Supplementary Material (ESI) for Organic & Biomolecular Chemistry

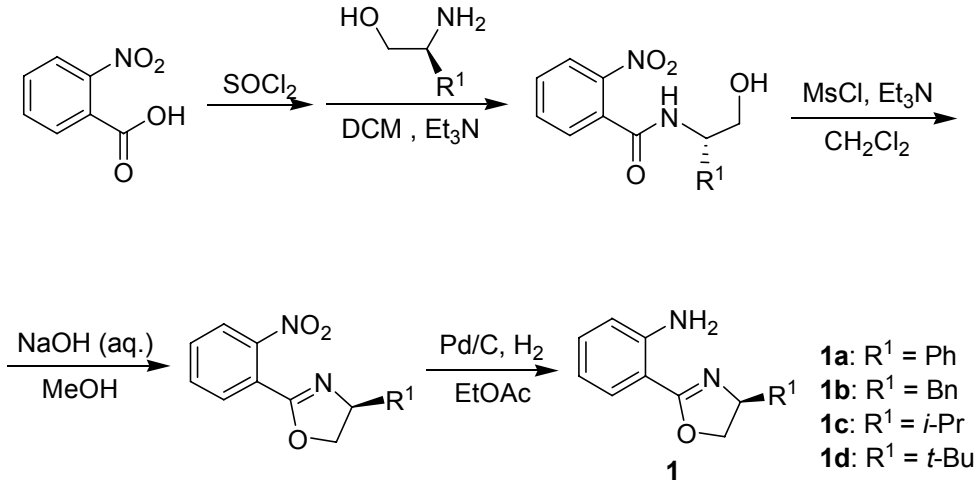
Efficient *in situ* Three-component Formation of Chiral Oxazoline-Schiff Base Copper(II) Complexes: Towards Combinatorial Library of Chiral Catalysts for Asymmetric Henry Reaction

Wen Yang,[a] Han Liu,[b] Da-Ming Du*[a]

[a]*School of Chemical Engineering and Environment, Beijing Institute of Technology, Beijing 100081, People's Republic of China*
[b]*Beijing National Laboratory for Molecular Sciences (BNLMS), College of Chemistry and Molecular Engineering, Peking University, Beijing 100871, Peoples's Republic of China*
E-mail: dudm@bit.edu.cn

Supporting information

Synthesis of 2-(2-aminophenyl)oxazolines 1a–d



Synthesis of nitrobenzamides

A solution of 2-nitrobenzoic acid (835 mg, 5.0 mmol) and thionyl chloride (5 mL) was refluxed for 3 h. The excess thionyl chloride was removed under reduced pressure to afford 2-nitrobenzoyl chloride. The above chloride in CH_2Cl_2 (25 mL) was added dropwise to a solution of chiral amino alcohol (5.0 mmoL) and Et_3N (1.8 mL, 12.5 mmol) in CH_2Cl_2 (20 mL) at 0 °C, then stirred at room temperature for 10 h. The reaction mixture was successively washed with saturated NH_4Cl (aq), HCl (1M), saturated $NaHCO_3$ (aq) and brine. The organic layer was dried over anhydrous Na_2SO_4, concentrated and purified by silica gel column chromatography (ethyl acetate-petroleum ether 1:1 *V*/*V*) to afford the nitrobenzamides.

N-((*S*)-2-Hydroxy-1-phenylethyl)-2-nitrobenzamide

White solid, yield 86%; m.p. 138–139 °C. $[\alpha]_D^{25}$ = +27.2 (*c* = 0.94, CH_2Cl_2); ^{1}H NMR (300M, d^6-DMSO): δ = 9.07 (d, *J* = 8.1 Hz, 1H), 8.03 (d, *J* = 7.8 Hz, 1H), 7.80 (t, *J* = 7.5 Hz, 1H), 7.72–7.66 (m, 2H), 7.41–7.26 (m, 5H), 5.00 (dd, *J* = 6.9 Hz, *J* = 14.1 Hz, 1H), 4.93 (t, *J* = 5.7 Hz, 1H), 3.71–3.58 (m, 2H); ^{13}C NMR (75M, d^6-DMSO): δ = 165.0, 147.0, 140.5, 133.4, 132.5, 130.6, 129.2, 128.0, 127.0, 126.8, 123.9, 64.4, 55.5; IR (KBr): ν 3315, 2960, 1648, 1575, 1531, 1453, 1350, 1318, 1052, 853, 735, 704 cm^{-1}; HR-ESIMS: m/z cacld for $C_{15}H_{15}N_2O_4$ (M+H): 287.09871. Found: 287.10263.

_N_-((_S_)-1-Hydroxymethyl-2-phenylethyl)-2-nitrobenzamide

Pale yellow oil, yield 98%. $[\alpha]_D^{25}$ = –6.8 (*c* = 1.68, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.83 (d, *J* = 7.8 Hz, 1H), 7.48–7.36 (m, 2H), 7.28–7.15 (m, 5H), 6.93 (d, *J* = 8.4 Hz, 1H), 4.21–4.19 (m, 1H), 3.59 (dd, *J* = 3.3 Hz, *J* = 10.8 Hz, 1H), 3.48 (dd, *J* = 4.5 Hz, *J* = 11.1 Hz, 1H), 3.37 (br s, 1H), 2.84 (d, *J* = 6.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 166.8, 145.8, 137.6, 133.6, 132.4, 130.1, 129.1, 128.5, 128.4, 126.4, 124.1, 62.7, 53.1, 36.3; IR (KBr): ν 3467, 2958, 1624, 1549, 1523, 1476, 1340, 1294, 1096, 825, 751 cm^{-1}; HR-ESIMS: m/z cacld for $C_{16}H_{17}N_2O_4$ (M+H): 301.11436. Found: 301.11828.

_N_-((_S_)-1-Hydroxymethyl-3-methylbutyl)-2-nitrobenzamide

White solid, yield 89%; m.p. 108–109 °C. $[\alpha]_D^{25}$ = –20.2 (*c* = 1.08, CH_2Cl_2); ^{1}H NMR (300M, $CDCl_3$): δ = 8.03 (d, *J* = 7.8 Hz, 1H), 7.66 (t, *J* = 7.2 Hz, 1H), 7.58–7.51 (m, 2H), 6.25 (d, *J* = 7.8 Hz, 1H), 3.92–3.73 (m, 3H), 2.37 (br s, 1H), 2.02–1.91 (m, 1H), 1.02 (d, *J* = 7.2 Hz, 6H); ^{13}C NMR (75M, d^6-DMSO): δ = 165.4, 146.9, 133.3, 133.1, 130.3, 129.2, 123.8, 60.9, 56.3, 28.1, 19.6, 17.9; IR (KBr): ν 3429, 3320, 2963, 1627, 1548, 1523, 1417, 1351, 1074, 853, 789, 698 cm^{-1}; HR-ESIMS: m/z cacld for $C_{12}H_{17}N_2O_4$ (M+H): 253.11436. Found: 253.11828.

_N_-((_S_)-1-Hydroxymethyl-2,2-dimethylpropyl)-2-nitrobenzamide

White solid, yield 85%; m.p. 137–138 °C. $[\alpha]_D^{25}$ = – 60.5 (*c* = 3.65, EtOH); ^{1}H NMR (300M, $CDCl_3$): δ = 8.04 (d, *J* = 7.8 Hz, 1H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.60–7.54 (m, 2H), 6.17 (d, *J* = 9.0 Hz, 1H), 4.08–4.01 (m, 1H), 3.95 (dd, *J* = 3.6 Hz, *J* = 11.4 Hz, 1H), 3.70 (dd, *J* = 7.2 Hz, *J* = 11.4 Hz, 1H), 2.25 (br s, 1H), 1.03 (s, 9H); ^{13}C NMR (75M, d^6-DMSO): δ = 165.6, 147.1, 133.14, 133.11, 130.3, 129.4, 123.7, 60.5, 59.5, 33.9, 26.9; IR (KBr): ν 3420, 3230, 3076, 2957, 1645, 1562, 1540, 1469, 1367, 1315, 1048, 871, 779, 728, 696 cm^{-1}; HR-ESIMS: m/z cacld for $C_{13}H_{19}N_2O_4$ (M+H): 267.13001. Found: 267.13393.

Synthesis of 2-(2-nitrophenyl)oxazolines

To a ice-cold solution of nitrobenzamide (4.0 mmol) and Et_3N (1.4 mL, 10.0 mmol) in CH_2Cl_2 (30 mL) was added methanesulfonyl chloride (0.39 mL, 0.5 mmol) via a syringe. After stirred for 6 h at room temperature, the reaction mixture was concentrated under reduced pressure to afford the crude product. The crude product was dissolved in methanol (15 mL), and a solution of NaOH(0.5 g) in water (5 mL) was added. The reaction mixture was refluxed for 3 h, then cooled to room temperature. The methanol was removed under reduced pressure and the residue was extracted with CH_2Cl_2 (3×25 mL). The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, concentrated and purified by silica gel column chromatography (ethyl acetate-petroleum ether 1:10 *V*/*V*) to afford the 2-(2-nitrophenyl)oxazolines.

(_S_)- 4-Phenyl-2-(2-nitrophenyl)- oxazoline

Pale yellow oil, yield 92%. $[\alpha]_D^{25}$ = –50.1 (*c* = 1.23, CH_2Cl_2); ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.93–7.86 (m, 2H), 7.66–7.60 (m, 2H), 7.38–7.27 (m, 5H), 5.40 (dd, *J* = 9.0 Hz, *J* = 10.0 Hz, 1H), 4.80 (dd, *J* = 8.4 Hz, *J* = 10.2 Hz, 1H), 4.28 (t, *J* = 8.6 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ = 162.3, 149.1, 141.5, 132.5, 131.5, 131.2, 128.7, 127.7, 126.8, 123.9, 123.1, 75.8, 70.4; IR (KBr): ν 3065, 1642, 1529, 1349, 1039, 850, 789, 701 cm^{-1}; HR-ESIMS: m/z cacld for $C_{15}H_{13}N_2O_3$ (M+H): 269.08815. Found: 269.09207.

(*S*)- 4-Benzyl-2-(2-nitrophenyl)-oxazoline

White soild, yield 89%; m.p. 55–56 °C. $[\alpha]_D^{25}$ = +9.0 (*c* = 1.15, CH_2Cl_2); ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.87–7.76 (m, 2H), 7.66–7.56 (m, 2H), 7.32–7.22 (m, 5H), 4.65–4.53 (m, 1H), 4.37 (t, *J* = 8.8 Hz, 1H), 4.16 (dd, *J* = 7.4 Hz, *J* = 8.4 Hz, 1H), 3.21 (dd, *J* = 5.6 Hz, *J* = 13.8 Hz, 1H), 2.81 (dd, *J* = 8.2 Hz, *J* = 13.8 Hz, 1H); ^{13}C NMR (50 MHz, $CDCl_3$): δ = 161.3, 149.1, 137.6, 132.3, 131.3, 130.9, 129.2, 128.5, 126.5, 123.8, 123.3, 72.8, 68.1, 41.2; IR (KBr): ν 3060, 1659, 1605, 1532, 1357, 1060, 948, 852, 754, 700 cm^{-1}; HR-ESIMS: m/z cacld for $C_{16}H_{15}N_2O_3$ (M+H): 235.10380. Found: 235.10772.

(*S*)-4-(1-Methylethyl)-2-(2-nitrophenyl)oxazoline

Pale yellow oil, yield 94%. $[\alpha]_D^{25}$ = –66.8 (*c* = 1.87, CH_2Cl_2); ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.86–7.80 (m, 2H), 7.64–7.57 (m, 2H), 4.30 (dd, *J* = 5.0 Hz, *J* = 6.6 Hz, 1H), 4.19–4.10 (m, 2H), 1.98–1.82 (m, 1H), 1.04 (d, *J* = 6.8 Hz), 0.97 (d, *J* = 6.8 Hz); ^{13}C NMR (50 MHz, $CDCl_3$): δ = 160.7, 149.0, 132.2, 131.2, 130.9, 123.7, 123.3, 72.9, 71.1, 32.5, 18.6, 18.2; IR (KBr): ν 2961, 1662, 1608, 1536, 1357, 1063, 955, 852, 784, 705 cm^{-1}; HR-ESIMS: m/z cacld for $C_{12}H_{15}N_2O_3$ (M+H): 283.10380. Found: 283.10772.

(*S*)-4-(1,1-Dimethylethyl)-2-(2-nitrophenyl)oxazoline

Pale yellow oil, yield 90%. $[\alpha]_D^{25}$ = –92.7 (*c* = 1.09, CH_2Cl_2); ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.86–7.80 (m, 2H), 7.64–7.57 (m, 2H), 4.37 (dd, *J* = 8.4 Hz, *J* = 10.2 Hz, 1H), 4.24 (t, *J* = 8.2 Hz, 1H), 4.08 (dd, *J* = 8.2 Hz, *J* = 10.2 Hz, 1H), 0.98 (s, 9H); ^{13}C NMR (50 MHz, $CDCl_3$): δ = 160.7, 149.1, 132.3, 131.2, 131.0, 123.7, 123.4, 76.6, 69.7, 34.0, 25.9; IR (KBr): ν 2956, 1663, 1536, 1357, 1062, 955, 851, 782, 705 cm^{-1}; HR-ESIMS: m/z cacld for $C_{13}H_{17}N_2O_3$ (M+H): 249.11945. Found: 249.12337.

Synthesis of 2-(2-aminophenyl)oxazolines 1a–d

To a solution of 2-(2-nitrophenyl)oxazoline (3.0 mmol) in EtOAc (25 mL) was added Pd/C (50 mg). The reaction was placed under an atmosphere of H_2 in a rubber balloon and stirred for 10 h. The reaction mixture was filtered through Celite to remove Pd/C, concentrated and purified by silica gel column chromatography (ethyl acetate-petroleum ether 1:20 *V*/*V*) to afford the 2-(2-aminophenyl)oxazolines.

(*S*)-2-(2-Aminophenyl)-4-phenyloxazoline 1a

White soild, yield 91%; m.p. 74–75 °C. $[\alpha]_D^{25}$ = +207.6 (*c* = 0.50, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.77 (d, *J* = 8.1 Hz, 1H), 7.36–7.18 (m, 6H), 6.69–6.65 (m, 2H), 6.11 (br, 2H), 5.42 (t, *J* = 9.0 Hz, 1H), 4.65 (t, *J* = 9.0 Hz, 1H), 4.10 (t, *J* = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 165.0, 148.8, 142.7, 132.2, 129.7, 128.6 127.5, 126.6, 116.0,115.7, 108.6, 73.0, 70.1. lit.[1] m.p. 74–77 °C. $[\alpha]_D$ = +185.1 (*c* = 1.0, $CHCl_3$).

(*S*)-2-(2-Aminophenyl)-4-benzyloxazoline 1b

White soild, yield 91%; m.p. 57–58 °C. $[\alpha]_D^{25}$ = +43.3 (*c* = 0.51, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.68 (d, *J* = 7.8 Hz, 1H), 7.32–7.16 (m, 6H), 6.68–6.61 (m, 2H), 6.08 (br s, 2H), 4.64–4.53 (m, 1H), 4.25 (t, *J* = 8.7 Hz, 1H), 4.00 (t, *J* = 7.8 Hz, 1H), 3.11 (dd, *J* = 6.0 Hz, *J* = 13.5 Hz, 1H), 2.74 (dd, *J* = 8.1 Hz, *J* = 13.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 164.0, 148.6, 138.3, 132.0, 129.5, 129.1, 128.4, 126.3, 115.9, 115.6, 108.9, 70.2, 68.0, 42.2. lit.[1] m.p. 56–57 °C. $[\alpha]_D$ = +24.7 (*c* = 1.0, $CHCl_3$).

(*S*)-2-(2-Aminophenyl)-4-isopropyloxazoline 1c

White soild, yield 91%; m.p. 64–65 °C. $[\alpha]_D^{25}$ = +23.2 (*c* = 0.50, CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.67 (d, *J* = 7.8 Hz, 1H), 7.24–7.16 (m, 1H), 6.69–6.62 (m, 2H), 5.98 (br, 2H), 4.30 (t, *J* = 8.4 Hz, 1H), 4.14–4.06 (m, 1H), 3.99 (t, *J* = 7.8 Hz, 1H), 1.81–1.72 (m, 1H), 1.02 (d, *J* = 6.6 Hz, 1H), 0.93 (d, *J* = 6.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 163.5, 148.5, 131.9, 129.5, 115.9, 115.6, 109.1, 72.8, 68.7, 33.1, 18.9, 18.5. lit.[1] m.p. 64–65 °C. $[\alpha]_D$ = +10.2 (*c* = 1.0, $CHCl_3$).

(*S*)-2-(2-Aminophenyl)-4-tert-butyloxazoline 1d

White soild, yield 90%; m.p. 67–68 °C. $[\alpha]_D^{25}$ = +34.8 (*c* = 0.50 , CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.67 (d, *J* = 7.8 Hz, 1H), 7.24–7.16 (m, 1H), 6.70–6.62 (m, 2H), 6.15 (br s, 2H), 4.27–4.19 (m, 1H), 4.13–4.08 (m, 2H), 0.93 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 163.4, 148.6, 131.8, 129.5, 115.9, 115.6, 109.1, 76.3, 66.8, 33.8, 25.8. lit.[1] m.p. 66–67 °C. $[\alpha]_D$ = +35 (*c* = 1.0, EtOH).

Table S1 Validation of the reproducibility and optimization of reaction temperature.[a]

Entry	T(°C)	Additive	Time (h)	Yield (%)[b]	ee (%)[c]
1	r.t.	none	24	90	69
2	r.t.	10 mol% Et_3N	12	99	20
3	r.t.	10 mol% NaOH	12	94	6
4	r.t.	100 mg 4 Å MS	20	98	56
5	r.t.	Na_2SO_4	24	81	70
6	50	none	7	95	63
7	10	none	72	94	71
8	0	none	168	95	78

[a] Reactions were performed with 0.5 mmol of 4-nitrobenzaldehyde and 5.0 mmol of nitromethane in 2 mL of EtOH in the presence of 10 mol % catalyst **4db** at room temperature. [b] Isolated yields after column chromatography purification. [c] Determined by HPLC using a Daicel Chiracel OD-H column (*n*-hexane–isopropanol 80:20 *V*/*V*, 1.0 mL/min, 254 nm).

Table S2 Optimization of solvents and catalyst loading.[a]

Entry	Solvent	Yield (%)[b]	ee (%)[c]
1	EtOH	90	69
2	95% EtOH	95	66
3	CF_3CH_2OH	trace	nd
4	MeOH	79	64
5	*i*-PrOH	87	67
6	THF	96	74
7	dioxane	trace	nd
8	TBME	93	76
9	toluene	34	80
10[d]	toluene	39	77
11	benzene	32	77
12	CH_2Cl_2	38	76
13	Et_2O	96	82
14[e]	Et_2O	90	74
15[f]	Et_2O	61	70

[a] Reactions were performed with 0.5 mmol of 4-nitrobenzaldehyde and 5.0 mmol of nitromethane in 2 mL of solvent in the presence of 10 mol % catalyst **4db** at room temperature for 24 h. [b] Isolated yields after column chromatography purification. [c] Determined by HPLC using a Daicel Chiracel OD-H column (*n*-hexane–isopropanol 80:20 *V*/*V*, 1.0 mL/min, 254 nm). [d] Catalyst **4db** was formed in EtOH, while the Henry reaction was carried out in toluene. [e] 5 mol % catalyst loading was employed. [f] 2.5 mol % catalyst loading was employed.

General procedure for asymmetric Henry reaction catalyzed by *in situ* three-component generated complex catalyst

A solution of 2-(2-aminophenyl)oxazoline **1d (**10.9 mg, 0.05 mmol), 5-nitrosalicylaldehyde **2b** (8.4 mg, 0.05 mmol), and diethyl ether (2 mL) was stirred for 30 min at room temperature. Anhydrous $Cu(OAc)_2$ (9.1 mg, 0.05 mmol) was added and stirred for another 30 min, and the reaction mixture turned dark green. Aldehyde (0.5 mmol) and nitromethane (5.0 mmol, 0.26 mL) were added and stirred for 24–96 h at room temperature. The reaction mixture was concentrated and purified by silica gel column chromatography using petroleum ether-ethyl acetate 5:1 as eluent.

(*S*)-1-(4-Nitrophenyl)-2-nitroethanol 7a

Compound **7a** was prepared according to the general procedure to give a colourless solid (97 mg, 92% yield); m.p. 84–85 ^oC. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 80:20 *V*/*V*, 1.0 mL/min, 254 nm); minor enantiomer t_r =

11.1 min, major enantiomer t_r = 13.3 min; 82% ee; $[\alpha]_D^{25}$ = +29.1 (*c* = 0.75, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 8.28–8.23 (m, 2H), 7.67–7.60 (m, 2H), 5.64–5.58 (m, 1H), 4.62–4.58 (m, 2H), 3.28 (d, *J* = 4.0 Hz, 1H). lit.[2] $[\alpha]_D^{25}$ = +29.4 (*c* = 2.36, CH_2Cl_2).

(*S*)-1-(3-Nitrophenyl)-2-nitroethanol 7b

Compound **7b** was prepared according to the general procedure to give a colourless solid (101 mg, 95% yield); m.p. 70–71 °C. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 85:15 *V*/*V*, 1.0 mL/min, 254 nm); minor enantiomer t_r = 14.5 min, major enantiomer t_r = 16.2 min; 76% ee; $[\alpha]_D^{25}$ = +34.1 (*c* = 1.12, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 8.34–8.32 (m, 1H), 8.25–8.21 (m, 1H), 7.82–7.79 (m, 1H), 7.62 (t, *J* = 7.8 Hz, 1H), 5.64–5.58 (m, 1H), 4.71–4.61 (m, 2H), 3.25 (d, *J* = 4.0 Hz, 1H). lit.[3] $[\alpha]_D^{25}$ = +32.6 (*c* = 0.13, CH_2Cl_2).

(*S*)-1-(2-Nitrophenyl)-2-nitroethanol 7c

Compound **7c** was prepared according to the general procedure to give a brown solid (103 mg, 97% yield); m.p. 80–82 °C. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 85:15 *V*/*V*, 1.0 mL/min, 254 nm); minor enantiomer t_r = 9.5 min, major enantiomer t_r = 10.3 min; 84% ee; $[\alpha]_D^{25}$ = –158.8 (*c* = 0.17, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 8.08 (dd, *J* = 8.4 Hz, *J* = 1.4 Hz, 1H), 7.96 (dd, *J* = 7.8 Hz, *J* = 1.4 Hz, 1H), 7.80–7.72 (m, 1H), 7.60–7.52 (m, 1H), 6.09–6.03 (m, 1H), 4.88 (dd, *J* = 13.6 Hz, *J* = 2.4 Hz, 1H), 4.55 (dd, *J* = 13.8 Hz, *J* = 9.0 Hz, 1H), 3.28 (d, *J* = 4.0 Hz, 1H). lit.[3] $[\alpha]_D^{25}$ = –185 (*c* = 0.19, CH_2Cl_2).

(*S*)-1-(4-Chlorophenyl)-2-nitroethanol 7d

Compound **7d** was prepared according to the general procedure to give a yellow oil (87 mg, 86% yield); Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 80:20 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 7.1 min, major enantiomer t_r = 8.5 min; 87% ee; $[\alpha]_D^{25}$ = +29.3 (*c* = 0.71, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.42–7.32 (m, 4H), 5.45 (d, *J* = 8.8 Hz, 1H), 4.64–4.44 (m, 2H), 2.99 (br s, 1H). lit.[3] $[\alpha]_D^{25}$ = +29.5 (*c* = 0.11, CH_2Cl_2).

(*S*)-1-(4-Bromophenyl)-2-nitroethanol 7e

Compound **7e** was prepared according to the general procedure to give a yellow oil (99 mg, 81% yield). Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 80:20 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 8.2 min, major enantiomer t_r = 10.1 min; 79% ee; $[\alpha]_D^{25}$ = +26.0 (*c* = 1.63, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.56–7.49 (m, 2H), 7.28–7.23 (m, 2H), 5.44–5.36 (m, 1H), 4.61–4.43 (m, 2H), 3.04 (d, *J* = 3.6 Hz, 1H). lit.[4] $[\alpha]_D^{23}$ = +29.4 (*c* = 0.67, CH_2Cl_2).

(*S*)- 2-nitro-1-phenyl-ethanol 7f

Compound **7f** was prepared according to the general procedure to give a colourless oil (71 mg, 85% yield). Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 80:20 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 7.6 min, major enantiomer t_r = 8.9 min; 83% ee; $[\alpha]_D^{25}$ = +36.8 (*c* = 1.06, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.43–7.35 (m, 5H), 5.51–5.43 (m, 1H), 4.62 (dd, *J* = 13.8 Hz, *J* = 9.2 Hz, 1H), 4.46 (dd, *J* = 13.2 Hz, *J* = 5.4 Hz, 1H), 2.84 (d, *J* = 4.0 Hz, 1H). lit.[2] $[\alpha]_D^{25}$ = +36.8 (*c* = 4.04, CH_2Cl_2).

(*S*)-1-(4-Methoxyphenyl)-2-nitroethanol 7g

Compound **7g** was prepared according to the general procedure to give a yellow oil (42 mg, 43% yield). Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 80:20 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 9.7 min, major enantiomer t_r = 11.6 min; 75% ee; $[\alpha]_D^{25}$ = +30.0 (*c* = 0.32, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.35–7.29 (m, 2H), 6.96–6.90 (m, 2H), 5.41 (d, *J* = 9.4 Hz, 1H), 4.61 (dd, *J* = 13.8 Hz, *J* = 9.0 Hz, 1H), 4.47 (dd, *J* = 13.8 Hz, *J* = 3.2 Hz, 1H), 3.82 (s, 3H), 2.76 (d, *J* = 2.2 Hz, 1H). lit.[5] $[\alpha]_D^{23}$ = +32.3 (*c* = 1.05, CH_2Cl_2).

(*S*)-1-(4-Methylphenyl)-2-nitroethanol 7h

Compound **7h** was prepared according to the general procedure to give a yellow oil (64 mg, 71% yield). Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 85:15 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 9.4 min, major enantiomer t_r = 11.5 min; 80% ee; $[\alpha]_D^{25}$ = +35.7 (*c* = 0.51, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.29–7.16 (m, 4H), 5.41–5.35 (m, 1H), 4.58 (dd, *J* = 13.2 Hz, *J* = 9.2 Hz, 1H), 4.45 (dd, *J* = 13.2 Hz, *J* = 3.4 Hz, 1H), 2.89 (d, *J* = 3.6 Hz, 1H), 2.35 (s, 3H). lit.[6] $[\alpha]_D^{25}$ = +20.2 (*c* = 0.85, CH_2Cl_2).

(*S*)-1-(1-Naphthyl)-2-nitroethanol 7i

Compound **7i** was prepared according to the general procedure to give a yellow oil (70 mg, 65% yield); Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 80:20 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 9.5 min, major enantiomer t_r = 13.0 min; 80% ee; $[\alpha]_D^{25}$ = +29.1 (*c* = 1.10, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 8.04 (d, *J* = 7.8 Hz, 1H), 7.95–7.85 (m, 2H), 7.78 (d, *J* = 7.0 Hz, 1H), 7.65–7.49 (m, 3H), 6.32–6.24 (m, 1H), 4.70–4.67 (m, 2H), 2.86 (d, *J* = 3.6 Hz, 1H). lit.[7] $[\alpha]_D^{25}$ = +24.1 (*c* = 1.03, CH_2Cl_2).

(*R*)-Nitromethyl-2-furanmethanol 7j

Compound **7j** was prepared according to the general procedure to give a yellow oil (44 mg, 56% yield). Enantiomeric excess was determined by HPLC with a Chiralcel IA column (*n*-hexane-isopropanol 95:5 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 18.3 min, major enantiomer t_r = 20.4 min; 88% ee; $[\alpha]_D^{25}$ = +34.3 (*c* = 1.05, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.43–7.42 (m ,1H), 6.41–6.37 (m, 1H), 5.52–5.44 (m, 1H), 4.80 (dd, *J* = 13.6 Hz, *J* = 8.8 Hz, 1H), 4.67 (dd, *J* = 13.4 Hz, *J* = 3.8 Hz, 1H), 3.00 (br s, 1H). lit.[4] $[\alpha]_D^{22}$ = +34.0 (*c* = 0.3, CH_2Cl_2).

(2*S*, 3*E*)-1-Nitro-4-phenyl-3-buten-2-ol 7k

Compound **7k** was prepared according to the general procedure to give a colourless solid (76 mg, 79% yield); m.p. 82–83 ^{o}C. Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 80:20 *V*/*V*, 1.0 mL/min, 254nm); major enantiomer t_r = 16.6 min, minor enantiomer t_r = 18.4 min; 75% ee; $[\alpha]_D^{25}$ = +7.4 (*c* = 2.48, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.39–7.28 (m, 5H), 6.76 (d, *J* = 16.2 Hz, 1H), 6.12 (dd, *J* = 15.8 Hz, *J* = 6.2 Hz, 1H), 5.08–4.96 (m, 1H), 4.84 (d, *J* = 6.4 Hz, 2H), 2.81 (d, *J* = 4.4 Hz, 1H). lit.[8] $[\alpha]_D^{22}$ = +11.8 (*c* = 0.64, CH_2Cl_2).

(*S*)-1-Nitro-4-phenylbutan-2-ol 7l

Compound **7l** was prepared according to the general procedure to give a yellow oil (67 mg, 69% yield); Enantiomeric excess was determined by HPLC with a Chiralcel IA column (n-hexane-isopropanol 90:10 *V*/*V*, 1.0 mL/min, 210nm); minor enantiomer t_r = 10.2 min, major enantiomer t_r = 12.4 min; 86% ee; $[\alpha]_D^{25}$ = –16.7 (*c* = 1.74, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.35–7.12 (m, 5H), 4.39–4.24 (m, 3H), 2.89–2.68 (m, 3H), 1.87–1.74 (m, 2H). lit.[8] $[\alpha]_D^{25}$ = –14.8 (*c* = 0.97, CH_2Cl_2).

(*S*)-1-Cyclohexyl-2-nitroethanol 7m

Compound **7m** was prepared according to the general procedure to give a colourless oil (80 mg, 92% yield). Enantiomeric excess was determined by HPLC with a Chiralcel OF column (*n*-hexane-isopropanol 90:10 *V*/*V*, 0.4 mL/min, 210nm); minor enantiomer t_r = 36.5 min, major enantiomer t_r = 39.6 min; 82% ee; $[\alpha]_D^{25}$ = +17.5 (*c* = 1.28, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 4.60–4.37 (m, 2H), 4.15–4.03 (m, 1H), 3.05 (d, *J* = 5.6 Hz, 1H), 1.86–1.64 (m, 5H), 1.58–1.35 (m, 1H), 1.30–1.01 (m, 5H). lit.[8] $[\alpha]_D^{25}$ = +16.9 (*c* = 0.89, CH_2Cl_2).

(*S*)-3-Methyl-1-nitro-2-butanol 7n

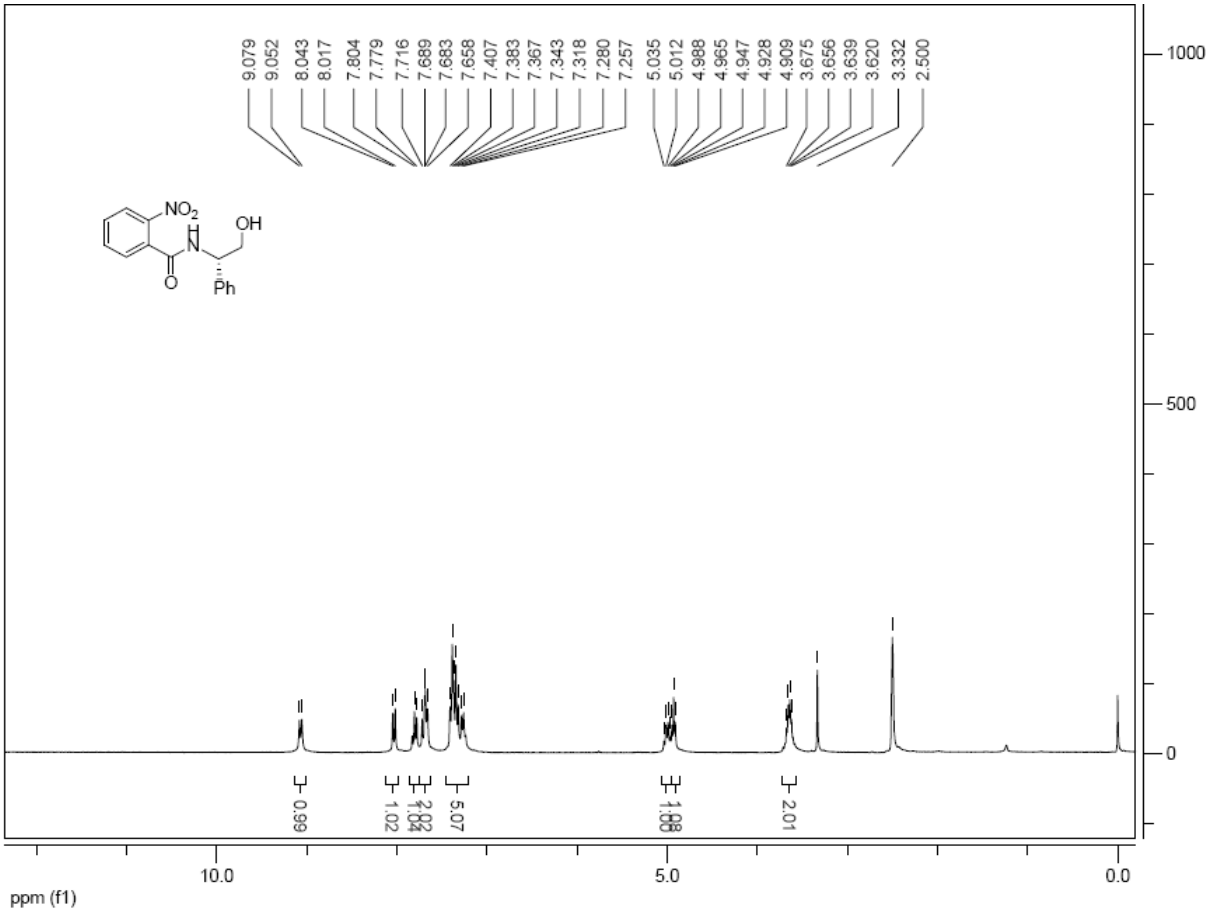
Compound **7n** was prepared according to the general procedure to give a yellow oil (54 mg, 82% yield). Enantiomeric excess was determined by HPLC with a Chiralcel OF column (*n*-hexane-isopropanol 95:5 *V*/*V*, 0.8 mL/min, 210nm); minor enantiomer t_r = 26.7 min, major enantiomer t_r = 29.0 min; 87% ee; $[\alpha]_D^{25}$ = +17.5 (*c* = 1.28, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 4.58–4.36 (m, 2H), 4.16–4.05 (m, 1H), 2.81 (d, *J* = 5.2 Hz, 1H), 1.88–1.72 (m, 1H), 1.01 (d, *J* = 3.0 Hz, 1H), 0.97 (d, *J* = 3.0 Hz, 1H). lit.[3] $[\alpha]_D^{25}$ = +13.9 (*c* = 0.14, $CHCl_3$).

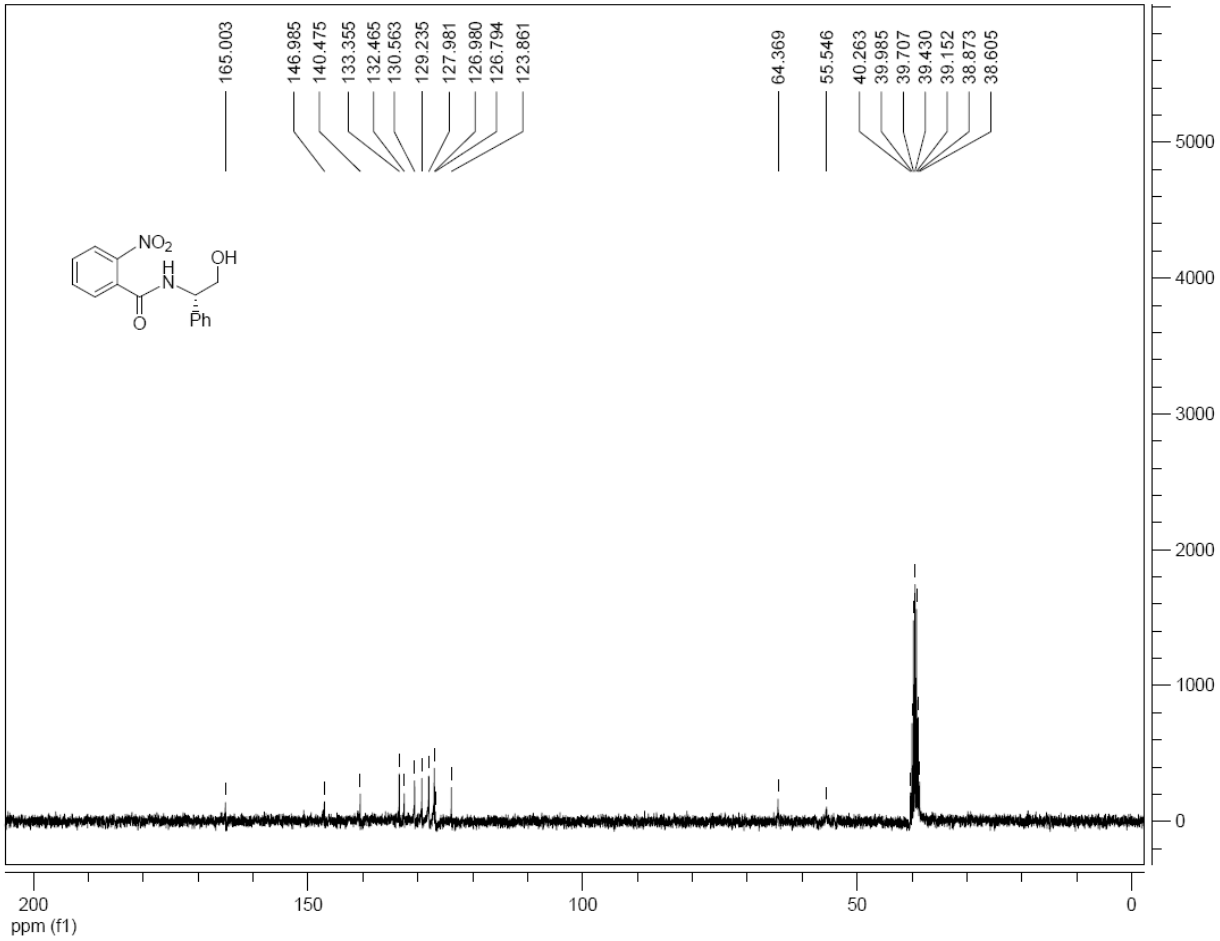
(*S*)-3,3-Dimethyl-1-nitro-2-butanol 7o

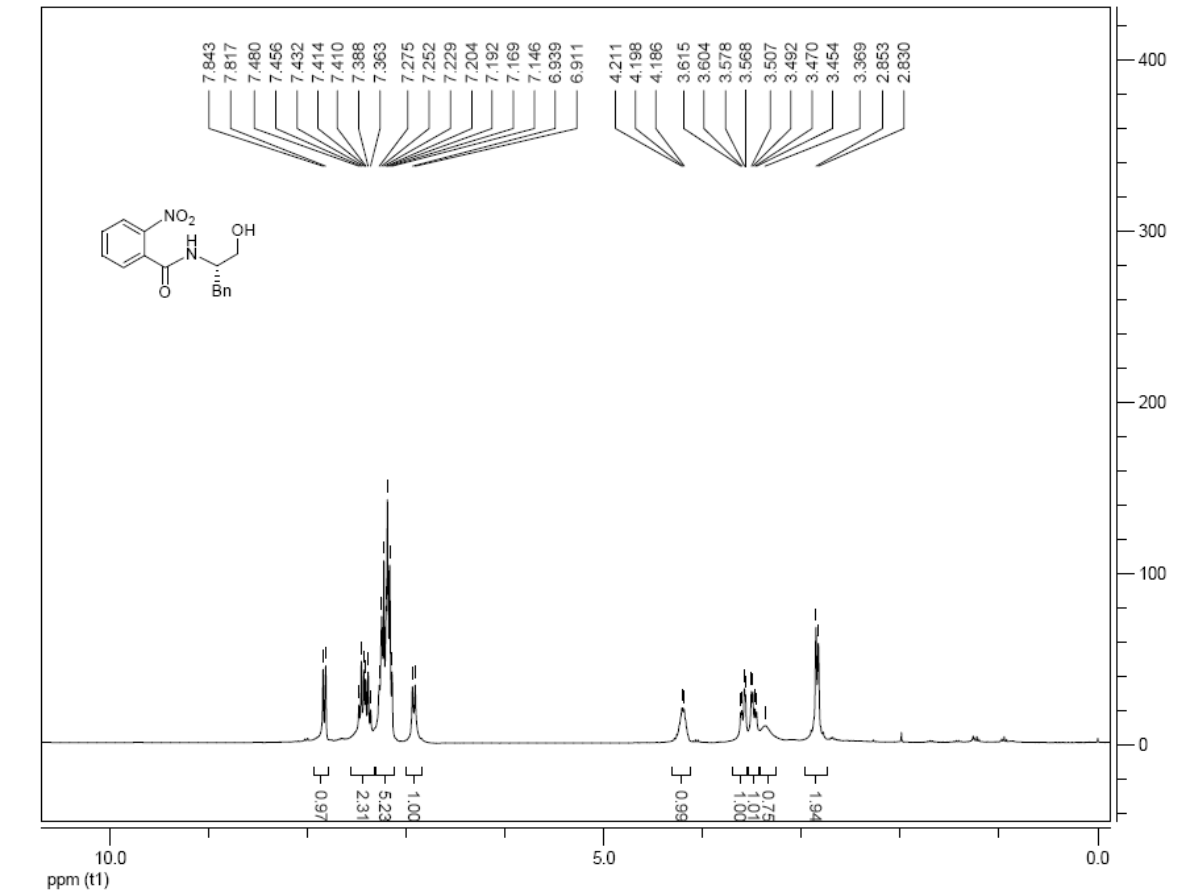
Compound **7o** was prepared according to the general procedure to give a colourless oil (60 mg, 81% yield). Enantiomeric excess was determined by HPLC with a Chiralcel OD-H column (*n*-hexane-isopropanol 95:5 *V*/*V*, 0.8 mL/min, 220nm); minor enantiomer t_r = 10.6 min, major enantiomer t_r = 12.0 min; 92% ee; $[\alpha]_D^{25}$ = +16.8 (*c* = 1.25, CH_2Cl_2). ^{1}H NMR (200 MHz, $CDCl_3$): δ = 4.50 (dd, *J* = 13.2 Hz, *J* = 2.4 Hz, 1H), 4.34 (dd, *J* = 12.8 Hz, *J* = 10.0 Hz, 1H), 4.04–3.96 (m, 1H), 2.47 (d, *J* = 4.8 Hz, 1H), 0.95 (s, 9H). lit.[9] $[\alpha]_D$ = +36.1 (*c* = 0.6, $CHCl_3$).

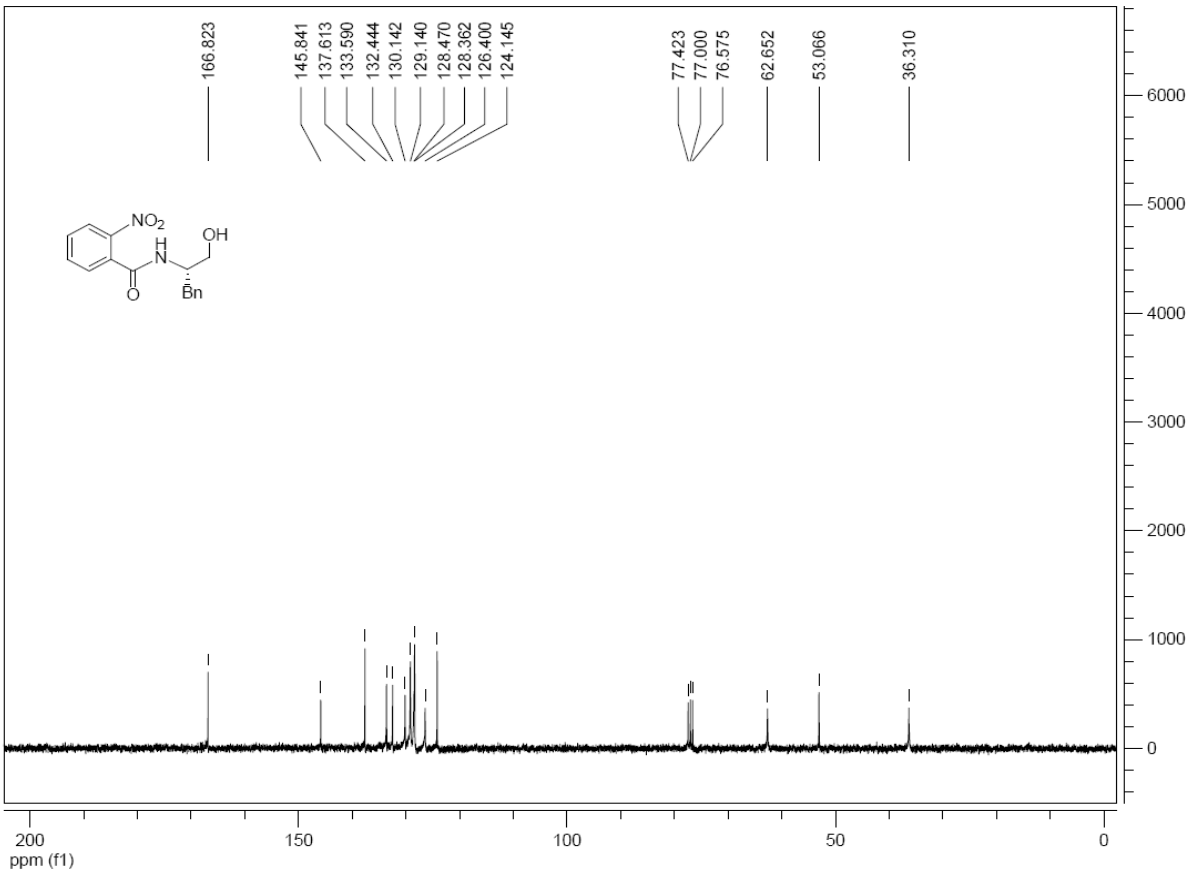
Reference:

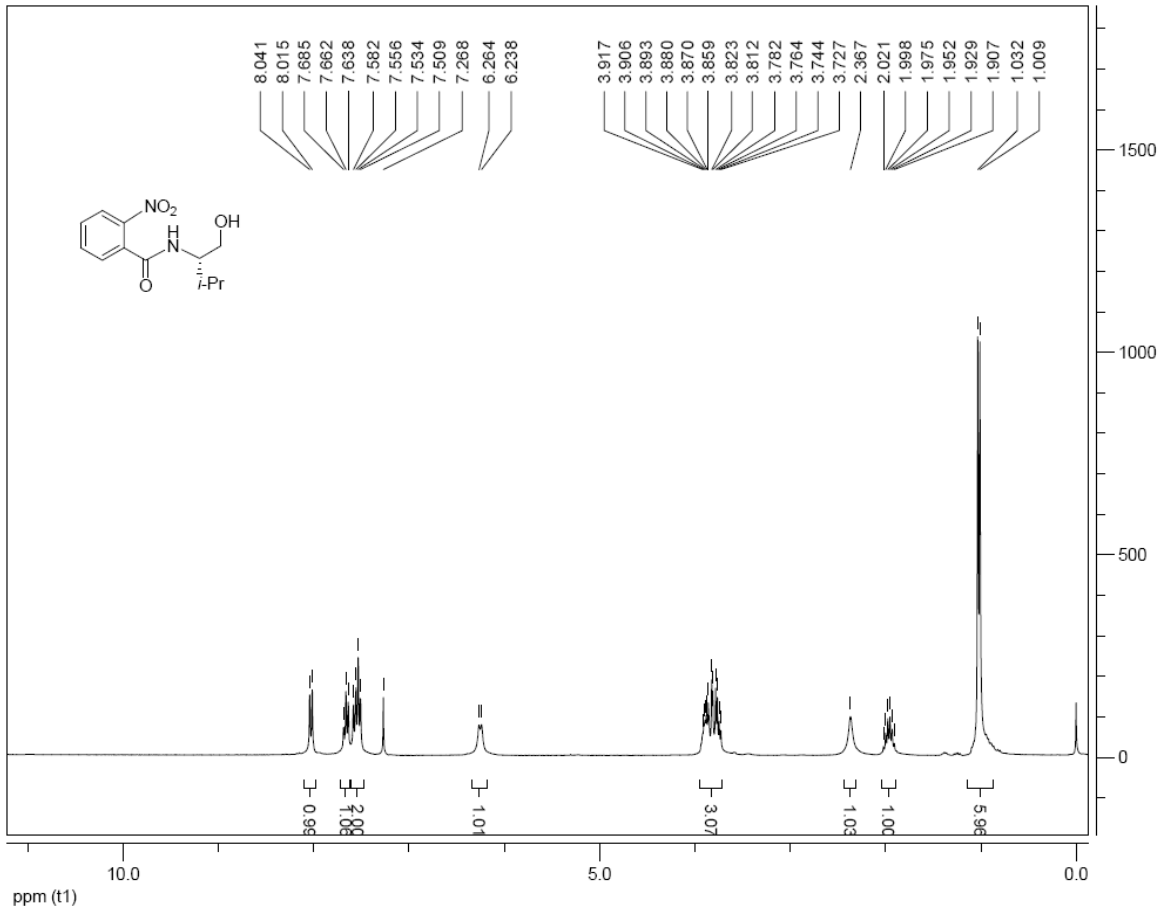
1 H. A. McManus, P. J. Guiry, *J. Org. Chem.* **2002**, *67*, 8566–8573.
2. S. K. Ginotra, V. K. Singh, *Org. Biomol. Chem.* **2007,** *5*, 3932–3937.
3. Y. Xiong, F. Wang, X. Huang, Y. Wen, X. Feng, *Chem. Eur. J.* **2007**, *13*, 829–833;
4. R. Kowalczyk, P. Kwiatkowski, J. Skarzewski, J. Jurczak, *J. Org. Chem.* 2009, 74(2), 753-756.
5. G. Blay, E. Climent, I. Fernández, J. R. Pedro, *Tetrahedron: Asymmetry* **2007**, *18*, 1603–1612.
6. J.-J. Jiang, M. Shi, *Tetrahedron: Asymmetry* **2007**, *18*, 1376–1382;
7. G. Lai, S. Wang, Z. Wang, *Tetrahedron: Asymmetry* **2008**, *19*, 1813–1819;
8. G. Blay, L. R. Domingo, V. Hernandez-Olmos, J. R. Pedro, *Chem. Eur. J.* **2008**, *14*, 4725–4730.
9. M. Bandini, F. Piccinelli, S. Tommasi, A. Umani-Ronchi, C. Ventrici, *Chem. Commun.* **2007**, 616–618.

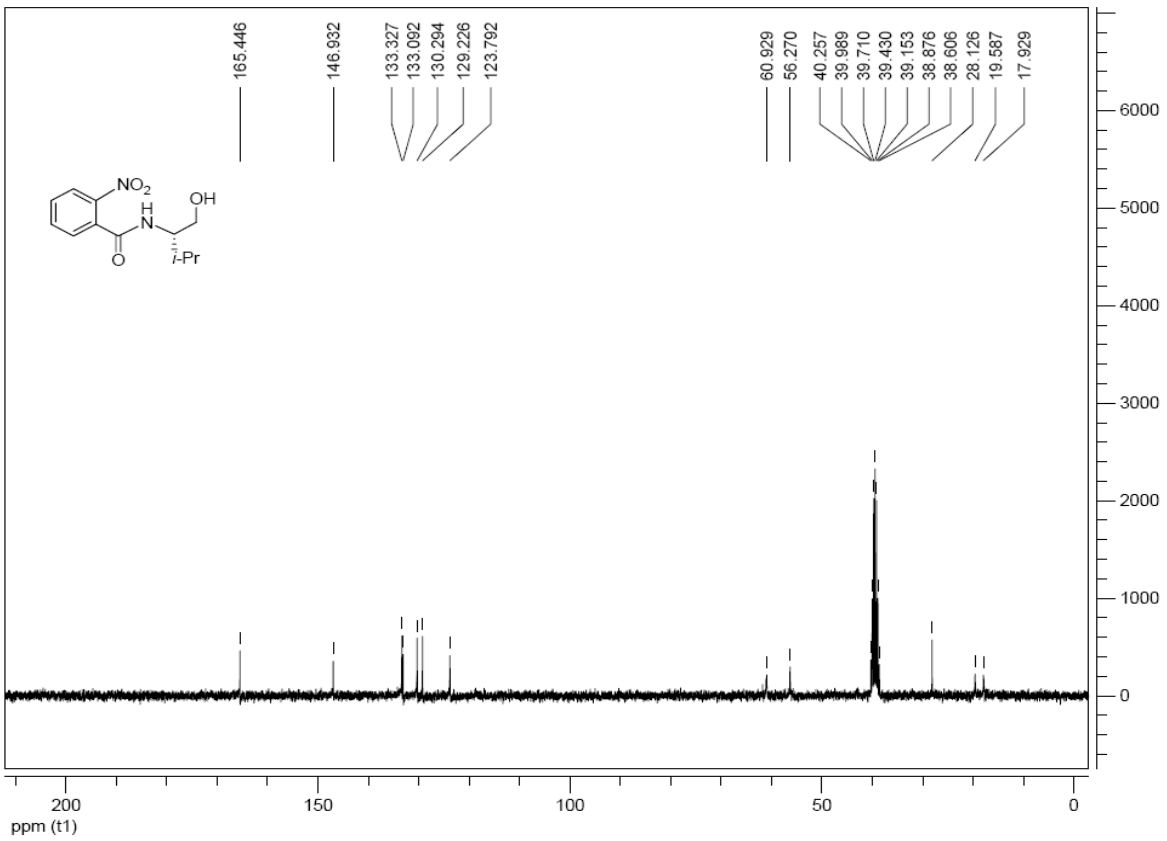

9.079
9.052
8.043
8.017
7.804
7.779
7.716
7.689
7.683
7.658
7.407
7.383
7.367
7.343
7.318
7.280
7.257
5.035
5.012
4.988
4.965
4.947
4.928
4.909
3.675
3.656
3.639
3.620
3.332
2.500
NO2
OH
H
N
O
Ph
0.99
1.02
2.02
5.07
1.00
2.01
1000
500
0
10.0
5.0
0.0
ppm (f1)

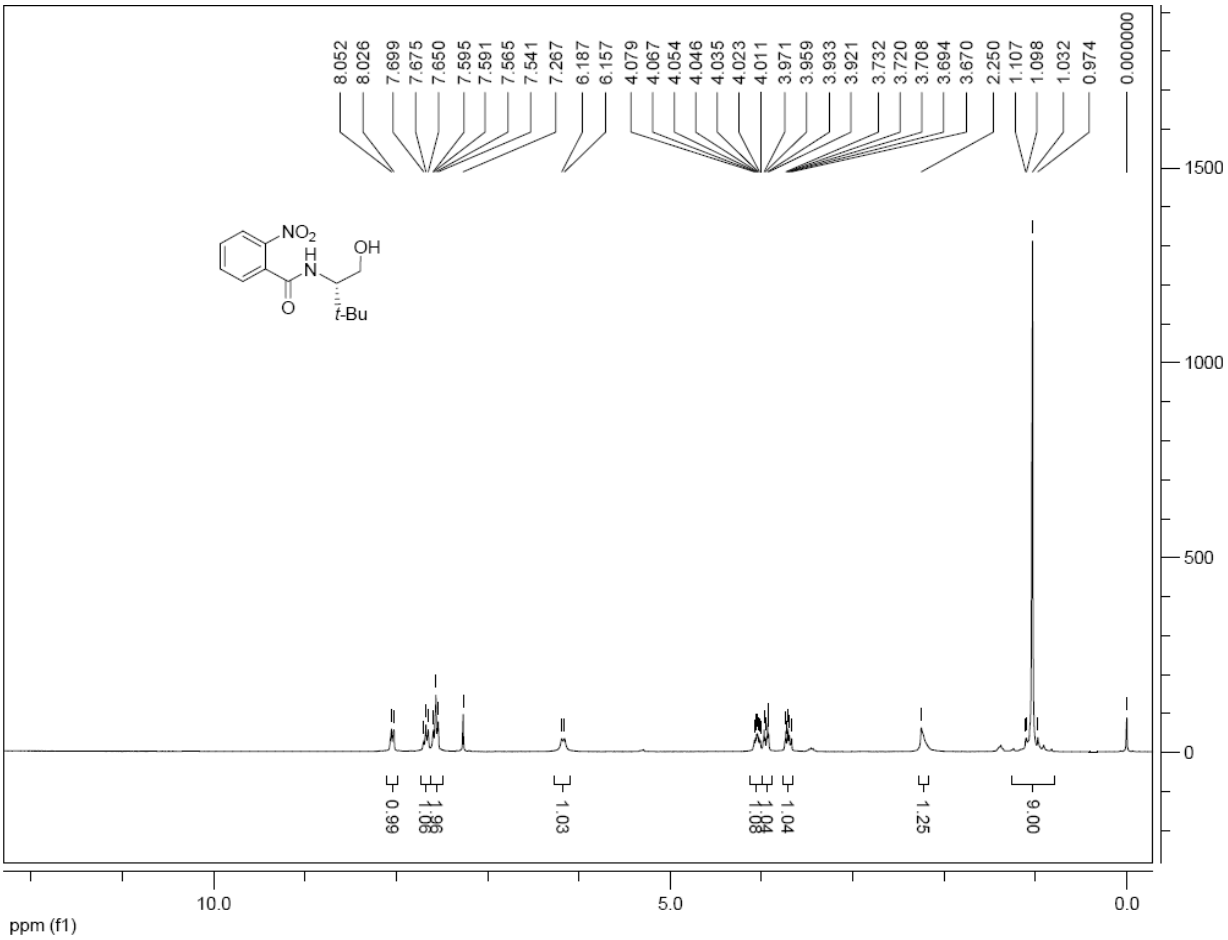

165.003
146.985
140.475
133.355
132.465
130.563
129.235
127.981
126.980
126.794
123.861
64.369
55.546
40.263
39.985
39.707
39.430
39.152
38.873
38.605
NO2
OH
H
N
O
Ph
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

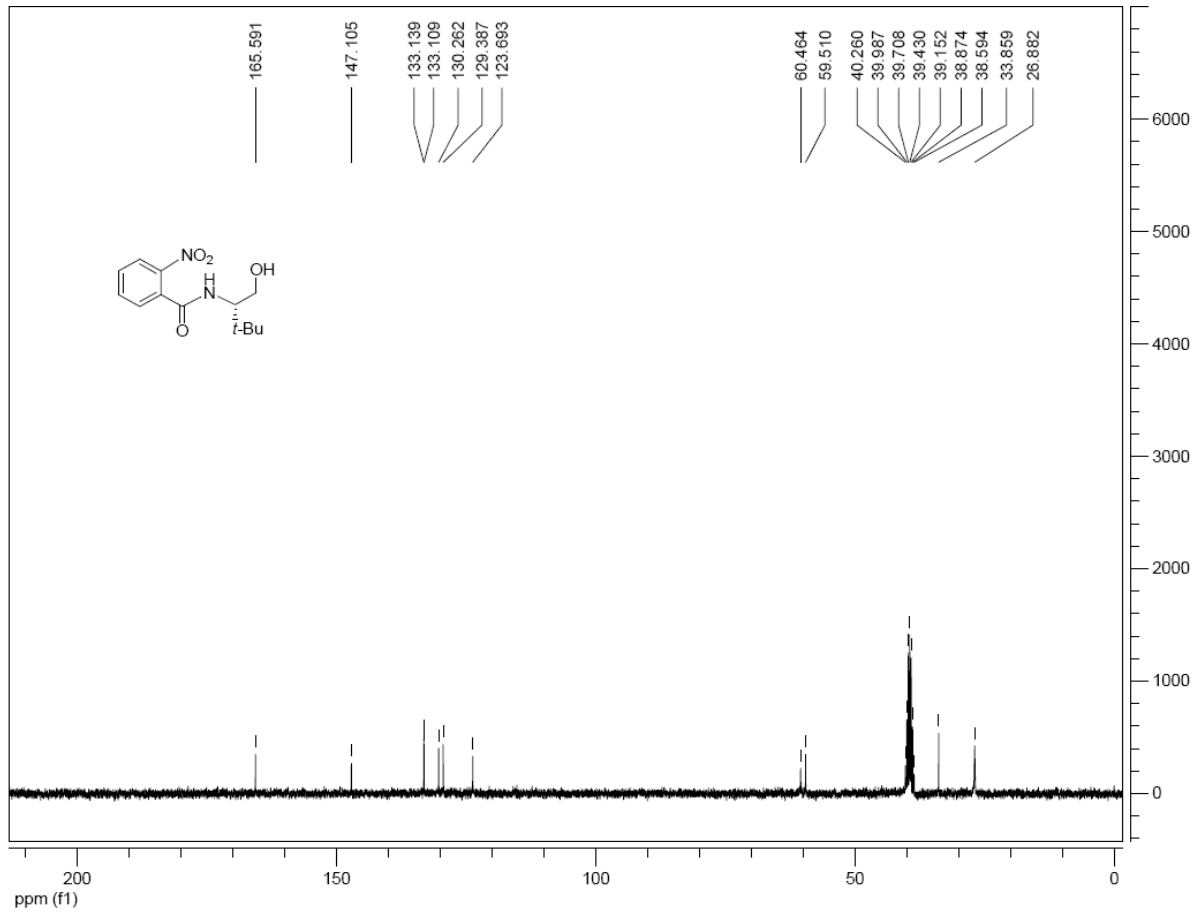

7.843
7.817
7.480
7.456
7.432
7.414
7.410
7.388
7.363
7.275
7.252
7.229
7.204
7.192
7.169
7.146
6.939
6.911
4.211
4.198
4.186
3.615
3.604
3.578
3.568
3.507
3.492
3.470
3.454
3.369
2.853
2.830
NO2
OH
H
N
O
Bn
0.97
2.31
5.23
1.00
0.99
1.00
1.01
0.75
1.94
400
300
200
100
0
10.0
5.0
0.0
ppm (t1)

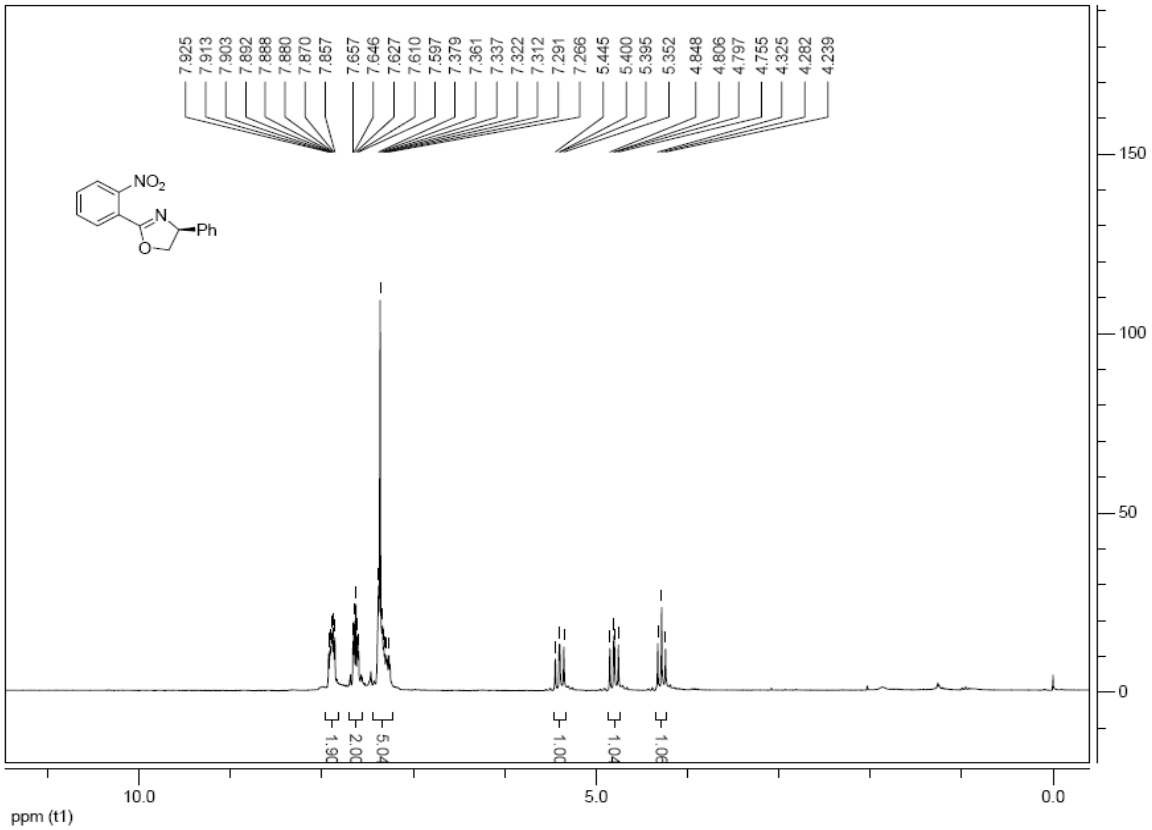

166.823
145.841
137.613
133.590
132.444
130.142
129.140
128.470
128.362
126.400
124.145
77.423
77.000
76.575
62.652
53.066
36.310
NO2
OH
H
N
O
Bn
6000
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

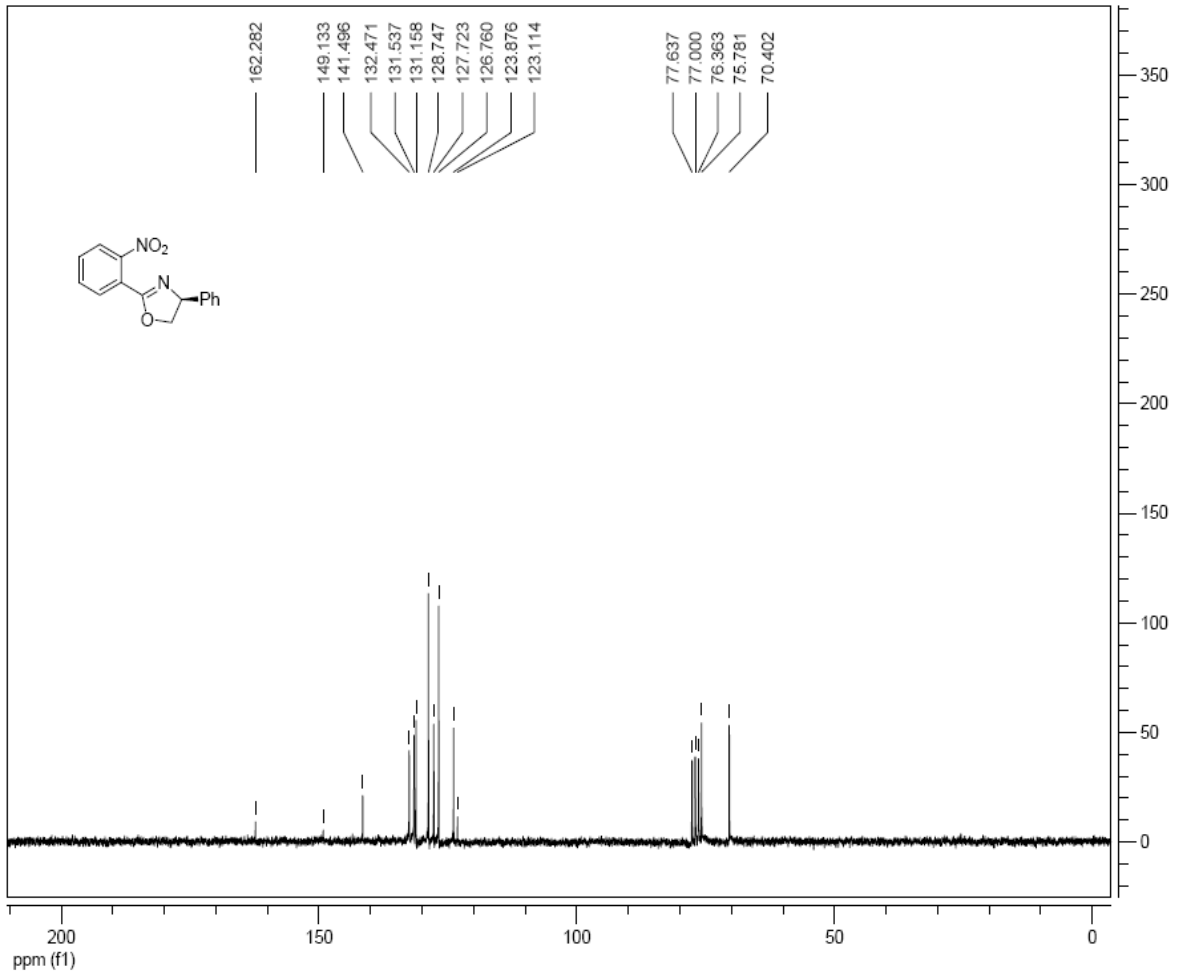

8.041
8.015
7.685
7.662
7.638
7.582
7.556
7.534
7.509
7.268
6.264
6.238
3.917
3.906
3.893
3.880
3.870
3.859
3.823
3.812
3.782
3.764
3.744
3.727
2.367
2.021
1.998
1.975
1.952
1.929
1.907
1.032
1.009
NO2
OH
H
N
O
i-Pr
0.96
1.08
2.08
1.01
3.07
1.03
1.00
5.96
1500
1000
500
0
10.0
5.0
0.0
ppm (t1)

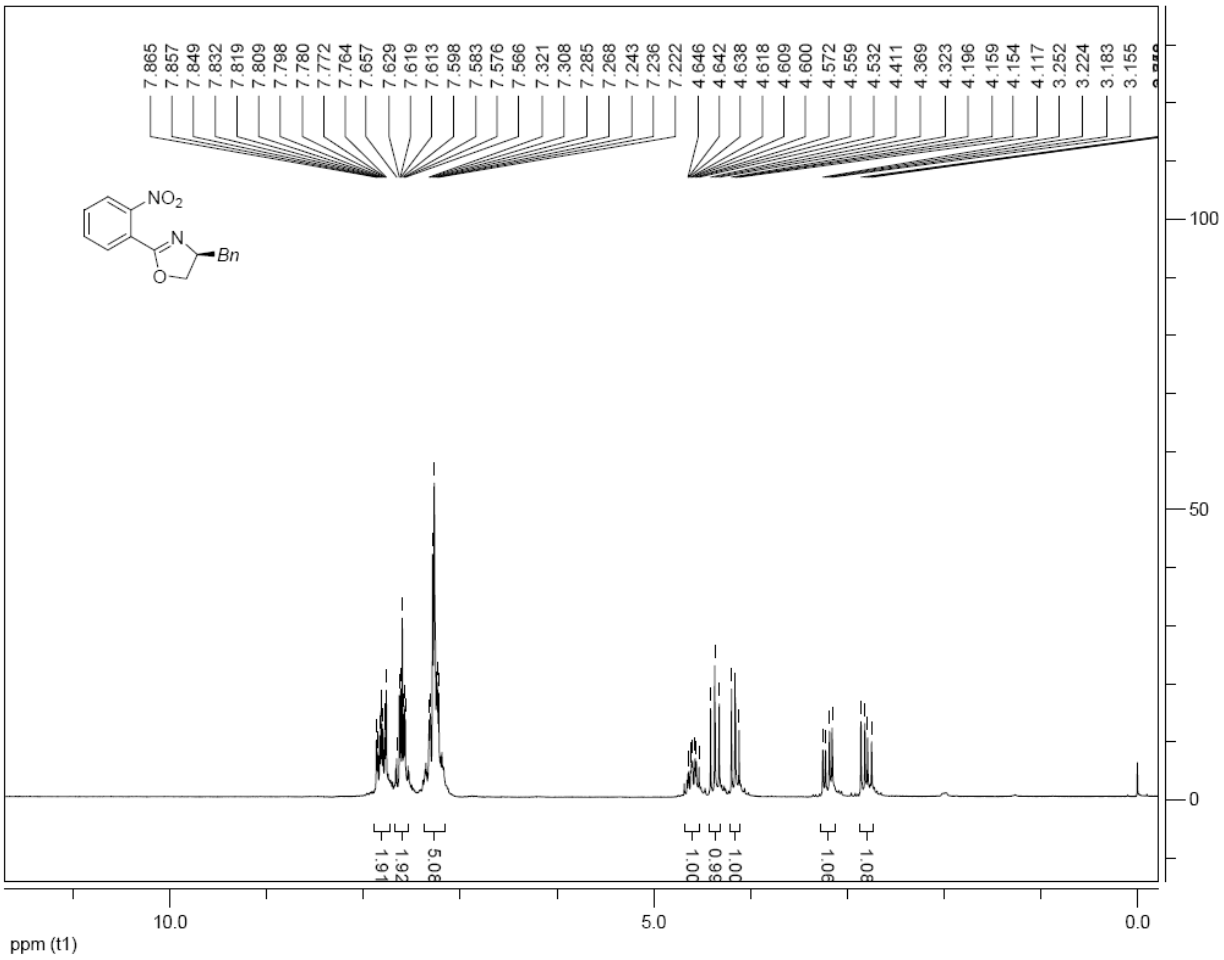

165.446
146.932
133.327
133.092
130.294
129.226
123.792
60.929
56.270
40.257
39.989
39.710
39.430
39.153
38.876
38.606
28.126
19.587
17.929
NO2
OH
H
N
O
i-Pr
6000
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (t1)

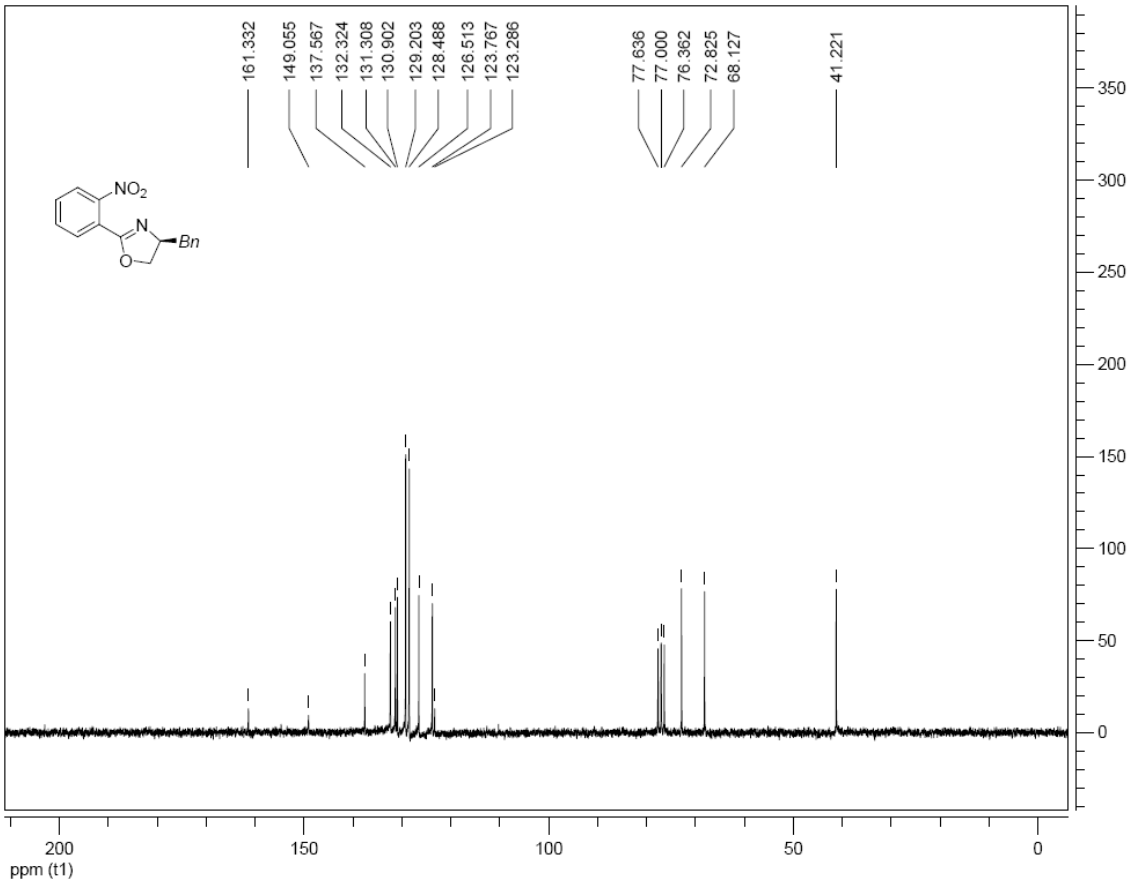

8.052
8.026
7.699
7.675
7.650
7.595
7.591
7.565
7.541
7.267
6.187
6.157
4.079
4.067
4.054
4.046
4.035
4.023
4.011
3.971
3.959
3.933
3.921
3.732
3.720
3.708
3.694
3.670
2.250
1.107
1.098
1.032
0.974
0.000000
NO2
OH
N
H
O
t-Bu
1500
1000
500
0
0.99
1.96
1.03
1.08
1.04
1.25
9.00
10.0
5.0
0.0
ppm (f1)

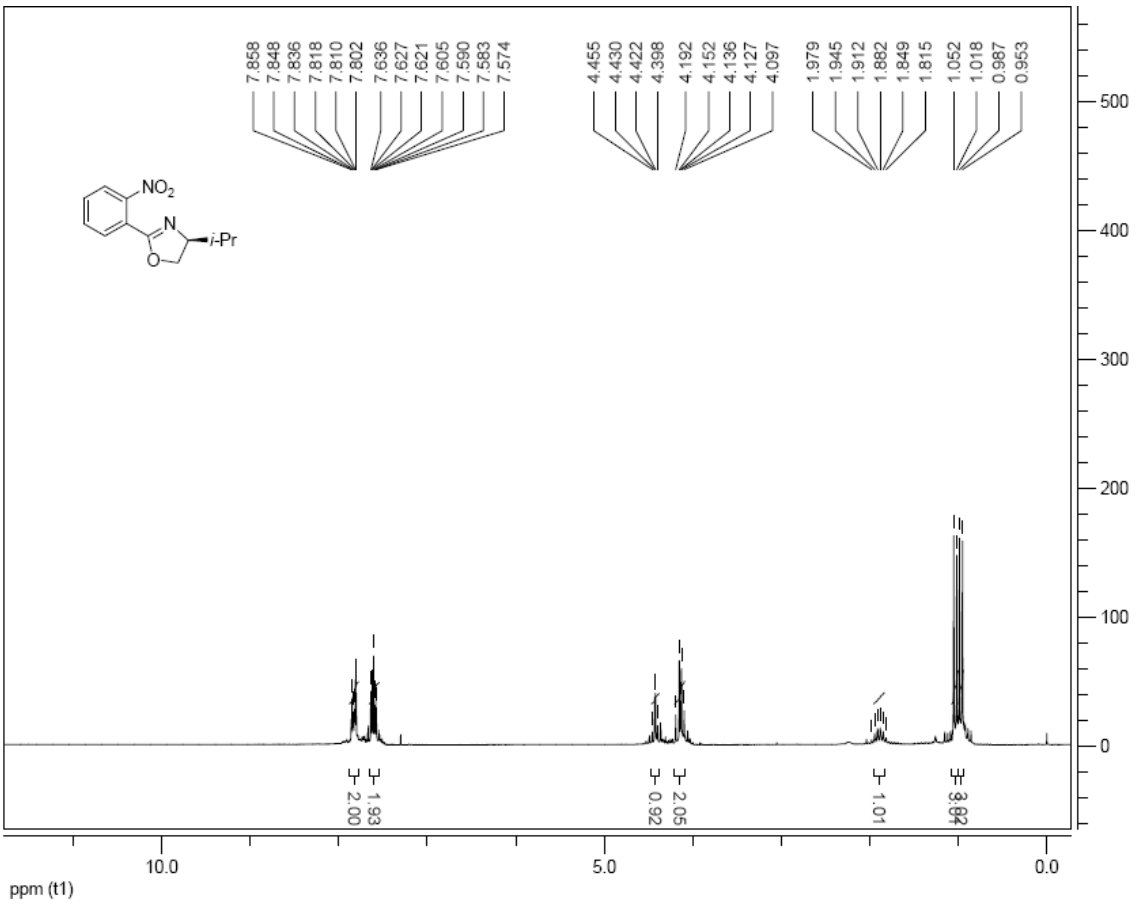

165.591
147.105
133.139
133.109
130.262
129.387
123.693
60.464
59.510
40.260
39.987
39.708
39.430
39.152
38.874
38.594
33.859
26.882
NO2
OH
N
H
O
t-Bu
6000
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

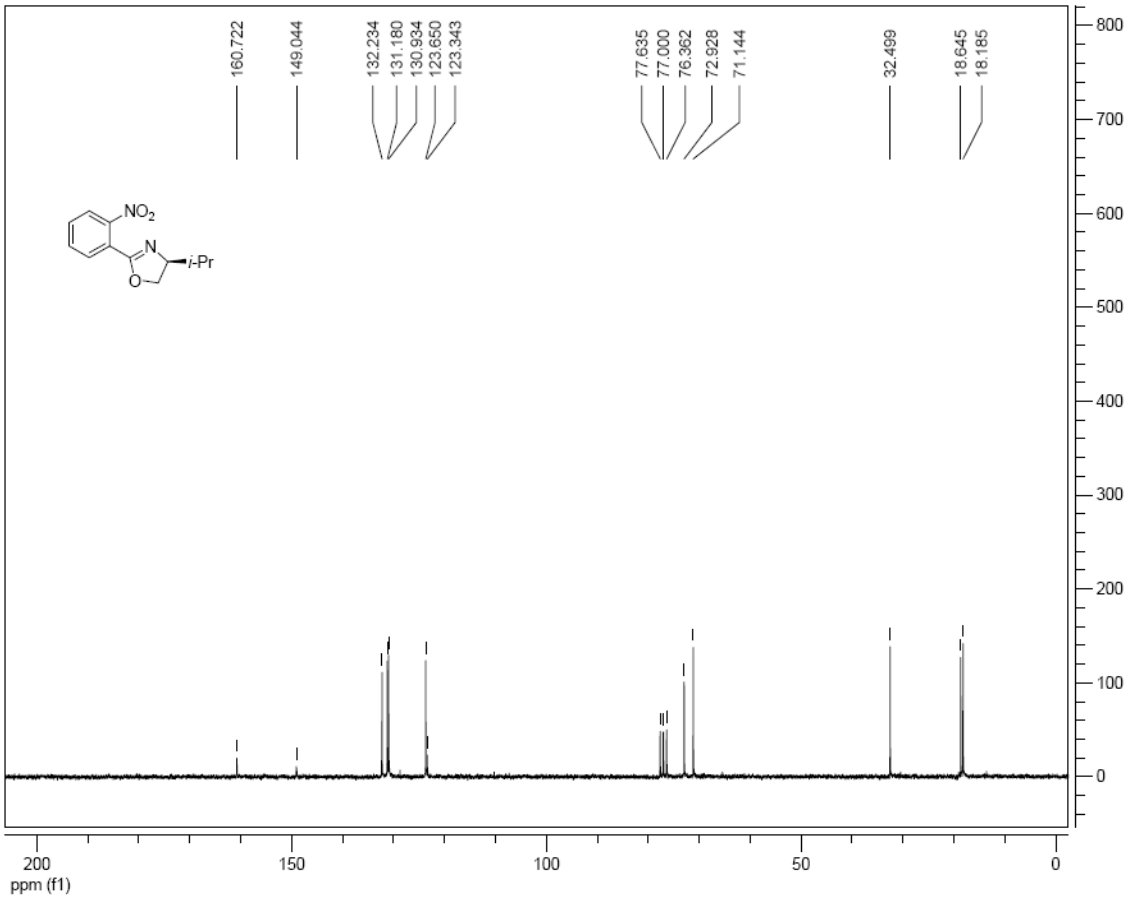

7.925
7.913
7.903
7.892
7.888
7.880
7.870
7.857
7.657
7.646
7.627
7.610
7.597
7.379
7.361
7.337
7.322
7.312
7.291
7.266
5.445
5.400
5.395
5.352
4.848
4.806
4.797
4.755
4.325
4.282
4.239
NO2
N
O
Ph
1.90
2.00
5.04
1.00
1.04
1.06
150
100
50
0
10.0
5.0
0.0
ppm (t1)

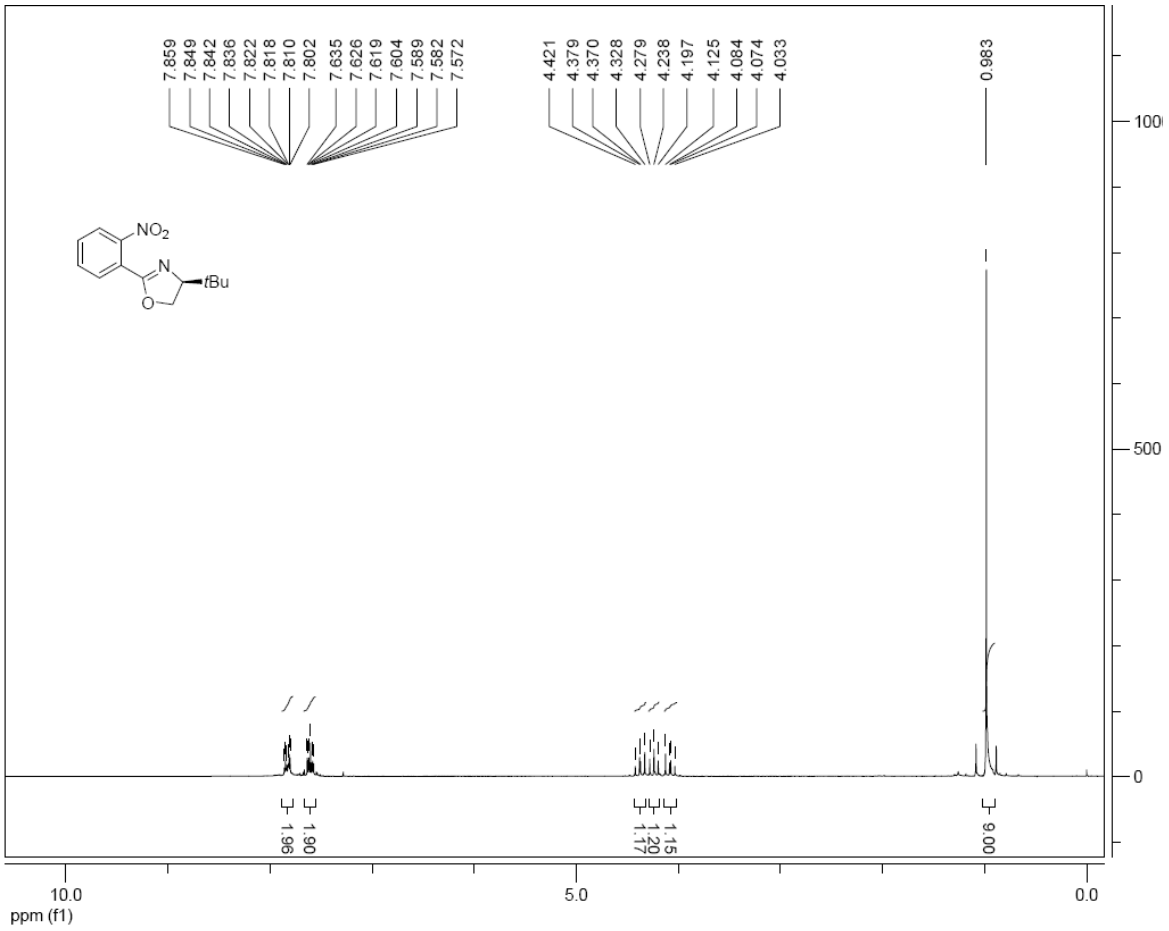

162.282
149.133
141.496
132.471
131.537
131.158
128.747
127.723
126.760
123.876
123.114
77.637
77.000
76.363
75.781
70.402
NO2
N
O
Ph
350
300
250
200
150
100
50
0
200
150
100
50
0
ppm (f1)

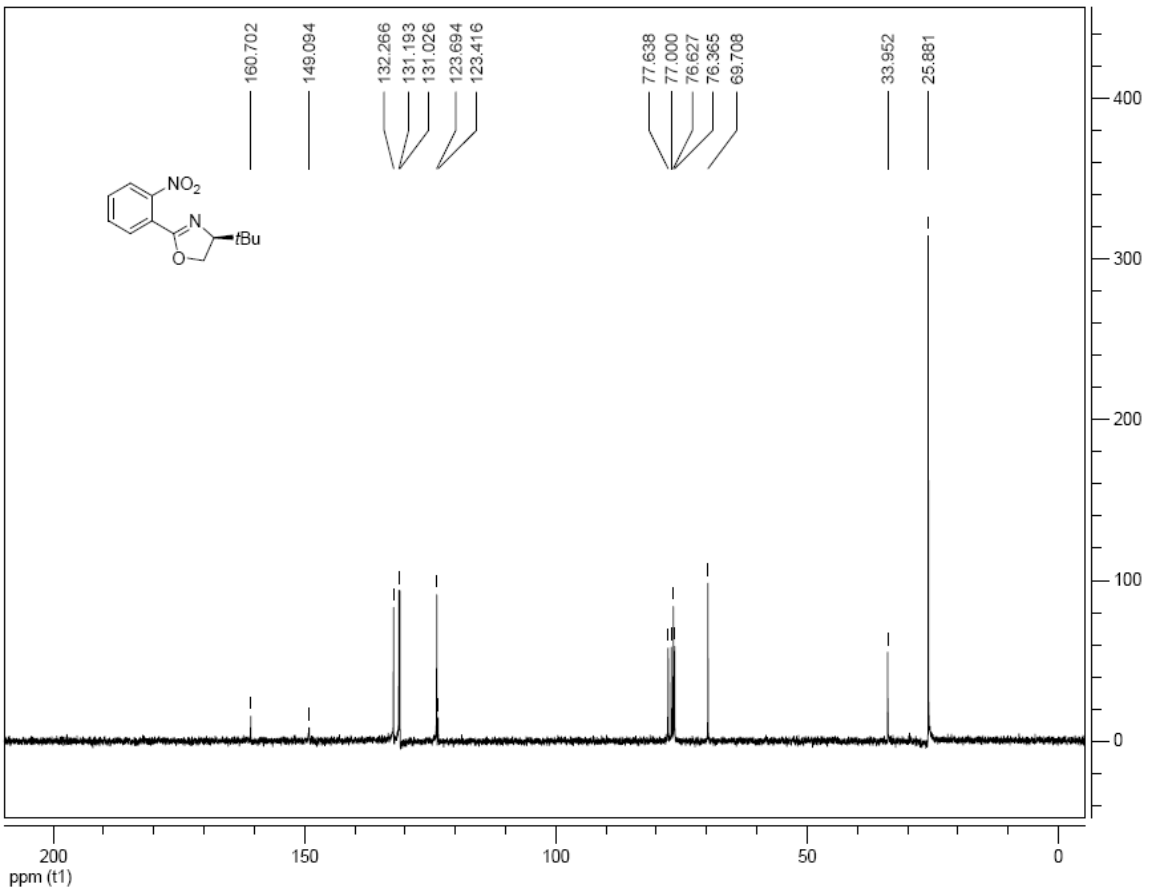

7.865
7.857
7.849
7.832
7.819
7.809
7.798
7.780
7.772
7.764
7.657
7.629
7.619
7.613
7.598
7.583
7.576
7.566
7.321
7.308
7.285
7.268
7.243
7.236
7.222
4.646
4.642
4.638
4.618
4.609
4.600
4.572
4.559
4.532
4.411
4.369
4.323
4.196
4.159
4.154
4.117
3.252
3.224
3.183
3.155
NO2
N
O
Bn
100
50
0
1.61
1.92
5.08
1.00
0.96
1.00
1.09
1.08
10.0
5.0
0.0
ppm (t1)

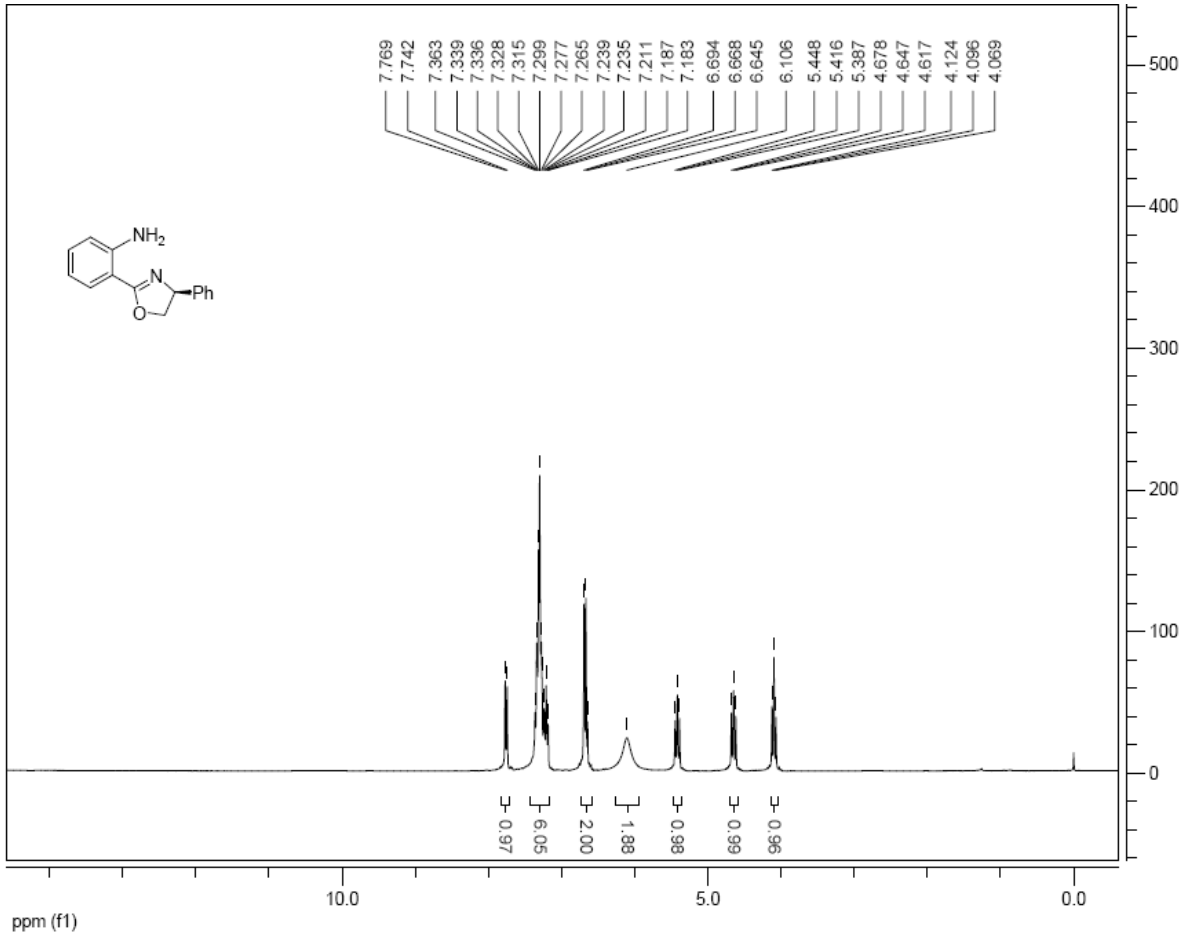

161.332
149.055
137.567
132.324
131.308
130.902
129.203
128.488
126.513
123.767
123.286
77.636
77.000
76.362
72.825
68.127
41.221
NO2
N
O
Bn
350
300
250
200
150
100
50
0
200
150
100
50
0
ppm (t1)

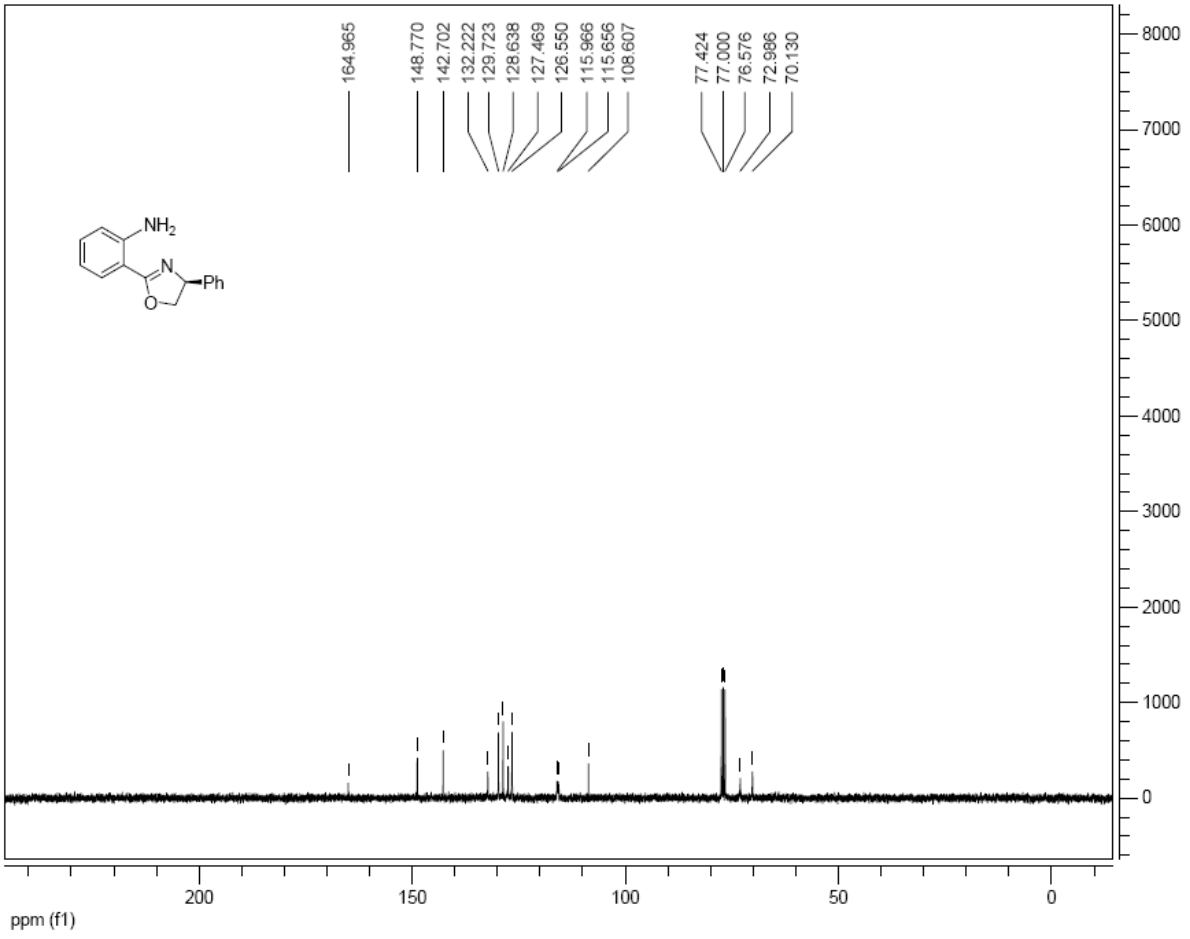

7.858
7.848
7.836
7.818
7.810
7.802
7.636
7.627
7.621
7.605
7.590
7.583
7.574
4.455
4.430
4.422
4.398
4.192
4.152
4.136
4.127
4.097
1.979
1.945
1.912
1.882
1.849
1.815
1.052
1.018
0.987
0.953
NO2
N
O
i-Pr
2.00
1.93
0.92
2.05
1.01
3.82
500
400
300
200
100
0
10.0
5.0
0.0
ppm (t1)

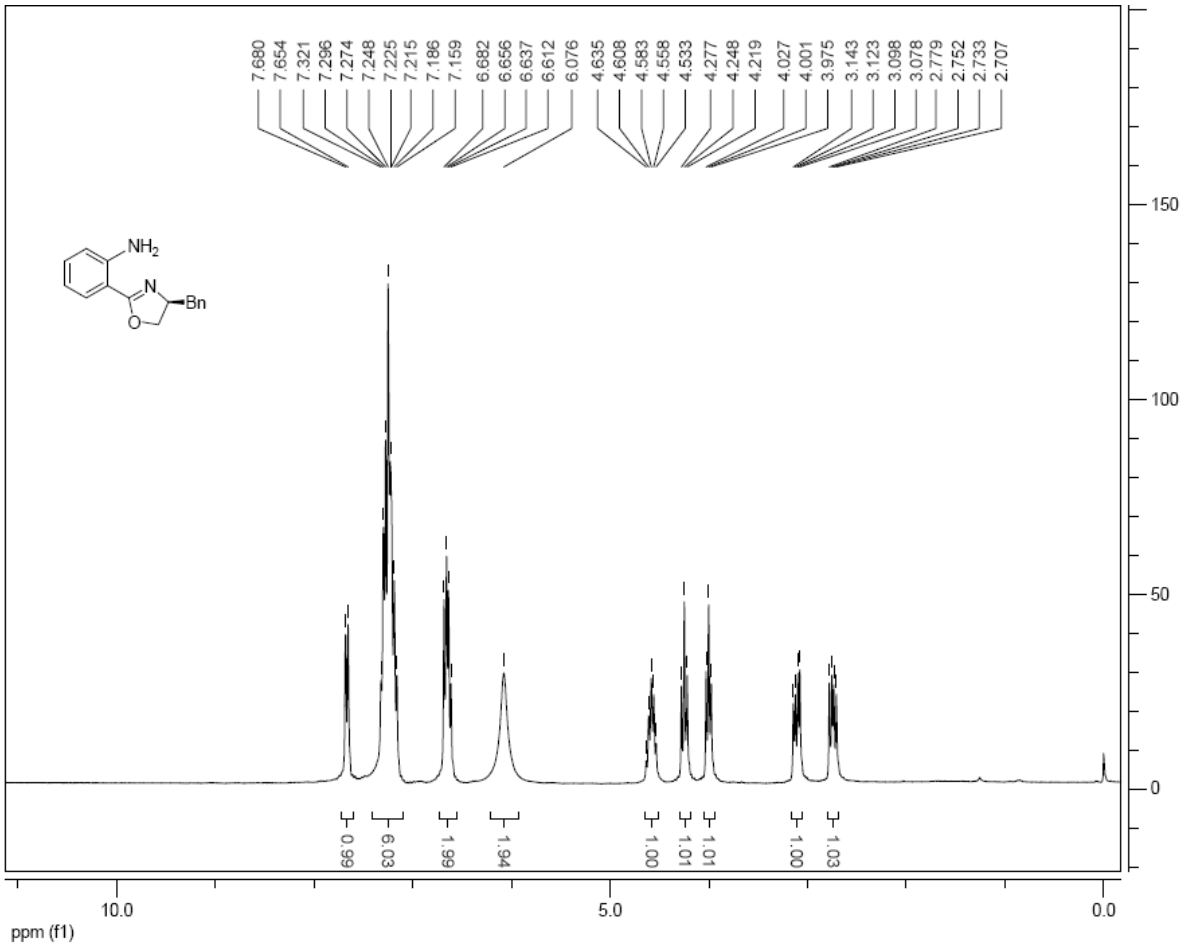

160.722
149.044
132.234
131.180
130.934
123.650
123.343
77.635
77.000
76.362
72.928
71.144
32.499
18.645
18.185
NO2
N
O
i-Pr
800
700
600
500
400
300
200
100
0
200
150
100
50
0
ppm (f1)

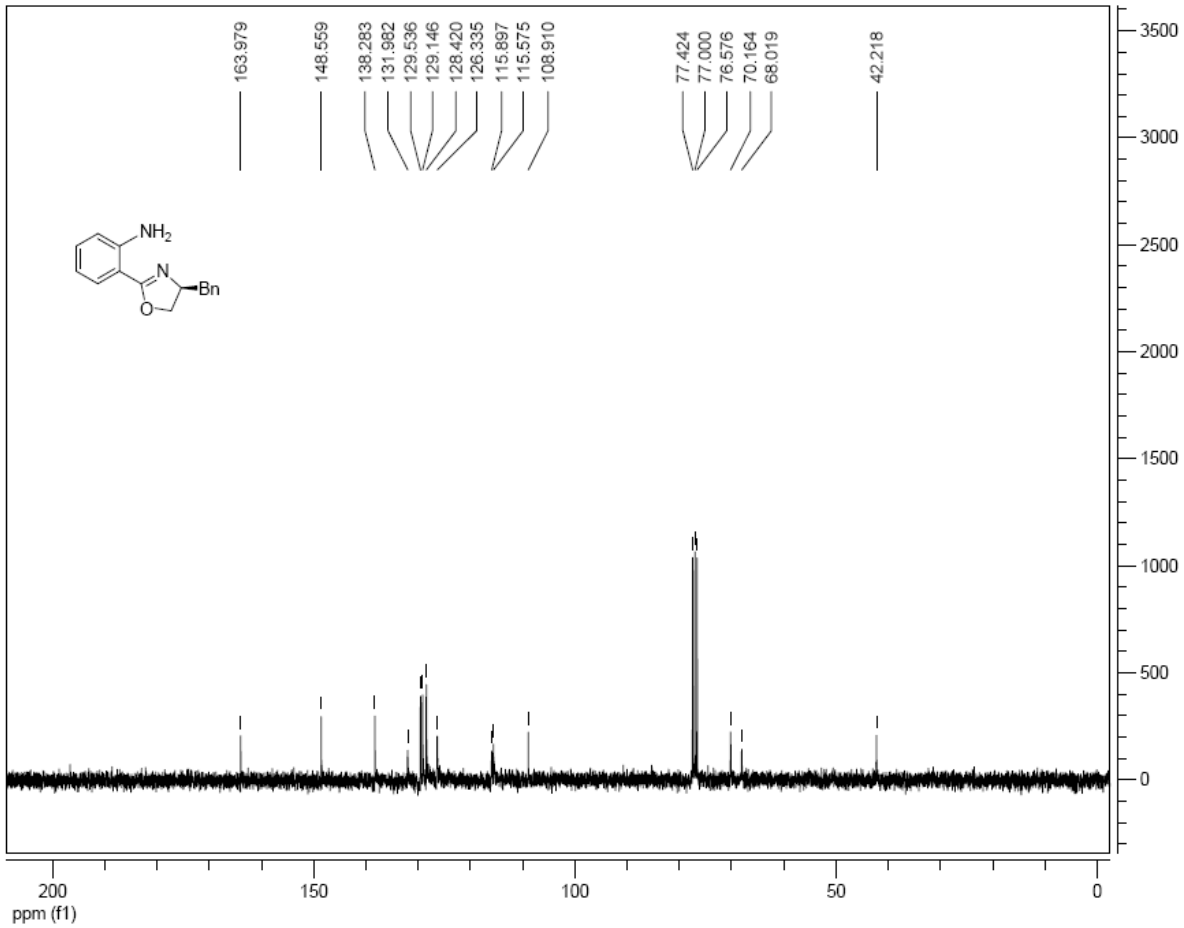

7.859
7.849
7.842
7.836
7.822
7.818
7.810
7.802
7.635
7.626
7.619
7.604
7.589
7.582
7.572
4.421
4.379
4.370
4.328
4.279
4.238
4.197
4.125
4.084
4.074
4.033
0.983
NO2
N
O
tBu
1.96
1.90
1.70
1.20
1.15
9.00
10.0
5.0
0.0
ppm (f1)

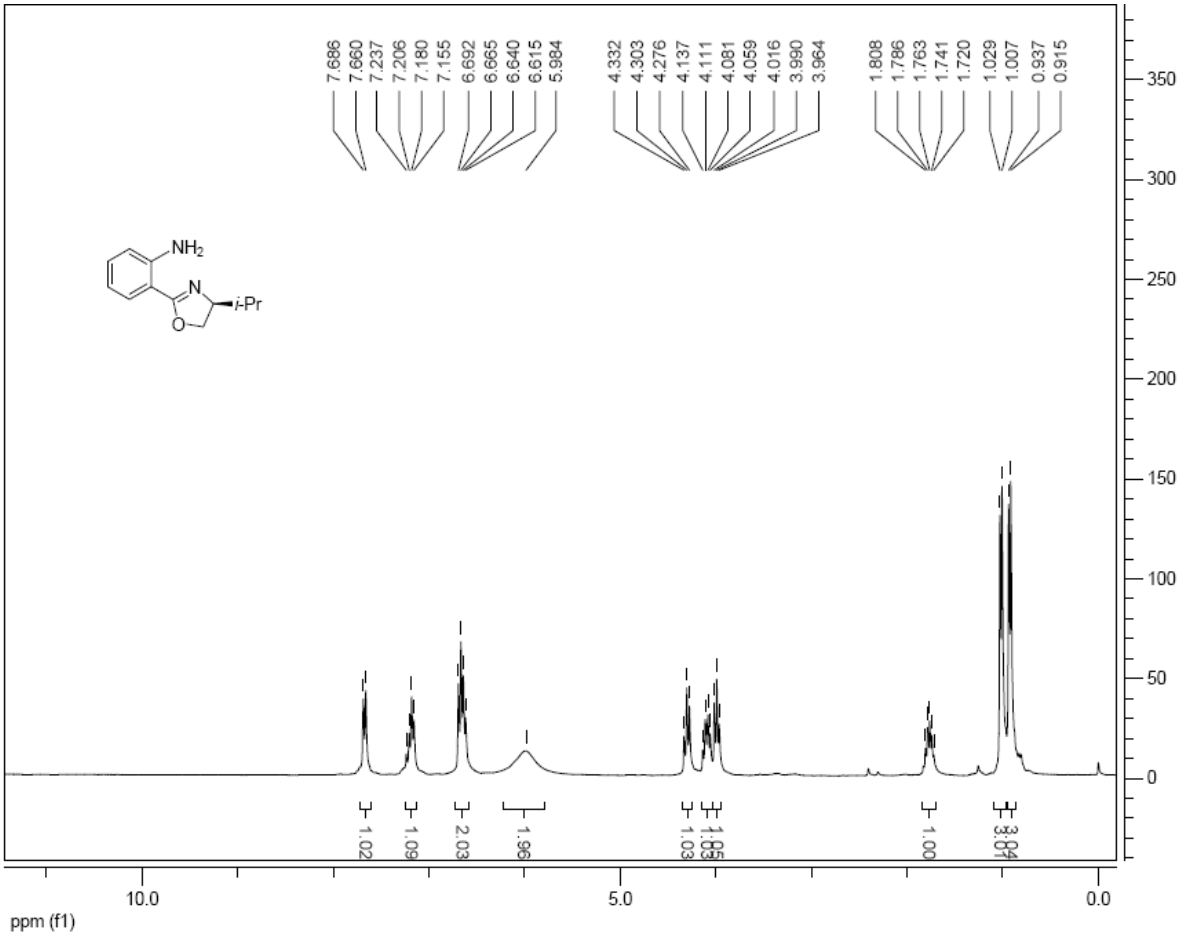

160.702
149.094
132.266
131.193
131.026
123.694
123.416
77.638
77.000
76.627
76.365
69.708
33.952
25.881
NO2
N
O
tBu
200
150
100
50
0
ppm (t1)

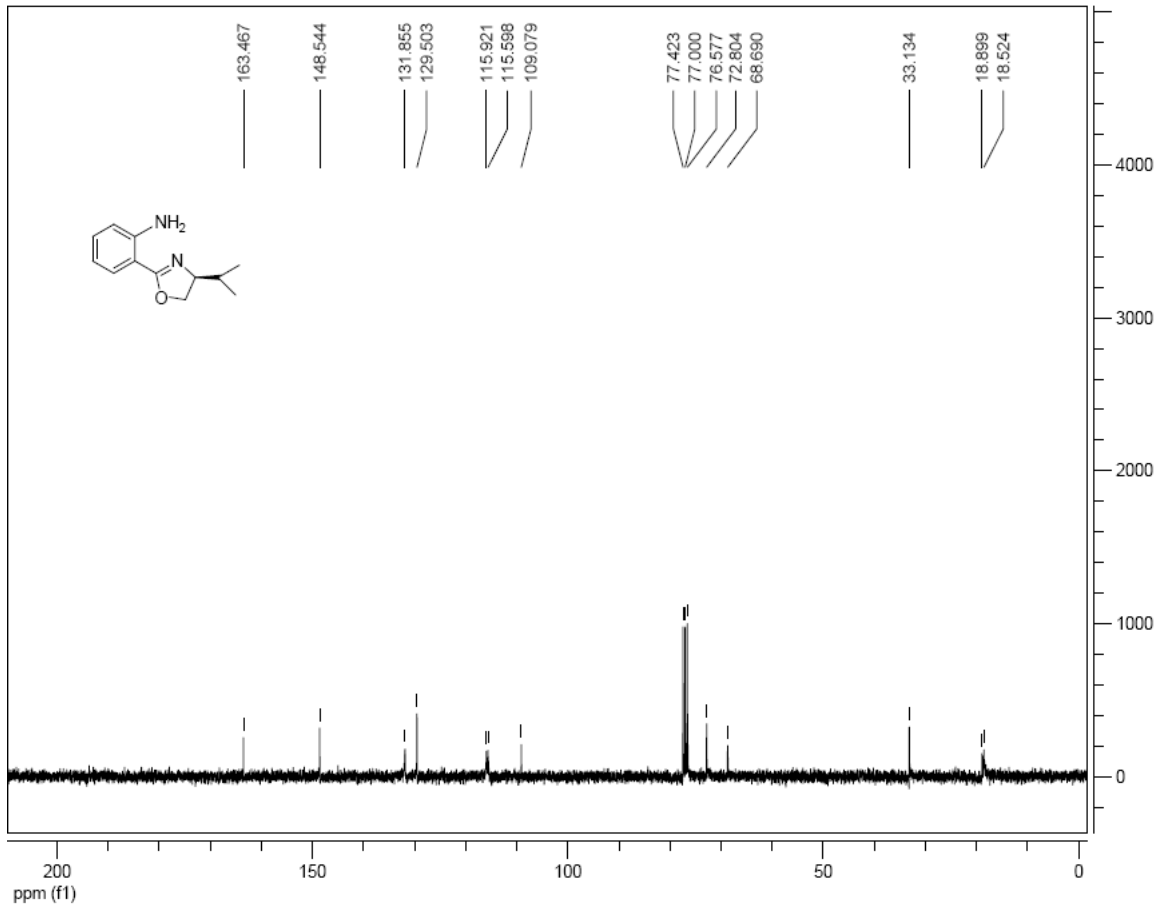

7.769
7.742
7.363
7.339
7.336
7.328
7.315
7.299
7.277
7.265
7.239
7.235
7.211
7.187
7.183
6.694
6.668
6.645
6.106
5.448
5.416
5.387
4.678
4.647
4.617
4.124
4.096
4.069
NH2
N
O
Ph
0.97
6.05
2.00
1.88
0.98
0.99
0.96
500
400
300
200
100
0
10.0
5.0
0.0
ppm (f1)

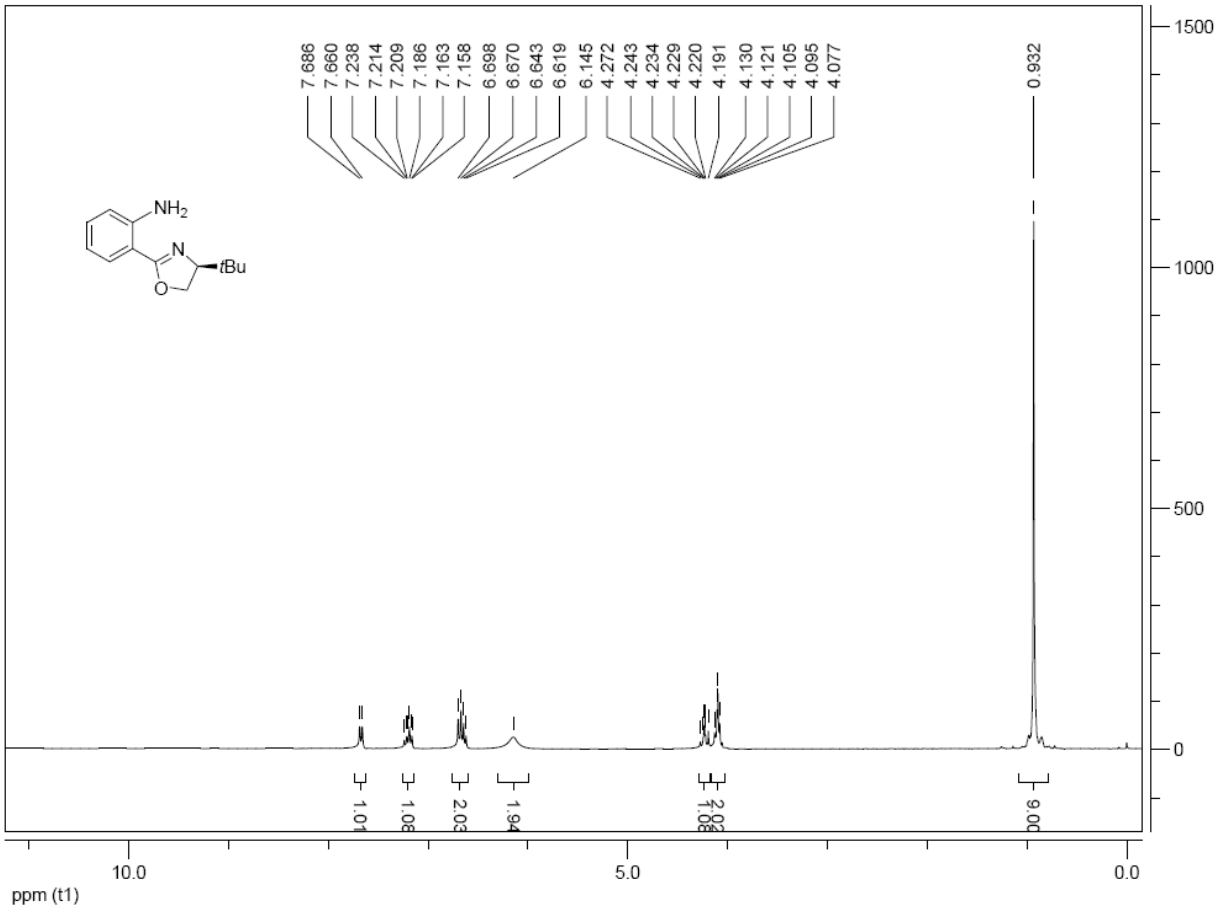

164.965
148.770
142.702
132.222
129.723
128.638
127.469
126.550
115.966
115.656
108.607
77.424
77.000
76.576
72.986
70.130
NH2
N
O
Ph
8000
7000
6000
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

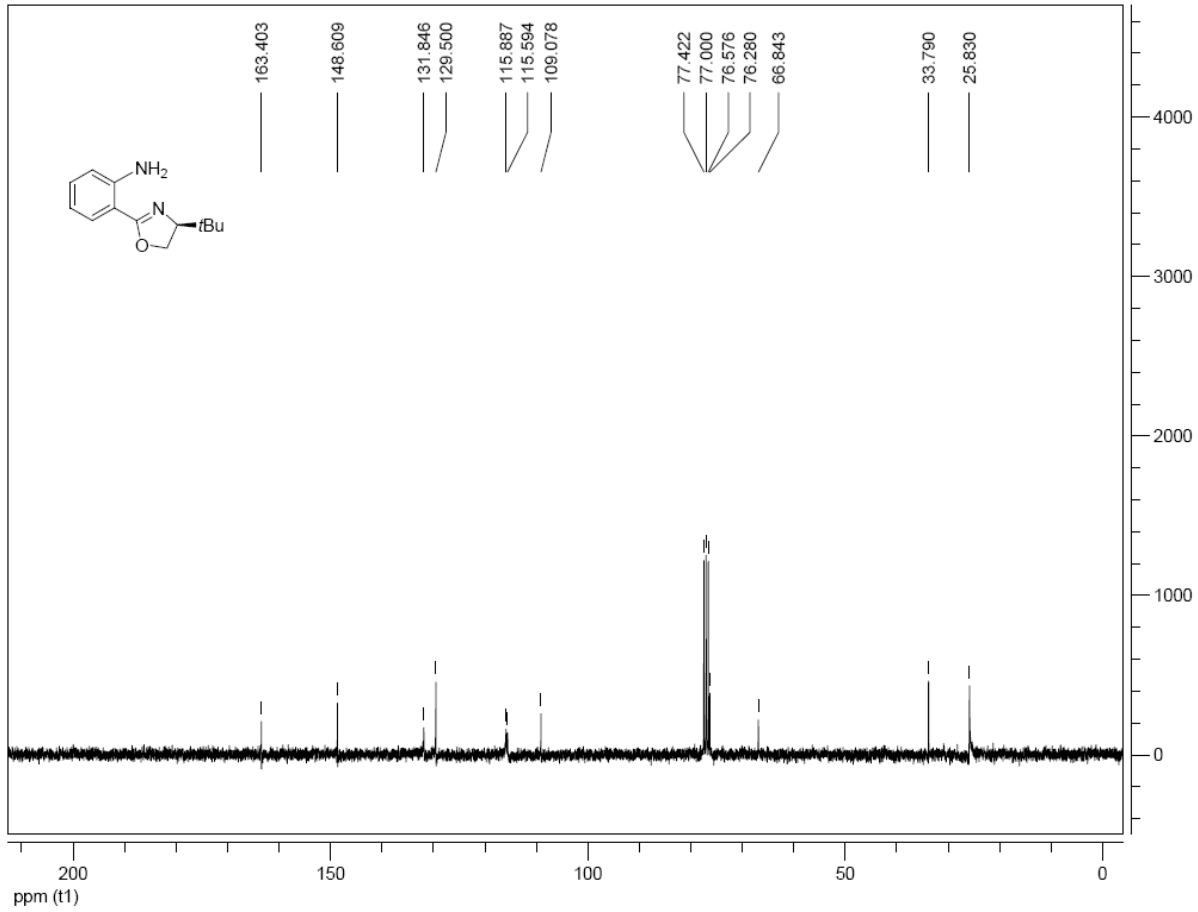

7.680
7.654
7.321
7.296
7.274
7.248
7.225
7.215
7.186
7.159
6.682
6.656
6.637
6.612
6.076
4.635
4.608
4.583
4.558
4.533
4.277
4.248
4.219
4.027
4.001
3.975
3.143
3.123
3.098
3.078
2.779
2.752
2.733
2.707
NH2
N
O
Bn
0.99
6.03
1.99
1.94
1.00
1.01
1.01
1.00
1.03
150
100
50
0
10.0
5.0
0.0
ppm (f1)

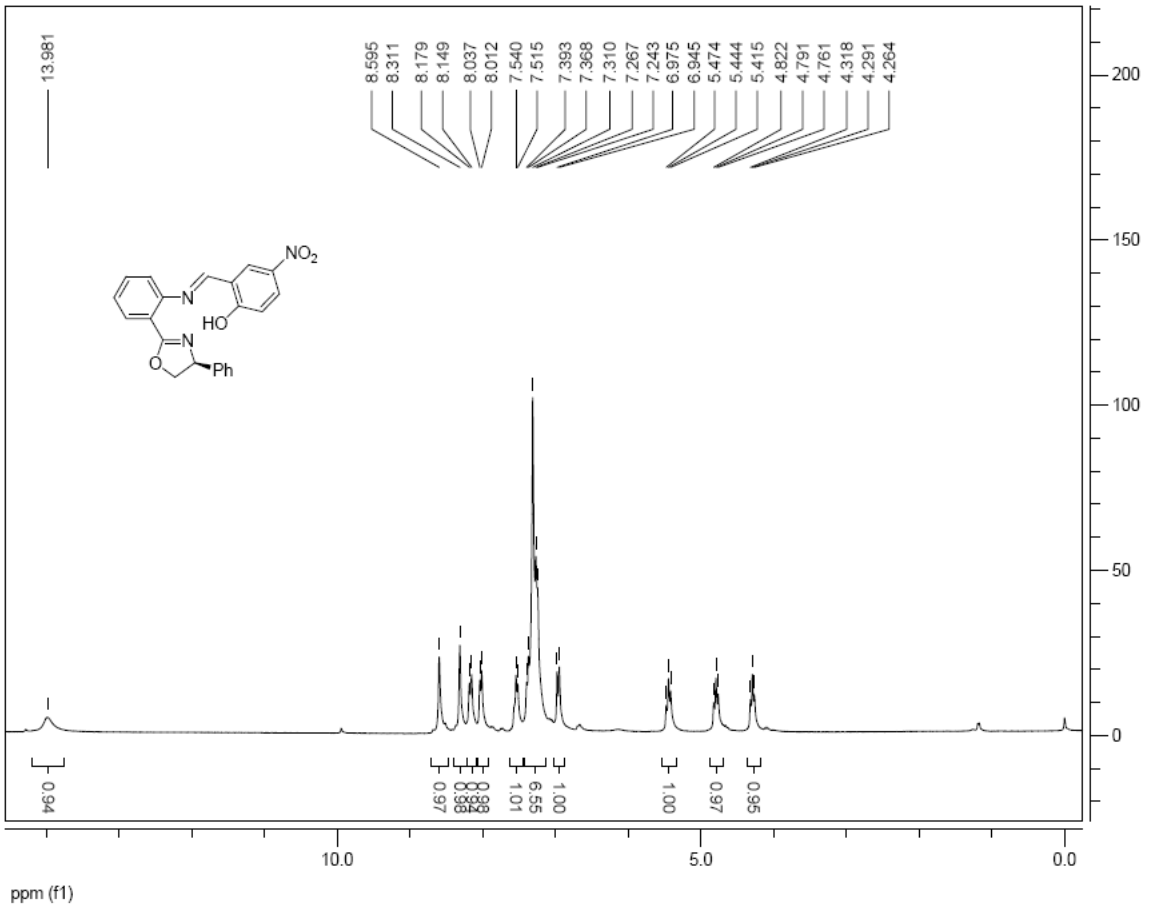

163.979
148.559
138.283
131.982
129.536
129.146
128.420
126.335
115.897
115.575
108.910
77.424
77.000
76.576
70.164
68.019
42.218
NH2
N
O
Bn
3500
3000
2500
2000
1500
1000
500
0
200
150
100
50
0
ppm (f1)

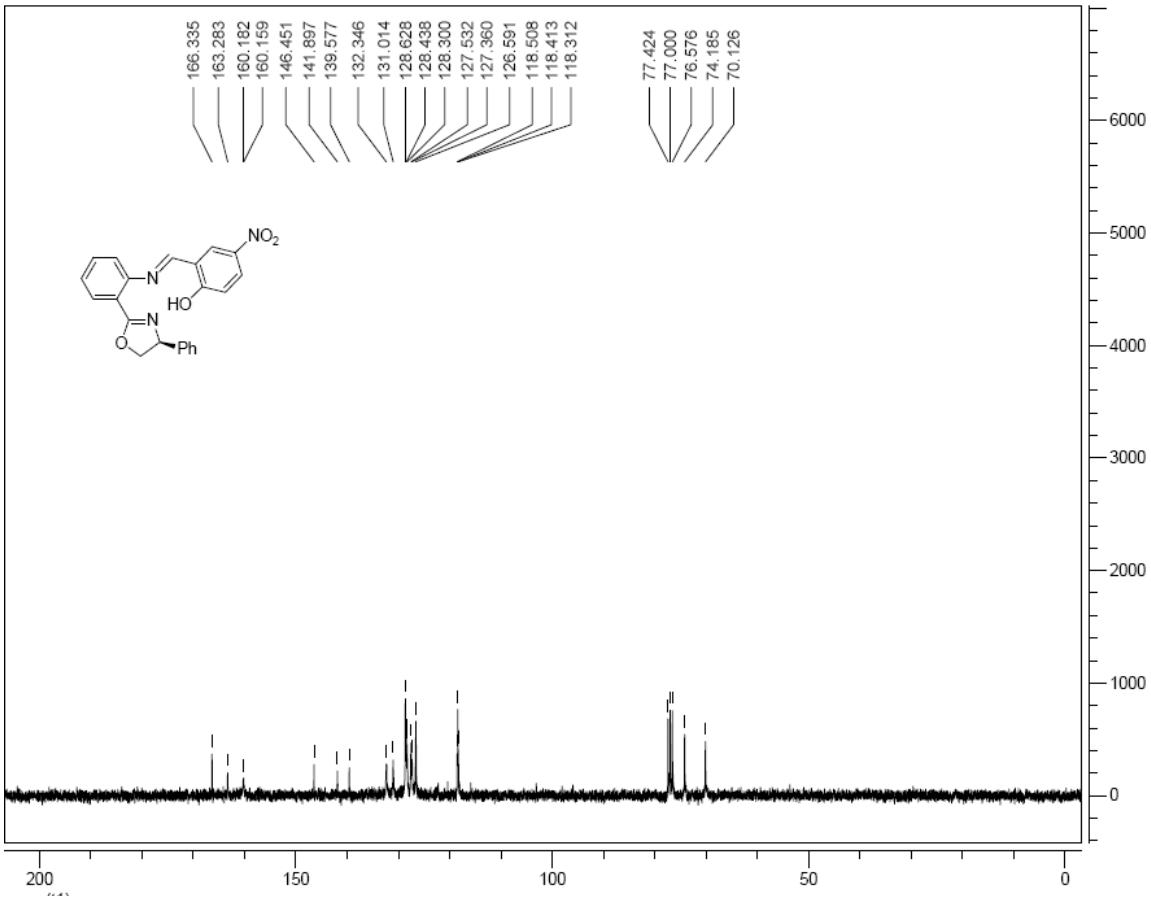

7.686
7.660
7.237
7.206
7.180
7.155
6.692
6.665
6.640
6.615
5.984
4.332
4.303
4.276
4.137
4.111
4.081
4.059
4.016
3.990
3.964
1.808
1.786
1.763
1.741
1.720
1.029
1.007
0.937
0.915
NH2
N
O
i-Pr
1.02
1.09
2.03
1.96
1.03
1.00
3.04
10.0
5.0
0.0
ppm (f1)

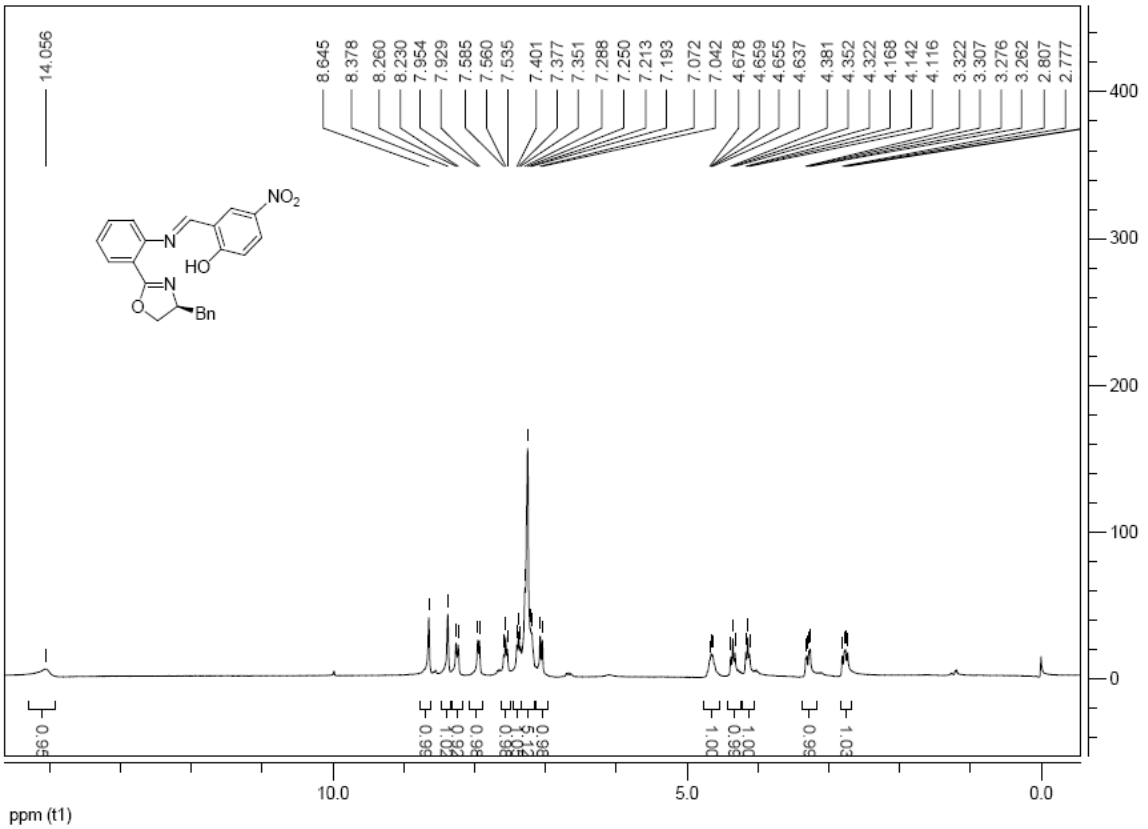

163.467
148.544
131.855
129.503
115.921
115.598
109.079
77.423
77.000
76.577
72.804
68.690
33.134
18.899
18.524
NH2
N
O
200
150
100
50
0
ppm (f1)

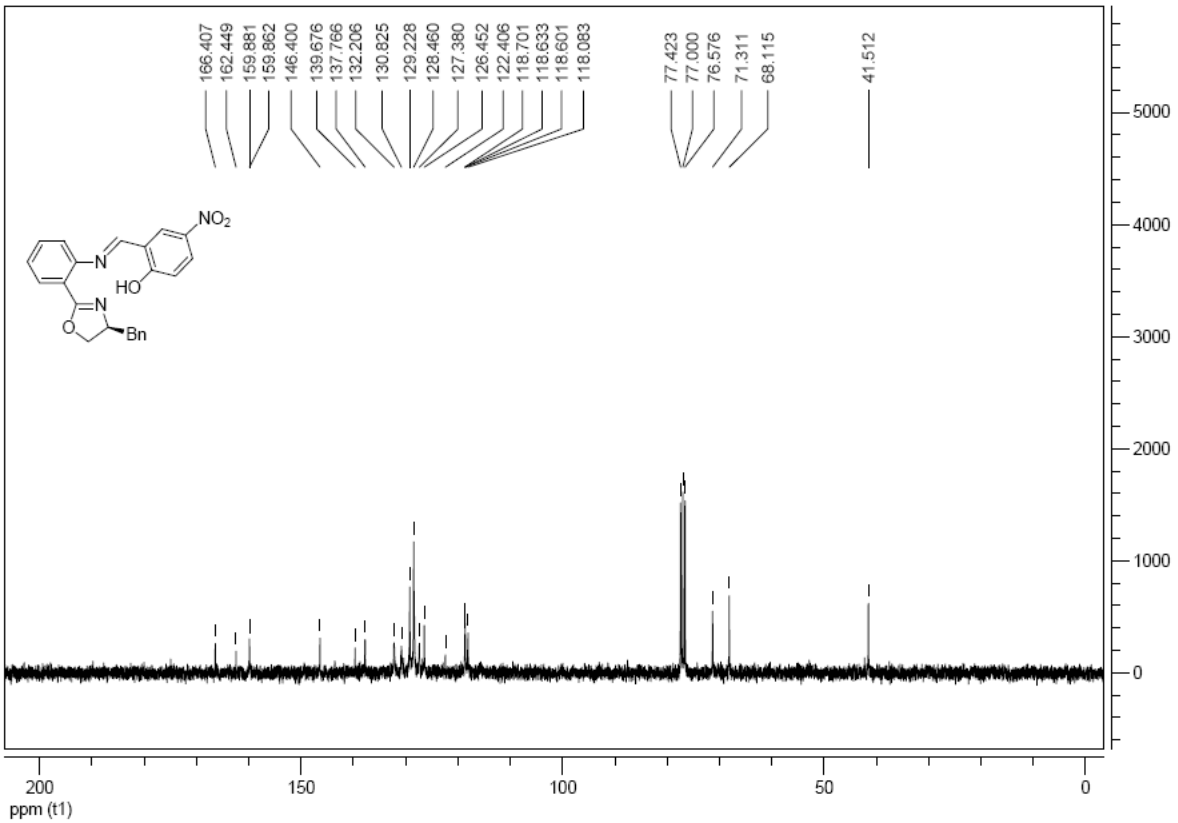

7.686
7.660
7.238
7.214
7.209
7.186
7.163
7.158
6.698
6.670
6.643
6.619
6.145
4.272
4.243
4.234
4.229
4.220
4.191
4.130
4.121
4.105
4.095
4.077
0.932
NH2
N
O
tBu
1.01
1.08
2.03
1.94
2.08
2.00
9.00
1500
1000
500
0
10.0
5.0
0.0
ppm (t1)

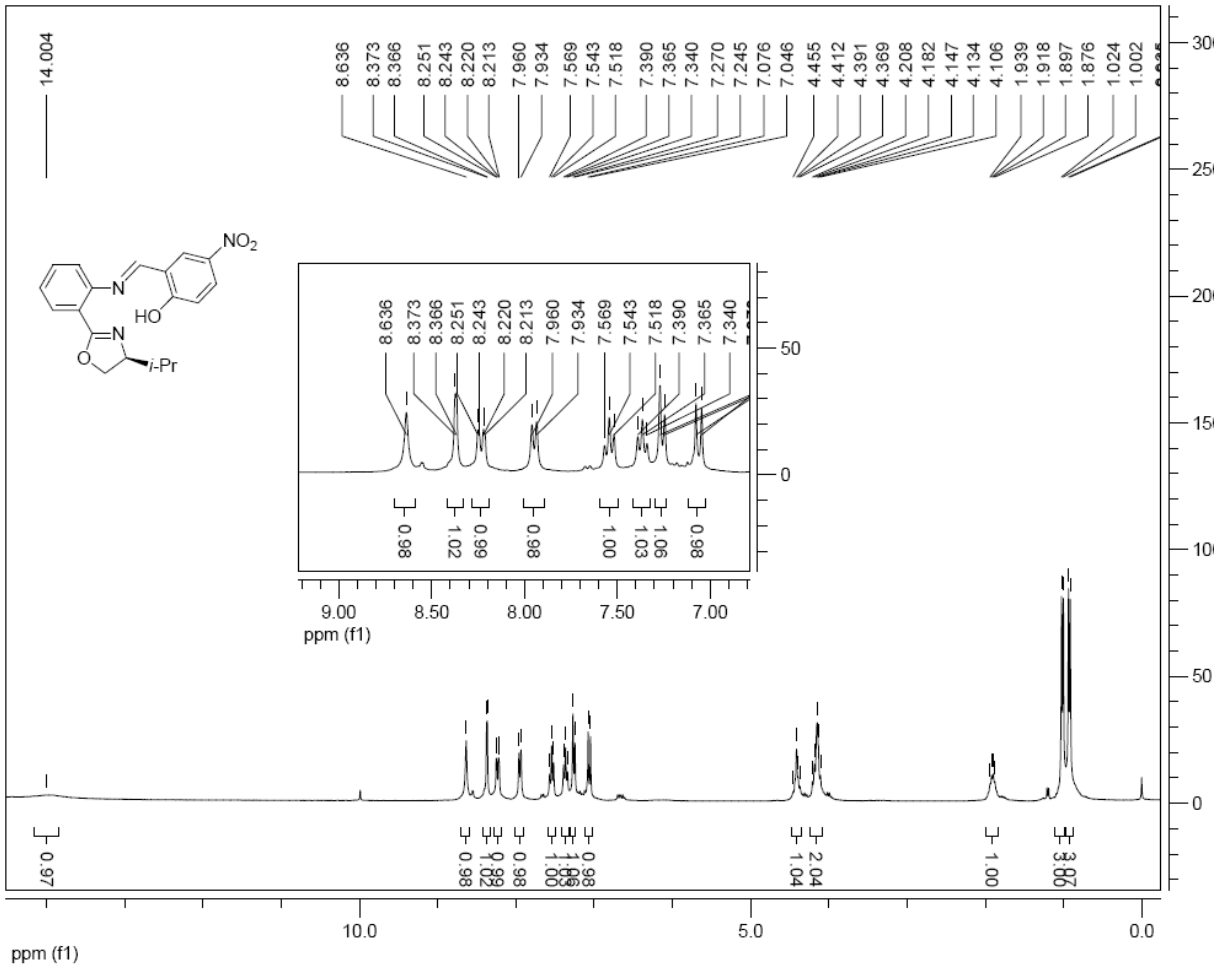

163.403
148.609
131.846
129.500
115.887
115.594
109.078
77.422
77.000
76.576
76.280
66.843
33.790
25.830
NH2
N
O
tBu
4000
3000
2000
1000
0
200
150
100
50
0
ppm (t1)

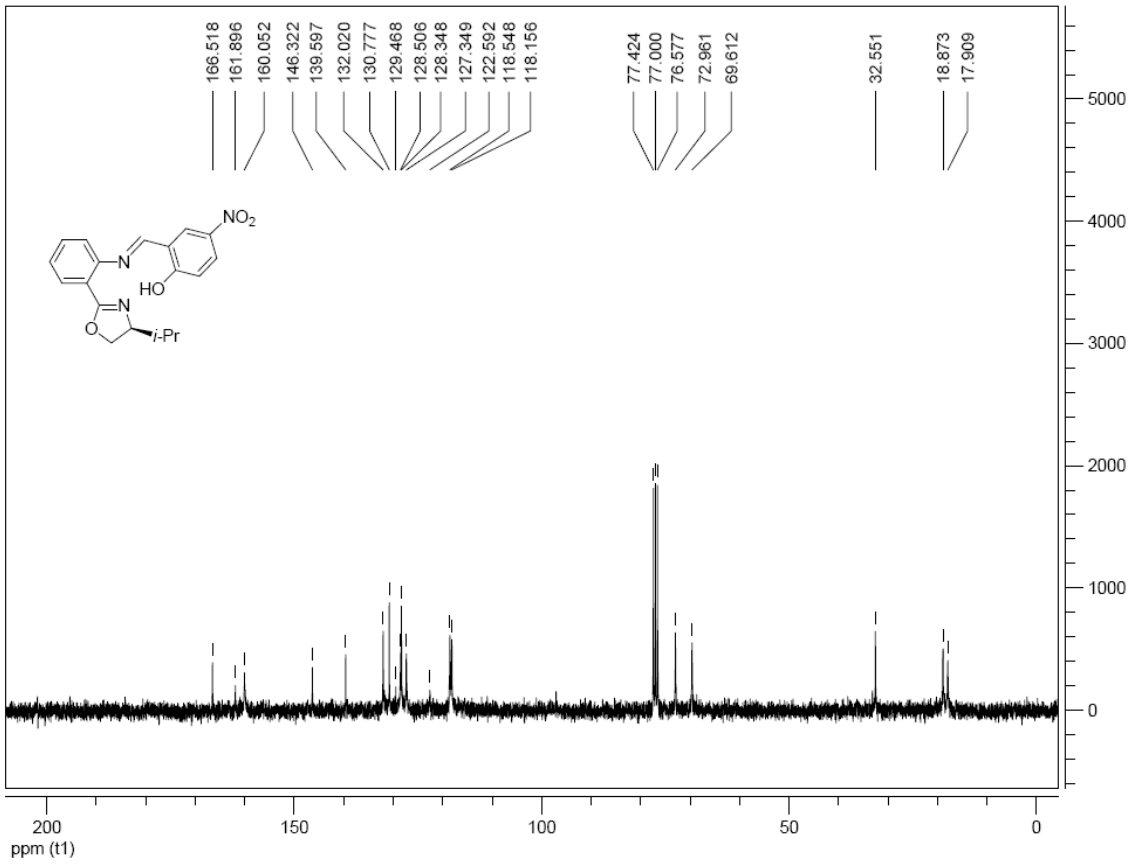

13.981
8.595
8.311
8.179
8.149
8.037
8.012
7.540
7.515
7.393
7.368
7.310
7.267
7.243
6.975
6.945
5.474
5.444
5.415
4.822
4.791
4.761
4.318
4.291
4.264
NO2
HO
N
O
Ph
0.94
0.97
1.07
6.55
1.00
1.00
0.97
0.96
200
150
100
50
0
10.0
5.0
0.0
ppm (f1)

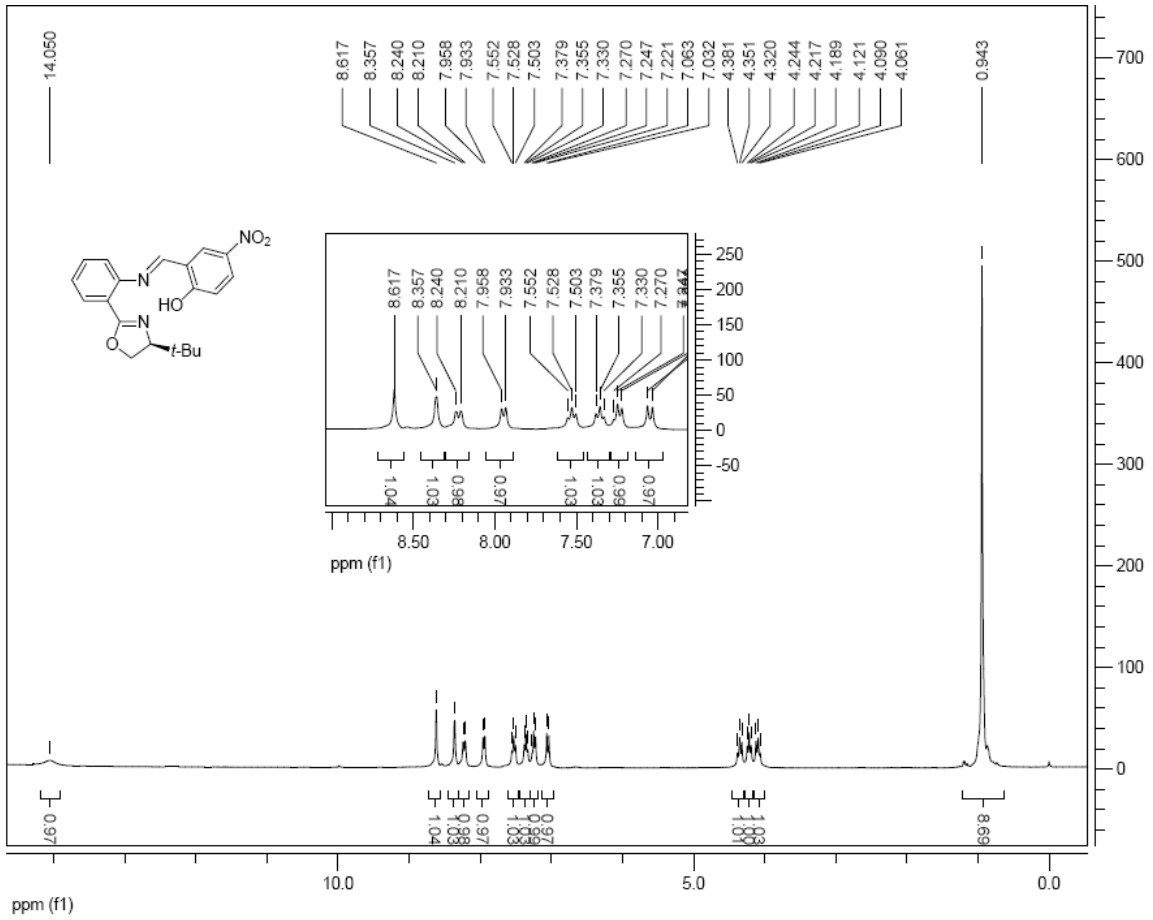

166.335
163.283
160.182
160.159
146.451
141.897
139.577
132.346
131.014
128.628
128.438
128.300
127.532
127.360
126.591
118.508
118.413
118.312
77.424
77.000
76.576
74.185
70.126
NO2
HO
N
O
Ph
6000
5000
4000
3000
2000
1000
0
200
150
100
50
0

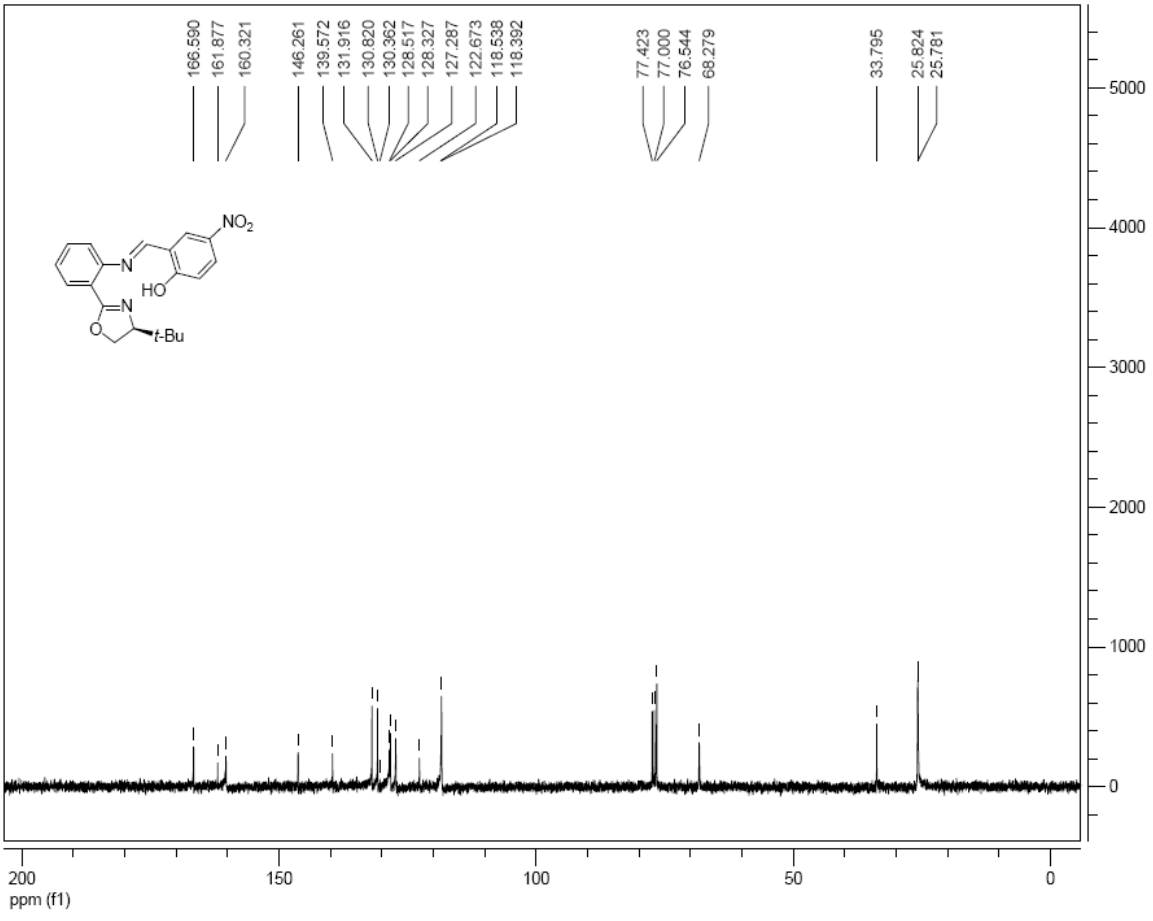

14.056
8.645
8.378
8.260
8.230
7.954
7.929
7.585
7.560
7.535
7.401
7.377
7.351
7.288
7.250
7.213
7.193
7.072
7.042
4.678
4.659
4.655
4.637
4.381
4.352
4.322
4.168
4.142
4.116
3.322
3.307
3.276
3.262
2.807
2.777
NO2
HO
N
O
Bn
400
300
200
100
0
10.0
5.0
0.0
ppm (t1)

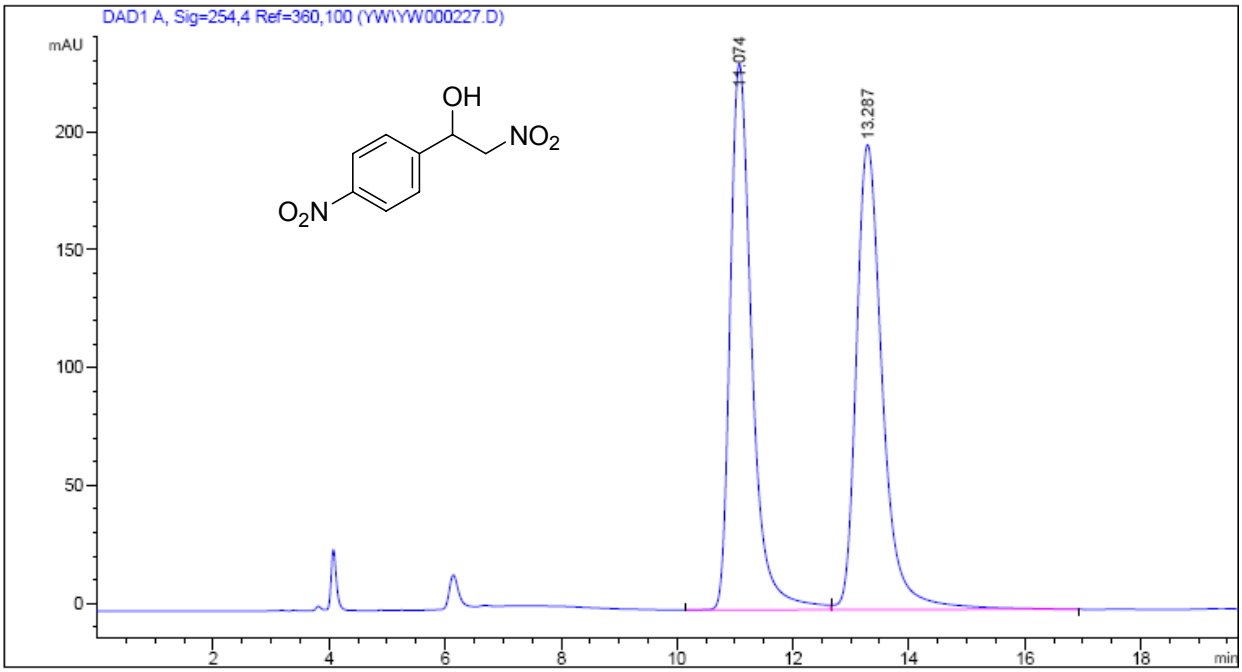

166.407
162.449
159.881
159.862
146.400
139.676
137.766
132.206
130.825
129.228
128.460
127.380
126.452
122.406
118.701
118.633
118.601
118.083
77.423
77.000
76.576
71.311
68.115
41.512
NO2
HO
N
O
Bn
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (t1)


14.004
8.636
8.373
8.366
8.251
8.243
8.220
8.213
7.960
7.934
7.569
7.543
7.518
7.390
7.365
7.340
7.270
7.245
7.076
7.046
4.455
4.412
4.391
4.369
4.208
4.182
4.147
4.134
4.106
1.939
1.918
1.897
1.876
1.024
1.002
NO2
HO
N
N
O
i-Pr
0.98
1.02
0.99
0.98
1.00
1.03
1.06
0.98
9.00
8.50
8.00
7.50
7.00
ppm (f1)
0.97
0.98
1.04
2.04
1.00
3.06
10.0
5.0
0.0
ppm (f1)

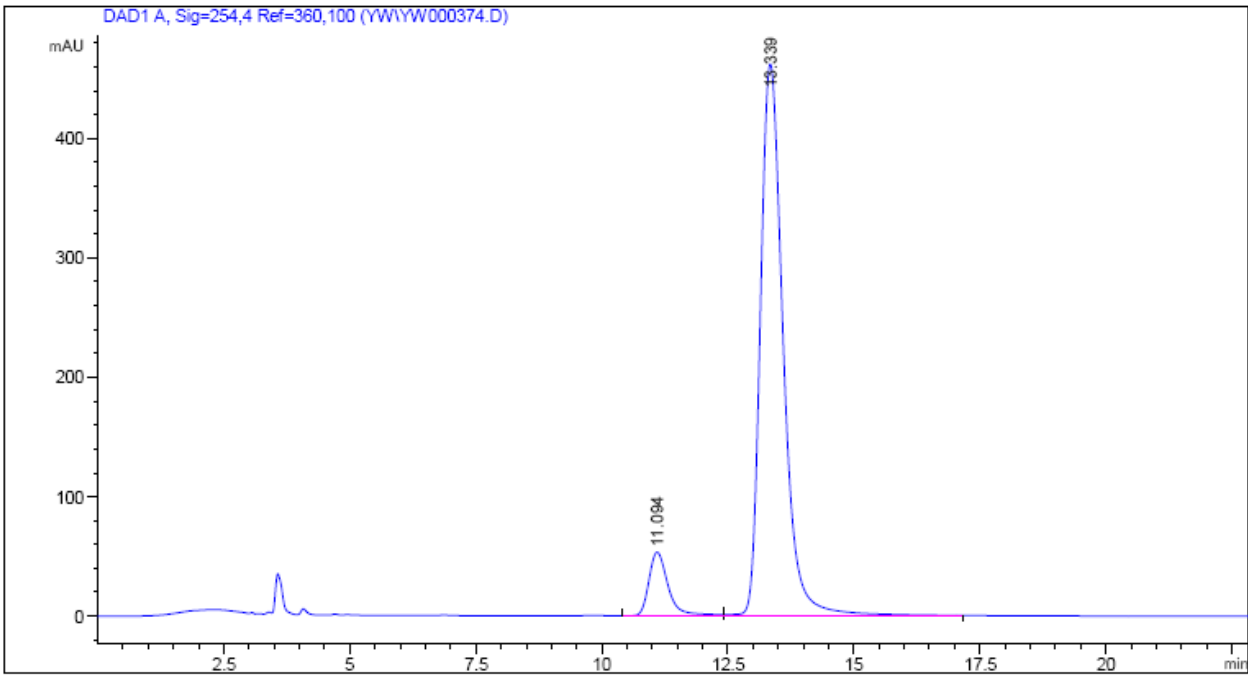

166.518
161.896
160.052
146.322
139.597
132.020
130.777
129.468
128.506
128.348
127.349
122.592
118.548
118.156
77.424
77.000
76.577
72.961
69.612
32.551
18.873
17.909
NO2
HO
N
N
O
i-Pr
200
150
100
50
0
ppm (t1)


14.050
8.617
8.357
8.240
8.210
7.958
7.933
7.552
7.528
7.503
7.379
7.355
7.330
7.270
7.247
7.221
7.063
7.032
4.381
4.351
4.320
4.244
4.217
4.189
4.121
4.090
4.061
0.943
NO2
HO
N
O
t-Bu
ppm (f1)
10.0
5.0
0.0

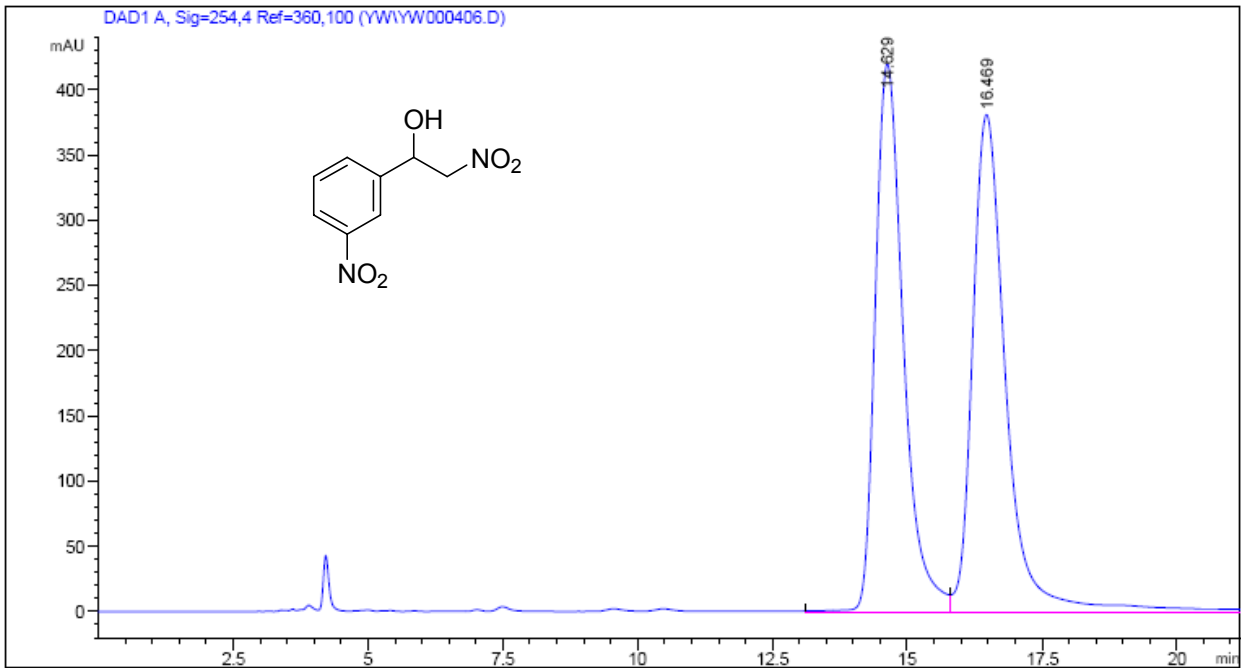

166.590
161.877
160.321
146.261
139.572
131.916
130.820
130.362
128.517
128.327
127.287
122.673
118.538
118.392
77.423
77.000
76.544
68.279
33.795
25.824
25.781
NO2
HO
N
O
t-Bu
ppm (f1)
200
150
100
50
0



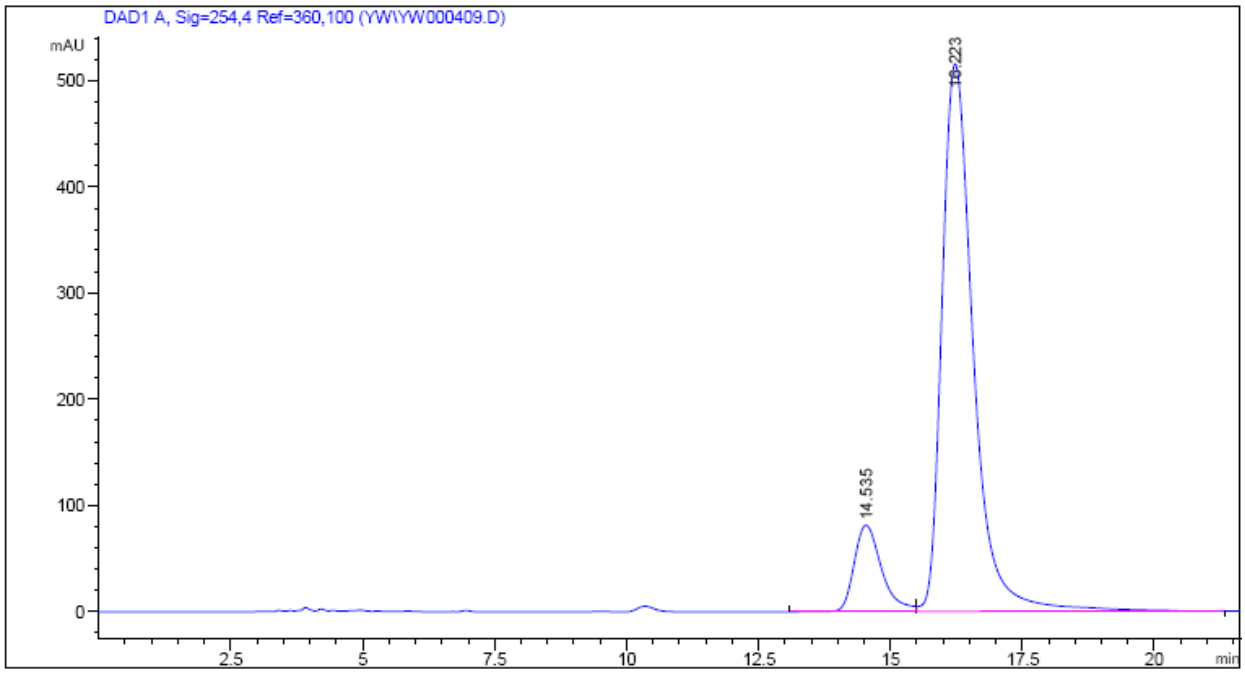
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.074	VV	0.3962	6041.87549	231.73190	49.3063
2	13.287	VB	0.4836	6211.89648	197.17511	50.6937



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.094	VV	0.4060	1413.33569	53.18480	8.8870
2	13.339	VB	0.4861	1.44902e4	461.74069	91.1130

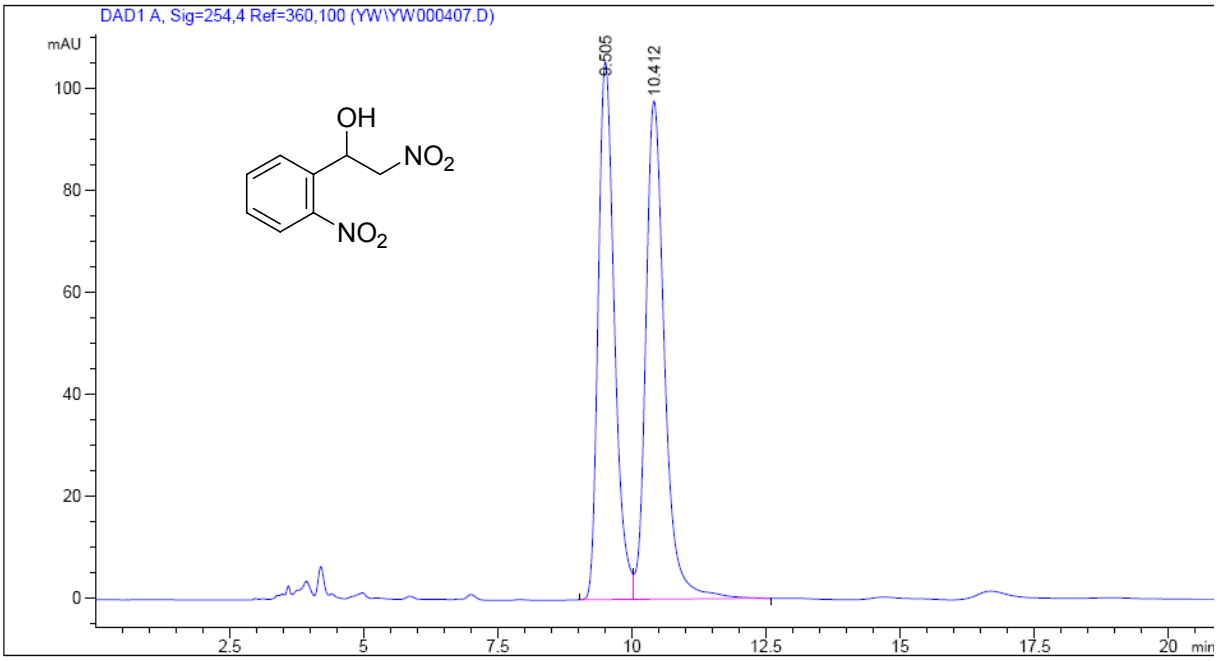


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	14.629	BV	0.5591	1.52261e4	419.84897	47.9883
2	16.469	VBA	0.6572	1.65027e4	381.08875	52.0117



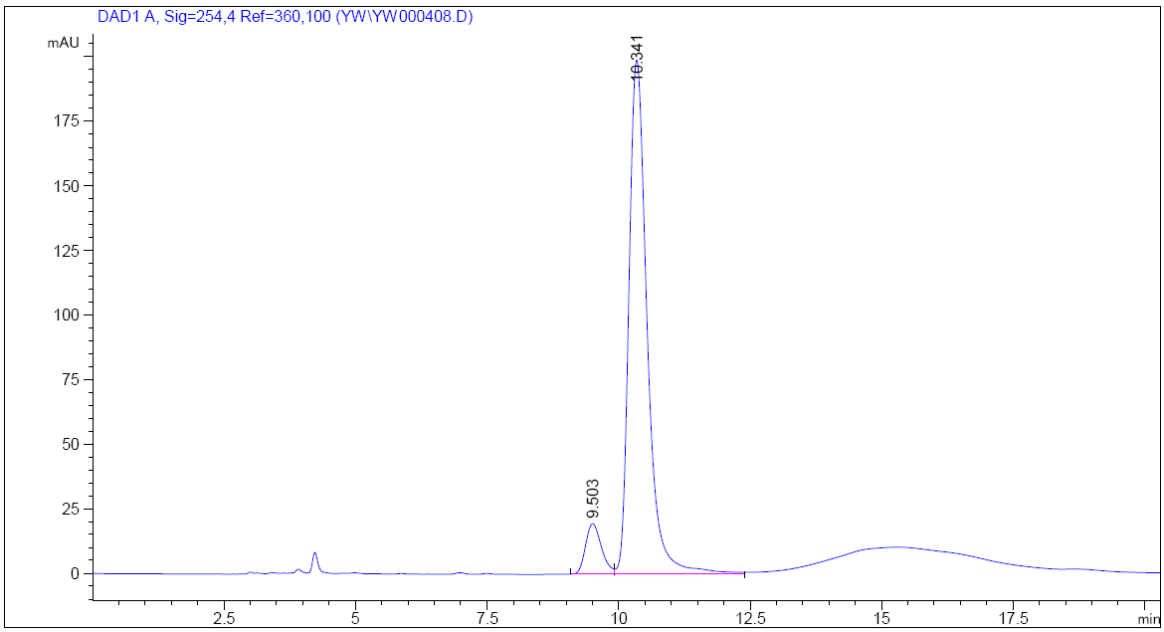
信号 1: DAD1 A, Sig=254,4 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	14.535	BV	0.5551	2953.59473	81.44023	12.1638
2	16.223	VB	0.6345	2.13283e4	515.68896	87.8362



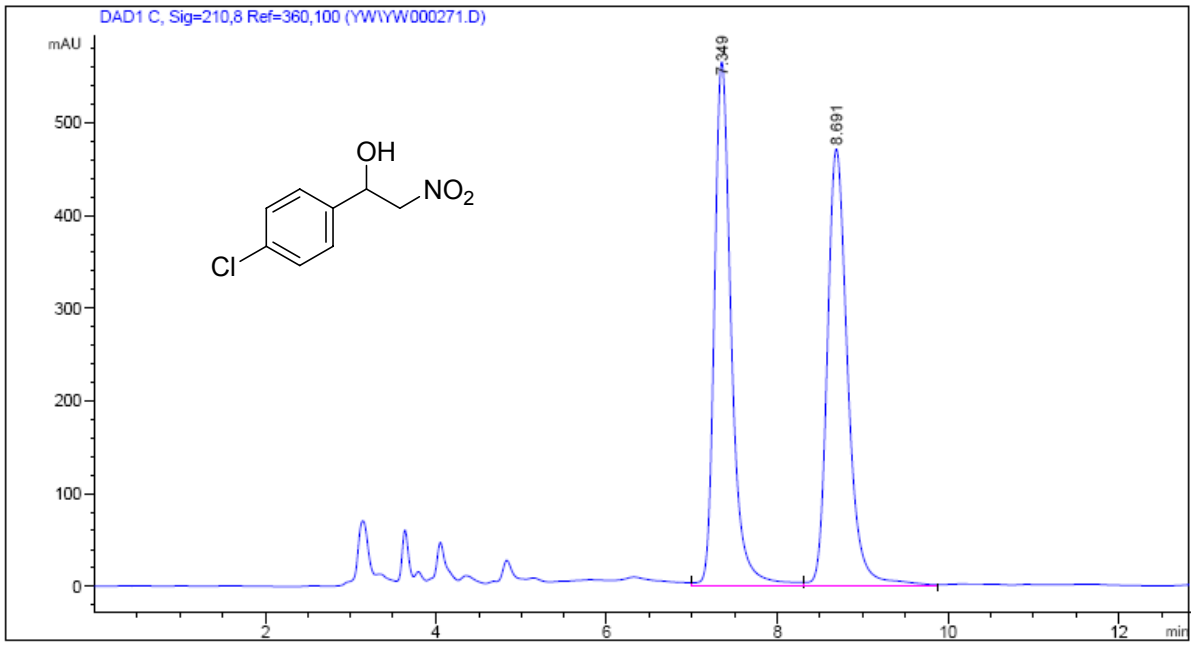
信号 1: DAD1 A, Sig=254,4 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.505	BV	0.3252	2216.81348	105.47421	48.2051
2	10.412	VB	0.3724	2381.89502	97.72131	51.7949



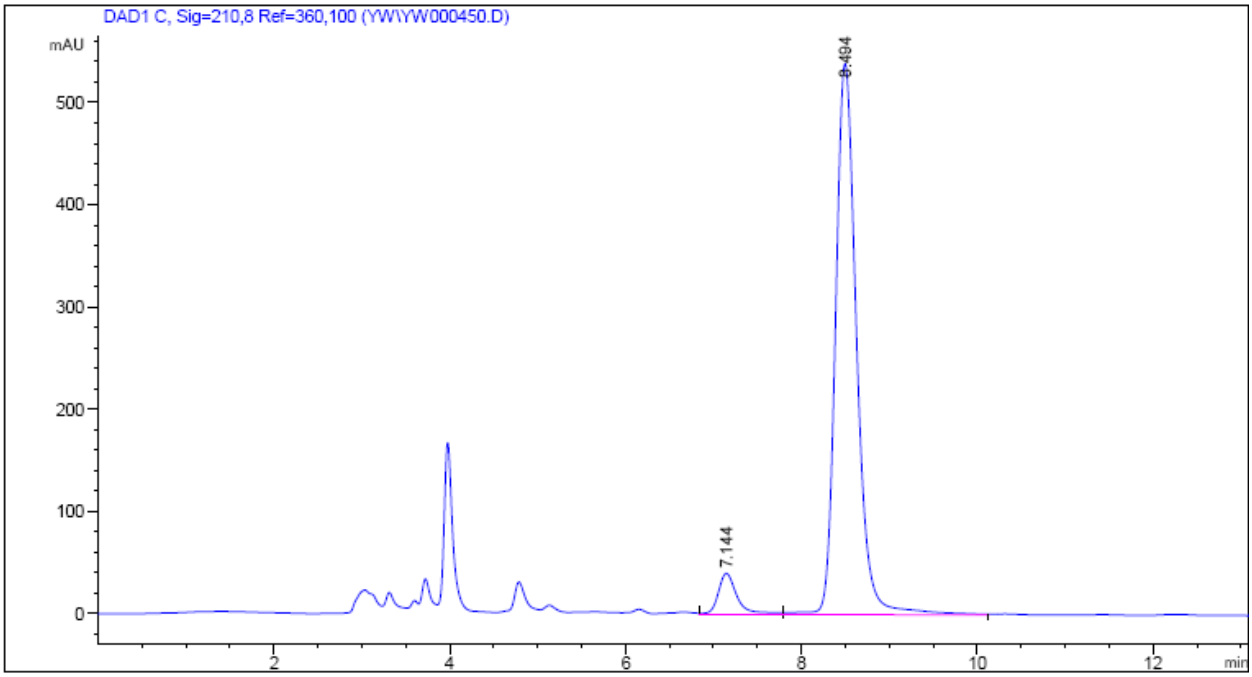
信号 1: DAD1 A, Sig=254,4 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.503	BV	0.3305	414.48679	19.61857	7.9371
2	10.341	VB	0.3698	4807.66992	199.04547	92.0629

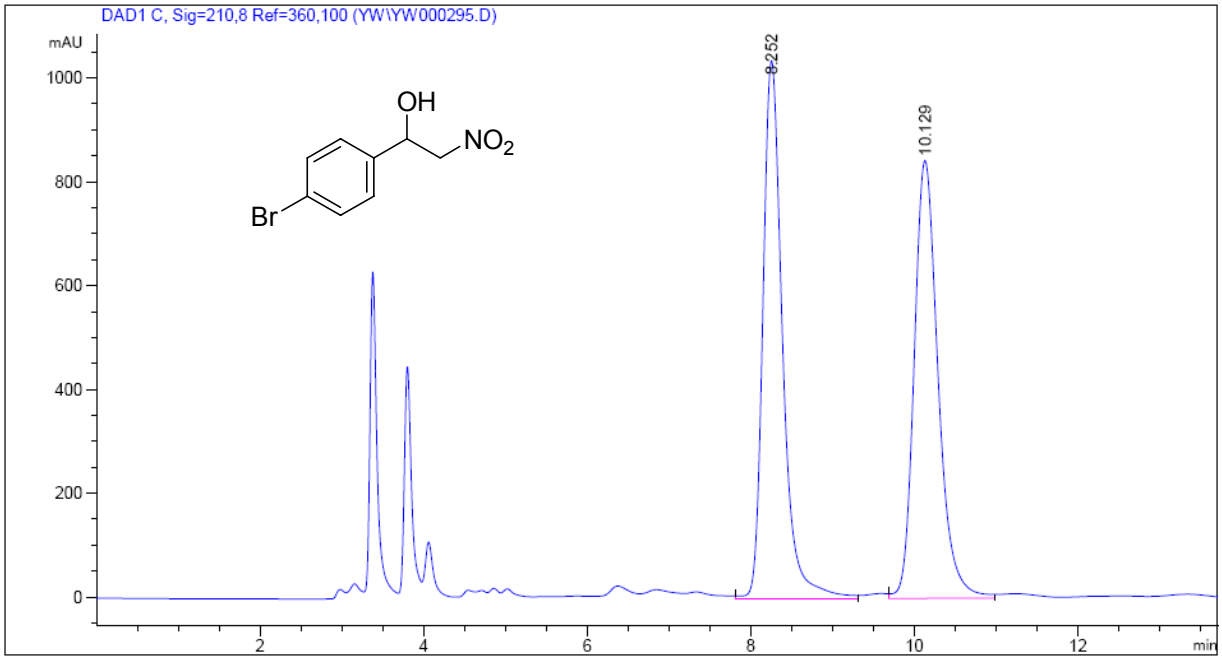


信号 1: DAD1 C, Sig=210,8 Ref=360,100

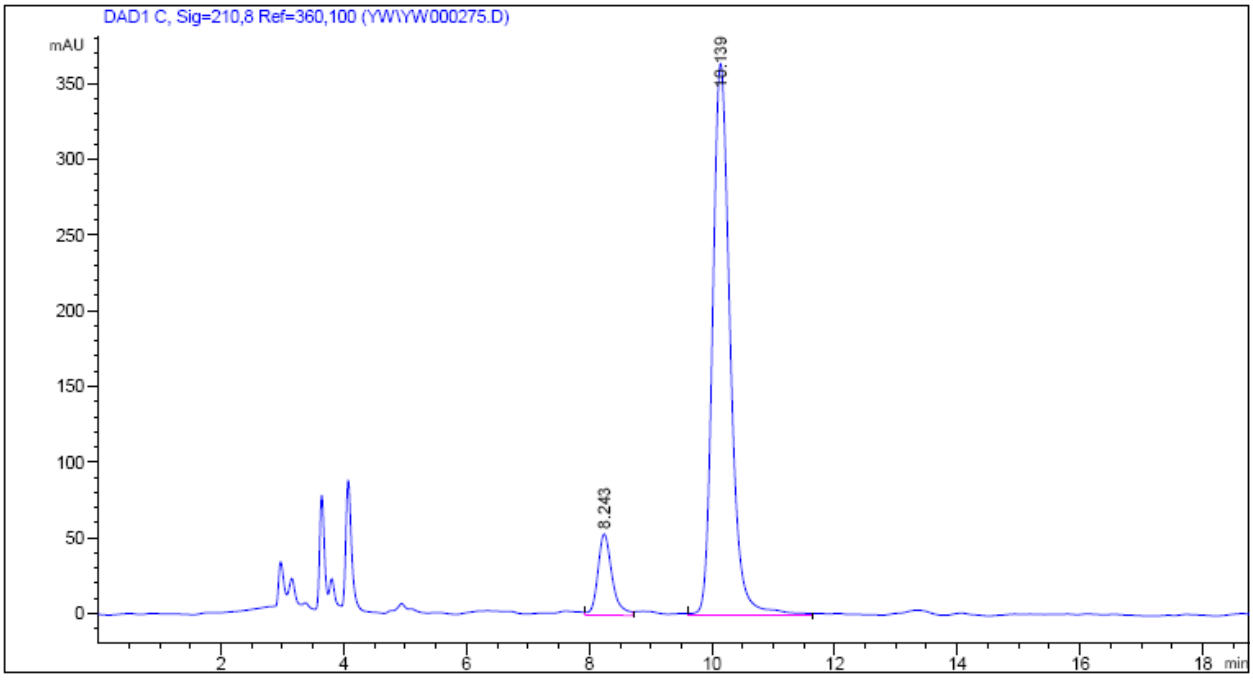
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	7.349	VV	0.2101	7871.92285	566.46179	49.9720
2	8.691	VV	0.2548	7880.75439	471.94147	50.0280



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	7.144	VV	0.2330	629.68567	40.59426	6.5226
2	8.494	VV	0.2591	9024.23730	539.50366	93.4774

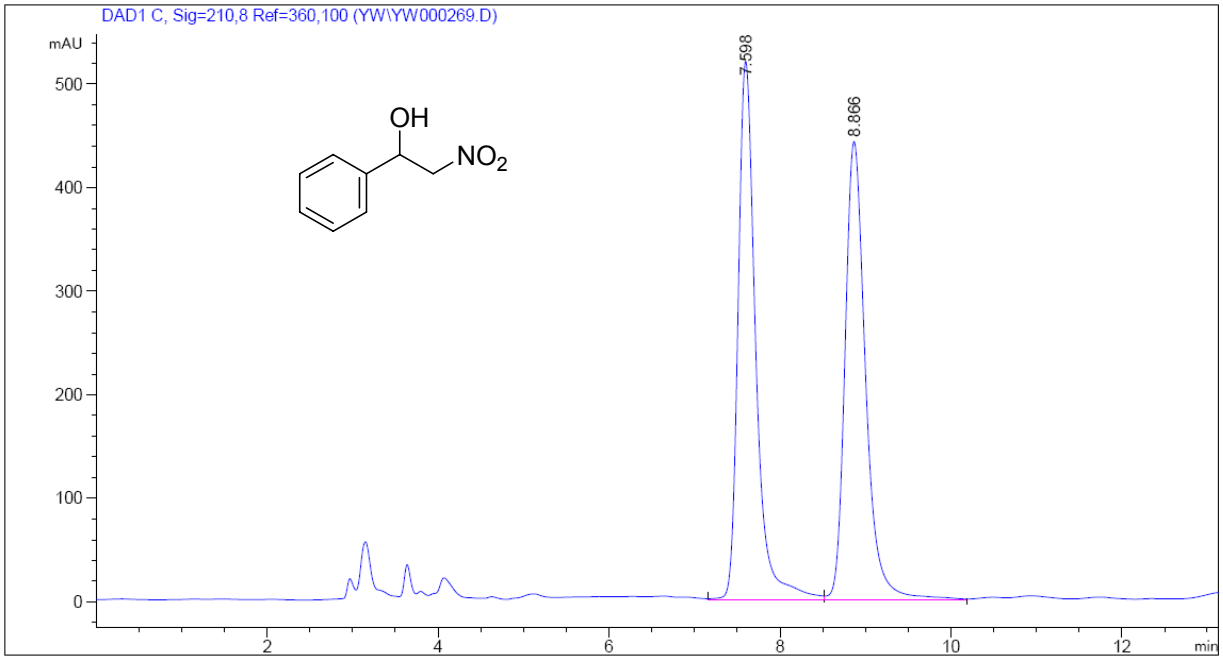


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	8.252	VV	0.2586	1.72623e4	1034.71265	50.1315
2	10.129	VV	0.3136	1.71718e4	843.01593	49.8685

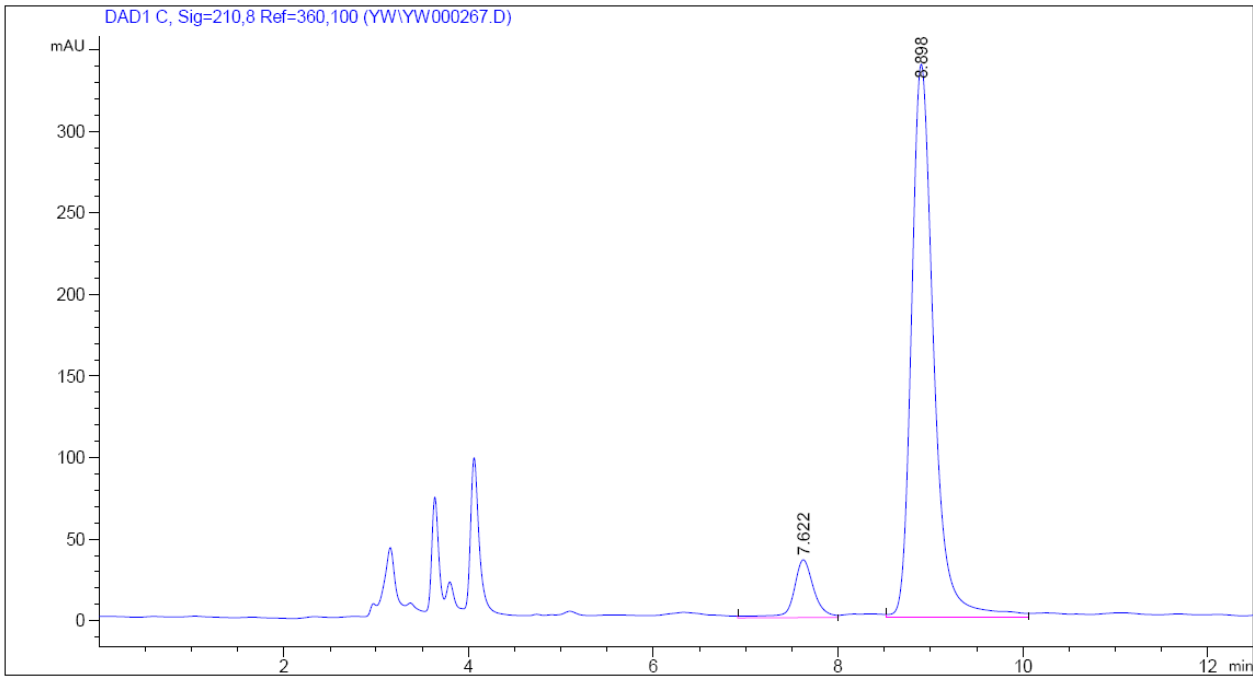


信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	8.243	VV	0.2495	873.66132	53.78755	10.7057
2	10.139	VV	0.3092	7287.06934	364.55786	89.2943

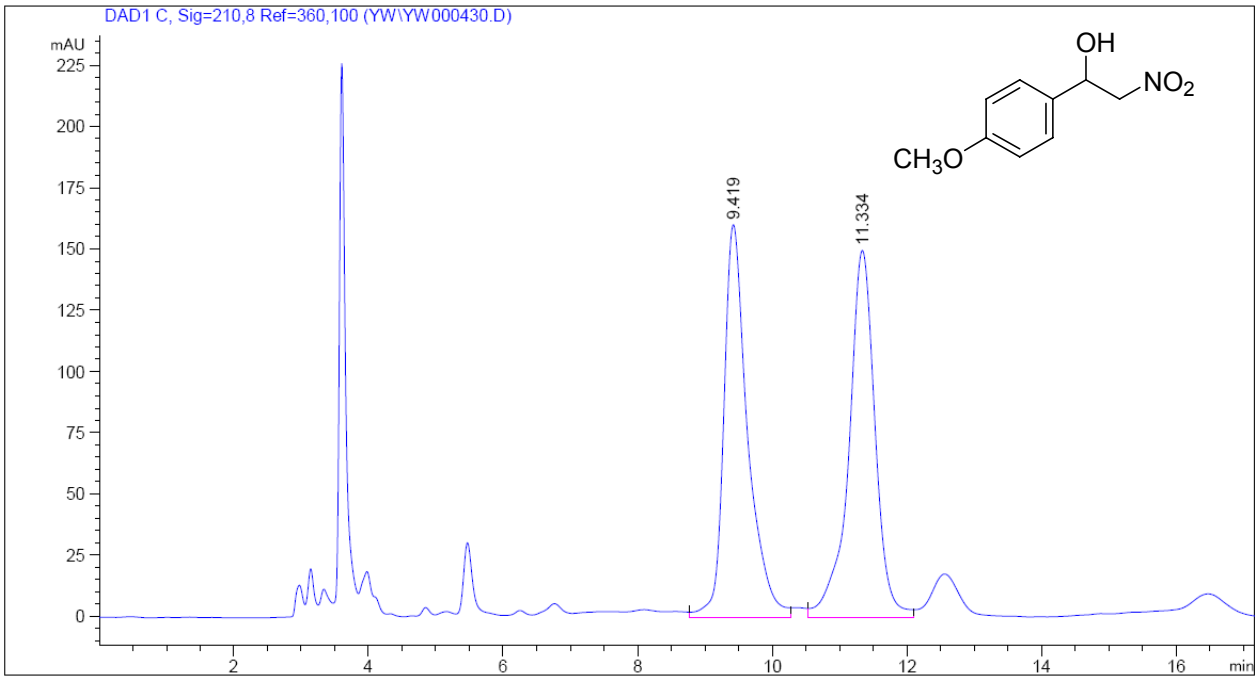


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	7.598	VV	0.2202	7494.69238	519.87598	50.3259
2	8.866	VV	0.2551	7397.62451	442.33167	49.6741



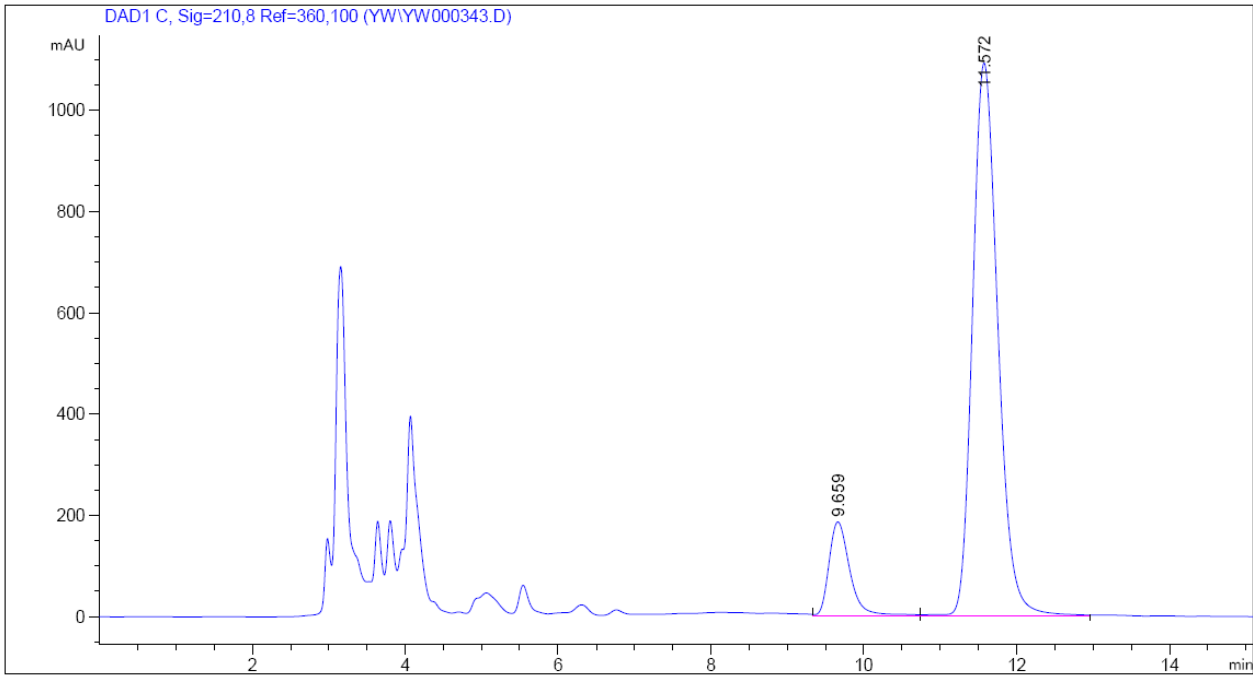
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	7.622	VV	0.2316	547.75702	35.58683	8.7597
2	8.898	VV	0.2562	5705.37793	339.17325	91.2403

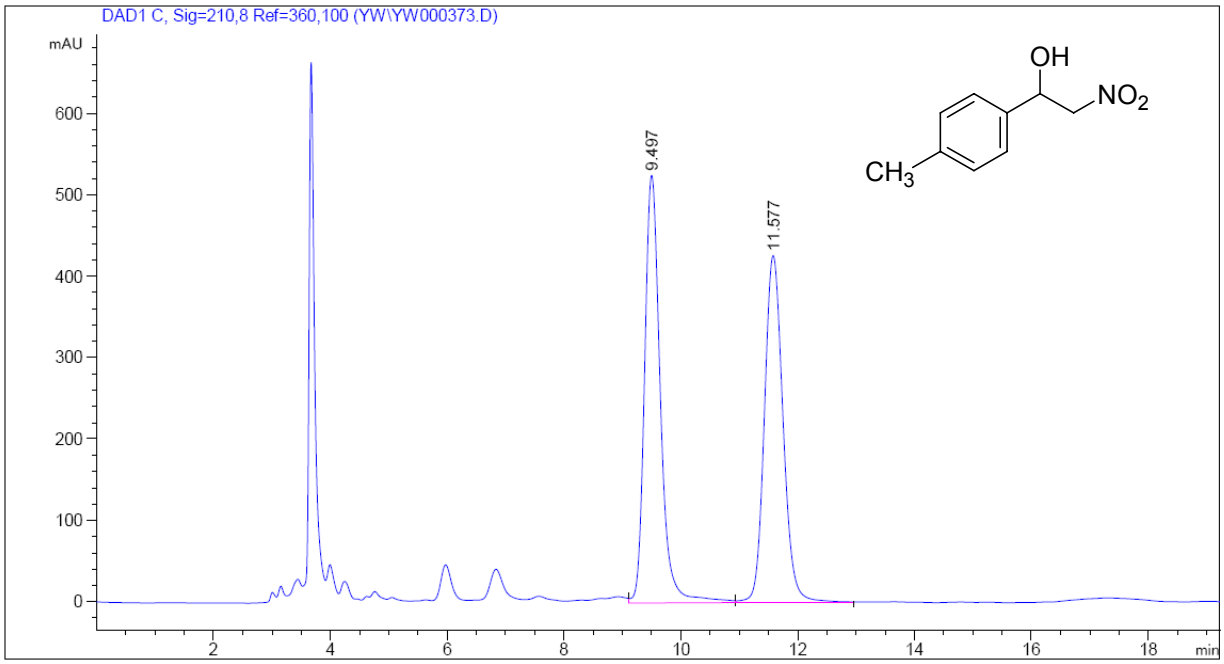


信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.419	VV	0.3642	3961.35840	160.35280	49.2486
2	11.334	VV	0.4098	4082.24292	149.86810	50.7514

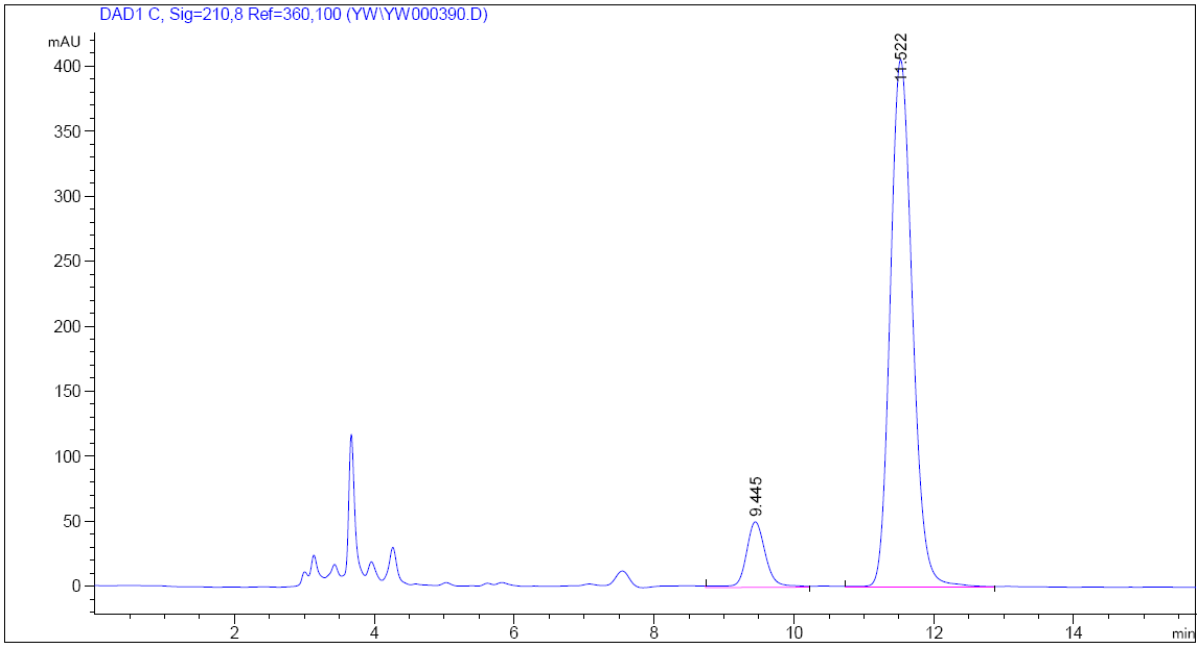


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.659	VV	0.2967	3668.70679	187.15082	12.6556
2	11.572	VV	0.3584	2.53202e4	1092.46655	87.3444



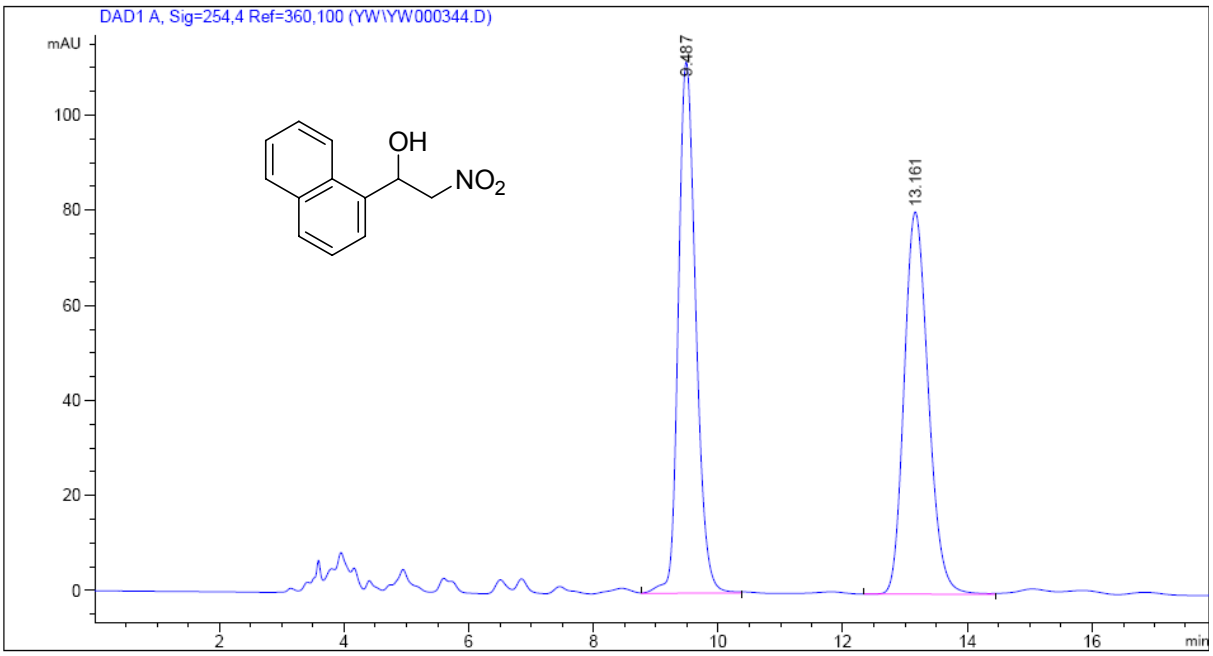
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.497	VV	0.2878	9895.47754	525.31006	50.9358
2	11.577	VV	0.3486	9531.87598	426.72028	49.0642



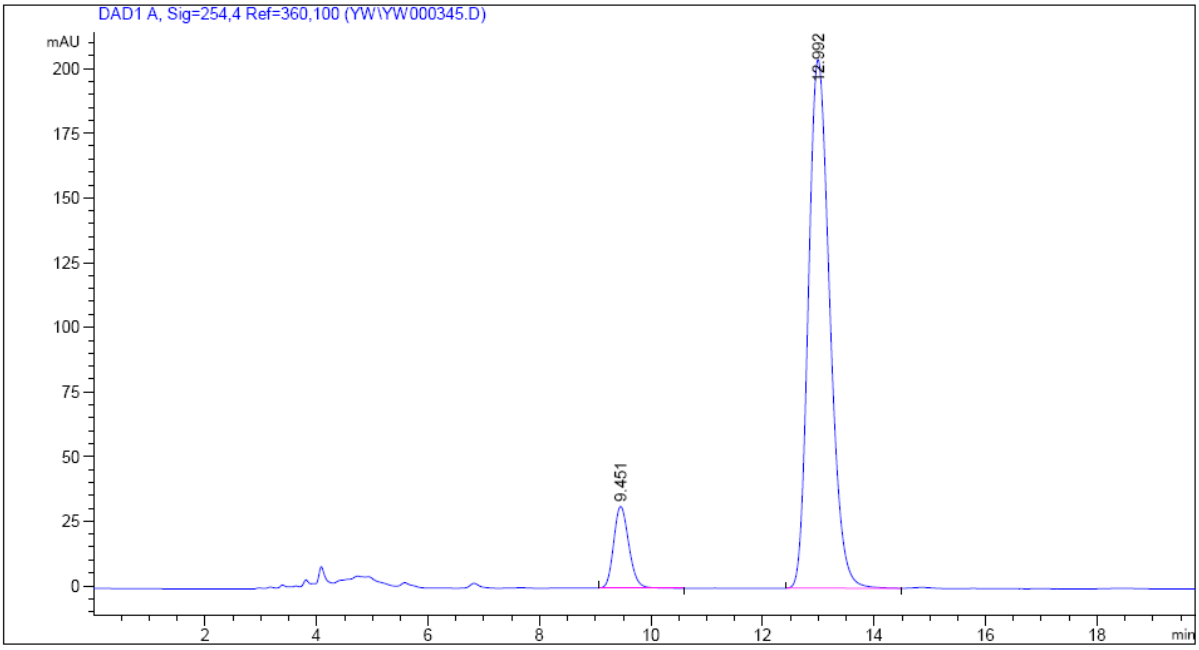
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.445	VV	0.2965	991.15161	50.61300	9.9293
2	11.522	VV	0.3463	8990.92969	406.05750	90.0707



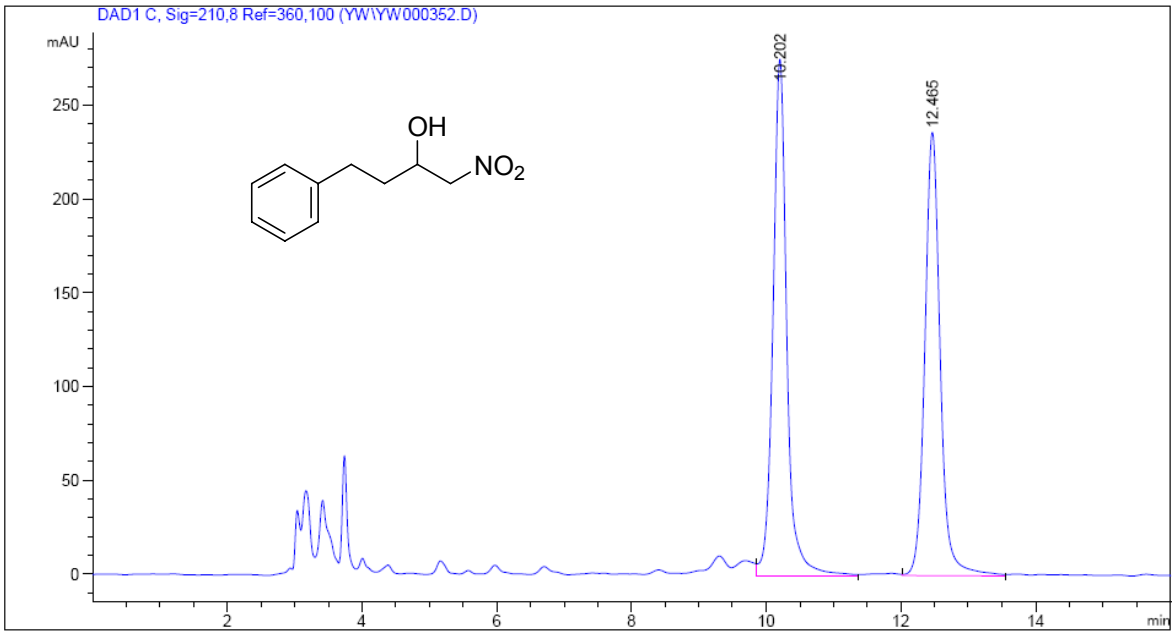
信号 1: DAD1 A, Sig=254,4 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.487	VB	0.3045	2189.45508	111.79115	50.2623
2	13.161	VB	0.4183	2166.60229	80.41850	49.7377



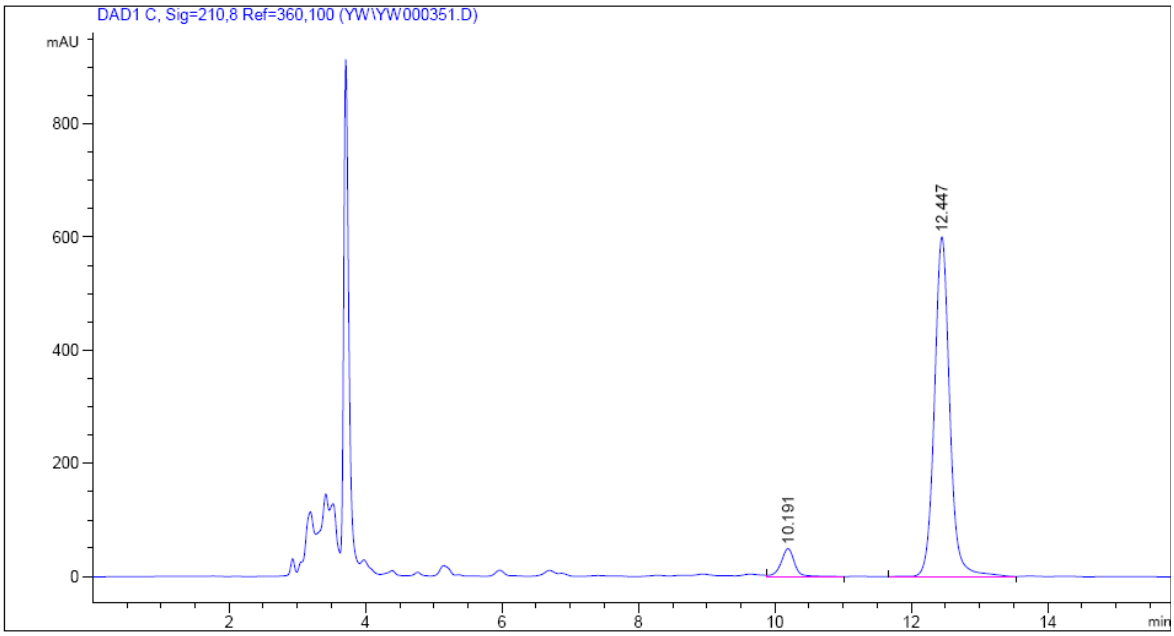
信号 1: DAD1 A, Sig=254,4 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.451	BB	0.2931	598.37616	31.56934	9.9137
2	12.992	BV	0.4143	5437.46143	204.41353	90.0863



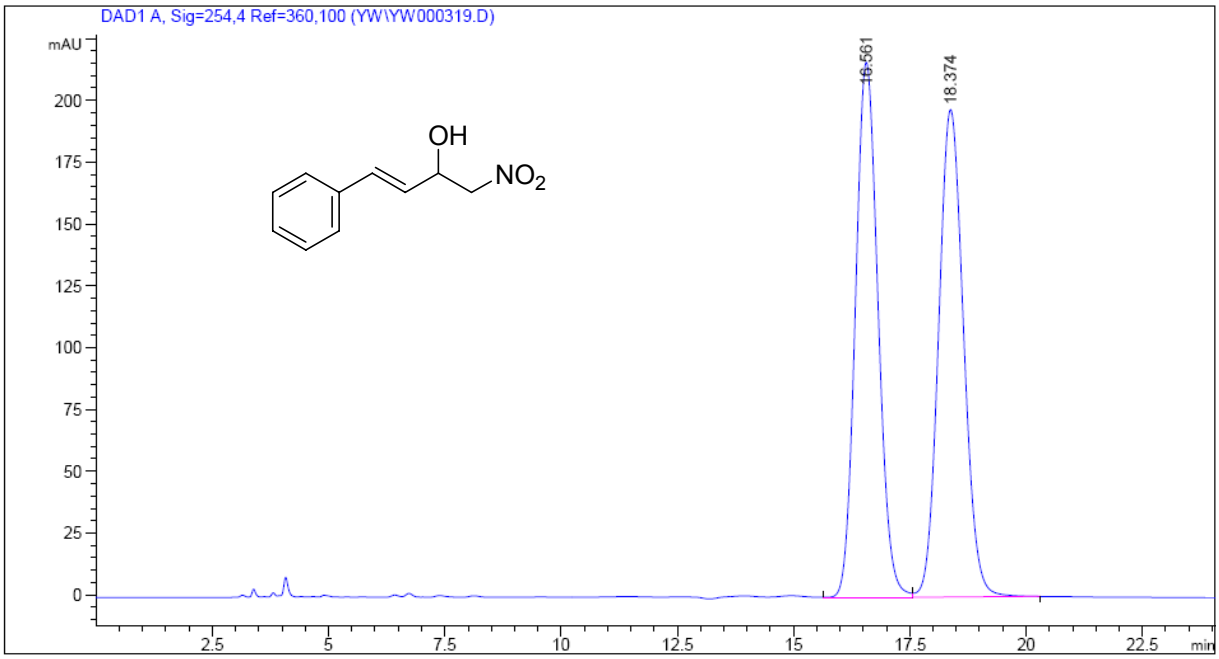
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.202	VV	0.2148	3936.18115	275.28439	51.1073
2	12.465	VV	0.2417	3765.61206	236.54498	48.8927



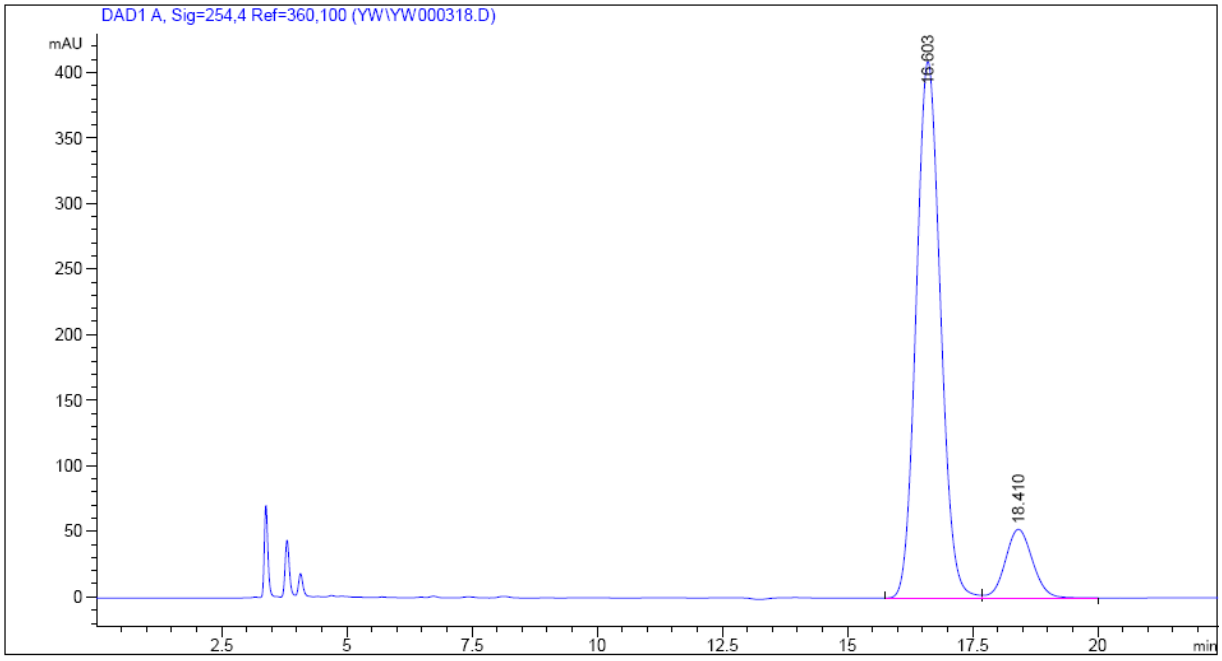
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.191	VV	0.2163	724.70532	50.22583	7.1516
2	12.447	VV	0.2427	9408.79199	600.83606	92.8484



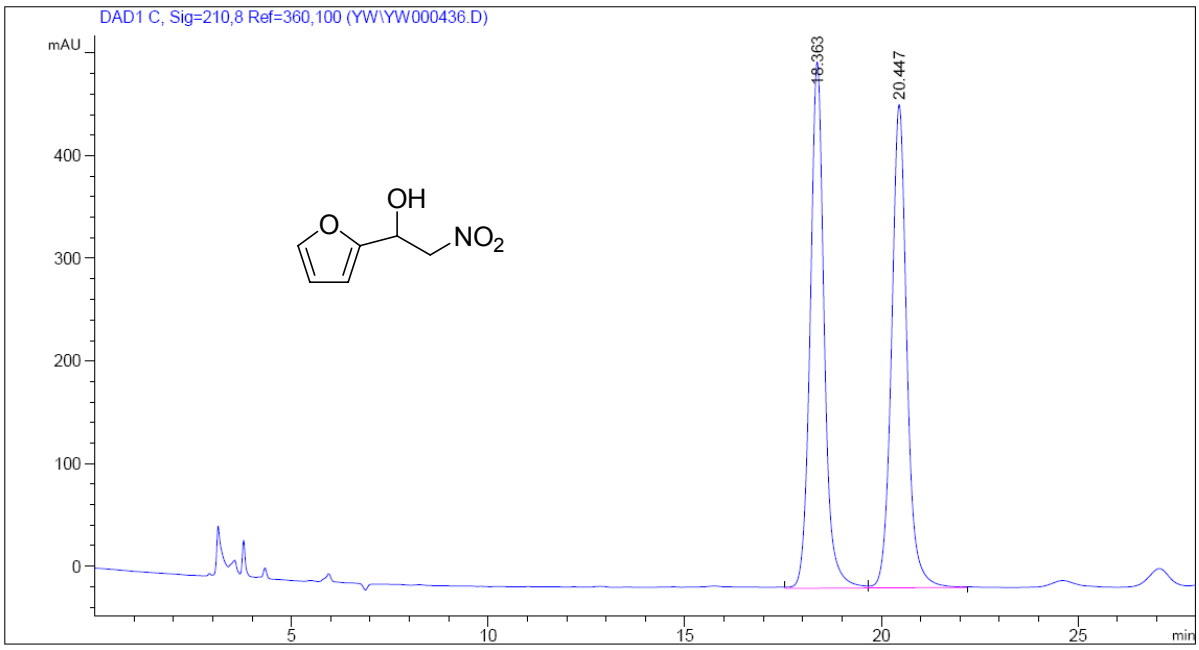
信号 1: DAD1 A, Sig=254,4 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.561	VV	0.5255	7301.83252	216.50389	49.8066
2	18.374	VB	0.5833	7358.53857	197.20122	50.1934



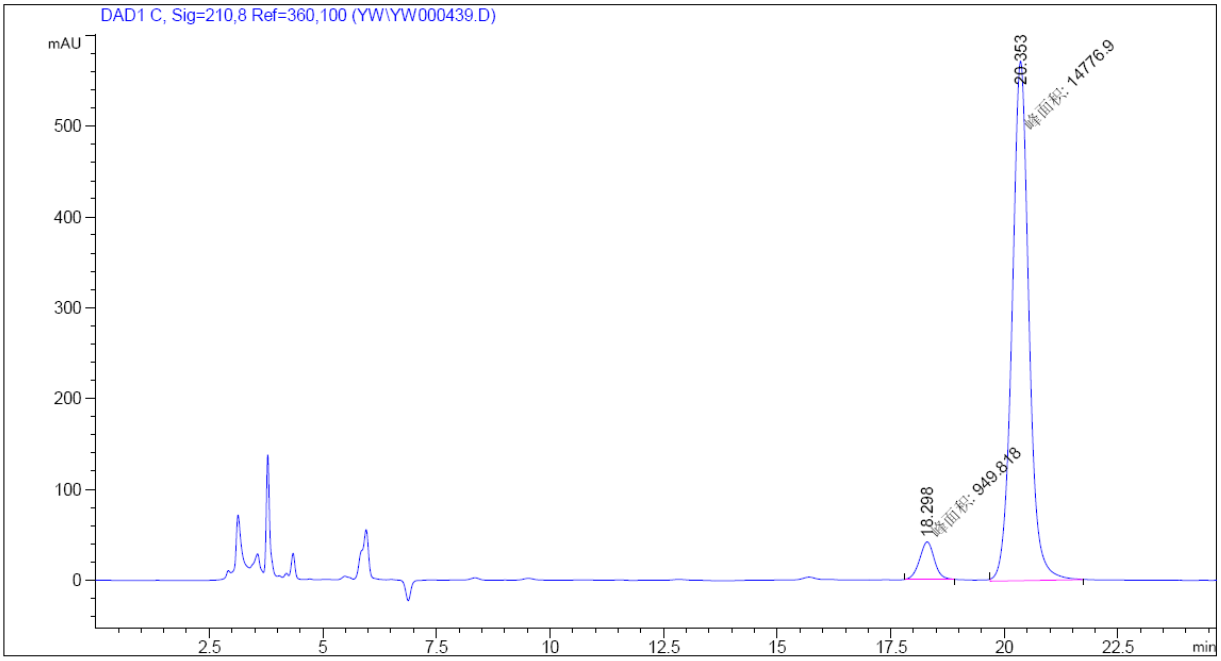
信号 1: DAD1 A, Sig=254,4 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	16.603	BV	0.5247	1.37747e4	409.22116	87.3994
2	18.410	VB	0.5898	1985.93591	52.44614	12.6006



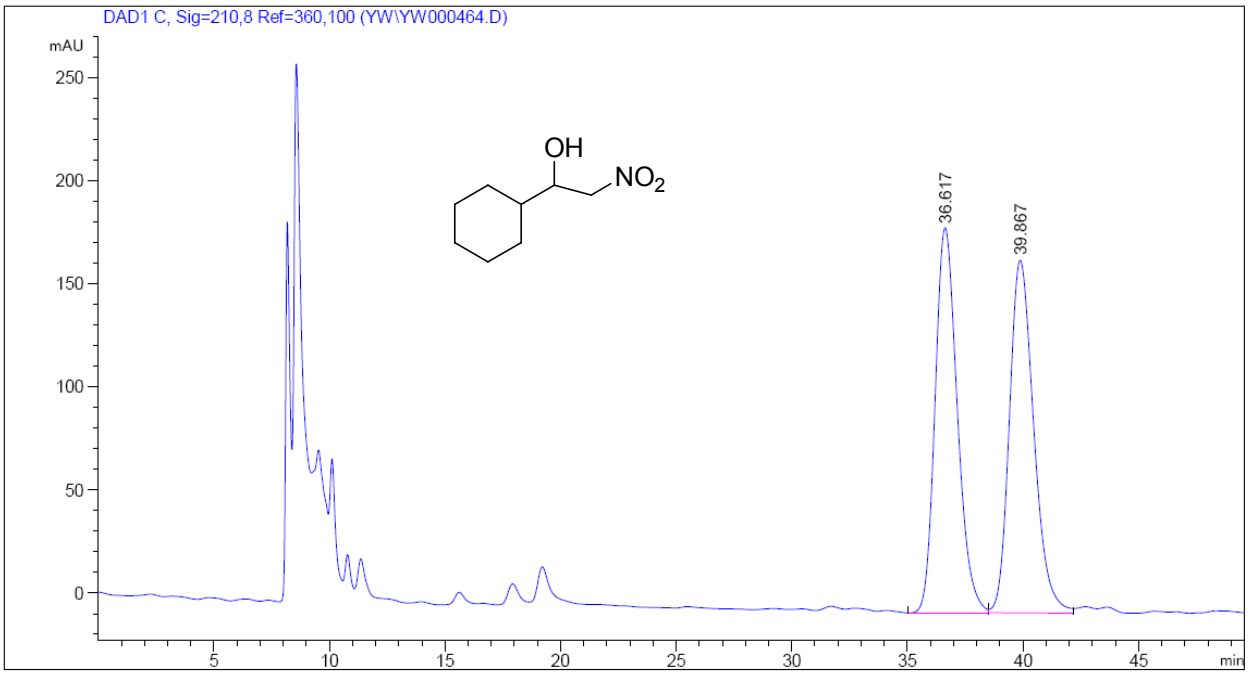
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	18.363	VV	0.3742	1.27476e4	512.55273	49.9855
2	20.447	VB	0.4081	1.27550e4	470.72137	50.0145



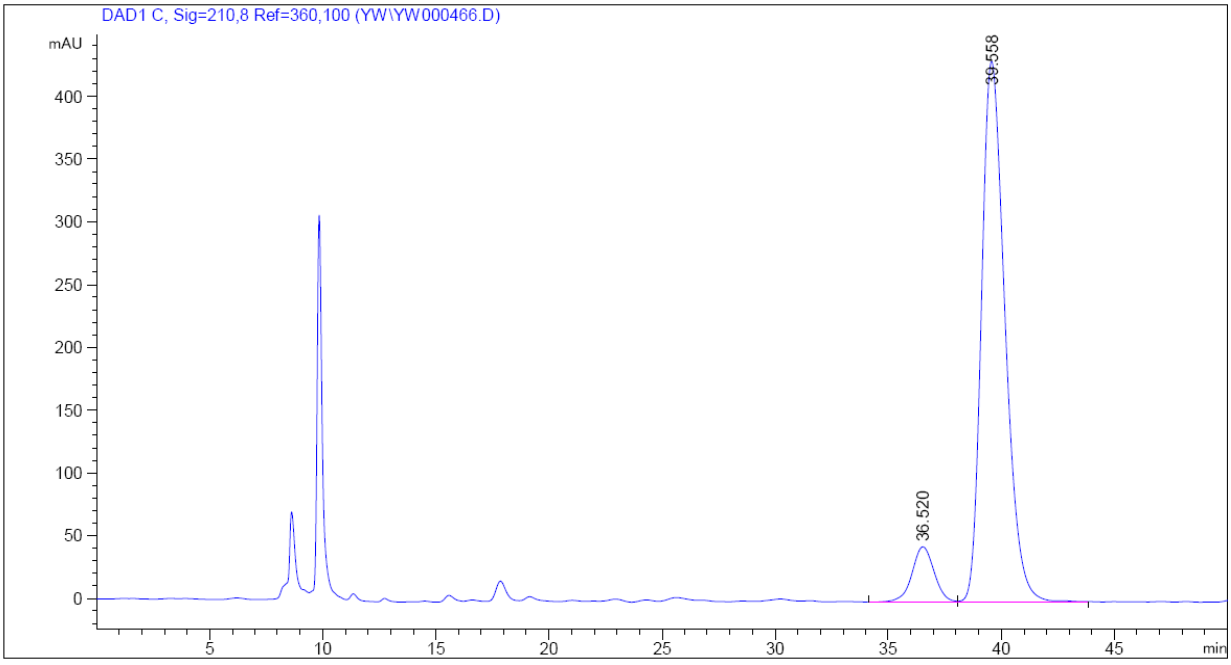
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	18.298	MM	0.3806	949.81750	41.59605	6.0395
2	20.353	MM	0.4301	1.47769e4	572.60583	93.9605



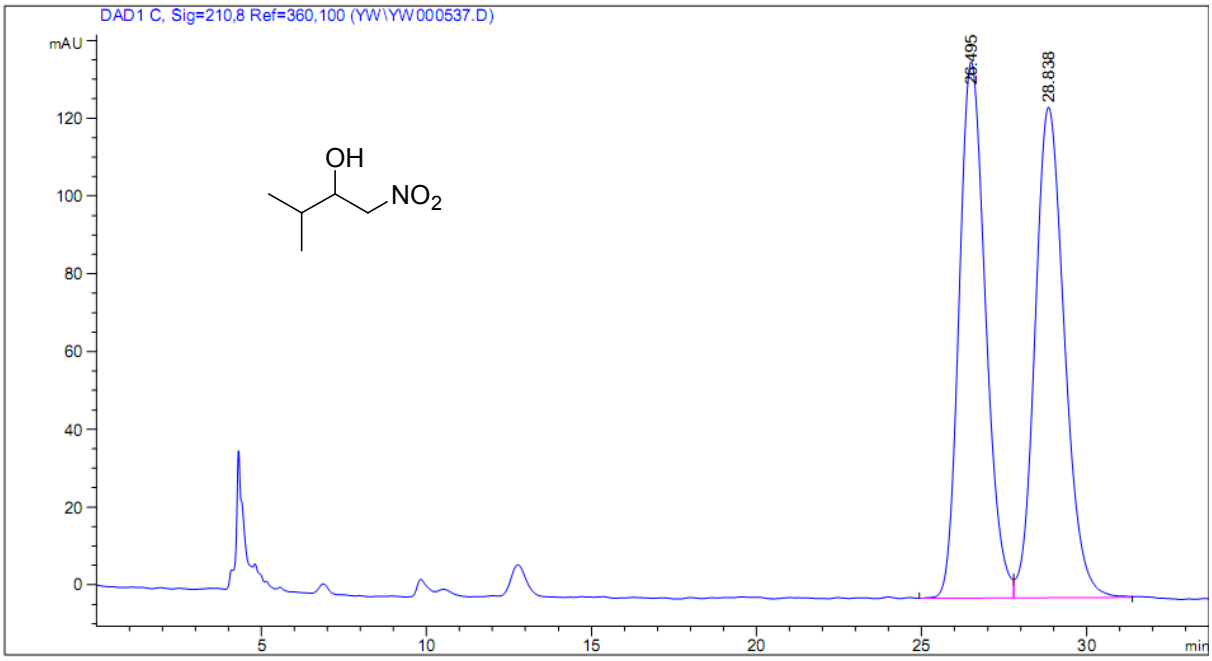
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	36.617	VV	1.0341	1.24673e4	186.92174	49.8436
2	39.867	VV	1.1268	1.25456e4	171.22792	50.1564



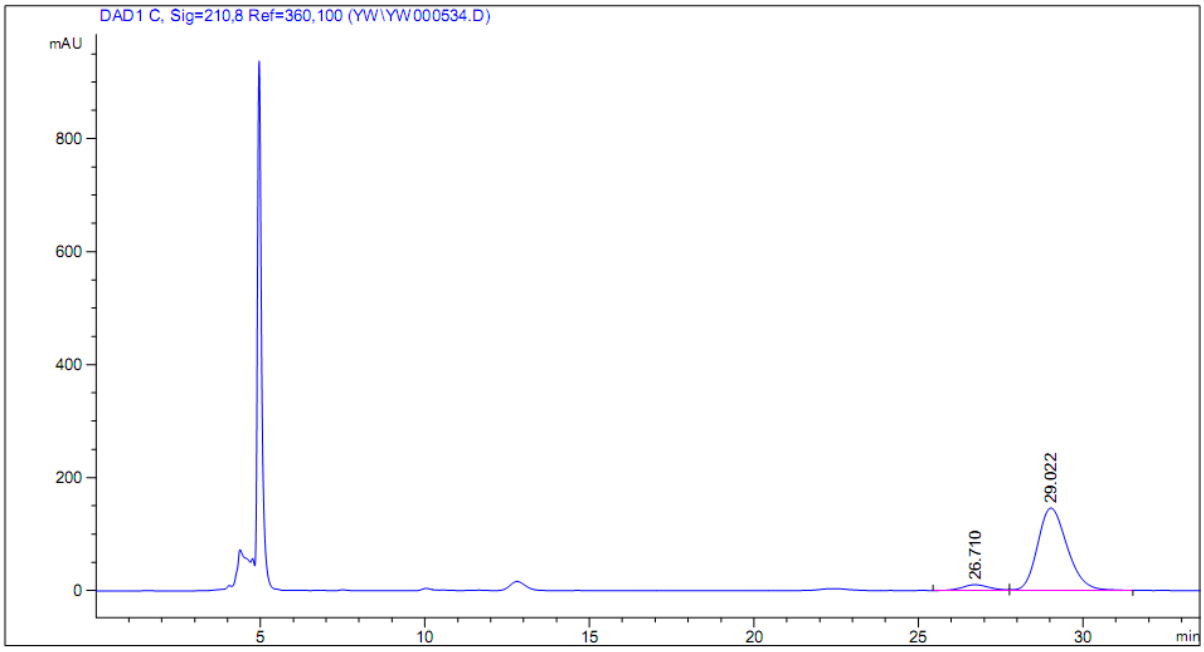
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	36.520	VV	1.0667	3034.31104	44.10318	8.8968
2	39.558	VB	1.1140	3.10714e4	430.51724	91.1032



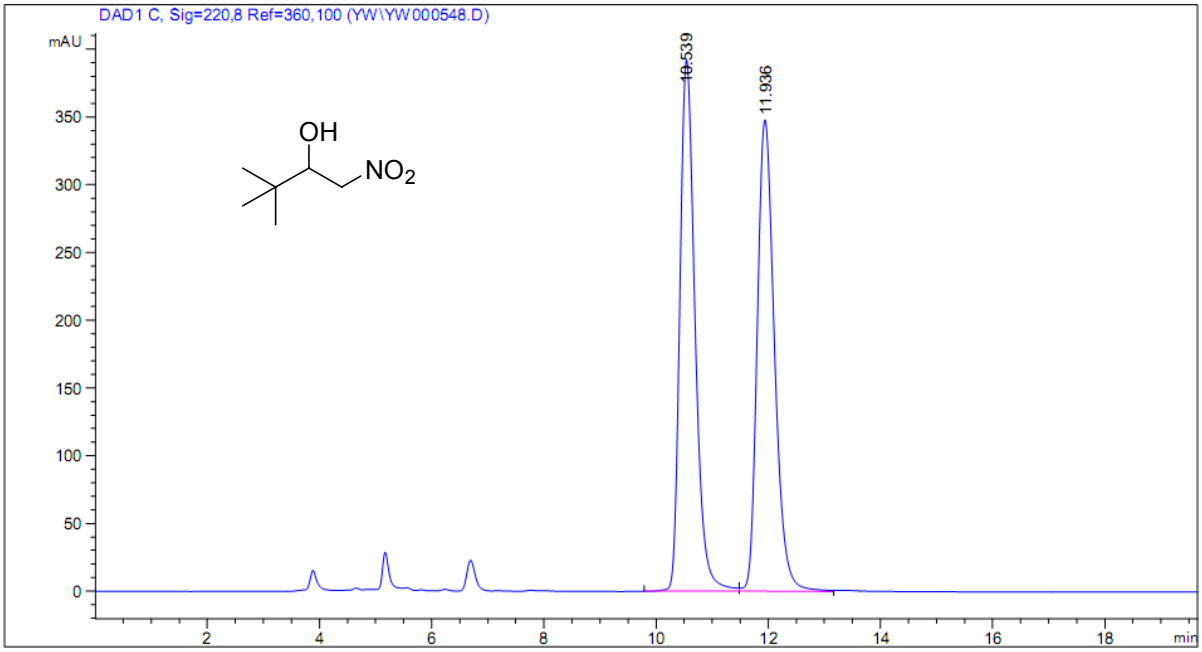
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	26.495	VV	0.8612	7676.32813	137.95648	49.5171
2	28.838	VB	0.9594	7826.04395	126.20803	50.4829



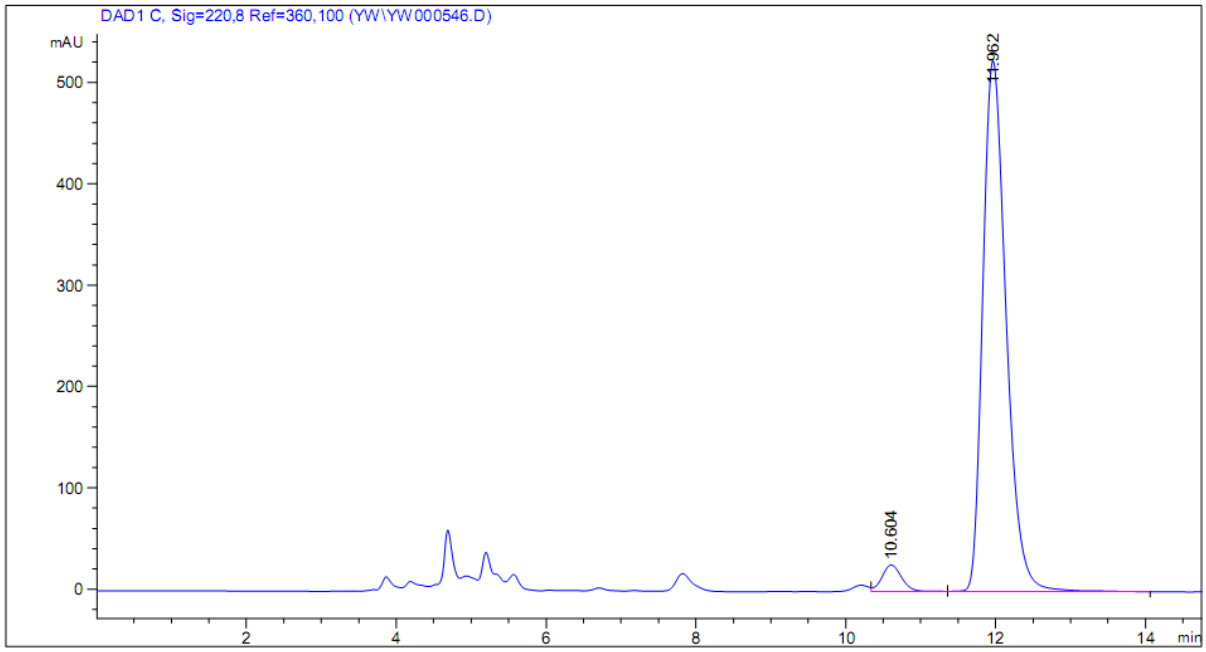
信号 1: DAD1 C, Sig=210,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	26.710	VV	0.9009	636.23981	10.59101	6.4658
2	29.022	VB	0.9704	9203.86230	146.21352	93.5342



信号 1: DAD1 C, Sig=220,8 Ref=360,100

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.539	BV	0.2915	7379.69043	392.16843	49.8224
2	11.936	VV	0.3291	7432.29834	348.12430	50.1776



信号 1: DAD1 C, Sig=220,8 Ref=360,100

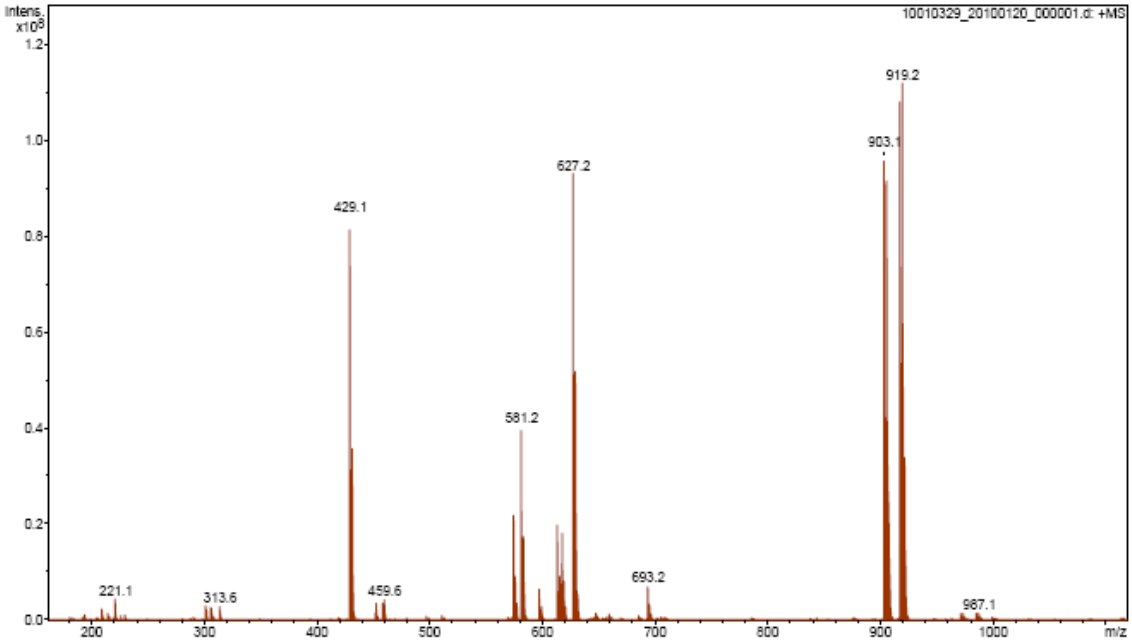
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.604	VV	0.2857	492.93130	26.41952	4.1615
2	11.962	VB	0.3369	1.13522e4	523.71454	95.8385

Mass spectra of catalyst **4db**

Peking University Mass Spectrometry Analysis Report

Analysis Info

Analysis Name	10010329_20100120_000001.d	Acquisition Date	1/20/2010 1:09:24 PM
Sample	Complex	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Bruker Compass DataAnalysis 4.0 printed: 1/20/2010 1:10:29 PM Page 1 of 1