Supporting Information

Hypoiodite mediated synthesis of isoxazolines from aldoximes and alkenes using catalytic KI and Oxone as the terminal oxidant

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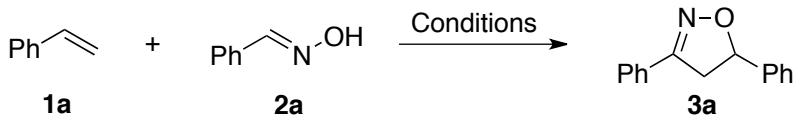
1. General experimental remarks

All reactions were performed under dry argon atmosphere with flame-dried glassware. All commercial reagents were ACS reagent grade and used without further purification. Dichloromethane was distilled from CaH_2 immediately prior to use. Diethyl ether was distilled from Na/benzophenone. All commercial reagents were ACS reagent grade and used without further purification. Melting points were determined in an open capillary tube with a Mel-temp II melting point apparatus. Infrared spectra were recorded as a KBr pellet on a Perkin-Elmer 1600 series FT-IR spectrophotometer. NMR spectra were recorded on a Varian Inova 500 MHz NMR spectrometer at 500 MHz (^{1}H NMR) and 125 MHz (^{13}C NMR). Chemical shifts are reported in parts per million (ppm). ^{1}H and ^{13}C chemical shifts are referenced relative to the tetramethylsilane.

2. Optimization study of the iodine-mediated catalytic cyclization of styrene 1a and benzaldoxime 2a.[a]



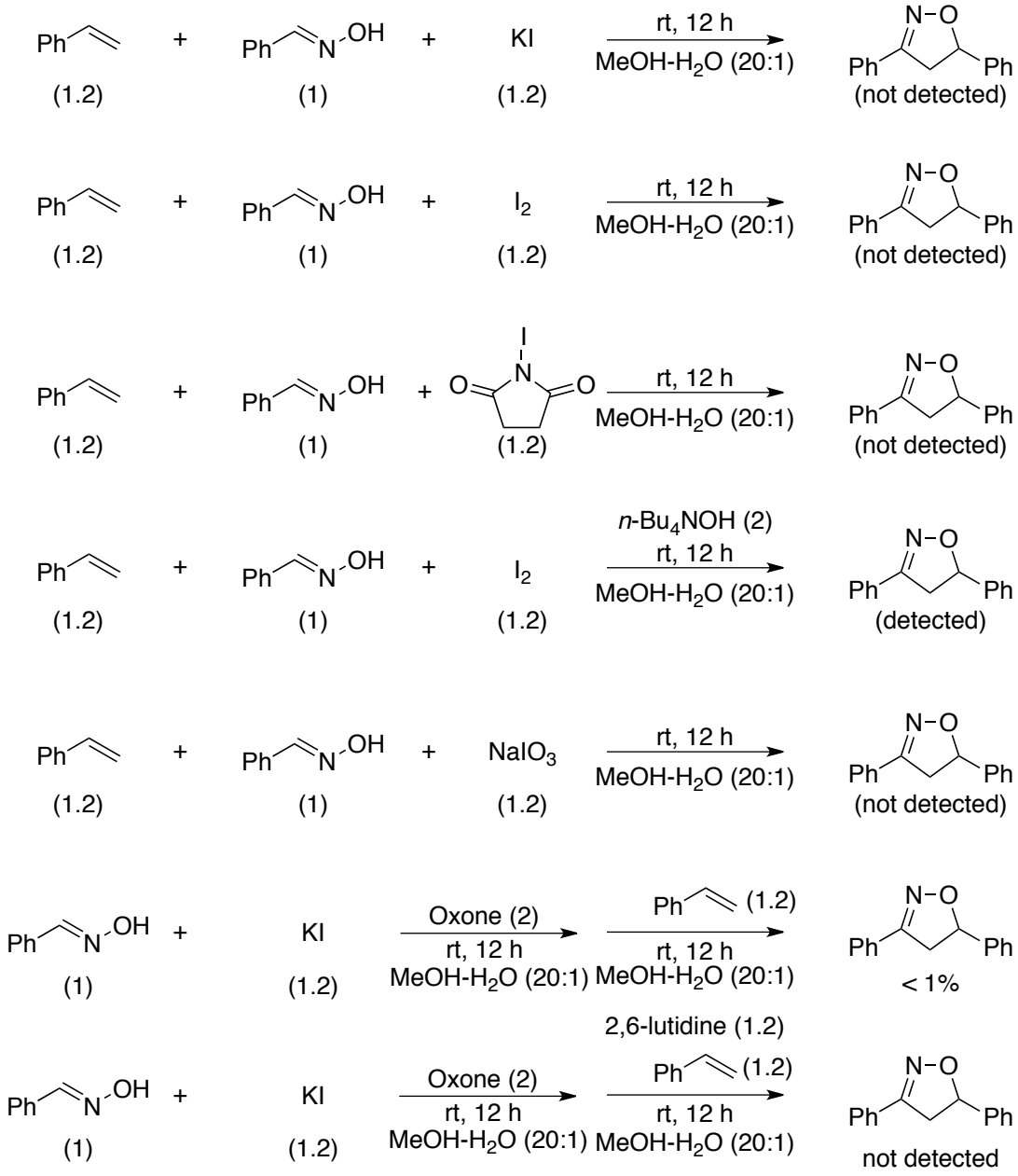
1a **2a** **3a**

Entry	Conditions	Solvent (ratio)	Oxidant (eq.)	Source of iodine (eq.)	**3a** (%)[b]
1	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	PhI (1)	(68)
2	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	PhI (0.5)	75 (75)
3	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	PhI (0.2)	(66)
4	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	*n*-Bu_4NI (0.2)	(70)
5	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	KI (0.2)	(78)

6	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	NaI (0.2)	(72)
7	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	I_2 (0.2)	(60)
8	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	*n*-Bu_4NBr (0.2)	(3)
9	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	KBr (0.2)	-
10	4 h, 40 ^{O}C	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	KI (0.2)	(80)
11	12 h, rt	MeOH-HFIP-H_2O (10:10:1)	Oxone (3)	KI (0.1)	(92)
12	12 h, rt	MeOH-H_2O (20:1)	Oxone (3)	KI (0.2)	88 (91)
13	12 h, rt	MeOH-H_2O (20:1)	Oxone (3)	KI (0.1)	89 (92)
14	12 h, rt	MeCN-H_2O (20:1)	Oxone (3)	KI (0.1)	(7)
15	12 h, rt	CH_2Cl_2-H_2O (20:1)	Oxone (3)	KI (0.1)	(1)
16	12 h, rt	AcOEt-H_2O (20:1)	Oxone (3)	KI (0.1)	(3)
17	12 h, rt	THF-H_2O (20:1)	Oxone (3)	KI (0.1)	(3)
18	12 h, rt	Toluene-H_2O (20:1)	Oxone (3)	KI (0.1)	(3)
19	12 h, rt	Hexane-H_2O (20:1)	Oxone (3)	KI (0.1)	(12)
20	12 h, rt	EtOH-H_2O (20:1)	Oxone (3)	KI (0.1)	(77)
21	12 h, rt	CH_3NO_2-H_2O (20:1)	Oxone (3)	KI (0.1)	(15)
22	12 h, rt	MeOH	Oxone (3)	KI (0.1)	-
23	12 h, rt	MeOH-H_2O (20:1)	*m*-CPBA (3)	KI (0.1)	(22)
24	12 h, rt	MeOH-H_2O (20:1)	30% H_2O_2 (3)	KI (0.1)	-
25	12 h, rt	MeOH-H_2O (20:1)	70% TBHP (3)	KI (0.1)	-
26	12 h, rt	MeOH-H_2O (20:1)	AcOOH (3)	KI (0.1)	(19)
27	12 h, rt	MeOH-H_2O (20:1)	-	KI (0.1)	-
28	12 h, rt	MeOH-H_2O (20:1)	Oxone (3)	KI (0.05)	(68)
29	24 h, rt	MeOH-H_2O (20:1)	Oxone (3)	KI (0.05)	82 (86)
30	24 h, rt	MeOH-H_2O (20:1)	Oxone (3)	KI (0.01)	(36)
31	12 h, rt	MeOH-H_2O (20:1)	Oxone (2)	KI (0.1)	86 (90)
32	12 h, rt	MeOH-H_2O (20:1)	Oxone (1.5)	KI (0.1)	88 (89)

[a] Reaction of benzaldoxime (1 equiv.) was carried out with styrene (1.4 equiv.), oxidant and iodine source. [b] Isolated yields. In parentheses are ^{1}H NMR yields.

3. *Control Experments*

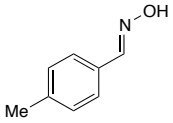


4. *Typical procedure for the synthesis of aldoximes for aldehydes*

To a mixture solution of aldehyde (5.0 mmol) and hydroxylamine hydrochloride (10 mmol) in CH_2Cl_2 (25 mL), pyridine (20.0 mmol) was added while the reaction was stirred, and the mixture was stirred at room temperature for 20 h. Upon completion, aqueous HCl (2.0 M, 15 mL) was added to the reaction mixture and the solution was

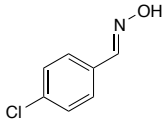
extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4 and the solvent was evaporated in vacuum. The residue was separated by column chromatography using the mixture EtOAc-hexanes (1:2) to afford the pure product **2**.

4-Methylbenzaldehyde Oxime (2b).[1]



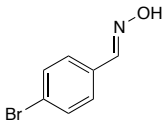
Reaction of hydroxylamine hydrochloride with 4-Methylbenzaldehyde (601 mg, 5.0 mmol) according to the general procedure afforded 541 mg (80%) of product **2b**, isolated as white solid: colorless needles (recrystallized from dichloromethane-hexane); mp 76.8-77.7 °C (lit.[2], mp 74-76 °C); IR (KBr) cm^{-1}: 3286, 2995, 2915, 1607, 1446, 1287, 712; ^1H NMR (500 MHz, $CDCl_3$): δ 8.12 (s, 1H), 7.47 (d, J = 8.0 Hz, 2H), 7.19 (d, J = 8.0 Hz, 2H), 2.37 (s, 3H).

4-Chlorobenzaldehyde Oxime (2c).[1]



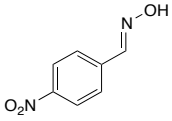
Reaction of hydroxylamine hydrochloride with 4-Chlorobenzaldehyde (703 mg, 5.0 mmol) according to the general procedure afforded 643 mg (83%) of product **2c**, isolated as white solid: colorless needles (recrystallized from dichloromethane-hexane); mp 110.3-110.7 °C (lit.[2], mp 106-108 °C); IR (KBr) cm^{-1}: 3294, 1596, 1493, 1318, 1088, 691; ^1H NMR (500 MHz, $CDCl_3$): δ 8.10 (s, 1H), 7.52 (d, J = 8.5 Hz, 2H), 7.36 (d, J = 8.5 Hz, 2H).

4-Bromobenzaldehyde Oxime (2d).[1]



Reaction of hydroxylamine hydrochloride with 4-Bromobenzaldehyde (925 mg, 5.0 mmol) according to the general procedure afforded 872 mg (87%) of product **2d**, isolated as white solid: colorless needles (recrystallized from dichloromethane-hexane); mp 113.5-114.1 °C (lit.[2], mp 109-111 °C); IR (KBr) cm^{-1}: 3294, 1588, 1489, 1317, 685; ^1H NMR (500 MHz, $CDCl_3$): δ 8.08 (s, 1H), 7.52 (d, J = 8.5 Hz, 2H), 7.45 (d, J = 8.5 Hz, 2H).

4-Nitrobenzaldehyde Oxime (2e).[1]



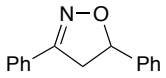
Reaction of hydroxylamine hydrochloride with 4-Nitrobenzaldehyde (756 mg, 5.0 mmol) according to the general procedure afforded 733 mg (88%) of product **2e**, isolated as yellow solid: yellow needles (recrystallized from dichloromethane-hexane); mp 131.3-132.0 °C (lit.[2], mp 132-134 °C); IR (KBr) cm^{-1}: 3306, 1606, 1538, 1350, 1324, 688; ^1H NMR (500 MHz, $CDCl_3$): δ 8.25 (d, J = 9.3 Hz, 2H), 8.20 (s, 1H), 7.75 (d, J = 9.3 Hz, 2H).

5. Typical procedure for the synthesis of aldoximes for aldehydes

To a solution of alkene **1** (0.3 mmol) in a solvent mixture of MeOH (1.5 mL), and H_2O (0.075 mL) were added Oxone (307 mg, 0.75 mmol), benzalde oxime **2** (0.25 mmol) and potassium iodide (4.2 mg, 0.025 mmol). The reaction was stirred at room temperature for 12-24 h. After reaction, the mixture was filtered, the inorganic solids on the filter were washed with CH_2Cl_2, then 5% aqueous $Na_2S_2O_3$ (5 mL) was added to the filtrate, and the

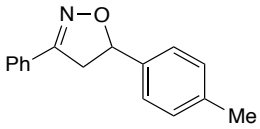
resulting solution was extracted with dichloromethane. The organic phase was dried over anhydrous Na_2SO_4 and concentrated. Purification by preparative TLC (hexane-ethyl acetate = 9 : 1 or 95 : 5) afforded analytically pure isoxazolines **3**.

3,5-Diphenyl-2-isoxazoline (3a)[1]



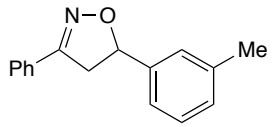
Reaction of styrene **1a** (31 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 50 mg (89%) of product **3a**, isolated as a yellow solid: light yellow needles (recrystallized from dichloromethane-hexane); mp 71.2-72.3 °C (lit.[1], mp 73-74 °C); IR (KBr) cm^{-1} 3029, 2971, 2877, 1448, 1364, 1064, 894, 860, 752, 687; ^1H NMR (500 MHz, $CDCl_3$): δ 7.72-7.65 (m, 2H), 7.43-7.28 (m, 8H), 5.73 (dd, *J* = 11.0, 8.3 Hz, 1H), 3.77 (dd, *J* = 16.8, 11.0 Hz, 1H), 3.33 (dd, *J* = 16.8, 8.3 Hz, 1H).

5-(*p*-Methylphenyl)-3-phenyl-2-isoxazoline (3b)[3]



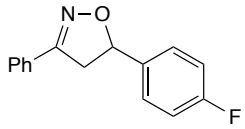
Reaction of *p*-methylstyrene **1b** (35 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 51 mg (86%) of product **3b**, isolated as a yellow solid: colorless needles (recrystallized from dichloromethane-hexane); mp 84.2-84.8 °C (lit.[3], mp 94-95 °C); IR (KBr) cm^{-1} 3043, 2929, 2917, 1364, 906, 869, 760, 691; ^1H NMR (500 MHz, $CDCl_3$): δ 7.72-7.65 (m, 2H), 7.44-7.36 (m, 3H), 7.28 (d, *J* = 8.3 Hz, 2H), 7.18 (d, *J* = 8.3 Hz, 2H), 5.70 (dd, *J* = 11.0, 8.5 Hz, 1H), 3.74 (dd, *J* = 16.5, 11.0 Hz, 1H), 3.32 (dd, *J* = 16.5, 8.5 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.2, 138.1, 138.0, 130.1, 129.6, 129.5, 128.8, 126.8, 126.0, 82.6, 43.1, 21.2.

5-(*m*-Methylphenyl)-3-phenyl-2-isoxazoline (3c)[4]



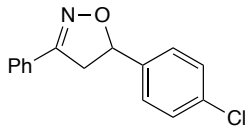
Reaction of *m*-methylstyrene **1c** (35 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 49 mg (83%) of product **3c**, isolated as a yellow oil; IR (neat) cm^{-1} 3030, 2920, 1446, 1359, 1077, 902, 848, 761, 693; ^1H NMR (500 MHz, $CDCl_3$) δ 7.74-7.65 (m, 2H), 7.45-7.36 (m, 3H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.21 (s, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 1H), 5.69 (dd, *J* = 10.8, 8.4 Hz, 1H), 3.74 (dd, *J* = 16.5, 10.8 Hz, 1H), 3.32 (dd, *J* = 16.5, 8.4 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.2, 140.9, 138.6, 130.1, 129.0, 128.7, 126.8, 126.6, 123.0, 82.7, 43.2, 21.5.

5-(*p*-Fluorophenyl)-3-phenyl-2-isoxazoline (3d)[5]



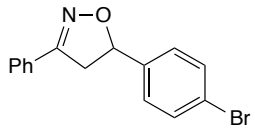
Reaction of *p*-fluorostyrene **1d** (37 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 52 mg (87%) of product **3d**, isolated as a yellow solid: colorless plates (recrystallized from dichloromethane-hexane); mp 89.2-89.8 °C (lit.[5], mp 90-91.5 °C); IR (KBr) cm^{-1} 3048, 2976, 2869, 1446, 1310, 1062, 899, 869, 759, 691; ^1H NMR (500 MHz, $CDCl_3$) δ 7.73-7.66 (m, 2H), 7.45-7.39 (m, 3H), 7.37 (dd, *J* = 8.5, 5.5 Hz, 2H), 7.05 (dd, *J* = 9.0, 8.5 Hz, 2H), 5.71 (dd, *J* = 10.8, 8.4 Hz, 1H), 3.77 (dd, *J* = 16.6, 10.8 Hz, 1H), 3.30 (dd, *J* = 16.8, 8.4 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.6 (d, J_{CF} = 245.3 Hz), 156.2, 136.8 (d, $^4J_{CF}$ = 3.1 Hz), 130.3, 129.4, 128.8, 127.7 (d, $^3J_{CF}$ = 7.9 Hz), 126.8, 115.7 (d, $^2J_{CF}$ = 21.5 Hz), 82.0, 43.2.

5-(*p*-Chlorophenyl)-3-phenyl-2-isoxazoline (3e)[4]



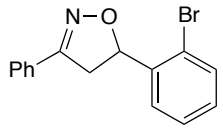
Reaction of *p*-chlorostyrene **1e** (42 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 59 mg (92%) of product **3e**, isolated as a yellow solid: colorless blocks (recrystallized from dichloromethane-hexane); mp 109.7-111.1 °C (lit.[4], mp 110.0-111.5 °C); IR (KBr) cm^{-1} 3048. 2988, 2856, 1448, 1367, 1094, 1069, 896, 860, 760, 688; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71-7.65 (m, 2H), 7.44-7.38 (m, 3H), 7.36-7.29 (m, 4H), 5.70 (dd, *J* = 11.0, 8.3 Hz, 1H), 3.77 (dd, *J* = 16.5, 11.0 Hz, 1H), 3.28 (dd, *J* = 16.5, 8.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 139.6, 134.0, 130.3, 129.3, 129.0, 128.8, 127.3, 126.8, 81.8, 43.2.

5-(*p*-Bromophenyl)-3-phenyl-2-isoxazoline (3f)[6]



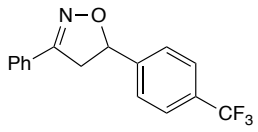
Reaction of *p*-bromostyrene **1f** (55 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 70 mg (92%) of product **3f**, isolated as a yellow solid: colorless plates (recrystallized from dichloromethane-hexane); mp 129.8-130.3 °C (lit.[6], mp 130-131 °C); IR (KBr) cm^{-1} 3048, 2976, 2895, 1369, 1327, 1055, 887, 862, 762, 690; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.70-7.64 (m, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.43-7.36 (m, 3H), 7.25 (d, *J* = 8.5 Hz, 2H), 5.67 (dd, *J* = 11.0, 8.0 Hz, 1H), 3.76 (dd, *J* = 16.5, 11.0 Hz, 1H), 3.27 (dd, *J* = 16.5, 8.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 140.1, 131.9, 130.3, 129.3, 128.8, 127.6, 126.8, 122.1, 81.8, 43.2.

5-(*o*-Bromophenyl)-3-phenyl-2-isoxazoline (3g)



Reaction of *o*-bromostyrene **1g** (55 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 66 mg (87%) of product **3g**, isolated as a yellow oil; IR (neat) cm^{-1} 3062, 2926, 2857, 1446, 1357, 1076, 901, 863, 756, 692; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71-7.66 (m, 2H), 7.57 (dd, *J* = 7.8, 0.8 Hz, 1H), 7.56 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.42-7.36 (m, 3H), 7.33 (td, *J* = 7.8, 0.8 Hz, 1H), 7.17 (td, *J* = 7.8, 1.8 Hz, 1H), 5.99 (dd, *J* = 11.1, 7.1 Hz, 1H), 3.96 (dd, *J* = 16.8, 11.1 Hz, 1H), 3.20 (dd, *J* = 16.8, 7.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 140.7, 132.8, 130.3, 129.4, 129.3, 128.8, 127.9, 126.9, 126.8, 120.9, 81.5, 43.0.

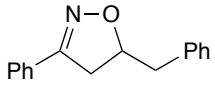
3-Phenyl-5-(*p*-trifluoromethyl-phenyl)-2-isoxazoline (3h)



Reaction of *p*-trifluoromethylstyrene **1h** (52 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 67 mg (92%) of product **3h**, isolated as a yellow solid: colorless plates (recrystallized from dichloromethane-hexane); mp 143.2-144.1 °C; IR (KBr) cm^{-1} 3062, 2914, 1423, 1332, 1121, 1072, 903, 840, 766, 694; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71-7.66 (m, 2H), 7.64 (d, *J* = 8.3, 2H), 7.52 (d, *J* = 8.3, 2H), 7.46-7.36 (m, 3H), 5.80 (dd, *J* = 11.8, 7.5 Hz, 1H), 3.77 (dd, *J* = 16.5, 11.8 Hz, 1H), 3.32 (dd, *J* = 16.5, 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.0, 145.1, 130.4 (q, $^{2}J_{CF}$ = 32.3 Hz), 130.4, 129.1, 128.8, 126.8, 126.1, 125.8 (q, $^{3}J_{CF}$ = 3.8 Hz), 124.0 (q, $^{1}J_{CF}$ = 270.5 Hz),

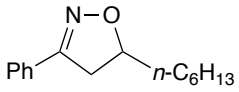
81.6, 43.4.

5-Benzyl-3-phenyl-2-isoxazoline (3i)[7]



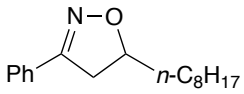
Reaction of allylbenzene **1i** (35 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 48 mg (81%) of product **3i**, isolated as a yellow solid: colorless needles (recrystallized from dichloromethane-hexane); mp 67.5-68.2 °C (lit.[7], mp 67.5-68.2 °C); IR (KBr) cm^{-1} 3024, 2940, 2914, 1446, 1360, 1083, 916, 881, 754, 700; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.67-7.60 (m, 2H), 7.42-7.35 (m, 3H), 7.34-7.29 (m, 2H), 7.28-7.21 (m, 3H), 5.03-4.94 (m, 1H), 3.30 (dd, *J* = 16.5, 11.0 Hz, 1H), 3.17 (dd, *J* = 13.5, 6.0 Hz, 1H), 3.05 (dd, *J* = 16.5, 7.8 Hz, 1H), 2.88 (dd, *J* = 13.5, 7.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.5, 137.0, 130.0, 129.4, 128.7, 128.7, 126.8, 126.7, 81.9, 41.1, 39.4.

5-Hexyl-3-phenyl-2-isoxazoline (3j)[1]



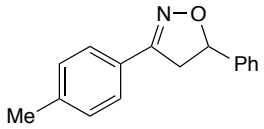
Reaction of 1-octene **1j** (34 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 45 mg (78%) of product **3j**, isolated as a yellow solid: colorless plates (recrystallized from dichloromethane-hexane); mp 49.4-50.7 °C (lit.[1], mp 47-48 °C); IR (KBr) cm^{-1} 2952, 2922, 2857, 1450, 1363, 755, 696; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71-7.64 (m, 2H), 7.43-7.37 (m, 3H), 4.79-4.68 (m, 1H), 3.39 (dd, *J* = 16.3, 11.0 Hz, 1H), 2.97 (dd, *J* = 16.3, 8.0 Hz, 1H), 1.86-1.74 (m, 1H), 1.68-1.58 (m, 1H), 1.53-1.24 (m, 8H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.4, 130.0, 129.9, 128.7, 126.6, 81.5, 40.0, 35.4, 31.7, 29.1, 25.5, 22.6, 14.1.

5-Octyl-3-phenyl-2-isoxazoline (3k)[8]



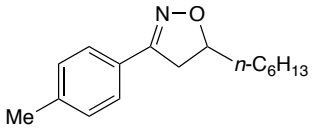
Reaction of 1-decene **1k** (42 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25 mmol) according to the general procedure afforded 45 mg (69%) of product **3k**, isolated as a yellow solid: colorless plates (recrystallized from dichloromethane-hexane); mp 60.4-61.0 °C (lit.[8], mp 60 °C); IR (KBr) cm^{-1} 2954, 2921, 2849, 1447, 1361, 755, 690; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.70-7.64 (m, 2H), 7.42-7.36 (m, 3H), 4.77-4.67 (m, 1H), 3.38 (dd, *J* = 16.5, 10.5 Hz, 1H), 2.96 (dd, *J* = 16.5, 8.5 Hz, 1H), 1.83-1.74 (m, 1H), 1.68-1.58 (m, 1H), 1.54-1.20 (m, 12H), 0.88 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.4, 130.0, 129.9, 128.7, 126.6, 81.5, 40.0, 35.4, 31.9, 29.5, 29.5, 29.2, 25.6, 22.7, 14.1.

5-Phenyl-3-(*p*-methylphenyl)-2-isoxazoline (3l)[1]



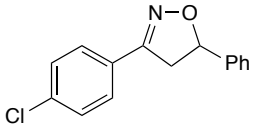
Reaction of styrene **1a** (31 mg, 0.30 mmol) and *p*-methylbenzaldehyde oxime **2b** (31 mg, 0.25 mmol) according to the general procedure afforded 45 mg (76%) of product **3l**, isolated as a yellow solid: colorless blocks (recrystallized from dichloromethane-hexane); mp 94.9-96.0 °C (lit.[1], mp 97 °C); IR (KBr) cm^{-1} 3048, 2905, 2869, 1450, 1352, 901, 824, 758, 698; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.42-7.26 (m, 5H), 7.19 (d, *J* = 8.0 Hz, 2H), 5.69 (dd, *J* = 10.9, 8.4 Hz, 1H), 3.30 (dd, *J* = 16.5, 8.4 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 141.1, 140.4, 129.4, 128.7, 128.2, 126.7, 126.7, 125.9, 82.4, 43.3, 21.4.

5-Hexyl-3-(*p*-methylphenyl)-2-isoxazoline (3m)



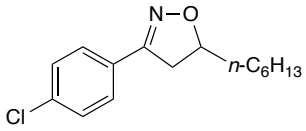
Reaction of 1-octene **1j** (34 mg, 0.30 mmol) and *p*-methylbenzaldehyde oxime **2b** (34 mg, 0.25 mmol) according to the general procedure afforded 39 mg (64%) of product **3m**, isolated as a yellow solid: white solid (recrystallized from dichloromethane-hexane); mp 49.5-50.0 °C; IR (KBr) cm^{-1} 2953, 2927, 2849, 1450, 1363, 1058, 898, 816; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56 (d, *J* = 7.5 Hz, 2H), 7.20 (d, *J* = 7.5 Hz, 2H), 4.76-4.64 (m, 1H), 3.37 (dd, *J* = 16.3, 10.3 Hz, 1H), 2.94 (dd, *J* = 16.3, 8.3 Hz, 1H), 2.37 (s, 3H), 1.84-1.73 (m, 1H), 1.67-1.56 (m, 1H), 1.53-1.23 (m, 8H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.3, 140.1, 129.3, 127.2, 126.5, 81.3, 40.1, 35.4, 31.7, 29.1, 25.5, 22.6, 21.4, 14.1.

5-Phenyl-3-(*p*-chlorophenyl)-2-isoxazoline (3n)[1]



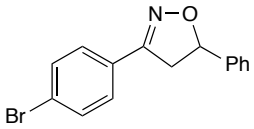
Reaction of styrene **1a** (31 mg, 0.30 mmol) and *p*-chlorobenzaldehyde oxime **2c** (39 mg, 0.25 mmol) according to the general procedure afforded 58 mg (91%) of product **3n**, isolated as a yellow solid: colorless plates (recrystallized from dichloromethane-hexane); mp 124.2-125.2 °C (lit.[1], mp 127-129 °C); IR (KBr) cm^{-1} 3043, 2905, 1417, 1348, 1093, 907, 837, 758, 699; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.5 Hz, 2H), 7.42-7.27 (m, 7H), 5.73 (dd, *J* = 11.1, 8.5 Hz, 1H), 3.73 (dd, *J* = 16.6, 11.1 Hz, 1H), 3.29 (dd, *J* = 16.6, 8.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.2, 140.7, 136.1, 129.0, 128.8, 128.3, 128.0, 128.0, 125.9, 82.8, 43.0.

5-Hexyl-3-(*p*-chlorophenyl)-2-isoxazoline (3o)



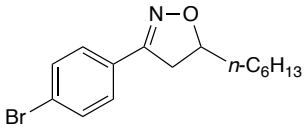
Reaction of 1-octene **1j** (34 mg, 0.30 mmol) and *p*-chlorobenzaldehyde oxime **2c** (39 mg, 0.25 mmol) according to the general procedure afforded 61 mg (92%) of product **3o**, isolated as a yellow solid: white solide (recrystallized from dichloromethane-hexane); mp 75.3-75.9 °C; IR (KBr) cm^{-1} 2952, 2926, 2848, 1470, 1357, 1097, 903, 829; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.59 (d, *J* = 8.5 Hz, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 4.79-4.68 (m, 1H), 3.35 (dd, *J* = 16.5, 10.5 Hz, 1H), 2.92 (dd, *J* = 16.5, 8.5 Hz, 1H), 1.83-1.74 (m, 1H), 1.66-1.57 (m, 1H), 1.53-1.24 (m, 8H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.5, 135.8, 128.9, 128.5, 127.8, 81.8, 39.8, 35.3, 31.7, 29.1, 25.5, 22.6, 14.1.

5-Phenyl-3-(*p*-bromophenyl)-2-isoxazoline (3p)[1]



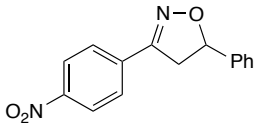
Reaction of styrene **1a** (31 mg, 0.30 mmol) and *p*-bromobenzaldehyde oxime **2d** (50 mg, 0.25 mmol) according to the general procedure afforded 60 mg (79%) of product **3p**, isolated as a yellow solid: colorless plates (recrystallized from dichloromethane-hexane); mp 136.9-137.4 °C (lit.[1], mp 141-142 °C); IR (KBr) cm^{-1} 3071, 3024, 2898, 1435, 1351, 1071, 1008, 906, 832, 758, 698; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.58-7.51 (m, 4H), 7.40-7.29 (m, 5H), 5.75 (dd, *J* = 11.0, 8.3 Hz, 1H), 3.75 (dd, *J* = 16.8, 11.0 Hz, 1H), 3.31 (dd, *J* = 16.8, 8.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.3, 140.6, 132.0, 128.8, 128.4, 128.3, 128.2, 125.8, 124.4, 82.9, 42.9.

5-Hexyl-3-(*p*-bromophenyl)-2-isoxazoline (3q)



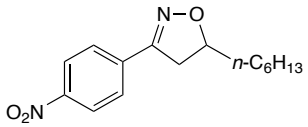
Reaction of 1-octene **1j** (34 mg, 0.30 mmol) and *p*-bromobenzaldehyde oxime **2d** (50 mg, 0.25 mmol) according to the general procedure afforded 58 mg (74%) of product **3q**, isolated as a yellow solid: white solide (recrystallized from dichloromethane-hexane); mp 82.4-83.3 °C; IR (KBr) cm^{-1} 2950, 2926, 2848, 1466, 1400, 1076, 1010, 903, 826, 708; ^1H NMR (500 MHz, $CDCl_3$): δ 7.52 (s, 4H), 4.79-4.69 (m, 1H), 3.35 (dd, *J* = 16.5, 10.5 Hz, 1H), 2.93 (dd, *J* = 16.5, 8.5 Hz, 1H), 1.84-1.74 (m, 1H), 1.67-1.57 (m, 1H), 1.53-1.24 (m, 8H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.6, 131.9, 128.9, 128.0, 124.1, 81.9, 39.7, 35.3, 31.7, 29.1, 25.5, 22.6, 14.1.

5-Phenyl-3-(*p*-nitrophenyl)-2-isoxazoline (3r)[1]



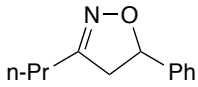
Reaction of styrene **1a** (31 mg, 0.30 mmol) and *p*-nitrobenzaldehyde oxime **2e** (42 mg, 0.25 mmol) according to the general procedure afforded 34 mg (51%) of product **3r**, isolated as a yellow solid: light yellow needles (recrystallized from dichloromethane-hexane); mp 128.8-129.3 °C (lit.[1], mp 127-128 °C); IR (KBr) cm^{-1} 3036, 2929, 2845, 1510, 1429, 1335, 1107, 918, 846, 753, 691; ^1H NMR (500 MHz, $CDCl_3$): δ 8.27 (d, *J* = 8.5 Hz, 2H), 7.86 (d, *J* = 8.5 Hz, 2H), 7.46-7.32 (m, 5H), 5.84 (dd, *J* = 11.1, 8.5 Hz, 1H), 3.81 (dd, *J* = 16.6, 11.1 Hz, 1H), 3.39 (dd, *J* = 16.6, 8.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.6, 148.5, 140.2, 135.6, 128.9, 128.6, 127.4, 125.8, 124.0, 83.7, 42.5.

5-Hexyl-3-(*p*-nitrophenyl)-2-isoxazoline (3s)



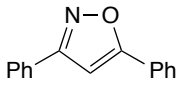
Reaction of 1-octene **1j** (34 mg, 0.30 mmol) and *p*-nitrobenzaldehyde oxime **2e** (42 mg, 0.25 mmol) according to the general procedure afforded 46 mg (78%) of product **3s**, isolated as a yellow solid: light yellow needles (recrystallized from dichloromethane-hexane); mp 64.7-65.5 °C; IR (KBr) cm^{-1} 2952, 2919, 2854, 1512, 1346, 1108, 918, 843, 751, 691; ^1H NMR (500 MHz, $CDCl_3$): δ 8.26 (d, *J* = 8.0 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 2H), 4.90-4.76 (m, 1H), 3.43 (dd, *J* = 16.3, 10.5 Hz, 1H), 3.00 (dd, *J* = 16.3, 8.5 Hz, 1H), 1.89-1.77 (m, 1H), 1.73-1.59 (m, 1H), 1.55-1.20 (m, 8H), 0.90 (t, *J* = 6.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.9, 148.3, 136.1, 127.2, 124.0, 82.8, 39.3, 35.3, 31.7, 29.1, 25.4, 22.5, 14.1.

5-Phenyl-3-propyl-2-isoxazoline (3t)[1]



Reaction of styrene **1a** (31 mg, 0.30 mmol) and butylaldehyde oxime **2f** (22 mg, 0.25 mmol) according to the general procedure afforded 6 mg (13 %) of product **3t**, isolated as a colorless oil; IR (neat) cm^{-1} 3031, 2962, 2933, 2874, 1455, 1327, 874, 758, 700; ^1H NMR (500 MHz, $CDCl_3$): δ 7.39-7.27 (m, 5H), 5.55 (dd, *J* = 11.0, 8.0 Hz, 1H), 3.35 (dd, *J* = 17.0, 11.0 Hz, 1H), 2.89 (dd, *J* = 17.0, 8.0 Hz, 1H), 2.37 (t, *J* = 7.8 Hz, 2H), 1.67-1.57 (m, 2H), 0.97 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.4, 141.4, 128.7, 128.0, 125.7, 81.2, 45.3, 29.6, 19.8, 13.8.

3,5-Diphenylisoxazole (5)[1]



Reaction of phenylacetylene **4** (31 mg, 0.30 mmol) and benzaldehyde oxime **2a** (30 mg, 0.25

mmol) according to the general procedure afforded 4 mg (7%) of product **5**, isolated as a yellow solid: light colorless needles (recrystallized from dichloromethane-hexane); mp 138.8-139.4 °C (lit.[1], mp 139-140 °C); IR (KBr) cm^{-1} 3381, 3119, 765, 692; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90-7.82 (m, 4H), 7.54-7.41 (m, 6H), 6.83 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.4, 163.0, 130.2, 130.0, 129.2, 129.0, 128.9, 127.5, 126.8, 125.8, 97.5.

References:

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2. M. Javad, N.-J. Mohammad R, H. Hamideh, D. Mohammad G, K. Gerd, *ChemSusChem* 2009, **3**, 248-254.
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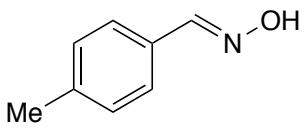
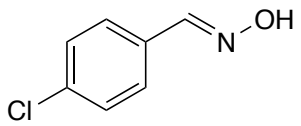
^{1}H NMR (500 MHz, $CDCl_3$):

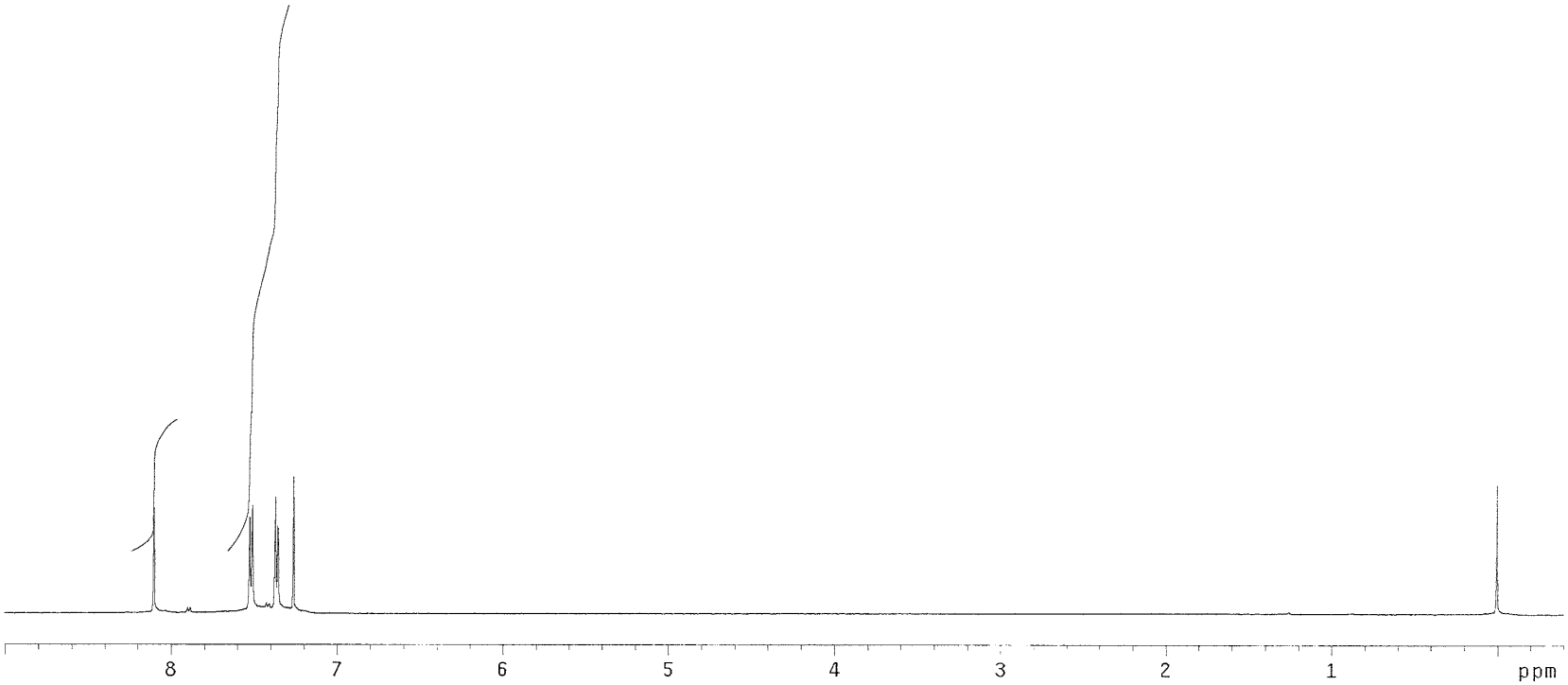


2b

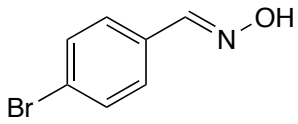
^{1}H NMR (500 MHz, $CDCl_3$):



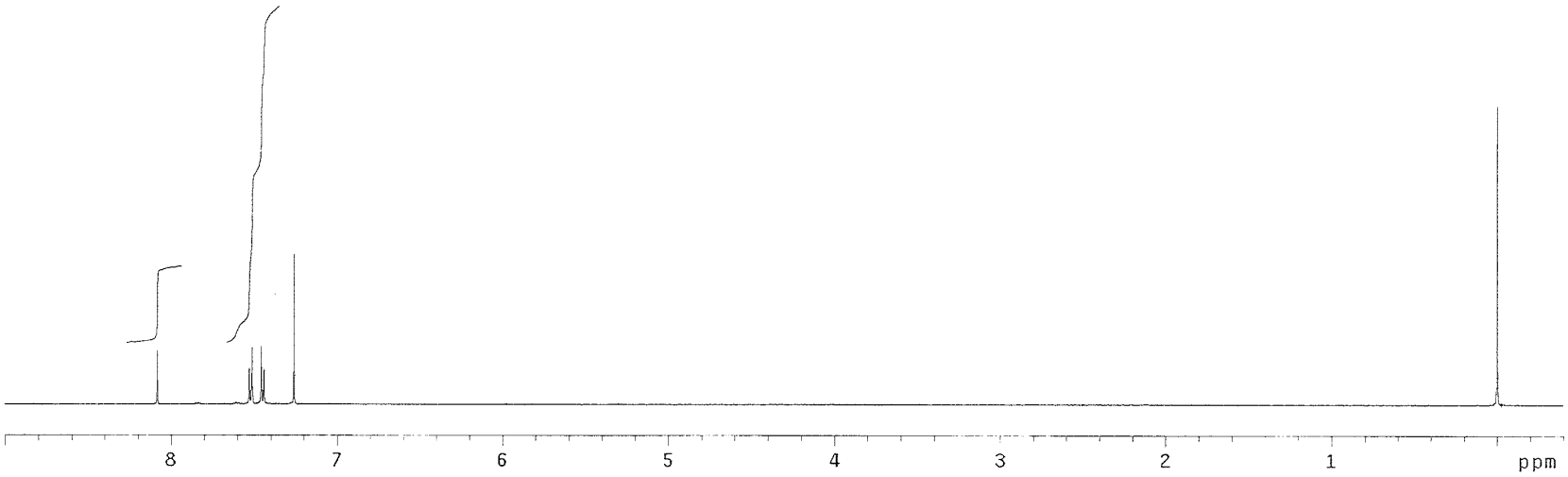
2c



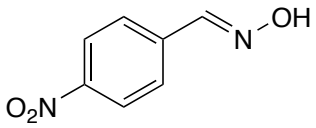
^{1}H NMR (500 MHz, $CDCl_3$):



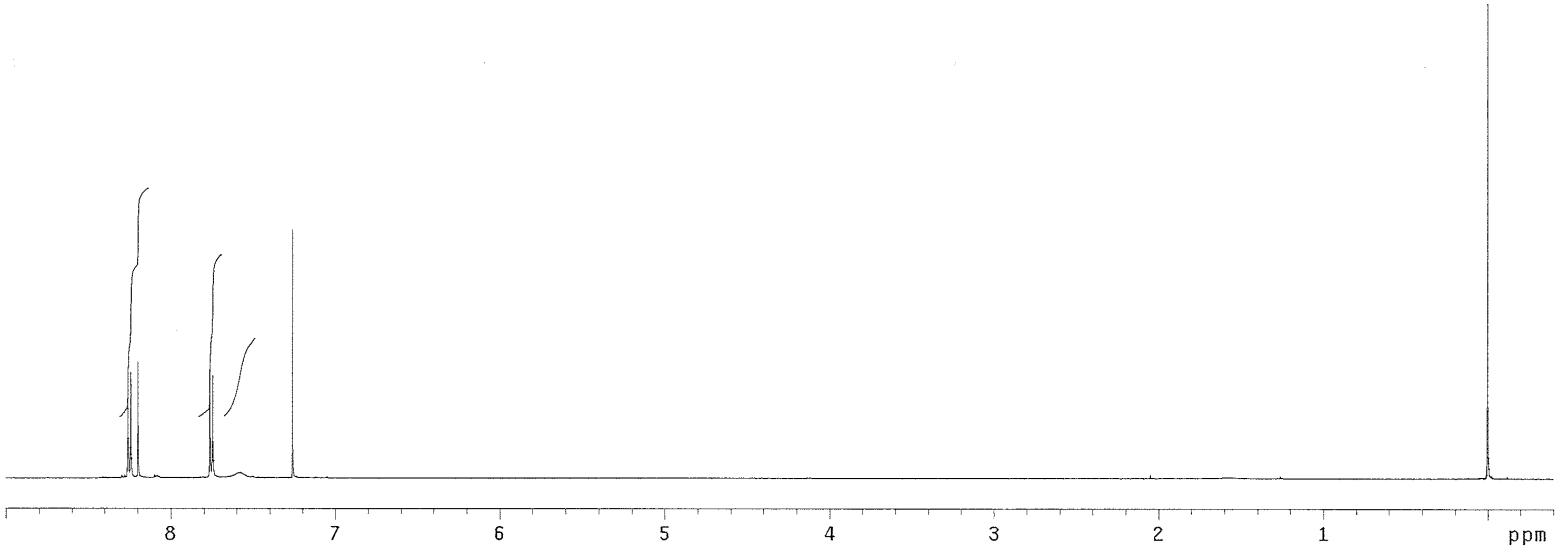
2d



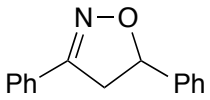
^{1}H NMR (500 MHz, $CDCl_3$):



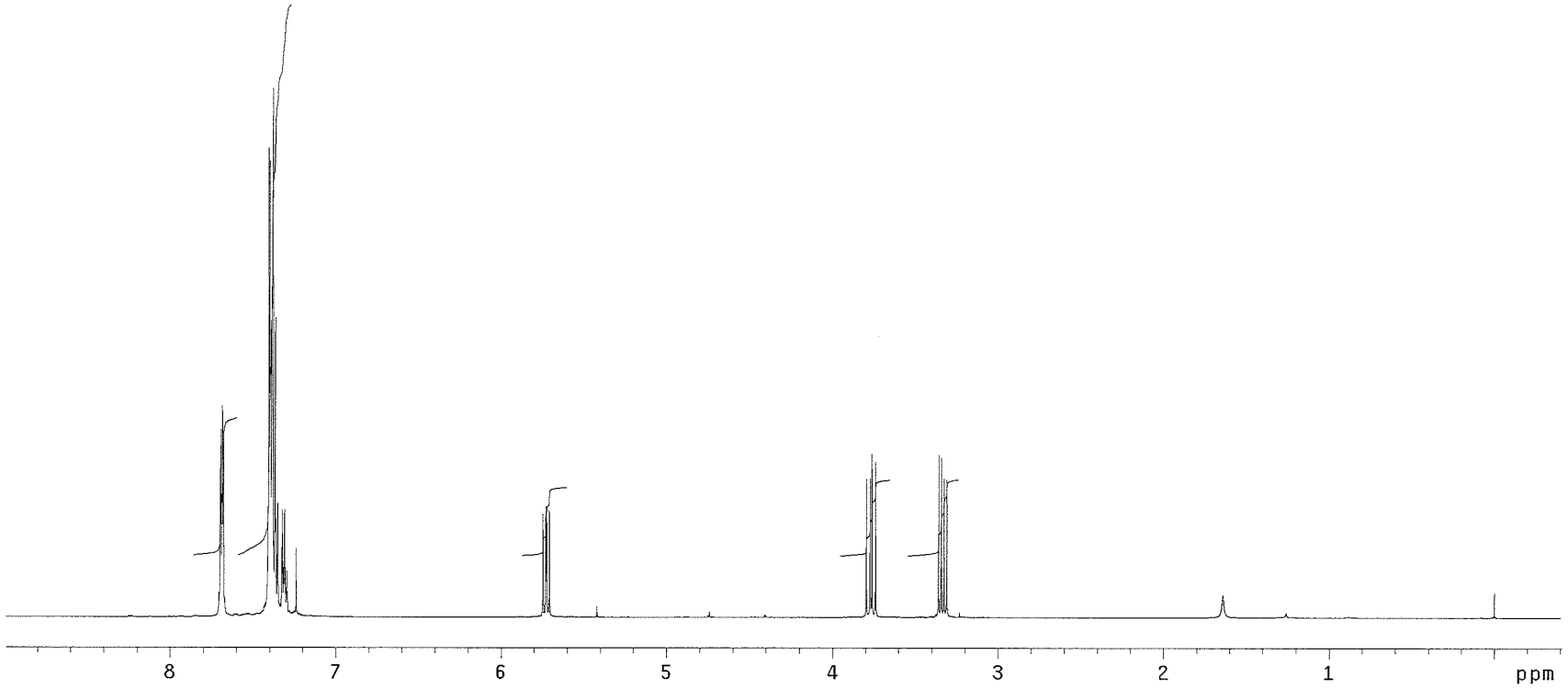
2e



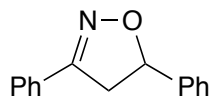
^{1}H NMR (500 MHz, $CDCl_3$):



3a



^{13}C NMR (125 MHz, $CDCl_3$):



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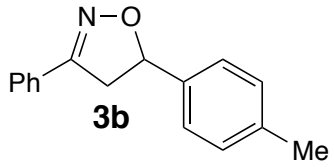
^{1}H NMR (500 MHz, $CDCl_3$):



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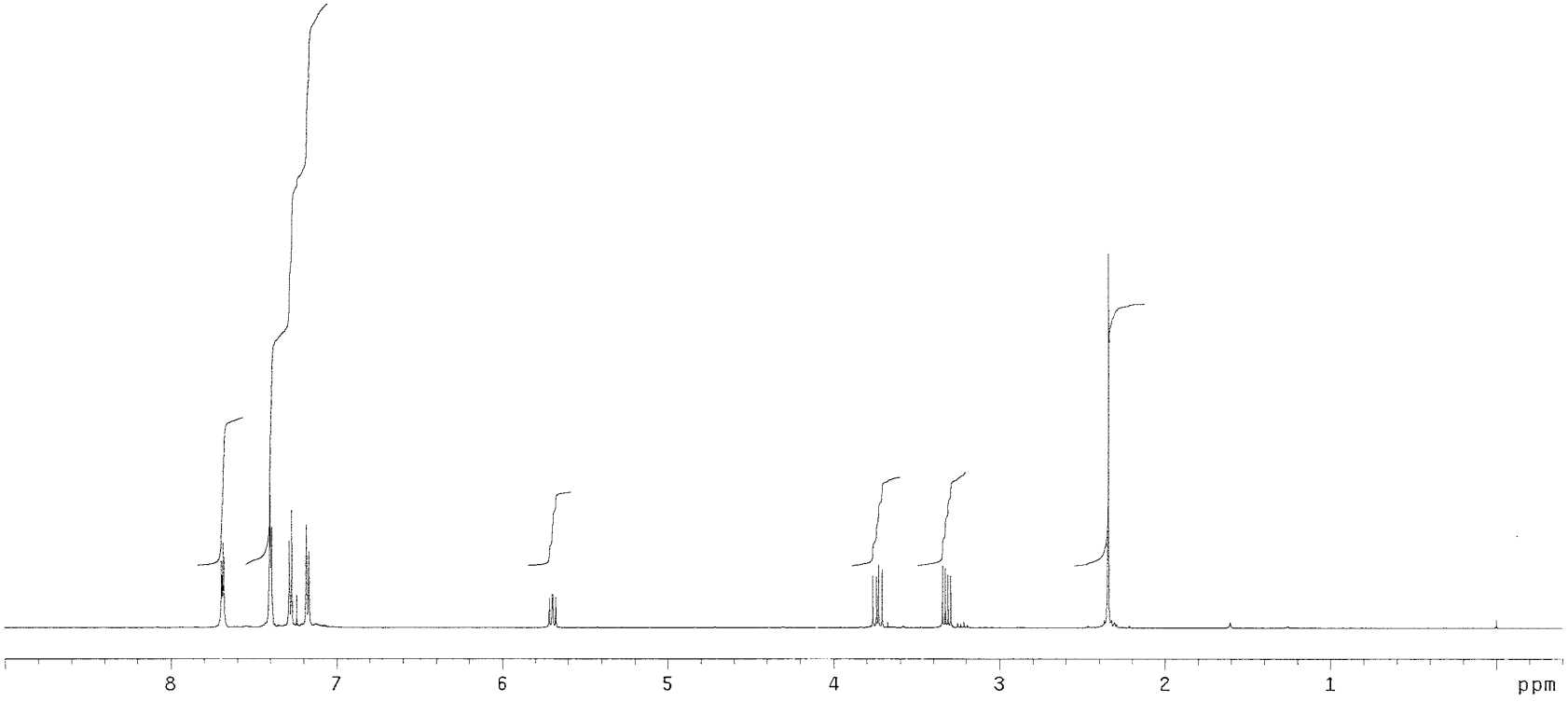
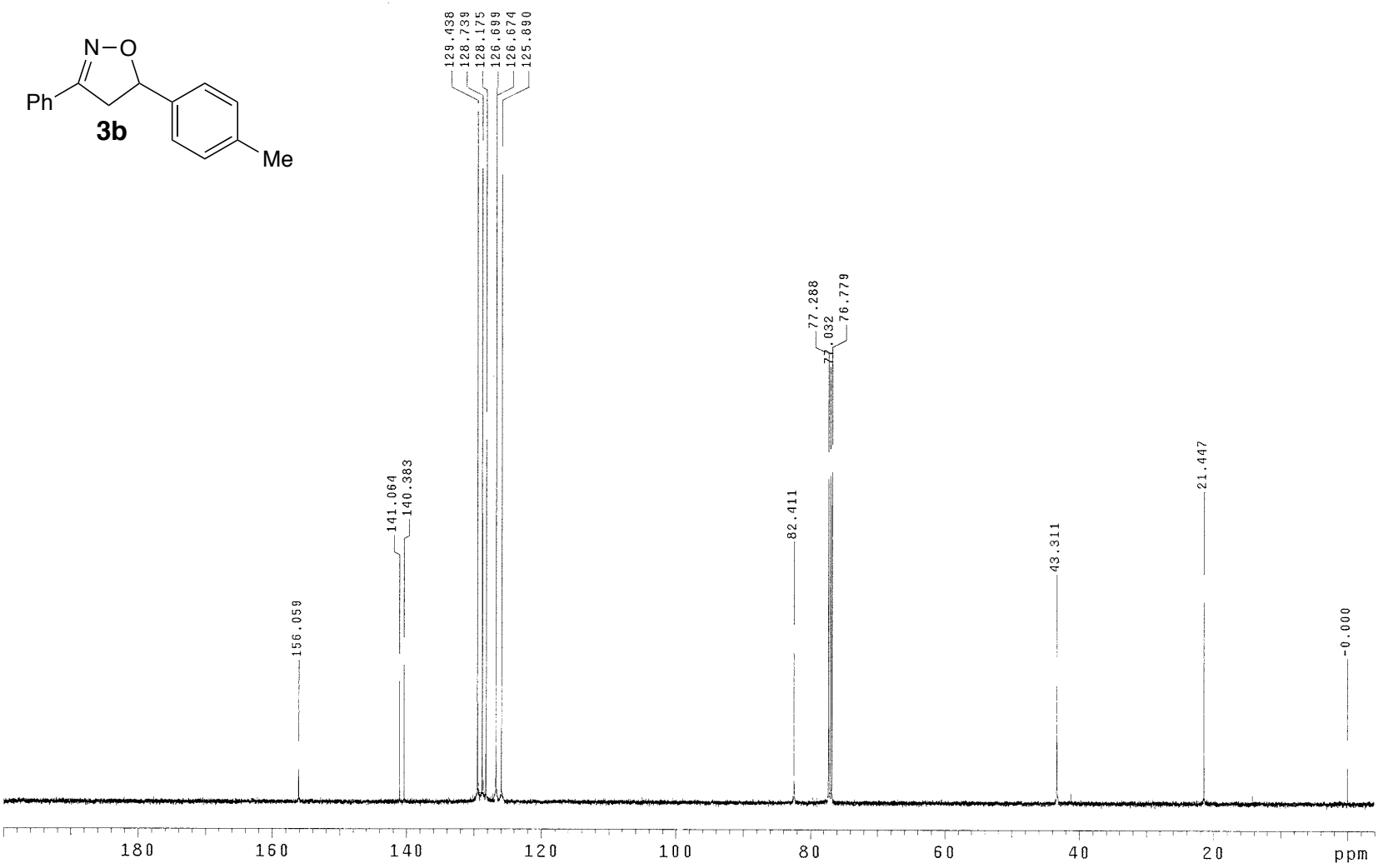


^{13}C NMR (125 MHz, $CDCl_3$):



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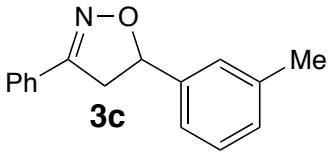

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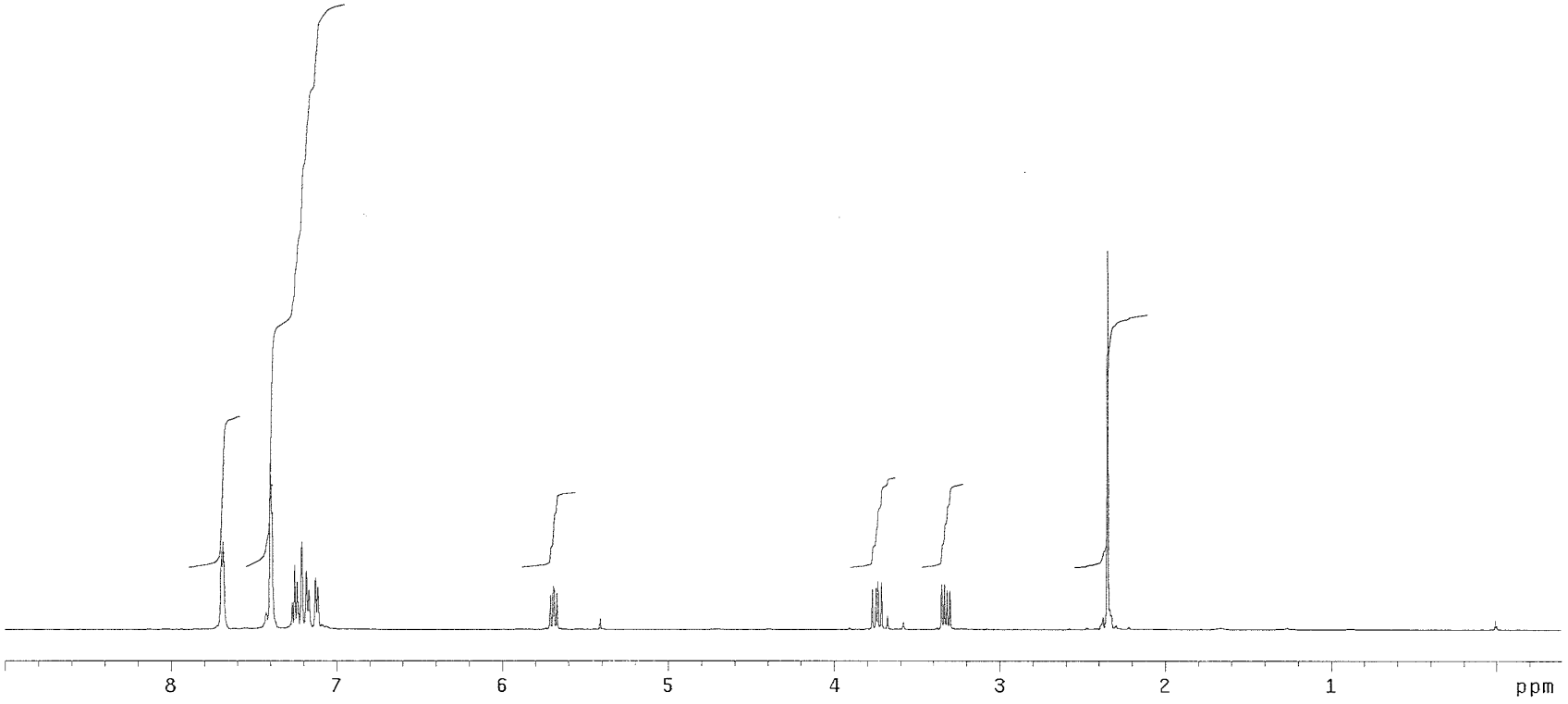

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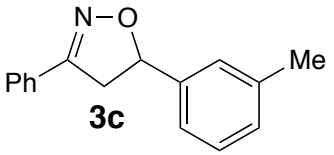

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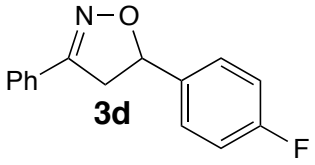

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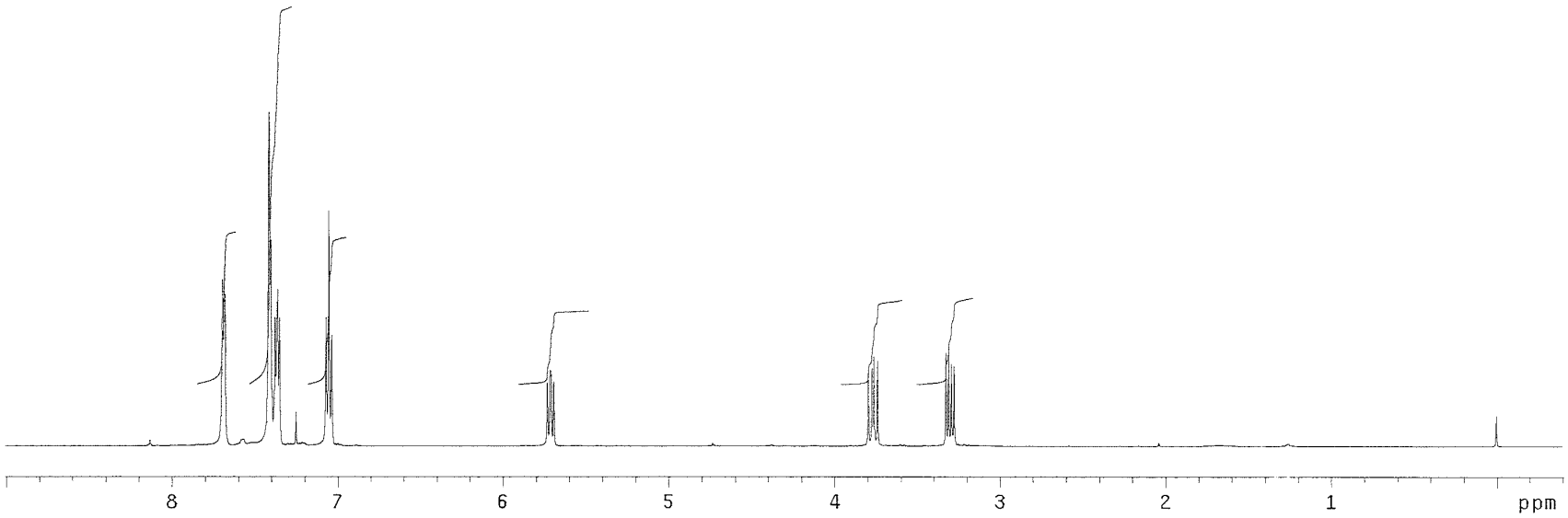

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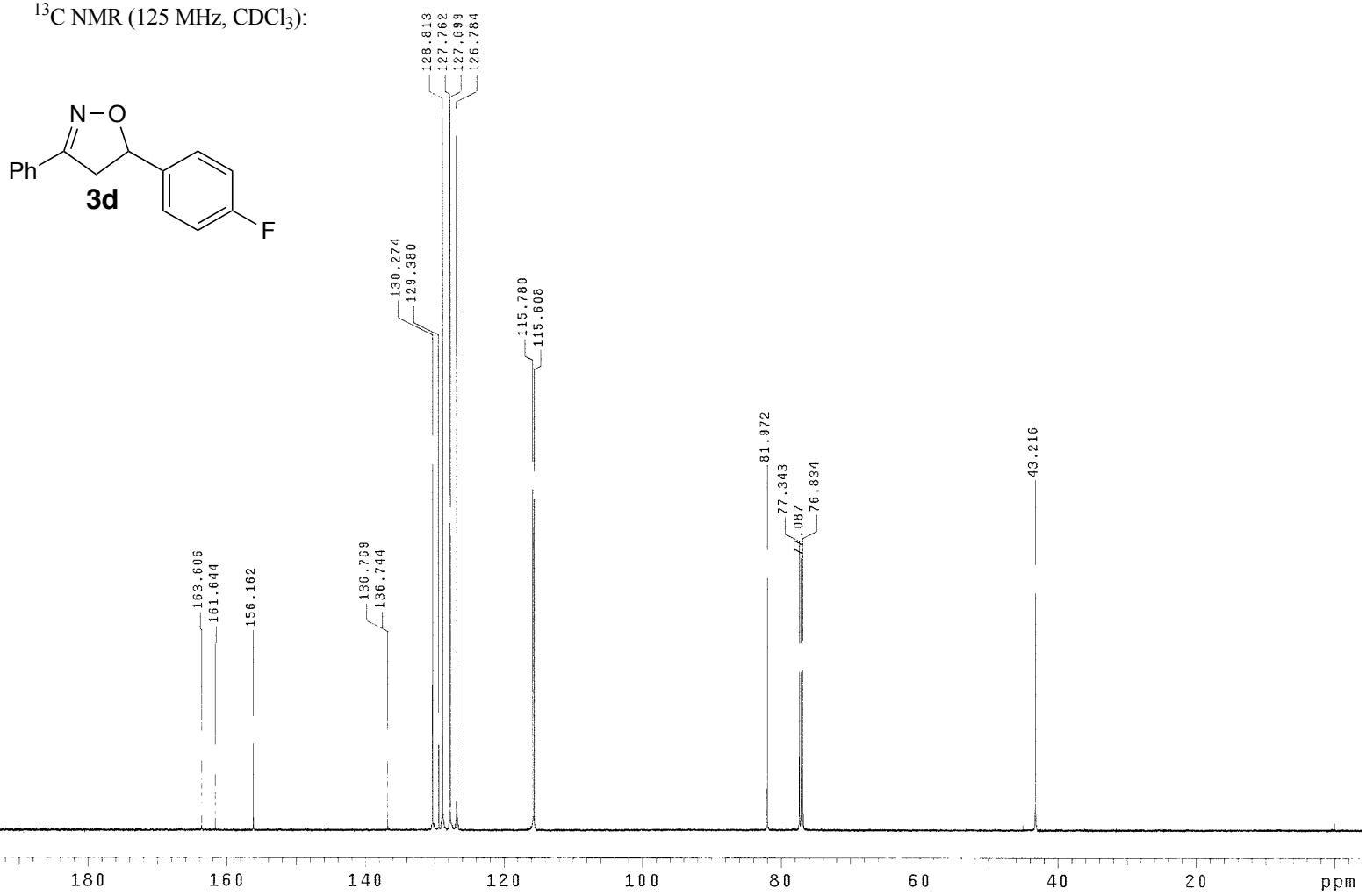

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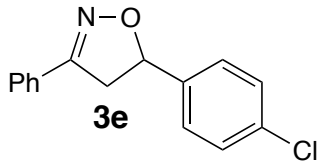

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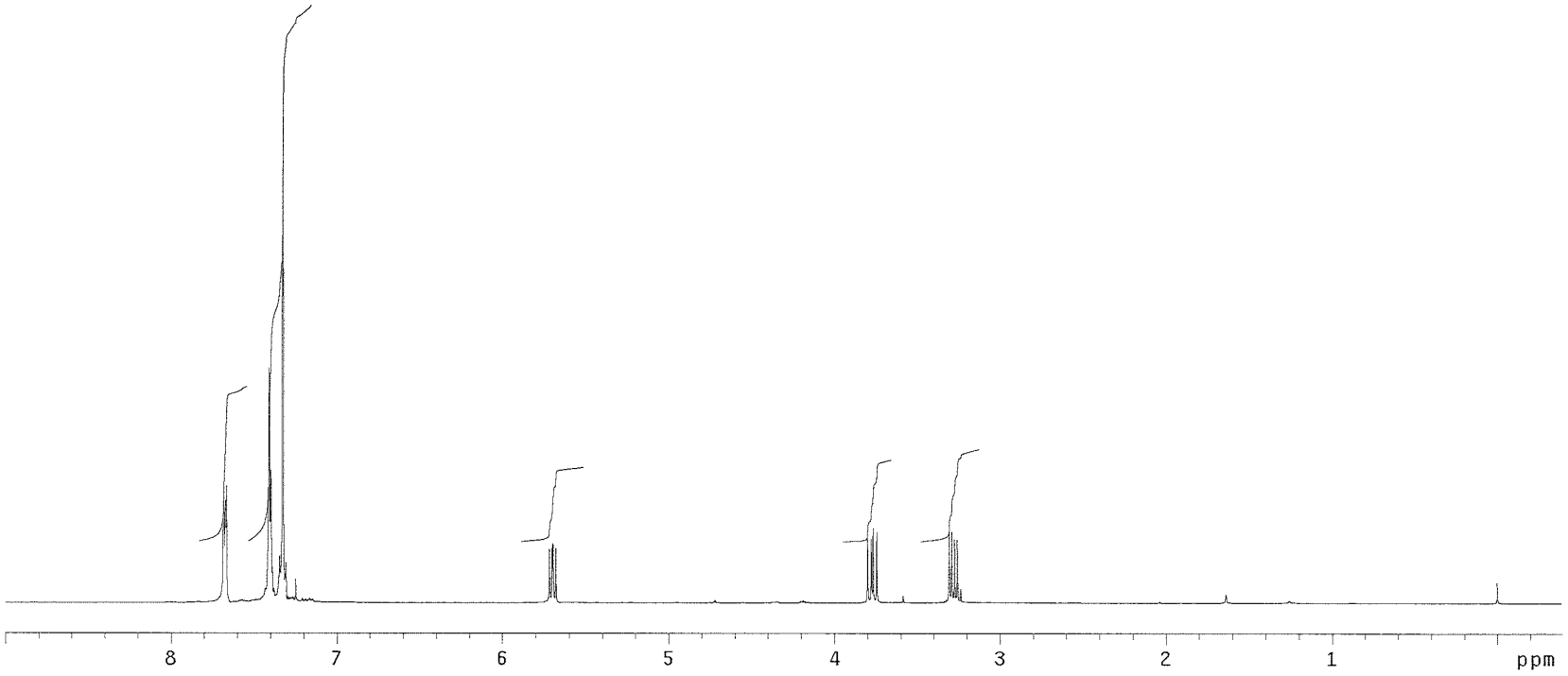

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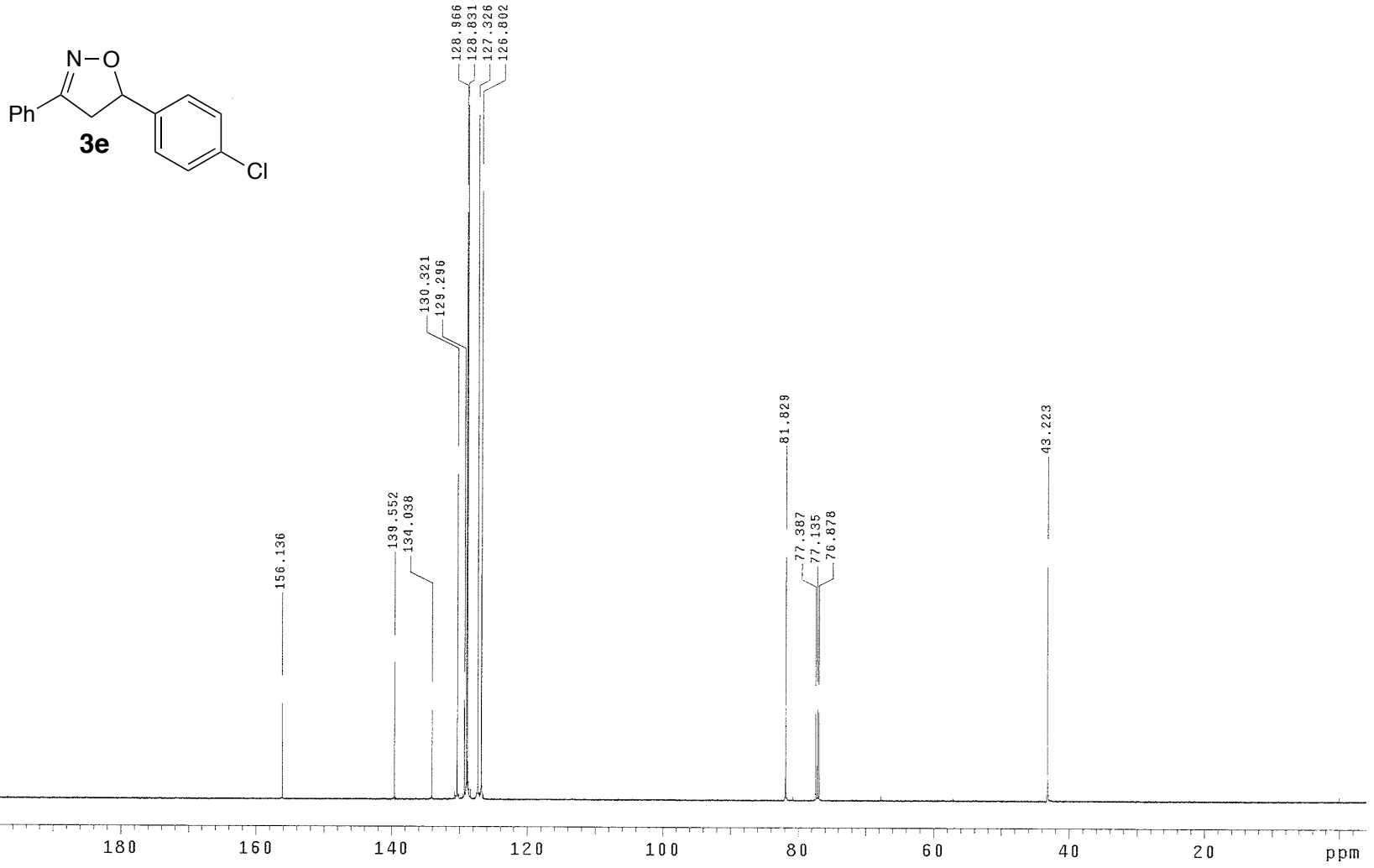

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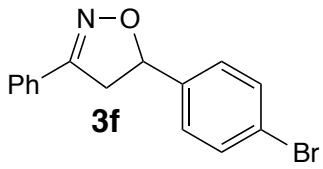

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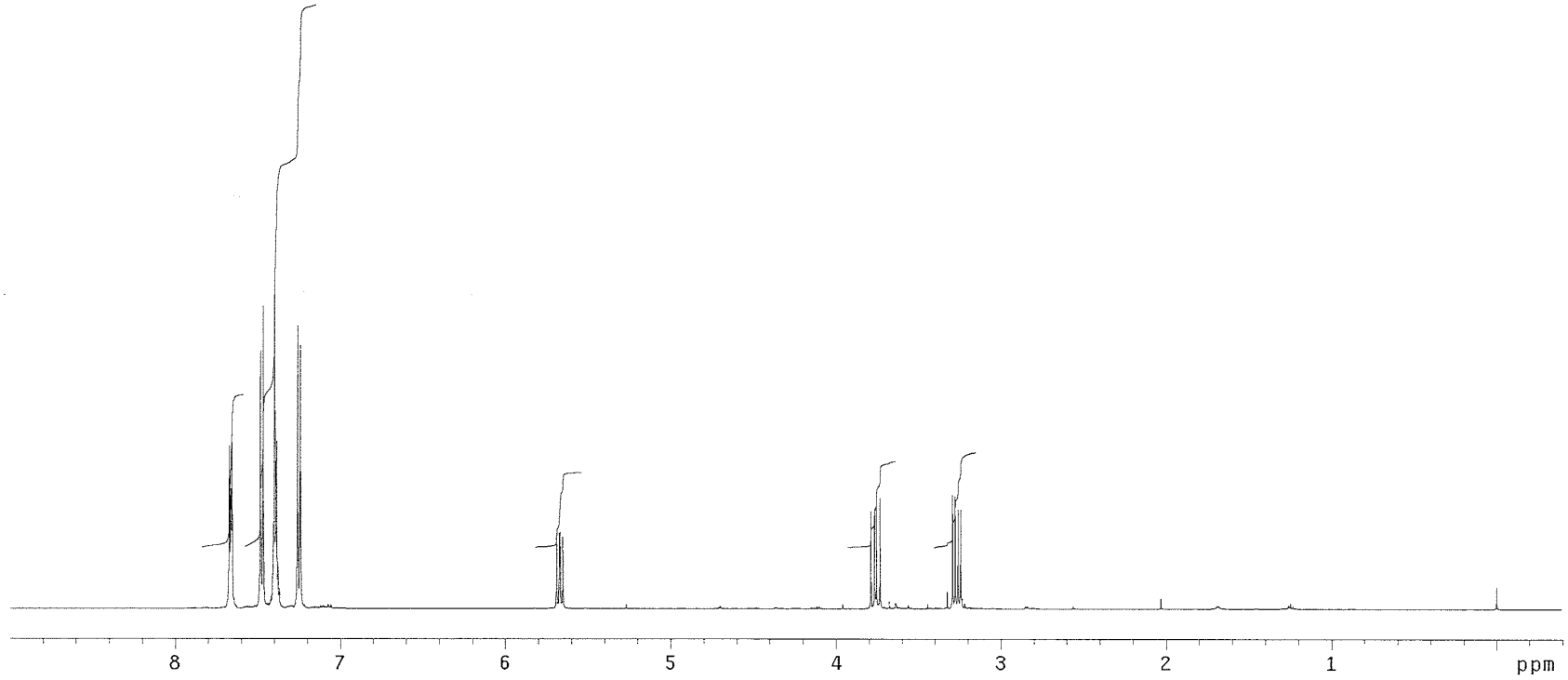

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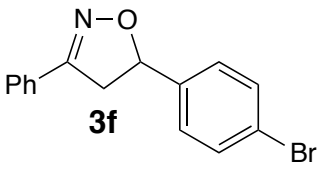

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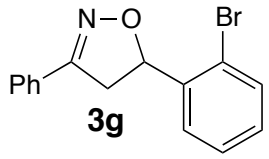

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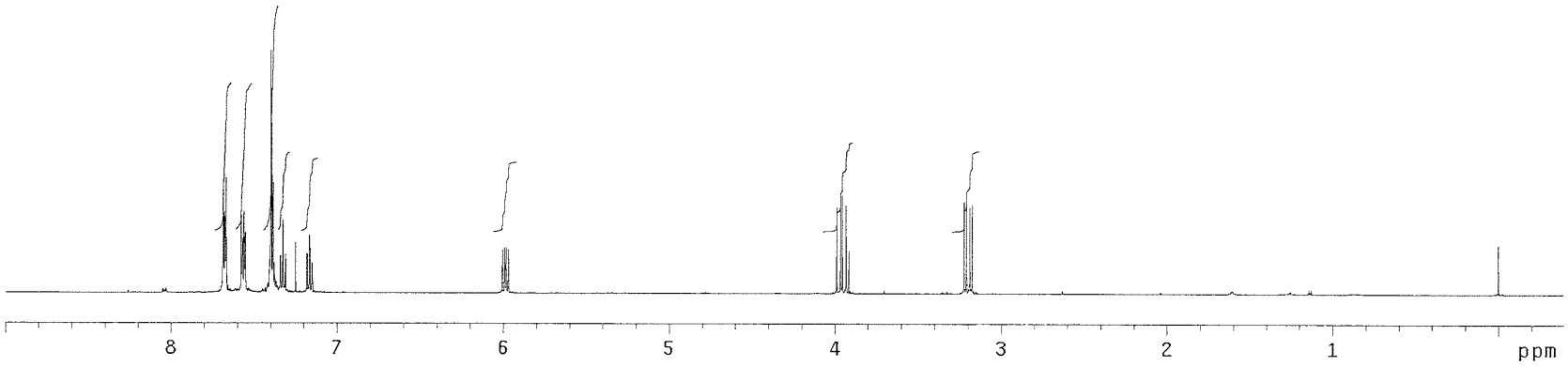

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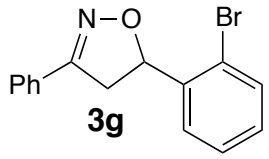

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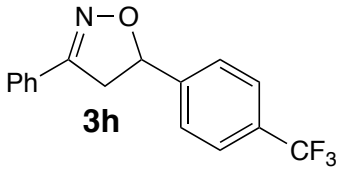

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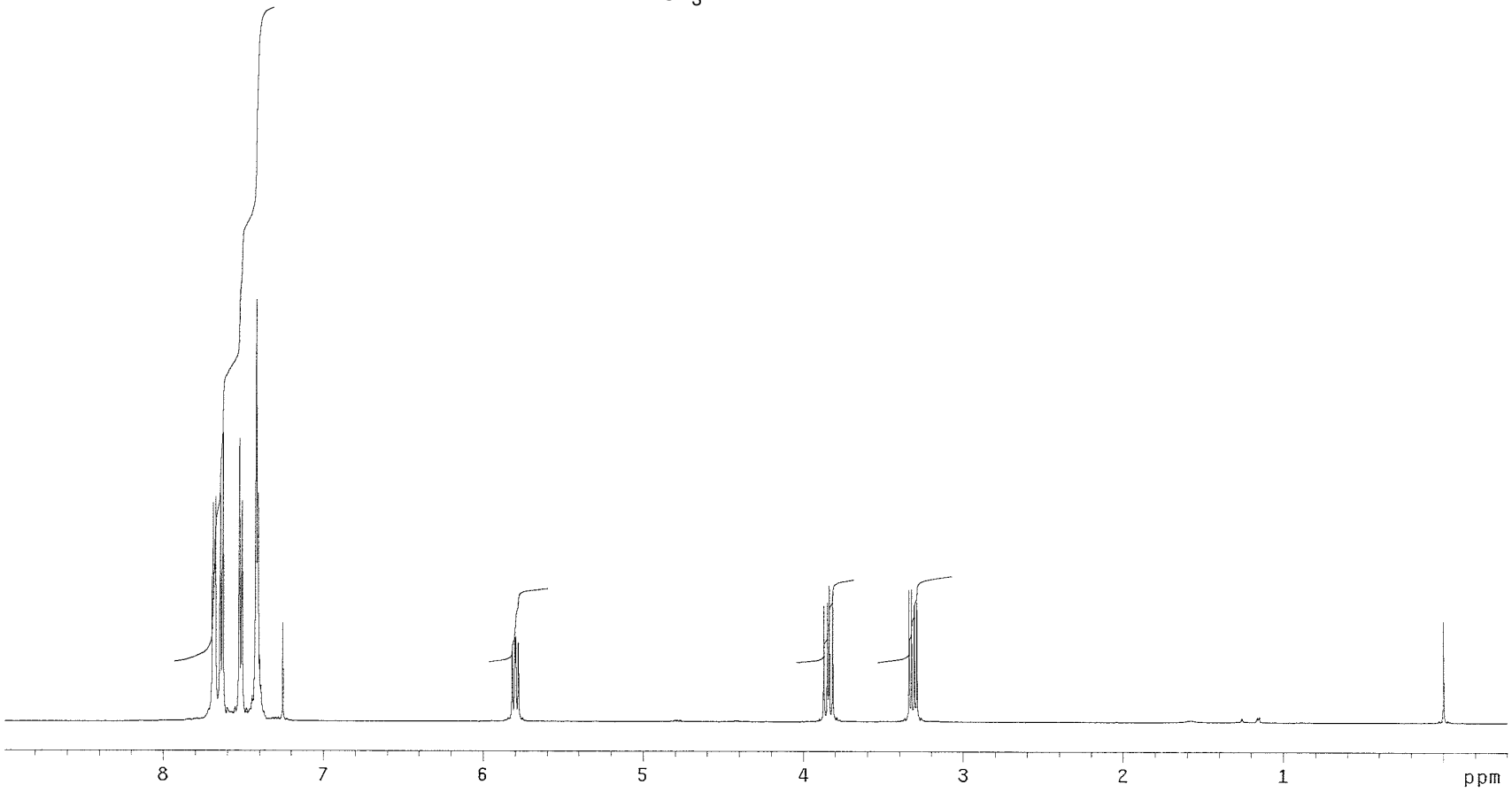

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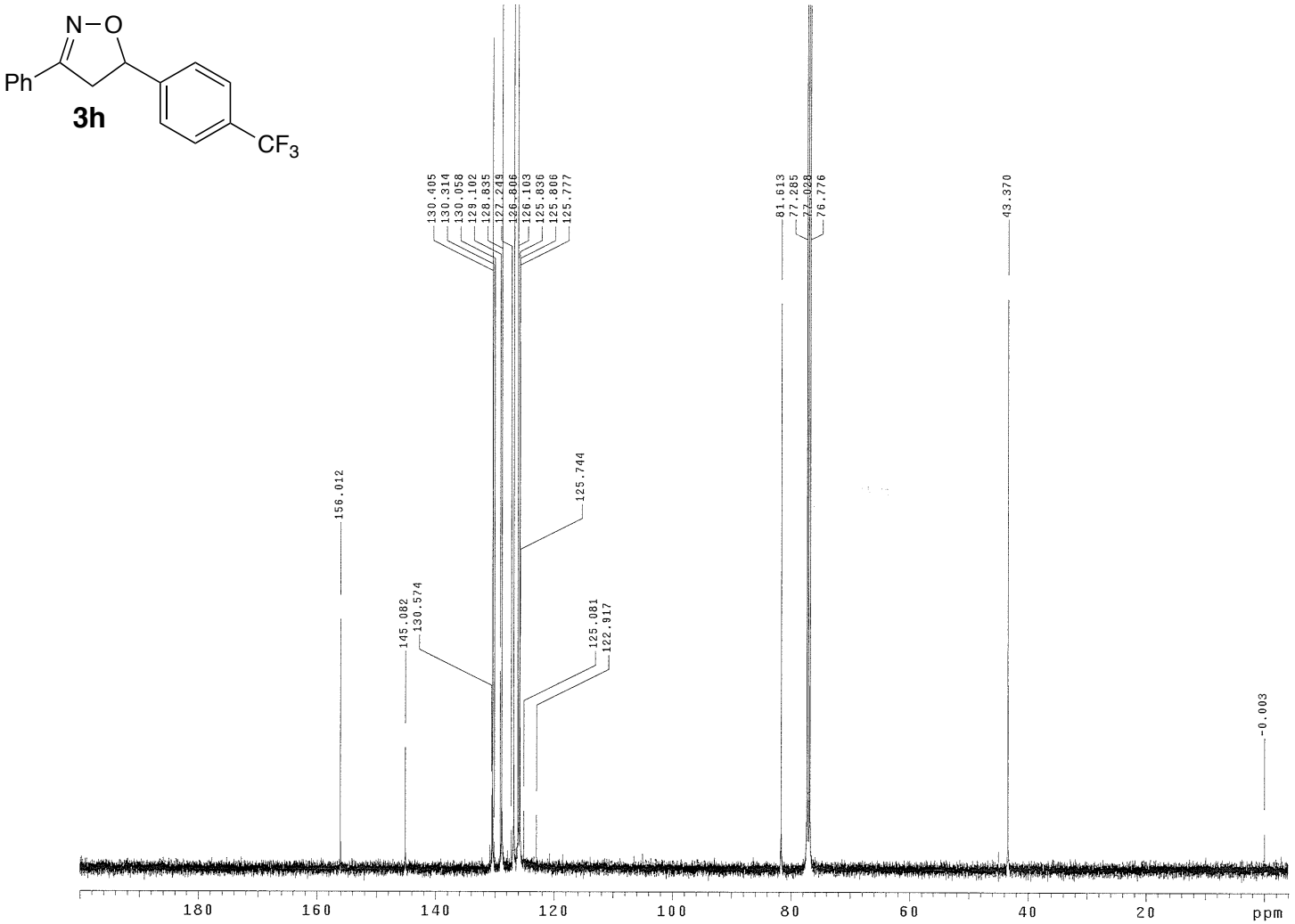

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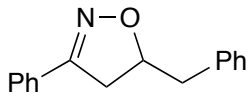

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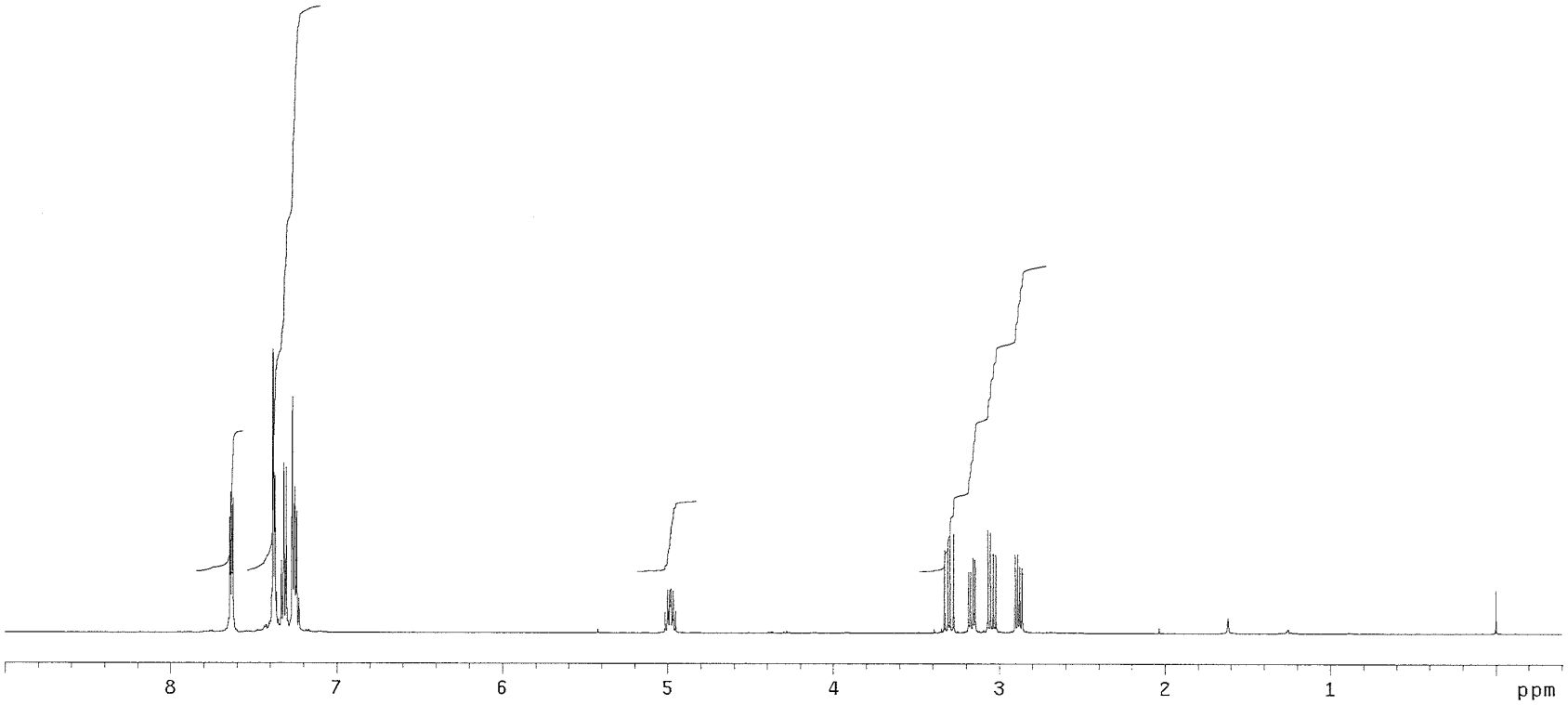

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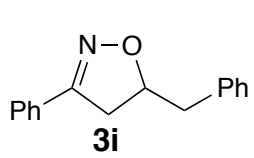

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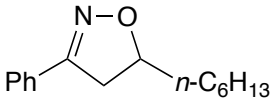

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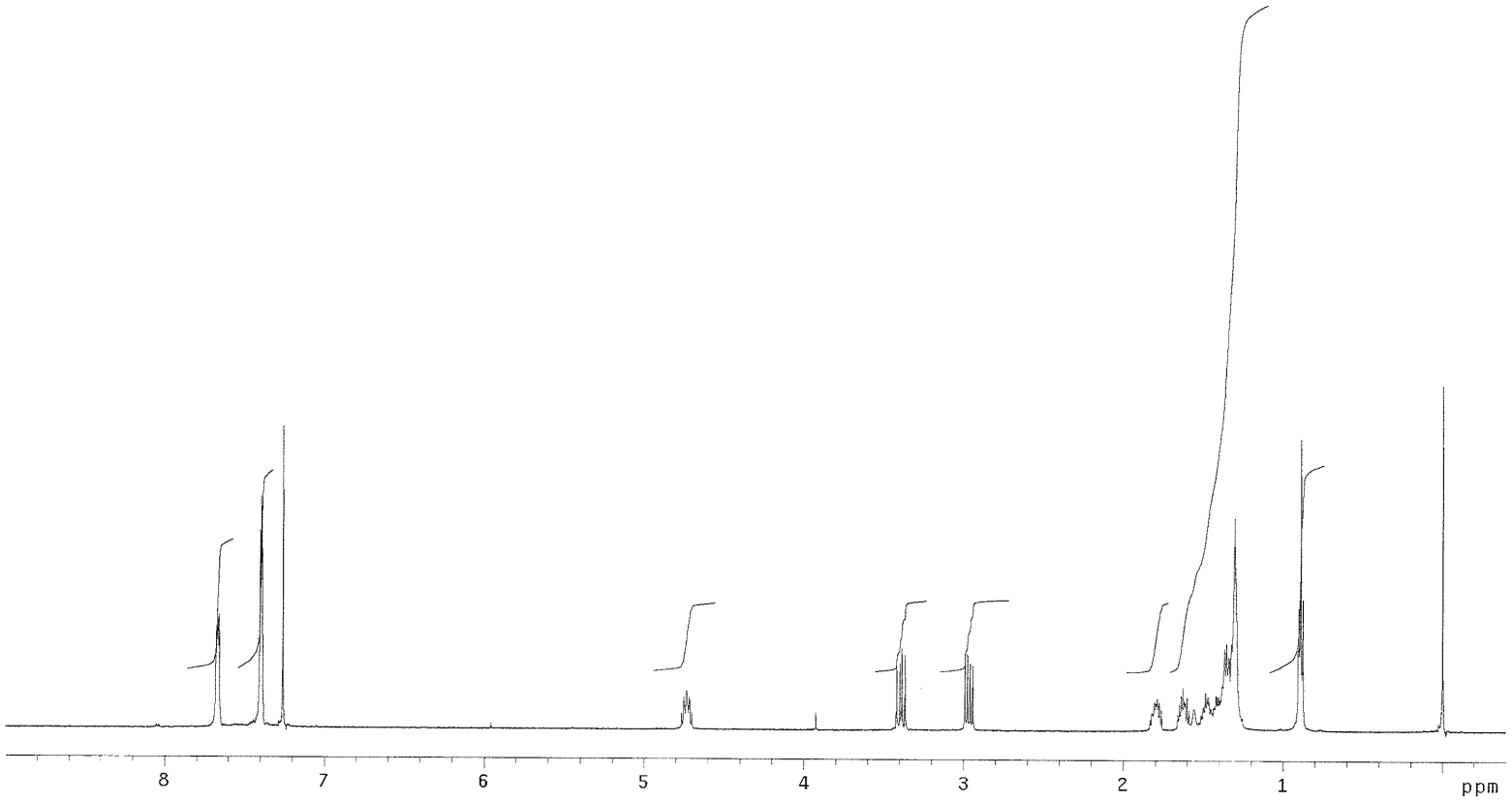

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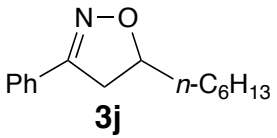

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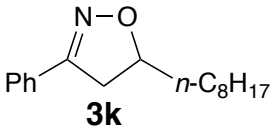

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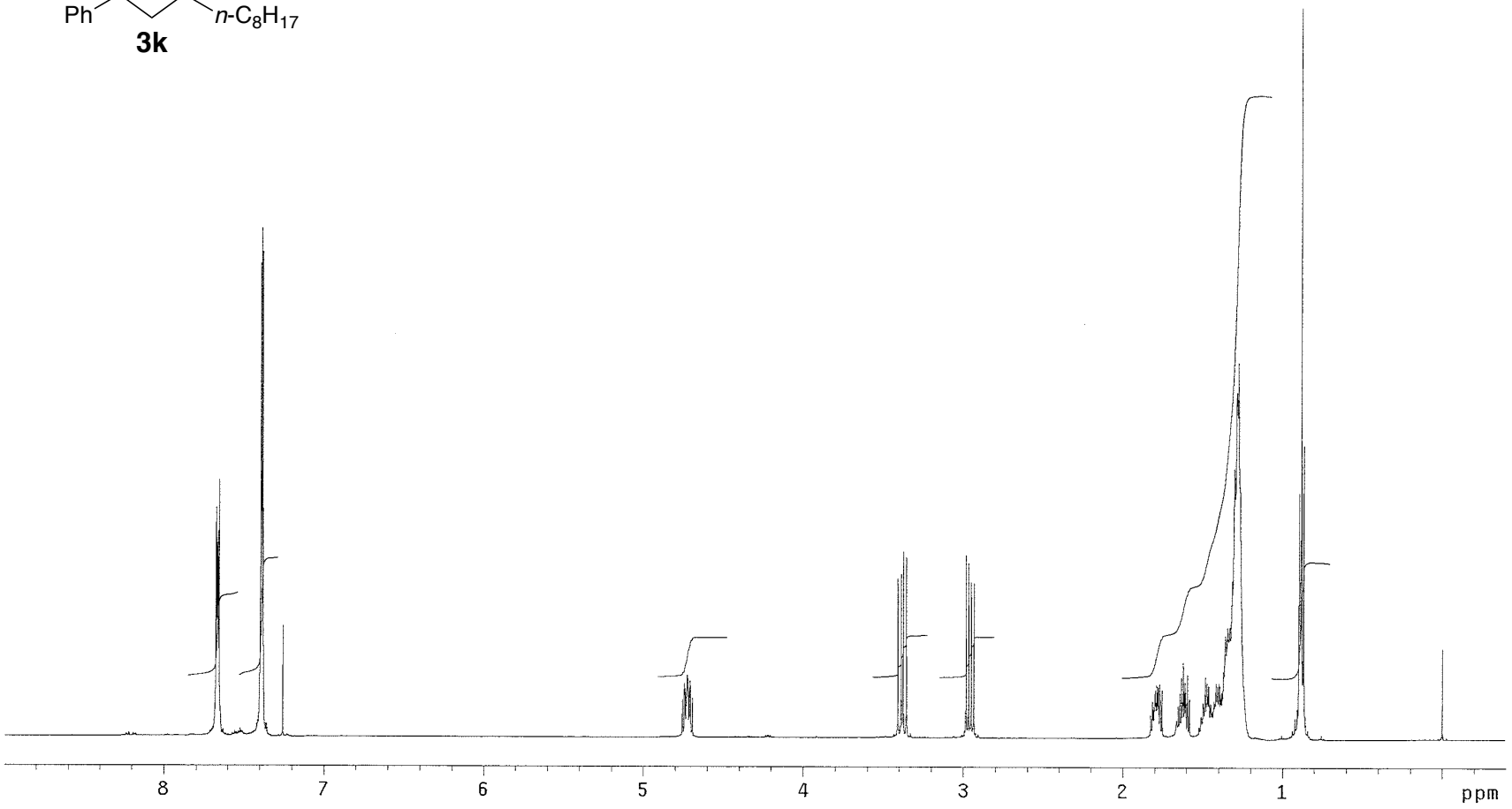

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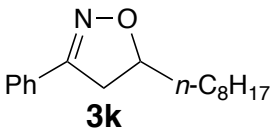

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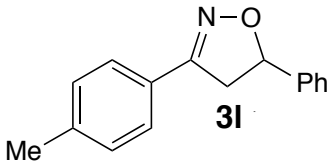

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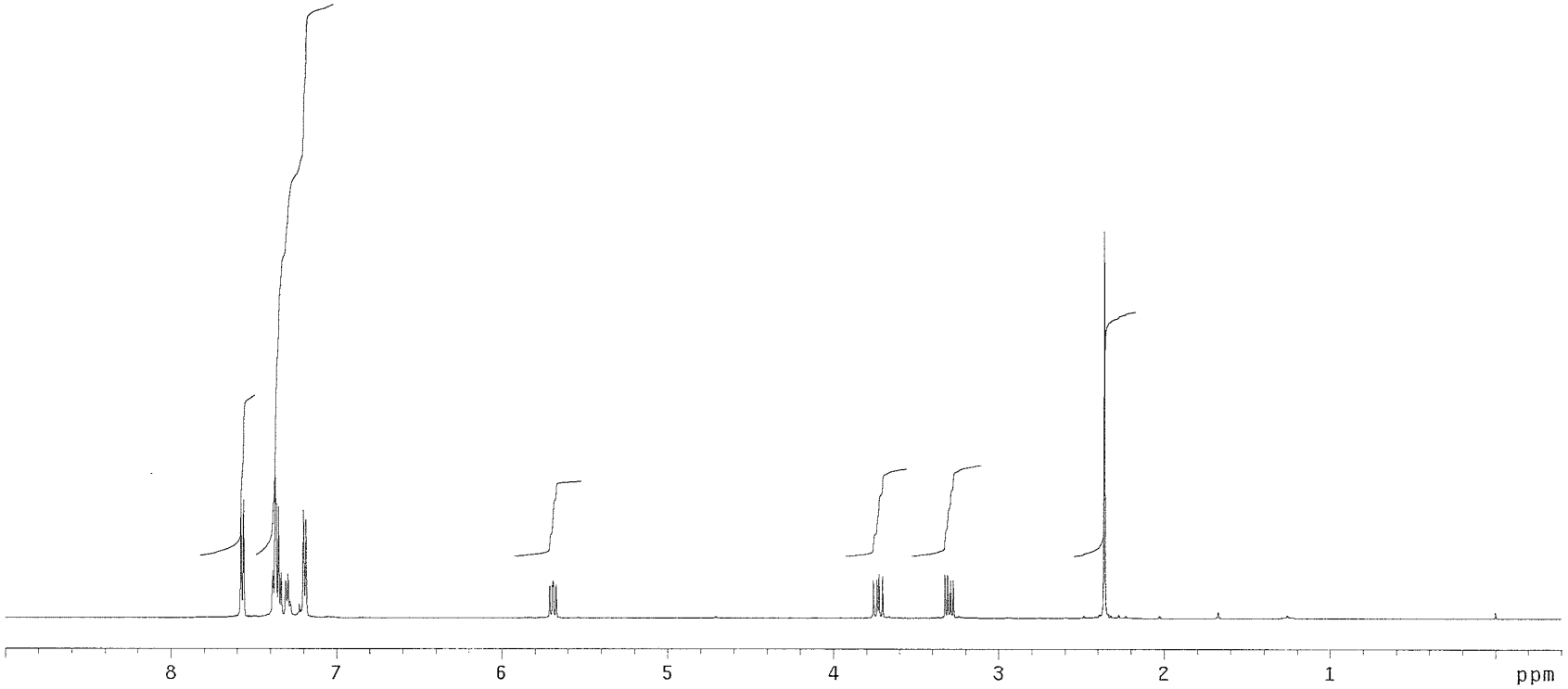

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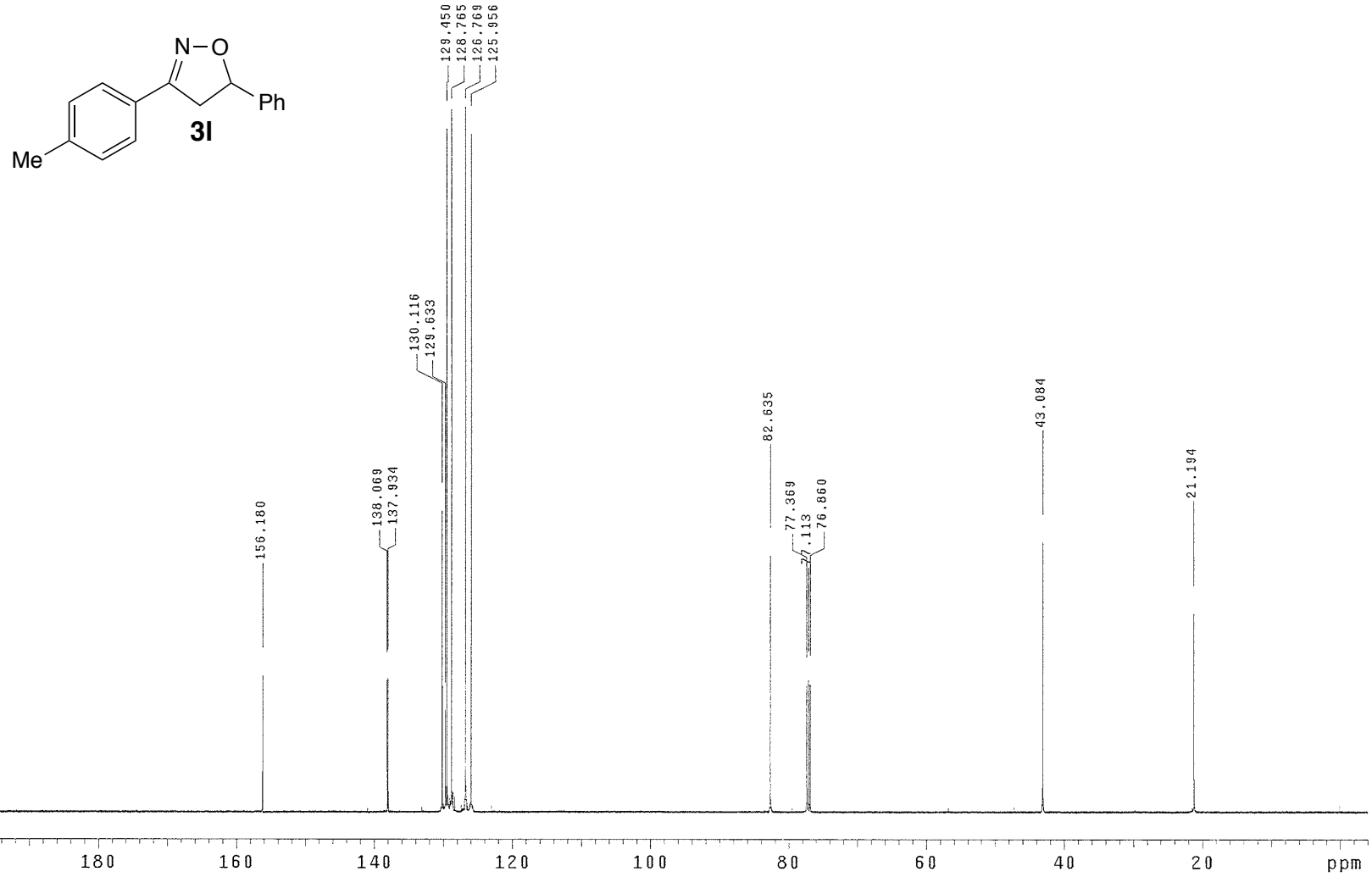

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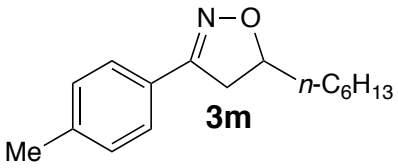

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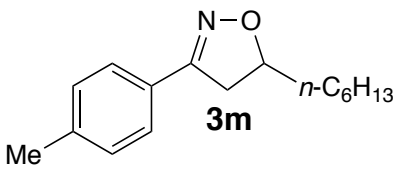

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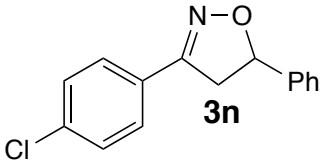

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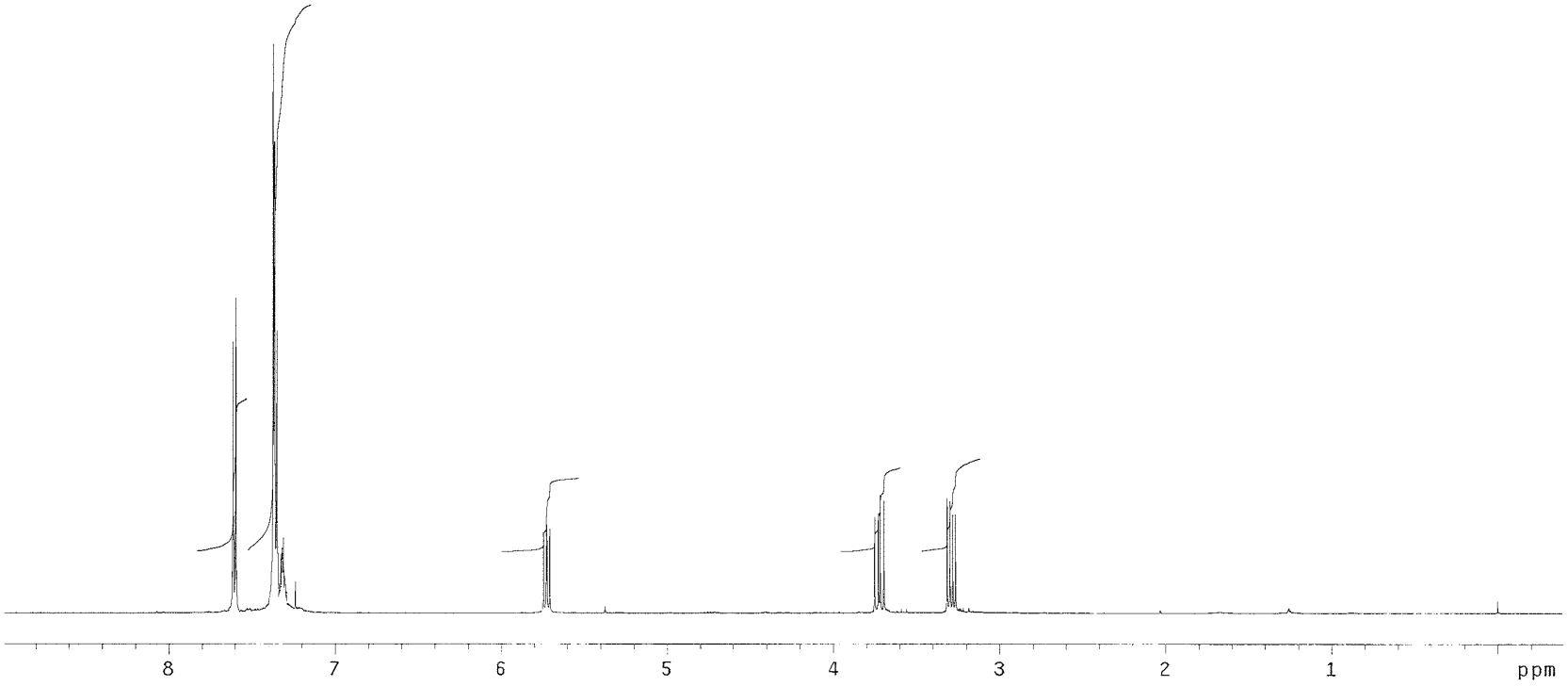

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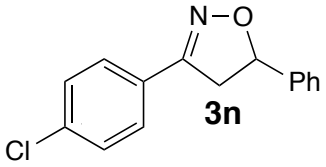

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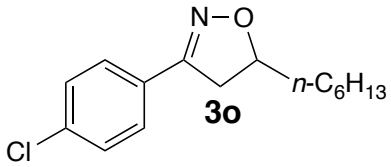

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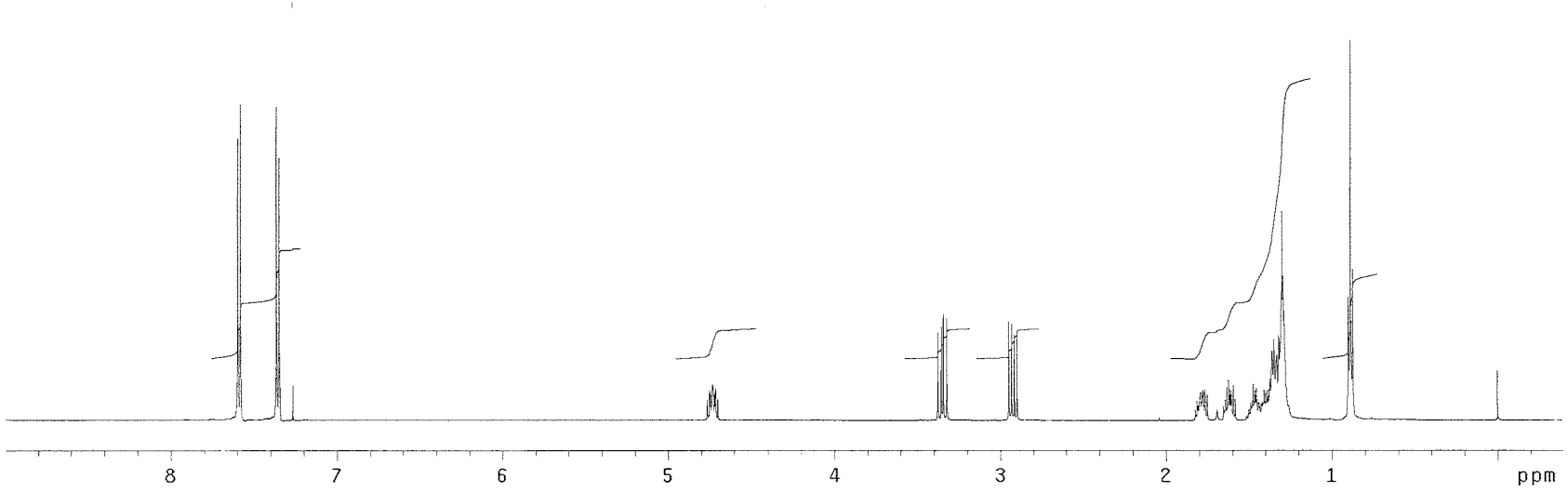

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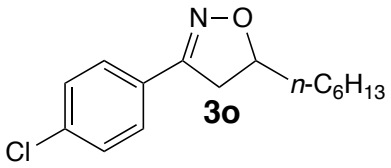

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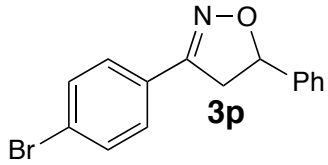

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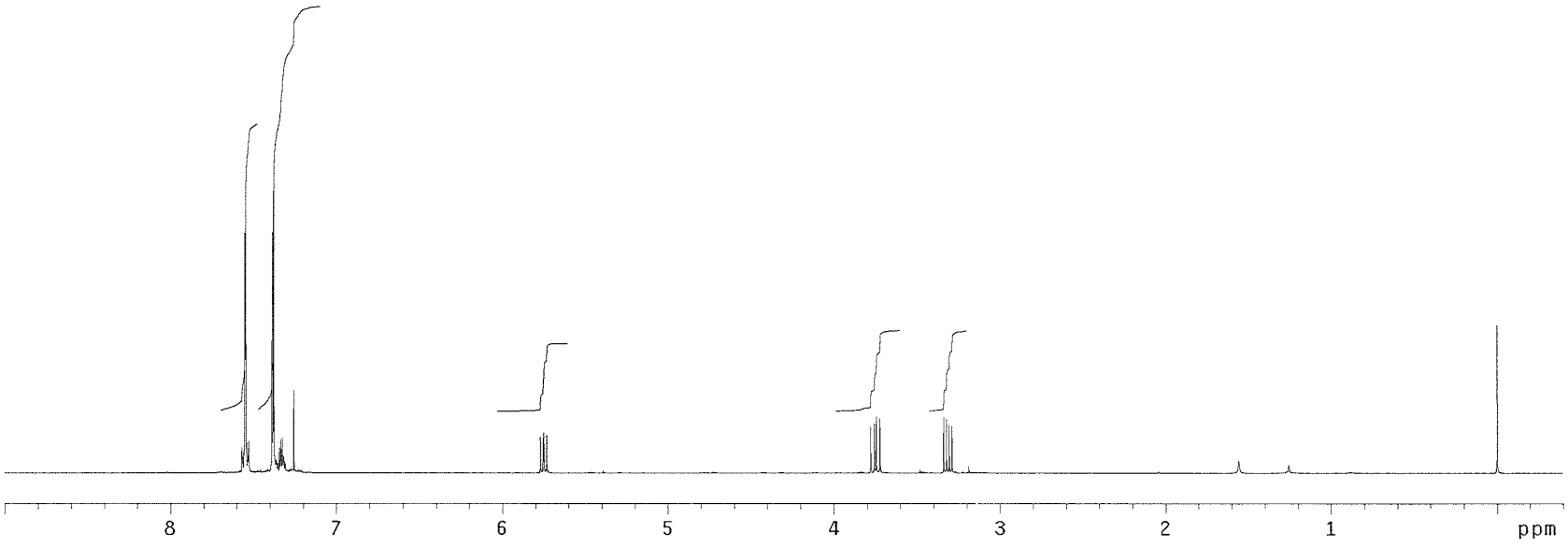

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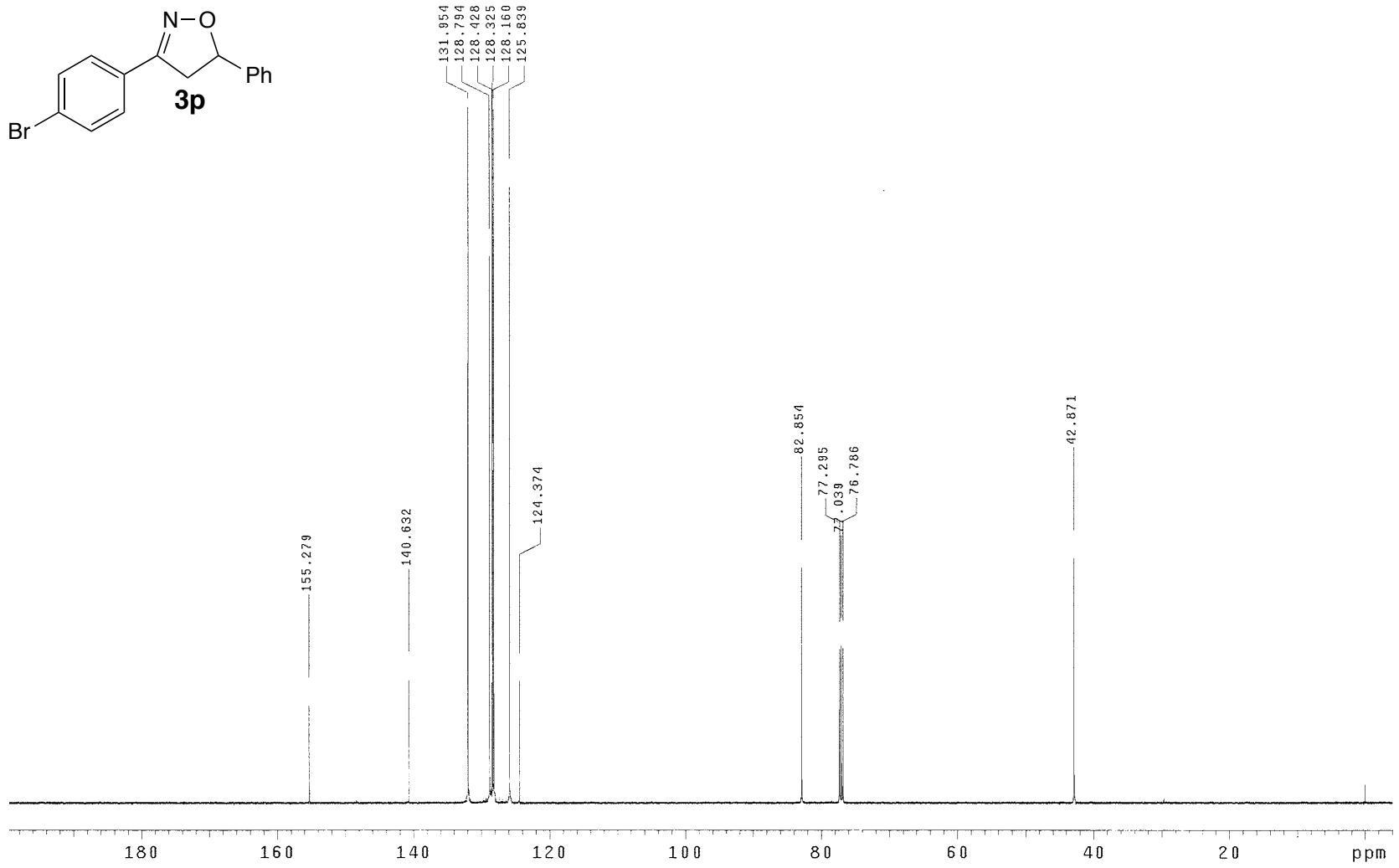

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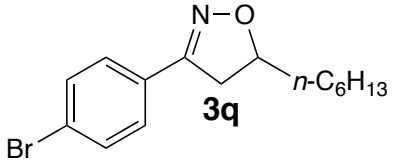

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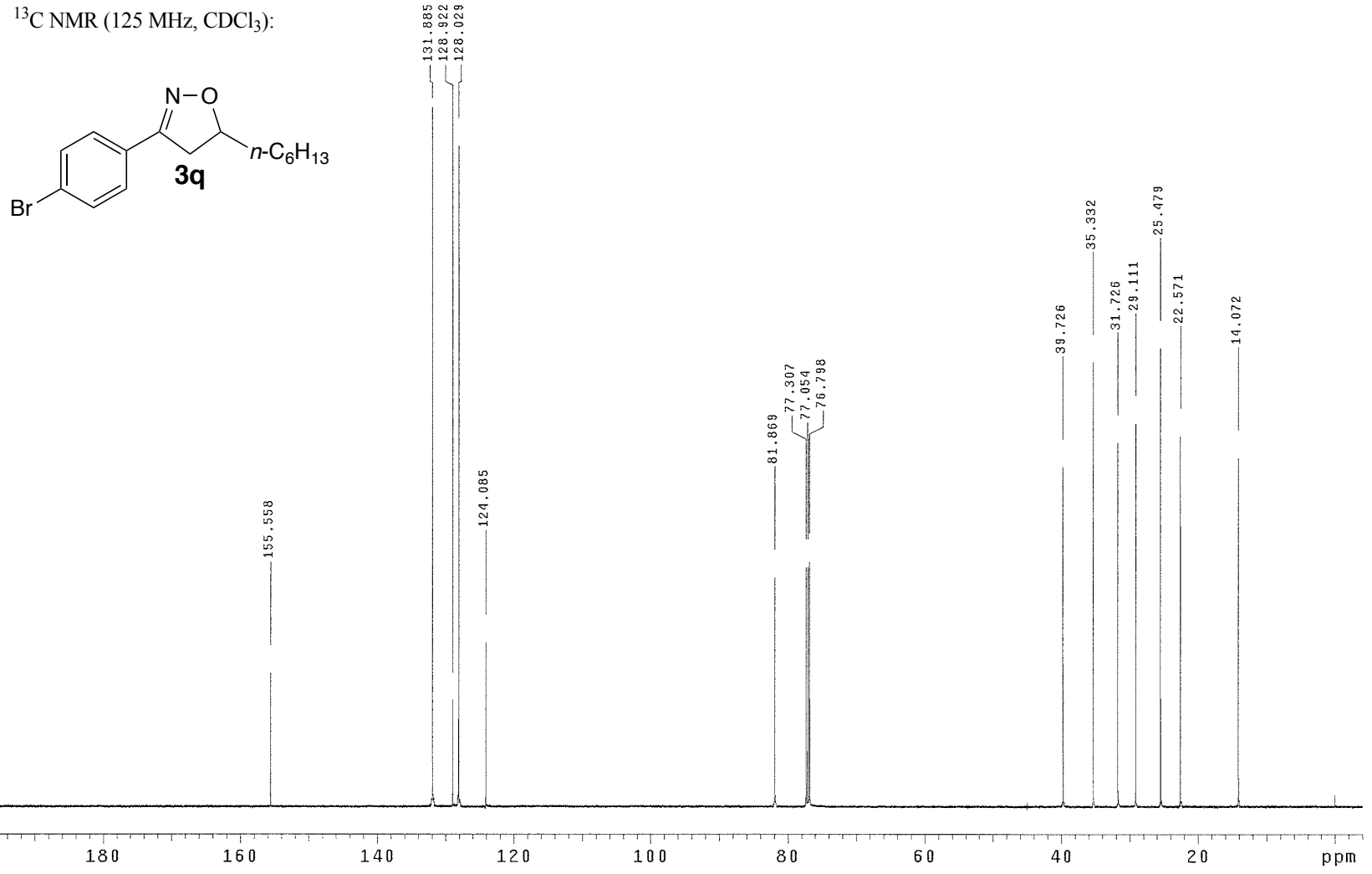

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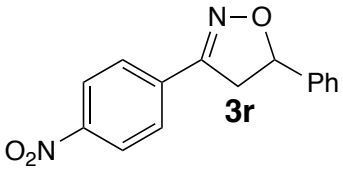

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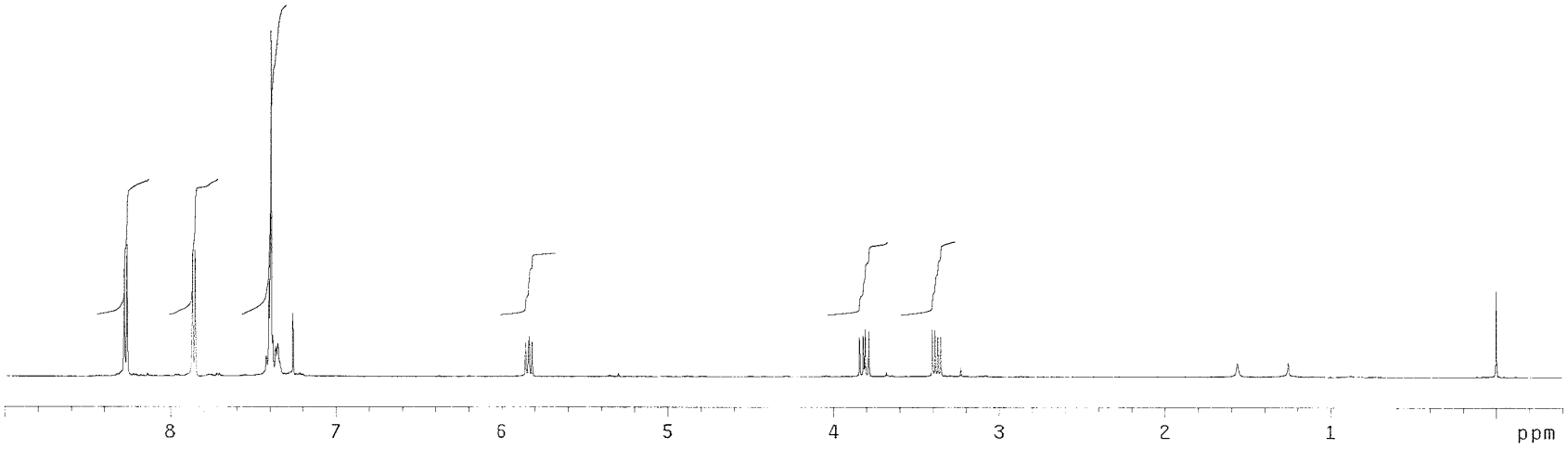

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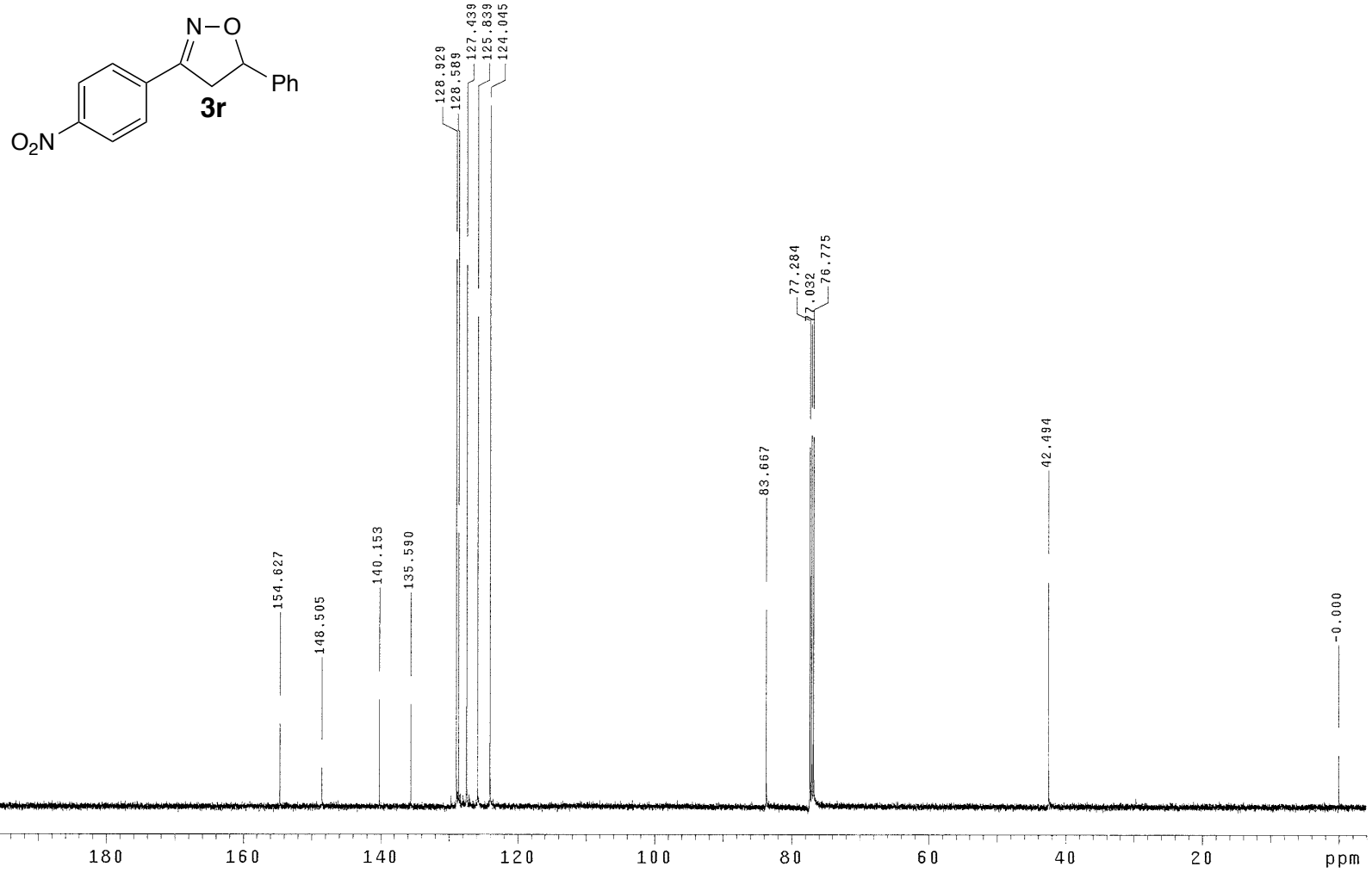

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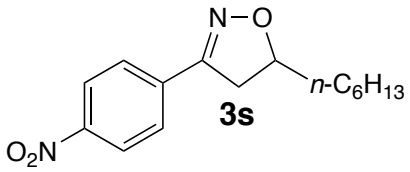

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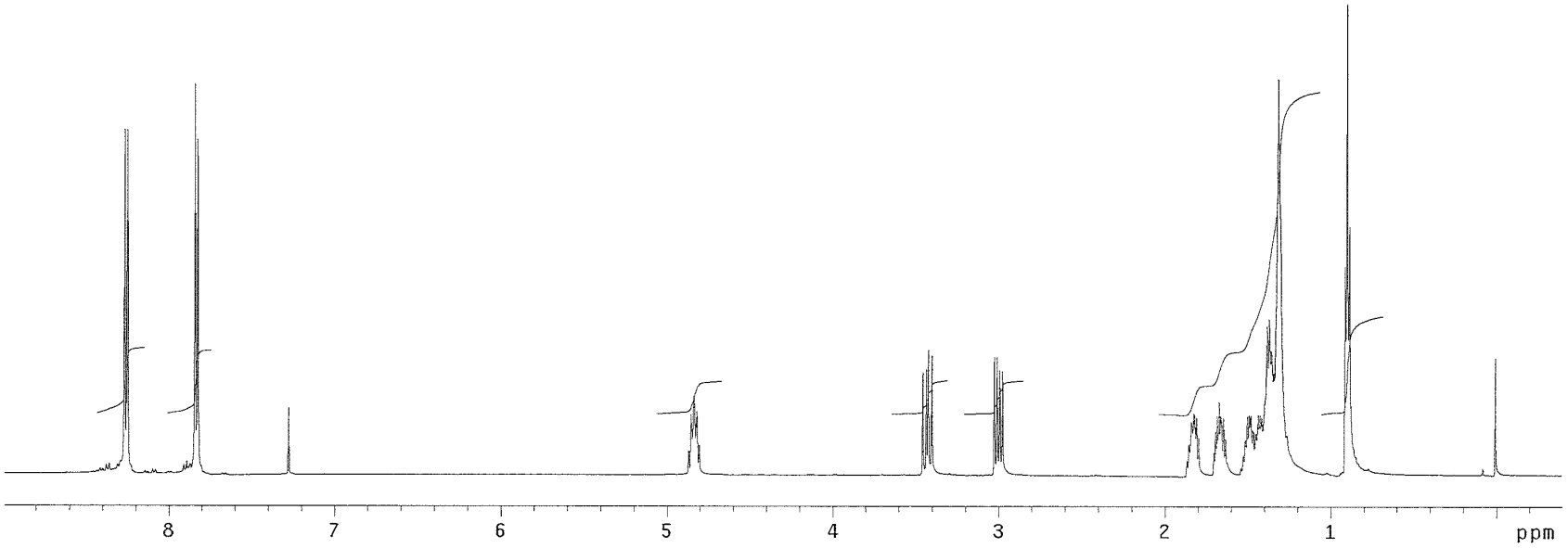

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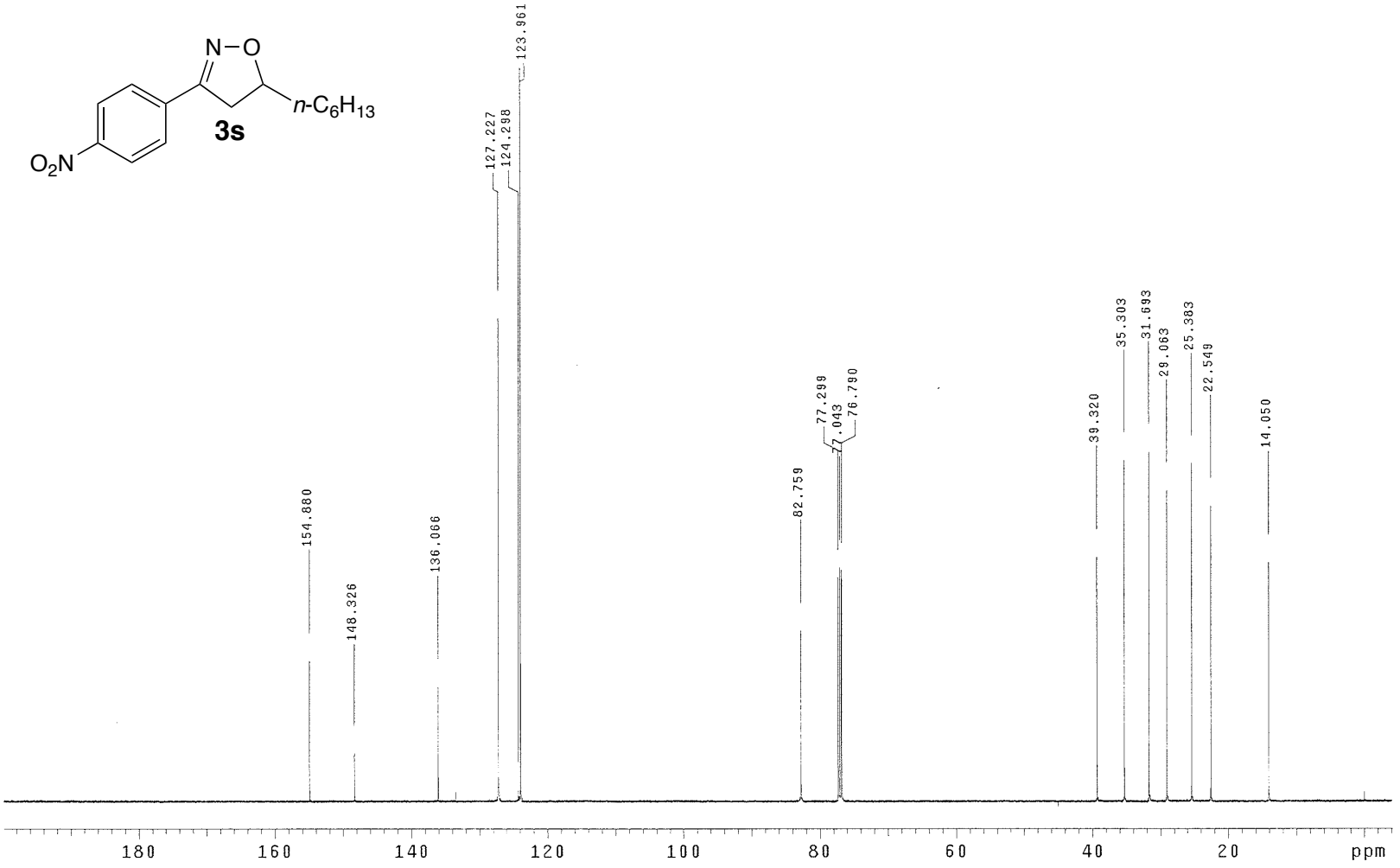

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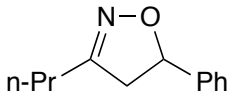
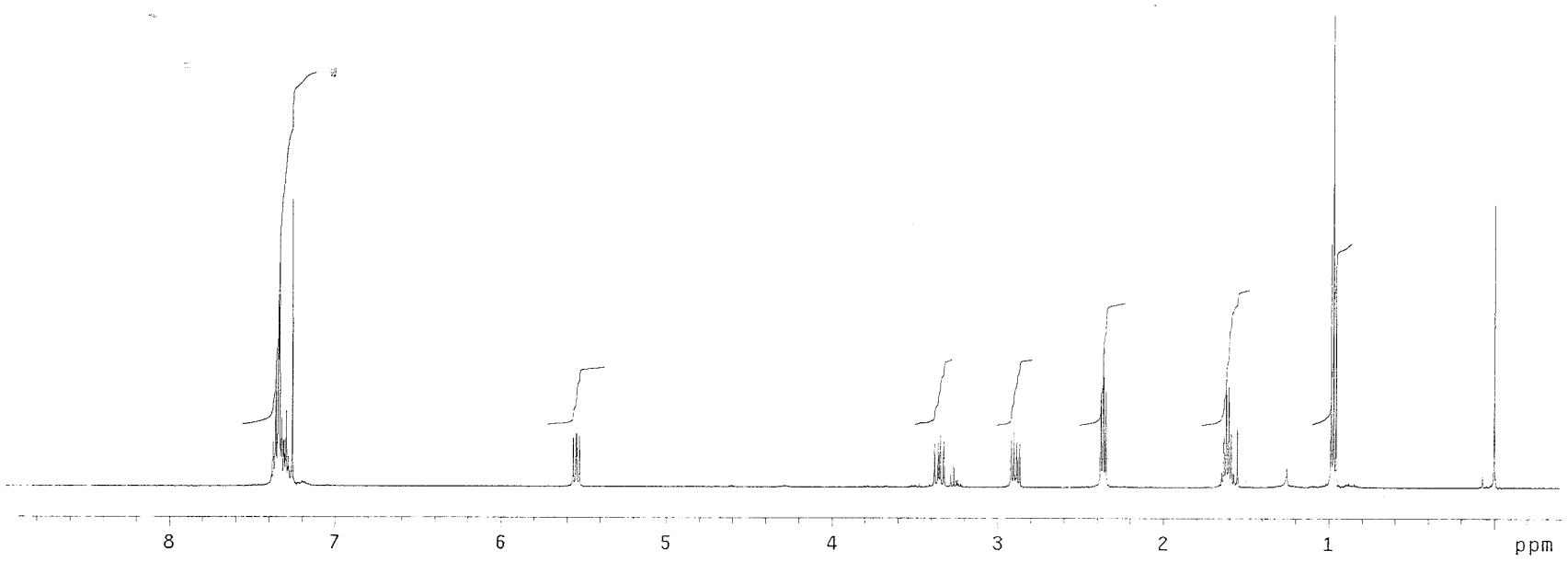
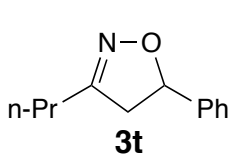


3t

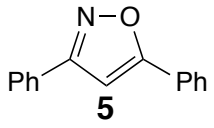


^{13}C NMR (125 MHz, $CDCl_3$):

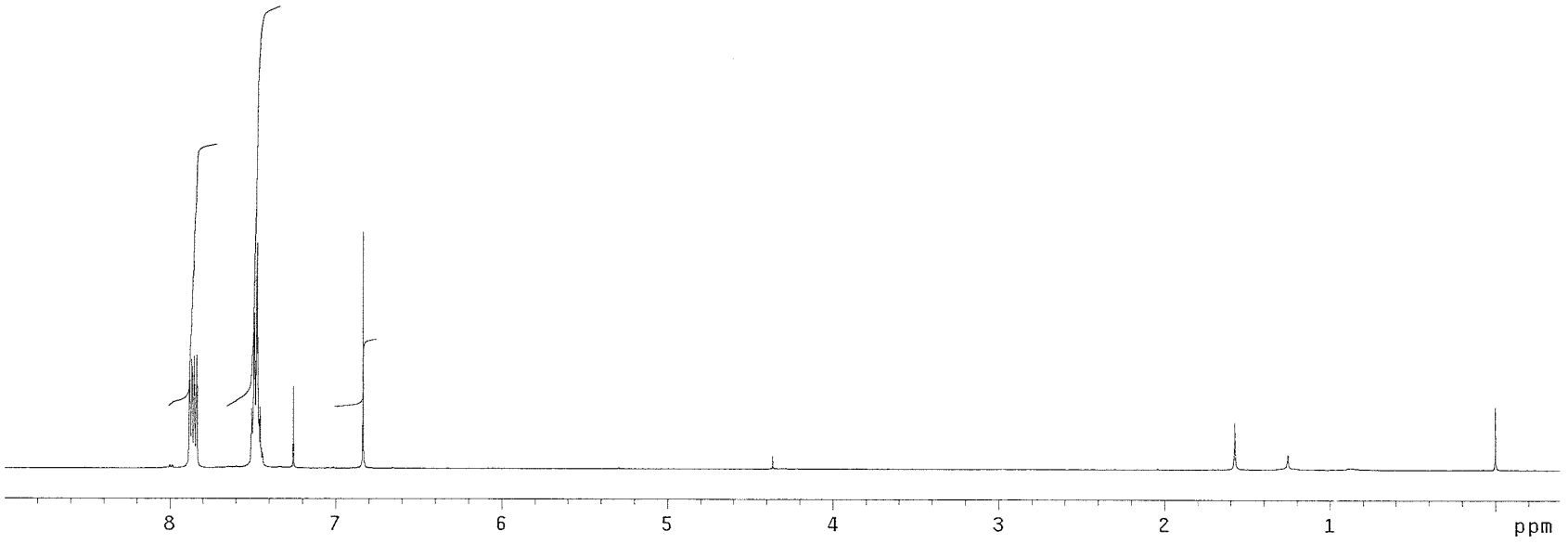


3t

^{1}H NMR (500 MHz, $CDCl_3$):



5



^{13}C NMR (125 MHz, $CDCl_3$):