Rh-Catalysed Asymmetric Conjugate Addition of Boronic Acids to Nitroalkenes Employing a *P*-Chiral *P*,π-Hybrid Ligand

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Supplementary Material

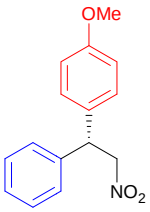
Table of Contents:

General. ^{1}H NMR spectra were recorded on Bruker 400 MHz or 500 MHz spectrometers. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as an internal standard ($CDCl_3$: 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, h = hextet, hept = heptet, br = broad, m = multiplet), and coupling constants (Hz). ^{13}C NMR was recorded on a Bruker 500 MHz (125 MHz) or 400 MHz (100 MHz) instrument with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: 77.0 ppm). Liquid chromatography was performed using forced flow (flash chromatography) on silica gel using a Biotage system. Thin layer chromatography (TLC) was performed on EMD silica gel F254 2.5x7.5 cm plates. Visualization was achieved using UV light or potassium permanganate in water followed by heating. All reactions were conducted in oven or flame dried glassware under an inert atmosphere of nitrogen or argon with magnetic stirring unless otherwise noted. Anhydrous solvents were purchased from Aldrich Chemical Company. Hexanes and ethyl acetate were purchased from Aldrich Chemical Company as ACS reagent grade. Solvents were degassed by Ar sparge. Chlorobis(ethylene)rhodium dimer was purchased from Strem Chemical Company. 3.0 M aqueous KHF_2 was purchased from Aldrich Chemical Company and degassed by Ar sparge before use (*caution: the pH of this solution is ~2 – 3, and therefore, may contain HF*). Ligands were prepared according to our previously published procedure.[1] All other reagents were purchased from either Fisher or Aldrich Chemical Companies and used directly.

General Procedure for the asymmetric conjugate addition of boronic acids (Scheme 3):

To a 20 mL crimp-cap vial with magnetic stir-bar was sequentially charged ligand (6.2 mg, 0.020 mmol, 4 mol %), chlorobis(ethylene)rhodium dimer (2.9 mg, 0.0075 mmol, 1.5 mol %), and 1.9 mL of THF in a glove-box under a N_2 atmosphere. The mixture was allowed to stir at rt for 5 min. The boronic acid (1.5 mmol, 3 equiv) and the nitroalkene (0.5 mmol, 1 equiv) were then charged, and the vial was sealed with a septum crimp-cap and removed from the glove-box. To the reaction mixture was then charged 0.50 mL of a 3.0 M aqueous KHF_2 (1.5 mmol, 3 equiv) solution that was freshly degassed by Ar sparge (*caution: the pH of this solution is ~2 – 3, and therefore, may contain HF*). The vial was then heated in an oil bath at 60 ^{o}C for the indicated time period. The mixture was cooled to rt, and 6 mL of 10% NH_4Cl was added (*caution: the pH of the aqueous layer was ~5.5; HF may be present*). The mixture was extracted with EtOAc (2x10mL), and the combined organic layers were dried with Na_2SO_4 and concentrated *in vacuo*. The crude residue was then purified by flash chromatography on silica gel (Biotage, hexanes/EtOAc mixtures). Enantioselectivity was determined by chiral HPLC analysis.

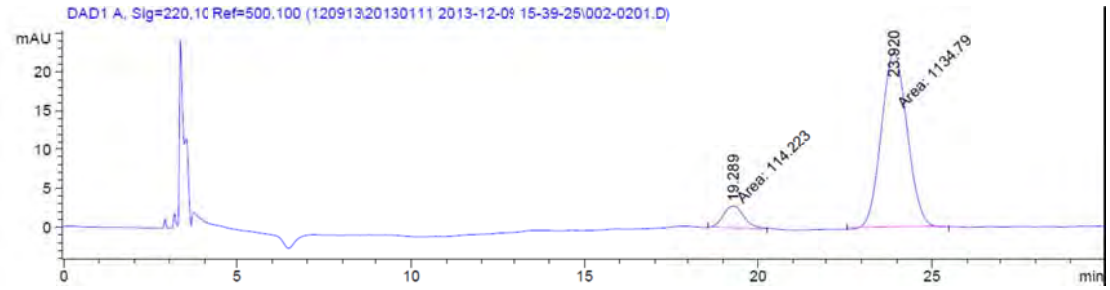
[1] Sieber, J. D.; Chennamadhavuni, D.; Fandrick, K. R.; Qu, B.; Han, Z. S.; Savoie, J. Ma, S.; Samankumara, L. P.; Grinberg, N.; Lee, H.; Song, J. J.; Senanayake, C. H. *Org. Lett.* **2014**, *16*, 5494.



(*R*)-1-methoxy-4-(2-nitro-1-phenylethyl)benzene (10a): A colorless oil. 112 mg (85%). R_f = 0.21 (SiO_2, 10% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 11.6 (*c* = 0.40, $CHCl_3$); Lit.: $[\alpha]^{20}_D$ = + 12 (*c* = 0.15, $CHCl_3$).[2] Spectral data was consistent with the literature.[2]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

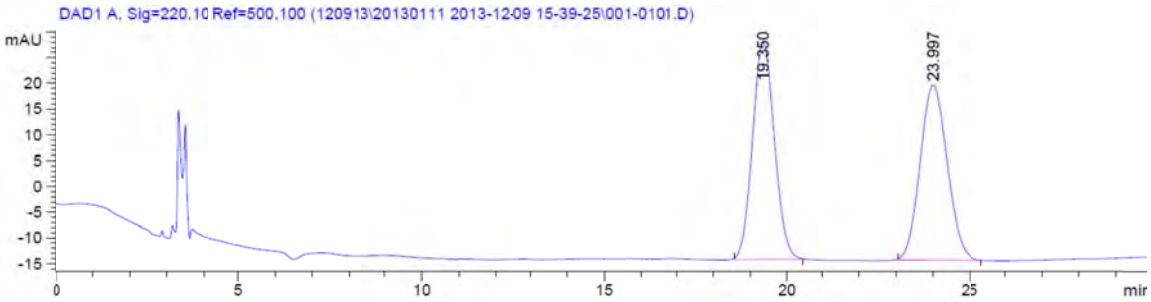
Asymmetric Reaction:



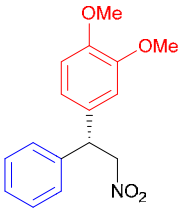
Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.289	MM	0.6803	114.22320	2.79817	9.1451
2	23.920	MM	0.8410	1134.78503	22.48932	90.8549
Totals :				1249.00823	25.28748	

Racemic Reaction:



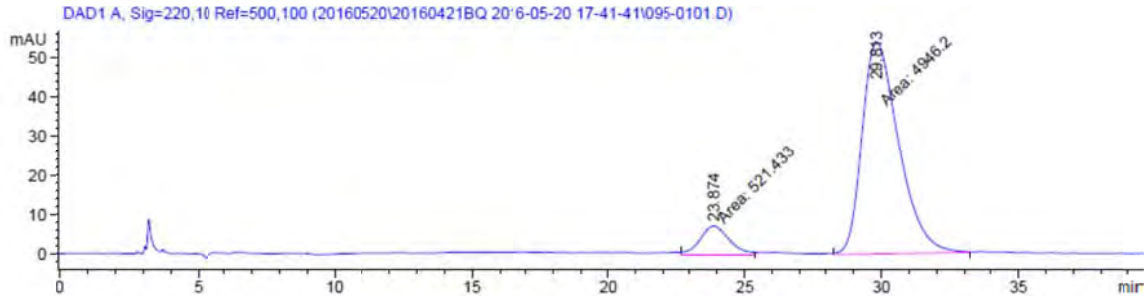
[2] F. Lang, G. Chen, L. Li, J. Xing, F. Han, L. Cun, J. Liao, *Chem. Eur. J.* 2011, **17**, 5242.



(*R*)-1,2-dimethoxy-4-(2-nitro-1-phenylethyl)benzene (10b): A colorless oil. 109 mg (76%). R_f = 0.28 (SiO_2, 30% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 8.8 (*c* = 0.50, $CHCl_3$); Lit.[3] (*S*-enantiomer): $[\alpha]^{20}_D$ = – 2.9 (*c* = 1.0, $CHCl_3$). Spectral data was consistent with the literature.[3]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

Asymmetric Reaction:

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Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  23.874 MM    1.2111  521.43286   7.17557   9.5367
   2  29.813 MM    1.5215 4946.19727  54.17947  90.4633

Totals :                  5467.63013  61.35504
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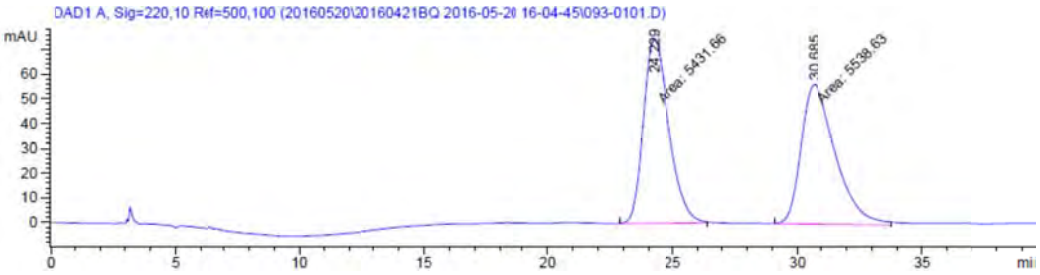
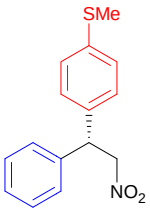
Racemic Reaction:



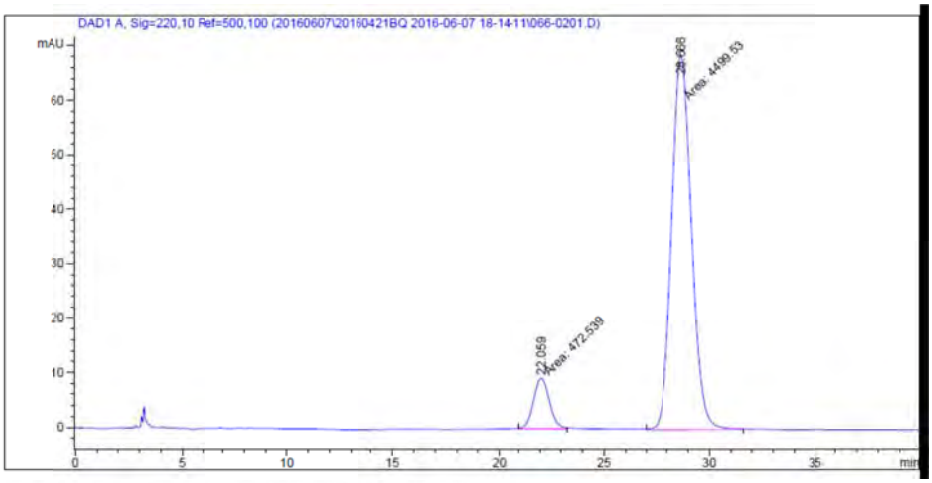
[3] K. –C.; Huang, B. Gopula, T. –S. Kuo, C. –W. Chiang, P. –Y. Wu, J. P. Henschke, H. –L. Wu, *Org. Lett.* 2013, **15**, 5730.



(*R*)-methyl(4-(2-nitro-1-phenylethyl)phenyl)sulfane (10c): A colorless oil. 104 mg (76%). R_f = 0.22 (SiO_2, 10% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 2.0 (*c* = 0.50, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33 (2H, t, *J* = 7.7 Hz), 7.26 (1H, t, *J* = 7.2 Hz), 7.18 – 7.24 (4H, m), 7.15 (2H, t, *J* = 8.4 Hz), 4.95 (2H, d, *J* = 8.2 Hz), 4.86 (1H, d, *J* = 8.2 Hz), 2.45 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.1, 138.1, 135.9, 129.1, 128.1, 127.7, 127.6, 127.0, 79.16, 48.47, 15.68. HRMS (ESI+) Calcd for $C_{15}H_{15}NO_2S$ $[M + NH_4]^+$: 291.1167; Found $[M + NH_4]^+$: 291.1153.

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

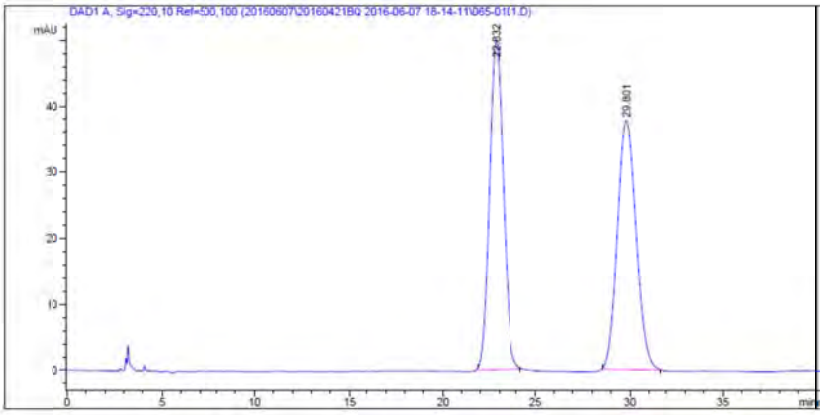
Asymmetric Reaction:

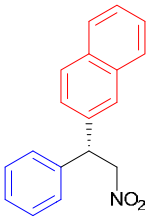


Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.059	MM	0.8602	472.53912	9.15582	9.5039
2	28.666	MM	1.0921	4499.53271	68.67033	90.4961
Totals :				4972.07184	77.82615	

Racemic Reaction:

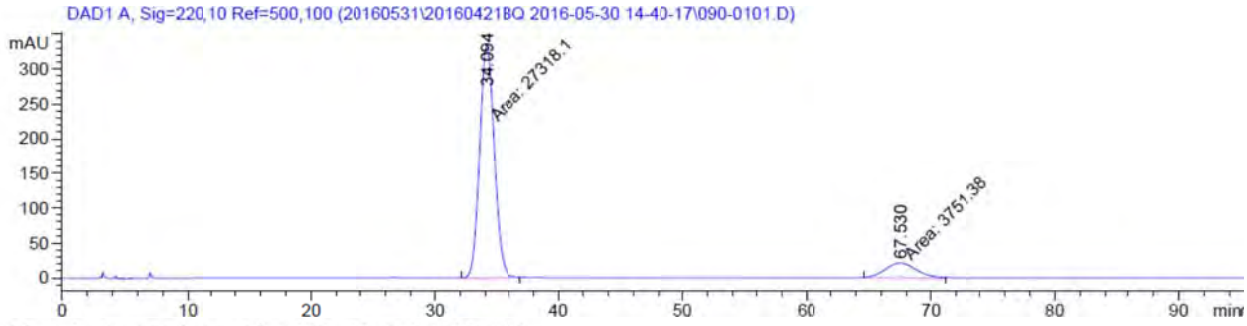




(*R*)-2-(2-nitro-1-phenylethyl)naphthalene (10d): A colorless oil. 111 mg (80%). R_f = 0.21 (SiO_2, 10% EtOAc in hexanes). $[\alpha]^{20}_D$ = – 26.9 (*c* = 0.50, $CHCl_3$); Lit.[4] (*S*-enantiomer): $[\alpha]^{20}_D$ = + 30.7 (*c* = 1.1, $CHCl_3$). Spectral data was consistent with the literature.[4]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

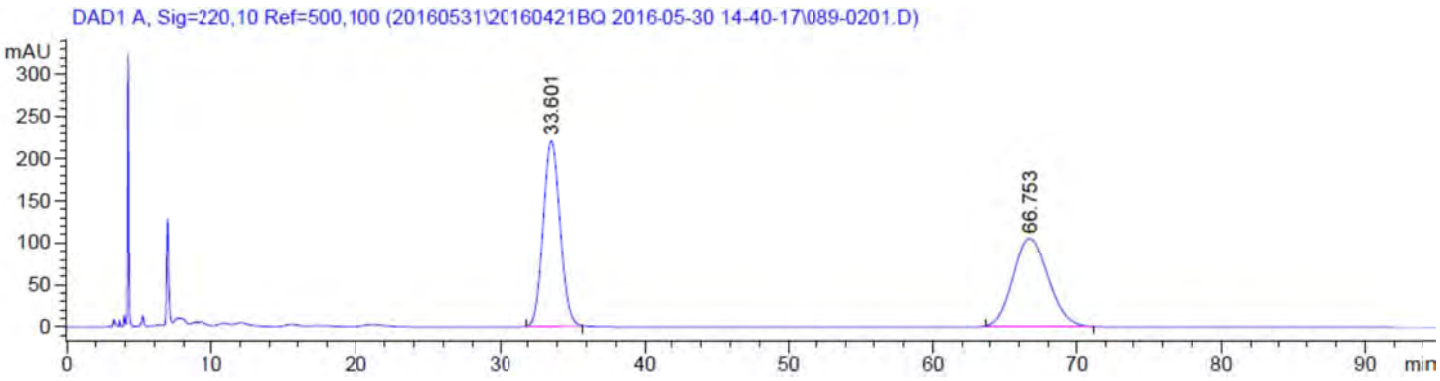
Asymmetric Reaction:



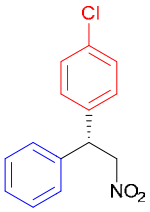
Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	34.094	MM	1.3588	2.73181e4	335.07977	87.9258
2	67.530	MM	2.9100	3751.38232	21.48572	12.0742
Totals :				3.10695e4	356.56549	

Racemic Reaction:



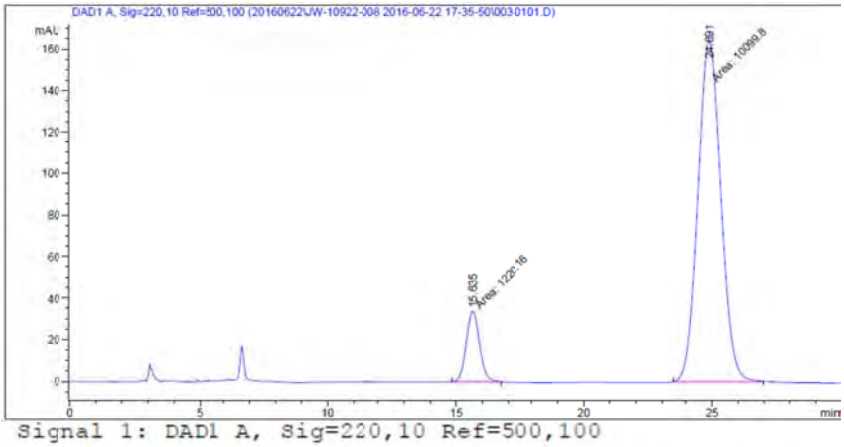
[4] F. Xue, D. Wang, X. Li, B. Wan, *J. Org. Chem.* 2012, **77**, 3071.



(*R*)-1-chloro-4-(2-nitro-1-phenylethyl)benzene (10e): A colorless oil. The reaction was performed employing 5 equiv of *p*-chlorophenylboronic acid: 78.8 mg (60%). R_f = 0.19 (SiO_2, 8% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 1.2 (*c* = 0.51, $CHCl_3$); Lit.[3]: $[\alpha]^{20}_D$ = + 1.3 (*c* = 1.0, $CHCl_3$). Spectral data was consistent with the literature.[3]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

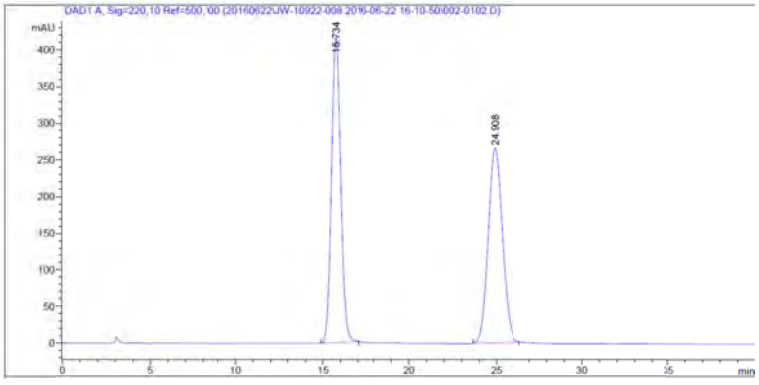
Asymmetric Reaction:

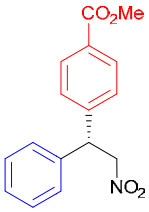


Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.635	MM	0.5984	1220.16357	33.98463	10.7789
2	24.891	MM	1.0251	1.00998e4	164.20747	89.2211
Totals :				1.13200e4	198.19210	

Racemic Reaction:

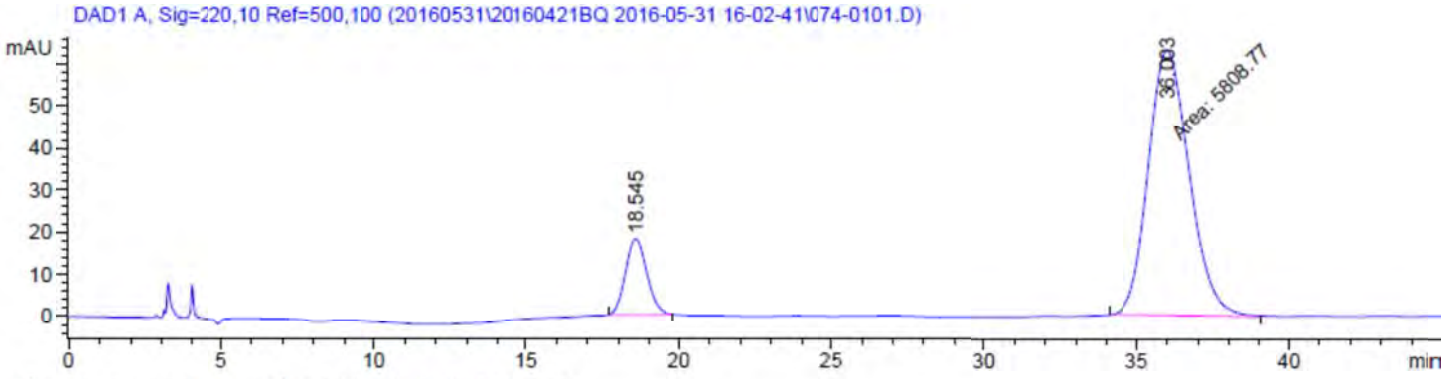




Methyl (*R*)-4-(2-nitro-1-phenylethyl)benzoate (10f): A colorless oil. 99.6 mg (70%). R_f = 0.21 (SiO_2, 20% EtOAc in hexanes). $[\alpha]^{20}_D$ = – 8.6 (*c* = 0.55, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 (2H, d, *J* = 8.3 Hz), 7.27 – 7.37 (5H, m), 7.22 (2H, d, *J* = 8.3 Hz), 4.93 – 5.06 (3H, m), 3.90 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6, 144.3, 138.5, 130.3, 129.6, 129.2, 127.9, 127.8, 127.7, 78.79, 52.19, 48.84. HRMS (ESI+) Calcd for $C_{16}H_{16}NO_4$ $[M + H]^+$: 286.1079; Found $[M + H]^+$: 286.1086.

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

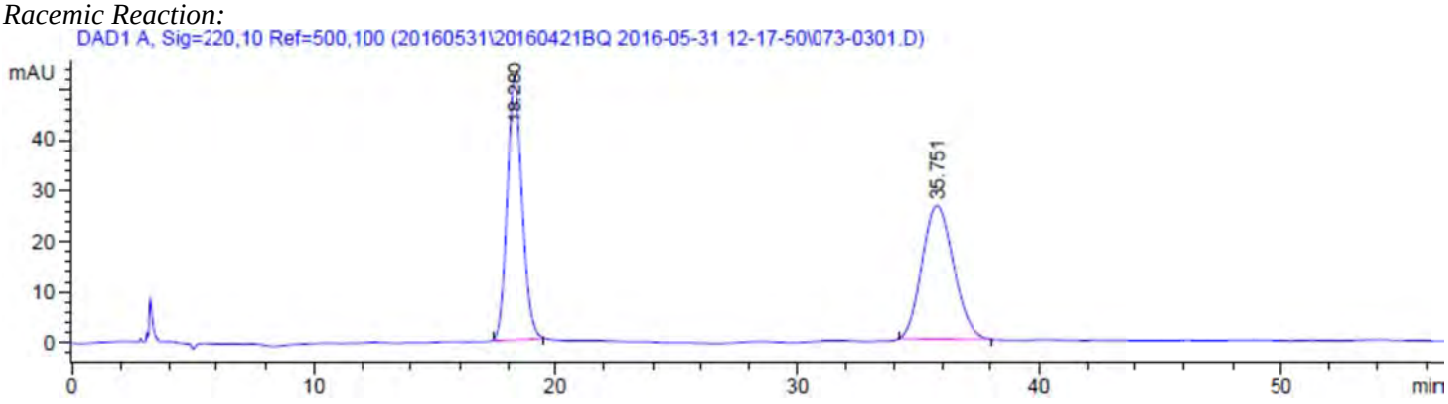
Asymmetric Reaction:

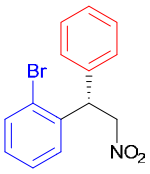


Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.545	BB	0.7186	866.06097	17.95971	12.9750
2	36.003	MM	1.5386	5808.76611	62.92257	87.0250
Totals :				6674.82709	80.88228	

Racemic Reaction:

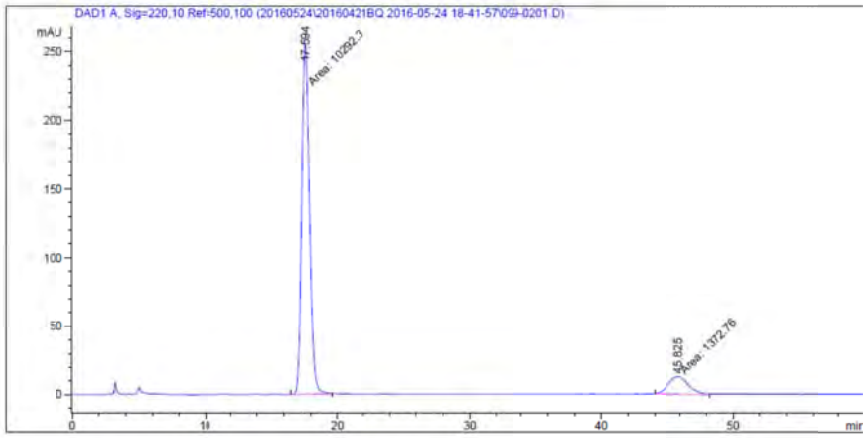




(*S*)-1-bromo-2-(2-nitro-1-phenylethyl)benzene (10g): A yellow oil. Reaction performed using 1.00 g (4.39 mmol) b-nitrostyrene: 1.14 g (85%). R_f = 0.17 (SiO_2, 8% EtOAc in hexanes). $[\alpha]^{20}_D$ = – 25.8 (*c* = 0.53, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60 (1H, dd, *J* = 8.2 Hz, *J* = 1.0 Hz), 7.24 – 7.36 (6H, m), 7.21 (1H, dd, *J* = 7.9 Hz, *J* = 1.6 Hz), 7.14 (1H, td, *J* = 7.8 Hz, *J* = 1.6 Hz), 5.45 (1H, t, *J* = 8.2 Hz), 4.98 (1H, dd, *J* = 13 Hz, *J* = 8.2 Hz), 4.94 (1H, dd, *J* = 13 Hz, *J* = 8.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.2, 137.7, 133.8, 129.2, 129.1, 128.4, 128.06, 127.96, 127.8, 125.0, 77.92, 47.64. HRMS (ESI+) Calcd for $C_{14}H_{12}BrNO_2$ $[M + NH_4]^+$: 323.0395; Found $[M + NH_4]^+$: 323.0383.

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

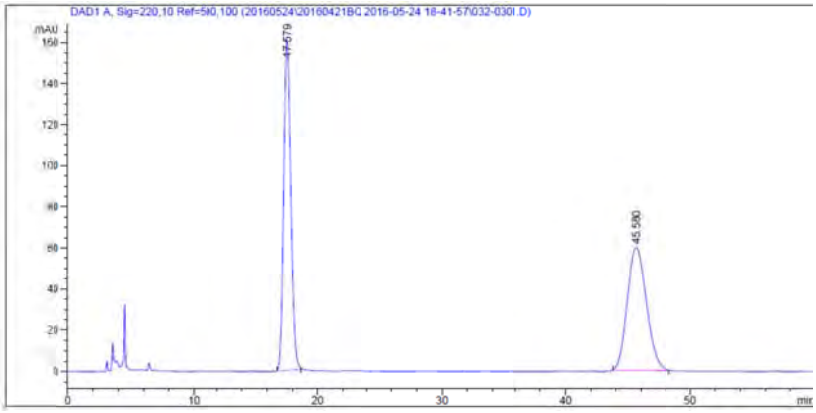
Asymmetric Reaction:

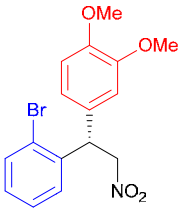


Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.594	MM	0.6715	1.02923e4	255.47009	88.2318
2	45.825	MM	1.7734	1372.76428	12.90144	11.7682
Totals :				1.16651e4	268.37154	

Racemic Reaction:

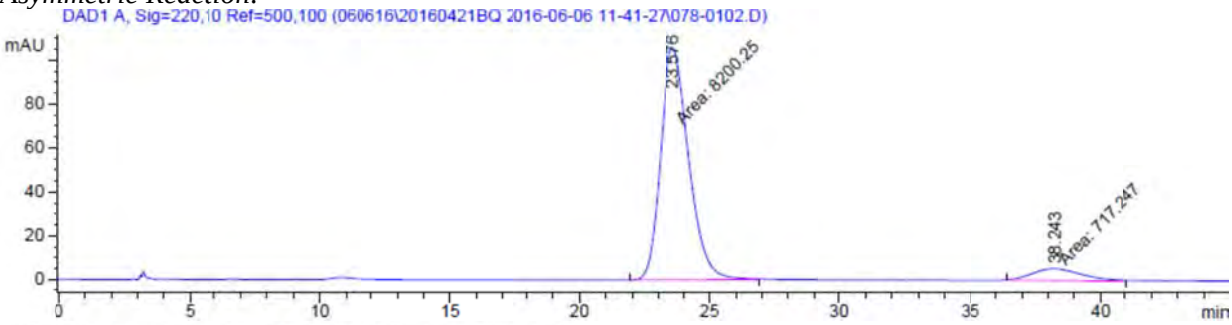




(*S*)-4-(1-(2-bromophenyl)-2-nitroethyl)-1,2-dimethoxybenzene (10h): A yellow oil. 156 mg (85%). R_f = 0.14 (SiO_2, 20% EtOAc in hexanes). $[\alpha]^{20}_D$ = – 36.5 (*c* = 0.55, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.61 (1H, dd, *J* = 7.8 Hz, *J* = 1.0 Hz), 7.30 (1H, t, *J* = 7.6 Hz), 7.20 (1H, dd, *J* = 7.8 Hz, *J* = 1.5 Hz), 7.14 (1H, td, *J* = 7.8 Hz, *J* = 1.6 Hz), 6.82 (1H, d, *J* = 8.3 Hz), 6.81 (1H, dd, *J* = 8.3 Hz, *J* = 1.8 Hz), 6.76 (1H, d, *J* = 1.6 Hz), 5.38 (1H, t, *J* = 8.7 Hz), 4.97 (1H, dd, *J* = 13 Hz, *J* = 8.7 Hz), 4.91 (1H, dd, *J* = 13 Hz, *J* = 8.7 Hz), 3.85 (3H, s), 3.84 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.3, 148.6, 138.4, 133.8, 130.1, 129.1, 128.3, 128.0, 124.8, 119.8, 111.7, 111.4, 77.94, 55.95, 55.87, 47.34. HRMS (ESI+) Calcd for $C_{16}H_{20}BrNO_4$ $[M + NH_4]^+$: 383.0606; Found $[M + NH_4]^+$: 383.0603.

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

Asymmetric Reaction:

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Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  23.576 MM    1.2843 8200.25195  106.41695  91.9569
   2  38.243 MM    2.2057  717.24744    5.41968   8.0431

Totals :                  8917.49939  111.83663
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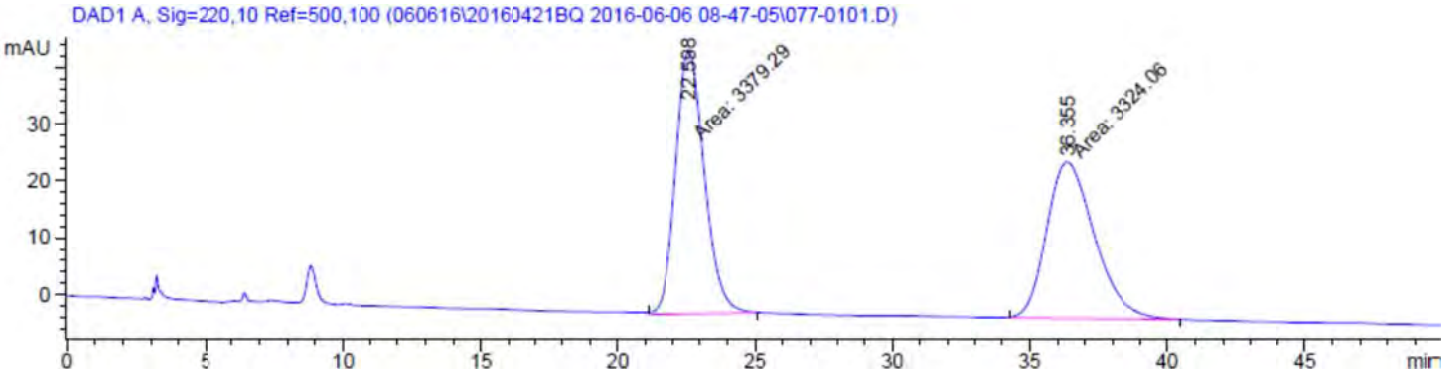
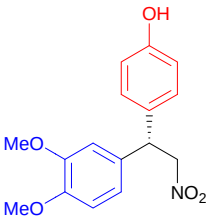
Racemic Reaction:

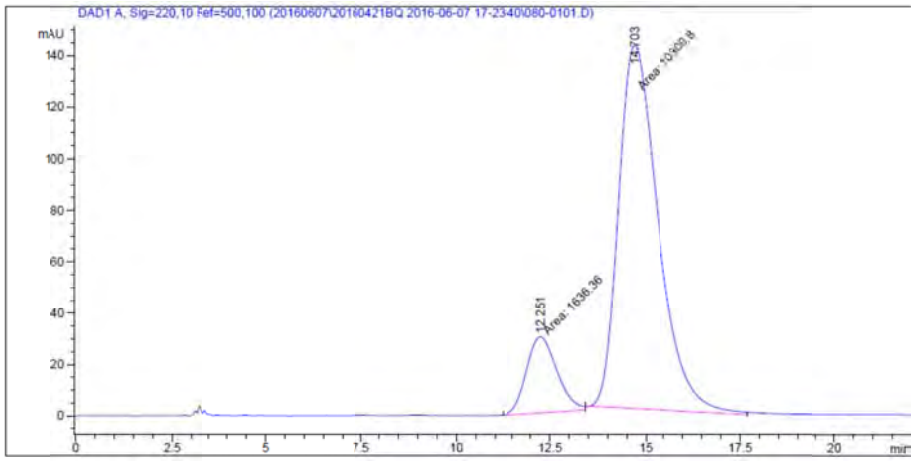




(*S*)-4-(1-(3,4-dimethoxyphenyl)-2-nitroethyl)phenol (10i): A white solid. 134 mg (88%). Mp = 38 – 42 °C. R_f = 0.25 (SiO_2, 50% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 3.1 (*c* = 0.76, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.09 (2H, d, *J* = 8.6 Hz), 6.74 – 6.84 (4H, m), 6.69 (1H, d, *J* = 2.0 Hz), 4.86 – 4.96 (2H, m), 4.79 (1H, t, *J* = 7.8 Hz), 4.63 – 4.84 (1H, br s), 3.85 (3H, s), 3.83 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.0, 149.0, 148.2, 131.9, 131.2, 128.7, 119.3, 115.8, 111.4, 111.2, 79.55, 55.79, 55.77, 47.77. HRMS (ESI+) Calcd for $C_{14}H_{13}NO_3$ [M + NH_4OAc + H]$^+$: 321.1450; Found [M + NH_4OAc + H]$^+$: 321.1444.

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

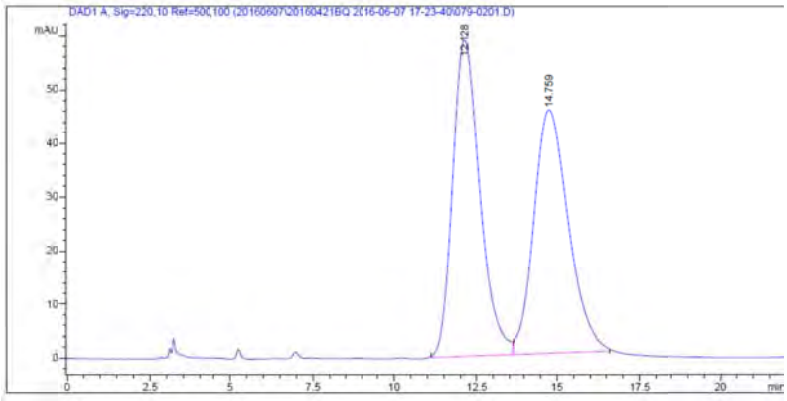
Asymmetric Reaction:

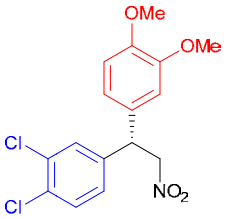


Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.251	MM	0.9204	1636.36292	29.63276	13.7082
2	14.703	MM	1.2165	1.03008e4	141.12134	86.2918
Totals :				1.19371e4	170.75410	

Racemic Reaction:

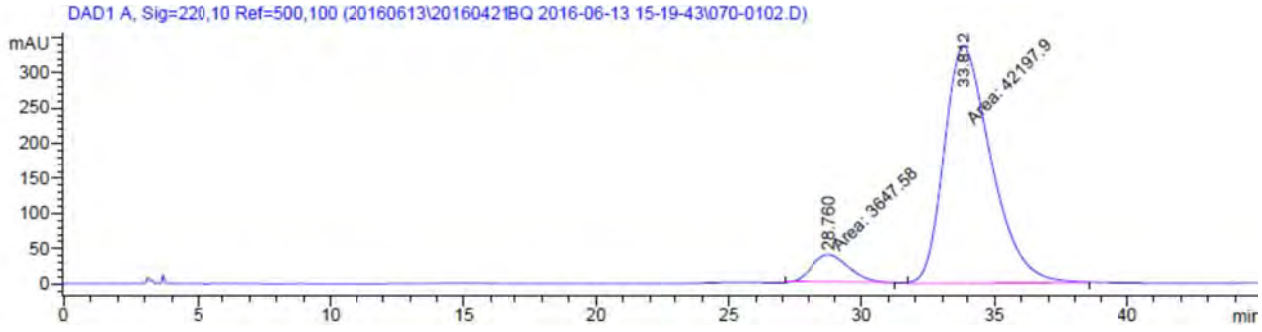




(*S*)-1,2-dichloro-4-(1-(3,4-dimethoxyphenyl)-2-nitroethyl)benzene (10j): A white solid. Mp 76 – 80 °C. CPME was used as the reaction solvent. 172 mg (96%). R_f = 0.24 (SiO_2, 30% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 5.7 (*c* = 0.56, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40 (1H, d, *J* = 8.4 Hz), 7.31 (1H, d, *J* = 2.1 Hz), 7.08 (1H, dd, *J* = 8.3 Hz, *J* = 2.1 Hz), 6.83 (1H, d, *J* = 8.3 Hz), 6.74 (1H, dd, *J* = 8.3 Hz, *J* = 2.1 Hz), 6.65 (1H, d, *J* = 2.1 Hz), 4.91 (2H, app. d, *J* = 8.0 Hz), 4.81 (1H, t, *J* = 8.0 Hz), 3.86 (3H, s), 3.84 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.5, 148.8, 139.7, 133.1, 131.8, 130.9, 130.4, 129.7, 126.9, 119.4, 111.6, 111.1, 78.84, 56.00, 55.93, 47.67. HRMS (ESI+) Calcd for $C_{16}H_{15}Cl_2NO_4$ $[M + NH_4]^+$: 373.0722; Found $[M + NH_4]^+$: 373.0712.

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

Asymmetric Reaction:

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Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  28.760 MM    1.5744 3647.57812   38.61463   7.9562
   2  33.812 MM    2.0813 4.21979e4   337.91702  92.0438

Totals :                  4.58455e4   376.53165
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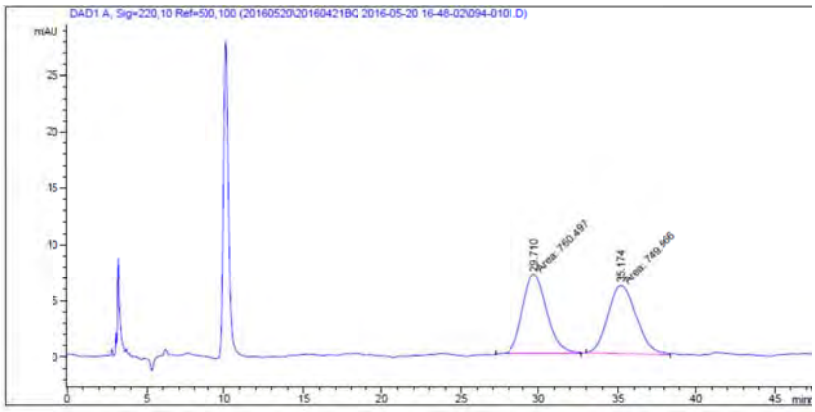
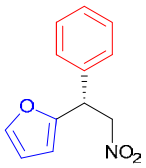
Racemic Reaction:

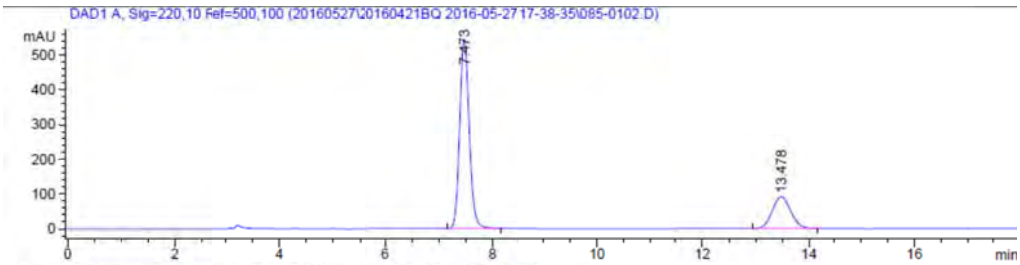




(*R*)-2-(2-nitro-1-phenylethyl)furan (10k): A colorless oil. 79.7 mg (74%). R_f = 0.22 (SiO_2, 8% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 34.2 (*c* = 0.26, $CHCl_3$); Lit.[5] (*S*-enantiomer): $[\alpha]^{20}_D$ = – 57 (*c* = 0.1, $CHCl_3$). Spectral data was consistent with the literature.[5]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

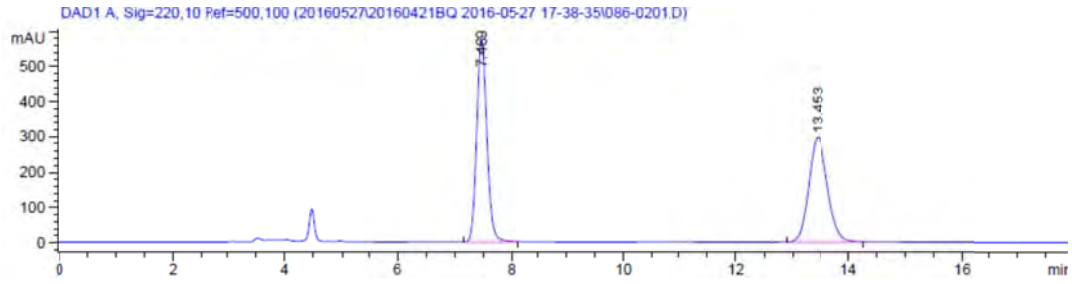
Asymmetric Reaction:



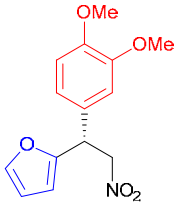
Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.473	BE	0.1900	6723.88672	544.86218	75.2442
2	13.478	BE	0.3762	2212.20190	91.46703	24.7558
Totals :				8936.08862	536.32922	

Racemic Reaction:



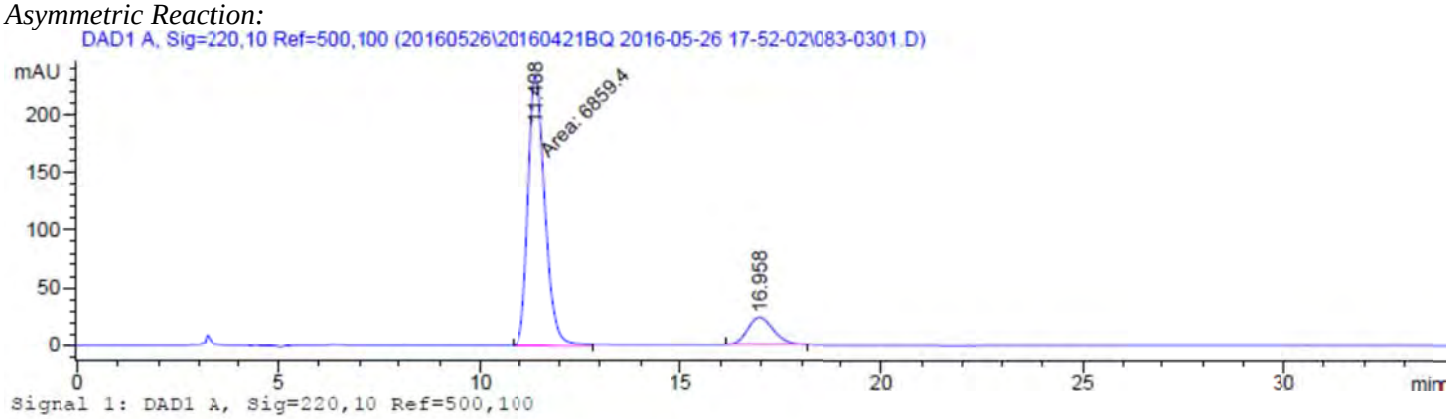
[5] J. Xing, G. Chen, P. Cao, J. Lia, *Eur. J. Org. Chem.* 2012, 1230.



(*R*)-2-(1-(3,4-dimethoxyphenyl)-2-nitroethyl)furan (10l): A colorless oil. 87.5 mg (63%). R_f = 0.25 (SiO_2, 30% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 42 (*c* = 0.49, $CHCl_3$); Lit.[4] (*S*-enantiomer): $[\alpha]^{20}_D$ = – 35.5 (*c* = 1.0, $CHCl_3$). Spectral data was consistent with the literature.[4]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

Asymmetric Reaction:

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Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.408 MM    0.4874 6859.40430  234.55846  86.3436
   2  16.958 BB    0.6856 1084.91125   23.98911  13.6564

Totals :                  7944.31555  258.54756
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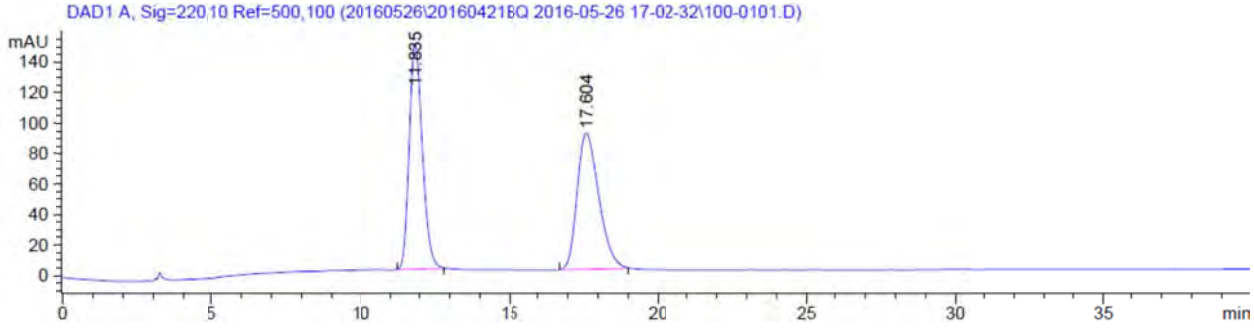
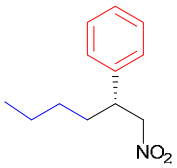
Racemic Reaction:

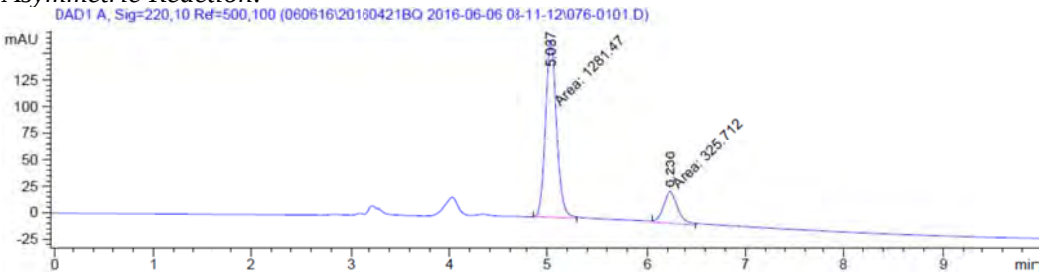




(*R*)-(1-nitrohexan-2-yl)benzene (10m): A colorless oil. 78.7 mg (76%). R_f = 0.23 (SiO_2, 5% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 14 (*c* = 0.53, $CHCl_3$); Lit.[4] (*S*-enantiomer): $[\alpha]^{20}_D$ = – 19.4 (*c* = 1.0, $CHCl_3$). Spectral data was consistent with the literature.[4]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

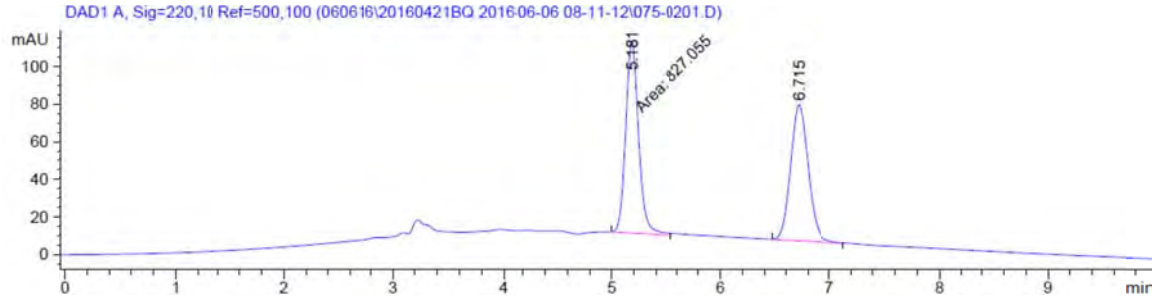
Asymmetric Reaction:

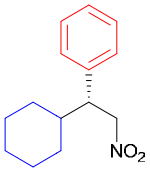


Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.037	MM	0.1281	1281.47119	166.76703	79.7340
2	6.236	MM	0.1804	325.71164	30.08503	20.2660
Totals :				1607.18283	196.85206	

Racemic Reaction:

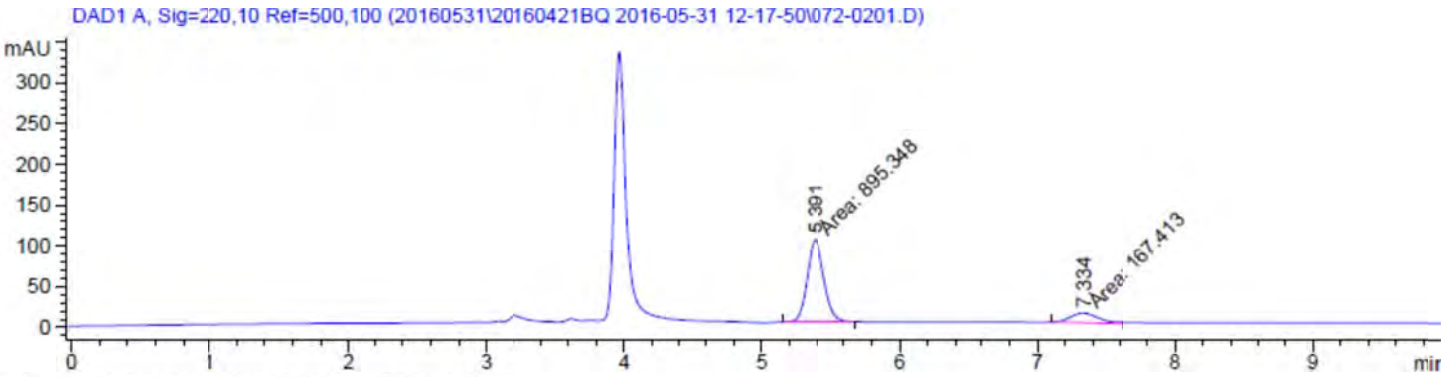




(*R*)-(1-cyclohexyl-2-nitroethyl)benzene (10n): A colorless oil. The nitroalkene starting material could not be separated from the product by chromatography; therefore the purity of the isolated material was assayed by ^{1}HNMR spectroscopy using dimethylfumarate as the analytical standard: 83.2 mg; 49.2 wt% purity (35%). R_f = 0.22 (SiO_2, 5% EtOAc in hexanes). $[\alpha]^{20}_D$ = + 32.4 (*c* = 0.27, $CHCl_3$); Lit.[4] (*S*-enantiomer): $[\alpha]^{20}_D$ = – 28.9 (*c* = 1.0, $CHCl_3$). Spectral data was consistent with the literature.[4]

Chiral HPLC (Chiralcel OD-H, 55:45 heptane:isopropanol, 220 nm) analysis:

Asymmetric Reaction:




```
Signal 1: DAD1 A, Sig=220,10 Ref=500,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.391 MM    0.1476  895.34753  101.10936  84.2474
   2   7.334 MM    0.2337  167.41266   11.94150  15.7526

Totals :                  1062.76019  113.05086
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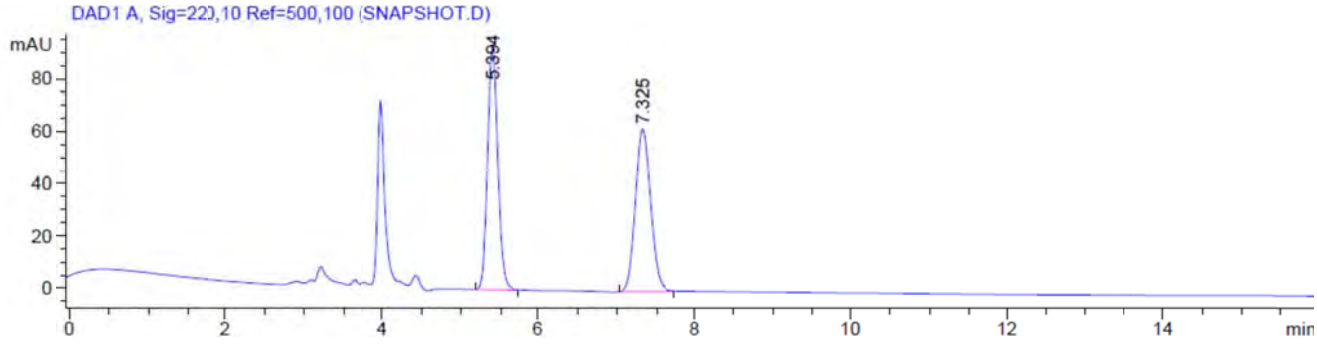
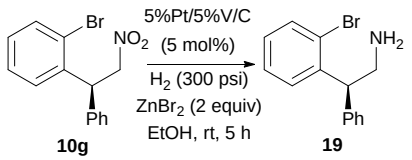
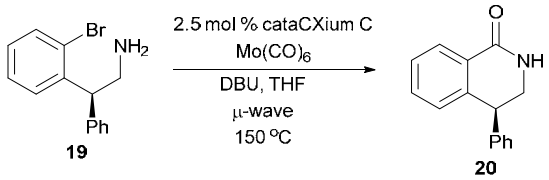
Racemic Reaction:



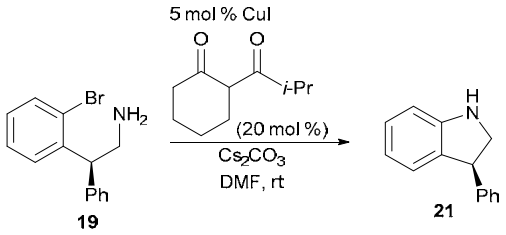


To an Endeavor tube was charged 0.500 g (1.63 mmol) of **10g**, 0.640 g (0.0728 mmol Pt) of 5%Pt/5%V on carbon (55.6 wt% wetted), 0.736 g (3.27 mmol) of $ZnBr_2$, and 5 mL of EtOH. The tube was then placed in an Endeavor system with an overhead mechanical stirrer and sealed. The mixture was then hydrogenated at 300 psi H_2 at rt with an agitator speed of 750 rpm for 5 h. An aliquot was checked by UPLCMS to confirm complete consumption of **10g**. The mixture was filtered through celite using MeOH, and then, concentrated *in vacuo*. To the crude residue was added 6 mL of water and 4 mL of 28 wt% NH_4OH, and the mixture was extracted with CH_2Cl_2 (3x15 mL). The combined organics were washed with satd. brine (1x15mL), dried with Na_2SO_4, and volatile materials removed *in vacuo* to provide 397 mg (88%) of amine **19** as a slight yellow oil. $[\alpha]^{20}_D$ = – 24.5 (*c* = 0.28, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.57 (1H, d, *J* = 8.2 Hz), 7.19 – 7.34 (7H, m), 7.04 – 7.10 (1H, m), 4.52 (1H, t, *J* = 7.5 Hz), 3.30 (2H, d, *J* = 7.5 Hz), 1.34 – 1.61 (2H, br s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.8, 141.4, 133.3, 128.63, 128.59, 128.47, 128.0, 127.7, 126.7, 125.8, 53.58, 46.73. HRMS (ESI+) Calcd for $C_{14}H_{14}BrN$ $[M + H]^+$: 276.0388; Found $[M + H]^+$: 276.0385.



The reaction conditions were adapted from the literature procedure.[6] To a conical 2 mL microwave vial with stir bar was charged 50.0 mg (0.181mmol) of **19**, 4.2 mg (0.0045 mmol) *trans*-Bis(acetato)bis[*o*-(di-*o*-tolylphosphino)benzyl]dipalladium (cataCXium C), 47.8 mg (0.181 mmol) $Mo(CO)_6$, 0.081 mL (0.54 mmol) DBU, and 1.25 mL of THF. The vial was then sealed and reacted in a Biotage microwave at 150 °C for 15 min. The crude mixture was filtered through celite using EtOAc and concentrated *in vacuo*. The crude residue was purified by flash chromatography (Biotage, gradient, hexanes to 40% EtOAc in hexanes) to afford 24.4 mg (60%) of **20** as a white solid. Mp 140 – 144 °C. R_f = 0.19 (SiO_2, 50% EtOAc in hexanes); $[\alpha]^{20}_D$ = – 23.3 (*c* = 0.15, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (1H, dd, *J* = 7.0 Hz, *J* = 1.9 Hz), 7.26 – 7.46 (5H, m), 7.18 (2H, d, *J* = 6.8 Hz, 1H), 6.97 (1H, d, *J* = 6.8 Hz), 6.22 (1H, br s), 4.32 (1H, dd, *J* = 7.4 Hz, *J* = 5.5 Hz), 3.81 (1H, ddd, *J* = 12 Hz, *J* = 5.5 Hz, *J* = 3.1 Hz), 3.71 (1H, ddd, *J* = 12 Hz, *J* = 7.4 Hz, *J* = 3.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.0, 141.3, 140.6, 132.5, 129.0, 128.8, 128.6, 128.2, 127.7, 127.5, 127.4, 47.24, 44.29. HRMS (ESI+) Calcd for $C_{15}H_{13}NO$ $[M + H]^+$: 224.1075; Found $[M + H]^+$: 224.1073.

[6] J. Wannbert, M. Larhed, *J. Org. Chem.* 2003, **68**, 5750 – 5753.

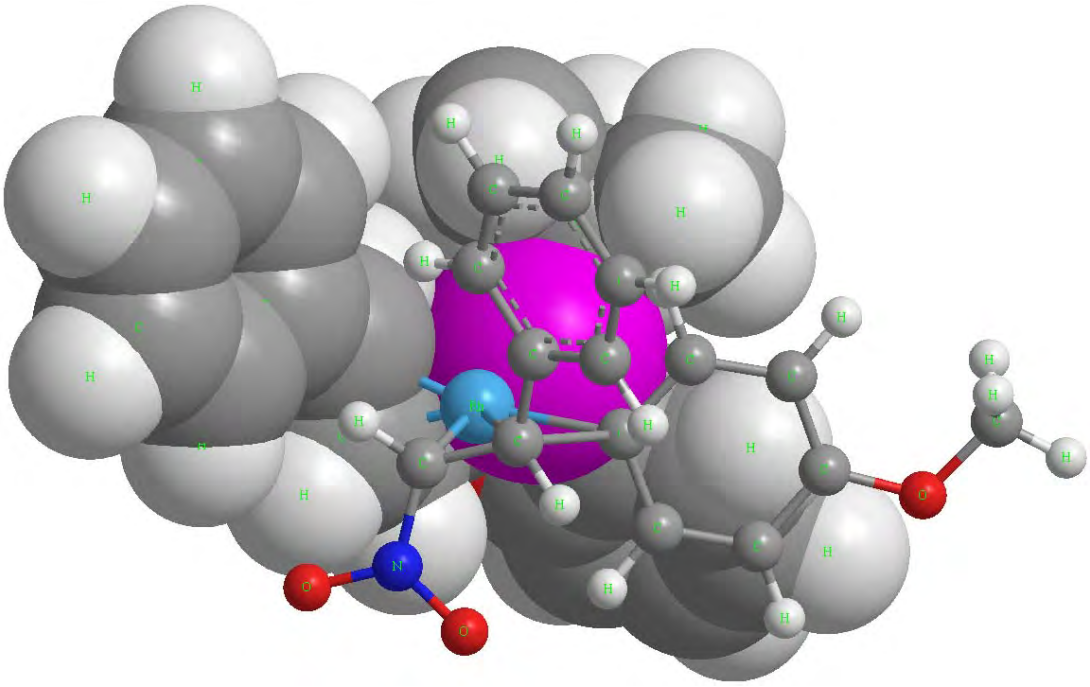


The reaction conditions were adapted from the literature procedure.[7] To a crimp-cap vial equipped with a magnetic stir-bar was charged 127.4 mg (0.461 mmol) of **19**, 4.4 mg (0.023 mmol) of CuI, 15 mg (0.092 mmol) of 2-isobutyrylcyclohexan-1-one, and 0.300 g (0.923 mmol) of Cs_2CO_3. The vial was sealed and inerted with Ar using vacuum-purge cycles (3x). DMF (1.2 mL) was added, and the reaction mixture was stirred at rt for 2 h. Water (20 mL) and MTBE (15 mL) were added. The layers were separated, and the aqueous phase was extracted with MTBE (15 mL). Combined organics were washed with water (10 mL), brine (10 mL), dried with $MgSO_4$, and concentrated *in vacuo*. Purification of the crude mixture by flash chromatography (gradient, 0-20% EtOAc in hexanes) afforded 88.2 mg (97%) of **21** as a pale yellow oil. Spectral data was consistent with the literature.[8] R_f = 0.43 (20% EtOAc/hexanes); $[\alpha]^{20}_D$ = + 24.6 (*c* = 0.30, $CHCl_3$).

[7] A. Shafir, S. L. Buchwald *J. Am. Chem. Soc*. 2006, **128**, 8742 – 8743.
[8] K. F. Johnson, R. Van Zeeland, L. M. Stanley, *Org. Lett.* 2013, **15**, 2798 – 2801.

Transition State Calculations

Transition state searches were performed with the Gaussian 09 program[9] on a Unix HPC platform. All structures were optimized at the B3LYP level of theory employing the LANL2DZ basis set.[10] All optimized structures were subjected to a frequency test to verify a single imaginary frequency was found and that the imaginary vibrational mode corresponds with the expected reaction pathway. The resulting structure was subjected to an IRC calculation (B3LYP/LANL2DZ) to verify a clean reaction pathway from the starting material (nitro-olefin complexes) to the addition product.



Cartesian Coordinates

Rh	0.49067200	-0.30869700	-0.45055900
P	-1.43094100	-1.31211100	0.70033200
C	-1.28019500	-3.14025400	0.10430900
C	-1.72523300	-1.40294400	2.63731700
C	-3.15872400	-1.29759900	-0.00818100
C	-4.04844800	-0.22548100	-0.18631300

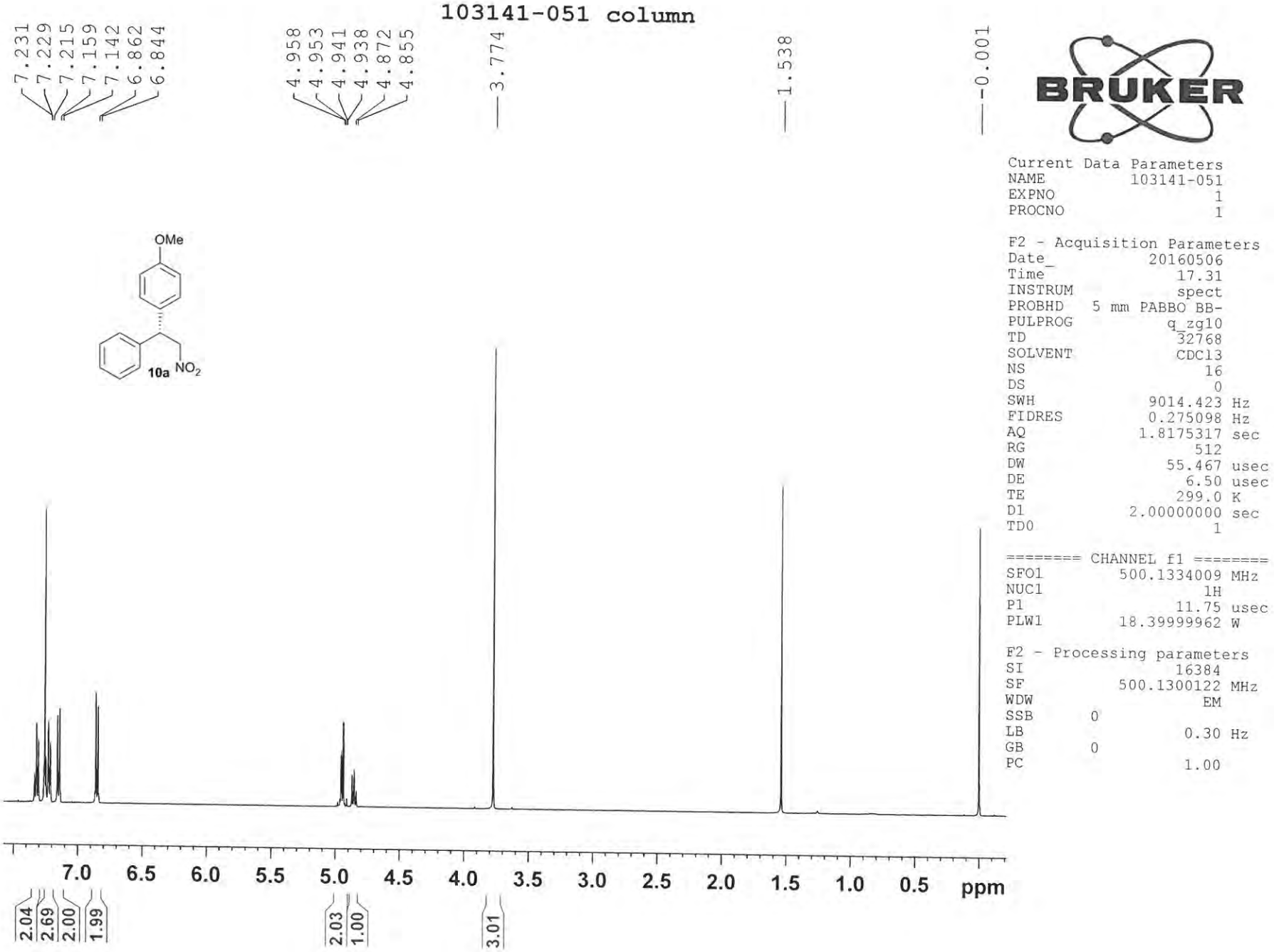
[9] Gaussian 09, Revision C.01, M. J. Frisch, W. G. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, D. J. Fox, Gaussian, Inc., Wallingford, CT 2010

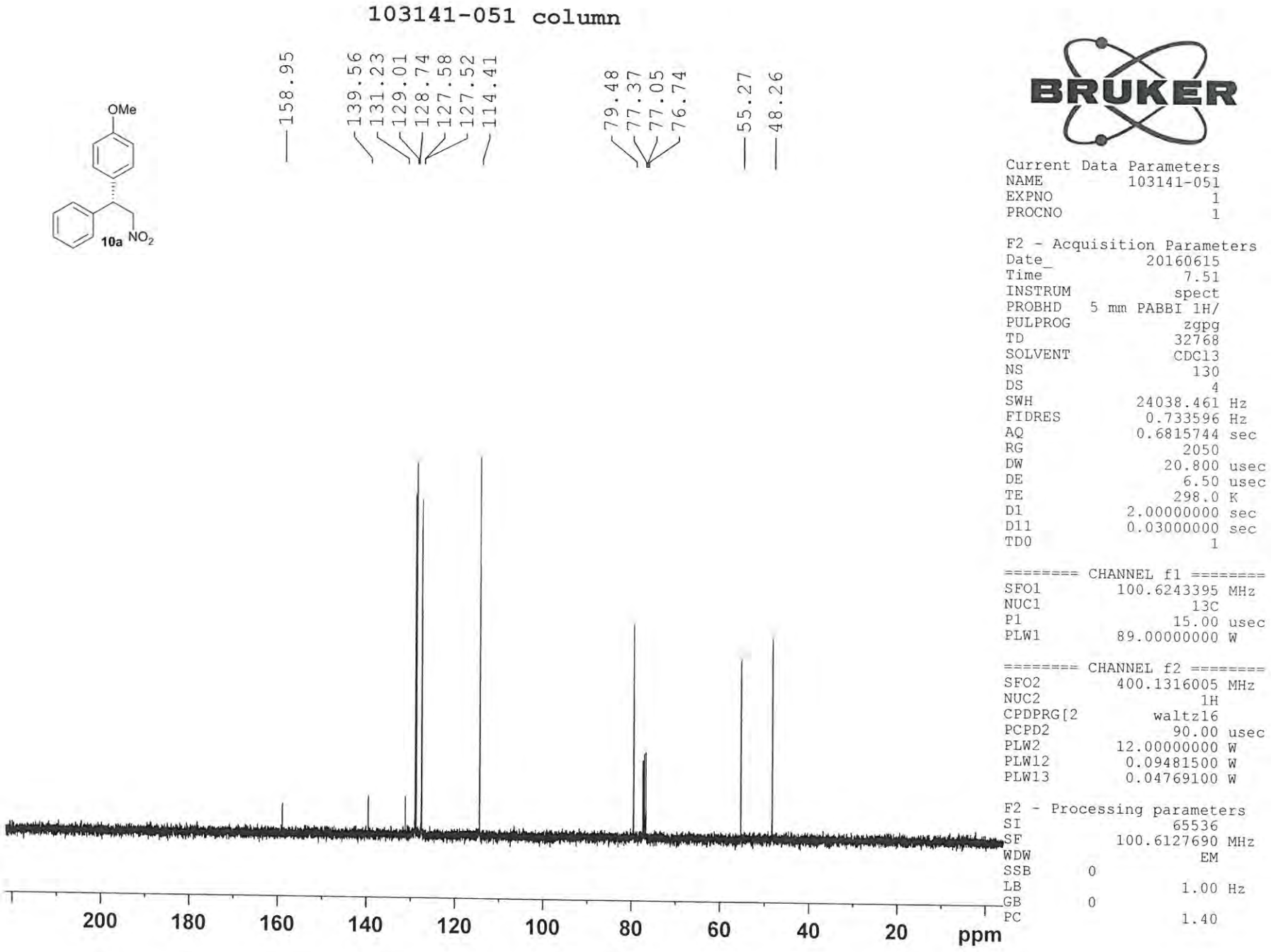
[10] (a) T. H. Dunning Jr. and P. J. Hay, in *Modern Theoretical Chemistry*, Ed. H. F. Schaefer III, Vol. 3, Plenum, New York, 1976, 1–28; (b) P. J. Hay and W. R. Wadt *J. Chem. Phys.* 1985, **82**, 270-283; (c) P. J. Hay, W. R. Wadt, *J. Chem. Phys.* 1985, **82**, 299-310; (d) W. R. Wadt, P. J. Hay, *J. Chem. Phys.* 1985, **82**, 284-298.

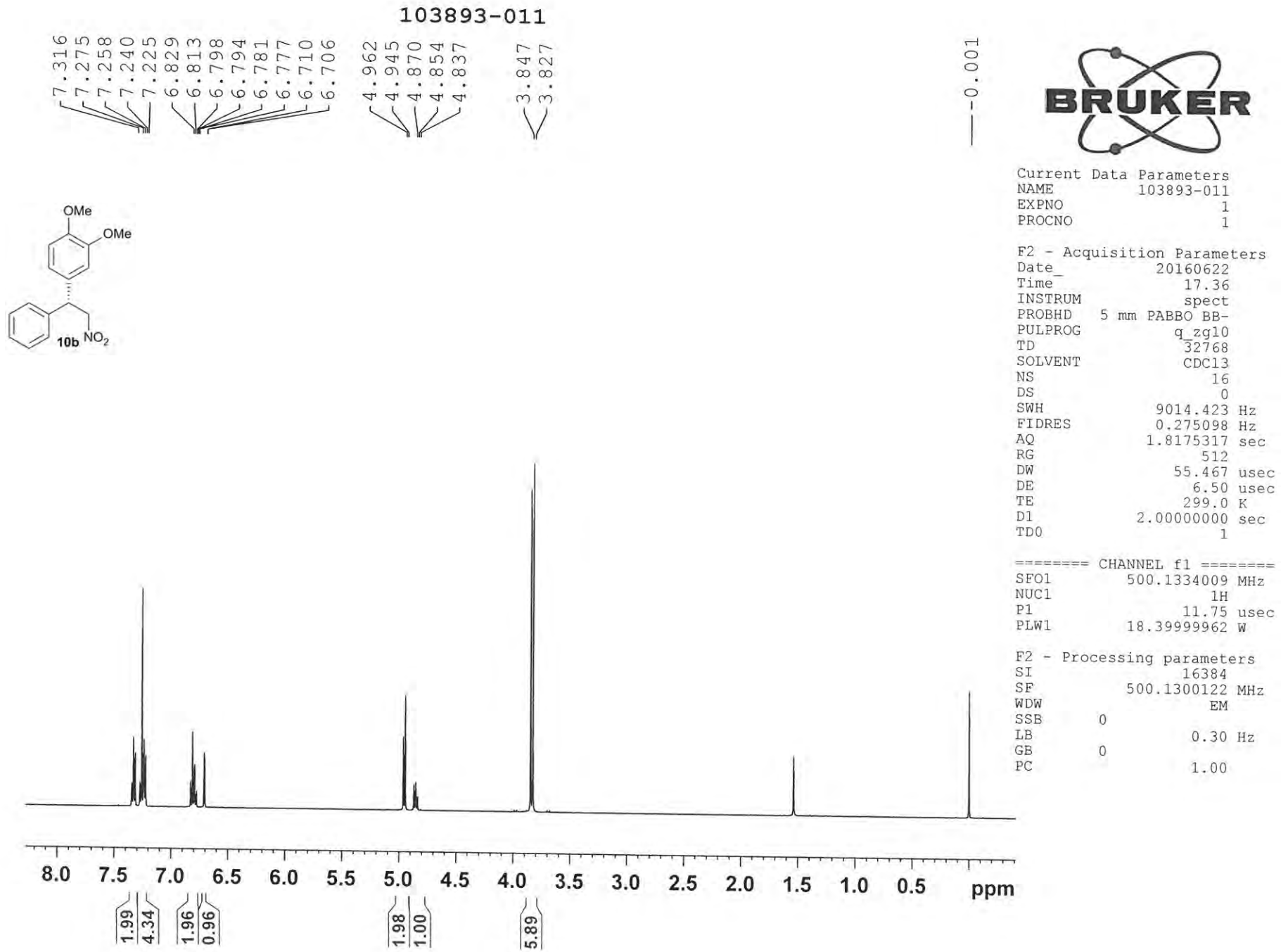
H	-3.74937000	0.78546000	0.07190100
C	-5.32934300	-0.46494200	-0.72316600
H	-6.01701700	0.36364500	-0.86746100
C	-5.71242100	-1.77380600	-1.08563100
H	-6.70011700	-1.95135100	-1.50441800
C	-4.82736900	-2.85671000	-0.92447800
H	-5.10043900	-3.86844500	-1.20724400
C	-3.55823100	-2.59840100	-0.38828400
C	-0.43434600	-3.17045700	-1.18154900
H	-0.22092500	-4.21937900	-1.44214500
H	-1.03868000	-2.76366700	-2.00375000
C	1.61607200	-2.23558900	0.10353800
C	0.87470500	-2.38972500	-1.09361700
C	-0.36716000	-1.72001300	3.30859700
H	0.40523000	-0.99683700	3.01830600
H	-0.48355400	-1.67533100	4.40058100
H	-0.01255500	-2.72945800	3.05961100
C	-2.77074000	-2.48236800	3.00063600
H	-2.89704100	-2.50566800	4.09298600
H	-3.74748300	-2.26559200	2.55390800
H	-2.46657500	-3.48493500	2.67823300
C	-2.22440900	-0.01048400	3.09443300
H	-1.50422100	0.77806800	2.85001100
H	-3.18470000	0.24597500	2.63068900
H	-2.37048900	-0.01784900	4.18412400
H	-0.87862300	-3.79498000	0.88109500
O	-2.64406700	-3.64148500	-0.18450600
H	1.40556400	-2.33536300	-2.04404700
H	1.14703500	-2.54787700	1.03458200
C	3.09196100	-2.14209500	0.24072900
C	3.99020900	-2.18198600	-0.85653100
C	3.64143000	-2.06890800	1.54690300
C	5.37910300	-2.13885500	-0.64740000
H	3.60971500	-2.24425500	-1.87135600
C	5.03001000	-2.02713300	1.75572900
H	2.96954600	-2.05220600	2.40409800
C	5.90960500	-2.06006800	0.65585200
H	6.04772300	-2.17024000	-1.50454800
H	5.42403100	-1.97682600	2.76847900
H	6.98546300	-2.03088800	0.81045900
C	2.09066100	0.51160600	-1.60559100
C	1.63383100	1.78876900	-1.10921400
H	3.02119800	0.07033100	-1.27019500
O	2.50833700	-0.76421400	-3.53804600
C	-0.42999000	1.65713400	-0.50497100
C	-0.67503800	2.42645600	0.65455800
C	-1.29447700	1.84733600	-1.62152600
C	-1.76786300	3.31230800	0.73760800
H	-0.00233300	2.35251200	1.50598300
C	-2.38460500	2.72485500	-1.55251100
H	-1.09794800	1.32143500	-2.55149600
C	-2.62932700	3.45824700	-0.37102600

H	-1.92464500	3.88106200	1.64964600
H	-3.04758200	2.86568700	-2.40130200
O	-3.74196700	4.30561100	-0.40437000
C	-4.02140300	5.13820000	0.75668500
H	-4.91036200	5.71413400	0.49157900
H	-4.22941900	4.52735600	1.64672800
H	-3.18693800	5.82171500	0.96722300
H	1.23664100	2.44955000	-1.87416900
O	1.06847900	0.96762000	-3.71116700
N	1.87751700	0.23734700	-3.02610000
C	2.36280600	2.48496100	-0.01353300
C	2.37074200	3.90000500	-0.00554600
C	3.07341800	1.79621300	0.99817700
C	3.08396600	4.61103500	0.97436800
H	1.81872300	4.44067100	-0.77163100
C	3.78885000	2.50633600	1.97578100
H	3.06070200	0.71018000	1.02681500
C	3.79861800	3.91616100	1.96947300
H	3.08471200	5.69827300	0.96102100
H	4.33706900	1.96221200	2.74105900
H	4.35265000	4.46340300	2.72848500

Zero-point correction=	0.643333 (Hartree/Particle)
Thermal correction to Energy=	0.683409
Thermal correction to Enthalpy=	0.684353
Thermal correction to Gibbs Free Energy=	0.569310
Sum of electronic and zero-point Energies=	-1826.629137
Sum of electronic and thermal Energies=	-1826.589061
Sum of electronic and thermal Enthalpies=	-1826.588117
Sum of electronic and thermal Free Energies=	-1826.703161


103141-051 column
7.231
7.229
7.215
7.159
7.142
6.862
6.844
4.958
4.953
4.941
4.938
4.872
4.855
3.774
1.538
-0.001
OMe
10a
NO2
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.04
2.69
2.00
1.99
2.03
1.00
3.01
BRUKER
Current Data Parameters
NAME 103141-051
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160506
Time 17.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W
F2 - Processing parameters
SI 16384
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


103141-051 column
158.95
139.56
131.23
129.01
128.74
127.58
127.52
114.41
79.48
77.37
77.05
76.74
55.27
48.26
OMe
10a
NO2
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME 103141-051
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160615
Time 7.51
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 130
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 15.00 usec
PLW1 89.00000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.09481500 W
PLW13 0.04769100 W
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

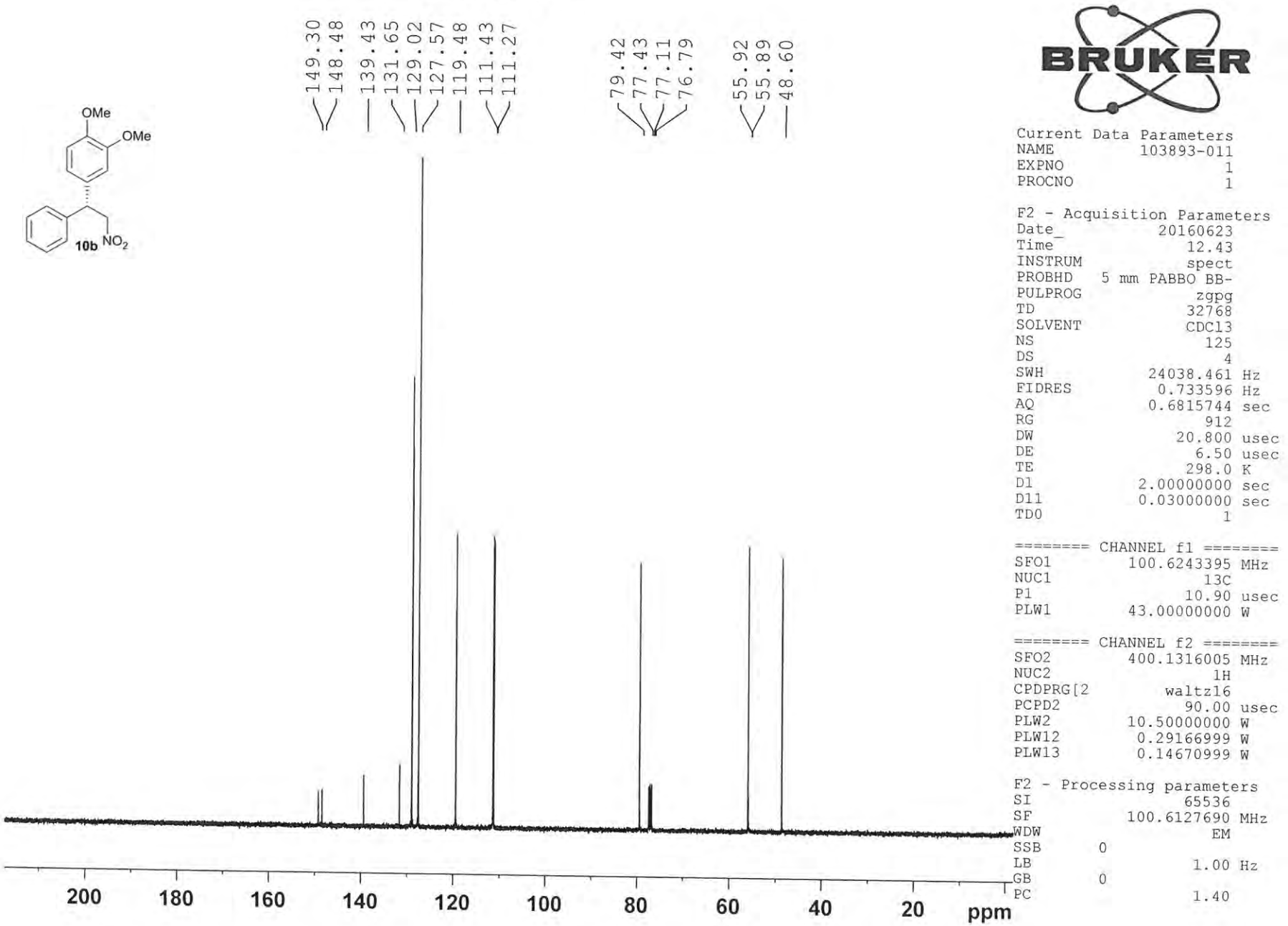
Current Data Parameters
NAME 103893-011
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160622
Time 17.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W

F2 - Processing parameters
SI 16384
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-011



BRUKER

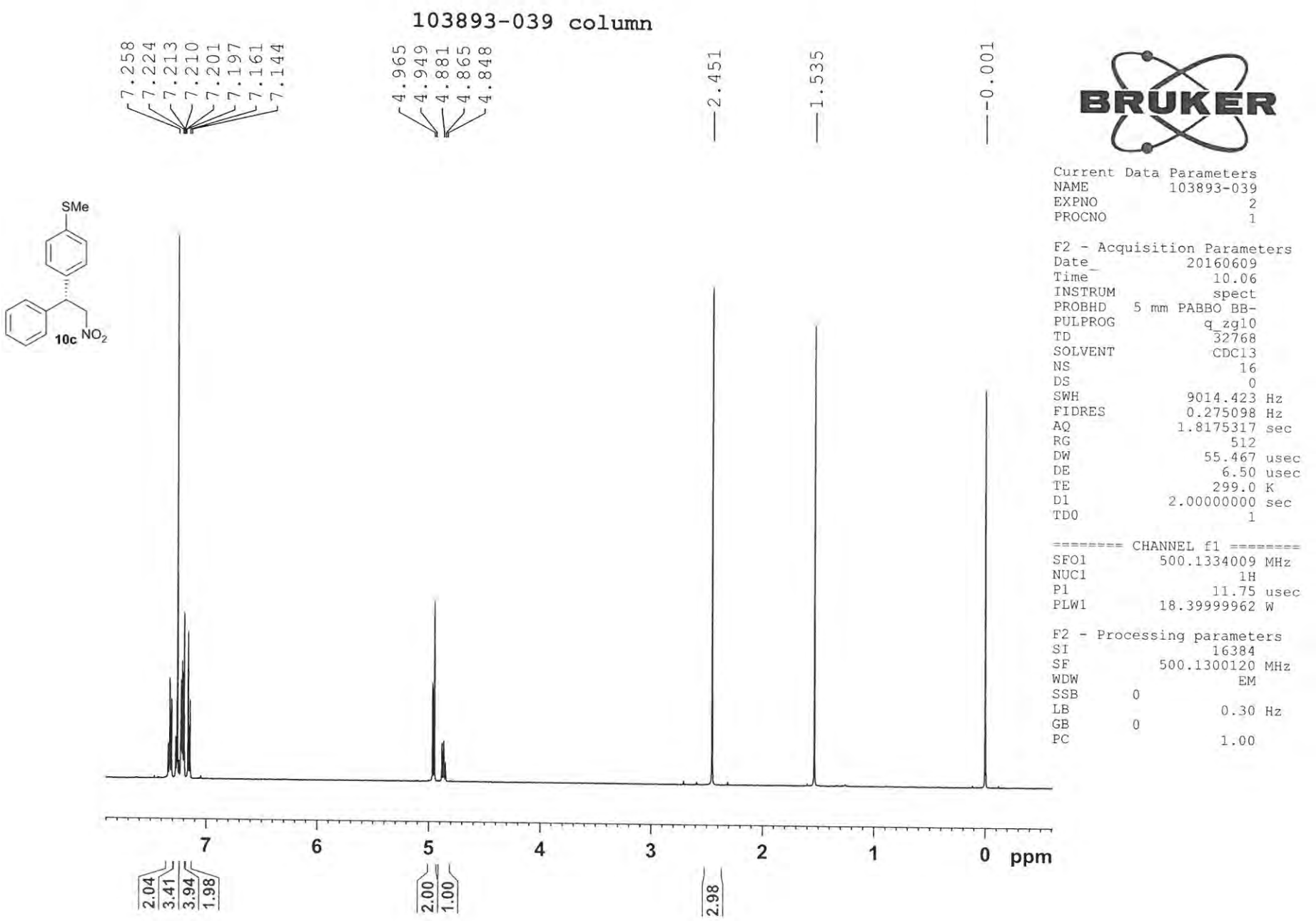
Current Data Parameters
NAME 103893-011
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160623
Time 12.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 912
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

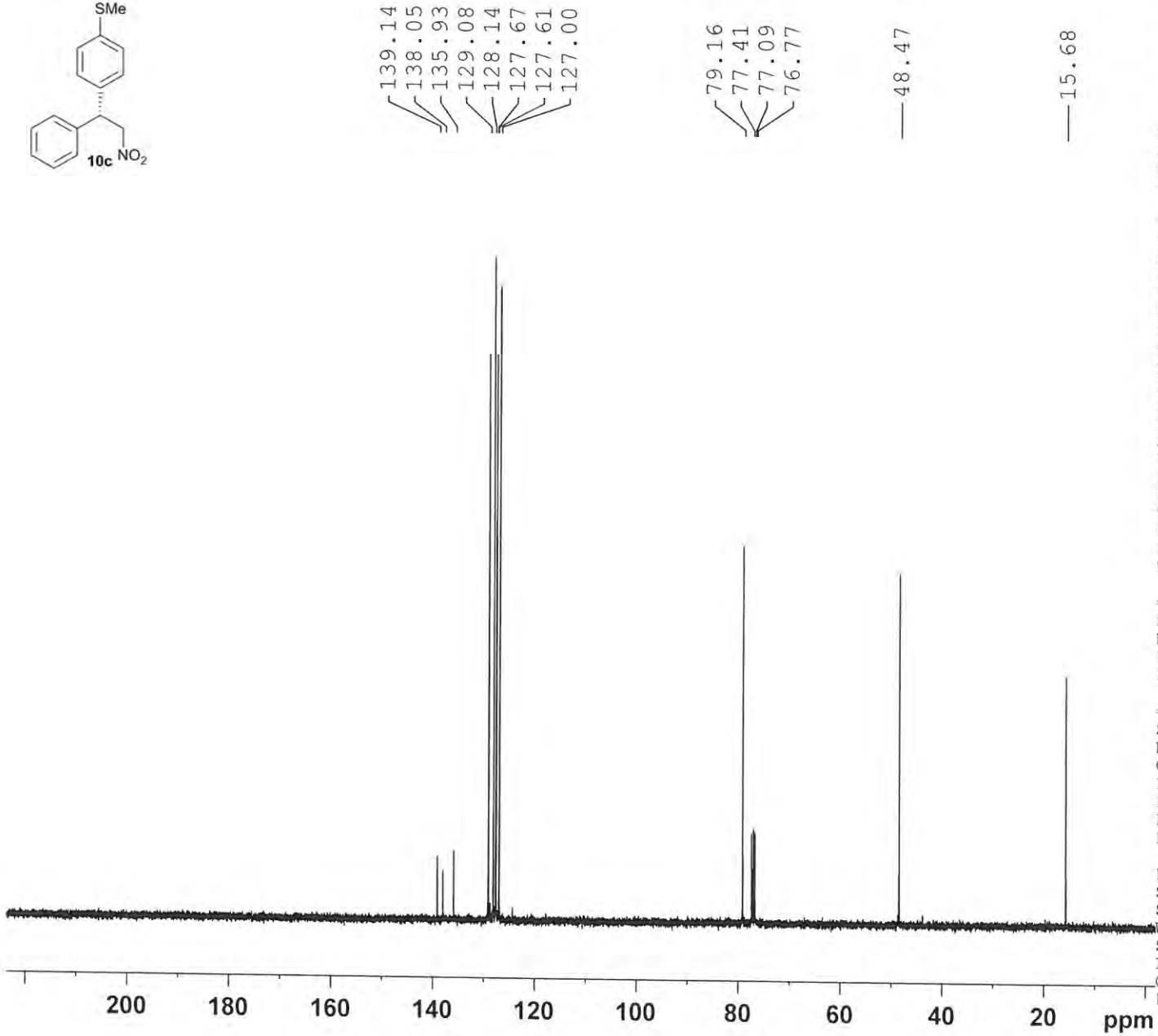
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


103893-039 column
7.258
7.224
7.213
7.210
7.201
7.197
7.161
7.144
4.965
4.949
4.881
4.865
4.848
2.451
1.535
0.001
SMe
10c NO2
7
6
5
4
3
2
1
0 ppm
2.04
3.41
3.94
1.98
2.00
1.00
2.98
Current Data Parameters
NAME 103893-039
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160609
Time 10.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W
F2 - Processing parameters
SI 16384
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-039



BRUKER

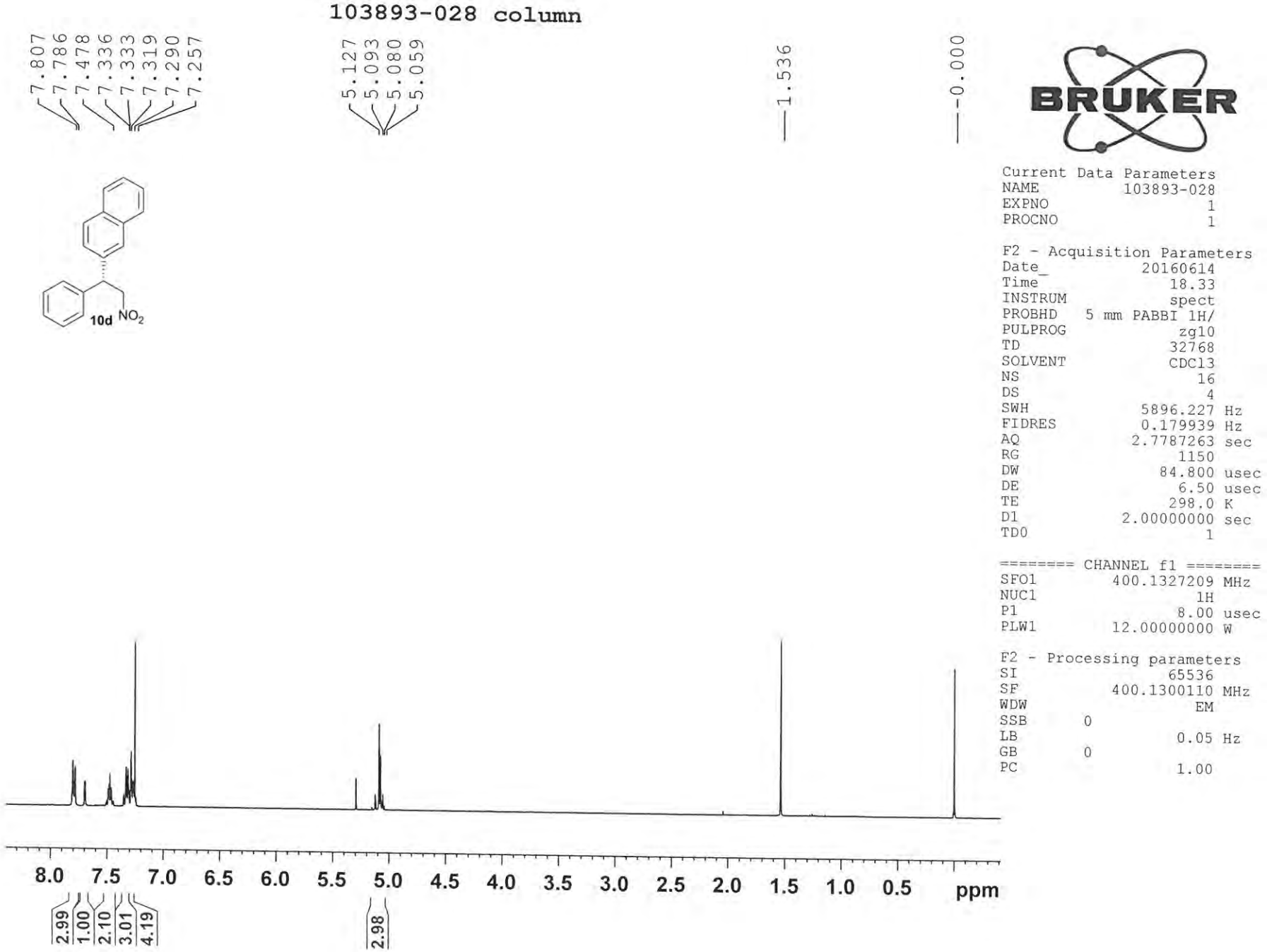
Current Data Parameters
NAME 103893-039
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160622
Time 15.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 645
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

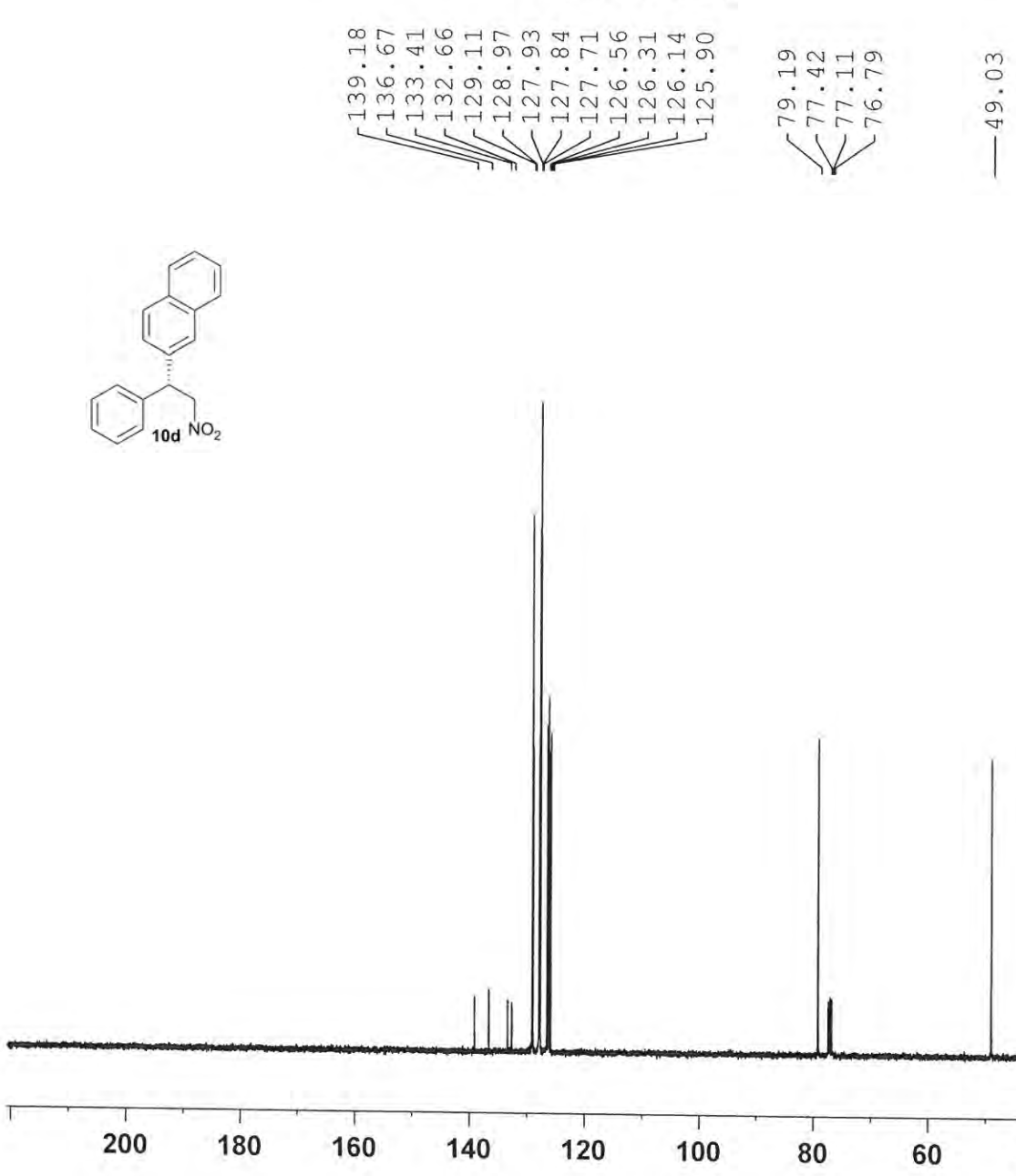
Current Data Parameters
NAME 103893-028
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160614
Time 18.33
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 5896.227 Hz
FIDRES 0.179939 Hz
AQ 2.7787263 sec
RG 1150
DW 84.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1327209 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300110 MHz
WDW EM
SSB 0
LB 0.05 Hz
GB 0
PC 1.00

103893-028



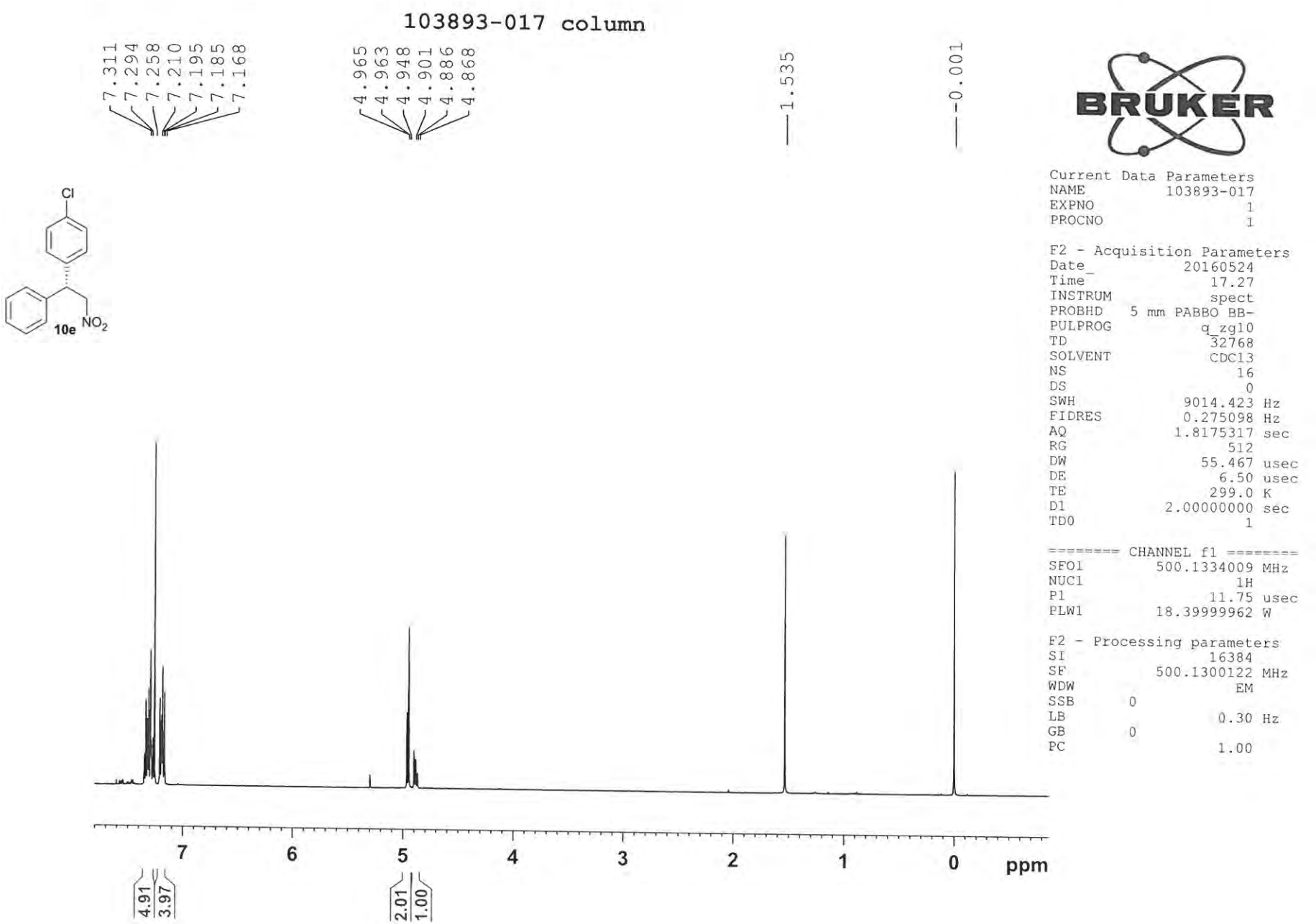
Current Data Parameters
NAME 103893-028
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160623
Time 12.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 724
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

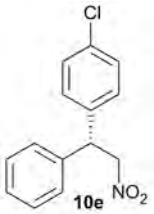
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

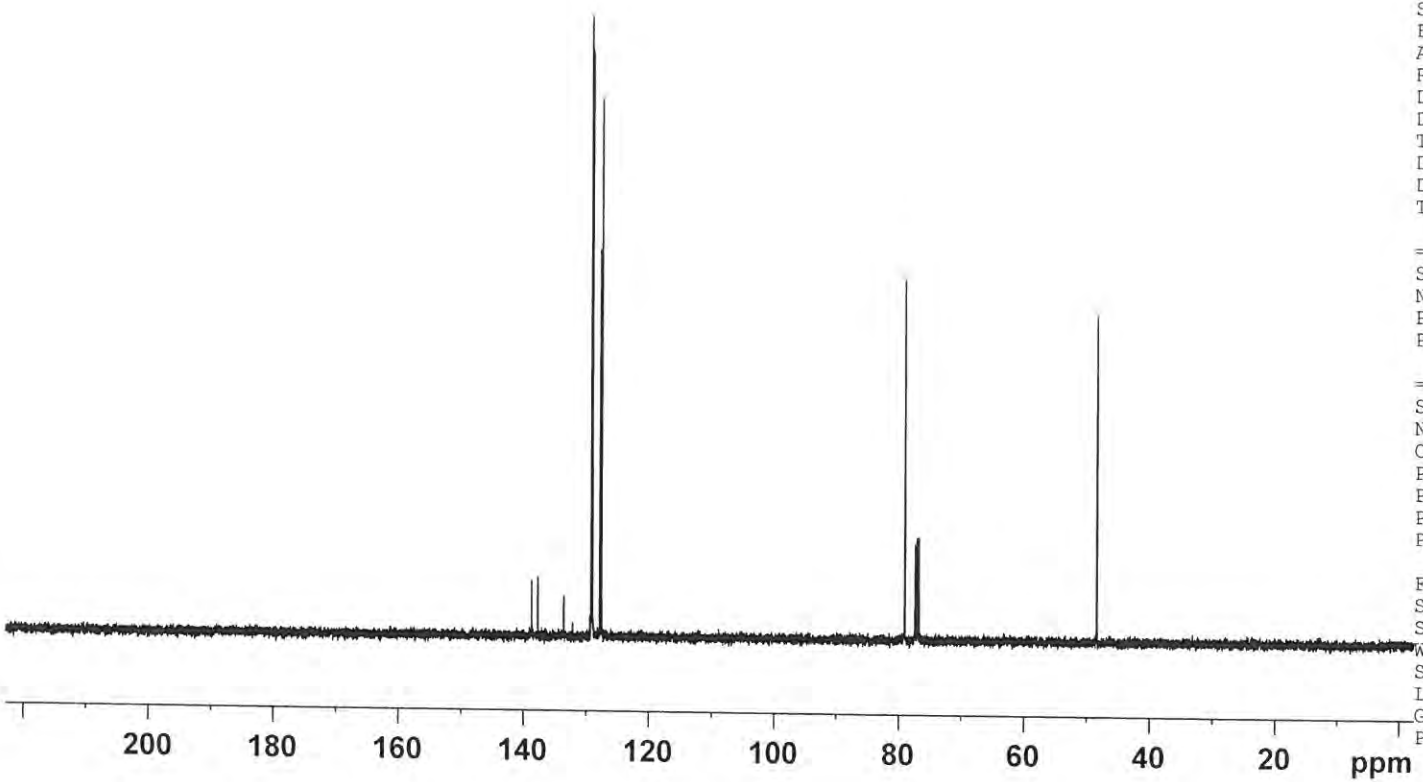

103893-017 column
7.311
7.294
7.258
7.210
7.195
7.185
7.168
4.965
4.963
4.948
4.901
4.886
4.868
1.535
0.001
Cl
10e
NO2
7
6
5
4
3
2
1
0
ppm
4.91
3.97
2.01
1.00
Current Data Parameters
NAME 103893-017
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160524
Time 17.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W
F2 - Processing parameters
SI 16384
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-017



10e

138.72
137.74
133.57
129.23
129.18
129.05
127.84
127.58
79.01
77.38
77.06
76.75
48.33



BRUKER

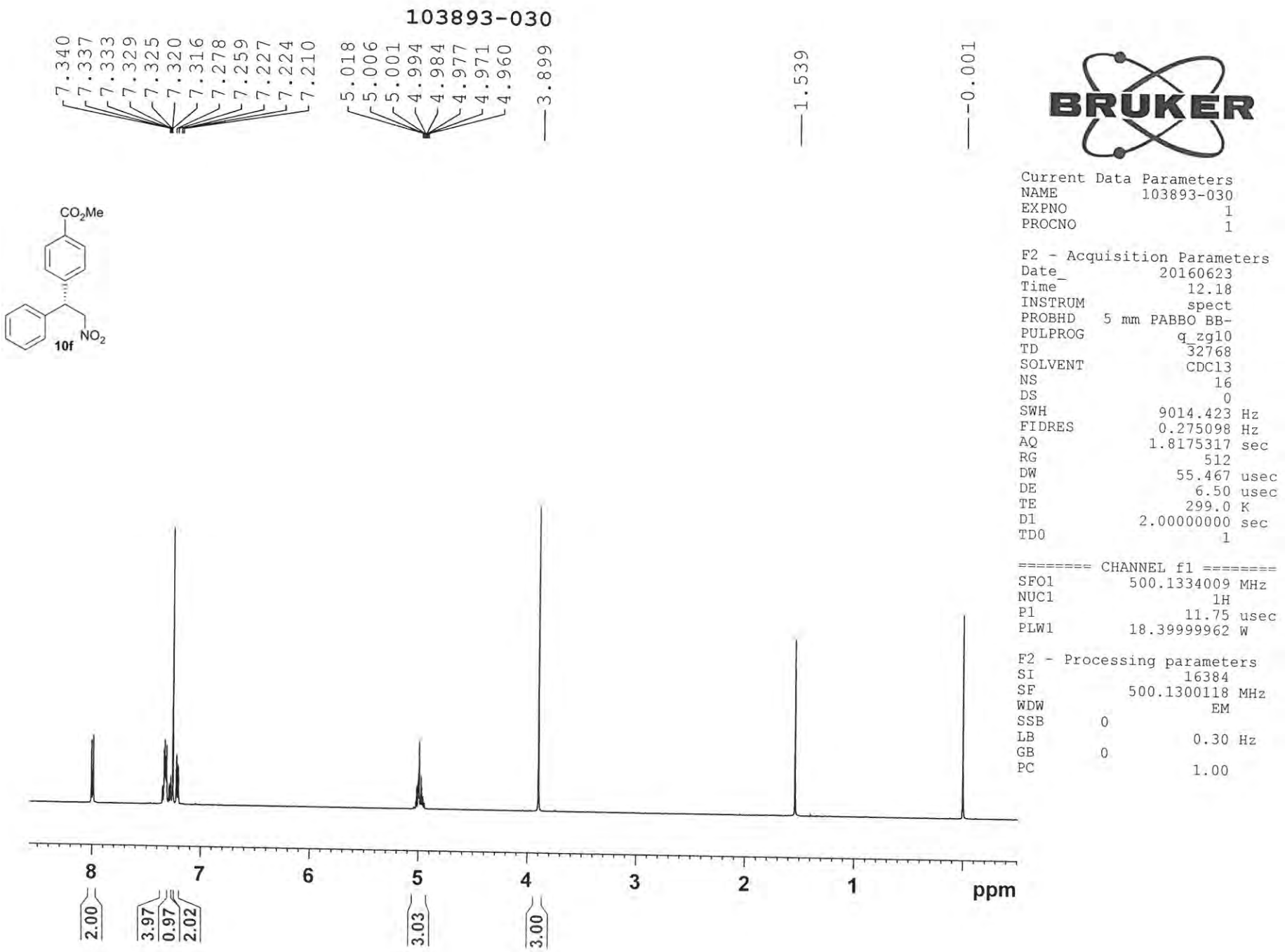
Current Data Parameters
NAME 103893-017
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160622
Time 15.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 645
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



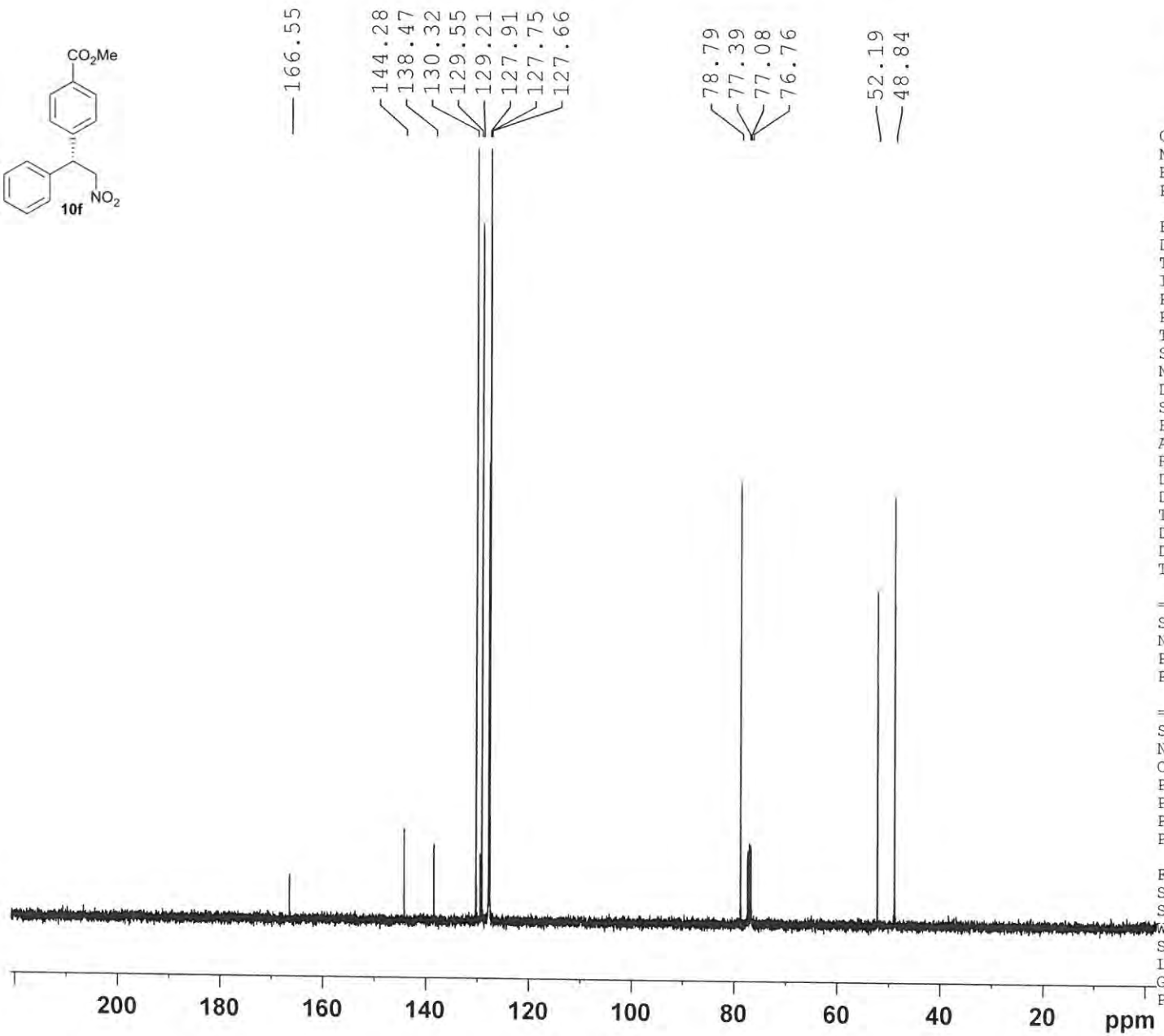
Current Data Parameters
NAME 103893-030
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160623
Time 12.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W

F2 - Processing parameters
SI 16384
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-030



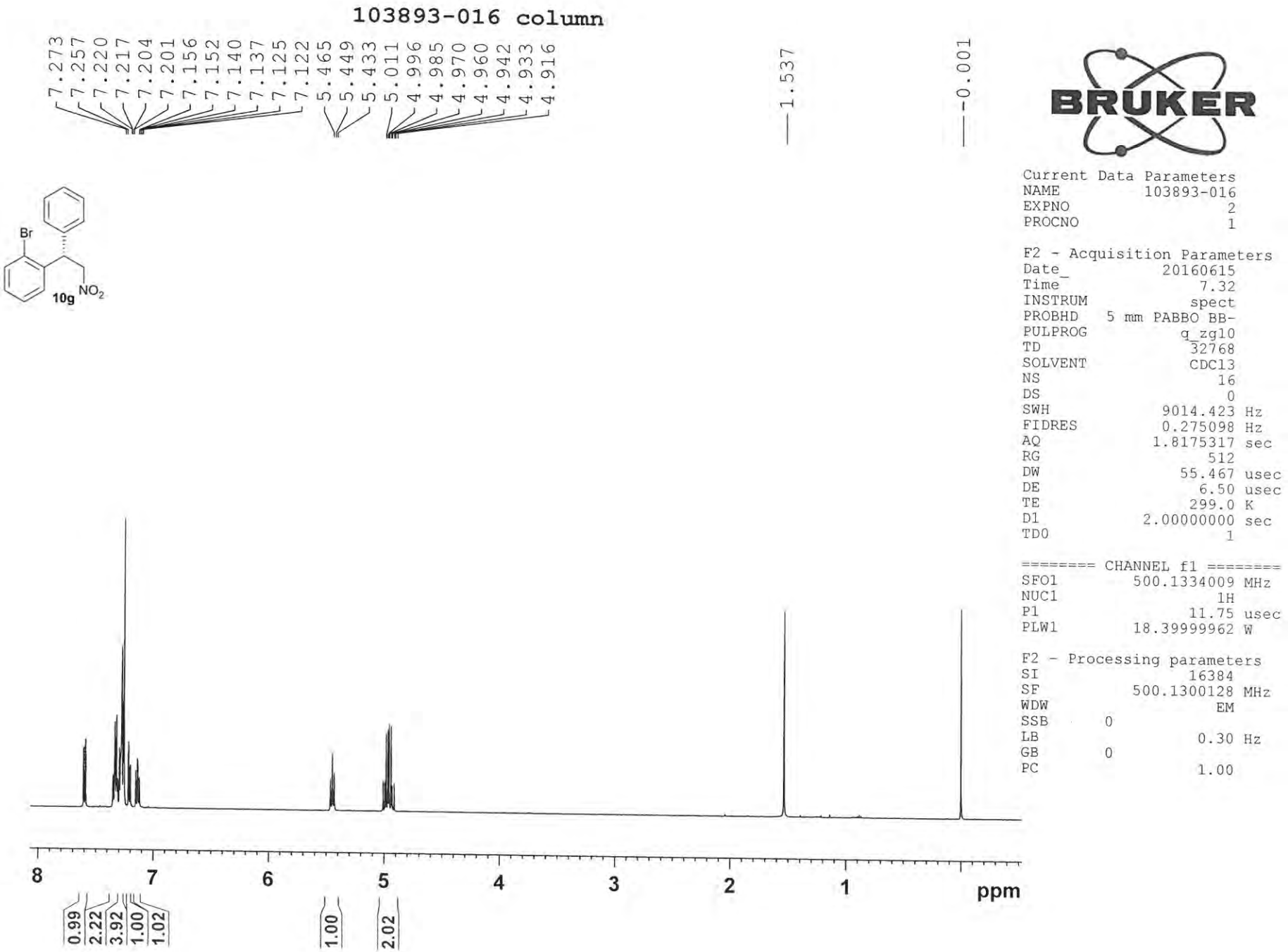
Current Data Parameters
NAME 103893-030
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160623
Time 13.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 1030
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

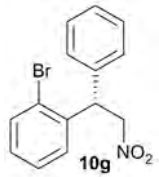
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


103893-016 column
7.273
7.257
7.220
7.217
7.204
7.201
7.156
7.152
7.140
7.137
7.125
7.122
5.465
5.449
5.433
5.011
4.996
4.985
4.970
4.960
4.942
4.933
4.916
1.537
0.001
Br
10g
NO2
8
7
6
5
4
3
2
1
ppm
0.99
2.22
3.92
1.00
1.02
1.00
2.02
Current Data Parameters
NAME 103893-016
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160615
Time 7.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W
F2 - Processing parameters
SI 16384
SF 500.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

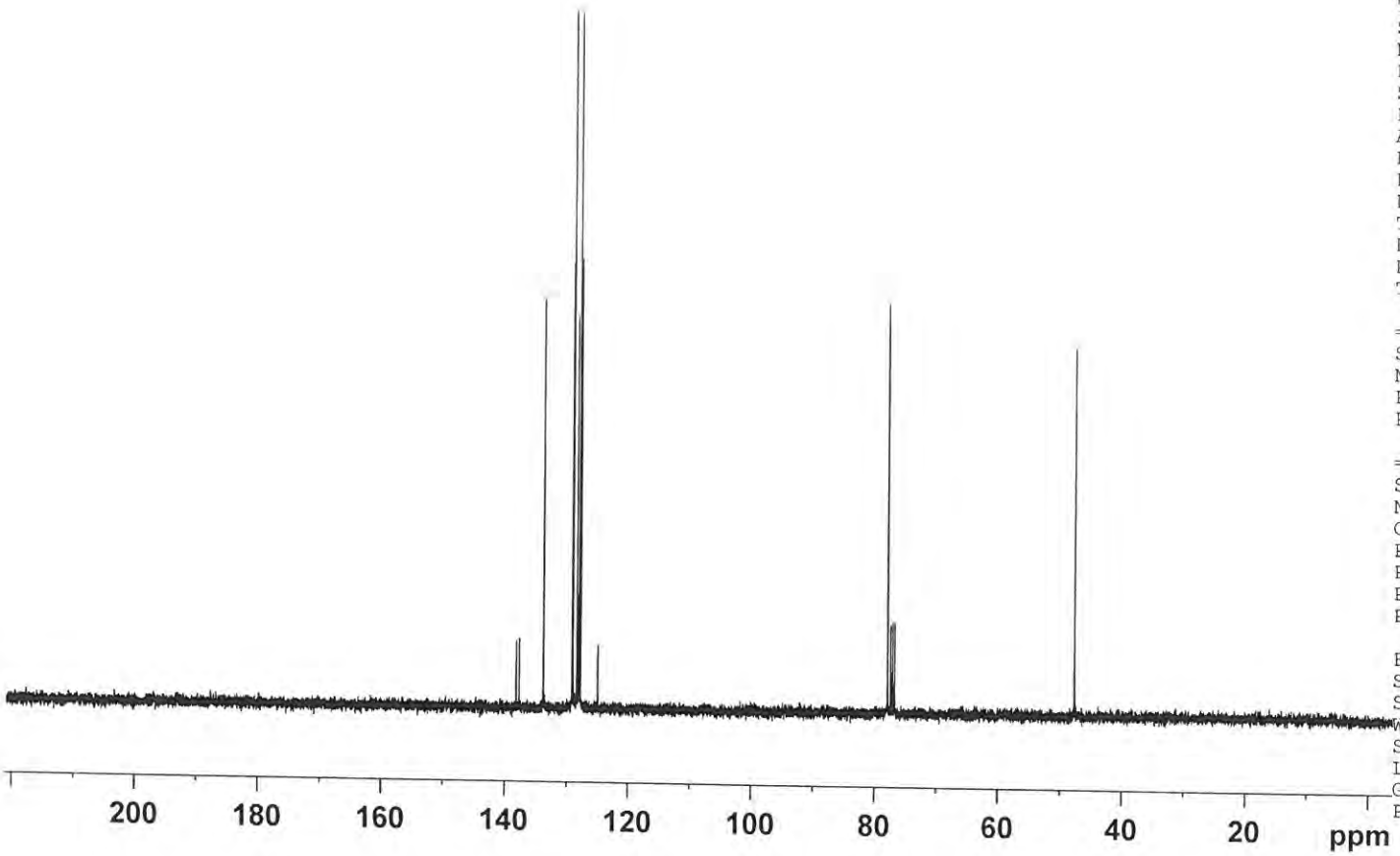
103893-016 column



138.20
137.74
133.83
129.17
129.05
128.41
128.06
127.96
127.79
124.96

77.92
77.40
77.08
76.76

47.64



BRUKER

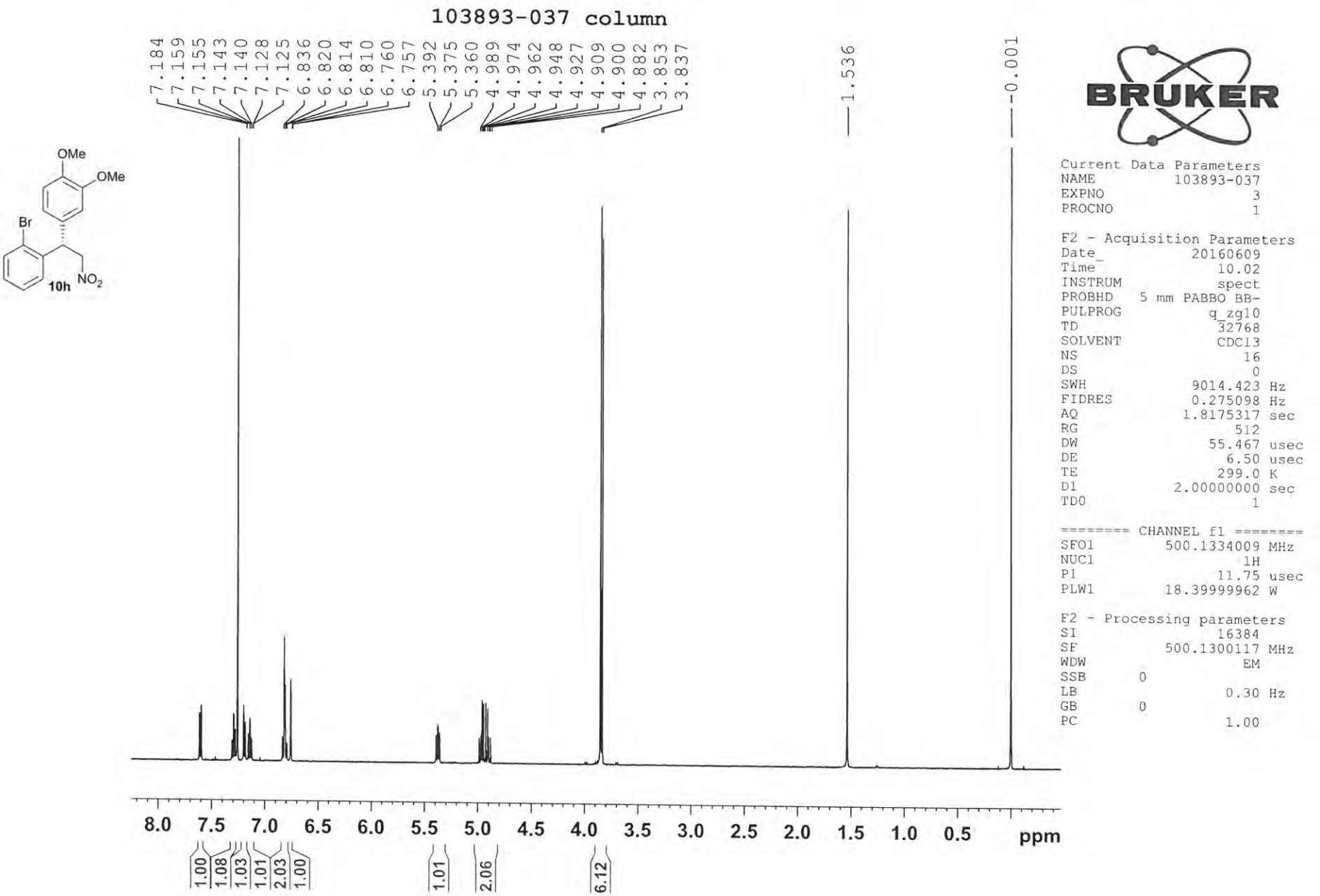
Current Data Parameters
NAME 103893-016
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160615
Time 7.42
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 15.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.09481500 W
PLW13 0.04769100 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

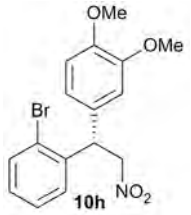
Current Data Parameters
NAME 103893-037
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160609
Time 10.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

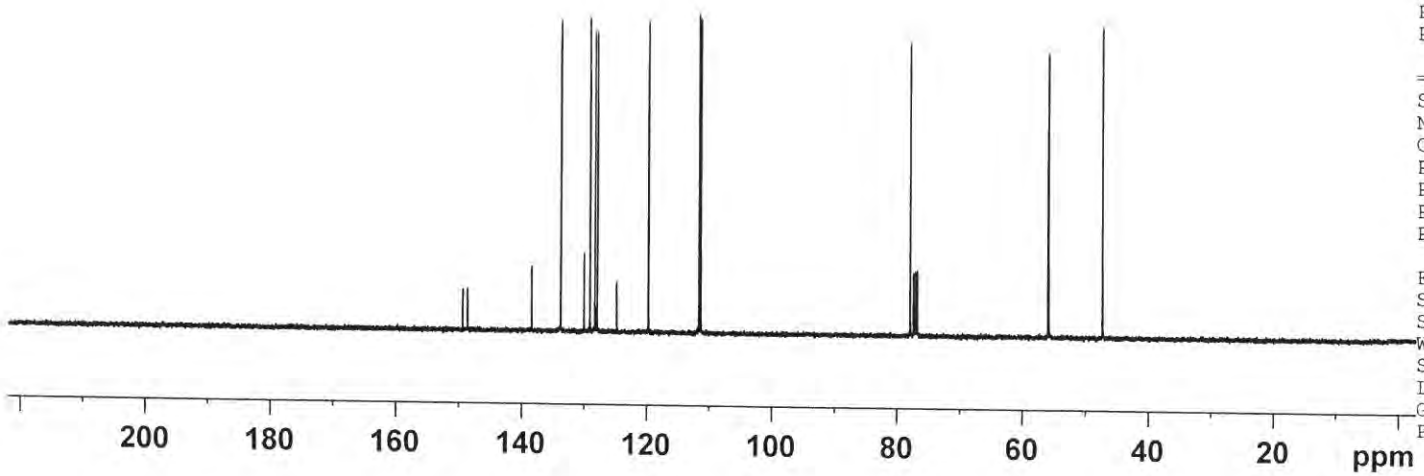
======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W

F2 - Processing parameters
SI 16384
SF 500.1300117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-037



149.33
148.59
138.38
133.78
130.08
129.13
128.33
127.95
124.84
119.77
111.68
111.37
77.94
77.42
77.10
76.79
55.95
55.87
47.34



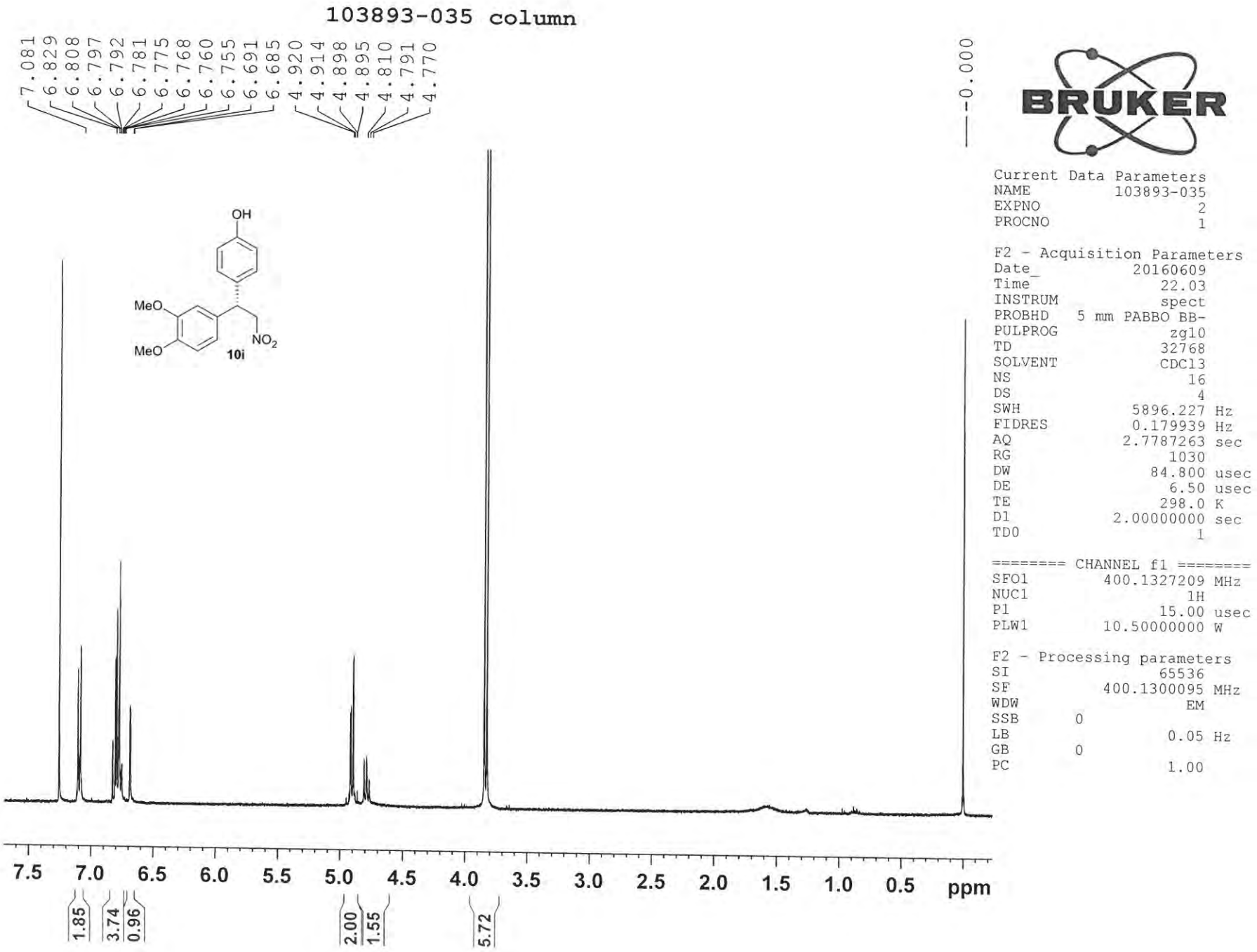
Current Data Parameters
NAME 103893-037
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160622
Time 16.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 575
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

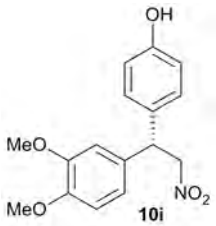
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

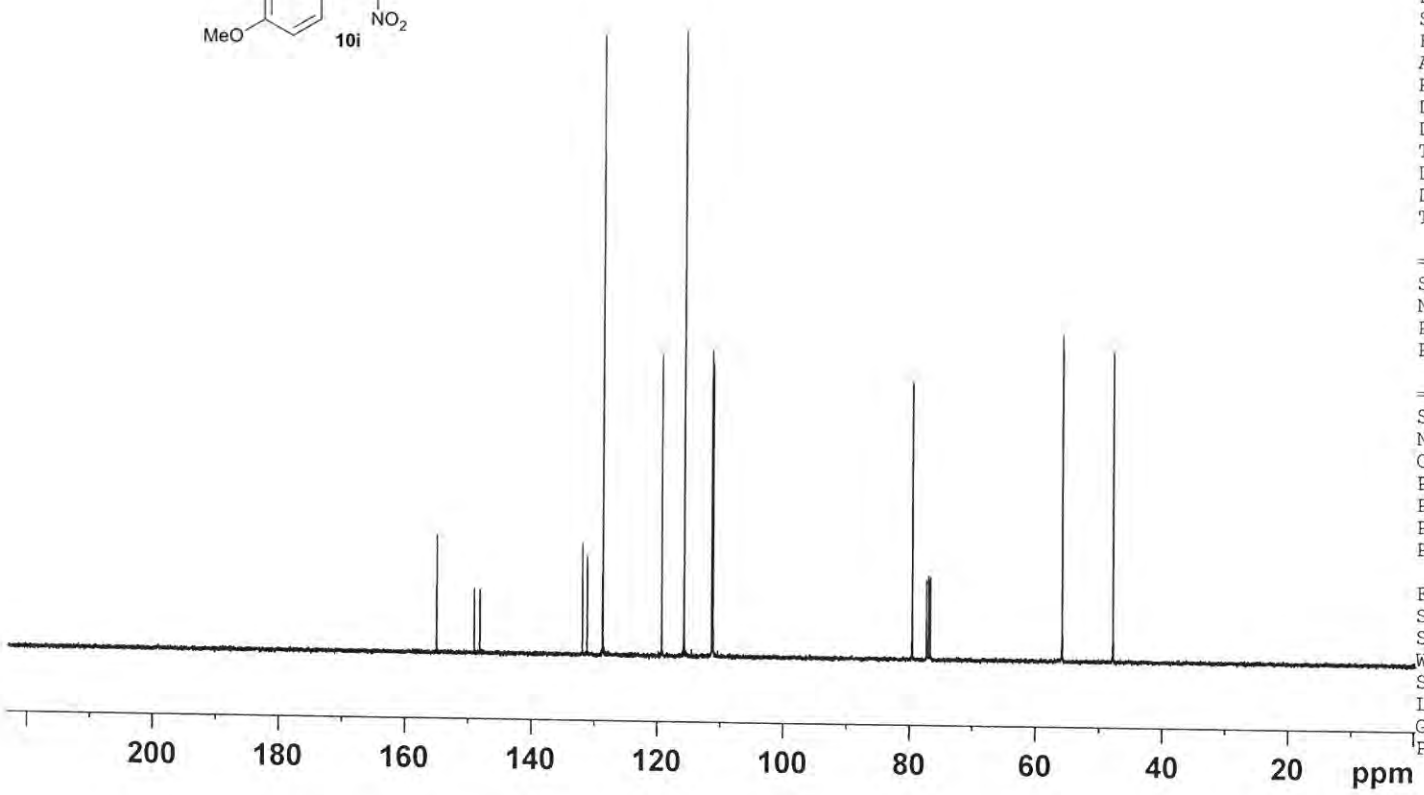
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


103893-035 column
7.081
6.829
6.808
6.797
6.792
6.781
6.775
6.768
6.760
6.755
6.691
6.685
4.920
4.914
4.898
4.895
4.810
4.791
4.770
-0.000
OH
MeO
MeO
NO2
10i
BRUKER
Current Data Parameters
NAME 103893-035
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160609
Time 22.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 5896.227 Hz
FIDRES 0.179939 Hz
AQ 2.7787263 sec
RG 1030
DW 84.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1327209 MHz
NUC1 1H
P1 15.00 usec
PLW1 10.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.05 Hz
GB 0
PC 1.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.85
3.74
0.96
2.00
1.55
5.72

103893-035 column

155.01
149.04
148.17
131.93
131.18
128.69
119.31
115.75
111.36
111.15
79.55
77.29
76.98
76.66
55.79
55.77
47.77





BRUKER

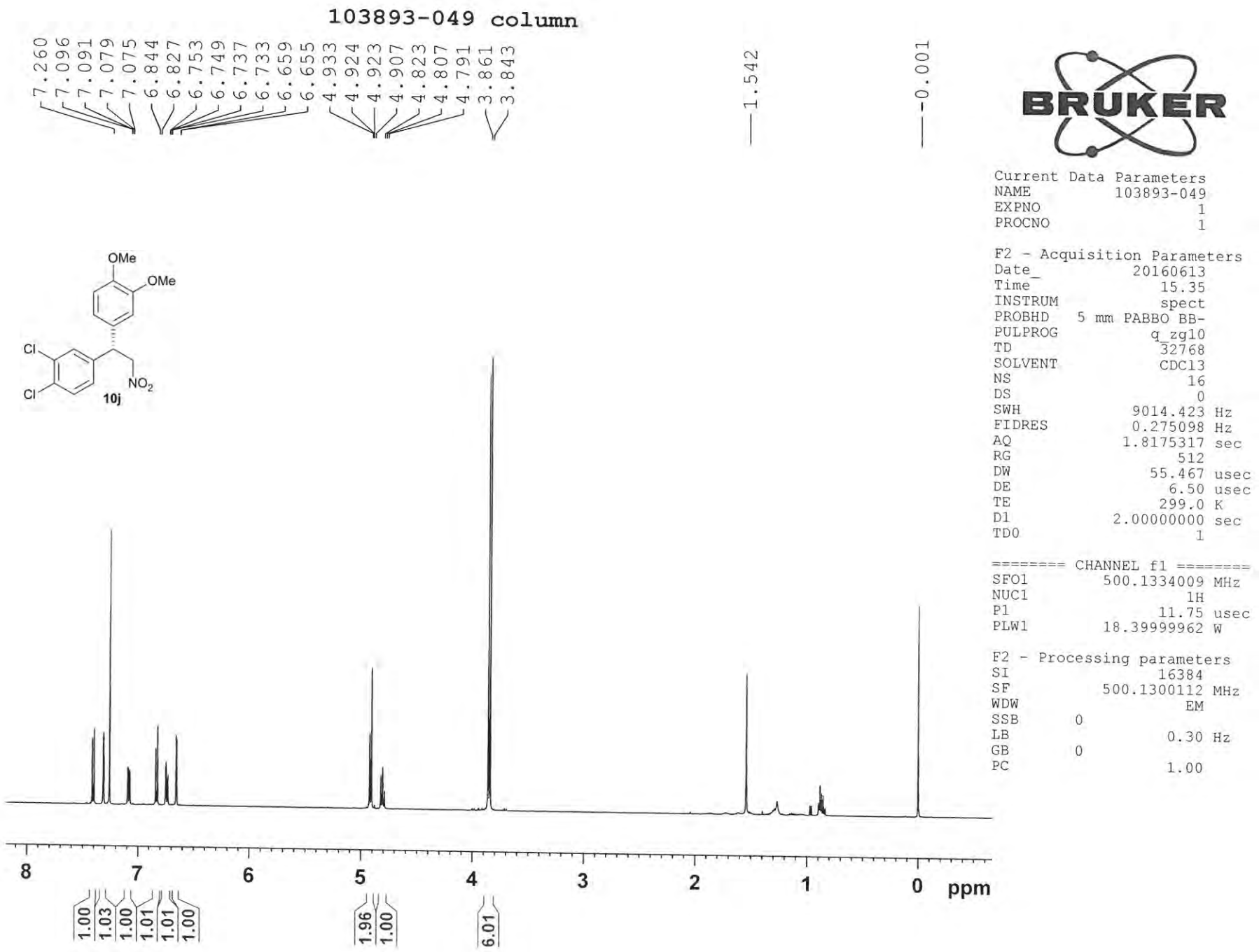
Current Data Parameters
NAME 103893-035
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160610
Time 16.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 150
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 1030
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127822 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME 103893-049
EXPNO 1
PROCNO 1

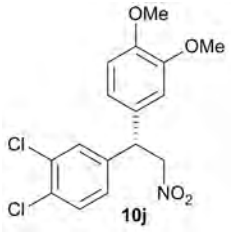
F2 - Acquisition Parameters
Date_ 20160613
Time 15.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W

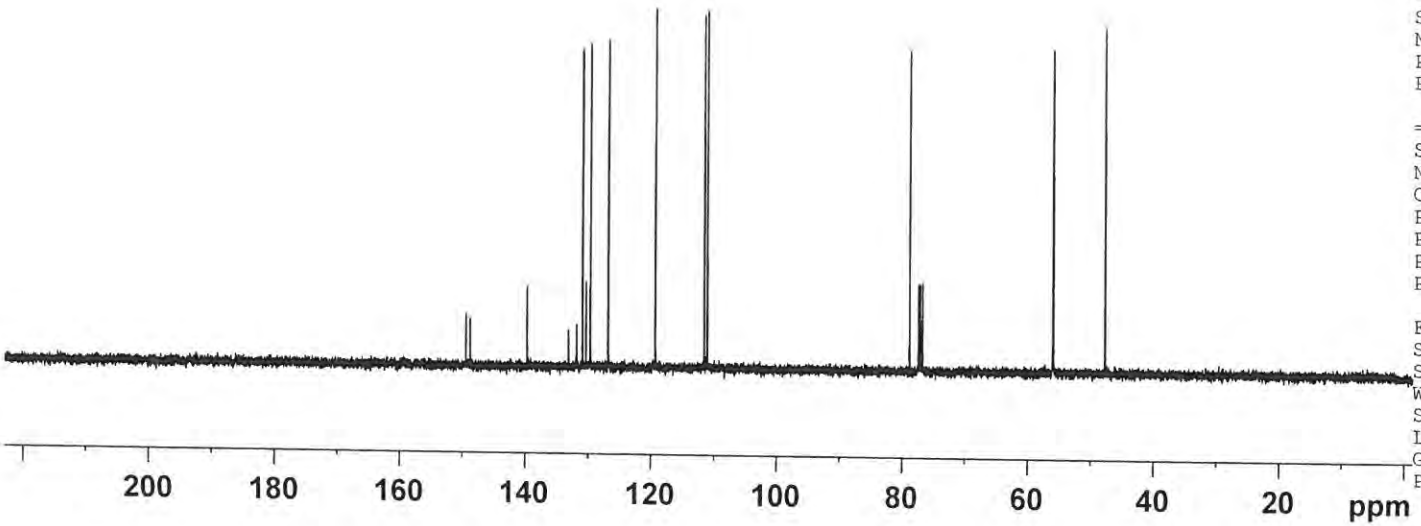
F2 - Processing parameters
SI 16384
SF 500.1300112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-049

149.51
148.83
139.72
133.14
131.84
130.94
130.38
129.70
126.87
119.36
111.58
111.08
78.84
77.40
77.08
76.76
56.00
55.93
47.67



10j



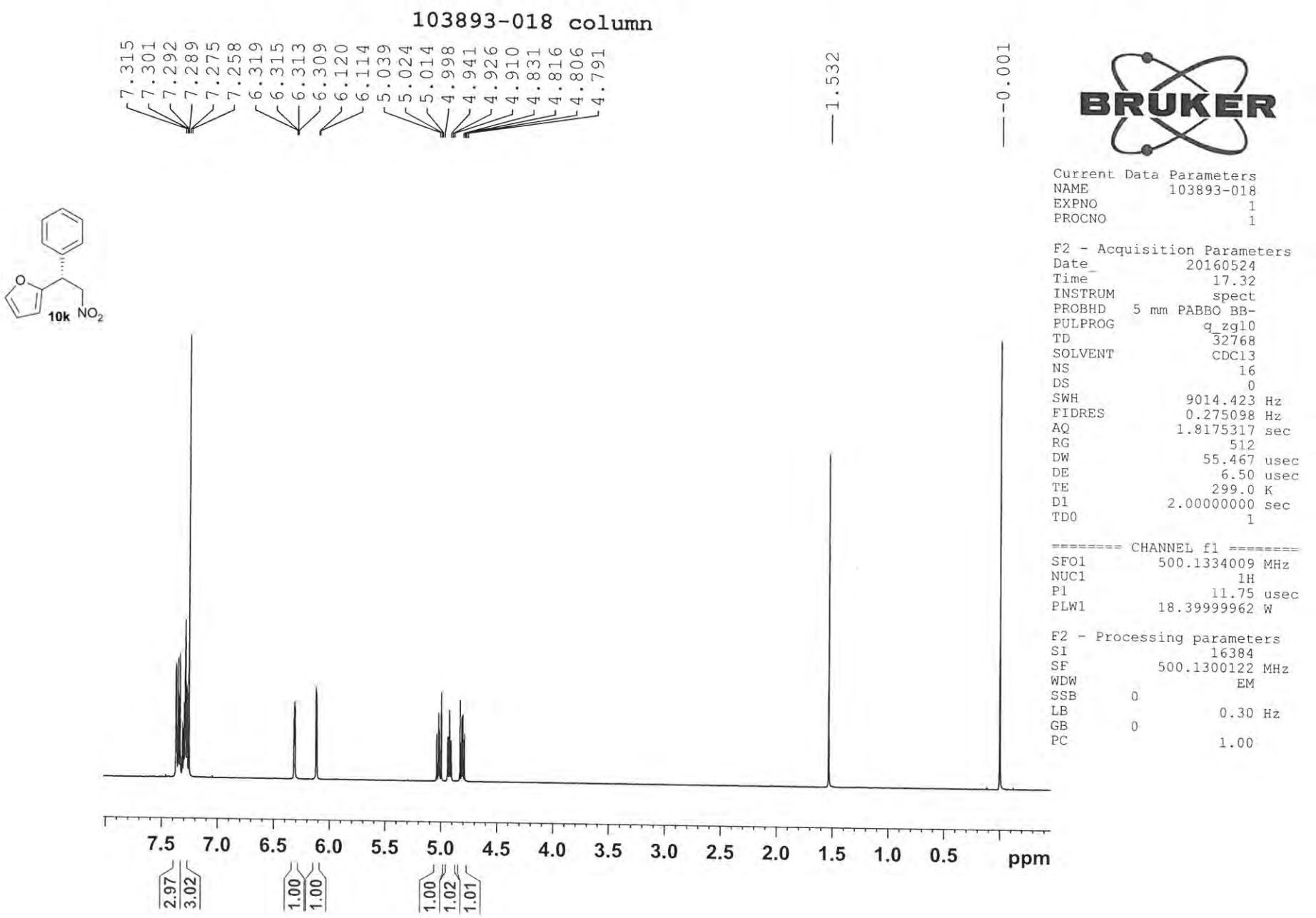
Current Data Parameters
NAME 103893-049
EXPNO 1
PROCNO 1

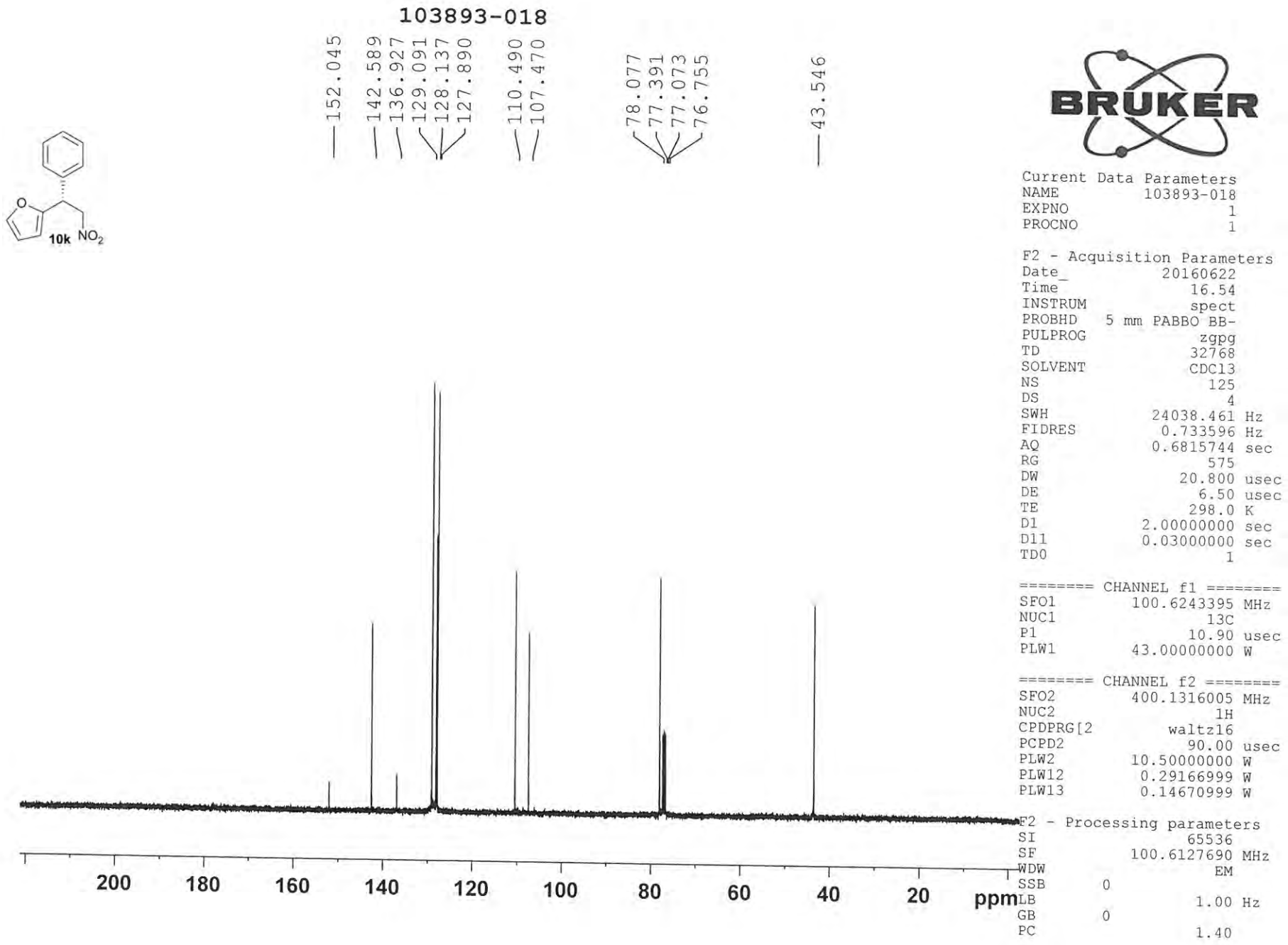
F2 - Acquisition Parameters
Date_ 20160624
Time 13.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 912
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


103893-018 column
7.315
7.301
7.292
7.289
7.275
7.258
6.319
6.315
6.313
6.309
6.120
6.114
5.039
5.024
5.014
4.998
4.941
4.926
4.910
4.831
4.816
4.806
4.791
1.532
0.001
10k
NO2
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.97
3.02
1.00
1.00
1.00
1.02
1.01
BRUKER
Current Data Parameters
NAME 103893-018
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160524
Time 17.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W
F2 - Processing parameters
SI 16384
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

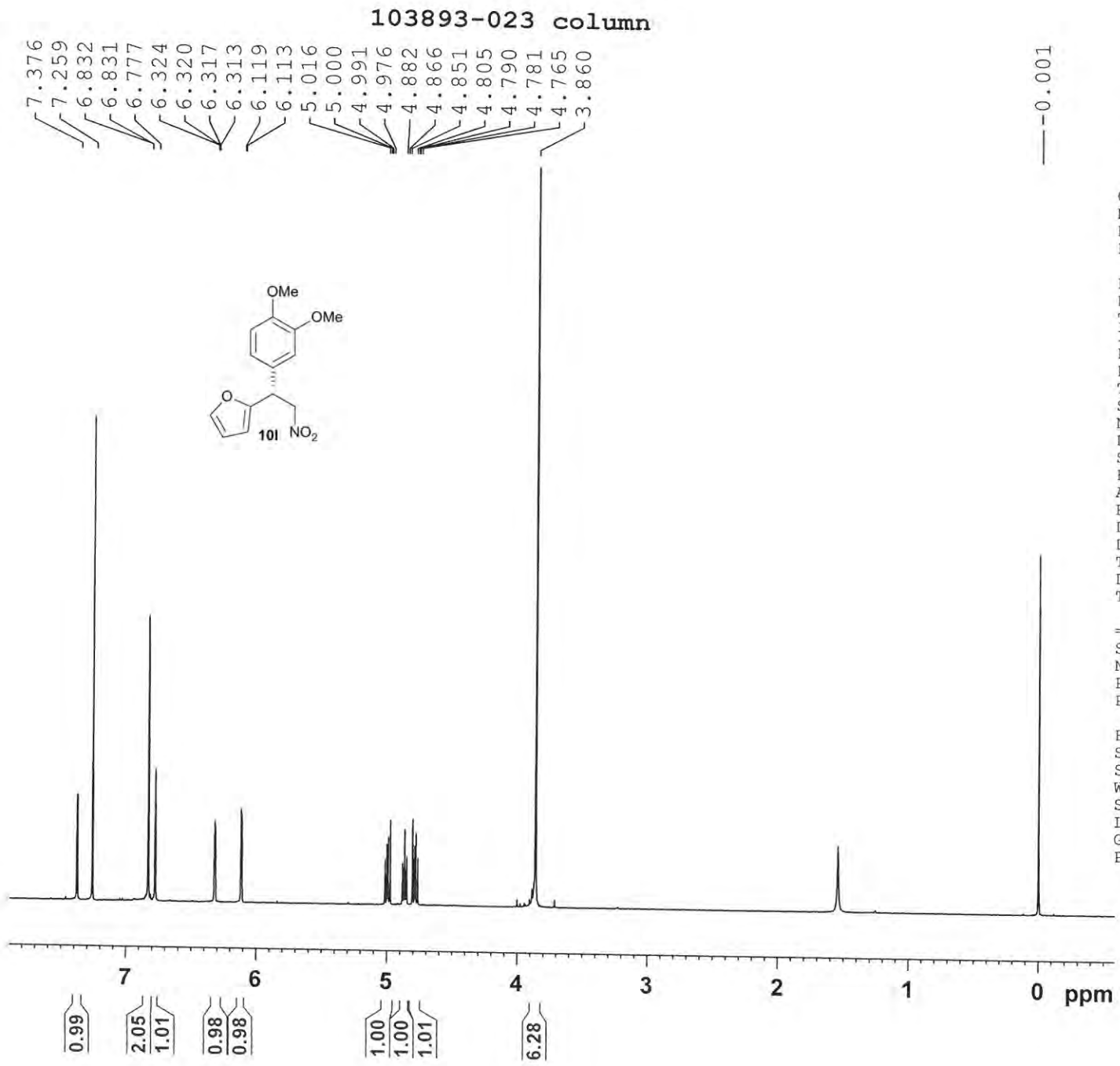
Current Data Parameters
NAME 103893-018
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160622
Time 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 575
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 10.90 usec
PLW1 43.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.50000000 W
PLW12 0.29166999 W
PLW13 0.14670999 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



BRUKER

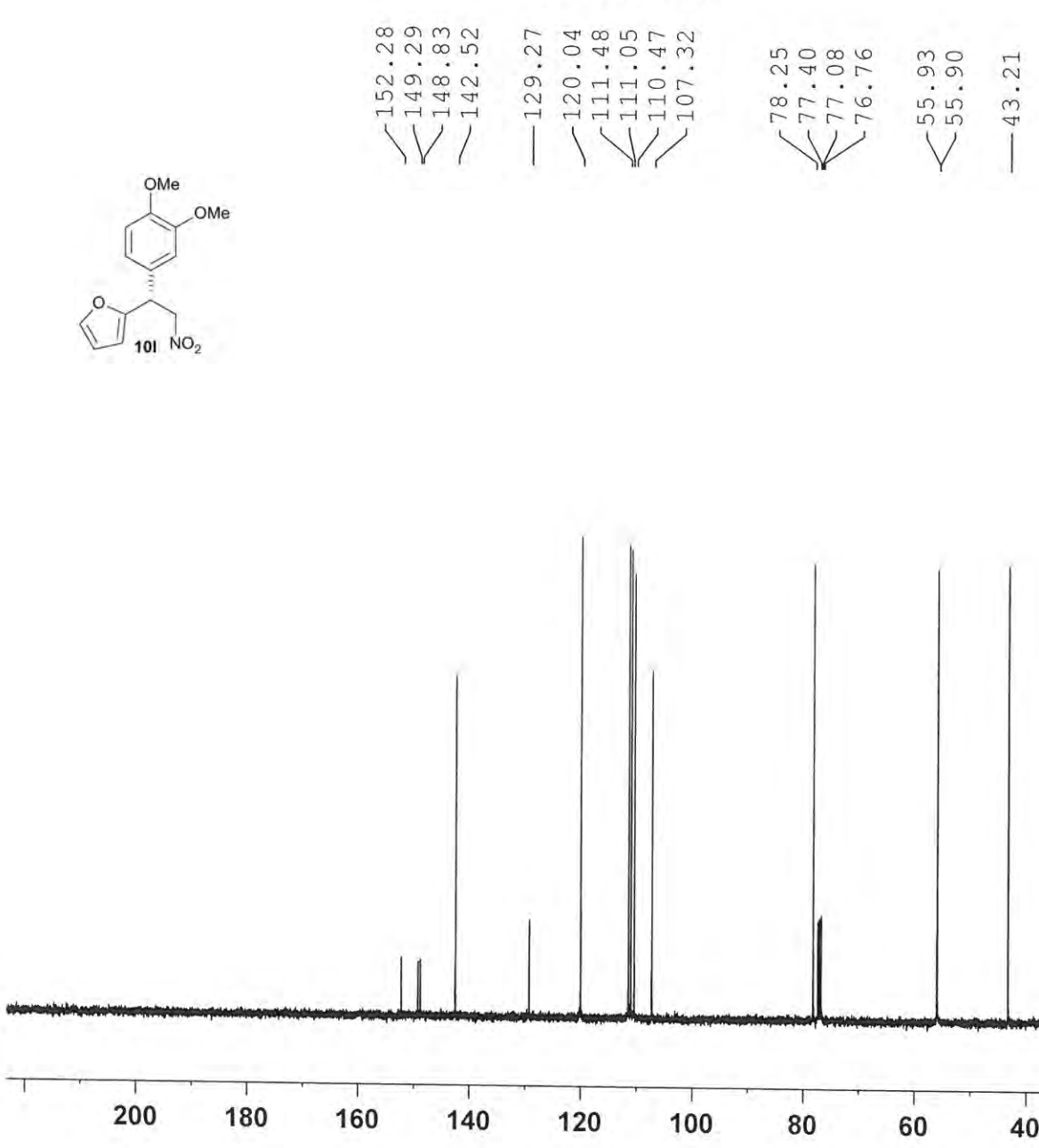
Current Data Parameters
NAME 103893-023
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160623
Time 12.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W

F2 - Processing parameters
SI 16384
SF 500.1300116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-023



BRUKER

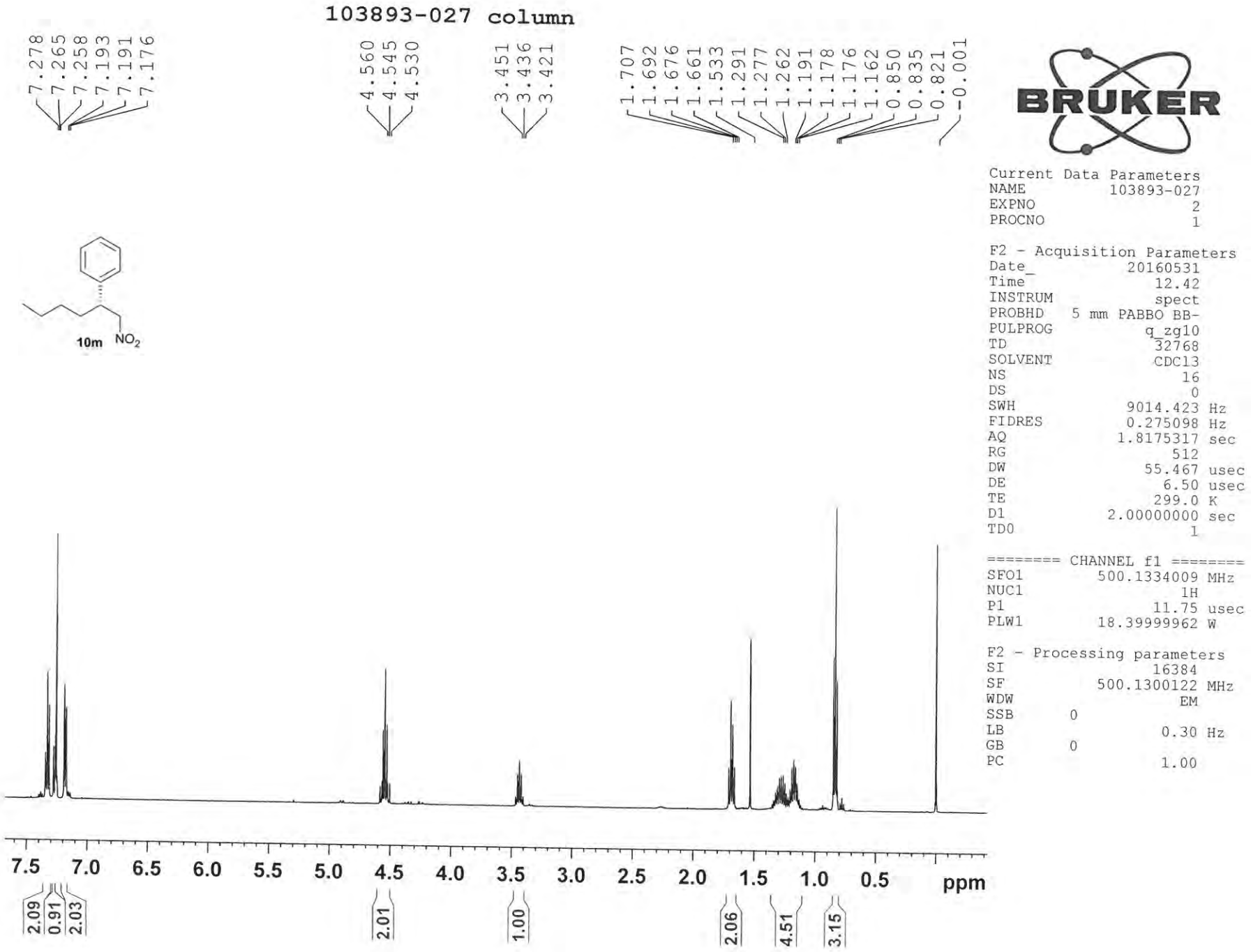
```
Current Data Parameters
NAME                103893-023
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20160623
Time                     13.29
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg
TD                      32768
SOLVENT                 CDCl3
NS                        125
DS                          4
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                  0.6815744 sec
RG                       1030
DW                     20.800 usec
DE                      6.50 usec
TE                     298.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

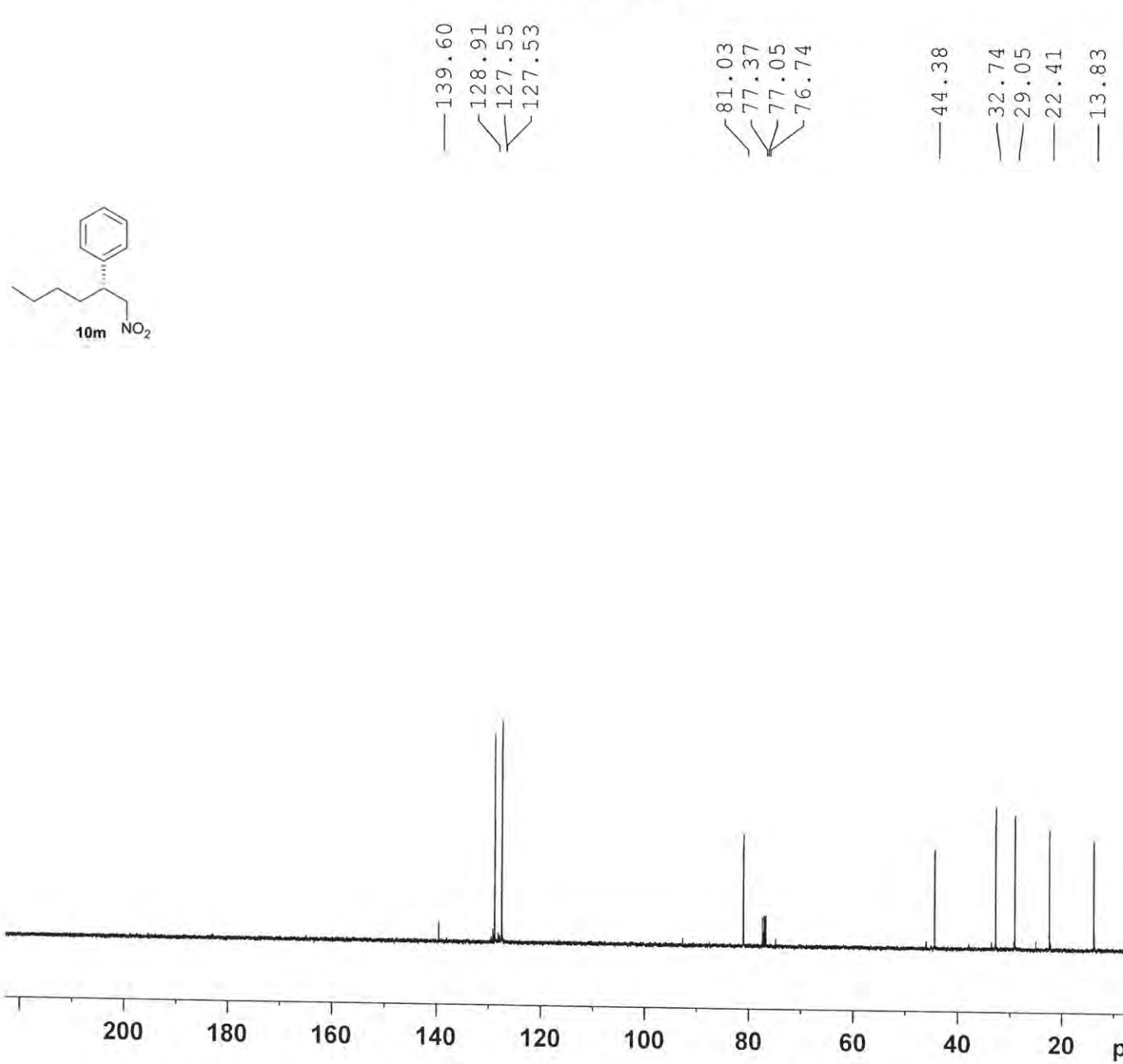
======== CHANNEL f1 ========
SFO1             100.6243395 MHz
NUC1                       13C
P1                       10.90 usec
PLW1              43.00000000 W

======== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2              10.50000000 W
PLW12              0.29166999 W
PLW13              0.14670999 W

F2 - Processing parameters
SI                         65536
SF               100.6127690 MHz
WDW                           EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```


103893-027 column
7.278
7.265
7.258
7.193
7.191
7.176
4.560
4.545
4.530
3.451
3.436
3.421
1.707
1.692
1.676
1.661
1.533
1.291
1.277
1.262
1.191
1.178
1.176
1.162
0.850
0.835
0.821
-0.001
10m
NO2
Current Data Parameters
NAME 103893-027
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160531
Time 12.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W
F2 - Processing parameters
SI 16384
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.09
0.91
2.03
2.01
1.00
2.06
4.51
3.15

103893-027 column



BRUKER

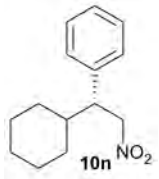
Current Data Parameters
NAME 103893-027
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160614
Time 18.22
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

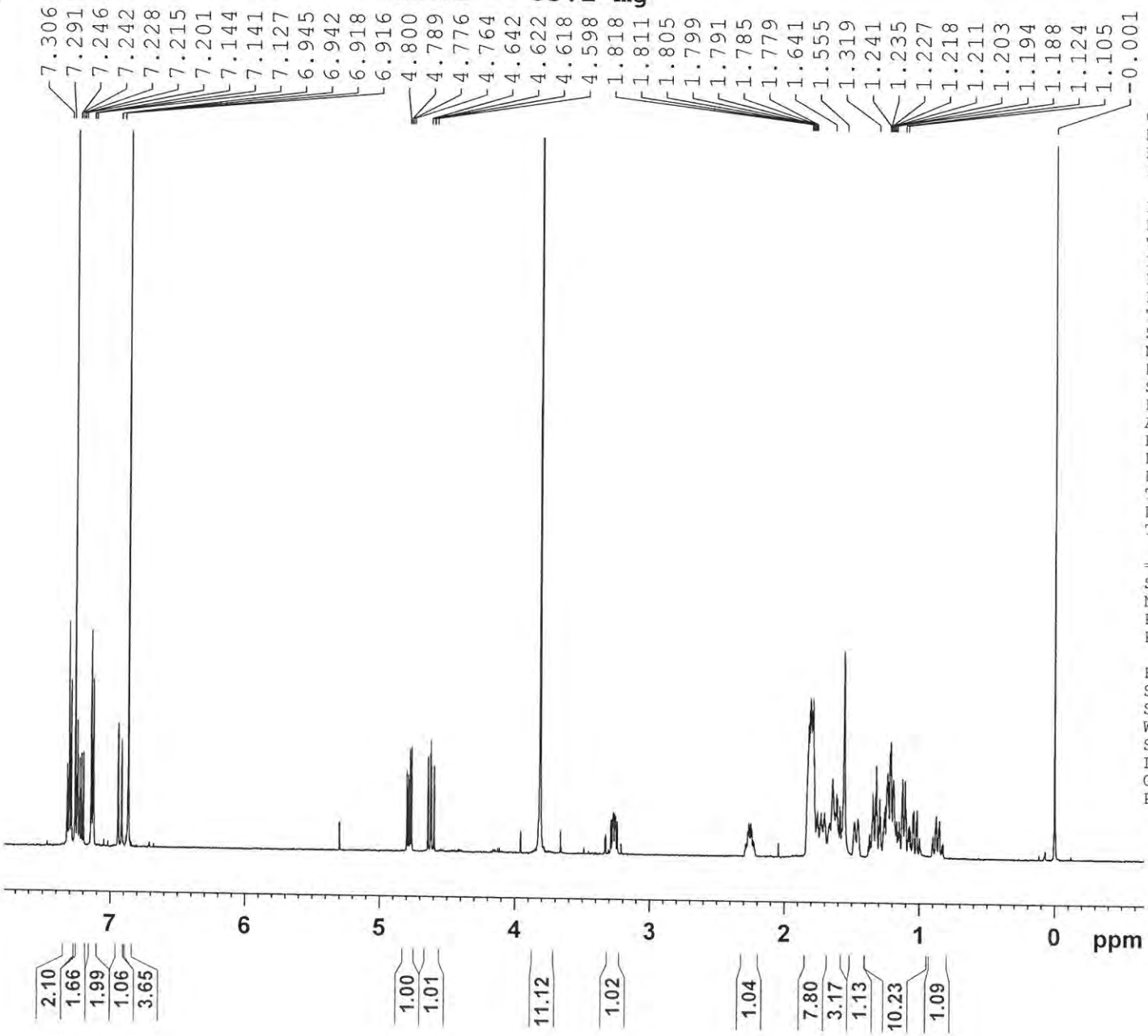
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 15.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.09481500 W
PLW13 0.04769100 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



103893-026 column
6.1 mg + 3.5 mg 96.9% dmf
total = 83.2 mg



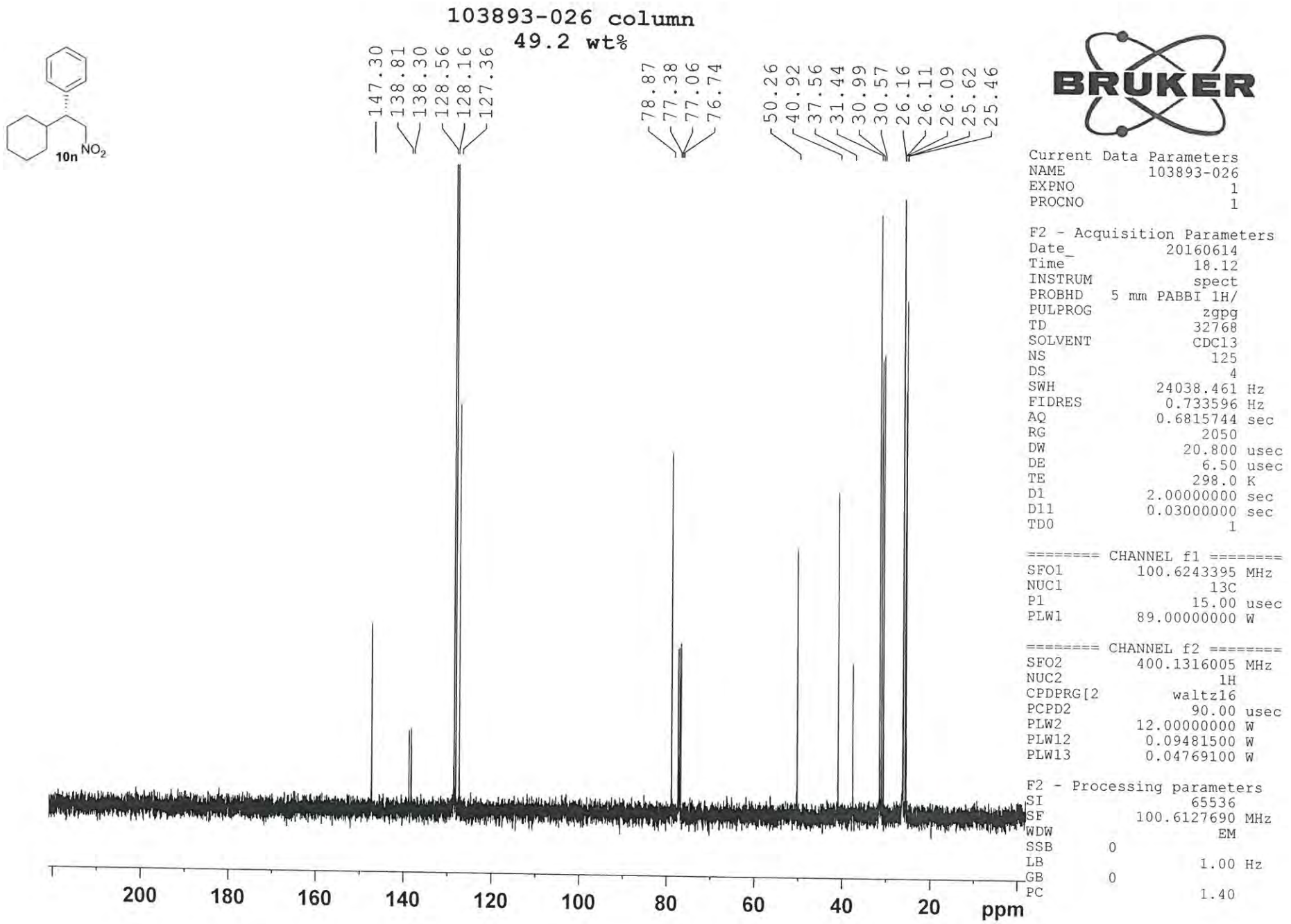
BRUKER

Current Data Parameters
NAME 103893-026
EXPNO 2
PROCNO 1

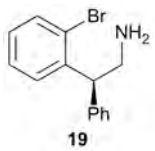
F2 - Acquisition Parameters
Date_ 20160531
Time 12.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W

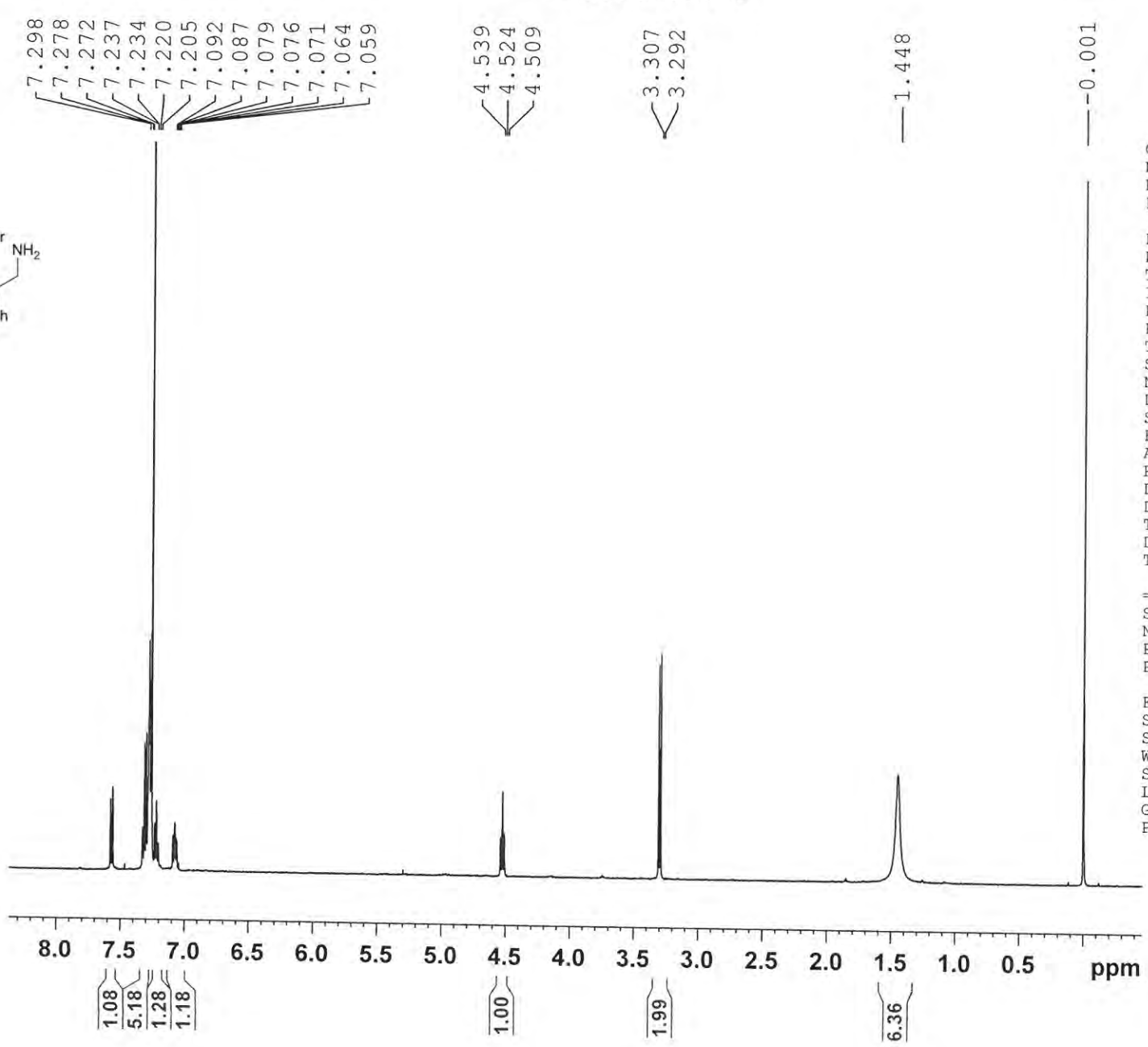
F2 - Processing parameters
SI 16384
SF 500.1300107 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


103893-026 column
49.2 wt%
147.30
138.81
138.30
128.56
128.16
127.36
78.87
77.38
77.06
76.74
50.26
40.92
37.56
31.44
30.99
30.57
26.16
26.11
26.09
25.62
25.46
10n NO2
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME 103893-026
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160614
Time 18.12
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 15.00 usec
PLW1 89.00000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.09481500 W
PLW13 0.04769100 W
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

103893-056-2



19



Current Data Parameters
NAME 103893-056-2
EXPNO 1
PROCNO 1

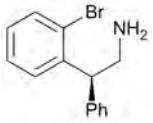
F2 - Acquisition Parameters
Date_ 20160617
Time 8.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG q_zg10
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 512
DW 55.467 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1334009 MHz
NUC1 1H
P1 11.75 usec
PLW1 18.39999962 W

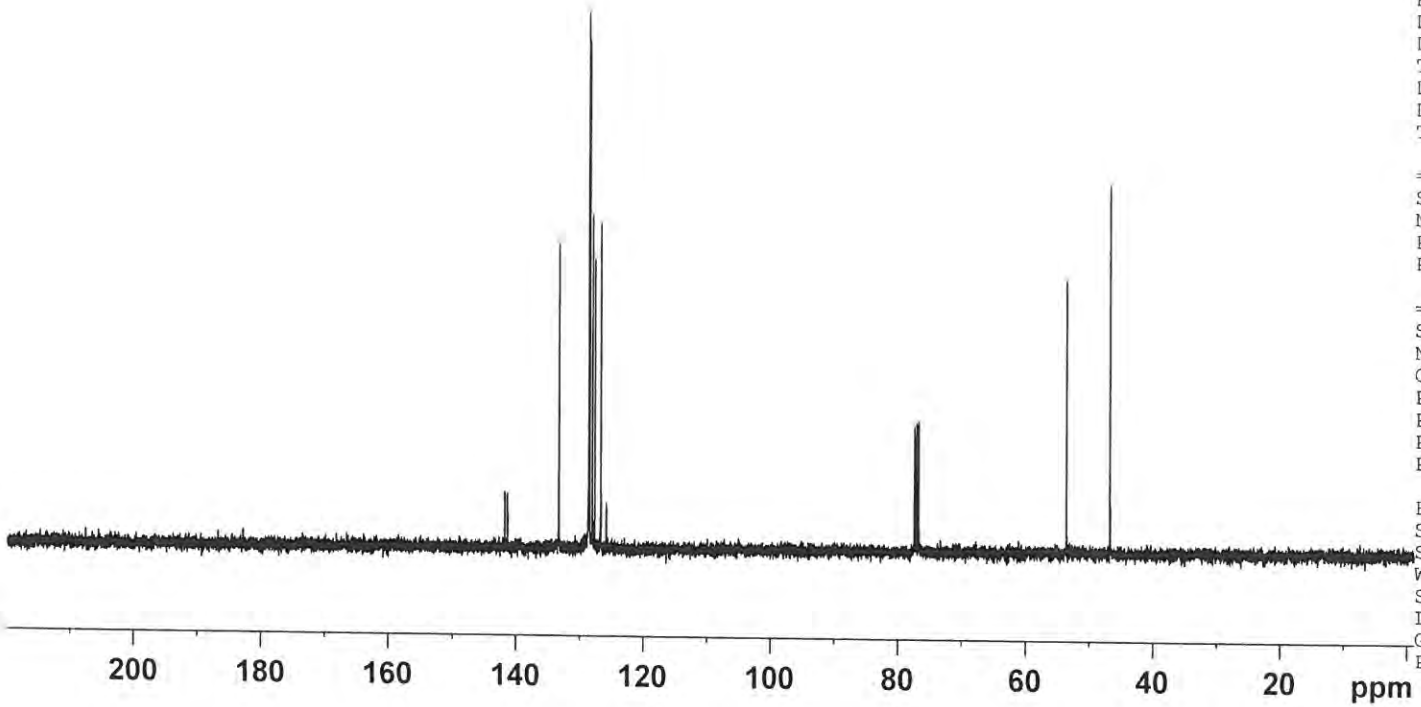
F2 - Processing parameters
SI 16384
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

103893-056-2

141.82
141.38
133.32
128.63
128.59
128.47
128.03
127.68
126.71
125.84
77.38
77.06
76.75
53.58
46.73



19



Current Data Parameters
NAME 103893-056-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160617
Time 10.33
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 200
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

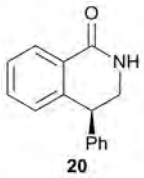
======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 15.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.09481500 W
PLW13 0.04769100 W

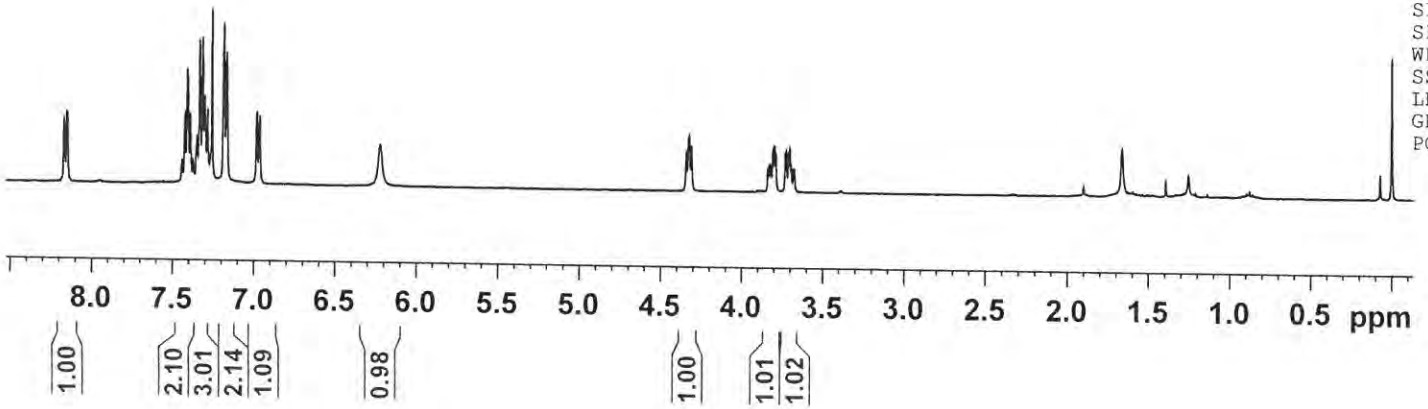
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

103893-057

7.409 7.395 7.392 7.353 7.336 7.318 7.304 7.293 7.286 7.259 7.186 7.169 6.983 6.966 6.223 4.343 4.324 4.311 3.841 3.833 3.827 3.820 3.810 3.802 3.797 3.789 3.731 3.724 3.712 3.704 3.694 3.681 3.674



20



—0.000

BRUKER

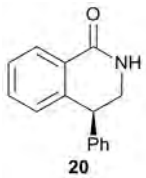
Current Data Parameters
NAME 103893-057
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160617
Time 10.54
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg10
TD 32768
SOLVENT CDCl3
NS 8
DS 4
SWH 5896.227 Hz
FIDRES 0.179939 Hz
AQ 2.7787263 sec
RG 645
DW 84.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1327209 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.00000000 W

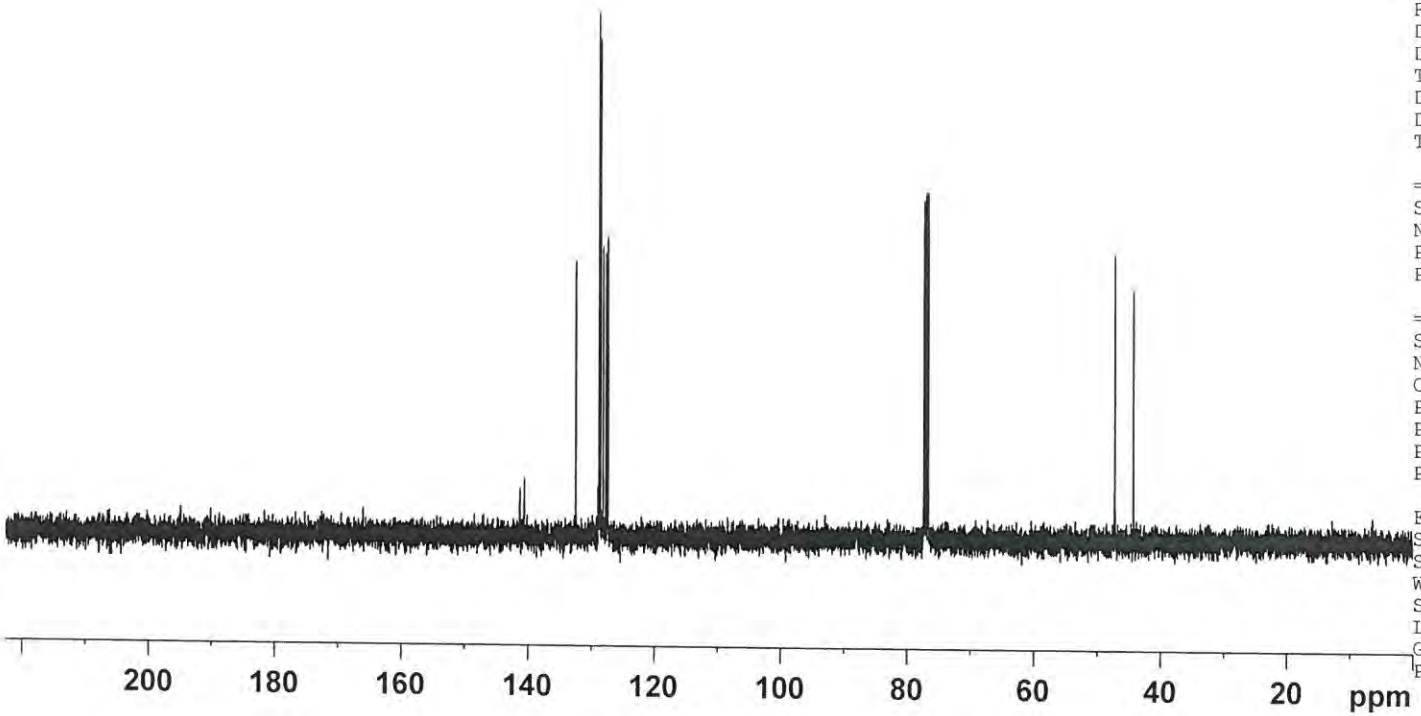
F2 - Processing parameters
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.05 Hz
GB 0
PC 1.00

103893-057



20

166.03
141.32
140.62
132.49
128.97
128.79
128.59
128.16
127.70
127.47
127.39
77.34
77.03
76.71
47.24
44.29



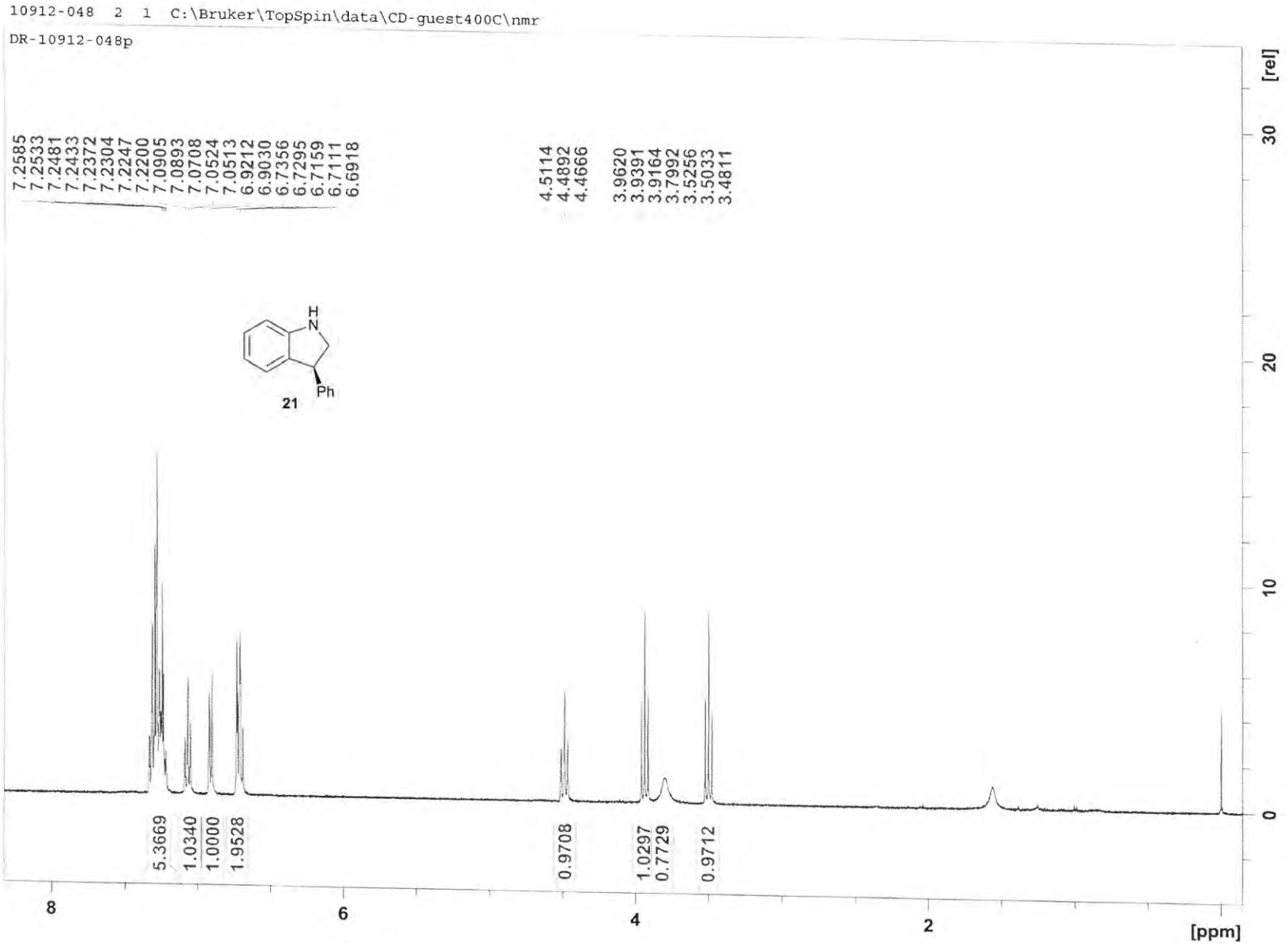
Current Data Parameters
NAME 103893-057
EXPNO 2
PROCNO 1

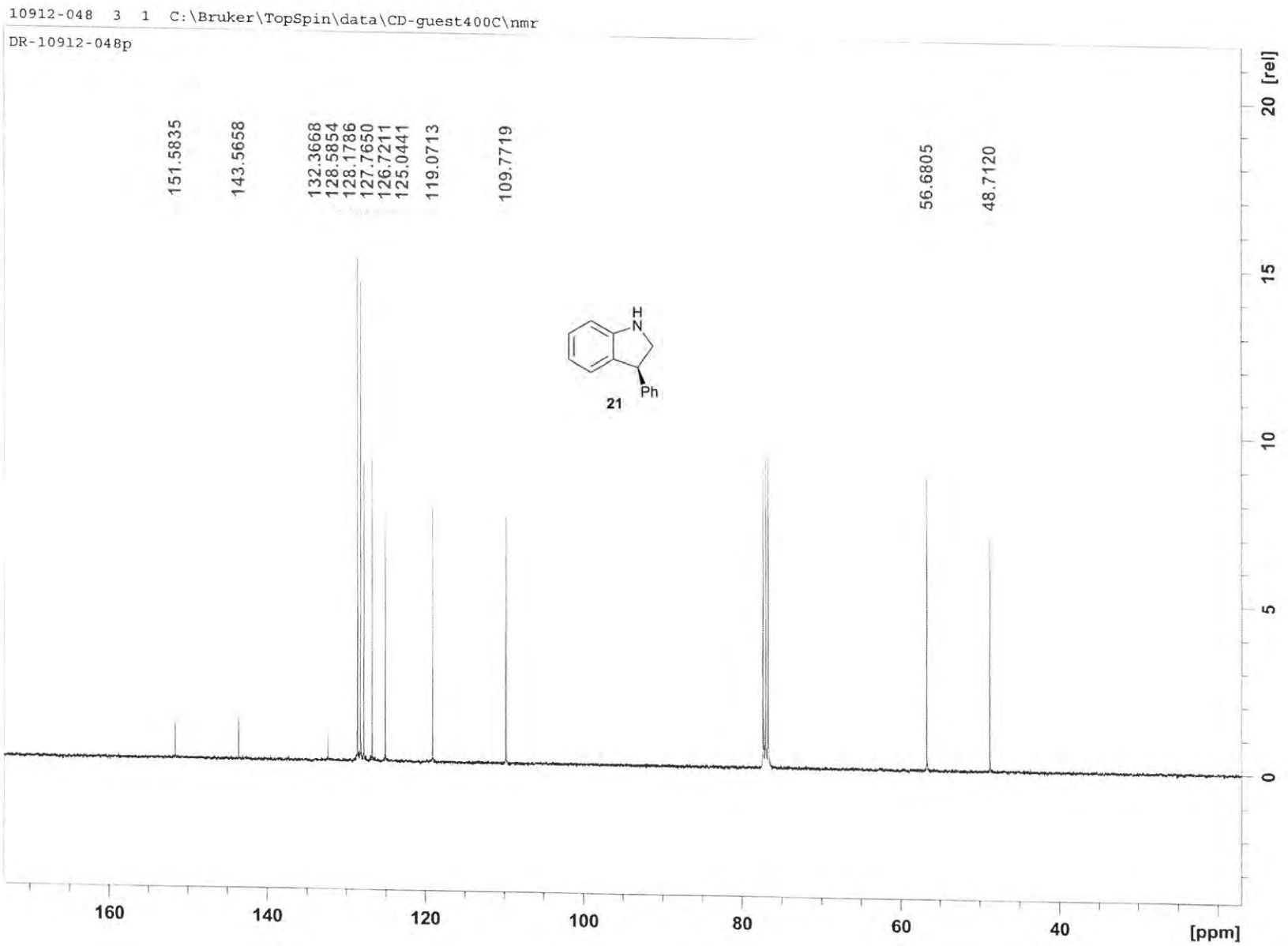
F2 - Acquisition Parameters
Date_ 20160617
Time 11.15
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6243395 MHz
NUC1 13C
P1 15.00 usec
PLW1 89.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.09481500 W
PLW13 0.04769100 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


10912-048 2 1 C:\Bruker\TopSpin\data\CD-guest400C\nmr
DR-10912-048p
7.2585
7.2533
7.2481
7.2433
7.2372
7.2304
7.2247
7.2200
7.0905
7.0893
7.0708
7.0524
7.0513
6.9212
6.9030
6.7356
6.7295
6.7159
6.7111
6.6918
4.5114
4.4892
4.4666
3.9620
3.9391
3.9164
3.7992
3.5256
3.5033
3.4811
H
N
Ph
21
5.3669
1.0340
1.0000
1.9528
0.9708
1.0297
0.7729
0.9712
8
6
4
2
[ppm]
[rel]
30
20
10
0


10912-048 3 1 C:\Bruker\TopSpin\data\CD-guest400C\nmr
DR-10912-048p
151.5835
143.5658
132.3668
128.5854
128.1786
127.7650
126.7211
125.0441
119.0713
109.7719
56.6805
48.7120
H
N
Ph
21
20 [rel]
15
10
5
0
160
140
120
100
80
60
40
[ppm]