

Supporting Information

**Visible Light Catalyzed Reaction of α -Bromochalcones with Chalcones:
Direct Access to the Urundevine Scaffold**

Bhupal Singh Karki,^{a,b} Mukund M. D. Pramanik,^{a,b} Ruchir Kant,^c and Namrata Rastogi^{a,b*}

^aMedicinal & Process Chemistry Division and ^cMolecular and Structural Biology Division, CSIR-Central Drug Research Institute, B.S. 10/1, Sector 10, Jankipuram extension, Sitapur Road, Lucknow 226031, India

^bAcademy of Scientific and Innovative Research, New Delhi 110001, India

* Corresponding author. e-mail: namrata.rastogi@cdri.res.in

Table of Contents

1. Experimental Section.....	1-3
2. Spectroscopic Data.....	3-13
3. Crystallographic Data.....	14-15
4. HPLC Chromatogram of the Product Mixture of Reaction between 1s & 2h	16
5. References.....	17
6. Copies of ¹ H and ¹³ C NMR Spectra.....	18-74

1. Experimental Section

1.1 General experimental information

All reactions were monitored by TLC, visualization was effected with UV and/or by developing in iodine. Melting points were recorded on a Precision melting point apparatus and are uncorrected. NMR spectra were recorded on a Bruker Avance spectrometer at 400 or 500 MHz (^1H) and 100 MHz (^{13}C). Chemical shifts are reported in δ (ppm) relative to TMS as the internal standard. To describe spin multiplicity, standard abbreviations such as s, d, t, q, m, dd referring to singlet, doublet, triplet, quartet, multiplet and doublet of doublet respectively, are used. The ESI-HRMS spectra were recorded on Agilent 6520-Q-ToF LC/MS system. The NMR yields of products were calculated through ^1H NMR of crude reaction mixture using dibromo methane as internal standard and isolated yields were calculated after purification by column chromatography. Preparative HPLC was conducted on a 1200 infinity series system (Pumps, 1260 Prep. Pumps; Diode Array Detector, 1260 DAD VL; Fraction collector, 1260 FC-PS; Sampler, 1260 manual injector and Open LAB CDS software) from Agilent Technologies. Reverse phase column (Agilent 10 Prep-C18, 150 x 30 mm) was used and acetonitrile (Pump A, flow rate 20 ml/min) and water with 0.1 % TFA (Pump B, flow rate 4 ml/min) were used as mobile phase with isocratic elution.

All the chemicals and catalysts were purchased from commercial sources and used as received except DMSO which was freshly distilled over CaH_2 before the reaction. The chalcones **1a-1s** are known compounds and were synthesized following literature protocols.¹ Similarly, all the α -bromochalcones except **2c** and **2f** are known compounds and were synthesized according to the reported procedure.²

1.2 General procedure for the photoredox catalyzed reaction

In an oven dried 5 mL snap vial equipped with a magnetic stirring bar, the α -bromochalcone **2** (0.3 mmol), chalcone **1** (0.6 mmol, 2.0 equiv), K_3PO_4 (0.13 g, 0.6 mmol, 2.0 equiv) and photocatalyst *fac*-Ir(ppy)₃ (0.002 g, 0.003 mmol, 1.0 mol%) were dissolved in anhydrous DMSO (3 mL). The resulting reaction mixture was degassed by three “pump-freeze-thaw” cycles via a syringe needle. The vial was irradiated using 450 nm blue LEDs with a cooling device maintaining the temperature around 25 °C. After 36 h of irradiation (TLC monitoring), the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layers were dried (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel using hexane/ethyl acetate as eluent to afford the pure product **3**.

1.3 General procedure for the oxidation of dihydronaphthalenes

The dihydronaphthalene **3** (0.1 mmol) and ammonium acetate (0.31 g, 0.4 mmol, 4.0 equiv) were dissolved in acetic acid (5 mL) and the reaction mixture was refluxed for 9-12 h (TLC monitoring). The reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate (3 x 10 mL). The combined organic layers were dried (Na₂SO₄) and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel using hexane/ethyl acetate as eluent to afford the pure product **4**.

2. Spectroscopic Data

(Z)-2-bromo-1-(5-bromo-2,4-dimethoxyphenyl)-3-(2-bromo-4,5-dimethoxyphenyl) prop-2-en-1-one (2c).

Yellow solid; *R_f* 0.50 (25% EtOAc/hexane); Mp 163-164 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.79 (s, 1H), 7.68 (s, 1H), 7.58 (s, 1H), 7.00 (s, 1H), 6.44 (s, 1H), 3.91 (s, 3H), 3.84 (s, 3H), 3.85 (s, 3H), 3.79 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 188.78, 159.21, 158.57, 150.94, 147.81, 141.81, 134.32, 126.06, 125.16, 120.56, 117.11, 115.31, 112.99, 102.23, 96.34, 56.47, 56.25, 56.23, 56.15; HRMS for C₁₉H₁₇Br₃O₅: calcd. (M+H)⁺: 562.8699, found: 562.8697

(Z)-2-bromo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (2f).

Yellow oil; *R_f* 0.50 (5% EtOAc/hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.77-7.80 (m, 3H), 7.74 (dd, *J* = 3.8 Hz, 1.1 Hz, 1H), 7.68 (dd, *J* = 5.0 Hz, 1.2 Hz, 1H), 7.36-7.40 (m, 3H), 7.10 (dd, *J* = 5.0 Hz, 3.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 183.03, 141.35, 140.02, 135.12, 134.81, 133.68, 130.25, 130.13, 128.57, 128.15, 120.78; HRMS for C₁₃H₉BrOS: calcd. (M+H)⁺: 292.9630, found: 292.9633

(1-(4-Methoxyphenyl)-1,2-dihydronaphthalene-2,3-diyl)bis(phenylmethanone) (3a).

White solid; isolated yield 75% (69 mg). *R_f* 0.50 (10% EtOAc/hexane); Mp 123-125 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.90-7.92 (m, 2H), 7.64-7.66 (m, 2H), 7.46-7.49 (m, 2H), 7.39 (d, *J* = 7.9 Hz, 3H), 7.36 (d, *J* = 3.2 Hz, 2H), 7.14-7.23 (m, 3H), 7.00-7.04 (m, 2H), 6.90-6.94 (m, 1H), 6.69-6.73 (m, 2H), 5.16 (d, *J* = 4.0 Hz, 1H), 4.43 (d, *J* = 4.0 Hz, 1H), 3.67 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 198.91, 196.36, 158.60, 142.07, 137.84, 137.31, 136.18, 135.04, 134.67, 132.92, 131.94, 131.61, 130.83, 129.49, 129.43, 129.07, 128.78, 128.74, 128.64, 128.28, 127.59, 114.19, 55.26, 49.68, 46.27; HRMS for C₃₁H₂₄O₃: calcd. (M+H)⁺: 445.1798, found: 445.1804

(1-Phenyl-1,2-dihydronaphthalene-2,3-diyl)bis(phenylmethanone) (3b). Yellow solid; isolated yield 72% (89 mg). *R_f* 0.50 (10% EtOAc/hexane); Mp 97-98 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, *J* = 7.6 Hz, 2H), 7.72 (d, *J* = 7.0 Hz, 2H), 7.53-7.57 (m, 2H), 7.44-

7.47 (m, 5H), 7.24-7.31 (m, 5H), 7.17-7.20 (m, 3H), 6.99-7.01 (m, 1H), 5.25 (d, $J = 3.9$ Hz, 1H), 4.54 (d, $J = 3.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.76, 196.31, 142.88, 142.12, 137.82, 136.88, 136.12, 134.61, 132.95, 131.95, 131.73, 130.85, 129.51, 129.42, 129.16, 128.82, 128.80, 128.65, 128.28, 127.74, 127.70, 127.11, 49.51, 47.02; HRMS for $\text{C}_{30}\text{H}_{22}\text{O}_2$: calcd. (M+H) $^+$: 415.1693, found: 415.1688

(3-Benzoyl-1-(p-tolyl)-1,2-dihydronaphthalen-2-yl)(4-methoxyphenyl)methanone (3c).

White solid; isolated yield 51% (70 mg). R_f 0.50 (10% EtOAc/hexane); Mp 112-113 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.92-7.95 (m, 2H), 7.64-7.66 (m, 2H), 7.45-7.49 (m, 1H), 7.35-7.39 (m, 3H), 7.14-7.22 (m, 3H), 7.00 (br s, 4H), 6.92-6.94 (m, 1H), 6.86-6.88 (m, 2H), 5.12 (d, $J = 3.3$ Hz, 1H), 4.41 (d, $J = 3.3$ Hz, 1H), 3.80 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.96, 196.40, 163.52, 142.12, 140.36, 137.97, 137.25, 136.66, 134.56, 131.86, 131.66, 131.19, 130.77, 129.50, 129.45, 129.16, 128.68, 128.25, 127.53, 127.47, 113.89, 55.49, 48.99, 46.78, 20.99; HRMS for $\text{C}_{32}\text{H}_{26}\text{O}_3$: calcd. (M+H) $^+$: 459.1955, found: 459.1960

(3-Benzoyl-1-(4-methoxyphenyl)-1,2-dihydronaphthalen-2-yl)(p-tolyl)methanone (3d).

Yellow gummy solid; isolated yield 55% (75 mg). R_f 0.50 (15% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.84 (d, $J = 8.6$ Hz, 2H), 7.63-7.67 (m, 2H), 7.46-7.50 (m, 1H), 7.35-7.40 (m, 3H), 7.14-7.23 (m, 5H), 7.01-7.05 (m, 2H), 6.91-6.93 (m, 1H), 6.71-6.74 (m, 2H), 5.14 (d, $J = 3.6$ Hz, 1H), 4.42 (d, $J = 3.3$ Hz, 1H), 3.69 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.26, 196.38, 158.56, 143.76, 142.04, 137.93, 137.33, 135.32, 134.61, 133.42, 131.88, 131.63, 130.77, 129.44, 129.38, 129.10, 128.97, 128.66, 128.25, 127.55, 114.18, 55.26, 49.41, 46.28, 21.65; HRMS for $\text{C}_{32}\text{H}_{26}\text{O}_3$: calcd. (M+H) $^+$: 459.1955, found: 459.1958

(3-Benzoyl-1-(4-methoxyphenyl)-1,2-dihydronaphthalen-2-yl)(2-methoxyphenyl)

methanone (3e). Yellow gummy solid; isolated yield 45% (64 mg). R_f 0.50 (20% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.56-7.58 (m, 2H), 7.43-7.47 (m, 1H), 7.32-7.39 (m, 4H), 7.21 (s, 1H), 7.17-7.19 (m, 3H), 6.95-7.01 (m, 3H), 6.85-6.90 (m, 2H), 6.67-6.70 (m, 2H), 5.27 (d, $J = 2.3$ Hz, 1H), 4.53 (d, $J = 1.9$ Hz, 1H), 3.74 (s, 3H), 3.67 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 200.60, 196.21, 158.24, 157.70, 140.85, 138.11, 137.68, 135.52, 134.66, 132.98, 131.88, 131.68, 130.50, 130.41, 129.41, 129.29, 129.21, 128.45, 128.13, 127.91, 127.47, 120.79, 113.79, 111.46, 55.60, 55.21, 52.99, 44.87; HRMS for $\text{C}_{32}\text{H}_{26}\text{O}_4$: calcd. (M+H) $^+$: 475.1904, found: 475.1902

(3-Benzoyl-1-(4-methoxyphenyl)-1,2-dihydronaphthalen-2-yl)(3-methoxyphenyl)

methanone (3f). Yellow gummy solid; isolated yield 50% (71 mg). R_f 0.50 (20% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.65-7.67 (m, 2H), 7.58 (d, $J = 7.6$ Hz, 1H),

7.46-7.50 (m, 1H), 7.37-7.40 (m, 4H), 7.31 (t, $J = 8.0$ Hz, 1H), 7.16-7.24 (m, 3H), 7.02-7.05 (m, 3H), 6.93-6.95 (m, 1H), 6.73 (d, $J = 8.6$ Hz, 2H), 5.13 (d, $J = 3.7$ Hz, 1H), 4.45 (d, $J = 3.5$ Hz, 1H), 3.74 (s, 3H), 3.68 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.58, 196.30, 159.91, 158.63, 142.00, 137.87, 137.52, 137.33, 135.11, 134.59, 131.94, 131.62, 130.84, 129.60, 129.48, 129.42, 129.12, 128.72, 128.28, 127.60, 121.36, 119.82, 114.22, 112.85, 55.40, 55.26, 49.84, 46.26; HRMS for $\text{C}_{32}\text{H}_{26}\text{O}_4$: calcd. (M+H) $^+$: 475.1904, found: 475.1898

(3-Benzoyl-1-(4-methoxyphenyl)-1,2-dihydronaphthalen-2-yl)(4-methoxyphenyl)

methanone (3g). Yellow gummy solid; isolated yield 62% (88 mg). R_f 0.50 (15% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, $J = 8.8$ Hz, 2H), 7.66 (d, $J = 7.1$ Hz, 2H), 7.46-7.49 (m, 1H), 7.34-7.40 (m, 3H), 7.15-7.22 (m, 3H), 7.03 (d, $J = 8.6$ Hz, 2H), 6.91-6.93 (m, 1H), 6.87 (d, $J = 8.8$ Hz, 2H), 6.72 (d, $J = 8.6$ Hz, 2H), 5.12 (d, $J = 3.8$ Hz, 1H), 4.42 (d, $J = 3.7$ Hz, 1H), 3.80 (s, 3H), 3.68 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.25, 196.45, 163.50, 158.56, 142.00, 137.94, 137.46, 135.39, 134.73, 131.89, 131.61, 131.15, 130.76, 129.46, 129.07, 128.82, 128.69, 128.26, 127.51, 114.19, 113.88, 55.49, 55.26, 49.19, 46.50; HRMS for $\text{C}_{32}\text{H}_{26}\text{O}_4$: calcd. (M+H) $^+$: 475.1904, found: 475.1894

(3-Benzoyl-1-(4-(methylthio)phenyl)-1,2-dihydronaphthalen-2-yl)(4-methoxyphenyl)

methanone (3h). Yellow solid; isolated yield 60% (88 mg). R_f 0.50 (15% EtOAc/hexane); Mp 108-109 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3) δ 7.92 (d, $J = 8.9$ Hz, 2H), 7.66 (d, $J = 7.1$ Hz, 2H), 7.46-7.49 (m, 1H), 7.34-7.40 (m, 3H), 7.15-7.22 (m, 3H), 7.06 (dd, $J = 21.1$ Hz, 8.4 Hz, 4H), 6.91-6.93 (m, 1H), 6.82-6.88 (m, 2H), 5.12 (d, $J = 3.6$ Hz, 1H), 4.41 (d, $J = 3.6$ Hz, 1H), 3.79 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.00, 196.36, 163.56, 141.93, 140.18, 137.88, 137.13, 136.92, 134.60, 131.93, 131.63, 131.14, 130.81, 129.51, 129.45, 129.08, 128.72, 128.28, 128.16, 127.67, 127.18, 113.92, 55.49, 48.91, 46.72, 15.96; HRMS for $\text{C}_{32}\text{H}_{26}\text{O}_3\text{S}$: calcd. (M+H) $^+$: 491.1675, found: 491.1674

(3-Benzoyl-1-(4-fluorophenyl)-1,2-dihydronaphthalen-2-yl)(4-ethoxyphenyl)methanone

(3i). White solid; isolated yield 57% (79 mg). R_f 0.50 (10% EtOAc/hexane); Mp 99-101 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, $J = 8.8$ Hz, 2H), 7.65 (d, $J = 7.2$ Hz, 2H), 7.45-7.49 (m, 1H), 7.33-7.39 (m, 3H), 7.16-7.22 (m, 3H), 7.05-7.08 (m, 2H), 6.84-6.90 (m, 5H), 5.10 (d, $J = 4.2$ Hz, 1H), 4.45 (d, $J = 4.1$ Hz, 1H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.24, 196.36, 163.58, 161.81 (d, $J = 244.5$ Hz), 141.73, 138.82 (d, $J = 3.5$ Hz), 137.81, 136.98, 134.77, 132.00, 131.61, 131.06, 130.84, 129.53, 129.44, 129.32 (d, $J = 7.9$ Hz), 128.96, 128.87, 128.29, 127.74, 115.64 (d, $J = 21.3$ Hz), 113.92, 55.48, 49.16, 46.60; HRMS for $\text{C}_{31}\text{H}_{23}\text{FO}_3$: calcd. (M+H) $^+$: 463.1704, found: 463.1705

(3-Benzoyl-1-(4-fluorophenyl)-1,2-dihydronaphthalen-2-yl)(4-fluorophenyl)methanone

(3j). White solid; isolated yield 45% (60 mg). R_f 0.50 (15% EtOAc/hexane); Mp 136-138 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.89 (dd, $J = 8.6$ Hz, 5.4 Hz, 2H), 7.65 (d, $J = 7.2$ Hz, 2H), 7.49 (t, $J = 7.5$ Hz, 1H), 7.39 (t, $J = 7.6$ Hz, 2H), 7.34 (s, 1H), 7.19-7.22 (m, 3H), 7.01-7.08 (m, 4H), 6.85-6.91 (m, 3H), 5.10 (d, $J = 5.2$ Hz, 1H), 4.46 (d, $J = 5.2$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 197.98, 196.31, 165.70 (d, $J = 253.4$ Hz), 161.87 (d, $J = 244.5$ Hz), 141.92, 138.16, 137.55, 136.91, 134.86, 132.78, 132.7, 131.48, 131.27 (d, $J = 9.3$ Hz), 131.01, 129.60, 129.54, 129.46, 129.40, 128.81, 128.37, 127.83, 115.72 (d, $J = 21.1$ Hz), 49.78, 46.73; **HRMS** for $\text{C}_{30}\text{H}_{20}\text{F}_2\text{O}_2$: calcd. (M+H) $^+$: 451.1504, found: 451.1503

(1-(4-Chlorophenyl)-1,2-dihydronaphthalene-2,3-diyl)bis(phenylmethanone) (3k). White solid; isolated yield 65% (87 mg). R_f 0.50 (10% EtOAc/hexane); Mp 119-120 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.86-7.88 (m, 2H), 7.60-7.62 (m, 2H), 7.45-7.49 (m, 2H), 7.35-7.39 (m, 4H), 7.33 (s, 1H), 7.12-7.20 (m, 5H), 7.01 (d, $J = 8.9$ Hz, 2H), 6.88-6.90 (m, 1H), 5.12 (d, $J = 4.0$ Hz, 1H), 4.43 (d, $J = 4.0$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 198.58, 196.21, 141.87, 141.29, 137.67, 136.43, 136.09, 134.42, 133.07, 132.95, 132.06, 131.60, 130.97, 129.63, 129.40, 129.11, 129.02, 128.96, 128.71, 128.32, 127.91, 49.33, 46.34; **HRMS** for $\text{C}_{30}\text{H}_{21}\text{ClO}_2$: calcd. (M+H) $^+$: 449.1303, found: 449.1313

(3-Benzoyl-1-(3-nitrophenyl)-1,2-dihydronaphthalen-2-yl)(4-methoxyphenyl) methanone

(3l). Yellow gummy solid; isolated yield 52% (76 mg). R_f 0.50 (20% EtOAc/hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.99-8.00 (m, 2H), 7.89 (d, $J = 8.7$ Hz, 2H), 7.65 (d, $J = 7.3$ Hz, 2H), 7.45-7.49 (m, 2H), 7.37-7.41 (m, 4H), 7.19-7.28 (m, 4H), 6.92 (br d, $J = 6.8$ Hz, 1H), 6.86 (br d, $J = 8.7$ Hz, 2H), 5.13 (d, $J = 3.7$ Hz, 1H), 4.59 (d, $J = 3.5$ Hz, 1H), 3.79 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 196.45, 196.18, 163.75, 148.55, 144.99, 141.74, 137.56, 135.68, 134.26, 133.93, 132.18, 131.59, 131.15, 131.04, 129.89, 129.83, 129.43, 128.88, 128.55, 128.38, 128.29, 122.79, 122.24, 114.05, 55.53, 48.65, 46.82; **HRMS** for $\text{C}_{31}\text{H}_{23}\text{NO}_5$: calcd. (M+H) $^+$: 490.1649, found: 490.1646

(3-Benzoyl-1-phenyl-1,2-dihydronaphthalen-2-yl)(pyridin-2-yl)methanone (3m).

White solid; isolated yield 60% (74 mg). R_f 0.50 (15% EtOAc/hexane); Mp 135-136 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.69-8.71 (m, 1H), 7.90-7.92 (m, 1H), 7.71-7.75 (m, 1H), 7.58-7.61 (m, 2H), 7.43-7.48 (m, 1H), 7.38-7.41 (m, 1H), 7.33-7.37 (m, 3H), 7.16-7.25 (m, 7H), 7.08-7.12 (m, 1H), 6.90-6.92 (m, 1H), 5.78 (d, $J = 4.0$ Hz, 1H), 4.74 (d, $J = 4.0$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 198.58, 196.08, 152.32, 148.79, 142.80, 141.86, 137.82, 136.93, 135.29, 132.51, 131.80, 130.61, 129.43, 129.29, 129.22, 128.39, 128.26, 128.20, 127.66, 127.02,

126.73, 122.92, 48.92, 46.35; **HRMS** for $C_{29}H_{21}NO_2$: calcd. (M+H)⁺: 416.1645, found: 416.1641

(3-Benzoyl-1-phenyl-1,2-dihydronaphthalen-2-yl)(thiophen-2-yl)methanone (3n). Yellow solid; isolated yield 47% (60 mg). *R_f* 0.50 (15% EtOAc/hexane); Mp 97-98 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.77 (dd, *J* = 3.8 Hz, 1.0 Hz, 1H), 7.64-7.66 (m, 2H), 7.57 (dd, *J* = 5.0 Hz, 1.1 Hz, 1H), 7.46-7.50 (m, 1H), 7.36-7.40 (m, 3H), 7.18-7.24 (m, 5H), 7.13-7.15 (m, 3H), 7.06 (dd, *J* = 5.0 Hz, 3.8 Hz, 1H), 6.96-6.98 (m, 1H), 4.97 (d, *J* = 4.1 Hz, 1H), 4.56 (d, *J* = 4.2 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 196.22, 191.25, 142.96, 142.91, 142.27, 137.75, 136.94, 134.09, 133.91, 132.67, 132.01, 131.68, 130.96, 129.54, 129.44, 129.17, 128.85, 128.31, 128.26, 127.85, 127.70, 127.16, 51.44, 47.65; **HRMS** for $C_{28}H_{20}O_2S$: calcd. (M+H)⁺: 421.1257, found: 421.1260

1-(3-Benzoyl-1-phenyl-1,2-dihydronaphthalen-2-yl)butan-1-one (3o). Yellow gummy solid; isolated yield 52% (59 mg). *R_f* 0.50 (10% EtOAc/hexane); **¹H NMR** (400 MHz, CDCl₃) δ 7.62 (d, *J* = 7.1 Hz, 2H), 7.47-7.50 (m, 1H), 7.37-7.40 (m, 2H), 7.15-7.23 (m, 7H), 7.07 (d, *J* = 7.1 Hz, 2H), 7.00 (d, *J* = 7.0 Hz, 1H), 4.57 (d, *J* = 5.5 Hz, 1H), 4.23 (d, *J* = 5.5 Hz, 1H), 2.47-2.55 (m, 1H), 2.14-2.25 (m, 1H), 1.38-1.45 (m, 2H), 0.71 (t, *J* = 7.4 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 209.68, 196.71, 142.26, 141.40, 137.84, 137.67, 135.01, 132.02, 131.62, 130.95, 129.31, 129.26, 128.93, 128.74, 128.36, 128.10, 127.59, 127.04, 54.92, 46.45, 44.12, 16.78, 13.58; **HRMS** for $C_{27}H_{24}O_2$: calcd. (M+H)⁺: 381.1849, found: 381.1853

(3-Benzoyl-1-phenyl-1,2-dihydronaphthalen-2-yl)(cyclopropyl)methanone (3p). White solid; isolated yield 45% (51 mg). *R_f* 0.50 (10% EtOAc/hexane); Mp 147-148 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.63-7.65 (m, 2H), 7.47-7.51 (m, 1H), 7.37-7.41 (m, 2H), 7.13-7.24 (m, 7H), 7.05-7.09 (m, 3H), 4.67 (d, *J* = 4.4 Hz, 1H), 4.52 (d, *J* = 4.5 Hz, 1H), 1.92-1.99 (m, 1H), 0.83-0.92 (s, 1H), 0.63-0.82 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 208.64, 196.67, 142.52, 141.36, 137.98, 137.83, 134.25, 131.99, 131.47, 130.98, 129.37, 129.30, 129.05, 128.66, 128.34, 127.97, 127.53, 126.93, 55.20, 45.94, 20.09, 11.40, 11.28; **HRMS** for $C_{27}H_{22}O_2$: calcd. (M+H)⁺: 379.1693, found: 379.1686

Ethyl 3-benzoyl-1-phenyl-1,2-dihydronaphthalene-2-carboxylate (3q). White gummy solid; isolated yield 52% (59 mg). *R_f* 0.50 (15% EtOAc/hexane); **¹H NMR** (400 MHz, CDCl₃) δ 7.65 (d, *J* = 7.0 Hz, 2H), 7.46-7.50 (m, 1H), 7.36-7.40 (m, 2H), 7.16-7.23 (m, 6H), 7.13 (d, *J* = 7.1 Hz, 1H), 7.09 (d, *J* = 7.0 Hz, 1H), 7.04 (d, *J* = 7.1 Hz, 2H), 4.75 (d, *J* = 4.0 Hz, 1H), 4.29 (d, *J* = 4.1 Hz, 1H), 3.98 (q, *J* = 7.1 Hz, 2H), 1.00 (t, *J* = 7.1 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 192.52, 168.54, 138.41, 136.01, 134.26, 133.56, 129.95, 128.22,

127.85, 127.08, 125.69, 125.51, 124.87, 124.57, 124.13, 123.96, 123.23, 57.47, 43.39, 42.38, 10.28; **HRMS** for C₂₆H₂₂O₃: calcd. (M+H)⁺: 383.1642, found: 383.1638

(3-Benzoyl-6-methyl-4-phenyl-3,4-dihydronaphthalen-2-yl)(p-tolyl)methanone (3r).

Yellow oil; isolated yield 56% (74 mg). *R_f* 0.50 (10% EtOAc/hexane); **¹H NMR** (400 MHz, CDCl₃) δ 7.95 (d, *J* = 7.3 Hz, 2H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.47-7.51 (m, 1H), 7.41 (d, *J* = 7.8 Hz, 2H), 7.37-7.38 (poorly resolved m, 1H), 7.17-7.22 (m, 5H), 7.11-7.15 (m, 4H), 6.99-7.01 (poorly resolved m, 1H), 6.76 (br s, 1H), 5.14 (d, *J* = 3.6 Hz, 1H), 4.42 (d, *J* = 3.5 Hz, 1H), 2.36 (s, 3H), 2.19 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 198.70, 196.01, 143.16, 142.46, 141.92, 141.23, 136.77, 136.13, 135.20, 133.57, 132.88, 130.00, 129.56, 129.44, 129.23, 128.92, 128.83, 128.79, 128.62, 128.44, 127.70, 127.02, 49.57, 46.98, 21.61; **HRMS** for C₃₂H₂₆O₂: calcd. (M+H)⁺: 443.2006, found: 443.2010

(1-(4-Methoxyphenyl)-7-methyl-1,2-dihydronaphthalene-2,3-diyl)bis(p-tolylmethanone) (3s).

Yellow solid; isolated yield 66% (92 mg). *R_f* 0.50 (15% EtOAc/hexane); Mp 120-121 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.85 (d, *J* = 8.2 Hz, 2H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.34 (s, 1H), 7.16-7.20 (m, 4H), 7.10 (br d, *J* = 7.7 Hz, 1H), 7.01-7.03 (m, 2H), 6.96 (br d, *J* = 7.7 Hz, 1H), 6.71-6.73 (m, 3H), 5.08 (d, *J* = 3.3 Hz, 1H), 4.35 (d, *J* = 3.2 Hz, 1H), 3.68 (s, 3H), 2.35 (s, 3H), 2.34 (s, 3H), 2.17 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 198.21, 196.07, 158.51, 143.65, 142.38, 141.83, 141.12, 137.22, 135.60, 135.32, 133.60, 133.45, 129.93, 129.59, 129.39, 129.35, 129.14, 129.00, 128.90, 128.63, 128.30, 114.16, 55.25, 49.48, 46.27, 21.64, 21.60; **HRMS** for C₃₄H₃₀O₃: calcd. (M+H)⁺: 487.2268, found: 487.2267

(3-(2-Methoxybenzoyl)-4-(4-methoxyphenyl)-6-methyl-3,4-dihydronaphthalen-2-yl)(p-tolyl) methanone (3t).

Brown solid; isolated yield 60% (90 mg). *R_f* 0.50 (20% EtOAc/hexane); Mp 145-146 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.45 (d, *J* = 7.2 Hz, 2H), 7.38-7.40 (m, 1H), 7.30-7.35 (m, 1H), 7.19 (s merged with CDCl₃ peak, 1H), 7.13 (d, *J* = 7.6 Hz, 2H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 8.3 Hz, 3H), 6.83-6.89 (m, 3H), 6.68 (d, *J* = 7.6 Hz, 2H), 5.21 (br s, 1H), 4.49 (br s, 1H), 3.72 (s, 3H), 3.67 (s, 3H), 2.33 (s, 3H), 2.20 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 200.67, 195.90, 158.21, 157.68, 142.16, 140.81, 140.64, 137.72, 135.81, 135.46, 133.67, 132.86, 130.40, 130.01, 129.55, 129.32, 129.20, 128.77, 128.48, 128.19, 128.05, 120.73, 113.78, 111.44, 55.57, 55.21, 53.08, 44.86, 21.62, 21.58; **HRMS** for C₃₄H₃₀O₄: calcd. (M+H)⁺: 503.2217, found: 503.2213

(3-(3-Methoxybenzoyl)-4-(4-methoxyphenyl)-6-methyl-3,4-dihydronaphthalen-2-yl)(p-tolyl)methanone (3u).

Yellow solid; isolated yield 53% (80 mg). *R_f* 0.50 (20% EtOAc/hexane); Mp 98-101 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.57 (d, *J* = 7.8 Hz, 1H), 7.54 (d, *J* = 8.1 Hz, 2H), 7.40 (t, *J* = 2.2 Hz, 1H), 7.34 (s, 1H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.17 (d, *J* =

8.0 Hz, 2H), 7.11 (d, $J = 7.7$ Hz, 1H), 7.02 (d, $J = 8.8$ Hz, 3H), 6.98 (d, $J = 7.3$ Hz, 1H), 6.71-6.76 (m, 3H), 5.06 (d, $J = 3.4$ Hz, 1H), 4.38 (d, $J = 3.4$ Hz, 1H), 3.74 (s, 3H), 3.68 (s, 3H), 2.35 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.51, 196.02, 159.87, 158.55, 142.47, 141.82, 141.22, 137.49, 137.20, 135.38, 135.23, 133.54, 129.95, 129.57, 129.41, 129.10, 128.93, 128.68, 128.35, 121.41, 119.83, 114.17, 112.80, 55.40, 55.25, 49.88, 46.21, 21.63, 21.61; HRMS for $\text{C}_{34}\text{H}_{30}\text{O}_4$: calcd. (M+H) $^+$: 503.2217, found: 503.2218

(3-(4-Methoxybenzoyl)-6-methyl-4-(4-(methylthio)phenyl)-3,4-dihydronaphthalen-2-yl)(p-tolyl)methanone (3v). Yellow solid; isolated yield 82% (127 mg). R_f 0.50 (15% EtOAc/hexane); Mp 98-100 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, $J = 8.8$ Hz, 2H), 7.55 (d, $J = 8.0$ Hz, 2H), 7.33 (s, 1H), 7.17 (d, $J = 8.0$ Hz, 2H), 7.02-7.12 (m, 5H), 6.98 (d, $J = 7.8$ Hz, 1H), 6.87 (d, $J = 8.8$ Hz, 2H), 6.74 (s, 1H), 5.07 (d, $J = 3.2$ Hz, 1H), 4.35 (d, $J = 3.0$ Hz, 1H), 3.80 (s, 3H), 2.35, 2.37 (2s merged, 6H), 2.17 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.00, 196.06, 163.51, 142.45, 141.73, 141.18, 140.46, 137.00, 136.84, 135.28, 133.60, 131.17, 129.93, 129.59, 129.45, 129.13, 128.93, 128.77, 128.41, 128.15, 127.18, 113.89, 55.48, 48.98, 46.72, 21.61, 15.98; HRMS for $\text{C}_{34}\text{H}_{30}\text{O}_3\text{S}$: calcd. (M+H) $^+$: 519.1988, found: 519.1992

(3-Benzoyl-4-(4-fluorophenyl)-6-methyl-3,4-dihydronaphthalen-2-yl)(p-tolyl) methanone (3w). White solid; isolated yield 45% (62 mg). R_f 0.50 (10% EtOAc/hexane); Mp 92-94 °C; ^1H NMR (500 MHz, CDCl_3) δ 7.90 (d, $J = 7.4$ Hz, 2H), 7.54 (d, $J = 8.0$ Hz, 2H), 7.47 (t, $J = 7.6$ Hz, 1H), 7.37 (t, $J = 7.8$ Hz, 2H), 7.33 (s, 1H), 7.17 (d, $J = 8.0$ Hz, 2H), 7.12 (d, $J = 7.7$ Hz, 1H), 7.04-7.07 (m, 2H), 7.00 (d, $J = 7.5$ Hz, 1H), 6.86 (t, $J = 8.6$ Hz, 2H), 6.72 (s, 1H), 5.10 (d, $J = 4.0$ Hz, 1H), 4.41 (d, $J = 4.0$ Hz, 1H), 2.35 (s, 3H), 2.17 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.88, 195.99, 161.78 (d, $J = 244.0$ Hz), 142.60, 141.65, 141.35, 138.75 (d, $J = 2.7$ Hz), 136.75, 136.25, 135.08, 133.61, 132.94, 129.81, 129.55, 129.51, 129.31 (d, $J = 8.0$ Hz), 129.09, 128.96, 128.72, 128.64, 128.55, 115.60 (d, $J = 21.2$ Hz), 49.69, 46.31, 21.62, 21.61; HRMS for $\text{C}_{32}\text{H}_{25}\text{FO}_2$: calcd. (M+H) $^+$: 461.1911, found: 461.1907

(5-Bromo-2,4-dimethoxyphenyl)(8-bromo-3-(2,4-dimethoxybenzoyl)-5,6-dimethoxy-4-(4-methoxyphenyl)-3,4-dihydronaphthalen-2-yl)methanone (3x). Yellow solid; isolated yield 40% (93 mg). R_f 0.50 (50% EtOAc/hexane); Mp 120-121 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.62-7.64 (m, 1H), 7.57 (s, 1H), 7.51 (s, 1H), 7.14 (d, $J = 8.6$ Hz, 2H), 7.00 (s, 1H), 6.77 (d, $J = 8.7$ Hz, 2H), 6.50-6.53 (m, 3H), 5.40 (s, 1H), 4.91 (s, 1H), 3.98 (s, 3H), 3.88 (s, 3H), 3.87 (s, 3H), 3.85 (s, 3H), 3.77 (s, 6H), 3.43 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.66, 193.33, 164.19, 160.15, 158.45, 158.30, 158.23, 154.85, 145.87, 140.26, 135.16, 135.03, 134.55, 134.29, 132.96, 128.54, 125.00, 122.31, 120.23, 119.90, 114.99, 113.61, 105.29,

101.86, 98.52, 96.60, 60.36, 56.35, 56.08, 55.91, 55.73, 55.50, 55.21, 50.46, 39.45; **HRMS** for $C_{37}H_{34}Br_2O_9$: calcd. (M+H)⁺: 781.0642, found: 781.0643

(7-Chloro-1-(4-chlorophenyl)-1,2-dihydronaphthalene-2,3-diyl)bis(phenylmethanone)

(3y). Yellow solid; isolated yield 50% (72 mg). R_f 0.50 (15% EtOAc/hexane); Mp 115-117 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.87-7.89 (m, 2H), 7.60-7.62 (m, 2H), 7.47-7.52 (m, 2H), 7.36-7.41 (m, 4H), 7.30 (s, 1H), 7.16-7.19 (m, 4H), 7.00-7.03 (m, 2H), 6.91 (br s, 1H), 5.13 (d, J = 3.9 Hz, 1H), 4.42 (d, J = 3.9 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 198.24, 195.94, 140.50, 140.45, 138.24, 137.44, 136.54, 135.83, 134.70, 133.26, 132.21, 130.59, 130.13, 129.37, 129.24, 129.15, 129.01, 128.79, 128.73, 128.38, 128.14, 49.05, 46.19; **HRMS** for $C_{30}H_{20}Cl_2O_2$: calcd. (M+H)⁺: 483.0913, found: 483.0914

(7-Fluoro-1-phenyl-1,2-dihydronaphthalene-2,3-diyl)bis(phenylmethanone) (3z). White

solid; isolated yield 54% (70 mg). R_f 0.50 (10% EtOAc/hexane); Mp 90-92 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, J = 7.6 Hz, 2H), 7.63 (d, J = 7.5 Hz, 2H), 7.48 (t, J = 7.5 Hz, 2H), 7.38 (t, J = 7.7 Hz, 4H), 7.33 (s, 1H), 7.13-7.23 (m, 4H), 7.09-7.11 (m, 2H), 6.85-6.90 (m, 1H), 6.66 (dd, J = 9.2 Hz, 2.4 Hz, 1H), 5.17 (d, J = 4.2 Hz, 1H), 4.45 (d, J = 4.1 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 198.81, 196.13, 164.03 (d, J = 250.2 Hz), 142.07, 140.91, 139.83 (d, J = 7.9 Hz), 137.70, 136.02, 134.21 (d, J = 2.4 Hz), 133.08, 132.01, 131.24 (d, J = 8.6 Hz), 129.37, 128.97, 128.77, 128.68, 128.32, 128.01 (d, J = 3.1 Hz), 127.69, 127.40, 116.51 (d, J = 22.4 Hz), 114.70 (d, J = 21.8 Hz), 49.10, 47.22; **HRMS** for $C_{30}H_{21}FO_2$: calcd. (M+H)⁺: 433.1598, found: 433.1603

(3-Benzoyl-7-fluoro-1-phenyl-1,2-dihydronaphthalen-2-yl)(thiophen-2-yl)methanone

(3za). Yellow gummy solid; isolated yield 58% (76 mg). R_f 0.50 (15% EtOAc/hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, J = 3.6 Hz, 1H), 7.62 (d, J = 7.3 Hz, 2H), 7.57 (d, J = 4.8 Hz, 1H), 7.46-7.49 (m, 1H), 7.35-7.39 (m, 2H), 7.31 (s, 1H), 7.10-7.23 (m, 6H), 7.05 (t, J = 4.6 Hz, 1H), 6.85-6.92 (m, 1H), 6.69 (dd, J = 9.2 Hz, 2.2 Hz, 1H), 4.95 (d, J = 4.2 Hz, 1H), 4.52 (d, J = 4.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 196.03, 191.25, 164.10 (d, J = 250.8 Hz), 142.86, 142.10, 141.04, 139.89 (d, J = 7.9 Hz), 137.64, 134.28, 133.48, 132.79, 132.06, 131.26 (d, J = 8.7 Hz), 129.39, 128.98, 128.33, 127.98, 127.78, 127.44, 116.54 (d, J = 22.7 Hz), 114.69 (d, J = 21.7 Hz), 50.98, 47.78; **HRMS** for $C_{28}H_{19}FO_2S$: calcd. (M+H)⁺: 439.1163, found: 439.1161

(3-Benzoyl-4-phenyl-3,4-dihydronaphthalen-2-yl)(thiophen-2-yl)methanone (3zb).

Yellow solid; isolated yield 55% (69 mg). R_f 0.50 (10% EtOAc/hexane); Mp 111-113 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.88-7.90 (m, 2H), 7.68 (dd, J = 3.8 Hz, 1.1 Hz, 1H), 7.66 (s, 1H), 7.57 (dd, J = 5.0 Hz, 1.1 Hz, 1H), 7.44-7.48 (m, 1H), 7.34-7.38 (m, 2H), 7.30-7.32 (m,

1H), 7.15-7.23 (m, 4H), 7.06-7.11 (m, 4H), 6.93 (br d, $J = 6.8$ Hz, 1H), 5.12 (d, $J = 4.2$ Hz, 1H), 4.45 (d, $J = 4.2$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.56, 187.30, 142.75, 142.71, 140.04, 136.89, 136.10, 134.82, 133.34, 133.28, 132.93, 131.69, 130.77, 129.45, 129.12, 128.80, 128.78, 128.60, 127.77, 127.73, 127.12, 50.03, 47.08; HRMS for $\text{C}_{28}\text{H}_{20}\text{O}_2\text{S}$: calcd. (M+H) $^+$: 421.1257, found: 421.1257

Ethyl 3-benzoyl-4-phenyl-3,4-dihydronaphthalene-2-carboxylate (3zc). White solid; isolated yield 57% (65 mg). R_f 0.50 (15% EtOAc/hexane); Mp 101-102 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.94-7.66 (m, 2H), 7.85 (s, 1H), 7.52-7.56 (m, 1H), 7.42-7.46 (m, 2H), 7.34-7.36 (m, 1H), 7.14-7.23 (m, 5H), 7.05-7.08 (m, 2H), 6.88 (d, $J = 7.3$ Hz, 1H), 4.98 (d, $J = 3.0$ Hz, 1H), 4.35 (d, $J = 3.0$ Hz, 1H), 4.10 (q, $J = 7.1$ Hz, 2H), 1.15 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.28, 166.58, 143.51, 138.98, 136.23, 135.84, 133.06, 131.57, 130.46, 129.33, 129.12, 128.83, 128.82, 128.73, 127.69, 127.54, 127.05, 126.11, 60.90, 49.18, 46.93, 14.08; HRMS for $\text{C}_{26}\text{H}_{22}\text{O}_3$: calcd. (M+H) $^+$: 383.1642, found: 383.1644

(6,7-Dimethoxy-1-(4-methoxyphenyl)-1,2-dihydronaphthalene-2,3-diyl)bis((2,4-dimethoxy phenyl)methanone) (3zd). Yellow solid; R_f 0.50 (50% EtOAc/hexane); Mp 185-187 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.65 (d, $J = 8.6$ Hz, 1H), 7.21 (d, $J = 8.4$ Hz, 1H), 7.16 (s, 1H), 7.06 (d, $J = 8.3$ Hz, 2H), 6.71 (d, $J = 8.3$ Hz, 2H), 6.48 (s, 1H), 6.42-6.46 (m, 4H), 5.26 (s, 1H), 4.31 (s, 1H), 3.79 (s, 6H), 3.76 (s, 3H), 3.74 (s, 3H), 3.70 (2 s merged, 6H), 3.65 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 197.67, 195.04, 164.23, 162.36, 160.35, 158.94, 158.19, 150.59, 147.86, 141.75, 136.84, 134.59, 133.59, 131.62, 131.37, 128.42, 124.86, 122.06, 120.07, 113.65, 112.10, 105.35, 104.15, 98.88, 98.52, 55.91, 55.88, 55.71, 55.68, 55.51, 55.45, 55.24, 51.58, 45.50; HRMS for $\text{C}_{37}\text{H}_{36}\text{O}_9$: calcd. (M+H) $^+$: 625.2432, found: 625.2431

(7,8-Dimethoxy-1-(4-methoxyphenyl)-1,2-dihydronaphthalene-2,3-diyl)bis((2,4-dimethoxy phenyl)methanone) (3ze). White solid; R_f 0.50 (50% EtOAc/hexane); Mp 125-127 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.56 (d, $J = 8.9$ Hz, 1H), 7.02-7.09 (m, 4H), 6.71 (s, 1H), 6.65 (d, $J = 8.5$ Hz, 2H), 6.51 (s, 1H), 6.39-6.48 (m, 4H), 4.93 (s, 1H), 4.58 (s, 1H), 3.81 (s, 9H), 3.77 (s, 3H), 3.73 (s, 3H), 3.67 (s, 3H), 3.61 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 199.01, 195.34, 164.33, 162.31, 159.76, 158.90, 158.24, 150.50, 148.17, 140.10, 137.10, 135.09, 133.26, 130.97, 128.63, 127.78, 126.34, 122.33, 120.14, 113.54, 112.78, 111.80, 105.56, 104.13, 98.91, 98.44, 57.55, 55.90, 55.84, 55.66, 55.57, 55.44, 55.14, 39.58; HRMS for $\text{C}_{37}\text{H}_{36}\text{O}_9$: calcd. (M+H) $^+$: 625.2432, found: 625.2431

(3-Benzoyl-1-(p-tolyl)naphthalen-2-yl)(4-methoxyphenyl)methanone (4a). Yellow solid; isolated yield 56% (25 mg). R_f 0.50 (20% EtOAc/hexane); Mp 138-140 °C; ^1H NMR (400

MHz, CDCl₃) δ 8.11 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.85-7.88 (m, 2H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.51-7.61 (m, 5H), 7.43-7.47 (m, 2H), 7.04, 7.11 (ABq, *J* = 7.9 Hz, 4H), 6.67-6.71 (m, 2H), 3.76 (s, 3H), 2.28 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 196.77, 196.25, 162.90, 138.98, 137.55, 137.46, 137.26, 135.04, 133.78, 133.44, 132.88, 131.97, 131.54, 131.16, 130.68, 130.50, 129.12, 128.69, 128.54, 128.30, 127.40, 126.99, 113.14, 55.33, 21.22; HRMS for C₃₂H₂₄O₃: calcd. (M+H)⁺: 457.1798, found: 457.1796

(3-Benzoyl-1-(4-methoxyphenyl)naphthalen-2-yl)(3-methoxyphenyl)methanone (4b).

Yellow solid; isolated yield 60% (28 mg). *R_f* 0.50 (25% EtOAc/hexane); Mp 169-171 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.06 (s, 1H), 7.87 (d, *J* = 7.5 Hz, 1H), 7.80-7.82 (m, 2H), 7.63 (d, *J* = 8.3 Hz, 1H), 7.46-7.55 (m, 3H), 7.38-7.42 (m, 2H), 7.00-7.07 (m, 5H), 6.81-6.84 (m, 1H), 6.68-6.72 (m, 2H), 3.69 (s, 3H), 3.64 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 198.17, 196.07, 159.21, 159.03, 140.01, 138.95, 137.72, 137.40, 134.96, 133.98, 132.88, 132.10, 132.04, 131.45, 130.48, 129.20, 128.85, 128.80, 128.48, 128.33, 127.51, 126.93, 122.50, 119.21, 113.37, 112.65, 55.30, 55.20; HRMS for C₃₂H₂₄O₄: calcd. (M+H)⁺: 473.1747, found: 473.1752

(3-Benzoyl-1-(4-methoxyphenyl)naphthalen-2-yl)(4-methoxyphenyl)methanone (4c).

White solid; isolated yield 30% (14 mg). *R_f* 0.50 (15% EtOAc/hexane); Mp 194-195 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.04 (s, 1H), 7.86 (d, *J* = 7.9 Hz, 1H), 7.80 (d, *J* = 7.3 Hz, 2H), 7.63 (d, *J* = 8.2 Hz, 1H), 7.46-7.54 (m, 5H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.07 (d, *J* = 8.2 Hz, 2H), 6.70 (d, *J* = 8.5 Hz, 2H), 6.62 (d, *J* = 8.8 Hz, 2H), 3.69, 3.70 (2 s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 196.91, 196.25, 162.89, 158.97, 138.56, 137.79, 137.44, 135.08, 133.91, 132.89, 132.01, 131.91, 131.51, 131.14, 130.52, 129.15, 128.70, 128.63, 128.30, 127.38, 126.90, 113.35, 113.18, 55.32, 55.19; HRMS for C₃₂H₂₄O₄: calcd. (M+H)⁺: 473.1747, found: 473.1747

(3-(3-Methoxybenzoyl)-4-(4-methoxyphenyl)-6-methylnaphthalen-2-yl)(p-

tolyl)methanone (4d). White solid; isolated yield 50% (25 mg). *R_f* 0.50 (25% EtOAc/hexane); Mp 130-132 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.02 (s, 1H), 7.77 (d, *J* = 8.7 Hz, 1H), 7.71 (d, *J* = 7.8 Hz, 2H), 7.37 (br d, *J* = 5.2 Hz, 2H), 7.19 (d merged with CDCl₃ peak, *J* = 7.6 Hz, 2H), 7.00-7.06 (m, 5H), 6.82 (d, *J* = 6.8 Hz, 1H), 6.70 (d, *J* = 8.4 Hz, 2H), 3.71 (s, 3H), 3.64 (s, 3H), 2.37 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 198.33, 195.75, 159.18, 158.91, 143.59, 140.13, 139.09, 138.18, 137.91, 134.90, 134.29, 134.12, 132.11, 131.23, 130.64, 130.23, 129.68, 129.04, 128.99, 128.74, 128.68, 125.84, 122.50, 119.16, 113.32, 112.55, 55.28, 55.18, 22.16, 21.70; HRMS for C₃₄H₂₈O₄: calcd. (M+H)⁺: 501.2060, found: 501.2067

(3-(4-Methoxybenzoyl)-6-methyl-4-(4-(methylthio)phenyl)naphthalen-2-yl)(p-tolyl) methanone (4e). White solid; isolated yield 26% (13 mg). R_f 0.50 (20% EtOAc/hexane); Mp 205-207 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.01 (s, 1H), 7.76 (d, $J = 8.3$ Hz, 1H), 7.69 (d, $J = 8.2$ Hz, 2H), 7.45-7.48 (m, 2H), 7.35-7.37 (m, 1H), 7.33 (br s, 1H), 7.18 (d, $J = 7.9$ Hz, 2H), 7.05 (s, 4H), 6.60-6.64 (m, 2H), 3.70 (s, 3H), 2.36, 2.37, 2.38 (3s merged, 9H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 196.85, 195.83, 162.88, 143.65, 139.09, 137.86, 137.50, 134.91, 134.34, 133.77, 133.32, 132.01, 131.47, 131.23, 131.12, 130.66, 130.18, 129.66, 129.02, 128.98, 125.69, 113.17, 55.33, 22.16, 21.71, 15.56; **HRMS** for $\text{C}_{34}\text{H}_{28}\text{O}_3\text{S}$: calcd. (M+H) $^+$: 517.1832, found: 517.1829

(6,7-Dimethoxy-1-(4-methoxyphenyl)naphthalene-2,3-diyl)bis((2,4-dimethoxyphenyl) methanone) (4f). White solid; isolated yield 14% (9 mg). R_f 0.50 (60% EtOAc/hexane); Mp 138-140 °C; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.80 (s, 1H), 7.36 (d, $J = 8.7$ Hz, 1H), 7.32 (d, $J = 8.1$ Hz, 1H), 7.09 (s, 1H), 7.04 (d, $J = 7.7$ Hz, 2H), 6.80 (s, 1H), 6.70 (d, $J = 7.7$ Hz, 2H), 6.38-6.40 (br m, 2H), 6.22 (d, $J = 8.4$ Hz, 1H), 6.13 (s, 1H), 3.92 (s, 3H), 3.78 (s, 3H), 3.70 (s, 6H), 3.67 (s, 3H), 3.63 (s, 3H), 3.42 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 195.07, 194.53, 164.02, 163.57, 160.64, 160.35, 158.57, 150.99, 149.91, 139.07, 135.42, 135.11, 133.69, 133.40, 131.59, 130.30, 129.83, 129.33, 127.88, 122.37, 121.86, 113.22, 107.33, 105.63, 104.47, 104.37, 98.79, 98.06, 55.95, 55.80, 55.72, 55.48, 55.42, 55.35, 55.15; **HRMS** for $\text{C}_{37}\text{H}_{34}\text{O}_9$: calcd. (M+H) $^+$: 623.2276, found: 623.2270

(7,8-Dimethoxy-1-(4-methoxyphenyl)naphthalene-2,3-diyl)bis((2,4-dimethoxyphenyl) methanone) (4g). White solid; isolated yield 13% (8 mg). R_f 0.50 (60% EtOAc/hexane); Mp 147-149 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.86 (s, 1H), 7.09-7.19 (m, 4H), 6.82 (d, $J = 7.8$ Hz, 2H), 6.37 (d, $J = 8.3$ Hz, 2H), 6.16-6.19 (m, 1H), 6.10-6.13 (m, 1H), 6.07 (br s, 2H), 3.95 (s, 3H), 3.80 (s, 3H), 3.68 (s, 3H), 3.66 (s, 3H), 3.55 (s, 3H), 3.48 (s, 3H), 3.41 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 197.17, 196.70, 164.38, 163.87, 160.76, 160.02, 158.19, 151.14, 149.81, 139.07, 138.43, 133.75, 133.46, 132.99, 131.37, 130.78, 127.79, 127.38, 127.02, 122.29, 122.23, 112.61, 107.03, 104.40, 104.15, 104.09, 98.22, 98.11, 55.96, 55.91, 55.58, 55.40, 55.11; **HRMS** for $\text{C}_{37}\text{H}_{34}\text{O}_9$: calcd. (M+H) $^+$: 623.2276, found: 623.2274

3. Crystallographic Data

3.1 Crystallographic data for **31**

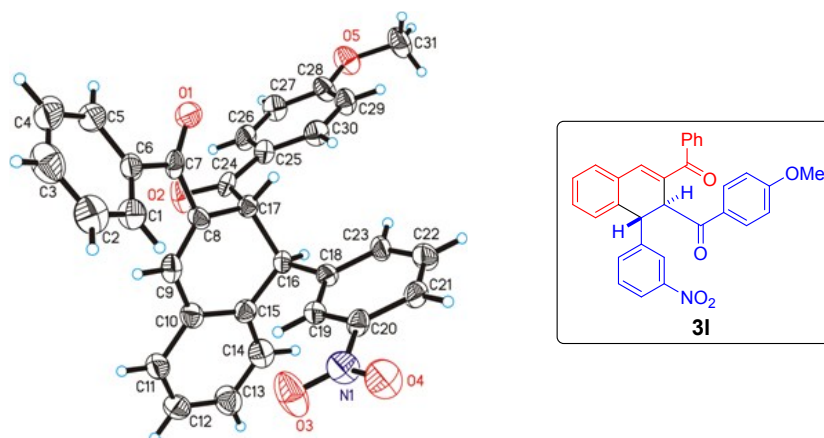


Figure 1. ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **31** determined at 293 K.

Crystallization: Crystals of compound **31** were grown from the solvent DCM:EtOH (1:3) by slow evaporation method.

Table 1. Crystal data and structure refinement details for **31**

Compound	31
Empirical formula	C ₃₁ H ₂₃ N O ₅
Formula weight	489.50
Crystal System	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> (Å)	13.541(8)
<i>b</i> (Å)	9.928(6)
<i>c</i> (Å)	20.222(15)
α (°)	90.00
β (°)	90.670(14)
γ (°)	90.00
<i>V</i> (Å ³)	2718(3)
<i>Z</i>	4
<i>D</i> _c (g/cm ³)	1.196
<i>F</i> ₀₀₀	1024
μ (mm ⁻¹)	0.081
θ_{\max} (°)	25.40
Total reflections	13071
Unique reflections	4683
Reflections [<i>I</i> > 2 σ (<i>I</i>)]	1282
Parameters	335
<i>R</i> _{int}	0.1222
Goodness-of-fit	0.859
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)]	0.0936
<i>wR</i> (<i>F</i> ² , all data)	0.2884
CCDC No.	1816363

3.2 Crystallographic data for 3x

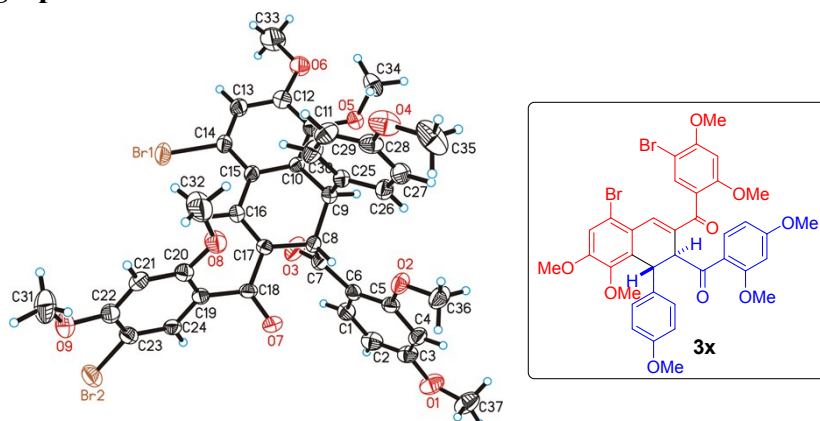


Figure 2 ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **3x** determined at 293 K.

Crystallization: Crystals of compound **3x** were grown from the solvent DCM:EtOH (1:3) by slow evaporation method.

Table 2 Crystal data and structure refinement details for **3x**

Compound	3x
Empirical formula	C ₃₈ H ₃₄ Br ₂ O ₁₀
Formula weight	810.47
Crystal System	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> (Å)	17.651(4)
<i>b</i> (Å)	11.598(3)
<i>c</i> (Å)	18.625(5)
<i>α</i> (°)	90.00
<i>β</i> (°)	98.778(5)
<i>γ</i> (°)	90.00
<i>V</i> (Å ³)	3768.2(16)
<i>Z</i>	4
<i>D_c</i> (g/cm ³)	1.429
<i>F</i> ₀₀₀	1648
<i>μ</i> (mm ⁻¹)	2.206
<i>θ</i> _{max} (°)	25.38
Total reflections	23722
Unique reflections	6762
Reflections [<i>I</i> > 2σ(<i>I</i>)]	3424
Parameters	458
<i>R</i> _{int}	0.0752
Goodness-of-fit	0.999
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)]	0.0647
<i>wR</i> (<i>F</i> ² , all data)	0.1871
CCDC No.	1816362

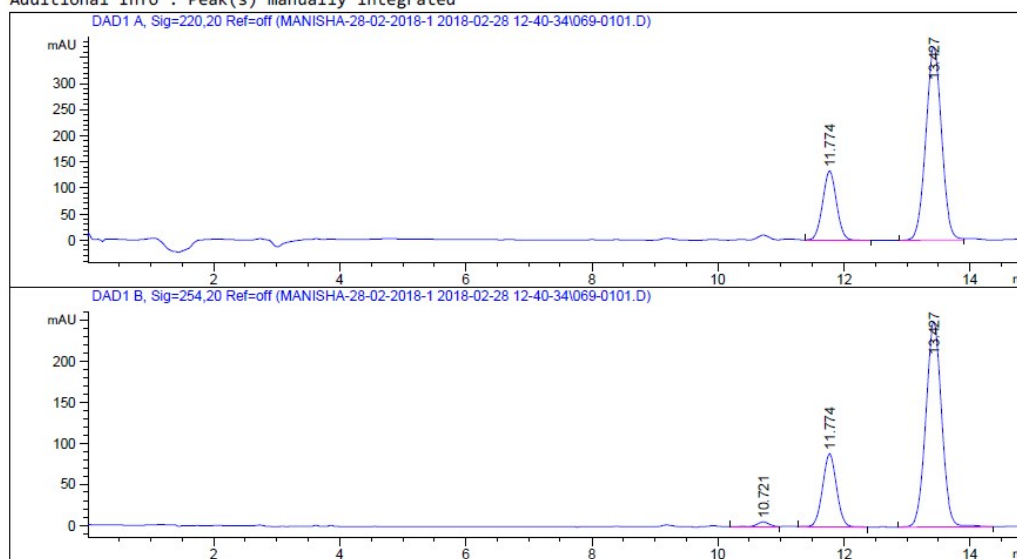
4. Experimental HPLC Chromatogram of the Product Mixture of Reaction between 1s & 2h

Data File C:\CHEM32\1\DATA\MANISHA-28-02-2018-1 2018-02-28 12-40-34\069-0101.D
 Sample Name: NAM-1

```

=====
Acq. Operator   : Dr. Anil Kumar K.S.           Seq. Line :    1
Acq. Instrument : Instrument 1                   Location  : Vial 69
Injection Date  : 2/28/2018 12:42:25 PM        Inj       :    1
                                                Inj Volume: 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\MANISHA-28-02-2018-1 2018-02-28 12-40-34\ACN-METH-WATER-35
                                                -35-30.M
Last changed    : 2/28/2018 11:52:20 AM by Dr. Anil Kumar K.S.
Analysis Method : C:\CHEM32\1\METHODS\ACN-METH-WATER-35-35-30.M
Last changed    : 2/28/2018 11:52:20 AM by Dr. Anil Kumar K.S.
Method Info     : OSDD
  
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=220,20 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.774	VB	0.2420	2053.78418	133.10472	24.1867
2	13.427	BV	0.2710	6437.61035	370.04031	75.8133

Totals : 8491.39453 503.14503

Signal 2: DAD1 B, Sig=254,20 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.721	BV	0.2118	79.16134	5.77861	1.3644
2	11.774	BB	0.2407	1362.77673	88.97102	23.4882
3	13.427	BB	0.2718	4360.01416	249.59172	75.1474

Totals : 5801.95223 344.34136

*** End of Report ***

5. References

- (1) For **1a**: (a) P. Ahmad, H. Woo, K. Jun, A. A. Kadi, H. A. Abdel-Aziz, K. Kwon and A. F. M. M. Rahman, *Bioorg. Med. Chem.*, 2016, **24**, 1898-1908; For **1b**, **1d**, **1g**: (b) I. Kazi, S. Guha and G. Sekar, *Org. Lett.*, 2017, **19**, 1244-1247; For **1c**, **1i**, **1k**: (c) L. Zhang, A. Wang, W. Wang, Y. Huang, X. Liu, S. Miao, J. Liu and T. Zhang, *ACS Catal.*, 2015, **5**, 6563-6572; For **1e**: (d) S. M. Stevenson, R. F. Higgins, M. P. Shores and E. M. Ferreira, *Chem. Sci.*, 2017, **8**, 654-660; For **1f**: (e) M. D. Bowman, M. M. Jacobson and H. E. Blackwell, *Org. Lett.*, 2016, **8**, 1645-1648; For **1h**: (f) B. Umesha and Y. B. Basavaraju, *Russ. J. Bioorg. Chem.*, 2014, **40**, 467-476; For **1j**, **1r**: (g) C. Chan, Y. Tsai and M. Y. Chang, *Tetrahedron*, 2017, **73**, 3368-3376; For **1l**: (h) P. Gao, K. Zhang, M. Yang, S. Xu, H. Sun, J. Zhang, Z. Gao, W. Zhang and L. Xu, *Chem. Commun.*, 2018, **54**, 5074-5077; For **1m**: (i) M. Li, V. Carreras, A. Jalba and T. Ollevier, *Org. Lett.*, 2018, **20**, 995-998; For **1n**: (j) B. Liu, Y. Bao, F. Du, H. Wang, J. Tian and R. Bai, *Chem. Commun.*, 2011, **47**, 1731-1733; For **1o**: (k) A. Ueda, T. Umeno, M. Doi, K. Akagawa, K. Kudo and M. Tanaka, *J. Org. Chem.*, 2016, **81**, 6343-6356; For **1p**: (l) D. Wang, Y. Zhang, A. Harris, L. N. S. Gautam, Y. Chen and X. Shi, *Adv. Synth. Catal.*, 2011, **353**, 2584-2588; For **1q**: (m) P. Gao, K. Zhang, M. Yang, S. Xu, H. Sun, J. Zhang, Z. Gao, W. Zhang and L. Xu, *Chem. Commun.*, 2018, **54**, 5074-5077; For **1s**: (n) A. Bianco, C. Cavarischia and M. Guis, *Eur. J. Org. Chem.*, 2004, 2894-2898.
- (2) S. Paria and O. Reiser, *Adv. Synth. Catal.*, 2014, **356**, 557-562.

6. Copies of ^1H and ^{13}C NMR Spectra

NRMP-604

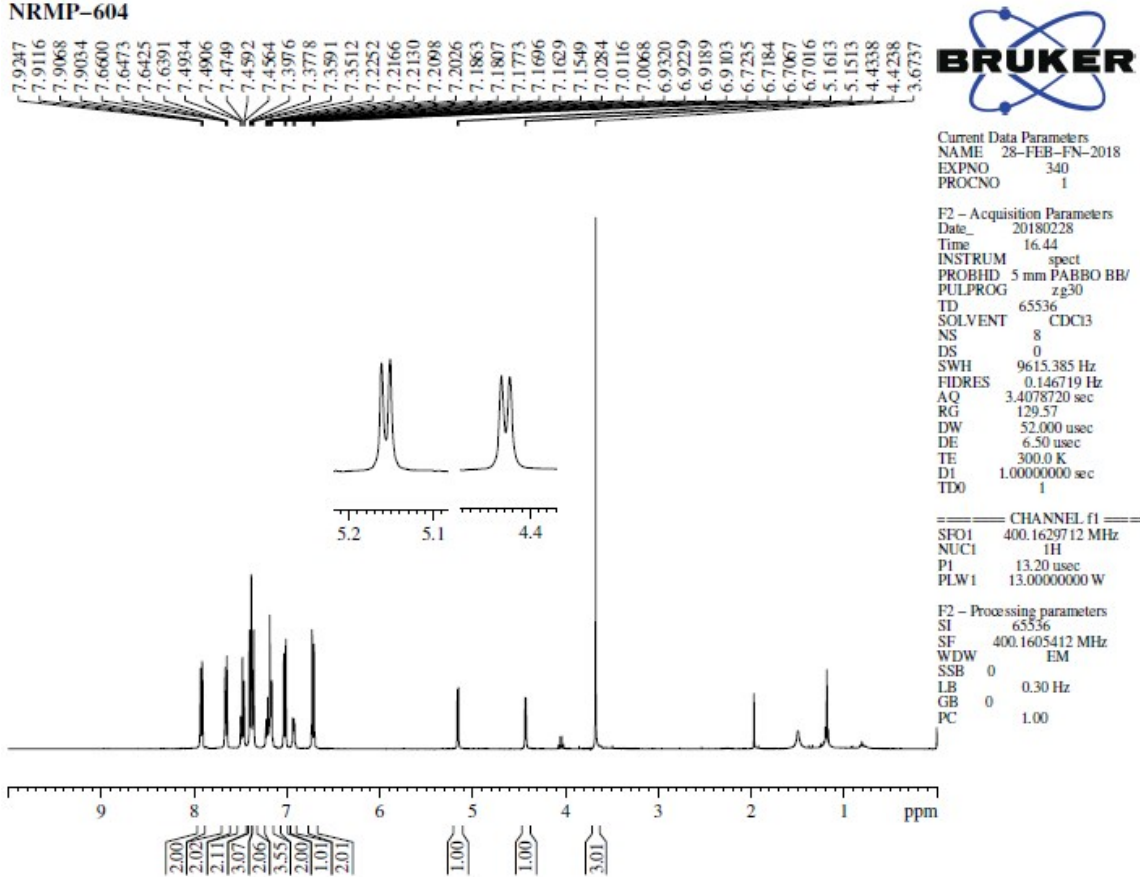


Figure 1: ^1H NMR spectrum of 3a

NRMP-604

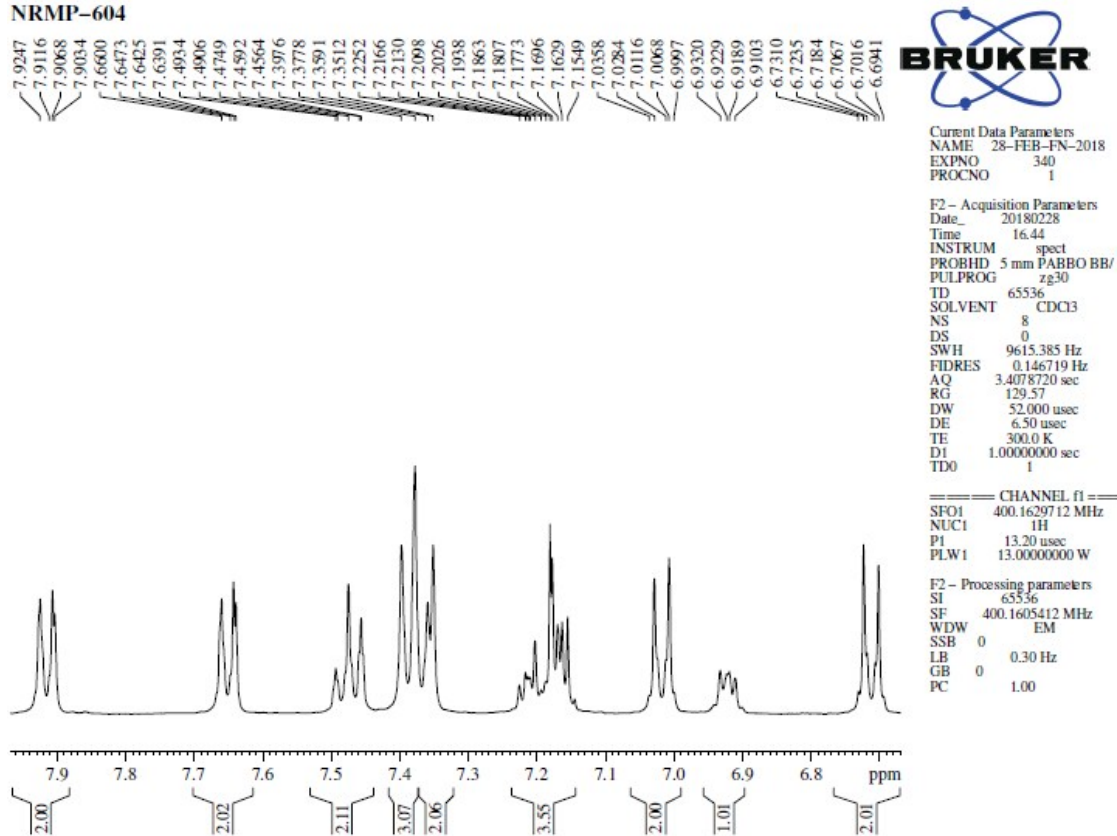


Figure 2: ^1H NMR spectrum of 3a (expansion)

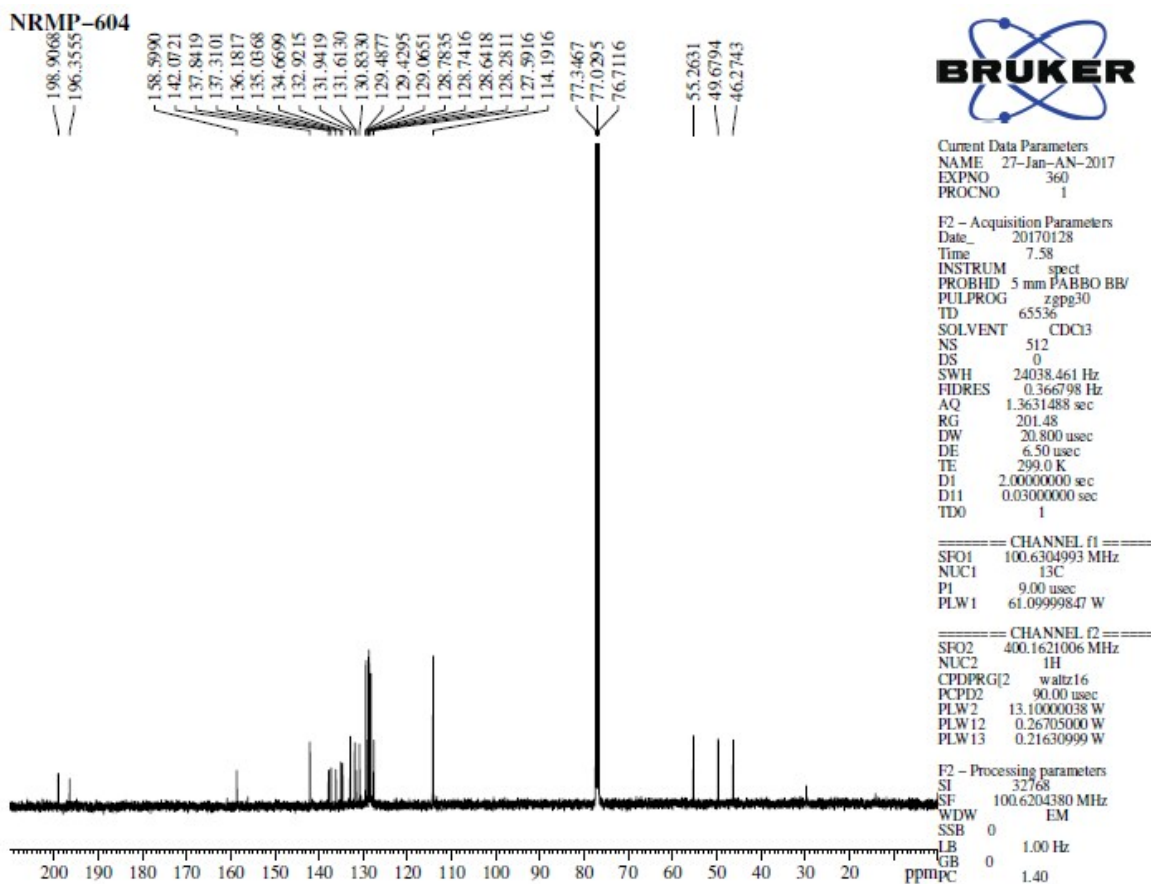


Figure 3: ^{13}C NMR spectrum of 3a

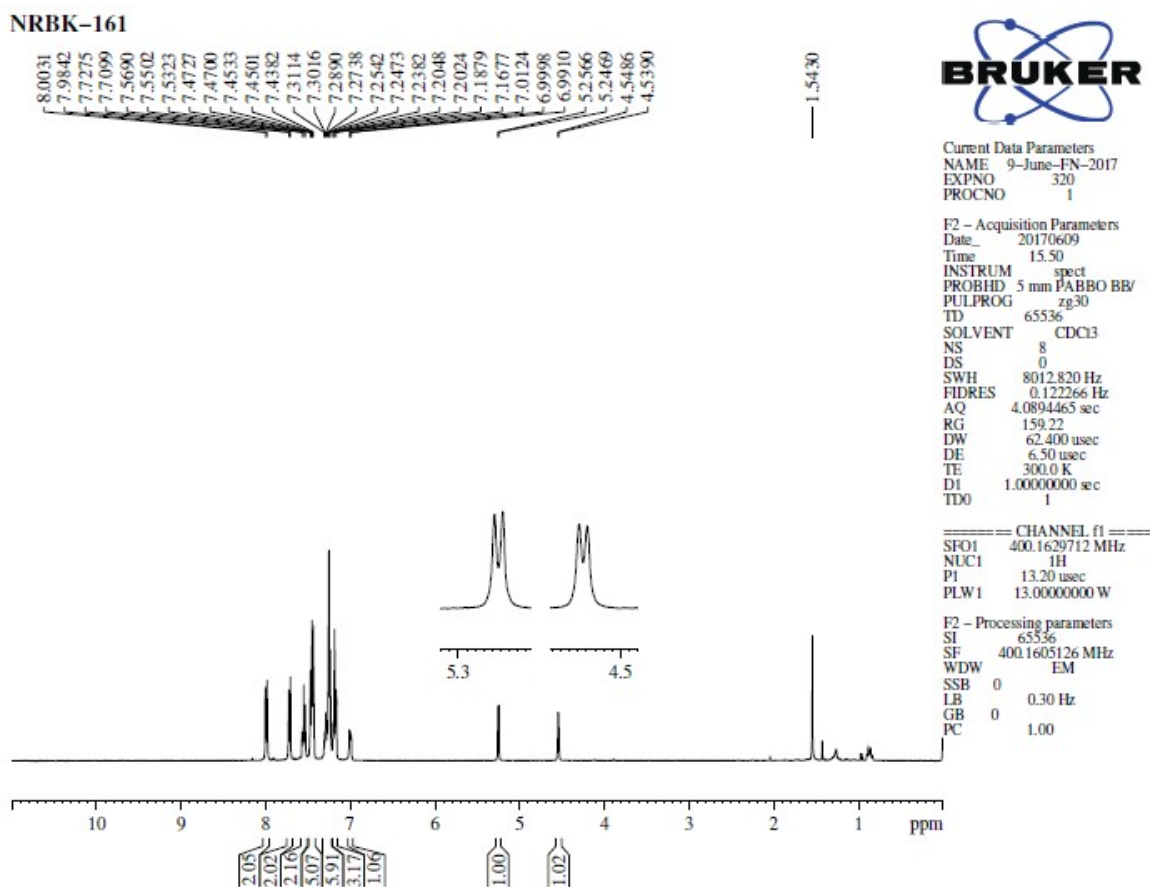
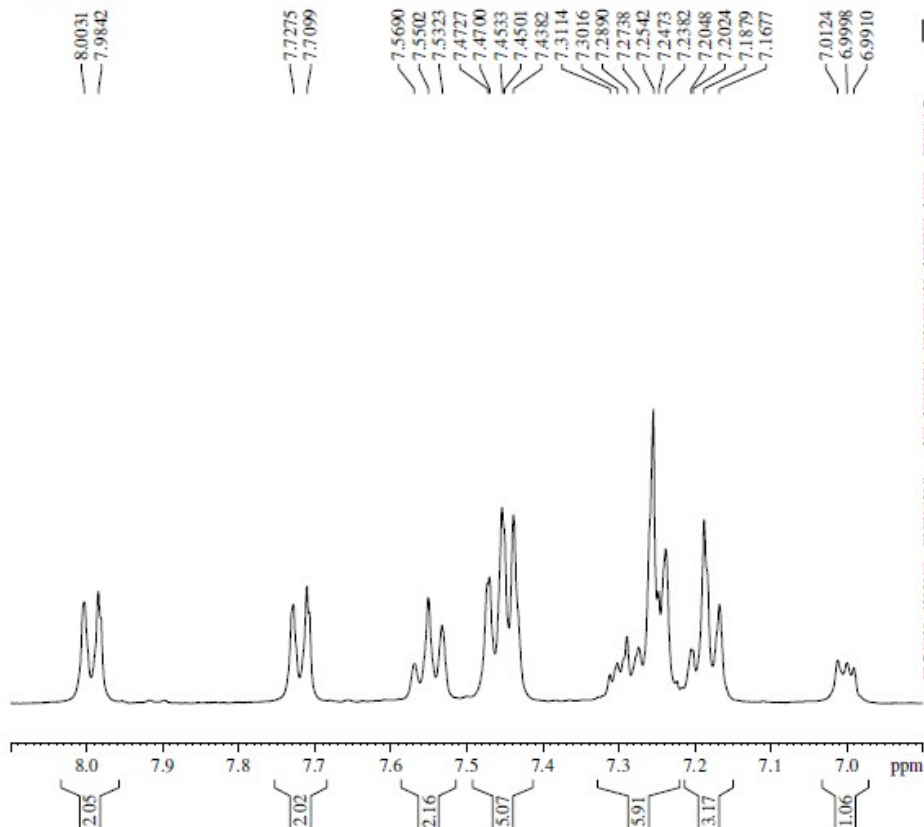


Figure 4: ^1H NMR spectrum of 3b

NRBK-161



Current Data Parameters
 NAME 9-June-FN-2017
 EXPNO 320
 PROCNO 1

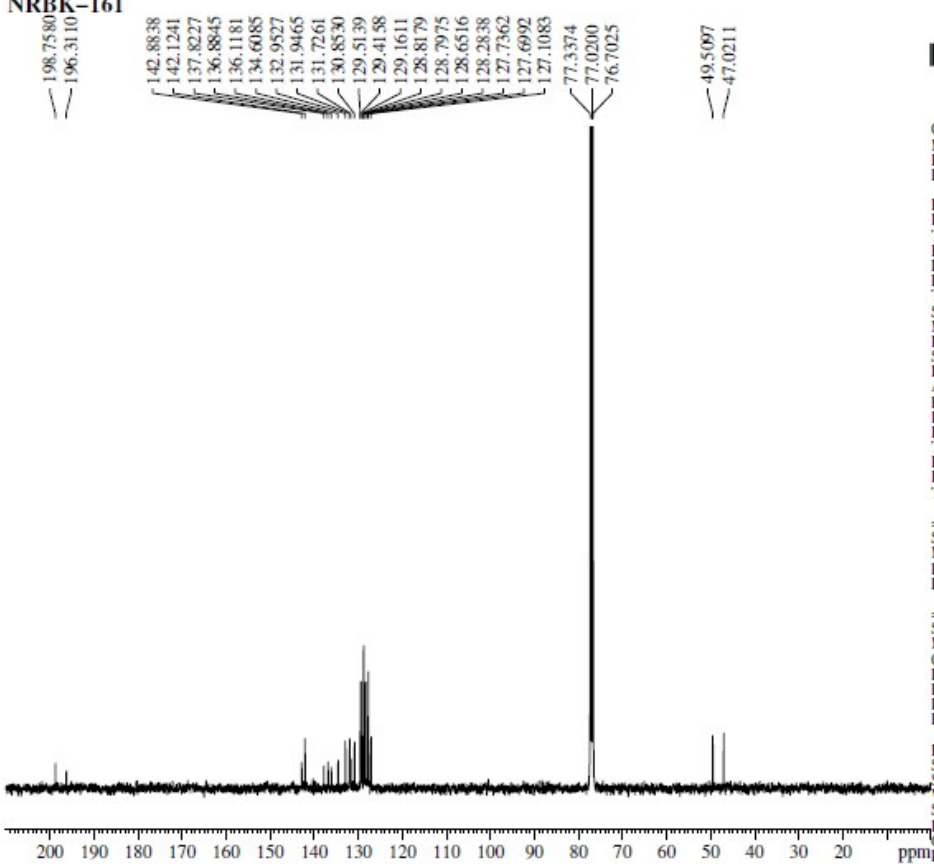
F2 - Acquisition Parameters
 Date_ 20170609
 Time 15.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 159.22
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605126 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 5: ¹H NMR spectrum of 3b (expansion)

NRBK-161



Current Data Parameters
 NAME 12-Jun-FN-2017
 EXPNO 320
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170612
 Time 18.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

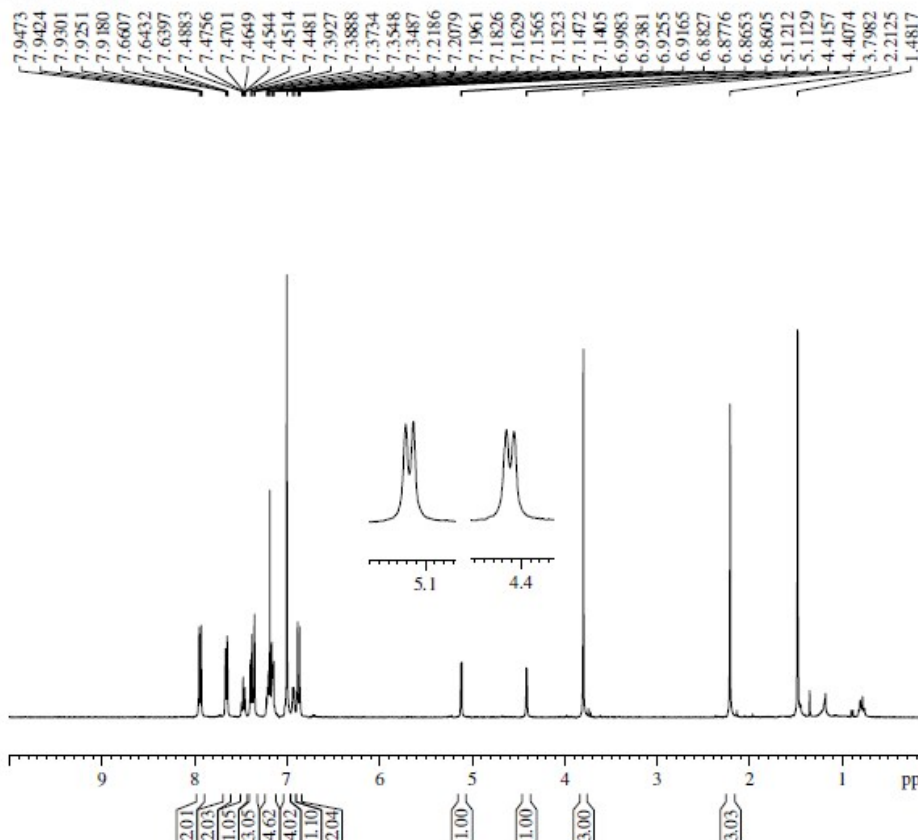
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG12 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

Figure 6: ¹³C NMR spectrum of 3b

NRBK-113



Current Data Parameters
NAME 24-July-AN-2017
EXPNO 340
PROCNO 1

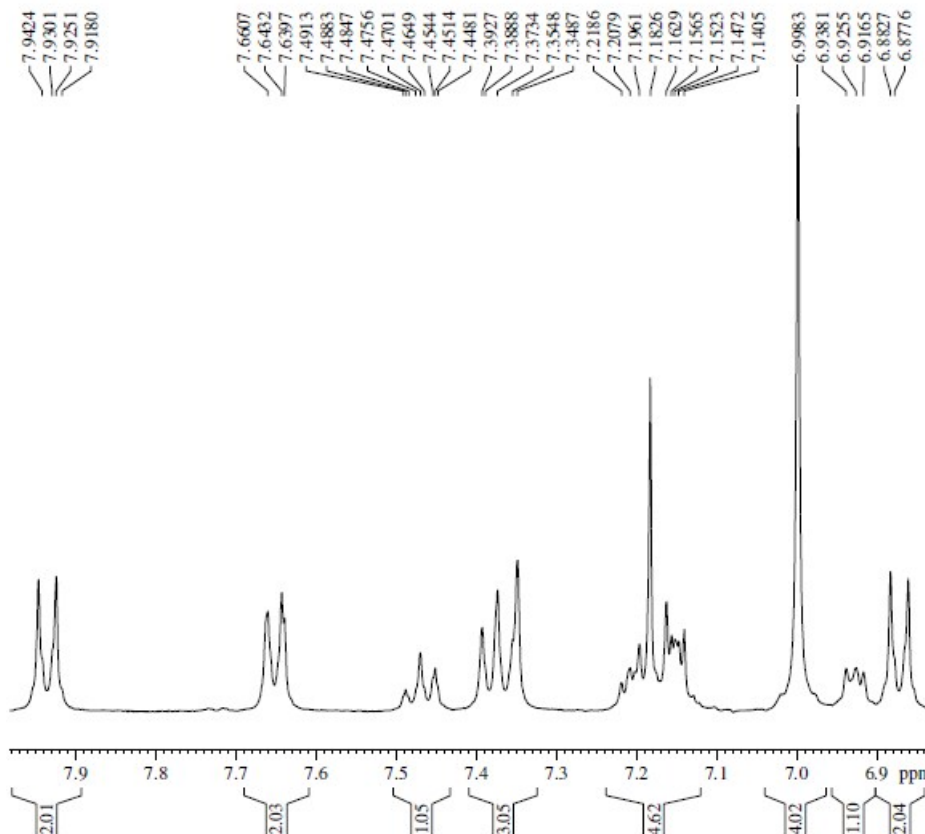
F2 - Acquisition Parameters
Date_ 20170725
Time 7.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 159.22
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605411 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 7: ¹H NMR spectrum of 3c

NRBK-113



Current Data Parameters
NAME 24-July-AN-2017
EXPNO 340
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170725
Time 7.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 159.22
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605411 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 8: ¹H NMR spectrum of 3c (expansion)

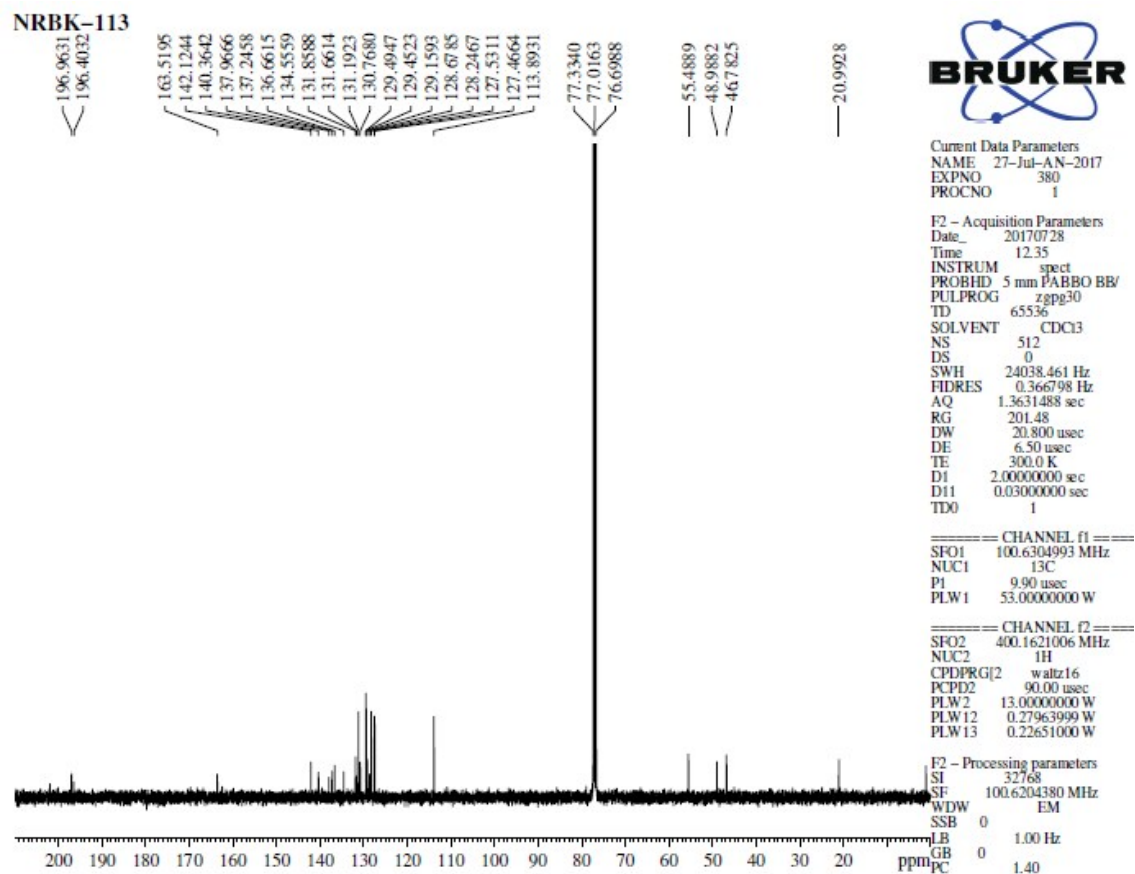


Figure 9: ^{13}C NMR spectrum of 3c

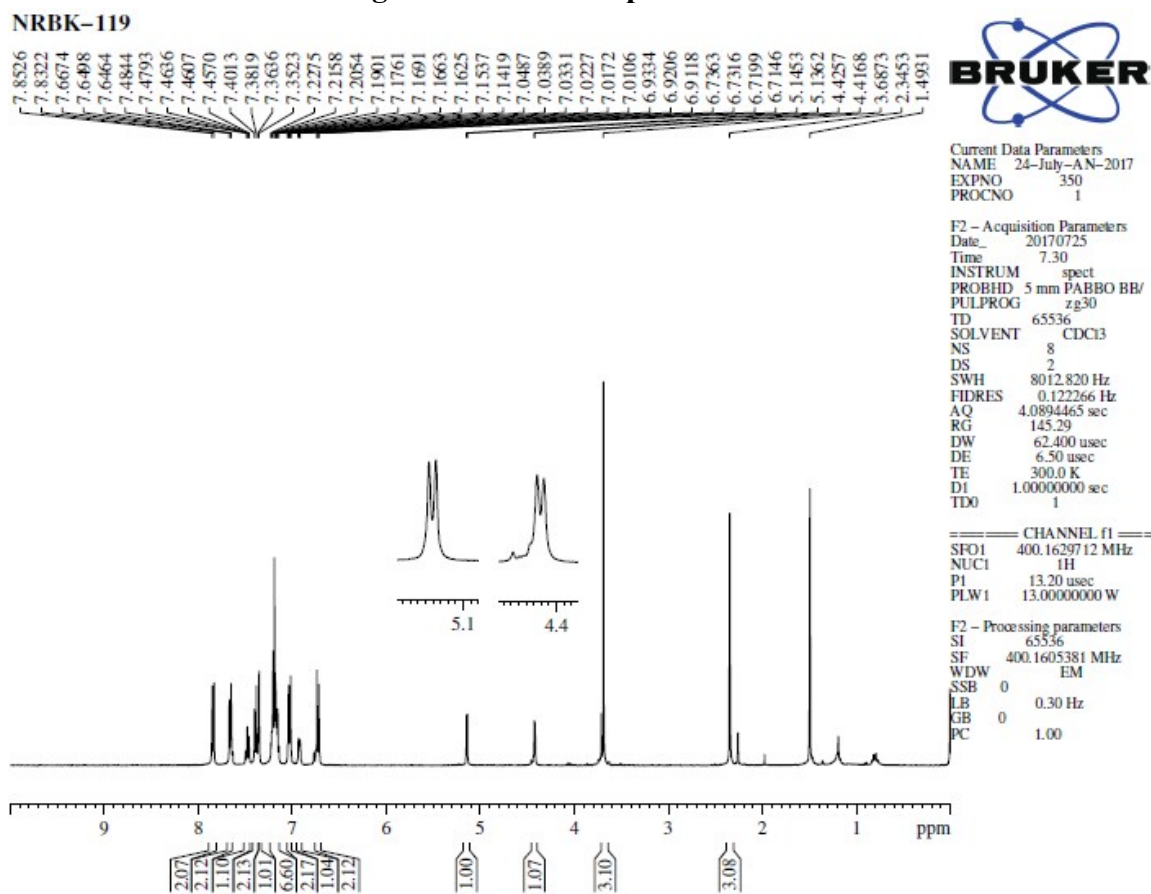
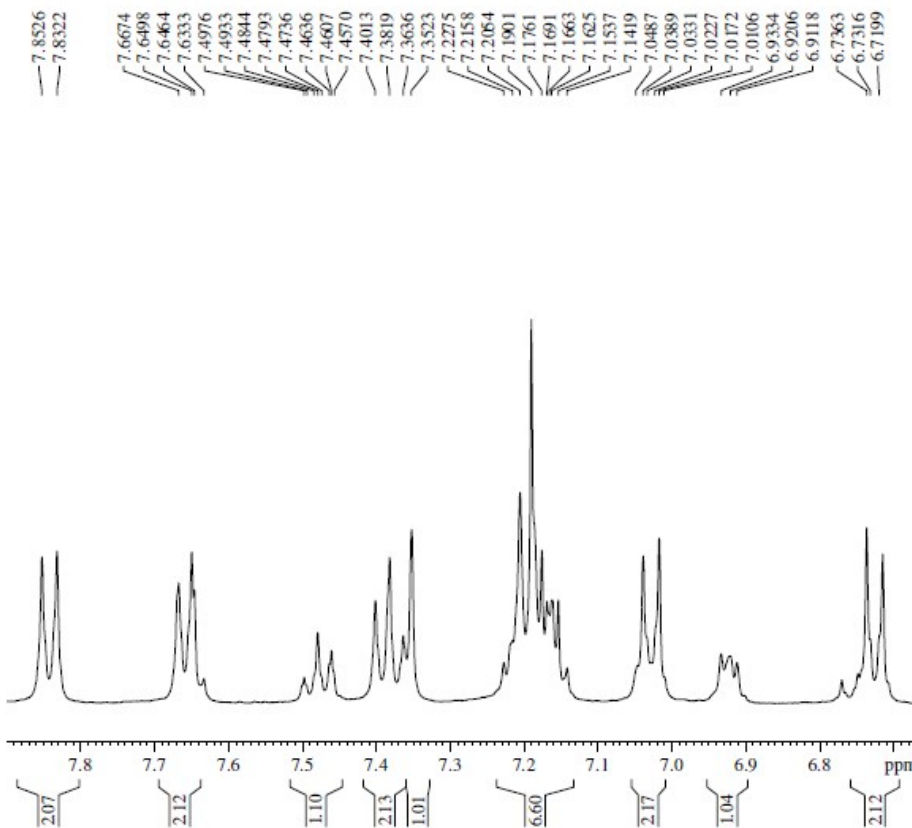


Figure 10: ^1H NMR spectrum of 3d

NRBK-119



Current Data Parameters
 NAME 24-July-AN-2017
 EXPNO 350
 PROCNO 1

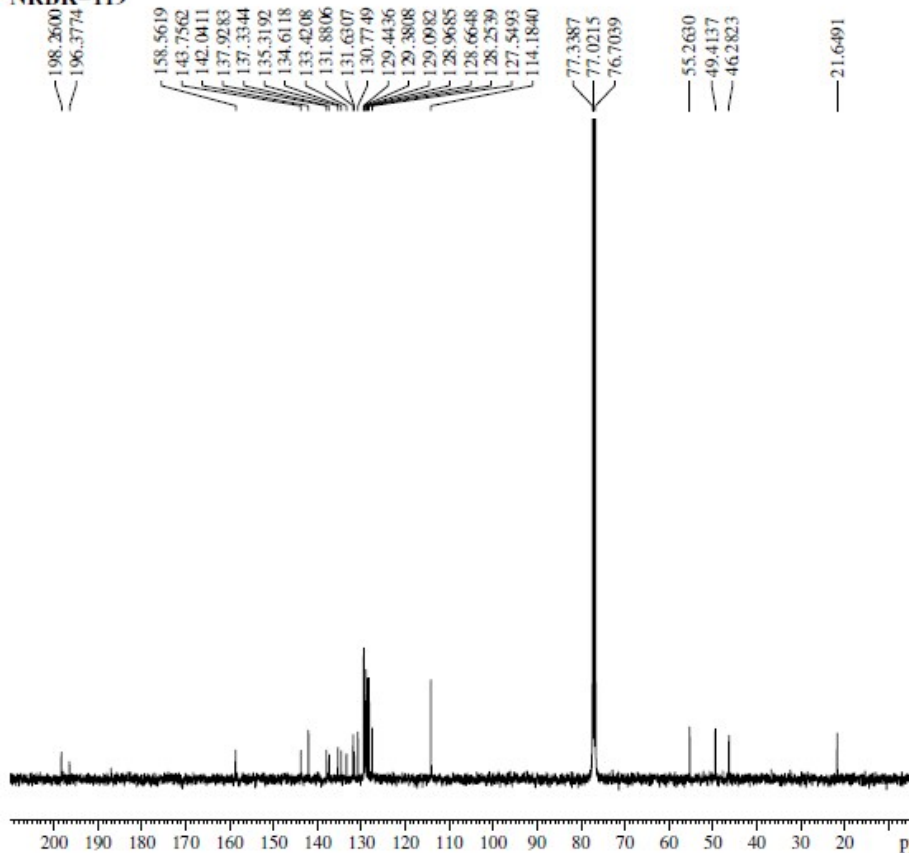
F2 - Acquisition Parameters
 Date_ 20170725
 Time 7.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 2
 SWH 8012.820 Hz
 HDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 145.29
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605381 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 11: ¹H NMR spectrum of 3d (expansion)

NRBK-119



Current Data Parameters
 NAME 27-Jul-AN-2017
 EXPNO 400
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170728
 Time 12.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

Figure 12: ¹³C NMR spectrum of 3d

NRBK-121

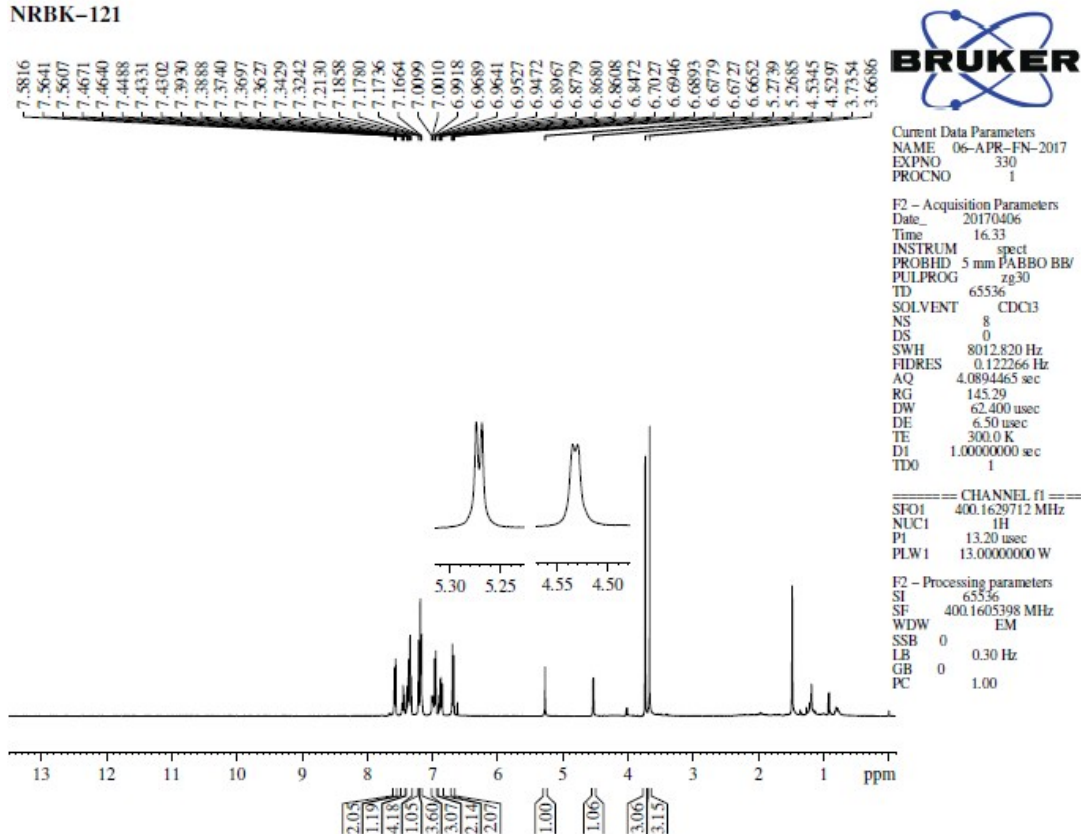


Figure 13: ¹H NMR spectrum of 3e

NRBK-121

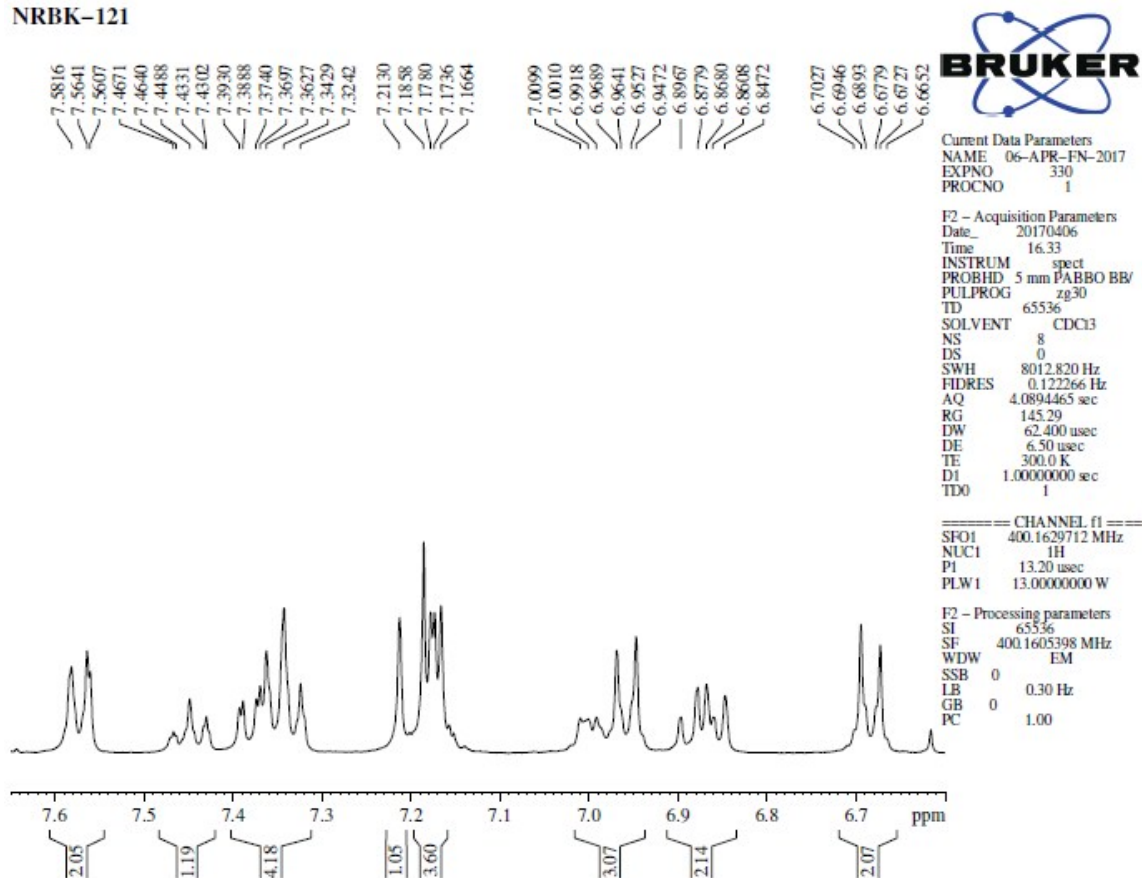


Figure 14: ¹H NMR spectrum of 3e (expansion)

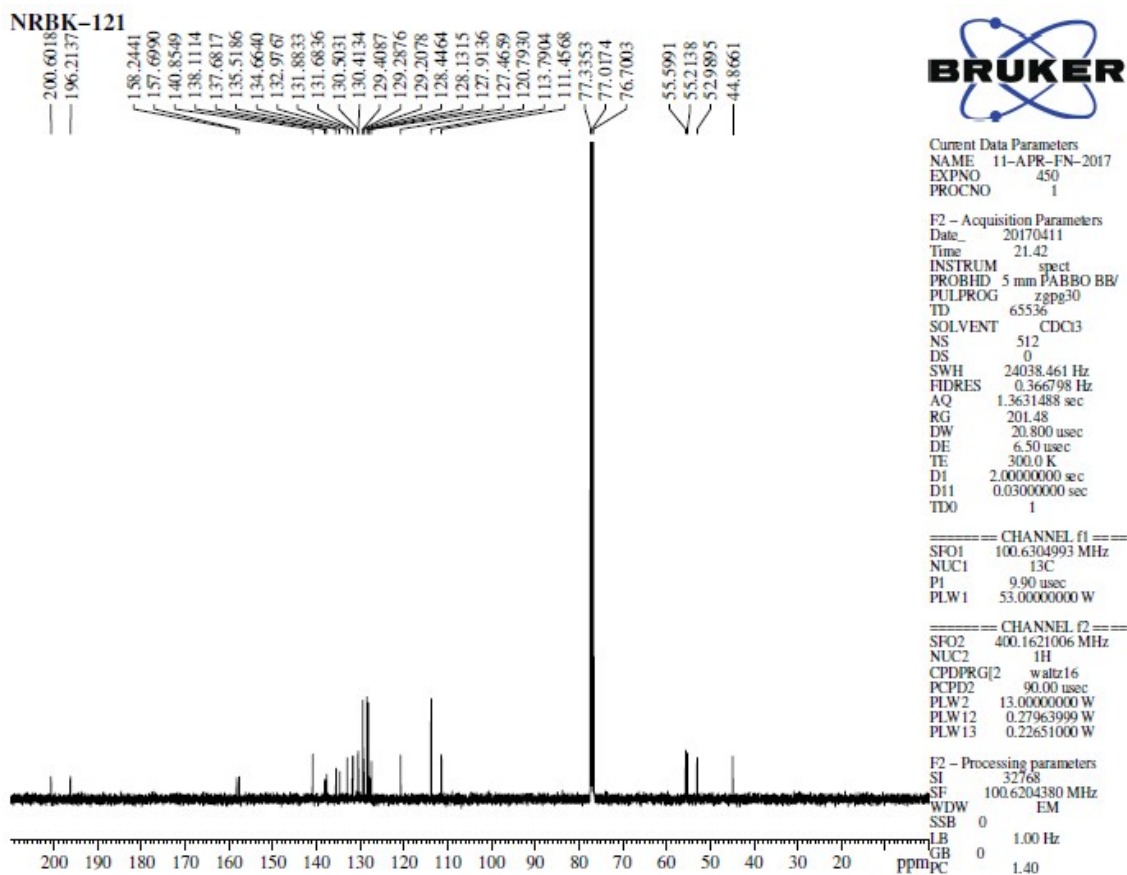


Figure 15: ¹³C NMR spectrum of 3e

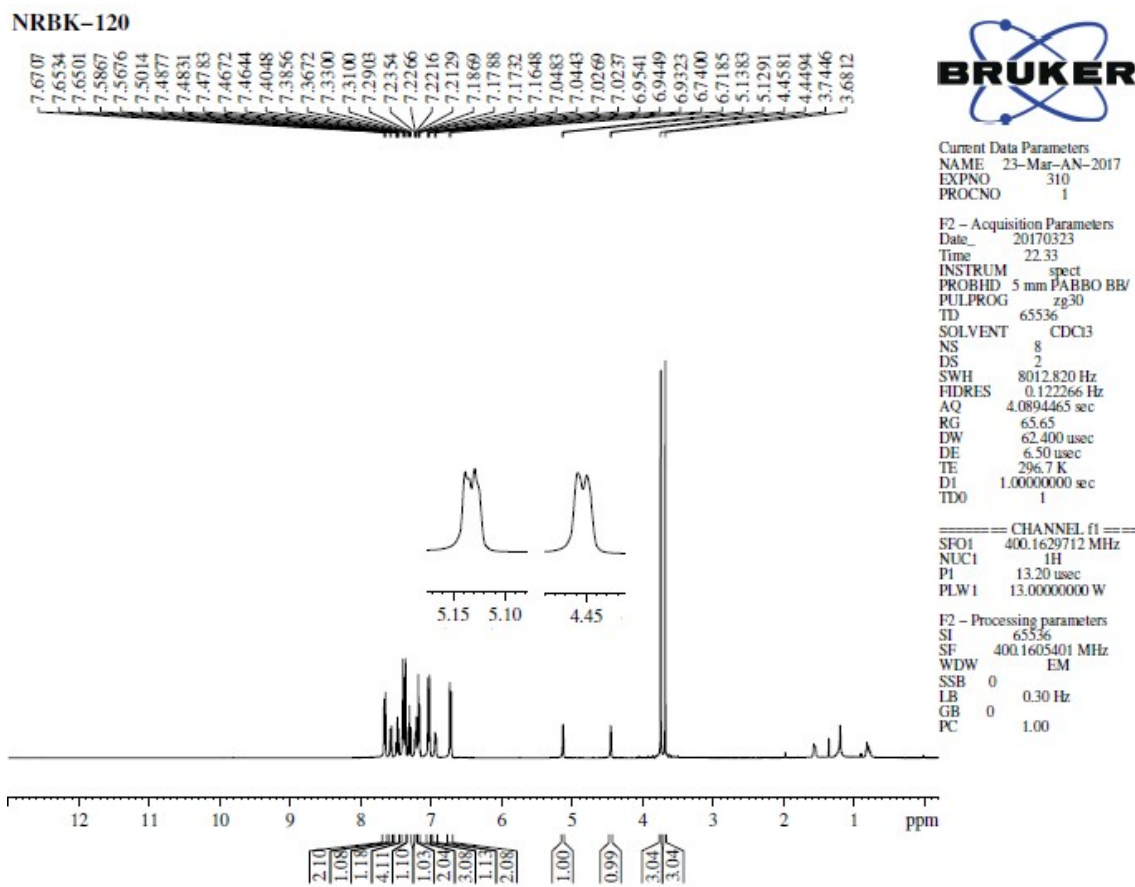


Figure 16: ¹H NMR spectrum of 3f

NRBK-120

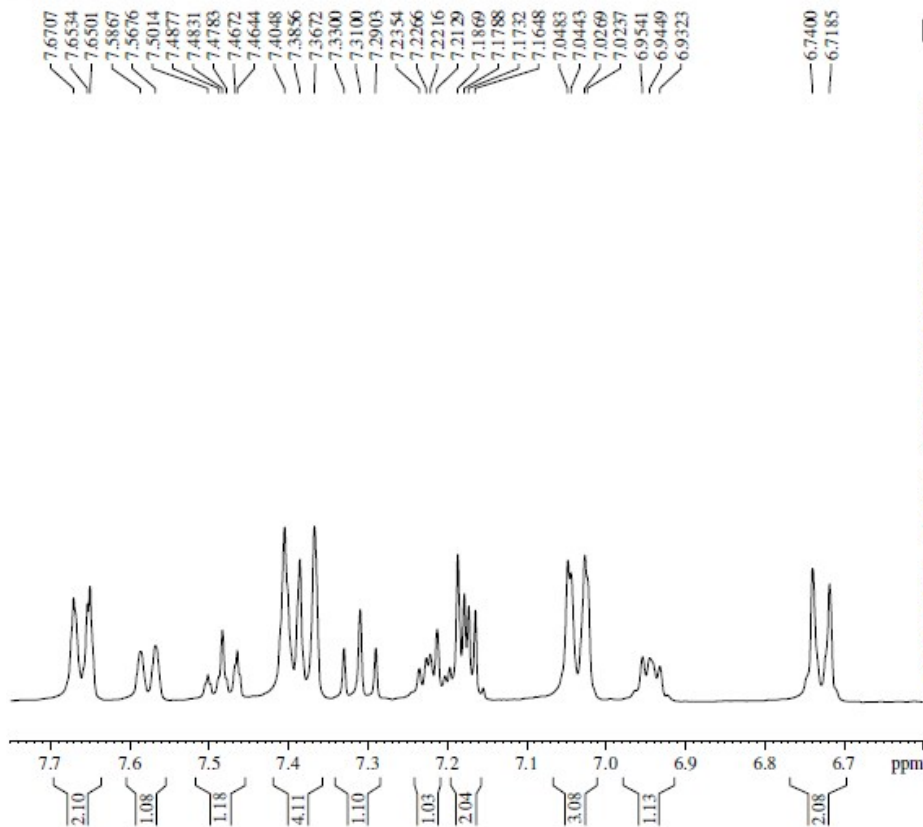


Figure 17: ¹H NMR spectrum of 3f (expansion)



Current Data Parameters
 NAME 23-Mar-AN-2017
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170323
 Time 22.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 65.65
 DW 62.400 usec
 DE 6.50 usec
 TE 296.7 K
 D1 1.0000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605401 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NRBK-120

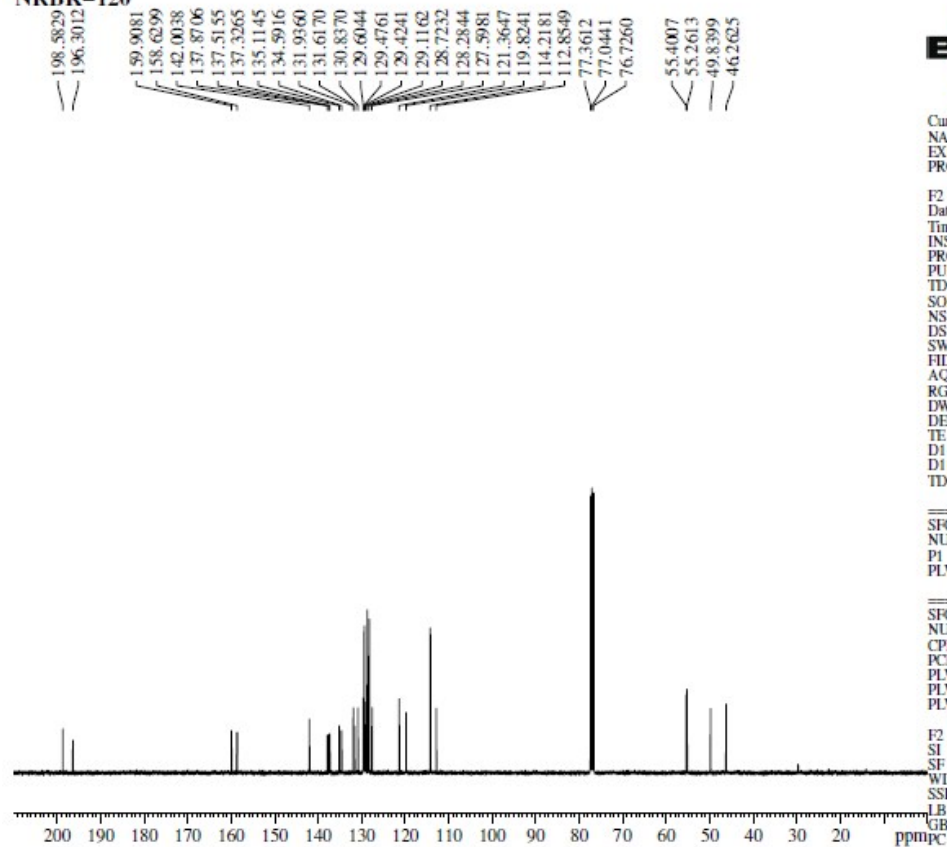


Figure 18: ¹³C NMR spectrum of 3f



Current Data Parameters
 NAME 29-Mar-FN-2017
 EXPNO 340
 PROCNO 1

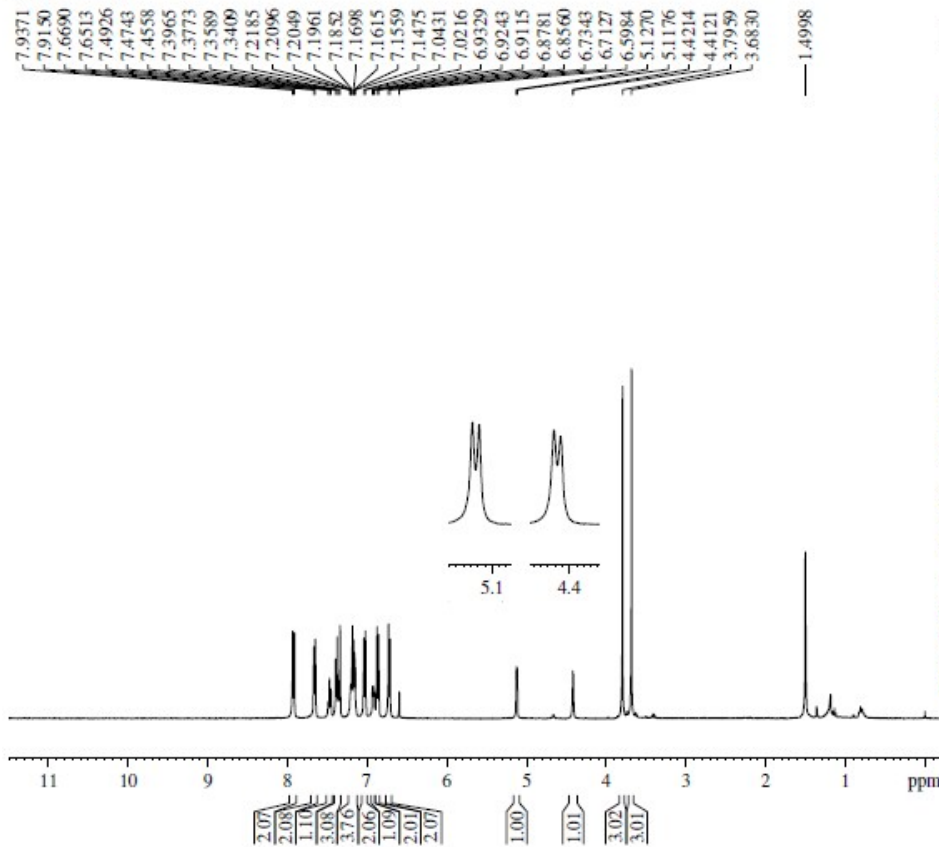
F2 - Acquisition Parameters
 Date_ 20170330
 Time 0.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 299.5 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRBK-114



Current Data Parameters
 NAME 06-APR-FN-2017
 EXPNO 320
 PROCNO 1

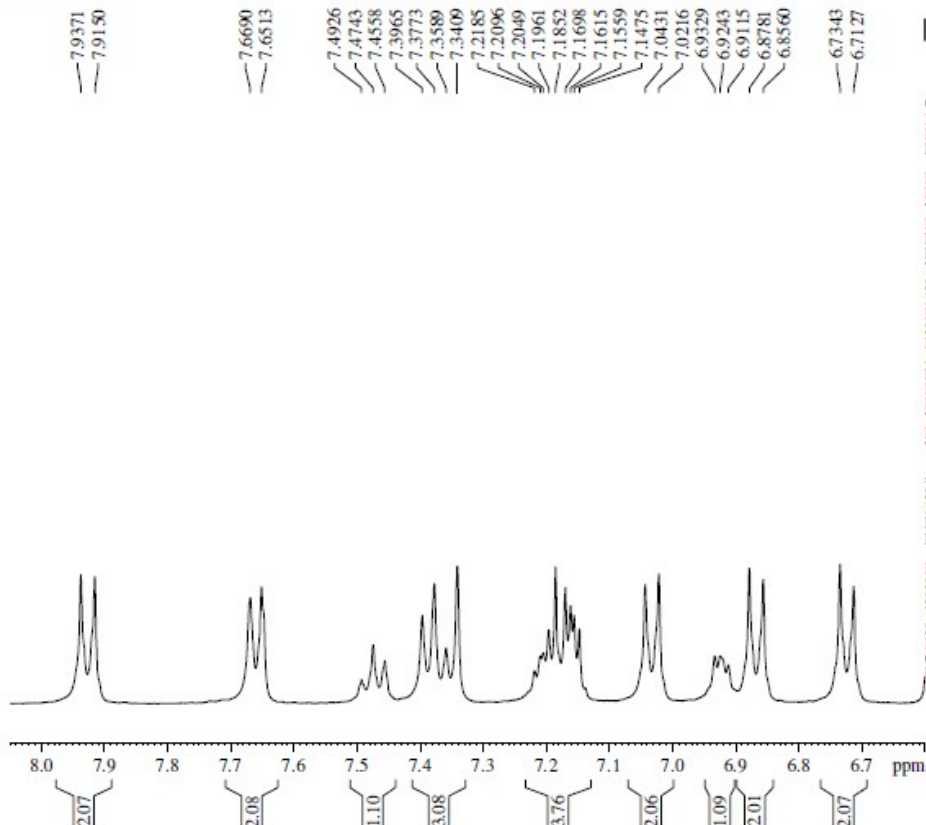
F2 - Acquisition Parameters
 Date_ 20170406
 Time 16.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 145.29
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 PI 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605401 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 19: ¹H NMR spectrum of 3g

NRBK-114



Current Data Parameters
 NAME 06-APR-FN-2017
 EXPNO 320
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170406
 Time 16.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 145.29
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 PI 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605401 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 20: ¹H NMR spectrum of 3g (expansion)

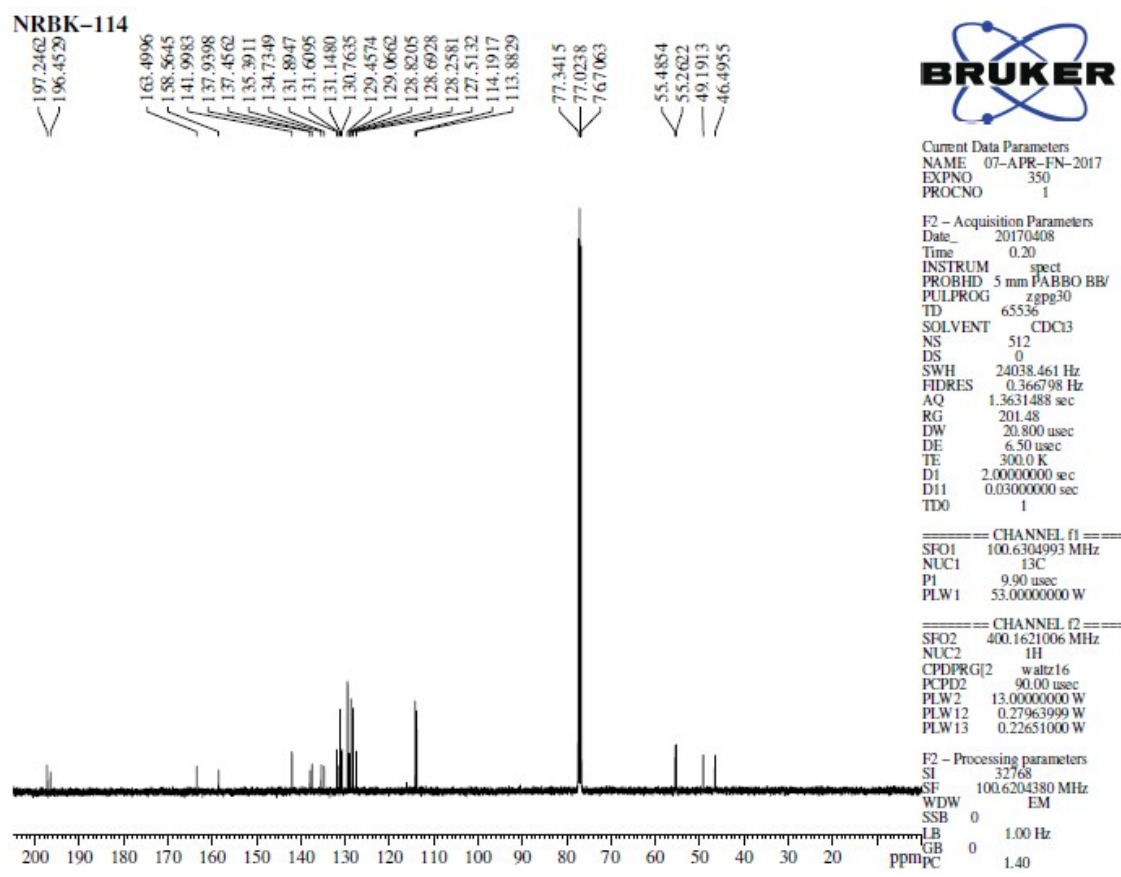


Figure 21: ¹³C NMR spectrum of 3g

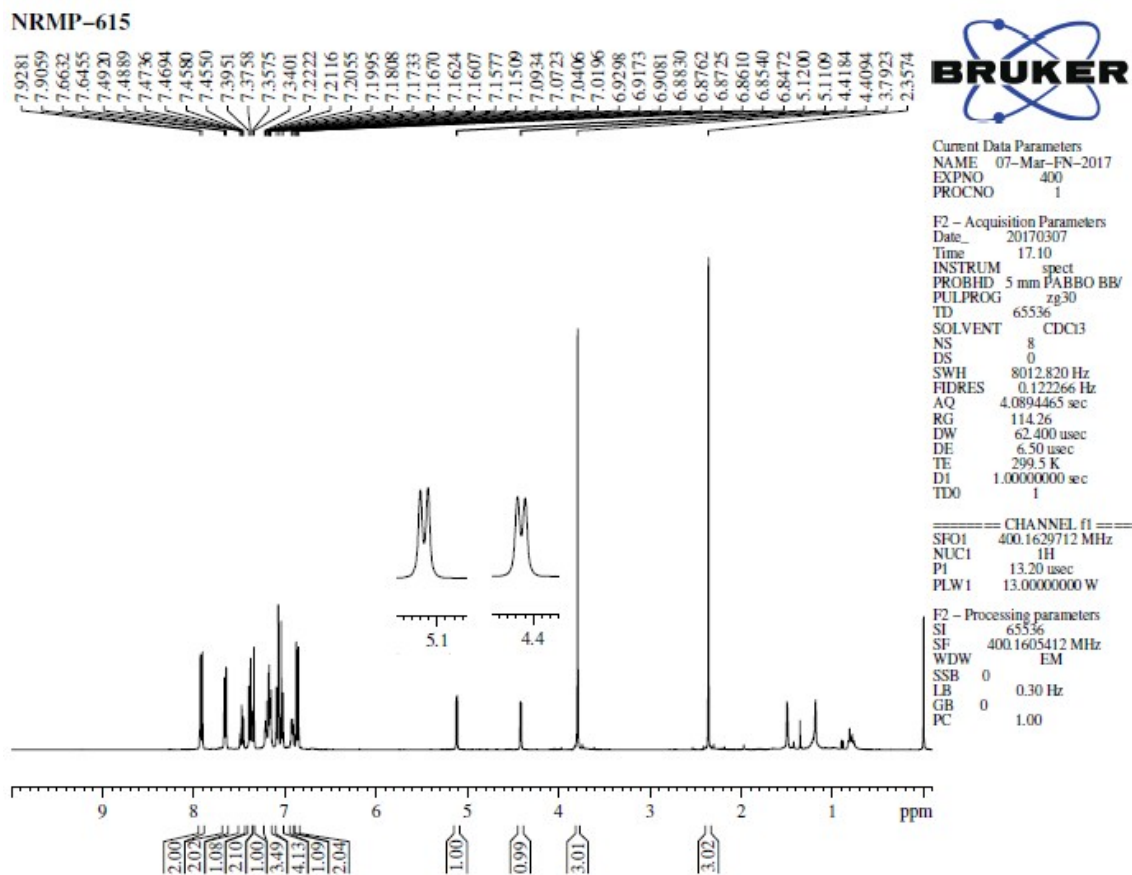
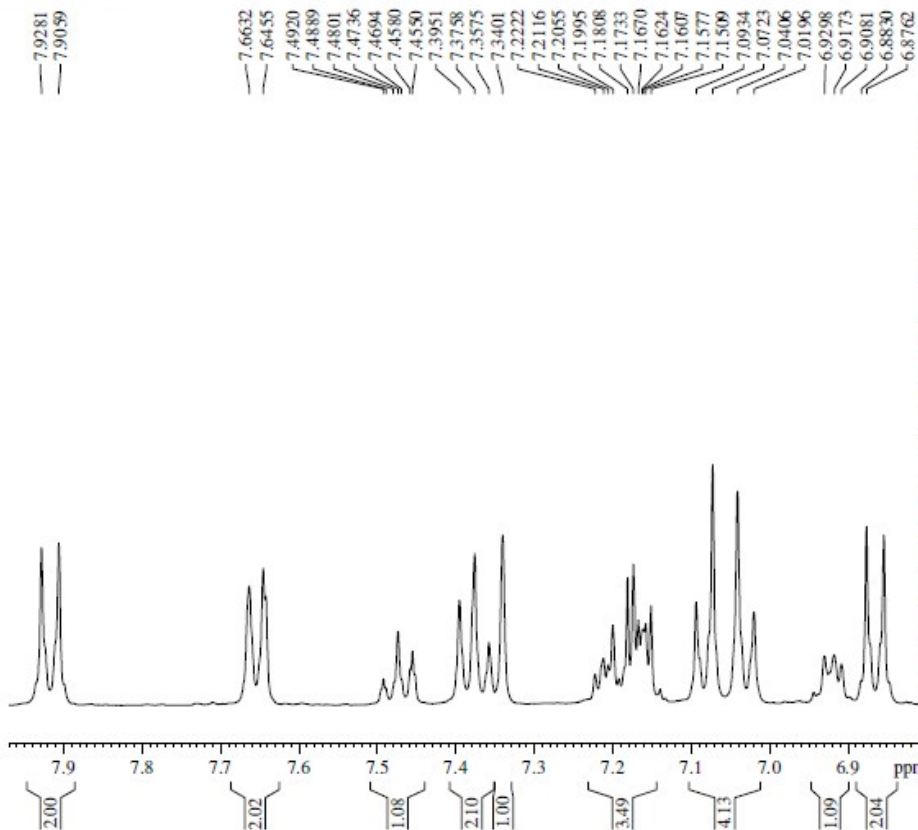


Figure 22: ¹H NMR spectrum of 3h

NRMP-615



Current Data Parameters
 NAME 07-Mar-FN-2017
 EXPNO 400
 PROCNO 1

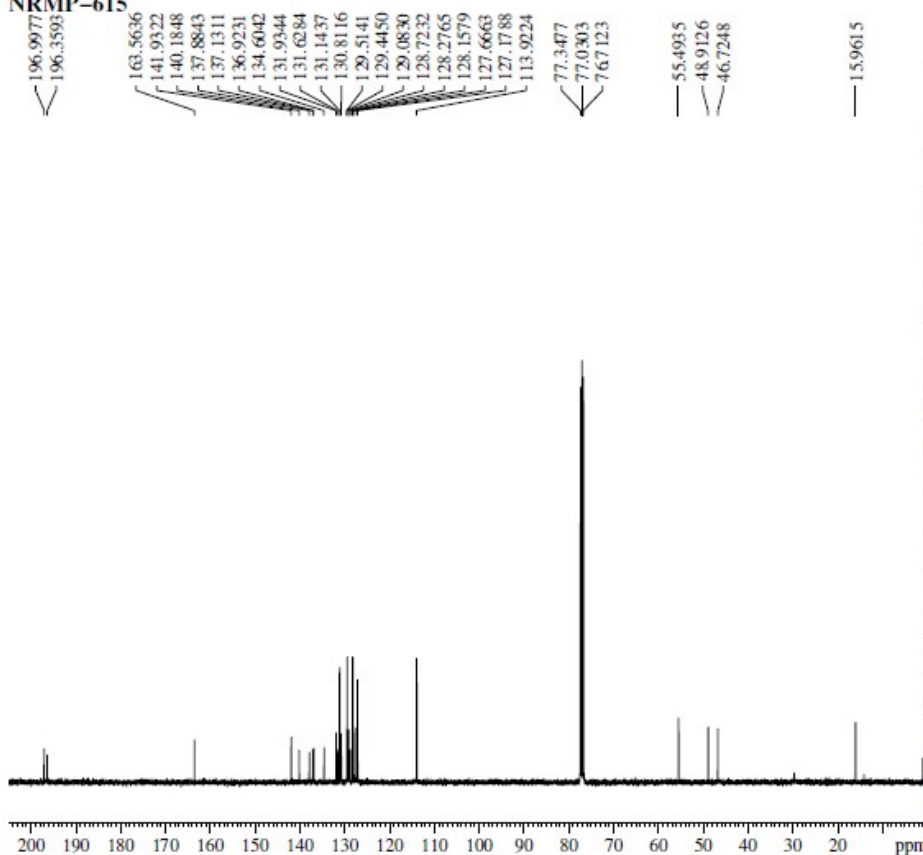
F2 - Acquisition Parameters
 Date_ 20170307
 Time 17.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 114.26
 DW 62.400 usec
 DE 6.50 usec
 TE 299.5 K
 D1 1.0000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605412 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 23: ¹H NMR spectrum of 3h (expansion)

NRMP-615



Current Data Parameters
 NAME 24-Mar-AN-2017
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170325
 Time 18.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

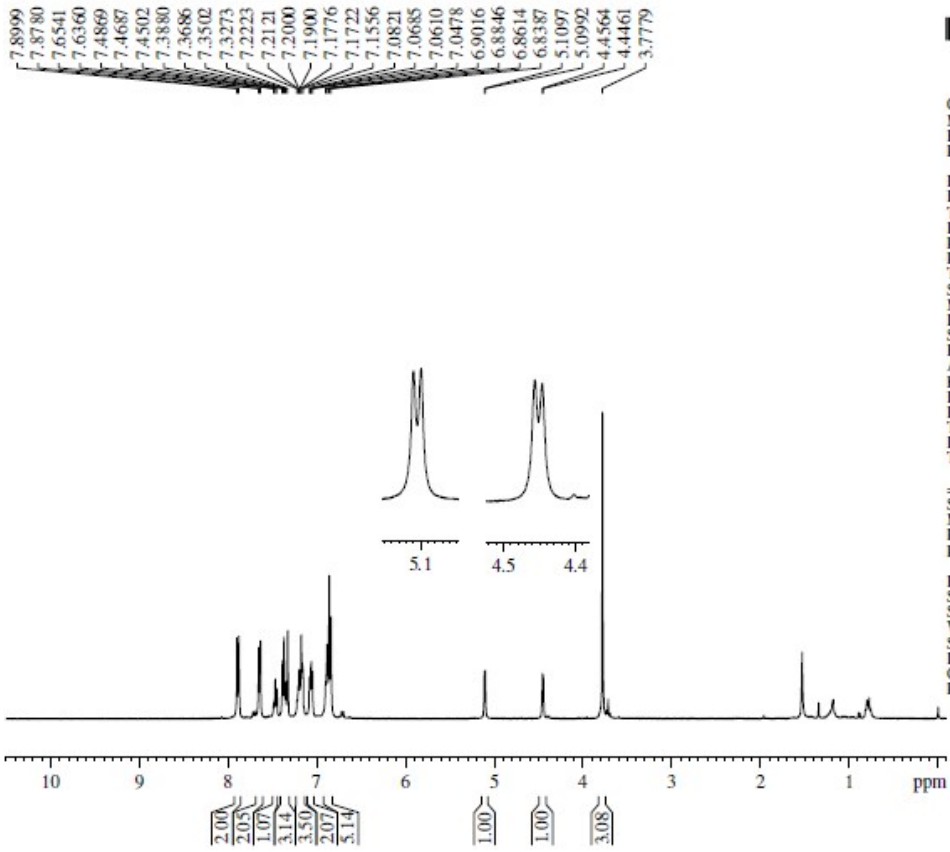
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 24: ¹³C NMR spectrum of 3h

NRBK-112



Current Data Parameters
NAME 24-Mar-FN 2017
EXPNO 310
PROCNO 1

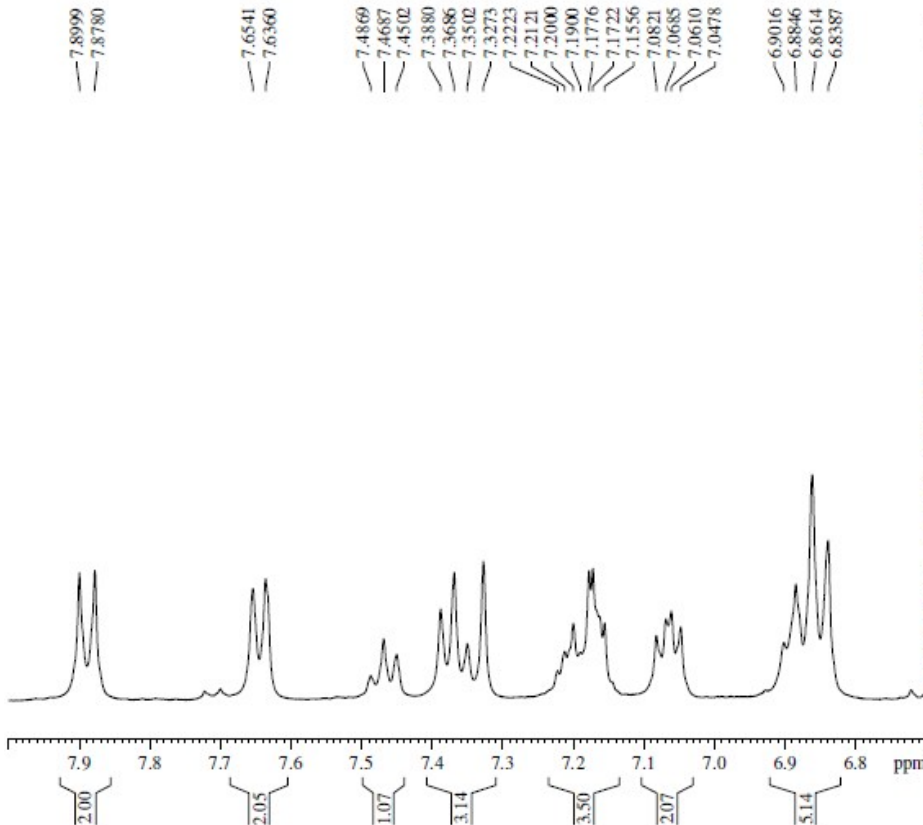
F2 - Acquisition Parameters
Date_ 20170324
Time 15.33
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 89.7
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605455 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

Figure 25: ¹H NMR spectrum of 3i

NRBK-112



Current Data Parameters
NAME 24-Mar-FN 2017
EXPNO 310
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170324
Time 15.33
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 89.7
DW 62.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605455 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

Figure 26: ¹H NMR spectrum of 3i (expansion)

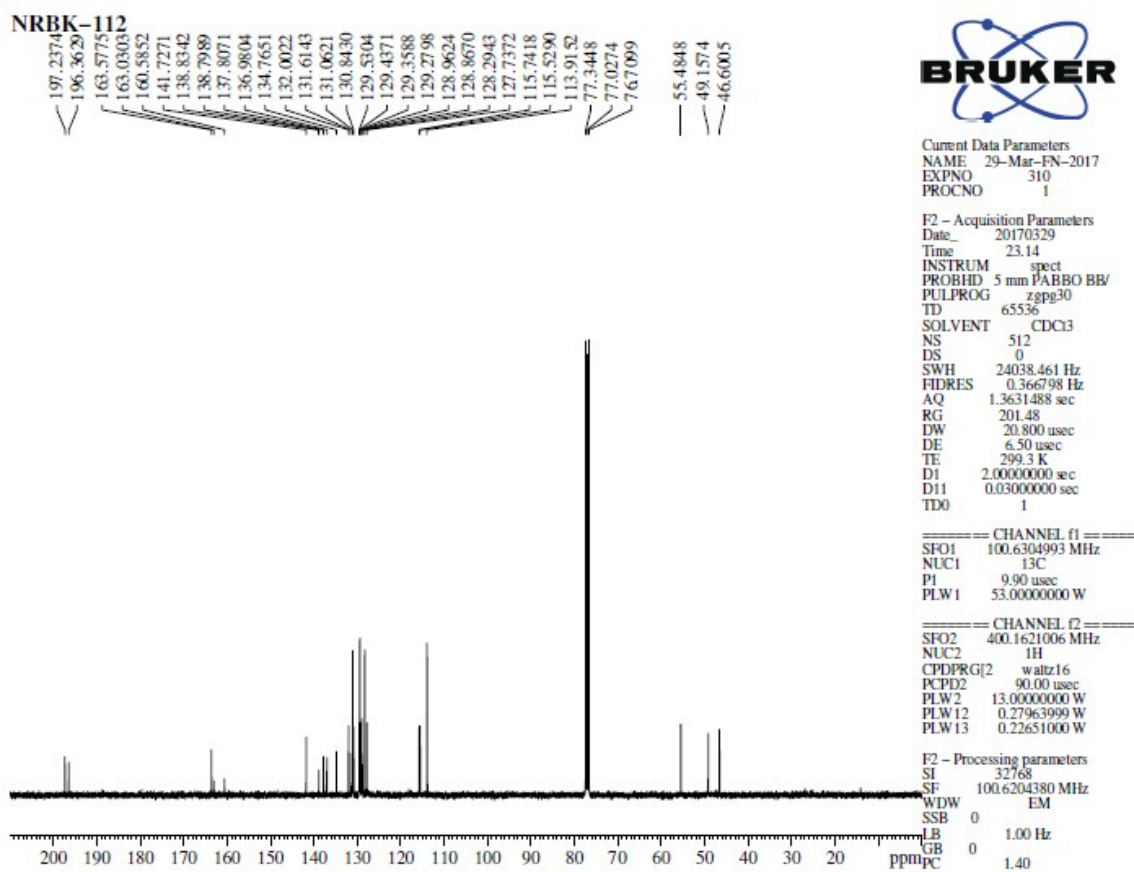


Figure 27: ^{13}C NMR spectrum of 3i

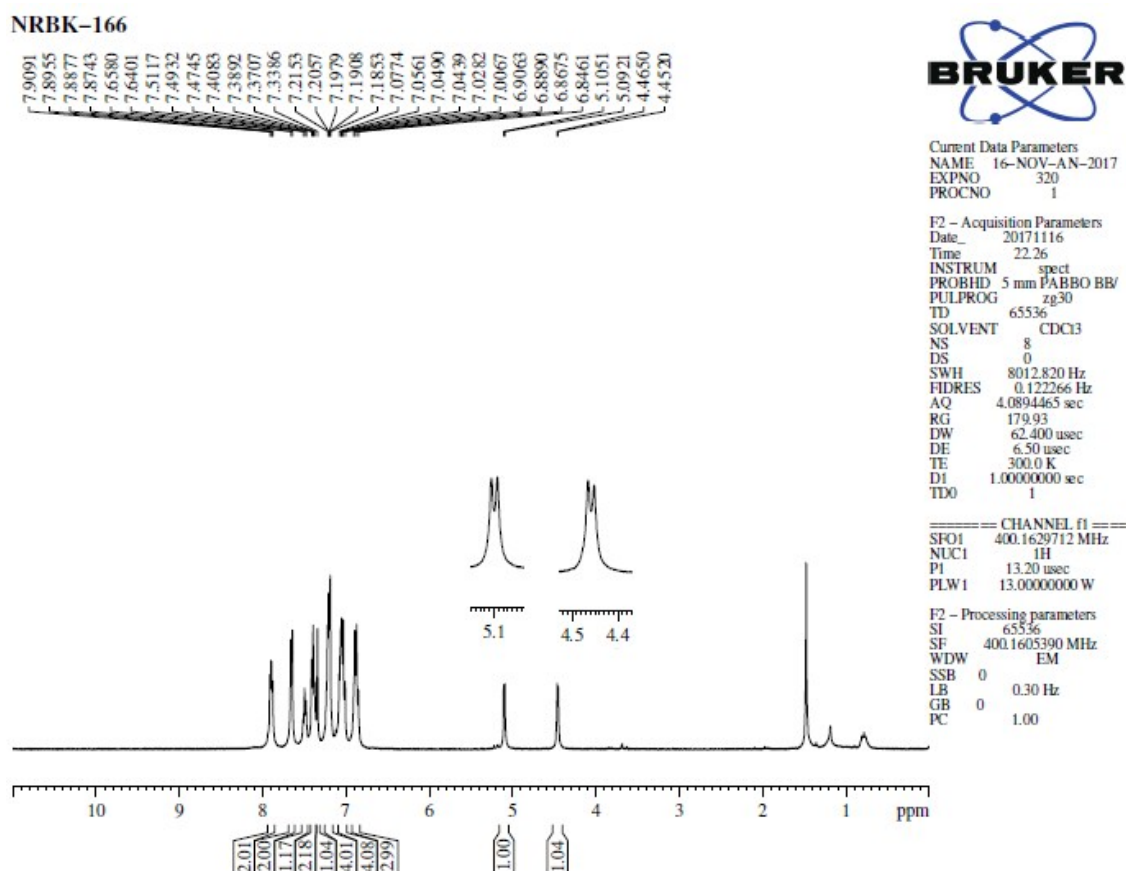
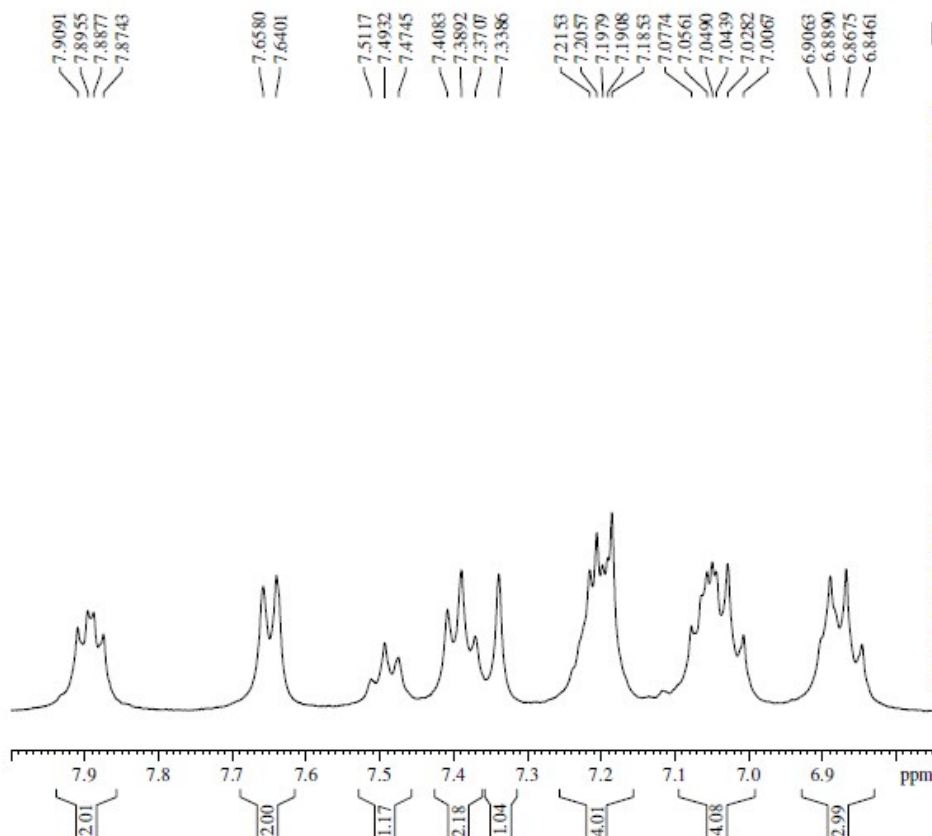


Figure 28: ^1H NMR spectrum of 3j

NRBK-166



Current Data Parameters
 NAME 16-NOV-AN-2017
 EXPNO 320
 PROCNO 1

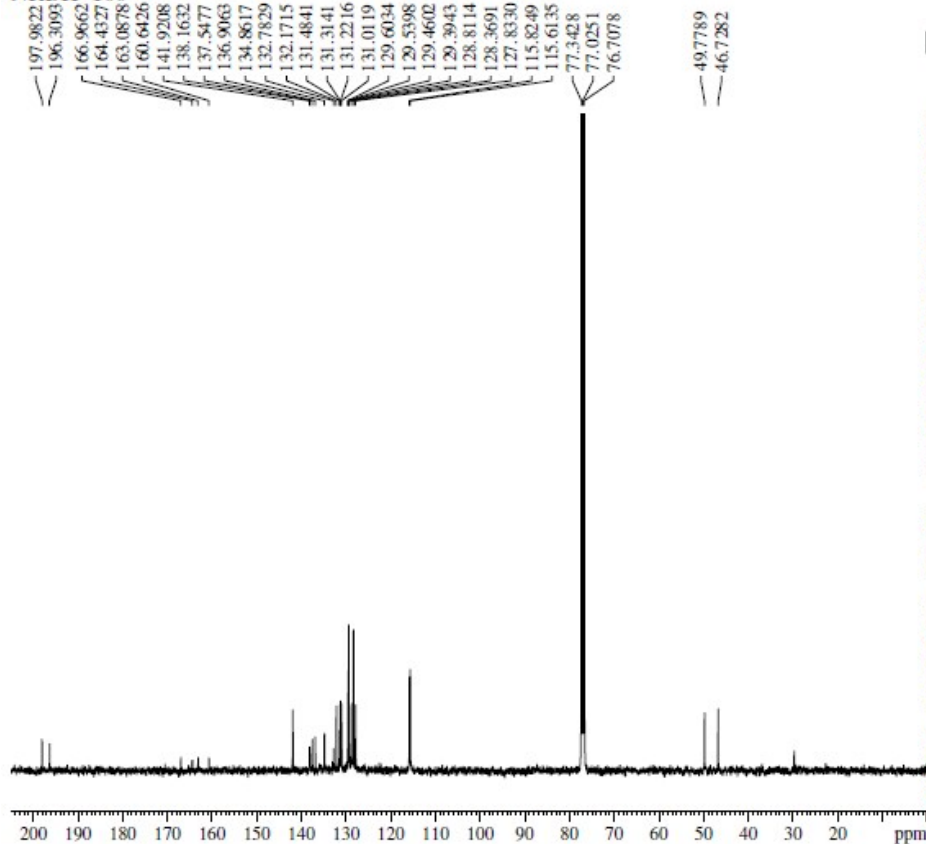
F2 - Acquisition Parameters
 Date_ 20171116
 Time 22.26
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 179.93
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605390 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 29: ¹H NMR spectrum of 3j (expansion)

NRBK-166



Current Data Parameters
 NAME 11-Aug-AN-2017
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170811
 Time 19.27
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.5631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

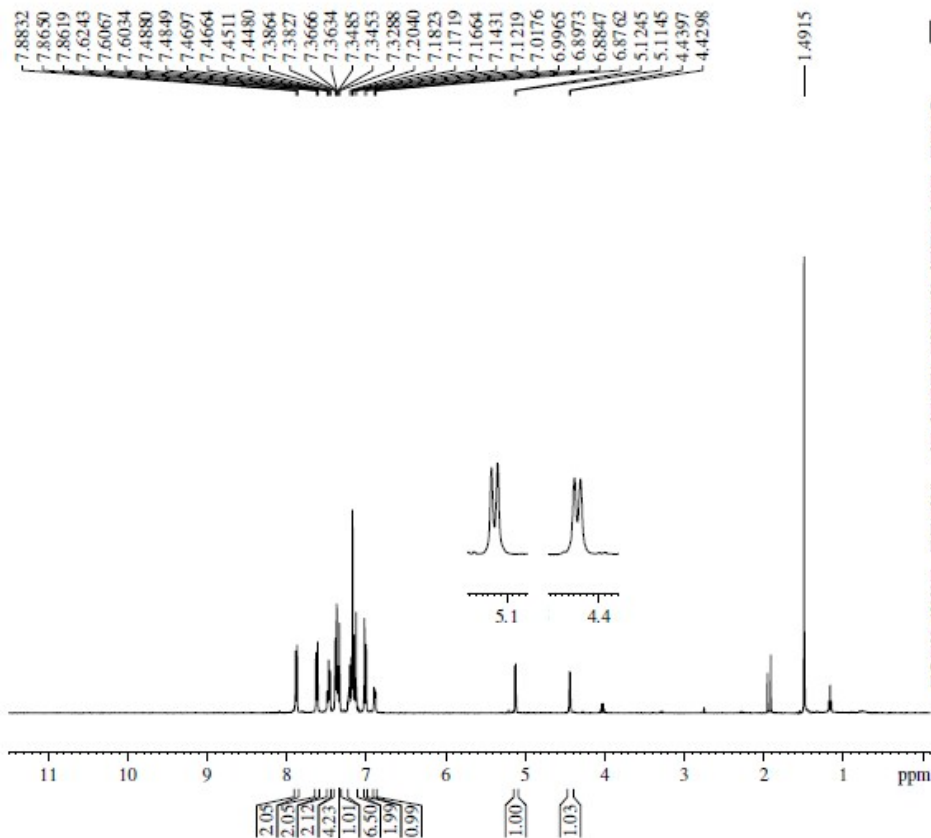
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

Figure 30: ¹³C NMR spectrum of 3j

NRBK-145



Current Data Parameters
 NAME 15-May-FN-2017
 EXPNO 330
 PROCNO 1

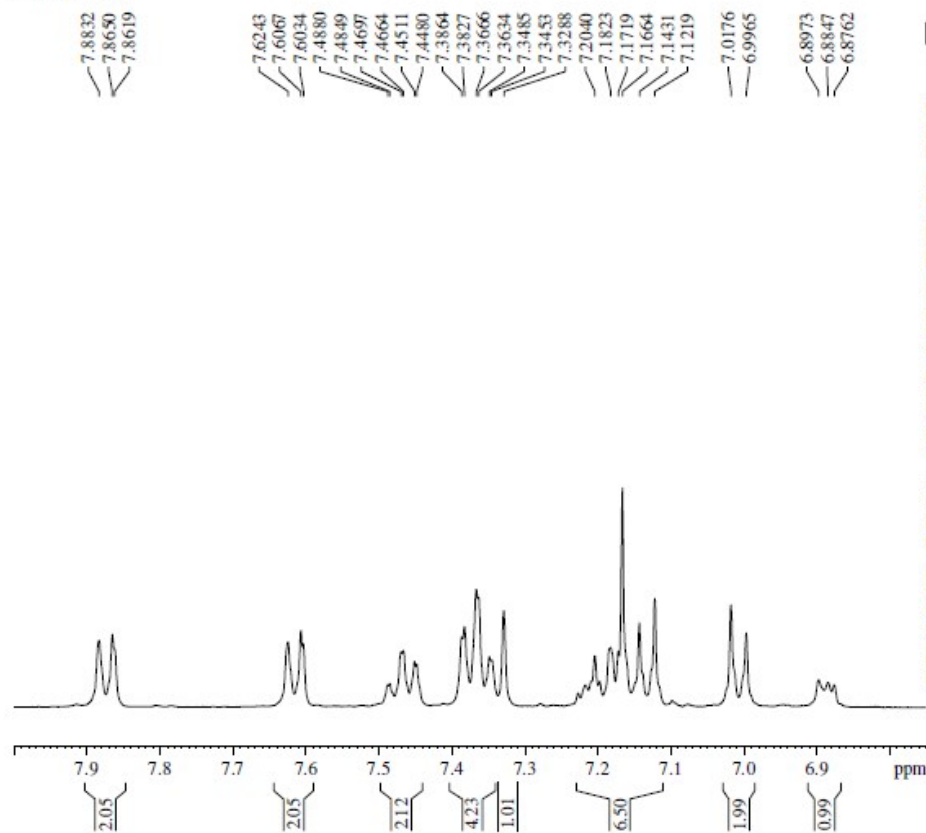
F2 - Acquisition Parameters
 Date_ 20170515
 Time 15.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 159.22
 DW 62.400 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605477 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 31: ¹H NMR spectrum of 3k

NRBK-145



Current Data Parameters
 NAME 15-May-FN-2017
 EXPNO 330
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170515
 Time 15.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 159.22
 DW 62.400 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605477 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 32: ¹H NMR spectrum of 3k (expansion)

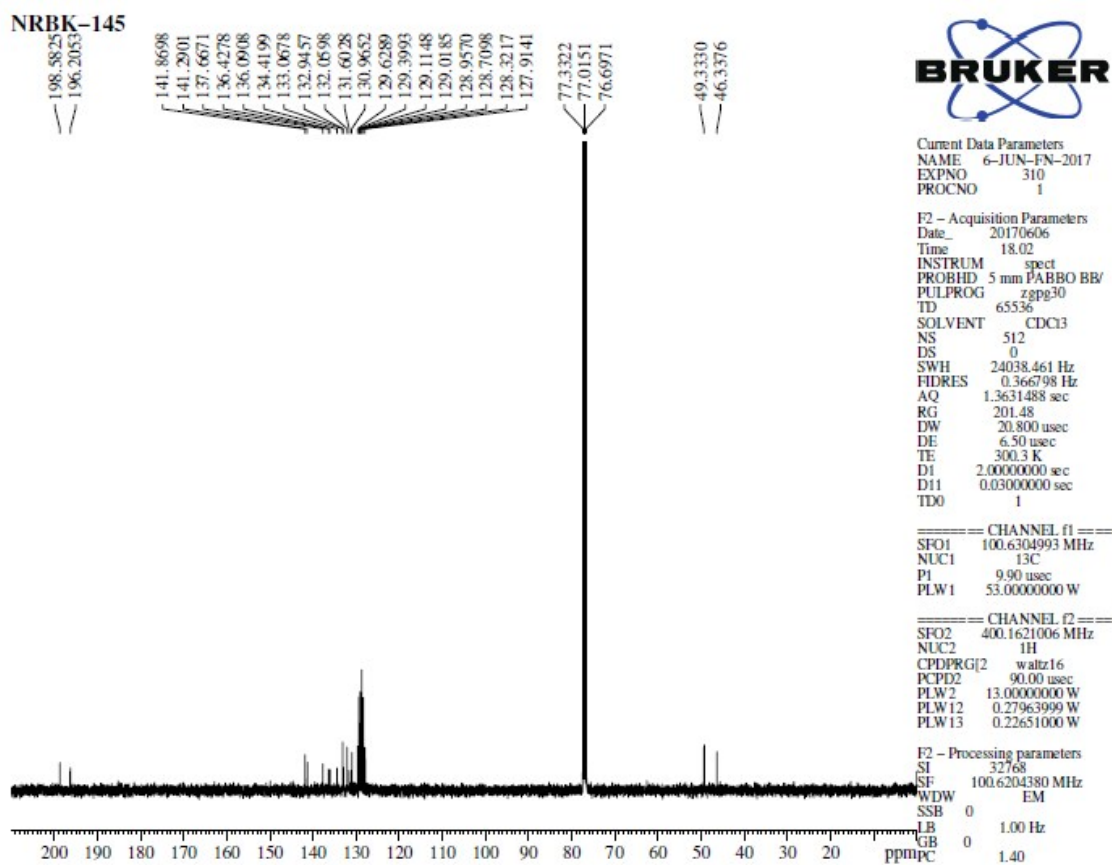


Figure 33: ¹³C NMR spectrum of 3k

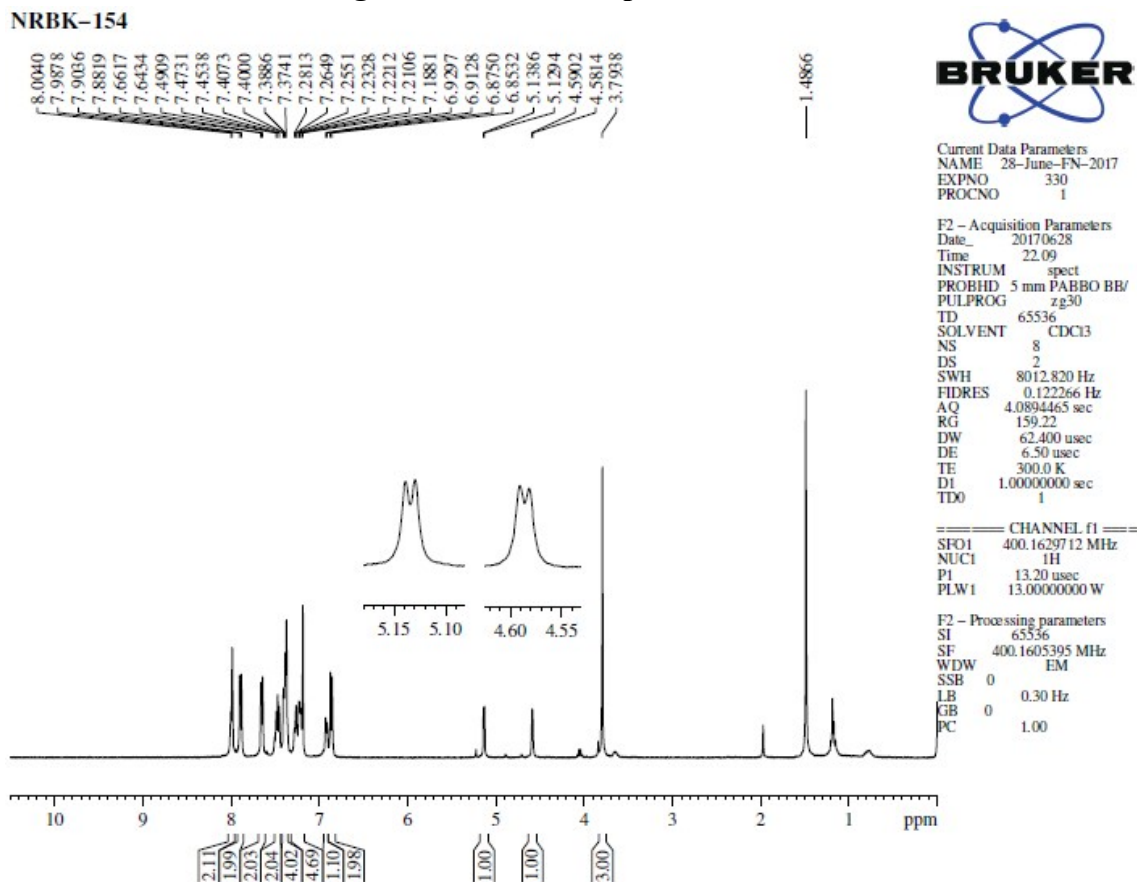
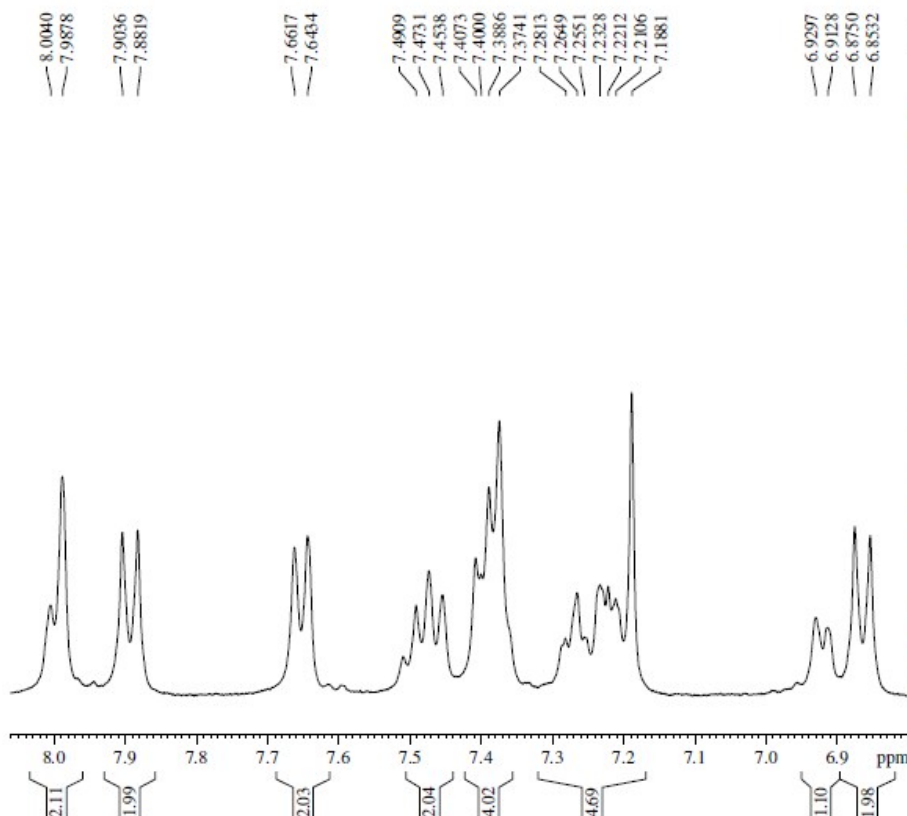


Figure 34: ¹H NMR spectrum of 3l

NRBK-154



Current Data Parameters
 NAME 28-Jun-FN-2017
 EXPNO 330
 PROCNO 1

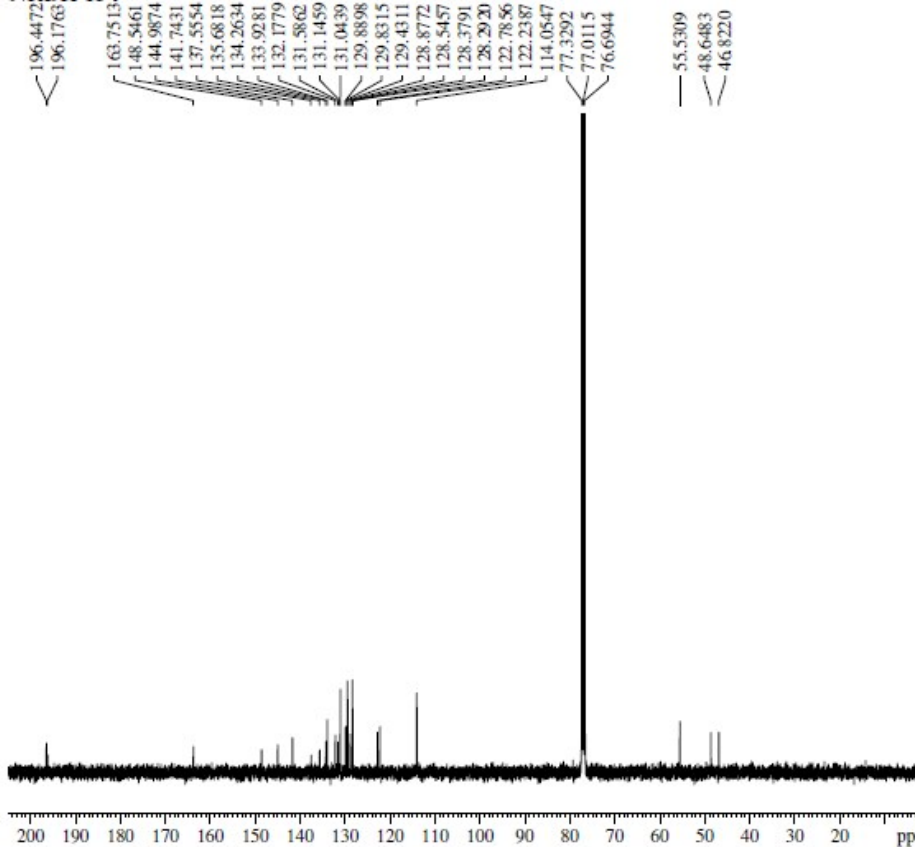
F2 - Acquisition Parameters
 Date_ 20170628
 Time 22.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 159.22
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605395 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 35: ¹H NMR spectrum of 3l (expansion)

NRBK 154



Current Data Parameters
 NAME 30-Jun-FN-2017
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170630
 Time 19.44
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 301.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

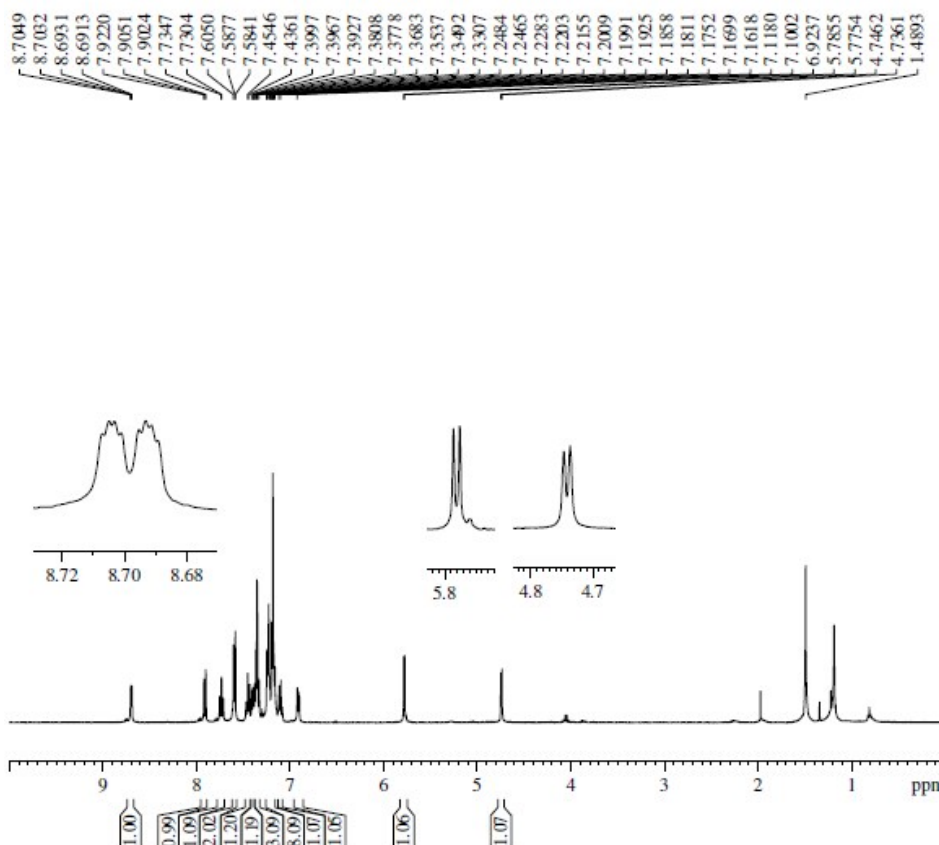
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 36: ¹³C NMR spectrum of 3l

NRBK-205



Current Data Parameters
NAME 13-NOV-FN-2017
EXPNO 350
PROCNO 1

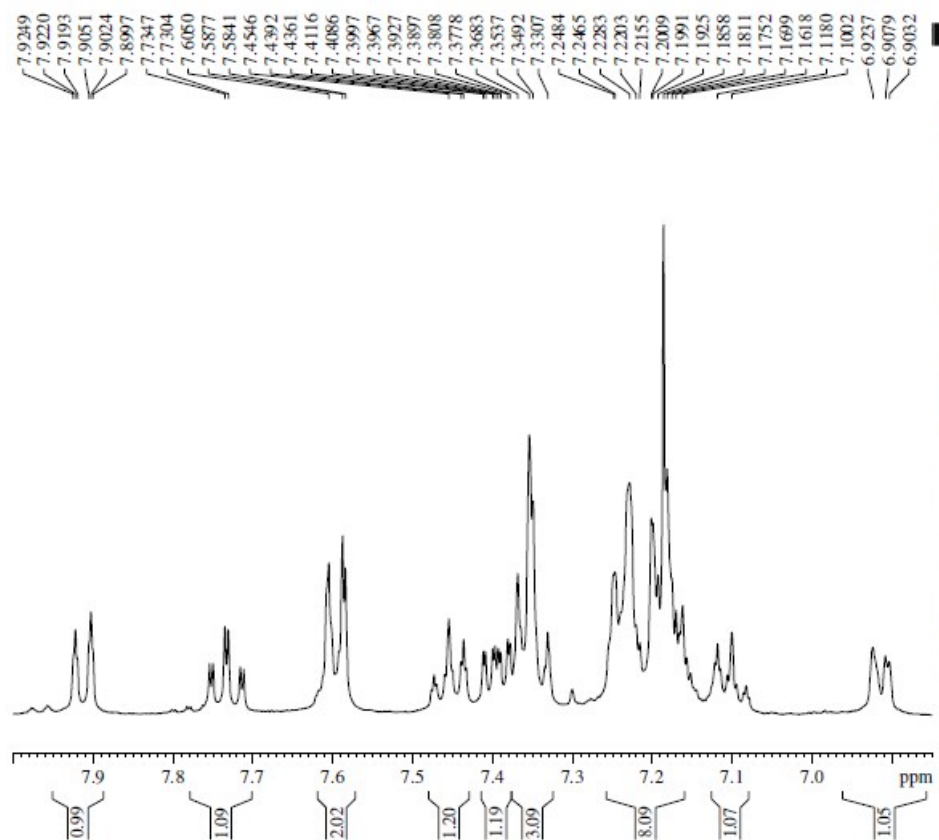
F2 - Acquisition Parameters
Date_ 20171113
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 159.22
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TDO 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605390 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 37: ¹H NMR spectrum of 3m

NRBK-205



Current Data Parameters
NAME 13-NOV-FN-2017
EXPNO 350
PROCNO 1

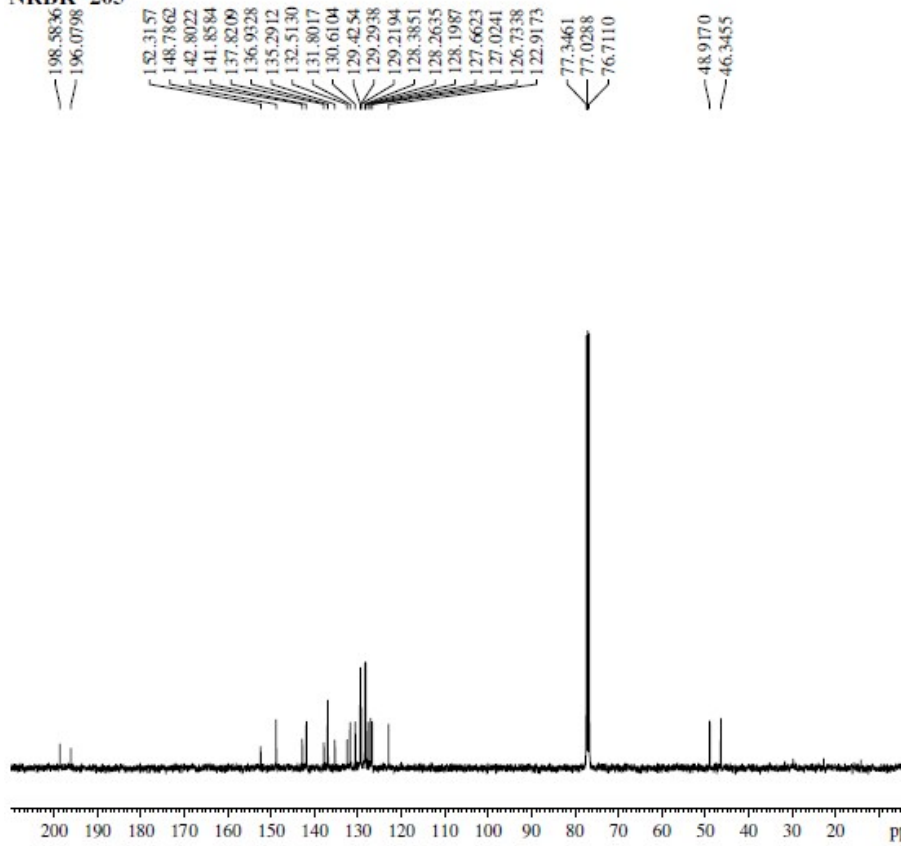
F2 - Acquisition Parameters
Date_ 20171113
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 159.22
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TDO 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605390 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 38: ¹H NMR spectrum of 3m (expansion)

NRBK-205



Current Data Parameters
 NAME 05-Oct-FN-2017
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171005
 Time 20.20
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

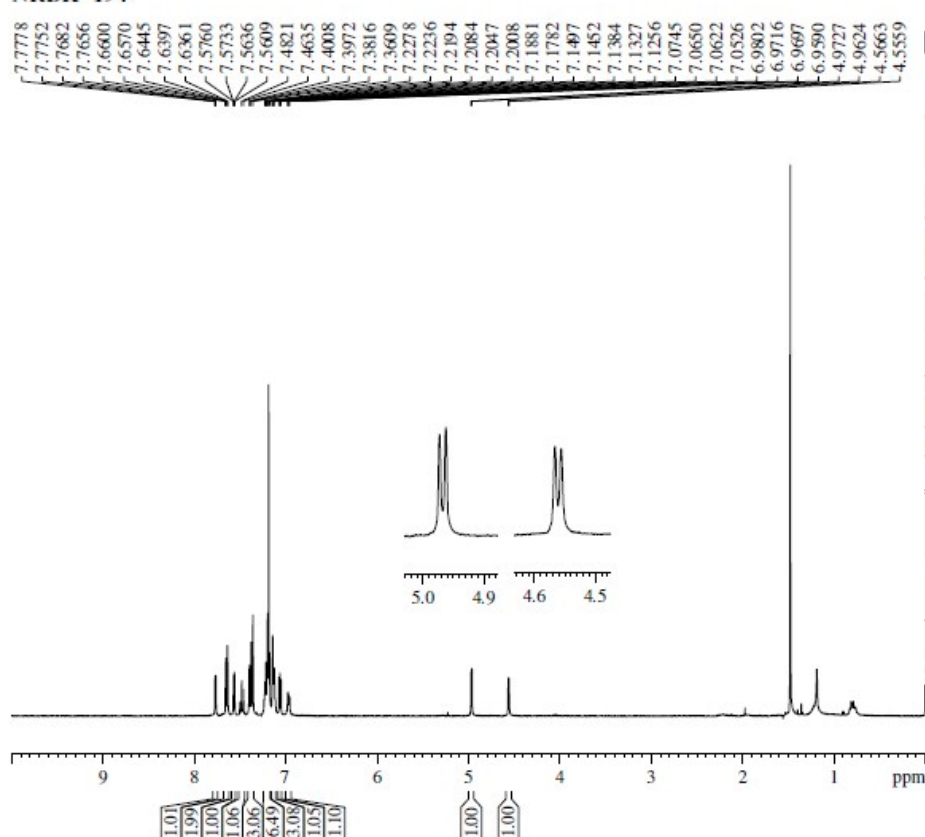
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW12 13.0000000 W
 PLW13 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

Figure 39: ¹³C NMR spectrum of 3m

NRBK-194



Current Data Parameters
 NAME 16-NOV-FN-2017
 EXPNO 320
 PROCNO 1

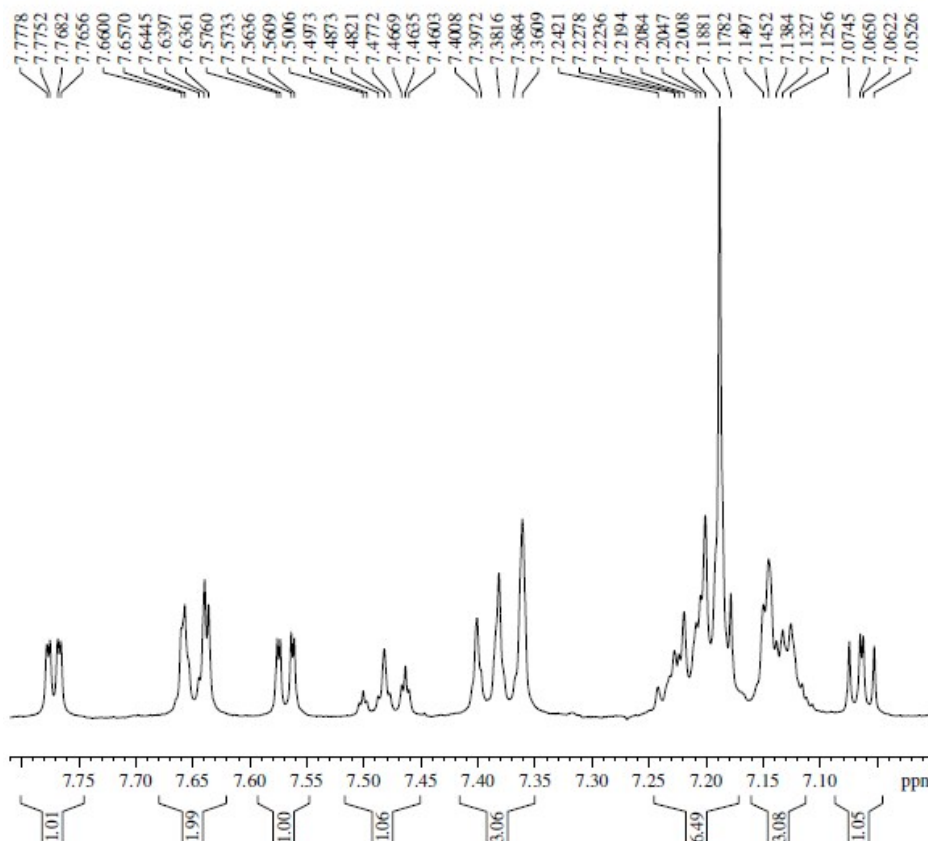
F2 - Acquisition Parameters
 Date_ 20171116
 Time 16.49
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 179.93
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605382 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 40: ¹H NMR spectrum of 3n

NRBK-194



Current Data Parameters
 NAME 16-NOV-FN-2017
 EXPNO 320
 PROCNO 1

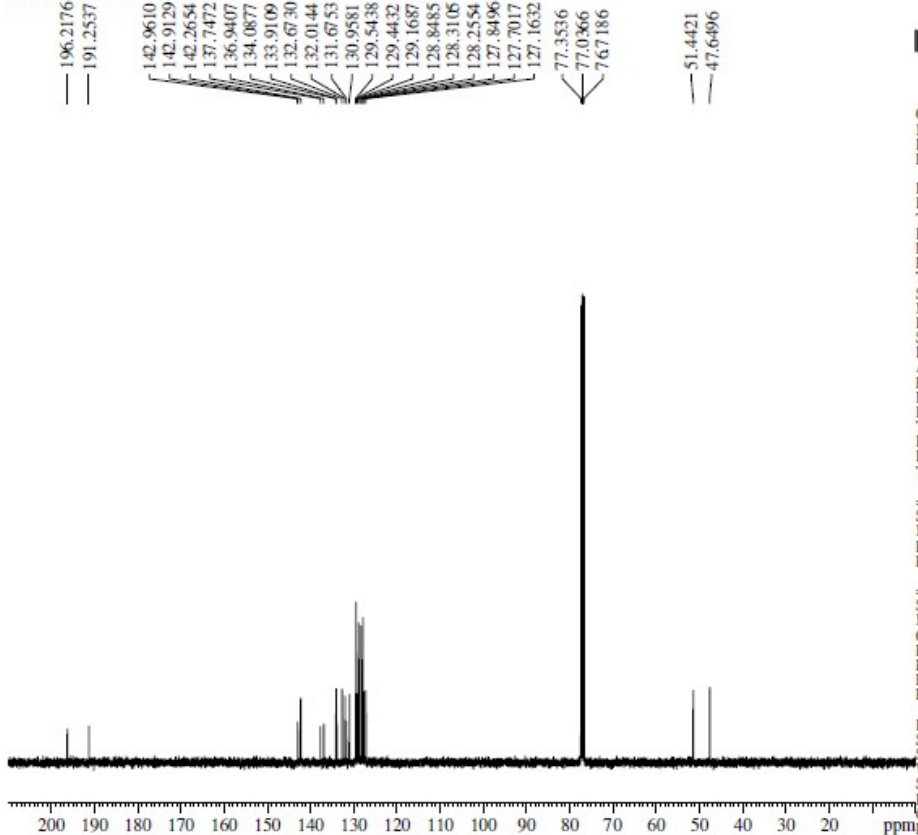
F2 - Acquisition Parameters
 Date_ 20171116
 Time 16.49
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 179.93
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605382 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 41: ¹H NMR spectrum of 3n (expansion)

NRBK-194



Current Data Parameters
 NAME 22-Aug-FN-2017
 EXPNO 370
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170822
 Time 15.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

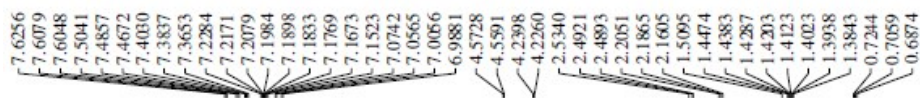
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

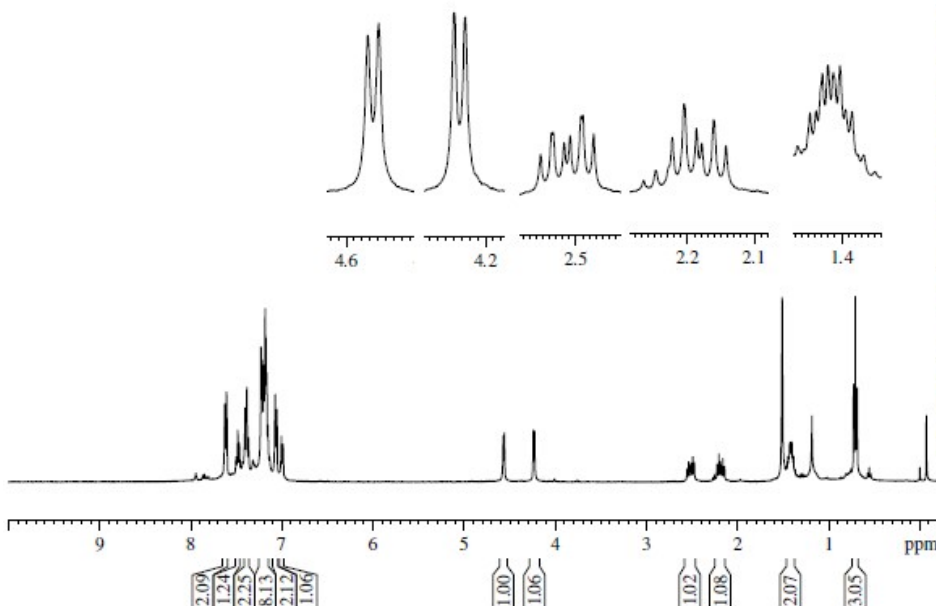
Figure 42: ¹³C NMR spectrum of 3n

NRBK-193



Current Data Parameters
NAME 21-Aug-FN-2017
EXPNO 440
PROCNO 1

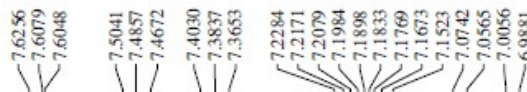
F2 - Acquisition Parameters
Date_ 20170821
Time 15.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 114.26
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1



==== CHANNEL f1 ====
SFO1 400.162912 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1605409 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

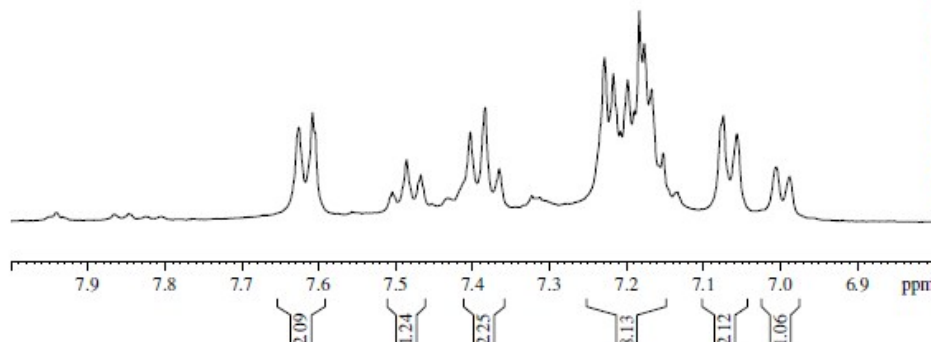
Figure 43: ¹H NMR spectrum of 3o

NRBK-193



Current Data Parameters
NAME 21-Aug-FN-2017
EXPNO 440
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170821
Time 15.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 114.26
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1



==== CHANNEL f1 ====
SFO1 400.162912 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1605409 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 44: ¹H NMR spectrum of 3o (expansion)

NR-BK-193

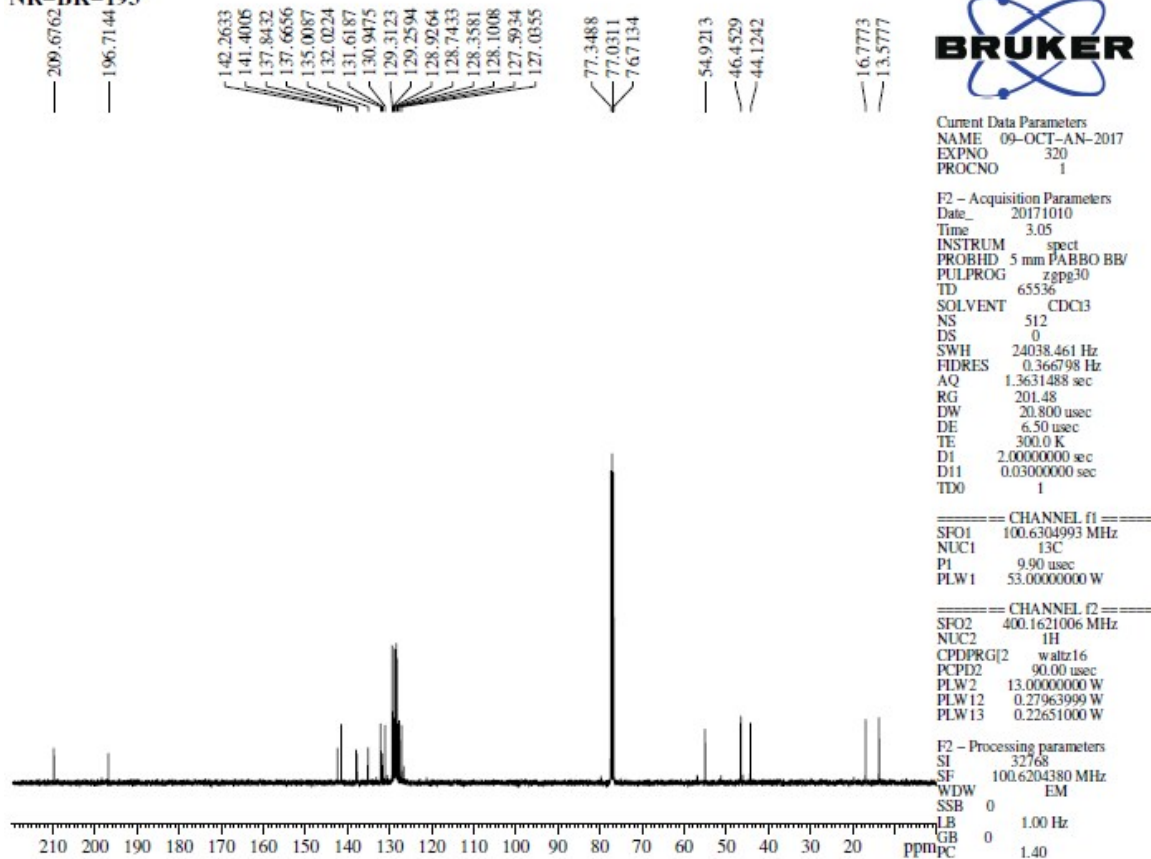


Figure 45: ¹³C NMR spectrum of 3o

NRBK-189

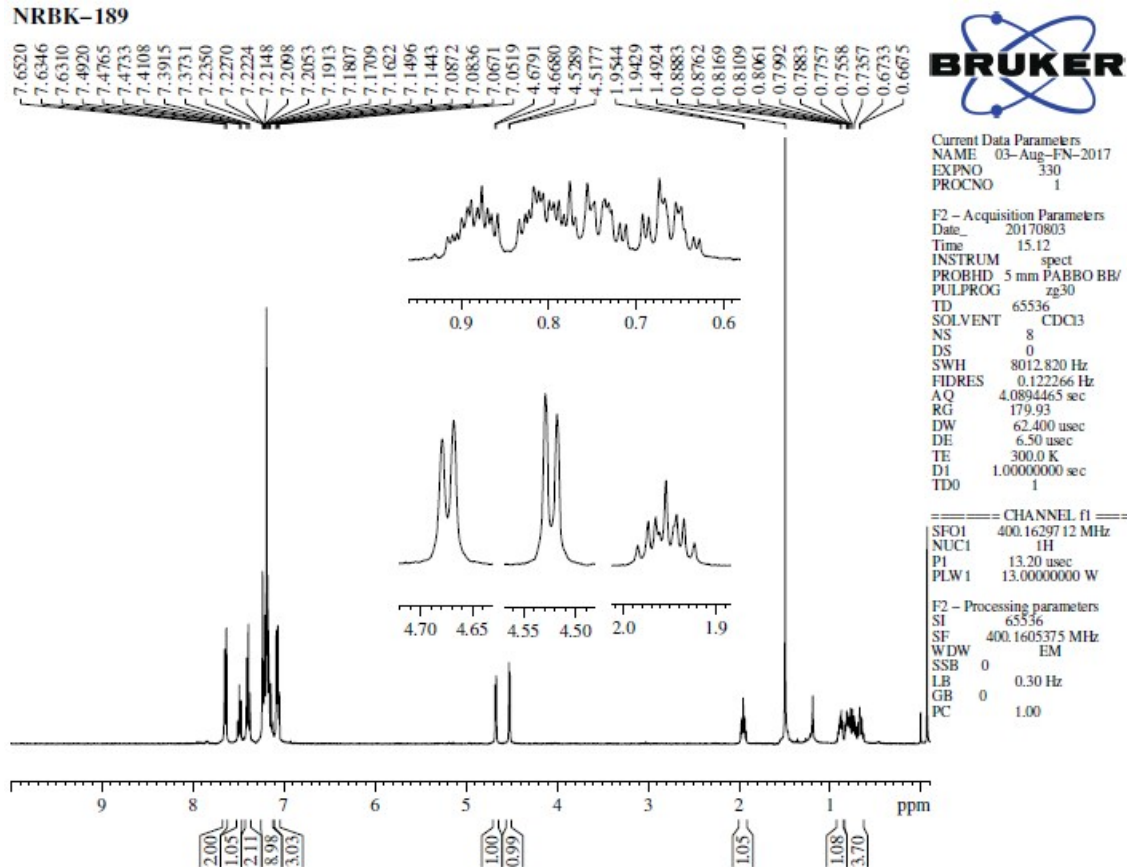


Figure 46: ¹H NMR spectrum of 3p

NRBK-189

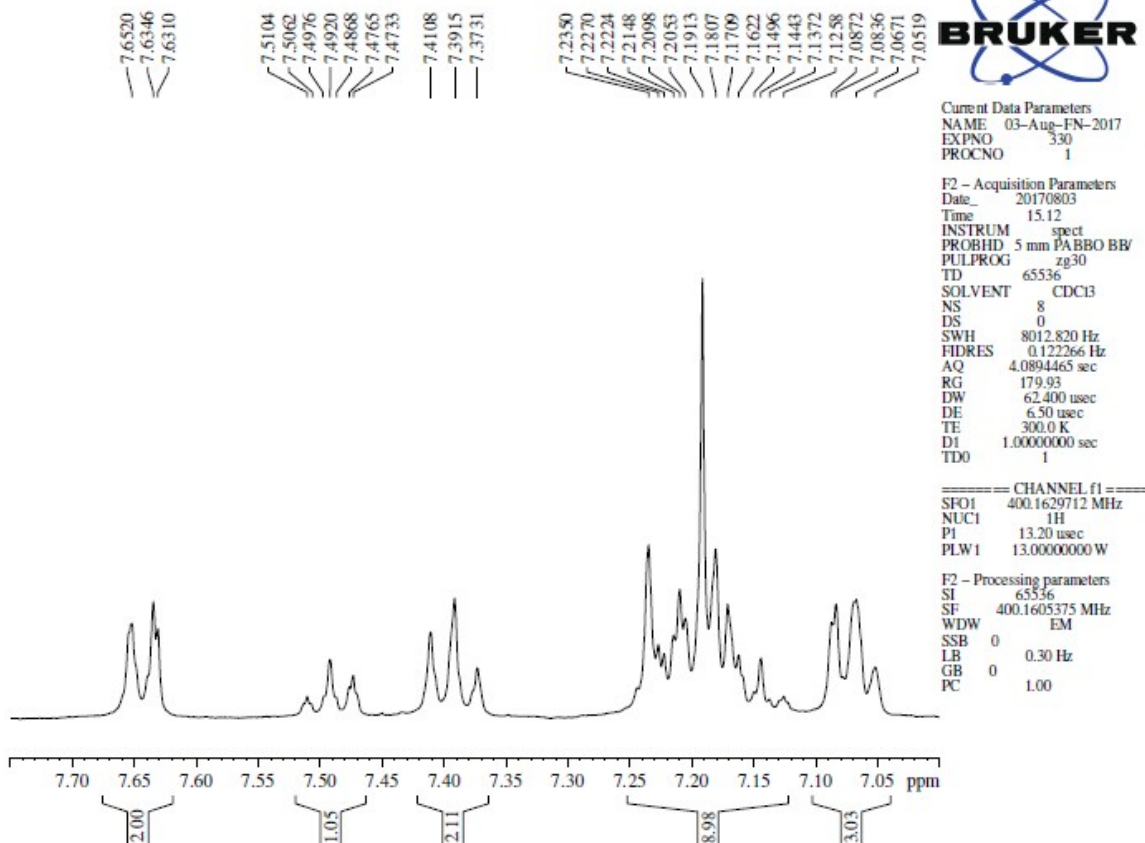


Figure 47: ¹H NMR spectrum of 3p (expansion)

NRBK-189

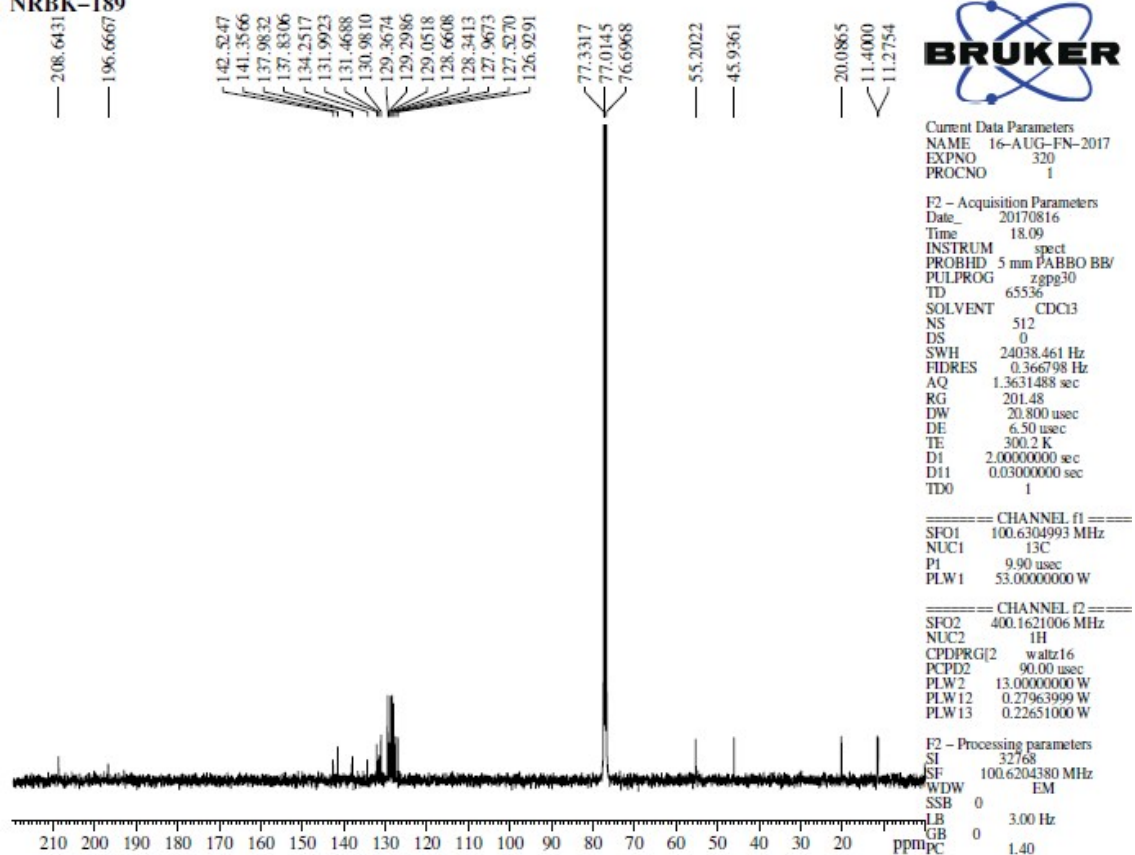
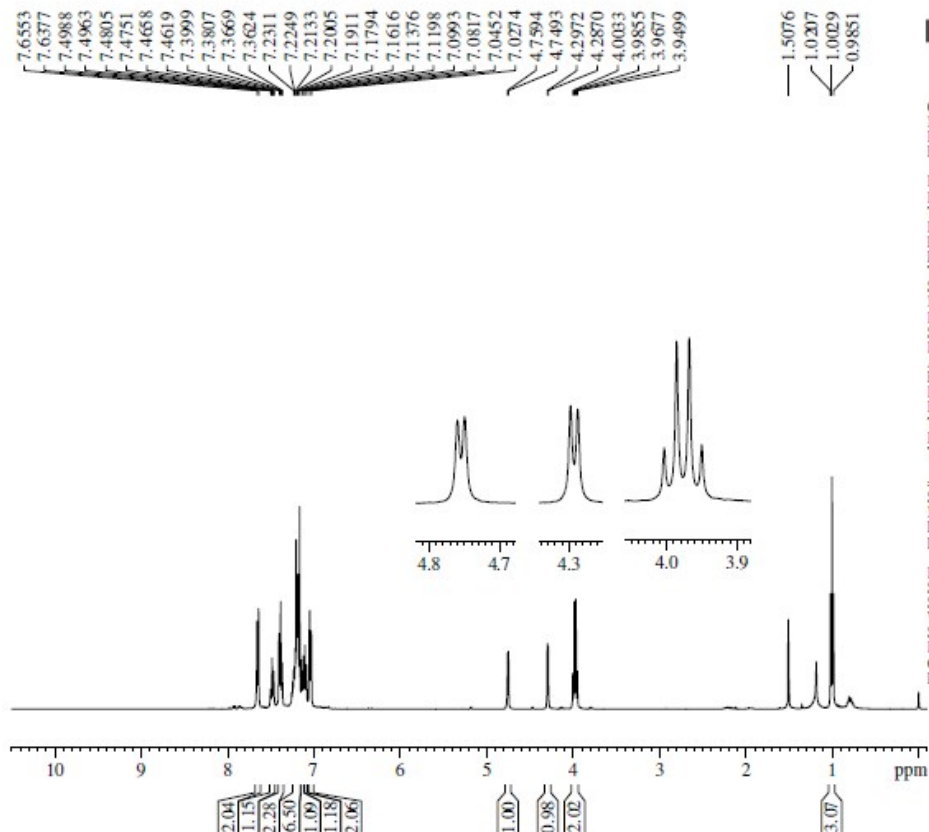


Figure 48: ¹³C NMR spectrum of 3p

NRBK-218



Current Data Parameters
 NAME 06-OCT-AN-2017
 EXPNO 320
 PROCNO 1

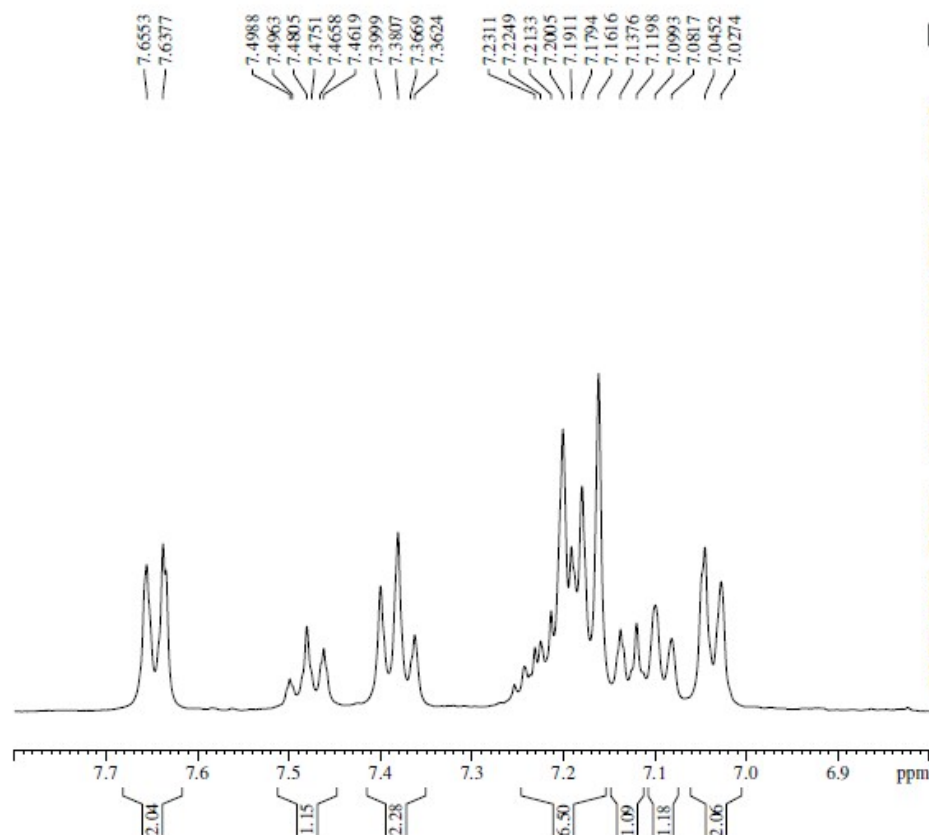
F2 - Acquisition Parameters
 Date_ 20171006
 Time 20.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 100.41
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605419 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 49: ¹H NMR spectrum of 3q

NRBK-218



Current Data Parameters
 NAME 06-OCT-AN-2017
 EXPNO 320
 PROCNO 1

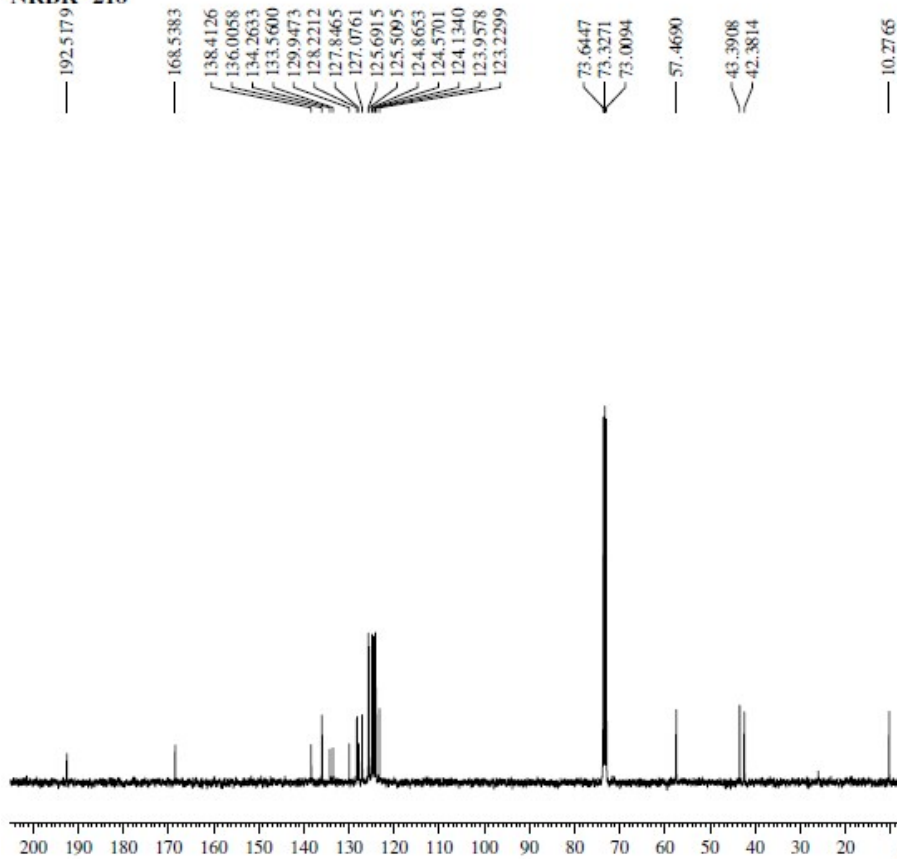
F2 - Acquisition Parameters
 Date_ 20171006
 Time 20.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 100.41
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605419 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 50: ¹H NMR spectrum of 3q (expansion)

NRBK-218



Current Data Parameters
 NAME 09-OCT-FN-2017
 EXPNO 340
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171009
 Time 20.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT Dioxane
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

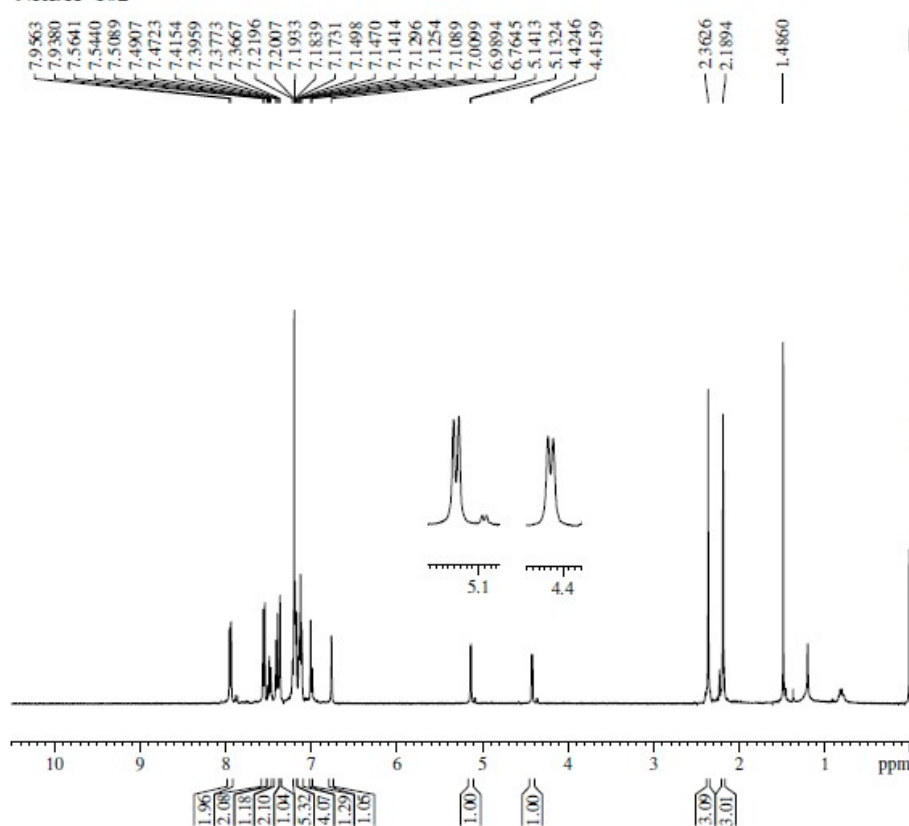
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

Figure 51: ¹³C NMR spectrum of 3q

NRBK-162



Current Data Parameters
 NAME 10-JULY-FN-2017
 EXPNO 370
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170710
 Time 14.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 159.22
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605340 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 52: ¹H NMR spectrum of 3r

NRBK-162

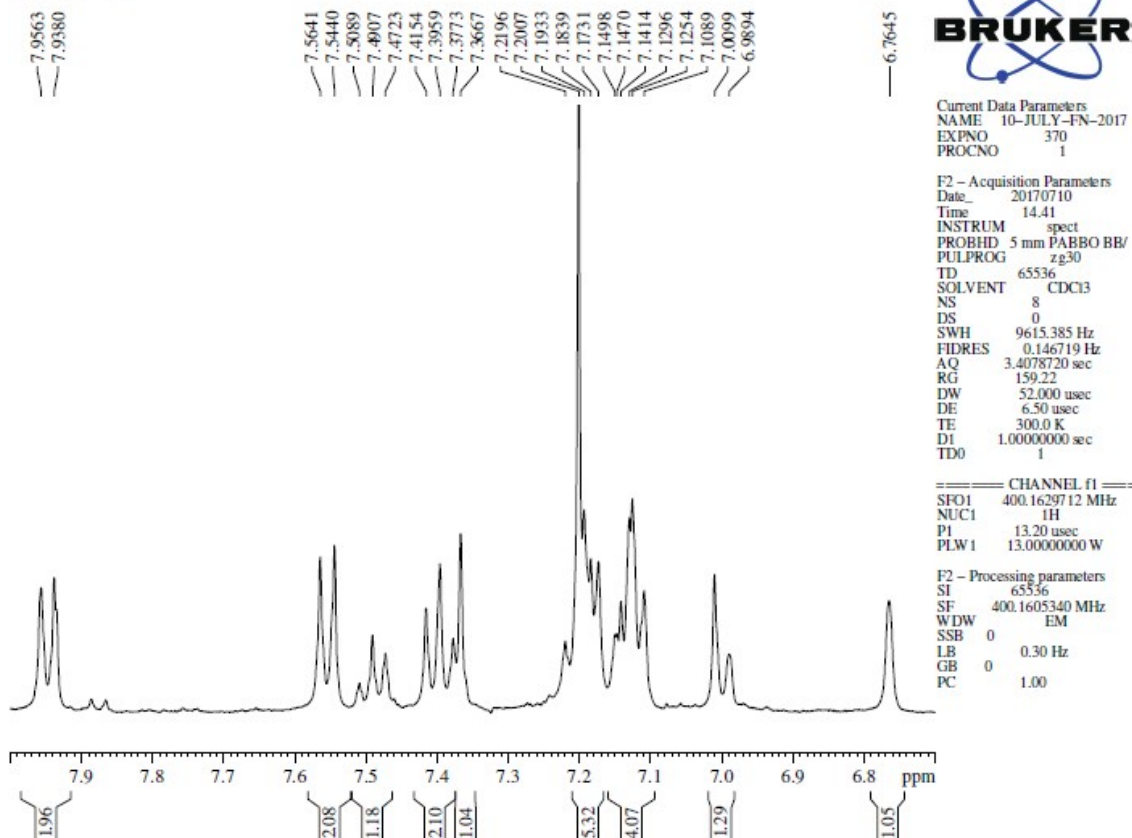


Figure 53: ¹H NMR spectrum of 3r (with expansion)

NRBK 162

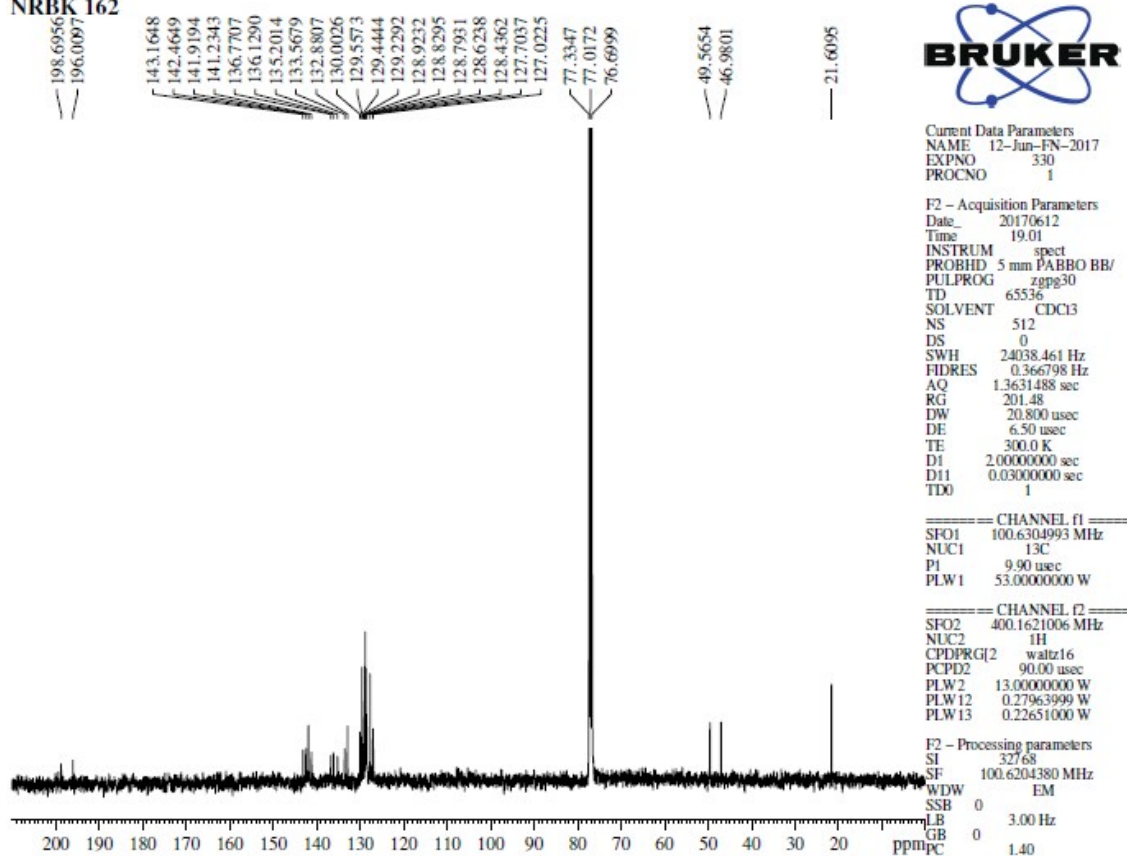
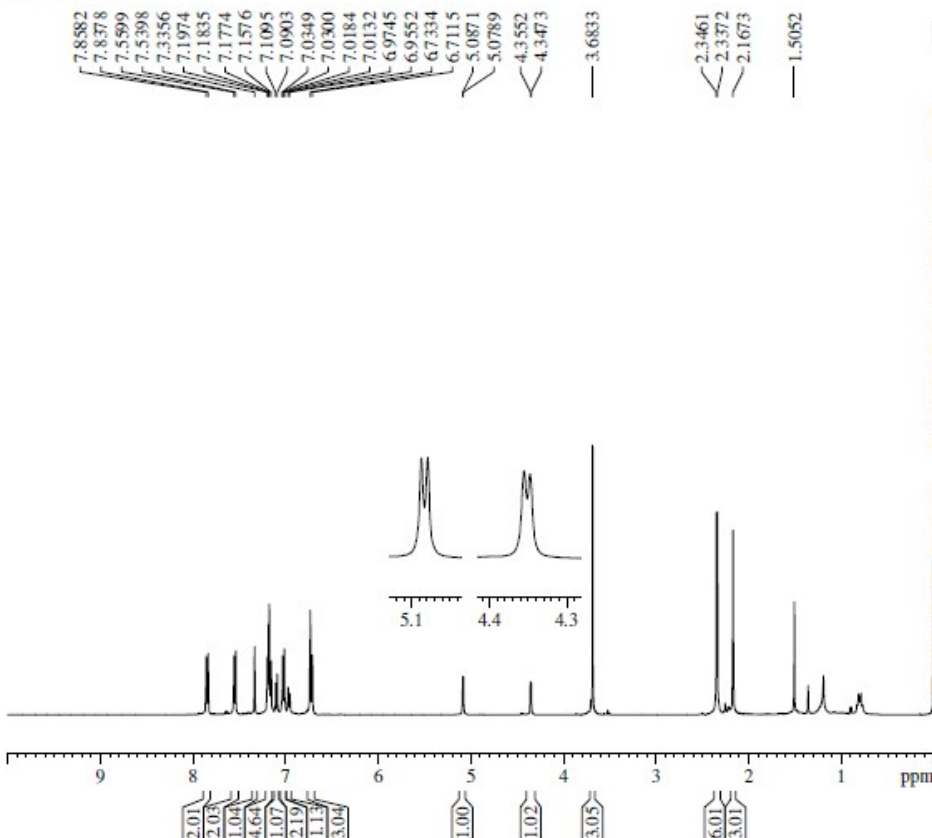


Figure 54: ¹³C NMR spectrum of 3r

NRBK-149



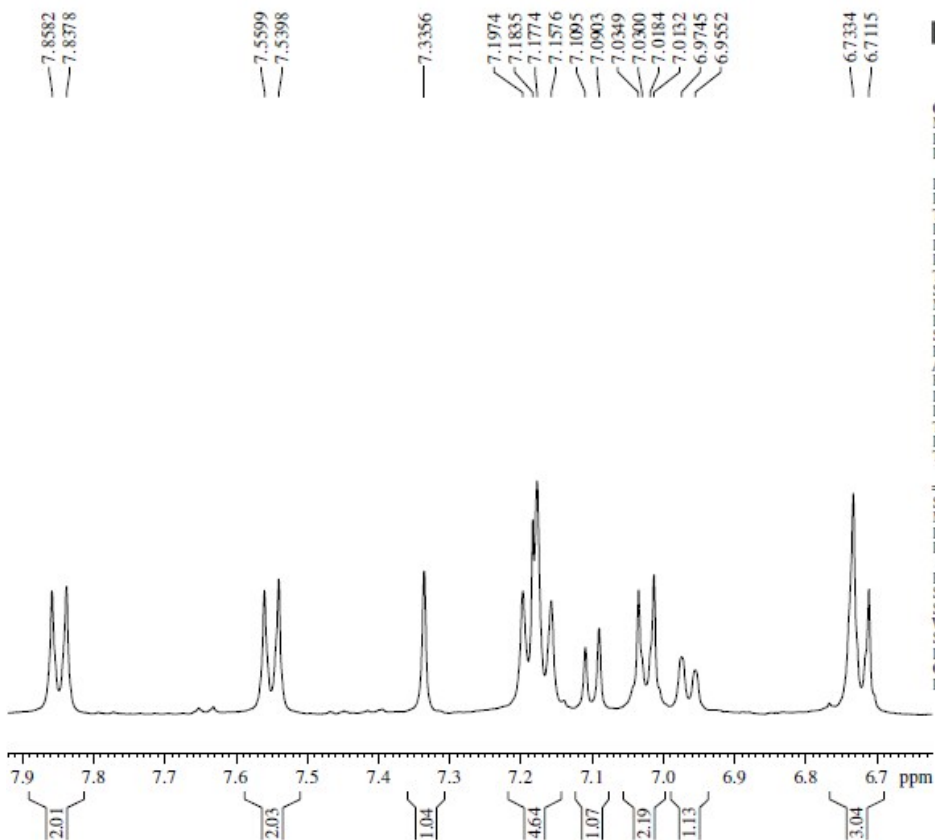
Current Data Parameters
 NAME 10-JULY-FN-2017
 EXPNO 360
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170710
 Time 14.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 100.41
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 400.1605407 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 55: ¹H NMR spectrum of 3s

NRBK-149



Current Data Parameters
 NAME 10-JULY-FN-2017
 EXPNO 360
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170710
 Time 14.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 100.41
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 400.1605407 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 56: ¹H NMR spectrum of 3s (expansion)

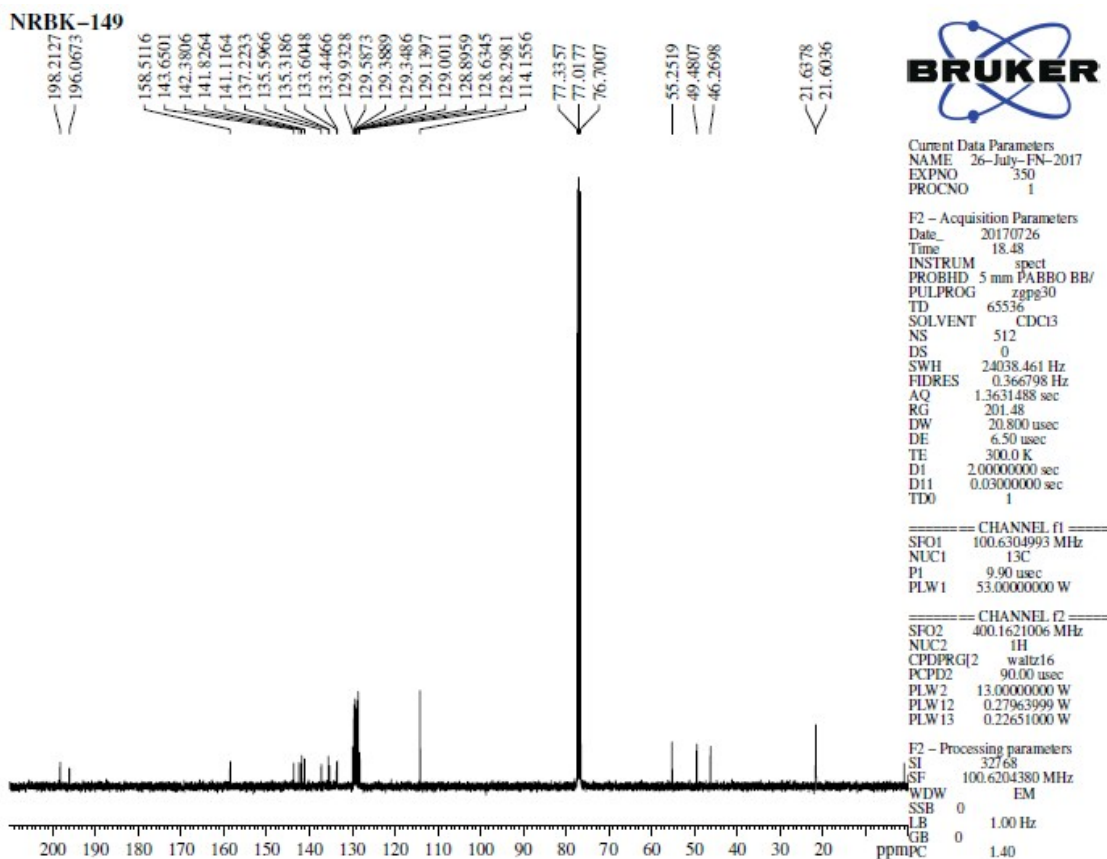


Figure 57: ¹³C NMR spectrum of 3s

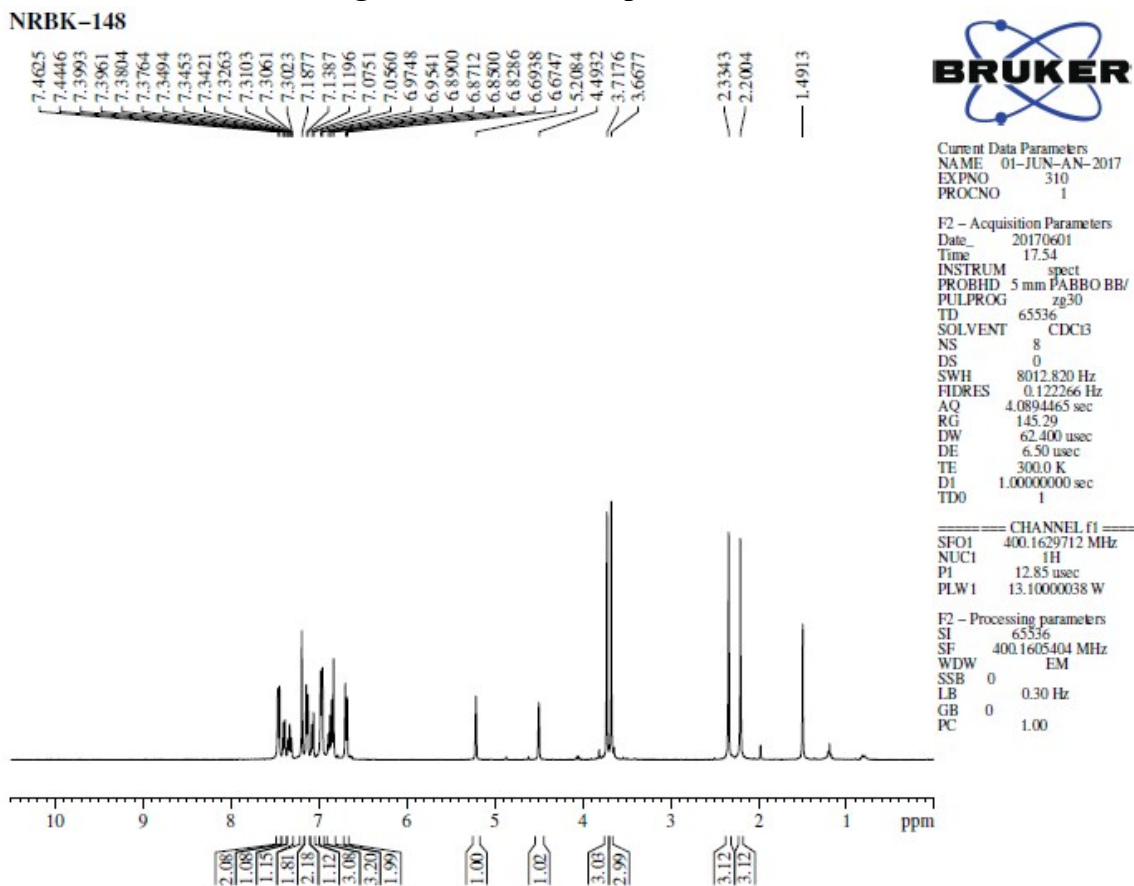


Figure 58: ¹H NMR spectrum of 3t

NRBK-148

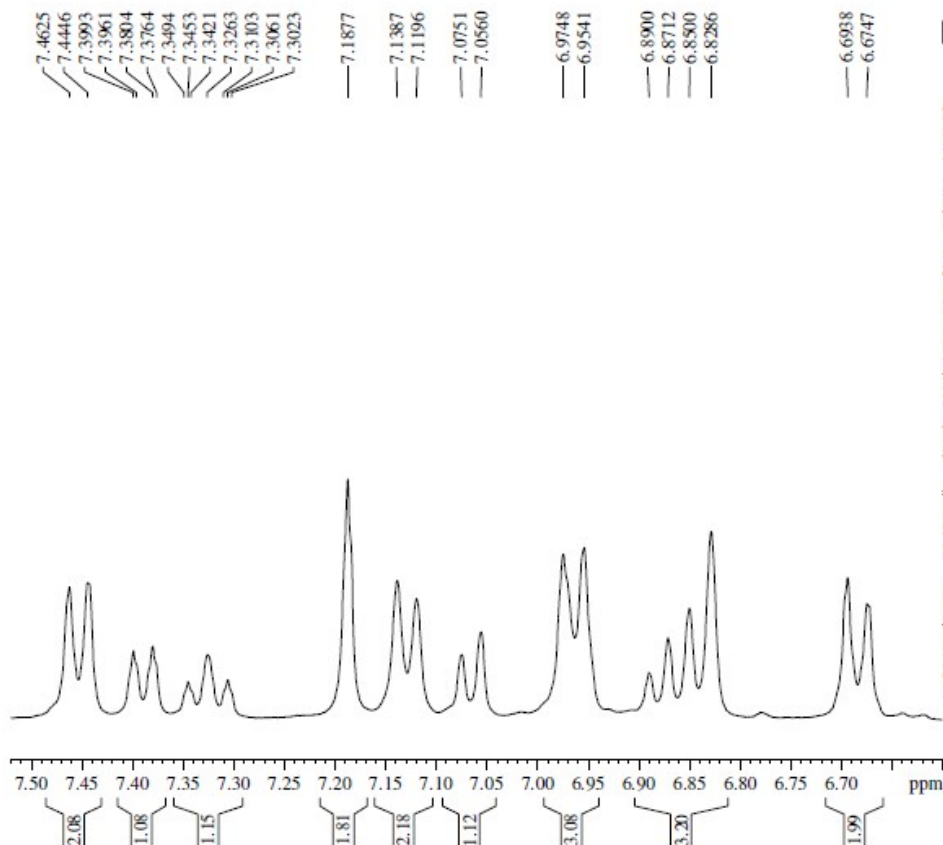


Figure 59: ¹H NMR spectrum of 3t (expansion)



Current Data Parameters
NAME 01-JUN-AN-2017
EXPNO 310
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170601
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 145.29
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 12.85 usec
PLW1 13.10000038 W

F2 - Processing parameters
SI 65536
SF 400.1605404 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NRBK-148

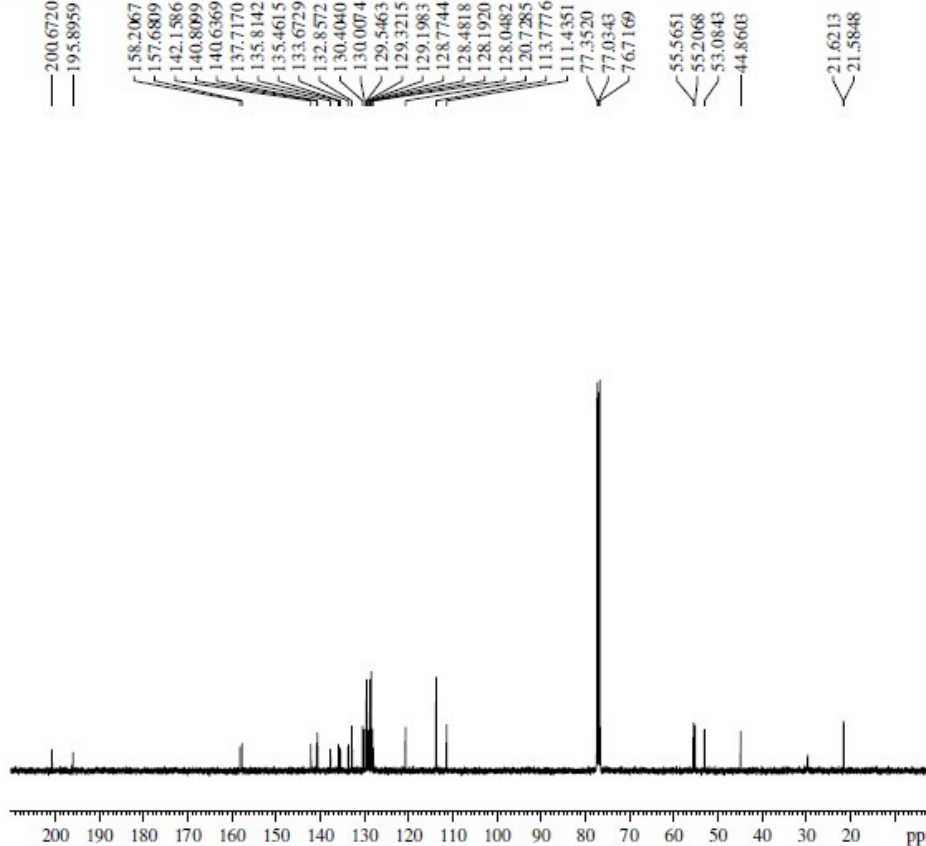


Figure 60: ¹³C NMR spectrum of 3t



Current Data Parameters
NAME 29-MAY-AN-2017
EXPNO 340
PROCNO 1

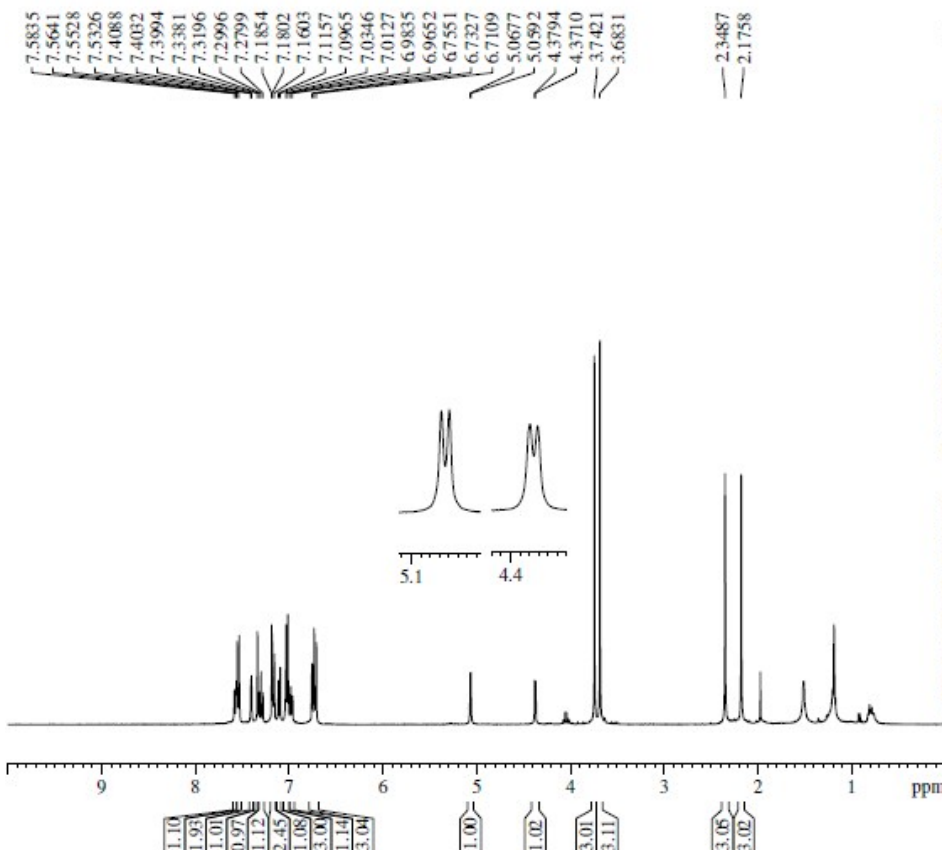
F2 - Acquisition Parameters
Date_ 20170529
Time 22.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 13C
P1 9.00 usec
PLW1 61.09999847 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.10000038 W
PLW12 0.26705000 W
PLW13 0.21630999 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

NRBK 146



Current Data Parameters
 NAME 22-MAY-FN-2017
 EXPNO 370
 PROCNO 1

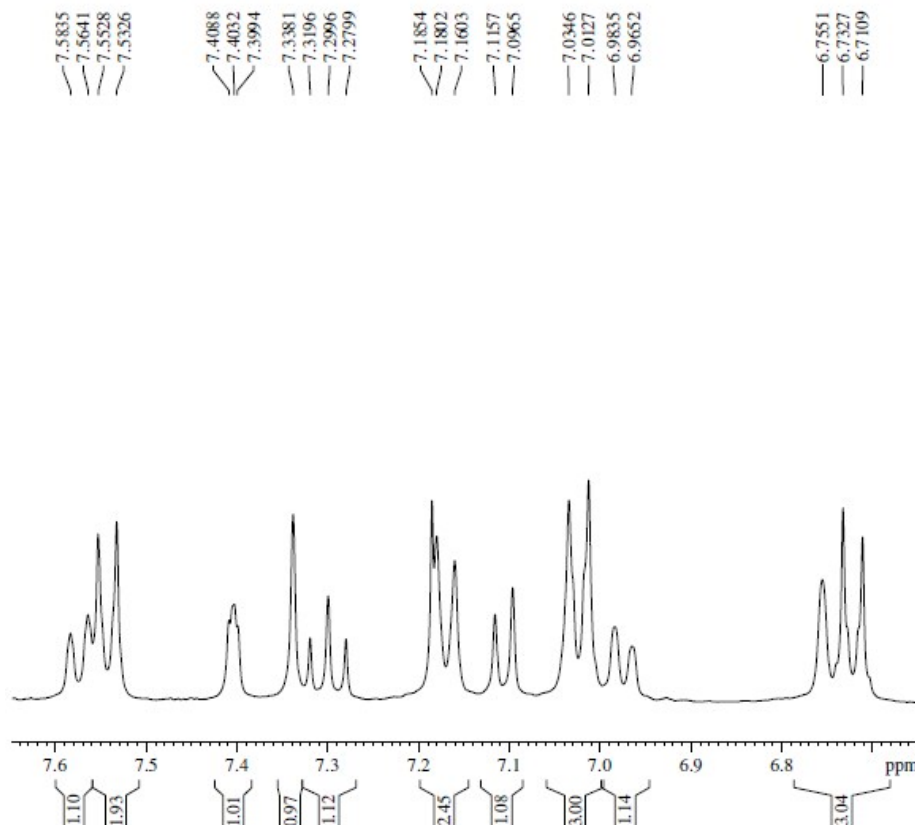
F2 - Acquisition Parameters
 Date_ 20170522
 Time 15.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605401 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 61: ¹H NMR spectrum of 3u

NRBK 146



Current Data Parameters
 NAME 22-MAY-FN-2017
 EXPNO 370
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170522
 Time 15.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605401 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 62: ¹H NMR spectrum of 3u (expansion)

NRBK-146

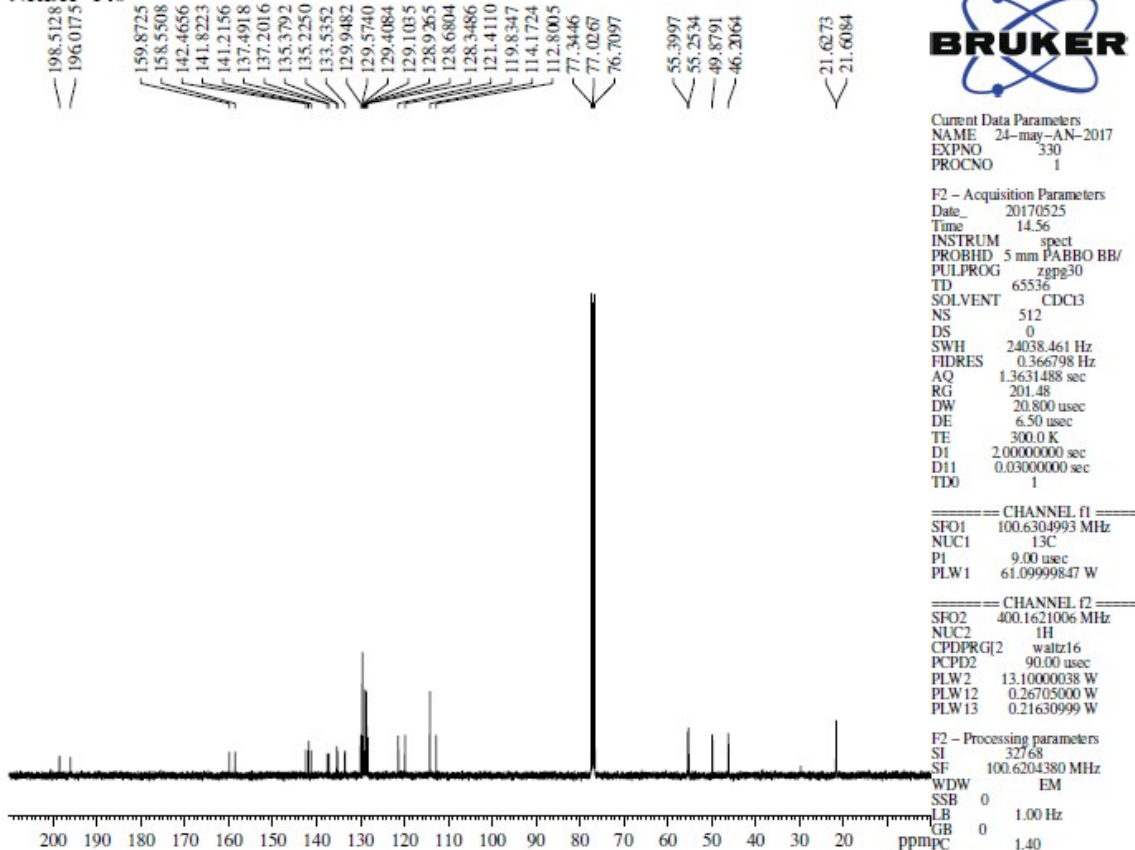


Figure 63: ¹³C NMR spectrum of 3u

NRBK 147

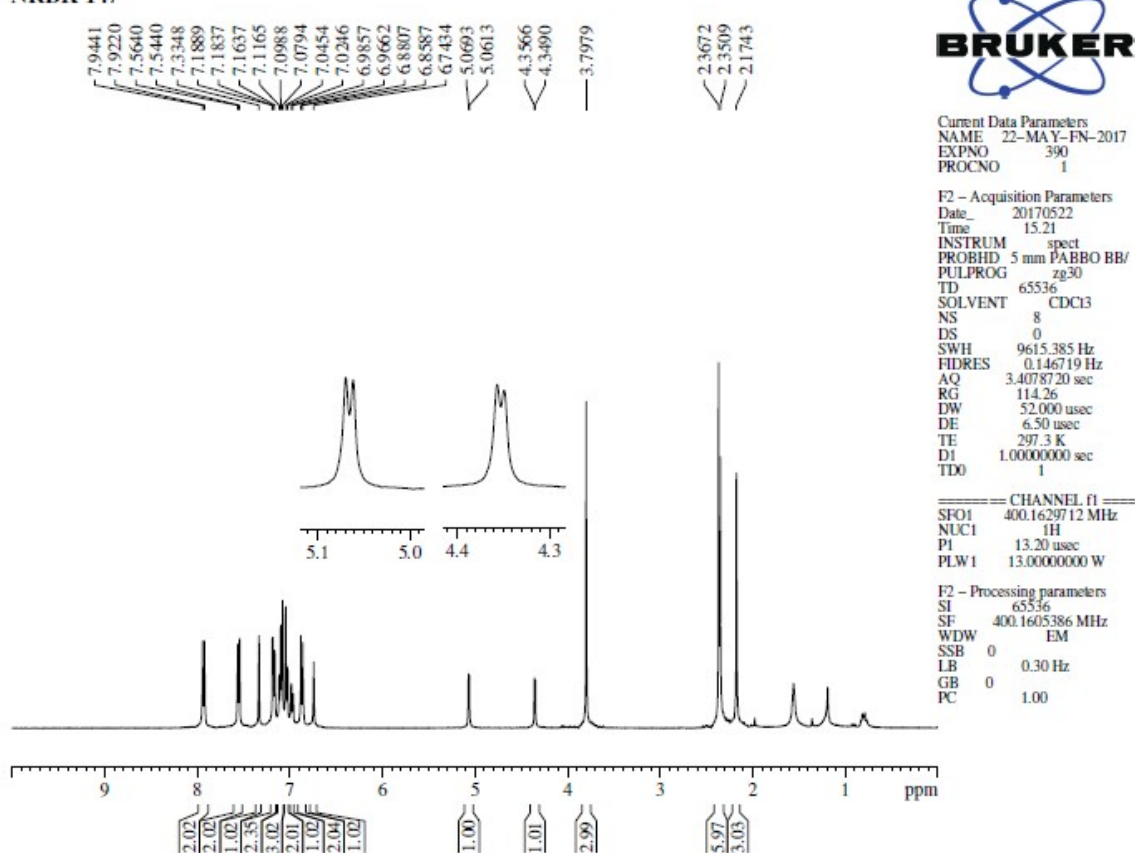
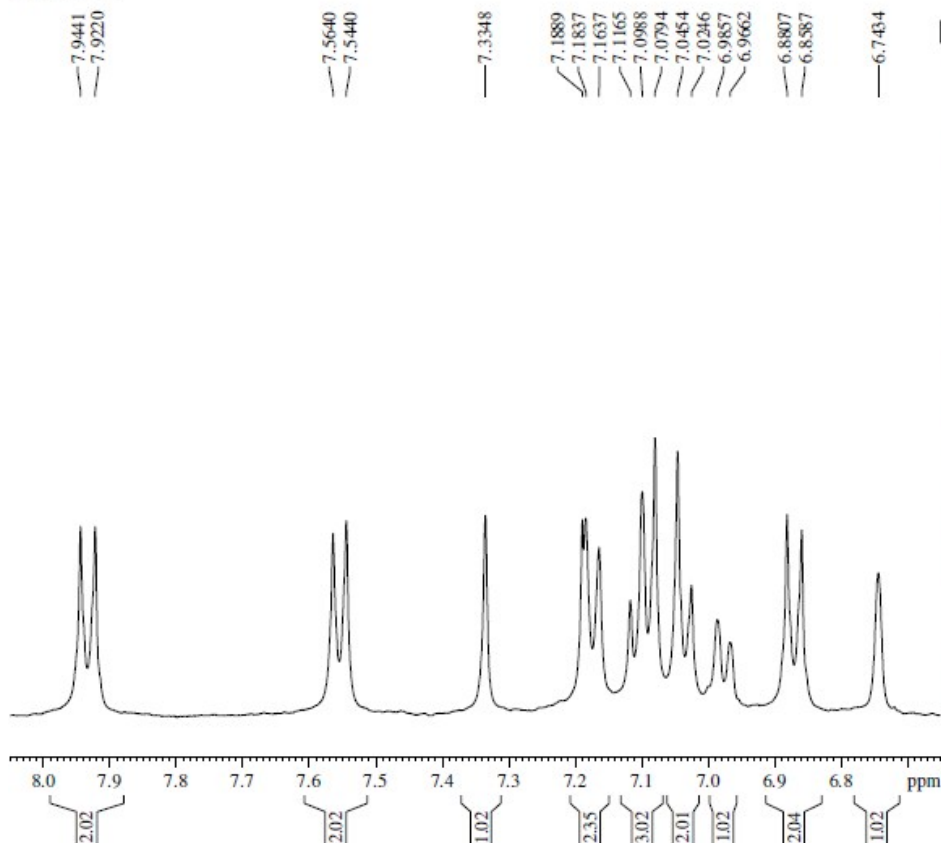


Figure 64: ¹H NMR spectrum of 3v

NRBK 147



Current Data Parameters
 NAME 22-MAY-FN-2017
 EXPNO 390
 PROCNO 1

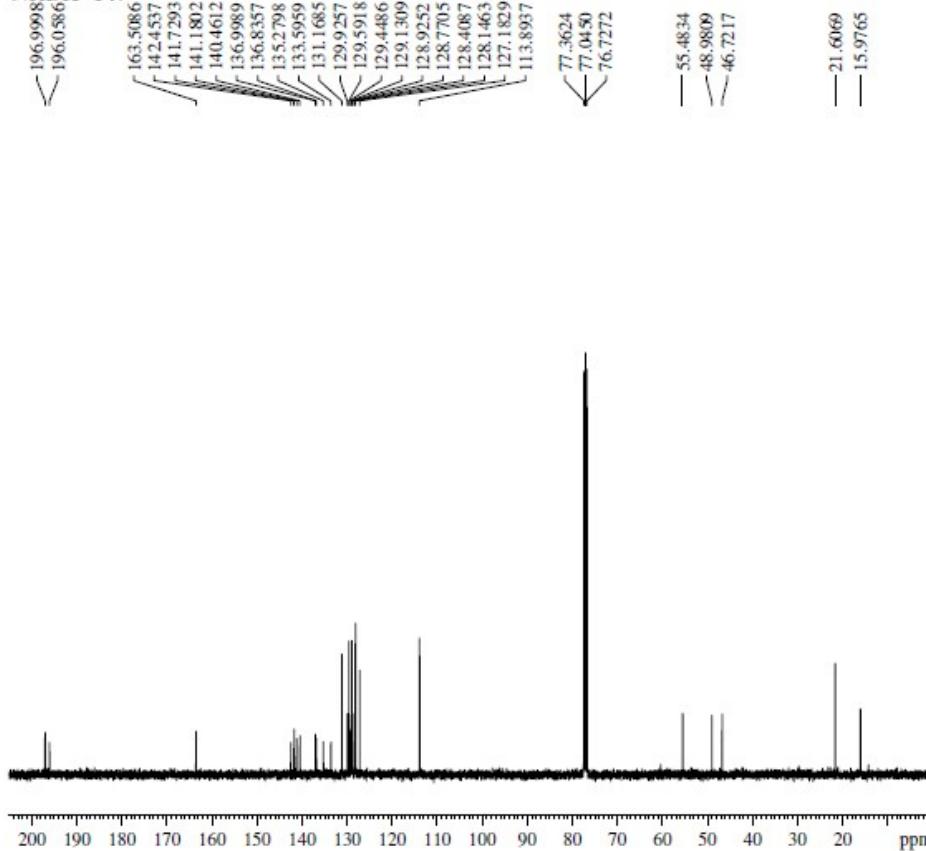
F2 - Acquisition Parameters
 Date_ 20170522
 Time 15.21
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 297.3 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605386 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 65: ¹H NMR spectrum of 3v (expansion)

NRBK-147



Current Data Parameters
 NAME 18-May-FN-2017
 EXPNO 340
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170518
 Time 17.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 184
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 299.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

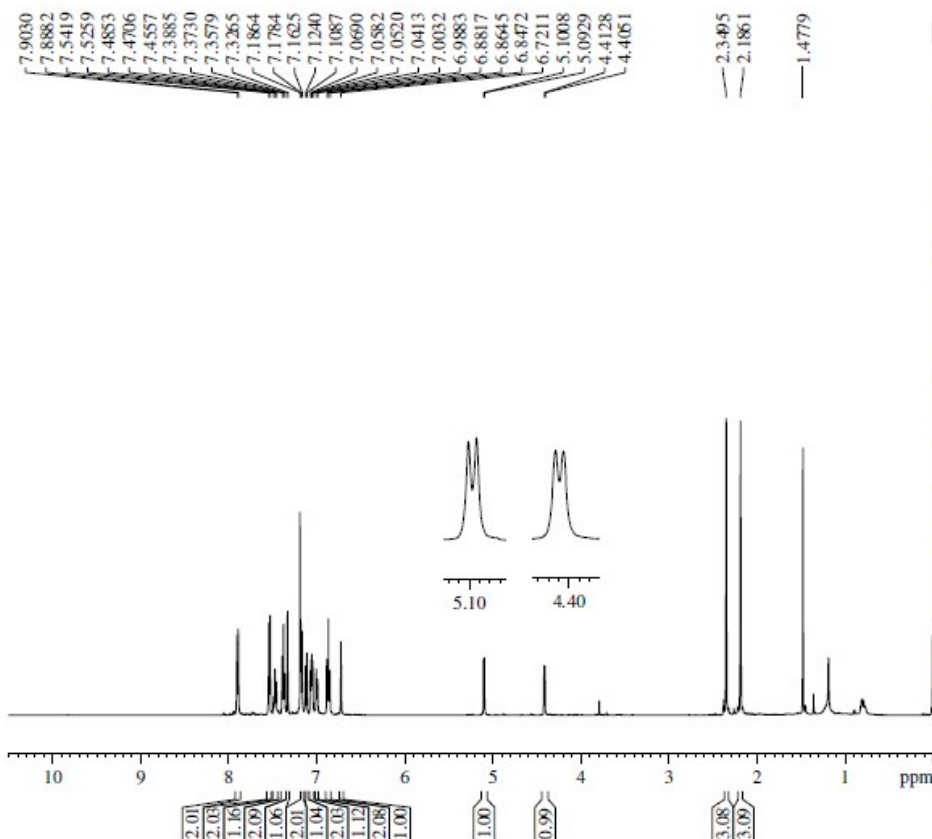
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 66: ¹³C NMR spectrum of 3v

NRBK-171



Current Data Parameters
 NAME 07-JULY-FN-2017
 EXPNO 310
 PROCNO 1

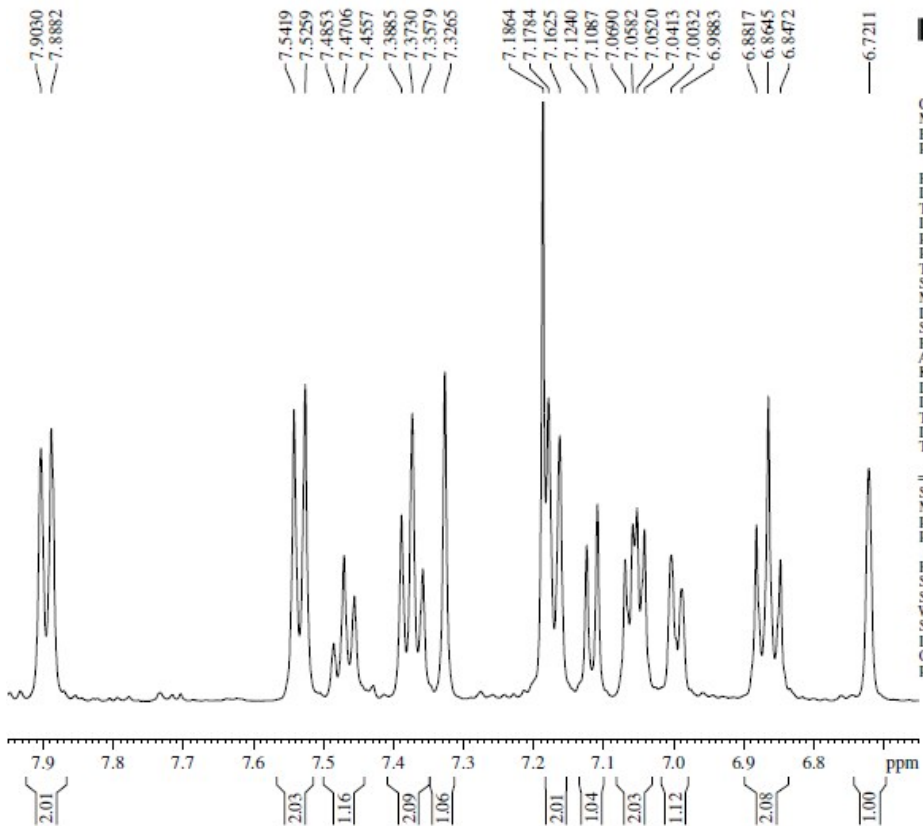
F2 - Acquisition Parameters
 Date_ 20170707
 Time 8.06
 INSTRUM spect
 PROBHD 5 mm PATX1 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 10026.738 Hz
 FIDRES 0.152996 Hz
 AQ 3.2680619 sec
 RG 201.78
 DW 49.867 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 500.6794419 MHz
 NUC1 1H
 P1 6.45 usec
 PLW1 13.6000038 W

F2 - Processing parameters
 SI 65536
 SF 500.6763986 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 67: ¹H NMR spectrum of 3w

NRBK-171



Current Data Parameters
 NAME 07-JULY-FN-2017
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170707
 Time 8.06
 INSTRUM spect
 PROBHD 5 mm PATX1 1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 10026.738 Hz
 FIDRES 0.152996 Hz
 AQ 3.2680619 sec
 RG 201.78
 DW 49.867 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 500.6794419 MHz
 NUC1 1H
 P1 6.45 usec
 PLW1 13.6000038 W

F2 - Processing parameters
 SI 65536
 SF 500.6763986 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 68: ¹H NMR spectrum of 3w (expansion)

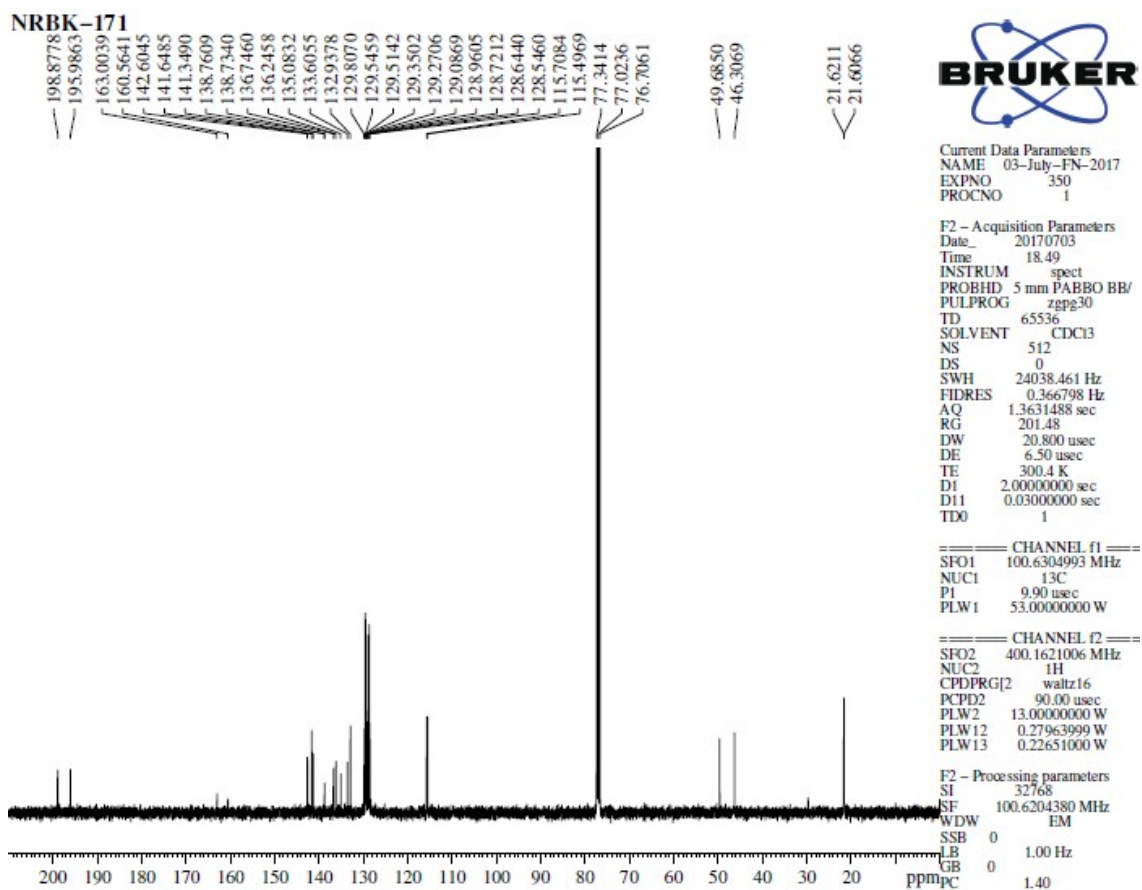


Figure 69: ^{13}C NMR spectrum of 3w

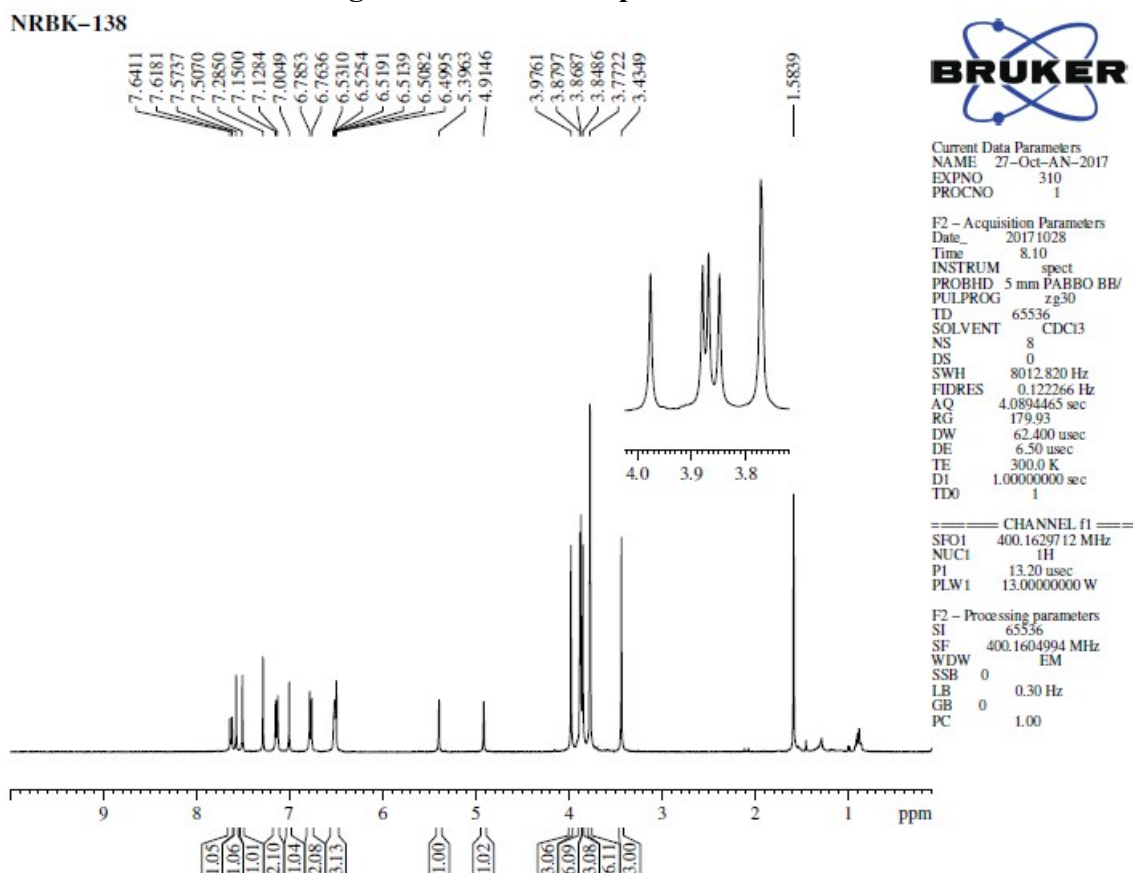
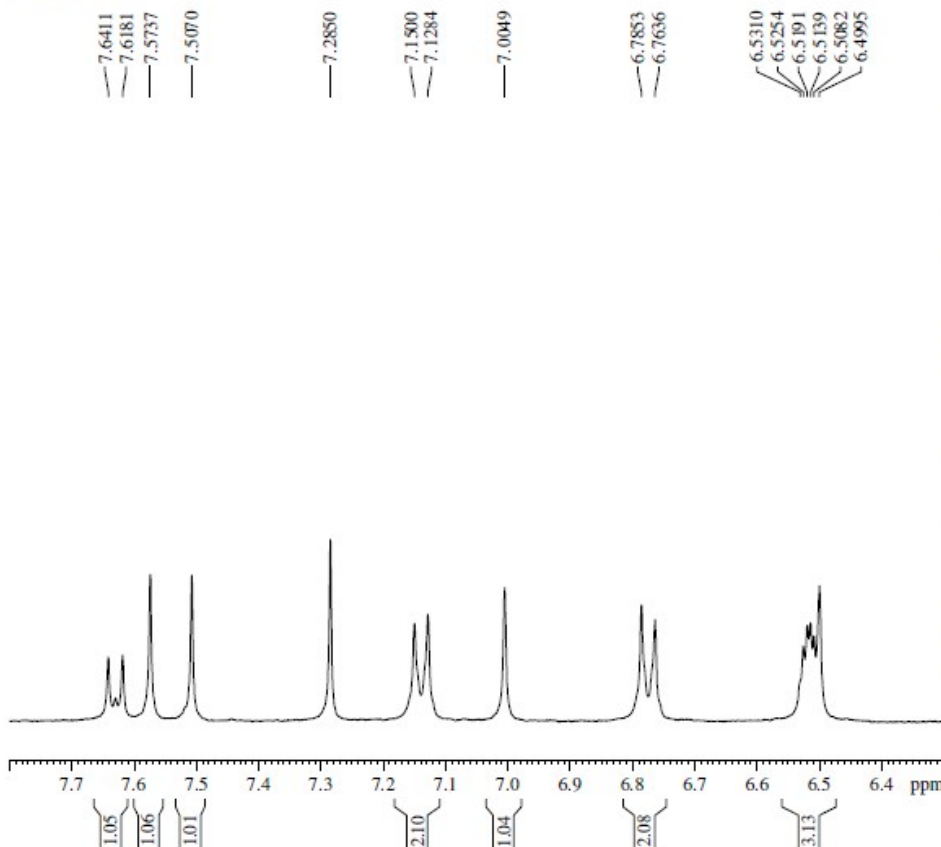


Figure 70: ^1H NMR spectrum of 3x

NRBK-138



Current Data Parameters
 NAME 27-Oct-AN-2017
 EXPNO 310
 PROCNO 1

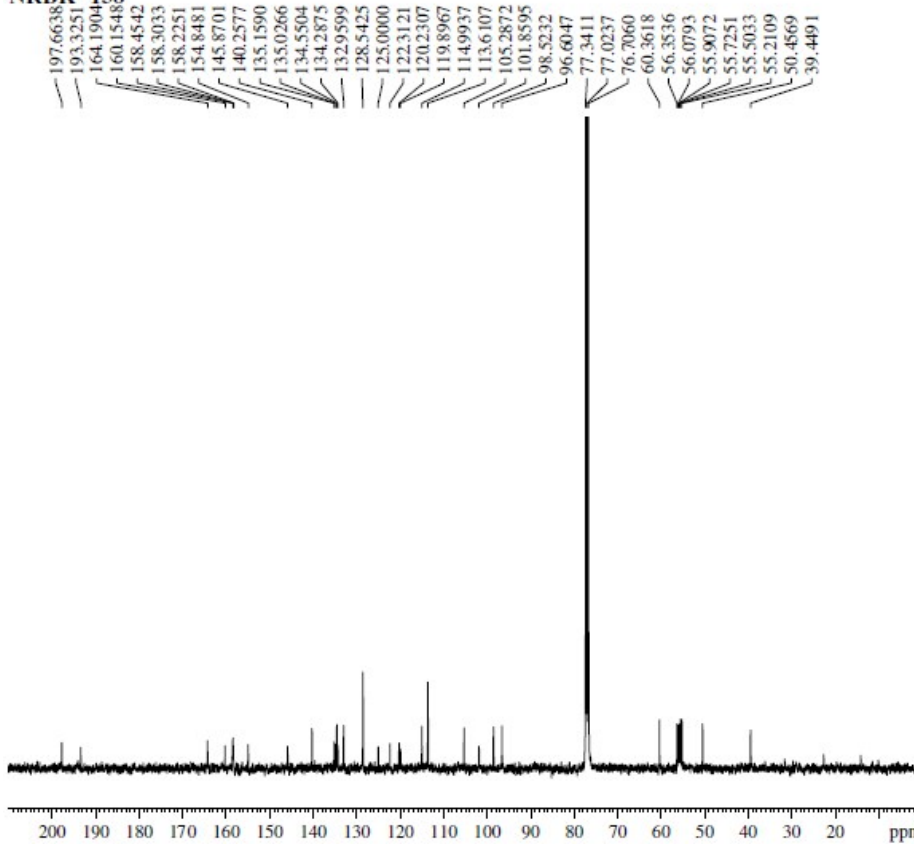
F2 - Acquisition Parameters
 Date_ 20171028
 Time 8.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 179.93
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1604994 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 71: ¹H NMR spectrum of 3x (expansion)

NRBK-138



Current Data Parameters
 NAME 03-NOV-FN-2017
 EXPNO 330
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171103
 Time 20.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

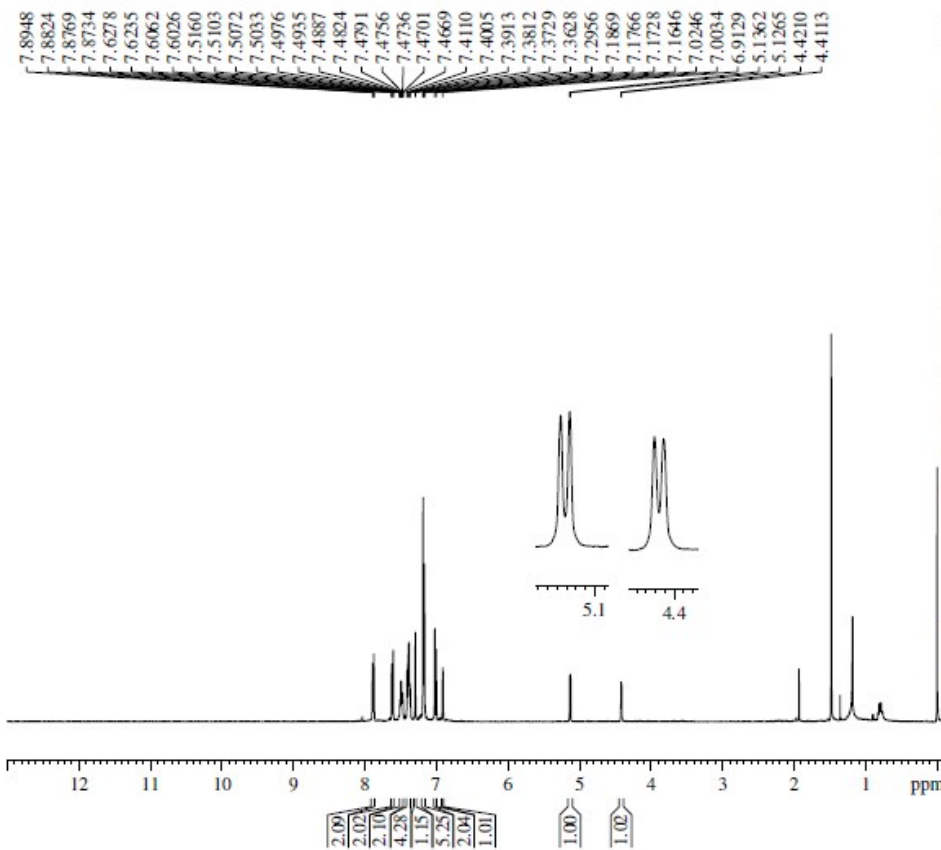
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

Figure 72: ¹³C NMR spectrum of 3x

NRBK-155



Current Data Parameters
 NAME 04-Oct-FN-2017
 EXPNO 340
 PROCNO 1

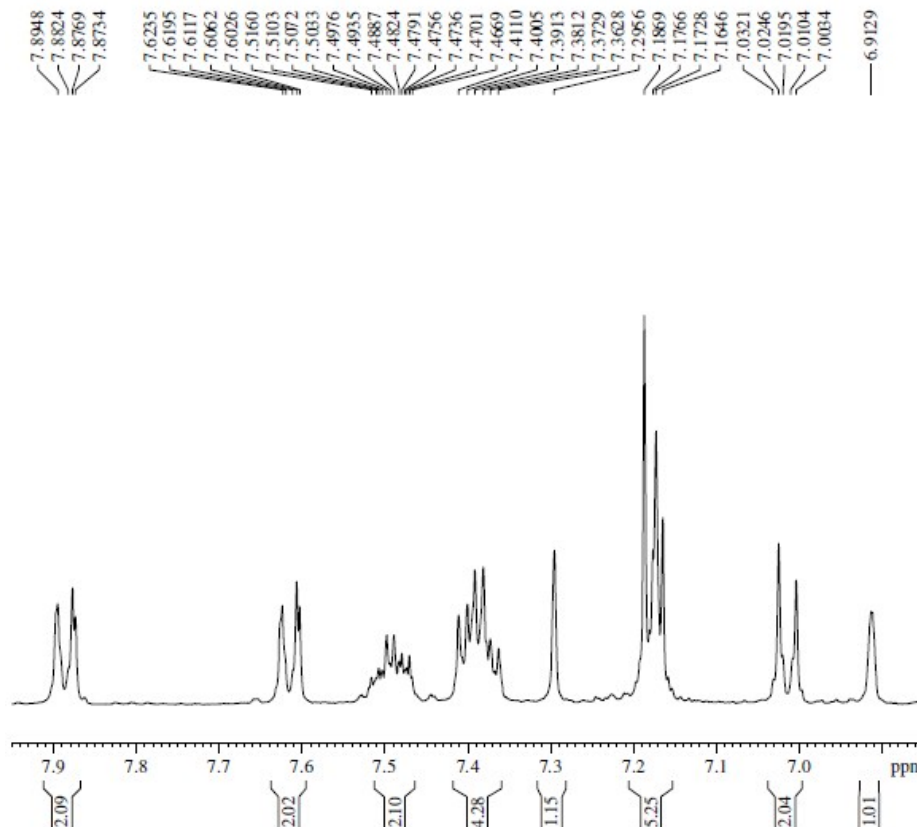
F2 - Acquisition Parameters
 Date_ 20171004
 Time 14.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 159.22
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605387 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 73: ¹H NMR spectrum of 3y

NRBK-155



Current Data Parameters
 NAME 04-Oct-FN-2017
 EXPNO 340
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171004
 Time 14.42
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 159.22
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605387 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 74: ¹H NMR spectrum of 3y (expansion)

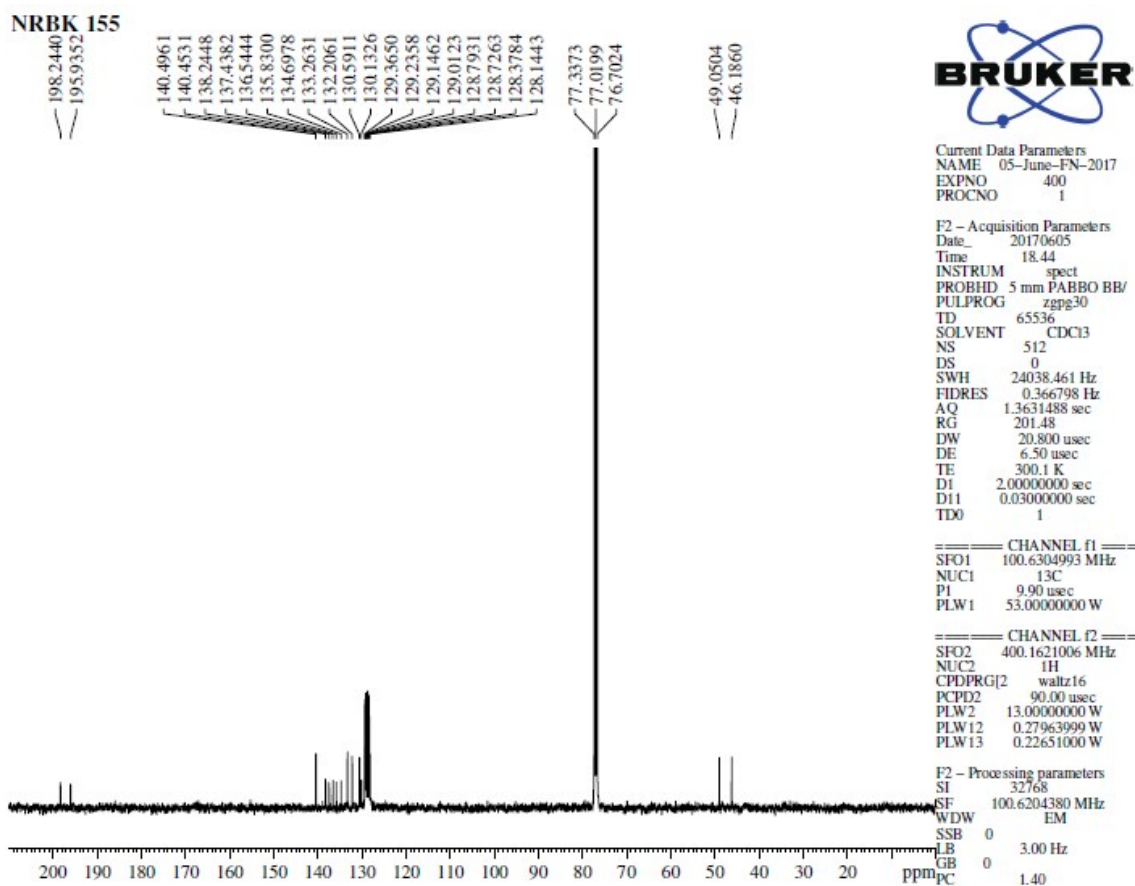


Figure 75: ^{13}C NMR spectrum of 3y

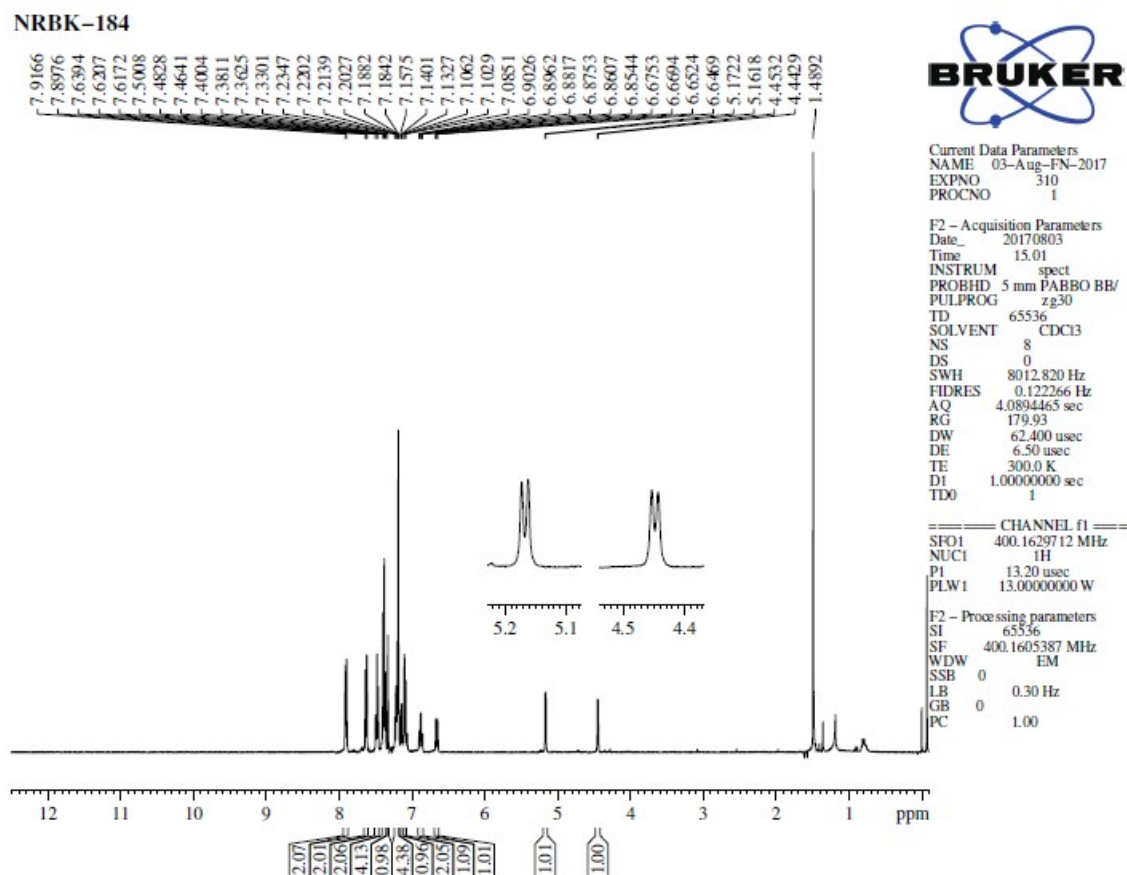
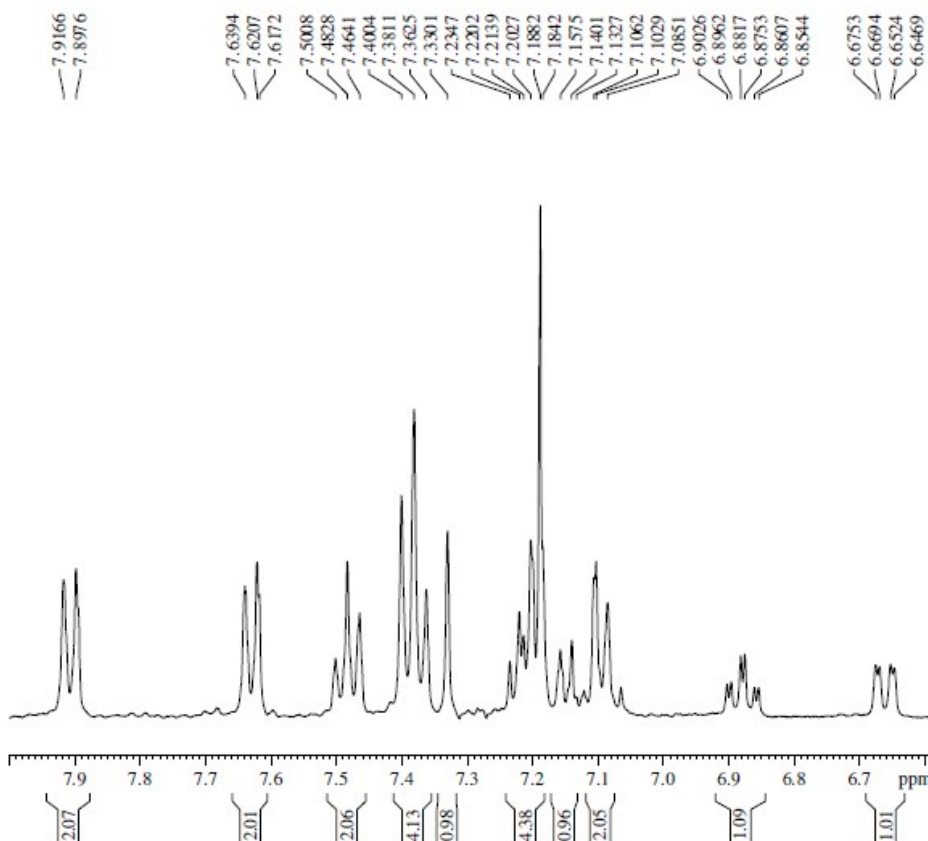


Figure 76: ^1H NMR spectrum of 3z

NRBK-184



Current Data Parameters
 NAME 03-Aug-FN-2017
 EXPNO 310
 PROCNO 1

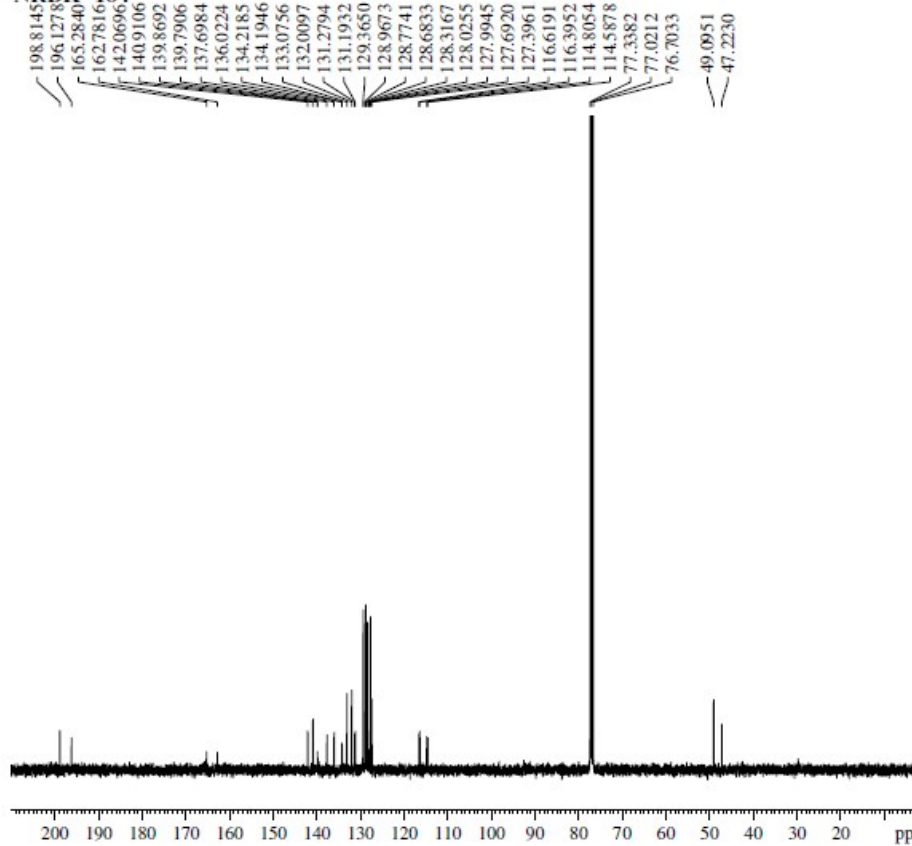
F2 - Acquisition Parameters
 Date_ 20170803
 Time 15.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 179.93
 DW 62.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605387 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 77: ¹H NMR spectrum of 3z (expansion)

NRBK-184



Current Data Parameters
 NAME 11-Aug-AN-2017
 EXPNO 450
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170812
 Time 17.57
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

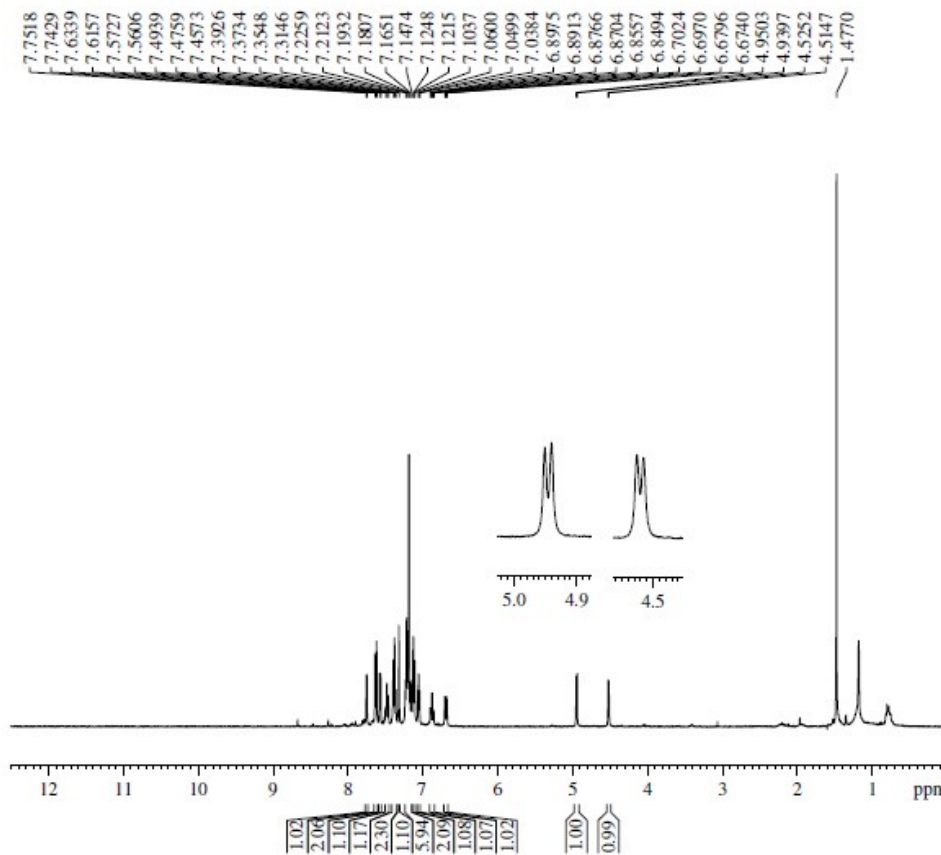
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 78: ¹³C NMR spectrum of 3z

NRBK-196



Current Data Parameters
NAME 24-aug-fn-2017
EXPNO 320
PROCNO 1

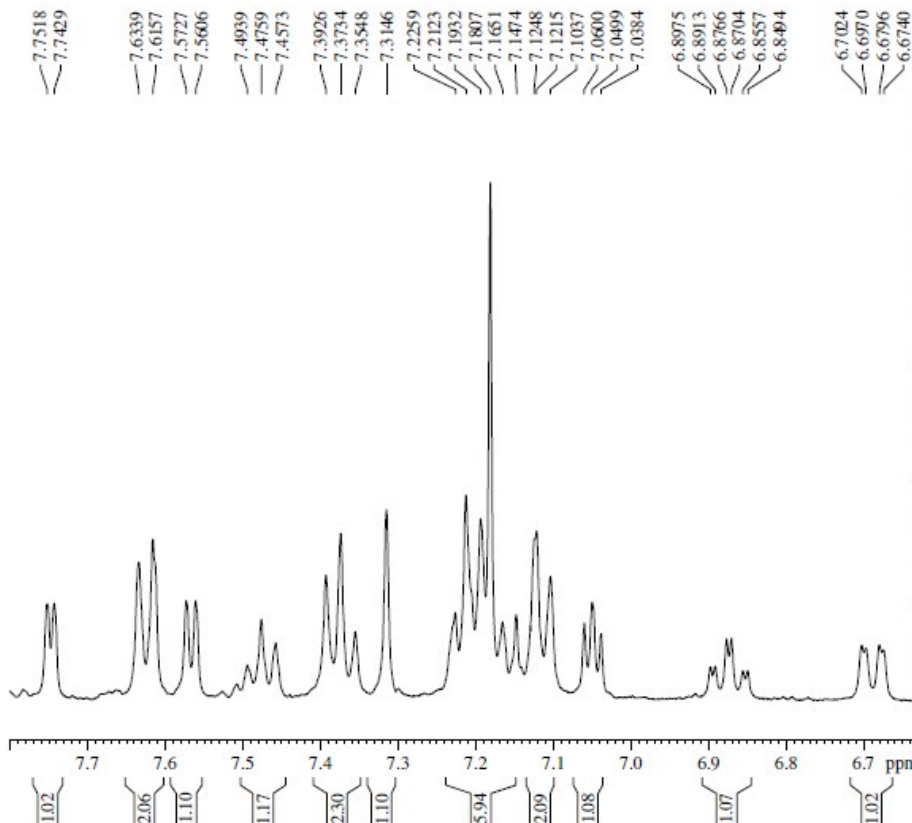
F2 - Acquisition Parameters
Date_ 20170824
Time 14.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 179.93
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605421 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 79: ¹H NMR spectrum of 3a

NRBK-196



Current Data Parameters
NAME 24-aug-fn-2017
EXPNO 320
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170824
Time 14.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 179.93
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605421 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 80: ¹H NMR spectrum of 3a (expansion)

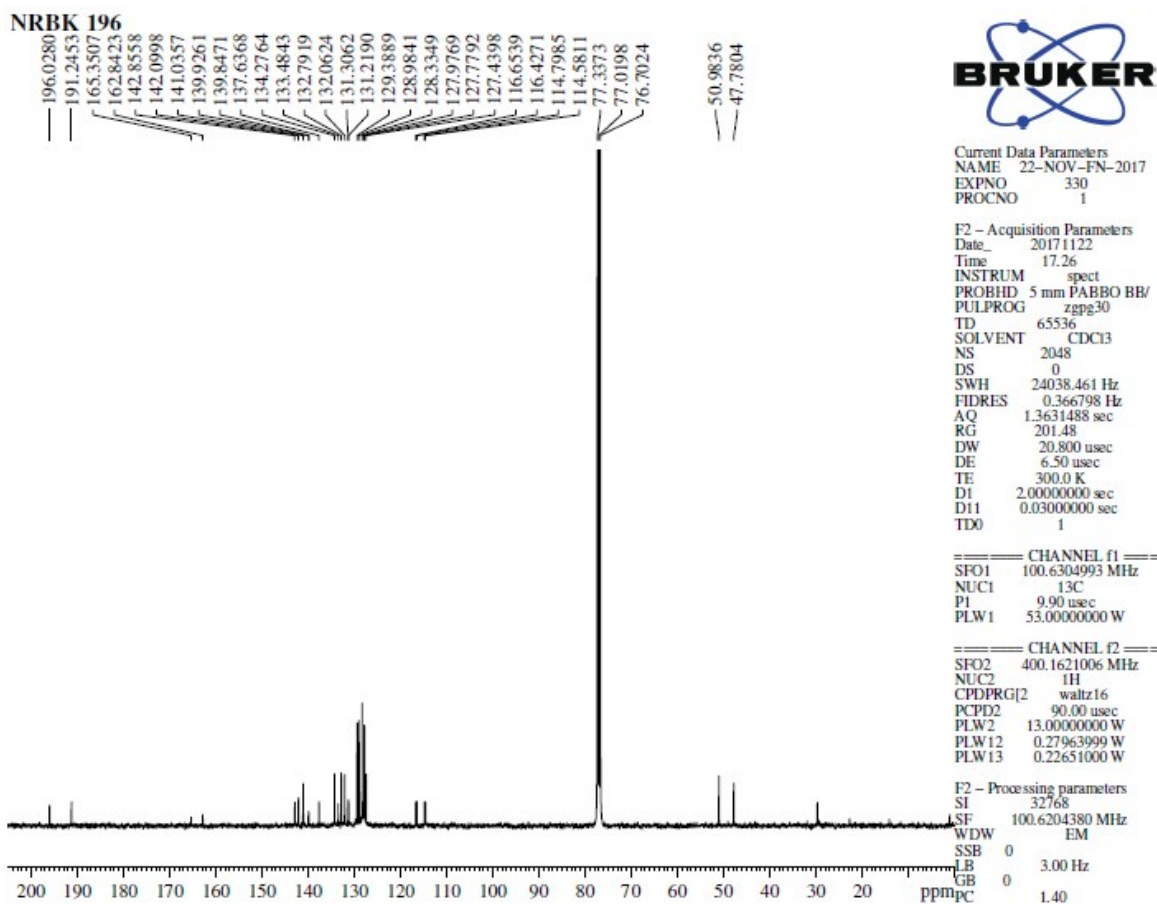


Figure 81: ¹³C NMR spectrum of 3a

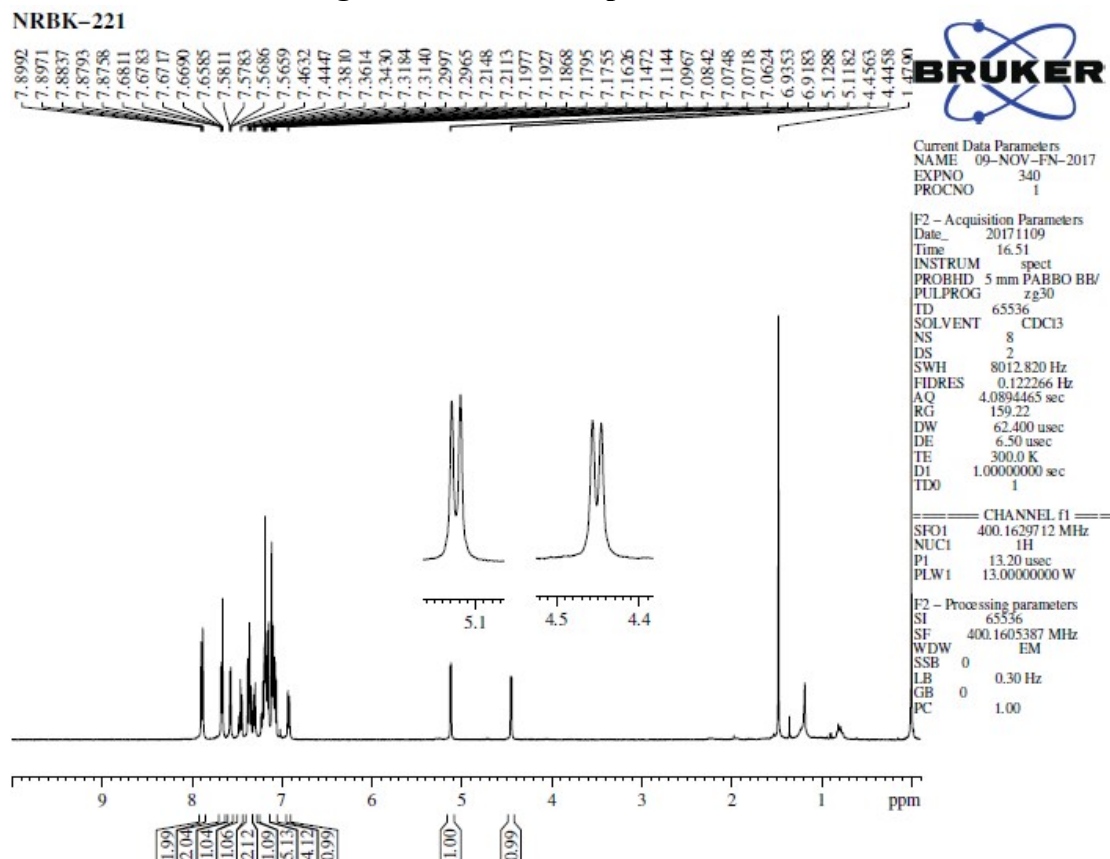
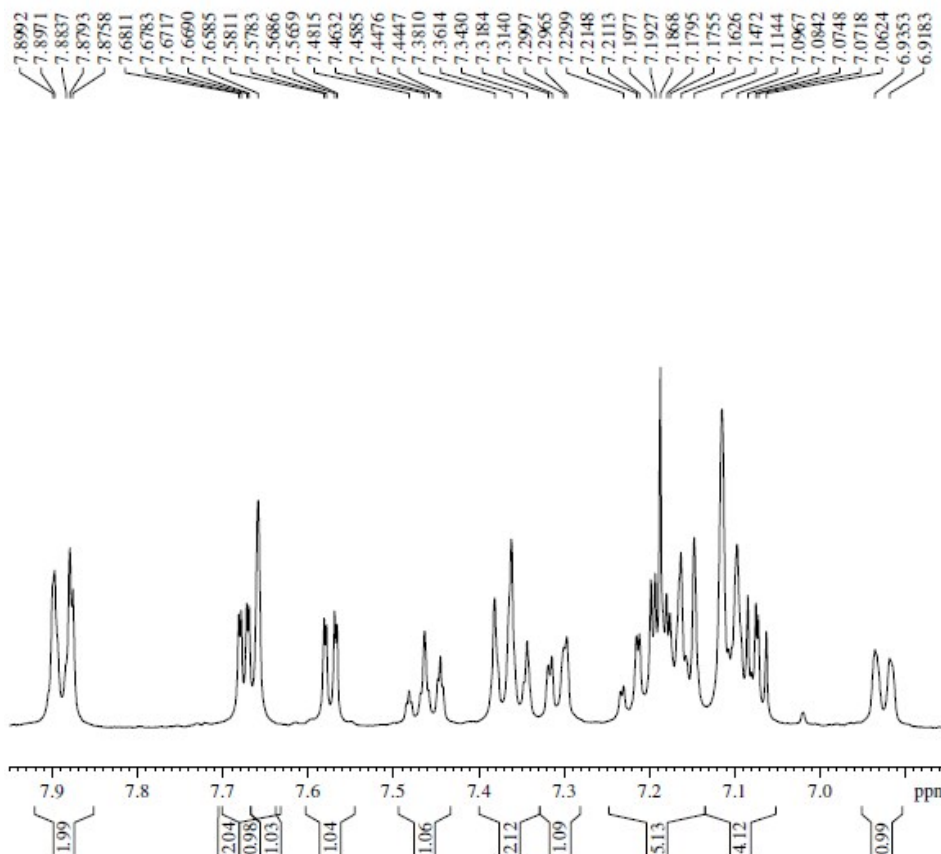


Figure 82: ¹H NMR spectrum of 3zb

NRBK-221



Current Data Parameters
NAME 09-NOV-FN-2017
EXPNO 340
PROCNO 1

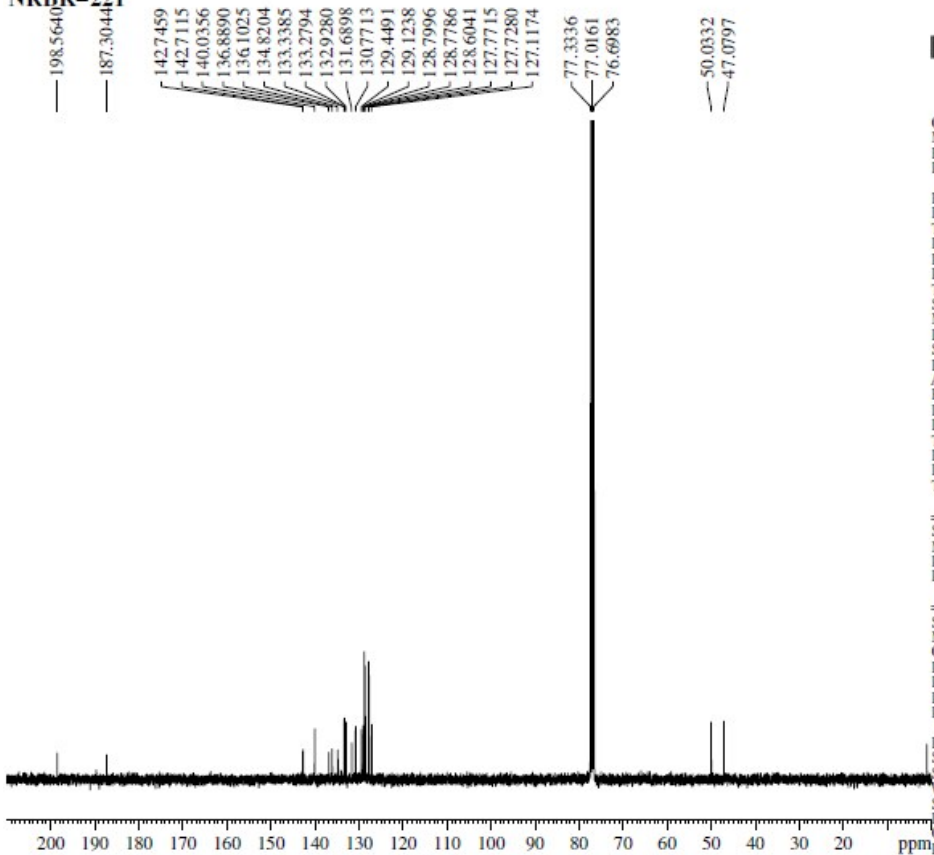
F2 - Acquisition Parameters
Date_ 20171109
Time 16.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 159.22
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605387 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 83: ¹H NMR spectrum of 3zb (expansion)

NRBK-221



Current Data Parameters
NAME 09-NOV-AN-2017
EXPNO 310
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171110
Time 7.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

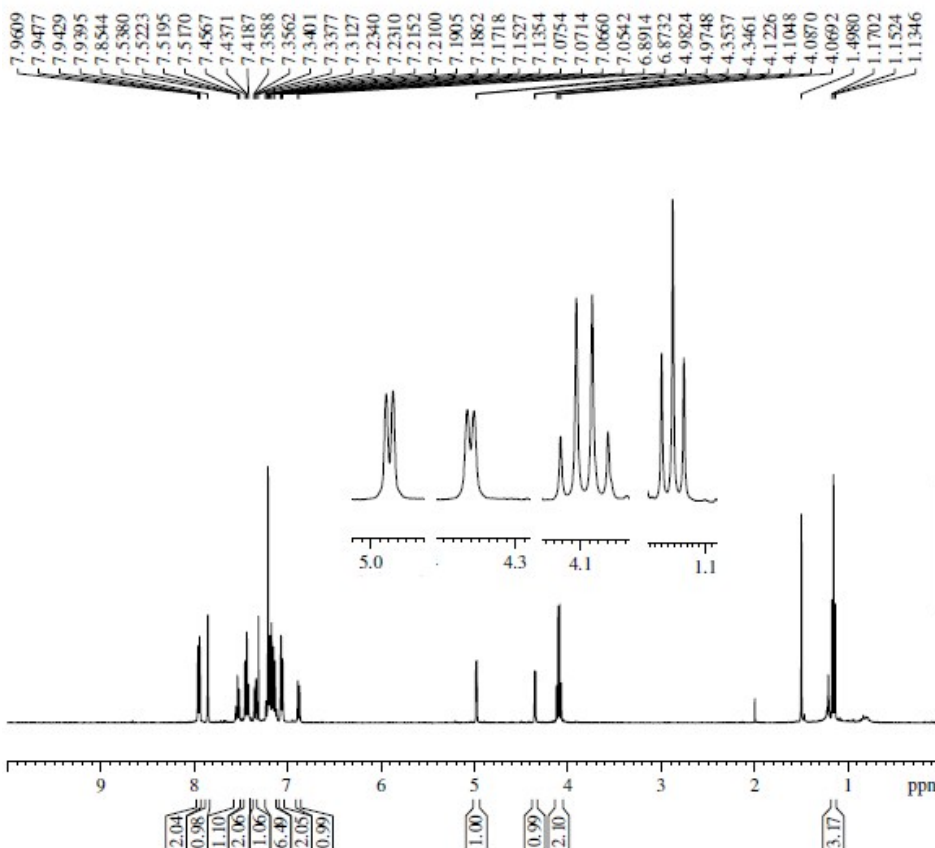
==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 13C
P1 9.90 usec
PLW1 53.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure 84: ¹³C NMR spectrum of 3zb

NRBK-219



Current Data Parameters
NAME 01-DEC-FN-2017
EXPNO 310
PROCNO 1

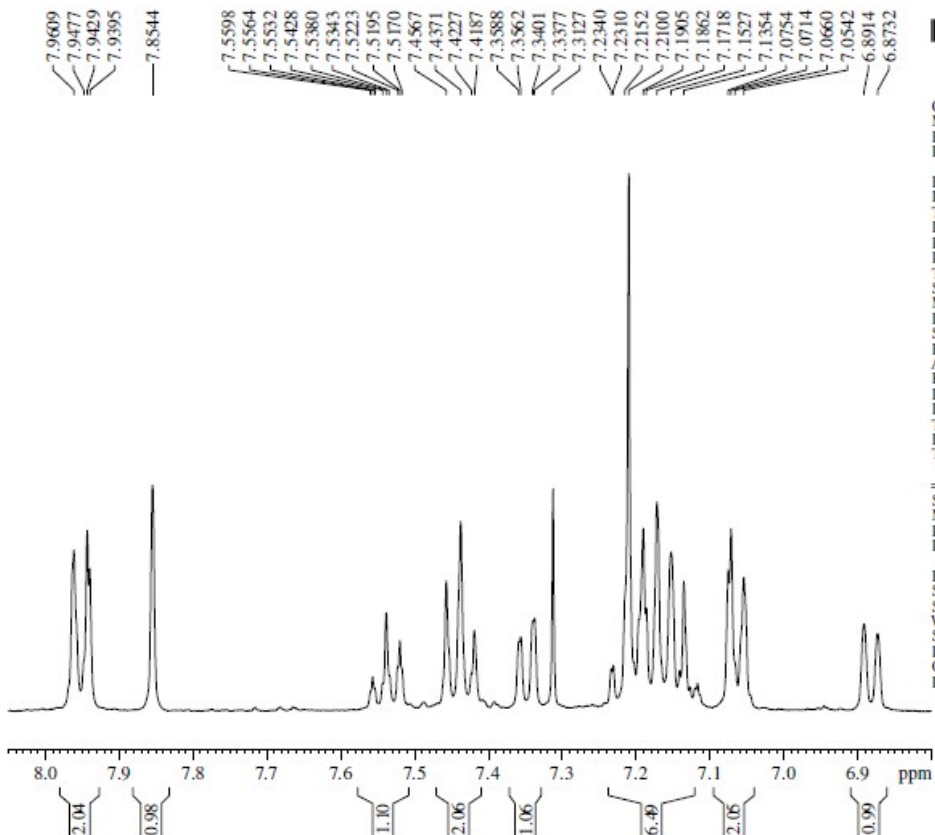
F2 - Acquisition Parameters
Date_ 20171201
Time 15.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 159.22
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605295 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 85: ¹H NMR spectrum of 3zc

NRBK-219



Current Data Parameters
NAME 01-DEC-FN-2017
EXPNO 310
PROCNO 1

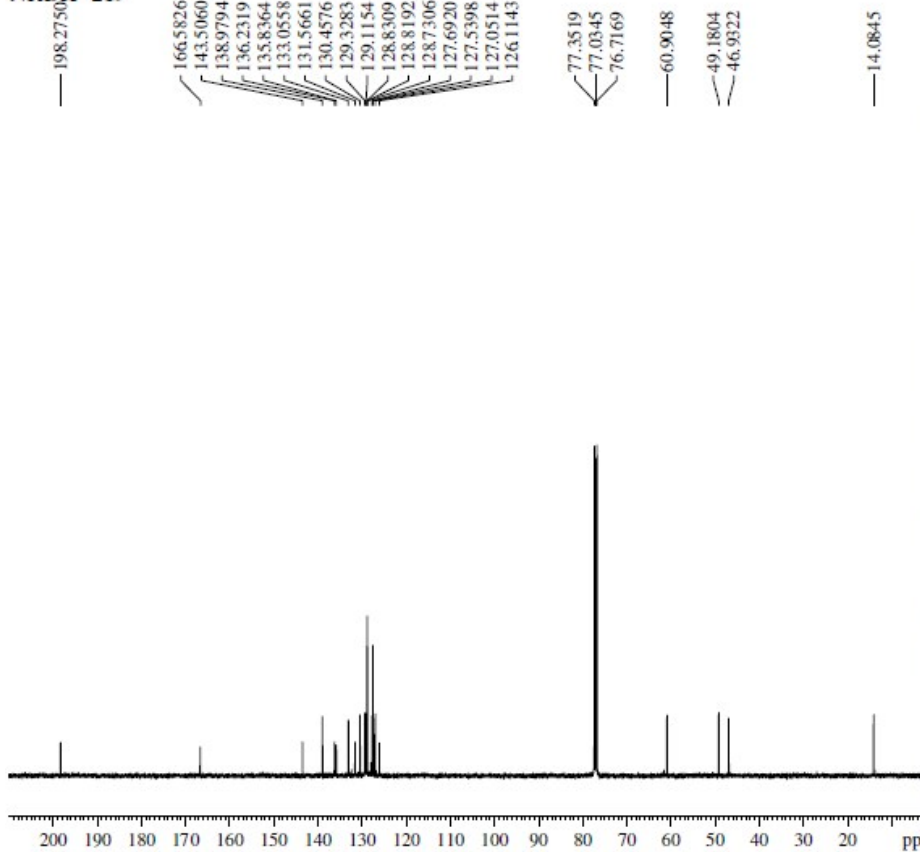
F2 - Acquisition Parameters
Date_ 20171201
Time 15.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 159.22
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605295 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 86: ¹H NMR spectrum of 3zc (expansion)

NRBK-219



Current Data Parameters
 NAME 09-OCT-FN-2017
 EXPNO 350
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20171009
 Time 21.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

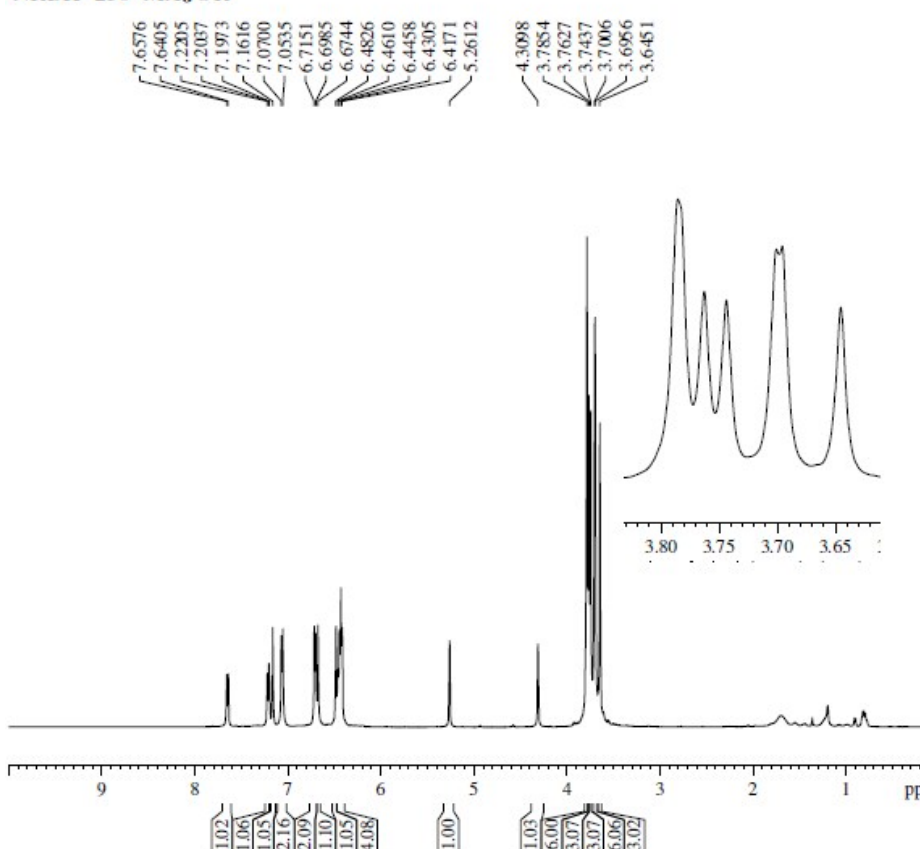
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 87: ¹³C NMR spectrum of 3zc

NRBK-256-MAJOR



Current Data Parameters
 NAME 09-APR-AN-2018
 EXPNO 310
 PROCNO 1

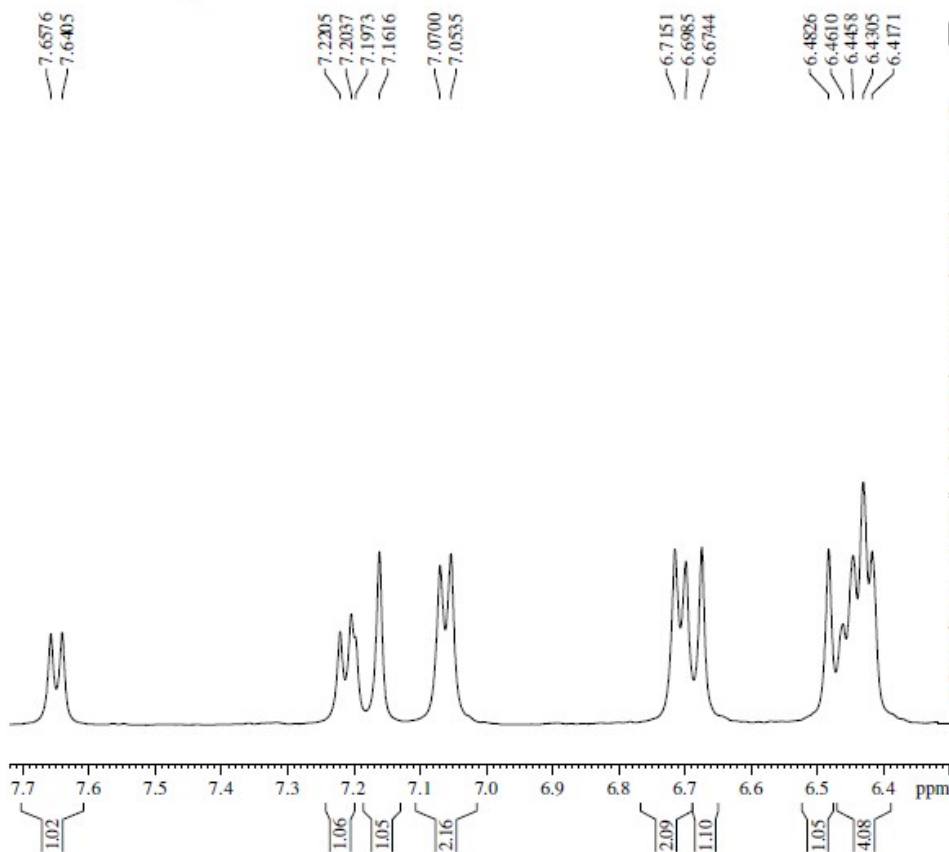
F2 - Acquisition Parameters
 Date_ 20180409
 Time 22.40
 INSTRUM spect
 PROBHD 5 mm PATXI 1H/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 10026.738 Hz
 FIDRES 0.152996 Hz
 AQ 3.2680619 sec
 RG 81.53
 DW 49.867 usec
 DE 6.50 usec
 TE 300.1 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 500.6794419 MHz
 NUC1 1H
 P1 6.45 usec
 PLW1 13.60000038 W

F2 - Processing parameters
 SI 65536
 SF 500.6763930 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 88: ¹H NMR spectrum of 3zd

NRBK-256-MAJOR



Current Data Parameters
NAME 09-APR-AN-2018
EXPNO 310
PROCNO 1

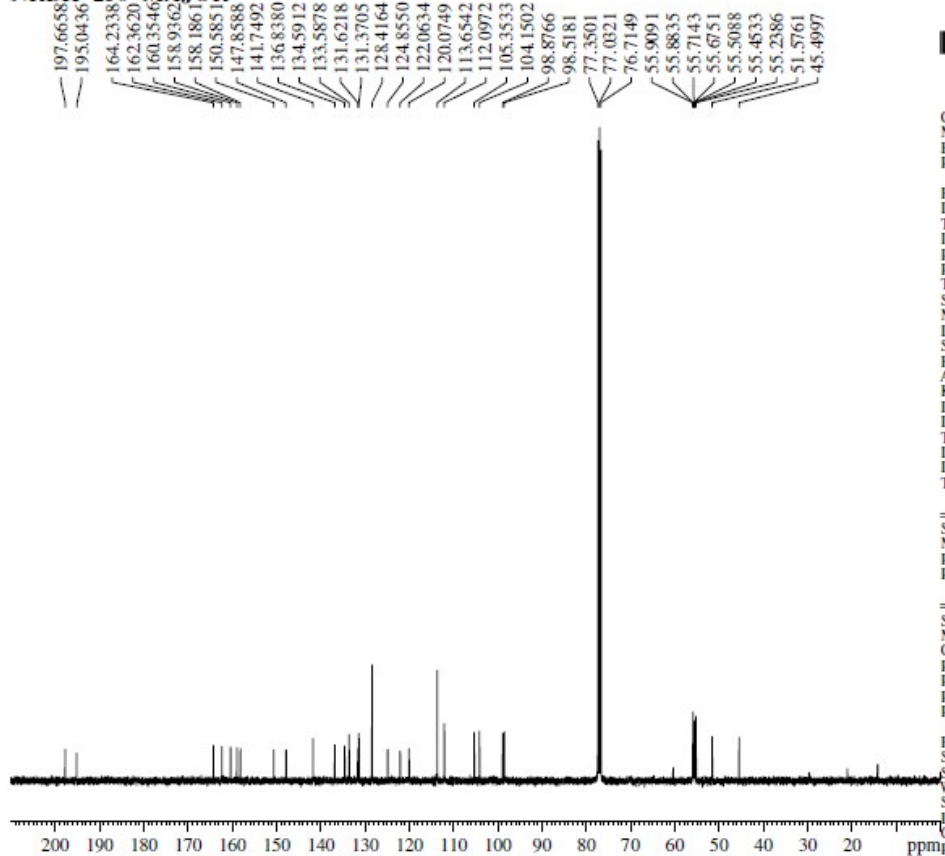
F2 - Acquisition Parameters
Date_ 20180409
Time 22.40
INSTRUM spect
PROBHD 5 mm PATX1 1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10026.738 Hz
FIDRES 0.152996 Hz
AQ 3.2680619 sec
RG 81.53
DW 49.867 usec
DE 6.50 usec
TE 300.1 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 500.6794419 MHz
NUC1 1H
P1 6.45 usec
PLW1 13.60000038 W

F2 - Processing parameters
SI 65356
SF 500.6763930 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 89: ¹H NMR spectrum of 3zd (expansion)

NRBK-256-MAJOR



Current Data Parameters
NAME 26-FEB-FN-2018
EXPNO 390
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180227
Time 2.43
INSTRUM spect
PROBHD 5 mm FABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

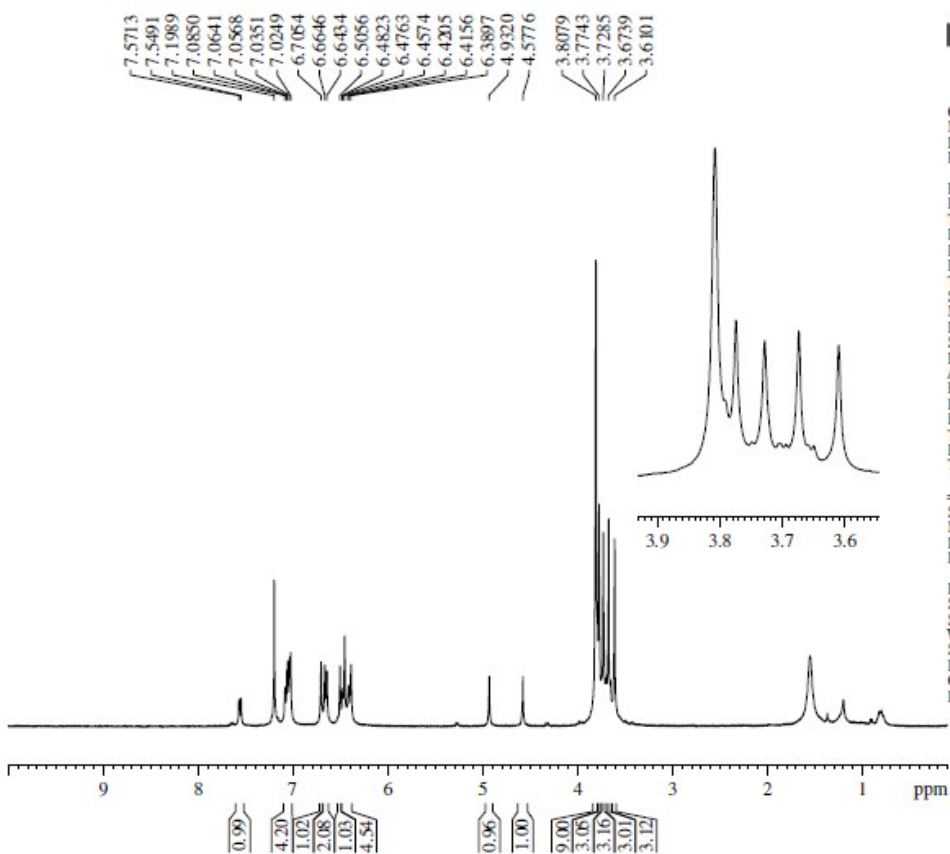
==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 13C
P1 9.90 usec
PLW1 53.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure 90: ¹³C NMR spectrum of 3zd

NRBK-256-minor



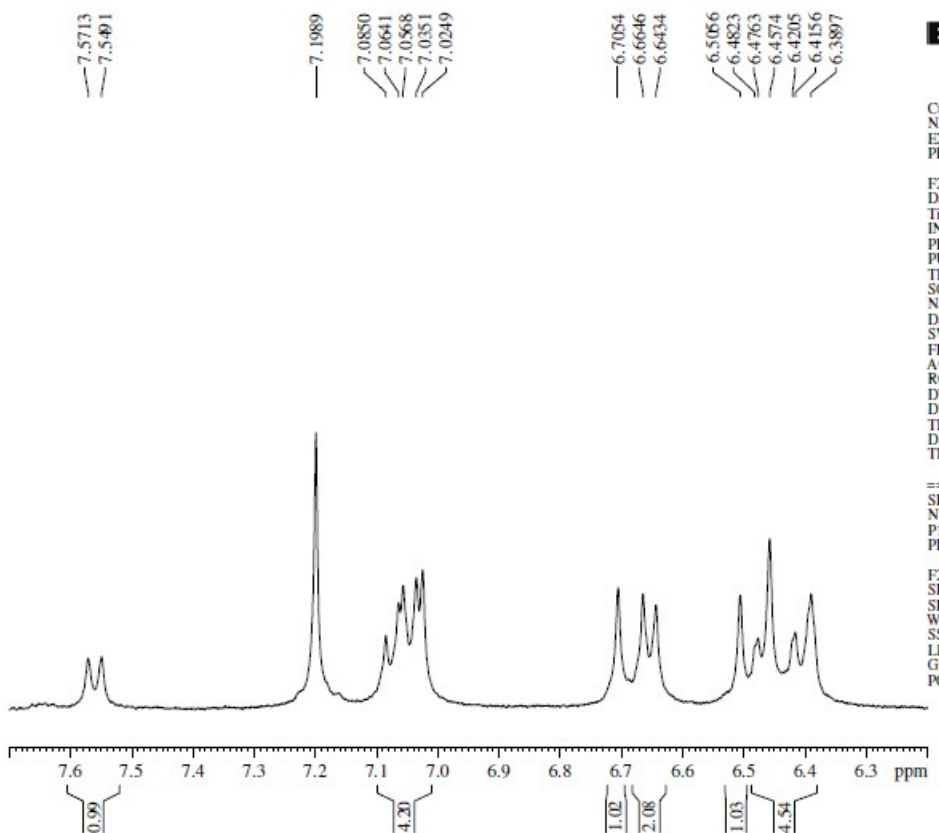
Current Data Parameters
 NAME 26-Apr-FN-2018
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180426
 Time 14.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 159.22
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 400.1605336 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 91: ¹H NMR spectrum of 3ze

NRBK-256-minor



Current Data Parameters
 NAME 26-Apr-FN-2018
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180426
 Time 14.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 159.22
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 400.1605336 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 92: ¹H NMR spectrum of 3ze (expansion)

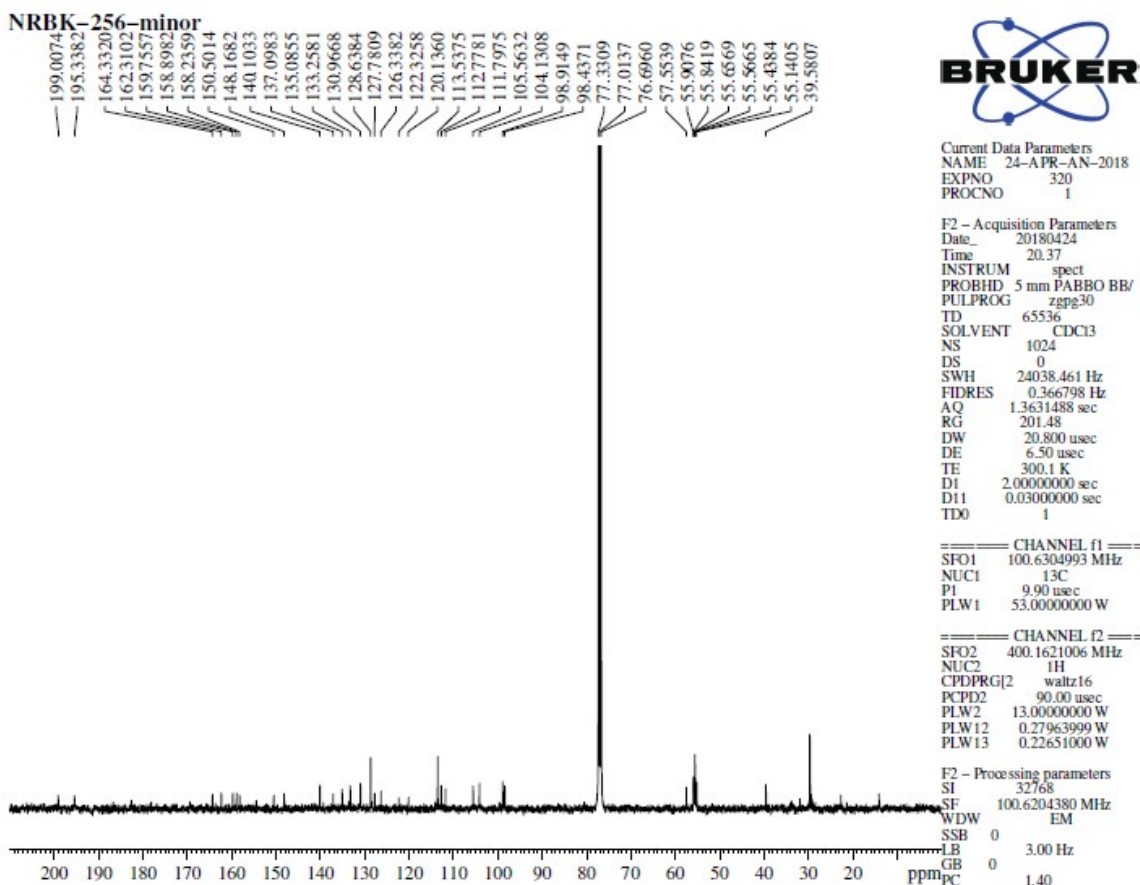


Figure 93: ^{13}C NMR spectrum of 3ze

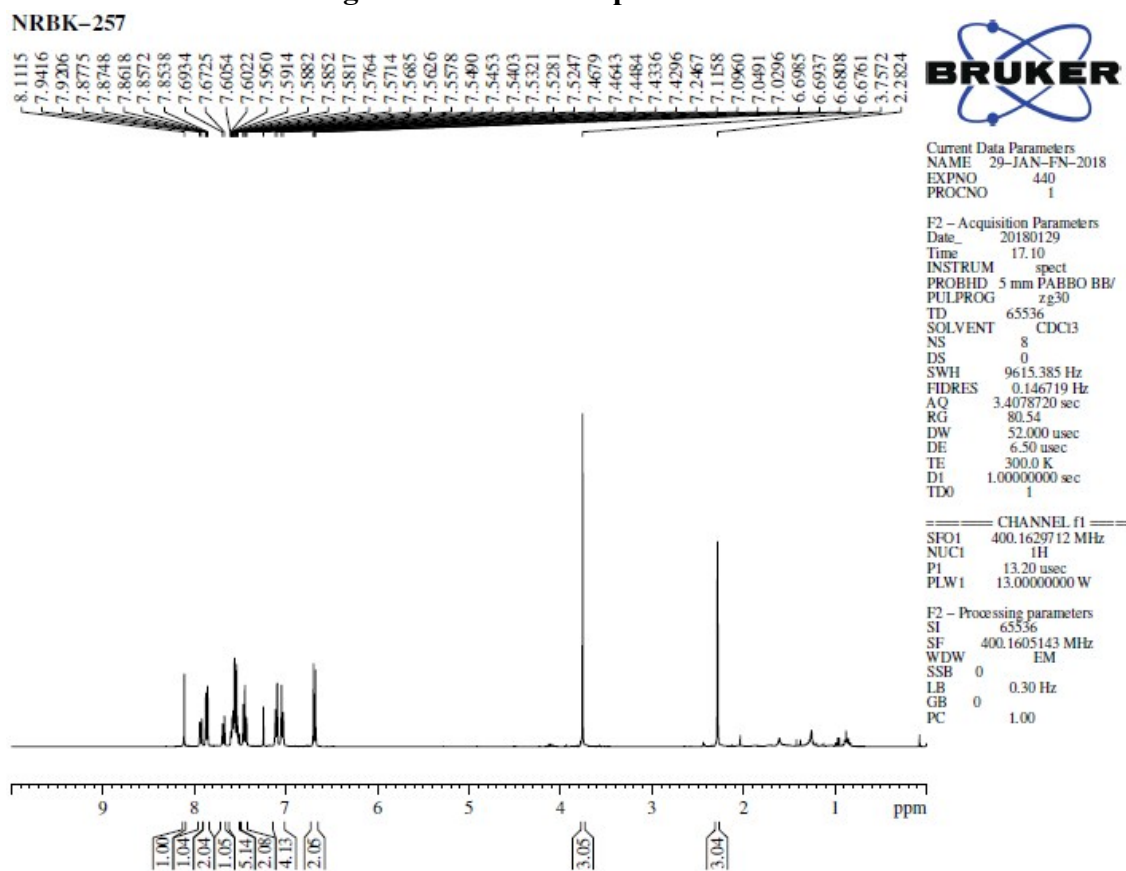


Figure 94: ^1H NMR spectrum of 4a

NRBK-257

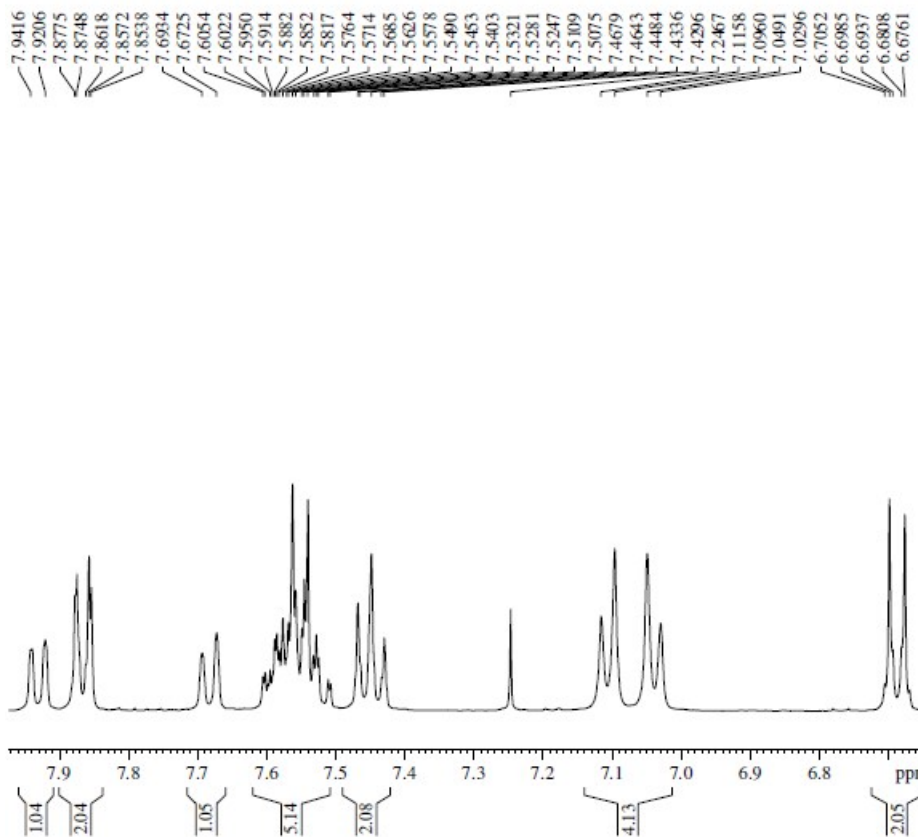


Figure 95: ¹H NMR spectrum of 4a (expansion)



Current Data Parameters
NAME 29-JAN-FN-2018
EXPNO 440
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180129
Time 17.10
INSTRUM spect
PROBHD 5 mm PABBO BE
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 80.54
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.0000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605143 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NRBK-257

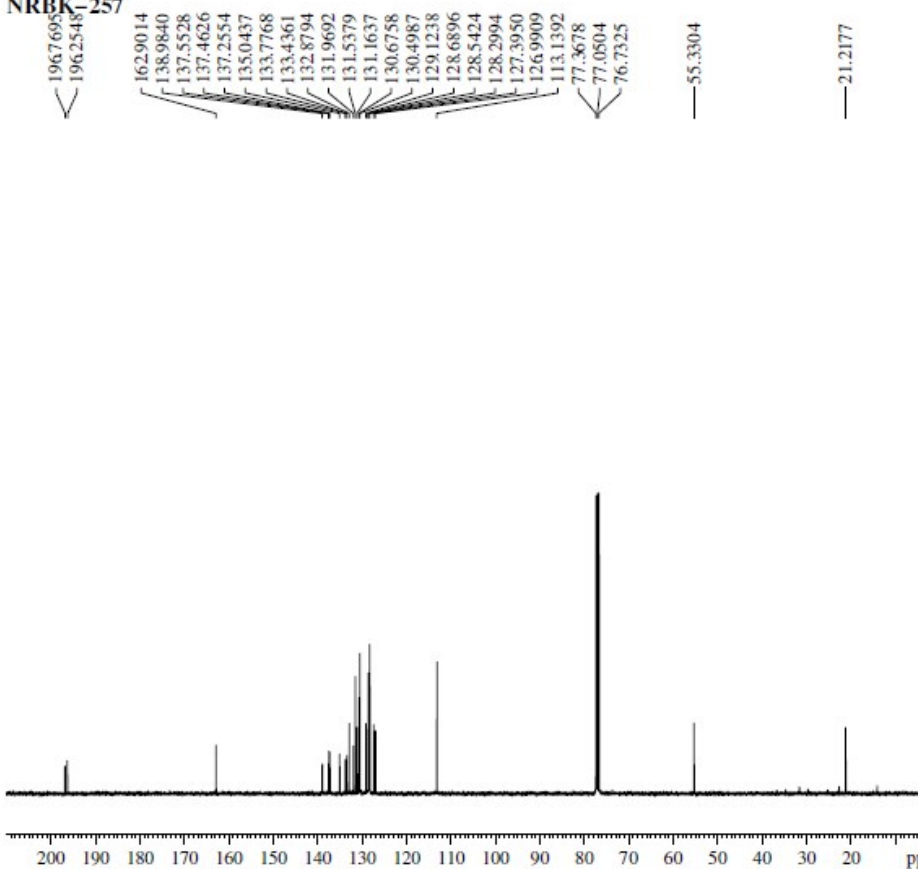


Figure 96: ¹³C NMR spectrum of 4a



Current Data Parameters
NAME 31-JAN-FN-2018
EXPNO 330
PROCNO 1

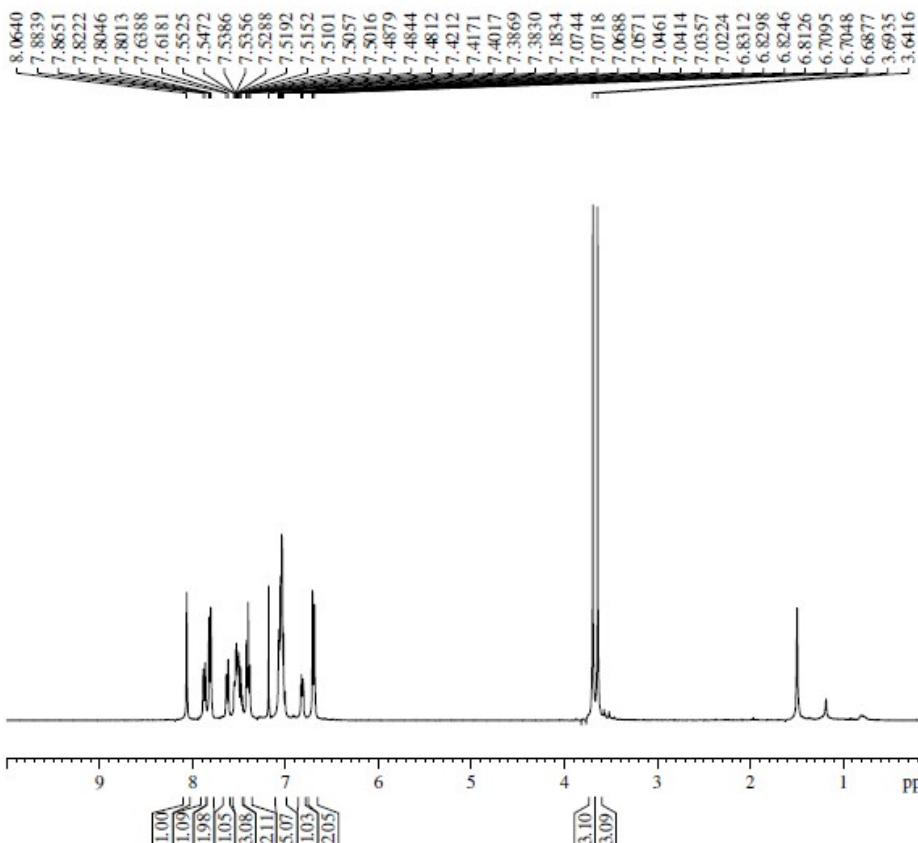
F2 - Acquisition Parameters
Date_ 20180201
Time 1.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 13C
P1 9.90 usec
PLW1 53.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
PCPD2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

NRBK-249



Current Data Parameters
NAME 28-FEB-FN-2018
EXPNO 310
PROCNO 1

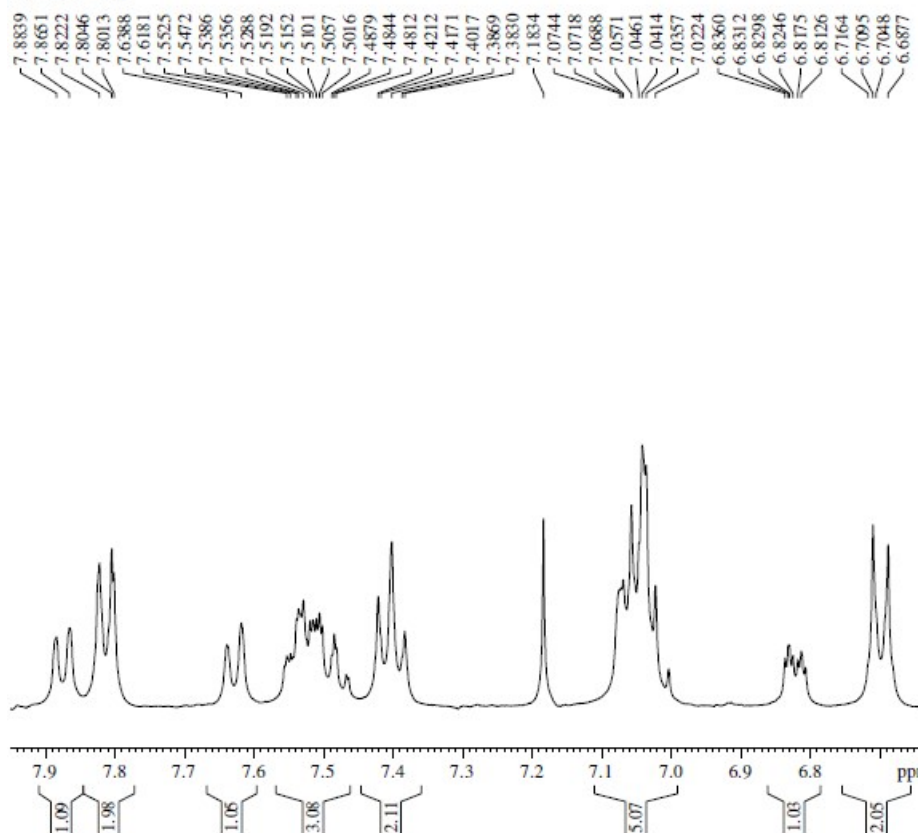
F2 - Acquisition Parameters
Date_ 20180228
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 129.57
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605401 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 97: ¹H NMR spectrum of 4b

NRBK-249



Current Data Parameters
NAME 28-FEB-FN-2018
EXPNO 310
PROCNO 1

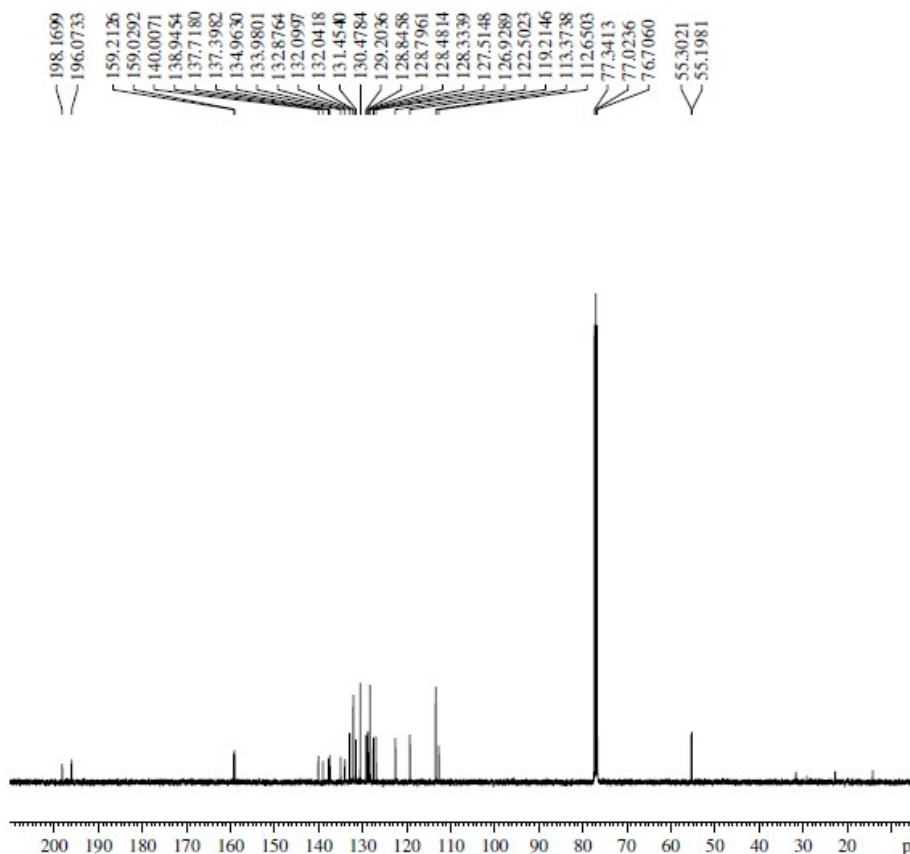
F2 - Acquisition Parameters
Date_ 20180228
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 129.57
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605401 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 98: ¹H NMR spectrum of 4b (expansion)

NRBK-249



Current Data Parameters
 NAME 24-JAN-FN-2018
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180124
 Time 17.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

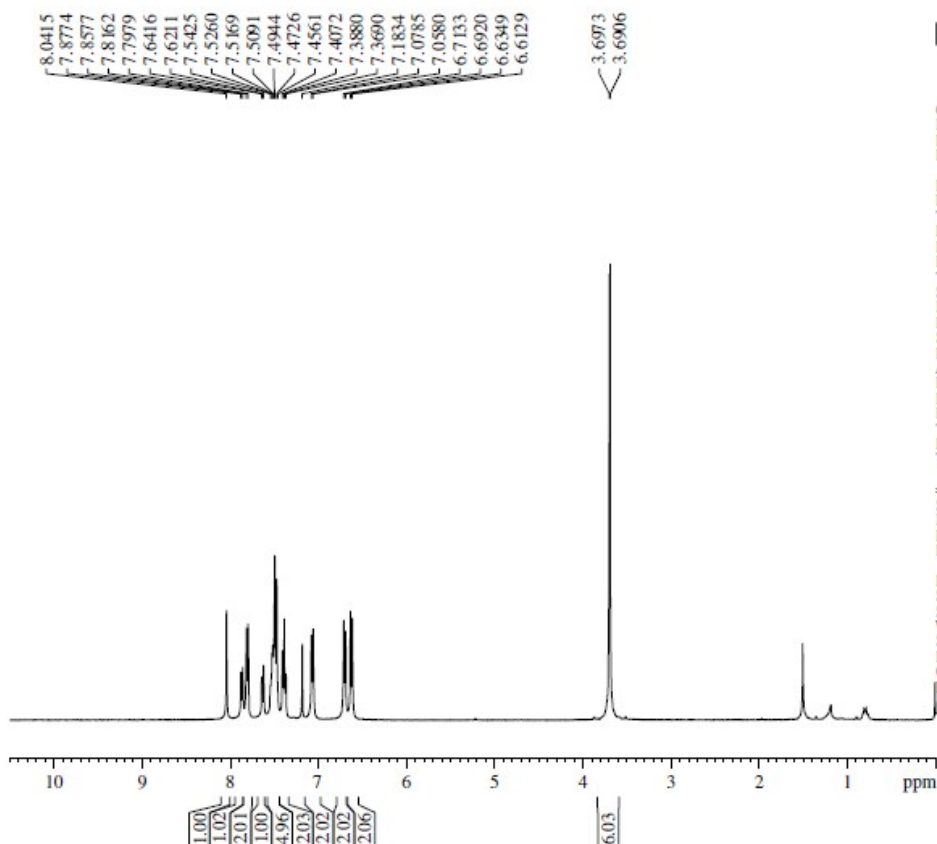
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 99: ¹³C NMR spectrum of 4b

NRBK-229



Current Data Parameters
 NAME 12-FEB-FN-2018
 EXPNO 310
 PROCNO 1

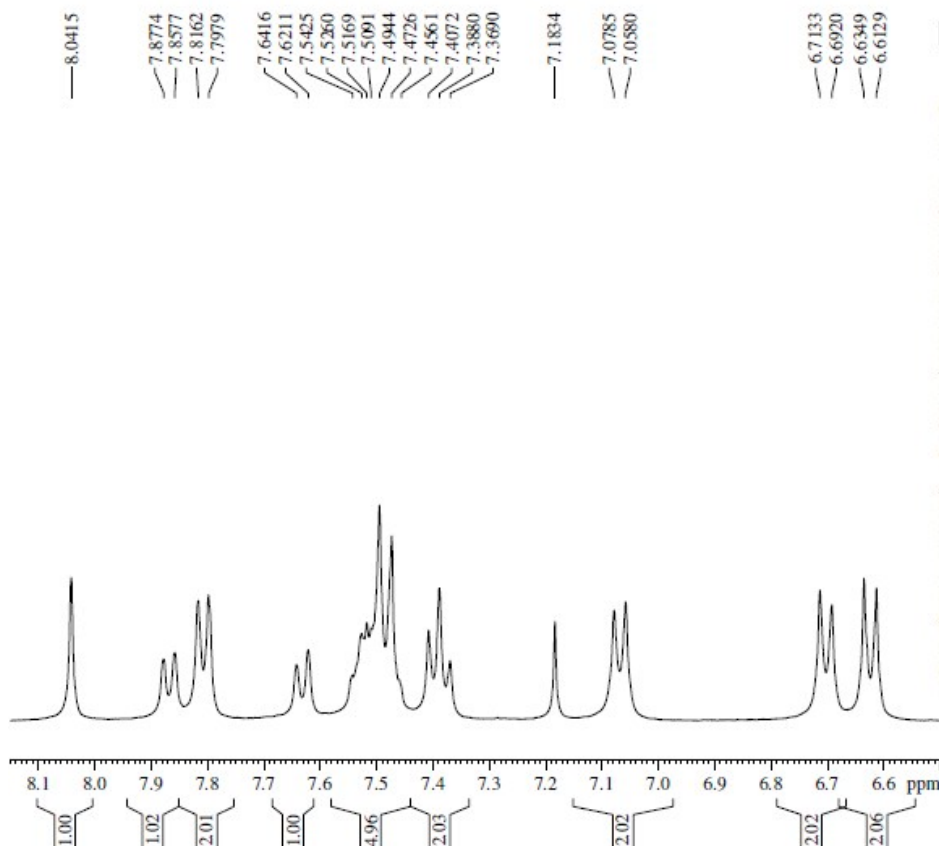
F2 - Acquisition Parameters
 Date_ 20180212
 Time 16.52
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605397 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 100: ¹H NMR spectrum of 4c

NRBK-229



Current Data Parameters
 NAME 12-FEB-FN-2018
 EXPNO 310
 PROCNO 1

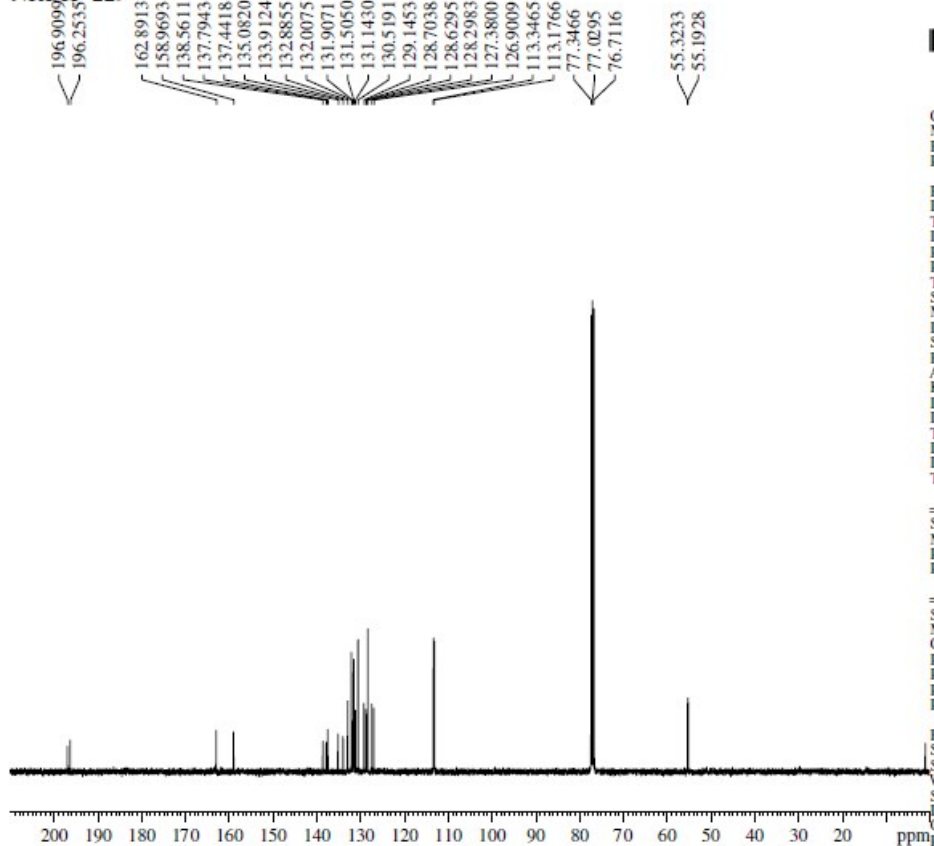
F2 - Acquisition Parameters
 Date_ 20180212
 Time 16.52
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605397 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 101: ¹H NMR spectrum of 4c (expansion)

NRBK-229



Current Data Parameters
 NAME 31-JAN-FN-2018
 EXPNO 350
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180201
 Time 2.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 102: ¹³C NMR spectrum of 4c

NRBK-250

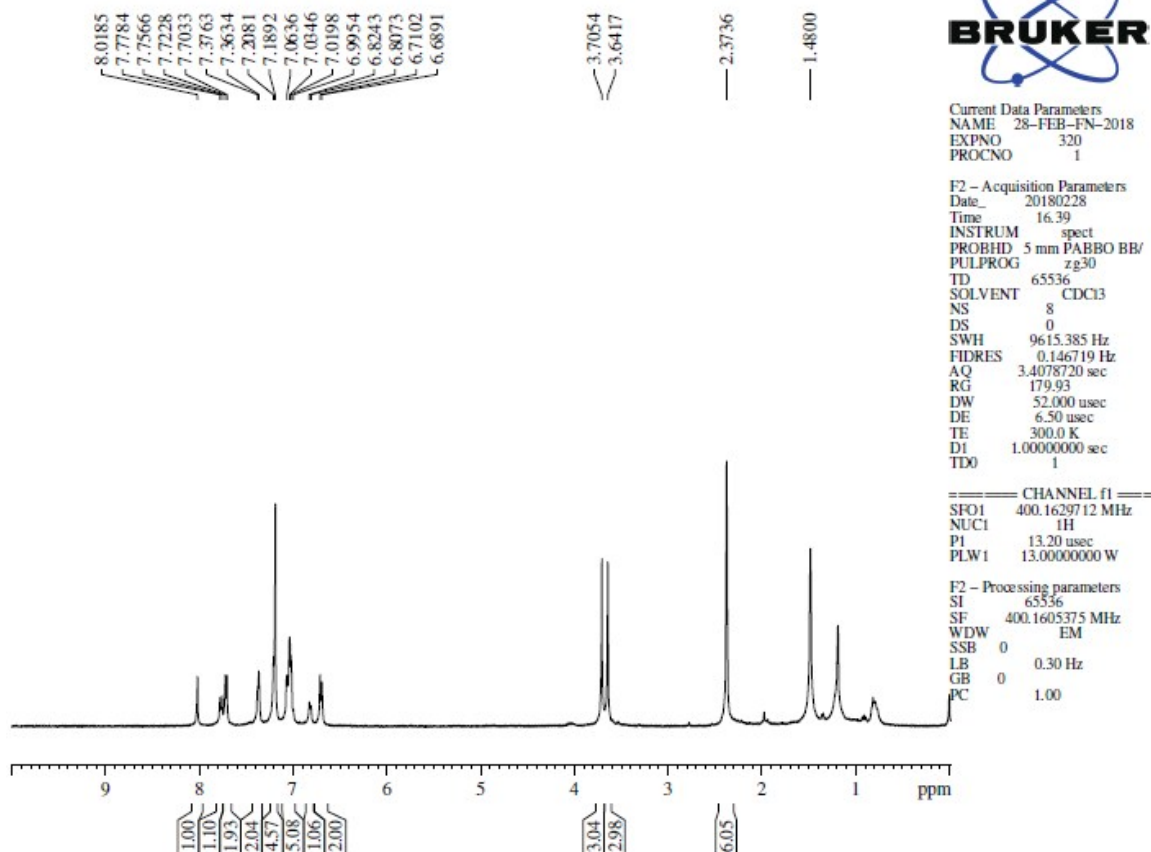


Figure 103: ¹H NMR spectrum of 4d

NRBK-250

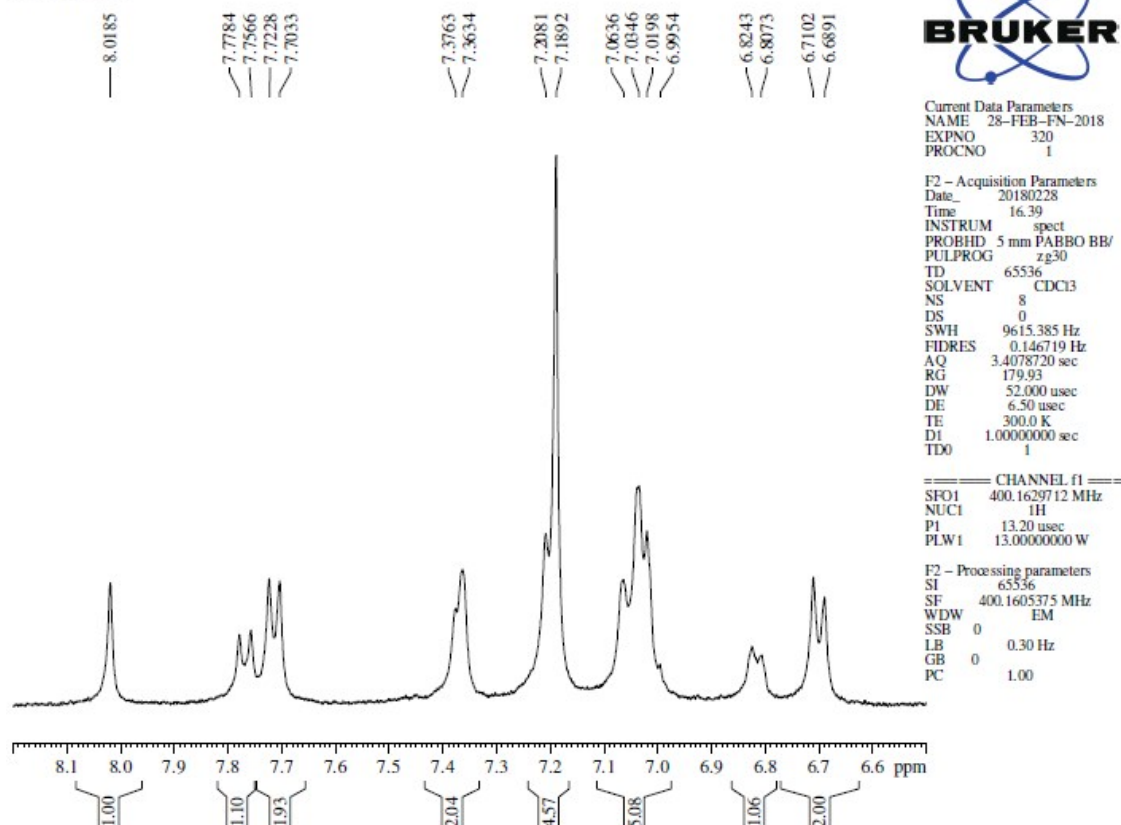


Figure 104: ¹H NMR spectrum of 4d (expansion)

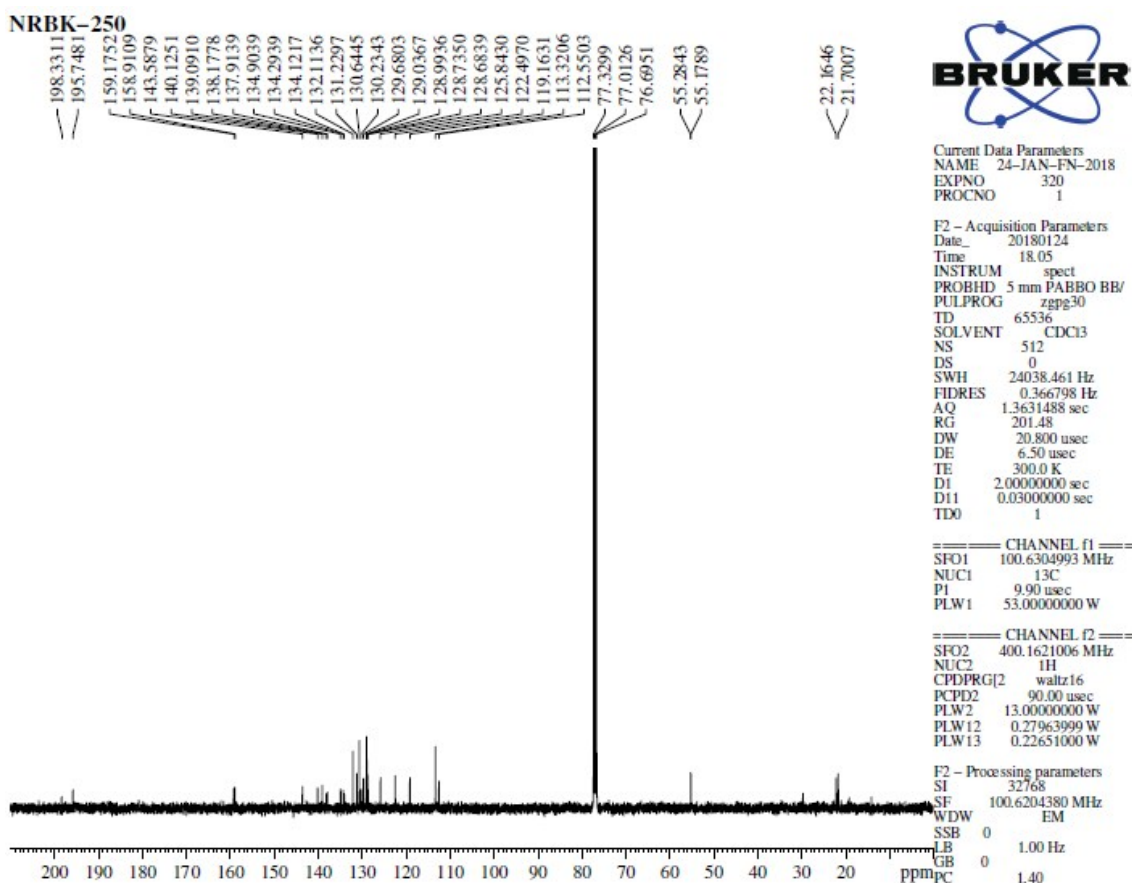


Figure 105: ¹³C NMR spectrum of 4d

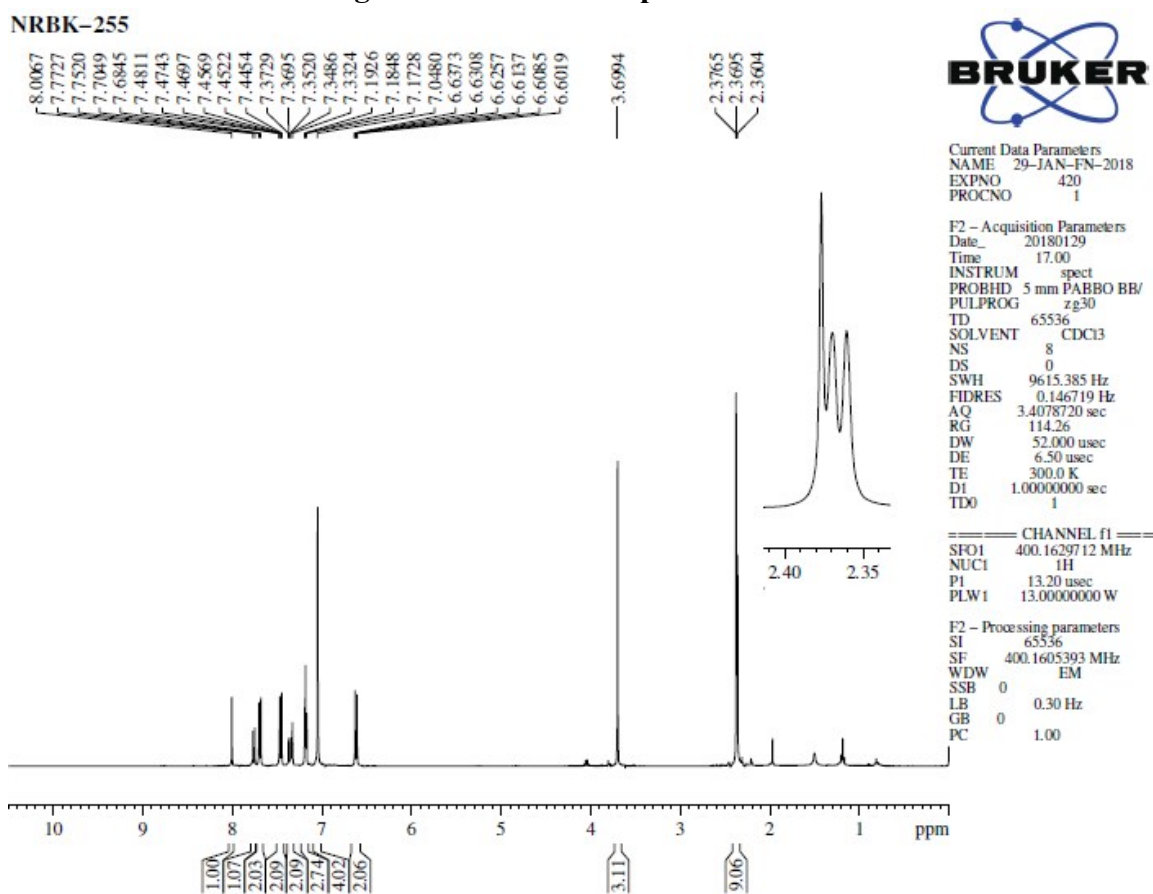
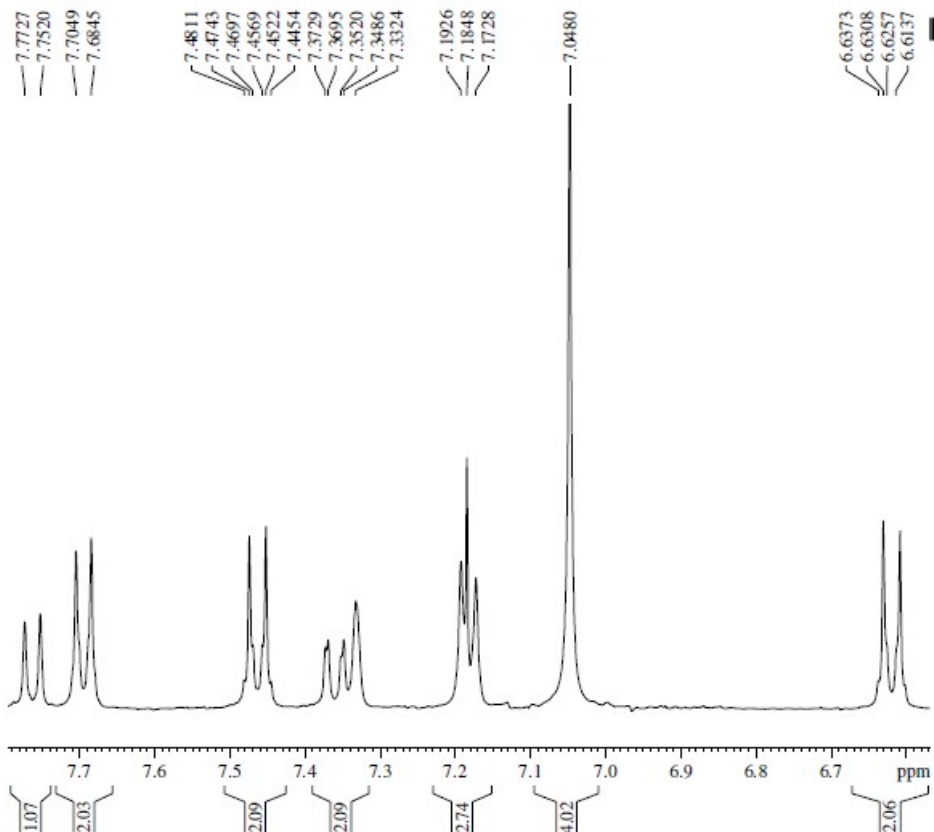


Figure 106: ¹H NMR spectrum of 4e

NRBK-255



Current Data Parameters
 NAME 29-JAN-FN-2018
 EXPNO 420
 PROCNO 1

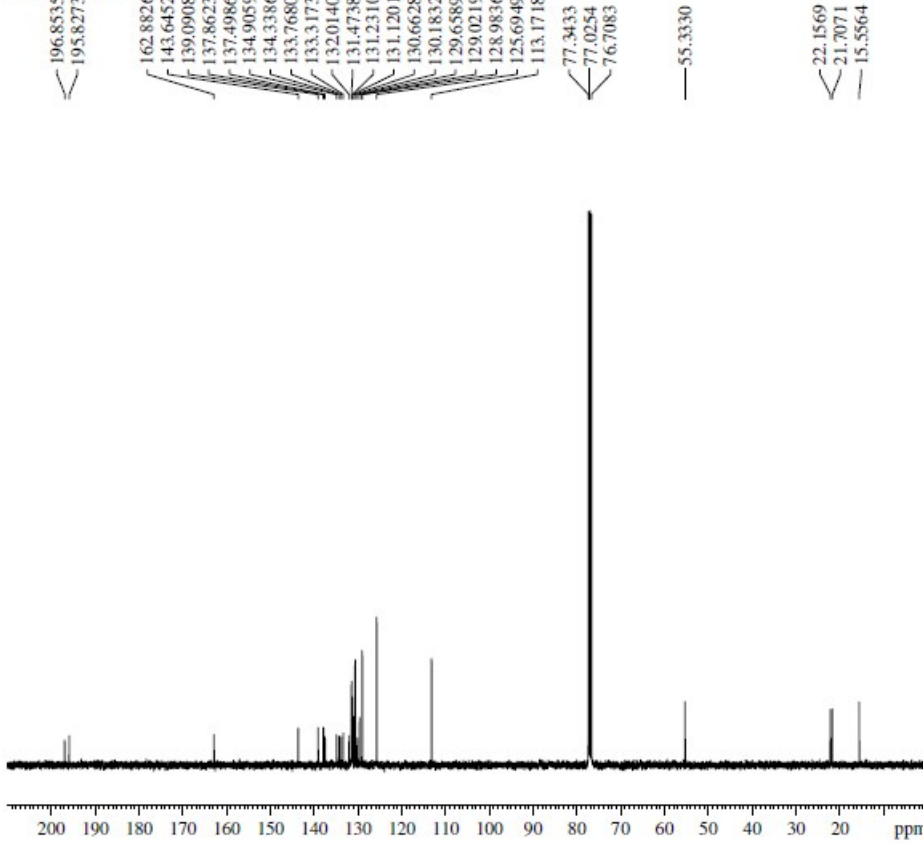
F2 - Acquisition Parameters
 Date_ 20180129
 Time 17.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605393 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 107: ¹H NMR spectrum of 4e (expansion)

NRBK-255



Current Data Parameters
 NAME 31-JAN-FN-2018
 EXPNO 320
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180201
 Time 1.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CDPDRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 108: ¹³C NMR spectrum of 4e

NRBK-272-major

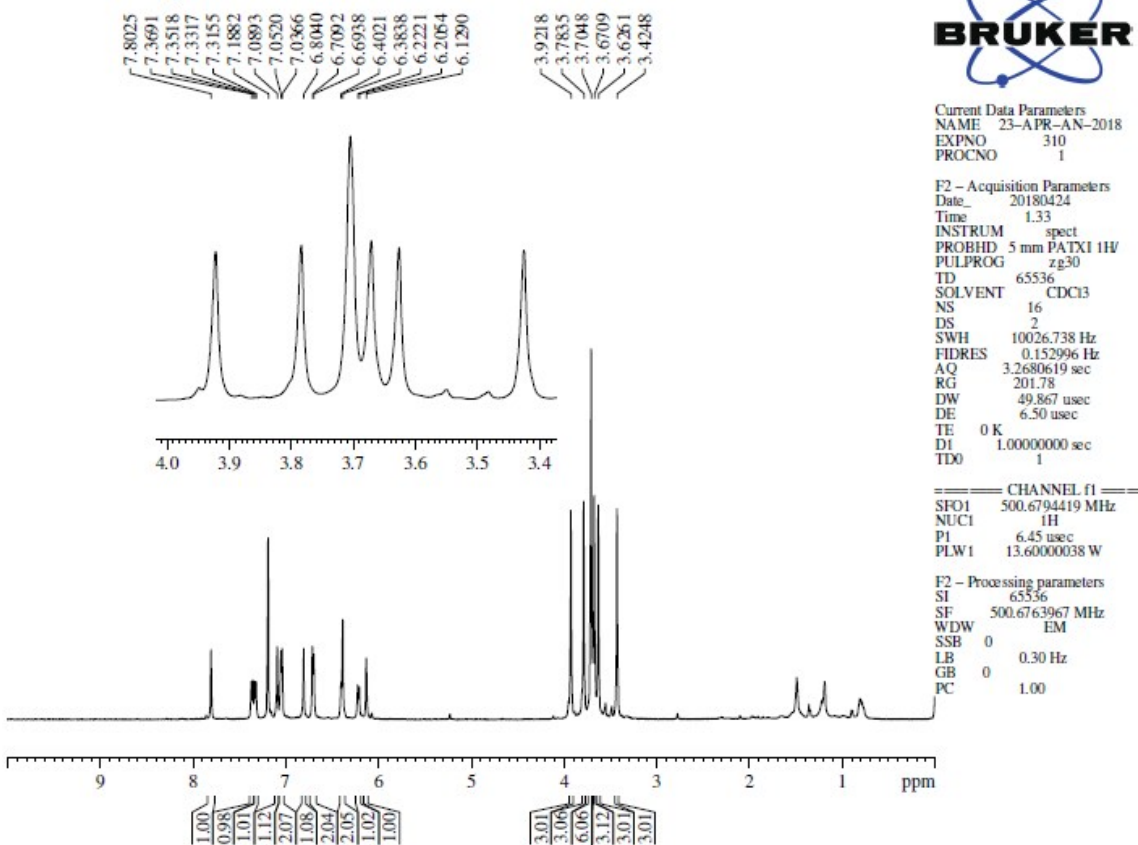


Figure 109: ¹H NMR spectrum of 4f

NRBK-272-major

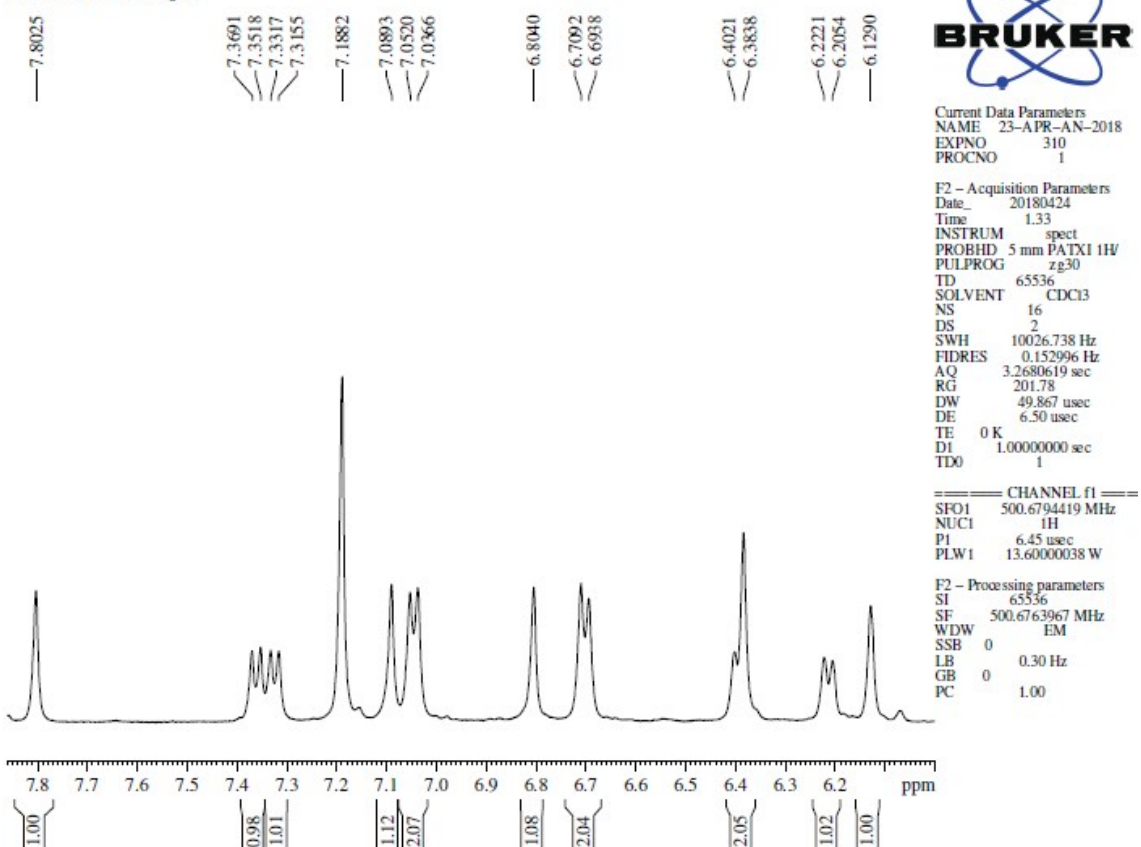
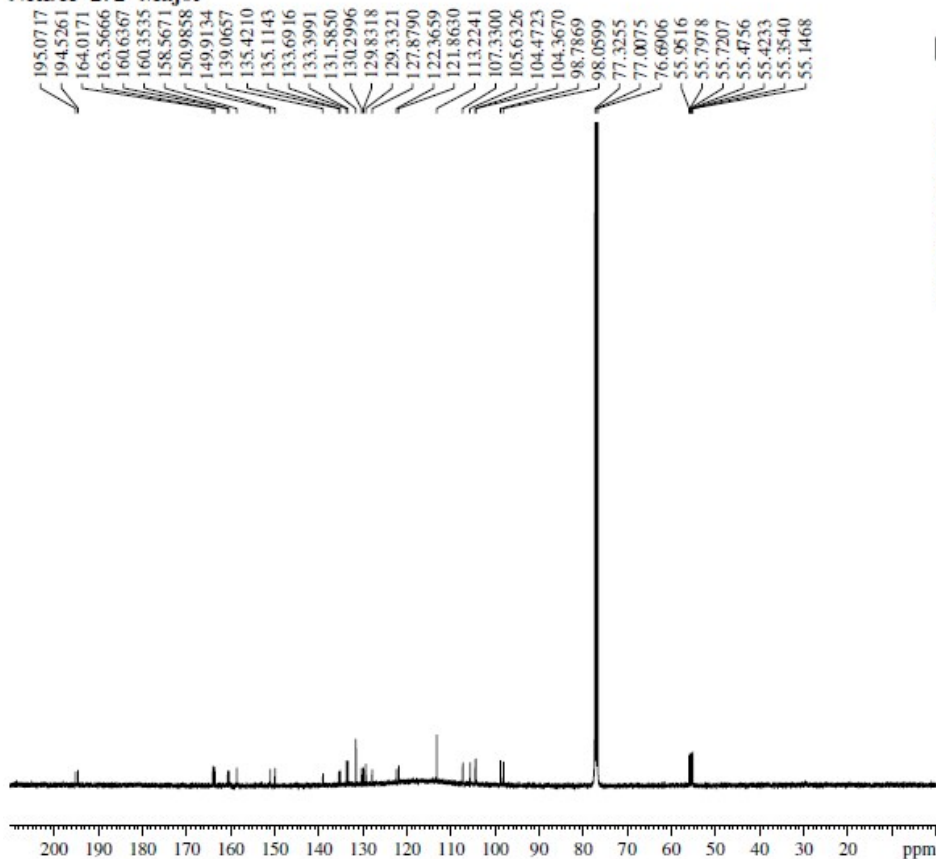


Figure 110: ¹H NMR spectrum of 4f (expansion)

NRBK-272-Major

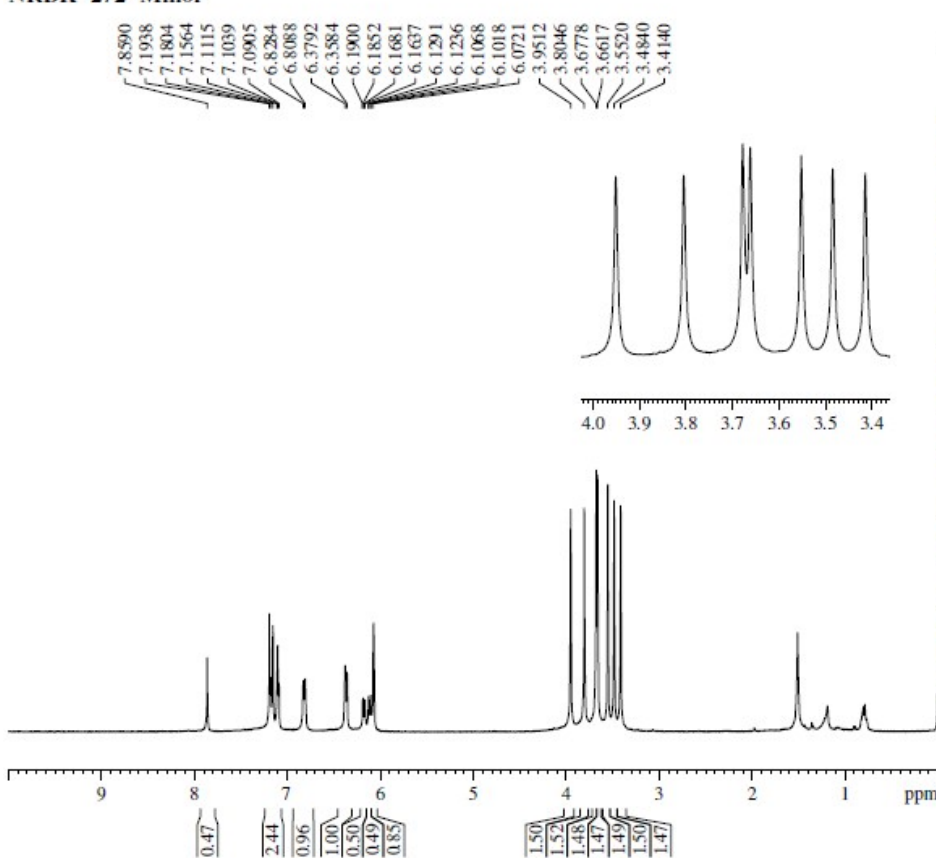


Current Data Parameters
 NAME 11-May-FN-2018
 EXPNO 310
 PROCNO 1

F2 - Processing parameters
 SI 32768
 SF 100.6569230 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 111: ¹³C NMR spectrum of 4f

NRBK-272-Minor



Current Data Parameters
 NAME 08-Mar-FN-2018
 EXPNO 330
 PROCNO 1

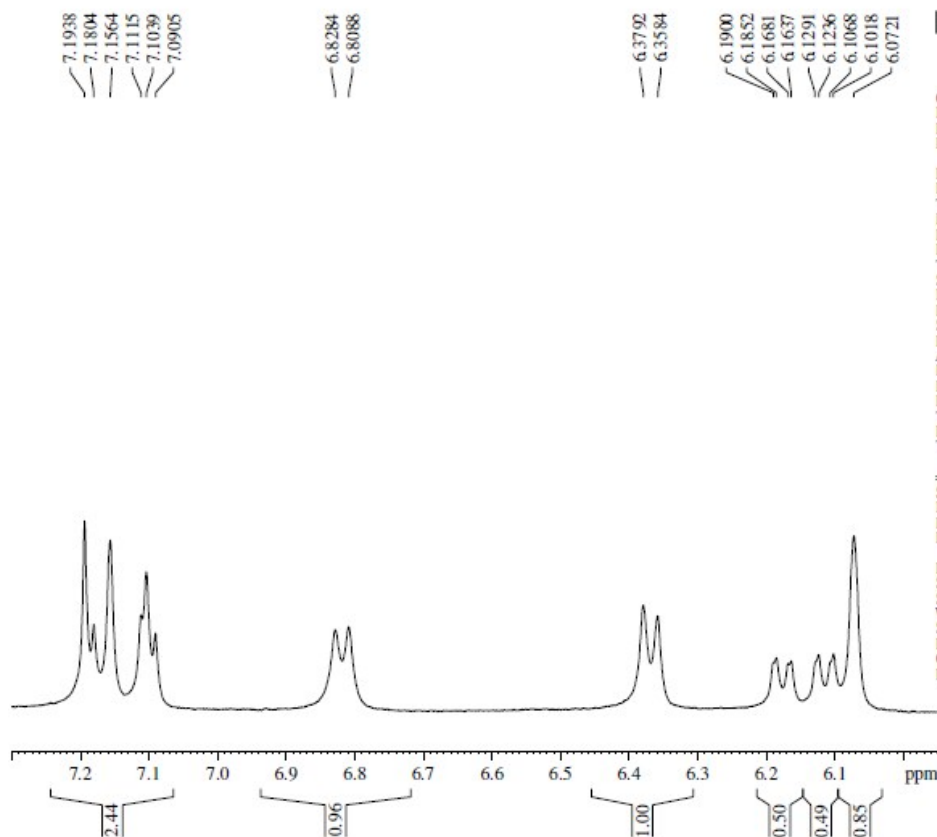
F2 - Acquisition Parameters
 Date_ 20180308
 Time 15.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 145.29
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 DI 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605358 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure 112: ¹H NMR spectrum of 4g

NRBK-272-minor



Current Data Parameters
NAME 08-Mar-FN-2018
EXPNO 330
PROCNO 1

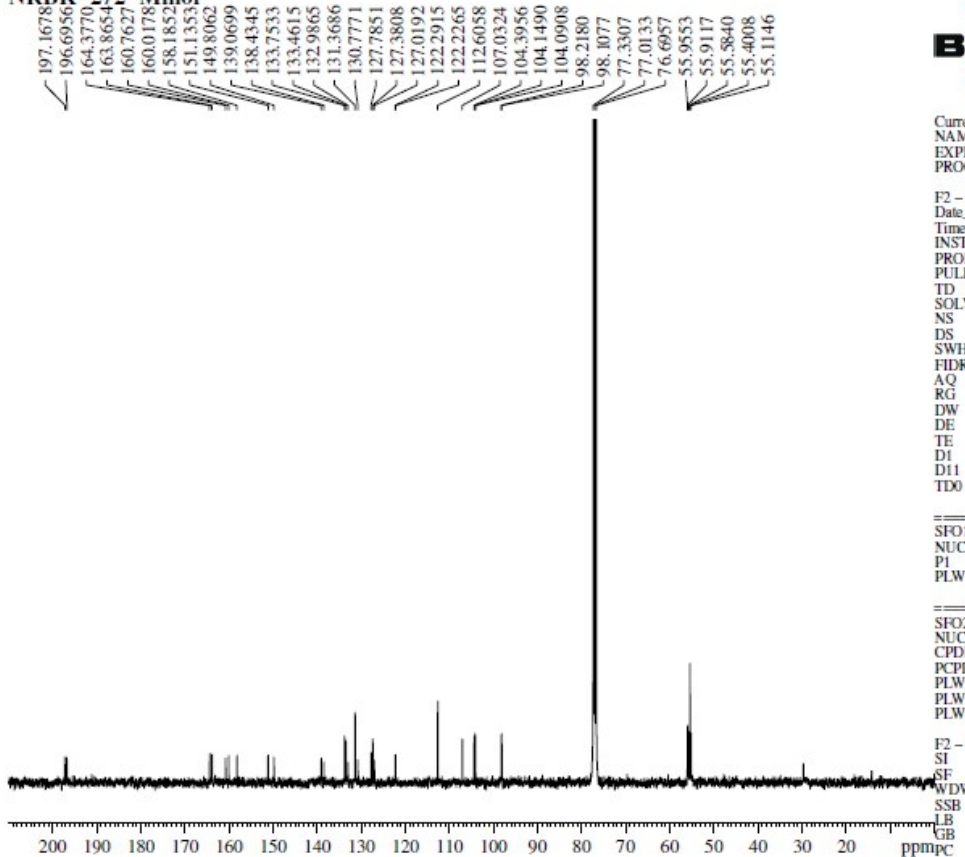
F2 - Acquisition Parameters
Date_ 20180308
Time 15.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 145.29
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605358 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 113: ¹H NMR spectrum of 4g (expansion)

NRBK-272-Minor



Current Data Parameters
NAME 06-Mar-AN-2018
EXPNO 320
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180307
Time 18.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 13C
P1 9.90 usec
PLW1 53.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

re 114: ¹³C NMR spectrum of 4g

Fig