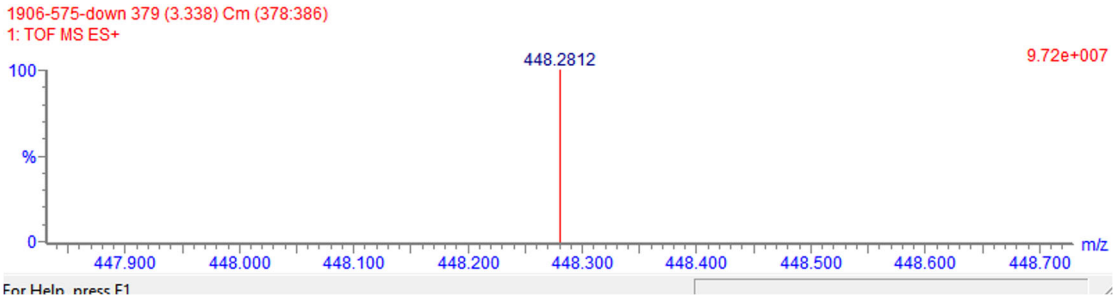
An oven-dried undivided electrochemical cell (10 mL) fitted with a stir bar was charged with β-hydroxyl oxime **51** (41.4 mg, 0.2 mmol), Cat-**A** (5.5 mg, 0.02 mmol), $^{n}Bu_4NBr$ (96.7 mg, 0.3 mmol), BHT (132.1 mg, 0.6 mmol) and CH_3CN (3 mL). The system was equipped with a graphite anode (1 cm x 1 cm) and a carbon paper cathode (1 cm x 1.3 cm). After purging the reaction system with nitrogen for 5 min to displace the air, the mixture was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography to afford the **52**.



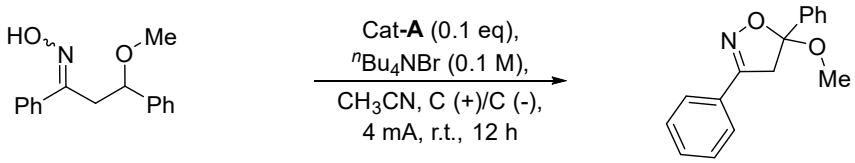
2,6-di-tert-butyl-4-(((3-hydroxy-1-phenylhexylidene)amino)oxy)-4-

methylcyclohexa-2,5-dien-1-one (52):

White solid (28.0 mg, 33% yield). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 7.5 Hz, 2H), 7.44 – 7.37 (m, 3H), 6.59 (s, 2H), 3.88 (tt, *J* = 7.9, 3.2 Hz, 1H), 3.24 (s, 1H), 2.65 – 2.49 (m, 2H), 1.57 – 1.51 (m, 1H), 1.47 – 1.41 (m, 1H), 1.36 (s, 3H), 1.36 – 1.30 (m, 2H), 1.25 (d, *J* = 7.9 Hz, 18H), 0.88 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 186.98, 156.23, 146.68, 146.64, 142.16, 142.08, 135.91, 129.30, 128.47, 126.42, 69.97, 40.07, 34.73, 34.62, 29.54, 25.43, 18.87, 14.10. HRMS (ESI) m/z: $[M + Na]^+$ calcd. for $C_{27}H_{39}NNaO_3$ 448.2822; Found 448.2812.

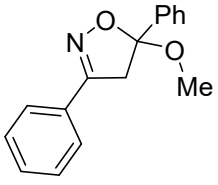


Electrooxidation of β-methoxyl oxime



55 **56**

An oven-dried undivided electrochemical cell (10 mL) fitted with a stir bar was charged with β-methoxyl oxime **55** (44.2 mg, 0.2 mmol), Cat-**A** (5.5 mg, 0.02 mmol), ^{n}Bu$_4$NBr (96.7 mg, 0.3 mmol), and CH_3CN (3 mL). The system was equipped with a graphite anode (1 cm x 1 cm) and a carbon paper cathode (1 cm x 1.3 cm). The mixture was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography (petroleum: ethyl acetate = 8: 1) to afford the desired products.



56

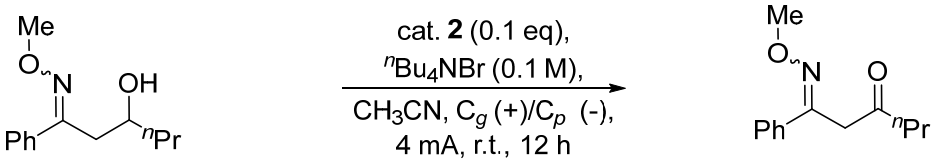
5-methoxy-5-methyl-3-phenyl-4,5-dihydroisoxazole (56):

White solid (44.5 mg, 88% yield). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.64 – 7.58 (m, 2H), 7.55 – 7.49 (m, 2H), 7.37 – 7.27 (m, 6H), 3.58 (d, *J* = 17.4 Hz, 1H), 3.32 (d, *J*

= 17.3 Hz, 1H), 3.18 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.2, 138.3, 130.4, 129.3, 128.8, 128.7, 128.6, 126.7, 126.4, 110.5, 50.83 48.9. HRMS (ESI) m/z: [M + H]$^+$ calcd. for $C_{16}H_{16}NO_2$ 254.1176; Found 254.1179.

Exclusion of A Route to the Isoxazolines via Annulation of Ketone and Oxime

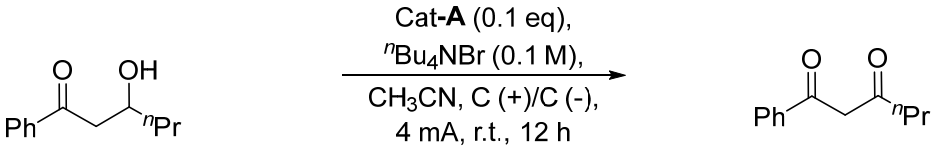
Electrooxidation of β-hydroxyl oxime ether



53 **54**, not detected

Oxime ether **54** (44.2 mg, 0.2 mmol), cat-A (5.5 mg, 0.02 mmol), ^{n}Bu$_4$NBr (96.7 mg, 0.3 mmol), and CH_3CN (3 mL) were added to oven-dried undivided electrochemical cell (10 mL) which equipped with a graphite anode (1 cm x 1 cm) and a carbon paper cathode (1 cm x 1.3 cm). The mixture was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. Conversion of **53** was traceless and none of the ketone product **54** was detected.

Electrooxidation of β-hydroxyl ketone



1 **57**, not detected

β-Hydroxyl ketone **1** (38.5 mg, 0.2 mmol), cat-**A** (5.5 mg, 0.02 mmol), ^{n}Bu$_4$NBr (96.7 mg, 0.3 mmol), and CH_3CN (3 mL) were added to oven-dried undivided electrochemical cell (10 mL) which equipped with a graphite anode (1 cm x 1 cm) and a carbon paper cathode (1 cm x 1.3 cm). The mixture was electrolyzed with constant current of 4.0 mA at room temperature for 12 h. Conversion of **1** was traceless and none of the ketone product **57** was detected.

Electrooxidation of β-hydroxyl ketone

Cyclic voltammograms were recorded in a solution of acetonitrile (MeCN) containing 0.1 M tetrabutylammonium tetrafluoroborate (^{n}Bu$_4$NBF$_4$) as the supporting electrolyte. The glassy carbon disk electrode (diameter, 1 mm) were used as both the working and counter electrode, while an Ag/AgCl electrode served as the reference electrode. The scan rate was maintained at 100 mV/s.

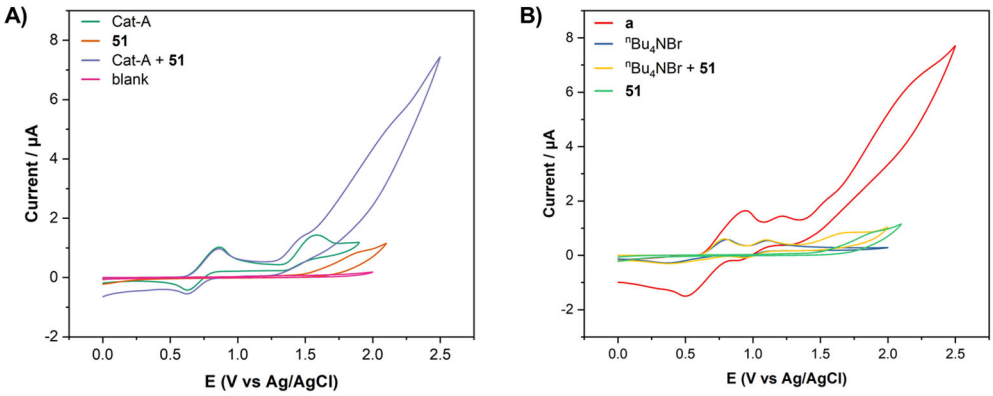
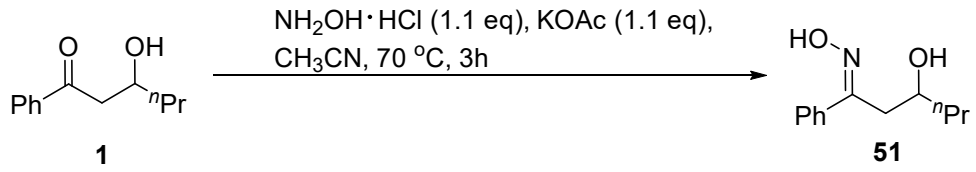


Figure. S4. Cyclic voltammograms. A) cyclic voltammograms of Cat-**A** (3 mM) in the absence or presence of **51** (3 mM). B) cyclic voltammograms of $^{n}Bu_4NBr$ (3 mM) in the absence or presence of **51** (3 mM). a: $^{n}Bu_4NBr$ (3 mM) + Cat-A (3 mM) + **51** (5 mM).

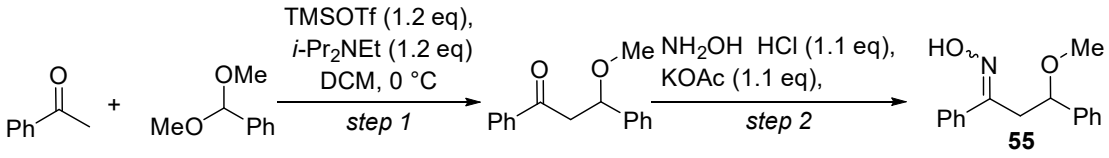
Synthesis of substrates

Synthesis of β-hydroxyl oxime 51



β-Hydroxyl ketone **1** (0.96 g, 5 mmol), hydroxylamine hydrochloride (0.42 g, 6 mmol), and KOAc (0.59 g, 6 mmol) in CH_3CN (15 mL) heated at 70 °C until complete consumption of the **1** (monitored by TLC). The solvent was removed under vacuum and the residue was purified by silica gel chromatography (petroleum: ethyl acetate = 3: 1) to provide the desired product as white solid (0.94 g, 91 %). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.60 (dd, *J* = 6.2, 2.7 Hz, 2H), 7.39 – 7.33 (m, 3H), 4.05 – 3.96 (m, 1H), 3.09 (dd, *J* = 13.5, 8.8 Hz, 1H), 2.92 (dd, *J* = 13.6, 3.9 Hz, 1H), 1.54 – 1.45 (m, 3H), 1.38 – 1.31 (m, 1H), 0.89 (t, *J* = 6.9 Hz, 3H).

Synthesis of 3-methoxy-1,3-diphenylpropan-1-one oxime 55



Step 1:

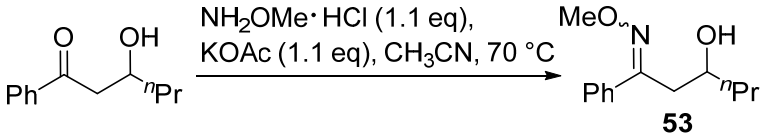
Following a modified literature procedure,[3] To an oven-dried 100 mL round-bottomed flask under N_2 was added CH_2Cl_2 (25 mL). The flask was cooled to 0 °C, and acetophenone (1.20 g, 10.0 mmol), *i*-Pr_2NEt (1.29 g, 12.0 mmol), and TMSOTf (2.67 g, 12.0 mmol) were added sequentially. After 30 min, benzaldehyde dimethyl acetal

(2.13 g, 14.0 mmol) was added, and the reaction was removed from the ice bath and allowed to warm to room temperature. After 2 h, the reaction mixture was filtered through a plug of silica and washed with Et_2O, then the solvent was removed by rotary evaporation. The residue was purified by silica gel chromatography (petroleum: ethyl acetate = 15: 1) to provide the desired product as colorless oil (2.09 g, 87 %). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.94 (dt, *J* = 8.5, 1.0 Hz, 2H), 7.54 (ddq, *J* = 7.8, 6.9, 1.1 Hz, 1H), 7.46 – 7.42 (m, 2H), 7.41 (d, *J* = 7.3 Hz, 2H), 7.39 – 7.35 (m, 1H), 7.33 – 7.27 (m, 1H), 4.88 (dd, *J* = 8.4, 4.4 Hz, 1H), 3.59 (ddd, *J* = 16.5, 8.4, 0.8 Hz, 1H), 3.24 (d, *J* = 0.9 Hz, 3H), 3.08 (ddd, *J* = 16.5, 4.4, 0.8 Hz, 1H).

Step 2:

To a 100 mL round-bottom flask, 3-methoxy-1,3-diphenylpropan-1-one (1.20 g, 5.0 mmol), hydroxylamine hydrochloride (0.42 g, 6.0 mmol), KOAc (0.59 g, 6 mmol), and CH_3CN (15 mL) were added. The mixture was stirred at 70 °C for 5 h. The solvent was removed by rotary evaporation. The residue was purified by silica gel chromatography (petroleum: ethyl acetate = 10: 1) to provide the desired product as white solid (1.16 g, 91 %). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.82 (s, 1H), 7.58 (dq, *J* = 5.2, 1.5 Hz, 2H), 7.44 – 7.31 (m, 7H), 7.28 – 7.26 (m, 1H), 4.66 (dd, *J* = 8.8, 5.1 Hz, 1H), 3.28 (ddd, *J* = 13.7, 8.7, 1.4 Hz, 1H), 3.18 (s, 3H), 3.15 – 2.98 (m, 1H).

Synthesis of 3-hydroxy-1-phenylhexan-1-one O-methyl oxime 53

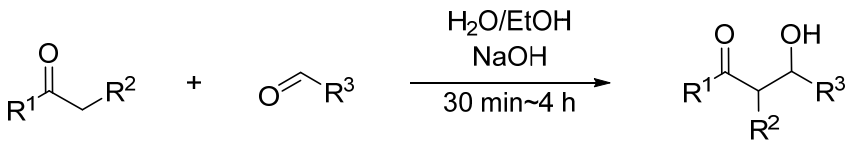


53

To a 100 mL round-bottom flask, 3-hydroxy-1-phenylhexan-1-one (0.96 g, 5.0 mmol), methoxyamine hydrochloride (0.50 g, 6.0 mmol), KOAc (0.59 g, 6 mmol), and CH_3CN (15 mL) were added. The mixture was stirred at 70 °C for 5 h. The solvent was removed by rotary evaporation. The residue was purified by silica gel chromatography (petroleum: ethyl acetate = 15: 1) to provide the desired product as white solid (0.92 g, 83 %). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.59 – 7.54 (m, 2H), 7.28 – 7.22 (m, 3H), 3.88 (s, 3H), 3.83 (s, 1H), 2.89 (dd, *J* = 13.4, 8.6 Hz, 1H), 2.78 (dd, *J* = 13.5, 4.1 Hz, 1H), 1.42 – 1.33 (m, 2H), 1.29 – 1.14 (m, 2H), 0.80 (t, *J* = 6.6 Hz, 3H).

Synthesis of β-Hydroxyl ketone

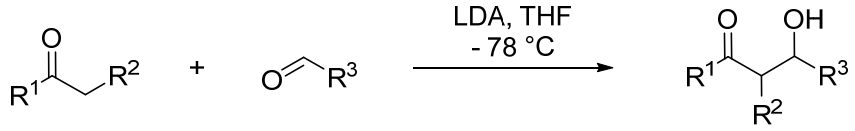
General Procedure A:



β-Hydroxyl ketone was synthesized following a literature procedure.[4] To a solution of NaOH (0.44 g, 11 mmol) in water (5 mL) and EtOH (10 mL), ketone (10 mmol) and aldehyde (13 mmol) were sequentially added at 0 °C. After stirring for 30

min~4 h (monitored by TLC), a saturated aqueous solution of NH_4Cl (15 mL) was added, and the solution was extracted with EtOAc. (3 × 15 mL). The organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel chromatography to provide the desired product.

General Procedure B:



β-Hydroxyl ketone was synthesized following a modified literature procedure.[5] LDA (2.0M in THF) (1.1 equiv.) was dissolved in dry THF and cooled to –78 °C under N_2. The ketone (1.0 equiv.) was added dropwise to the LDA solution. The reaction mixture was stirred at –78 °C for 2 h. Then aldehyde (1.1 equiv.) was added dropwise. The reaction mixture was then allowed to stir 2h at –78 °C under N_2. Then a saturated aqueous solution of NH_4Cl (35 mL) was added. The reaction was allowed to stir for 10 minutes at 0 °C. The reaction mixture was extracted with ethyl acetate (3 × 50 mL) and then washed with sodium bicarbonate, brine, and water. The organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by silica gel chromatography to provide the desired product.

Characterizations of substrates

3-hydroxy-1-phenylhexan-1-one (**1**): obtained from General Procedure B as white solid (93%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 – 7.93 (m, 2H), 7.58 (tt, *J* = 7.3, 1.2 Hz, 1H), 7.49 – 7.44 (m, 2H), 4.24 (dddd, *J* = 9.1, 7.4, 4.6, 2.6 Hz, 1H), 3.43 (br, 1H), 3.16 (ddd, *J* = 17.5, 2.7, 1.0 Hz, 1H), 3.05 (dd, *J* = 17.6, 9.1 Hz, 1H), 1.67 – 1.56 (m, 1H), 1.56 – 1.39 (m, 3H), 0.96 (t, *J* = 7.1 Hz, 3H).

3-hydroxy-1-(p-tolyl)hexan-1-one (S3): obtained from General Procedure B as white solid (91%) . ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.88 – 7.82 (m, 2H), 7.31 – 7.23 (m, 2H), 4.08 (q, *J* = 4.6 Hz, 1H), 3.48 – 3.34 (m, 1H), 3.17 – 2.95 (m, 1H), 2.41 (s, 3H), 1.77 – 1.15 (m, 4H), 0.87 (t, *J* = 7.1 Hz, 3H).

3-hydroxy-1-(4-pentylphenyl)hexan-1-one (S4): obtained from General Procedure A as white solid (59%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.83 – 7.76 (m, 2H), 7.26 – 7.13 (m, 2H), 4.20 – 4.11 (m, 1H), 3.28 (s, 1H), 3.07 (dd, *J* = 17.5, 2.6 Hz, 1H), 2.93 (dd, *J* = 17.5, 9.1 Hz, 1H), 2.66 – 2.51 (m, 2H), 1.62 – 1.51 (m, 3H), 1.48 – 1.33 (m, 3H), 1.31 – 1.19 (m, 4H), 0.88 (t, *J* = 7.1 Hz, 3H), 0.81 (t, *J* = 7.0 Hz, 3H).

3-hydroxy-1-(4-methoxyphenyl)hexan-1-one (S5): obtained from General Procedure A as white solid (52%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.06 – 7.84 (m, 2H), 7.20 – 6.42 (m, 2H), 4.09 (dtd, *J* = 15.7, 8.2, 2.9 Hz, 1H), 3.89 – 3.85 (m, 3H), 3.59 – 3.51 (m, 1H), 3.42 (d, *J* = 7.3 Hz, 1H), 1.55 – 1.48 (m, 1H), 1.38 – 1.26 (m, 3H), 0.88 (t, *J* = 7.1 Hz, 3H).

1-(4-fluorophenyl)-3-hydroxyhexan-1-one (S6): obtained from General Procedure A

as white solid (39%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.04 – 7.97 (m, 2H), 7.19 – 7.11 (m, 2H), 4.23 (dddd, *J* = 9.0, 7.6, 4.6, 2.7 Hz, 1H), 3.12 (dd, *J* = 17.5, 2.7 Hz, 1H), 3.03 (dd, *J* = 17.5, 9.0 Hz, 1H), 1.62 – 1.58 (m, 1H), 1.53 – 1.49 (m, 2H), 1.46 – 1.41 (m, 1H), 0.96 (t, *J* = 7.1 Hz, 3H).

1-(4-chlorophenyl)-3-hydroxyhexan-1-one (S7): obtained from General Procedure A as white solid (41%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.82 – 7.77 (m, 2H), 7.36 – 7.32 (m, 2H), 4.13 (dt, *J* = 8.6, 4.5 Hz, 1H), 3.18 (s, 1H), 3.04 – 2.88 (m, 2H), 1.58 – 1.48 (m, 1H), 1.45 – 1.30 (m, 3H), 0.86 (t, *J* = 7.2 Hz, 3H).

1-(4-bromophenyl)-3-hydroxyhexan-1-one (S8): obtained from General Procedure A as pale-yellow solid (63%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.83 – 7.77 (m, 2H), 7.63 – 7.57 (m, 2H), 4.22 (dddd, *J* = 8.7, 7.6, 4.5, 3.0 Hz, 1H), 3.27 (d, *J* = 25.3 Hz, 1H), 3.12 – 2.99 (m, 2H), 1.65 – 1.56 (m, 1H), 1.56 – 1.35 (m, 3H), 0.95 (t, *J* = 7.2 Hz, 3H).

3-hydroxy-1-(4-(trifluoromethyl)phenyl)hexan-1-one (S9): obtained from General Procedure A as grey solid (57%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.06 (dt, *J* = 7.9, 0.8 Hz, 2H), 7.74 (d, *J* = 8.1 Hz, 2H), 4.36 – 4.22 (m, 1H), 3.15 – 3.09 (m, 2H), 1.66 – 1.58 (m, 1H), 1.55 – 1.50 (m, 2H), 1.48 – 1.42 (m, 1H), 0.97 (t, *J* = 7.2 Hz, 3H).

3-hydroxy-1-(3-methoxyphenyl)hexan-1-one (S10): obtained from General Procedure A as white solid (61%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.49 (ddd, *J* = 7.6, 1.6, 0.9 Hz, 1H), 7.43 (dd, *J* = 2.7, 1.5 Hz, 1H), 7.27 (t, *J* = 7.9 Hz, 1H), 7.00 (ddd, *J* = 8.3, 2.7, 0.9 Hz, 1H), 3.76 (s, 3H), 3.00 (dd, *J* = 16.3, 6.9 Hz, 1H), 2.89 (dd, *J* = 16.3, 6.3 Hz, 1H), 2.67 (p, *J* = 6.5 Hz, 1H), 1.34 (ddd, *J* = 9.1, 6.7, 4.5 Hz, 2H), 1.32 – 1.24 (m, 2H), 0.80 (t, *J* = 7.1 Hz, 3H).

3-hydroxy-1-(2-methoxyphenyl)hexan-1-one (S11): obtained from General Procedure A as yellow oil (63%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.64 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.40 (ddd, *J* = 8.4, 7.3, 1.8 Hz, 1H), 6.92 (td, *J* = 7.5, 1.0 Hz, 1H), 6.89 (dd, *J* = 8.4, 1.0 Hz, 1H), 4.13 – 4.02 (m, 1H), 3.82 (s, 3H), 3.25 (s, 1H), 3.16 (dd, *J* = 17.9, 2.4 Hz, 1H), 2.94 (dd, *J* = 17.9, 9.3 Hz, 1H), 1.55 – 1.46 (m, 1H), 1.44 – 1.30 (m, 3H), 0.87 (t, *J* = 7.2 Hz, 3H).

1-(2-bromophenyl)-3-hydroxyhexan-1-one (S12): obtained from General Procedure A as yellow solid (39%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.60 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.43 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.37 (td, *J* = 7.5, 1.1 Hz, 1H), 7.31 – 7.28 (m, 1H), 4.24 – 4.19 (m, 1H), 3.19 – 3.08 (m, 2H), 3.00 (dd, *J* = 17.4, 8.9 Hz, 1H), 1.61 – 1.52 (m, 1H), 1.50 – 1.44 (m, 2H), 1.43 – 1.34 (m, 1H), 0.94 (t, *J* = 7.1 Hz, 3H).

1-(2-fluorophenyl)-3-hydroxyhexan-1-one (S13): obtained from General Procedure A as white solid (43%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.88 (td, *J* = 7.6, 1.9 Hz, 1H), 7.54 (dddd, *J* = 8.3, 7.1, 5.0, 1.9 Hz, 1H), 7.26 – 7.22 (m, 1H), 7.14 (ddd, *J* = 11.3, 8.3, 1.1 Hz, 1H), 4.28 – 4.18 (m, 1H), 3.21-3.17 (m, 2H), 3.06 (ddd, *J* = 18.2, 9.2, 3.4 Hz, 1H), 1.60 (dddd, *J* = 13.2, 11.3, 7.0, 3.1 Hz, 2H), 1.45 – 1.39 (m, 2H), 0.95 (t, *J* = 7.2 Hz, 3H).

3-hydroxy-1-(naphthalen-2-yl)hexan-1-one (S14): obtained from General Procedure A as white solid (55%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.42 (d, *J* = 1.7 Hz, 1H), 7.98 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.92 (dd, *J* = 8.2, 1.2 Hz, 1H), 7.88 – 7.78 (m, 2H), 7.57 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.52 (ddd, *J* = 8.1, 6.8, 1.3 Hz, 1H), 4.28 (qd, *J* = 7.6, 2.8 Hz, 1H), 3.42 (s, 1H), 3.24 (dd, *J* = 17.3, 2.8 Hz, 1H), 3.15 (dd, *J* = 17.3, 8.9 Hz, 1H), 1.71 – 1.60 (m, 1H), 1.59 – 1.43 (m, 3H), 0.97 (t, *J* = 7.1 Hz, 3H).

3-hydroxy-1-(naphthalen-1-yl)hexan-1-one (S15): obtained from General Procedure A as white solid (37%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.67 – 8.60 (m, 1H), 7.99 – 7.93 (m, 1H), 7.85 (td, *J* = 7.5, 1.1 Hz, 2H), 7.57 (ddd, *J* = 8.5, 6.8, 1.5 Hz, 1H), 7.51 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H), 7.46 (dd, *J* = 8.2, 7.2 Hz, 1H), 4.35 – 4.24 (m, 1H), 3.38 (s, 1H), 3.24 – 3.09 (m, 2H), 1.68 – 1.58 (m, 1H), 1.57 – 1.39 (m, 3H), 0.95 (t, *J* = 7.1 Hz, 3H).

1-(benzo[b]thiophen-2-yl)-3-hydroxyhexan-1-one (S16): obtained from General Procedure A as white solid (59%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 – 7.89 (m, 2H), 7.60 – 7.52 (m, 1H), 7.49 – 7.42 (m, 2H), 4.23 (dddd, *J* = 8.8, 7.6, 4.6, 2.8 Hz, 1H), 3.41 (br, 1H), 3.13 (dd, *J* = 17.5, 2.8 Hz, 1H), 3.05 (dd, *J* = 17.5, 8.9 Hz, 1H), 1.66 – 1.46 (m, 1H), 1.46 – 1.36 (m, 3H), 0.95 (t, *J* = 7.1 Hz, 3H).

3-hydroxy-1-(pyridin-3-yl)hexan-1-one (S17): obtained from General Procedure A as white solid (33%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 9.15 (dd, *J* = 2.3, 0.9 Hz, 1H), 8.77 (dd, *J* = 4.8, 1.7 Hz, 1H), 8.25 (dt, *J* = 7.9, 2.0 Hz, 1H), 7.44 (ddd, *J* = 7.9, 4.8, 0.9 Hz, 1H), 4.28 (tt, *J* = 7.9, 4.1 Hz, 1H), 3.26 – 3.05 (m, 2H), 1.71 – 1.58 (m, 1H), 1.56 – 1.48 (m, 2H), 1.48 – 1.40 (m, 1H), 0.96 (t, *J* = 7.1 Hz, 3H).

3-hydroxy-1-(pyridin-2-yl)hexan-1-one (S18): obtained from General Procedure A as white solid (41%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.68 (ddd, *J* = 4.8, 1.8, 0.9 Hz, 1H), 8.06 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.87 (td, *J* = 7.7, 1.7 Hz, 1H), 7.51 (ddd, *J* = 7.6, 4.8, 1.2 Hz, 1H), 4.24 – 4.14 (m, 1H), 3.40 (dd, *J* = 16.8, 2.6 Hz, 1H), 3.27 (dd, *J* = 16.8, 8.9 Hz, 1H), 1.65 – 1.56 (m, 1H), 1.56 – 1.49 (m, 2H), 1.48 – 1.42 (m, 1H), 0.95 (t, *J* = 7.1 Hz, 3H).

2-(1-hydroxybutyl)-3,4-dihydronaphthalen-1(2H)-one (S19): obtained from General Procedure A as white solid (39%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 (dt, *J* = 7.9, 1.4 Hz, 1H), 7.47 (qd, *J* = 7.6, 1.5 Hz, 1H), 7.35 – 7.25 (m, 1H), 7.23 (dd, *J* = 7.7, 1.3 Hz, 1H), 4.31 (ddd, *J* = 9.4, 4.1, 3.0 Hz, 0.5H), 4.03 (ddd, *J* = 8.2, 7.1, 2.7 Hz, 0.5H), 3.08 – 2.95 (m, 2H), 2.61 – 2.49 (m, 1H), 2.25 – 2.04 (m, 2H), 1.86 (tdd, *J* = 13.2, 11.5, 5.2 Hz, 0.5H), 1.66 – 1.52 (m, 2.5H), 1.51 – 1.34 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H).

3-(1-hydroxybutyl)chroman-4-one (S20): obtained from General Procedure A as yellow solid (55%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.87 (dt, *J* = 7.8, 2.2 Hz, 1H), 7.46 (tdd, *J* = 8.9, 7.2, 1.8 Hz, 1H), 7.00 (dddd, *J* = 10.4, 8.1, 7.2, 1.1 Hz, 1H), 6.95 (dd, *J* = 8.4, 1.0 Hz, 1H), 4.65 – 4.53 (m, 2H), 4.39 – 4.28 (m, 1H), 2.86 – 2.76 (m, 1H), 1.64 – 1.53 (m, 2H), 1.50 – 1.42 (m, 1H), 1.40 – 1.33 (m, 1H), 0.94 (t, *J* = 7.2 Hz, 3H).

3-hydroxy-1-phenylpentan-1-one (S21): obtained from General Procedure B as white

solid (87%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 – 7.91 (m, 2H), 7.60 – 7.55 (m, 1H), 7.49 – 7.44 (m, 2H), 4.14 (dtd, *J* = 12.9, 6.1, 3.6 Hz, 1H), 3.36 (s, 1H), 3.16 (dd, *J* = 17.5, 2.6 Hz, 1H), 3.04 (dd, *J* = 17.5, 9.1 Hz, 1H), 1.63 (dt, *J* = 13.7, 7.4 Hz, 1H), 1.60 – 1.52 (m, 1H), 1.01 (t, *J* = 7.4 Hz, 3H).

3-hydroxy-1-phenylbutan-1-one (S22): obtained from General Procedure A as white solid (62%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 7.7 Hz, 2H), 7.67 – 7.57 (m, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 4.51 – 4.37 (m, 1H), 3.20 (dd, *J* = 17.7, 2.8 Hz, 1H), 3.11 – 3.04 (m, 1H), 1.33 (d, *J* = 6.3 Hz, 3H).

3-hydroxy-1-phenyltetradecan-1-one (S23): obtained from General Procedure A as pale-yellow oil (56%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 – 7.92 (m, 2H), 7.60 – 7.55 (m, 1H), 7.50 – 7.44 (m, 2H), 4.22 (td, *J* = 8.0, 3.8 Hz, 1H), 3.31 (d, *J* = 2.9 Hz, 1H), 3.16 (dd, *J* = 17.6, 2.6 Hz, 1H), 3.04 (dd, *J* = 17.5, 9.0 Hz, 1H), 1.66 – 1.58 (m, 1H), 1.51 (dp, *J* = 10.7, 4.1 Hz, 2H), 1.32 – 1.24 (m, 16H), 0.88 (t, *J* = 7.0 Hz, 3H).

7-chloro-3-hydroxy-1-phenylheptan-1-one (S24): obtained from General Procedure A as yellow oil (33%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 – 7.93 (m, 2H), 7.59 (ddt, *J* = 8.7, 7.0, 1.3 Hz, 1H), 7.50 – 7.44 (m, 2H), 4.23 (dddd, *J* = 9.1, 7.5, 4.4, 2.7 Hz, 1H), 3.56 (t, *J* = 6.7 Hz, 2H), 3.17 (dd, *J* = 17.6, 2.7 Hz, 1H), 3.06 (dd, *J* = 17.6, 9.0 Hz, 1H), 1.89 – 1.78 (m, 2H), 1.73 – 1.61 (m, 2H), 1.60 – 1.52 (m, 2H).

5-(benzo[d][1,3]dioxol-5-yl)-3-hydroxy-4-methyl-1-phenylpentan-1-one (S25): obtained from General Procedure A as white solid (63%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.94 (ddd, *J* = 10.8, 8.3, 1.3 Hz, 2H), 7.60 – 7.55 (m, 1H), 7.46 (dt, *J* = 8.3, 7.4 Hz, 2H), 6.75 – 6.69 (m, 2H), 6.64 (td, *J* = 7.9, 1.7 Hz, 1H), 5.89 (d, *J* = 3.2 Hz, 2H), 4.17 (dt, *J* = 9.3, 2.9 Hz, 0.57H), 4.13 – 4.06 (m, 0.46H), 3.19 (dd, *J* = 17.4, 2.3 Hz, 0.50H), 3.15 – 3.03 (m, 1.59H), 2.92 (dd, *J* = 13.6, 4.6 Hz, 0.51H), 2.83 (dd, *J* = 13.5, 6.5 Hz, 0.58H), 2.43 (dd, *J* = 13.4, 8.4 Hz, 0.58H), 2.36 (dd, *J* = 13.6, 9.4 Hz, 0.46H), 1.99 – 1.90 (m, 0.51H), 1.83 (dddd, *J* = 13.5, 6.7, 3.4, 1.6 Hz, 0.58H), 0.96 (d, *J* = 6.8 Hz, 1.63H), 0.90 (d, *J* = 6.8 Hz, 1.41H).

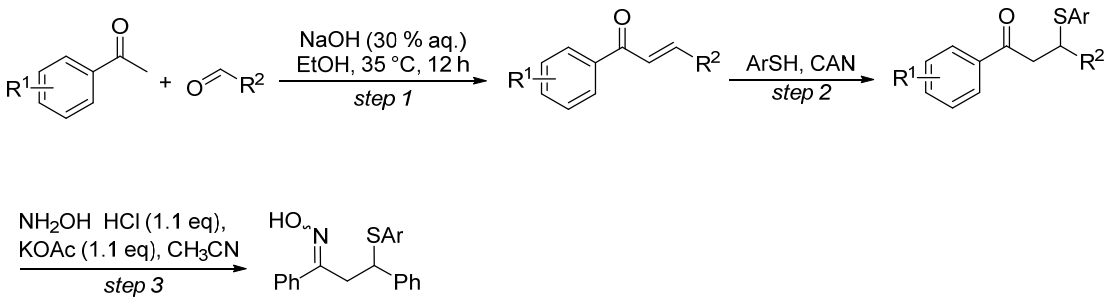
3-hydroxy-1-phenyl-3-(tetrahydrofuran-3-yl)propan-1-one (S26): obtained from General Procedure A as yellow oil (43%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 – 7.87 (m, 2H), 7.61 – 7.53 (m, 1H), 7.46 (dtd, *J* = 13.9, 7.7, 2.6 Hz, 2H), 5.36 (ddq, *J* = 12.5, 6.5, 2.2 Hz, 0.4H), 4.43 (dd, *J* = 10.2, 7.5 Hz, 0.1H), 4.35 (ddd, *J* = 10.1, 7.7, 4.2 Hz, 0.33H), 4.27 – 4.07 (m, 0.83H), 4.02 – 3.73 (m, 2H), 3.71 – 3.58 (m, 1H), 3.55 – 3.41 (m, 0.7H), 3.32 – 3.16 (m, 0.52H), 3.14 – 3.06 (m, 0.60H), 3.06 – 2.90 (m, 0.51H), 2.63 – 2.34 (m, 1.32H), 2.20 (dq, *J* = 16.3, 7.7 Hz, 0.29H), 2.11 – 1.92 (m, 0.49H), 1.91 – 1.84 (m, 0.29H), 1.69 – 1.56 (m, 0.58H).

3-hydroxy-1,4-diphenylbutan-1-one (S27): obtained from General Procedure A as white solid (61%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 – 7.90 (m, 2H), 7.64 – 7.57 (m, 1H), 7.49 (t, *J* = 7.7 Hz, 2H), 7.39 – 7.27 (m, 2H), 7.29 – 7.21 (m, 3H), 4.52 (tt, *J* = 7.2, 3.6 Hz, 1H), 3.19 – 3.12 (m, 2H), 3.02 – 2.98 (m, 1H), 2.88 (dd, *J* = 13.5, 6.4 Hz, 1H).

3-hydroxy-1,3-diphenylpropan-1-one (S28): obtained from General Procedure B as

white solid (89%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.95 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.61 – 7.56 (m, 1H), 7.49 – 7.42 (m, 4H), 7.38 (dd, *J* = 8.5, 6.9 Hz, 2H), 7.32 – 7.29 (m, 1H), 5.35 (td, *J* = 6.1, 2.7 Hz, 1H), 3.60 (d, *J* = 3.0 Hz, 1H), 3.44 – 3.28 (m, 2H).

Synthesis of β-arylthioketone oximes



Step 1:

Following a modified literature procedure,[6] to a 20 mL vial equipped with a magnetic stirrer bar were added the corresponding aldehyde (10.0 mmol, 1.0 equiv.), acetophenone (10.0 mmol, 1.0 equiv.), and ethanol (9.0 mL). NaOH (30% aq., 10.0 mL) was then added to the resulting solution and subsequently stirred for 12 h at room temperature. The reaction mixture was extracted with EtOAc, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The resulting residue was purified by column chromatography on silica gel to afford the corresponding chalcones.

Step 2:

Following a modified literature procedure,[7] In a 50 mL round bottom flask were added chalcone (5 mmol) and thiophenol (5.5 mmol), ceric ammonium nitrate (CAN) (0.5 mmol) and CH_3CN (2 mL). The mixture was stirred at 50 °C for 2 h. After completion of the reaction (monitored by TLC), the crude product was purified by silica gel column chromatography to give the pure product.

Step 3:

To a 100 mL round-bottom flask, ketone (2 mmol), hydroxylamine hydrochloride (0.15 g, 2.2 mmol), KOAc (0.59 g, 6 mmol), and CH_3CN (15 mL) were added. The mixture was stirred at 70 °C for 5 h. The solvent was removed by rotary evaporation. The residue was purified by silica gel chromatography to provide the desired product.

Characterizations of substrates

3-(naphthalen-2-ylthio)-3-phenyl-1-(p-tolyl)propan-1-one (29): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 – 7.75 (m, 3H), 7.69 (t, *J* = 9.1 Hz, 2H), 7.46 – 7.42 (m, 2H), 7.42 – 7.39 (m, 1H), 7.38 – 7.35 (m, 2H), 7.26 – 7.22 (m, 2H), 7.19 (t, *J* = 8.6 Hz, 3H), 5.08 (dd, *J* = 8.0, 6.1 Hz, 1H), 3.62 (qd, *J* = 17.1, 7.1 Hz, 2H), 2.38 (s, 3H).

3-(naphthalen-2-ylthio)-3-phenyl-1-(p-tolyl)propan-1-one oxime (30): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (q, *J* = 3.6 Hz, 2H), 7.56 – 7.51 (m, 2H), 7.32 (dt, *J* = 6.3, 3.4 Hz, 2H), 7.23 – 7.17 (m, 3H), 7.13 (d, *J* = 8.1 Hz, 2H), 7.11 – 7.04 (m, 3H), 7.00

(d, *J* = 7.8 Hz, 2H), 4.69 (dd, *J* = 8.6, 7.1 Hz, 1H), 3.41 (dd, *J* = 13.8, 7.1 Hz, 1H), 3.34 (dd, *J* = 13.8, 8.7 Hz, 1H), 2.25 (s, 3H).

3-(4-methoxyphenyl)-3-(phenylthio)-1-(p-tolyl)propan-1-one (S31): white solid (92%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 – 7.74 (m, 2H), 7.36 – 7.31 (m, 2H), 7.25 (d, *J* = 8.7 Hz, 2H), 7.22 – 7.16 (m, 5H), 6.76 (d, *J* = 8.7 Hz, 2H), 4.93 (dd, *J* = 8.5, 5.6 Hz, 1H), 3.71 (s, 3H), 3.59 (dd, *J* = 17.0, 8.6 Hz, 1H), 3.50 (dd, *J* = 17.0, 5.7 Hz, 1H), 2.35 (s, 3H).

3-(4-methoxyphenyl)-3-(phenylthio)-1-(p-tolyl)propan-1-one oxime (S32): white solid (80%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 9.27 (s, 1H), 7.25 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.21 – 7.19 (m, 2H), 7.18 – 7.15 (m, 3H), 7.15 – 7.12 (m, 2H), 7.09 (d, *J* = 7.9 Hz, 2H), 6.70 (d, *J* = 8.7 Hz, 2H), 4.59 (dd, *J* = 8.7, 7.0 Hz, 1H), 3.71 (s, 3H), 3.43 – 3.35 (m, 2H), 3.43 – 3.35 (m, 3H).

1-(4-chlorophenyl)-3-(4-methoxyphenyl)-3-(phenylthio)propan-1-one (S33): white solid (76%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.5 Hz, 2H), 7.42 (d, *J* = 8.5 Hz, 2H), 7.37 – 7.32 (m, 2H), 7.30 – 7.23 (m, 5H), 6.81 (d, *J* = 8.7 Hz, 2H), 4.92 (dd, *J* = 8.4, 5.8 Hz, 1H), 3.78 (s, 3H), 3.61 (dd, *J* = 17.0, 8.4 Hz, 1H), 3.52 (dd, *J* = 17.0, 5.8 Hz, 1H).

1-(4-chlorophenyl)-3-(4-methoxyphenyl)-3-(phenylthio)propan-1-one oxime (S34): pale-yellow solid (81%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.20 (m, 10H), 7.19 – 7.13 (m, 2H), 6.76 – 6.72 (m, 2H), 4.56 (t, *J* = 7.9 Hz, 1H), 3.77 (d, *J* = 1.1 Hz, 3H), 3.49 – 3.35 (m, 2H).

1-(4-bromophenyl)-3-(4-methoxyphenyl)-3-(phenylthio)propan-1-one (S35): white solid (76%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.72 – 7.67 (m, 2H), 7.55 – 7.51 (m, 2H), 7.34 – 7.30 (m, 2H), 7.27 – 7.18 (m, 5H), 6.80 – 6.74 (m, 2H), 4.89 (dd, *J* = 8.4, 5.7 Hz, 1H), 3.72 (s, 3H), 3.56 (dd, *J* = 17.0, 8.5 Hz, 1H), 3.48 (dd, *J* = 17.0, 5.7 Hz, 1H).

1-(4-bromophenyl)-3-(4-methoxyphenyl)-3-(phenylthio)propan-1-one oxime (S36): white solid (66%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.72 (s, 1H), 7.42 – 7.38 (m, 2H), 7.27 – 7.24 (m, 2H), 7.21 – 7.10 (m, 7H), 6.70 (d, *J* = 8.6 Hz, 2H), 4.53 (t, *J* = 7.9 Hz, 1H), 3.73 (s, 3H), 3.36 (d, *J* = 7.9 Hz, 2H).

3-(4-methoxyphenyl)-3-(phenylthio)-1-(p-tolyl)propan-1-one (S37): white solid (63%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.11 (t, *J* = 1.8 Hz, 2H), 7.95 (t, *J* = 1.8 Hz, 1H), 7.90 (ddd, *J* = 7.8, 1.7, 1.1 Hz, 2H), 7.80 (s, 1H), 7.77 (s, 1H), 7.76 – 7.74 (m, 1H), 7.66 (ddd, *J* = 7.9, 2.0, 1.0 Hz, 2H), 7.62 (ddd, *J* = 7.9, 2.0, 1.0 Hz, 1H), 7.49 – 7.46 (m, 1H), 7.31 (d, *J* = 1.3 Hz, 1H), 4.89 (dd, *J* = 8.4, 5.7 Hz, 1H), 3.73 (s, 3H), 3.57 (dd, *J* = 17.1, 8.5 Hz, 1H), 3.49 (dd, *J* = 17.1, 5.7 Hz, 1H).

1-(3-bromophenyl)-3-(4-methoxyphenyl)-3-(phenylthio)propan-1-one oxime (S38): yellow solid (71%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 9.30 (s, 1H), 7.35 – 7.29 (m, 1H), 7.24 (q, *J* = 2.0 Hz, 1H), 7.19 (dt, *J* = 8.2, 2.0 Hz, 2H), 7.14 – 7.07 (m, 4H), 7.06 – 6.98 (m, 3H), 6.64 – 6.58 (m, 2H), 4.50 (dd, *J* = 9.4, 6.2 Hz, 1H), 3.62 (s, 3H), 3.30

(dd, *J* = 13.7, 6.2 Hz, 1H), 3.21 (ddd, *J* = 12.3, 9.2, 2.4 Hz, 1H).

1-(2-chlorophenyl)-3-(4-methoxyphenyl)-3-(phenylthio)propan-1-one (S39): yellow solid (72%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.50 – 7.46 (m, 1H), 7.34 – 7.31 (m, 1H), 7.30 – 7.27 (m, 3H), 7.21 – 7.19 (m, 4H), 7.17 (d, *J* = 8.7 Hz, 2H), 6.77 – 6.74 (m, 2H), 4.79 (dd, *J* = 8.3, 6.6 Hz, 1H), 3.72 (s, 3H), 3.61 – 3.50 (m, 2H).

1-(2-chlorophenyl)-3-(4-methoxyphenyl)-3-(phenylthio)propan-1-one oxime (S40): white solid (62%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.31 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.23 – 7.21 (m, 3H), 7.19 – 7.16 (m, 4H), 7.03 (td, *J* = 7.5, 1.2 Hz, 1H), 7.02 – 6.99 (m, 1H), 6.70 (dd, *J* = 7.6, 1.7 Hz, 1H), 6.68 – 6.66 (m, 2H), 4.48 (dd, *J* = 9.5, 6.7 Hz, 1H), 3.72 (s, 3H), 3.55 (dd, *J* = 14.8, 6.8 Hz, 1H), 3.41 (dd, *J* = 14.7, 9.5 Hz, 1H).

3-(4-methoxyphenyl)-3-(naphthalen-2-ylthio)-1-(o-tolyl)propan-1-one (S41): white solid (63%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 – 7.78 (m, 2H), 7.73 (dd, *J* = 8.5, 5.8 Hz, 2H), 7.52 – 7.47 (m, 3H), 7.44 – 7.39 (m, 2H), 7.37 (t, *J* = 7.4 Hz, 1H), 7.30 – 7.18 (m, 3H), 6.84 – 6.79 (m, 2H), 5.00 (t, *J* = 7.4 Hz, 1H), 3.78 (s, 3H), 3.60 – 3.53 (m, 2H), 2.34 (s, 3H).

3-(4-methoxyphenyl)-3-(naphthalen-2-ylthio)-1-(o-tolyl)propan-1-one oxime (S42): yellow solid (61%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.69 – 7.65 (m, 1H), 7.60 (d, *J* = 1.8 Hz, 0.38H), 7.58 – 7.54 (m, 1.67H), 7.37 – 7.33 (m, 1.45H), 7.26 – 7.19 (m, 1.55H), 7.19 – 7.15 (m, 1.72H), 7.14 – 7.10 (m, 1.28H), 7.05 (dd, *J* = 14.2, 7.9 Hz, 1.50H), 7.02 – 6.97 (m, 1.51H), 6.75 (td, *J* = 8.2, 1.8 Hz, 1.34H), 6.68 (d, *J* = 8.6 Hz, 0.66H), 6.62 (d, *J* = 8.7 Hz, 1H), 4.49 (dd, *J* = 9.4, 6.6 Hz, 0.4H), 4.39 – 4.31 (m, 0.36H), 3.74 – 3.68 (m, 1H), 3.68 – 3.62 (m, 2H), 3.40 (dd, *J* = 14.5, 9.5 Hz, 0.5H), 3.33 (dd, *J* = 14.5, 6.6 Hz, 0.5H), 3.15 – 3.03 (m, 1H), 2.17 (d, *J* = 23.4 Hz, 1H), 2.02 – 1.93 (m, 2H).

3-(4-methoxyphenyl)-3-(naphthalen-2-ylthio)-1-(4-pentylphenyl)propan-1-one (S43): white solid (81%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.1 Hz, 3H), 7.73 (dd, *J* = 8.1, 5.4 Hz, 2H), 7.51 – 7.43 (m, 4H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 6.82 (d, *J* = 8.5 Hz, 2H), 5.11 (dd, *J* = 8.3, 5.8 Hz, 1H), 3.77 (s, 3H), 3.74 – 3.55 (m, 2H), 2.66 (t, *J* = 7.7 Hz, 2H), 1.75 – 1.58 (m, 2H), 1.42 – 1.28 (m, 4H), 0.92 (t, *J* = 6.8 Hz, 3H).

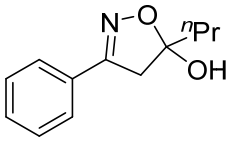
3-(4-methoxyphenyl)-3-(naphthalen-2-ylthio)-1-(4-pentylphenyl)propan-1-one oxime (S44): white solid (69%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 – 7.75 (m, 2H), 7.69 (dd, *J* = 7.4, 3.8 Hz, 2H), 7.50 – 7.44 (m, 2H), 7.34 (d, *J* = 8.7 Hz, 1H), 7.26 (d, *J* = 7.9 Hz, 2H), 7.22 – 7.18 (m, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 6.74 (d, *J* = 8.3 Hz, 2H), 4.71 (d, *J* = 7.9 Hz, 1H), 3.76 (d, *J* = 1.1 Hz, 3H), 3.47 (dd, *J* = 8.0, 3.6 Hz, 2H), 2.61 (t, *J* = 7.8 Hz, 2H), 1.62 (p, *J* = 7.4 Hz, 2H), 1.46 – 1.26 (m, 4H), 0.92 (t, *J* = 6.7 Hz, 3H).

3-(4-methoxyphenyl)-1-(naphthalen-2-yl)-3-(naphthalen-2-ylthio)propan-1-one (S45): white solid (76%).^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 – 7.84 (m, 3H), 7.81 (dt, *J* = 9.5, 3.3 Hz, 2H), 7.77 – 7.69 (m, 3H), 7.68 – 7.62 (m, 2H), 7.48 (td, *J* = 5.7, 2.3 Hz, 4H), 7.37 (d, *J* = 8.4 Hz, 2H), 6.82 (d, *J* = 8.3 Hz, 2H), 5.15 (dd, *J* = 8.0,

6.2 Hz, 1H), 3.83 – 3.74 (m, 5H).

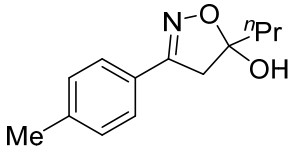
3-(4-methoxyphenyl)-1-(naphthalen-2-yl)-3-(naphthalen-2-ylthio)propan-1-one oxime (S46): pale yellow solid (59%). ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.59 (s, 1H), 7.86 (d, J = 7.7 Hz, 4H), 7.80 (d, J = 8.4 Hz, 3H), 7.68 (d, J = 8.5 Hz, 1H), 7.49 (tt, J = 9.7, 2.7 Hz, 5H), 7.41 – 7.37 (m, 1H), 7.32 (d, J = 8.2 Hz, 2H), 6.74 (d, J = 8.2 Hz, 2H), 4.93 (t, J = 7.8 Hz, 1H), 3.61 (s, 3H), 3.51 (d, J = 7.8 Hz, 2H).

Characterizations of Products



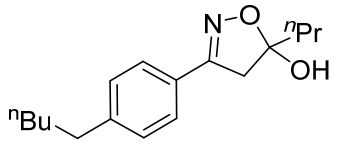
2

3-phenyl-5-propyl-4,5-dihydroisoxazol-5-ol (2).[8] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 5: 1).
White solid (32.6 mg, 87% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.62 – 7.57 (m, 2H), 7.39 – 7.32 (m, 3H), 4.22 (s, 1H), 3.31 – 3.11 (m, 2H), 1.94 (ddd, J = 13.9, 11.3, 5.2 Hz, 1H), 1.87 (ddd, J = 14.0, 11.2, 5.3 Hz, 1H), 1.56 – 1.42 (m, 2H), 0.95 (t, J = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.4, 130.2, 129.6, 128.7, 126.7, 109.0, 44.4, 40.3, 18.1, 14.2;
HRMS (ESI): Calcd for $C_{12}H_{16}NO_2$ $[M+H]^+$: 206.1176; found 206.1170.



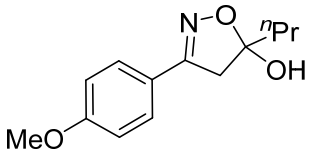
3

5-propyl-3-(p-tolyl)-4,5-dihydroisoxazol-5-ol (3). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (36.5 mg, 83% yield).
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 – 7.54 (m, 2H), 7.20 (d, J = 7.9 Hz, 2H), 3.25 (d, J = 0.8 Hz, 2H), 2.81 (s, 1H), 2.38 (s, 3H), 2.09 – 1.89 (m, 2H), 1.69 – 1.48 (m, 2H), 1.01 (t, J = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.4, 140.5, 129.4, 126.8, 126.6, 108.7, 44.6, 40.5, 21.5, 18.1, 14.2.;
HRMS (ESI): Calcd for $C_{13}H_{18}NO_2$ $[M+H]^+$: 220.1332; found 220.1337.



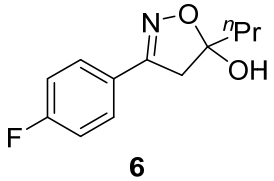
4

3-(4-pentylphenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (4). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
White solid (44.6 mg, 81% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.57 – 7.52 (m, 2H), 7.21 – 7.17 (m, 2H), 3.41 (s, 1H), 3.28 – 3.19 (m, 2H), 2.61 (t, *J* = 7.7 Hz, 2H), 2.00 – 1.87 (m, 2H), 1.66 – 1.58 (m, 2H), 1.57 – 1.47 (m, 2H), 1.35 – 1.31 (m, 4H), 0.98 (t, *J* = 7.4 Hz, 3H), 0.89 (t, *J* = 6.9 Hz, 3H);
^{13}C NMR (151 MHz, $CDCl_3$) δ 157.4, 145.5, 128.8, 126.9, 126.7, 108.7, 44.6, 40.4, 35.8, 31.4, 30.9, 22.5, 18.1, 14.2, 14.0;
HRMS (ESI): Calcd for $C_{17}H_{26}NO_2$ $[M+H]^+$: 276.1958; found 276.1962.



5

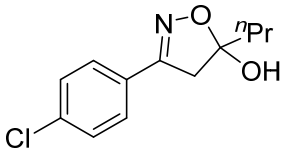
3-(4-methoxyphenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (5). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 3: 1).
White solid (39.8 mg, 85% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.8 Hz, 2H), 6.89 (d, *J* = 8.9 Hz, 2H), 3.83 (s, 3H), 3.49 – 3.37 (m, 1H), 3.22 (s, 2H), 1.99 – 1.87 (m, 2H), 1.63 – 1.46 (m, 2H), 0.99 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.1, 157.0, 128.3, 122.2, 114.1, 108.6, 55.4, 44.7, 40.4, 18.1, 14.2;
HRMS (ESI): Calcd for $C_{13}H_{18}NO_3$ $[M+H]^+$: 236.1281; found 236.1289.



6

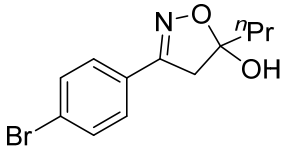
3-(4-fluorophenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (6). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
White solid (32.2 mg, 72% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.70 – 7.58 (m, 2H), 7.08 (t, *J* = 8.7 Hz, 2H), 3.39 (s, 1H), 3.23 (s, 2H), 2.01 – 1.89 (m, 2H), 1.59 – 1.47 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 163.8 (d, *J* = 207.5 Hz), 156.4, 128.7 (d, *J* = 7.5

Hz), 125.8(d, *J* = 2.5 Hz), 115.8 (d, *J* = 18.7 Hz), 109.0, 44.5, 40.4, 18.1, 14.1;
^{19}F NMR (565 MHz, Chloroform-*d*) δ -109.70;
HRMS (ESI): Calcd for $C_{12}H_{14}FNNaO_2$ $[M+Na]^+$: 246.0901; found 246.0906.



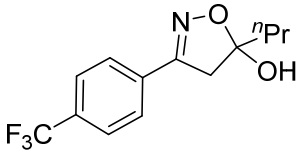
7

3-(4-chlorophenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (7). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
White solid (36.8 mg, 77% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.6 Hz, 2H), 7.33 (d, *J* = 8.6 Hz, 2H), 3.94 (s, 1H), 3.26 – 3.15 (m, 2H), 1.92 (dddd, *J* = 38.5, 14.0, 11.0, 5.2 Hz, 2H), 1.56 – 1.44 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).;
^{13}C NMR (151 MHz, Chloroform-*d*) δ 156.5, 136.2, 129.0, 128.1, 127.9, 109.2, 44.2, 40.3, 18.1, 14.1;
HRMS (ESI): Calcd for $C_{12}H_{15}ClNO_2$ $[M+H]^+$: 240.0786; found 240.0788.



8

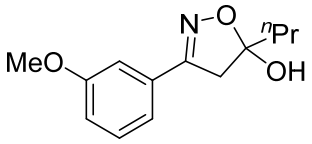
3-(4-bromophenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (8). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
Pale-yellow solid (44.6 mg, 79% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.39 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H), 4.41 – 4.17 (m, 1H), 3.17 – 3.05 (m, 2H), 1.92 – 1.74 (m, 2H), 1.53 – 1.34 (m, 2H), 0.87 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 156.6, 131.9, 128.5, 128.1, 124.5, 109.3, 44.1, 40.3, 18.1, 14.1.;
HRMS (ESI): Calcd for $C_{12}H_{15}BrNO_2$ $[M+H]^+$: 284.0281, 286.0261; found 284.0289, 286.0265.



9

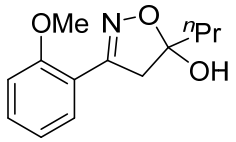
5-propyl-3-(4-(trifluoromethyl)phenyl)-4,5-dihydroisoxazol-5-ol (9). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
White solid (33.8 mg, 62% yield).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.81 – 7.71 (m, 2H), 7.64 (d, *J* = 8.2 Hz, 2H), 3.49 (s, 1H), 3.26 (s, 2H), 2.03 – 1.91 (m, 2H), 1.61 – 1.46 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 156.3, 133.0, 131.8 (q, *J* = 32.7 Hz), 126.9, 125.7 (q, *J* = 3.8 Hz), 123.8 (q, *J* = 272.3 Hz), 109.5, 44.0, 40.4, 18.1, 14.1;
^{19}F NMR (565 MHz, Chloroform-*d*) δ -62.92;
HRMS (ESI): Calcd for $C_{13}H_{15}F_3NO_2$ $[M+H]^+$: 274.1049; found 274.1041.



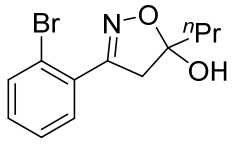
10

3-(3-methoxyphenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (10). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 3: 1).
White solid (33.3 mg, 71% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.31 – 7.27 (m, 1H), 7.25 (dd, *J* = 2.6, 1.5 Hz, 1H), 7.15 (ddd, *J* = 7.6, 1.5, 1.0 Hz, 1H), 6.95 (ddd, *J* = 8.3, 2.6, 0.9 Hz, 1H), 3.83 (s, 3H), 3.24 (s, 2H), 3.22 – 3.17 (m, 1H), 1.95 (qdd, *J* = 14.0, 10.8, 5.6 Hz, 2H), 1.61 – 1.48 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.7, 157.3, 130.8, 129.7, 119.4, 116.7, 111.2, 108.9, 55.4, 44.5, 40.5, 18.1, 14.1;
HRMS (ESI): Calcd for $C_{13}H_{18}NO_3$ $[M+H]^+$: 236.1281; found 236.1282.



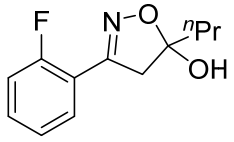
11

3-(2-methoxyphenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (11). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 2: 1).
White solid (29.6 mg, 63% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.68 (d, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.9 Hz, 1H), 6.90 (t, *J* = 7.4 Hz, 1H), 6.85 (d, *J* = 8.2 Hz, 1H), 3.78 (d, *J* = 4.0 Hz, 3H), 3.36 – 3.27 (m, 2H), 2.89 (s, 1H), 1.86 (tt, *J* = 13.6, 7.0 Hz, 2H), 1.55 – 1.40 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.5, 156.8, 131.4, 129.3, 120.9, 118.8, 111.4, 108.4, 55.5, 47.4, 40.4, 18.1, 14.2;
HRMS (ESI): Calcd for $C_{13}H_{18}NO_3$ $[M+H]^+$: 236.1281; found 236.1286.



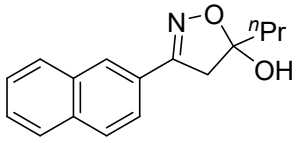
12

3-(2-bromophenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (12). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 5: 1).
White solid (26.1 mg, 46% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.52 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.47 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.25 (td, *J* = 7.5, 1.3 Hz, 1H), 7.17 (td, *J* = 7.7, 1.8 Hz, 1H), 3.67 (s, 1H), 3.42 (d, *J* = 17.8 Hz, 1H), 3.25 (d, *J* = 17.7 Hz, 1H), 1.92 – 1.83 (m, 2H), 1.51 – 1.40 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.3, 133.7, 131.2, 131.1, 131.0, 127.6, 121.8, 109.4, 47.0, 40.3, 18.1, 14.2;
HRMS (ESI): Calcd for $C_{12}H_{15}BrNO_2$ $[M+H]^+$: 284.0281, 286.0261; found 284.0280, 286.0269.



13

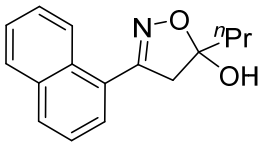
3-(2-fluorophenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (13). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
White solid (13.8 mg, 31% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.87 (td, *J* = 7.6, 1.8 Hz, 1H), 7.42 – 7.35 (m, 1H), 7.17 (td, *J* = 7.5, 1.2 Hz, 1H), 7.10 (ddd, *J* = 11.3, 8.4, 1.2 Hz, 1H), 3.41 – 3.32 (m, 2H), 3.17 (br, 1H), 1.96 (qdd, *J* = 14.0, 10.8, 5.6 Hz, 2H), 1.60 – 1.50 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 160.4 (d, *J* = 252.4 Hz), 154.0 (d, *J* = 2.9 Hz), 131.9 (d, *J* = 8.7 Hz), 128.9 (d, *J* = 3.2 Hz), 124.5 (d, *J* = 3.3 Hz), 117.7 (d, *J* = 11.5 Hz), 116.4 (d, *J* = 22.2 Hz), 108.9 (d, *J* = 2.7 Hz), 46.3 (d, *J* = 6.9 Hz), 40.4, 18.1, 14.1;
^{19}F NMR (565 MHz, Chloroform-*d*) δ -112.85;
HRMS (ESI): Calcd for $C_{12}H_{15}FNO_2$ $[M+H]^+$: 224.1081; found 224.1077.



14

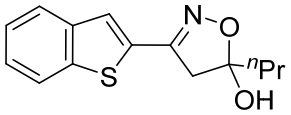
3-(naphthalen-2-yl)-5-propyl-4,5-dihydroisoxazol-5-ol (14). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 5: 1).
White solid (38.3 mg, 75% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.88 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.81 – 7.80 (m, 1H), 7.79 – 7.77 (m, 1H), 7.77 – 7.75 (m, 2H), 7.47 (dddd, *J* = 18.1, 8.1, 6.9, 1.4 Hz, 2H), 3.83 (s, 1H), 3.42 – 3.26 (m, 2H), 2.04 – 1.88 (m, 2H), 1.60 – 1.48 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.6, 134.1, 132.9, 128.5, 128.4, 127.8, 127.2, 127.1, 127.1, 126.7, 123.3, 109.1, 44.4, 40.4, 18.2, 14.2;

HRMS (ESI): Calcd for $C_{16}H_{18}NO_2$ $[M+H]^+$: 256.1332; found 256.1335.



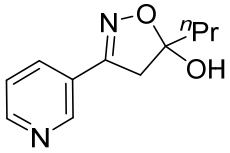
15

3-(naphthalen-1-yl)-5-propyl-4,5-dihydroisoxazol-5-ol (15). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 5: 1).
White solid (29.3 mg, 53% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.95 (dt, *J* = 8.6, 1.0 Hz, 1H), 7.89 (ddt, *J* = 13.3, 8.1, 1.0 Hz, 2H), 7.60 (ddd, *J* = 8.4, 6.8, 1.4 Hz, 1H), 7.57 – 7.52 (m, 2H), 7.47 (dd, *J* = 8.1, 7.2 Hz, 1H), 3.47 (q, *J* = 17.1 Hz, 2H), 2.82 (s, 1H), 2.08 – 1.99 (m, 2H), 1.69 – 1.60 (m, 2H), 1.05 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.2, 134.0, 131.0, 130.6, 128.6, 127.8, 127.6, 127.0, 126.4, 124.8, 107.5, 47.5, 40.4, 18.2, 14.2;
HRMS (ESI): Calcd for $C_{16}H_{17}NNaO_2$ $[M+H]^+$: 278.1151; found 278.1153.



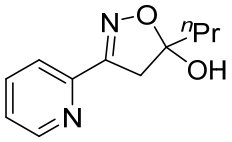
16

3-(benzo[b]thiophen-2-yl)-5-propyl-4,5-dihydroisoxazol-5-ol (16). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
Yellow solid (20.3 mg, 39% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.83 – 7.78 (m, 1H), 7.76 – 7.71 (m, 1H), 7.41 – 7.32 (m, 3H), 3.34 (s, 2H), 3.16 (d, *J* = 11.6 Hz, 1H), 1.98 (qdd, *J* = 14.0, 10.7, 5.6 Hz, 2H), 1.61 – 1.50 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 153.7, 140.4, 139.0, 132.4, 126.1, 125.8, 124.7, 124.2, 122.5, 109.6, 44.8, 40.4, 18.1, 14.1.;
HRMS (ESI): Calcd for $C_{14}H_{16}NO_2S$ $[M+H]^+$: 262.0896; found 262.0898.



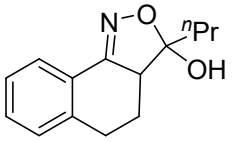
17

5-propyl-3-(pyridin-3-yl)-4,5-dihydroisoxazol-5-ol (17). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 2: 1).
White solid (18.9 mg, 46% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.63 (d, *J* = 2.2 Hz, 1H), 8.48 (dd, *J* = 4.9, 1.6 Hz, 1H), 7.91 (dt, *J* = 8.0, 1.9 Hz, 1H), 7.23 (dd, *J* = 8.0, 4.8 Hz, 1H), 3.16 (s, 2H), 1.93 (ddd, *J* = 14.0, 11.2, 5.3 Hz, 1H), 1.86 (ddd, *J* = 14.0, 11.1, 5.3 Hz, 1H), 1.55 – 1.41 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 154.6, 150.4, 147.2, 133.9, 126.3, 123.8, 109.6, 43.7, 40.4, 18.2, 14.2.;
HRMS (ESI): Calcd for $C_{11}H_{15}N_2O_2$ $[M+H]^+$: 207.1128; found 207.1129.



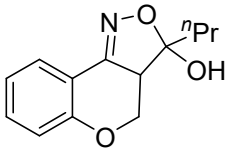
18

5-propyl-3-(pyridin-2-yl)-4,5-dihydroisoxazol-5-ol (18). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 2: 1).
White solid (16.9 mg, 41% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.54 (ddd, *J* = 5.0, 1.8, 0.9 Hz, 1H), 7.91 – 7.86 (m, 1H), 7.69 – 7.63 (m, 1H), 7.26 (ddd, *J* = 7.5, 4.9, 1.2 Hz, 1H), 5.05 (s, 1H), 3.45 (d, *J* = 18.2 Hz, 1H), 3.34 (d, *J* = 18.2 Hz, 1H), 2.05 – 1.88 (m, 2H), 1.67 – 1.46 (m, 2H), 0.98 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.6, 149.1, 149.0, 136.6, 124.4, 121.6, 110.0, 43.8, 40.3, 18.1, 14.2;
HRMS (ESI): Calcd for $C_{11}H_{15}N_2O_2$ $[M+H]^+$: 207.1128; found 207.1122.



19

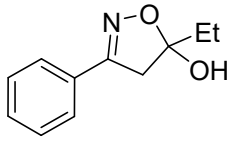
3-propyl-3,3a,4,5-tetrahydronaphtho[1,2-c]isoxazol-3-ol (19). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (32.8 mg, 71% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.94 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.33 – 7.27 (m, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 7.7 Hz, 1H), 3.39 (s, 1H), 3.10 (dd, *J* = 13.4, 5.1 Hz, 1H), 2.96 (ddd, *J* = 16.6, 4.8, 2.4 Hz, 1H), 2.88 (ddd, *J* = 16.8, 12.8, 4.9 Hz, 1H), 2.11 – 2.04 (m, 1H), 2.06 – 1.98 (m, 1H), 1.98 – 1.93 (m, 2H), 1.63 – 1.50 (m, 2H), 0.99 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 158.1, 139.1, 130.5, 128.9, 126.7, 125.7, 125.3, 107.9, 52.6, 40.2, 29.7, 22.2, 17.6, 14.3;
HRMS (ESI): Calcd for $C_{14}H_{18}NO_2$ $[M+H]^+$: 232.1332; found 232.1330.



20

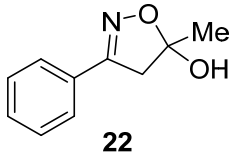
3-propyl-3a,4-dihydro-3H-chromeno[4,3-c]isoxazol-3-ol (20).[8] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 5: 1).
White solid (35.4 mg, 76% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.86 (td, *J* = 7.4, 6.8, 1.8 Hz, 0.30H), 7.80 (dd, *J* = 7.8, 1.7 Hz, 0.68H), 7.47 (tdd, *J* = 8.8, 7.1, 1.8 Hz, 0.30H), 7.35 – 7.29 (m, 0.71H), 7.04 – 6.91 (m, 2H), 4.61 – 4.56 (m, 0.38H), 4.53 (dt, *J* = 10.9, 5.8 Hz, 0.72H), 4.34 (t, *J* = 11.3 Hz, 0.32H), 4.28 (dd, *J* = 13.0, 10.9 Hz, 0.70H), 4.02 (td, *J* = 8.1, 7.6, 3.2 Hz, 0.19H), 3.94 – 3.85 (m, 0.63H), 3.55 (s, 0.16H), 3.41 (dd, *J* = 12.9, 6.0 Hz, 0.69H),

2.86 (ddd, J = 11.5, 6.7, 5.2 Hz, 0.16H), 2.81 (ddd, J = 8.8, 6.8, 4.3 Hz, 0.16H), 2.70 (s, 0.15H), 2.04 – 1.92 (m, 1.52H), 1.64 – 1.42 (m, 2.52H), 1.00 (t, J = 7.4 Hz, 2.06H), 0.97 – 0.91 (m, 1.07H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.9, 161.8, 155.8, 153.3, 136.4, 136.2, 132.5, 127.3, 127.2, 125.5, 121.7, 121.5, 121.4, 121.2, 120.9, 117.9, 117.8, 117.5, 113.7, 108.3, 69.5, 68.4, 67.6, 67.4, 66.4, 50.9, 50.6, 49.8, 40.2, 36.3, 36.1, 19.3, 18.4, 17.8, 14.2, 14.0, 13.9;
HRMS (ESI): Calcd for $C_{13}H_{16}NO_3$ $[M+H]^+$: 234.1125; found 234.1120.



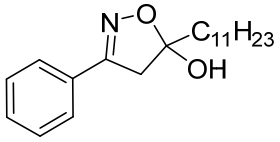
21

5-ethyl-3-phenyl-4,5-dihydroisoxazol-5-ol (21). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (34.1 mg, 89% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.65 – 7.60 (m, 2H), 7.40 – 7.34 (m, 3H), 3.92 (br, 1H), 3.23 (d, J = 3.7 Hz, 2H), 2.04 – 1.92 (m, 2H), 1.05 (t, J = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.4, 130.2, 129.6, 128.7, 126.7, 109.5, 43.9, 31.3, 9.0;
HRMS (ESI): Calcd for $C_{11}H_{14}NO_2$ $[M+H]^+$: 192.1019; found 192.1022.



22

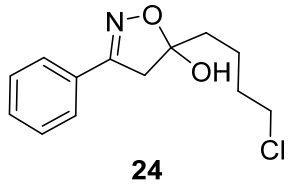
5-methyl-3-phenyl-4,5-dihydroisoxazol-5-ol (22).[9] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 5: 1).
White solid (30.5 mg, 86% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.65 – 7.59 (m, 2H), 7.44 – 7.35 (m, 3H), 3.92 (br, 1H), 3.35 (d, J = 17.3 Hz, 1H), 3.22 (d, J = 17.3 Hz, 1H), 1.76 (s, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.8, 130.3, 129.5, 128.7, 126.7, 107.0, 46.3, 25.3;
HRMS (ESI): Calcd for $C_{10}H_{12}NO_2$ $[M+H]^+$: 178.0863; found 178.0866.



23

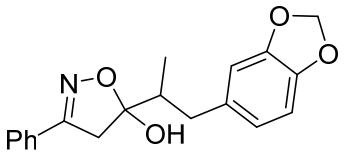
3-phenyl-5-undecyl-4,5-dihydroisoxazol-5-ol (23). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
Colorless oil (45.6 mg, 72% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.68 – 7.64 (m, 2H), 7.42 – 7.36 (m, 3H), 3.26 (s, 2H), 3.23 (br, 1H), 2.03 – 1.90 (m, 2H), 1.57 – 1.43 (m, 2H), 1.39 – 1.22 (m, 18H),

0.88 (t, *J* = 7.0 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.4, 130.3, 129.6, 128.7, 126.7, 108.9, 44.4, 38.4, 31.9, 29.7, 29.6, 29.6, 29.5, 29.5, 29.4, 24.8, 22.7, 14.1;
HRMS (ESI): Calcd for $C_{20}H_{32}NO_2$ $[M+H]^+$: 318.2428; found 318.2431.



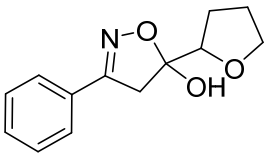
24

5-(4-chlorobutyl)-3-phenyl-4,5-dihydroisoxazol-5-ol (24). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 6: 1).
Colorless oil (33.4 mg, 66% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.62 (dd, *J* = 7.9, 1.8 Hz, 2H), 7.42 – 7.36 (m, 3H), 3.97 (s, 1H), 3.54 (t, *J* = 6.5 Hz, 2H), 3.32 – 3.21 (m, 2H), 1.97 (dddd, *J* = 34.6, 13.9, 10.9, 5.5 Hz, 2H), 1.86 – 1.80 (m, 2H), 1.72 – 1.58 (m, 2H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.5, 130.4, 129.4, 128.8, 126.7, 108.7, 44.8, 44.6, 37.4, 32.3, 22.1;
HRMS (ESI): Calcd for $C_{13}H_{17}ClNO_2$ $[M+H]^+$: 254.0942; found 254.0945.



25

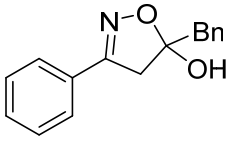
5-(1-(benzo[d][1,3]dioxol-5-yl)propan-2-yl)-3-phenyl-4,5-dihydroisoxazol-5-ol (25). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 3: 1).
White solid (42.5 mg, 65% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.65 (ddt, *J* = 8.1, 6.1, 1.9 Hz, 2H), 7.42 – 7.36 (m, 3H), 6.74 – 6.68 (m, 2H), 6.64 (ddd, *J* = 14.3, 7.8, 1.7 Hz, 1H), 5.92 (q, *J* = 1.6 Hz, 2H), 3.47 (br, 1H), 3.30 – 3.18 (m, 2H), 3.15 (dd, *J* = 13.2, 3.1 Hz, 0.5H), 2.96 (dd, *J* = 12.8, 3.3 Hz, 0.5H), 2.31 (ddd, *J* = 27.3, 13.1, 10.5 Hz, 1H), 2.27 – 2.20 (m, 1H), 1.03 (d, *J* = 6.4 Hz, 1.5H), 0.95 (d, *J* = 6.8 Hz, 1.5H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.3, 147.6, 133.9, 130.3, 129.5, 128.8, 126.7, 122.2, 1110.0, 110.8, 109.6, 109.5, 108.2, 100.9, 43.3, 43.2, 43.2, 43.1, 38.4, 37.3, 14.7, 14.2;
HRMS (ESI): Calcd for $C_{19}H_{20}NO_4$ $[M+H]^+$: 326.1387; found 326.1388.



26

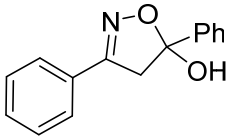
3-phenyl-5-(tetrahydrofuran-2-yl)-4,5-dihydroisoxazol-5-ol (26). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 3: 1).

White solid (33.6 mg, 71% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.58 – 7.53 (m, 2H), 7.35 – 7.28 (m, 3H), 4.31 – 3.86 (m, 2H), 3.79 – 3.58 (m, 2H), 3.35 – 3.26 (m, 0.5H), 3.23 – 3.18 (m, 1H), 3.18 – 3.11 (m, 0.5H), 2.83 – 2.72 (m, 1H), 2.12 – 2.00 (m, 1H), 1.83 – 1.71 (m, 1H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.4, 157.3, 130.4, 129.3, 129.3, 128.8, 126.7, 126.7, 109.8, 69.8, 69.4, 68.5, 68.2, 46.3, 46.1, 43.6, 43.3, 28.6, 28.1;
HRMS (ESI): Calcd for $C_{13}H_{16}NO_3$ $[M+H]^+$: 234.1125; found 234.1121.



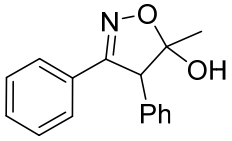
27

5-benzyl-3-phenyl-4,5-dihydroisoxazol-5-ol (27). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 5: 1).
White solid (43.5 mg, 86% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.56 – 7.50 (m, 2H), 7.30 (td, *J* = 6.8, 3.7 Hz, 4H), 7.28 – 7.24 (m, 2H), 7.22 – 7.17 (m, 2H), 3.28 – 3.21 (m, 2H), 3.20 – 3.15 (m, 2H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.6, 135.1, 130.5, 130.3, 129.4, 128.7, 128.7, 127.4, 126.7, 108.0, 44.6, 44.3;
HRMS (ESI): Calcd for $C_{16}H_{15}NNaO_2$ $[M+Na]^+$: 276.0995; found 276.0998.



28

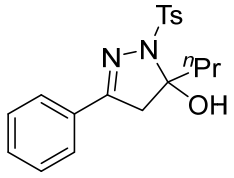
3,5-diphenyl-4,5-dihydroisoxazol-5-ol (28).[9] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (42.3 mg, 88% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.71 – 7.67 (m, 2H), 7.67 – 7.64 (m, 2H), 7.45 – 7.37 (m, 6H), 3.68 (d, *J* = 17.4 Hz, 1H), 3.49 (dd, *J* = 17.4, 1.1 Hz, 1H), 3.30 (s, 1H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 157.5, 140.7, 130.5, 129.3, 129.0, 128.8, 128.6, 126.8, 125.6, 107.7, 49.0;
HRMS (ESI): Calcd for $C_{15}H_{14}NO_2$ $[M+H]^+$: 240.1019; found 240.1015.



29

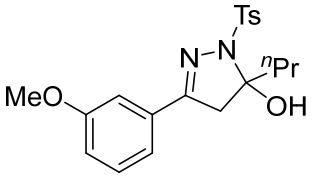
5-methyl-3,4-diphenyl-4,5-dihydroisoxazol-5-ol (29). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (45.1 mg, 89% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.63 – 7.60 (m, 1.71H), 7.52 – 7.49 (m, 0.28H), 7.38 – 7.26 (m, 6.11H), 7.21 – 7.13 (m, 1.86H), 4.52 (s, 0.86H), 4.46 (s, 0.14H), 3.40

(s, 0.83H), 2.72 (s, 0.13H), 1.77 (s, 0.42H), 1.27 (s, 2.59H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 160.7, 134.7, 132.3, 130.2, 130.0, 129.4, 129.2, 129.2, 128.9, 128.6, 128.6, 128.1, 127.4, 127.1, 109.4, 108.7, 63.1, 60.8, 26.2, 22.1;
HRMS (ESI): Calcd for $C_{16}H_{16}NO_2$ $[M+H]^+$: 254.1176; found 254.1172.



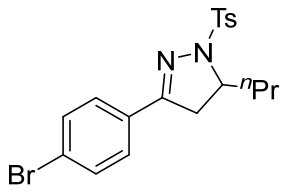
30

3-phenyl-5-propyl-1-tosyl-4,5-dihydro-1H-pyrazol-5-ol (30). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 3: 1).
White solid (55.2 mg, 77% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 – 7.88 (m, 2H), 7.63 – 7.58 (m, 2H), 7.37 – 7.31 (m, 3H), 7.27 – 7.22 (m, 2H), 3.27 – 3.13 (m, 2H), 2.37 – 2.31 (m, 4H), 2.21 (ddd, *J* = 14.0, 11.8, 4.9 Hz, 1H), 1.54 – 1.43 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 153.4, 143.8, 136.6, 131.1, 130.2, 129.3, 128.6, 128.3, 126.5, 98.5, 45.6, 43.1, 21.6, 18.7, 14.0.;
HRMS (ESI): Calcd for $C_{19}H_{23}N_2O_3S$ $[M+H]^+$: 359.1424; found 359.1426.



31

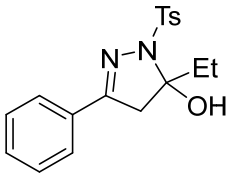
3-(3-methoxyphenyl)-5-propyl-1-tosyl-4,5-dihydro-1H-pyrazol-5-ol (31). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 2: 1).
White solid (53.2 mg, 69% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.29 – 7.23 (m, 3H), 7.20 (dd, *J* = 2.6, 1.5 Hz, 1H), 7.15 (dt, *J* = 7.8, 1.2 Hz, 1H), 6.91 (ddd, *J* = 8.2, 2.6, 0.9 Hz, 1H), 3.81 (s, 3H), 3.23 – 3.14 (m, 2H), 2.37 (s, 3H), 2.26 (dd, *J* = 31.8, 15.1 Hz, 2H), 1.53 – 1.44 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.6, 153.2, 143.9, 136.5, 132.4, 129.6, 129.3, 128.2, 119.2, 116.2, 111.5, 98.5, 55.4, 45.6, 43.2, 21.6, 18.7, 14.0.;
HRMS (ESI): Calcd for $C_{20}H_{25}N_2O_4S$ $[M+H]^+$: 389.1530; found 389.1530.



32

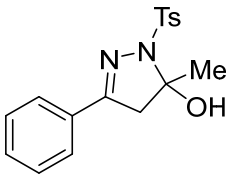
3-(2-fluorophenyl)-5-propyl-4,5-dihydroisoxazol-5-ol (32). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (72.1 mg, 86% yield).

^{1}H NMR (600 MHz, DMSO-*d*$_6$) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.64 – 7.61 (m, 2H), 7.59 (d, *J* = 8.6 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 6.64 (s, 1H), 3.33 (d, *J* = 17.8 Hz, 1H), 3.04 (d, *J* = 17.9 Hz, 1H), 2.34 (s, 3H), 2.28 (ddd, *J* = 13.5, 11.5, 4.9 Hz, 1H), 2.07 (ddd, *J* = 13.5, 11.5, 5.1 Hz, 1H), 1.50 – 1.36 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H);
^{13}C NMR (151 MHz, DMSO-*d*$_6$) δ 153.39, 143.65, 137.33, 132.14, 131.04, 129.39, 128.91, 128.74, 123.82, 99.27, 45.74, 41.92, 21.46, 18.89, 14.56;
HRMS (ESI): Calcd for $C_{19}H_{22}BrN_2O_2S$ $[M+H]^+$: 421.0580, 423.0560; found 421.0582, 423.0558.



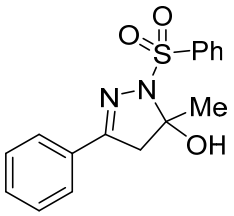
33

5-ethyl-3-phenyl-1-tosyl-4,5-dihydro-1H-pyrazol-5-ol (33). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (50.2 mg, 73% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 – 7.90 (m, 2H), 7.65 – 7.62 (m, 2H), 7.39 – 7.35 (m, 3H), 7.28 (d, *J* = 8.1 Hz, 2H), 3.29 (s, 1H), 3.27 – 3.14 (m, 2H), 2.43 – 2.33 (m, 4H), 2.34 – 2.24 (m, 1H), 1.06 (t, *J* = 7.4 Hz, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 153.3, 143.9, 136.6, 131.1, 130.2, 129.3, 128.6, 128.2, 126.5, 99.0, 45.0, 34.0, 21.6, 9.6;
HRMS (ESI): Calcd for $C_{18}H_{20}N_2NaO_3S$ $[M+Na]^+$: 367.1087; found 367.1088.



34

5-methyl-3-phenyl-1-tosyl-4,5-dihydro-1H-pyrazol-5-ol (34). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (52.0 mg, 79% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 – 7.90 (m, 2H), 7.64 – 7.61 (m, 2H), 7.39 – 7.34 (m, 3H), 7.27 (d, *J* = 8.0 Hz, 2H), 3.53 (br, 1H), 3.32 (d, *J* = 17.6 Hz, 1H), 3.19 (d, *J* = 17.6 Hz, 1H), 2.38 (s, 3H), 2.02 (s, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 153.4, 143.9, 136.4, 131.0, 130.3, 129.3, 128.6, 128.2, 126.5, 95.7, 48.3, 28.6, 21.6;
HRMS (ESI): Calcd for $C_{17}H_{19}N_2O_3S$ $[M+H]^+$: 331.1111; found 331.1113.



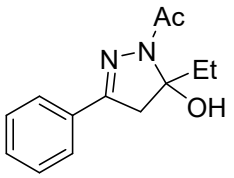
35

5-methyl-3-phenyl-1-(phenylsulfonyl)-4,5-dihydro-1H-pyrazol-5-ol (35). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1). White solid (38.5 mg, 61% yield).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.06 – 8.02 (m, 2H), 7.64 – 7.60 (m, 2H), 7.57 – 7.53 (m, 1H), 7.49 (dd, *J* = 8.4, 7.0 Hz, 2H), 7.40 – 7.34 (m, 3H), 3.61 – 3.54 (m, 1H), 3.33 (d, *J* = 17.6 Hz, 1H), 3.20 (d, *J* = 17.6 Hz, 1H), 2.03 (s, 3H);

^{13}C NMR (151 MHz, Chloroform-*d*) δ 153.6, 139.4, 133.0, 130.9, 130.4, 128.7, 128.6, 128.2, 126.5, 95.7, 48.3, 28.6;

HRMS (ESI): Calcd for $C_{16}H_{17}N_2O_3S$ $[M+H]^+$: 317.0954; found 317.0950.



36

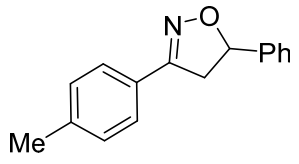
1-(5-ethyl-5-hydroxy-3-phenyl-4,5-dihydro-1H-pyrazol-1-yl)ethan-1-one (36). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).

White solid (37.7 mg, 81% yield).

^{1}H NMR (600 MHz, Chloroform-*d*)) δ 7.68 – 7.58 (m, 2H), 7.38 – 7.31 (m, 3H), 4.57 (s, 1H), 3.25 (d, *J* = 18.1 Hz, 1H), 3.17 (d, *J* = 18.1 Hz, 1H), 2.31 (s, 3H), 2.25 – 2.08 (m, 2H), 0.86 (t, *J* = 7.5 Hz, 3H);

^{13}C NMR (151 MHz, Chloroform-*d*) δ 171.3, 152.9, 131.4, 130.3, 128.7, 126.4, 95.2, 44.6, 32.1, 22.5, 8.7;

HRMS (ESI): Calcd for $C_{13}H_{17}N_2O_2$ $[M+H]^+$: 233.1285; found 233.1287.

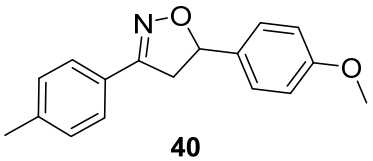


39

3-(4-methoxyphenyl)-5-phenyl-4,5-dihydroisoxazole (39).[10] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: DCM = 1: 4).

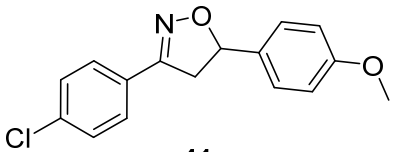
White solid (43.2 mg, 91% yield).

^{1}H NMR (600 MHz, Chloroform-*d*)) δ 7.57 (d, *J* = 7.7 Hz, 2H), 7.40 – 7.32 (m, 4H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.19 (d, *J* = 7.7 Hz, 2H), 5.69 (t, *J* = 9.6 Hz, 1H), 3.73 (dd, *J* = 16.5, 10.9 Hz, 1H), 3.30 (dd, *J* = 16.6, 8.3 Hz, 1H), 2.36 (s, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 156.1, 141.1, 140.4, 129.5, 128.8, 128.2, 126.8, 126.7, 126.0, 82.5, 43.3, 21.5;
HRMS (ESI): Calcd for $C_{16}H_{15}NNaO$ $[M+Na]^+$: 260.1046; found 260.1043.



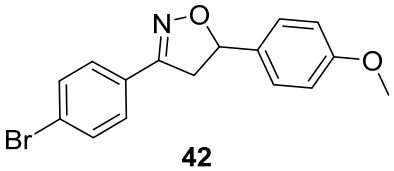
40

5-(4-methoxyphenyl)-3-(p-tolyl)-4,5-dihydroisoxazole (40).[11] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: DCM = 1: 4).
White solid (49.3 mg, 92% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.7 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 5.63 (dd, *J* = 10.8, 8.6 Hz, 1H), 3.77 (s, 3H), 3.67 (dd, *J* = 16.6, 10.8 Hz, 1H), 3.27 (dd, *J* = 16.6, 8.6 Hz, 1H), 2.36 (s, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.6, 156.3, 140.4, 132.9, 129.5, 127.5, 126.8, 126.7, 114.1, 82.4, 55.3, 43.0, 21.5;
HRMS (ESI): Calcd for $C_{17}H_{18}NO_2$ $[M+H]^+$: 268.1332; found 268.1330.



41

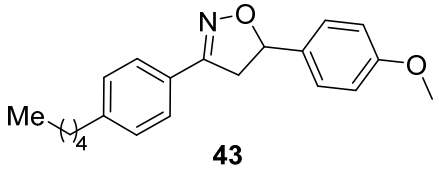
3-(4-chlorophenyl)-5-(4-methoxyphenyl)-4,5-dihydroisoxazole (41).[11] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: DCM = 1: 5).
Pale-yellow solid (46.5 mg, 81% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.63 – 7.58 (m, 2H), 7.36 (d, *J* = 8.6 Hz, 2H), 7.29 (d, *J* = 8.7 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 5.67 (dd, *J* = 10.9, 8.7 Hz, 1H), 3.79 (s, 3H), 3.67 (dd, *J* = 16.7, 10.9 Hz, 1H), 3.27 (dd, *J* = 16.7, 8.7 Hz, 1H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.7, 155.4, 136.0, 132.5, 129.0, 128.2, 128.0, 127.4, 114.2, 82.8, 55.4, 42.7;
HRMS (ESI): Calcd for $C_{16}H_{15}ClNO_2$ $[M+H]^+$: 288.0786; found 288.0787.



42

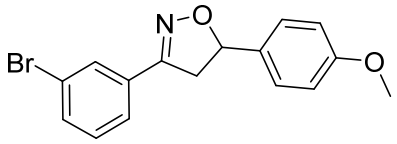
3-(4-bromophenyl)-5-(4-methoxyphenyl)-4,5-dihydroisoxazole (42). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: DCM = 1: 5).
Yellow solid (56.8 mg, 86% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.57 – 7.52 (m, 4H), 7.32 – 7.29 (m, 2H), 6.92 – 6.88 (m, 2H), 5.69 (dd, *J* = 10.9, 8.6 Hz, 1H), 3.80 (s, 3H), 3.69 (dd, *J* = 16.6, 10.9 Hz,

1H), 3.29 (dd, *J* = 16.7, 8.7 Hz, 1H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.7, 155.5, 132.5, 132.0, 128.6, 128.2, 127.4, 124.4, 114.2, 82.8, 55.4, 42.7;
HRMS (ESI): Calcd for $C_{16}H_{15}BrNO_2$ $[M+H]^+$: 332.0281, 334.0261; found 332.0283, 334.0260.



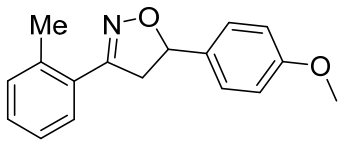
43

5-(4-methoxyphenyl)-3-(4-pentylphenyl)-4,5-dihydroisoxazole (43). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: DCM = 1: 5).
White solid (59.5 mg, 92% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.9 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 6.92 (d, *J* = 8.2 Hz, 2H), 5.69 (dd, *J* = 10.8, 8.5 Hz, 1H), 3.83 (s, 3H), 3.74 (dd, *J* = 16.6, 10.8 Hz, 1H), 3.34 (dd, *J* = 16.6, 8.5 Hz, 1H), 2.65 (t, *J* = 7.7 Hz, 2H), 1.66 (q, *J* = 7.5 Hz, 2H), 1.35 (h, *J* = 5.9 Hz, 4H), 0.96 – 0.86 (m, 3H);
^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.6, 156.2, 145.4, 133.0, 128.8, 127.4, 127.0, 126.7, 114.1, 82.3, 55.3, 43.1, 35.8, 31.4, 31.0, 22.5, 14.0.;
HRMS (ESI): Calcd for $C_{21}H_{26}NO_2$ $[M+H]^+$: 324.1958; found 324.1955.



44

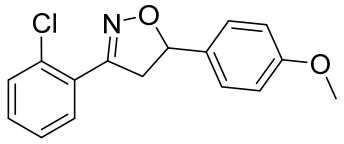
3-(3-bromophenyl)-5-(4-methoxyphenyl)-4,5-dihydroisoxazole (44). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: DCM = 1: 5).
Yellow solid (50.8 mg, 77% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.83 (t, *J* = 1.8 Hz, 1H), 7.61 (ddd, *J* = 7.8, 1.6, 1.0 Hz, 1H), 7.52 (ddd, *J* = 8.0, 2.0, 1.0 Hz, 1H), 7.31 – 7.28 (m, 2H), 7.27 (t, *J* = 7.9 Hz, 1H), 6.91 – 6.87 (m, 2H), 5.69 (dd, *J* = 11.0, 8.6 Hz, 1H), 3.80 (s, 3H), 3.68 (dd, *J* = 16.7, 11.0 Hz, 1H), 3.28 (dd, *J* = 16.7, 8.7 Hz, 1H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.7, 155.1, 133.0, 132.4, 131.7, 130.3, 129.6, 127.4, 125.2, 122.9, 114.2, 82.9, 55.4, 42.6.;
HRMS (ESI): Calcd for $C_{16}H_{15}BrNO_2$ $[M+H]^+$: 332.0281, 334.0261; found 332.0283, 334.0268.



45

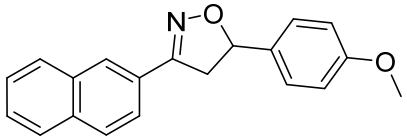
5-(4-methoxyphenyl)-3-(o-tolyl)-4,5-dihydroisoxazole (45). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 3: 1).

White solid (38.1 mg, 71% yield).
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (d, *J* = 8.0 Hz, 3H), 7.32 (d, *J* = 5.3 Hz, 2H), 7.26 (t, *J* = 4.2 Hz, 1H), 7.01 – 6.90 (m, 2H), 5.67 (dd, *J* = 10.7, 8.4 Hz, 1H), 3.84 (s, 3H), 3.82 – 3.77 (m, 1H), 3.40 (dd, *J* = 16.6, 8.4 Hz, 1H), 2.63 (s, 3H);
^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.6, 157.1, 138.1, 133.0, 131.6, 129.4, 128.9, 128.7, 127.3, 125.8, 114.2, 81.5, 55.4, 45.5, 23.0;
HRMS (ESI): Calcd for $C_{17}H_{18}NO_2$ $[M+H]^+$: 268.1332; found 268.1335.



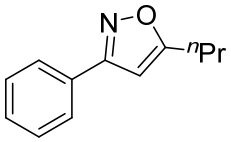
46

3-(2-chlorophenyl)-5-(4-methoxyphenyl)-4,5-dihydroisoxazole (46). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 3: 1).
White solid (41.6 mg, 73% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.67 (dd, *J* = 7.6, 1.9 Hz, 1H), 7.42 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.36 – 7.32 (m, 3H), 7.30 (td, *J* = 7.5, 1.4 Hz, 1H), 6.93 – 6.89 (m, 2H), 5.70 (dd, *J* = 10.7, 8.8 Hz, 1H), 3.85 (dd, *J* = 17.1, 10.7 Hz, 1H), 3.80 (s, 3H), 3.50 (dd, *J* = 17.1, 8.8 Hz, 1H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.7, 156.3, 132.9, 132.4, 130.9, 130.6, 130.6, 129.1, 127.5, 127.1, 114.2, 83.2, 55.4, 45.2;
HRMS (ESI): Calcd for $C_{16}H_{15}ClNO_2$ $[M+H]^+$: 288.0786; found 288.0787.



47

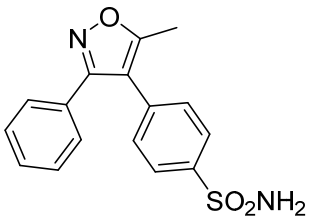
5-(4-methoxyphenyl)-3-(naphthalen-2-yl)-4,5-dihydroisoxazole (34).[12] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: DCM = 1: 4).
White solid (50.3 mg, 83% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (dd, *J* = 8.7, 1.7 Hz, 1H), 7.84 (d, *J* = 1.7 Hz, 1H), 7.81 – 7.78 (m, 3H), 7.49 – 7.44 (m, 2H), 7.31 (d, *J* = 8.7 Hz, 2H), 6.89 – 6.85 (m, 2H), 5.67 (dd, *J* = 10.8, 8.7 Hz, 1H), 3.78 – 3.70 (m, 4H), 3.38 (dd, *J* = 16.5, 8.7 Hz, 1H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 159.7, 156.5, 134.1, 133.1, 132.8, 128.6, 128.4, 127.9, 127.5, 127.3, 127.2, 127.0, 126.8, 123.6, 114.2, 82.8, 55.4, 42.8;
HRMS (ESI): Calcd for $C_{20}H_{18}NO_2$ $[M+H]^+$: 304.1332; found 304.1335.



48

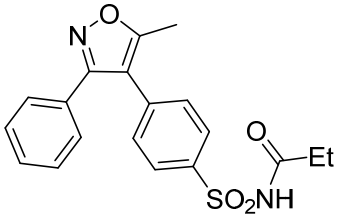
5-methyl-3-phenyl-1-tosyl-4,5-dihydro-1H-pyrazol-5-ol (34).[13] On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 20: 1).

Yellow solid (0.87g, 93% yield).
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 6.9 Hz, 2H), 7.46 (d, *J* = 6.1 Hz, 3H), 6.32 (s, 1H), 2.80 (t, *J* = 7.5 Hz, 2H), 1.81 (q, *J* = 7.5 Hz, 2H), 1.05 (t, *J* = 7.5 Hz, 3H);
^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.1, 162.3, 129.8, 129.5, 128.9, 126.8, 98.9, 28.8, 21.0, 13.7;
HRMS (ESI): Calcd for $C_{12}H_{14}NO$ $[M+H]^+$: 188.1070; found 188.1073.



Valdecoxib

4-(5-methyl-3-phenylisoxazol-4-yl)benzenesulfonamide (Valdecoxib). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 2: 1).
White solid (1.18 g, 75% yield).
^{1}H NMR (600 MHz, DMSO-*d*$_6$) δ 7.89 – 7.83 (m, 2H), 7.48 – 7.40 (m, 7H), 7.36 (dt, *J* = 7.0, 1.4 Hz, 2H), 2.48 (s, 3H);
^{13}C NMR (151 MHz, DMSO-*d*$_6$) δ 168.10, 161.15, 143.74, 133.75, 130.49, 130.25, 129.31, 128.88, 128.67, 126.56, 114.73, 11.86;
HRMS (ESI): Calcd for $C_{16}H_{14}N_2NaO_3S$ $[M+Na]^+$: 337.0617 ; found 337.0619.



Parecoxib

N-((4-(5-methyl-3-phenylisoxazol-4-yl)phenyl)sulfonyl)propionamide (Parecoxib). On 0.2 mmol scale. Purification by silica gel chromatography (petroleum: ethyl acetate = 4: 1).
White solid (1.13 g, 87% yield).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.57 (br, 1H), 7.99 – 7.94 (m, 2H), 7.37 – 7.25 (m, 7H), 2.43 (d, *J* = 1.1 Hz, 3H), 2.31 – 2.19 (m, 2H), 1.08 – 1.00 (m, 3H);
^{13}C NMR (151 MHz, Chloroform-*d*) δ 171.9, 167.6, 161.2, 137.8, 136.4, 130.1, 129.9, 128.8, 128.7, 128.5, 128.3, 114.5, 29.6, 11.9, 8.2;
HRMS (ESI): Calcd for $C_{19}H_{19}N_2O_4S$ $[M+H]^+$: 371.1060; found 371.1062.

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Single Crystal X-ray Diffraction Data

X-ray crystallographic data for 28

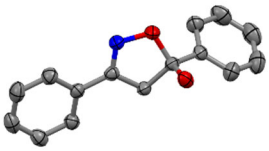


Table 1 Crystal data and structure refinement for compound 28.

Identification code	CCDC2386231
Empirical formula	$C_{15}H_{13}NO_2$
Formula weight	239.276
Temperature/K	298.15
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	8.8108(2)
b/Å	9.98924(19)
c/Å	13.7937(3)

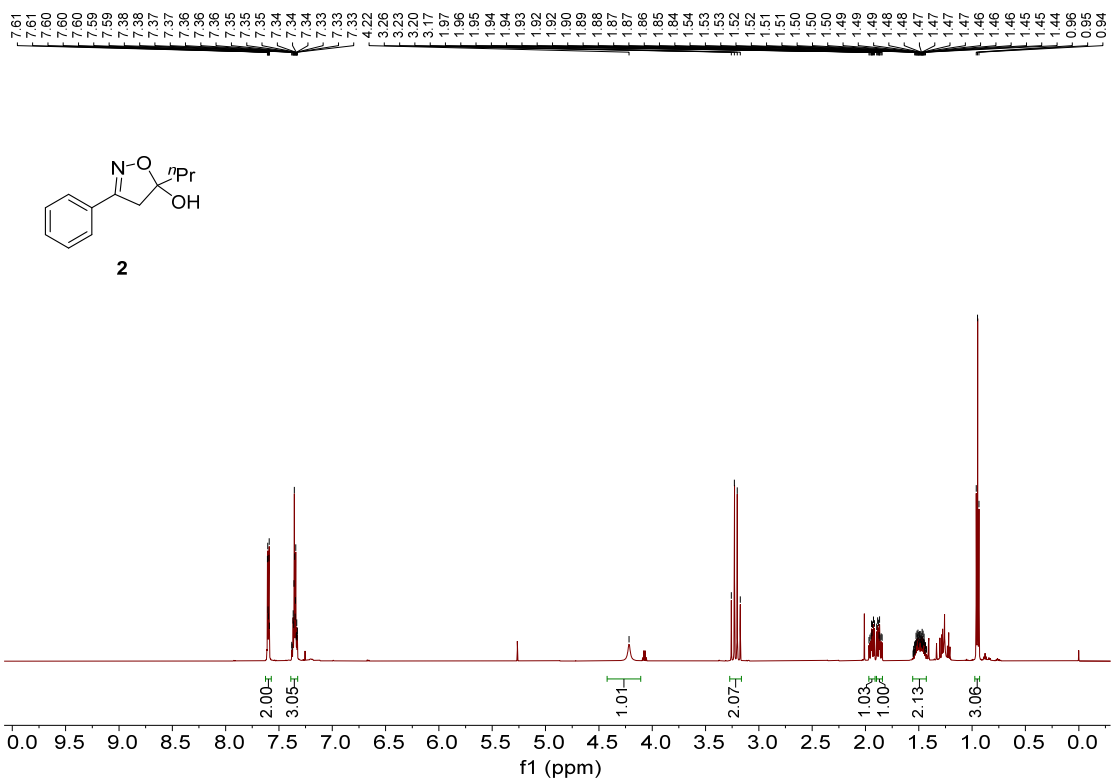
α/°	90
β/°	96.041(2)
γ/°	90
Volume/Å^3	1207.29(5)
Z	4
ρ_{calc}g/cm^3	1.316
μ/mm^{-1}	0.451
F(000)	505.2
Crystal size/mm^3	0.2 × 0.2 × 0.1
Radiation	synchrotron (λ = 1.34050)
2Θ range for data collection/°	9.52 to 112.7
Index ranges	-10 ≤ h ≤ 10, -12 ≤ k ≤ 12, -16 ≤ l ≤ 17
Reflections collected	13864
Independent reflections	2376 [R_{int} = 0.0308, R_{sigma} = 0.0181]
Data/restraints/parameters	2376/0/164
Goodness-of-fit on F^2	1.062
Final R indexes [I>=2σ (I)]	R_1 = 0.0465, wR_2 = 0.1182
Final R indexes [all data]	R_1 = 0.0504, wR_2 = 0.1228
Largest diff. peak/hole / e Å^{-3}	0.24/-0.41

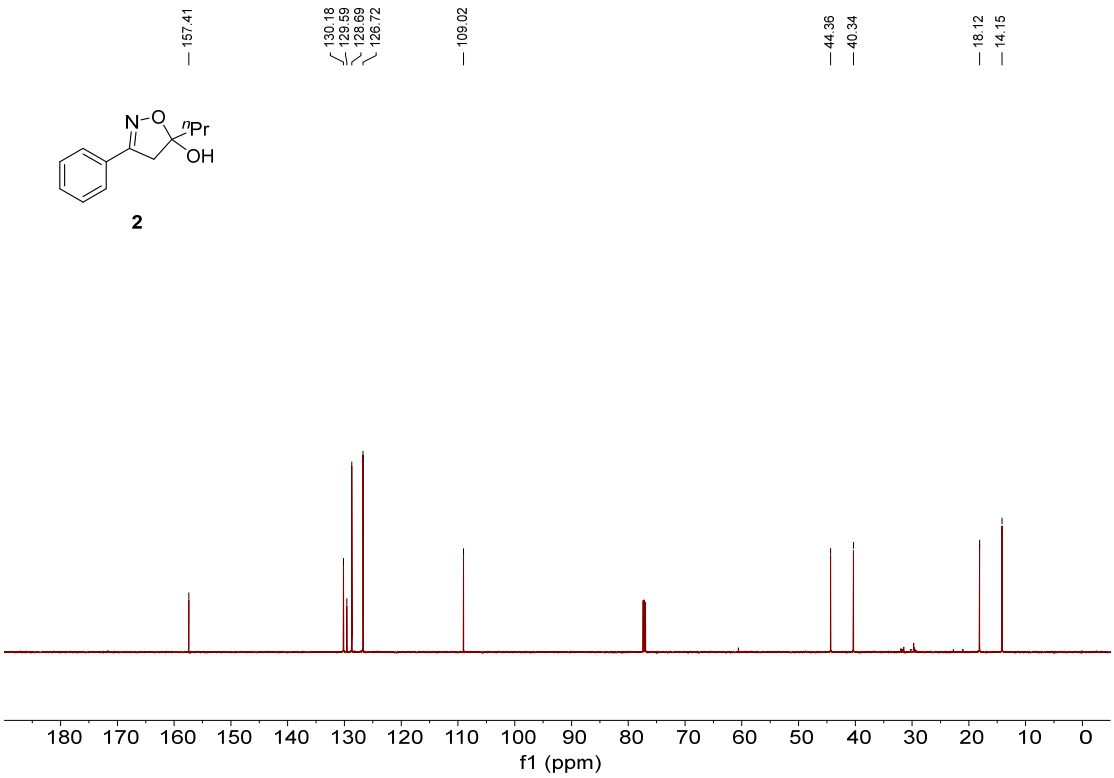
Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound1. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	4124.8(10)	3230.1(8)	7260.8(6)	41.1(2)
O2	4640.1(10)	5255.3(8)	6521.5(6)	42.6(3)
N2	5209.6(11)	2312.2(10)	6954.0(7)	39.2(3)
C	3671.9(13)	4142.5(11)	6460.0(8)	34.4(3)
C1	2011.3(13)	4497.8(12)	6506.8(8)	36.7(3)
C2	5184.4(12)	2374.6(11)	6022.7(8)	33.7(3)
C3	6157.3(13)	1513.6(11)	5488.3(8)	34.9(3)
C4	5922.9(14)	1456.2(12)	4477.0(9)	39.1(3)
C5	4029.3(13)	3348.0(11)	5571.5(8)	34.7(3)
C6	7297.0(15)	725.0(13)	5978.1(10)	45.1(3)
C7	1012.0(16)	3605.8(14)	6876.2(11)	49.7(3)
C8	1451.0(16)	5713.8(13)	6142.0(10)	49.9(3)
C9	6809.1(15)	622.3(14)	3965.6(10)	47.6(3)
C10	7924.1(16)	-157.8(14)	4456.2(11)	52.9(4)
C11	8162.9(16)	-108.0(14)	5463.9(12)	53.8(4)
C12	-83.0(19)	6026.9(17)	6156.6(13)	65.2(5)
C13	-514.6(18)	3936.5(18)	6889.5(13)	64.1(4)

C14	-1058.0(18)	5147(2)	6529.6(12)	67.2(5)

NMR Spectra

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109.02
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14.15
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O
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2
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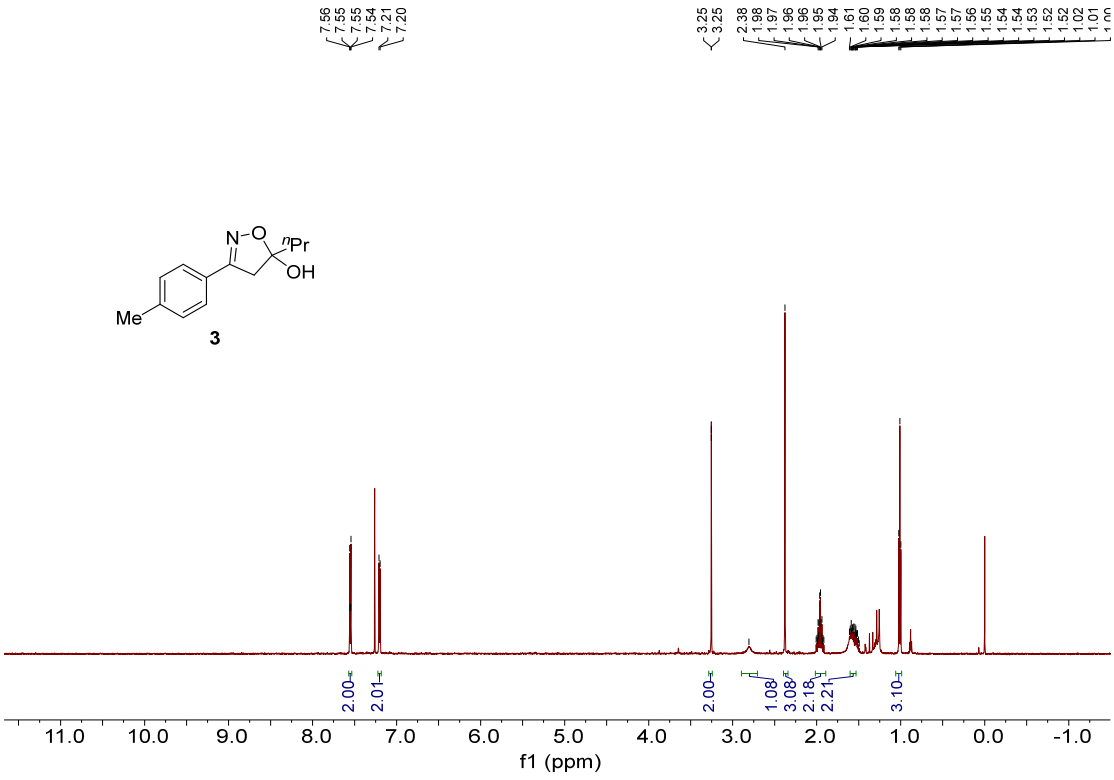
f1 (ppm)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1.58
1.57
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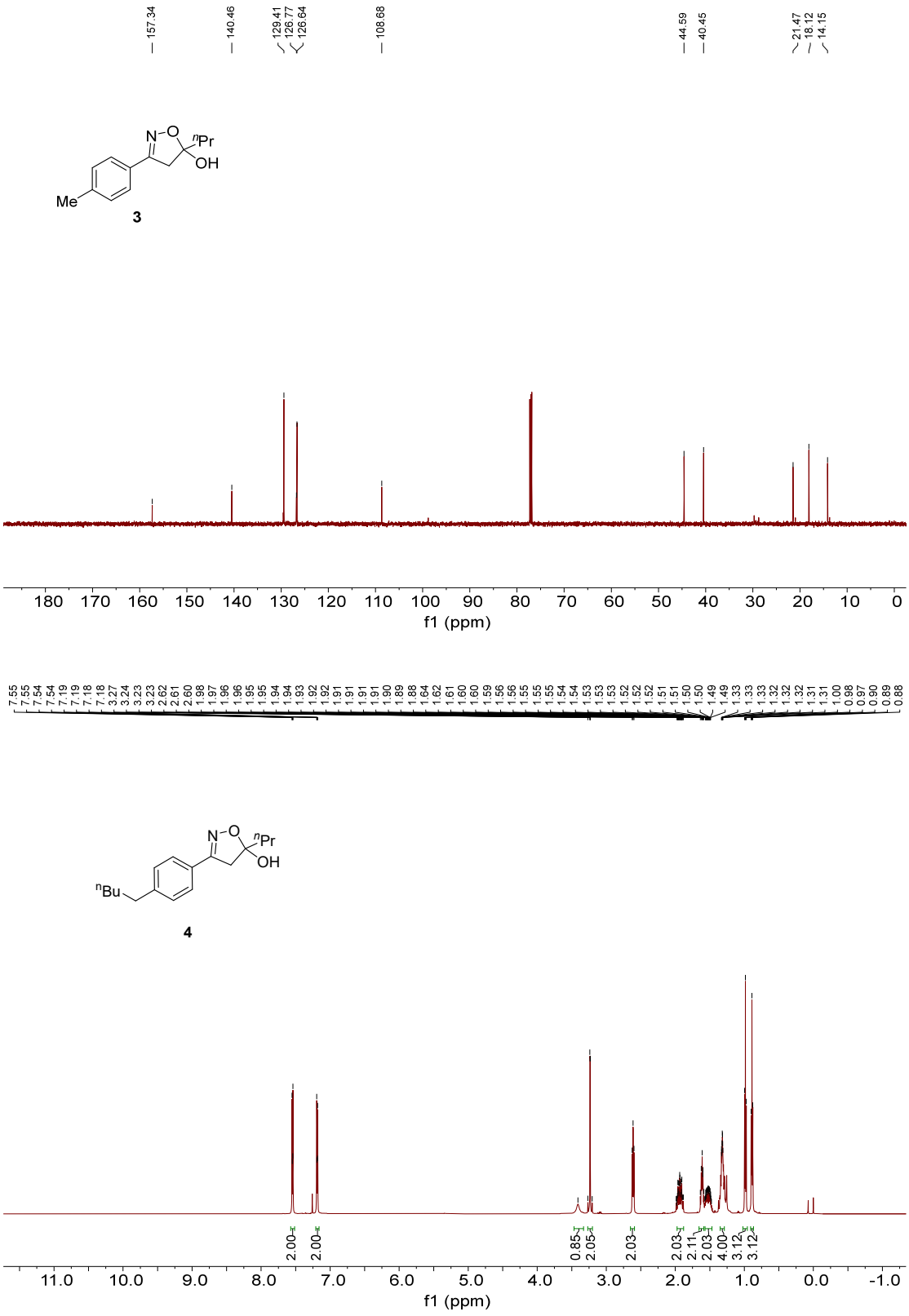
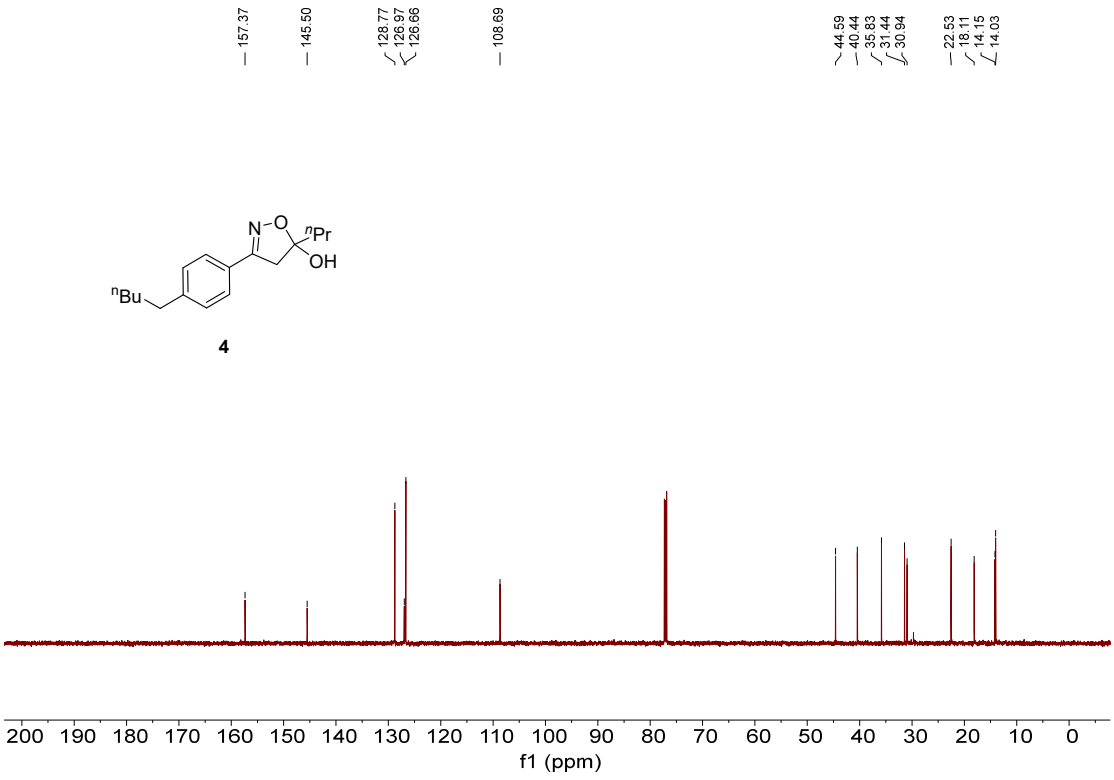
1.55
1.54
1.54
1.53
1.52
1.52
1.02
1.01
N
O
nPr
OH
Me
3
2.00
2.01
2.00
1.08
3.08
2.18
2.21
3.10
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)


157.34
140.46
129.41
126.77
126.64
108.68
44.59
40.45
21.47
18.12
14.15
N
O
nPr
OH
Me
3
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
7.55 7.55 7.54 7.54 7.54 7.19 7.19 7.18 7.18 3.27 3.24 3.23 3.23 2.62 2.61 2.60 1.98 1.97 1.96 1.96 1.95 1.95 1.94 1.94 1.93 1.92 1.92 1.91 1.91 1.91 1.91 1.90 1.89 1.88 1.64 1.62 1.61 1.60 1.60 1.59 1.56 1.56 1.55 1.55 1.55 1.54 1.54 1.53 1.53 1.53 1.52 1.52 1.52 1.52 1.51 1.51 1.50 1.50 1.49 1.49 1.33 1.33 1.33 1.32 1.32 1.32 1.31 1.31 1.00 0.98 0.97 0.90 0.89 0.88
N
O
nPr
OH
nBu
4
2.00
2.00
0.85
2.05
2.03
2.03
2.11
2.03
4.00
3.12
3.12
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)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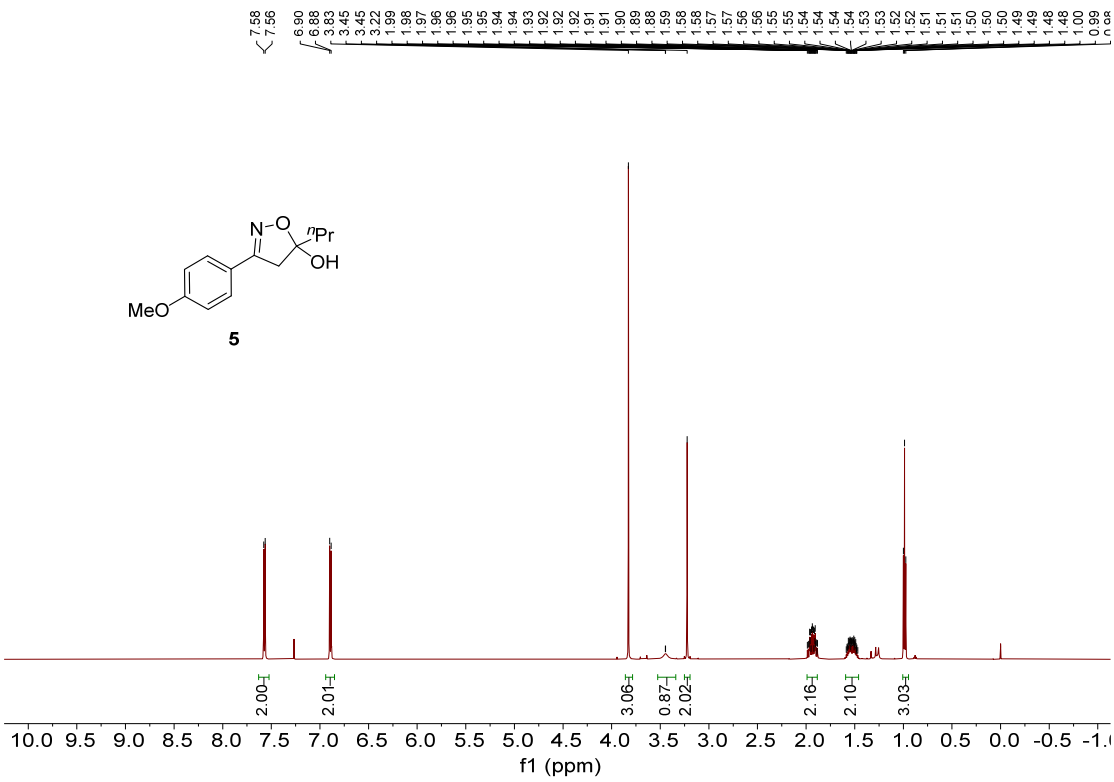
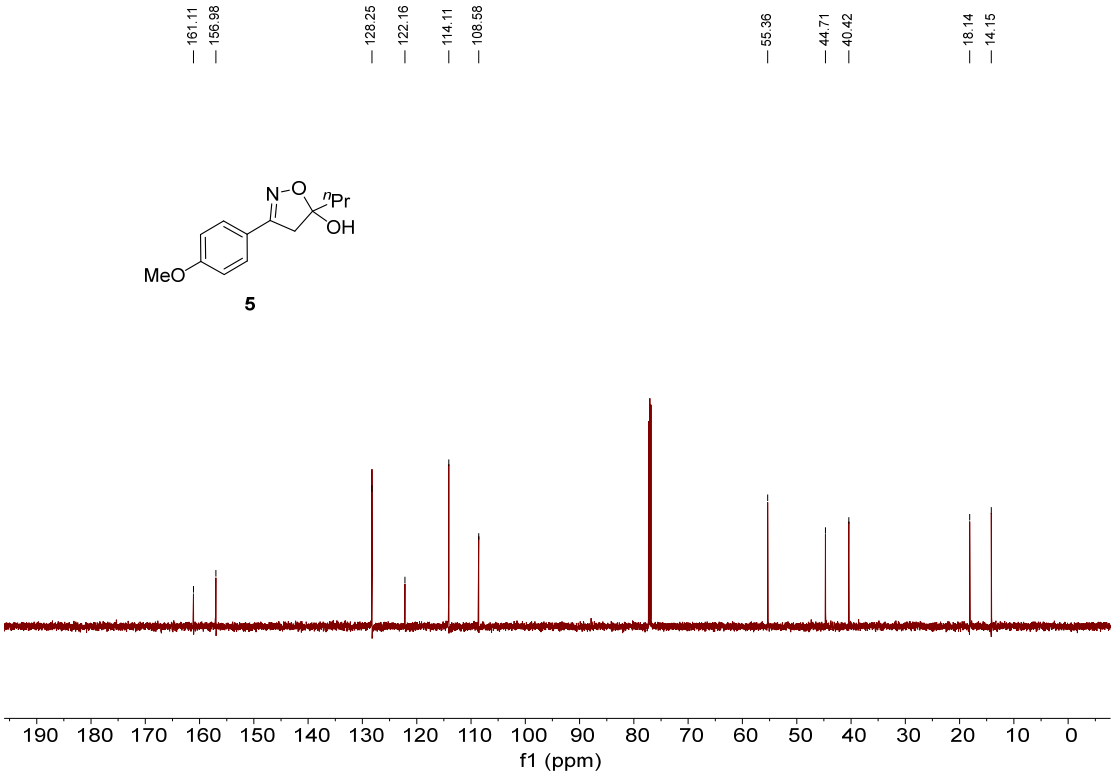
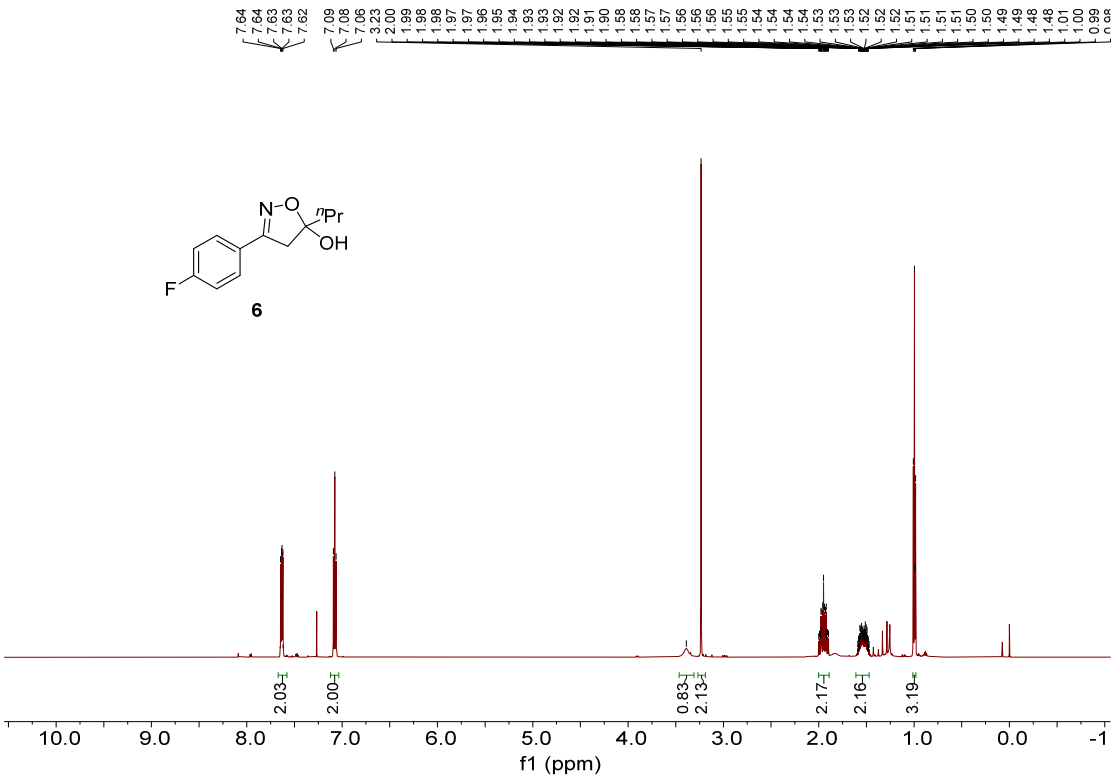
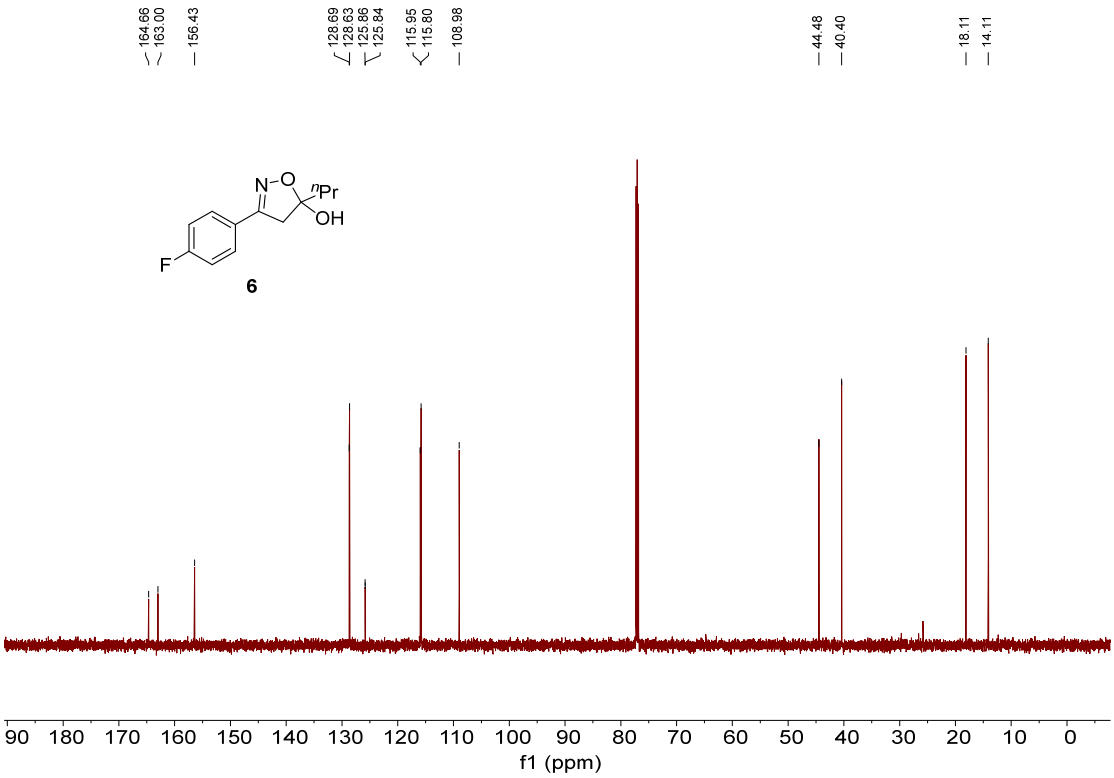
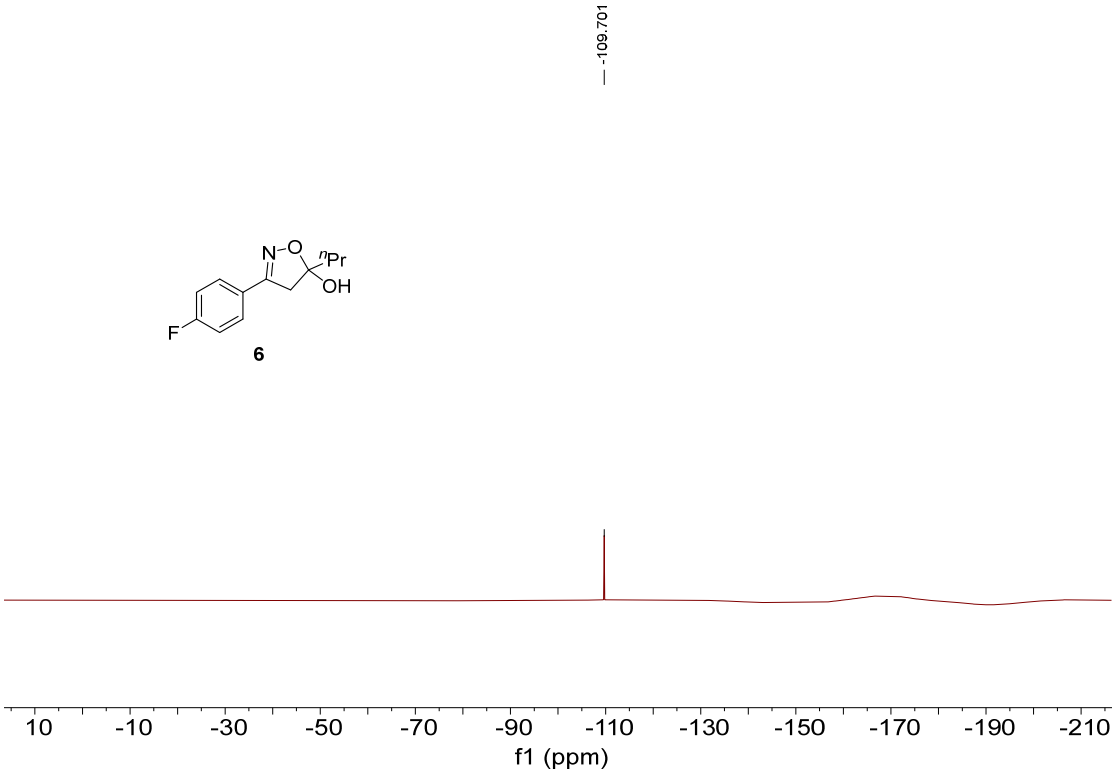
145.50
128.77
126.97
126.66
108.69
44.59
40.44
35.83
31.44
30.94
22.53
18.11
14.15
14.03
N
O
nPr
OH
nBu
4
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

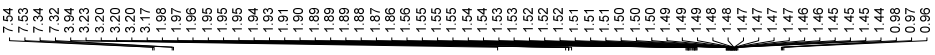

7.58
7.56
6.90
6.88
3.83
N
O
nPr
OH
MeO
5
2.00
2.01
3.06
0.87
2.02
2.16
2.10
3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

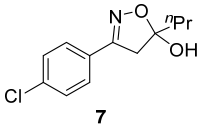

161.11
156.98
128.25
122.16
114.11
108.58
55.36
44.71
40.42
18.14
14.15
N
O
nPr
OH
MeO
5
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.64
7.64
7.63
7.63
7.62
7.09
7.08
7.06
3.23
2.00
1.99
1.98
1.98
1.97
1.97
1.96
1.95
1.94
1.93
1.93
1.92
1.92
1.91
1.90
1.58
1.58
1.57
1.57
1.56
1.56
1.56
1.55
1.55
1.54
1.54
1.54
1.54
1.53
1.53
1.53
1.52
1.52
1.52
1.51
1.51
1.51
1.51
1.50
1.50
1.49
1.49
1.48
1.48
1.01
1.00
0.99
N
O
nPr
OH
F
6
2.03
2.00
0.83
2.13
2.17
2.16
3.19
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1
f1 (ppm)

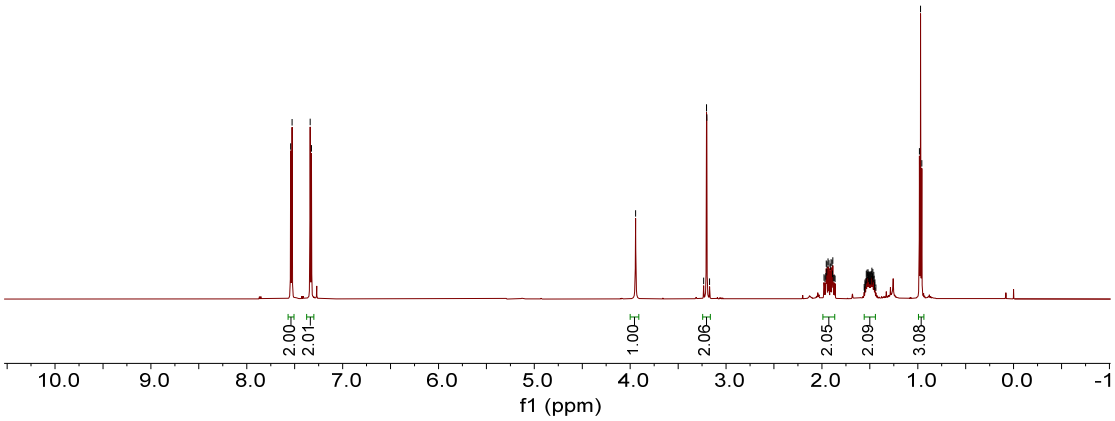

164.66
163.00
156.43
128.69
128.63
125.86
125.84
115.95
115.80
108.98
44.48
40.40
18.11
14.11
N
O
nPr
OH
F
6
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-109.701
N
O
nPr
OH
F
6
10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

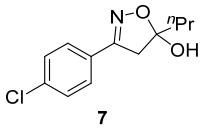

7.54
7.53
7.34
7.32
3.94
3.23
3.20
3.20
3.20
3.17
1.98
1.97
1.96
1.95
1.95
1.95
1.94
1.93
1.91
1.90
1.89
1.89
1.89
1.88
1.87
1.86
1.56
1.55
1.55
1.55
1.54
1.54
1.53
1.53
1.52
1.52
1.52
1.51
1.51
1.51
1.50
1.50
1.50
1.49
1.49
1.49
1.48
1.48
1.47
1.47
1.47
1.47
1.46
1.46
1.45
1.45
1.45
1.44
0.98
0.97
0.96


N
O
nPr
OH
Cl

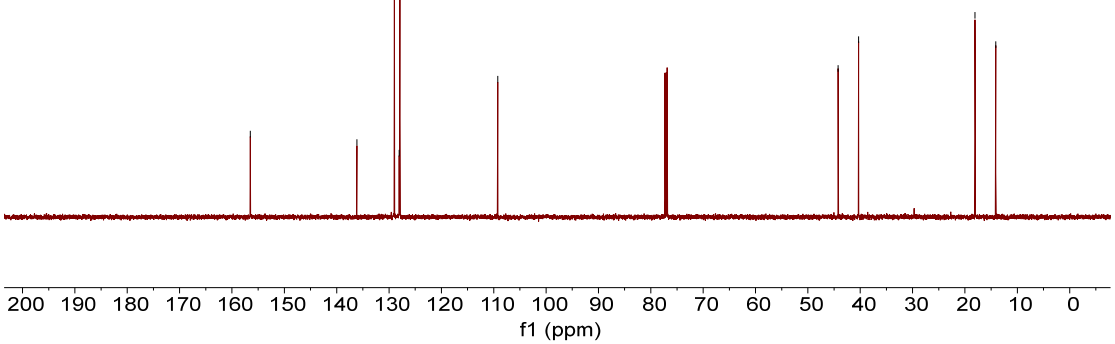
7

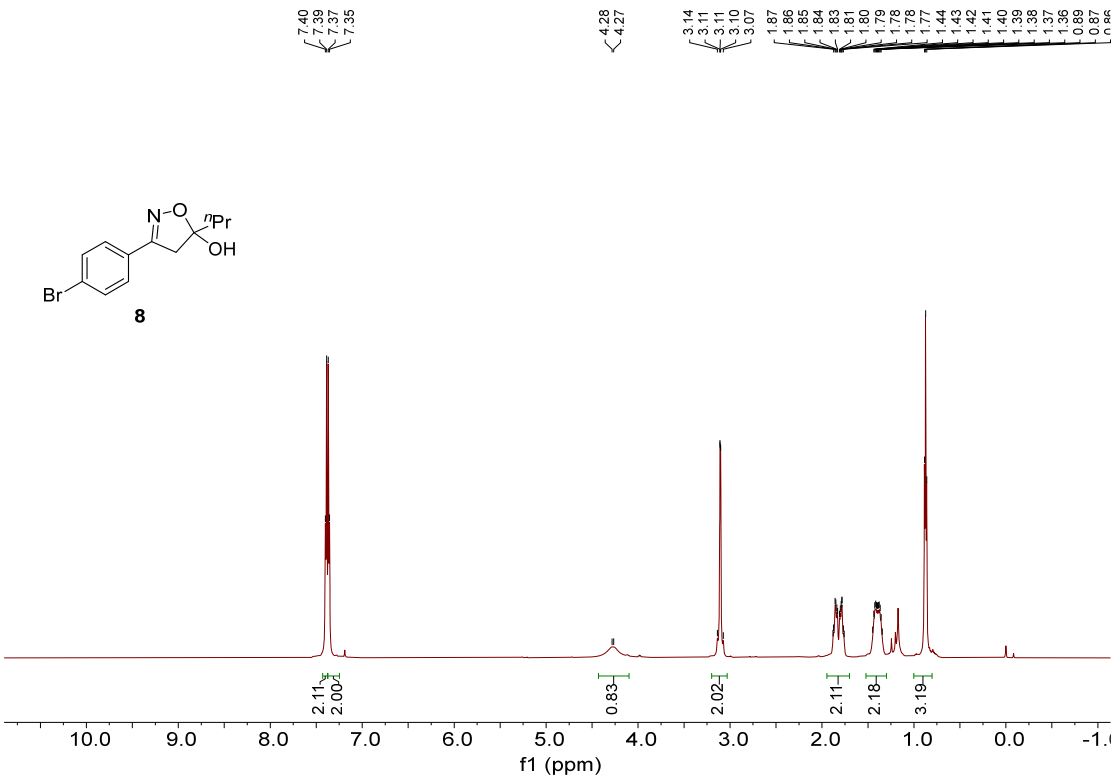

2.00
2.01
1.00
2.06
2.05
2.09
3.08
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1
f1 (ppm)

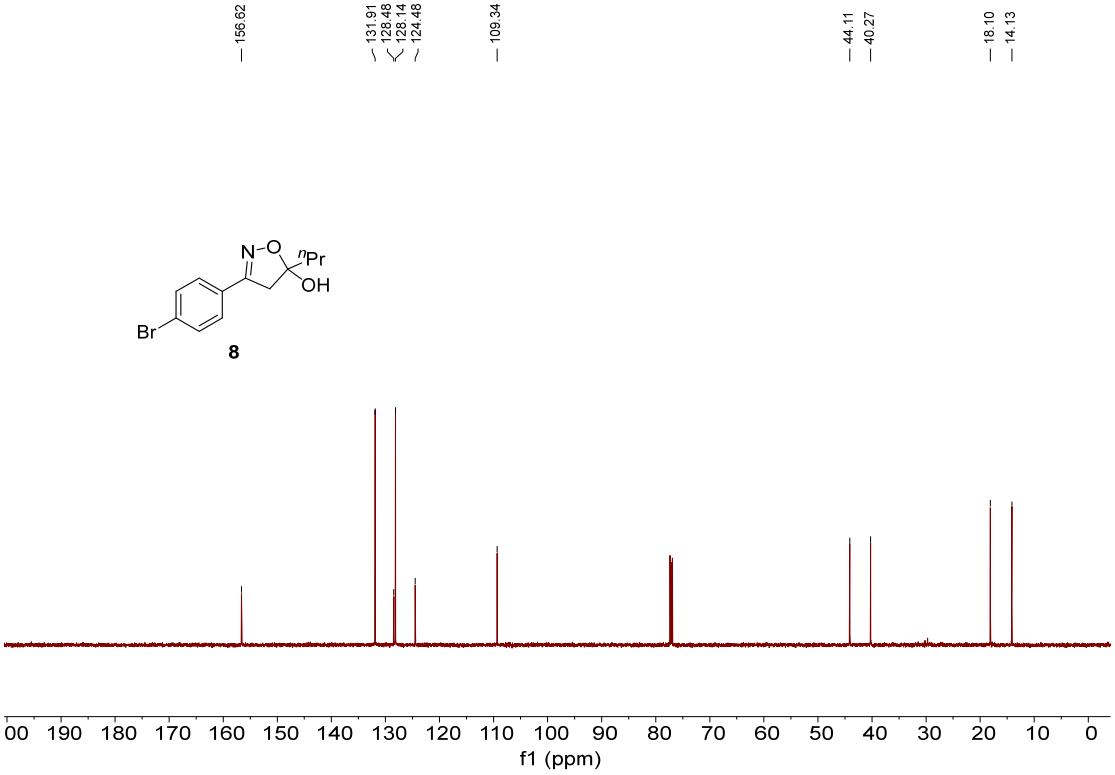

156.49
136.15
128.97
128.06
127.92
109.24
44.21
40.32
18.09
14.11

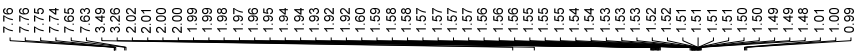

N
O
nPr
OH
Cl

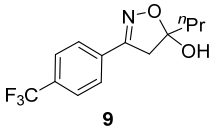
7


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

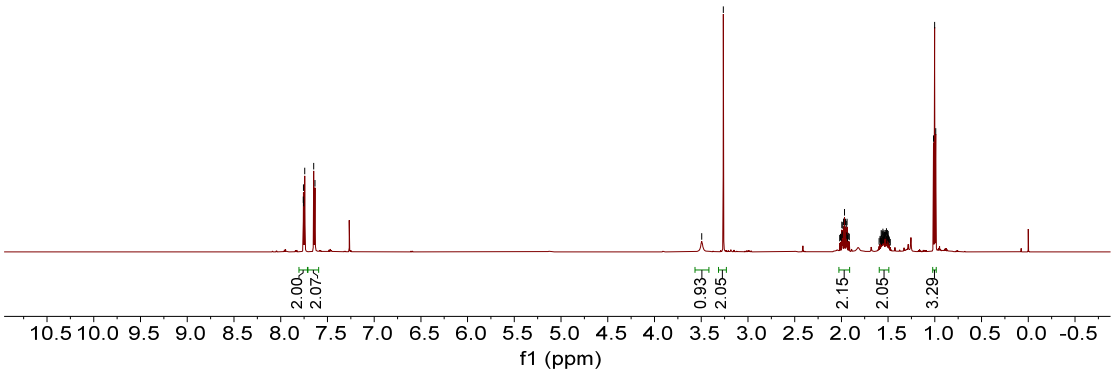

7.40
7.39
7.37
7.35
4.28
4.27
3.14
3.11
3.11
3.10
3.07
1.87
1.86
1.85
1.84
1.83
1.81
1.80
1.79
1.78
1.78
1.77
1.44
1.43
1.42
1.41
1.40
1.39
1.38
1.37
1.36
0.89
0.87
N
O
nPr
OH
Br
8
2.11
2.00
0.83
2.02
2.11
2.18
3.19
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


156.62
131.91
128.48
128.14
124.48
109.34
44.11
40.27
18.10
14.13
N
O
nPr
OH
Br
8
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

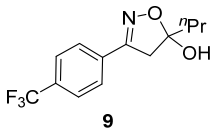

7.76
7.76
7.75
7.74
7.65
7.63
3.49
3.26
2.02
2.01
2.00
2.00
1.99
1.99
1.98
1.97
1.96
1.95
1.94
1.94
1.93
1.92
1.92
1.60
1.59
1.58
1.58
1.57
1.57
1.57
1.57
1.56
1.56
1.56
1.55
1.55
1.55
1.54
1.54
1.53
1.53
1.53
1.52
1.52
1.51
1.51
1.51
1.51
1.50
1.50
1.49
1.49
1.48
1.01
1.00
0.99


N
O
nPr
OH
F3C

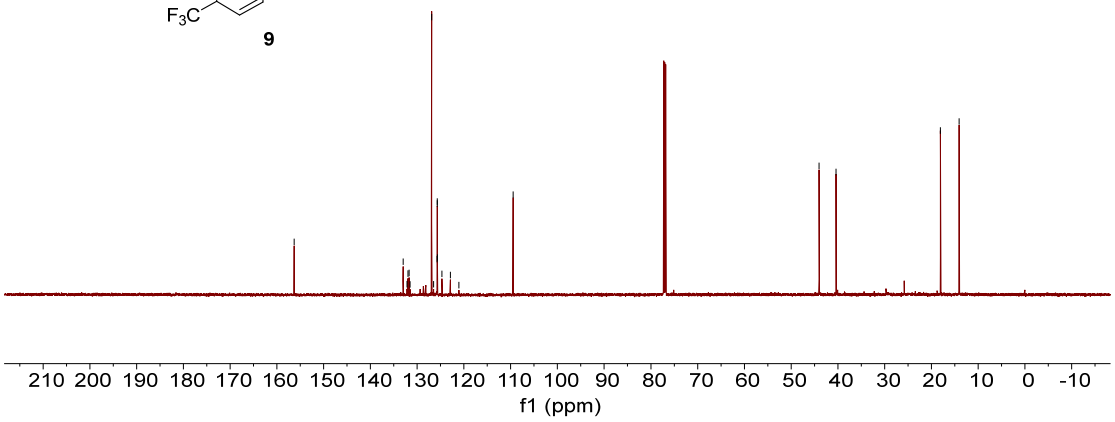
9

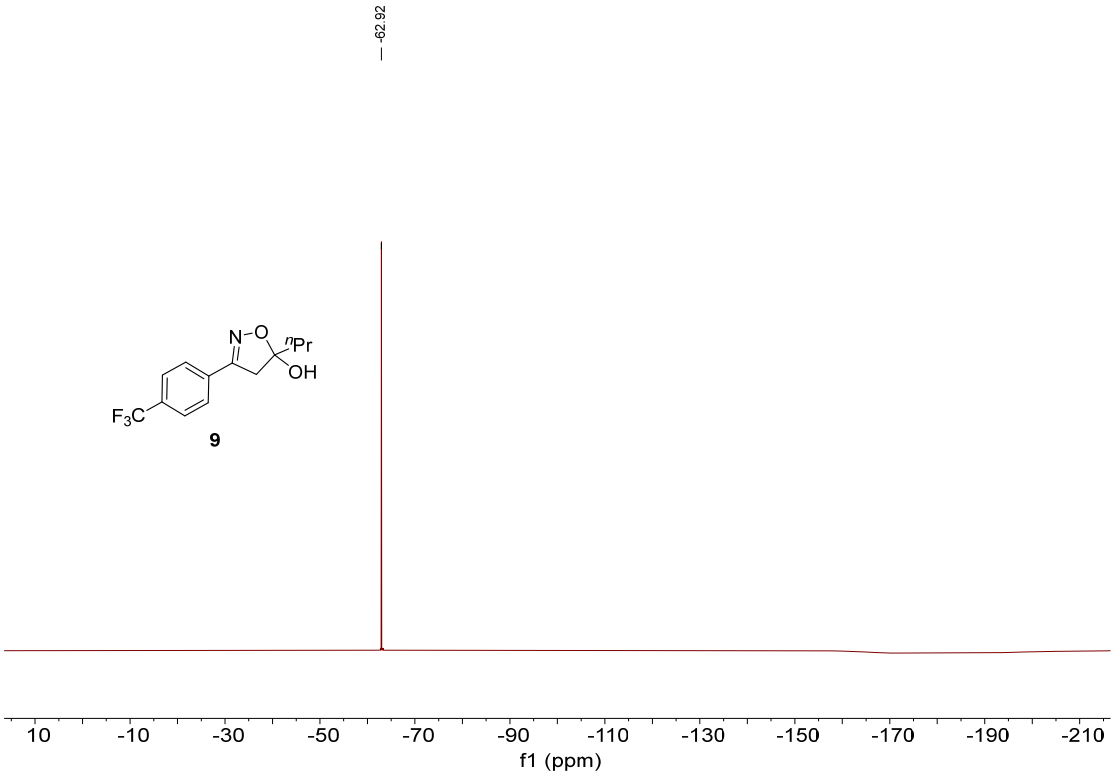

2.00
2.07
0.93
2.05
2.15
2.05
3.29
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

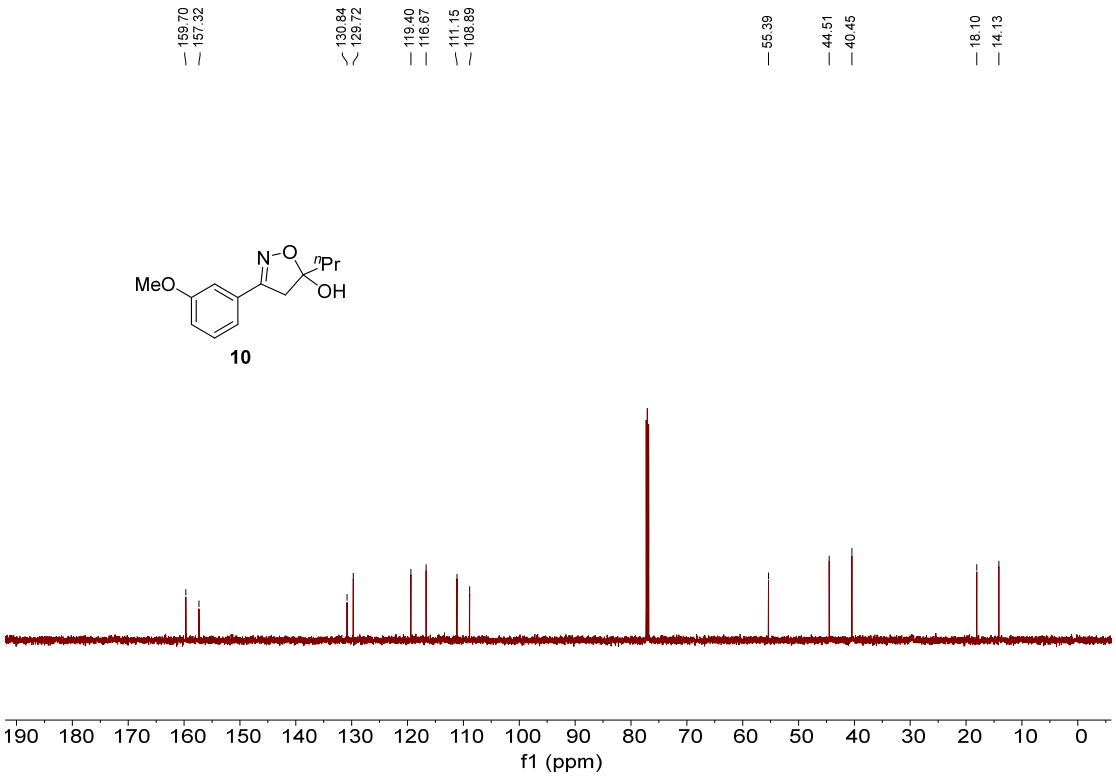

156.31
133.00
132.17
131.95
131.74
131.52
126.90
126.49
125.73
125.70
125.68
125.65
124.69
122.88
121.08
109.48
44.02
40.40
18.07
14.07

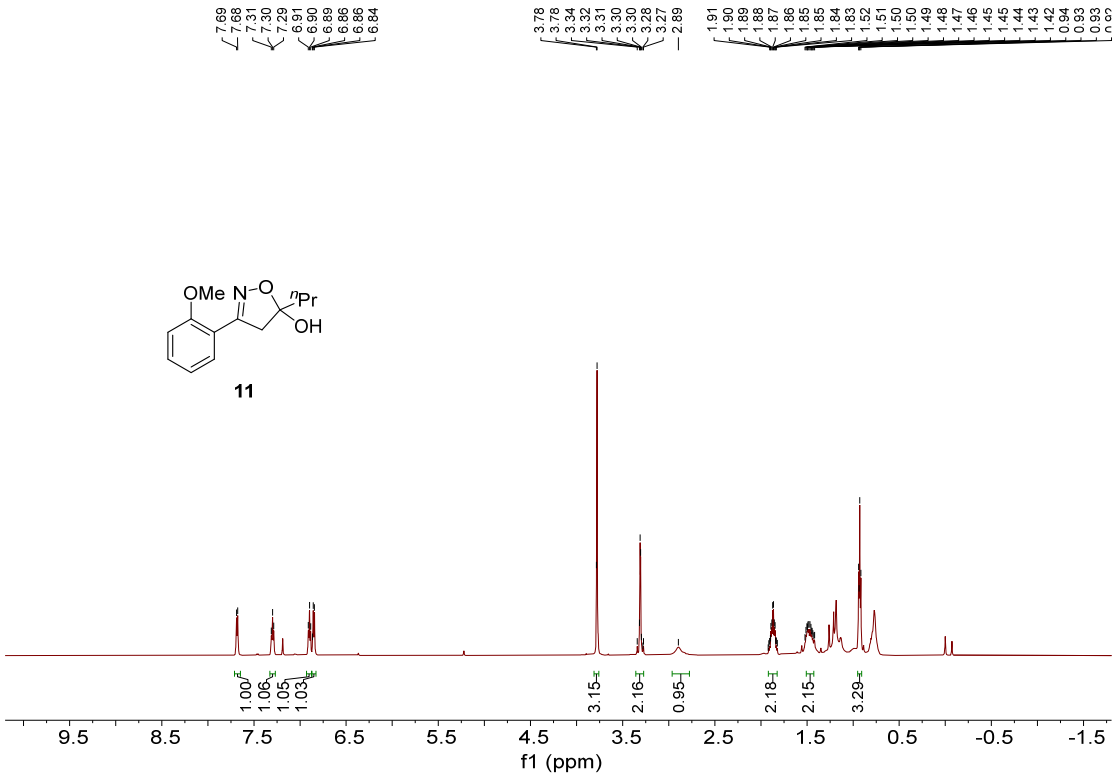

N
O
nPr
OH
F3C

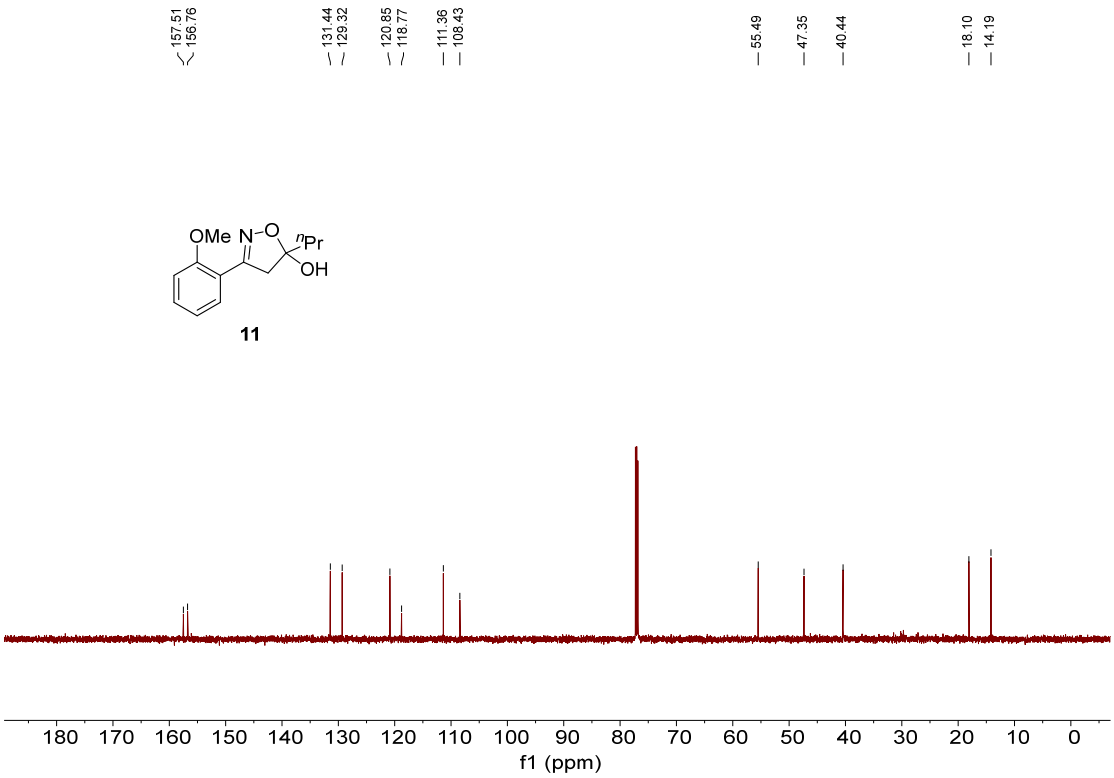
9


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

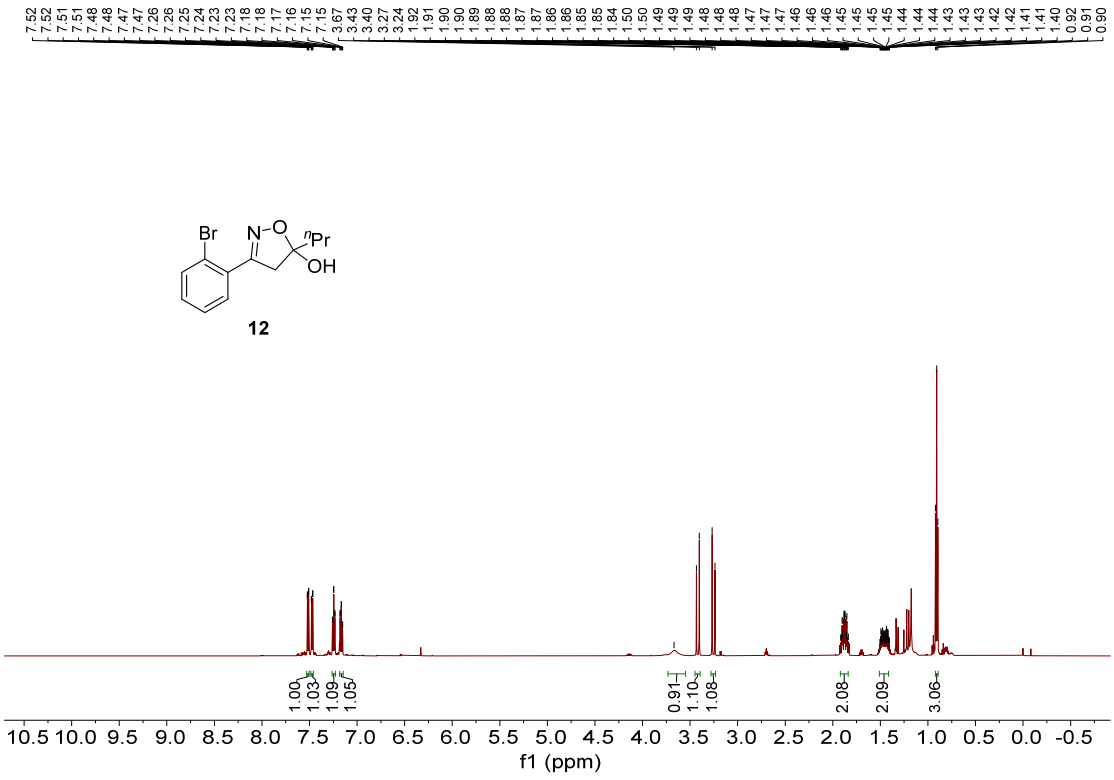

-62.92
N
O
nPr
OH
F3C
9
10
10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)
MeO
1.01
1.00
1.00
1.00
3.10
2.09
0.91
2.12
2.01
3.01
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


159.70
157.32
130.84
129.72
119.40
116.67
111.15
108.89
55.39
44.51
40.45
18.10
14.13
MeO
N
O
nPr
OH
10
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.69
7.68
7.31
7.30
7.29
6.91
6.90
6.89
6.86
6.86
6.84
3.78
3.78
3.34
3.32
3.31
3.30
3.30
3.28
3.27
2.89
1.91
1.90
1.89
1.88
1.87
1.86
1.85
1.85
1.84
1.83
1.52
1.51
1.50
1.50
1.49
1.48
1.47
1.46
1.45
1.45
1.44
1.43
1.42
0.94
0.93
0.93
OMe
N
O
nPr
OH
11
1.00
1.06
1.05
1.03
3.15
2.16
0.95
2.18
2.15
3.29
9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5 -1.5
f1 (ppm)


157.51
156.76
131.44
129.32
120.85
118.77
111.36
108.43
55.49
47.35
40.44
18.10
14.19
OMe
N
O
nPr
OH
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

11


7.52
7.52
7.51
7.51
7.48
7.48
7.47
7.47
7.26
7.25
7.24
7.23
7.23
7.18
7.18
7.17
7.16
7.15
7.15
3.67
3.43
3.40
3.27
3.24
1.92
1.91
1.90
1.90
1.89
1.88
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1.87
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1.87
1.86
1.86
1.85
1.85
1.84
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1.48
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1.47
1.47
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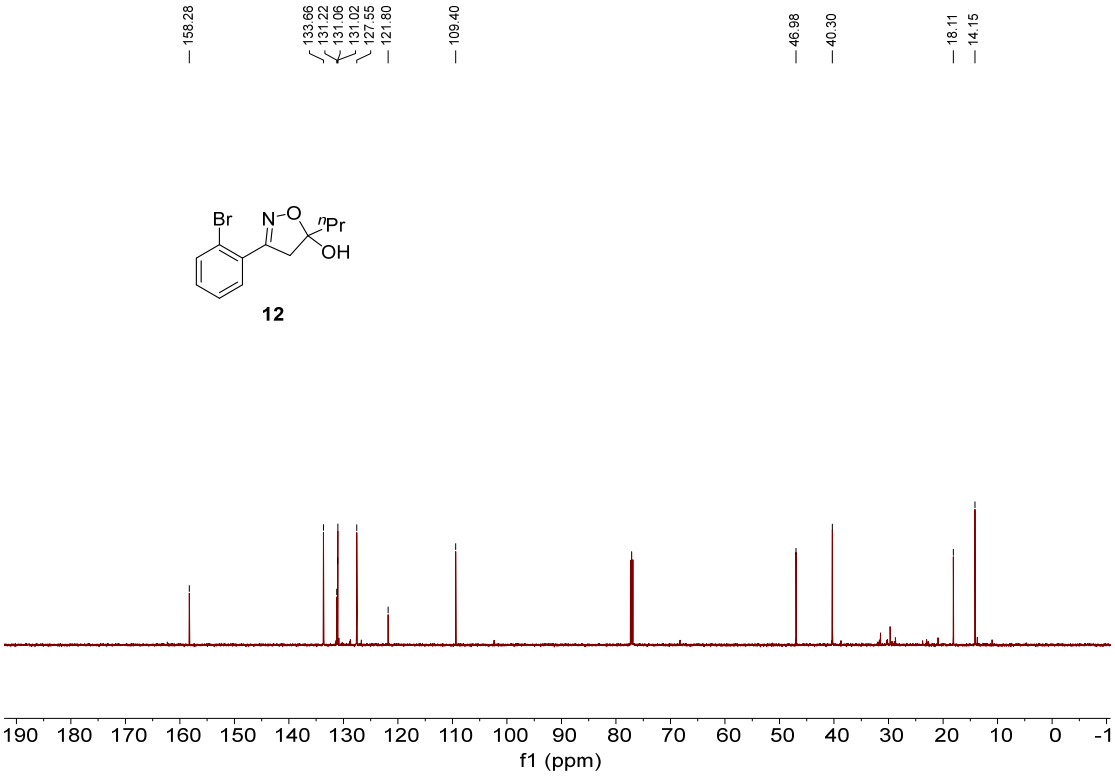
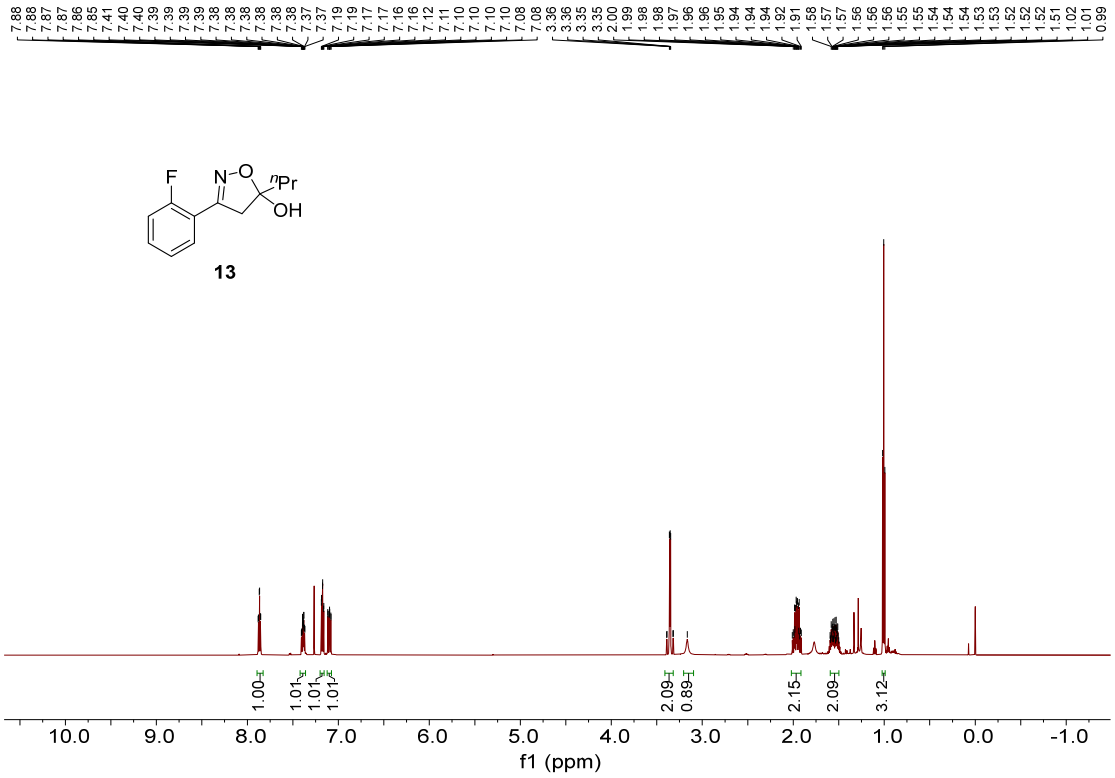
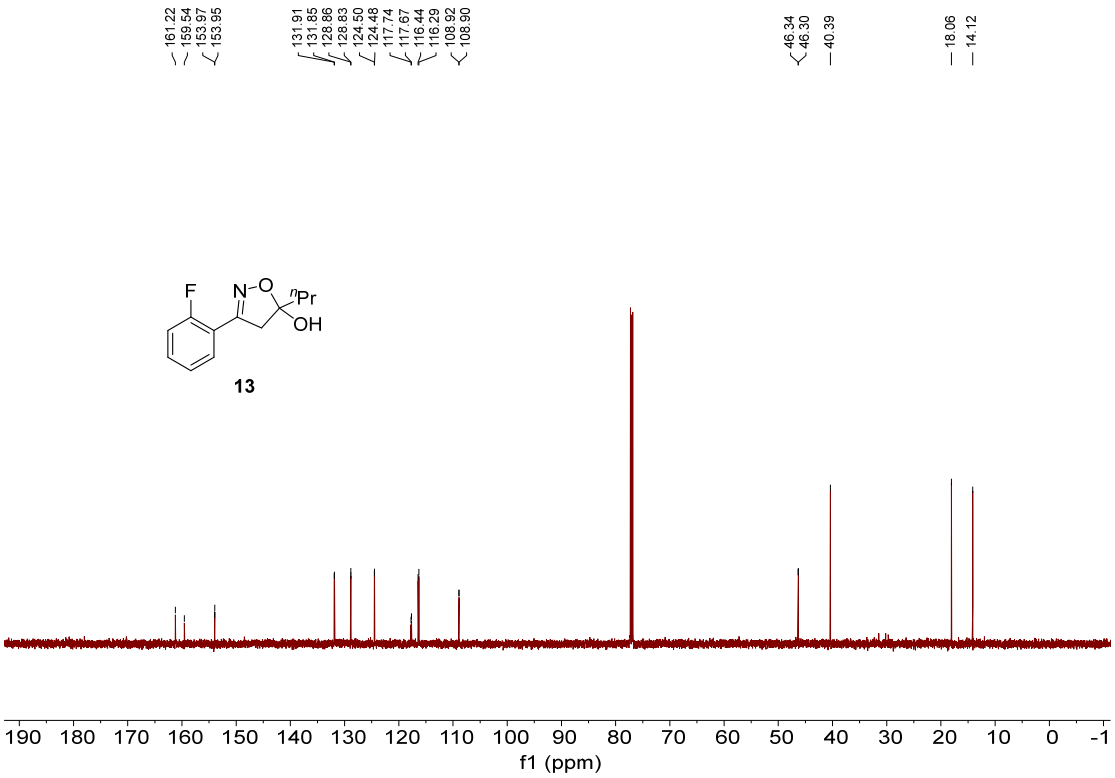
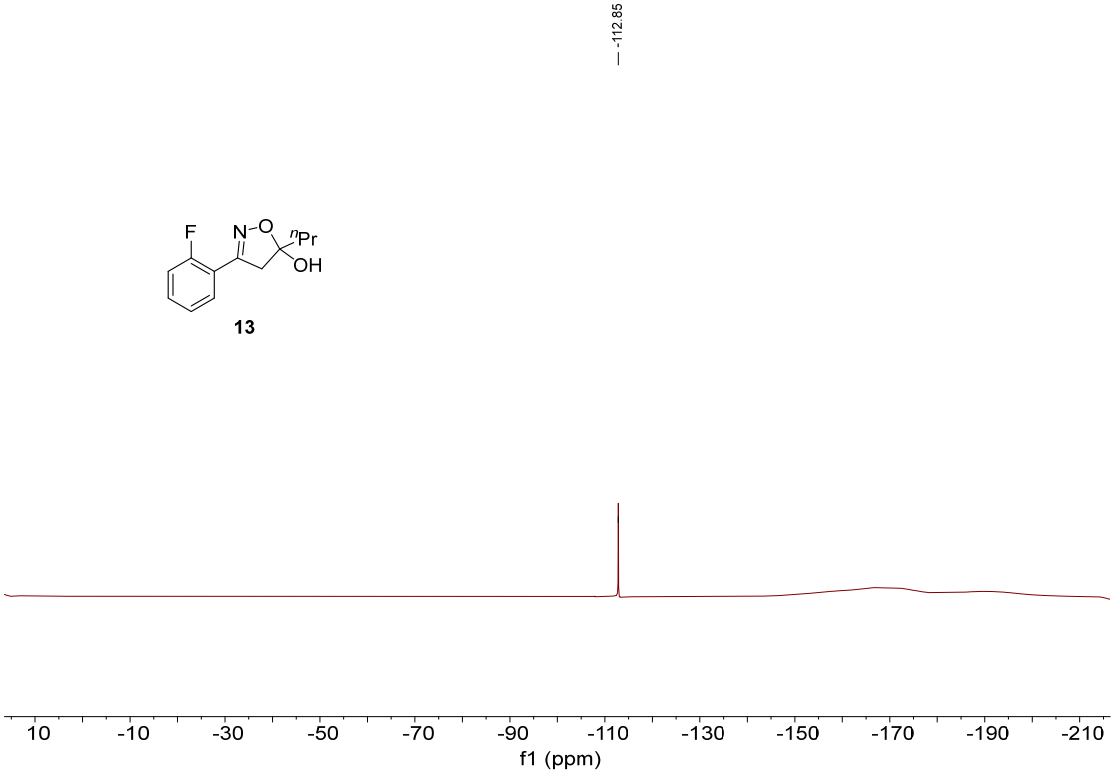
1.45
1.45
1.45
1.45
1.44
1.44
1.44
1.43
1.43
1.43
1.42
1.42
1.41
1.41
1.40
0.92
0.91
0.90
Br
N
O
nPr
OH
1.00
1.03
1.09
1.05
0.91
1.10
1.08
2.08
2.09
3.06
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

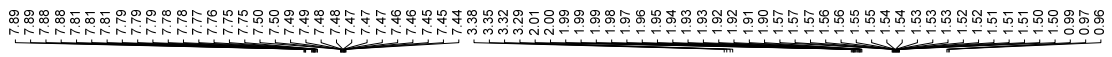
12

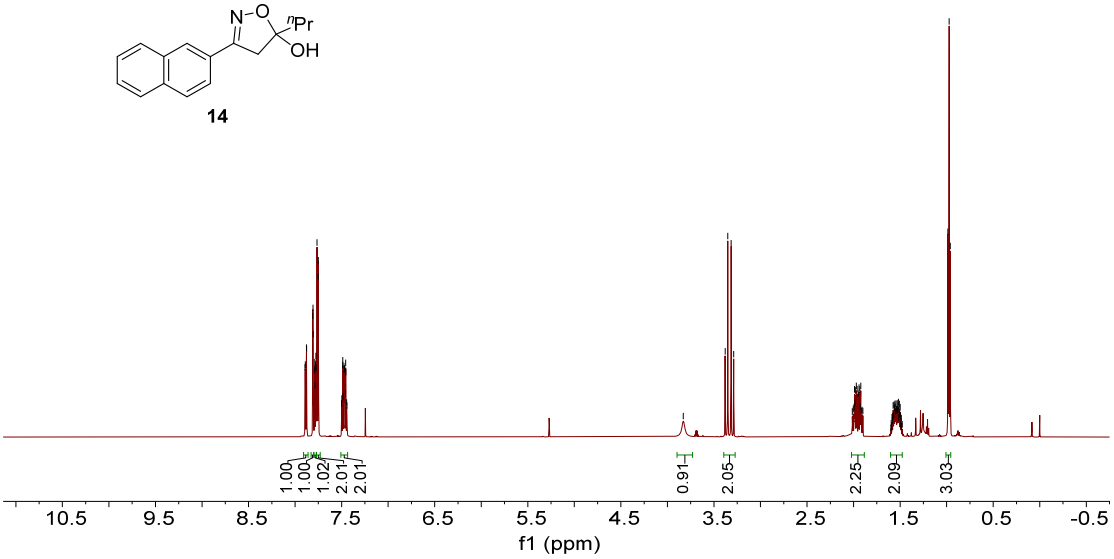

158.28
133.66
131.22
131.06
131.02
127.55
121.80
109.40
46.98
40.30
18.11
14.15
Br
N
O
nPr
OH
12
f1 (ppm)


F
N
O
nPr
OH
13
1.00
1.01
1.01
1.01
2.09
0.89
2.15
2.09
3.12
f1 (ppm)

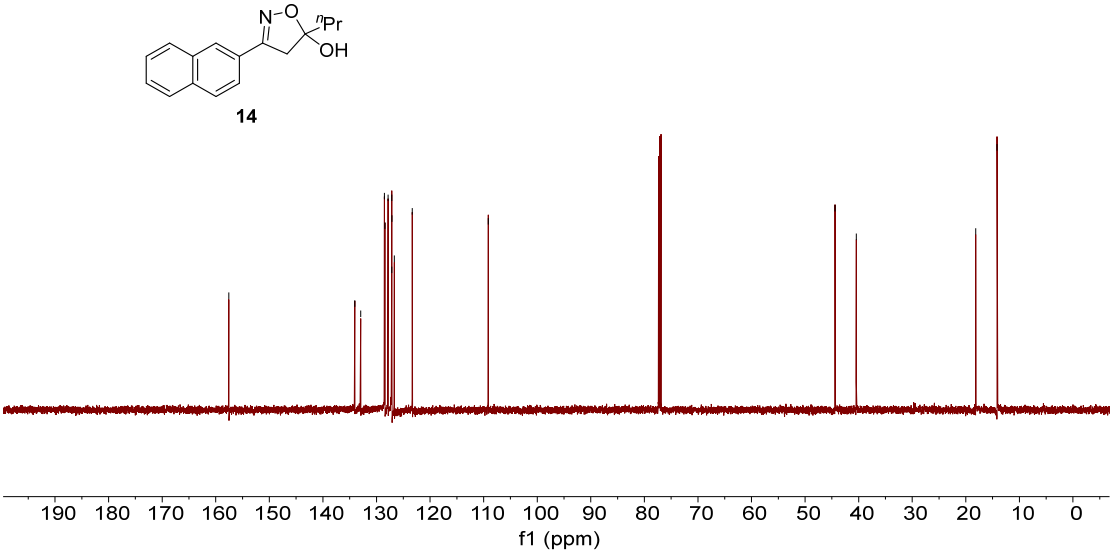

161.22
159.54
153.97
153.95
131.91
131.85
128.86
128.83
124.50
124.48
117.74
117.67
116.44
116.29
108.92
108.90
46.34
46.30
40.39
18.06
14.12
F
N
O
nPr
OH
13
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

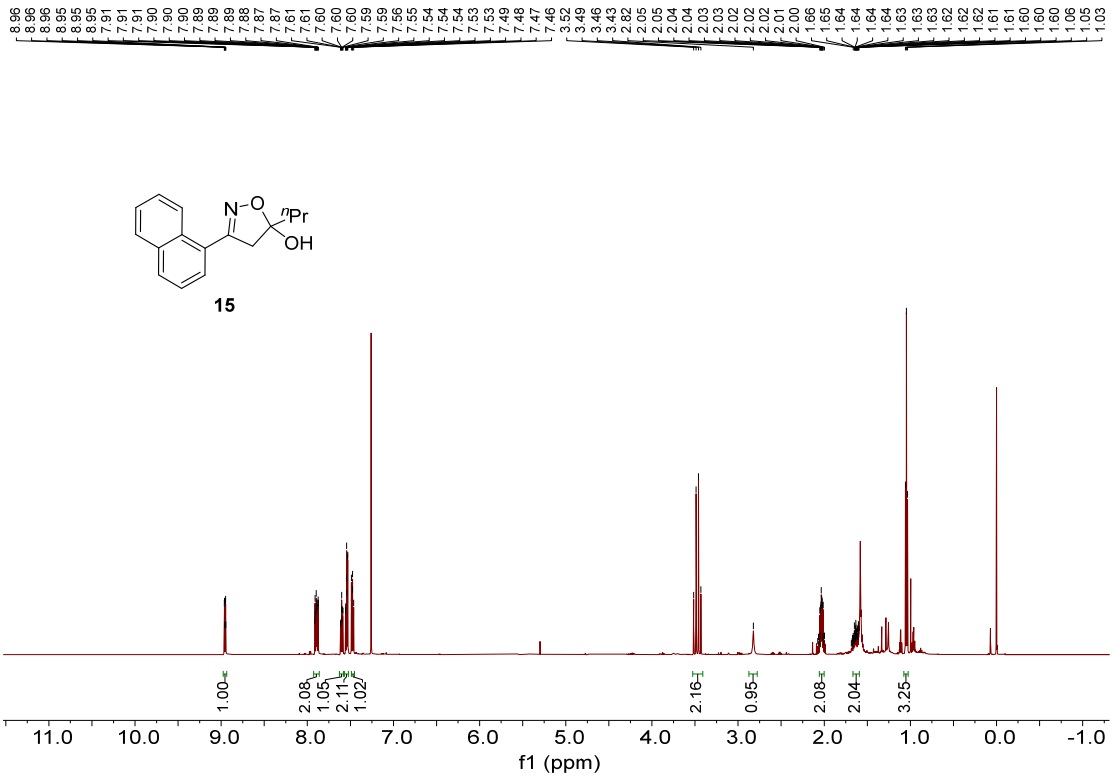

-112.85
F
N
O
nPr
OH
13
10 -10 -30 -50 -70 -90 -110 -130 -150 -170 -190 -210
f1 (ppm)

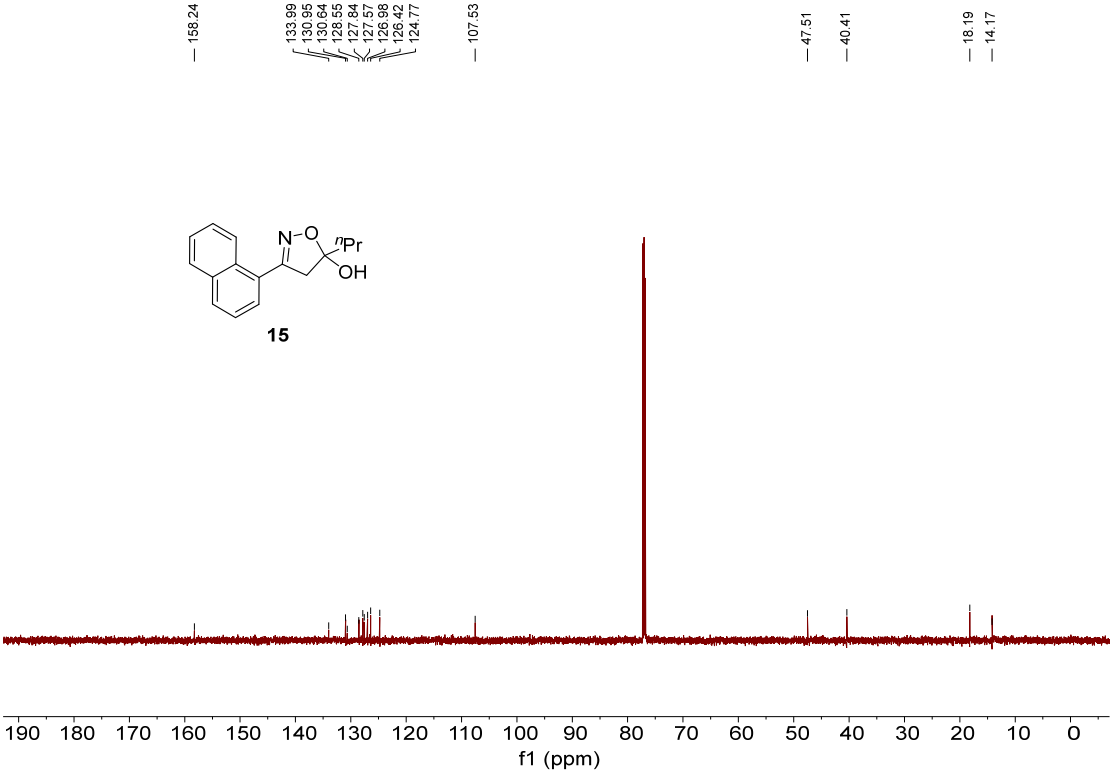

7.89 7.89 7.88 7.88 7.81 7.81 7.81 7.79 7.79 7.79 7.78 7.78 7.77 7.76 7.75 7.75 7.50 7.50 7.49 7.49 7.48 7.48 7.47 7.47 7.47 7.46 7.46 7.45 7.45 7.44
3.38 3.35 3.32 3.29 2.01 2.00 1.99 1.99 1.98 1.97 1.96 1.95 1.94 1.93 1.92 1.92 1.91 1.90 1.57 1.57 1.57 1.56 1.56 1.55 1.55 1.54 1.54 1.53 1.53 1.52 1.52 1.51 1.51 1.50 1.50 0.99 0.97 0.96


N
O
nPr
OH
14
1.00
1.00
1.02
2.01
2.01
0.91
2.05
2.25
2.09
3.03
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

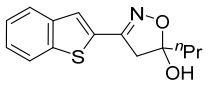

157.55
134.05
132.94
128.51
128.43
127.83
127.15
127.14
127.12
126.66
123.33
109.13
44.42
40.44
18.15
14.16


N
O
nPr
OH
14
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

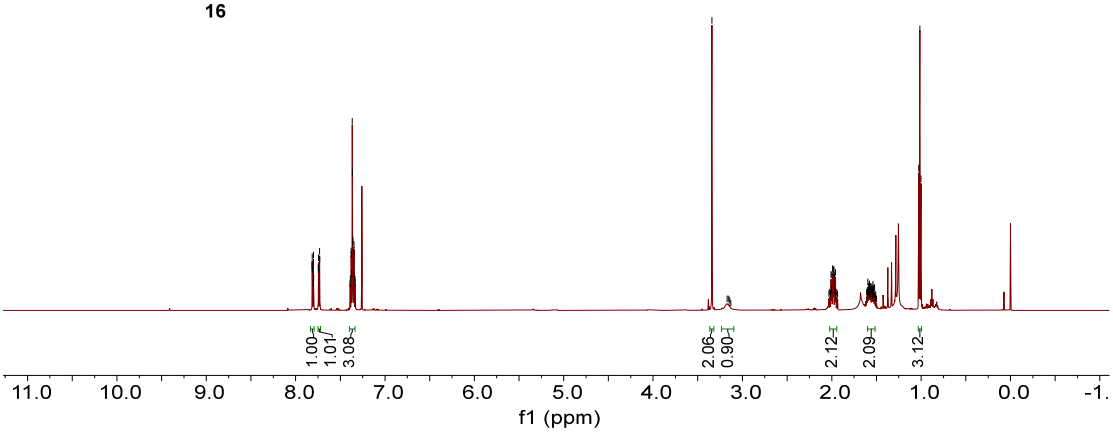

8.96 8.96 8.96 8.95 8.95 8.95 7.91 7.91 7.91 7.90 7.90 7.90 7.89 7.89 7.89 7.88 7.87 7.87 7.61 7.61 7.60 7.60 7.60 7.59 7.59 7.56 7.55 7.54 7.54 7.54 7.53 7.53 7.49 7.48 7.47 7.46
3.52 3.49 3.46 3.43 2.82 2.05 2.05 2.04 2.04 2.03 2.03 2.02 2.02 2.02 2.01 2.00 1.66 1.65 1.64 1.64 1.64 1.64 1.63 1.63 1.63 1.62 1.62 1.62 1.61 1.61 1.60 1.60 1.60 1.06 1.05 1.03
N O nPr OH
15
1.00 2.08 1.05 2.11 1.02 2.16 0.95 2.08 2.04 3.25
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

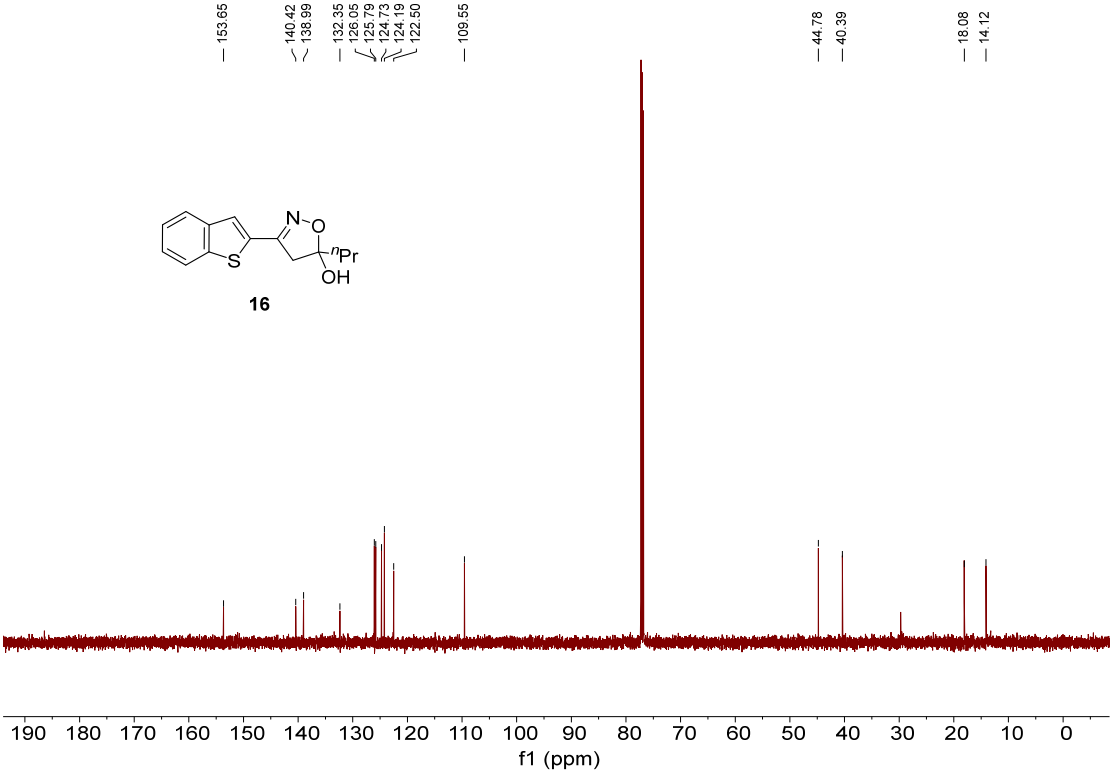

158.24
133.99 130.95 130.64 128.55 127.84 127.57 126.98 126.42 124.77
107.53
47.51
40.41
18.19
14.17
N O nPr OH
15
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.82 7.81 7.81 7.80 7.80 7.80 7.80 7.75 7.74 7.74 7.73 7.73 7.39 7.39 7.38 7.38 7.38 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.33 3.34 2.03 2.02 2.01 2.01 2.00 2.00 1.99 1.99 1.98 1.98 1.97 1.96 1.96 1.95 1.94 1.94 1.61 1.61 1.60 1.60 1.59 1.59 1.58 1.58 1.58 1.57 1.57 1.57 1.57 1.56 1.56 1.55 1.55 1.54 1.54 1.54 1.53 1.53 1.53 1.52 1.52 1.51 1.03 1.01 1.00


N
O
S
nPr
OH

16

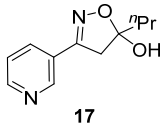

1.00
1.01
3.08
2.06
0.90
2.12
2.09
3.12
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.
f1 (ppm)


153.65
140.42
138.99
132.35
126.05
125.79
124.73
124.19
122.50
109.55
44.78
40.39
18.08
14.12
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

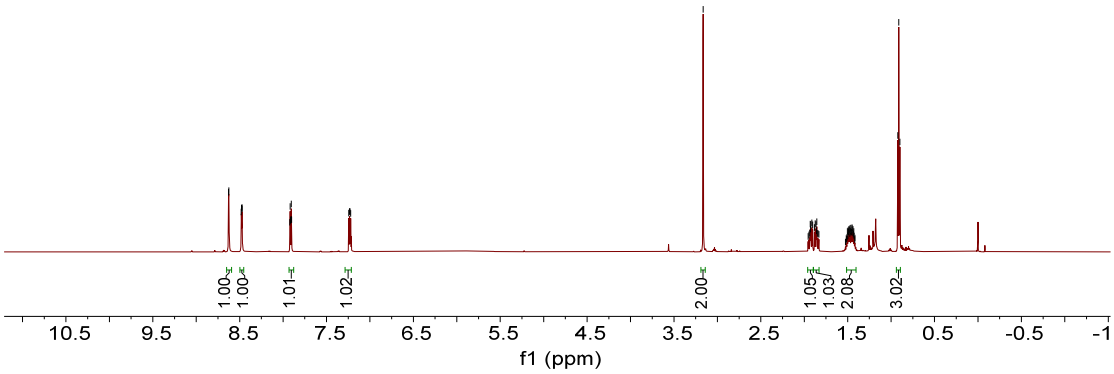
N
O
S
nPr
OH

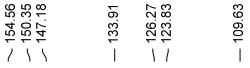
16

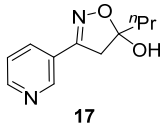

8.63
8.62
8.48
8.48
8.47
8.47
7.92
7.92
7.92
7.91
7.91
7.90
7.24
7.24
7.23
7.22
3.16
1.95
1.95
1.94
1.93
1.93
1.92
1.91
1.90
1.88
1.87
1.86
1.86
1.86
1.85
1.84
1.83
1.53
1.52
1.51
1.51
1.50
1.50
1.49
1.49
1.49
1.48
1.48
1.47
1.47
1.46
1.46
1.46
1.45
1.45
1.44
1.44
1.44
1.43
1.43
1.42
1.42
0.92
0.91
0.90


N
O
nPr
OH
N

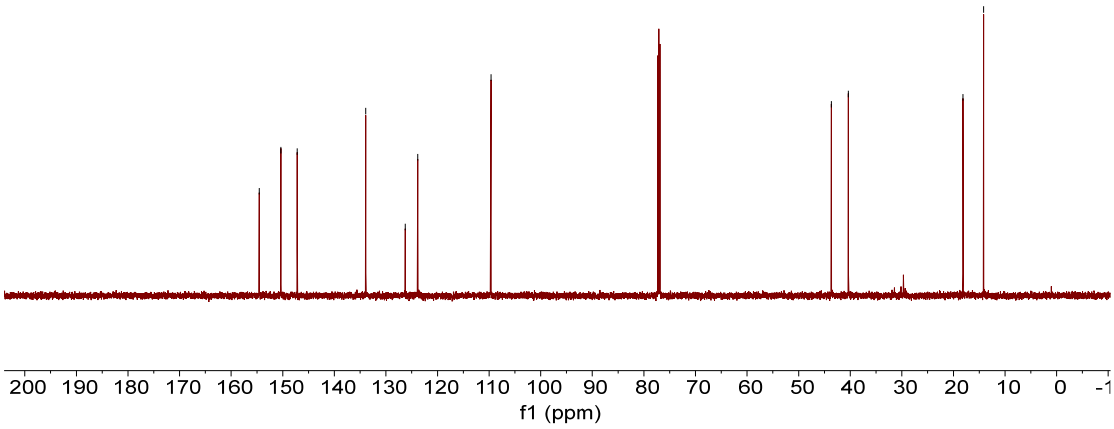
17

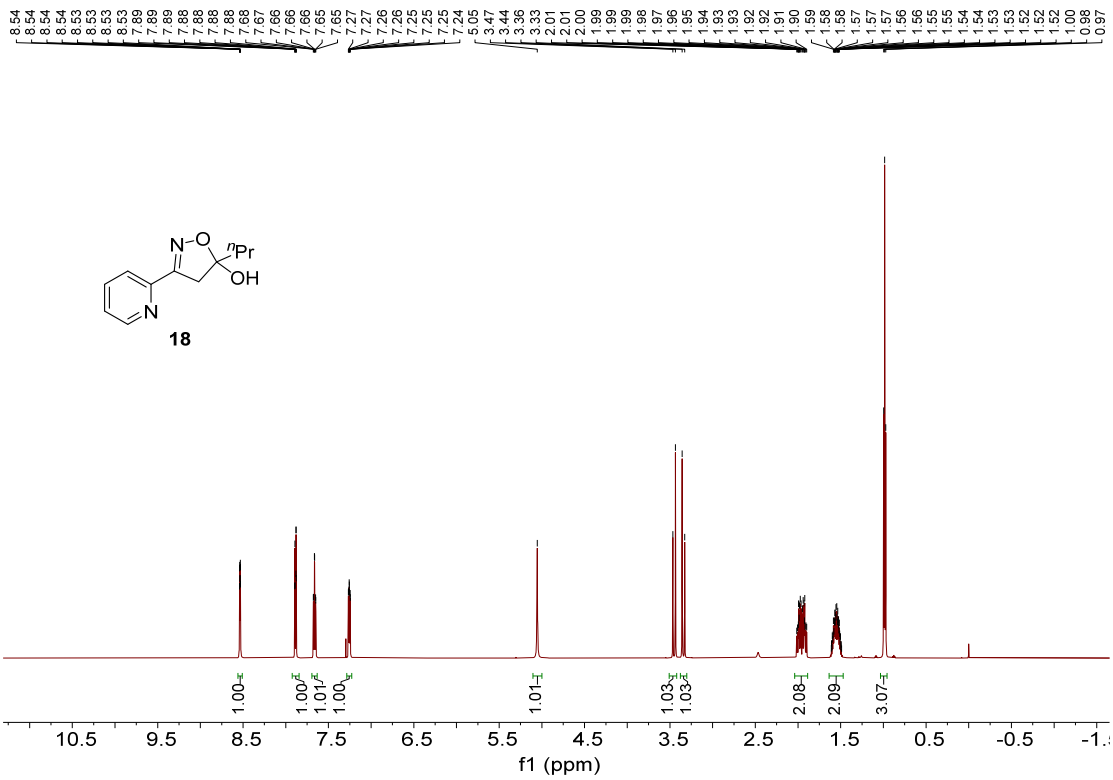

1.00
1.00
1.01
1.02
2.00
1.05
1.03
2.08
3.02
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1
f1 (ppm)

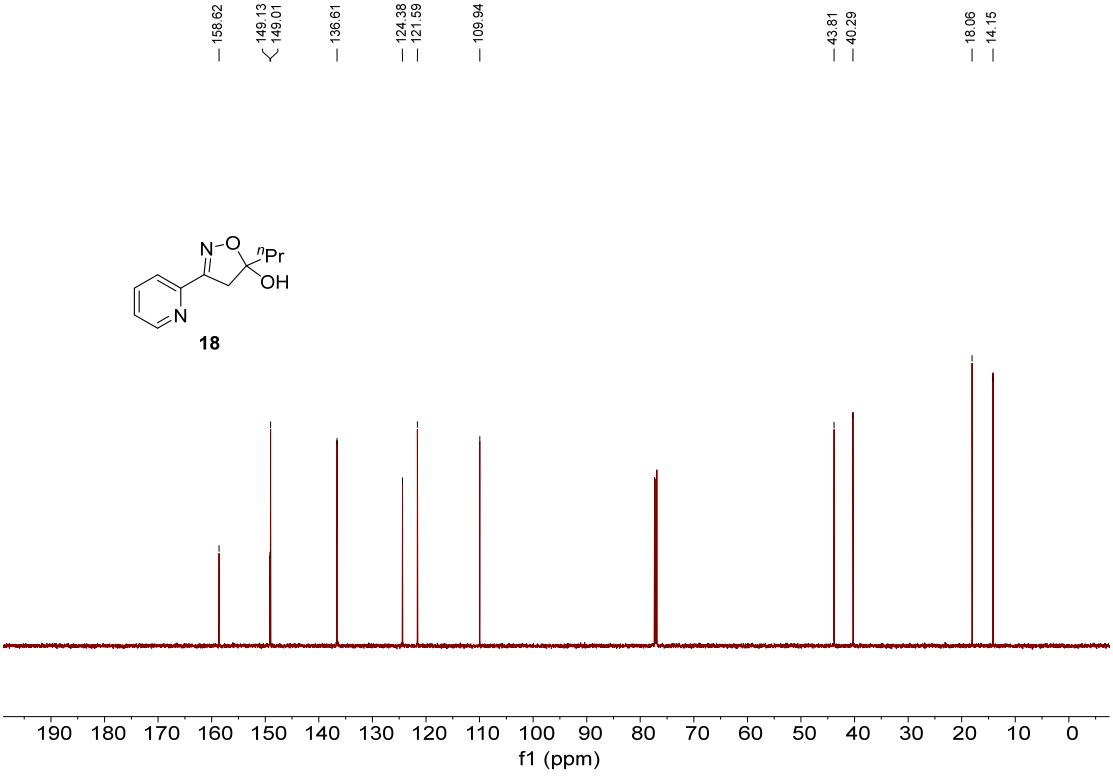

154.56
150.35
147.18
133.91
126.27
123.83
109.63
43.67
40.37
18.16
14.15


N
O
nPr
OH
N

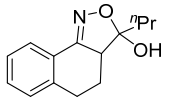
17


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


8.54 8.54 8.54 8.54 8.53 8.53 8.53 7.89 7.89 7.89 7.88 7.88 7.88 7.68 7.67 7.66 7.66 7.66 7.65 7.65 7.27 7.27 7.26 7.26 7.25 7.25 7.24 5.05 3.47 3.44 3.36 3.33 2.01 2.01 2.00 1.99 1.99 1.99 1.98 1.97 1.96 1.95 1.94 1.93 1.93 1.92 1.92 1.91 1.90 1.59 1.58 1.58 1.57 1.57 1.57 1.56 1.56 1.55 1.55 1.54 1.54 1.53 1.53 1.52 1.52 1.52 1.00 0.98 0.97
N
O
nPr
OH
N
18
1.00 1.00 1.01 1.00 1.01 1.03 1.03 2.08 2.09 3.07
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)


158.62
149.13
149.01
136.61
124.38
121.59
109.94
43.81
40.29
18.06
14.15
N
O
nPr
OH
N
18
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

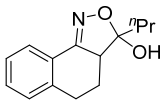

7.95 7.94 7.93 7.93 7.31 7.31 7.30 7.30 7.30 7.29 7.28 7.23 7.22 7.21 7.20 7.20 7.19 7.17 3.39 3.12 3.11 3.10 3.09 2.97 2.95 2.95 2.94 2.94 2.91 2.90 2.88 2.88 2.09 2.08 2.07 2.07 2.07 2.06 2.06 2.05 2.05 2.04 2.02 2.02 2.02 2.00 1.99 1.97 1.97 1.96 1.96 1.95 1.94 1.59 1.59 1.58 1.58 1.57 1.57 1.57 1.56 1.56 1.56 1.55 1.55 1.54 1.54 1.53 1.00 0.99 0.98


N
O
nPr
OH

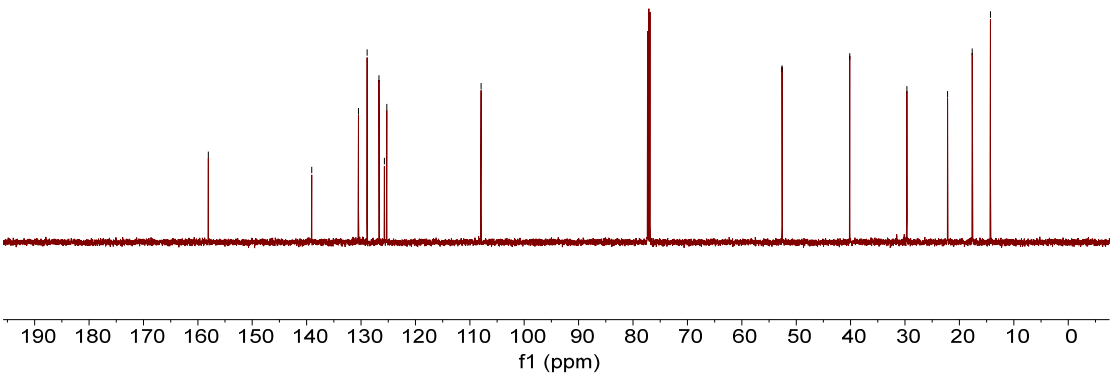
19

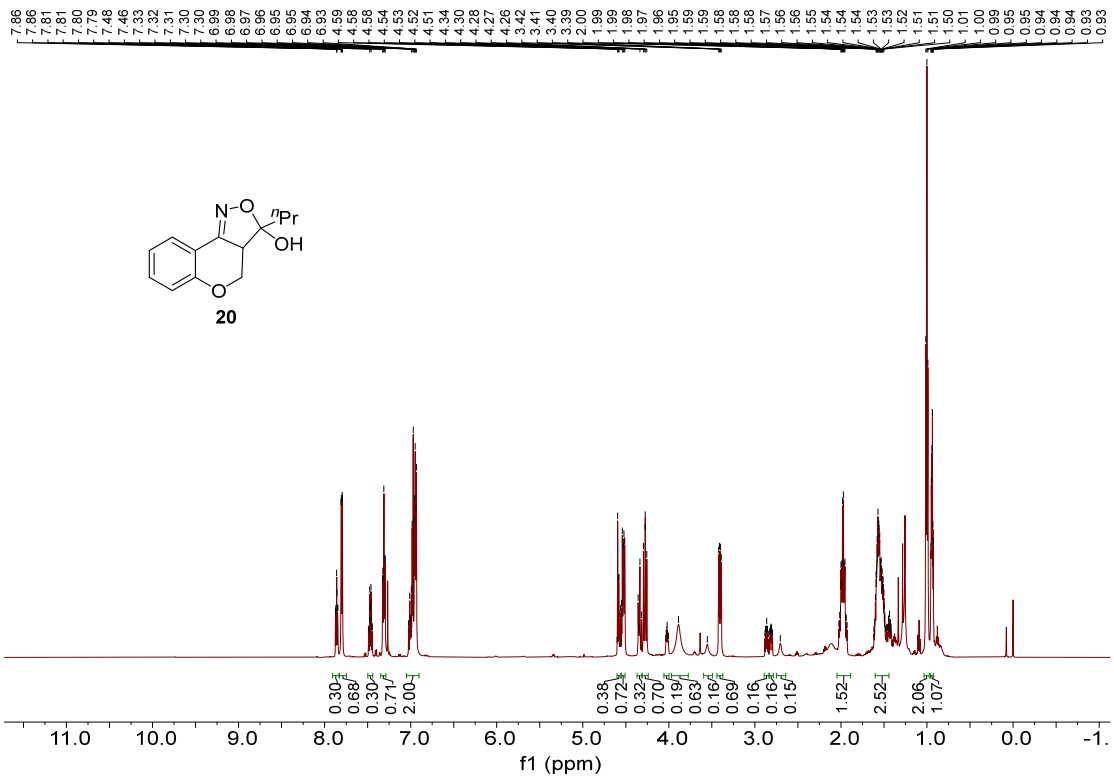
1.00
1.01
1.07
1.01
1.01
1.00
1.05
1.02
1.07
1.13
2.07
2.15
3.06
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0 -2
f1 (ppm)

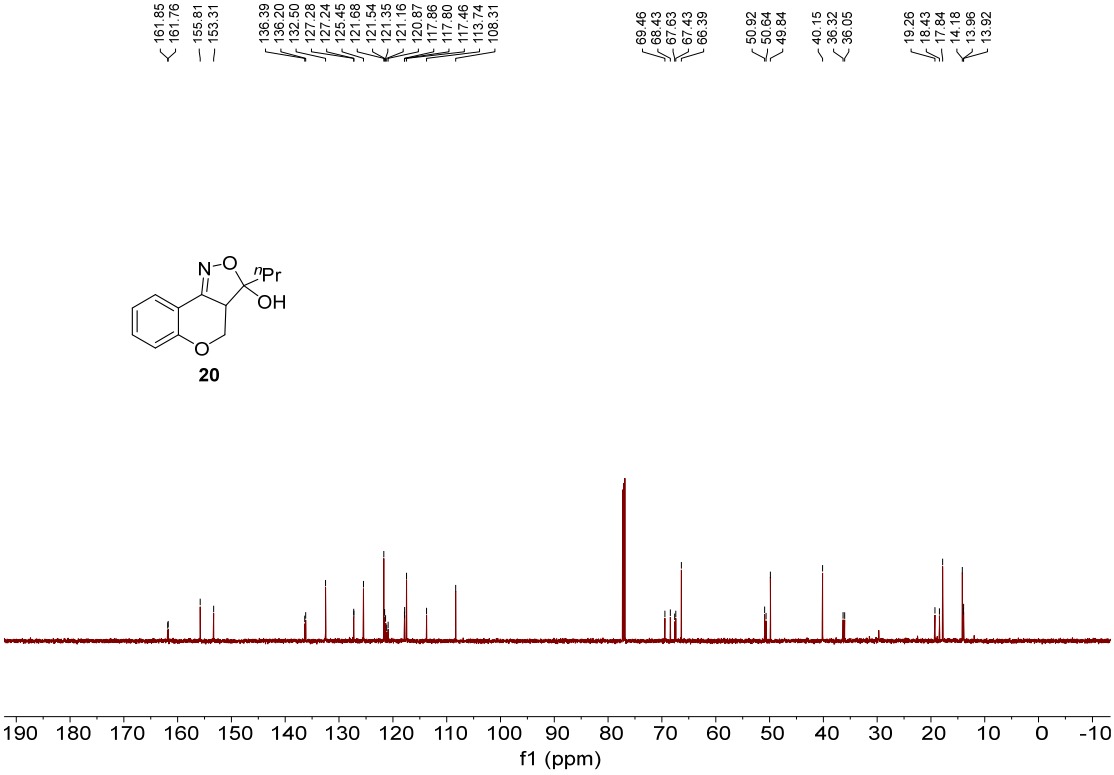

158.08
139.06
130.47
128.87
126.69
125.70
125.27
107.94
52.61
40.16
29.66
22.16
17.64
14.30

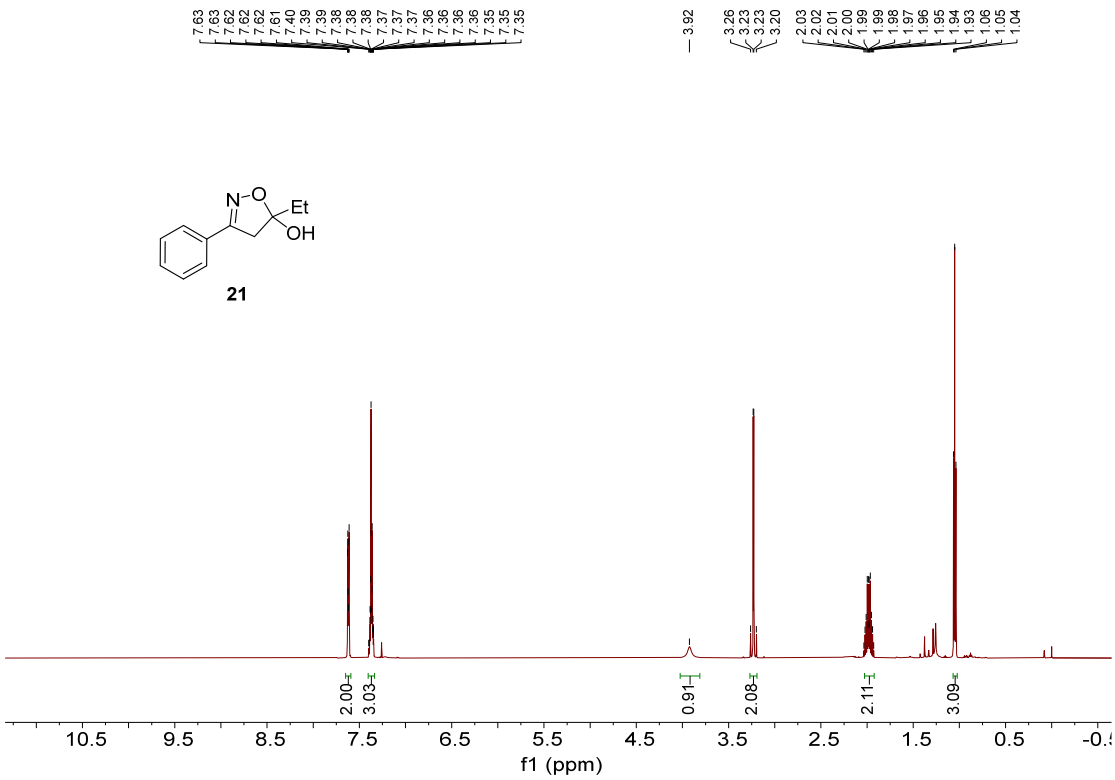

N
O
nPr
OH

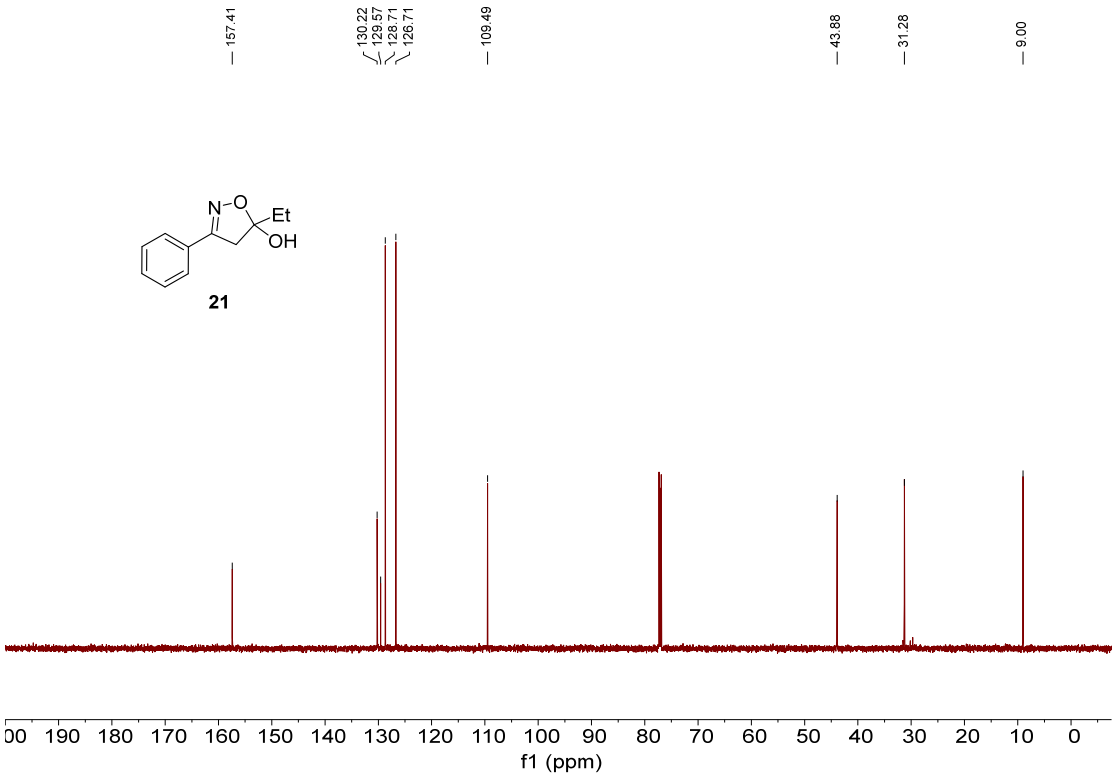
19

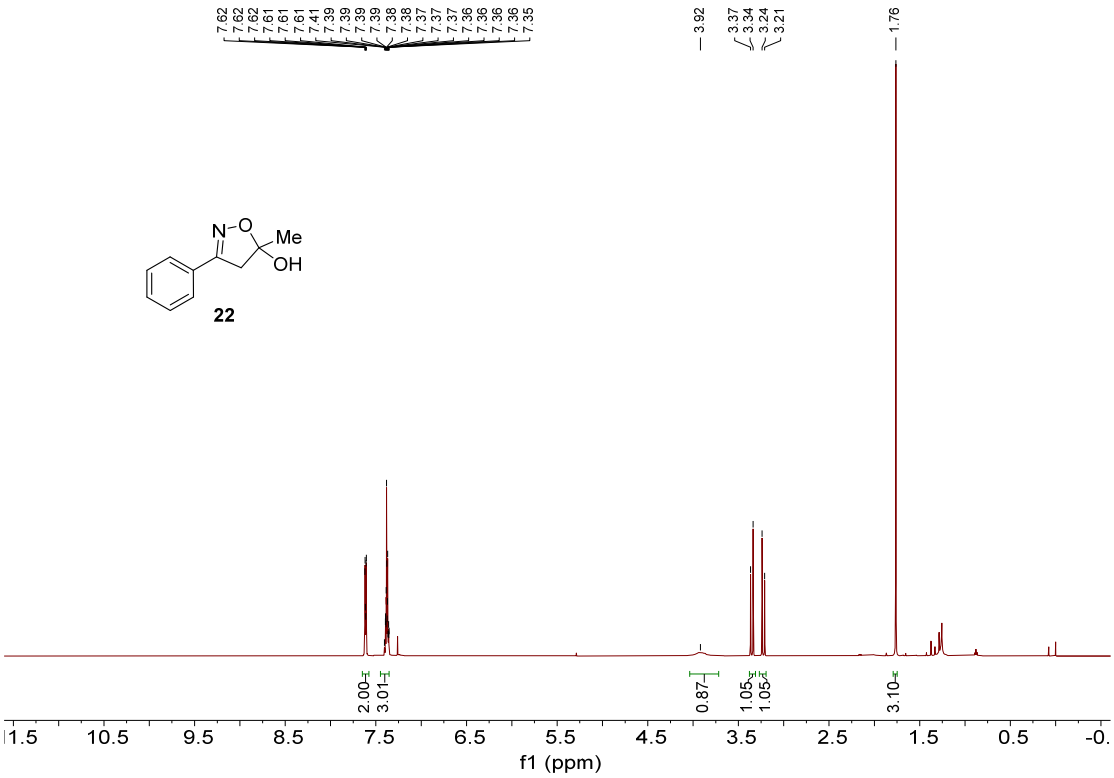

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

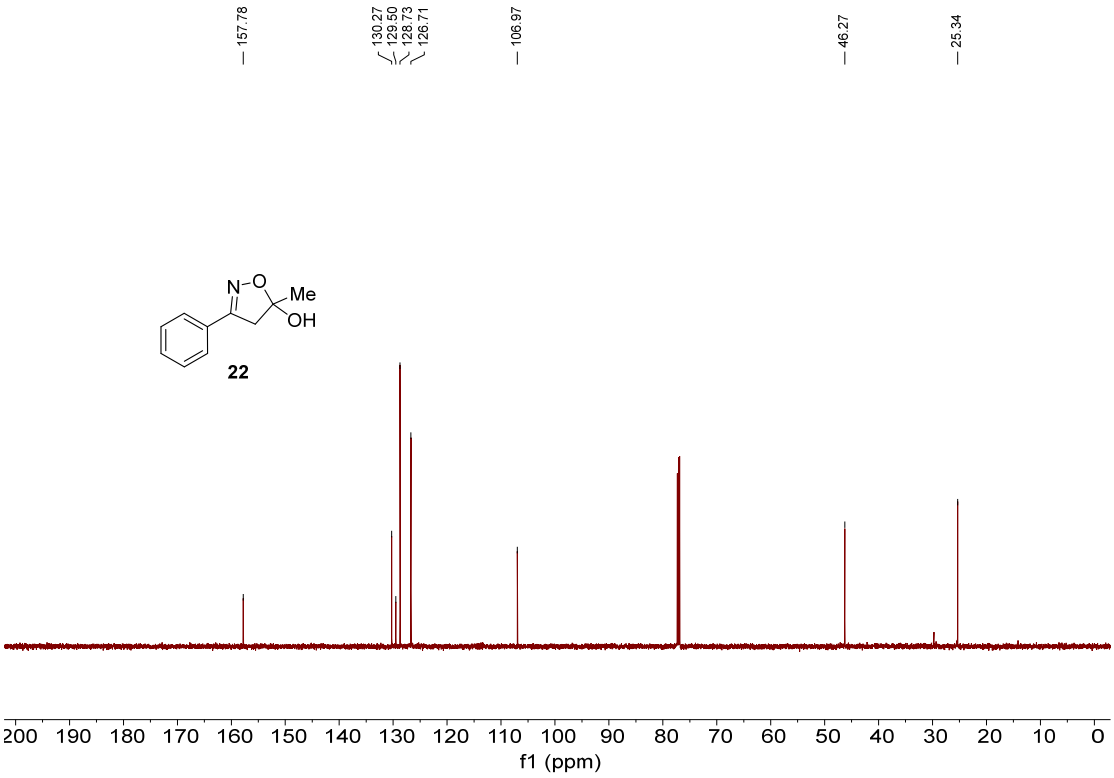

7.86 7.86 7.81 7.81 7.80 7.79 7.48 7.46 7.33 7.32 7.31 7.30 7.30 6.99 6.98 6.97 6.96 6.95 6.95 6.94 6.93 4.59 4.58 4.58 4.54 4.53 4.52 4.51 4.34 4.30 4.28 4.27 4.26 3.42 3.41 3.40 3.39 2.00 1.99 1.99 1.98 1.97 1.96 1.95 1.59 1.59 1.58 1.58 1.58 1.57 1.56 1.56 1.55 1.54 1.54 1.54 1.53 1.53 1.52 1.51 1.51 1.50 1.01 1.00 0.99 0.95 0.95 0.94 0.94 0.94 0.93 0.93
N O nPr OH O
20
0.30 0.68 0.30 0.71 2.00 0.38 0.72 0.32 0.70 0.19 0.63 0.16 0.69 0.16 0.16 0.15 1.52 2.52 2.06 1.07
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.
f1 (ppm)

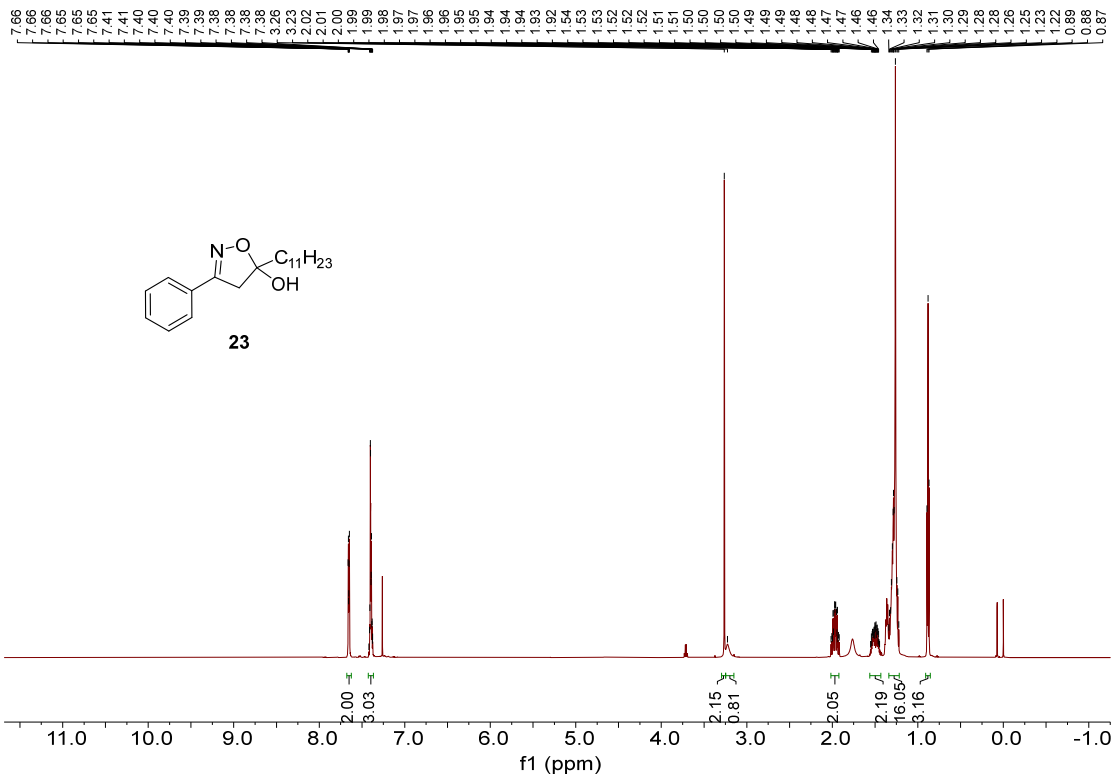

161.85 161.76 155.81 153.31 136.39 136.20 132.50 127.28 127.24 125.45 121.68 121.54 121.35 121.16 120.87 117.86 117.80 117.46 113.74 108.31 69.46 68.43 67.63 67.43 66.39 50.92 50.64 49.84 40.15 36.32 36.05 19.26 18.43 17.84 14.18 13.96 13.92
N O nPr OH O
20
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

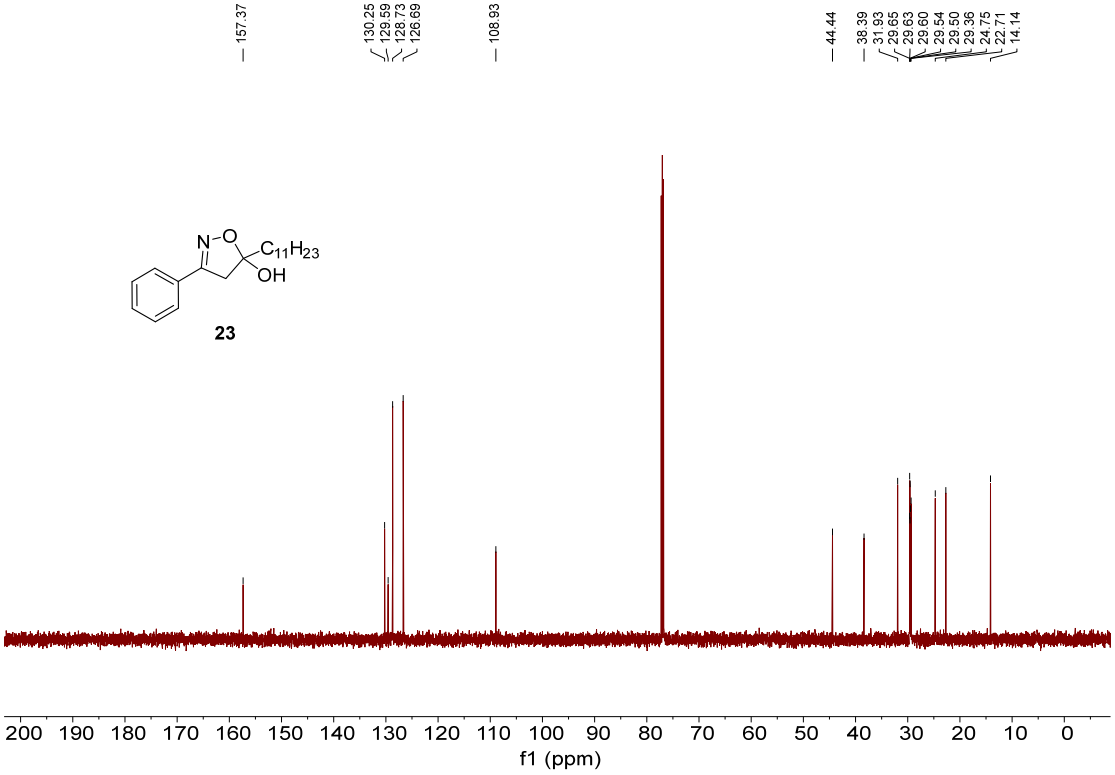

7.63
7.63
7.62
7.62
7.62
7.61
7.40
7.39
7.39
7.38
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.36
7.35
7.35
7.35
3.92
3.26
3.23
3.23
3.20
2.03
2.02
2.01
2.00
1.99
1.99
1.98
1.97
1.96
1.95
1.94
1.93
1.06
1.05
1.04
N
O
Et
OH
21
2.00
3.03
0.91
2.08
2.11
3.09
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

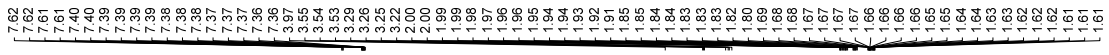

157.41
130.22
129.57
128.71
126.71
109.49
43.88
31.28
9.00
N
O
Et
OH
21
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

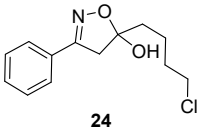

7.62
7.62
7.62
7.61
7.61
7.61
7.41
7.39
7.39
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.36
7.35
3.92
3.37
3.34
3.24
3.21
1.76
N
O
Me
OH
22
2.00
3.01
0.87
1.05
1.05
3.10
1.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.
f1 (ppm)


157.78
130.27
129.50
128.73
126.71
106.97
46.27
25.34
N
O
Me
OH
22
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

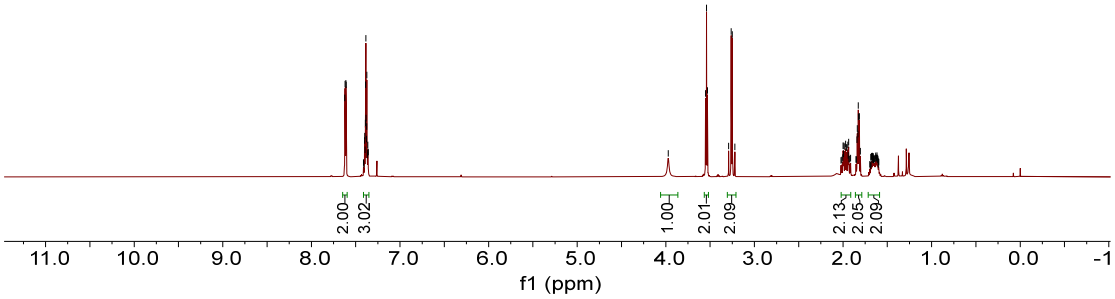

7.66 7.66 7.66 7.65 7.65 7.65 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.38 7.38 7.38 3.26 3.23 2.02 2.01 2.00 2.00 1.99 1.98 1.97 1.97 1.96 1.96 1.95 1.95 1.94 1.94 1.94 1.93 1.92 1.54 1.53 1.53 1.52 1.52 1.52 1.51 1.51 1.50 1.50 1.50 1.50 1.49 1.49 1.49 1.48 1.48 1.47 1.47 1.46 1.46 1.34 1.33 1.32 1.31 1.30 1.29 1.28 1.28 1.26 1.25 1.23 1.22 0.89 0.88 0.87
N O
C11H23
OH
23
2.00 3.03 2.15 0.81 2.05 2.19 16.05 3.16
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)


157.37
130.25 129.59 128.73 126.69
108.93
44.44
38.39 31.93 29.65 29.63 29.60 29.54 29.50 29.36 24.75 22.71 14.14
N O
C11H23
OH
23
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

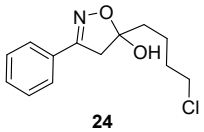

7.62 7.62 7.61 7.61 7.40 7.40 7.39 7.39 7.39 7.39 7.38 7.38 7.38 7.37 7.37 7.37 7.36 7.36 3.97 3.55 3.54 3.53 3.29 3.26 3.25 3.22 2.00 2.00 1.99 1.99 1.98 1.97 1.96 1.96 1.95 1.94 1.94 1.93 1.92 1.91 1.85 1.85 1.84 1.84 1.83 1.83 1.82 1.80 1.69 1.68 1.68 1.67 1.67 1.67 1.67 1.66 1.66 1.66 1.66 1.65 1.65 1.64 1.64 1.63 1.63 1.62 1.62 1.62 1.61 1.61 1.61


N
O
OH
Cl

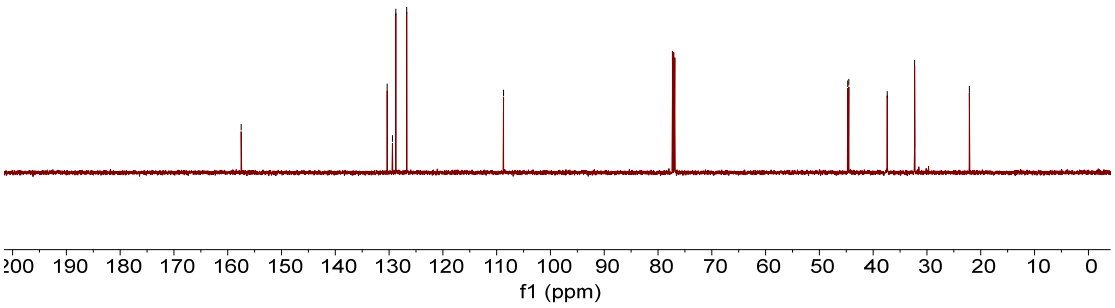
24

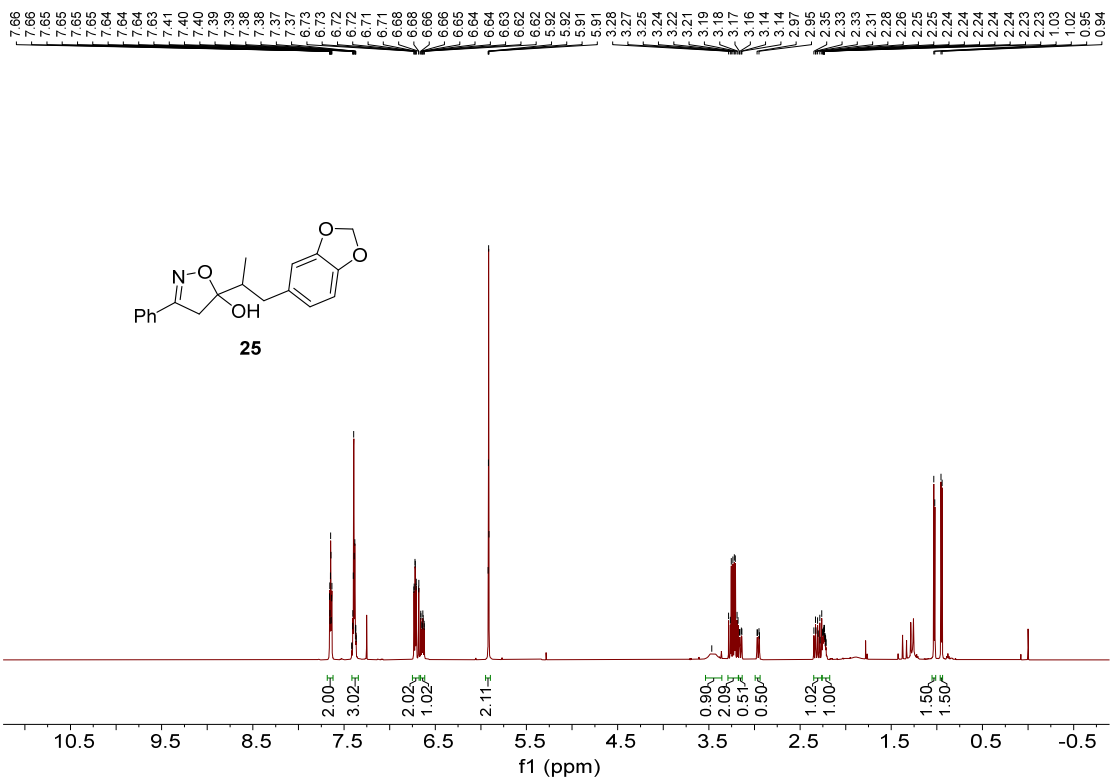

2.00
3.02
1.00
2.01
2.09
2.13
2.05
2.09
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1
f1 (ppm)

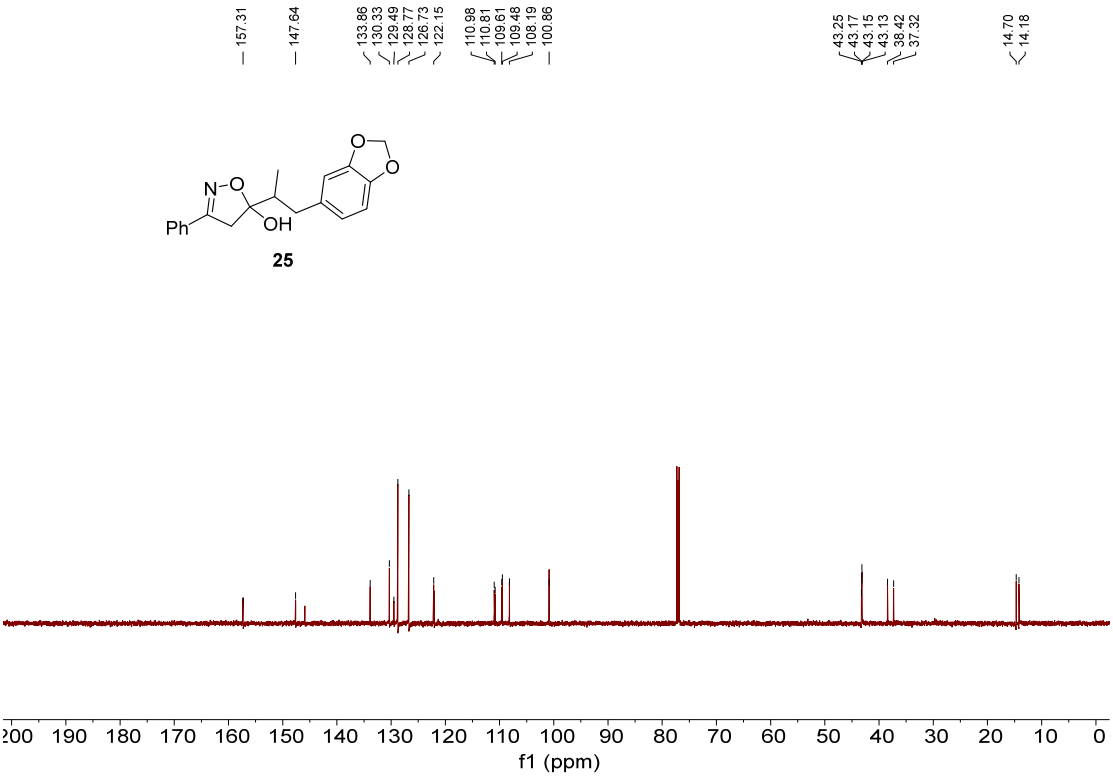

157.49
130.35
129.39
128.77
126.72
108.74
44.76
44.55
37.39
32.29
22.12

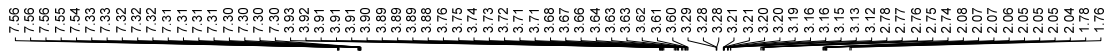

N
O
OH
Cl

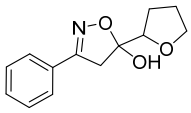
24


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

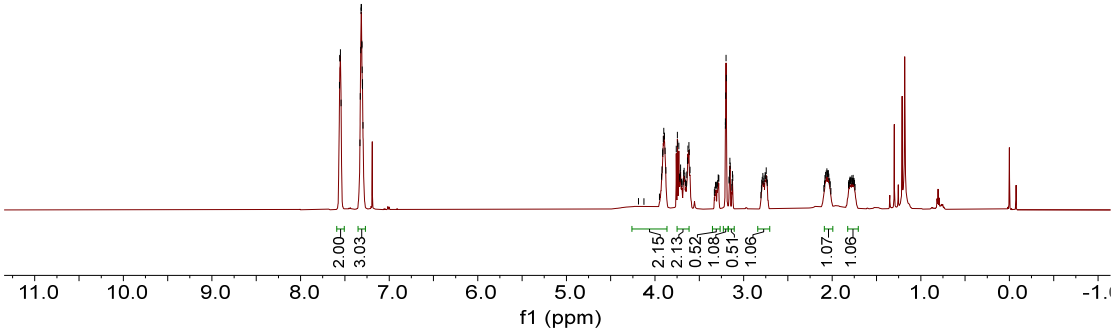

7.66 7.66 7.65 7.65 7.65 7.65 7.64 7.64 7.64 7.63 7.41 7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.37 6.73 6.73 6.72 6.72 6.71 6.71 6.68 6.68 6.66 6.66 6.65 6.64 6.64 6.63 6.62 6.62 5.92 5.92 5.91 5.91
3.28 3.27 3.25 3.24 3.22 3.21 3.19 3.18 3.17 3.16 3.14 3.14 2.97 2.95 2.35 2.33 2.31 2.28 2.26 2.25 2.25 2.24 2.24 2.24 2.24 2.24 2.23 2.23 1.03 1.02 0.95 0.94
N
O
O
O
Ph
OH
25
2.00 3.02 2.02 1.02 2.11 0.90 2.09 0.51 0.50 1.02 1.00 1.50 1.50
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

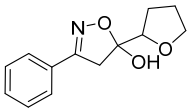

157.31 147.64 133.86 130.33 129.49 128.77 126.73 122.15 110.98 110.81 109.61 109.48 108.19 100.86
43.25 43.17 43.15 43.13 38.42 37.32 14.70 14.18
N
O
O
O
Ph
OH
25
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



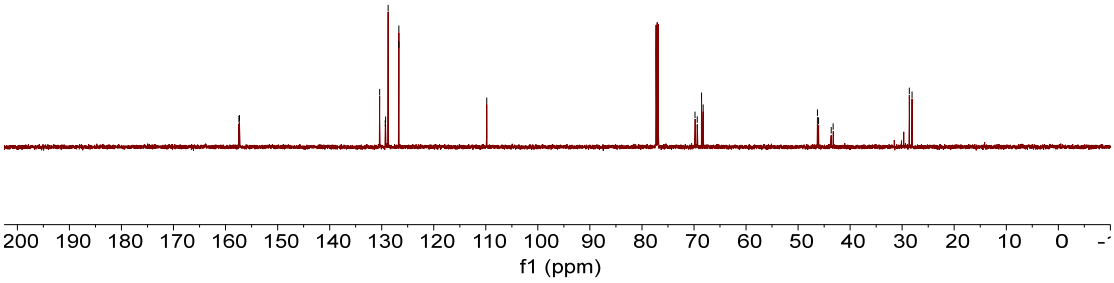


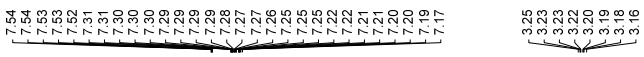
26

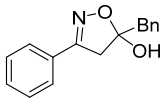

2.00
3.03
2.15
2.13
0.52
1.08
0.51
1.06
1.07
1.06
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)



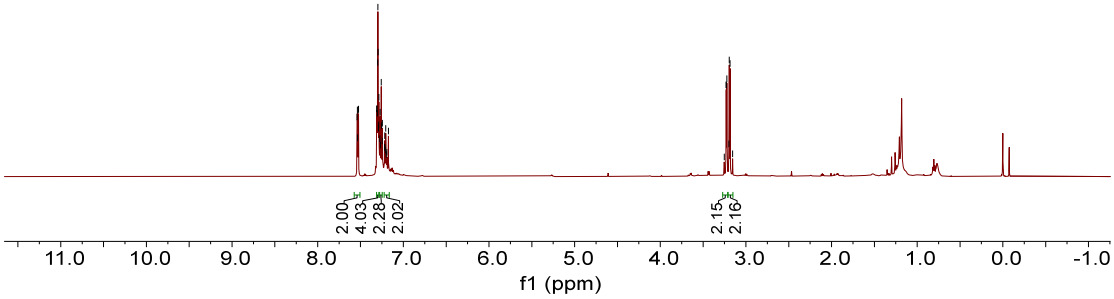
26

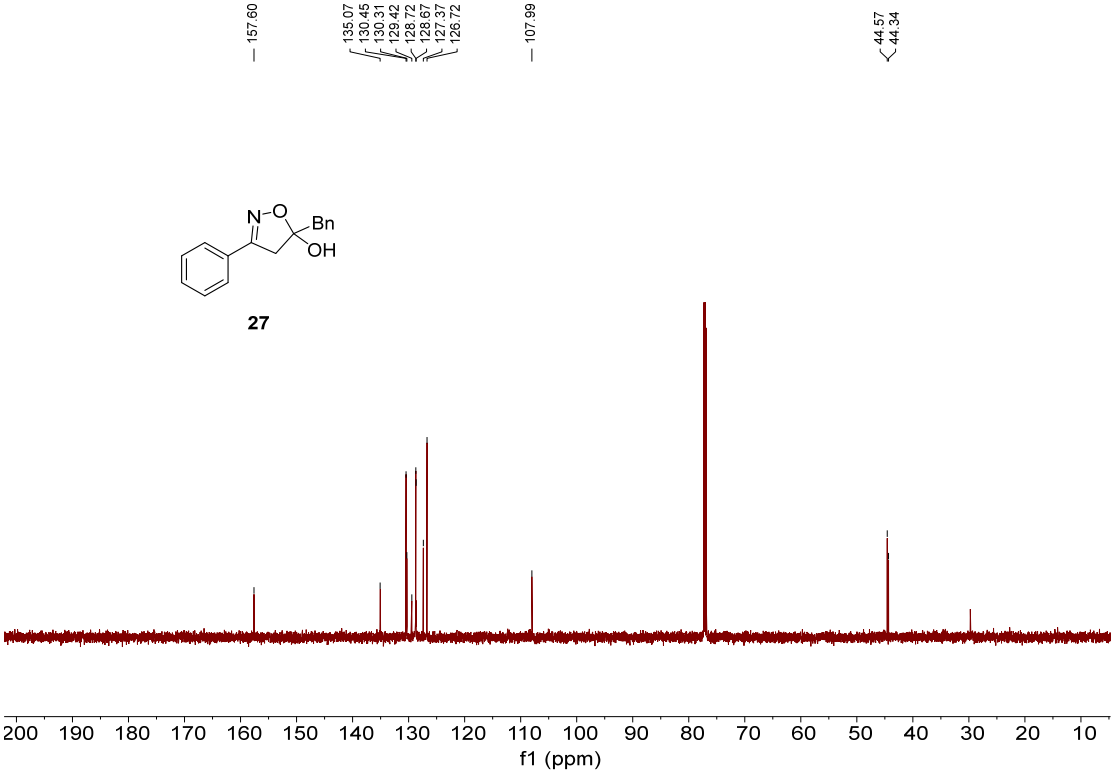

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

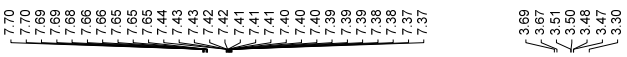

7.54
7.54
7.53
7.53
7.52
7.31
7.31
7.30
7.30
7.30
7.29
7.29
7.29
7.29
7.28
7.27
7.27
7.26
7.25
7.25
7.22
7.22
7.21
7.21
7.20
7.20
7.19
7.17
3.25
3.23
3.23
3.22
3.20
3.19
3.18
3.16

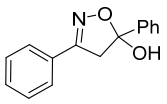

N
O
Bn
OH

27

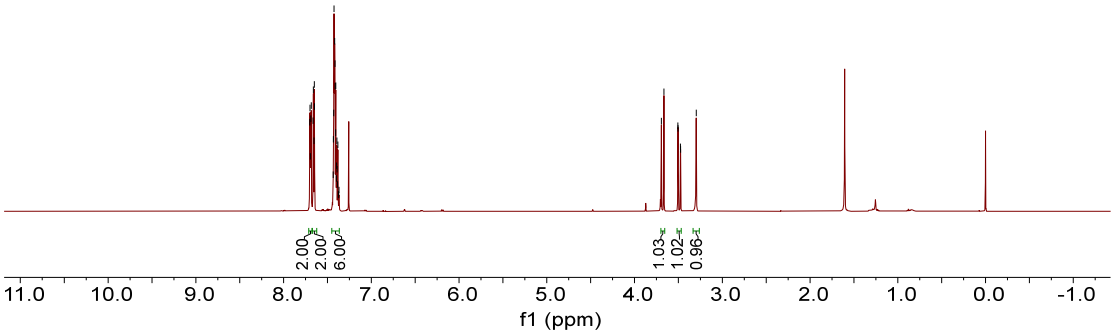

2.00
4.03
2.28
2.02
2.15
2.16
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


157.60
135.07
130.45
130.31
129.42
128.72
128.67
127.37
126.72
107.99
44.57
44.34
N
O
Bn
OH
27
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

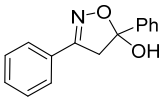

7.70
7.70
7.69
7.69
7.68
7.66
7.66
7.65
7.65
7.44
7.43
7.43
7.42
7.42
7.41
7.41
7.41
7.40
7.40
7.40
7.39
7.39
7.39
7.38
7.38
7.37
7.37
3.69
3.67
3.51
3.50
3.48
3.47
3.30


N
O
Ph
OH

28


2.00
2.00
6.00
1.03
1.02
0.96
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

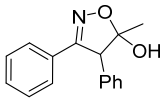

157.46
140.70
130.46
129.25
128.97
128.82
128.61
126.81
125.64
107.69
49.02


N
O
Ph
OH

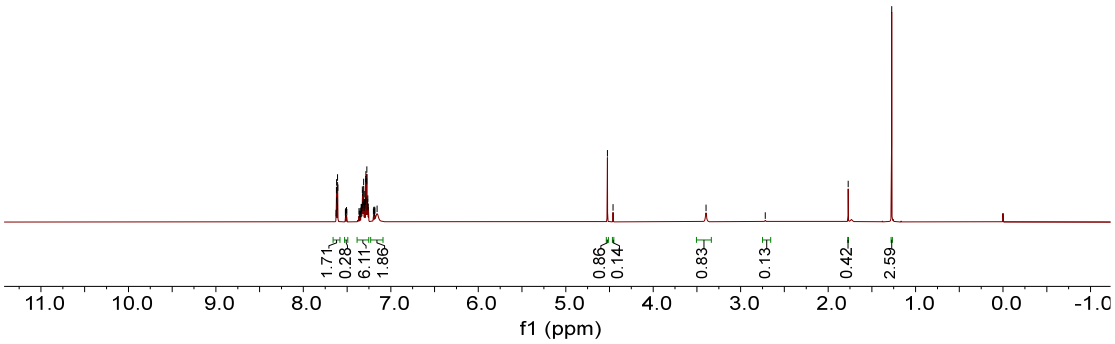
28

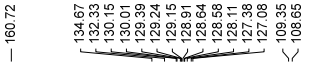
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

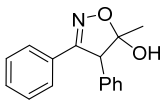

7.63
7.62
7.62
7.62
7.61
7.61
7.61
7.52
7.52
7.51
7.50
7.36
7.35
7.34
7.34
7.34
7.33
7.33
7.32
7.32
7.32
7.31
7.31
7.31
7.30
7.30
7.30
7.29
7.29
7.29
7.28
7.28
7.27
7.27
7.27
7.26
7.26
7.20
7.20
7.19
7.18
7.18
7.16
4.52
4.46
3.40
2.72
1.77
1.27


N
O
OH
Ph

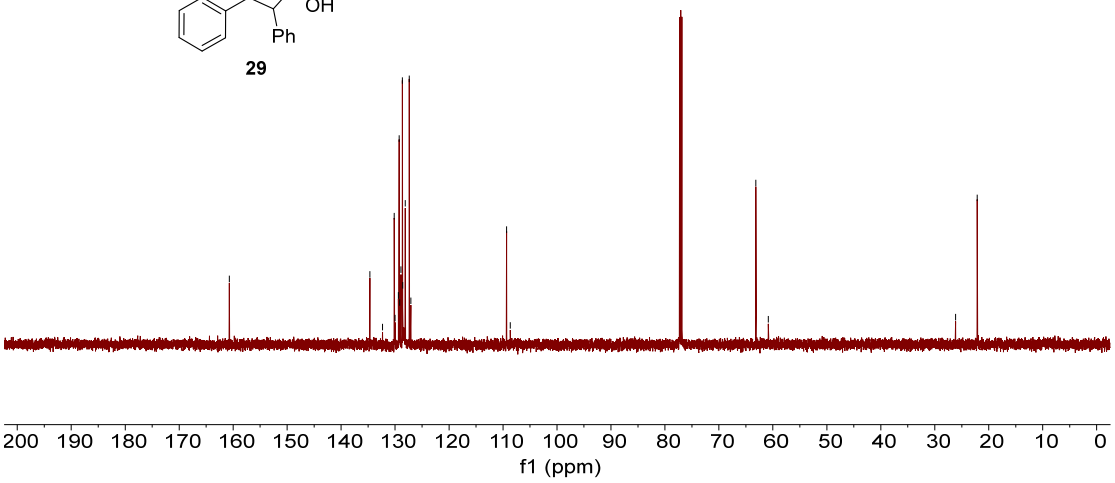
29


1.71
0.28
6.11
1.86
0.86
0.14
0.83
0.13
0.42
2.59
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

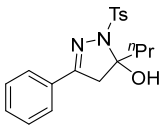

160.72
134.67
132.33
130.15
130.01
129.39
129.24
129.15
128.91
128.64
128.58
128.11
127.38
127.08
109.35
108.65
63.14
60.83
26.15
22.14


N
O
OH
Ph

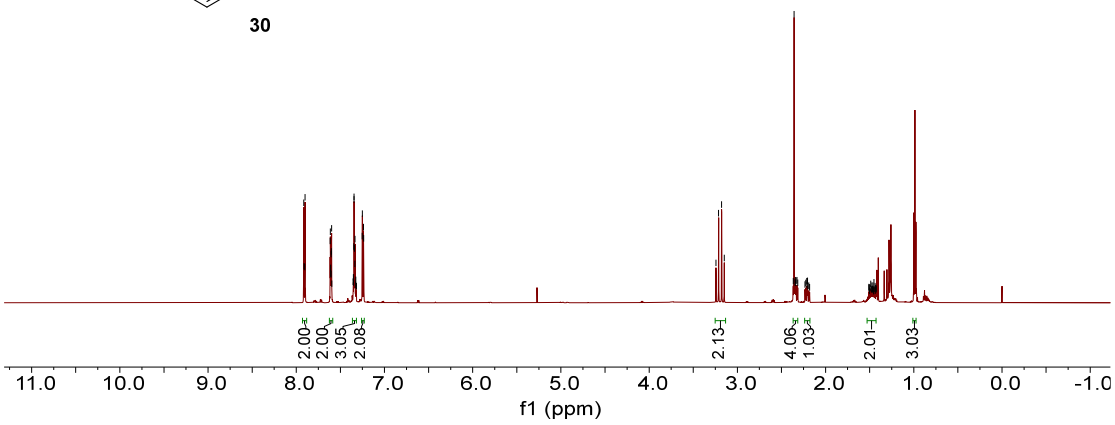
29


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

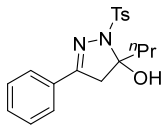

7.91
7.91
7.90
7.90
7.62
7.61
7.61
7.61
7.60
7.60
7.60
7.60
7.36
7.35
7.35
7.35
7.34
7.34
7.34
7.34
7.33
7.33
7.32
7.32
7.25
7.25
7.24
7.24
3.24
3.21
3.18
3.15
2.37
2.36
2.35
2.34
2.34
2.33
2.32
2.32
2.23
2.22
2.21
2.21
2.20
2.20
2.19
2.18
1.51
1.50
1.49
1.48
1.46
1.45


Ts
N
N
nPr
OH

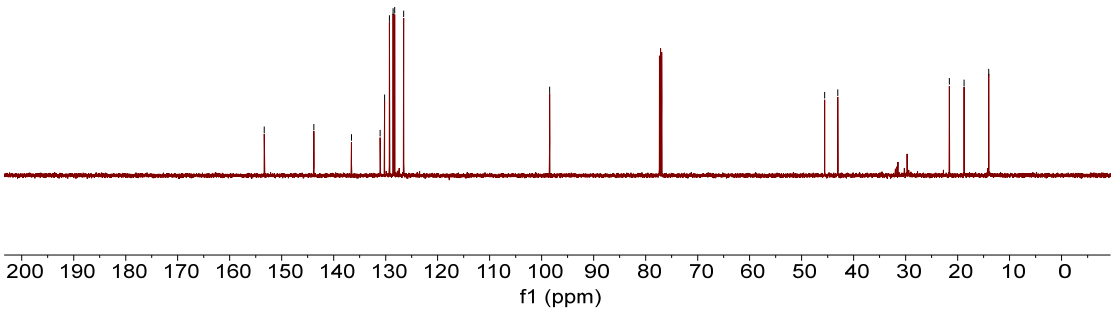
30

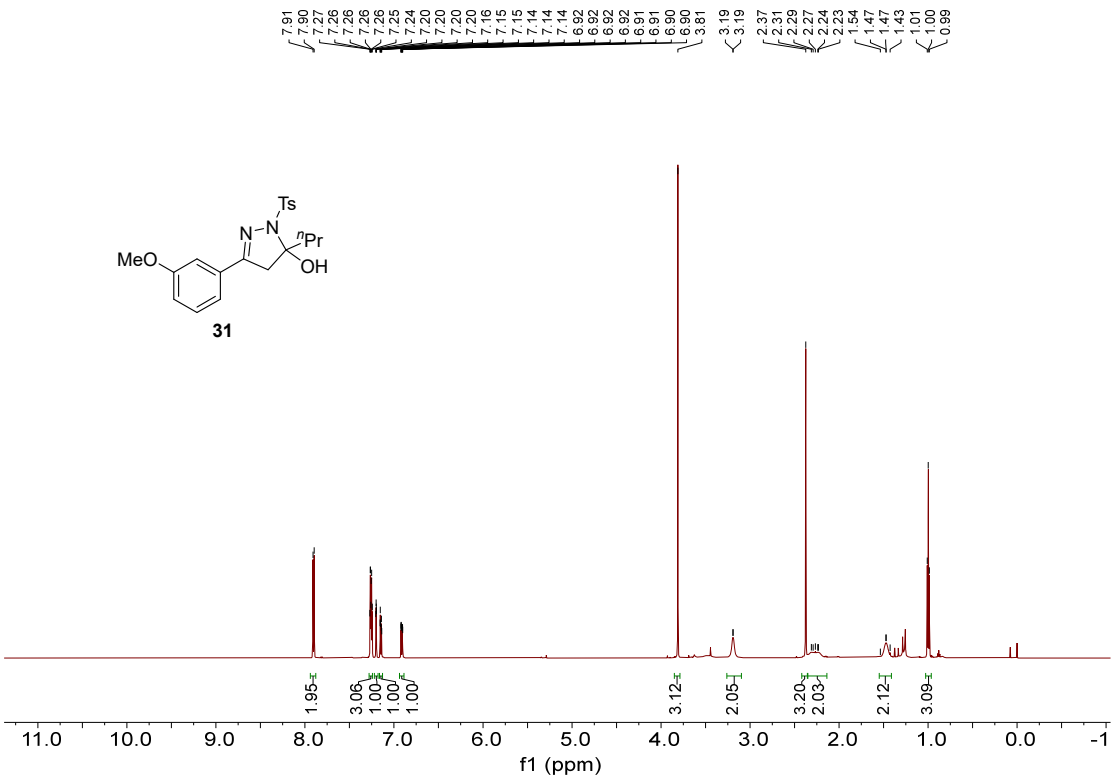

2.00
2.00
3.05
2.08
2.13
4.06
1.03
2.01
3.03
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

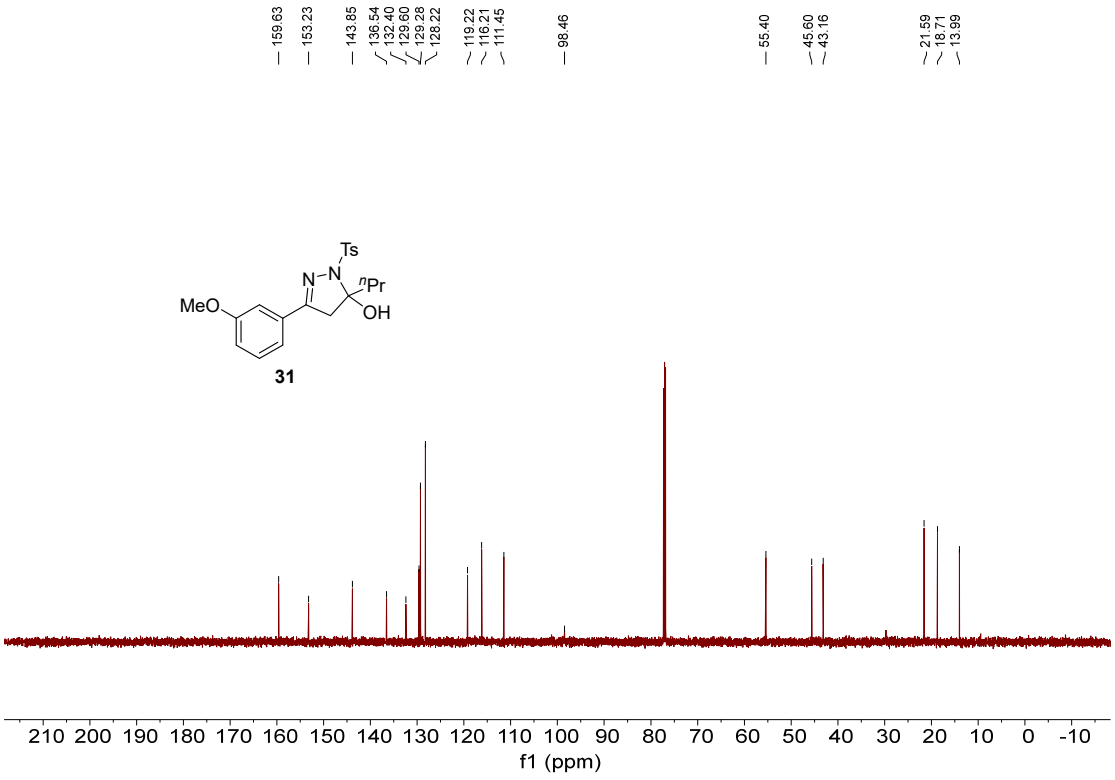

153.36
143.82
136.57
131.08
130.21
129.29
128.57
128.27
126.53
98.47
45.56
43.06
21.59
18.74
14.02

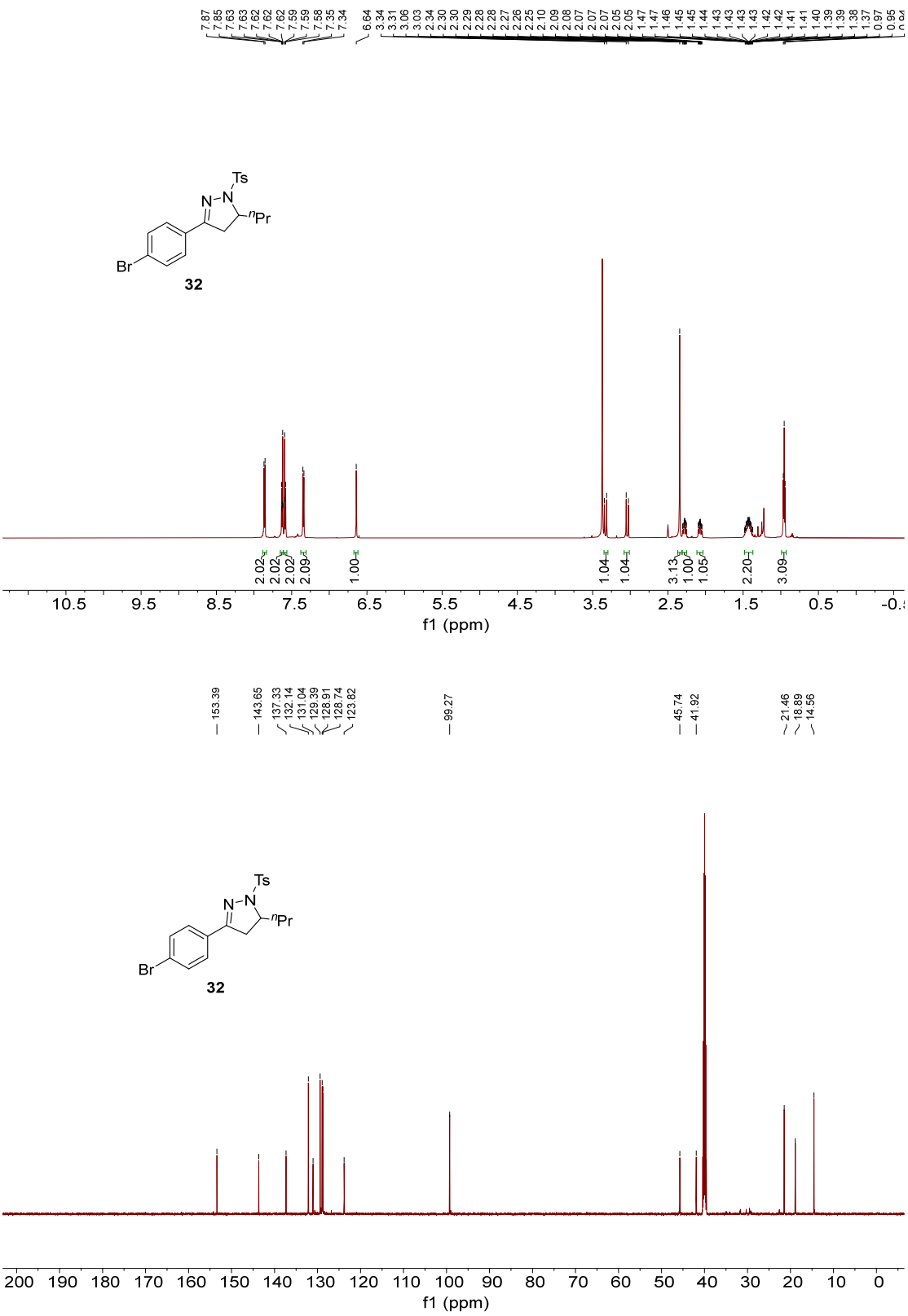

Ts
N
N
nPr
OH

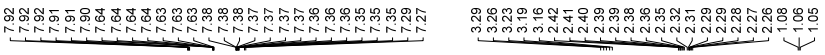
30


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.91
7.90
7.27
7.26
7.26
7.26
7.26
7.25
7.24
7.20
7.20
7.20
7.20
7.16
7.15
7.15
7.14
7.14
7.14
6.92
6.92
6.92
6.92
6.91
6.91
6.90
6.90
3.81
3.19
3.19
2.37
2.31
2.29
2.27
2.24
2.23
1.54
1.47
1.47
1.43
1.01
1.00
0.99
Ts
N
N
nPr
OH
MeO
31
1.95
3.06
1.00
1.00
1.00
3.12
2.05
3.20
2.03
2.12
3.09
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1
f1 (ppm)


159.63
153.23
143.85
136.54
132.40
129.60
129.28
128.22
119.22
116.21
111.45
98.46
55.40
45.60
43.16
21.59
18.71
13.99
Ts
N
N
nPr
OH
MeO
31
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.87 7.85 7.63 7.63 7.62 7.62 7.62 7.59 7.59 7.58 7.35 7.34
6.64
3.34 3.31 3.06 3.03 2.34 2.30 2.30 2.29 2.28 2.28 2.27 2.26 2.25 2.10 2.09 2.08 2.07 2.07 2.05 2.05 1.47 1.47 1.46 1.45 1.45 1.44 1.43 1.43 1.43 1.43 1.42 1.42 1.41 1.41 1.40 1.39 1.39 1.38 1.37 0.97 0.95
Ts
N
N
nPr
Br
32
2.02 2.02 2.02 2.09 1.00 1.04 1.04 3.13 1.00 1.05 2.20 3.09
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)
153.39 143.65 137.33 132.14 131.04 129.39 128.91 128.74 123.82
99.27
45.74 41.92
21.46 18.89 14.56
Ts
N
N
nPr
Br
32
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.92
7.92
7.91
7.91
7.90
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7.64
7.64
7.64
7.63
7.63
7.63
7.38
7.38
7.38
7.37
7.37
7.37
7.37
7.36
7.36
7.36
7.35
7.35
7.35
7.29
7.27
3.29
3.26
3.23
3.19
3.16
2.42
2.41
2.40
2.39
2.39
2.38
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2.32
2.31
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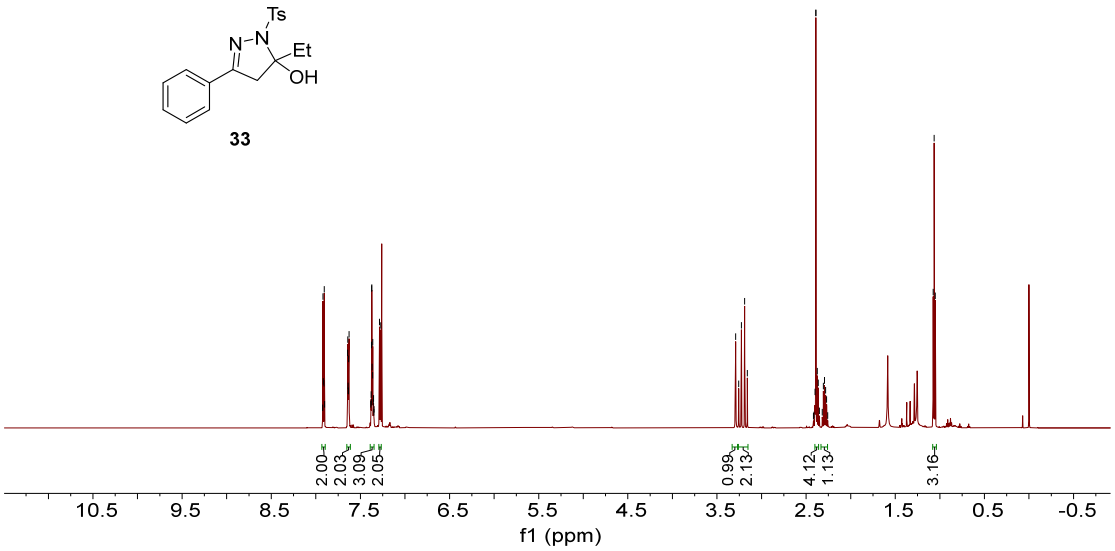
2.29
2.28
2.27
2.26
1.08
1.06
1.05


Ts
N
N
Et
OH
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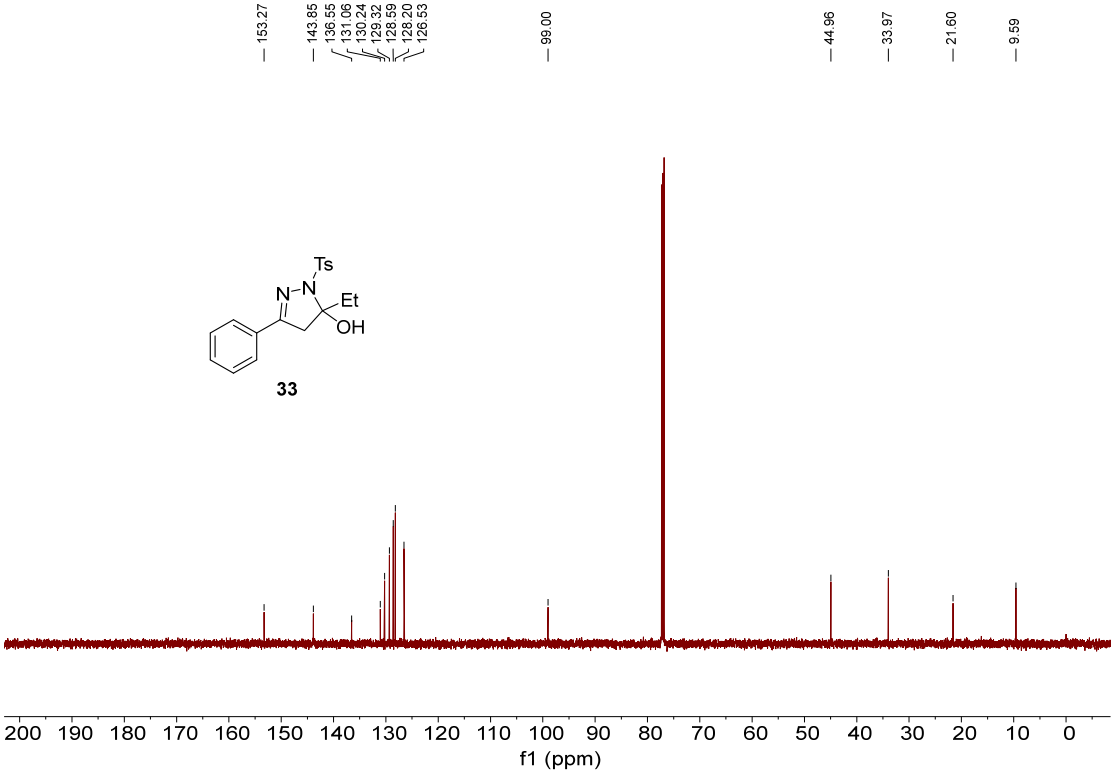
2.03
3.09
2.05
0.99
2.13
4.12
1.13
3.16
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)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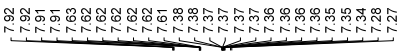
143.85
136.55
131.06
130.24
129.32
128.59
128.20
126.53
99.00
44.96
33.97
21.60
9.59
Ts
N
N
Et
OH
33
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.62
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7.61
7.38
7.38
7.37
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7.36
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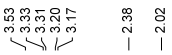
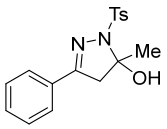
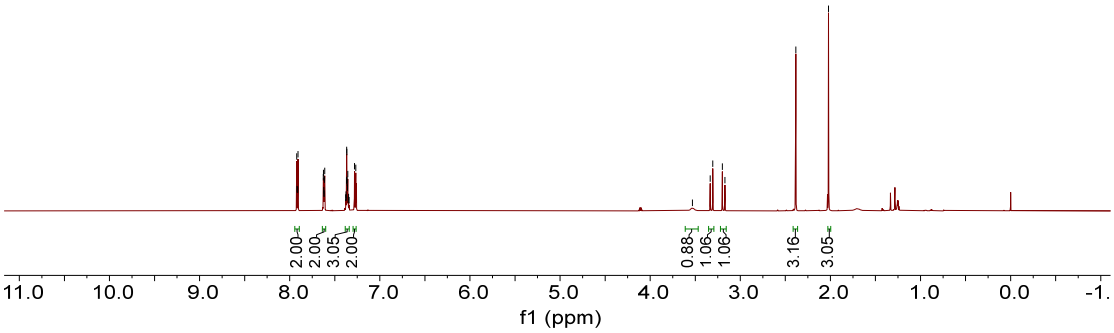
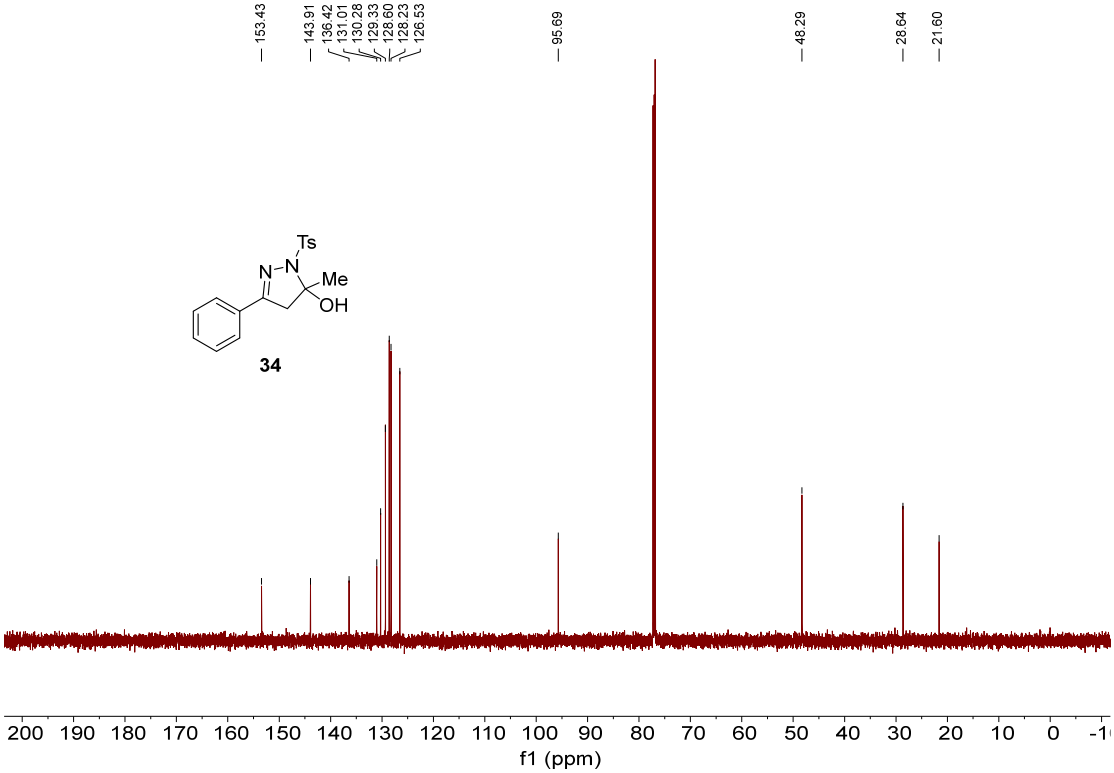
7.35
7.35
7.34
7.28
7.27


3.53
3.33
3.31
3.20
3.17
2.38
2.02


Ts
N
N
Me
OH

34


2.00
2.00
3.05
2.00
0.88
1.06
1.06
3.16
3.05
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)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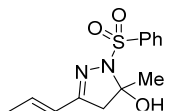
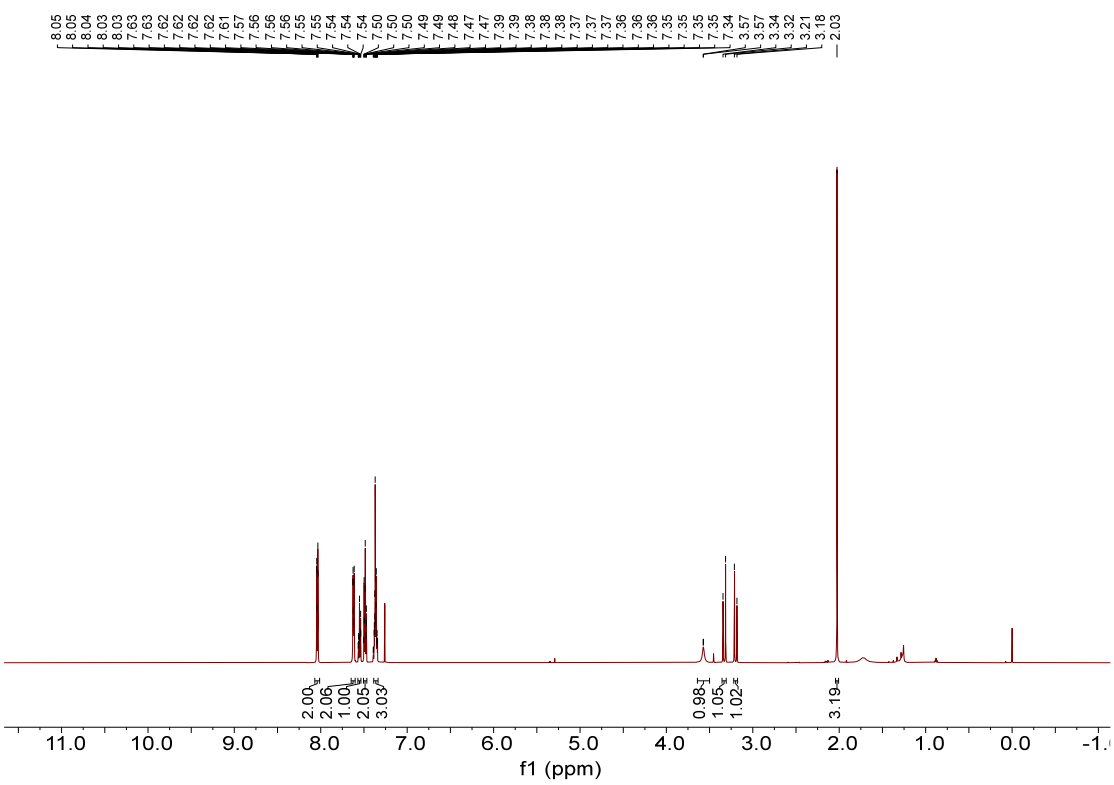
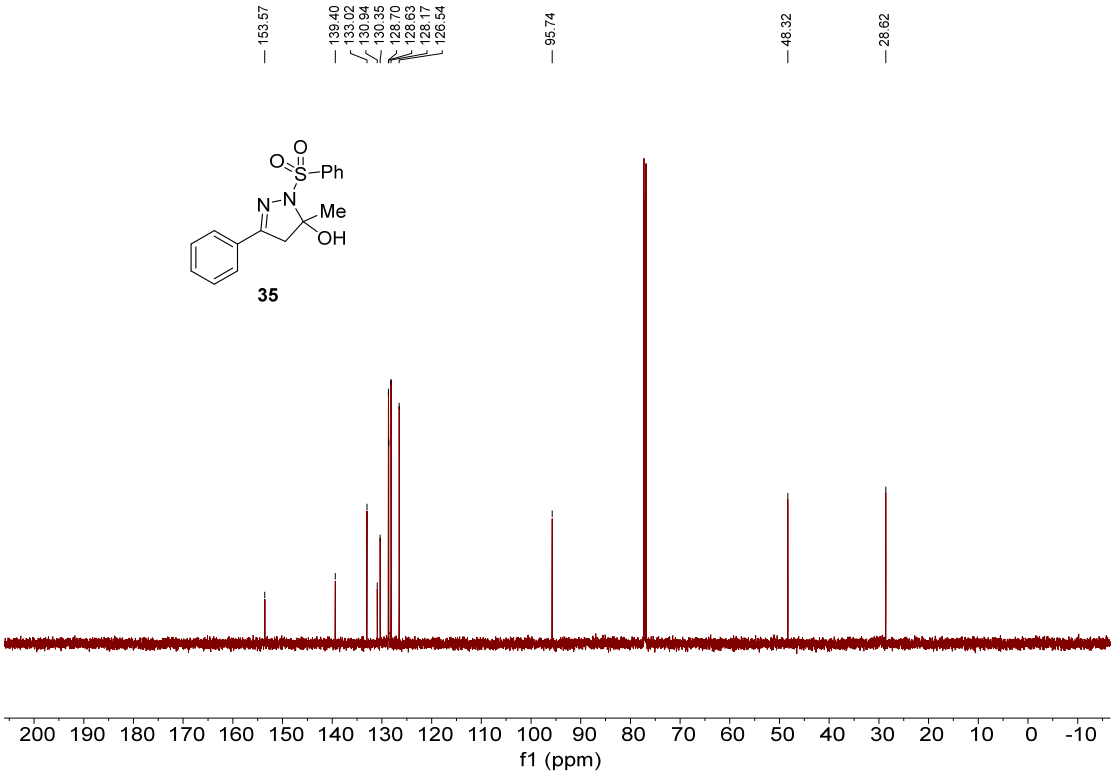
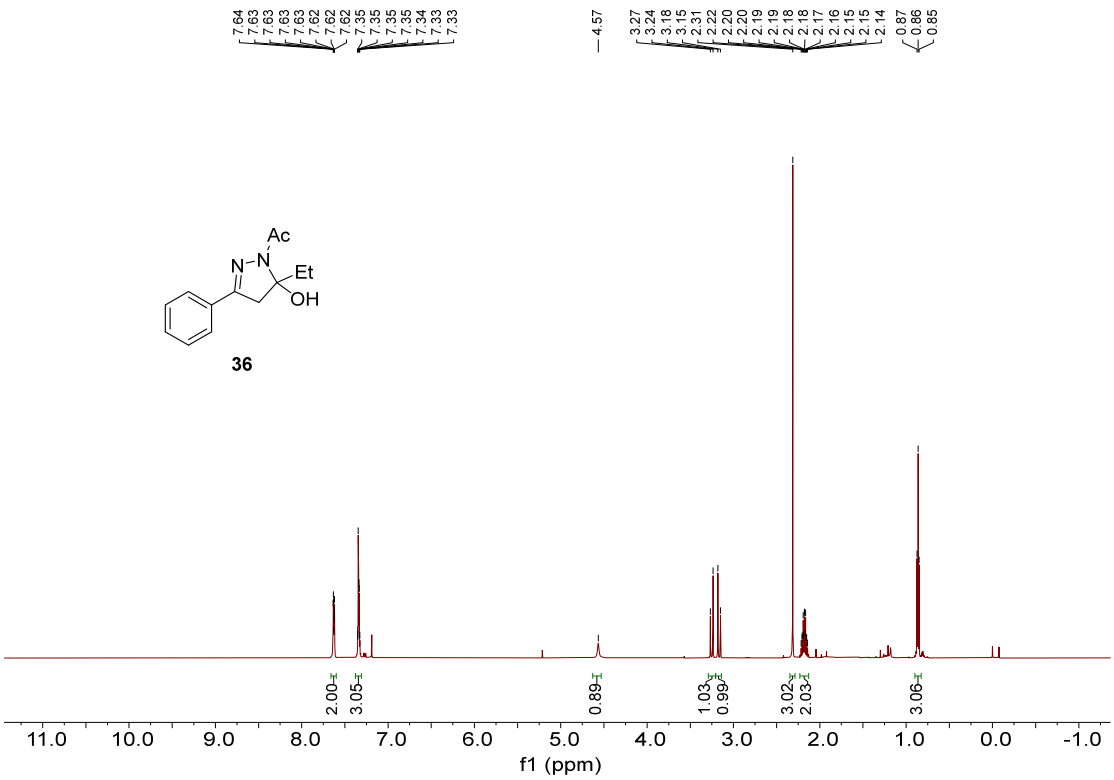
143.91
136.42
131.01
130.28
129.33
128.60
128.23
126.53
95.69
48.29
28.64
21.60
Ts
N
N
Me
OH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

34


O
O
S
Ph
N
N
Me
OH


8.05 8.05 8.04 8.03 8.03 7.63 7.63 7.62 7.62 7.62 7.62 7.61 7.57 7.56 7.56 7.56 7.55 7.55 7.54 7.54 7.54 7.50 7.50 7.50 7.49 7.49 7.48 7.47 7.47 7.39 7.39 7.38 7.38 7.38 7.37 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.35 7.34 3.57 3.57 3.34 3.32 3.21 3.18 2.03
2.00 2.06 1.00 2.05 3.03 0.98 1.05 1.02 3.19
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
f1 (ppm)


153.57 139.40 133.02 130.94 130.35 128.70 128.63 128.17 126.54 95.74 48.32 28.62
O
O
S–Ph
N–N
Me
OH
35
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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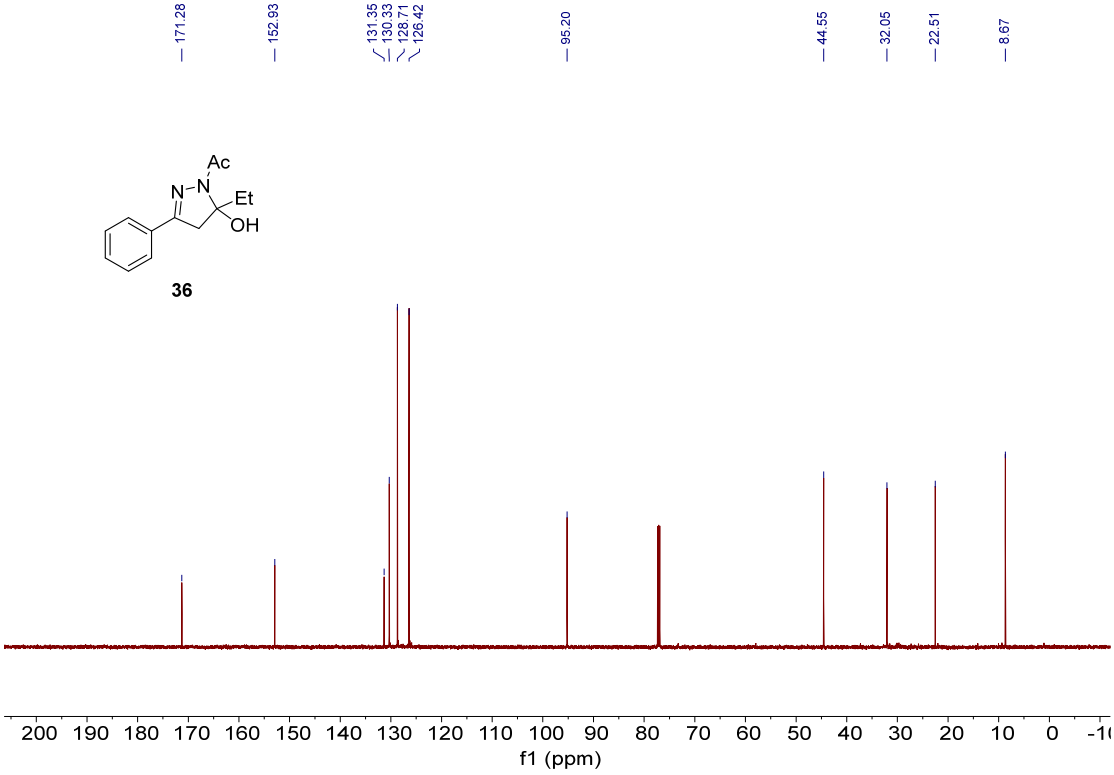
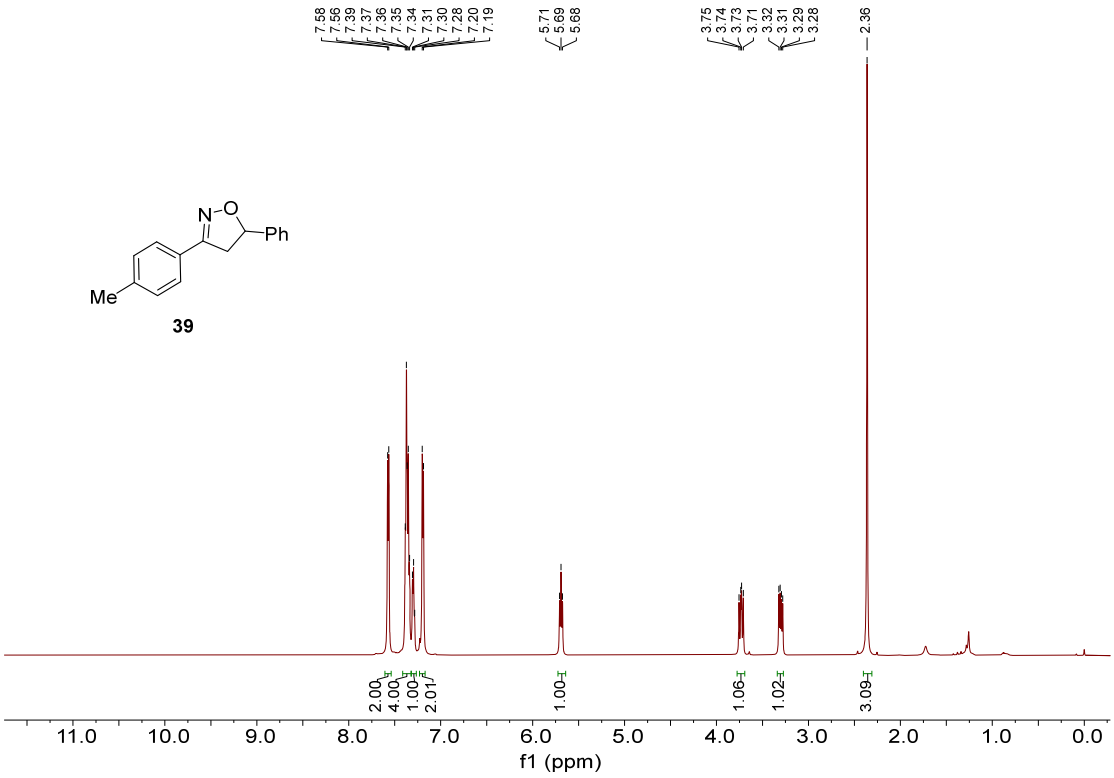
7.63
7.63
7.63
7.63
7.62
7.62
7.62
7.35
7.35
7.35
7.35
7.34
7.33
7.33
4.57
3.27
3.24
3.18
3.15
2.31
2.22
2.20
2.20
2.19
2.19
2.18
2.18
2.17
2.16
2.15
2.15
2.14
0.87
0.86
0.85
Ac
N
N
Et
OH
36
2.00
3.05
0.89
1.03
0.99
3.02
2.03
3.06
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


171.28
152.93
131.35
130.33
128.71
126.42
96.20
44.55
32.05
22.51
8.67
Ac
N
N
Et
OH
36
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.35
7.34
7.31
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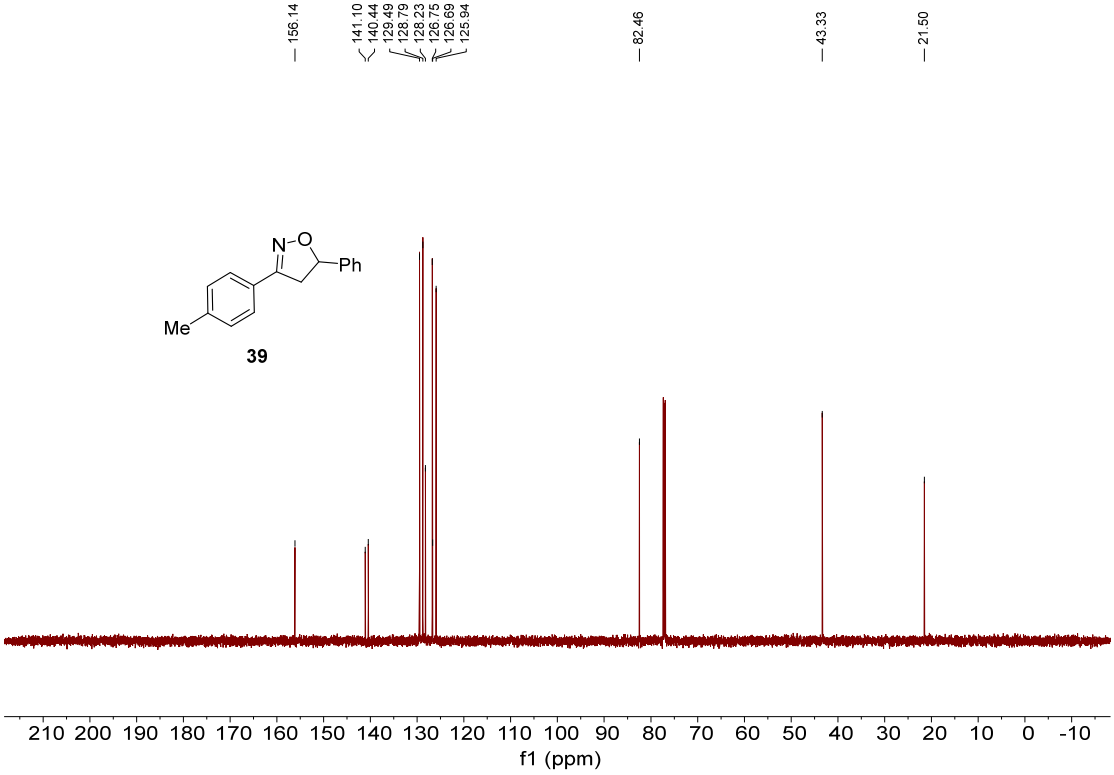
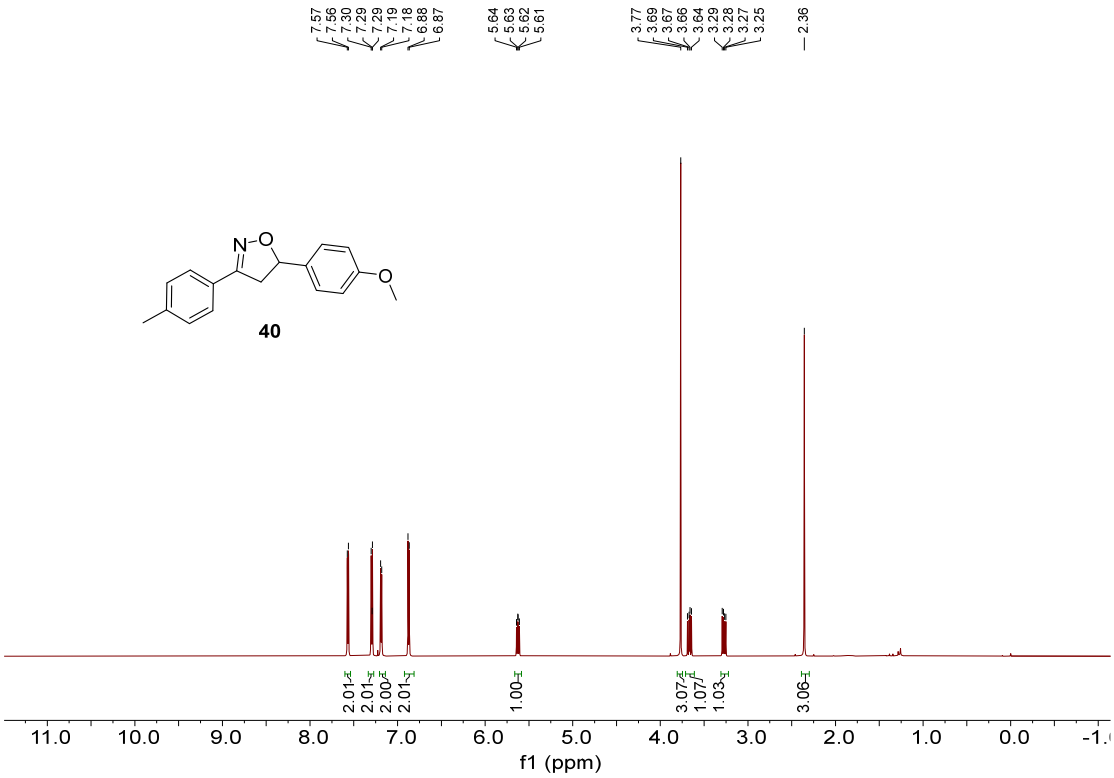
7.28
7.20
7.19
5.71
5.69
5.68
3.75
3.74
3.73
3.71
3.32
3.31
3.29
3.28
2.36
N
O
Ph
Me
39
2.00
4.00
1.00
2.01
1.00
1.06
1.02
3.09
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


156.14
141.10
140.44
129.49
128.79
128.23
126.75
126.69
125.94
82.46
43.33
21.50
N
O
Ph
Me
39
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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6.87
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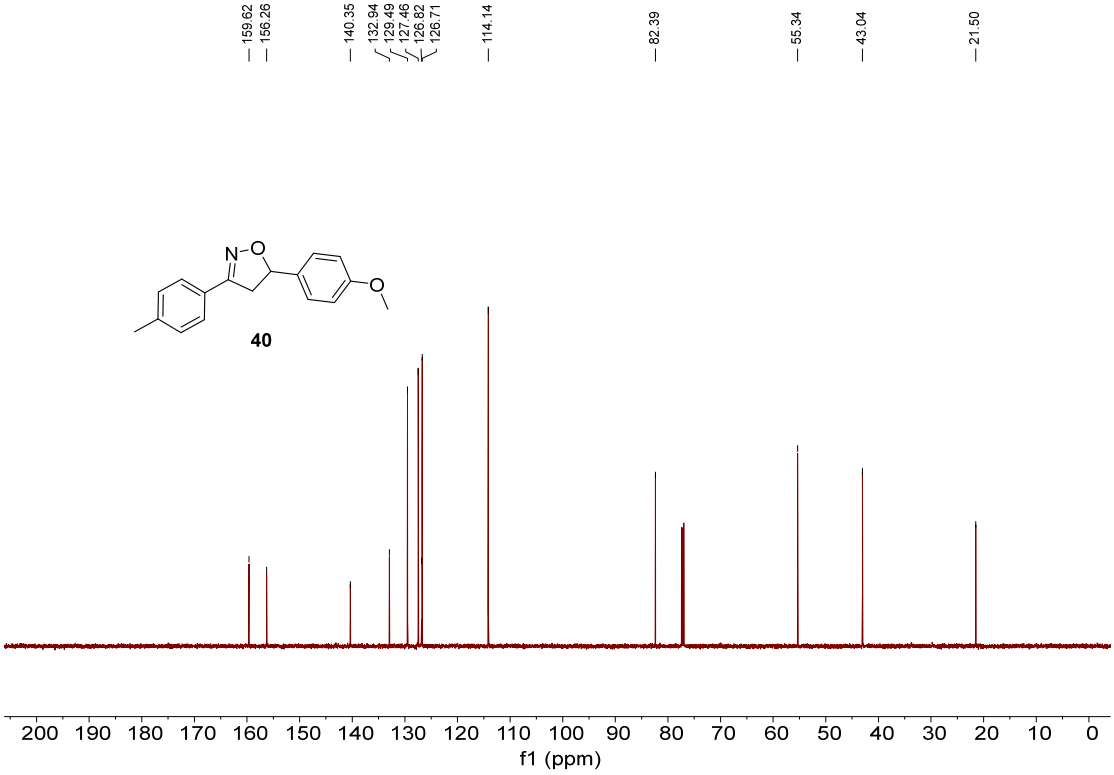
5.62
5.61
3.77
3.69
3.67
3.66
3.64
3.29
3.28
3.27
3.25
2.36
N
O
O
40
2.01
2.01
2.00
2.01
1.00
3.07
1.07
1.03
3.06
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)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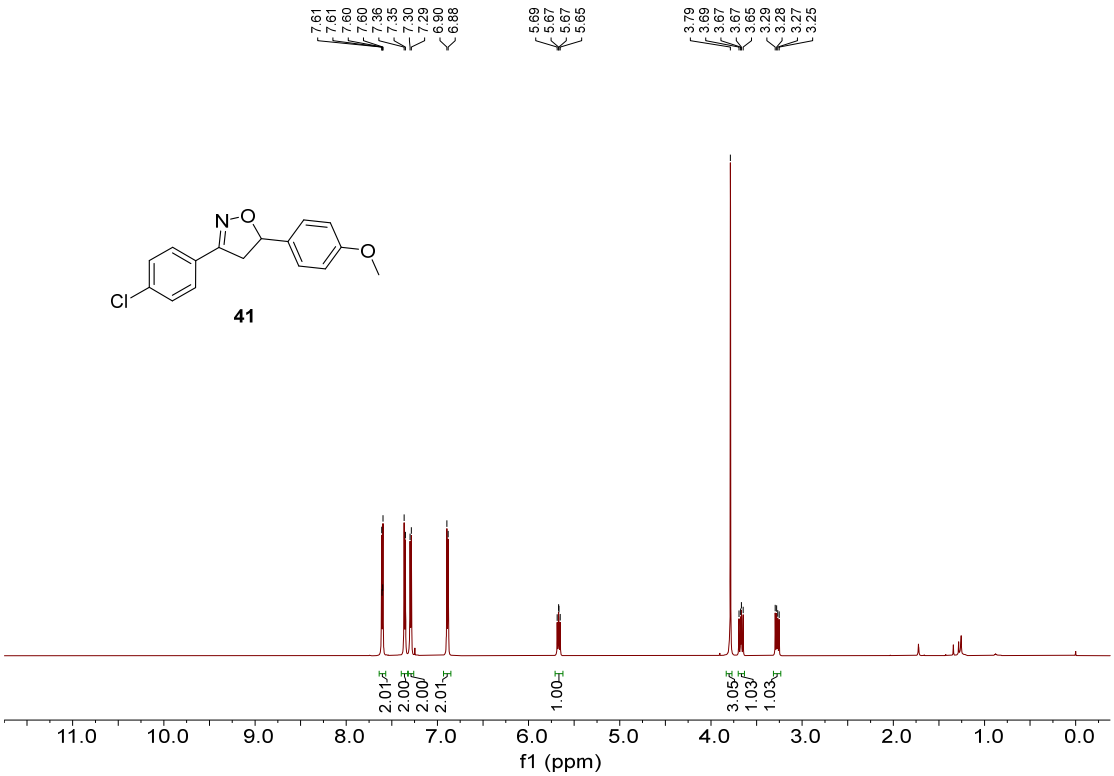
140.35
132.94
129.49
127.46
126.82
126.71
114.14
82.39
55.34
43.04
21.50
N
O
O
40
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.61
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7.60
7.36
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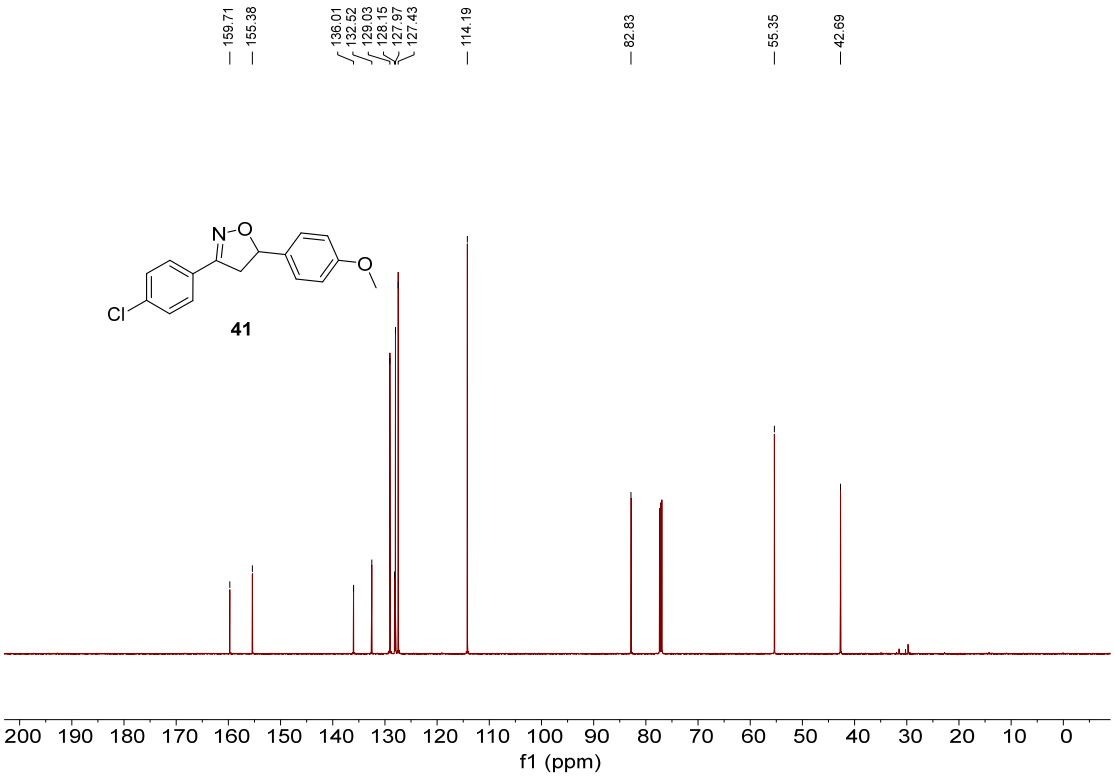
7.30
7.29
6.90
6.88
5.69
5.67
5.67
5.65
3.79
3.69
3.67
3.67
3.65
3.29
3.28
3.27
3.25
N
O
O
Cl
41
2.01
2.00
2.00
2.01
1.00
3.05
1.03
1.03
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)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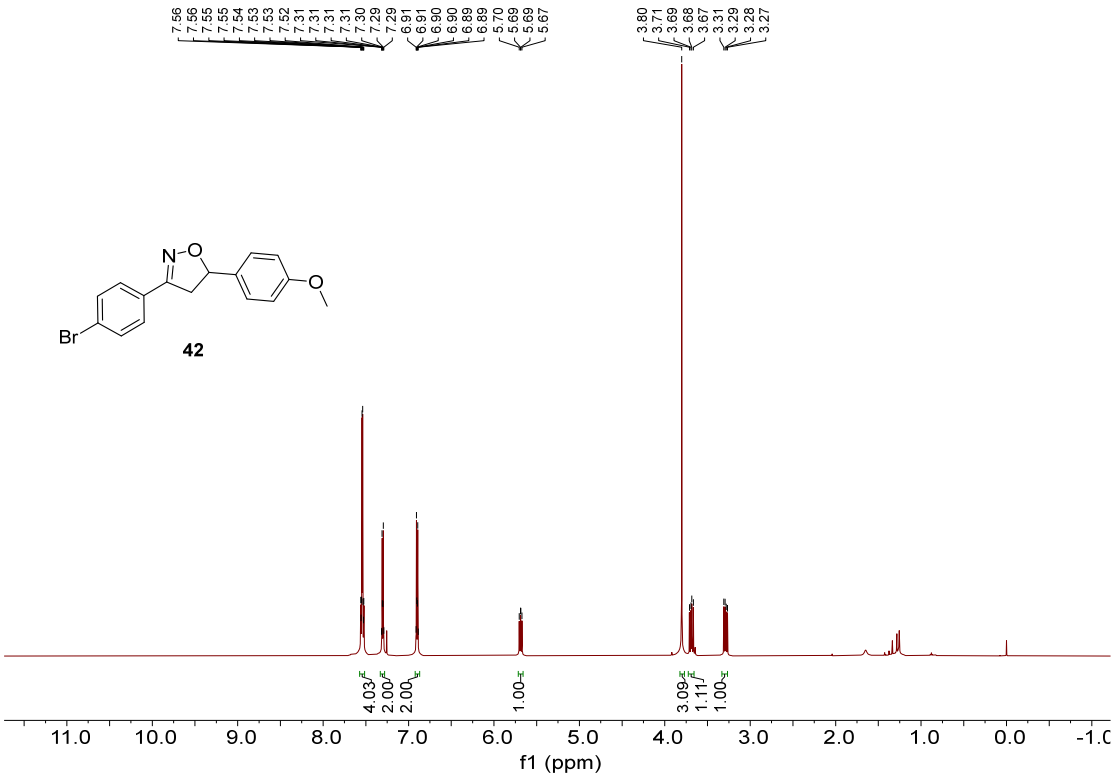
136.01
132.52
129.03
128.15
127.97
127.43
114.19
82.83
55.35
42.69
N
O
O
Cl
41
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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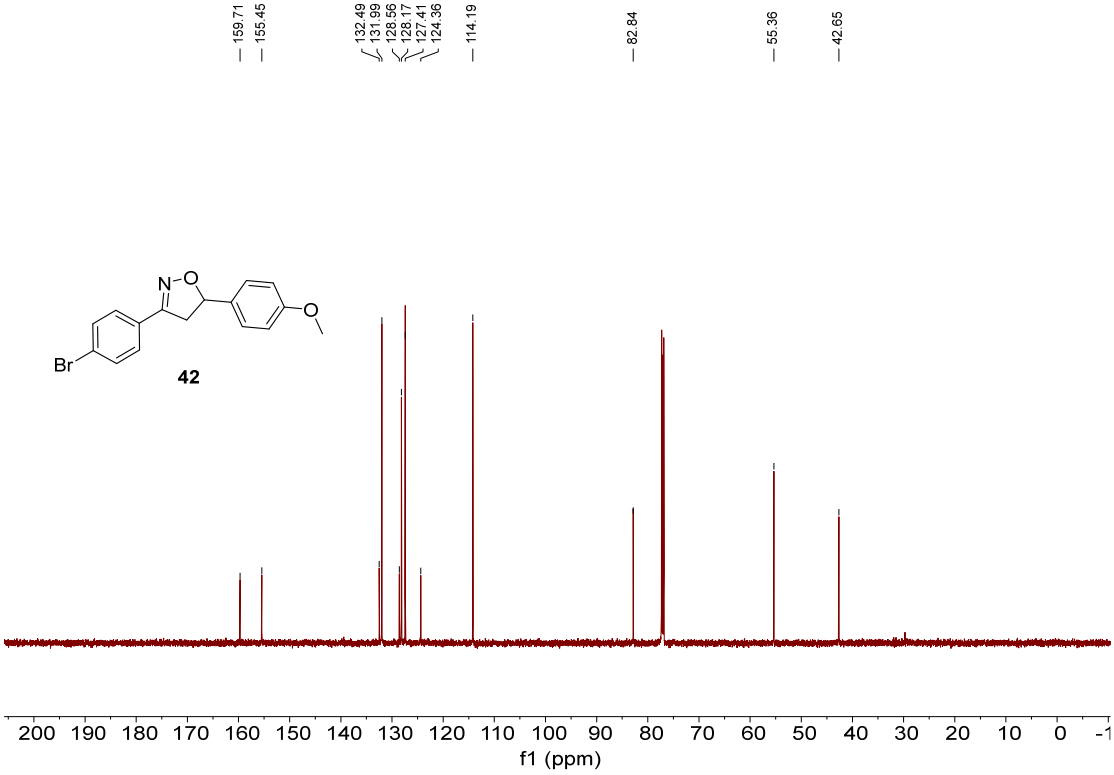
7.31
7.30
7.29
7.29
6.91
6.91
6.90
6.90
6.89
6.89
5.70
5.69
5.69
5.67
3.80
3.71
3.69
3.68
3.67
3.31
3.29
3.28
3.27
N
O
O
Br
42
4.03
2.00
2.00
1.00
3.09
1.11
1.00
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)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155.45
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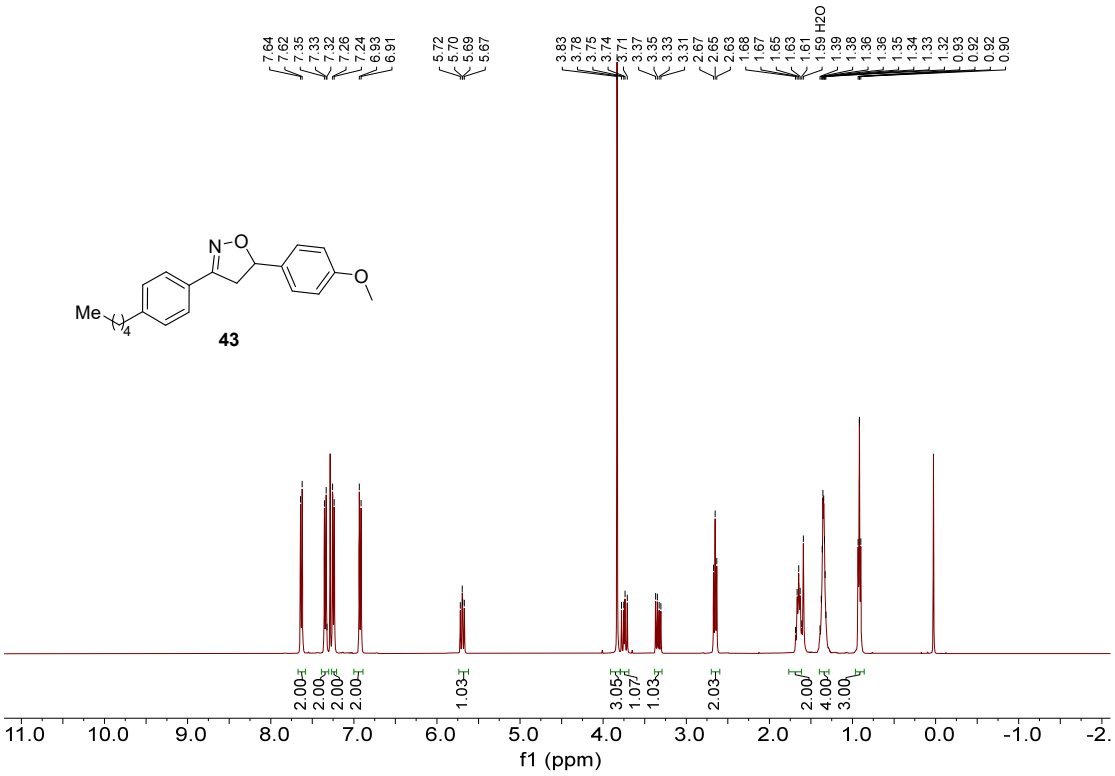
131.99
128.56
128.17
127.41
124.36
114.19
82.84
55.36
42.65
N
O
O
Br
42
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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5.67
3.83
3.78
3.75
3.74
3.71
3.37
3.35
3.33
3.31
2.67
2.65
2.63
1.68
1.67
1.65
1.63
1.61
1.59 H2O
1.39
1.38
1.36
1.36
1.35
1.34
1.33
1.32
0.93
0.92
0.92
0.90
N
O
O
Me
4
43
2.00
2.00
2.00
2.00
1.03
3.05
1.07
1.03
2.03
2.00
4.00
3.00
11.0
10.0
9.0
8.0
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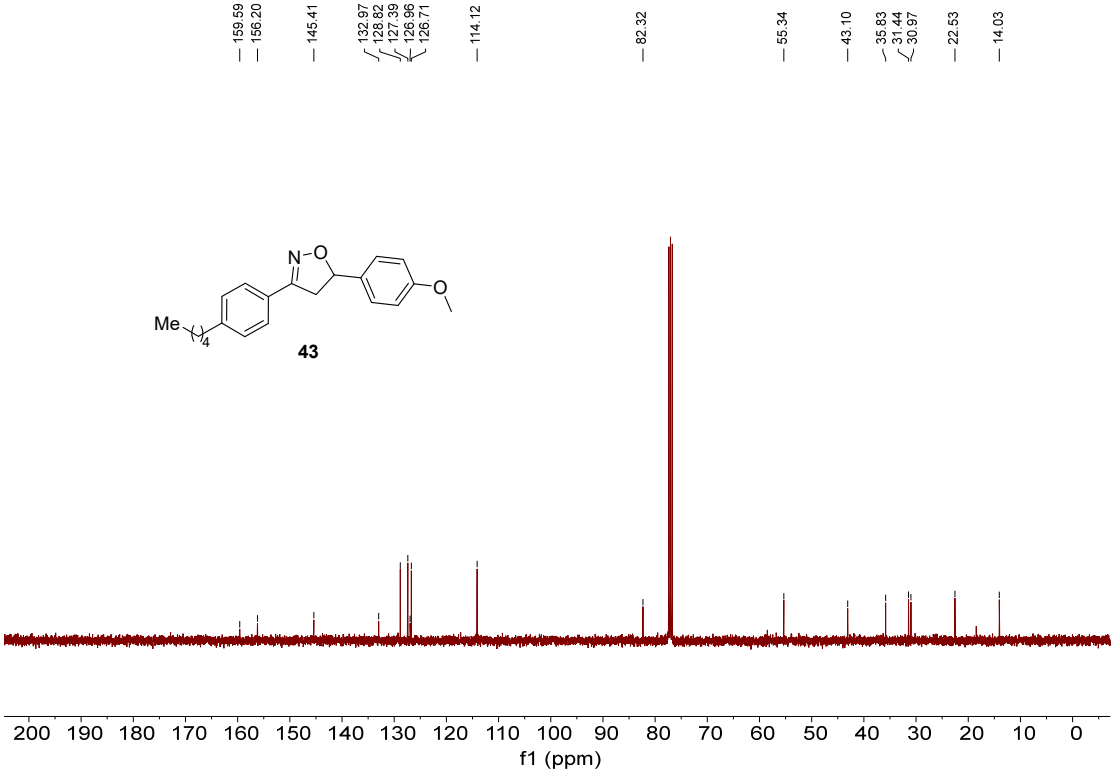
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2.
f1 (ppm)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126.96
126.71
114.12
82.32
55.34
43.10
35.83
31.44
30.97
22.53
14.03
N
O
O
Me
4
43
200
190
180
170
160
150
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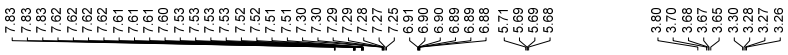
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.60
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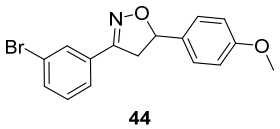
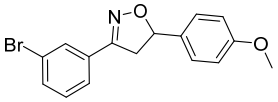
7.53
7.53
7.52
7.52
7.51
7.51
7.30
7.30
7.29
7.29
7.28
7.27
7.25
6.91
6.90
6.90
6.89
6.89
6.88
5.71
5.69
5.69
5.68
3.80
3.70
3.68
3.67
3.65
3.30
3.28
3.27
3.26


N
O
Br
O

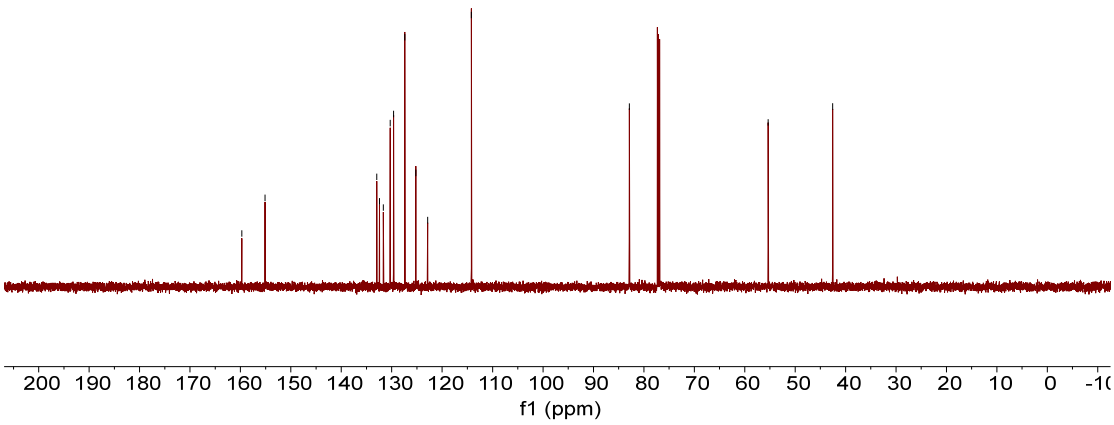
44

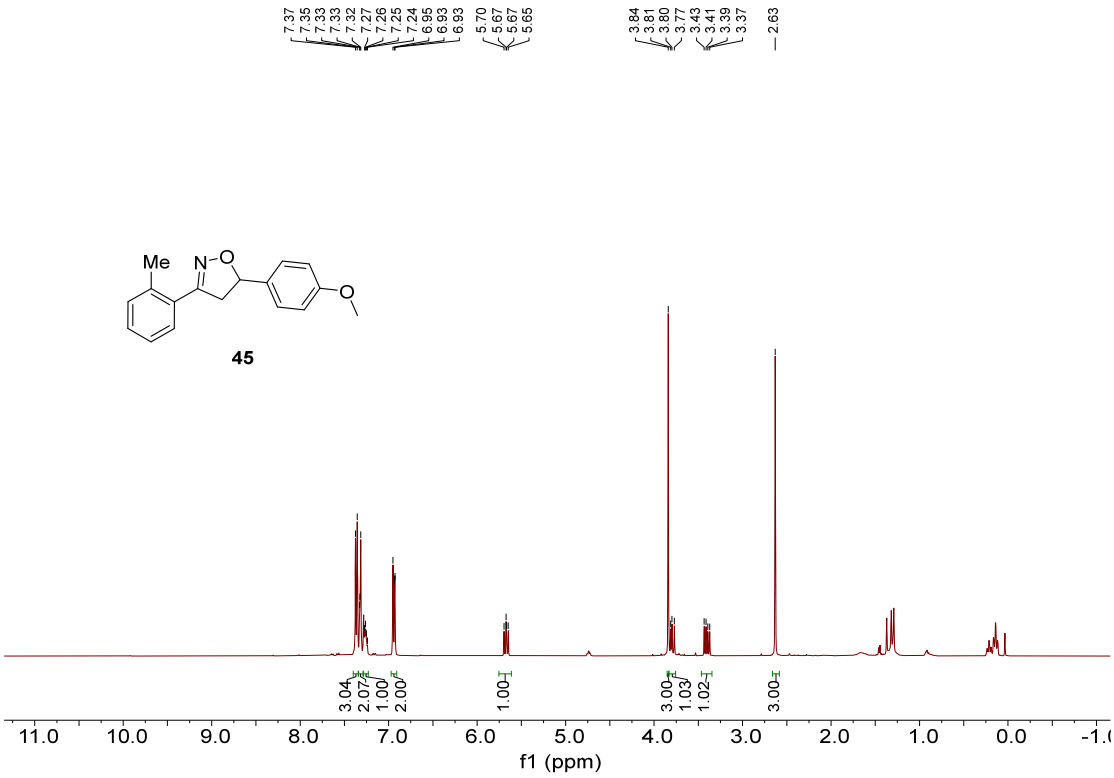
1.00
1.03
1.00
2.05
1.32
2.09
1.03
3.16
1.11
1.09
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


159.73
155.13
132.97
132.44
131.66
130.30
129.64
127.40
125.24
122.87
114.21
82.89
55.37
42.57


N
O
Br
O

44


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.35
7.33
7.33
7.32
7.27
7.26
7.25
7.24
6.95
6.93
6.93
5.70
5.67
5.67
5.65
3.84
3.81
3.80
3.77
3.43
3.41
3.39
3.37
2.63
Me
N
O
O
45
3.04
2.07
1.00
2.00
1.00
3.00
1.03
1.02
3.00
11.0
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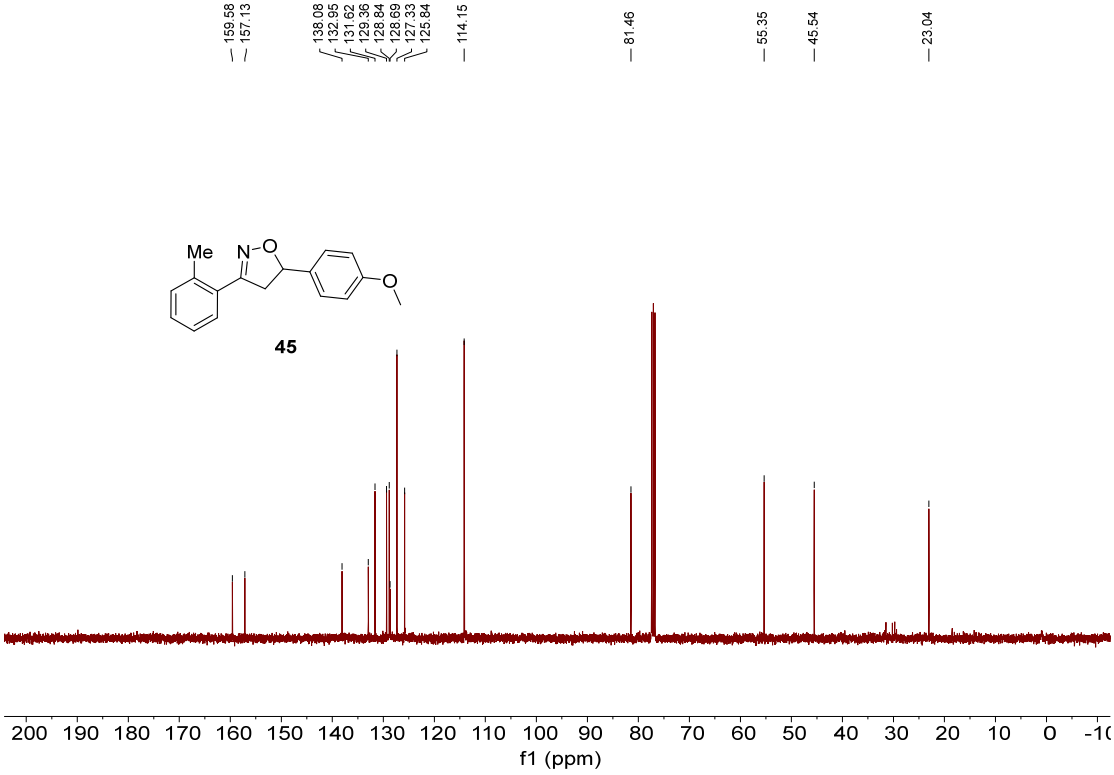
f1 (ppm)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128.69
127.33
125.84
114.15
81.46
55.35
45.54
23.04
Me
N
O
O
45
200
190
180
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90
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50
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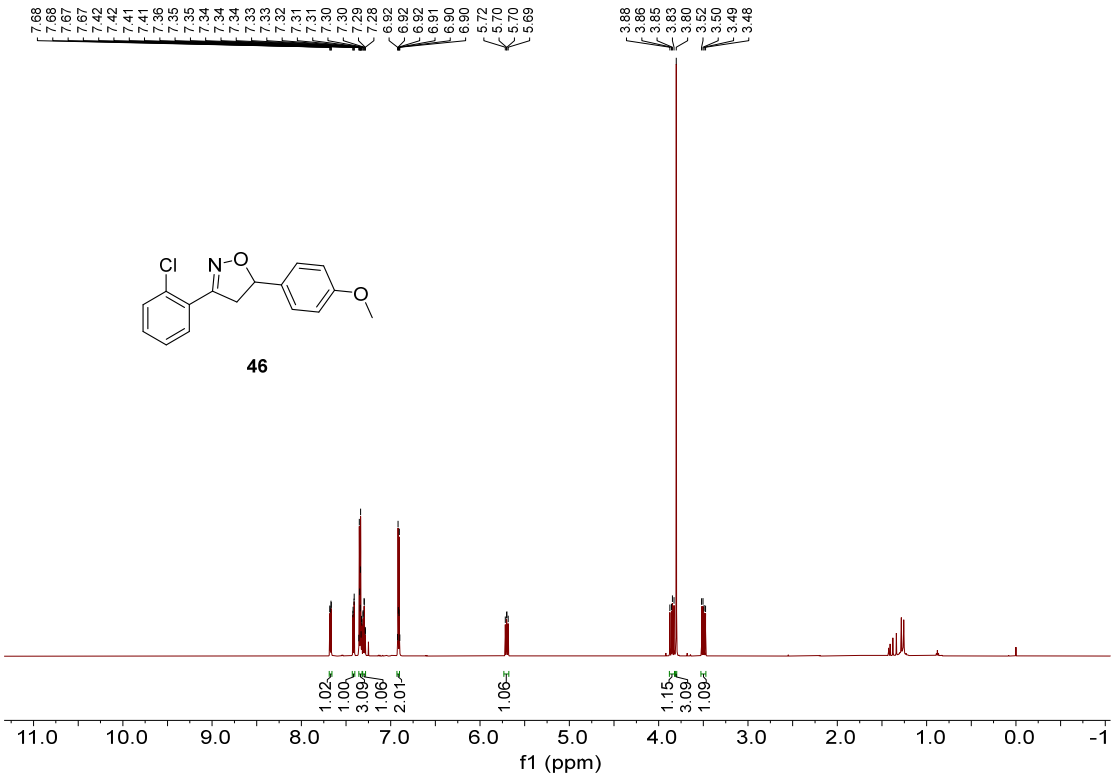
f1 (ppm)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7.34
7.33
7.33
7.32
7.31
7.31
7.30
7.30
7.29
7.28
6.92
6.92
6.92
6.91
6.90
6.90
5.72
5.70
5.70
5.69
3.88
3.86
3.85
3.83
3.80
3.52
3.50
3.49
3.48
Cl
N
O
O
46
1.02
1.00
3.09
1.06
2.01
1.06
1.15
3.09
1.09
11.0
10.0
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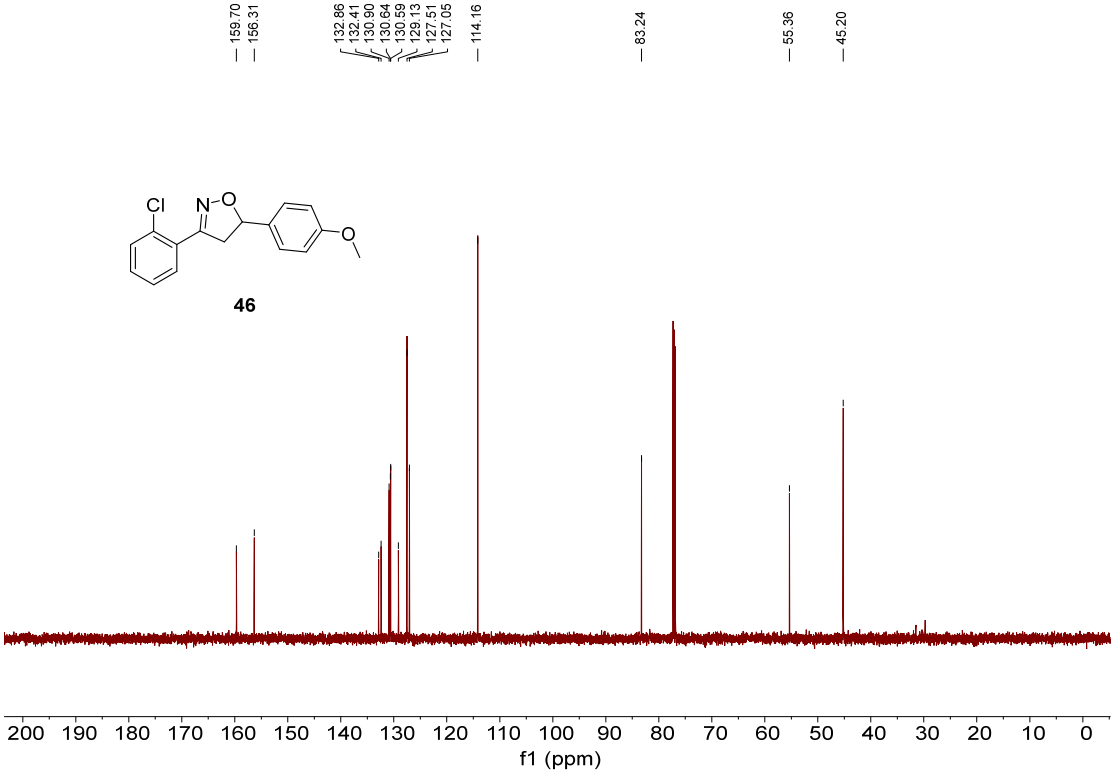
3.0
2.0
1.0
0.0
-1
f1 (ppm)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130.64
130.59
129.13
127.51
127.05
114.16
83.24
55.36
45.20
Cl
N
O
O
46
200
190
180
170
160
150
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130
120
110
100
90
80
70
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50
40
30
20
10
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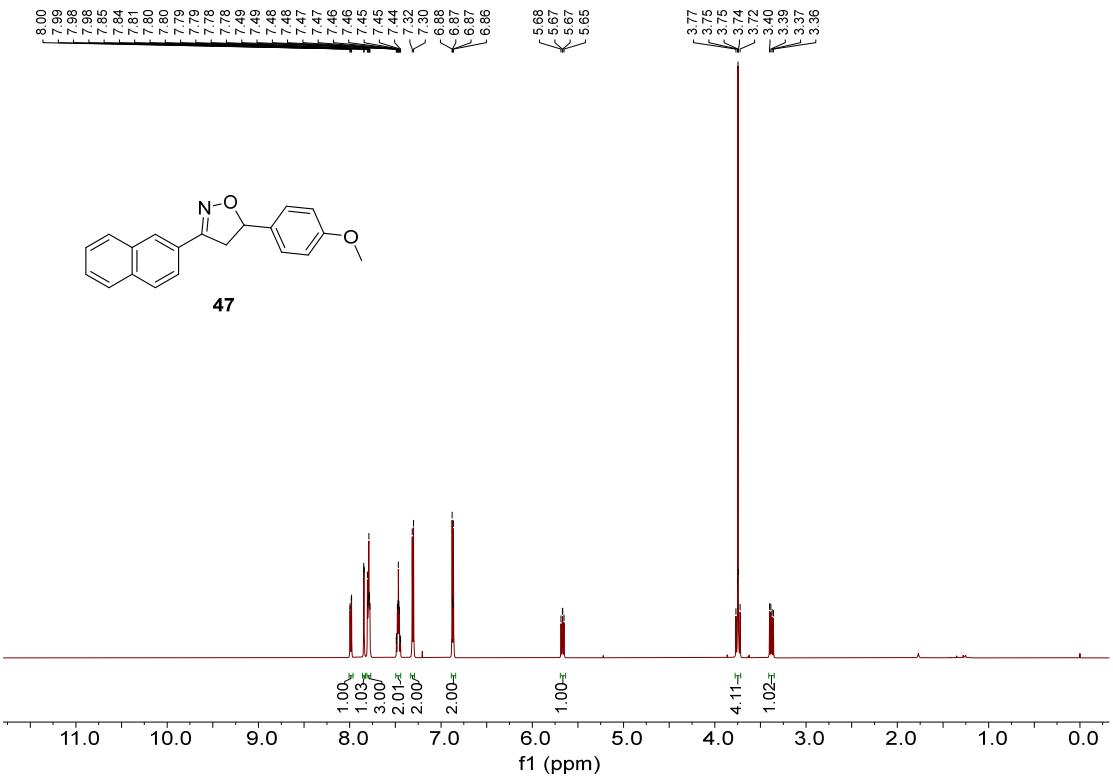
f1 (ppm)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7.49
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.45
7.44
7.32
7.30
6.88
6.87
6.87
6.86
5.68
5.67
5.67
5.65
3.77
3.75
3.75
3.74
3.72
3.40
3.39
3.37
3.36
N
O
O
47
1.00
1.03
3.00
2.01
2.00
2.00
1.00
4.11
1.02
11.0
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5.0
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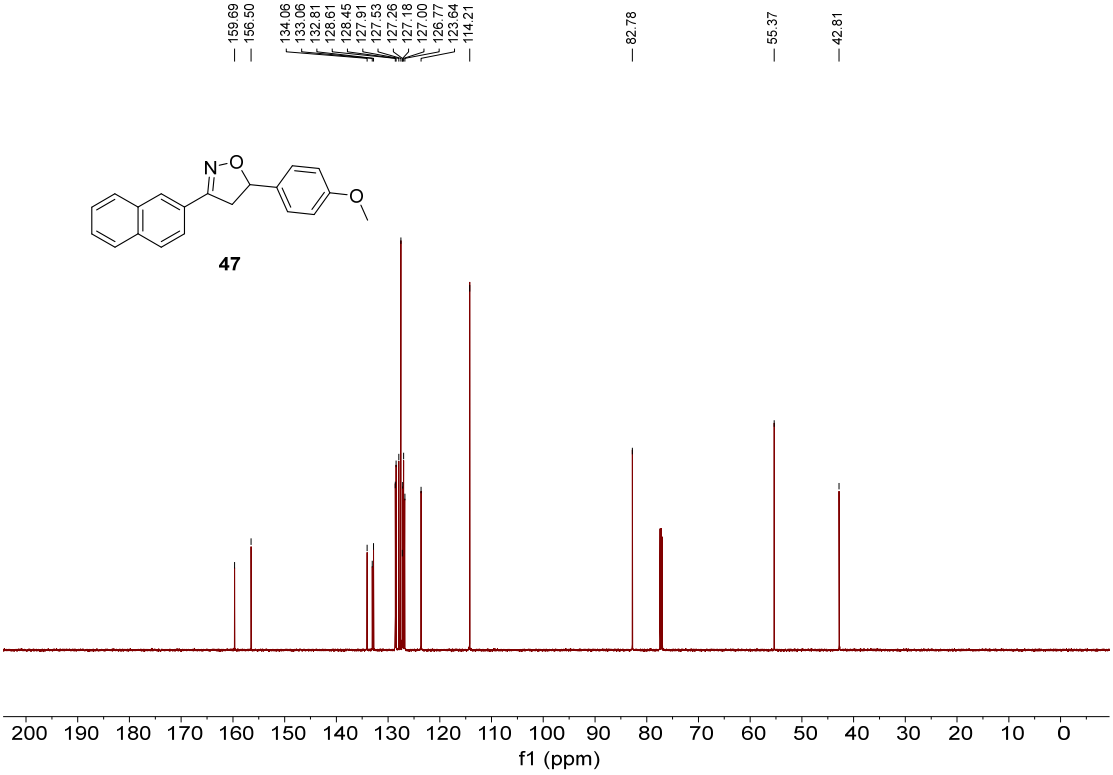
0.0
f1 (ppm)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133.06
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127.91
127.53
127.26
127.18
127.00
126.77
123.64
114.21
82.78
55.37
42.81
N
O
O
47
200
190
180
170
160
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130
120
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100
90
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70
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50
40
30
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10
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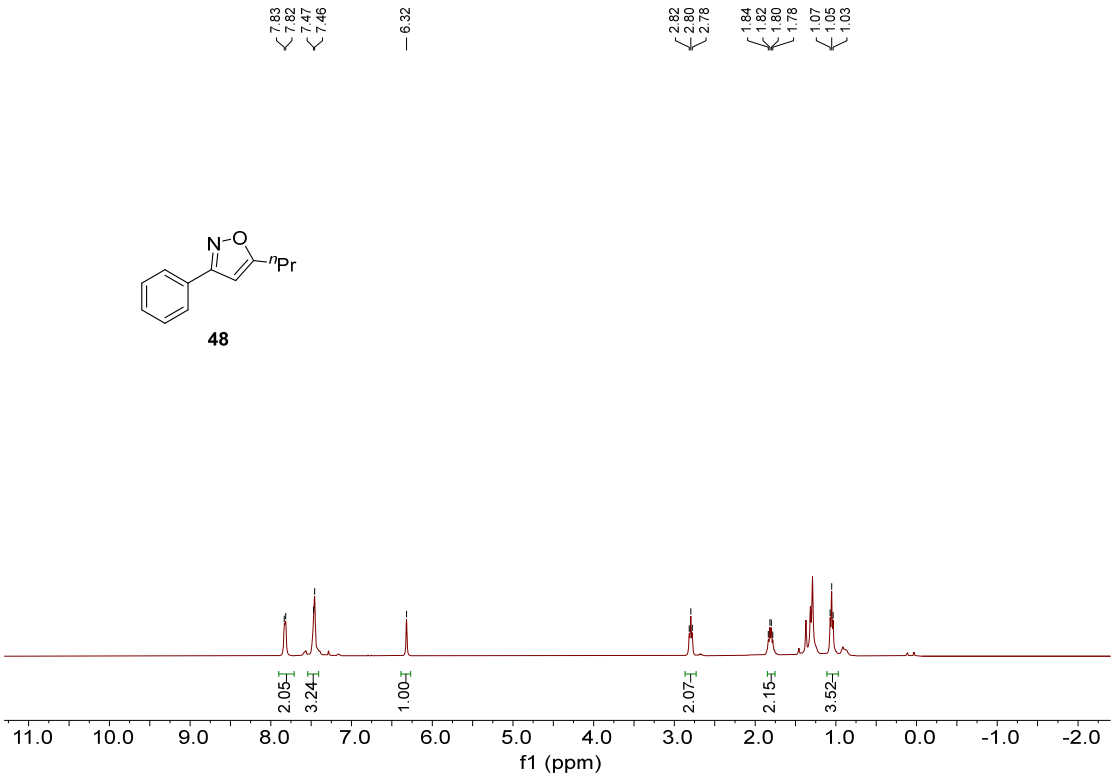
f1 (ppm)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2.78
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1.82
1.80
1.78
1.07
1.05
1.03
N
O
nPr
48
2.05
3.24
1.00
2.07
2.15
3.52
11.0
10.0
9.0
8.0
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5.0
4.0
3.0
2.0
1.0
0.0
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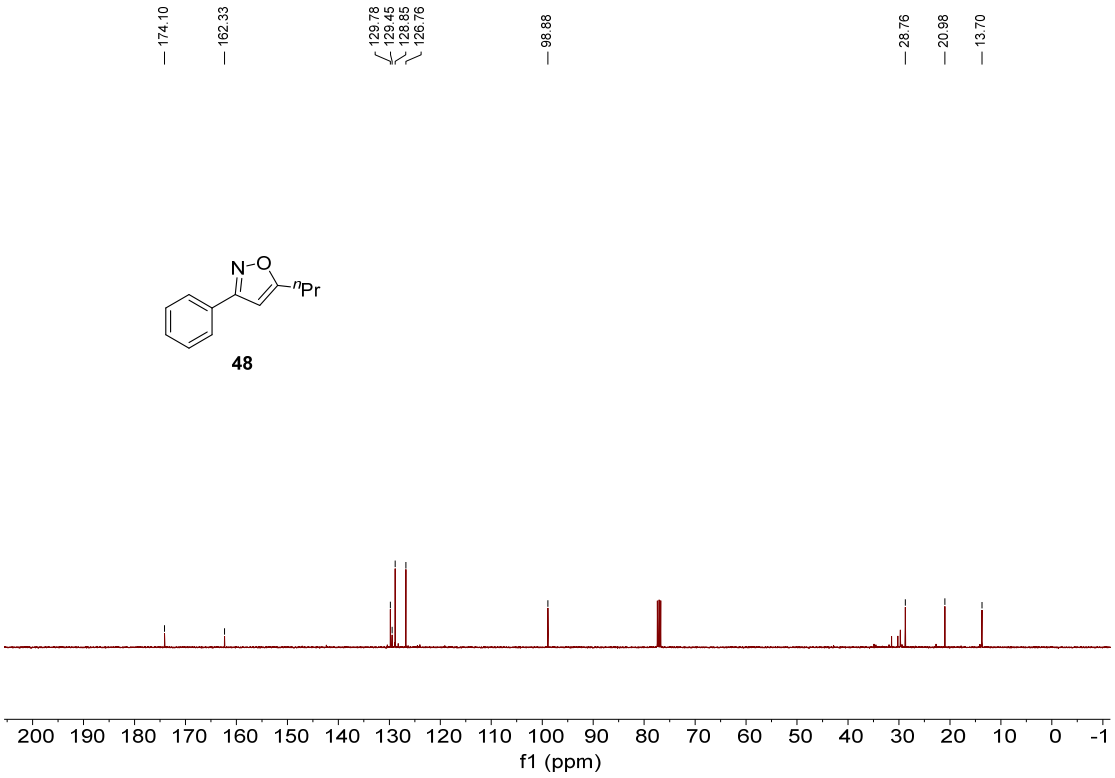
-2.0
f1 (ppm)


174.10
162.33
129.78
129.45
128.85
126.76
98.88
28.76
20.98
13.70
N
O
nPr
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200
190
180
170
160
150
140
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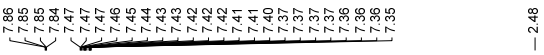
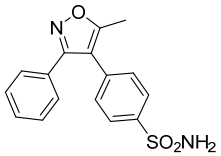
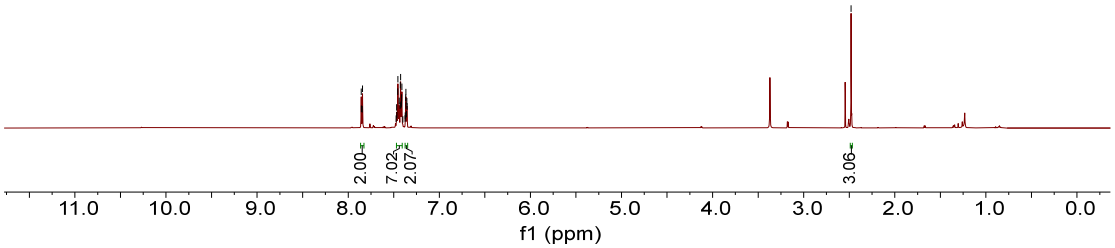
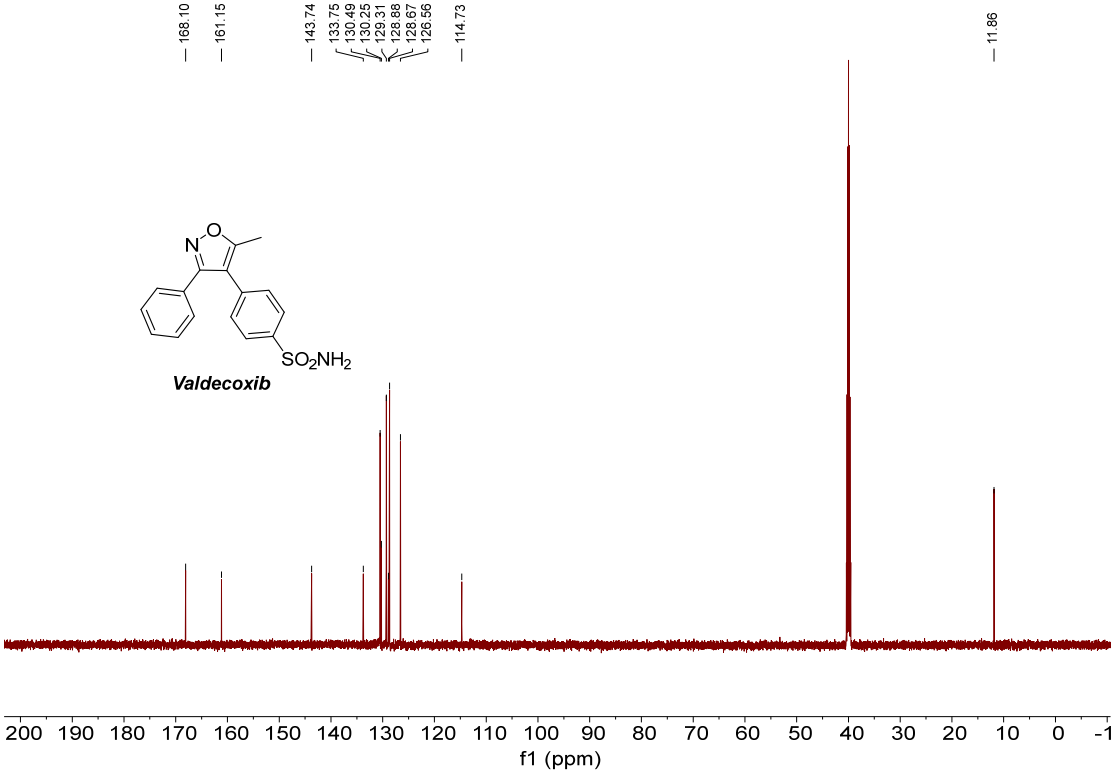
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)



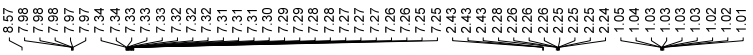


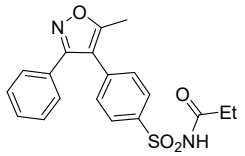
Valdecoxib



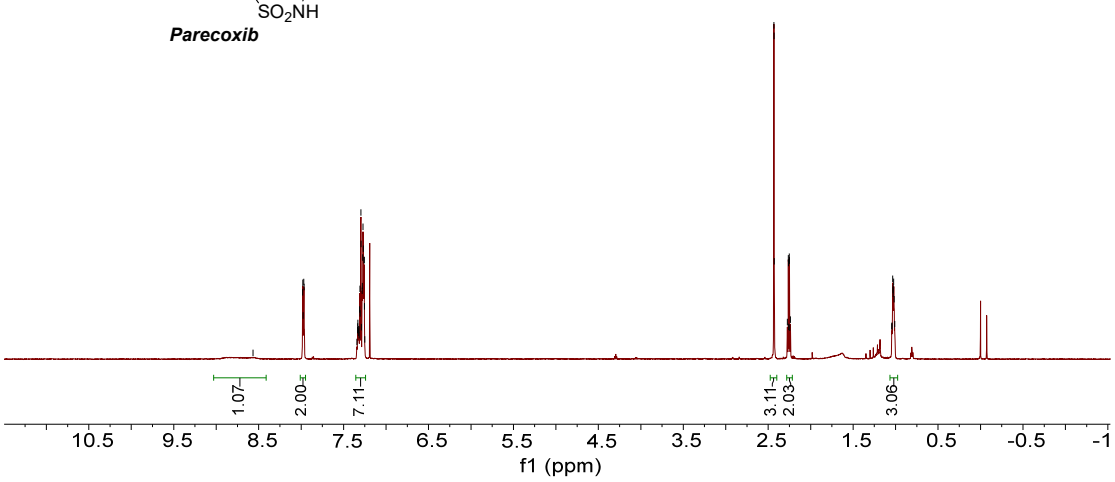


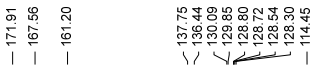
Valdecoxib

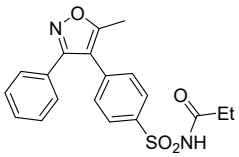




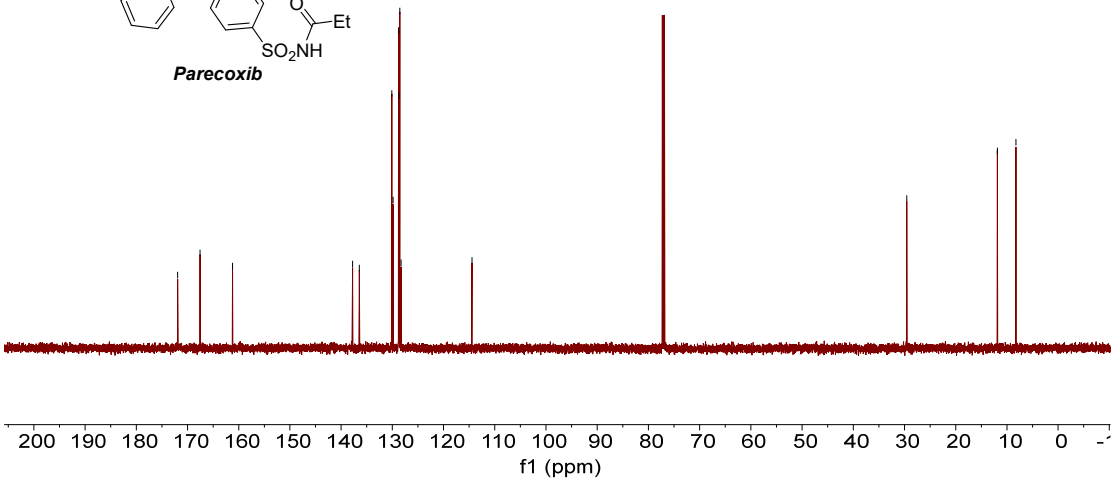
Parecoxib

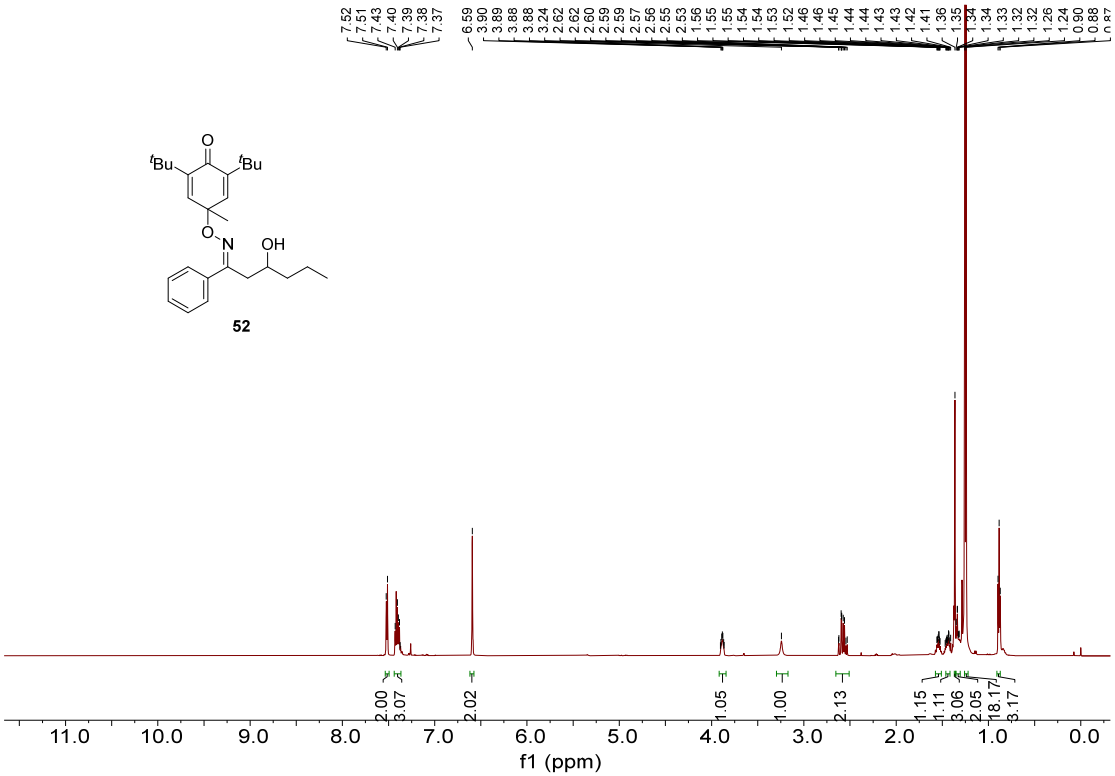






Parecoxib

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3.88
3.88
3.24
2.62
2.62
2.60
2.59
2.59
2.57
2.56
2.55
2.53
1.56
1.55
1.55
1.54
1.54
1.53
1.52
1.46
1.46
1.45
1.44
1.44
1.43
1.43
1.42
1.41
1.36
1.35
1.34
1.34
1.33
1.32
1.26
1.24
0.90
0.88
tBu
tBu
O
O
N
OH
52
2.00
3.07
2.02
1.05
1.00
2.13
1.15
1.11
3.06
2.05
18.17
3.17
11.0
10.0
9.0
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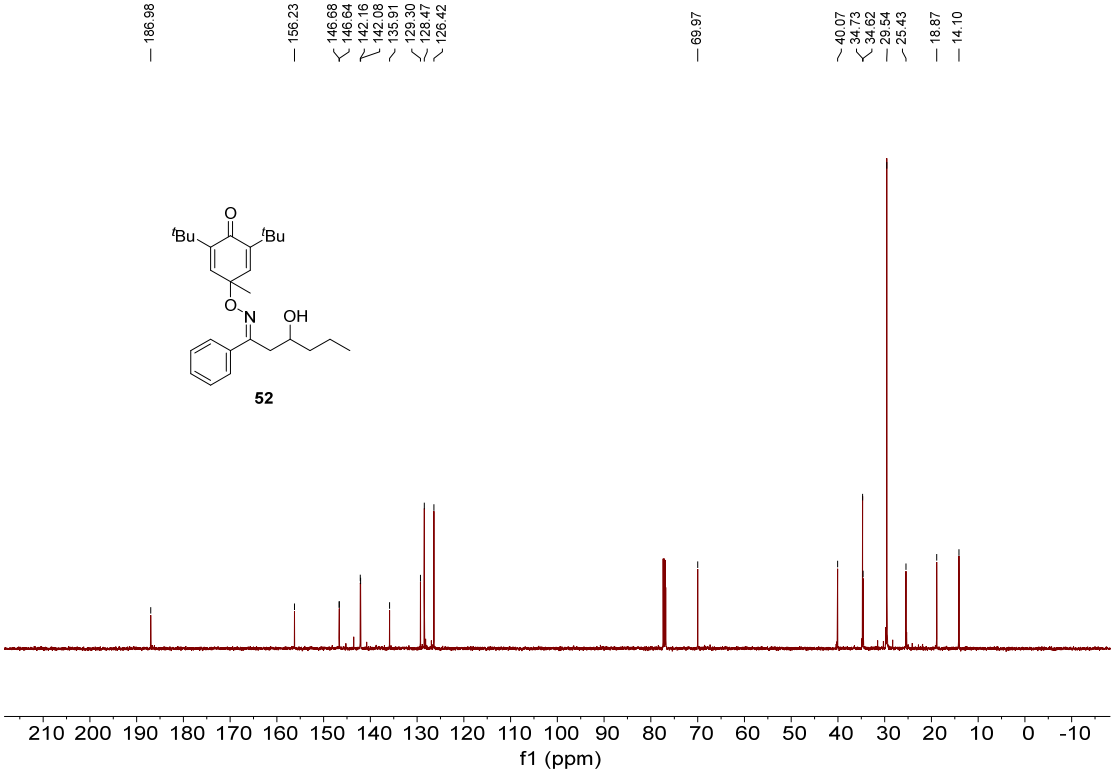
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)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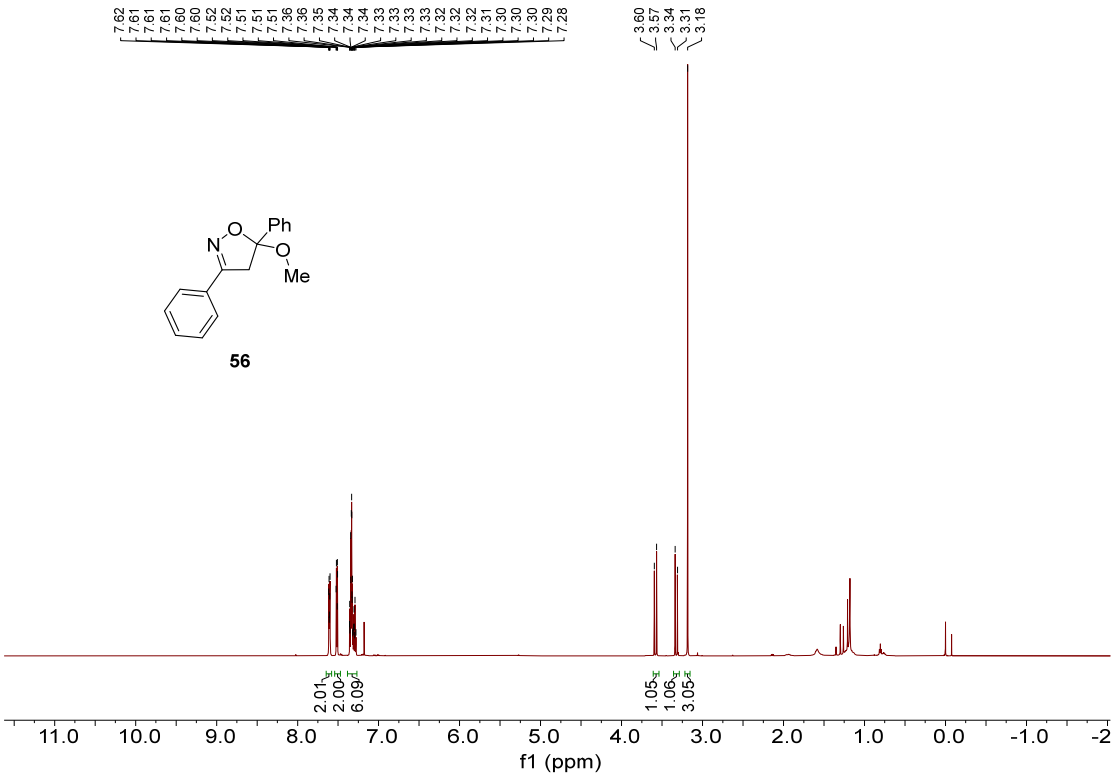
128.47
126.42
69.97
40.07
34.73
34.62
29.54
25.43
18.87
14.10
tBu
tBu
O
O
N
OH
52
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.51
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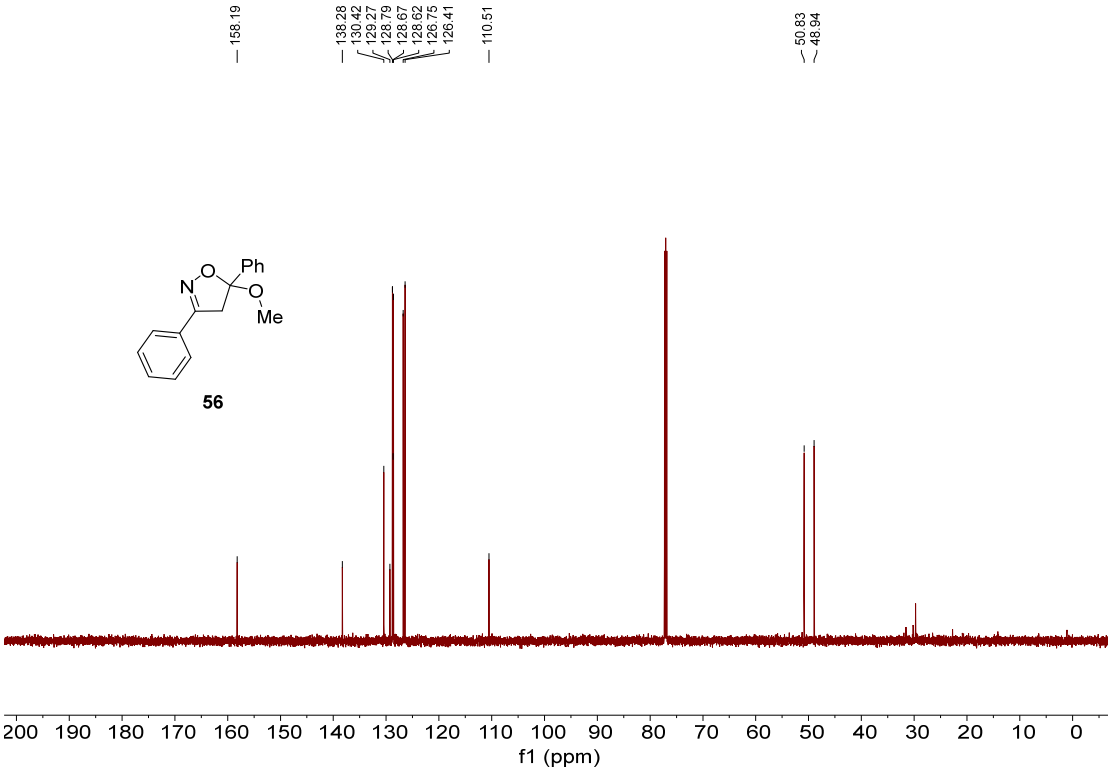
7.30
7.30
7.30
7.29
7.28
3.60
3.57
3.34
3.31
3.18
Ph
O
N
O
Me
56
2.01
2.09
6.09
1.05
1.06
3.05
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
-2
f1 (ppm)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129.27
128.79
128.67
128.62
126.75
126.41
110.51
50.83
48.94
Ph
O
N
O
Me
56
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)