Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.


# Chemical synthesis of a library of natural product-like derivatives based on pinnaic acid and initial evaluation of their anti-cancer activity.

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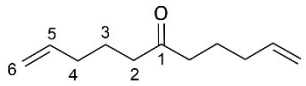
# *Supplementary Information*

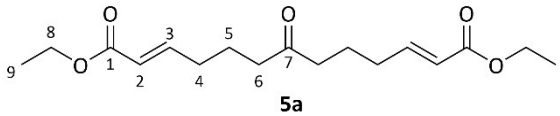
*Undeca-1,10-dien-6-one* **(4)**




**4**

To a solution of undeca-1,10-dien-6-ol (20.9 g, 124 mmol) in anhydrous DCM (200 mL) was added pyridinium chlorochromate (36.2 g, 168 mmol) and silica gel (50 g). The resulting dark brown solution was left to stir at r.t for 24 hours followed by the addition of a further portion of silica gel (25.0 g) and left to stir at r.t for a further 30 minutes. The mixture was concentrated, diluted with $Et_2O$ (100 mL) and filtered through a pad of celite and silica gel which was washed with $Et_2O$ successively until the filtrate ran clear. The filtrate was concentrated to give **4** as a yellow oil 16.4 g, 79 %. **IR** $\nu_{max}$ (neat)/$cm^{-1}$ 2932, 1712, 1640, 1440, 1370; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 5.77-5.67 (2H, m, 5-H), 5.00-4.91 (4H, m, 6-H), 2.36 (4H, t, *J* 7.40, 2-H), 2.04-1.98 (4H, m, 4-H), 1.62 (4H, p, *J* 7.40, 3-H); $\delta_c$ (75 MHz, $CDCl_3$), 210.8 (1-C), 138.0 (5-C), 115.1 (6-C), 41.9 (2-C), 33.1 (4-C), 22.8 (3-C); **MS** *m/z* (EI) 166 ($M^+$ 1), 125 (10), 97 (64), 84 (48), 69 (100).

*Diethyl (2E,11E)-7-oxotrideca-2,11-dienedioate* **(5a)**

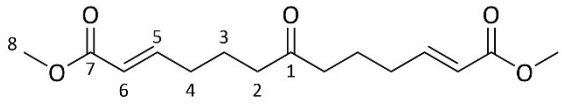



**5a**

A solution of **4** (5.0 g, 30 mmol) in anhydrous DCM (150 mL) and ethyl acrylate (21 g, 20 mL, 181 mmol) were added to Hoveyda Grubbs II catalyst (0.47 g, 2.5 mol %) and the resulting mixture left to stir at r.t for 24 hours. A further portion of Hoveyda Grubbs II catalyst (0.47 g, 2.5 mol %) was added and the mixture continued to stir until TLC analysis confirmed complete consumption of the starting material (2 days). The reaction was stopped and the solvent evaporated to give **5a** as a viscous brown oil. The crude product was purified via flash chromatography eluting with hexane/EtOAc 4:1 to give diethyl (2E,11E)-7-oxotrideca-2,11-

dienedioate as a brown oil (4.3 g, 83 %). **IR** $\nu_{max}$ (neat)/$cm^{-1}$ 2937, 1712, 1652, 1265, 1179; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 6.83 (2H, dtd, *J* 15.6, 6.96, 5-H), 5.73 (2H, dt, *J* 15.6, 1.56, 6-H), 4.09 (4H, q, *J* 7.14, 8-$H_2$), 2.36 (4H, t, *J* 7.30, 2-$H_2$), 2.16-2.11 (4H, m, 4-$H_2$), 1.67 (4H, p, *J* 7.33, 3-$H_2$), 1.21 (6H, t, *J* 7.14, 9-$H_3$); $\delta_c$ (75 MHz, $CDCl_3$); 209.3 (1-C), 166.3 (7-C), 147.9 (5-C), 121.9 (6-C), 60.1 (8-C), 41.6 (2-C), 31.3 (4-C), 21.8 (3-C), 14.2 (9-C); **MS** *m/z* (EI) 310 ($M^+$), 123 (51), 95 (100), 81 (82).

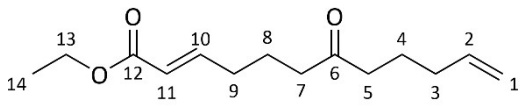
*Dimethyl (2E,11E)-7-oxotrideca-2,11-dienedioate* **(5b)**




**5b**

**5b** was obtained by following the synthesis for **5a** resulting in a yellow oil 2.7 g, 72%. **IR** $\nu_{max}$ (neat)/$cm^{-1}$ 2951, 1713, 1658, 1436, 1315, 1201; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 6.83 (2H, dt, *J* 15.6, 6.97, 5-H), 5.74 (2H, dt, *J* 15.6, 1.57, 6-H), 3.64 (6H, s, 8-$H_3$), 2.34 (4H, t, *J* 7.30, 2-$H_2$), 2.13 (4H, qd, *J* 14.5, 7.25, 4-$H_2$), 1.67 (4H, p, *J* 14.6, 7.30, 3-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 209.3 (1-C), 166.8 (7-C), 148.3 (5-C), 121.5 (6-C), 51.4 (8-C), 41.6 (2-C), 31.3 (4-C), 21.8 (3-C); **MS** *m/z* (EI) 282 ($M^+$ 1), 155 (25).

*Ethyl (2E)-7-oxododeca-2,11-dienoate* **(5d)**

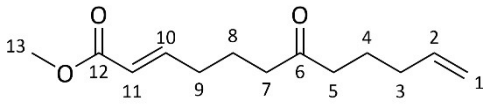



**5d**

**5d** was obtained as a by-product from the synthesis of **5a** resulting in a brown oil (0.63 g, 22 %). **IR** $\nu_{max}$ (neat)/$cm^{-1}$ 2936, 1712, 1654, 1267, 1179; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 6.84 (1H, dddd, *J* 15.6, 6.96, 10-H), 5.77-5.63 (2H, m, 2-H, 11-H), 4.96-4.87 (2H, m, 1-$H_2$), 4.10 (2H, q, *J* 7.14, 13-$H_2$), 2.37-2.31 (4H, m, 5-$H_2$, 7-$H_2$), 2.16-2.10 (2H, m, 9-$H_2$), 2.00-1.94 (2H, m, 3-$H_2$), 1.70-1.56 (4H, m, 4-$H_2$, 8-$H_2$), 1.21 (3H, t, *J* 7.14, 14-$H_3$); $\delta_c$ (75 MHz, $CDCl_3$) 210.1 (6-C), 166.5 (12-C), 148.1 (10-C), 137.9 (2-C), 122.0 (11-C), 115.2 (1-C), 60.2 (13-C), 41.9 (5-C), 41.7 (7-C),

33.1 (3-C), 31.4 (9-C), 22.7 (8-C), 21.9 (4-C), 14.2 (14-C); **MS** *m/z* (EI) 238 ($M^+$), 95 (100), 81 (71).

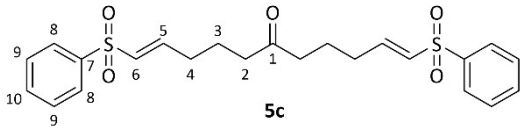
*Methyl (2E)-7-oxododeca-2,11-dienoate* **(5e)**




**5e**

**5e** was obtained as a by-product from the synthesis of **5b**, resulting in a yellow oil 1.1g, 17 %. **IR** $\nu_{max}$ (neat)/$cm^{-1}$ 2949, 1717, 1657, 1270, 1197; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 6.84 (1H, dddd, *J* 15.6, 6.96, 10-H), 5.75 (1H, dt, *J* 15.6, 1.58, 11-H), 5.71-5.63 (1H, m, 2-H), 4.95-4.87 (2H, m, 1-H), 3.64 (3H, s, 13-$H_3$), 2.39-2.31 (4H, m, 5-$H_2$, 7-$H_2$), 2.17-2.11 (2H, m, 9-$H_2$), 2.00-1.94 (2H, m, 3-$H_2$), 1.70-1.52 (4H, m, 8-$H_2$, 4-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 210.0 (6-C), 166.8 (12-C), 148.4 (10-C), 137.8 (2-C), 121.4 (11-C), 115.1 (11-C), 51.3 (13-C), 41.8 (7-C), 41.6 (5-C), 33.0 (3-C), 31.3 (9-C), 22.6 (8-C), 21.8 (4-C); **MS** *m/z* (EI) 224 ($M^+$ 1), 81 (76), 69 (100), 55 (95)

*Ethyl (3aS,4S,7S,10aS)-7-(2-ethoxy-2-oxoethyl)octahydro-1H-yclopenta[3,4][1,2]oxazolo[2,3-a]pyridine-4-carboxylate* **(5c)**

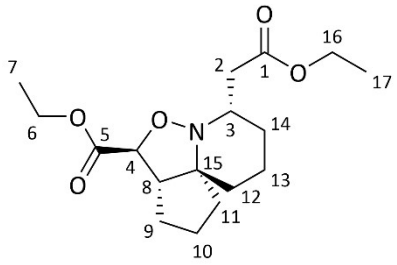



**5c**

To a solution of Hoveyda Grubbs II catalyst (0.094 g, 5 mol %) in anhydrous DCM (20 mL) was added a solution of **4** (0.50 g, 3.0 mmol) in anhydrous DCM (15 mL), followed by phenyl vinyl sulfone (3.0 g, 18 mmol) and the resulting mixture left to stir @ 50 °C. At 24 hours a second portion of Hoveyda Grubbs II catalyst (0.094 g, 5 mol %) was added to the reaction mixture which was then left to stir for a further 7 days. The reaction was stopped and the solvent

evaporated *in vacuo* to give a brown oil. The crude product was purified via flash chromatography eluting with hexane/EtOAc 1:1 to give **5c** as a brown oil, 1.2 g, 88 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2941, 1709, 1625, 1305, 1143; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 7.87-7.84 (4H, m, 8-$H_2$), 7.62-7.58 (2H, m, 10-H), 7.55-7.50 (4H, m, 9-$H_2$), 6.91 (2H, dt, *J* 15.1, 6.79, 5-H), 6.31 (2H, dt, *J* 15.1, 1.55, 6-H), 2.38 (4H, t, *J* 7.17, 2-$H_2$), 2.25-2.19 (4H, m, 4-$H_2$), 1.71 (4H, p, *J* 14.9, 3-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 208.7 (1-C), 145.9 (5-C), 140.5 (7-C), 133.4 (10-C), 131.1 (6-C), 129.3 (9-C), 127.6 (8-C), 41.5 (2-C), 30.6 (4-C), 21.3 (3-C); **MS** *m/z* (EI) 446 ($M^+$), 144 (100).

*2-[(3aS,4S,7S,10aS)-4-(hydroxymethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]ethan-1-ol* **(6a)**

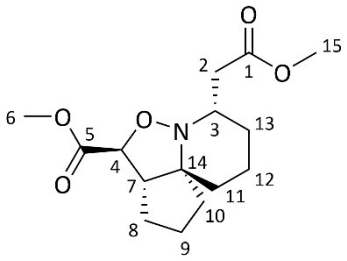



**6a**

To a solution of $NH_2OH{\cdot}HCl$ (0.36 g, 5.8 mmol) and NaOAc (1.0 g, 12 mmol) in anhydrous MeOH (50 mL) and anhydrous MeCN (10 mL) was added a solution of **5a** (0.80 g, 0.26 mmol) in anhydrous MeCN (5 mL) and the resulting mixture left to stir at r.t. Upon complete consumption of the starting material **5a** the reaction was stopped, and the solvent evaporated. The resulting orange solid was diluted with DCM (50 mL), filtered under suction and washed continuously with DCM until the solid became white in colour. The filtrate was concentrated, and the oil obtained dissolved in MeCN (50 mL). This solution was stirred at r.t and upon complete consumption of the intermediate nitrone the reaction was stopped, concentrated *in vacuo*, refluxed in hexane for 20 minutes, filtered hot and concentrated to give **6a** as a yellow oil, 1.4 g, 87 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$ 2939, 1731, 1445, 1262, 1184; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.25-4.08 (5H, m, 4-H, 6-$H_2$, 16-$H_2$), 3.16 (1H, dd, *J* 18.8, 12.3, $2_B$ -H), 3.05-3.02 (1H, m, 8-H), 2.90-2.85 (1H, m, 3-H), 2.29 (1H, dd, *J* 9.96, 5.77, $2_A$-H ), 2.05-1.35 (12H, m,

9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$, 13-$H_2$, 14-$H_2$), 1.28-1.22 (6H, m, 7-$H_3$, 17-$H_3$); $\delta_c$ (75 MHz, $CDCl_3$) 173.0 (1-C), 172.2 (5-C), 83.4 (4-C), 77.5 (15-C), 61.4 (16-C), 60.5 (3-C), 60.1 (6-C), 51.0 (8-C), 39.2 (14-C), 39.1 (2-C), 33.0, 29.9, 29.2, 21.1, 20.6 (9-C to 14-C), 14.3 (17-C), 14.0 (7-C); **MS** *m/z* (EI) 325 ($M^+$, 13), 252 (29), 238 (100), 210 (86).

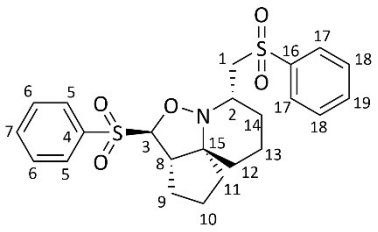
*Methyl [(4S,7S,7aS,10aS)-7-hydroxy-6-oxodecahydrocyclopenta[i]indolizin-4-yl]acetate* **(6b)**




**6b**

**6b** was obtained by reacting **5b** as described in the synthesis for **6a**, resulting in a yellow oil 1.9 g, 68 %. **IR** $\nu_{max}$ (neat)/$cm^{-1}$ 2948, 1730, 1436, 1262, 1163; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.12 (1H, d, *J* 6.42, 4-H), 3.72 (3H, s, 6-$H_3$), 3.63 (3H, s, 15-$H_3$), 3.13 (1H, dd, *J* 18.6, 12.3, 3.20, $2_A$-H), 3.02-2.99 (1H, m, 7-H), 2.88-2.82 (1H, m, 3-H), 2.28 (1H, dd, *J* 5.86, $2_B$-H), 1.95-1.21 (12H, m, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$, 13-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$), 171.5 (1-C), 170.7 (5-C), 81.4 (4-C), 75.7 (14-C), 58.6 (3-C), 50.5 (6-C), 49.5 (15-C), 49.2 (7-C), 37.3 (13-C), 37.0 (2-C), 31.0, 28.1, 27.3, 19.2, 18.7 (8-C to 12-C); **MS** *m/z* (EI) 297 ($M^+$ 16), 266 (5), 238 (25); **HRMS:** Found 373.2208 $C_{22}H_{29}O_3N$, $[M+H]^+$ Requires 373.2203.

*(3aS,7S,10aS)-4-(benzenesulfonyl)-7-[(benzenesulfonyl)methyl]octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridine* **(6c)**

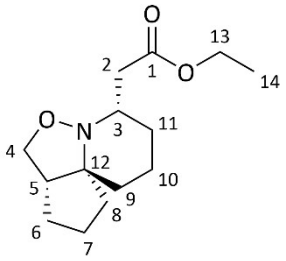


**6c**

To a solution of $NH_2OH{\cdot}HCl$ (0.037 g, 0.54 mmol) and NaOAc (0.092 g, 1.1 mmol) in anhydrous MeCN (9 mL) and anhydrous MeOH (7 mL) was added a solution of **5c** (0.20 g, 0.45 mmol) in anhydrous MeCN (1 mL) and the resulting mixture left to stir at r.t. overnight. Upon complete consumption of the starting material the solvent was evaporated and the resulting brown solid diluted with DCM (50 mL), filtered under suction and washed continuously with DCM until the solid became white in colour. The filtrate was concentrated, dissolved in MeCN (20 mL) and stirred at 50 °C for 3 hours. The crude mixture was concentrated and purified via flash chromatography eluting with hexane/EtOAc 1:2 to give **6c** as a yellow oil, 0.077 g, 37 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2927, 1629, 1305, 1146, 1085; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 7.95-7.87 (4H, m, 5-$H_2$, 17-$H_2$), 7.68-7.59 (2H, m, 7-H, 19-H), 7.56-7.52 (4H, m, 6-$H_2$, 18-$H_2$), 4.47 (1H, d, *J* 7.41, 3-H), 3.85-3.80 (1H, m, 2-H), 3.53 (1H, dd, *J* 14.2, 2.61, $1_B$-H), 3.32 (1H, t, *J* 6.62, 8-H), 3.20 (1H, dd, *J* 14.2, 9.41, $1_A$-H), 2.35-2.30 (1H, m, $14_B$-H), 1.97-1.39 (11H, m, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$, 13-$H_2$, $14_A$-H); $\delta_c$ (75 MHz, $CDCl_3$) 141.0 (4-C)137.6 (16-C), 134.2 (7-C), 133.6 (19-C), 129.3 (6-C), 129.2 (18-C), 129.0 (5-C), 127.8 (17-C), 99.7 (3-C), 77.3 (15-C), 60.4 (1-C), 60.3 (2-C), 48.6 (8-C), 38.5, 32.0, 29.7, 28.0, 20.3, 19.4 (9-C to 14-C); **MS** *m/z* (FT) 462 ($[M+H]^+$ 100), 320 (100), **HRMS** Found 462.1396. $C_{23}H_{27}O_5S_2N$ $[M+H]^+$, requires 462.1403.

*Ethyl [(3aS,7S,10aS)-octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]acetate*

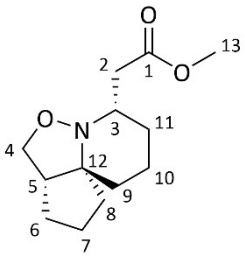
**(6d)**



**6d**

To a solution of $NH_2OH{\cdot}HCl$ (0.070 g, 1.0 mmol) and NaOAc (0.17 g, 2.1 mmol) in absolute EtOH (25 mL) was added a solution of **5d** in absolute EtOH (10 mL) and the resulting solution was refluxed for 4 hours. The reaction was stopped and the solvent evaporated to give **6d** as a yellow oil, 0.20 g, 94 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2936, 1730, 1184, 1120; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.22 (1H, t, *J* 8.83, $4_A$-H), 4.05 (2H, q, *J* 7.13, 13-$H_2$), 3.34 (1H, dd, *J* 4.39, $4_B$-H), 2.96-2.89 (1H, m, 3-H), 2.72 (1H, dd, *J* 15.3, 5.88, $2_A$-H), 2.64-2.60 (1H, m, 5-H), 2.16 (1H, dd, *J* 15.3, 4.78, $2_B$ -H), 1.93-1.24 (12H, m, 6-$H_2$, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$), 1.19 (3H, t, *J* 7.13, 14-$H_3$); $\delta_c$ (75 MHz, $CDCl_3$) 172.8 (1-C), 77.2 (12-C), 71.8 (4-C), 60.3 (13-C), 57.4 (3-C), 47.1 (5-C), 41.6 (6-C), 40.6 (2-C), 33.9, 31.5, 30.4, 22.7, 21.3 (7-C to 11-C), 14.2 (14-C); **MS** *m/z* (EI) 253 ($M^+$, 9), 224 (10), 208 (11), 166 (100).

*Methyl [(3aS,7S,10aS)-octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]acetate* **(6e)**

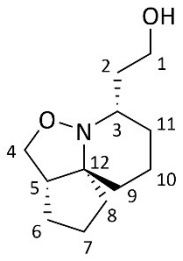


**6e**

**6e** was obtained by reacting **5e** in the synthesis described for **6d** resulting in a yellow oil 0.37 g, 37 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2944, 1733, 1435, 1249, 1159; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.21 (1H, t, *J* 8.34, $4_A$-H), 3.59 (3H, s, 13-$H_3$), 3.33 (1H, dd, *J* 12.9, 8.39, 4.08, $4_B$-H), 2.96-2.90 (1H, m, 3-H). 2.72 (1H, dd, *J* 15.2, 5.74, $2_B$-H). 2.64-2.60 (1H, m, 5-H), 2.18 (1H, dd, *J* 15.2, 6.55, $2_A$--H), 1.92-1.19 (12H, m, 6-$H_2$, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 173.1 (1-C),

77.1 (12-C), 71.7 (4-C), 57.4 (3-C), 51.5 (13-C), 47.0 (5-C), 41.5, 40.3, 33.8, 31.4, 30.4, 22.6, 21.3 (2-C and 6C to 11-C); **MS**, *m/z* (EI), 239 ($M^+$ 10), 166 (100).

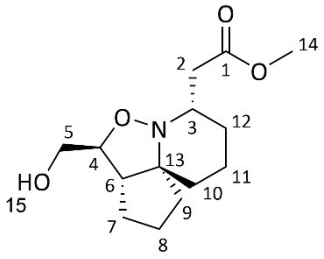
*2-[(3aS,7S,10aS)-octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]ethan-1-ol* **(7)**




**7**

To a solution of **6d** (0.051 g, 0.20 mmol) in anhydrous DCM (5 mL) pre-cooled to 0 °C was added DIBAL-H (0.23 mL, 0.22 mmol) in one portion and the resulting mixture left to stir at 0 °C for 2.5 hours. The reaction was quenched with MeOH (5 mL), diluted with $dH_2O$ (5 mL) and left to stir for 10 minutes. The solution was added to HCl (5 mL, 10 % sol'n) and the pH raised to 10 with the addition of NaOH (1M). The mixture was diluted with EtOAc (15 mL), extracted with EtOAc (3 x 15 mL), organic fractions combined, dried over $MgSO_4$, filtered and concentrated to give **7** as a brown oil 0.025 g, 59 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3360-OH, 2932, 2863, 1444, 1054; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.25 (1H, t *J* 8.80, $4_A$-H), 3.92-3.86 (1H, m, $1_A$-H), 3.67-3.62 (1H, m, $1_B$-H), 3.43 (1H, dd, *J* 8.62, 4.37, $4_B$-H), 2.82-2.77 (1H, m, 3-H), 2.67-2.62 (1H, m, 5-H), 2.10-2.02 (1H, m, $2_A$-H). 1.97-1.83 (2H, m, $6_B$ -H, 11-H), 1.77-1.30 (12H, m, $2_B$-H, $6_A$-H, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$), 76.7 (12-C), 71.5 (4-C), 60.4 (1-C), 58.9 (3-C), 47.8 (5-C), 41.8 (6-C), 35.6 (2-C), 33.8 (11-C), 31.5, 28.9, 22.9, 21.1 (7-C to 10-C); **MS** *m/z* (EI) 211 ($M^+$, 8), 166 (100); **HRMS** Found 212.1642, $C_{12}H_{22}NO_2$, $[M+H]^+$, Requires 212.1645.

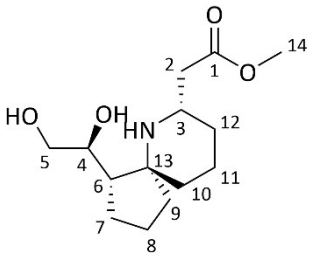
*Methyl [(3aS,4S,7S,10aS)-4-(hydroxymethyl)octahydro-1H-yclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]acetate* **(8)**




**8**

A solution of **6a** (0.53 g, 1.9 mmol) in anhydrous MeOH (40 mL) was added to Pd/C (0.53 g, 100 % wt) and left to stir under an atmosphere of $H_2$ at r.t for 18 hours. The catalyst was filtered off and the solvent evaporated under reduced pressure. The crude was purified via flash chromatography eluting with DCM/MeOH 9:1 to give **8** as a colourless oil, 0.085 g, 20 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$)3436-OH, 2937, 1732, 1436, 1195, 1114; **NMR** $\delta_H$ (400 MHz, $CDCl_3$), 4.60 (1H, s (br), 15-H), 4.51 (1H, d, *J* 9.80, $5_A$-H), 4.39 (1H, d, *J* 9.90, $5_B$-H), 3.66 (3H, s, 14-$H_3$), 3.54-3.48 (1H, m, 4-H), 3.32-3.28 (1H, m, 3-H), 2.48 (1H, dd, *J* 14.3, 9.86, 2-H), 2.34 (1H, dd, *J* 14.3, 8.85, 2-H), 2.14-2.07 (1H, m, $12_A$-H), 1.85-1.38 (12H, m, 6-H, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, $12_B$-H); $\delta_c$ (75 MHz, $CDCl_3$) 172.9 (1-C), 78.5 (4-C), 77.2 (5-C), 77.1 (13-C), 52.7 (3-C), 51.7 (13-C), 40.7 (6-C), 37.7 (2-C), 36.1, 30.8, 28.2, 23.7, 19.9, 17.6 (7-C to 12-C); **MS** *m/z* (EI) 252 (24), 224 (19), 210 (69), 196 (31); **HRMS** Found 270.1698, $C_{14}H_{24}NO_4$, $[M+H]^+$, Requires 270.1700.

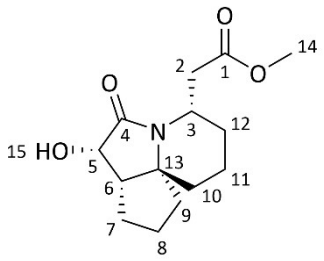
*Methyl {(1S,5S,7S)-1-[(1S)-1,2-dihydroxyethyl]-6-azaspiro[4.5]decan-7-yl}acetate* **(9)**




**9**

A solution of **8** (0.76 g, 2.7 mmol) in anhydrous MeOH (40 mL) followed by Pd/C (0.76 g, 100 % wt) was added to a pressure reactor and stirred at r.t for 18 hours under an atmosphere of $H_2$ at 5 bar pressure. The catalyst was filtered off and the solvent evaporated under reduced pressure. The crude mixture was purified via flash chromatography eluting with DCM/MeOH 7:3 to give **9** as a white residue 0.13 g, 17 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3311, 2931, 1728, 1195, 1173, 1116, 1070; **NMR** $\delta_H$ (400 MHz, $CDCl_3$), 3.71-3.67 (2H, m, 4-H, $5_B$-H), 3.65 (3H, s, 14-$H_3$), 3.46-3.42 (1H, m, $5_A$-H), 3.32-3.26 (1H, m, 3-$H_3$), 2.46-2.42 (2H, m, 2-$H_2$), 2.13-2.09 (1H, m, 6-H), 1.87-1.29 (12H, m, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 172.1 (1-C), 77.3 (13-C), 74.0 (4-C), 65.3 (5-C), 51.8 (14-C), 50.3 (3-C), 41.0 (6-C), 40.3 (2-C), 38.6, 36.0, 30.9, 26.6, 20.0, 19.6 (7-C to 12-C); MS (EI), 271 ($M^+$ 3), 254 (34), 240 (67), 224 (13), 198 (40); **HRMS** Found 272.1858 $C_4H_{25}NO_4$, $[M+H]^+$, Requires 272.1856.

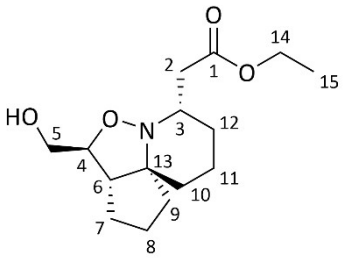
*Methyl [(4S,7S,7aS,10aS)-7-hydroxy-6-oxodecahydrocyclopenta[i]indolizin-4-yl]acetate* **(10)**




**10**

A solution of **6b** (0.11 g, 0.36 mmol) in anhydrous MeOH (8 mL) was added to Pd/C (0.11 g, 100 % wt) and the solution was left to stir at r.t under an atmosphere of $H_2$ for 18 hours. The catalyst was filtered off and the solvent evaporated under reduced pressure to give **10** as a colourless residue 0.071 g, 73 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3307-OH, 2945, 1733, 1663; **NMR** $\delta_H$ (400 MHz, $CDCl_3$), 4.74-4.68 (1H, m, 3-H), 4.61 (1H, S-br, 15-H), 4.32 (1H, d, *J* 9.83, 5-H), 3.65 (3H, s, 14-$H_3$), 2.61 (2H, ddd, *J* 15.1, 2-$H_2$), 2.39-2.33 (1H, m, 6-H), 2.01-1.95 (1H, m, 12-H), 1.82-1.28 (11H, m, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_1$); $\delta_c$ (75 MHz, $CDCl_3$) 174.2 (1-C), 171.2 (4-C), 69.4 (13-C), 69.1 (5-C), 51.8 (14-C), 48.3 (6-C), 45.7 (3-C), 38.8, 37.3, 36.8, 27.8, 25.9, 23.8, 17.3 (2-C and 7-C to 12-C), **MS** (EI) 267 ($M^+$ 36).

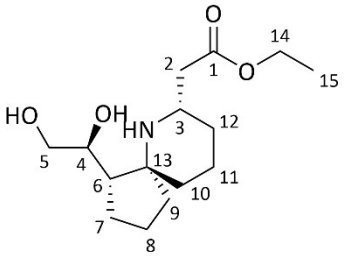
*Ethyl [(3aS,4S,7S,10aS)-4-(hydroxymethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]acetate* **(11)**




**11**

To solution of **6a** (1.1 g, 3.7 mmol) in absolute EtOH (30 mL) pre-cooled to 0 °C was added $NaBH_4$ (0.43 g, 11 mmol) in one portion. The reaction mixture was warmed to r.t and left to stir for 48 hours. The reaction was quenched with acetone (30 mL) and left to stir at r.t for 1 hour. The solvent was evaporated, and the resulting white solid dissolved in $dH_2O$ (30 mL). The crude was extracted with EtOAc (3 x 40 mL), dried over $MgSO_4$, filtered and concentrated to give a yellow oil. The crude was purified via flash chromatography eluting with hexane/EtOAc 1:1 to give **11** as a colourless oil 0.60 g, 63 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3435- OH, 2934, 1730, 1299, 1170, 1130; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.16-4.03 (2H, m, 14-$H_2$), 3.73-3.64 (2H, m, 4-H, $5_A$-H), 3.57-3.51 (1H, m, $5_B$-H), 3.21-3.15 (1H, m, 3-H), 2.66-2.60 (2H, m, $2_B$ -H, 6-H), 2.22 (1H, dd, *J* 14.9, 3.90, $2_A$-H), 2.03-1.95 (1H, m, $12_B$-H), 1.66-1.23 (11H, m, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, $12_A$-H), 1.21 (3H, t, *J* 7.15, 15-$H_3$); $\delta_c$ (75 MHz, $CDCl_3$) 174.9 (1-C), 88.0 (4-C), 76.7 (13-C), 62.1 (5-C), 61.8 (3-C), 61.0 (14-C), 46.1 (6-C), 42.0 (2-C), 38.0 (12-C), 31.9, 31.1, 26.6, 20.3, 20.2 (7-C to 11-C), 14.0 (15-C); **MS** *m/z* (EI) 283 ($M^+$, 14), 210 (40), 196 (100).

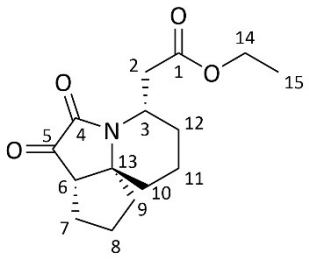
*Ethyl {(1S,5S,7S)-1-[(1S)-1,2-dihydroxyethyl]-6-azaspiro[4.5]decan-7-yl}acetate* **(12)**




**12**

A solution of **11** (0.62 g, 2.2 mmol) in anhydrous MeOH (40 mL) was added to Pd/C (0.25 g, 40 % wt) and the reaction mixture left to stir under an atmosphere of $H_2$ at r.t. Upon complete consumption of the starting material the reaction was stopped, and the mixture filtered to remove the catalyst. The solvent was evaporated to give **12** as a viscous opaque oil, 0.61 g, 99 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3304, 2934, 1731, 1253, 1185; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.15-4.09 (2H, m, 14-$H_2$), 3.90-3.86 (1H, m, 4-H), 3.78-3.72 (2H, m, 3-H, 5-H), 3.48 (1H, dd, *J* 11.4, 5.30 5-H), 2.92-2.85 (1H, m, $2_B$-H) 2.68 (1H, dd, *J* 16.0, 8.60, $2_A$-H), 2.24-2.20 (1H, m, 6-H), 2.14-2.08 (1H, m, $12_B$-H), 1.90-1.45 (11H, m, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, $12_A$-H), 1.23 (3H, t, *J* 7.15, 15-H); $\delta_c$ (75 MHz, $CDCl_3$), 170.5 (1-C), 72.7 (4-C), 66.8 (13-C), 65.5 (5-C), 60.9 (14-C), 51.5 (3-C), 42.2 (6-C), 38.3 (2-C), 36.5 (12-C), 34.3, 28.5, 26.1, 19.5, 19.0 (7-C to 11-C), 14.2 (15-C); **MS** *m/z* (EI) 285 ($M^+$ 6), 268 (38), 254 (84).

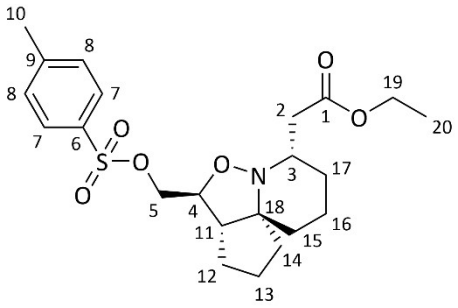
*Ethyl [(4S,7S,7aS,10aS)-7-hydroxy-6-oxodecahydrocyclopenta[i]indolizin-4-yl]acetate* **(13)**




**13**

To a stirring solution of DMSO (0.16 g, 0.41 mL, 5.7 mmol) in anhydrous DCM (2 mL), pre-cooled to -84 °C was added oxalyl chloride (0.22 g, 0.50 mL, 5.8 mmol) in one portion. After stirring at -84 °C for one hour **12** (0.20 g, 0.70 mmol) was added dropwise over 30 minutes and the mixture left to stir at - 15 °C for one hour. The reaction temperature was lowered to -84 °C followed by the addition of $Et_3N$ (0.43 g, 0.60 mL, 4.2 mmol) dropwise over 30 minutes. Upon complete addition the reaction temperature was raised to r.t and the mixture was left to stir for a further 45 minutes. The reaction was quenched with $dH_2O$ (8 mL) and left to stir for 1 hour, followed by dilution with DCM (15 mL). The crude product was extracted with DCM (2 x 25 mL), washed with brine (15 mL), dried over $MgSO_4$, filtered and concentrated to give **13** as an orange oil. The crude product was purified via flash chromatography eluting with hexane/EtOAc 4:1 to give a yellow oil, 0.027 g, 27 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2934, 2870, 1758, 1728, 1703, 1448, 1174; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 5.02-4.96 (1H, m, 3-H), 4.17-4.05 (2H, m, 14-$H_2$), 2.75-2.61 (2H, m, 2-$H_2$), 2.50 (1H, dd, *J* 9.54, 4.90, 6-H), 2.04-1.57 (12H, m, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$), 1.23 (3H, t, *J* 7.14, 15-$H_3$); $\delta_c$ (75 MHz, $CDCl_3$) 202.0 (5-C), 170.2 (1-C), 158.8 (4-C), 66.3 (13-C), 61.1 (14-C), 55.0 (6-C), 46.4 (3-C), 37.9 (2-C), 37.6, 37.3, 28.1, 27.6, 26.3, 16.7 (7C to 12-C), 14.2 (15-C); **MS** *m/z* (EI) 279 ($M^+$ 82).

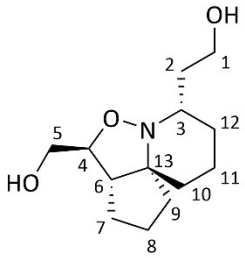
*Ethyl [(3aS,4S,7S,10aS)-4-{[(4-methylbenzene-1-sulfonyl)oxy]methyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]acetate* **(14)**




**14**

To a solution of **11** (0.10 g, 0.39 mmol) in anhydrous DCM (6 mL) was added $Et_3N$ (0.051 g, 0.07 mL, 0.51 mmol) and the resulting mixture was left to stir at r.t for 30 minutes. The temperature was lowered to 0 °C, followed by the addition of TsCl (0.071 g, 0.37 mmol) and the mixture left to stir at the same temperature for 5 minutes, then warmed to r.t and left to stir for 2 weeks. The reaction was stopped and quenched with brine (10 mL) at 0 °C, extracted with DCM (2 x 30 mL), organics combined, dried over $MgSO_4$, filtered and concentrated to give a yellow residue. The crude was purified via flash chromatography eluting with hexane / EtOAc 1:1 to give **14** as a colourless oil, 0.60 g, 41 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$)2938, 1728, 1364, 1189, 1175, 1096; **NMR** $\delta_H$ (400 MHz, $CDCl_3$), 7.81-7.78 (2H, m, 7-$H_2$), 7.35-7.32 (2H, m, 8-$H_2$), 4.16 (2H, d, *J* 6.08, 5-$H_2$), 4.08 (2H, q, *J* 7.14, 19-$H_2$), 3.81-3.76 (1H, m, 4-H), 2.95-2.88 (1H, m, 3-H), 2.75 (1H, dd, *J* 15.1, $2_A$-H), 2.46-2.41 (4H, m, 10-$H_2$, 11-$H_2$), 2.20 (1H, dd, *J* 15.1, 8.30, $2_B$-H), 1.96-1.45 (12H, m, 12-$H_2$, 13-$H_2$, 14-$H_2$, 15-$H_2$, 16-$H_2$, 17-$H_2$), 1.23 (3H, t, *J* 7.14, 20-$H_3$); $\delta_c$ (75 MHz, $CDCl_3$) 172.2 (1-C), 144.9 (6-C), 133.0 (9-C), 129.9 (8-C), 128.0 (7-C), 82.5 (4-C), 76.7 (18-C), 71.6 (5-C), 61.4 (3-C), 60.3 (19-C), 51.2 (11-C), 40.1 (2-C), 38.3, 31.6, 29.2, 27.7, 21.7, 21.3, 19.6 (10-C and 12-C to 14-C), 14.2 (20-C); **MS** *m/z* (EI) 237 ($M^+$ 12), 208 (14), 194 (100), **HRMS** Found 438.1938, $C_{22}H_{32}NO_6S$, $[M+H]^+$ Requires 438.1945.

*2-[(3aS,4S,7S,10aS)-4-(hydroxymethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]ethan-1-ol* **(15)**




**15**

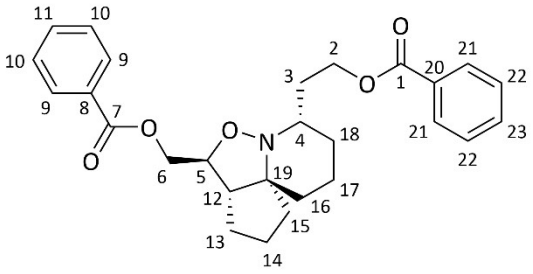
To a solution of **6a** (0.90 g, 2.8 mmol) in anhydrous DCM (25 mL) pre-cooled to 0 °C was added a solution of Red-Al in PhMe (60 % wt, 1.6 mL, 8.3 mmol) dropwise. Upon complete addition, the cooling bath was removed and the mixture left to stir for 10 minutes. The mixture was quenched with Rochelle's salt (10 % sol'n, 40 mL) and left to stir overnight. The organic phase was extracted with DCM (7 x 50 mL), combined, dried over $MgSO_4$, filtered and concentrated to give a yellow oil. The crude mixture was purified via flash chromatography, eluting with DCM/MeOH 8:2 to give **15** as an opaque oil, 0.40 g, 55 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$), 3330-OH, 2929, 2868, 1445, 1124, 1056; **NMR** $\delta_H$ (400 MHz, $CDCl_3$), 3.95-3.90 (1H, m, $1_A$-H), 3.82-3.79 (1H, dd, *J* 9.51, 3.81, $5_B$-H), 3.74-3.70 (1H, m, 4-H), 3.66-3.57 (2H, m, $1_B$-H, $5_A$-H), 3.05-2.99 (1H, m, 3-H), 2.53-2.50 (1H, m, 6-H), 2.04-1.97 (1H, m, $12_A$-H), 1.94-1.34 (13H, m, 2-$H_2$, 7$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, $12_B$-H); $\delta_c$ (75 MHz, $CDCl_3$) 86.1 (4-C), 76.6 (13-C), 62.9, (5-C) 60.9 (3-C), 60.3 (1-C), 49.6 (6-C), 38.3 (12-C), 37.0 (2-C), 31.2, 28.5, 27.5, 21.8, 19.4 (7-C to 11-C), **HRMS** Found 264.1567 $C_{13}H_{23}NO_3Na$, $[M + Na]^+$, Requires 264.1570.

Synthesis for compounds **17a-l**

To a solution of **15** (1 eq) in anhydrous DCM (4 mL) cooled to 0 °C was added $Et_3N$ (2 eq) followed by a solution of acid chloride (see each compound below for equivalents of acid chloride) in anhydrous DCM (2 mL) dropwise. Once the addition was complete the cooling

bath was removed, and the mixture left to stir at r.t. Upon complete consumption of the starting material the reaction was quenched with $dH_2O$ (20 mL) and washed with $dH_2O$ (3 x 20 mL). The crude was extracted with DCM (3 x 20 mL), the organic fractions combined, dried over $MgSO_4$, filtered and concentrated. The crude mixture was purified via flash chromatography eluting with hexane/EtOAc 1:1.

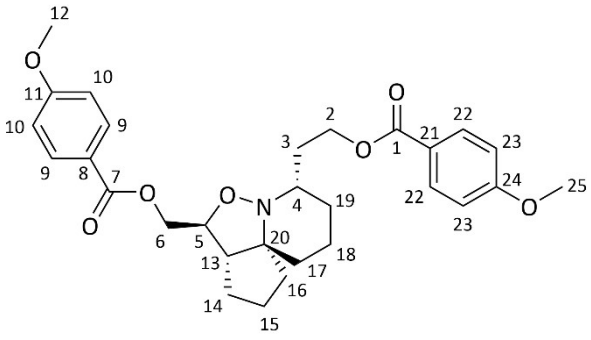
*{(3aS,4S,7S,10aS)-7-[2-(benzoyloxy)ethyl]octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl}methyl benzoate* **(17a)**



**17a**

**15** (1 eq) was treated with benzoyl chloride (1.1 eq) to give **17a** as an opaque oil, 0.18 g, 54 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$), 2938, 2866, 1714, 1601, 1450, 1176, 1111; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.05-7.99 (4H, m, 9-H, 21-H), 7.55-7.47 (2H, m, 1-H, 23-H), 7.43-7.36 (4H, m, 8-H, 22-H), 4.55-4.37 (4H, m, 6-$H_2$, 2-$H_2$), 4.00-3.95 (1H, m, 5-H), 2.97-2.90 (1H, m, 4-H), 2.52-2.48 (1H, m, 12-H), 2.31-2.23 (1H, m, $18_A$-H), 1.97-1.33 (13H, m, 13-$H_2$, 14-$H_2$, 15-$H_2$, 16-$H_2$, 17-$H_2$, $18_B$-H), ); $\delta_c$ (75 MHz, $CDCl_3$) 196.6 (1-C), 166.6 (7-C), 133.1 (11-C), 132.7 (23-C), 130.5 (8-C), 129.9 (20-C), 129.7 (9-C), 129.5 (21-C), 128.4 (10-C), 128.3 (22-C), 82.6 (5-C), 76.3 (19-C), 66.9 (6-C), 62.8 (2-C), 60.8 (4-C), 52.6 (12-C), 38.6, 34.4, 31.5, 28.1, 22.3, 19.5 (13-C to 18-C); **MS** *m/z* (FT) 472 ([M + Na]$^+$ 13), 450 (100), 328 (100); **HRMS** Found 450.2269 $C_{27}H_{31}NO_5$, $[M+H]^+$, Requires 450.2275.

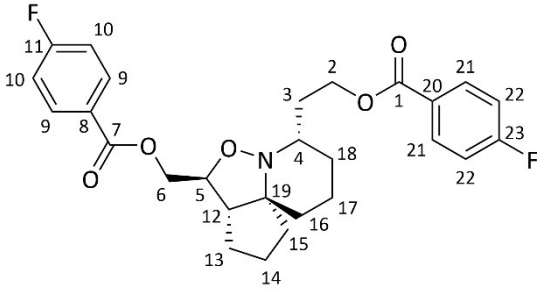
*[(3aS,4S,7S,10aS)-7-{2-[(4-methoxybenzoyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl 4-methoxybenzoate* **(17b)**



**17b**

**15** (1 eq) was treated with 4-methoxybenzoyl chloride (1.1 eq) to give **17b** as a colourless oil 0.013 g, 6.0 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$), 2924, 2850, 1711, 1606, 1511, 1460, 1257, 1167; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.01-7.95 (4H, m, 9-H, 13-H), 6.93-6.96 (4H, m, 10-H, 23-H), 4.51 (1H, dd, *J* 4.80, $6_A$-H), 4.47-4.35 (3H, m, 2-H, $6_B$-H), 4.00-3.95 (1H, m, 5-H), 3.85 (3H, s, 12-$H_3$), 3.83 (3H, s, 25-$H_3$). 2.95-2.89 (1H, m, 4-H), 2.53-2.49 (1H, 13-H), 2.32-2.24 (1H, m, $19_A$-H), 2.05-1.29 (13H, m, 3-$H_2$, 14-$H_2$, 15-$H_2$, 16-$H_2$ 17-$H_2$,-18-$H_2$, $19_B$-H); $\delta_c$ (75 MHz, $CDCl_3$) 166.4 (7-C), 166.0 (1-C), 163.5 (24-C), 163.2 (11-C), 131.7 (22-C), 131.5 (9-C), 122.9 (21-C), 122.3 (8-C), 113.7 (23-C), 113.6 (10-C), 82.8 (5-C), 76.3 (20-C), 66.7 (6-C), 62.5 (2-C), 60.9 (4-C), 55.4 (25-C), 55.3 (12-C), 52.3 (13-C), 38.6, 34.0, 31.5, 28.2, 28.0, 22.1, 19.6 (3-C and 17-C to 22-C); **LCMS** *m/z* (EI) 510 ($[M+H]^+$ 100), 358 (17); **HRMS** Found 510.2474 $C_{29}H_{36}NO_7$, $[M + H]^+$, Requires 510.2486.

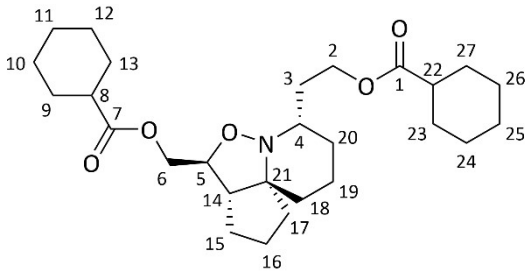
*[(3aS,4S,7S,10aS)-7-{2-[(4-fluorobenzoyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl 4-fluorobenzoate* **(17c)**



**17c**

**15** (1 eq) was treated with 4-fluorobenzoyl chloride (1.1 eq) to give **17c** as a white residue, 0.022 g, 9.4 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$), 2937, 2867, 1716, 1602, 1507, 1270, 1238, 1152; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.07-8.00 (4H, m, 9-H, 21-H), 7.14-7.04 (4H, m, 10-H, 22-H), 4.54 (1H, dd, *J* 11.6, 4.58, $6_A$-H), 4.47-4.43 (2H, m, 2-$H_2$), 4.36 (1H, dd, *J* 11.5, 6.90, $6_B$-H), 3.99-3.98 (1H, m, 5-H), 2.93-2.92 (1H, m, 4-H), 2.50-2.47 (1H, m, 12-H), 2.26-2.23 (1H, m, $18_B$-H), 2.01-1.28 (13H, m, 3-$H_2$, 13-$H_2$, 14-$H_2$, 15-$H_2$, 16-$H_2$, 17-$H_2$, $18_A$-H); $\delta_c$ (75 MHz, $CDCl_3$) 165.7 (1-C), 165.3 (7-C), 164.6 (23-C), 164.4 (11-C), 132.2 (9-C), 132.1 (21-C), 126.7 (20-C), 126.1 (8-C), 115.7 (22-C), 115.4 (10-C), 82.6 (5-C), 76.3 (19-C), 67.1 (6-C), 63.0 (2-C), 60.8 (4-C), 52.6 (12-C), 34.1, 31.9, 31.4, 29.4, 28.1, 22.7, 19.4 (3-C and 13-C to 18-C); **MS** *m/z* (EI) 486 ($M^+$ 100), 346 (17), 318 (58), **HRMS** Requires 486.2073 $C_{27}H_{30}F_2NO_5$, $[M + H]^+$, Found 486.2087.

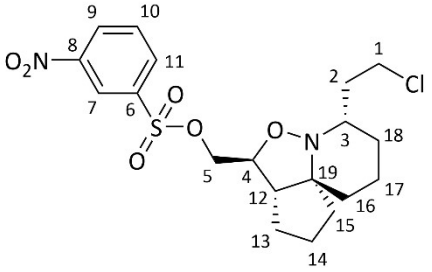
*[(3aS,4S,7S,10aS)-7-{2-[(cyclohexanecarbonyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl cyclohexanecarboxylate* **(17d)**




**17d**

**15** (1 eq) was treated with cyclohexane carbonoyl chloride (1.1 eq) to give **17d** as a colourless oil, 0.11 g, 50 %. No further purification was required. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$), 2929, 2854, 1728, 1450, 1225, 1168; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 4.26-4.21 (1H, dd, *J* 4.97, $6_B$-H), 4.17-4.11 (3H, m, 2-H, $6_A$-H), 3.83-3.78 (1H, m, 5-H), 2.79-2.73 (1H, m, 4-H), 2.40-2.37 (1H, m, 14-H), 2.35-2.22 (2H, m, 8-H, 22-H), 2.11-2.04 (1H, m, $20_A$-H), 1.98-1.19 (33H, m, 3-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$, 13-$H_2$, 15-$H_2$, 16-$H_2$, 17-$H_2$, 18-$H_2$, 19-$H_2$, $20_B$-H, 23-$H_2$, 24-$H_2$, 25-$H_2$, 26-$H_2$, 27-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 176.1 (1-C), 175.8 (7-C), 82.7 (5-C), 76.3 (21-C), 66.4 (6-C), 61.9 (2-C), 60.8 (4-C), 52.3 (14-C), 43.2 (22-C), 43.1 (8-C), 38.5, 34.5, 33.9, 31.5, 29.1, 29.0, 29.0, 28.2, 28.1, 27.7, 25.8, 25.7, 25.5, 25.4, 25.4, 22.1, 19.6 (3-C, 9-C to 13-C, 15-C to 20-C and 23-C to 27-C); **LCMS** *m/z* (EI) 462 $[M + H]^+$ 100), 334 (100); **HRMS** Found 462.3209 $C_{27}H_{44}NO_5$, $[M + H]^+$, Requires 462.3214.

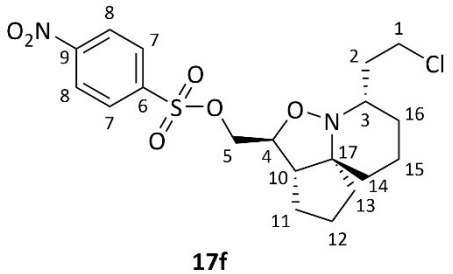
*[(3aS,4S,7S,10aS)-7-(2-chloroethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl 4-nitrobenzene-1-sulfonate* **(17e)**




**17e**

**15** (1 eq) was treated with 3-nitrobenzene sulfonoyl chloride (3.3 eq) to give **17e** as a brown oil, 0.025 g, 12 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2936, 2872, 1607, 1532, 1447, 1372, 1351, 1189, 733; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.77-8.74 (1H, m, 7-H), 8.53-8.50 (1H, m, 9-H), 8.27-8.25 (1H, m, 11-H), 7.81 (1H, t, *J* 8.05, 10-H), 4.34 (1H, ddd, *J* 4.64, $5_A$-H), 4.25 (1H, ddd, *J* 6.28, $5_B$-H), 3.86-3.82 (1H, m, 4-H), 3.66-3.48 (2H, m, 1-$H_2$), 2.72-2.65 (1H, m, 3-H), 2.43-2.40 (1H, m, 12-H), 2.15-2.06 (1H, m, $18_A$-H), 1.93-1.32 (13H, m, 2-$H_2$, 13-$H_2$, 14-$H_2$, 15-$H_2$, 16-$H_2$, 17-$H_2$, $18_B$-H); $\delta_c$ (75 MHz, $CDCl_3$) 148.3 (8-C), 138.2 (6-C), 133.4 (11-C), 130.8 (10-C), 128.4 (9-C), 123.2 (7-C), 81.9 (4-C), 76.5 (19-C), 72.5 (5-C), 60.7 (3-C), 51.9 (12-C), 42.5 (1-C), 38.3 (2-C), 38.0 (18-C), 31.2, 29.7, 27.8, 22.0, 19.3 (13-C to 17-C); **MS** *m/z* (EI) 445 ($[M + H]^+$ 100), 242 (18), **HRMS** Found 445.1195 $C_{19}H_{25}ClN_2O_6S$, $[M + H]^+$, Requires 445.1200.

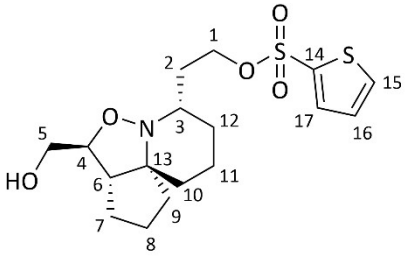
*[(3aS,4S,7S,10aS)-7-(2-chloroethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl 4-nitrobenzene-1-sulfonate* **(17f)**



**17f**

**15** (1 eq) was treated with 4-nitrobenzene sulfonoyl chloride (2.2 eq) to give **17f** as a white residue, 0.046 g, 26 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2938, 2871, 1604, 1532, 1446, 1403, 1370, 1185, 640; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.42-8.39 (2H, m, 8-H), 8.14-8.11 (2H, m, 7-H), 4.28 (2H, ddd, *J* 6.34, 5-$H_2$), 3.85-3.81 (1H, m, 4-H), 3.64-3.49 (2H, m, 1-$H_2$), 2.72-2.65 (1H, m, 3-H), 2.41-2.38 (1H, m, 10-H), 2.15-2.06 (1H, m, 2-H), 1.92-1.19 (13H, m, 2-H, 11-$H_2$, 12-$H_2$, 13-$H_2$, 14-$H_2$, 15-$H_2$, 16-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 150.8 (9-C), 141.7 (6-C), 129.3 (7-C), 124.5 (8-C), 81.9 (4-C), 76.5 (17-C), 72.6 (5-C), 60.7 (3-C), 51.8 (10-C), 42.5 (1-C), 38.3, 38.0, 31.2, 27.8, 27.7, 21.9, 19.3 (2-C and 11-C to 16-C); **HRMS** (TOF MS) 445 ($[M + H]^+$ 100).

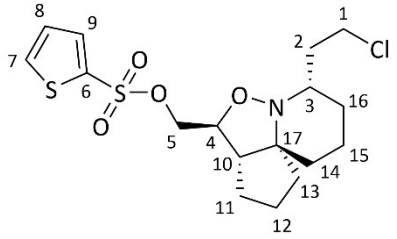
*2-[(3aS,4S,7S,10aS)-4-(hydroxymethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]ethyl thiophene-2-sulfonate* **(17ga)**




**17ga**

**15** (1 eq) was treated with 2-thiophenesulfonoyl chloride (2.2 eq) to give **17ga** as a colourless oil, 0.048 g, 22 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3383-OH, 2936, 2871, 1403, 1445, 1368, 1227, 1177; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 7.74-7.73 (1H, m, 15-H), 7.71-7.70 (1H, m, 17-H), 7.15-7.14 (1H, m, 16-H), 4.24-4.23 (2H, m, 1-$H_2$), 3.84-3.75 (2H, m, 4-H, $5_B$-H), 3.66-3.62 (1H, m, $5_A$-H), 2.94-2.90 (1H, m, 3-H), 2.42-2.38 (1H, m, 6-H), 1.97-1.45 (2-$H_2$, 7-$H_2$, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 135.6 (14-C), 134.5 (15-C), 133.1 (17-C), 128.2 (16-C), 80.8 (4-C), 76.3 (13-C), 71.1 (1-C), 60.9 (3-C), 60.4 (5-C), 52.6 (6-C), 37.9, 35.5, 34.7, 30.9, 27.3, 23.7, 18.9 (2-C and 7-C to 12-C); **MS** (EI) 388 ([M + H]$^+$ 100), 370 (60), **HRMS** Found 388.1240, $C_{17}H_{26}NO_5S_2$ [M + H]$^+$, Requires 388.1247.

*[7-(2-chloroethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl thiophene-2-sulfonate* **(17gb)**

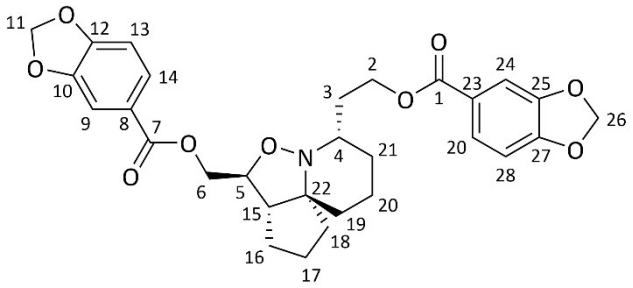



**17gb**

**15** (1 eq) was treated with 2-thiophenesulfonoyl chloride (2.2 eq) to give **17gb** as a colourless oil, 0.051 g, 24 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2938, 2870, 1445, 1368, 1228, 1177, 727; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 7.73 (1H, dd, *J* 3.79, 1.36, 7-H), 7.72 (1H, dd, *J* 5.02, 1.35, 9-H), 7.16, (1H, dd, *J* 5.01, 3.80, 8-H), 4.28-4.21 (2H, m, 5-$H_2$), 3.84-3.80 (1H, M, 4-H), 3.65-3.65-3.52 (2H, m, 1-$H_2$), 2.71-2.65 (1H, m, 3-H), 2.44-2.41 (1H, m, 10-H), 2.19-2.11 (1H, m, $16_A$-H), 1.95-1.36 (13H, m, 2-$H_2$, 11-$H_2$, 12-$H_2$, 13-$H_2$, 14-$H_2$, 15-$H_2$, $16_B$-H); $\delta_c$ (75 MHz, $CDCl_3$) 135.4 (6-C), 134.5 (9-C), 133.9 (7-C), 127.6 (8-C), 82.0 (4-C), 76.6 (17-C), 72.0 (5-C), 61.1 (3-C), 51.5 (10-C), 42.5 (1-C), 38.3, 38.2, 31.4, 28.3, 27.8, 21.7, 19.5 (2-C and 11-C to 16-C); **MS** (EI) 428 ($[M + Na]^+$ 100), 406 ($[M + H]^+$ 85), 370 (43); **HRMS** Found 406.0904 $C_{17}H_{25}ClNO_4S_2$, $[M + H]^+$, Requires 406.0908.

*[(3aS,4S,7S,10aS)-7-{2-[(2H-1,3-benzodioxole-5-carbonyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl 2H-1,3-benzodioxole-5-carboxylate*

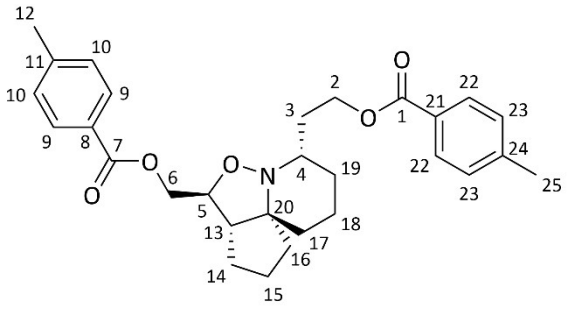
**(17h)**



**17h**

**15** (1 eq) was treated with piperonyloyl chloride (1.8 eq) to give **17h** as an opaque oil, 0.071 g, 29%. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2929, 2250, 1709, 1505, 1442, 1277; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 7.63 (1H, dd, *J* 14.8, 1.64, 14-H), 7.60 (1H, dd, *J* 14.8, 1.62, 29-H), 7.43 (1H, d, *J* 15.74, 1.74,13-H), 7.41 (1H, dd, *J* 15.74, 1.74, 28-H), 6.81-6.77 (2H, m, 9-H, 24-H), 6.01 (2H, s, 11-H), 5.99 (2H, s, 26-H), 4.48 (1H, dd, *J* 11.6, 4.63, $6_B$-H), 4.41-4.38 (2H, m, 2-$H_2$), 4.32 (1H, dd, *J* 11.6, 6.67, $6_A$-H), 3.97-3.92 (1H, m, 5-H), 2.92-2.88 (1H, m, 4-H), 2.49-2.46 (1H, m, 15-H), 2.25-2.20 (1H, m, 3-H), 2.01-1.50 (13H, m, 3-H, 16-$H_2$, 17-$H_2$, 18-$H_2$, 19-$H_2$, 20-$H_2$, 21-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 165.9 (1-C), 165.6 (7-C), 151.7 (12-C), 151.4 (27-C), 147.7 (10-C), 147.6 (25-C), 125.5 (14-C), 125.2 (29-C), 124.5 (8-C), 123.9 (23-C), 109.5 (13-C), 109.4 (28-C), 108.0 (9-C), 107.9 (24-C), 101.8 (11-C), 101.7 (26-C), 82.7 (5-C), 76.3 (22-C), 66.8 (6-C), 62.7 (2-C), 60.8 (4-C), 52.4 (15-C), 38.6, 34.1, 31.5, 29.8, 28.1, 22.2, 19.5 (3-C and 16-C to 21-C); **MS** *m/z* (FT) 538 ($[M+H]^+$ 100), 372 (13), **HRMS** Found 538.2065, $C_{29}H_{31}O_9N$ ($M+H^+$), requires 538.2072.

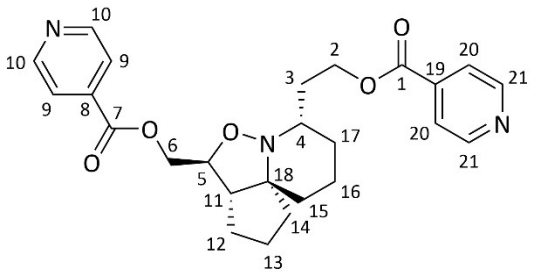
*[(3aS,4S,7S,10aS)-7-{2-[(4-methylbenzoyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl 4-methylbenzoate* **(17i)**



**17i**

**15** (1 eq) was treated with *p*-toluoyl chloride (4 eq) to give **17i** as a brown oil, 0.076 g, 38 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$)2937, 1713, 1611, 1447, 1271, 1209, 1177; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 7.91 (4H, dd, *J* 15.1, 8.21, 9-H, 22-H), 7.20 (4H, dd, *J* 13.5, 8.21, 10-H, 23-H), 4.52 (1H, dd, *J* 11.6, 4.79, $6_B$-H), 4.48-4.36 (3H, m, 2-$H_2$, $6_A$-H), 4.01-3.96 (1H, m, 5-H), 2.94-2.90 (1H, m, 4-H), 2.53-2.50 (1H, m, 13-H), 2.39 (3H, s, 12-$H_3$), 2.37 (3H, s, 25-$H_3$), 2.32-2.24 (1H, m, $3_B$-H), 2.03-1.33 (13H, m, $3_A$-H, 14-$H_2$, 15-$H_2$, 16-$H_2$, 17-$H_2$, 18-$H_2$, 19-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 166.7 (1-C), 166.2 (7-C), 143.8 (11-C), 143.3 (24-C), 129.7 (22-C), 129.5 (9-C), 129.1 (23-C), 129.0 (10-C), 127.7 (21-C), 127.1 (8-C), 82.7 (5-C), 76.3 (20-C), 66.7 (6-C), 62.6 (2-C), 60.9 (4-C), 52.3 (13-C), 38.5, 33.9, 31.5, 28.2, 28.0, 22.1, 21.7, 21.6, 19.5 (3-C, 12-C, 14-C to 19-C and 25-C); **MS** (EI) 500 ($[M+Na]^+$ 14), 478 (100), 342 (12), **HRMS** Requires 478.2581 $C_{29}H_{36}NO_5$, $[M+H]^+$, Found 478.2588.

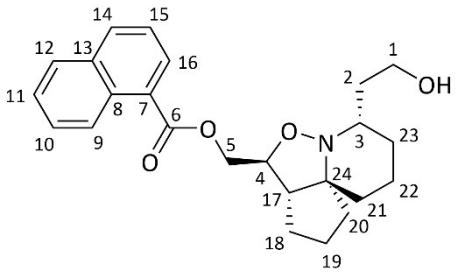
*[(3aS,4S,7S,10aS)-7-{2-[(pyridine-4-carbonyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl pyridine-4-carboxylate* **(17j)**



**17j**

**15** (1 eq) was treated with isonicotinoyl chloride (3 eq) to give **17j** as a yellow oil, 0.067 g, 36 %. No further purification was required. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2931, 2361, 1727, 1597, 1446, 1124; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.76-8.71 (4H, m, 10-H, 21-H), 7.81-7.76 (4H, m, 9-H, 20-H), 4.54-4.46 (3H, m, 2-$H_2$, $6_A$-H), 4.37 (1H, dd, *J* 11.6, 7.11, $6_B$-H), 3.97-3.92 (1H, m, 5-H), 2.91-2.88 (1H, m, 4-H), 2.45-2.42 (1H, m, 11-H), 2.21-2.13 (1H, m, $3_B$-H), 1.97-1.32 (13H, m, $3_A$-H, 12-$H_2$, 13-$H_2$, 14-$H_2$, 15-$H_2$, 16-$H_2$, 17-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 165.1 (1-C), 164.8 (7-C), 150.7 (21-C), 150.6 (10-C), 137.5 (19-C), 137.0 (8-C), 122.9 (9-C), 122.8 (20-C), 82.1 (5-C), 76.2 (18-C), 67.6 (6-C), 63.7 (2-C), 60.4 (3-C), 52.7 (11-C), 38.6, 34.2, 31.1, 28.1, 27.9, 22.4, 19.3 (3-C and 12-C to 17-C); **MS** (EI) 474 ($[M+Na]^+$ 12), 452 (100), 329 (15); **HRMS** Found 452.2172 $C_{25}H_{30}N_3O_5$, $[M+H]^+$, Requires 452.2180.

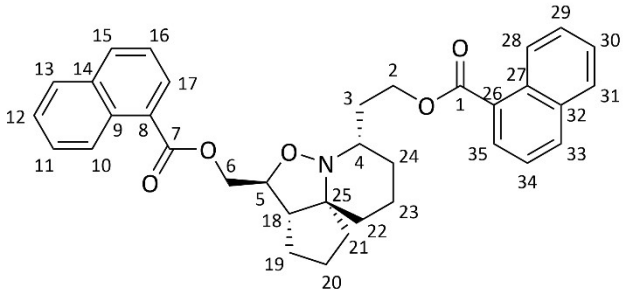
*[(3aS,4S,7S,10aS)-7-(2-hydroxyethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl naphthalene-1-carboxylate* **(17ka)**




**17ka**

**15** (1 eq) was treated with 1-napthoyl chloride (1.5 eq) to give **17ka** as a white residue, 0.023 g, 7.0 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3397-OH, 2936, 2867, 1714, 1593, 1510, 1445, 1245, 1196, 1134; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.93 (1H, d, *J* 8.66, 9-H), 8.23 (1H, dd, *J* 7.28, 1.19, 16-H), 8.04-8.02 (1H, m, 14-H), 7.90-7.88 (1H, m, 12-H), 7.65-7.60 (1H, m, 10-H), 7.56-7.49 (2H, m, 11-H, 15-H), 4.62-4.51 (2H, m, 5-$H_2$), 4.03-3.99 (1H, m, 4-H), 3.96-3.90 (1H, m, $1_B$-H), 3.75-3.70 (1H, m, $1_A$-H), 3.18-3.13 (1H, m, 3-H), 2.52-2.48 (1H, m, 17-H), 2.09-1.49 (14H, m, 2-$H_2$, 18-$H_2$, 19-$H_2$, 20-$H_2$, 21-$H_2$, 22-$H_2$, 23-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 167.3 (6-C), 133.8 (13-C), 133.6 (14-C), 131.4 (7-C), 130.5 (16-C), 128.6 (12-C), 127.9 (10-C), 126.7 (8-C), 126.3 (11-C), 125.8 (9-C), 124.5 (15-C), 81.9 (4-C), 76.0 (24-C), 66.2 (5-C), 61.3 (3-C), 60.5 (1-C), 54.0 (17-C), 38.7, 35.5, 30.7, 28.4, 26.2, 23.1, 18.8 (2-C and 18-C to 23-C); **MS** 418 ([M+Na]$^+$ 100), 396 (14); **HRMS** Found 396.2161 $C_{24}H_{30}NO_4$, [M+H]$^+$, Requires 396.2169.

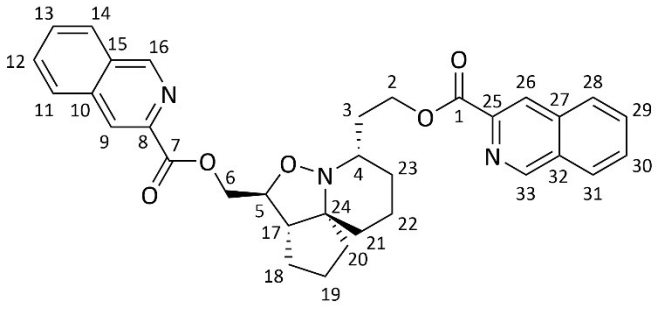
*[(3aS,4S,7S,10aS)-7-{2-[(naphthalene-1-carbonyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl naphthalene-1-carboxylate* **(17kb)**



**17kb**

**15** (1 eq) was treated with 1-napthoyl chloride (1.5 eq) to give **17kb** as a white residue, 0.14 g, 32 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 2924, 2855, 1711, 1593, 1510, 1455, 1241, 1195, 1133; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.91 (2H, t, *J* 9.30, 10-H, 28-H), 8.22 (1H, dd, *J* 7.28, 17-H), 8.15 (1H, dd, *J* 7.26, 33-H), 8.00 (1H, d, *J* 8.20, 15-H), 7.94 (1H, d, *J* 8.20, 35-H), 7.87-7.82 (2H, m, 13-H, 31-H), 7.61-7.41 (6H, m, 11-H, 12-H, 16-H, 29-H. 30-H, 34-H), 4.62 (1H, m, $6_B$-H), 4.58 (2H, t, *J* 6.81, 2-$H_2$), 4.50 (1H, dd, *J* 6.85, $6_A$-H), 4.09-4.04 (1H, m, 5-H), 3.05-3.00 (1H, m, 4-H), 2.57-2.53 (1H, m, 18-H), 2.40-2.33 (1H, m, $3_B$-H), 2.08-1.38 (13H, m, $3_A$-H, 19-$H_2$, 20-$H_2$, 21-$H_2$, 22-$H_2$, 23-$H_2$, 24-$H_2$); $\delta_c$ (75 MHz, $CDCl_3$) 167.6 (1-C), 167.2 (7-C), 133.8 (14-C), 133.7 (32-C), 133.5 (15-C), 133.1 (35-C), 131.4 (8-C), 131.3 (26-C), 130.5 (17-C), 130.1 (33-C), 128.5 (13-C), 128.4 (31-C), 127.8 (11-C), 127.6 (29-C), 127.4 (9-C), 126.7 (27-C), 126.2 (12-C), 126.1 (30-C), 125.8 (10-C), 125.7 (28-C), 124.5 (16-C, 34-C), 82.5 (5-C), 76.3 (25-C), 67.0 (6-C), 62.9 (2-C), 60.7 (4-C), 52.7 (18-C), 38.6 (3-C), 34.2, 31.3, 28.2, 22.4, 28.0, 19.4 (19-C to 24-C); **MS** (EI) 572 ([M+Na]$^+$ 10), 550 (100), 378 (22); **HRMS** Found 550.2587 $C_{35}H_{36}NO_5$, [M+H]$^+$, Requires 550.2588.

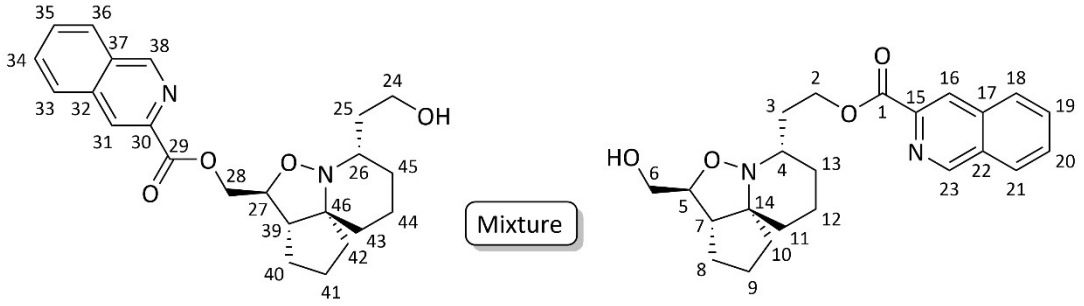
*[(3aS,4S,7S,10aS)-7-{2-[(isoquinoline-3-carbonyl)oxy]ethyl}octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl isoquinoline-3-carboxylate* **(18la)**



**17la**

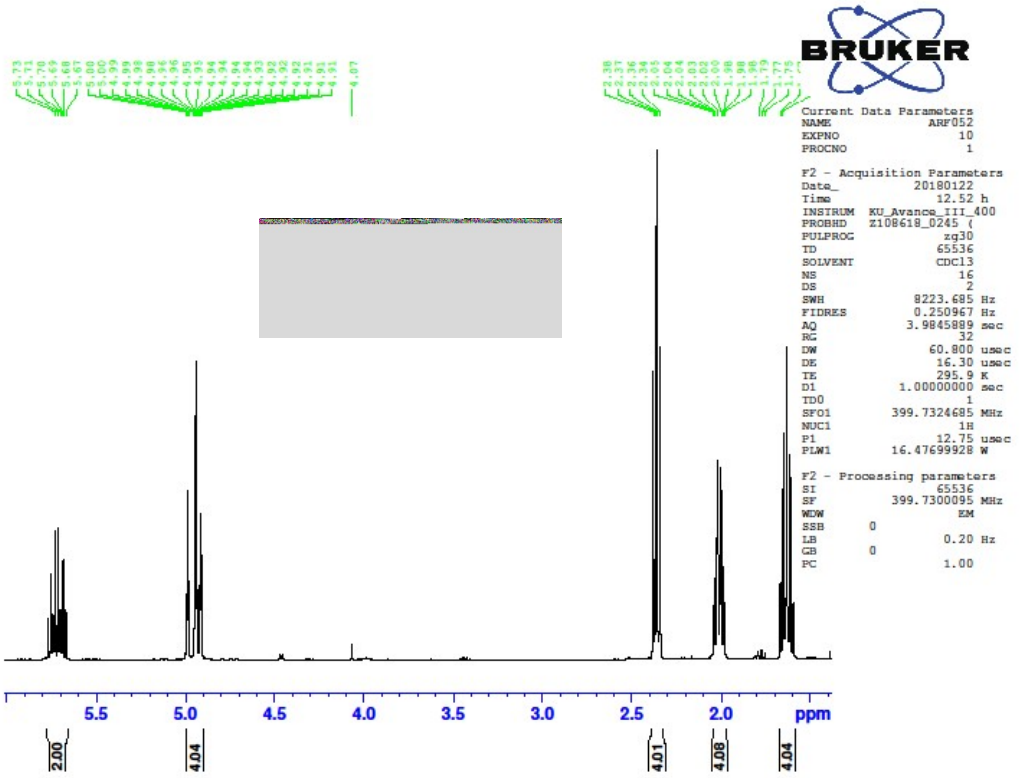
**15** (1 eq) was treated with quinaldoyl chloride (1.3 eq) to give **17la** as a yellow oil. The crude mixture was purified via flash chromatography, eluting with DCM/MeOH 9:1 to give a yellow oil, 0.13 g, 39 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$)2937, 2236, 1721, 1593, 1564, 1504, 1462, 1243, 1136, 1106; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.26-8.19 (4H, m, 9-H, 16-H, 26-H, 33-H), 8.12-8.08 (2H, m, 11-H, 28-H), 7.90-7.56 (6H, m, 13-H, 13-H, 14-H, 29-H, 30-H, 31-H), 4.70 (1H, dd, *J* 7.47, 4.25, 6-H), 4.66-4.57 (3H, m, 2-$H_2$, 6-H), 4.10-4.05 (1H, m, 5-H), 3.15-3.08 (1H, m, 4-H), 2.64-2.60 (1H, m, 17-H), 2.41-2.33 (1H, m, 3-H), 2.01-1.37 (13H, m, 3-H, 18-$H_2$, 19-$H_2$, 20-$H_2$, 21-$H_2$, 22-$H_2$, 23-$H_2$); $\delta_C$ (75 MHz, $CDCl_3$) 165.3 (7-C), 164.9 (1-C), 148.2 (8-C), 147.6 (25-C), 147.6 (15-C), 147.5 (32-C), 137.2, 137.1, 130.7, 130.2, 130.1, 129.3 (10-C), 129.2 (17-C), 128.6, 128.4, 127.5, 127.4, 121.1, 121.0, 82.7 (5-C), 76.4 (24-C), 67.4 (6-C), 64.1 (2-C), 60.7 (4-C), 51.9 (17-C), 38.5, 34.0, 31.6, 28.5, 27.9, 22.1, 19.6 (3-C and 18-C to 23-C); **MS** (EI) 552 ($[M+H]^+$ 100), 379 (85), 365 (17), 351 (36); **HRMS** Found 552.2481 $C_{33}H_{34}N_3O_5$, $[M+H]^+$, Requires 552.2493.

*[7-(2-hydroxyethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-4-yl]methyl isoquinoline-3-carboxylate and 2-[4-(hydroxymethyl)octahydro-1H-cyclopenta[3,4][1,2]oxazolo[2,3-a]pyridin-7-yl]ethyl isoquinoline-3-carboxylate* **(17lb)** and **(17lc)**




**17lc** **17lb**

**15** was treated with quinaldoyl chloride (1.3 eq) to give a mixture of **17lb** and **17lc** as an opaque residue, 0.027 g, 11 %. **IR** $\nu_{max}$ (thin film)/$cm^{-1}$) 3374, 2934, 2867, 1720, 1657, 1593, 1564, 1505, 1463, 1293, 1243, 1139, 1107; **NMR** $\delta_H$ (400 MHz, $CDCl_3$) 8.39-3.19 (6H, m, 16-H, 18-H, 23-H, 31-H, 33-H, 38-H), 7.90-7.64 (6H, m, 19-H, 30-H, 21-H, 34-H, 35-H, 36-H), 5.34 (1H, Br s, O-H), 4.79-4.74 (1H, m, 28-H), 4.65 (2H, d, *J* 5.05, 2-$H_2$), 4.59-4.53 (1H, m, 28-H), 4.09-4.05 (1H, m, 27-H), 3.99-3.90 (2H, m, 6-H, 24-H), 3.84-3.81 (1H, m, 5-H), 3.77-3.68 (2-H, m, 6-H, 24-H), 3.41-3.34 (1H, m, 4-H), 3.24-3.20 (1H, m, 26-H), 2.85-2.82 (1H, m, 39-H), 2.57-2.53 (1H, m, 7-H), 2.35-2.27 (1H, m, 3-H), 2.11-1.41 (27H, m, 3-H, 8-$H_2$, 9-$H_2$, 10-$H_2$, 11-$H_2$, 12-$H_2$, 13-$H_2$, 25-$H_2$, 40-$H_2$, 41-$H_2$, 42-$H_2$, 43-$H_2$, 44-$H_2$, 45-$H_2$; $\delta_c$ (75 MHz, $CDCl_3$) 165.5 (29-C), 165.1 (1-C), 148.1 (30-C), 147.6 (15-C), 147.5 (37-C), 147.1 (22-C), 137.8, 137.4, 130.6, 130.4, 129.8, 129.5 (32-C), 129.4 (17-C), 128.7, 127.6, 121.2, 121.1, 87.5 (5-C), 82.3 (27-C), 77.0 (14-C), 76.2 (46-C), 66.8 (2-C), 64.1 (28-C), 61.4 (6-C), 61.3 (26-C), 60.4 (24-C), 59.9 (4-C), 52.5 (7-C), 46.2 (39-C), 38.6, 38.3, 36.0, 35.6, 32.1, 31.2, 30.5, 28.1, 27.2, 27.1, 22.5, 20.7, 20.0, 19.2 (3-C, 8-C to 13-C, 25-C and 40-C to 45-C); **MS** (EI) 419 ($[M+Na]^+$ 100), 397 (13), 224 (10), **HRMS** Found 397.2119 $C_{23}H_{29}N_2O_4$, $[M+H]^+$, Requires 397.2122.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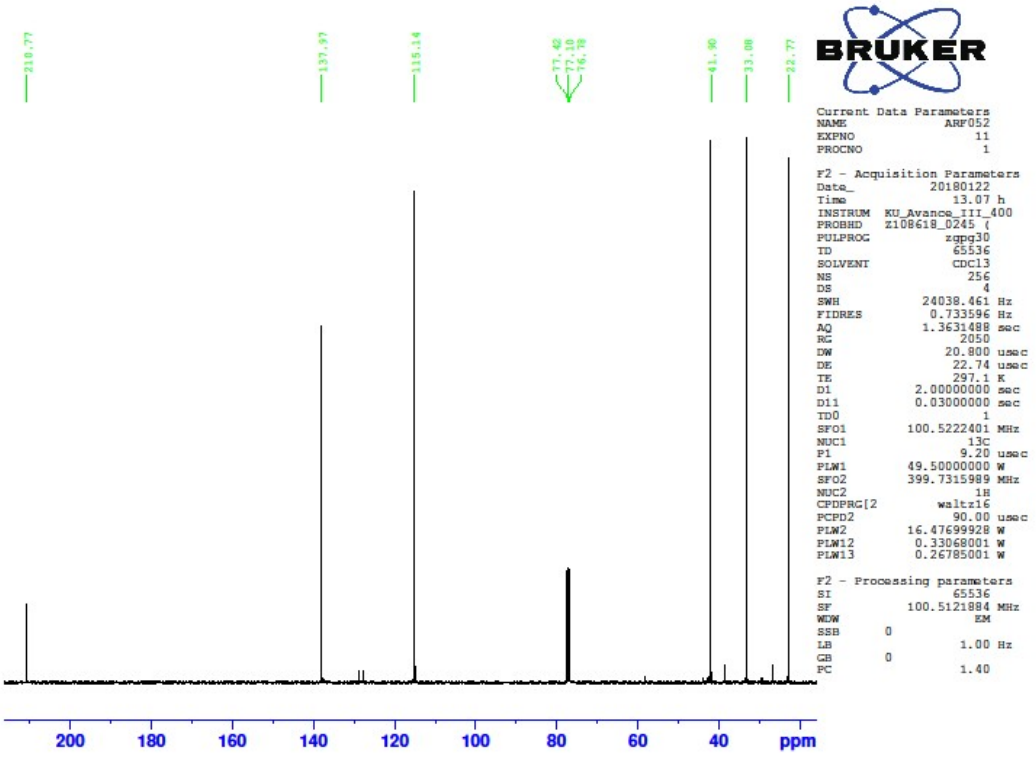
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SFO2 399.7315989 MHz
NUC2 1H
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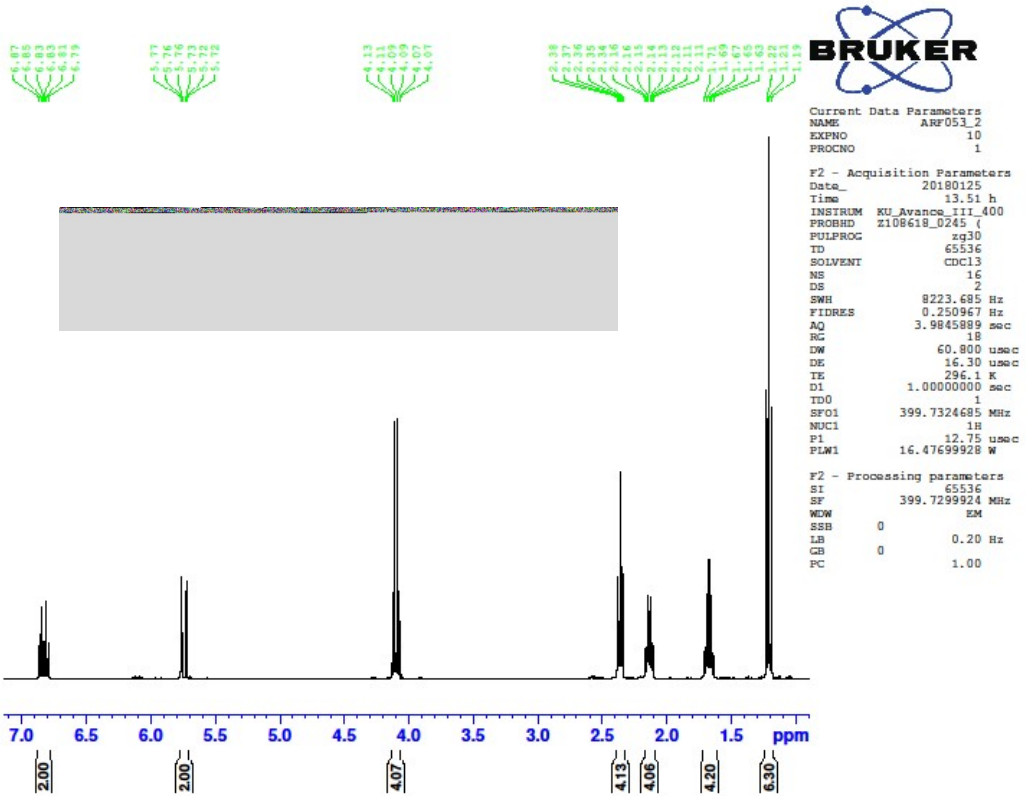
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NUC1 1H
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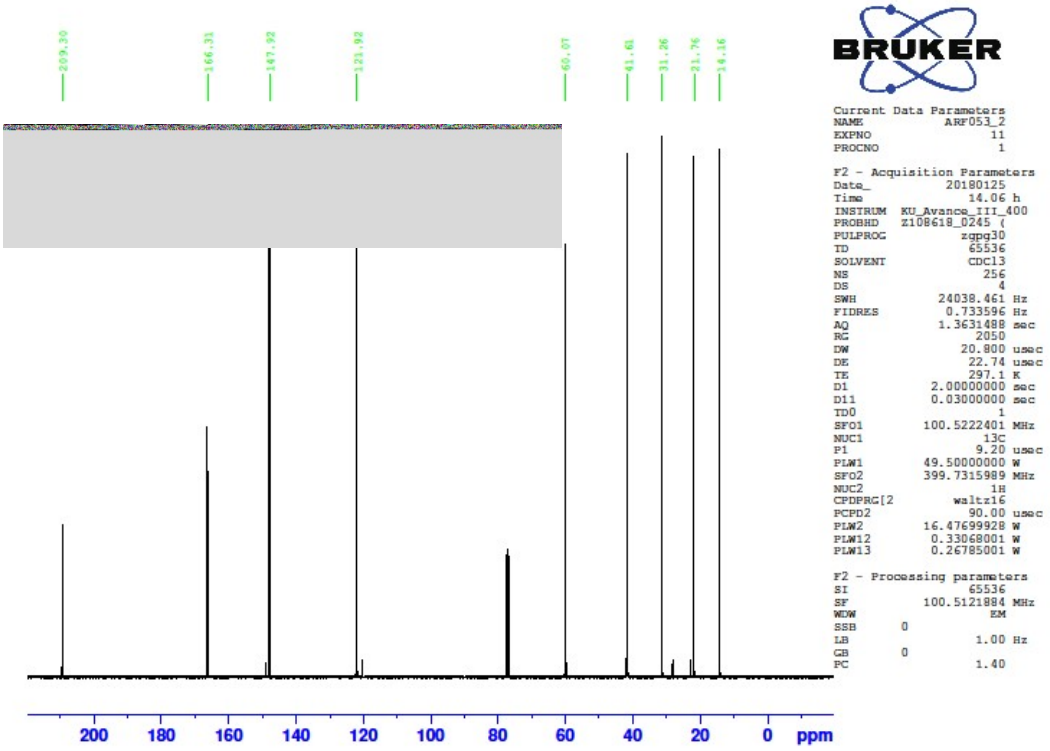
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D11 0.03000000 sec
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NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
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PLW13 0.26785001 W
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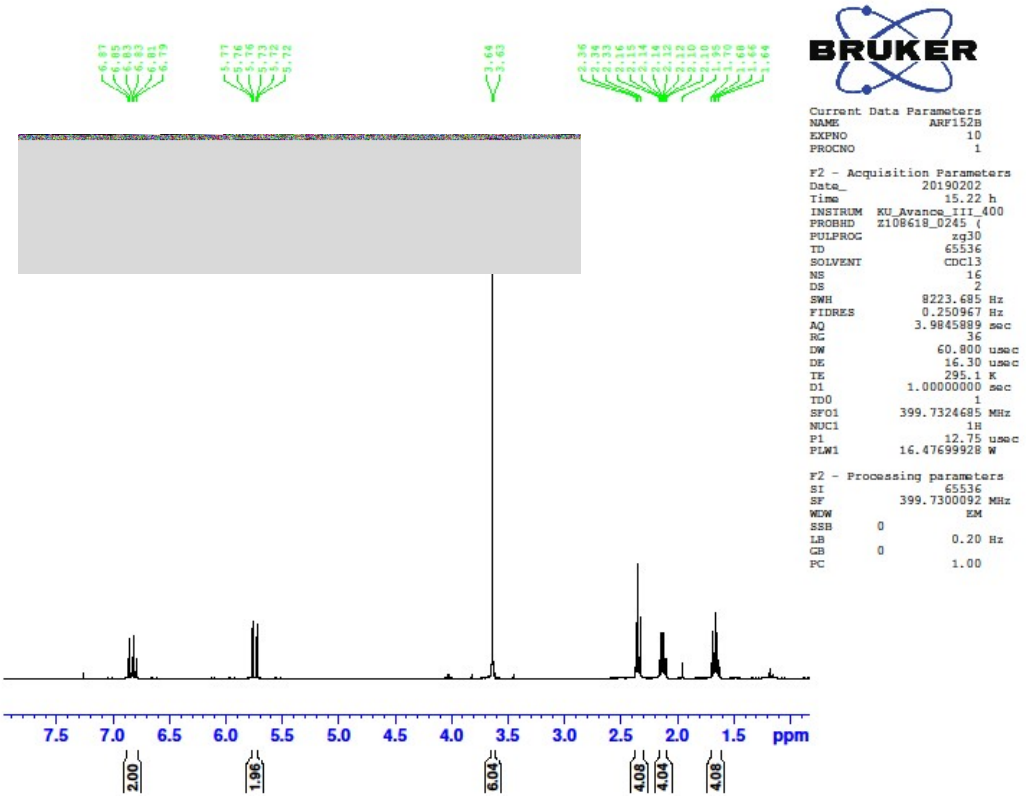
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NUC1 1H
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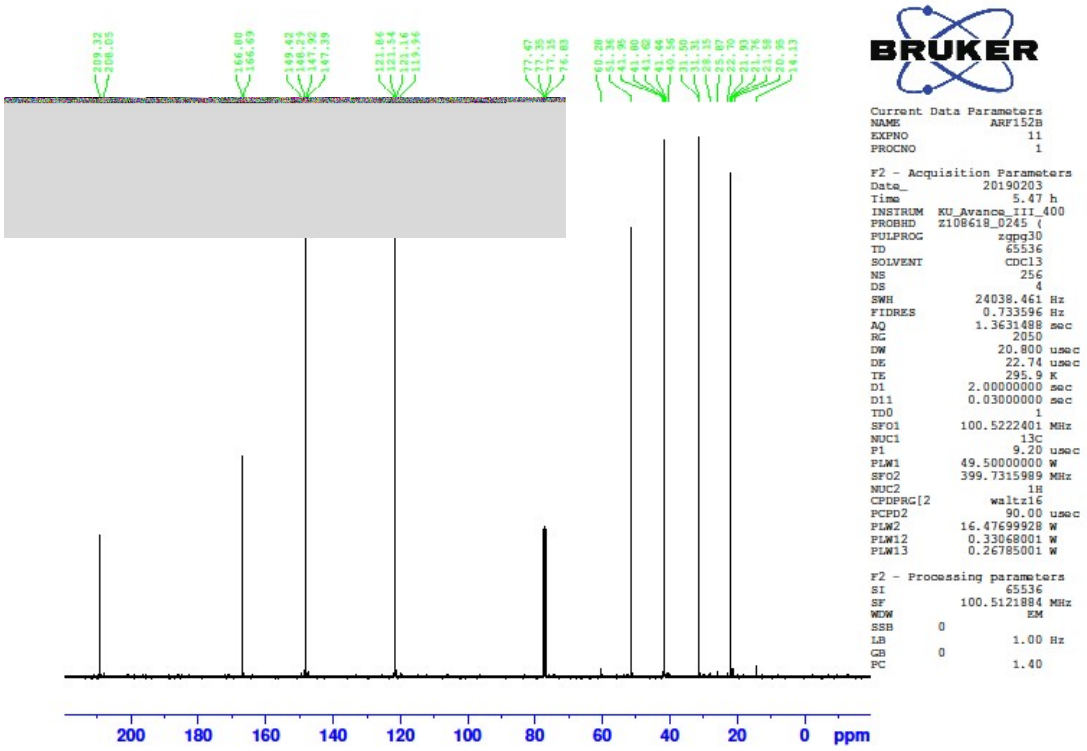
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DE 22.74 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
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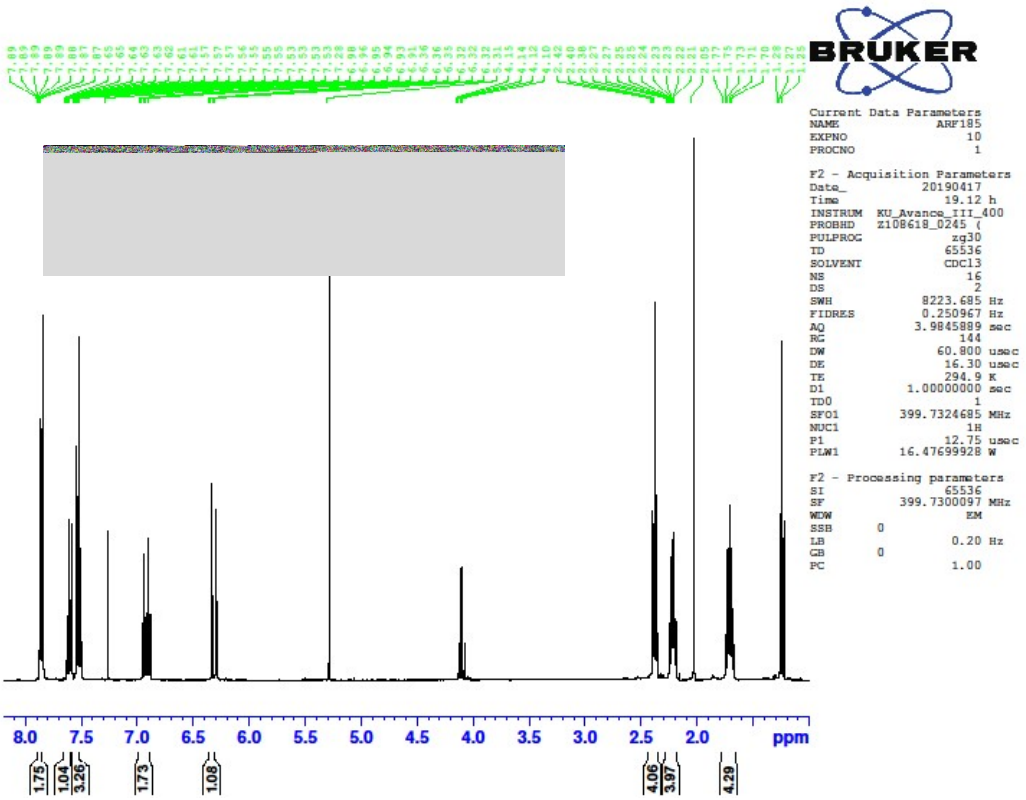
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NUC1 1H
P1 12.75 usec
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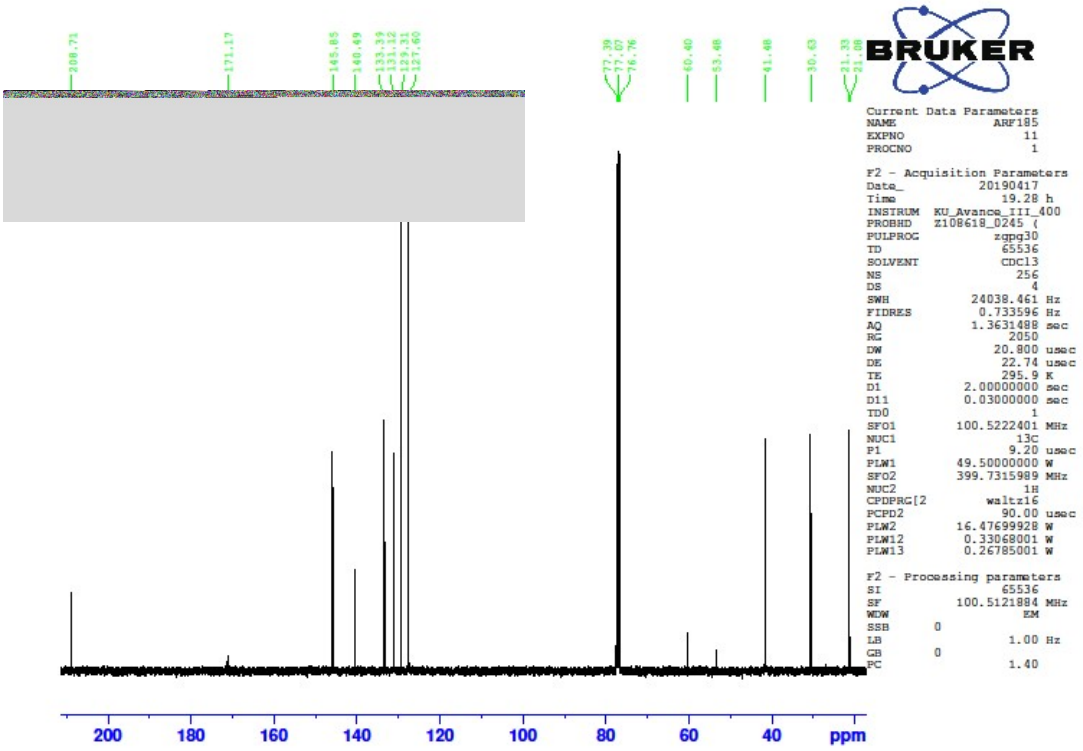
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PROCNO 1
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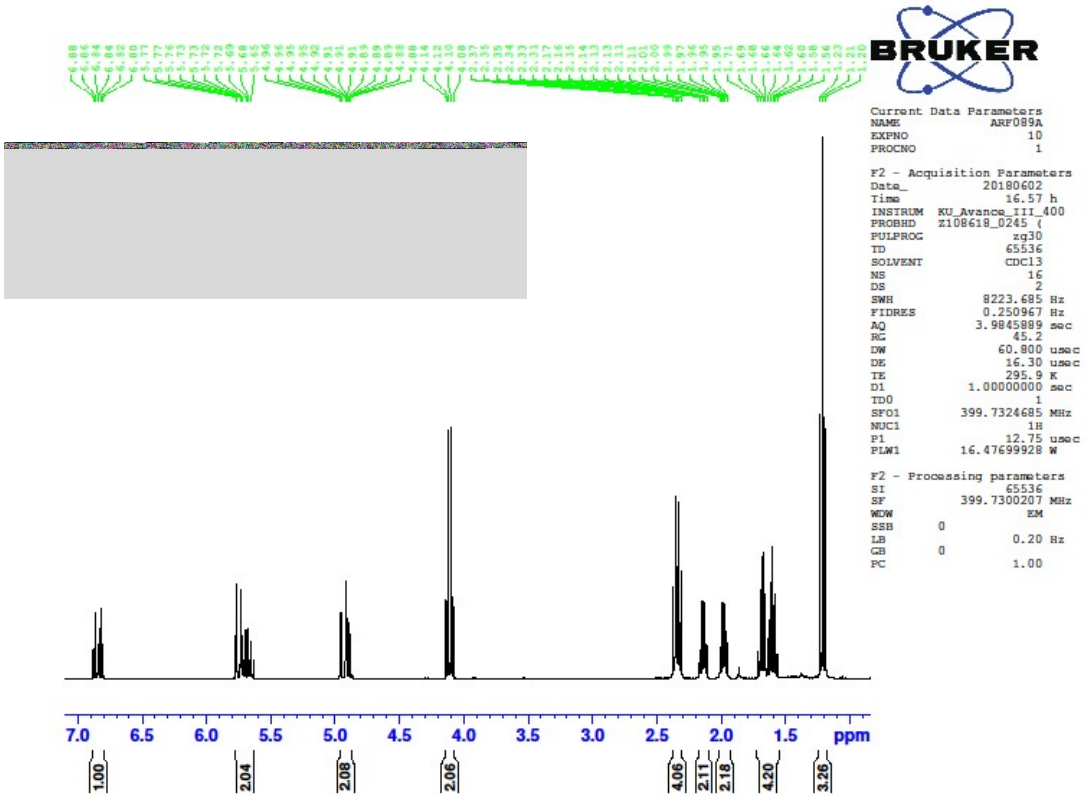
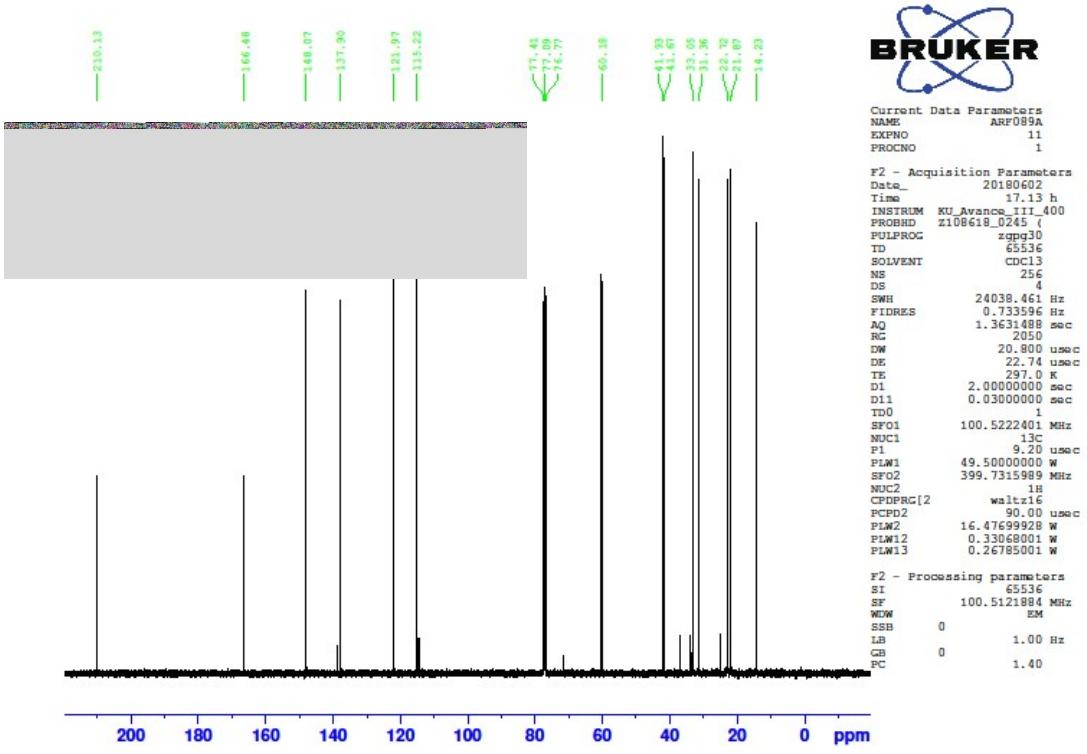
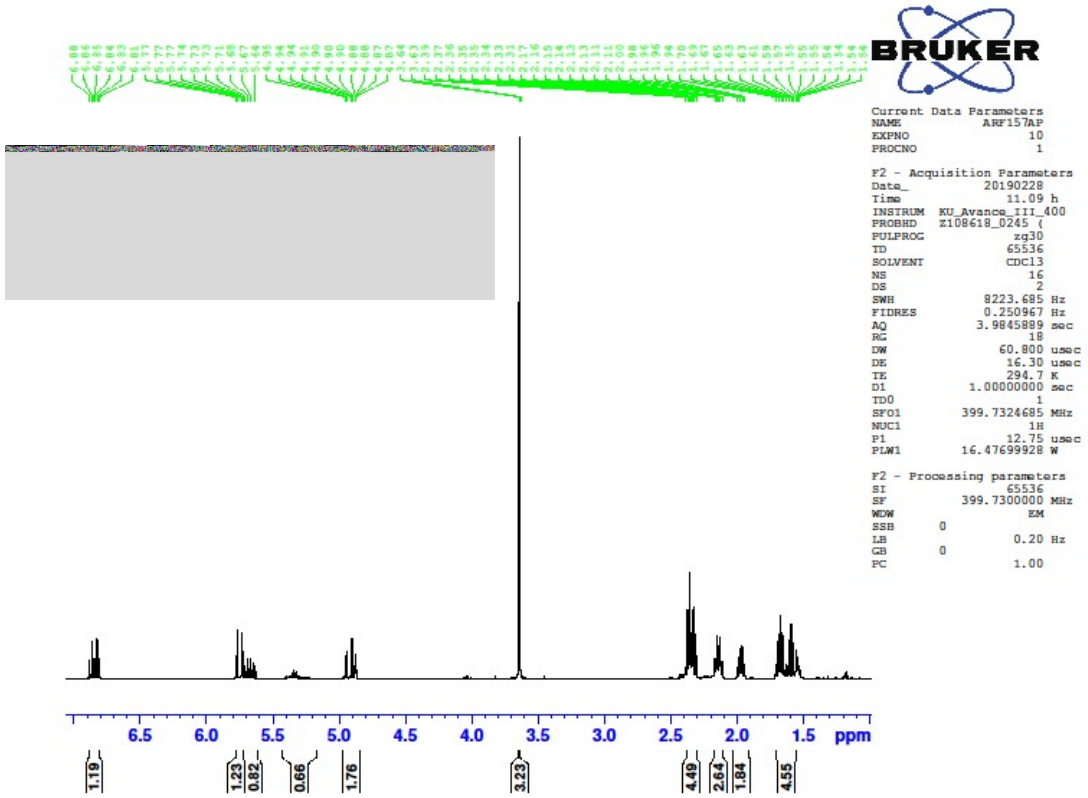
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
ppm



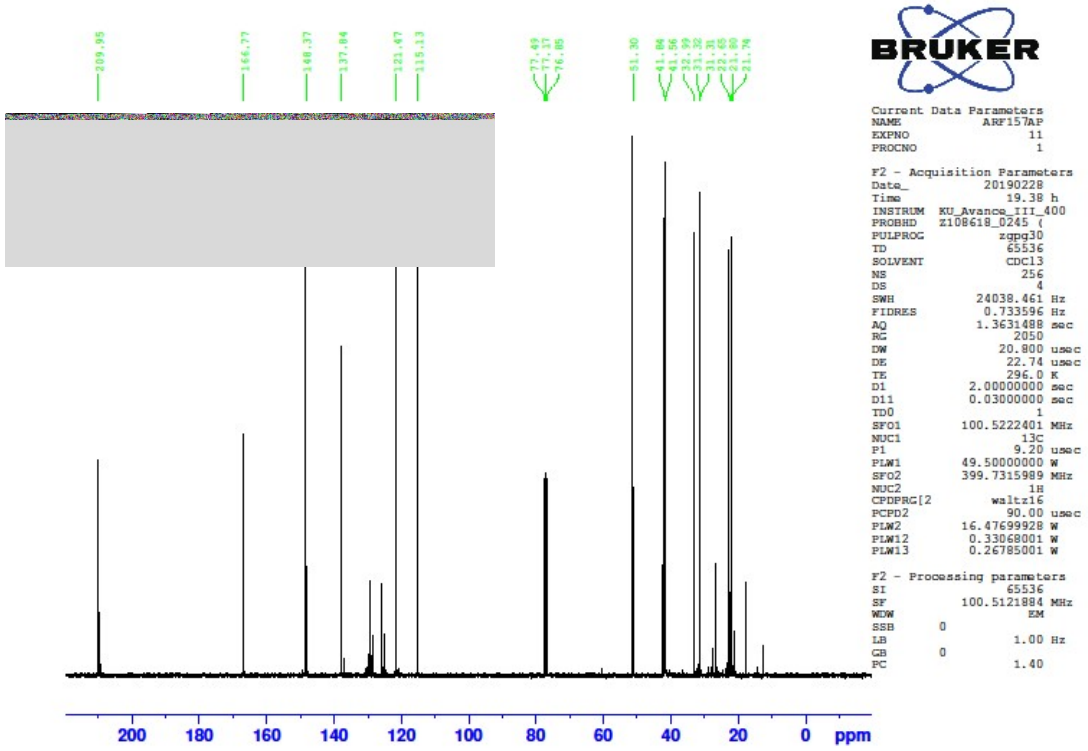

```
Current Data Parameters
NAME                 ARF089A
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180602
Time                  16.57 h
INSTRUM   KU_Avance_III_400
PROBHD    Z108618_0245 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.250967 Hz
AQ                 3.9845889 sec
RG                      45.2
DW                   60.800 usec
DE                   16.30 usec
TE                   295.9 K
D1                1.00000000 sec
TD0                       1
SFO1             399.7324685 MHz
NUC1                      1H
P1                    12.75 usec
PLW1              16.47699928 W

F2 - Processing parameters
SI                    65536
SF               399.7300207 MHz
WDW                      EM
SSB          0
LB                    0.20 Hz
GB           0
PC                    1.00
```



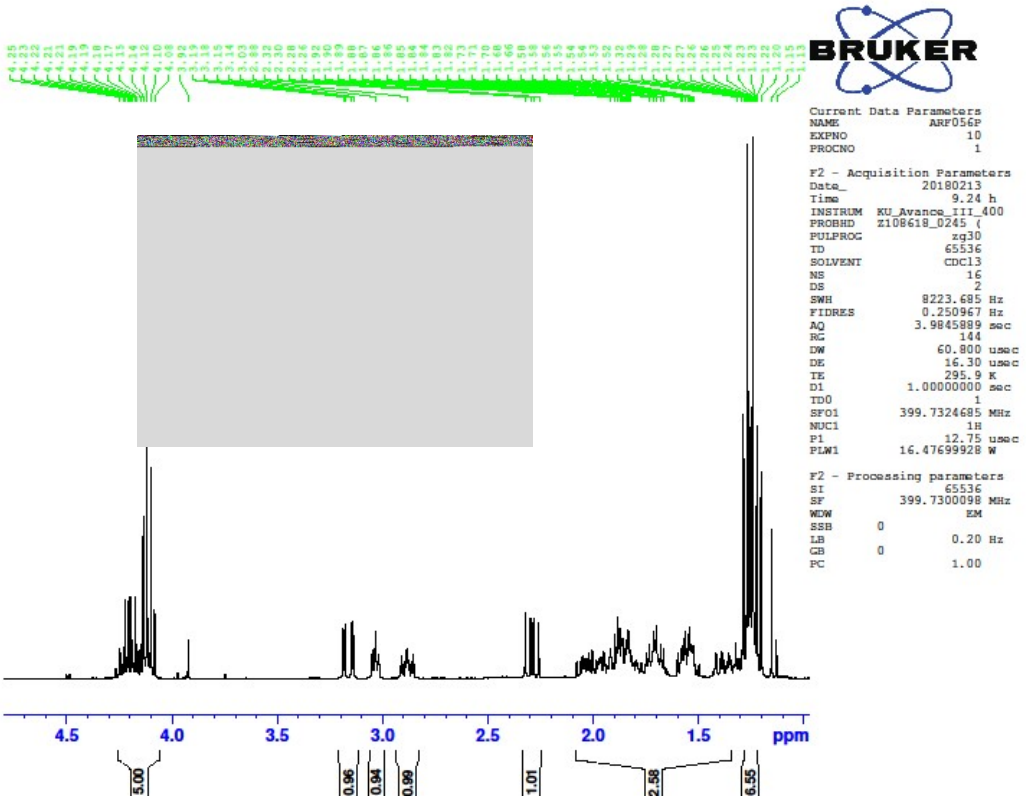

```
Current Data Parameters
NAME                 ARF089A
EXPNO                     11
PROCNO                     1

F2 - Acquisition Parameters
Date_              20180602
Time                  17.13 h
INSTRUM   KU_Avance_III_400
PROBHD    Z108618_0245 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH               24038.461 Hz
FIDRES             0.733596 Hz
AQ                 1.3631488 sec
RG                     2050
DW                   20.800 usec
DE                   22.74 usec
TE                   297.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1             100.5222401 MHz
NUC1                      13C
P1                    9.20 usec
PLW1              49.50000000 W
SFO2             399.7315989 MHz
NUC2                      1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2              16.47699928 W
PLW12              0.33068001 W
PLW13              0.26785001 W

F2 - Processing parameters
SI                    65536
SF               100.5121884 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

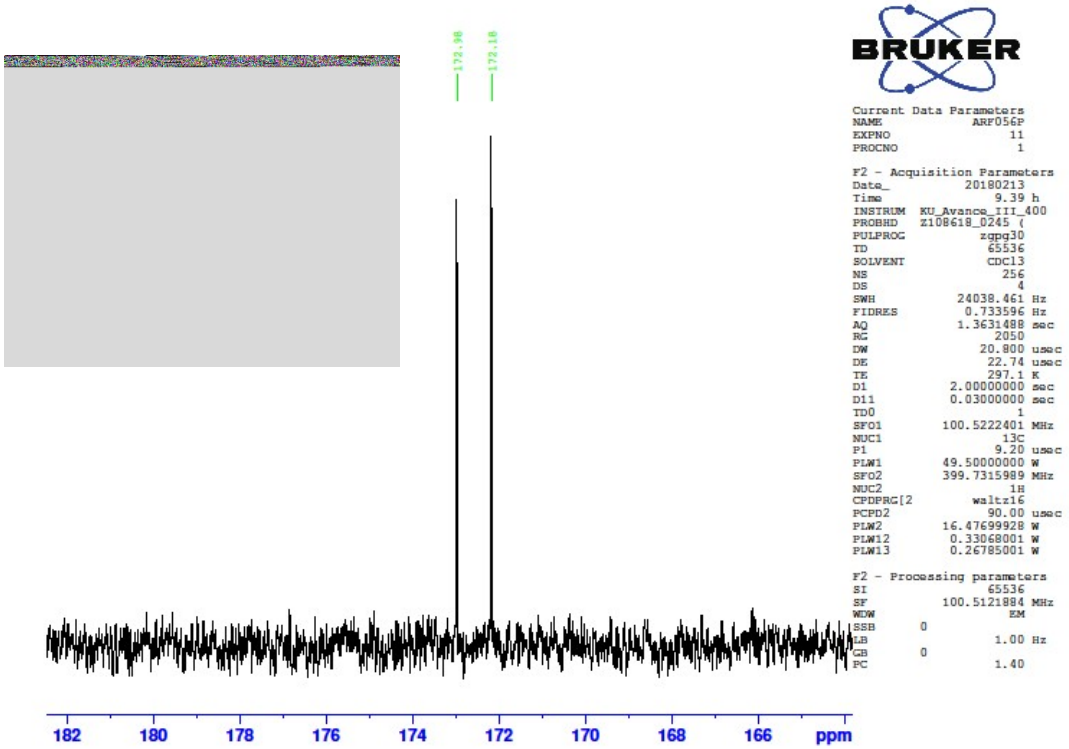


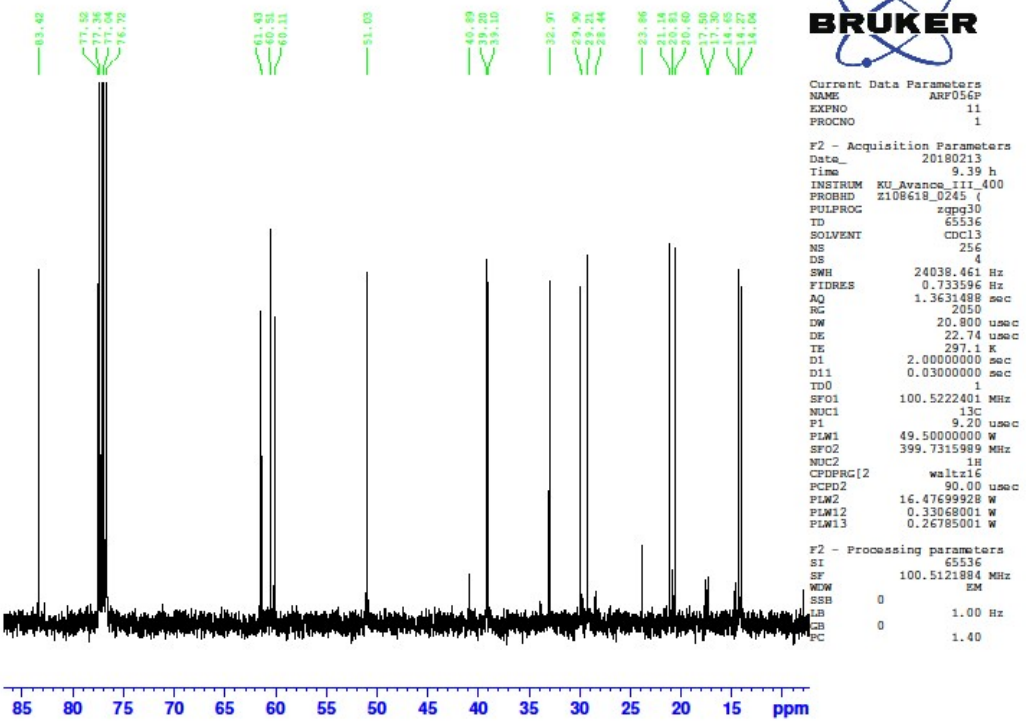

```
Current Data Parameters
NAME                 ARF157AP
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20190228
Time                    11.09 h
INSTRUM          KU_Avance_III_400
PROBHD           Z108618_0245 (
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                  8223.685 Hz
FIDRES               0.250967 Hz
AQ                   3.9845889 sec
RG                        18
DW                     60.800 usec
DE                     16.30 usec
TE                     294.7 K
D1                   1.00000000 sec
TD0                        1
SFO1               399.7324685 MHz
NUC1                      1H
P1                     12.75 usec
PLW1                16.47699928 W

F2 - Processing parameters
SI                     65536
SF                 399.7300000 MHz
WDW                       EM
SSB                0
LB                     0.20 Hz
GB                 0
PC                     1.00
```




```
Current Data Parameters
NAME                 ARF157AP
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_               20190228
Time                    19.38 h
INSTRUM          KU_Avance_III_400
PROBHD           Z108618_0245 (
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       256
DS                         4
SWH                 24038.461 Hz
FIDRES               0.733596 Hz
AQ                   1.3631488 sec
RG                      2050
DW                     20.800 usec
DE                     22.74 usec
TE                     296.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                        1
SFO1               100.5222401 MHz
NUC1                      13C
P1                      9.20 usec
PLW1                49.50000000 W
SFO2               399.7315989 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2                16.47699928 W
PLW12                0.33068001 W
PLW13                0.26785001 W

F2 - Processing parameters
SI                     65536
SF                 100.5121884 MHz
WDW                       EM
SSB                0
LB                     1.00 Hz
GB                 0
PC                     1.40
```


BRUKER
Current Data Parameters
NAME ARF056P
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180213
Time 9.24 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 144
DW 60.800 usec
DE 16.30 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300098 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
5.00
0.96
0.94
0.99
1.01
2.58
6.55


172.98
172.18
BRUKER
Current Data Parameters
NAME ARF056P
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180213
Time 9.39 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
182
180
178
176
174
172
170
168
166
ppm

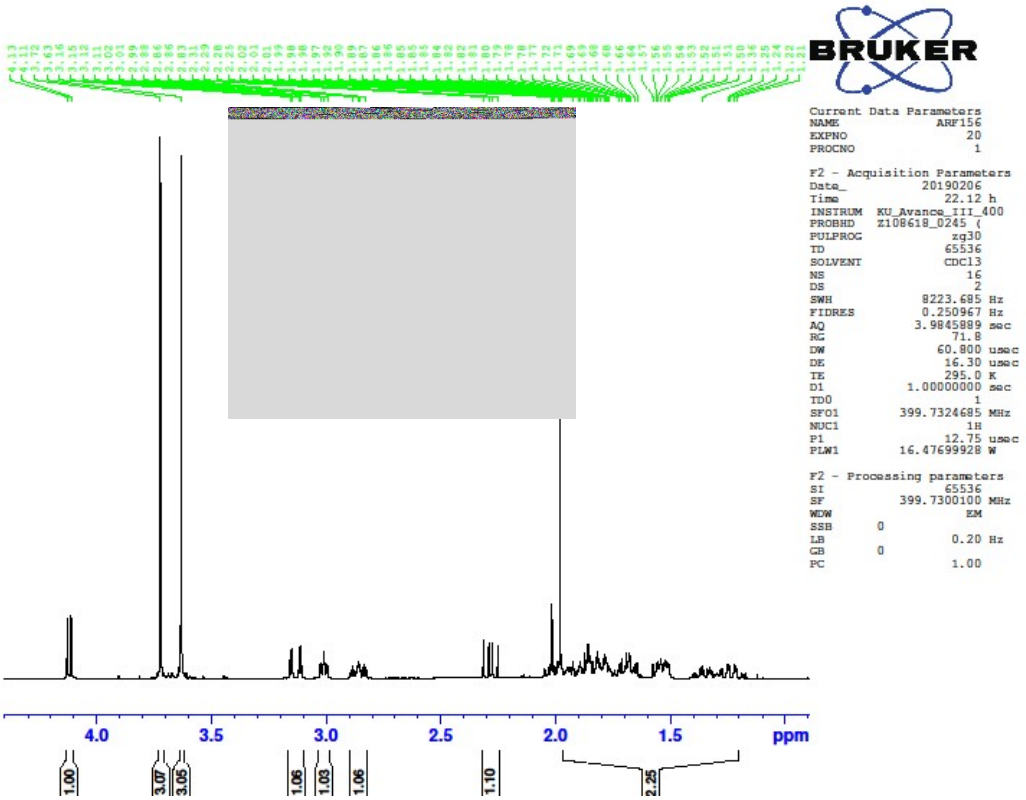


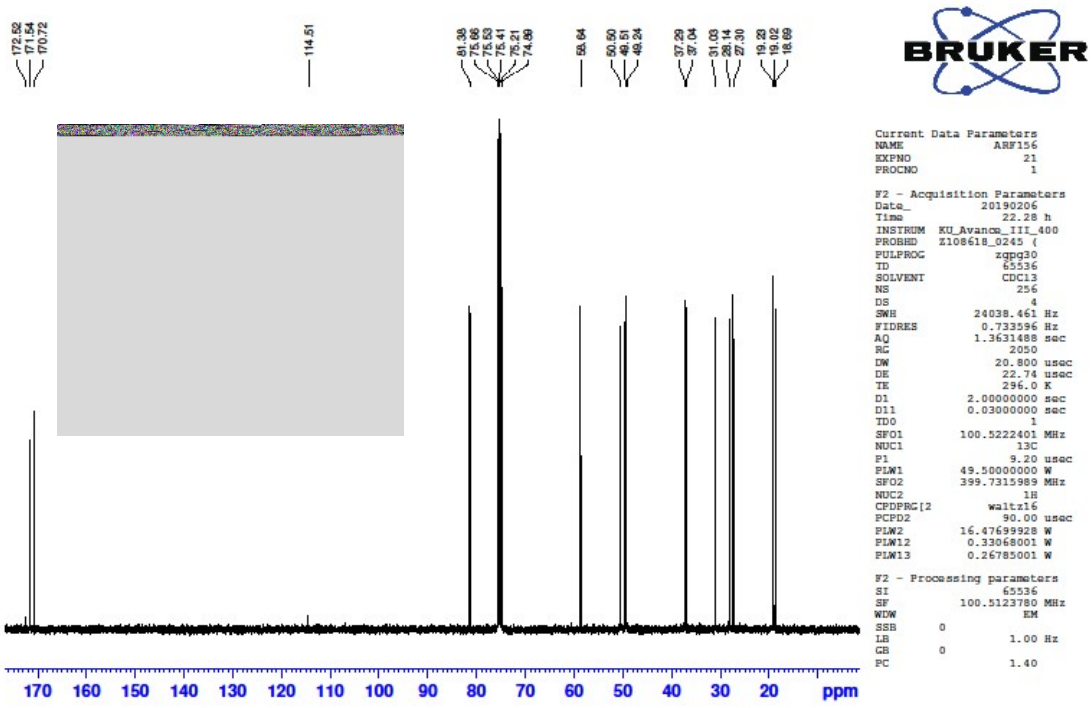

BRUKER

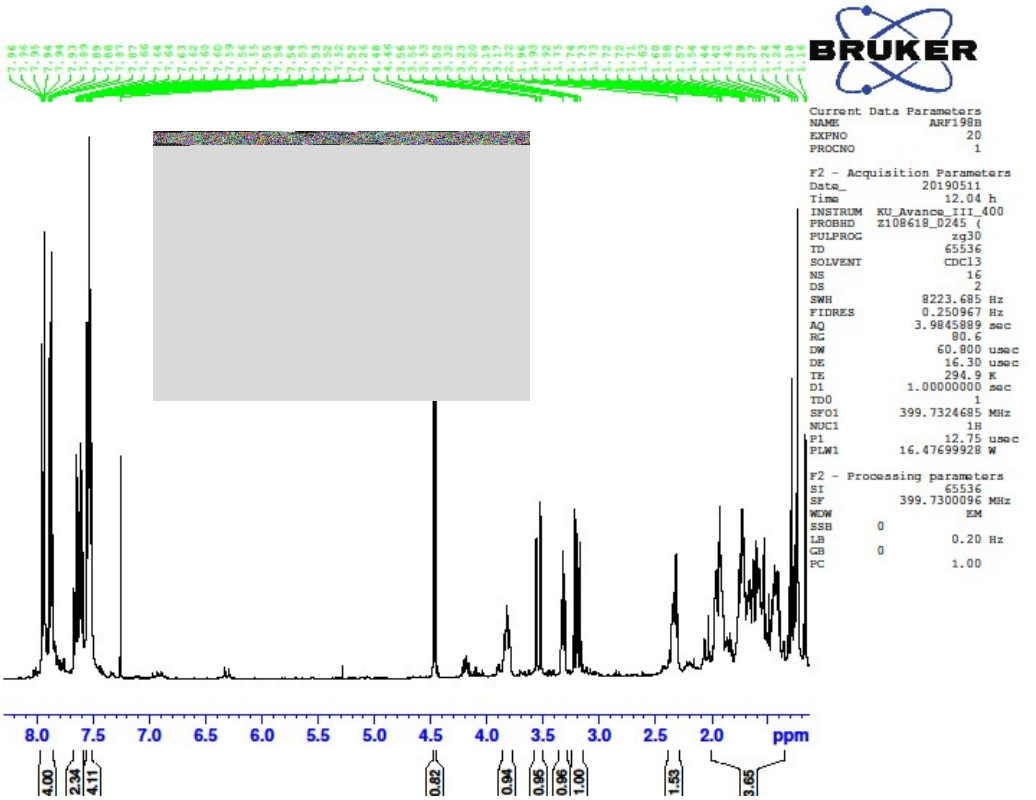
Current Data Parameters
NAME ARF056P
EXPNO 11
PROCNO 1

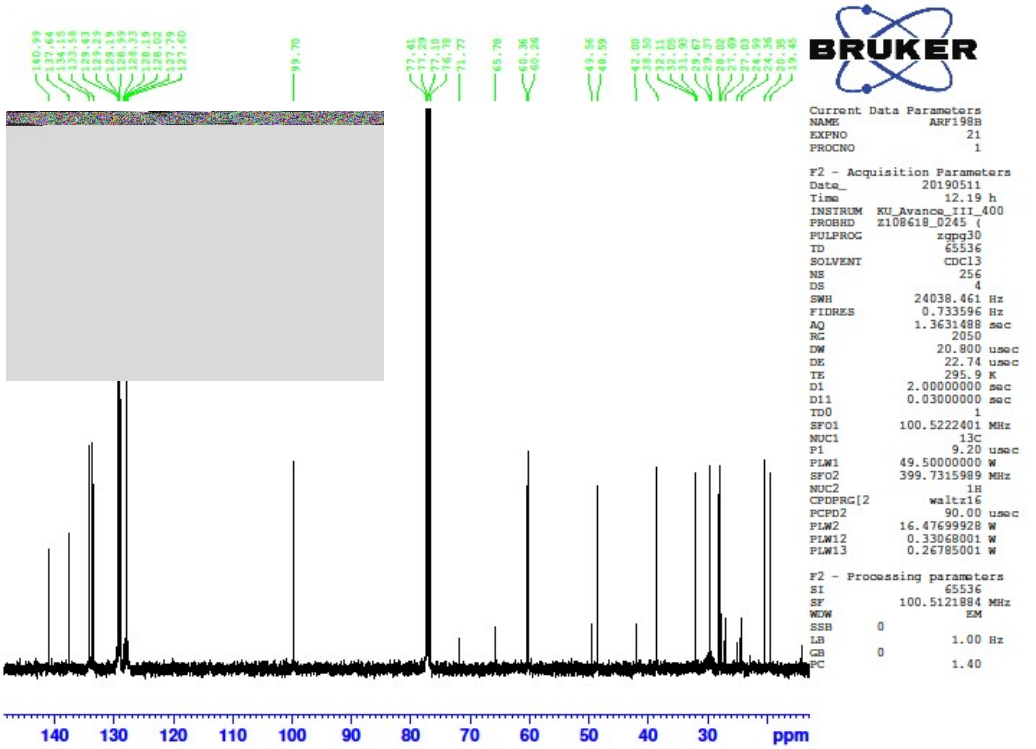
F2 - Acquisition Parameters
Date_ 20180213
Time 9.39 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W

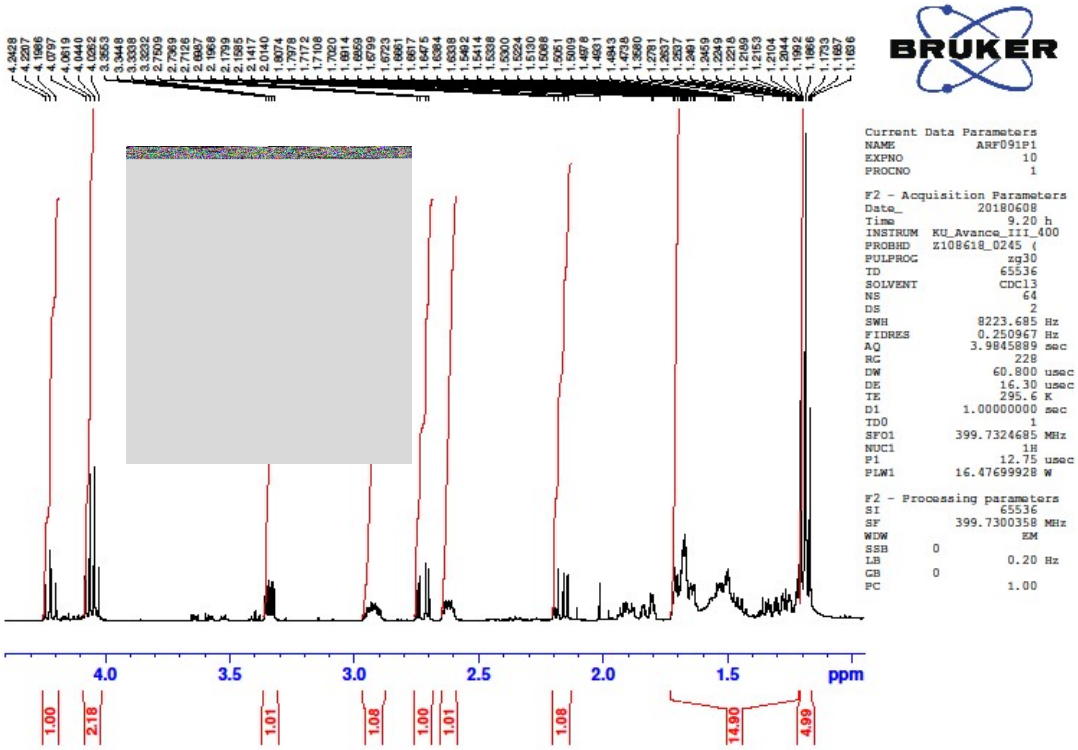
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

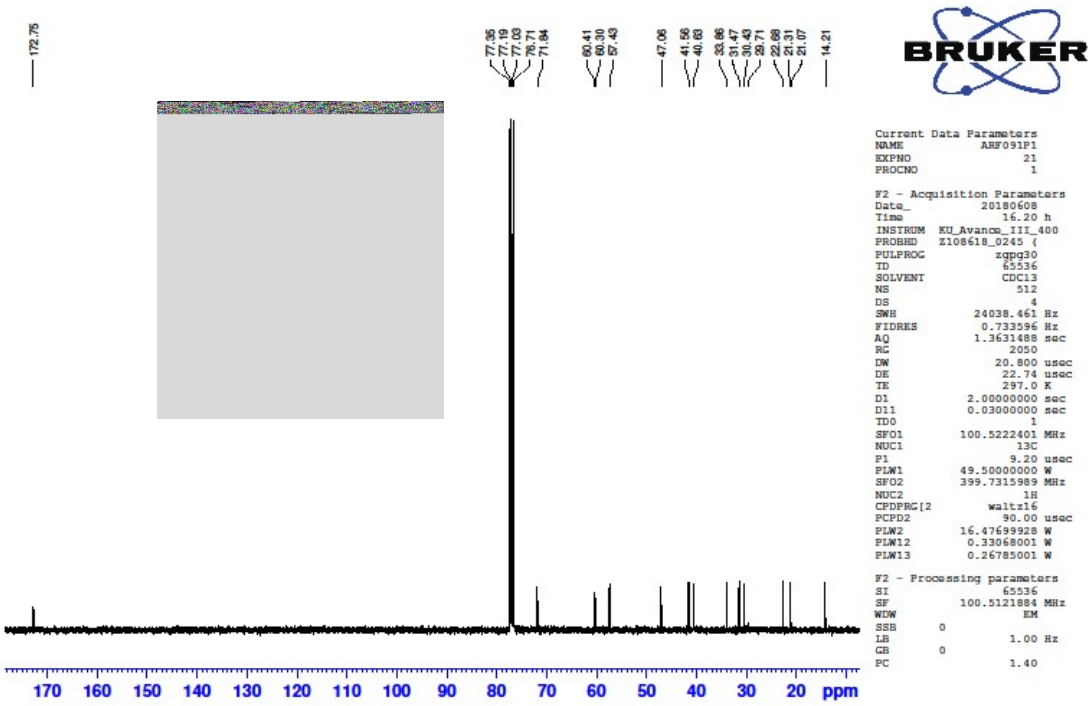

BRUKER
Current Data Parameters
NAME ARF156
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190206
Time 22.12 h
INSTRUM KU_Avanca_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 71.8
DW 60.800 usec
DE 16.30 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300100 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.00
3.07
3.05
1.06
1.03
1.06
1.10
2.26

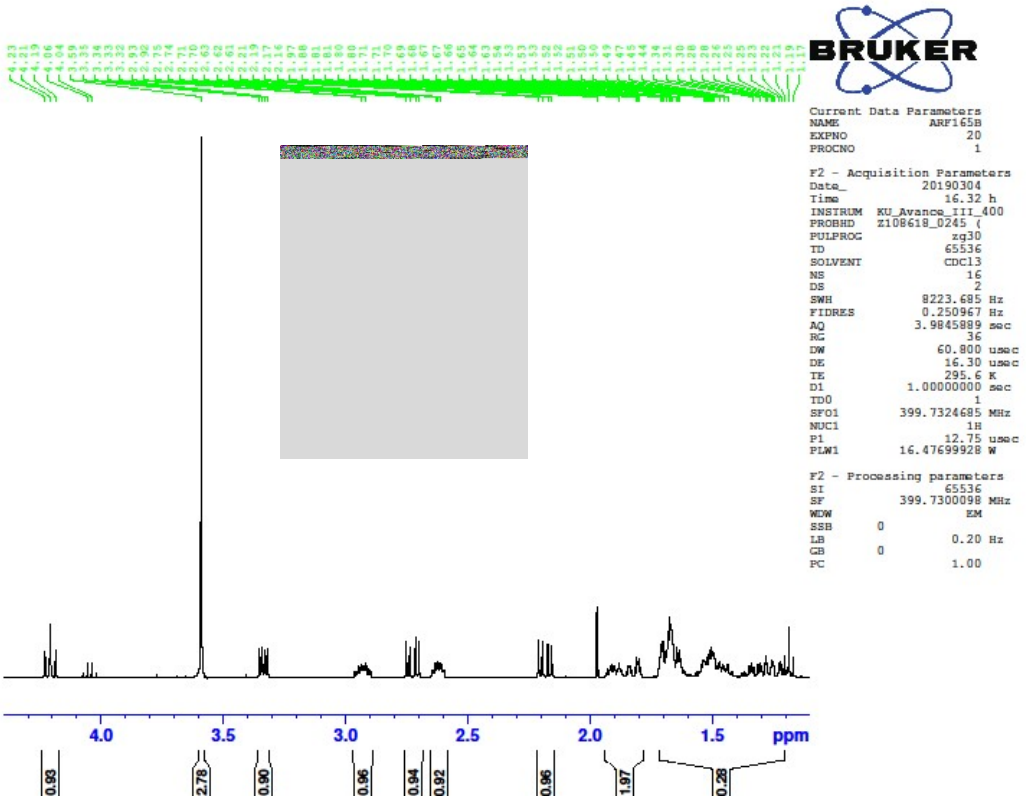

172.52
171.54
170.72
114.51
81.38
75.66
75.53
75.41
75.21
74.69
59.64
50.50
49.51
49.24
37.29
37.04
31.03
28.14
27.30
19.23
19.02
18.69
BRUKER
Current Data Parameters
NAME ARF156
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190206
Time 22.28 h
INSTRUM KU_Avanca_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5123780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

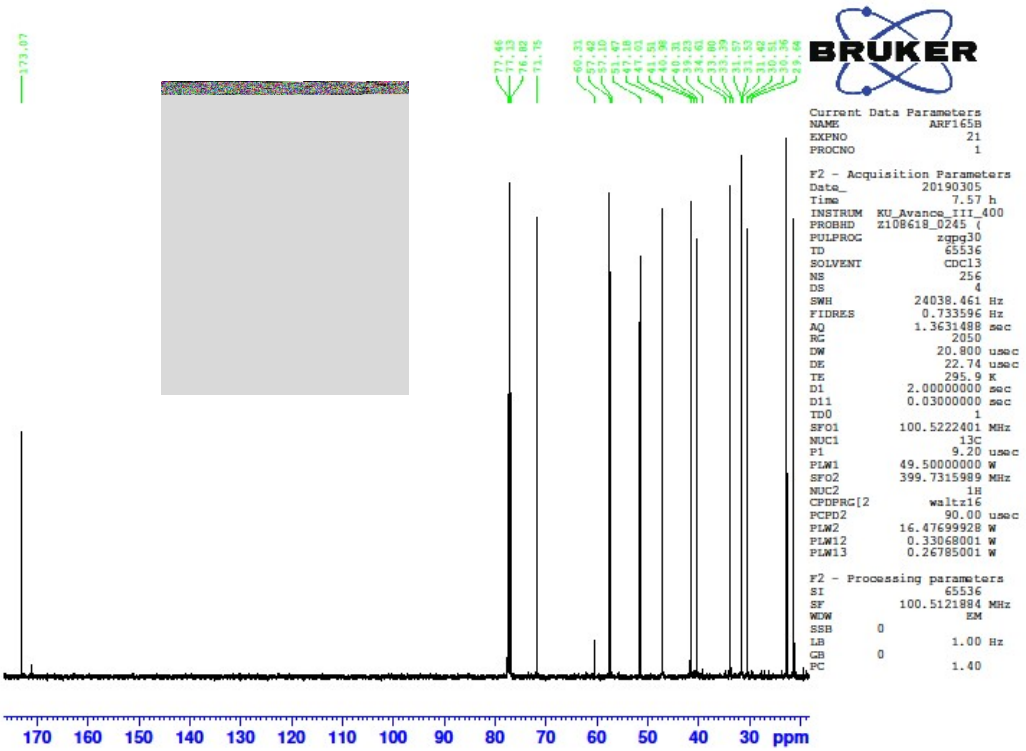

BRUKER
Current Data Parameters
NAME ARF198B
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190511
Time 12.04 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 80.6
DW 60.800 usec
DE 16.30 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300096 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
4.00 2.34 4.11 0.82 0.94 0.98 0.96 1.00 1.53 3.65

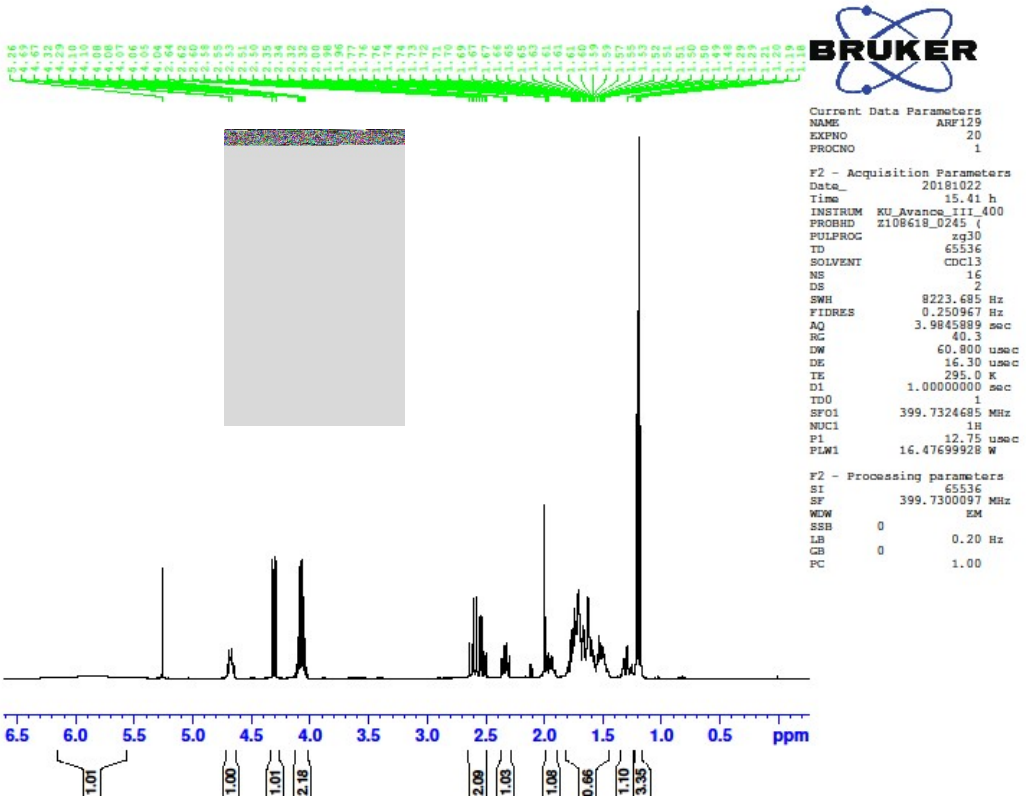

BRUKER
Current Data Parameters
NAME ARF198B
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190511
Time 12.19 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140 130 120 110 100 90 80 70 60 50 40 30 ppm

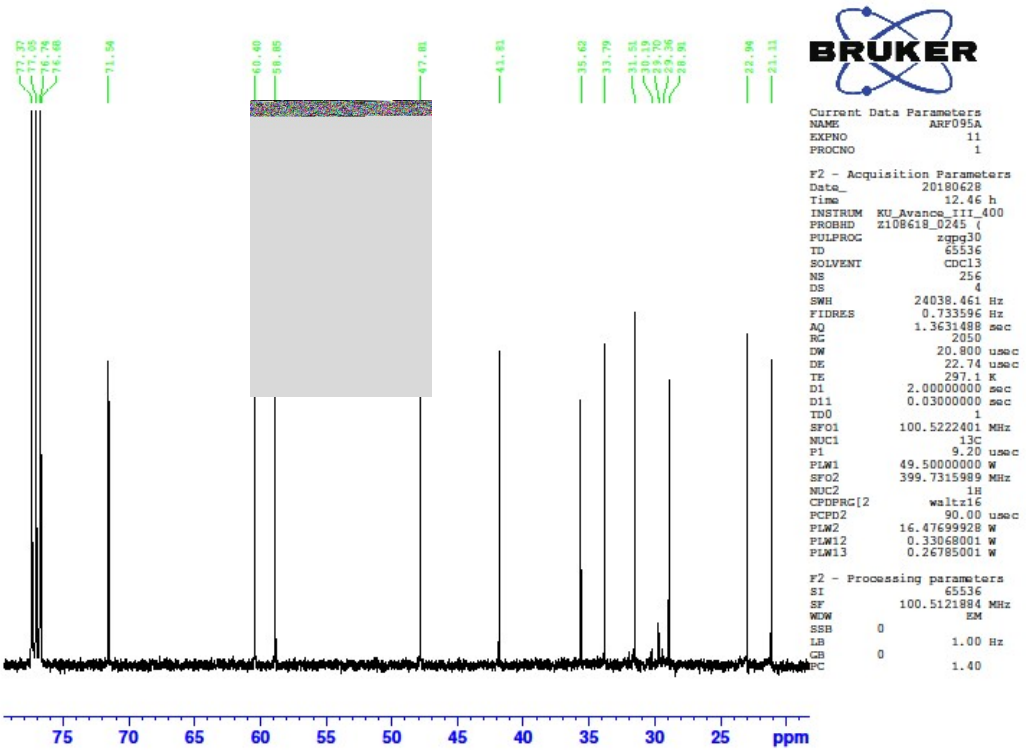

4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.00
2.18
1.01
1.08
1.00
1.01
1.08
14.90
4.99
Current Data Parameters
NAME ARF091P1
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180608
Time 9.20 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 228
DW 60.800 usec
DE 16.30 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300358 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00


172.75
77.35
77.19
77.03
76.71
71.84
60.41
60.30
57.43
47.06
41.56
40.63
33.86
31.47
30.43
29.71
22.68
21.31
21.07
14.21
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm
Current Data Parameters
NAME ARF091P1
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180608
Time 16.20 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

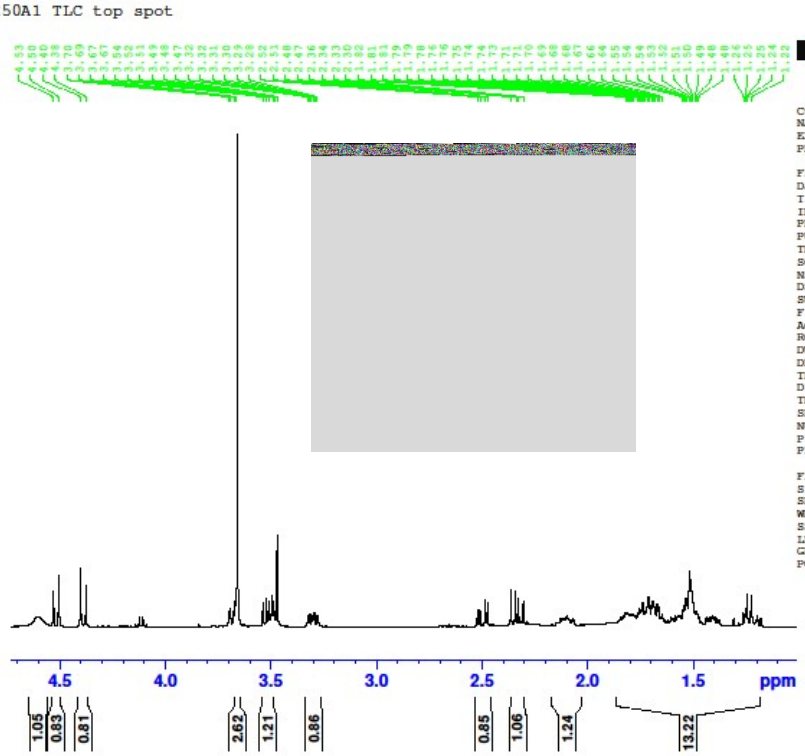

BRUKER
Current Data Parameters
NAME ARF165B
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190304
Time 16.32 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 36
DW 60.800 usec
DE 16.30 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300098 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
4.0
3.5
3.0
2.5
2.0
1.5
ppm
0.93
2.78
0.90
0.96
0.94
0.92
0.96
1.97
0.28


BRUKER
Current Data Parameters
NAME ARF165B
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190305
Time 7.57 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


BRUKER
Current Data Parameters
NAME ARF129
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181022
Time 15.41 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 40.3
DW 60.800 usec
DE 16.30 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300097 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.01 1.00 1.01 2.18 2.09 1.03 1.08 0.66 1.10 3.35

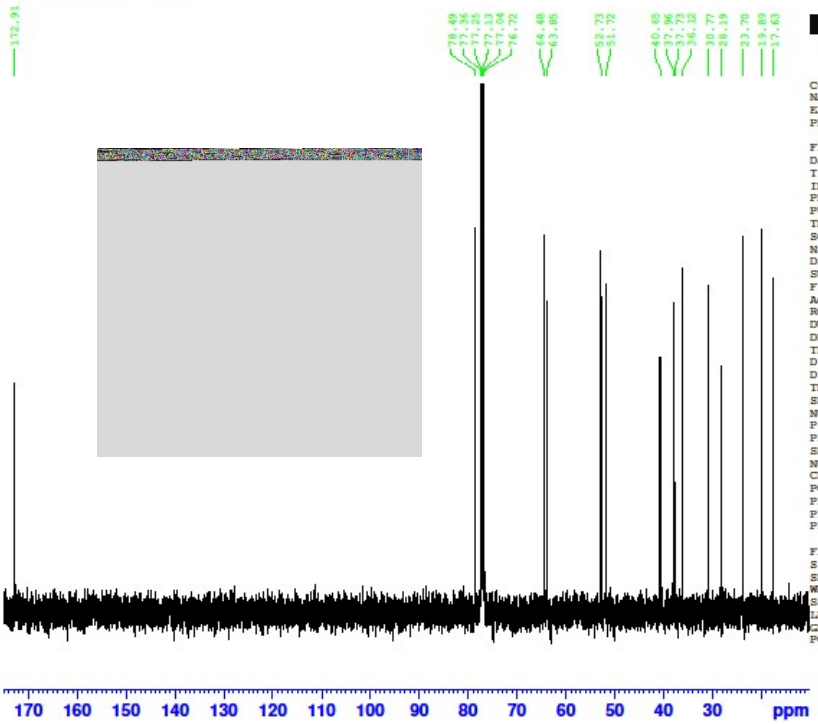

BRUKER
77.37
77.05
76.74
76.48
71.54
60.40
58.85
47.81
41.81
35.62
33.79
31.51
30.13
29.70
29.36
28.91
22.94
21.11
Current Data Parameters
NAME ARF095A
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 12.46 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
75 70 65 60 55 50 45 40 35 30 25 ppm

250A1 TLC top spot

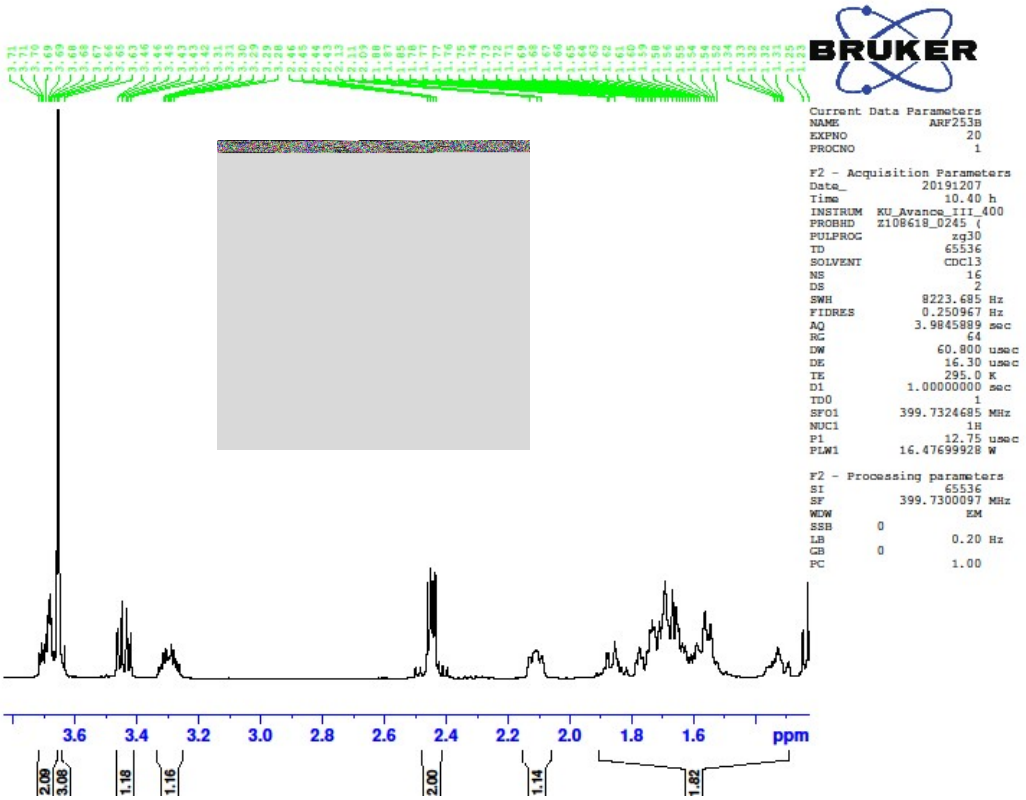


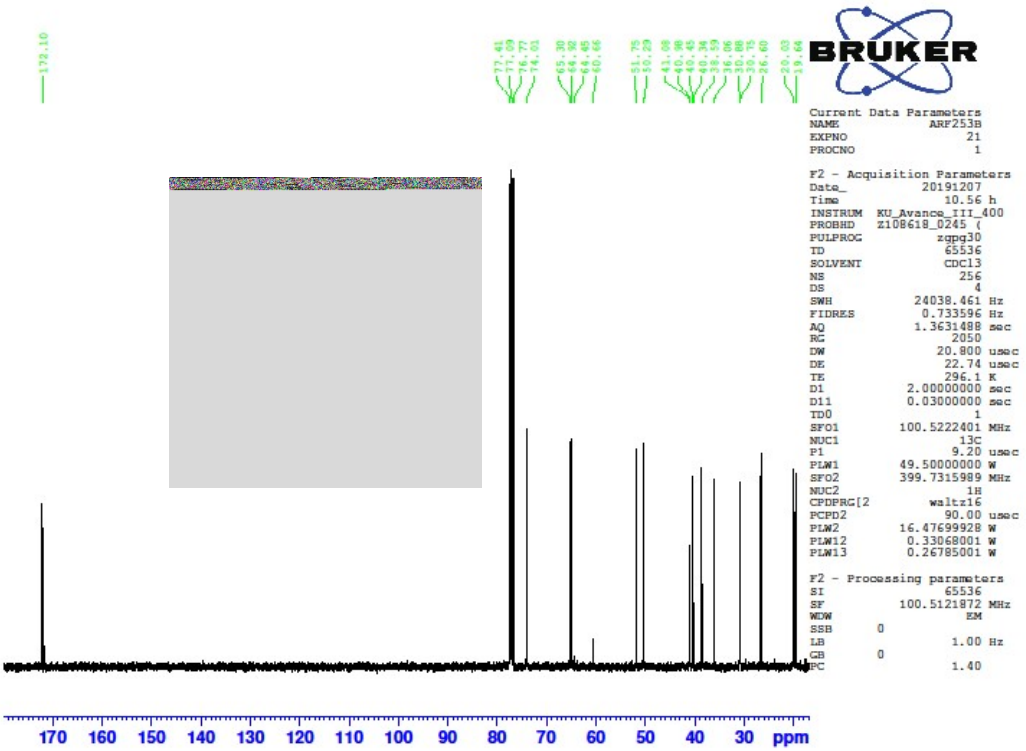

```
Current Data Parameters
NAME                 ARF250A1
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20191207
Time                   14.50 h
INSTRUM  KU_Avance_III_400
PROBHD   Z108618_0245 (
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         16
DS                          2
SWH                 8223.685 Hz
FIDRES              0.250967 Hz
AQ                  3.9845889 sec
RG                        128
DW                     60.800 usec
DE                     16.30 usec
TE                     295.2 K
D1                  1.00000000 sec
TD0                         1
SFO1               399.7324685 MHz
NUC1                       1H
P1                     12.75 usec
PLW1               16.47699928 W

F2 - Processing parameters
SI                         65536
SF                 399.7300098 MHz
WDW                        EM
SSB                 0
LB                          0.20 Hz
GB                  0
PC                          1.00
```

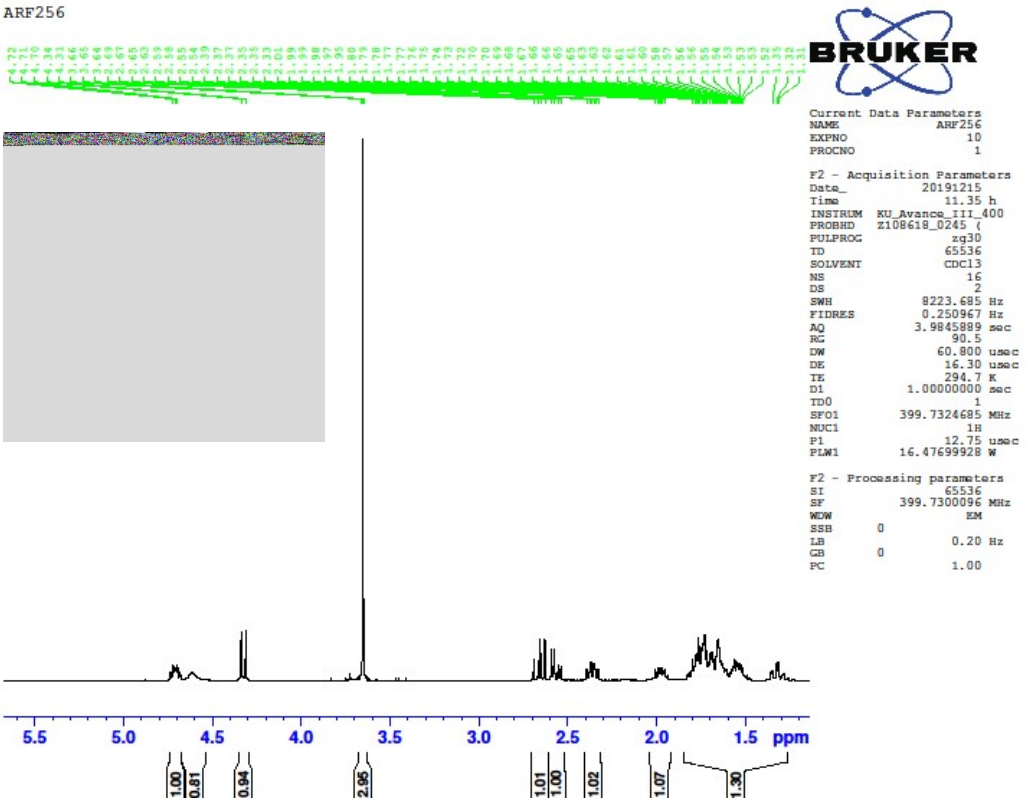
250A1 TLC top spot

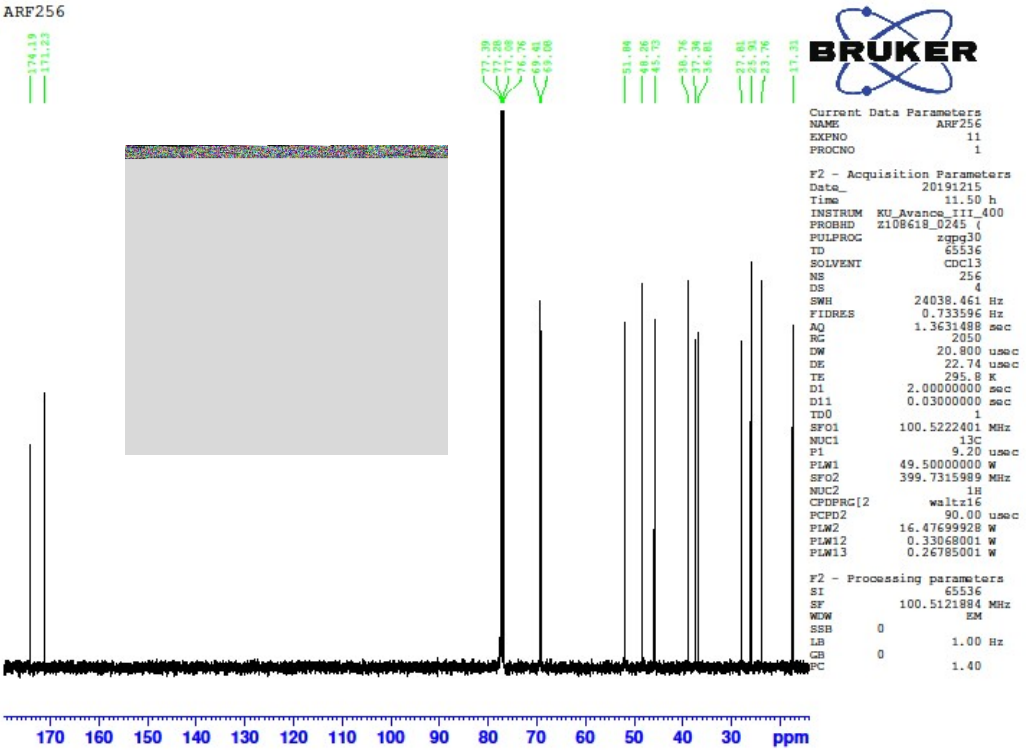


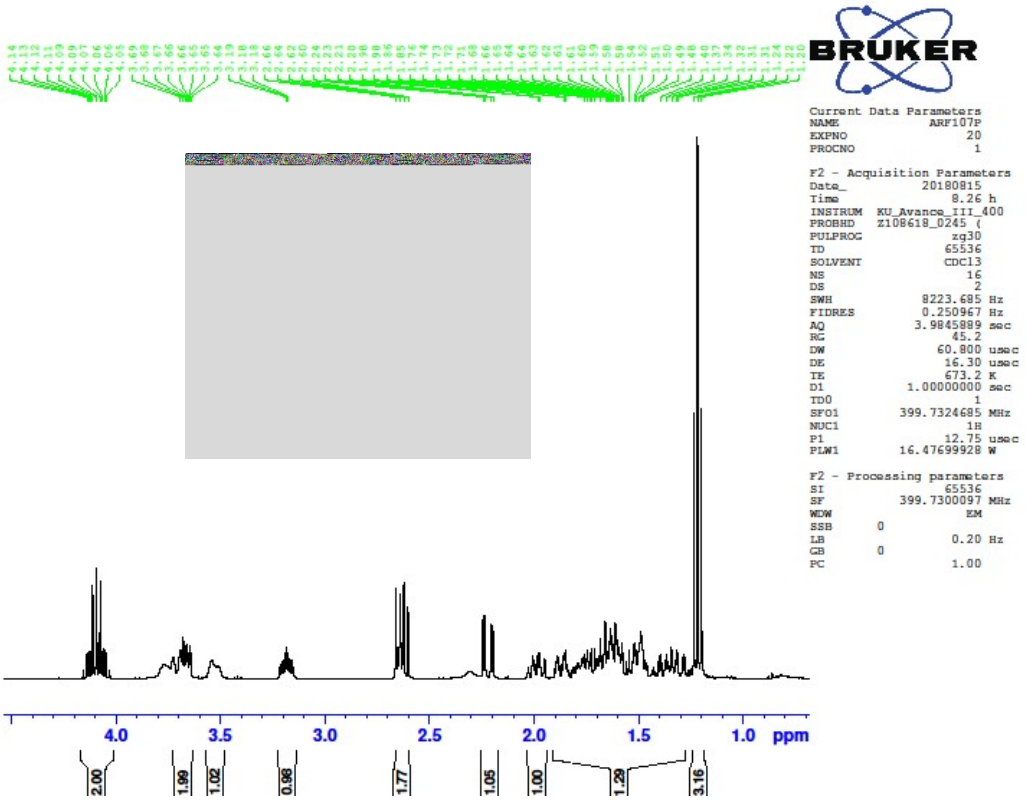

```
Current Data Parameters
NAME                 ARF250A1
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_               20191207
Time                   15.05 h
INSTRUM  KU_Avance_III_400
PROBHD   Z108618_0245 (
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        256
DS                          4
SWH                24038.461 Hz
FIDRES              0.733596 Hz
AQ                  1.3631488 sec
RG                       2050
DW                     20.800 usec
DE                     22.74 usec
TE                     296.1 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1
SFO1               100.5222401 MHz
NUC1                       13C
P1                      9.20 usec
PLW1                49.50000000 W
SFO2               399.7315989 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                  90.00 usec
PLW2               16.47699928 W
PLW12               0.33068001 W
PLW13               0.26785001 W

F2 - Processing parameters
SI                         65536
SF                 100.5121884 MHz
WDW                        EM
SSB                 0
LB                          1.00 Hz
GB                  0
PC                          1.40
```

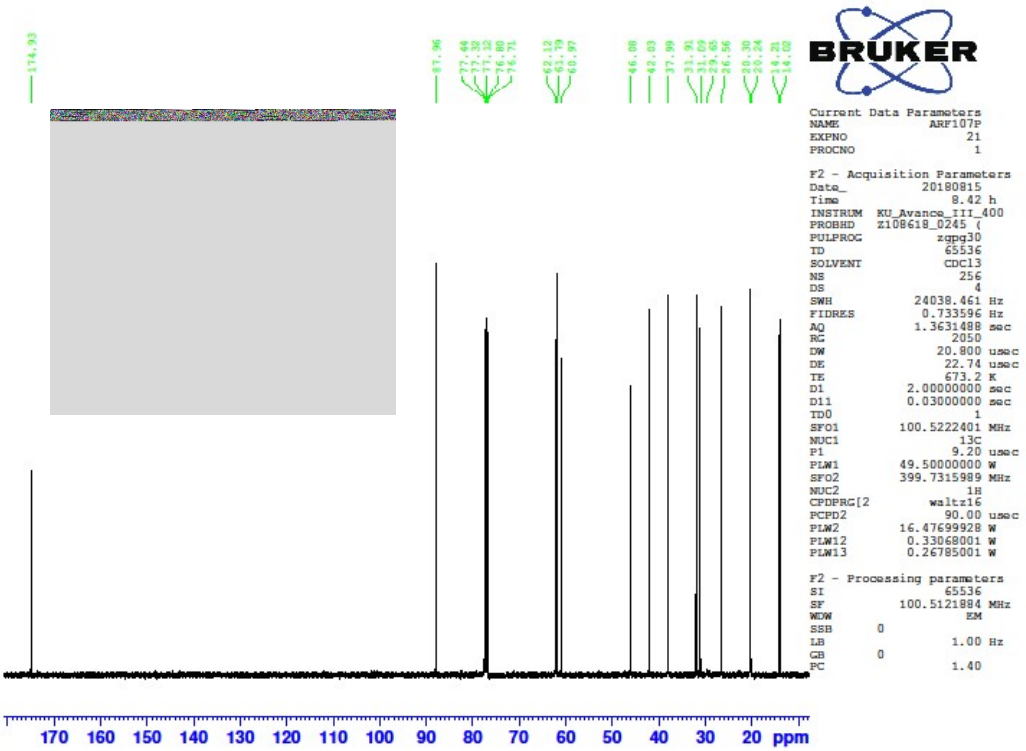

BRUKER
Current Data Parameters
NAME ARF253B
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191207
Time 10.40 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 64
DW 60.800 usec
DE 16.30 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300097 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 ppm
2.09 3.08 1.18 1.16 2.00 1.14 1.82

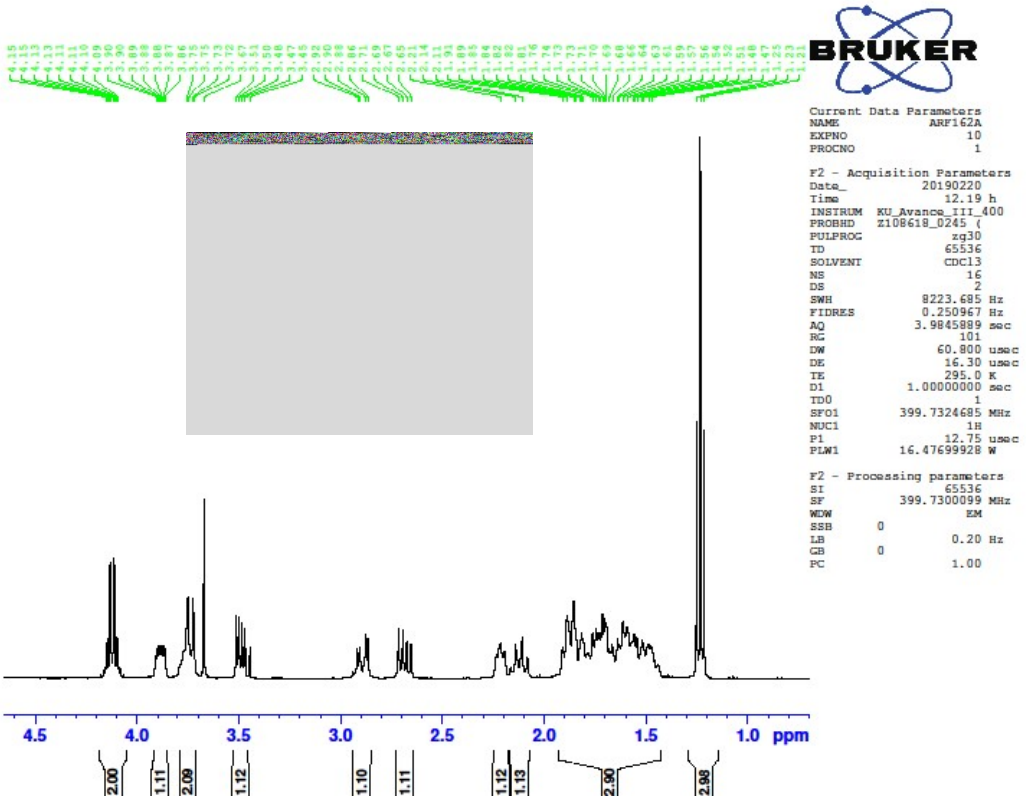

BRUKER
Current Data Parameters
NAME ARF253B
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191207
Time 10.56 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121872 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

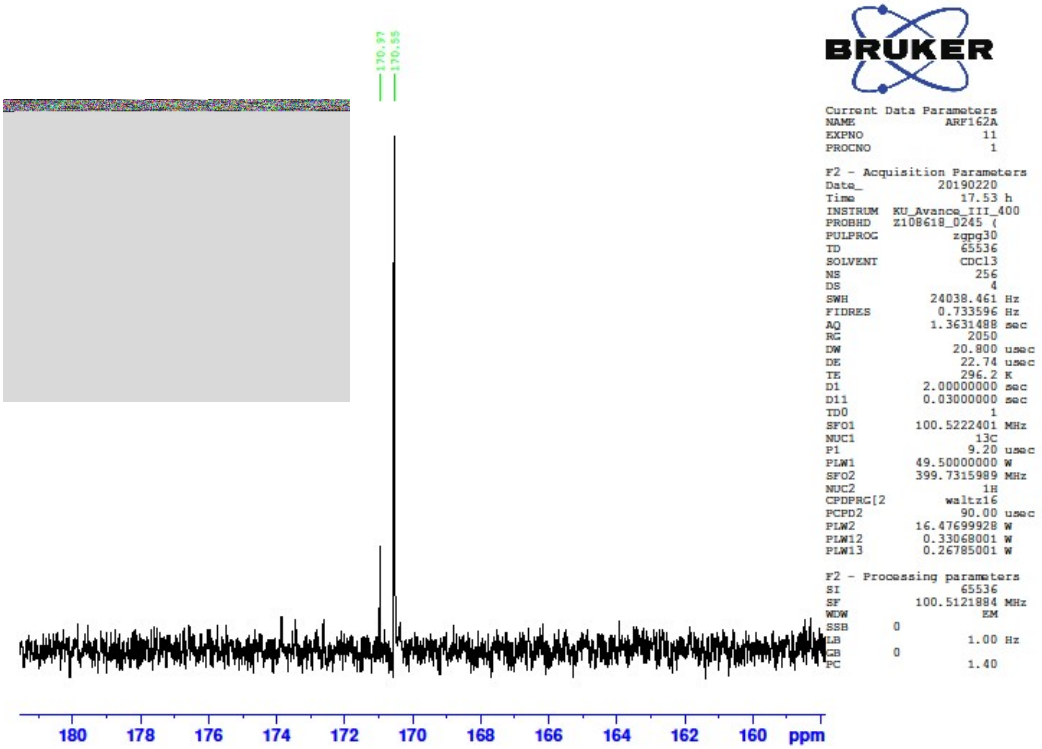

ARF256
BRUKER
Current Data Parameters
NAME ARF256
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191215
Time 11.35 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 90.5
DW 60.800 usec
DE 16.30 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300096 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
1.00 0.81 0.94 2.95 1.01 1.00 1.02 1.07 1.30

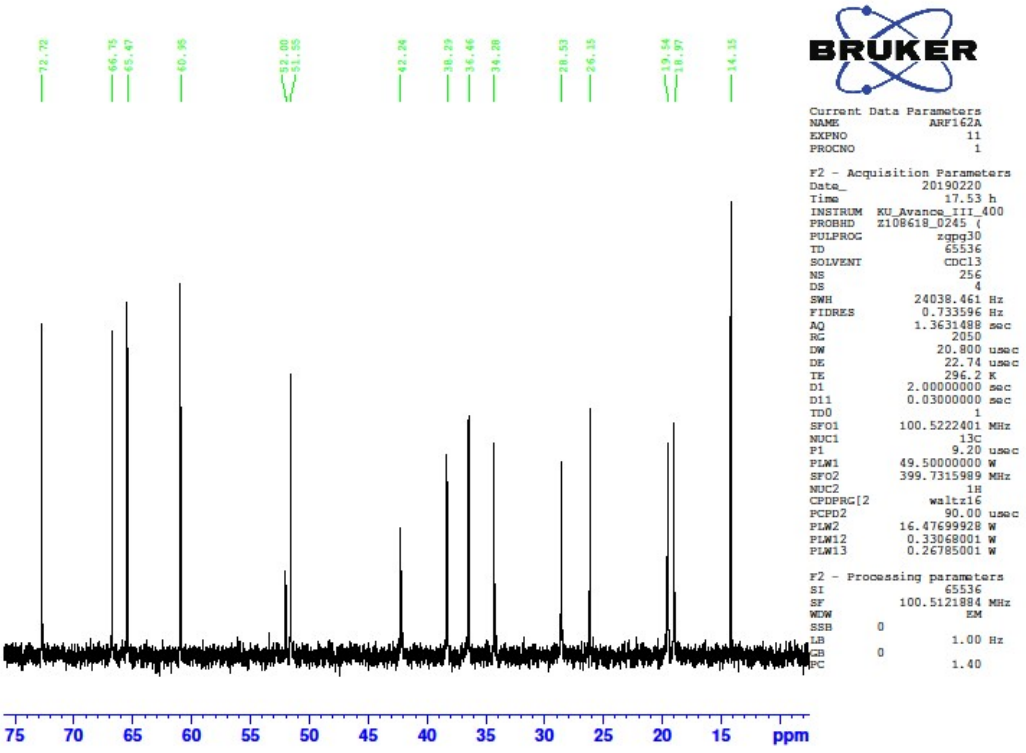

ARF256
BRUKER
Current Data Parameters
NAME ARF256
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191215
Time 11.50 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

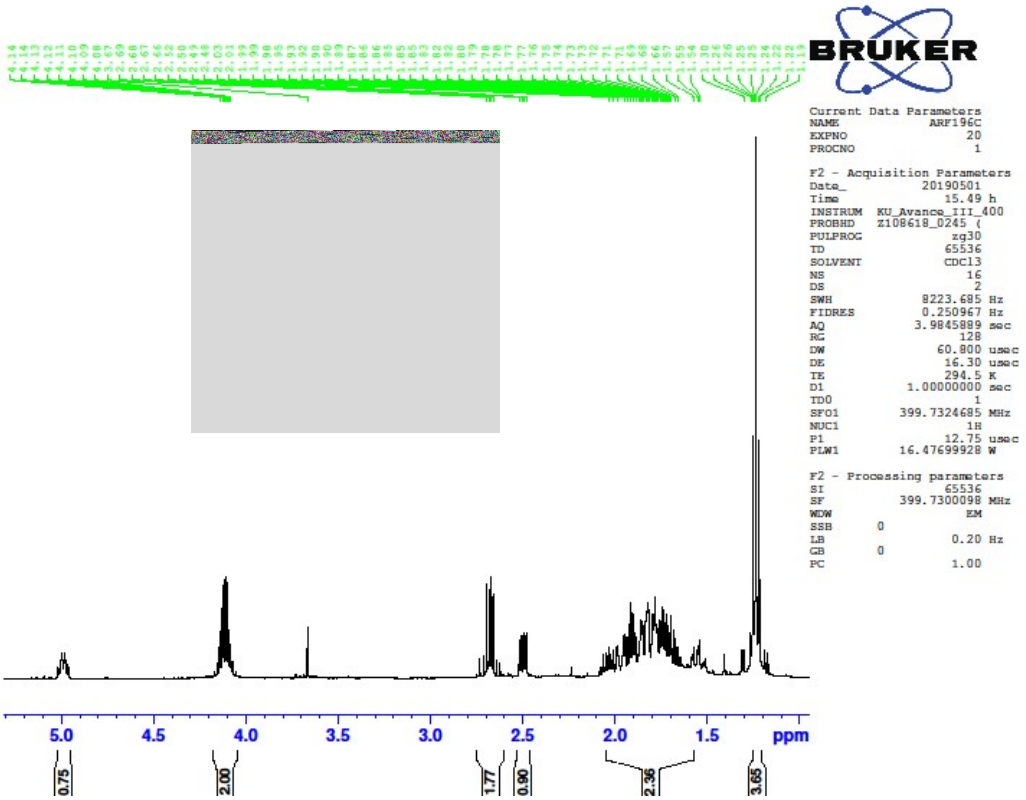

BRUKER
Current Data Parameters
NAME ARF107P
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180815
Time 8.26 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 45.2
DW 60.800 usec
DE 16.30 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300097 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.00
1.99
1.02
0.98
1.77
1.05
1.00
1.29
3.16

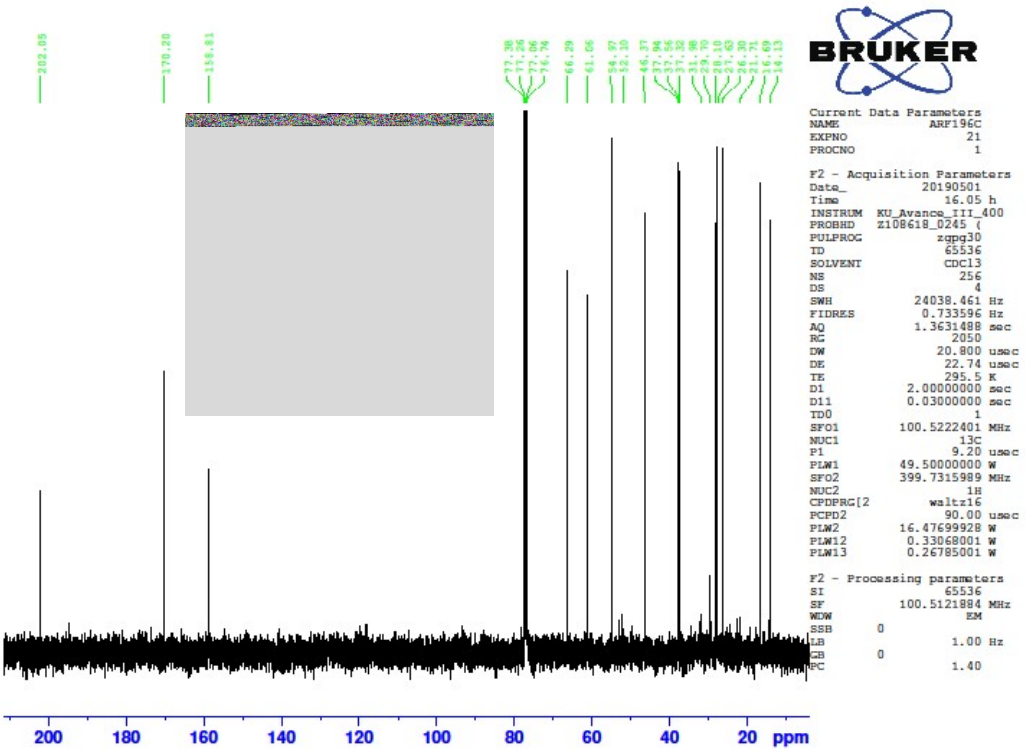

BRUKER
Current Data Parameters
NAME ARF107P
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180815
Time 8.42 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
174.93
97.96
77.44
77.32
77.12
76.80
76.71
62.12
61.79
60.97
46.08
42.03
37.99
33.91
31.09
29.65
26.56
20.30
20.24
14.21
14.02
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

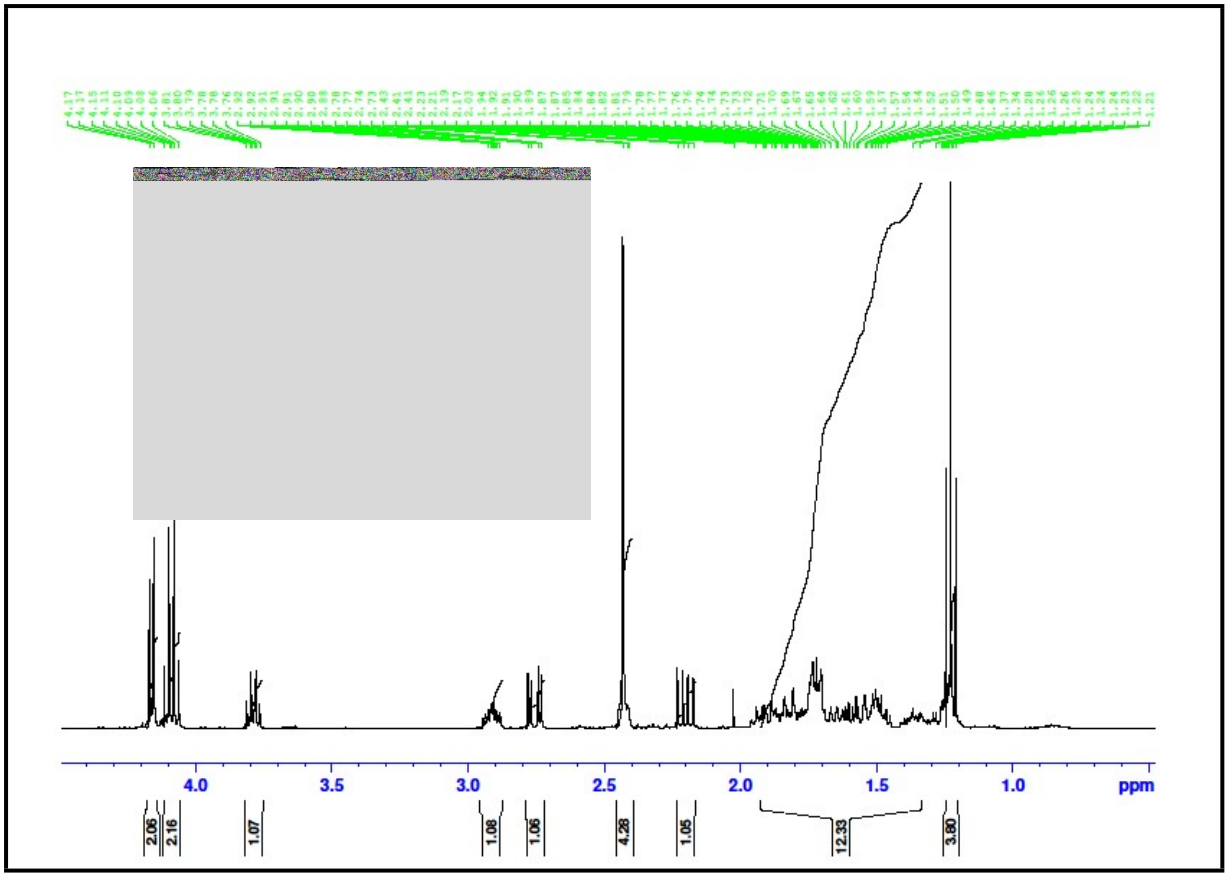

BRUKER
Current Data Parameters
NAME ARF162A
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190220
Time 12.19 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 101
DW 60.800 usec
DE 16.30 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300099 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0 ppm
2.00
1.11
2.09
1.12
1.10
1.11
1.12
1.13
2.90
2.98

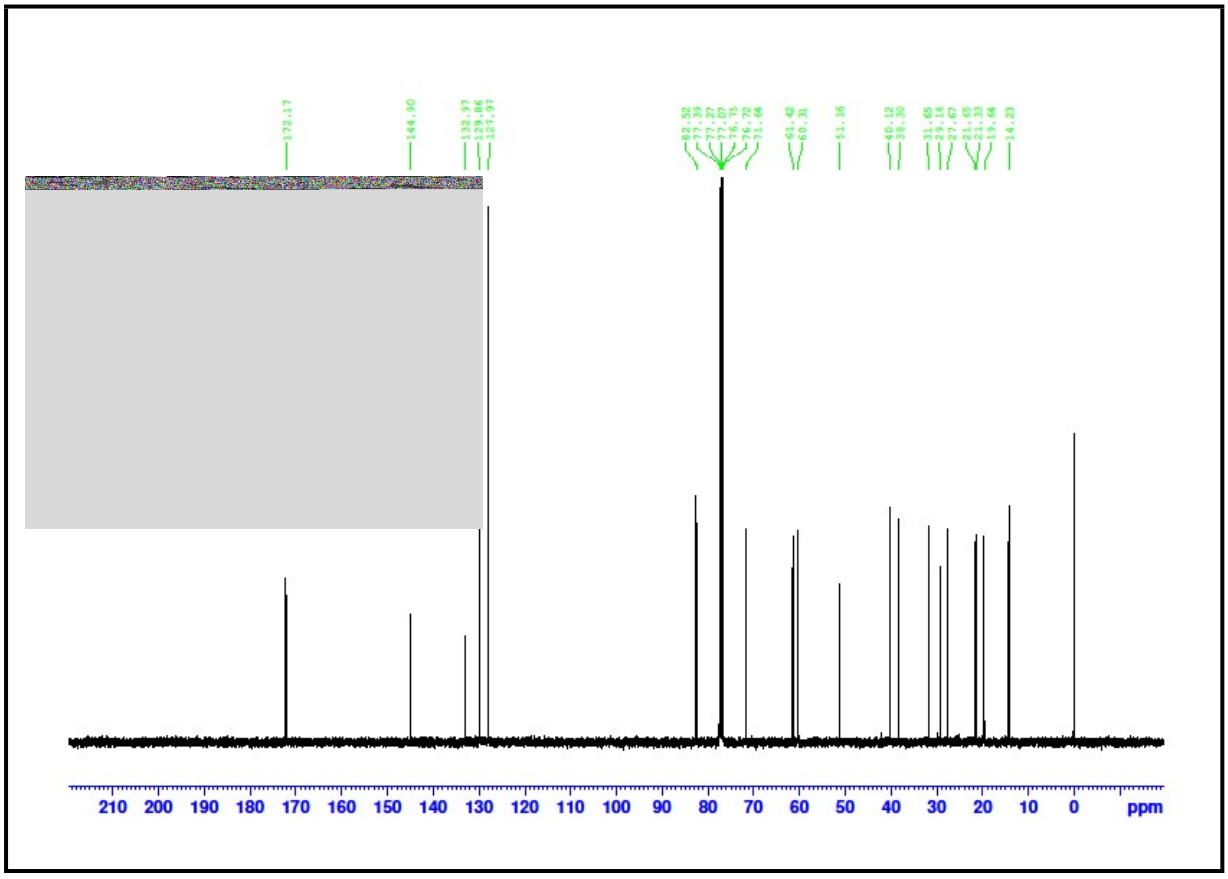

170.97
170.55
BRUKER
Current Data Parameters
NAME ARF162A
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190220
Time 17.53 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
178
176
174
172
170
168
166
164
162
160 ppm

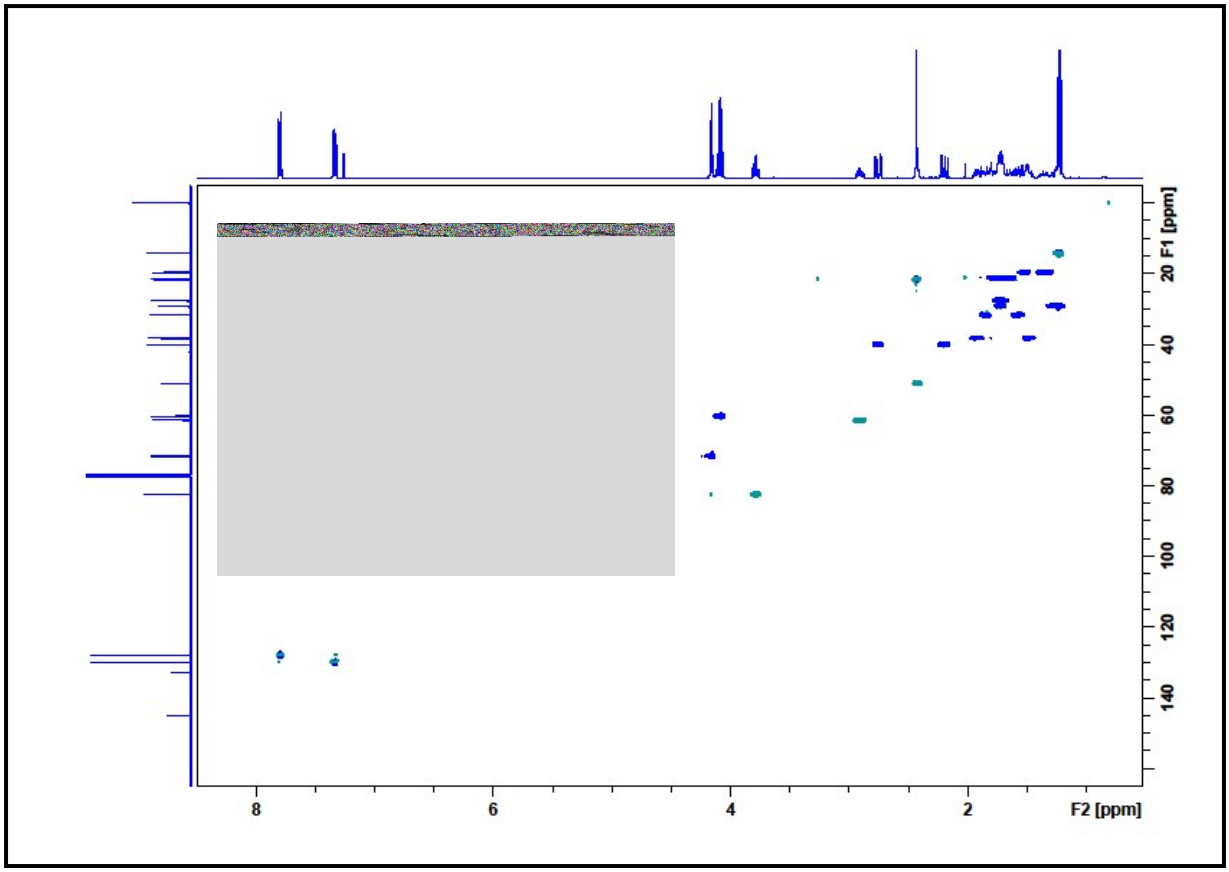

72.70
66.75
65.47
60.95
52.08
51.85
42.24
38.29
36.46
34.28
28.53
26.35
19.54
18.97
14.35
75
70
65
60
55
50
45
40
35
30
25
20
15
ppm
Current Data Parameters
NAME ARF162A
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190220
Time 17.53 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

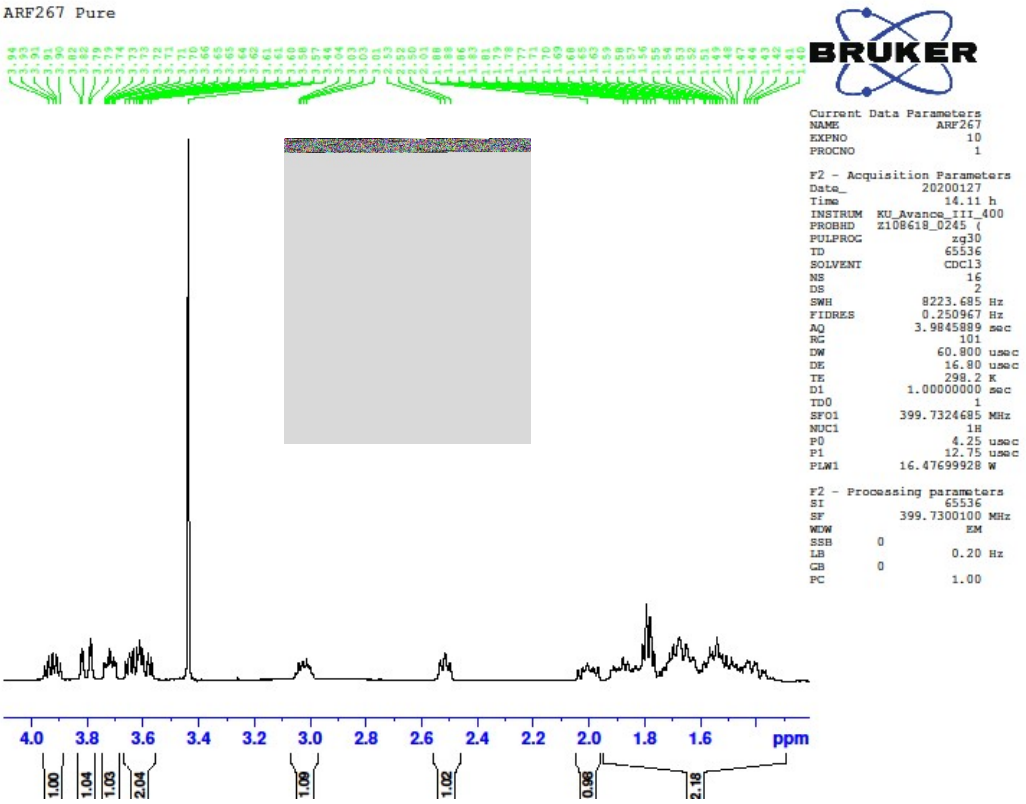

BRUKER
Current Data Parameters
NAME ARF196C
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190501
Time 15.49 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 128
DW 60.800 usec
DE 16.30 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324685 MHz
NUC1 1H
P1 12.75 usec
PLW1 16.47699928 W
F2 - Processing parameters
SI 65536
SF 399.7300098 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
0.75
2.00
1.77
0.90
2.36
3.65


202.05
170.20
158.81
77.38
77.06
76.74
66.29
61.06
54.97
52.30
46.37
37.94
37.56
37.32
31.98
29.70
28.10
27.40
26.30
21.71
16.69
14.13
BRUKER
Current Data Parameters
NAME ARF196C
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190501
Time 16.05 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 2050
DW 20.800 usec
DE 22.74 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.5222401 MHz
NUC1 13C
P1 9.20 usec
PLW1 49.50000000 W
SFO2 399.7315989 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
PLW12 0.33068001 W
PLW13 0.26785001 W
F2 - Processing parameters
SI 65536
SF 100.5121884 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm


4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.06
2.16
1.07
1.08
1.06
4.28
1.05
12.33
3.80


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


20
40
60
80
100
120
140
F1 [ppm]
8
6
4
2
F2 [ppm]


ARF267 Pure
BRUKER
Current Data Parameters
NAME ARF267
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200127
Time 14.11 h
INSTRUM KU_Avance_III_400
PROBHD Z108618_0245 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 3.9845889 sec
RG 101
DW 60.800 usec
DE 16.80 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 399.7324695 MHz
NUC1 1H
P0 4.25 usec
P1 12.75 usec
PLW1 16.47699928 W
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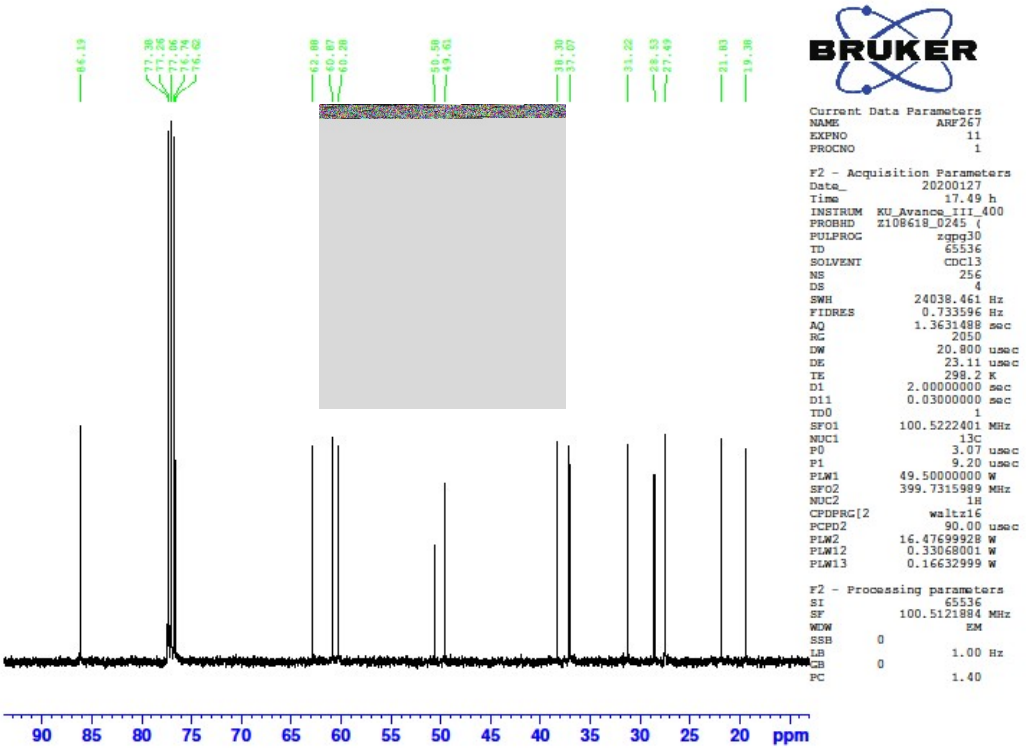
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 ppm
1.00 1.04 1.03 2.04 1.09 1.02 0.98 2.18




BRUKER

Current Data Parameters
NAME ARF267
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
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Time 17.49 h
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DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
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TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
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CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.47699928 W
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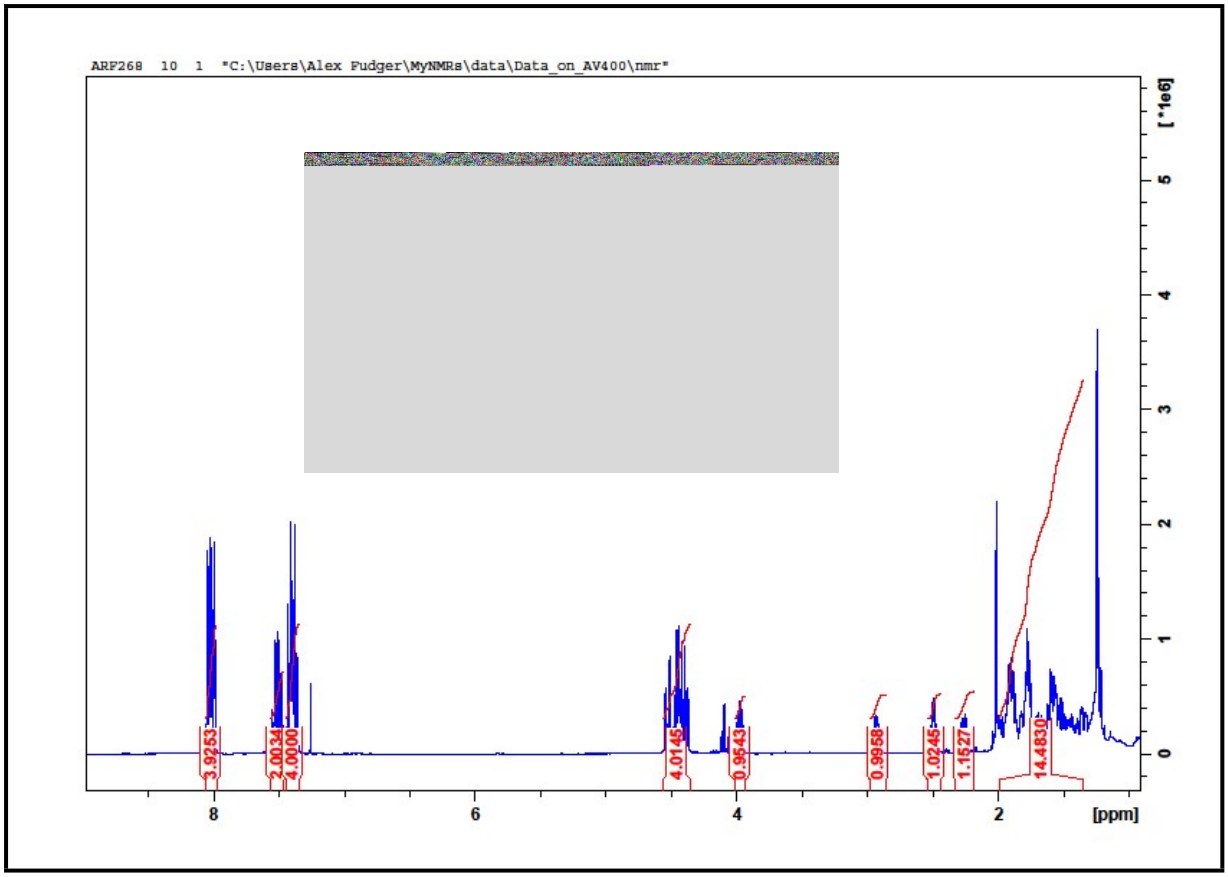
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0.3958
1.0245
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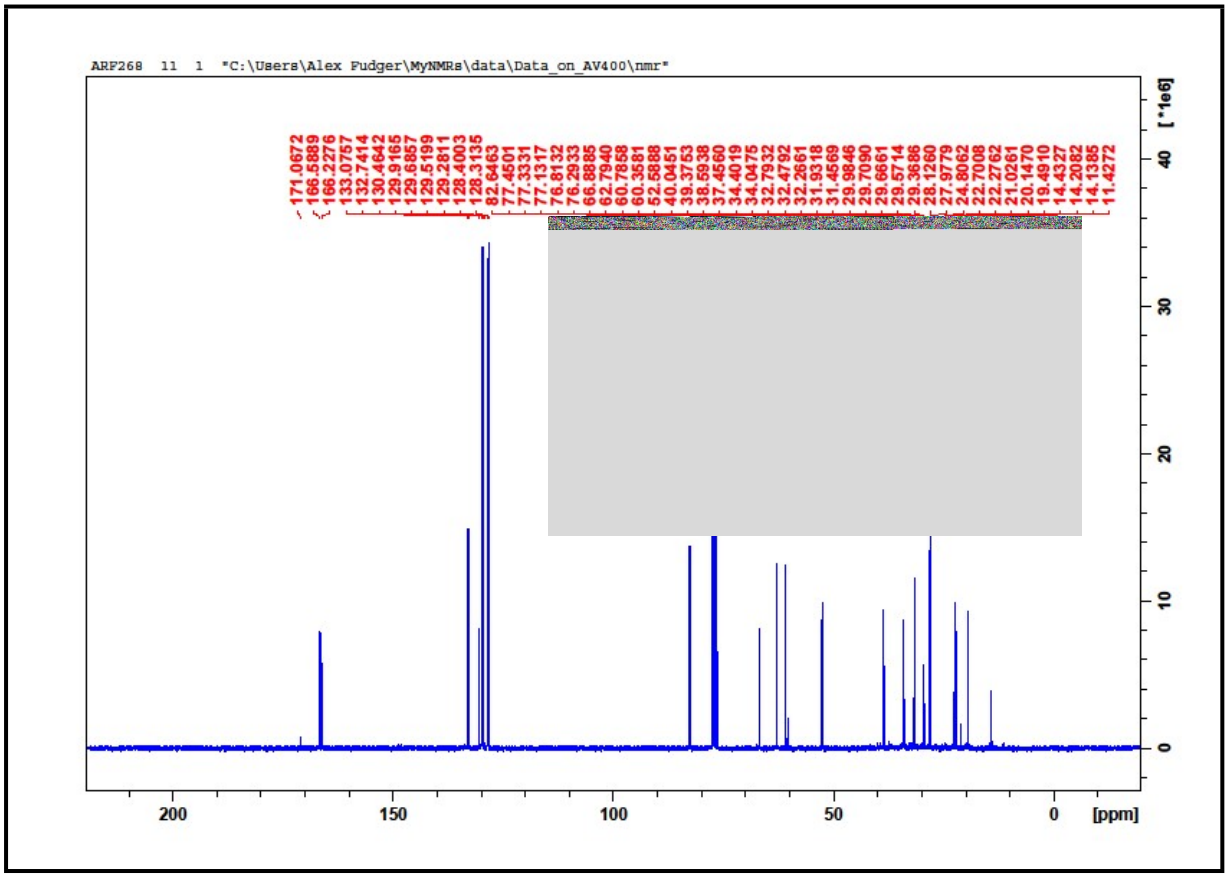
14.4830
8
6
4
2
[ppm]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[*1e6]
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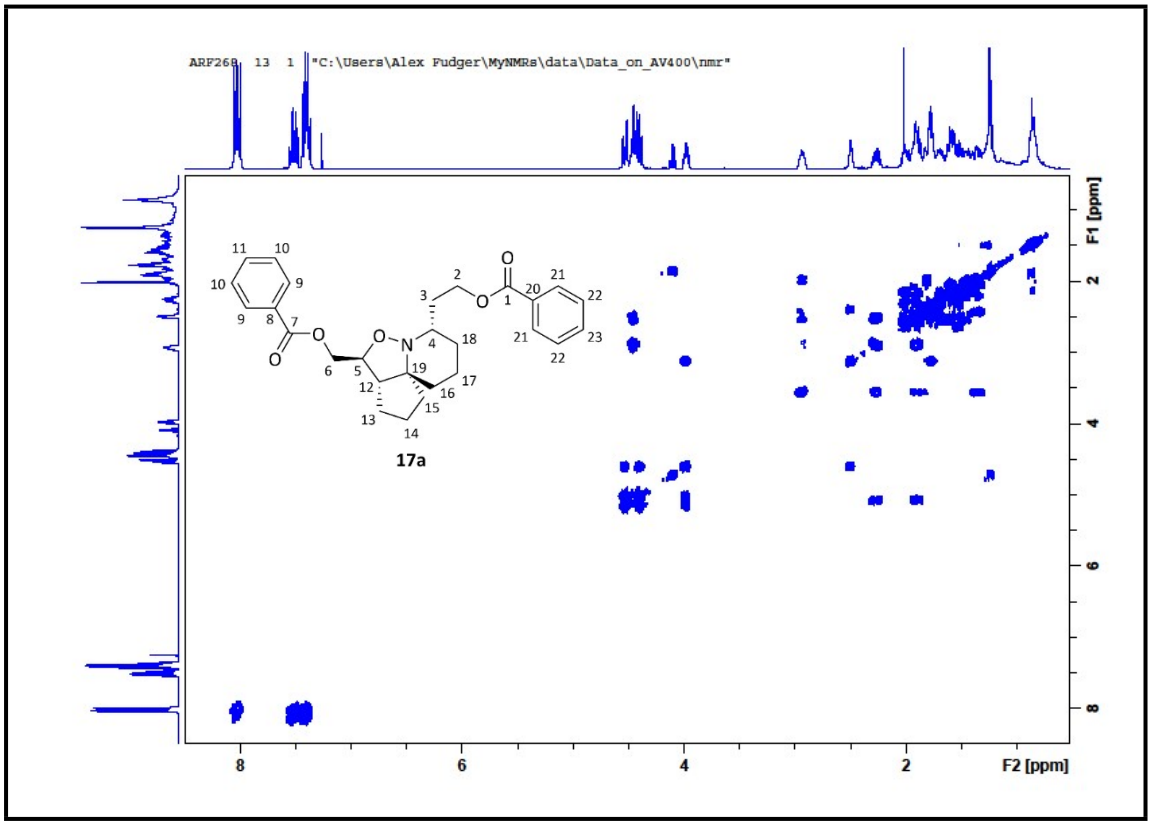
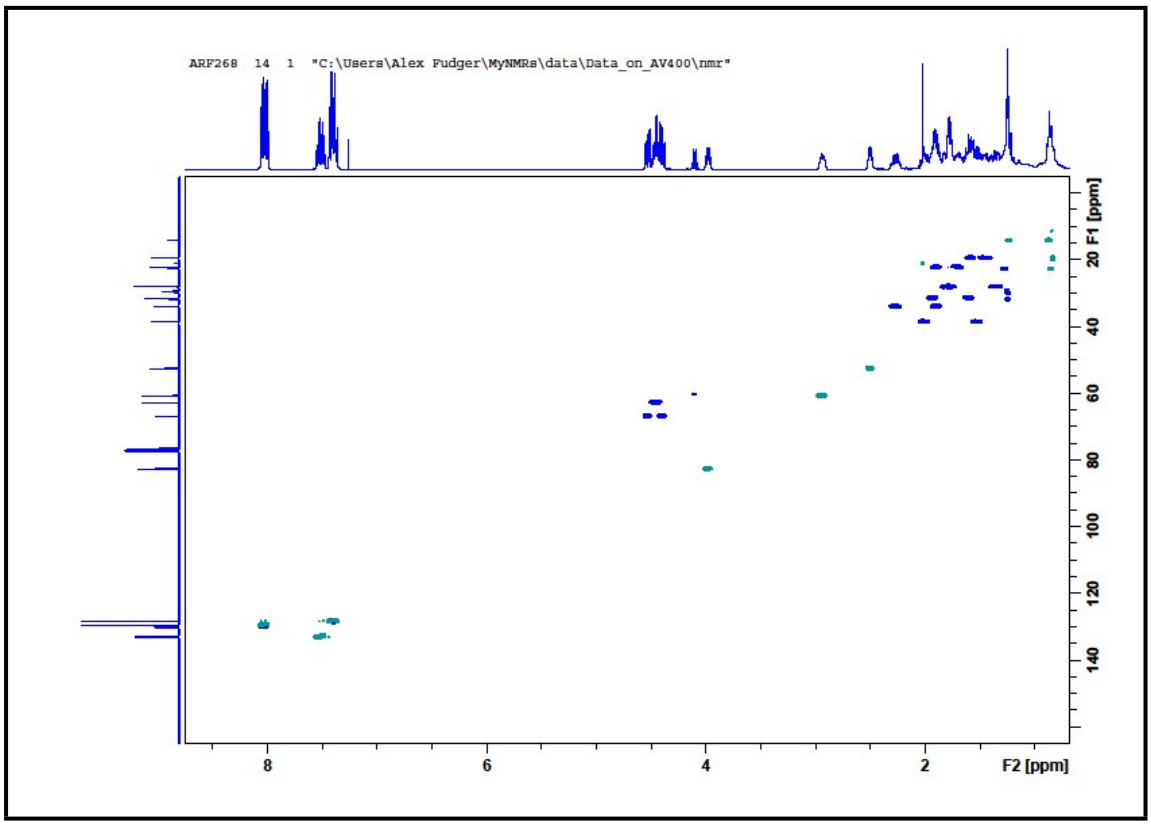
166.2276
133.0757
132.7414
130.4642
129.9165
129.6857
129.5199
129.2811
128.4003
128.3135
82.6463
77.4501
77.3331
77.1317
76.8132
76.2933
66.8885
62.7940
60.7858
60.3581
52.5888
40.0451
39.3753
38.5938
37.4560
34.4019
34.0475
32.7932
32.4792
32.2661
31.9318
31.4569
29.9846
29.7090
29.6661
29.5714
29.3686
28.1260
27.9779
24.8062
22.7008
22.2762
21.0261
20.1470
19.4910
14.4327
14.2082
14.1385
11.4272
200
150
100
50
0
[ppm]

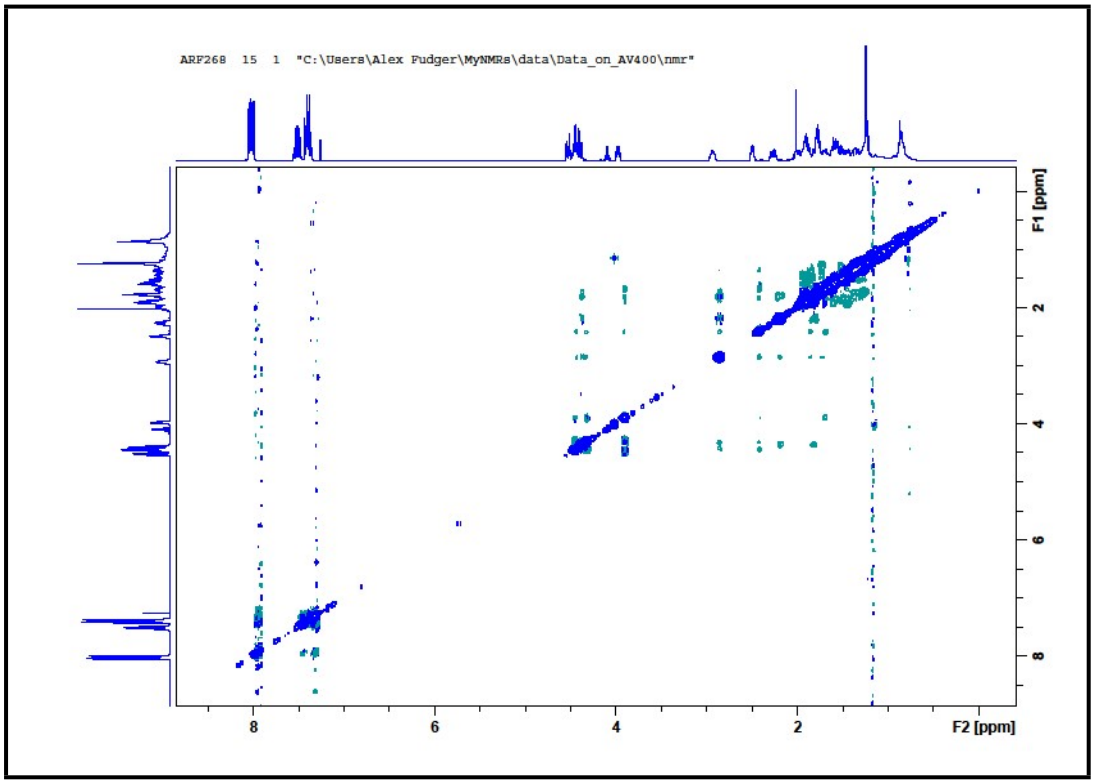
COSY NMR spectrum for compound **17a**

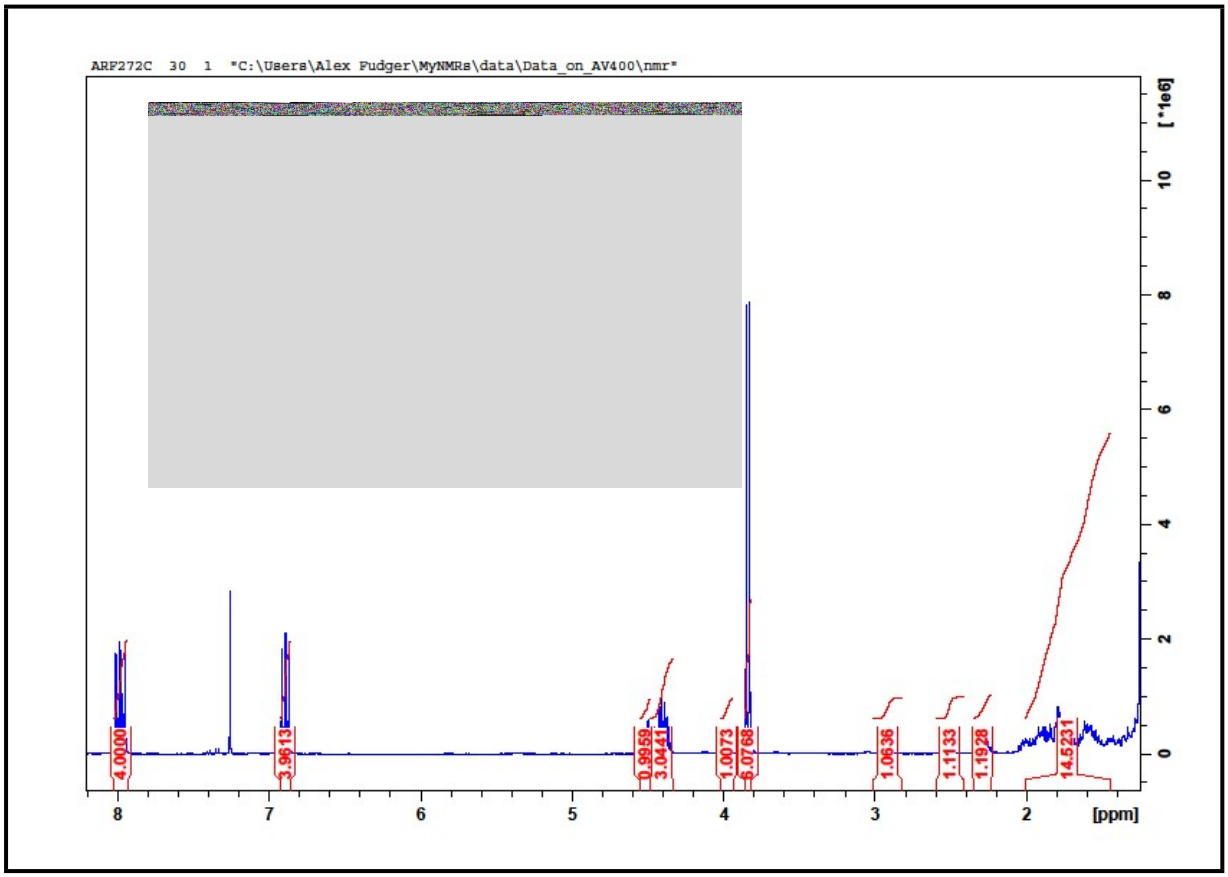



HSQC NMR spectrum for compound **17a**



NOESY NMR spectrum for compound **17a**

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6
4
2
0
4.0000
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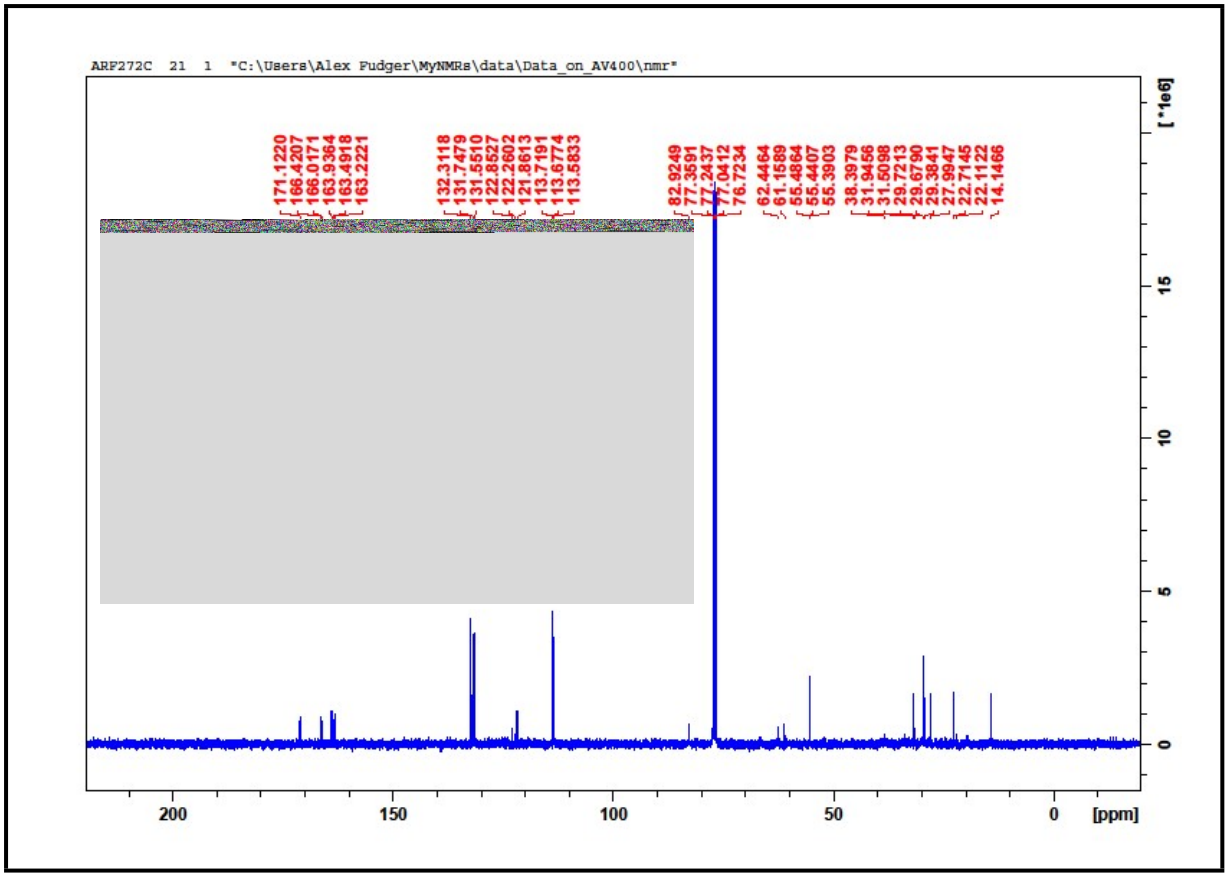
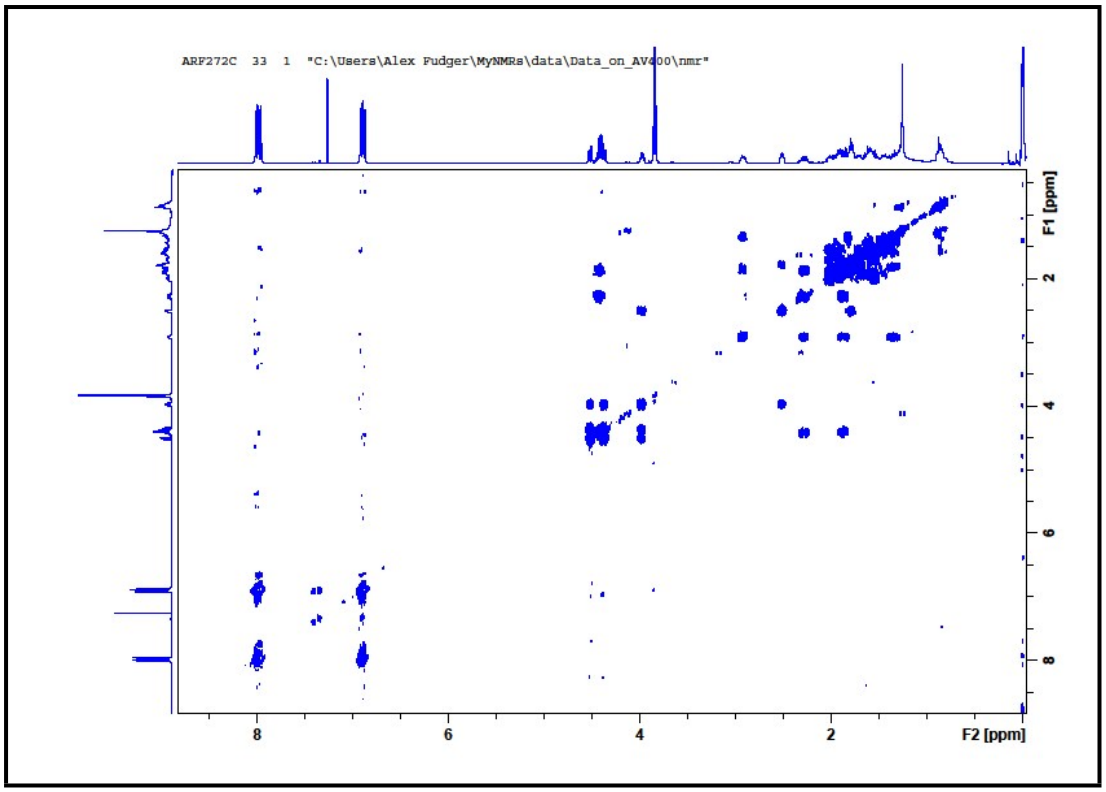
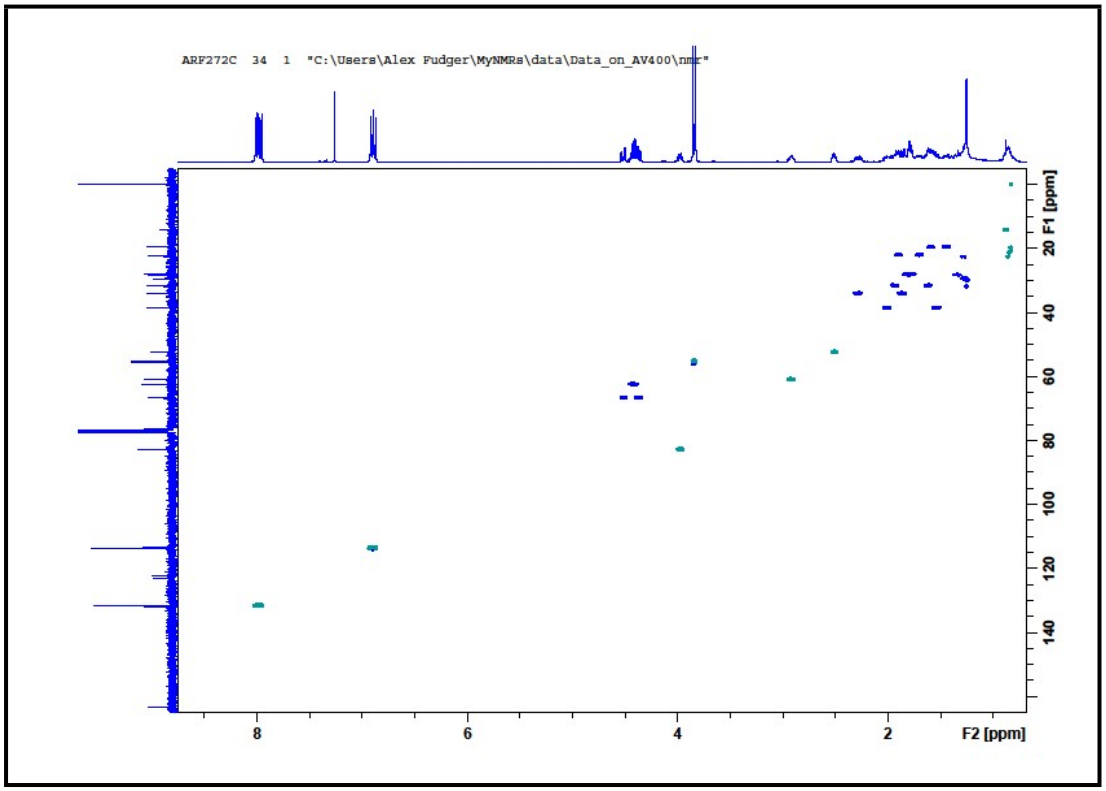
0.9959
3.0441
1.0073
6.0768
1.0636
1.1133
1.1928
14.5231
8
7
6
5
4
3
2
[ppm]



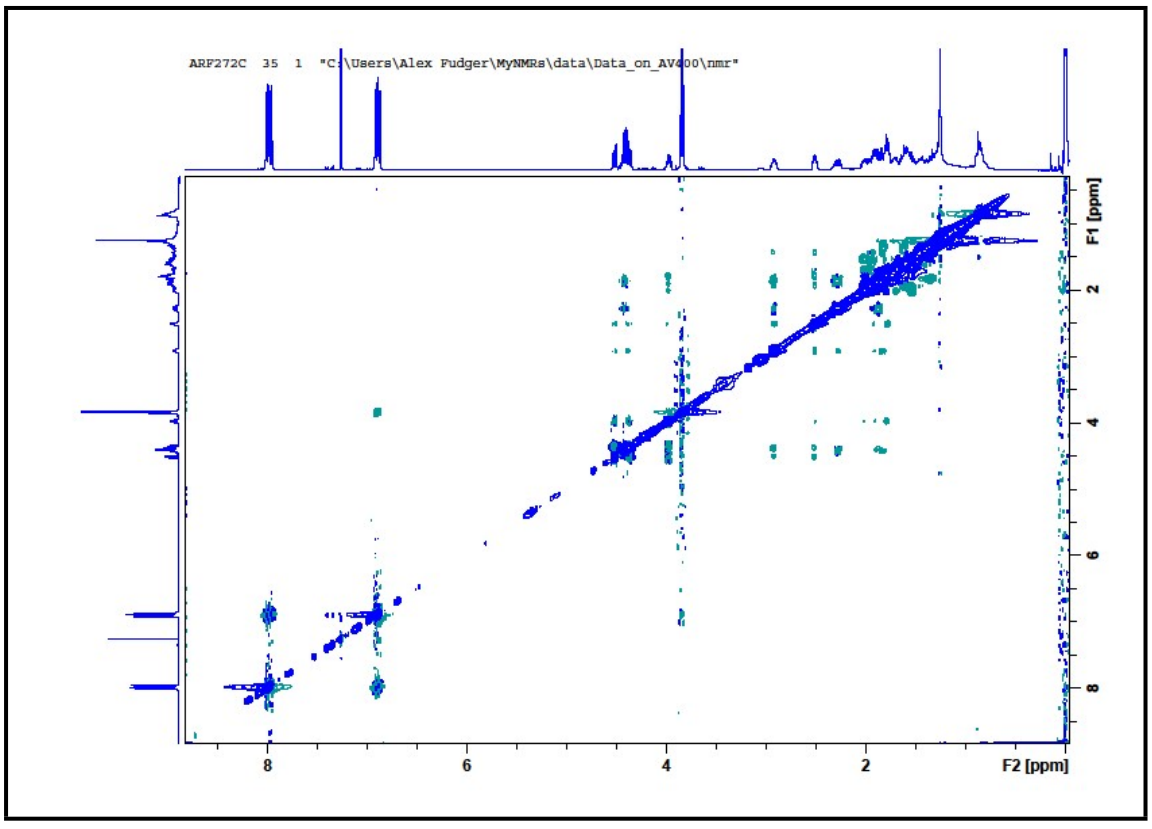

COSY NMR spectrum for compound **17b**

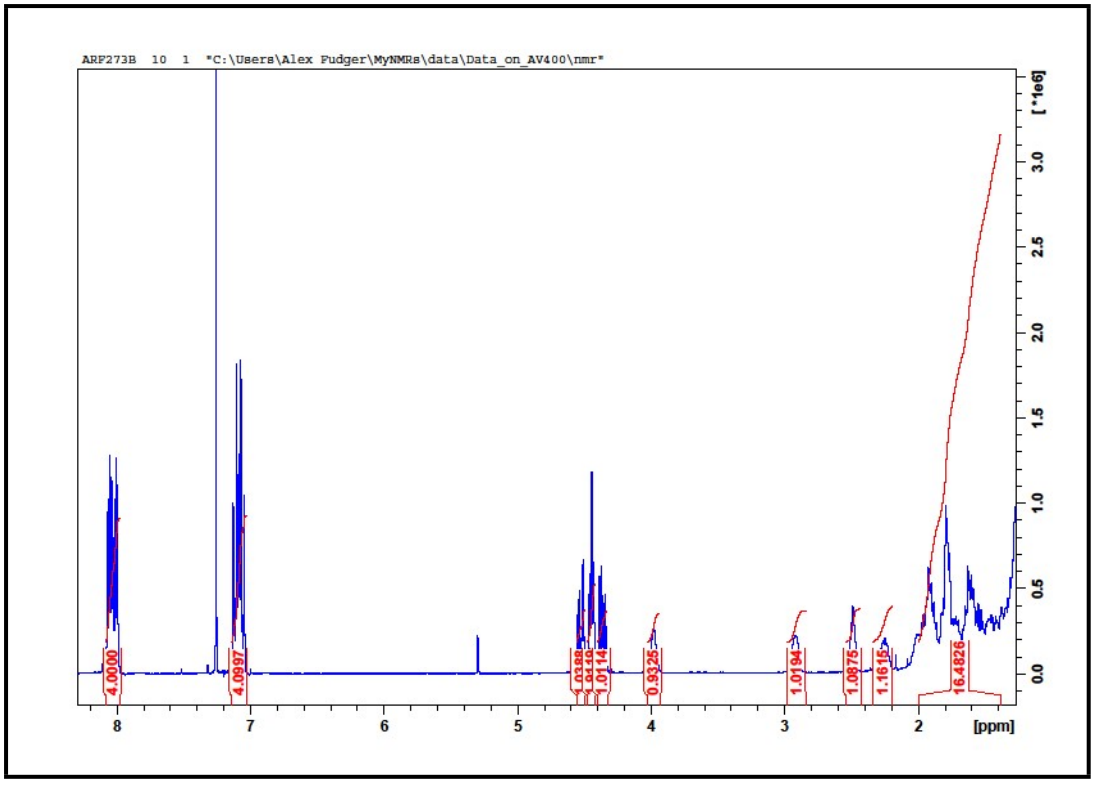


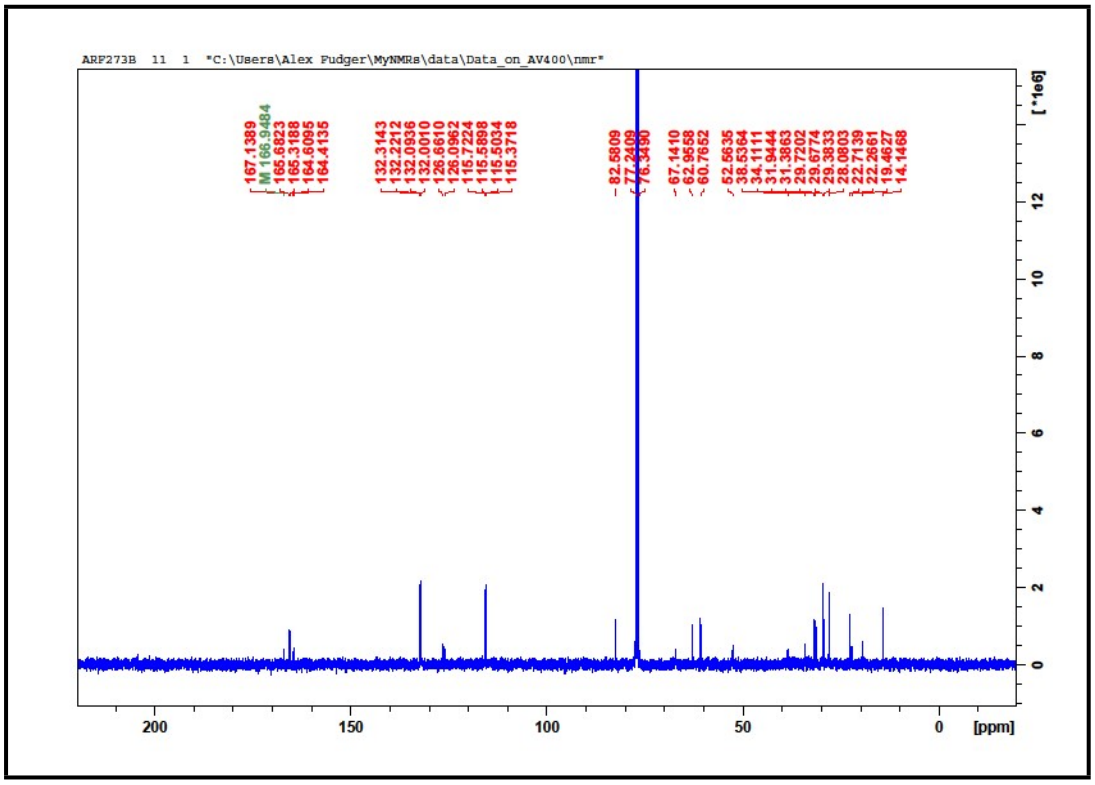
HSQC NMR spectrum for compound **17b**




NOESY NMR spectrum for compound **17b**




ARF273B 10 1 "C:\Users\Alex Fudger\MyNMRs\data\Data_on_AV400\nmr"
4.0000
4.0097
1.0114
0.9325
1.0194
1.0875
1.1615
16.4826
8
7
6
5
4
3
2
[ppm]
[*1e6]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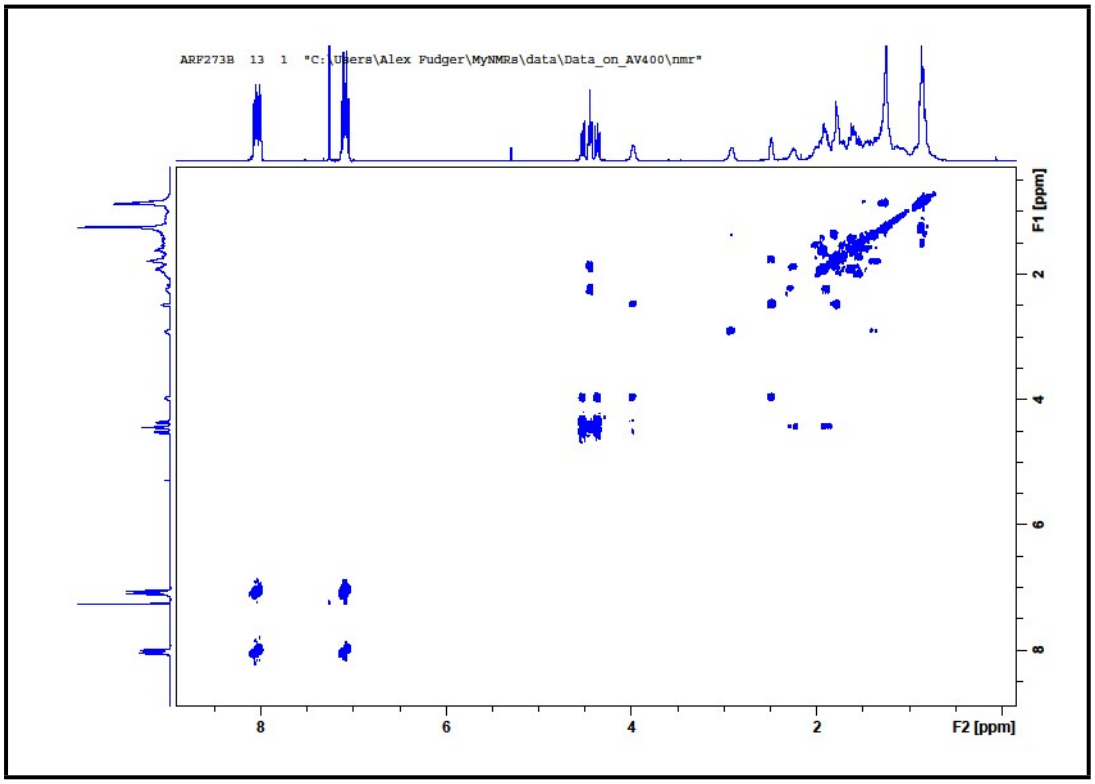
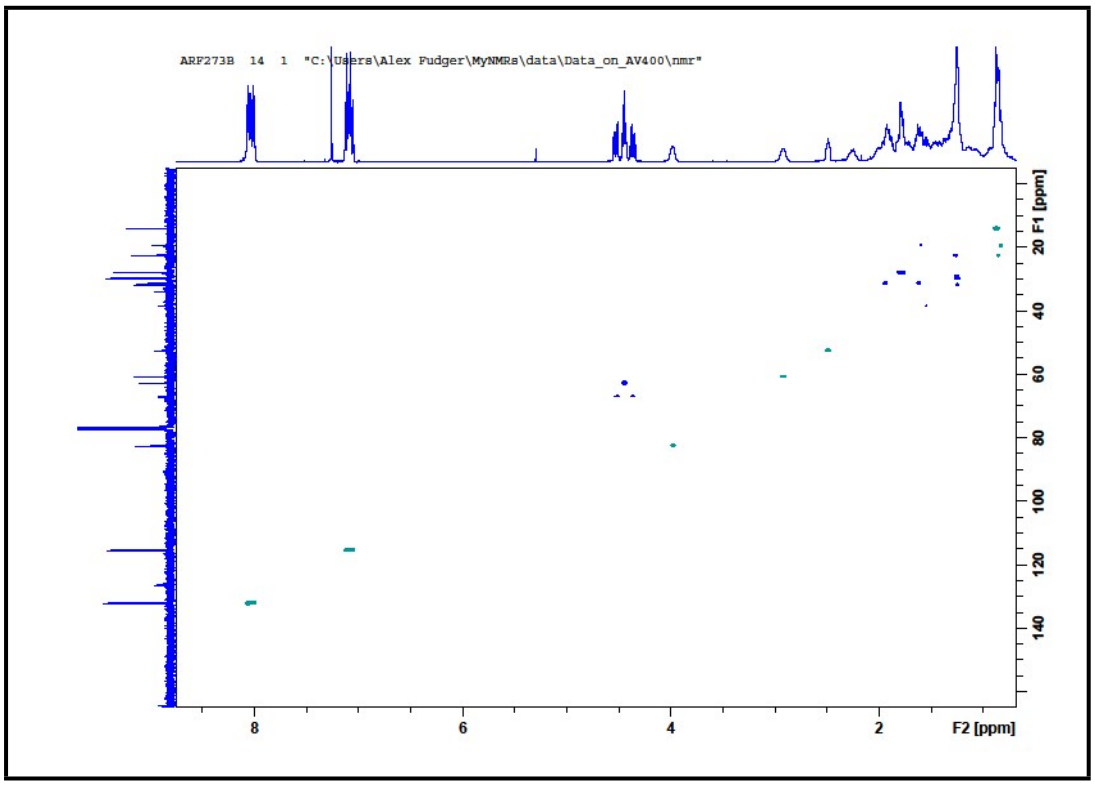
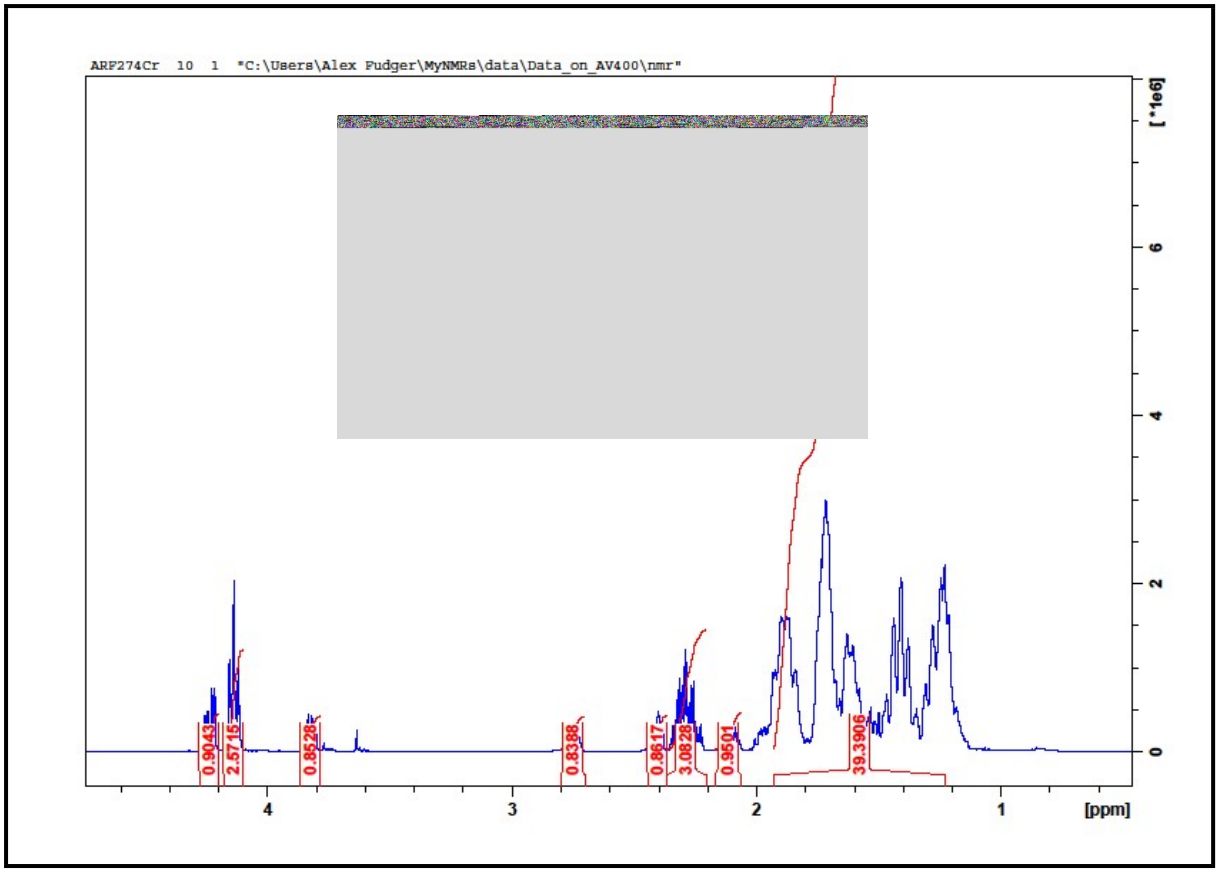
M 166.9484
165.6823
165.3188
164.6095
164.4135
132.3143
132.2212
132.0936
132.0010
126.6610
126.0962
115.7224
115.5898
115.5034
115.3718
82.5809
77.2409
76.3490
67.1410
62.9558
60.7652
52.5635
38.5364
34.1111
31.9444
31.3863
29.7202
29.6774
29.3833
28.0803
22.7139
22.2661
19.4627
14.1468
200
150
100
50
0
[ppm]
[*1e6]

COSY NMR spectrum for compound **17c**




HSQC NMR spectrum for compound **17c**

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2
4
6
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2.5745
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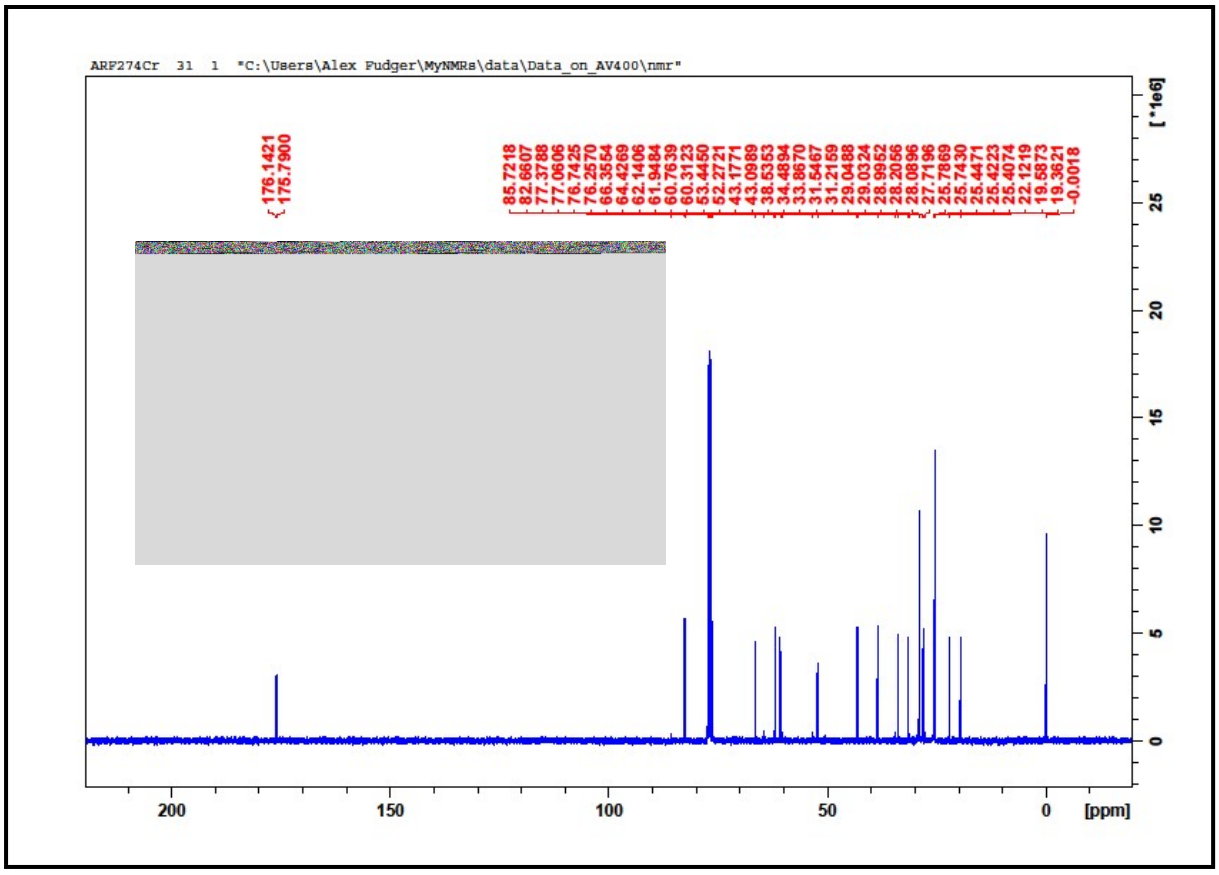
0.8388
0.8617
3.0828
0.9501
39.3906
4
3
2
1
[ppm]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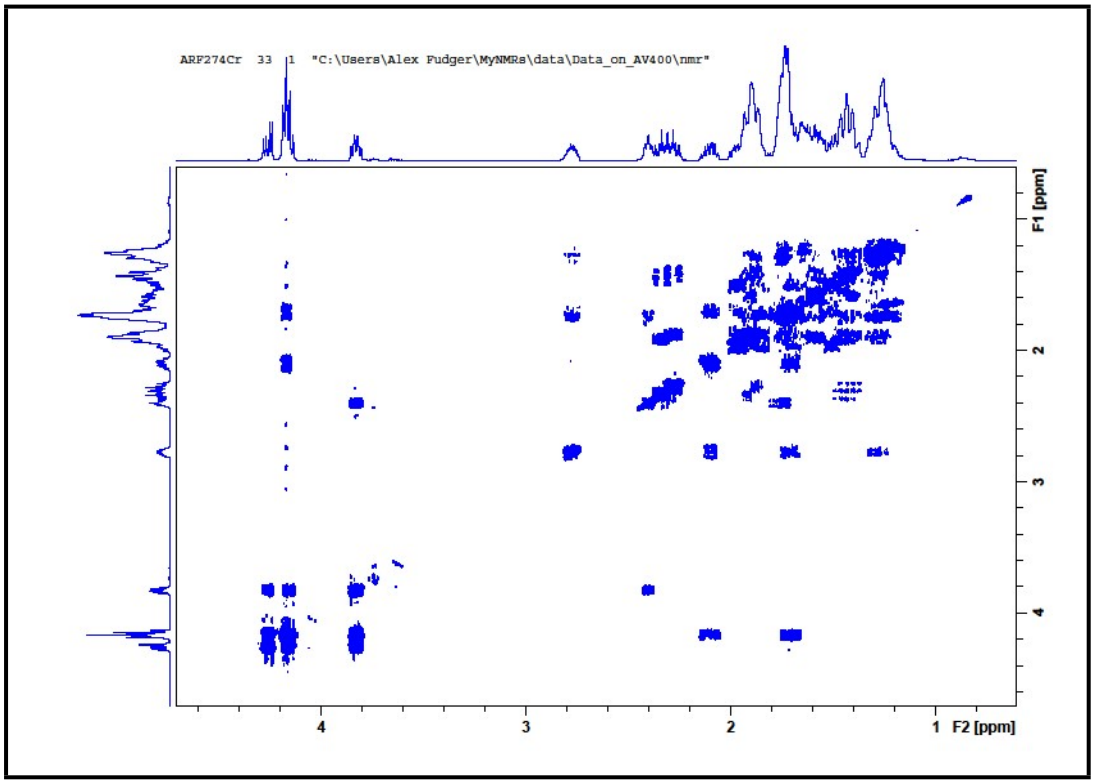
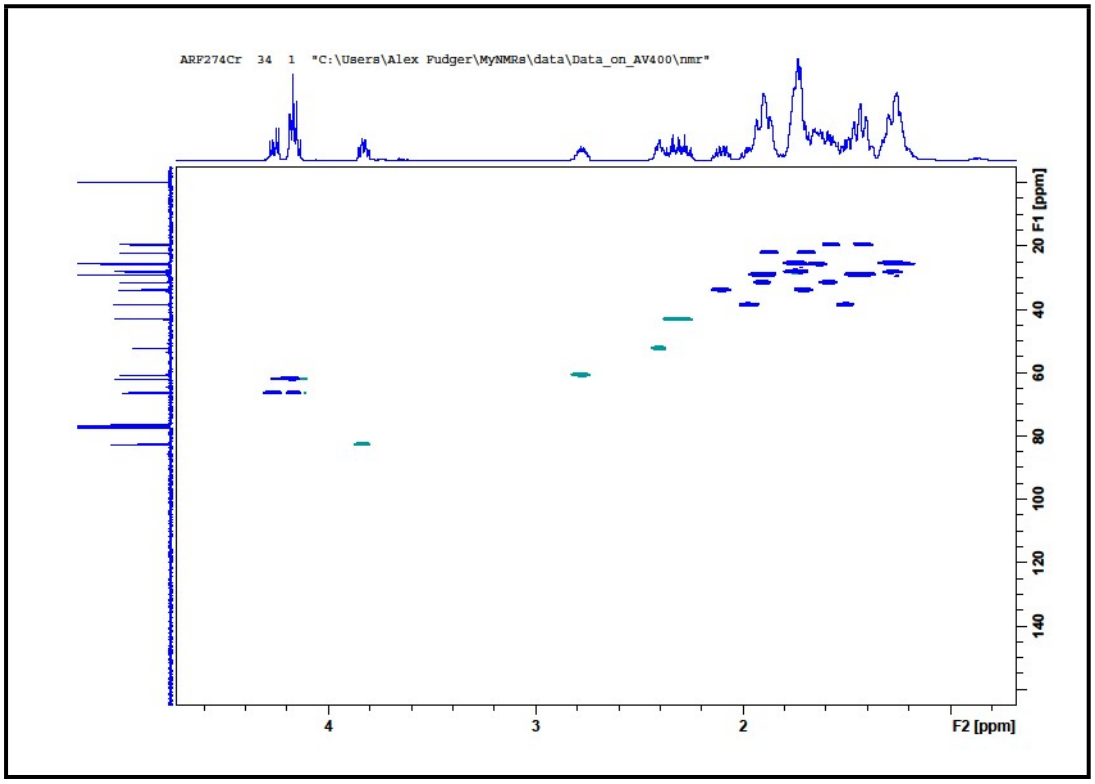
[*1e6]
0
5
10
15
20
25
176.1421
175.7900
85.7218
82.6607
77.3788
77.0606
76.7425
76.2570
66.3554
64.4269
62.1406
61.9484
60.7639
60.3123
53.4450
52.2721
43.1771
43.0989
38.5353
34.4894
33.8670
31.5467
31.2159
29.0488
29.0324
28.9952
28.2056
28.0896
27.7196
25.7869
25.7430
25.4471
25.4223
25.4074
22.1219
19.5873
19.3621
-0.0018
200
150
100
50
0
[ppm]

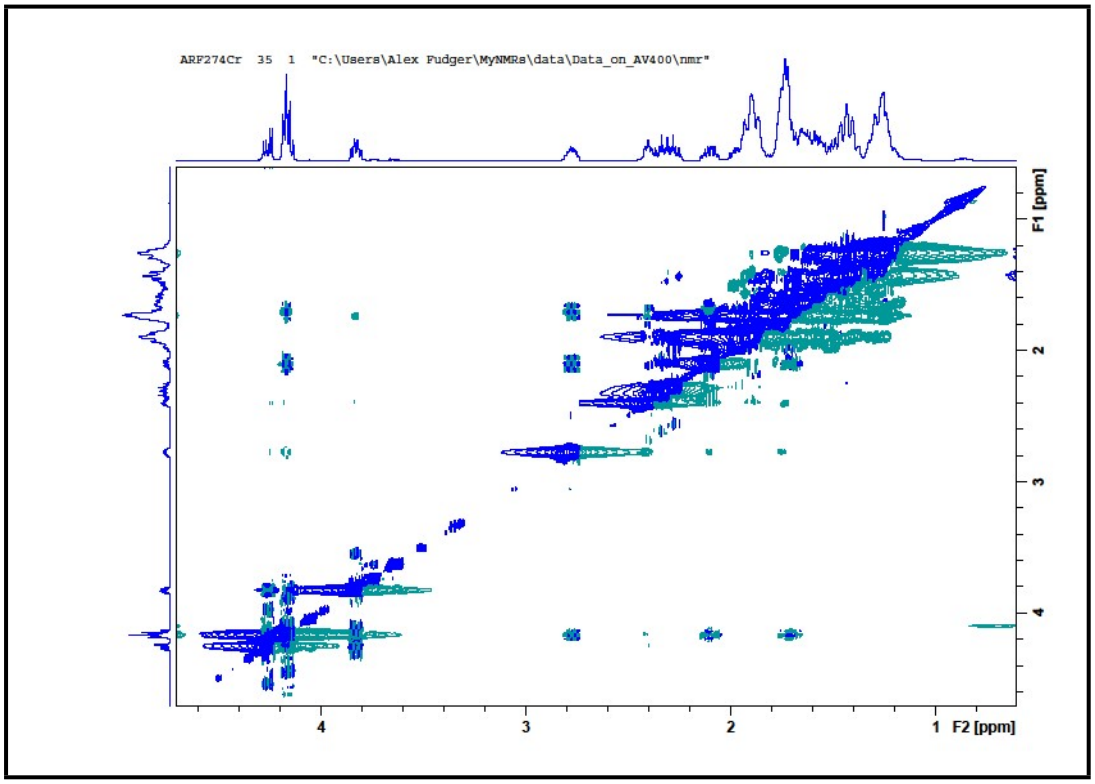
COSY NMR spectrum for compound **17d**

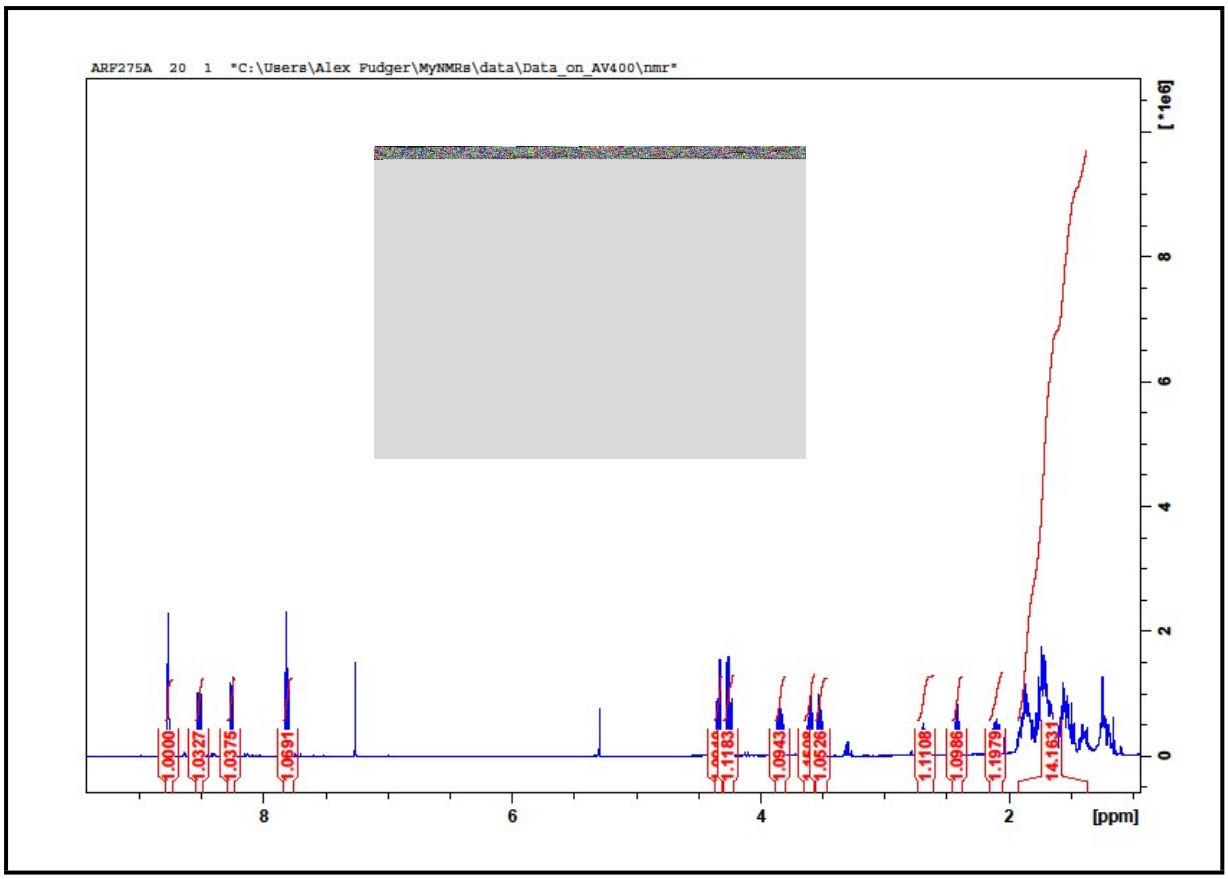



HSQC NMR spectrum for compound **17d**



NOESY NMR spectrum for compound **17d**

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[*1e6]
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6
4
2
[ppm]
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1.0691
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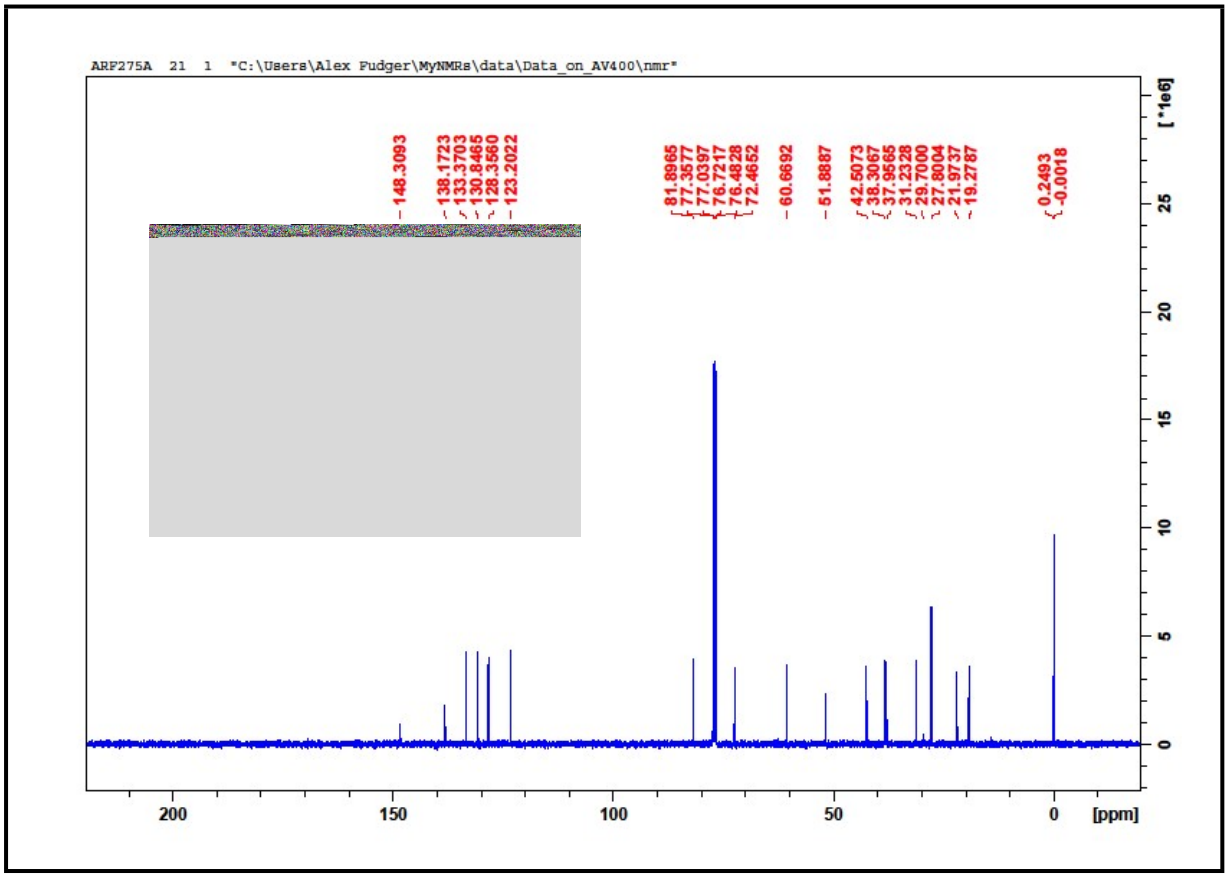
1.0943
1.0528
1.1108
1.0986
1.1979
14.1631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130.8465
128.3560
123.2022
81.8965
77.3577
77.0397
76.7217
76.4828
72.4652
60.6692
51.8887
42.5073
38.3067
37.9565
31.2328
29.7000
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0.2493
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200
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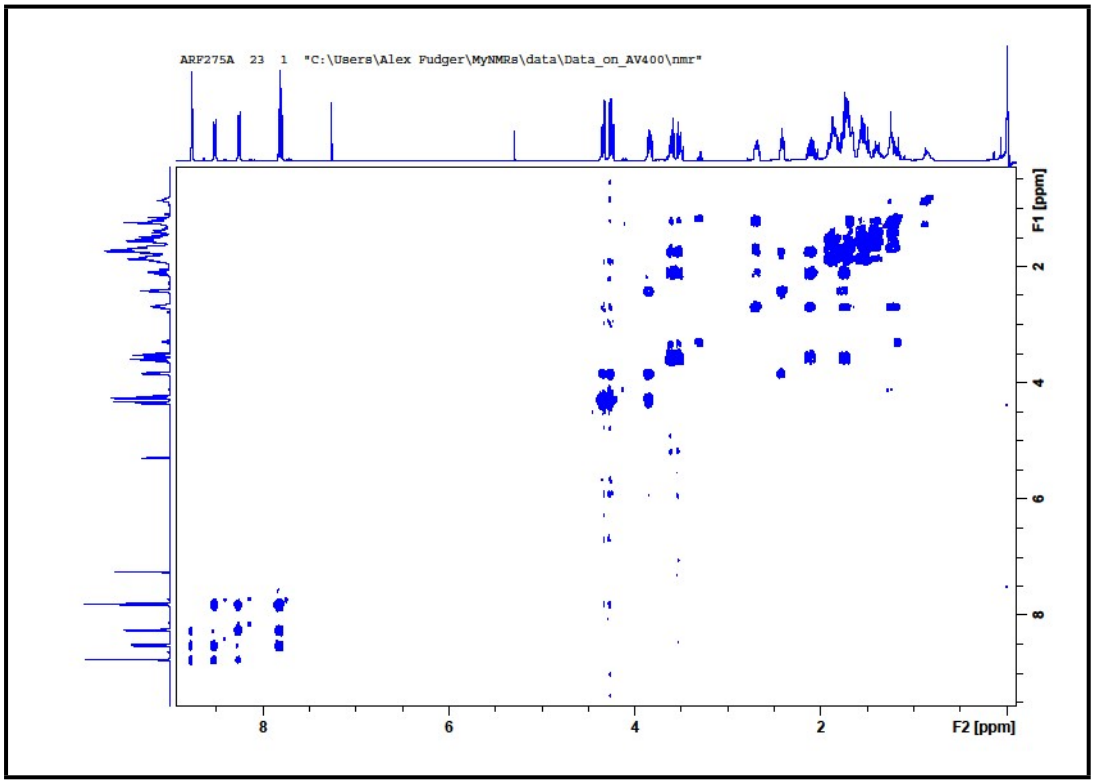
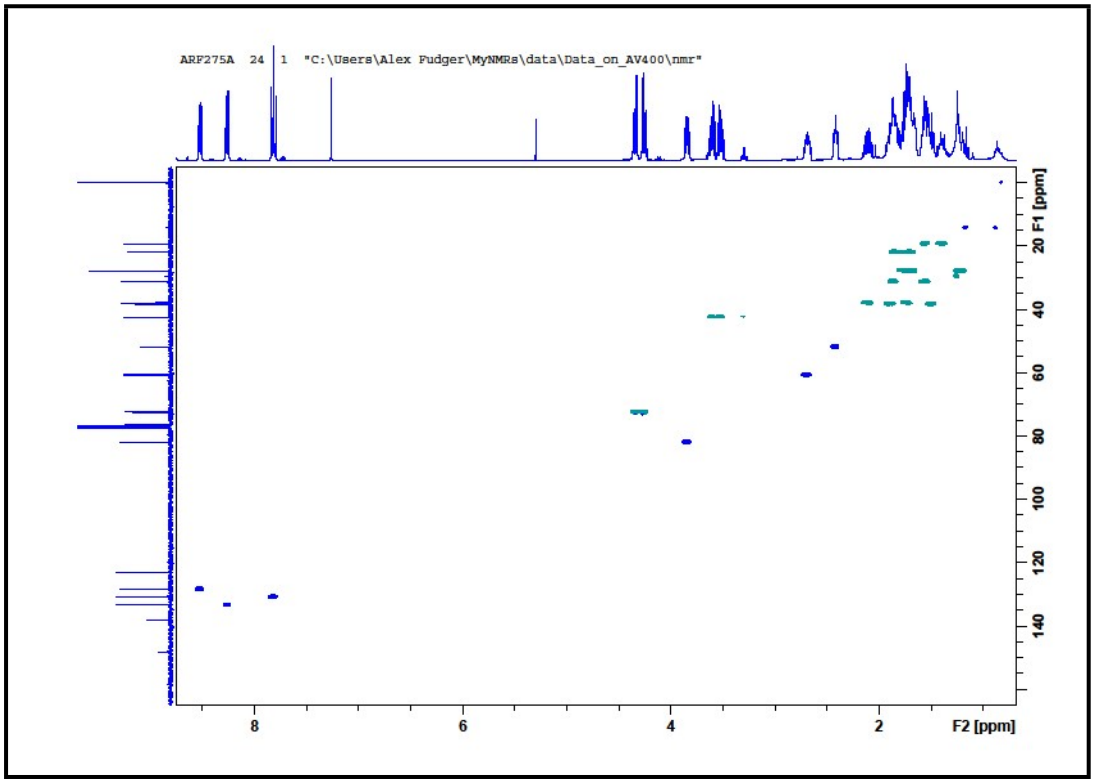
100
50
0
[ppm]

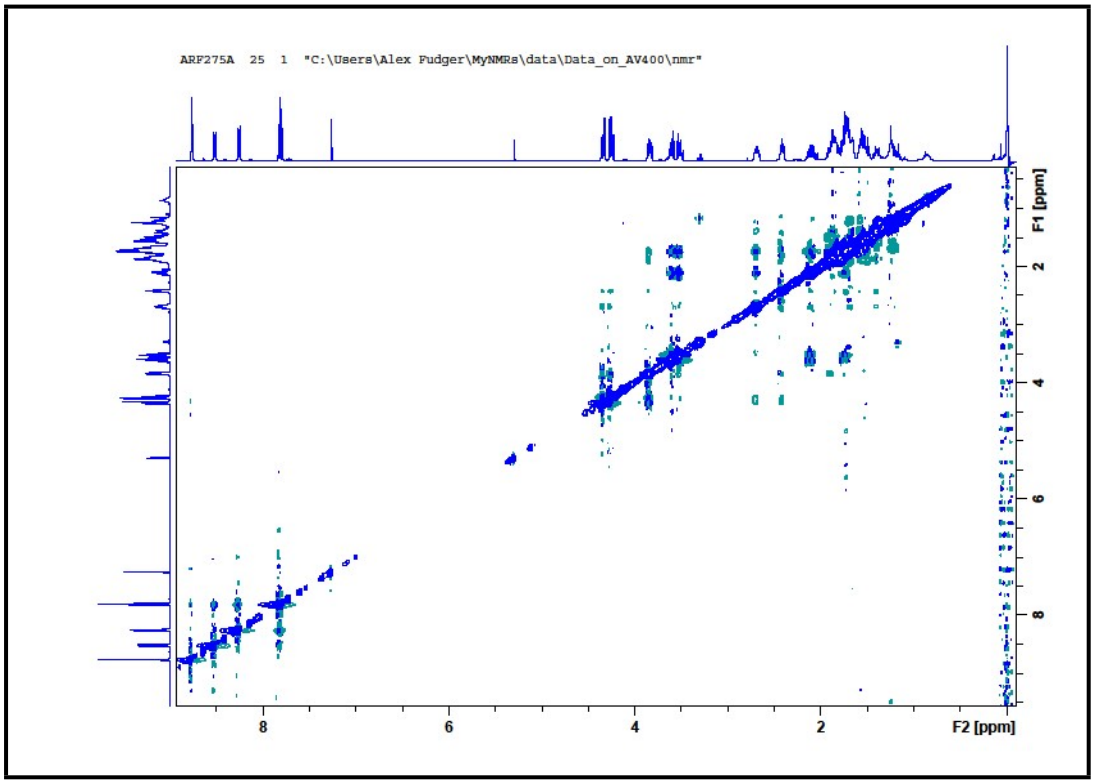
COSY NMR spectrum for compound **17e**

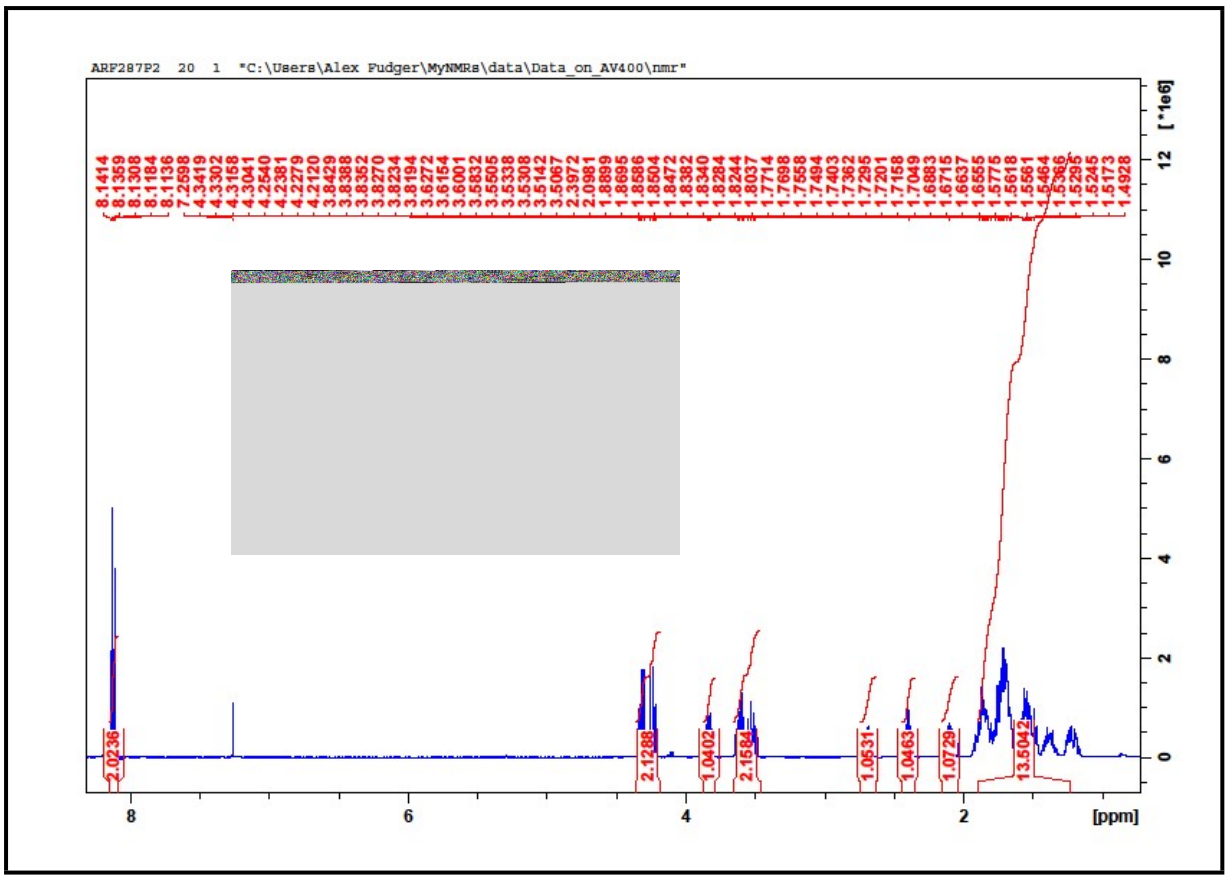



HSQC NMR spectrum for compound **17e**



NOESY NMR spectrum for compound **17e**

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4.3041
4.2640
4.2381
4.2279
4.2120
3.8429
3.8388
3.8352
3.8270
3.8234
3.8194
3.6272
3.6154
3.6001
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3.5308
3.5142
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2.3972
2.0981
1.8899
1.8695
1.8686
1.8504
1.8472
1.8382
1.8340
1.8284
1.8244
1.8037
1.7714
1.7698
1.7558
1.7494
1.7403
1.7362
1.7295
1.7201
1.7158
1.7049
1.6883
1.6715
1.6637
1.6555
1.5775
1.5618
1.5561
1.5464
1.5366
1.5295
1.5245
1.5173
1.4928
2.0236
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2.1584
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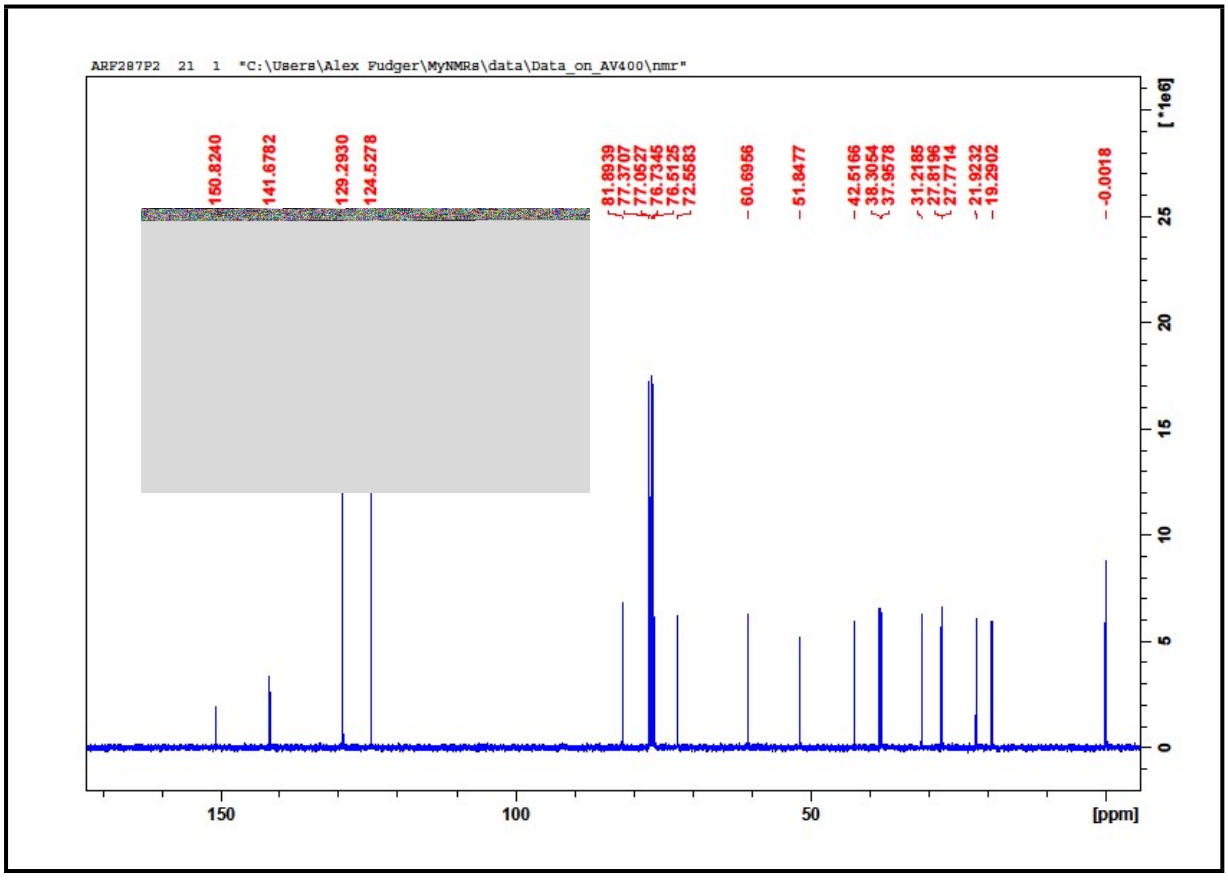
1.0463
1.0729
13.5042
[*1e6]
[ppm]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77.3707
77.0527
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72.5583
60.6956
51.8477
42.5166
38.3054
37.9578
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19.2902
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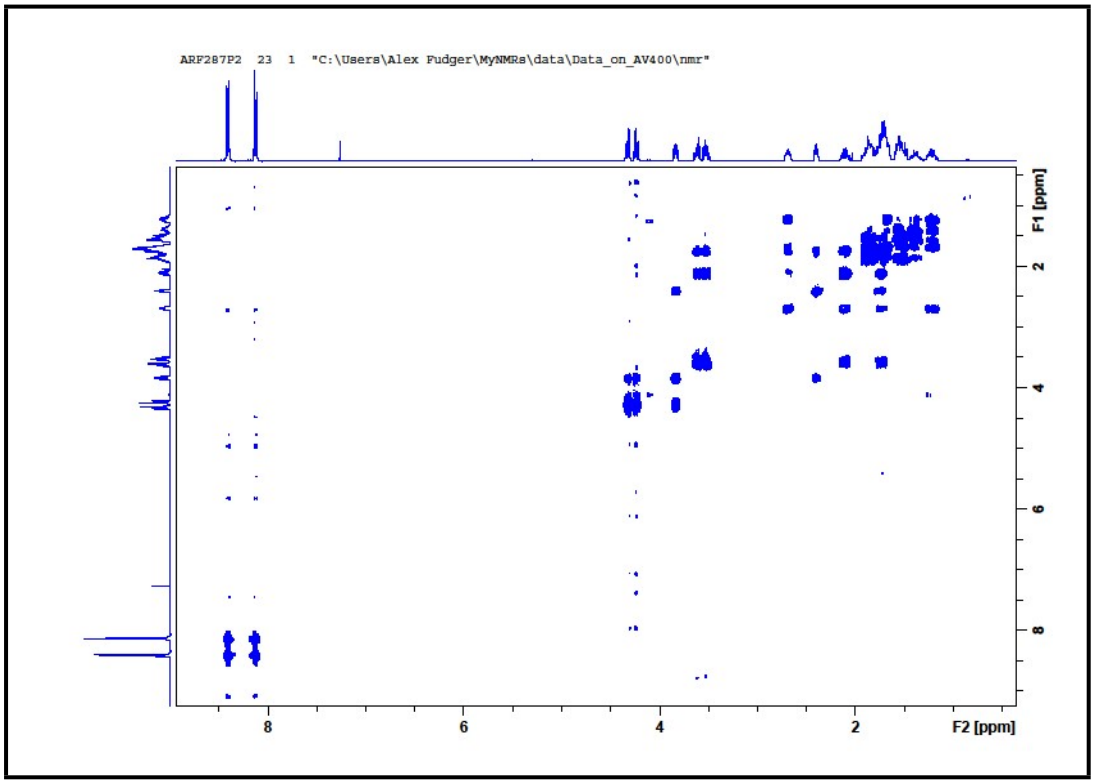
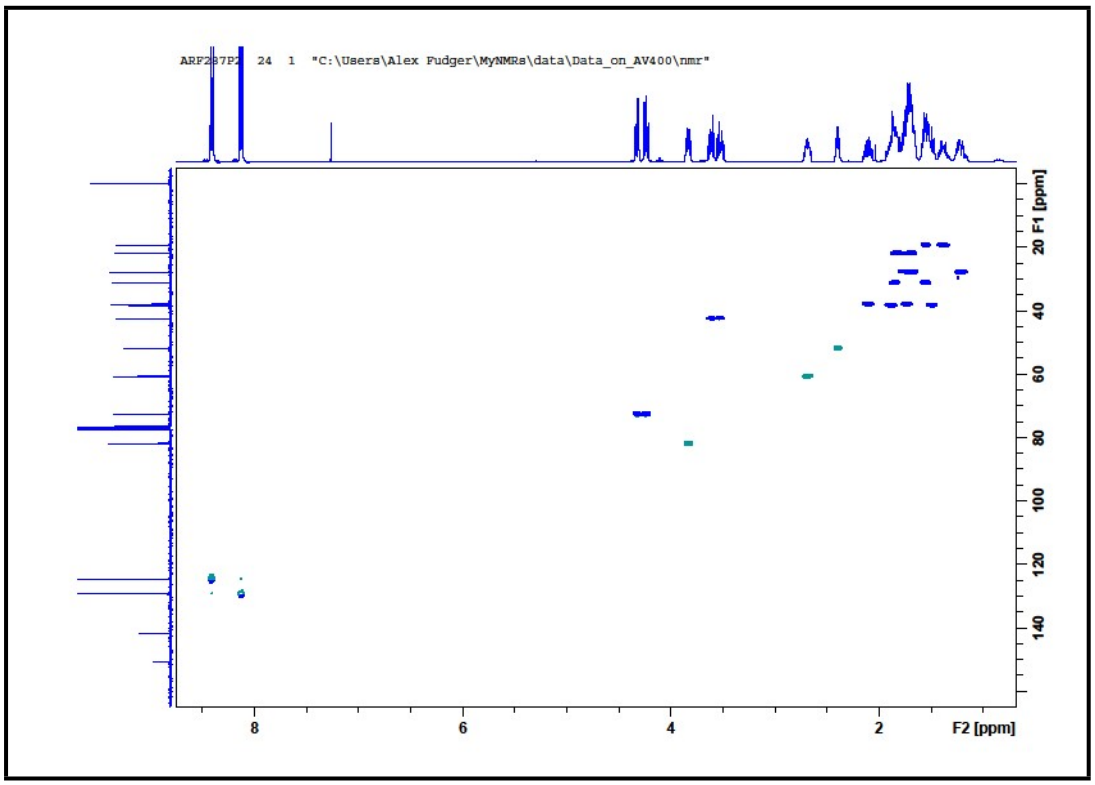
[*1e6]
[ppm]

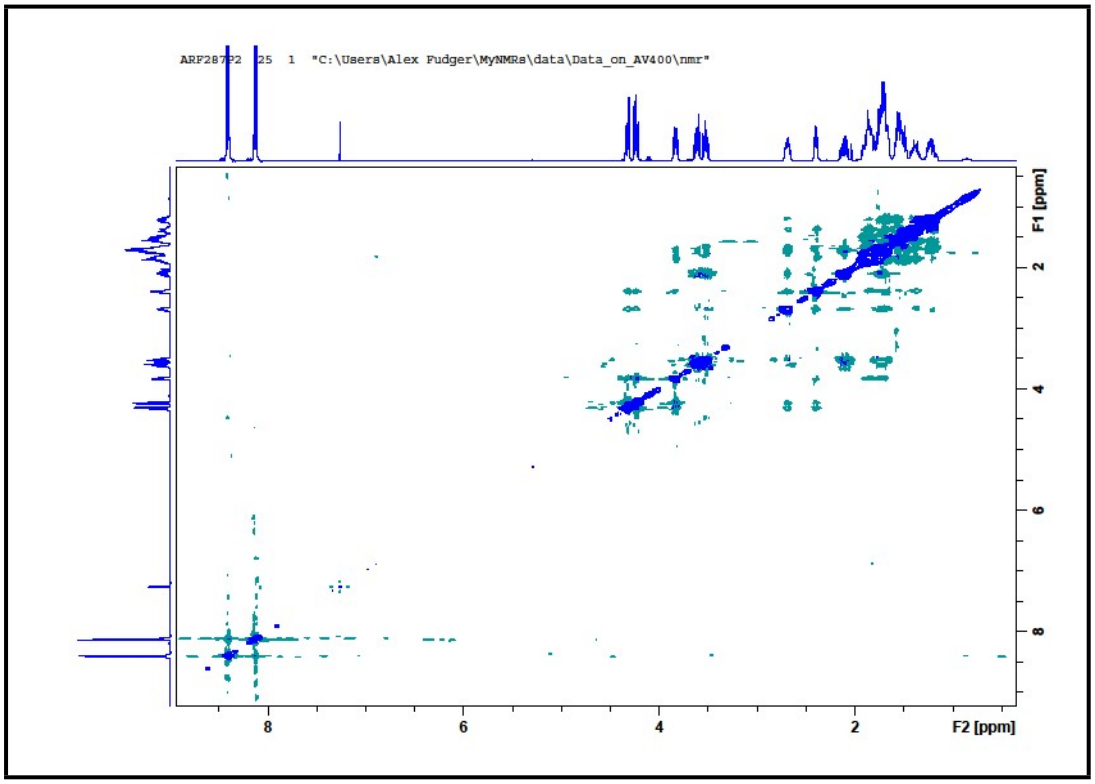
COSY NMR spectrum for compound **17f**

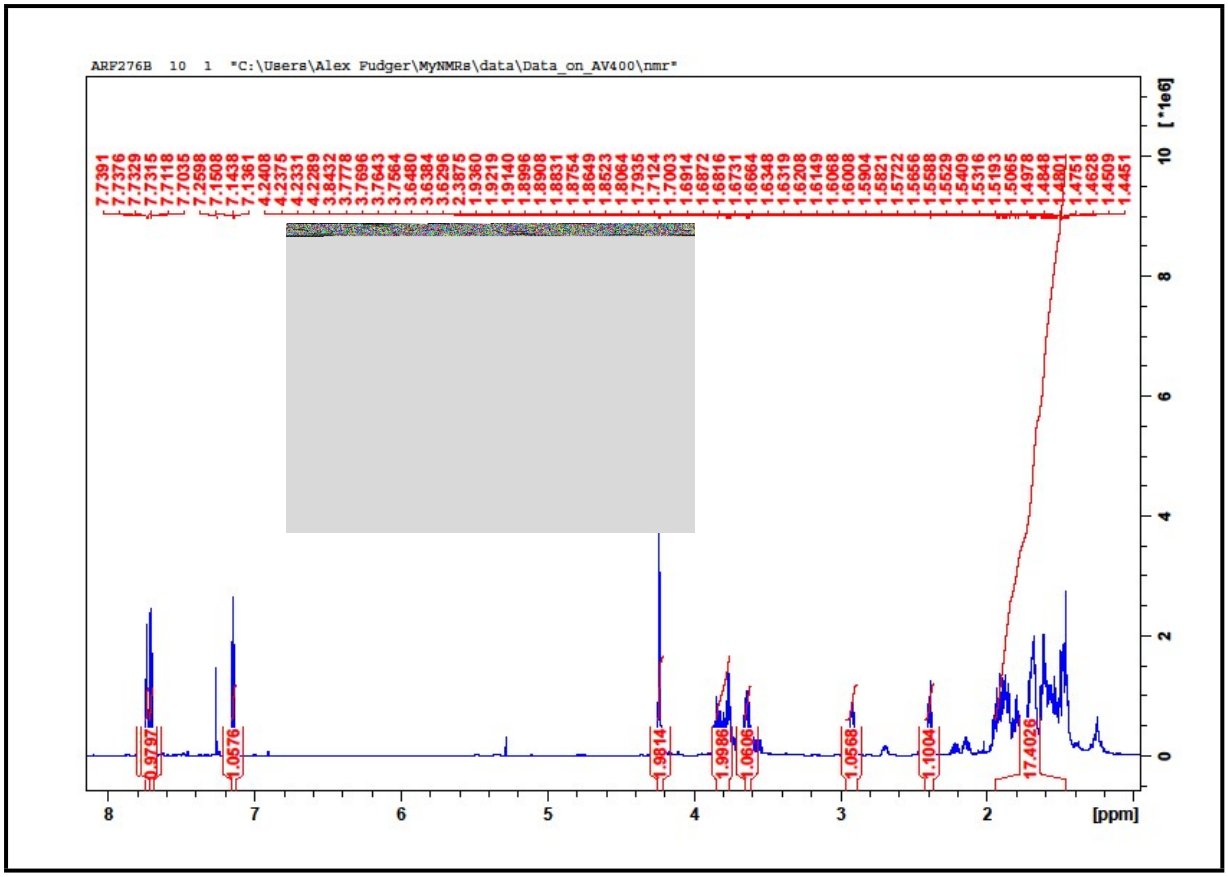



HSQC NMR spectrum for compound **17f**

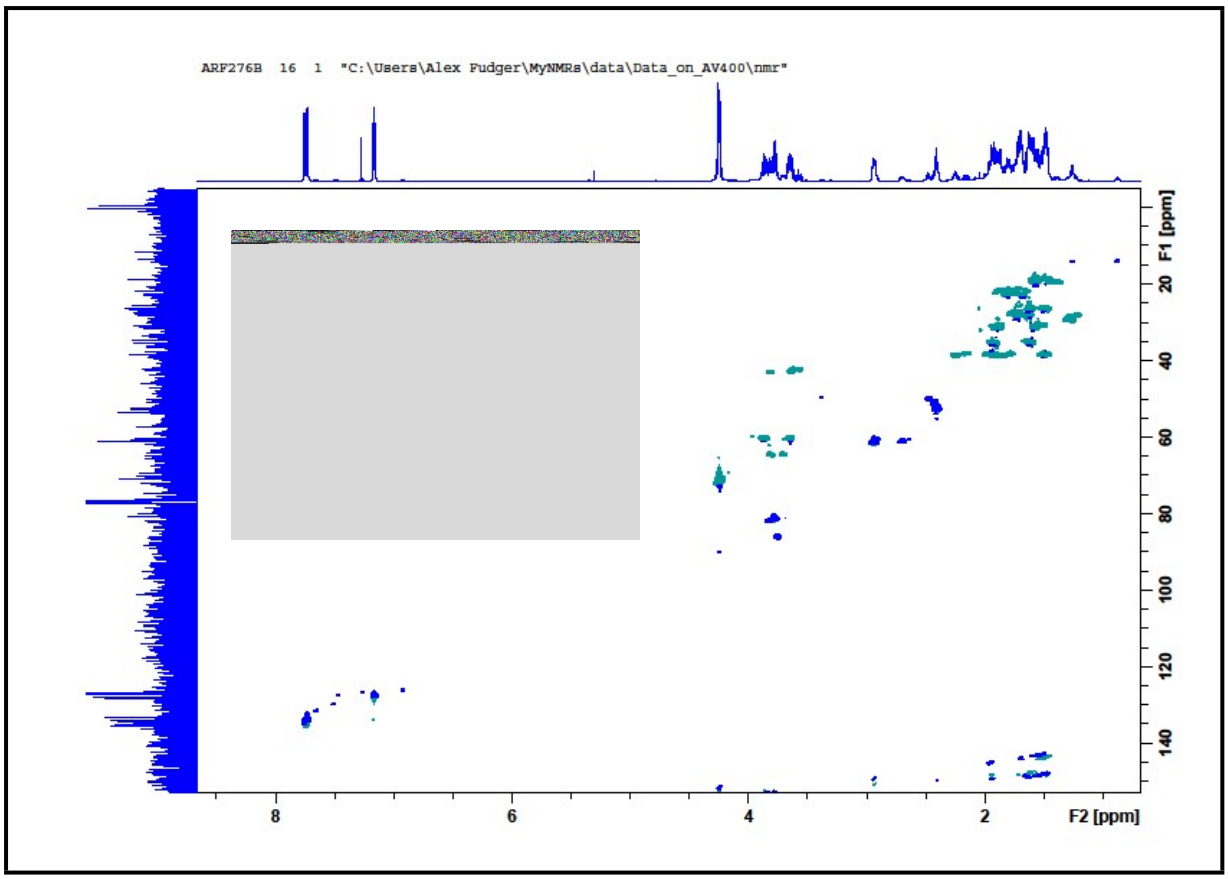


NOESY NMR spectrum for compound **17f**

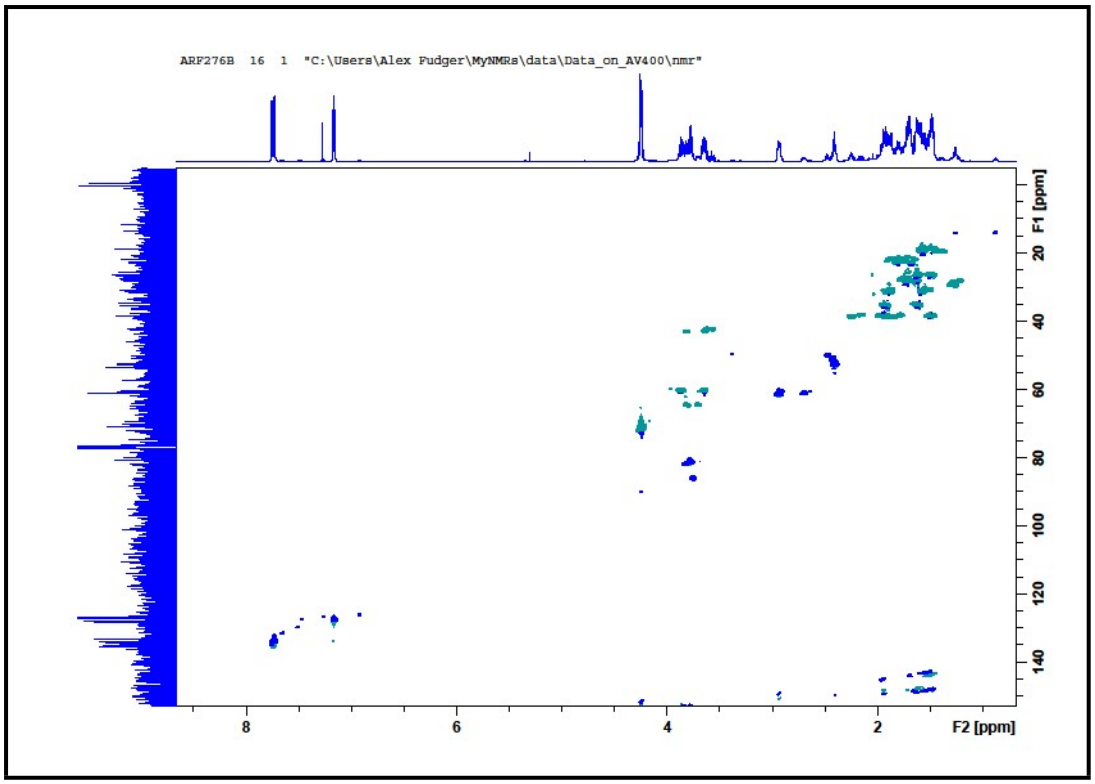




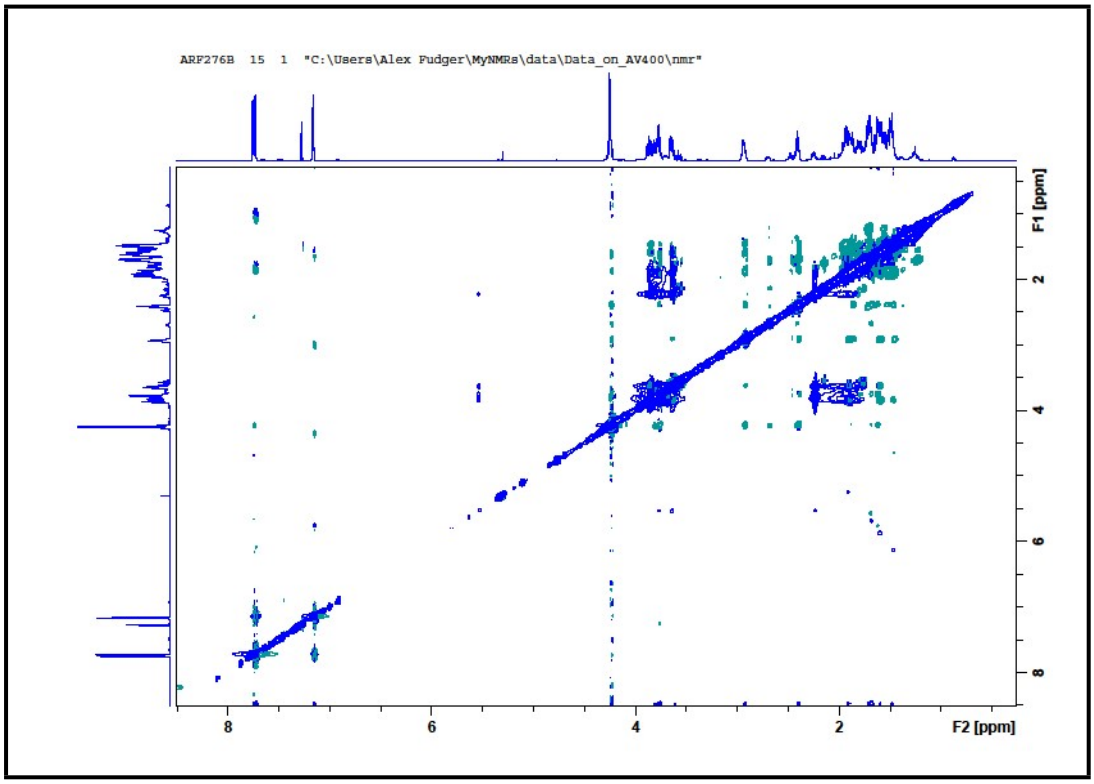

COSY NMR spectrum for compound **17ga**

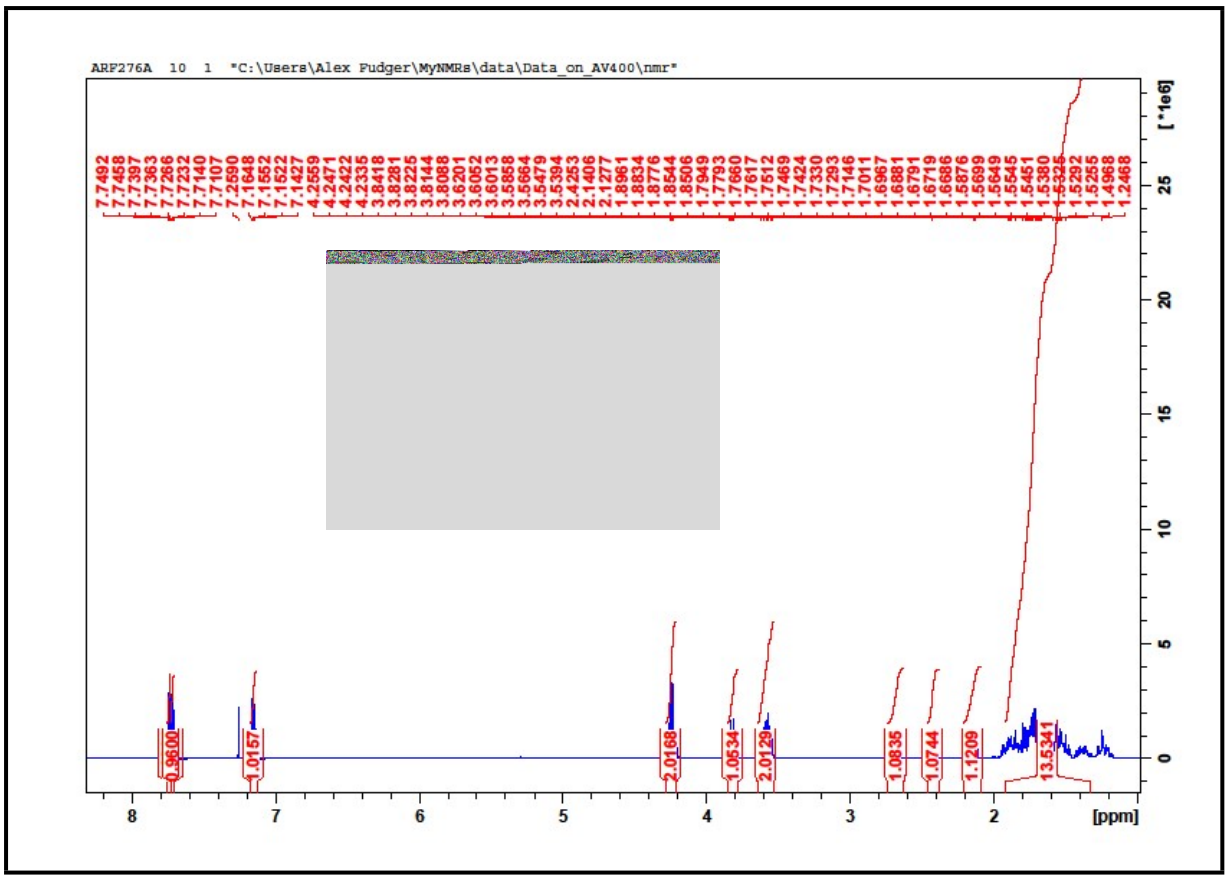



HSQC NMR spectrum for compound **17ga**



NOESY NMR spectrum for compound **17ga**

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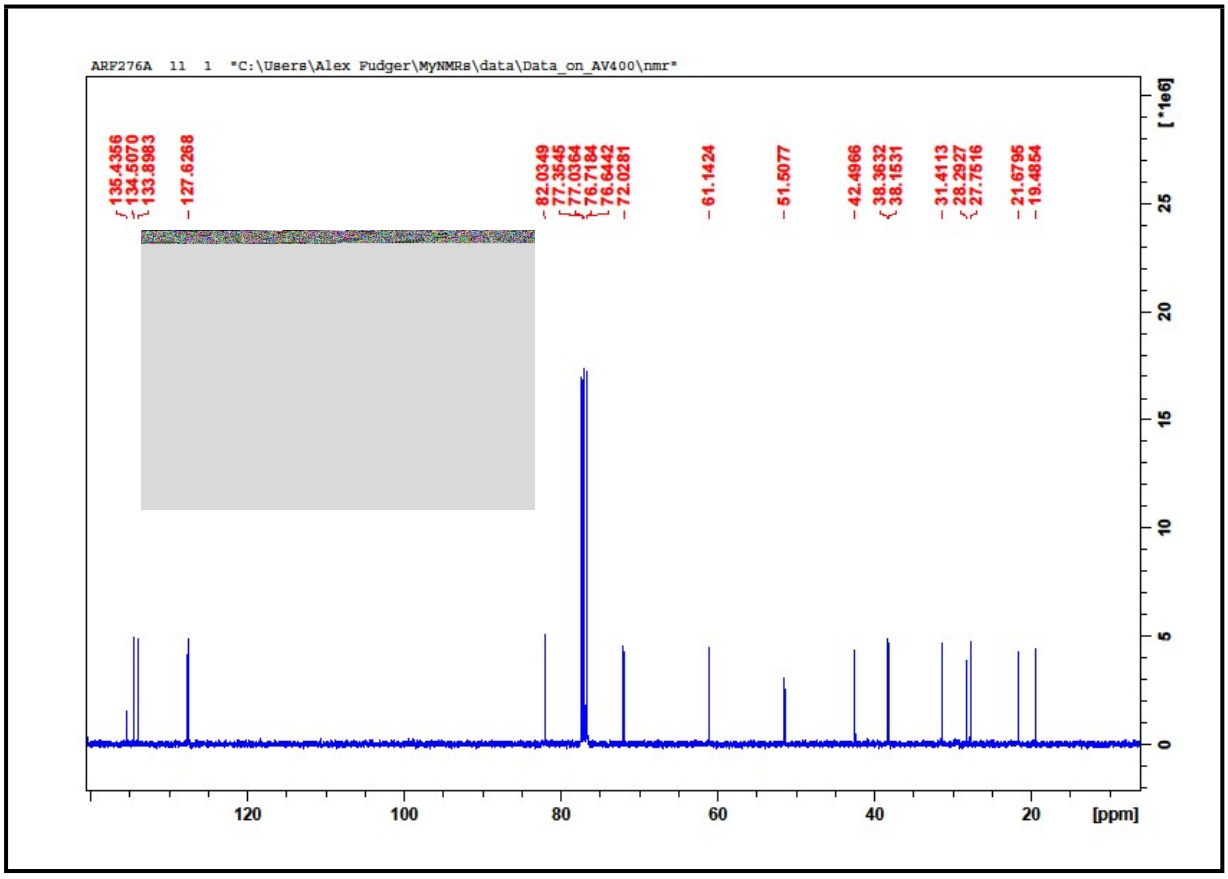
1.0744
1.1209
13.5341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[*1e6]
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127.6268
82.0349
77.3545
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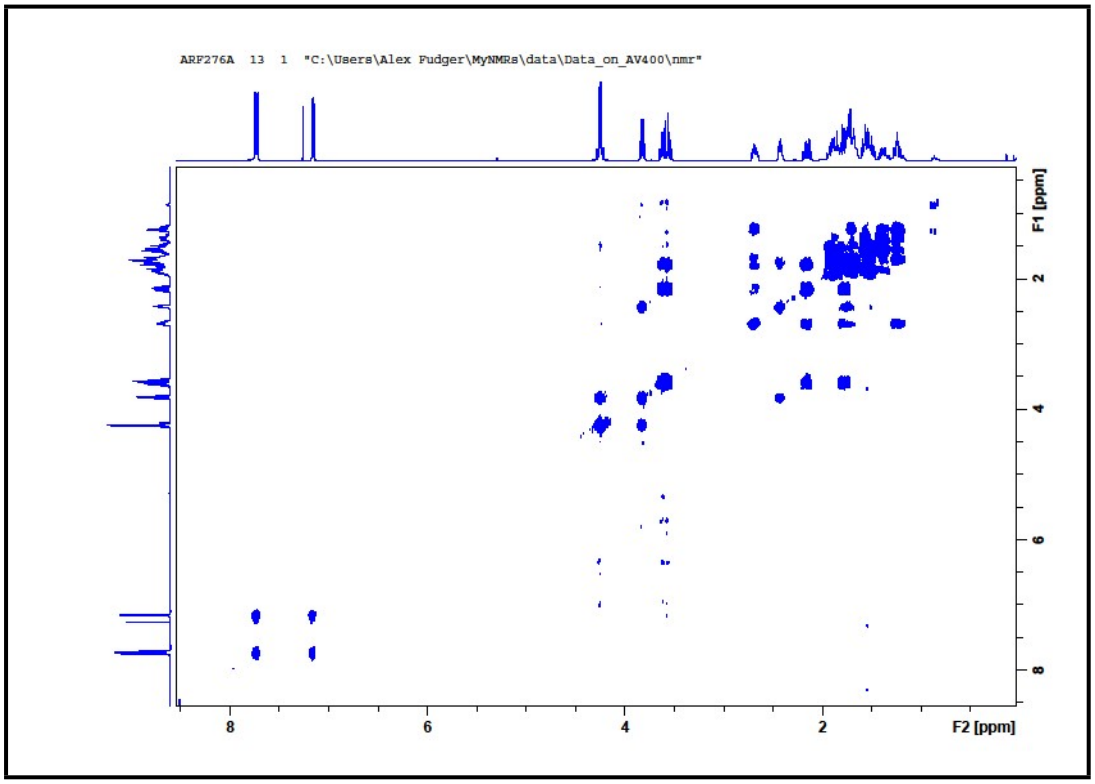
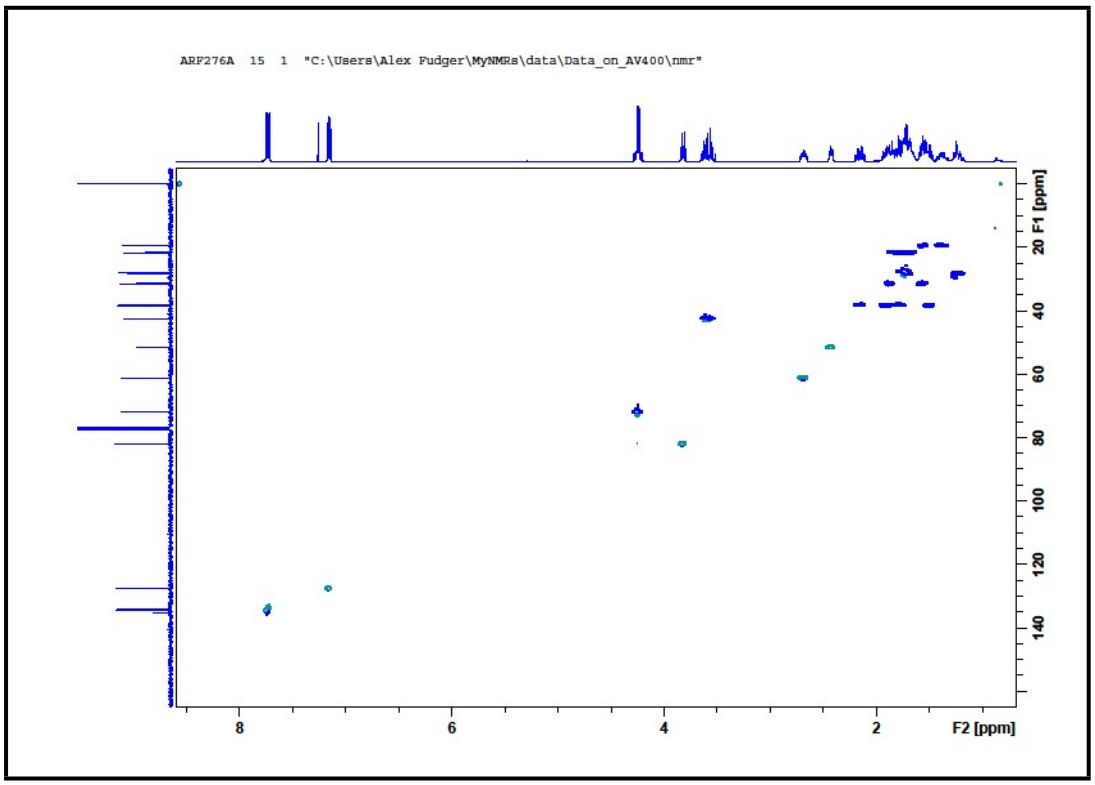
76.7184
76.6442
72.0281
61.1424
51.5077
42.4966
38.3632
38.1531
31.4113
28.2927
27.7516
21.6795
19.4854

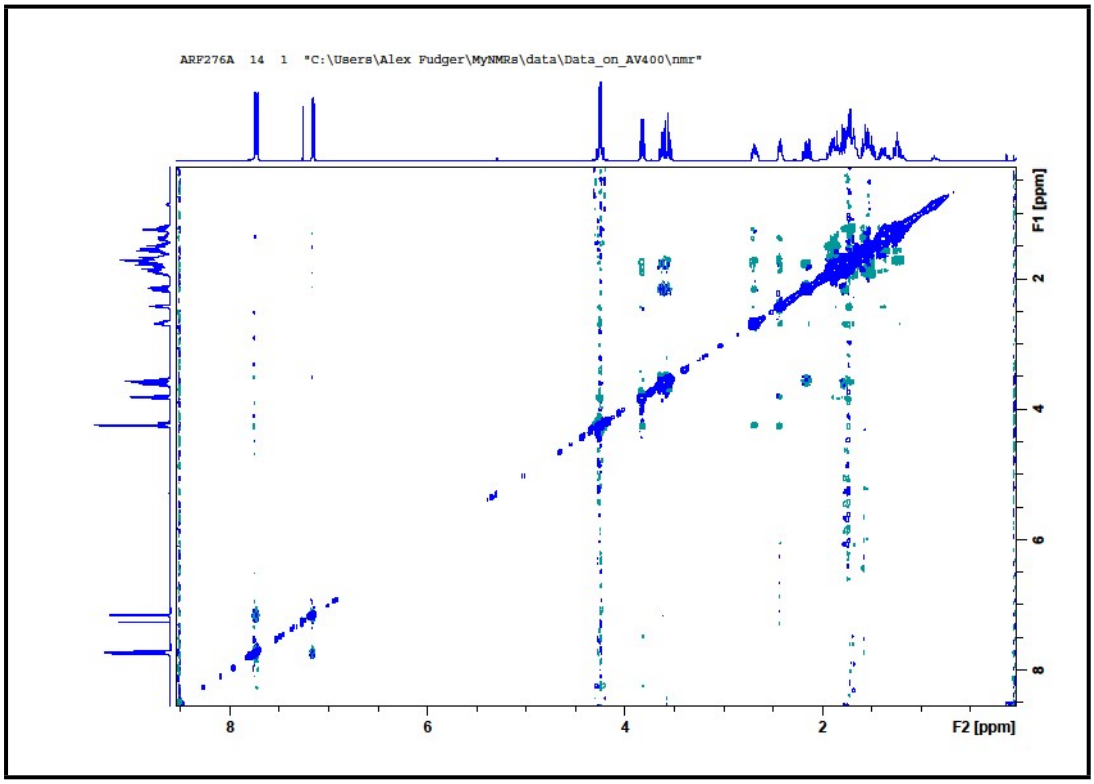
COSY NMR spectrum for compound **17gb**

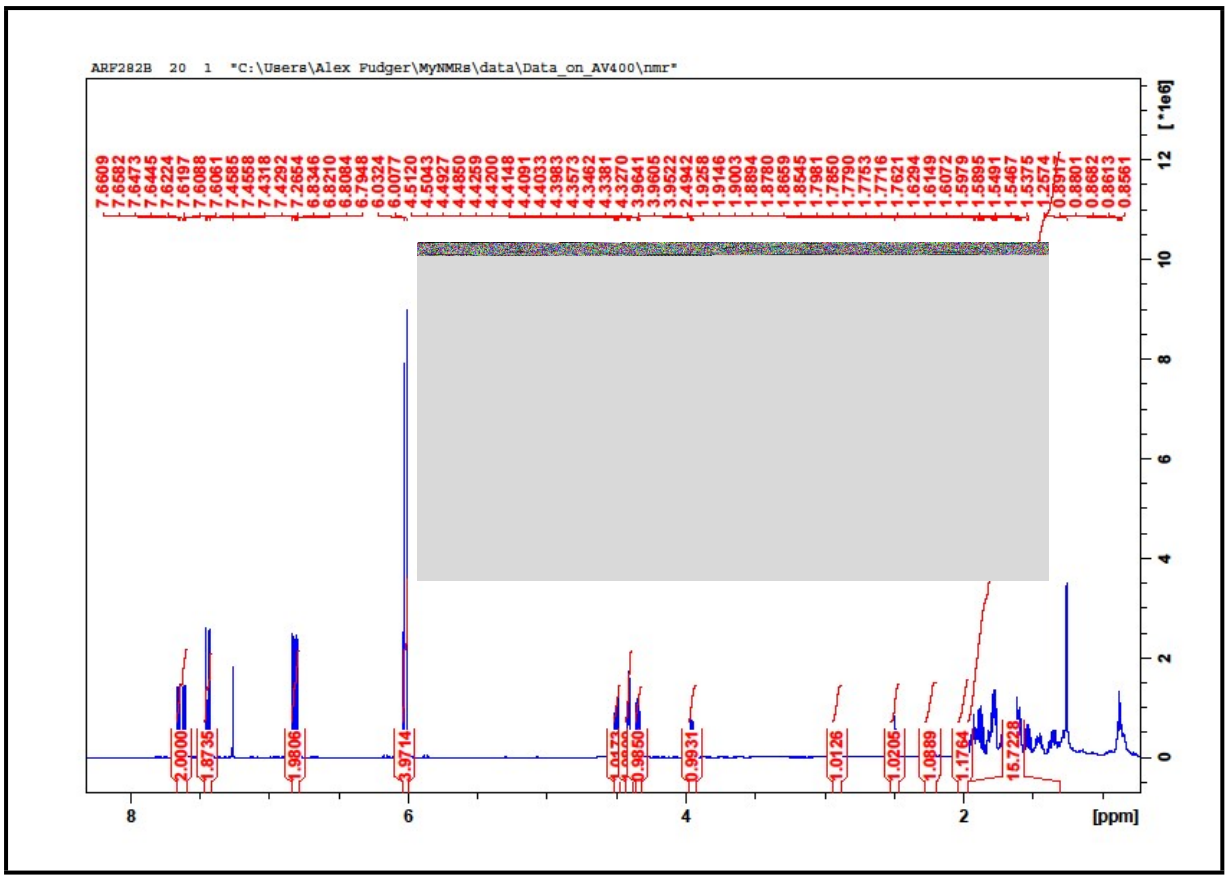


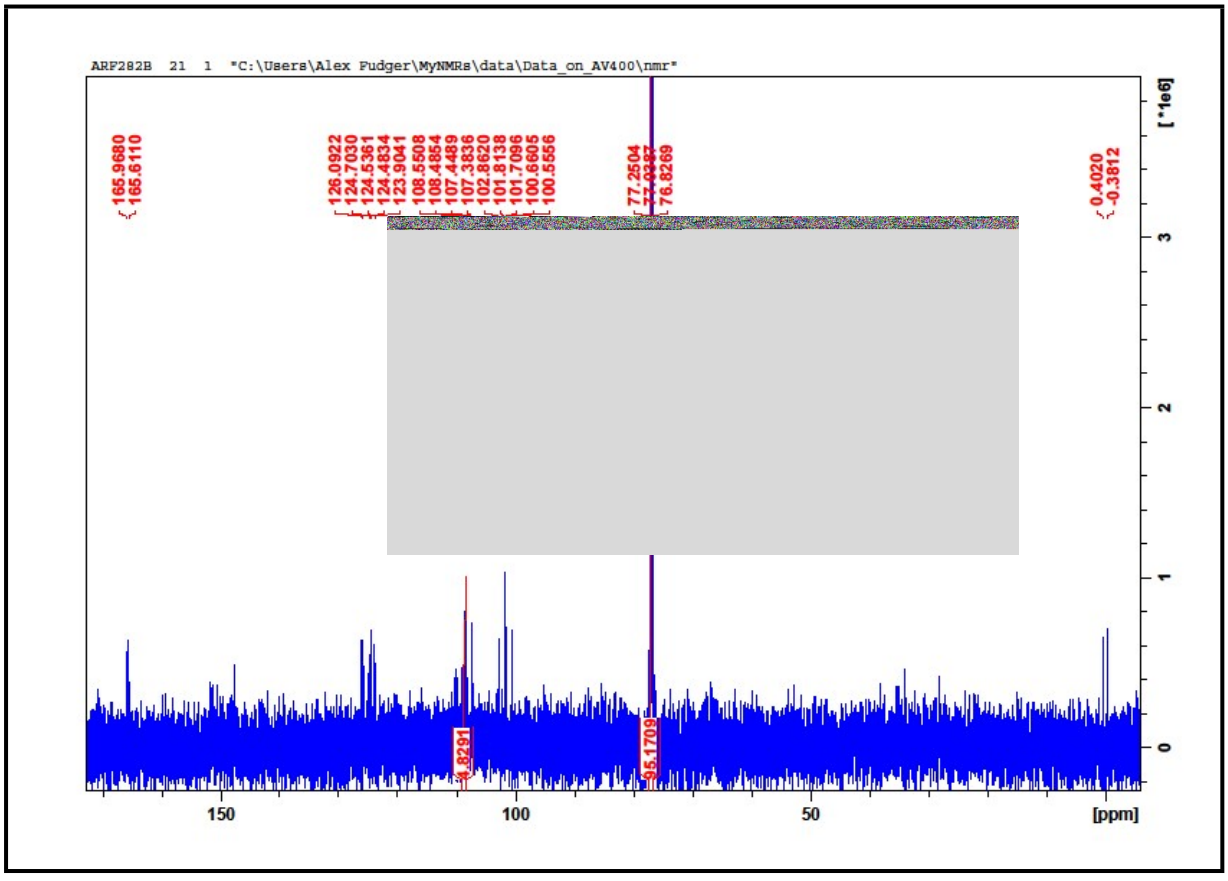

HSQC NMR spectrum for compound **17gb**



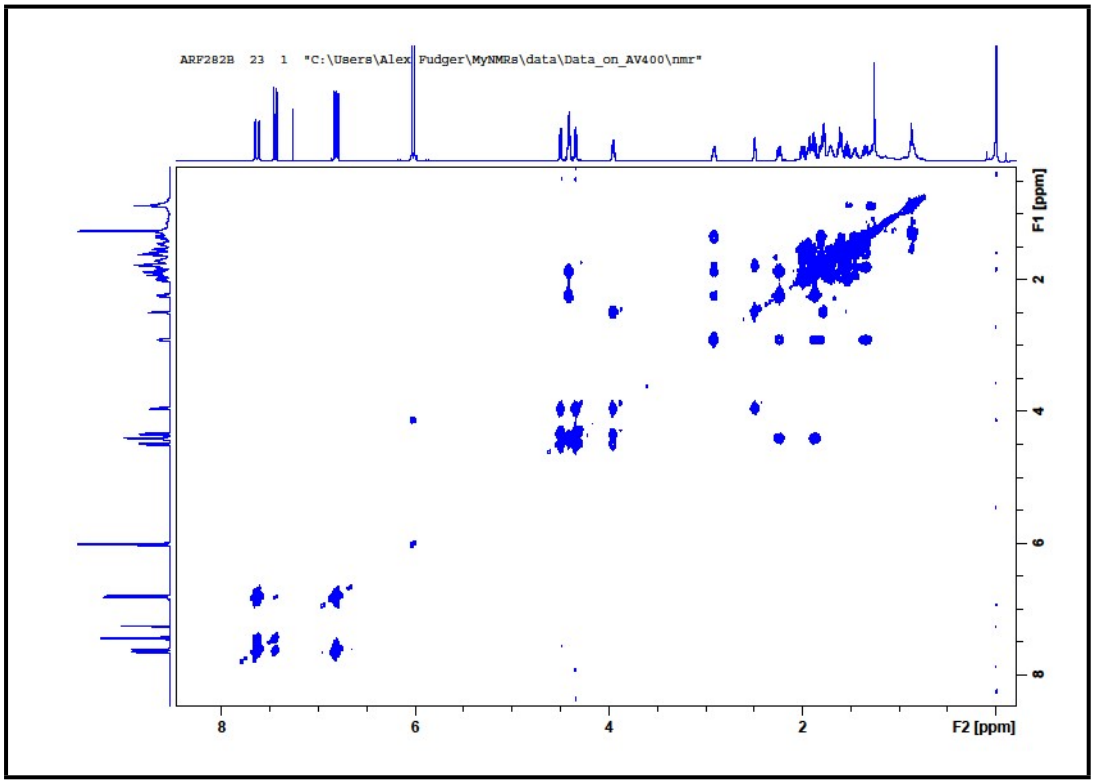
NOESY NMR spectrum for compound **17gb**



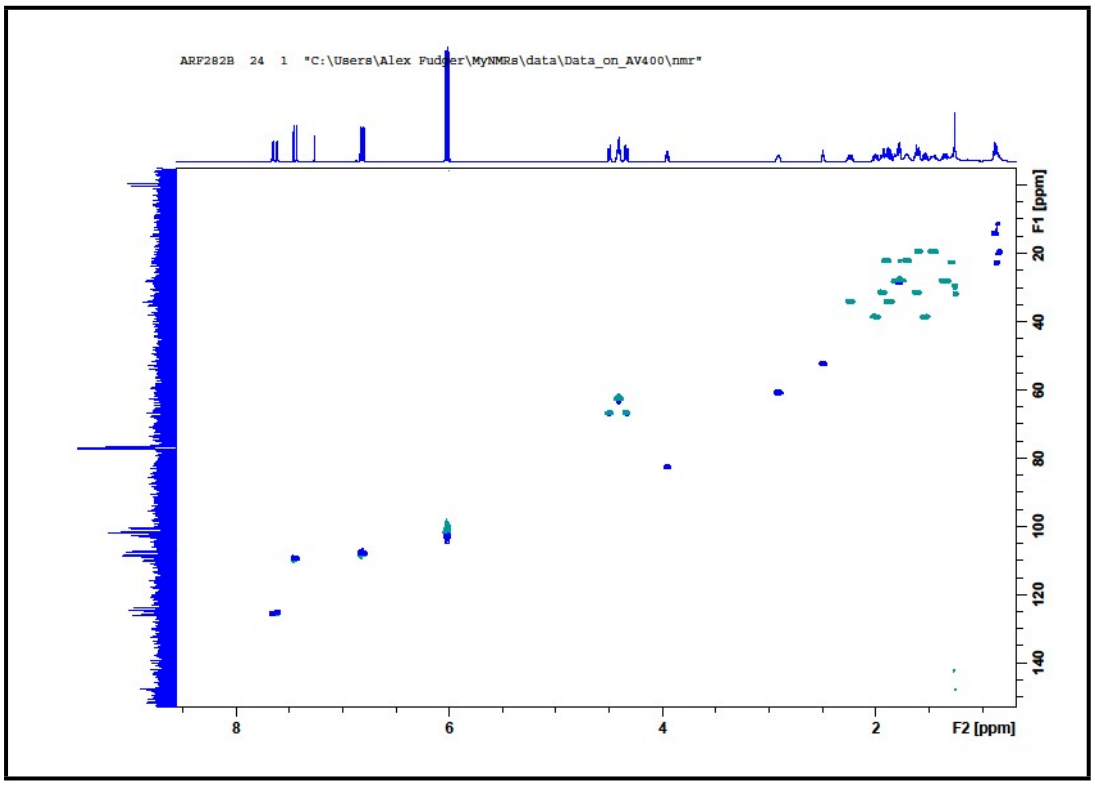

ARF282B 20 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
[*1e6]
[ppm]


ARF282B 21 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
[*1e6]
[ppm]

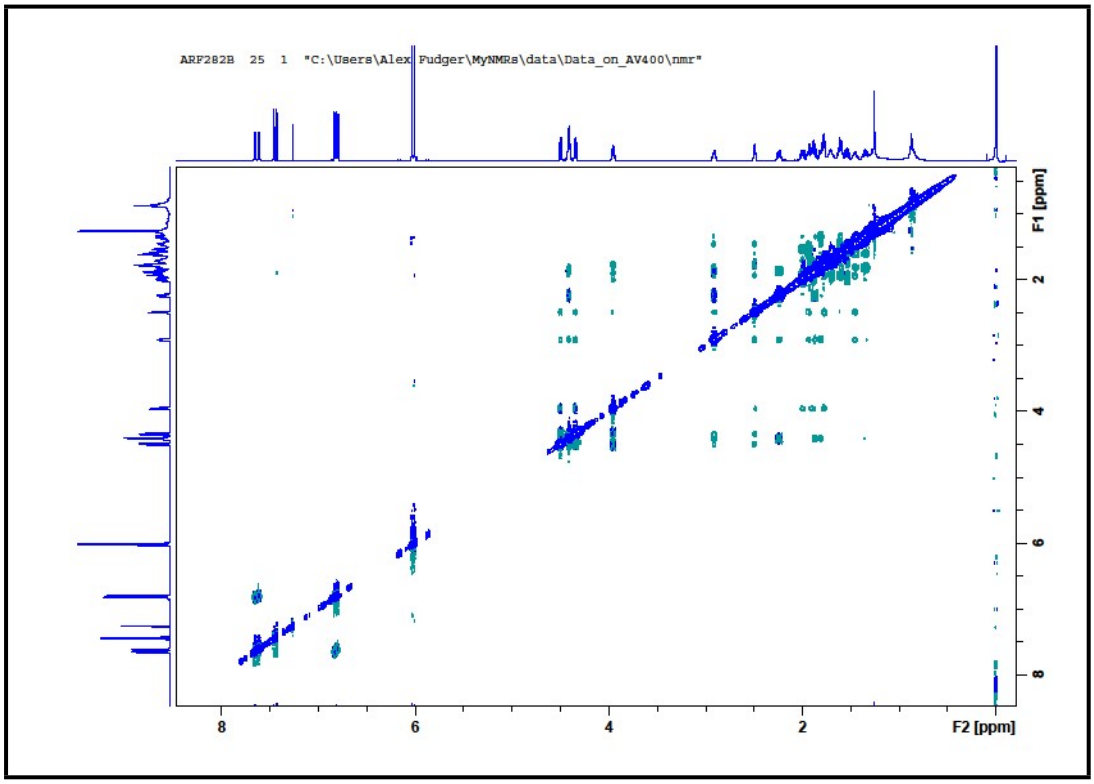
COSY spectrum for compound **17h**

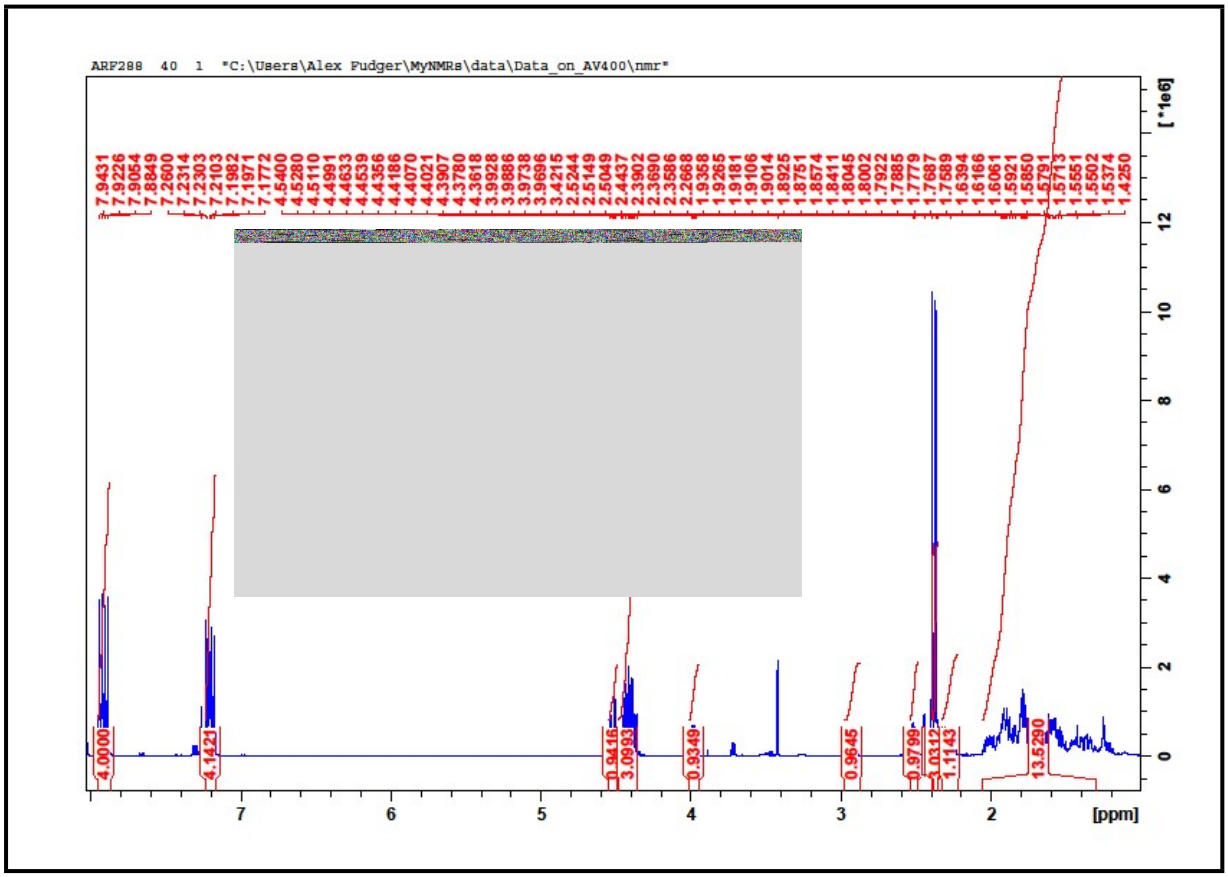


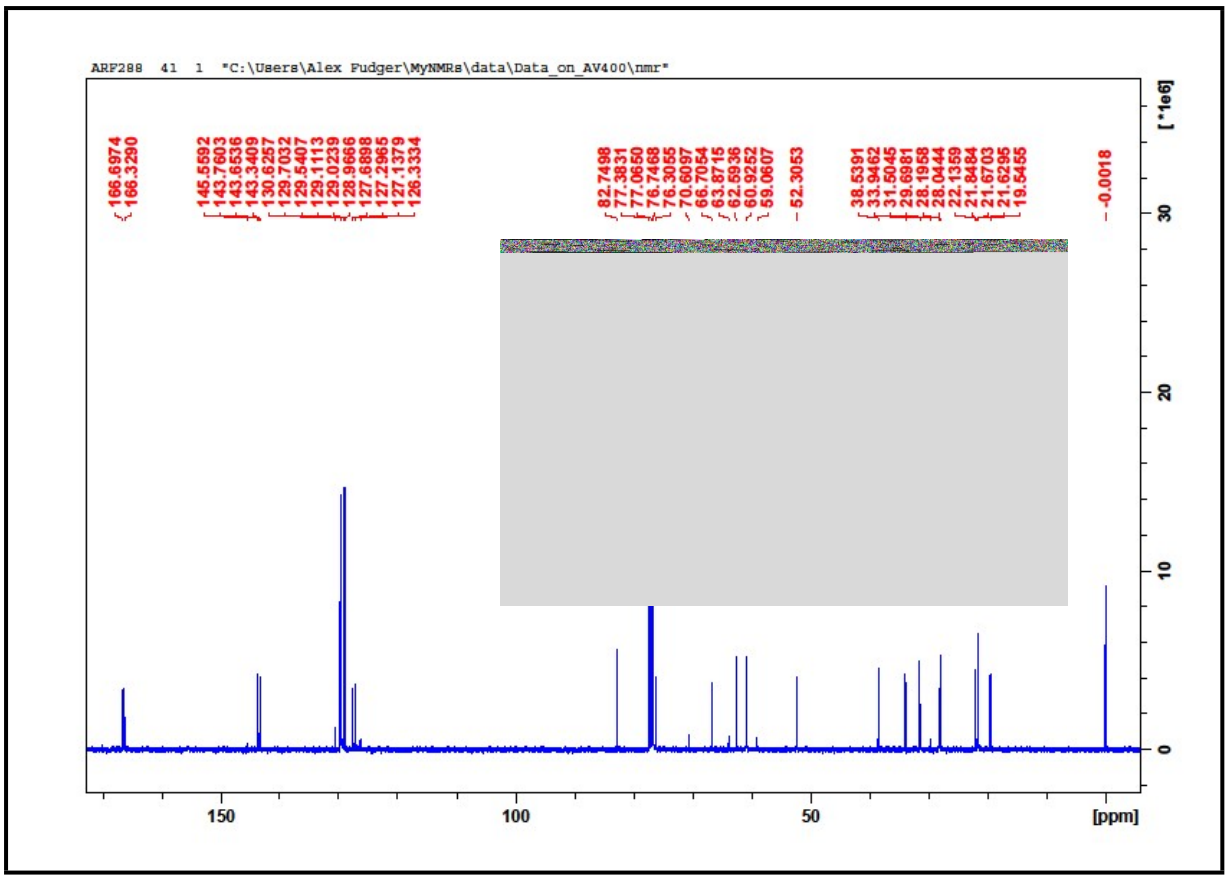

HSQC NMR spectrum for compound **17h**



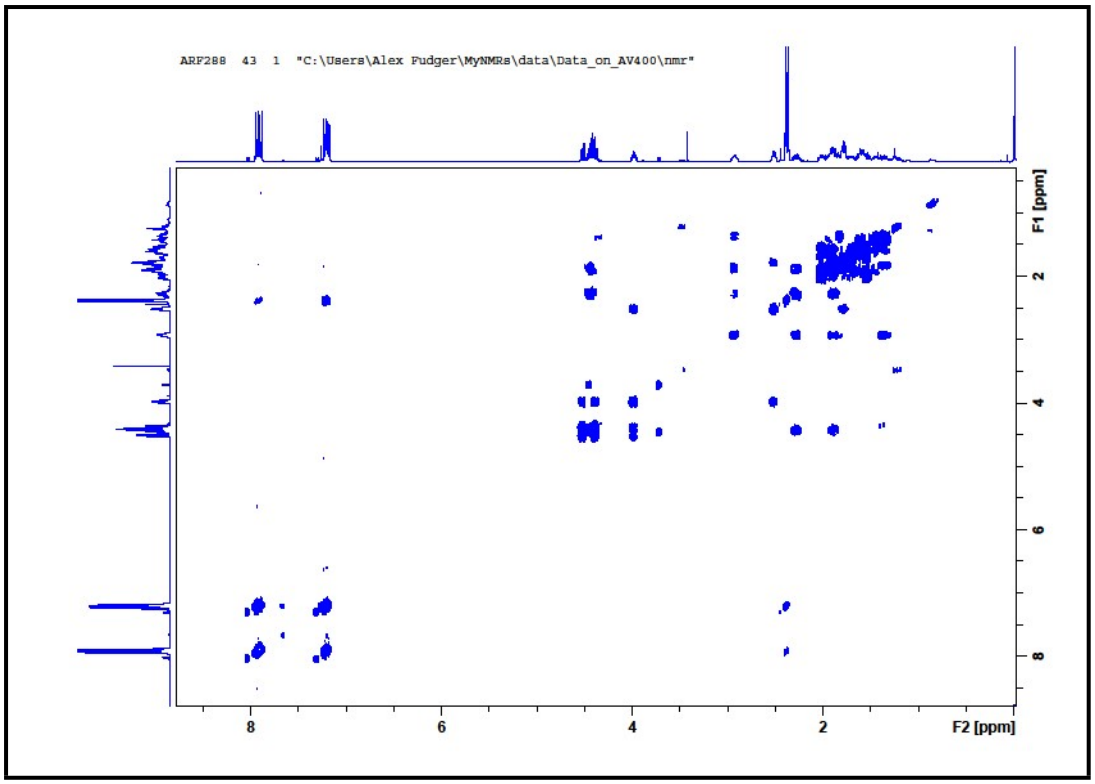
NOESY NMR spectrum for compound **17h**



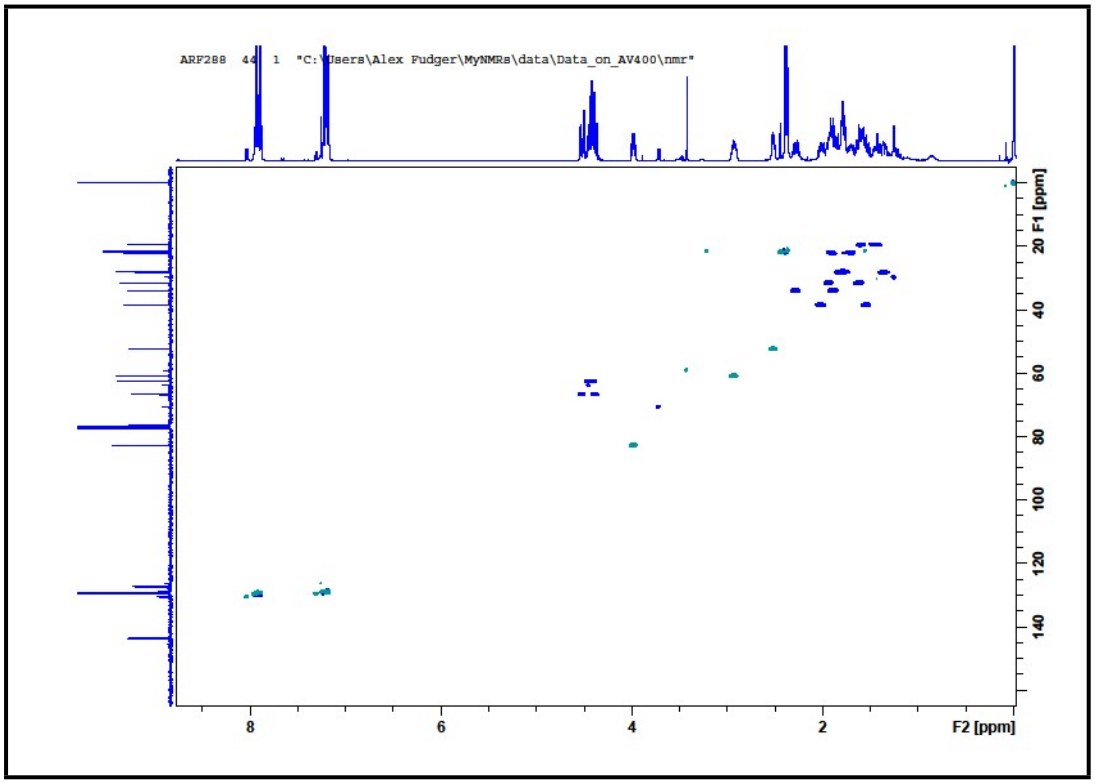

ARF288 40 1 "C:\Users\Alex Fudger\MyNMRs\data\Data_on_AV400\nmr"
[*1e6]
[ppm]



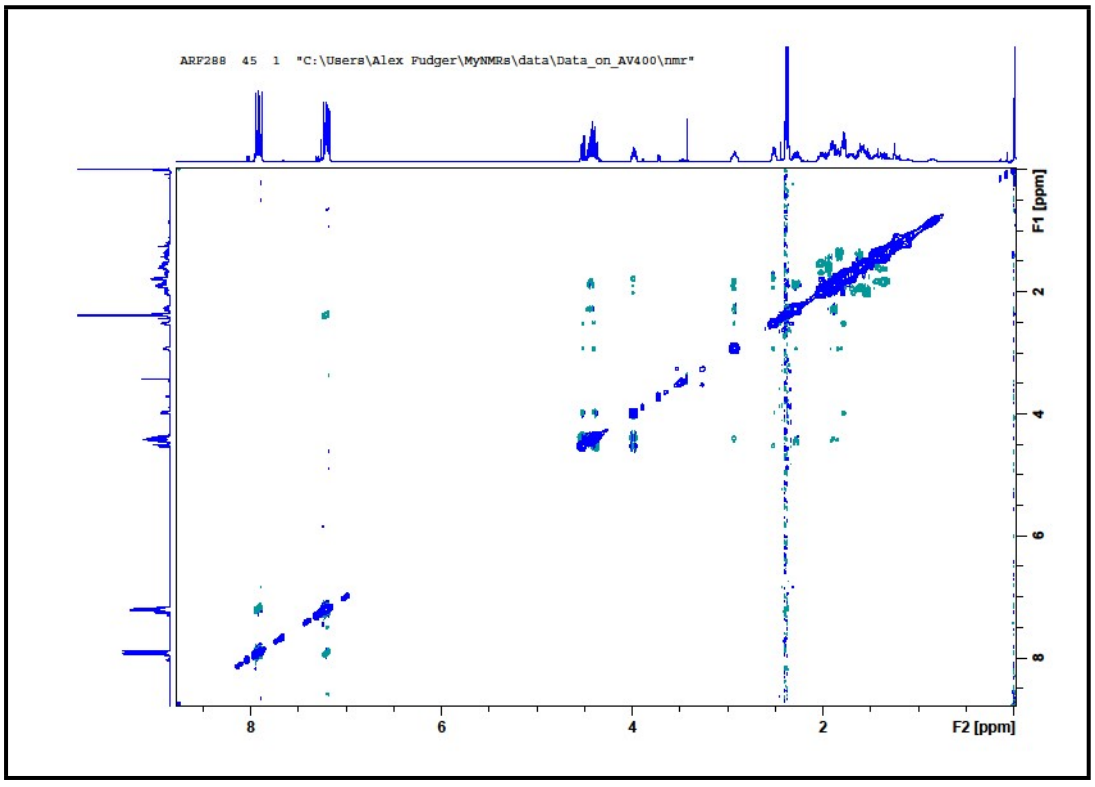

COSY NMR spectrum for compound **17i**

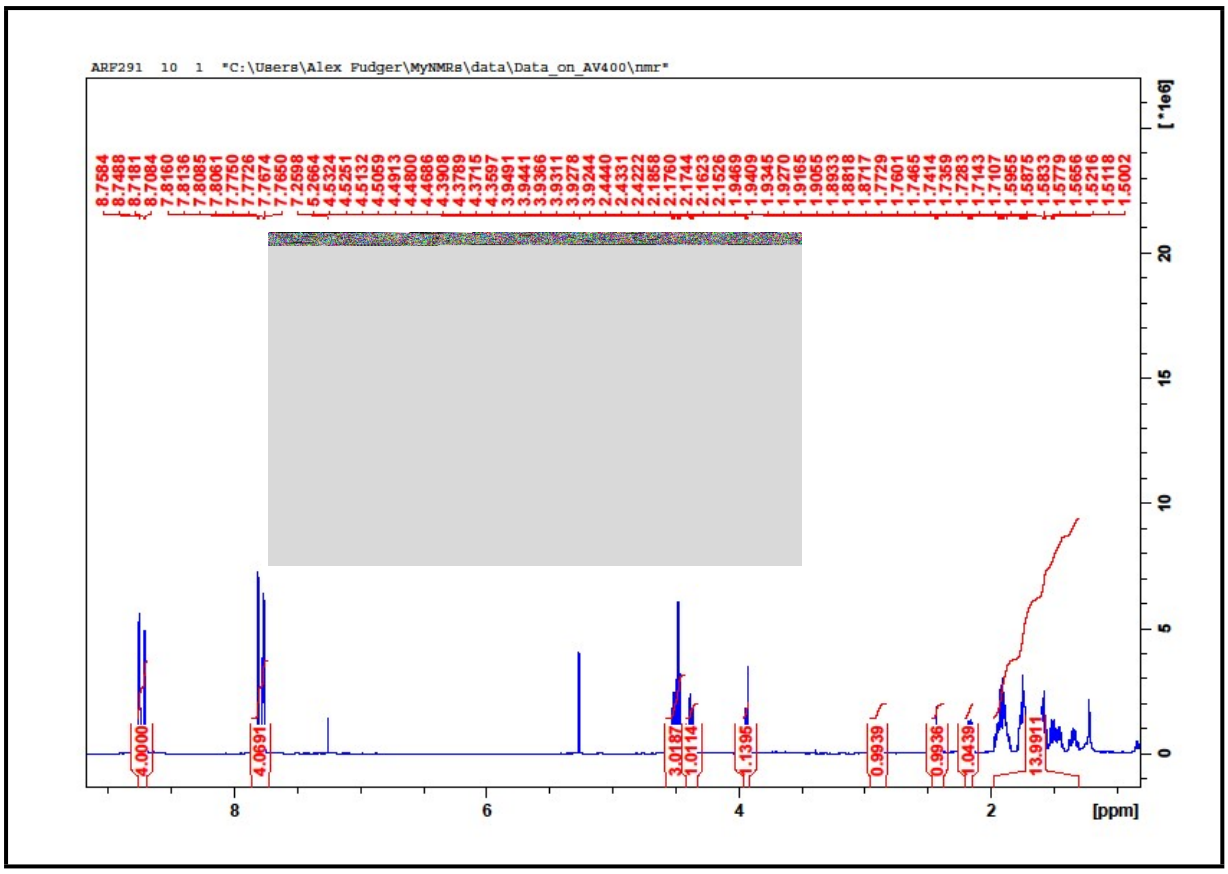


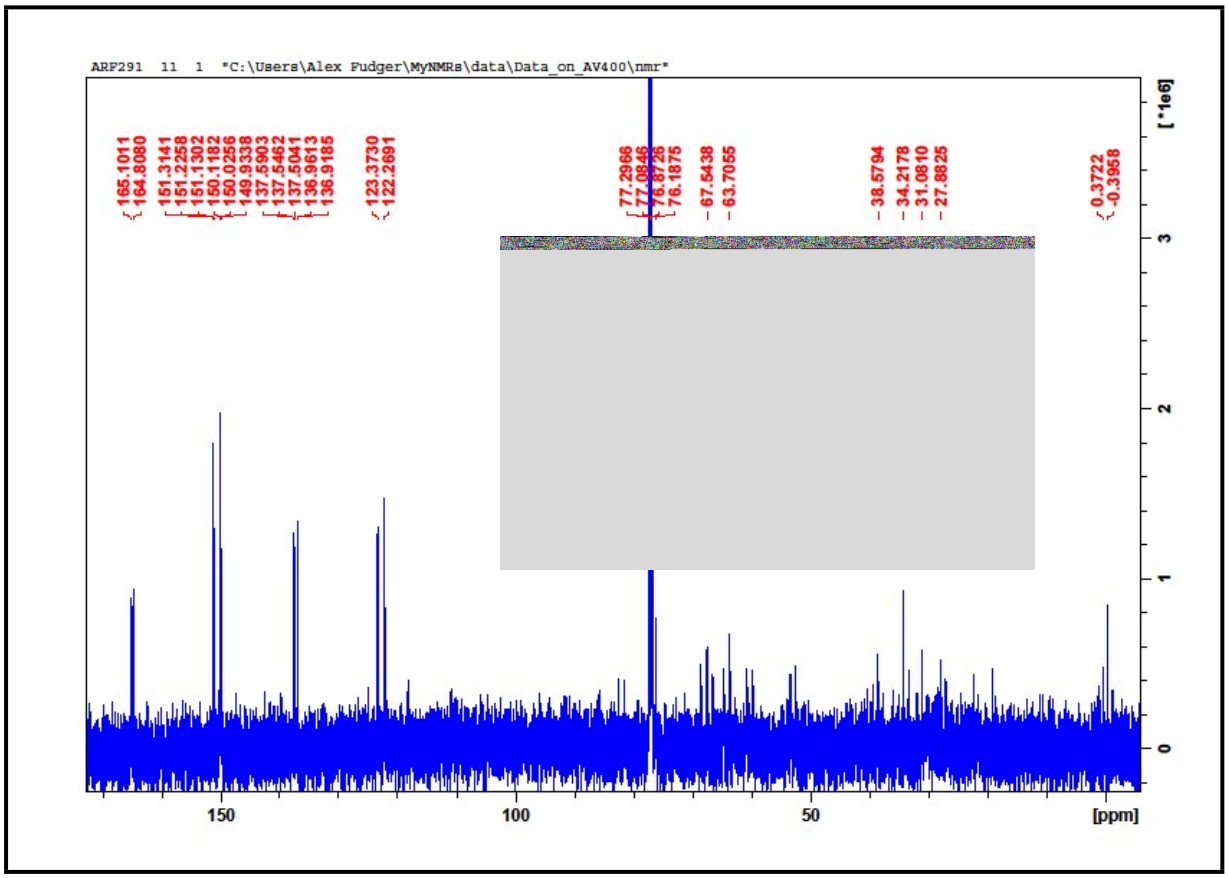
HSQC NMR spectrum for compound **17i**




NOESY NMR spectrum for compound **17i**




ARF291 10 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
8.7584
8.7488
8.7181
8.7084
7.8160
7.8136
7.8085
7.8061
7.7760
7.7726
7.7674
7.7650
7.2598
5.2664
4.5324
4.5251
4.5132
4.5059
4.4913
4.4800
4.4686
4.3908
4.3789
4.3715
4.3597
3.9491
3.9441
3.9366
3.9311
3.9278
3.9244
2.4440
2.4331
2.4222
2.1858
2.1760
2.1744
2.1623
2.1526
1.9469
1.9409
1.9345
1.9270
1.9165
1.9055
1.8833
1.8818
1.8717
1.7729
1.7601
1.7465
1.7414
1.7359
1.7283
1.7143
1.7107
1.5955
1.5875
1.5833
1.5779
1.5656
1.5216
1.5118
1.5002
4.0000
4.0691
3.0187
1.0114
1.1395
0.9939
0.9936
1.0439
13.9911
[*1e6]
[ppm]


ARF291 11 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
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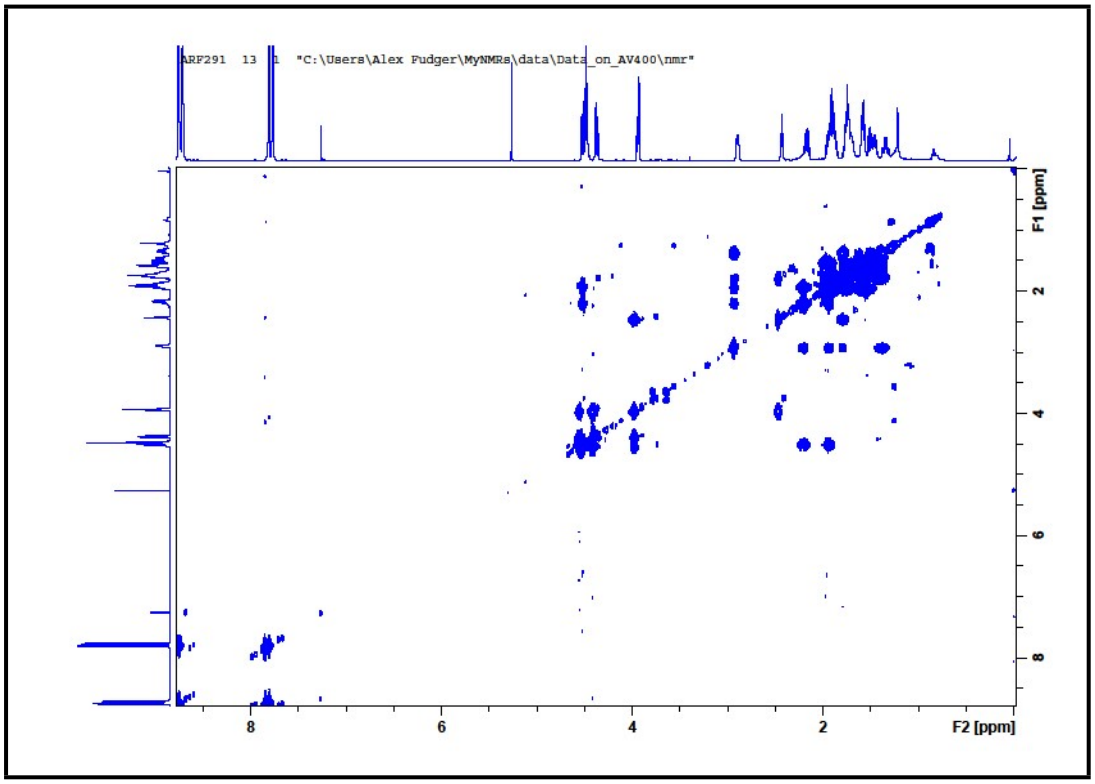
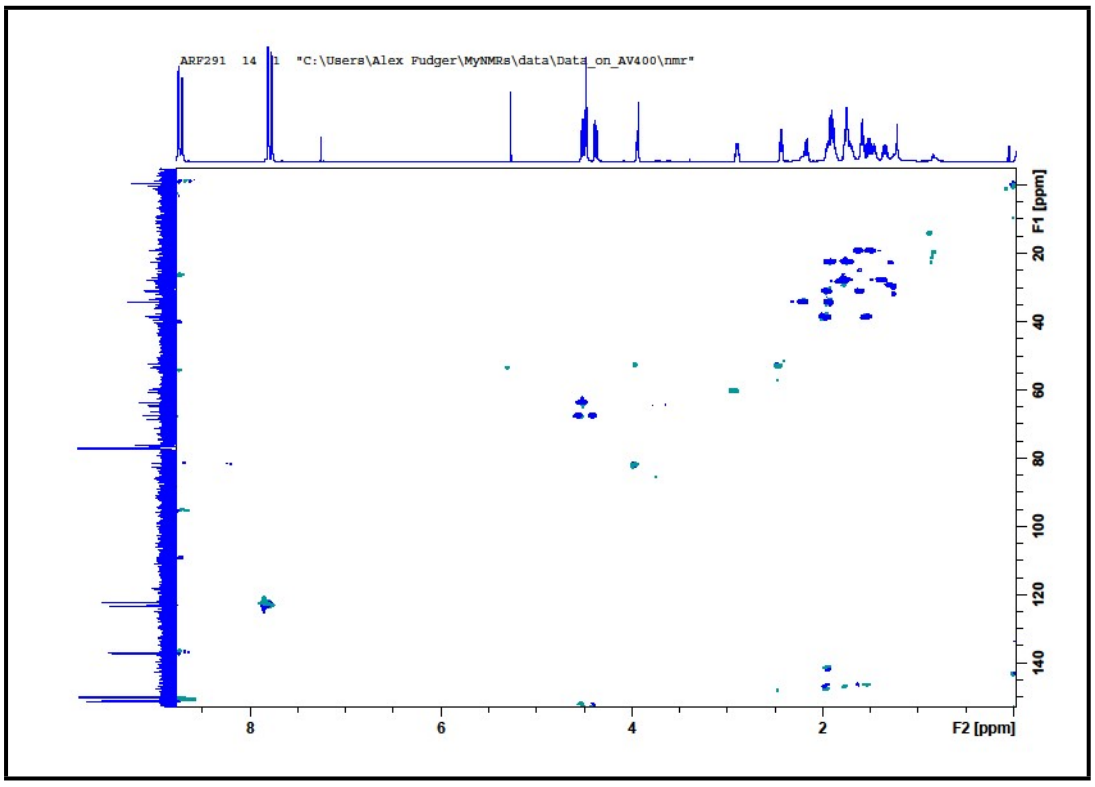
164.8080
151.3141
151.2258
151.1302
150.1182
150.0256
149.9338
137.5903
137.5462
137.5041
136.9613
136.9185
123.3730
122.2691
77.2966
77.0846
76.8726
76.1875
67.5438
63.7055
38.5794
34.2178
31.0810
27.8825
0.3722
-0.3958
[*1e6]
[ppm]

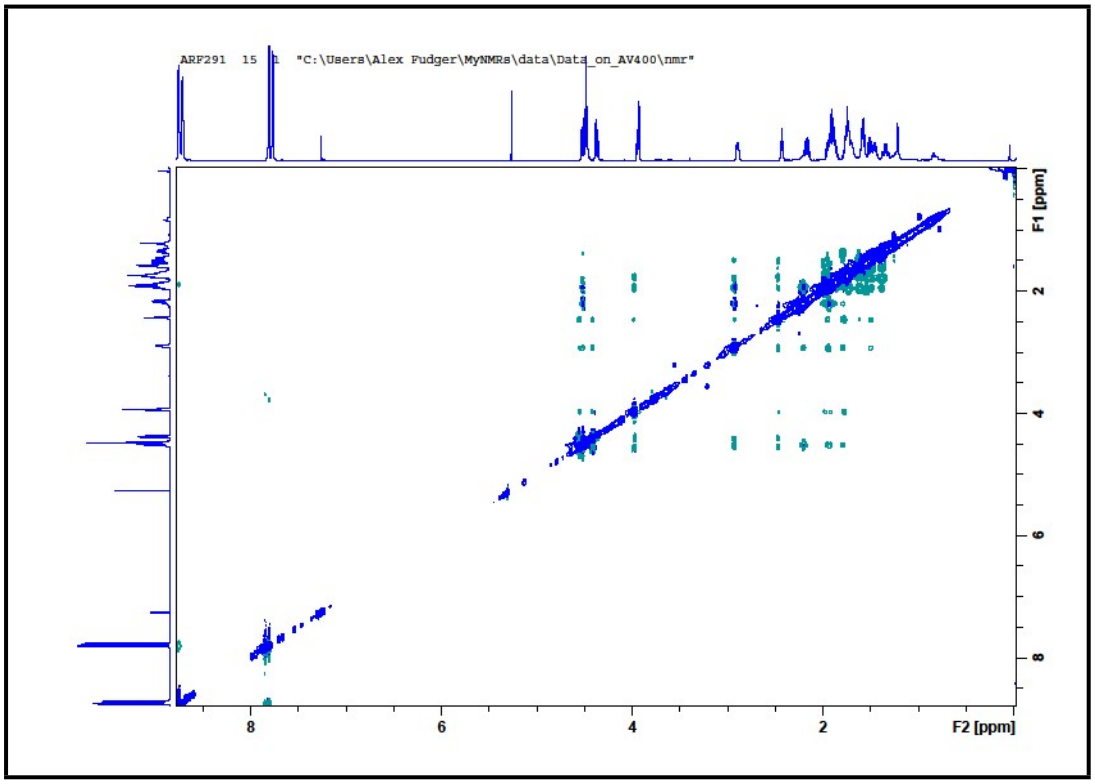
COSY NMR spectrum for compound **17j**

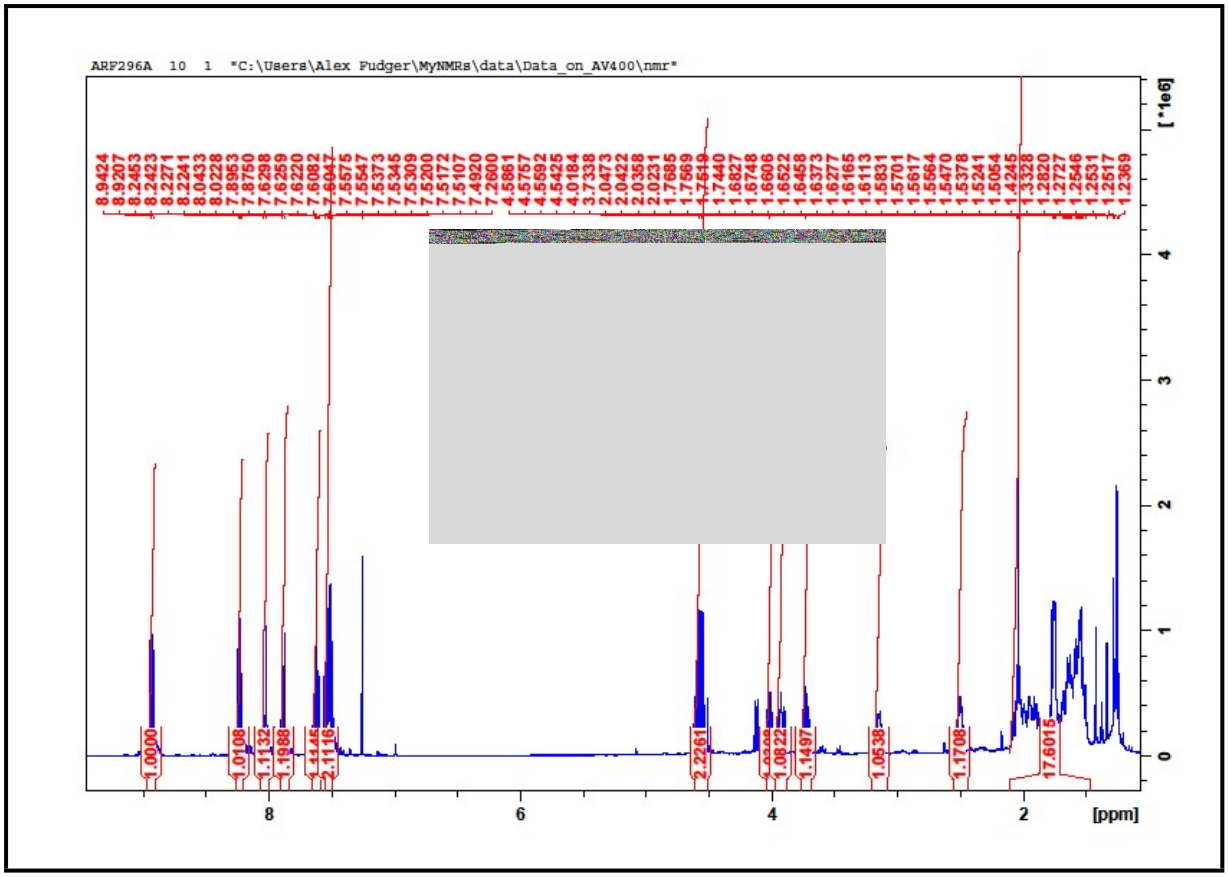



HSQC NMR spectrum for compound **17j**



NOESY NMR spectrum for compound **(242)**




ARF296A 10 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
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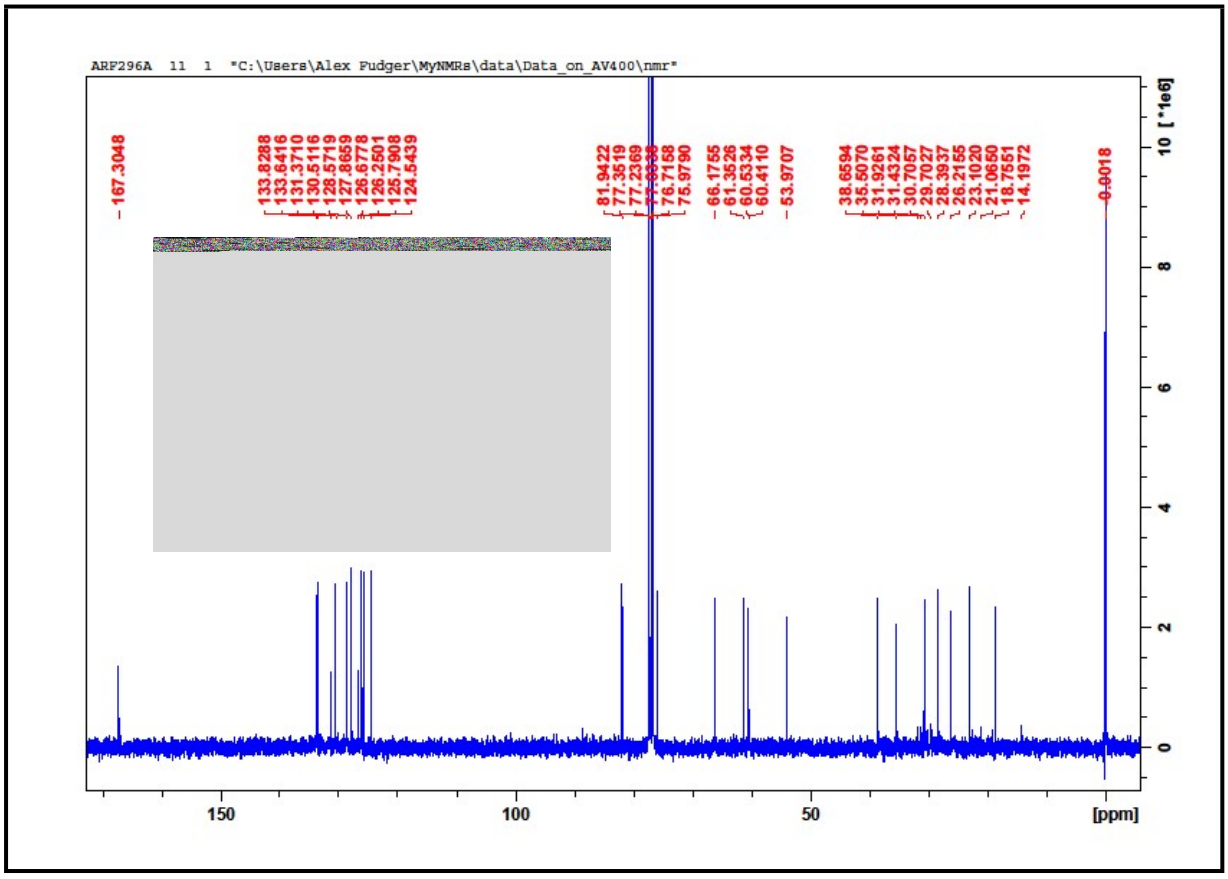
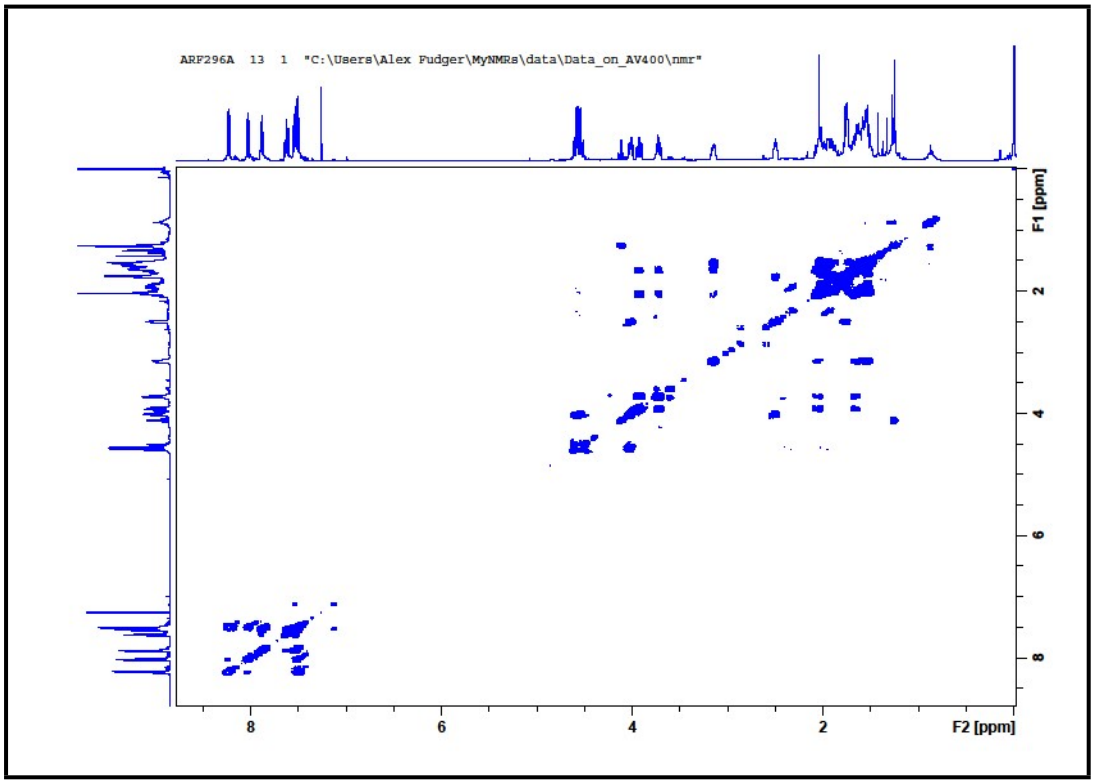
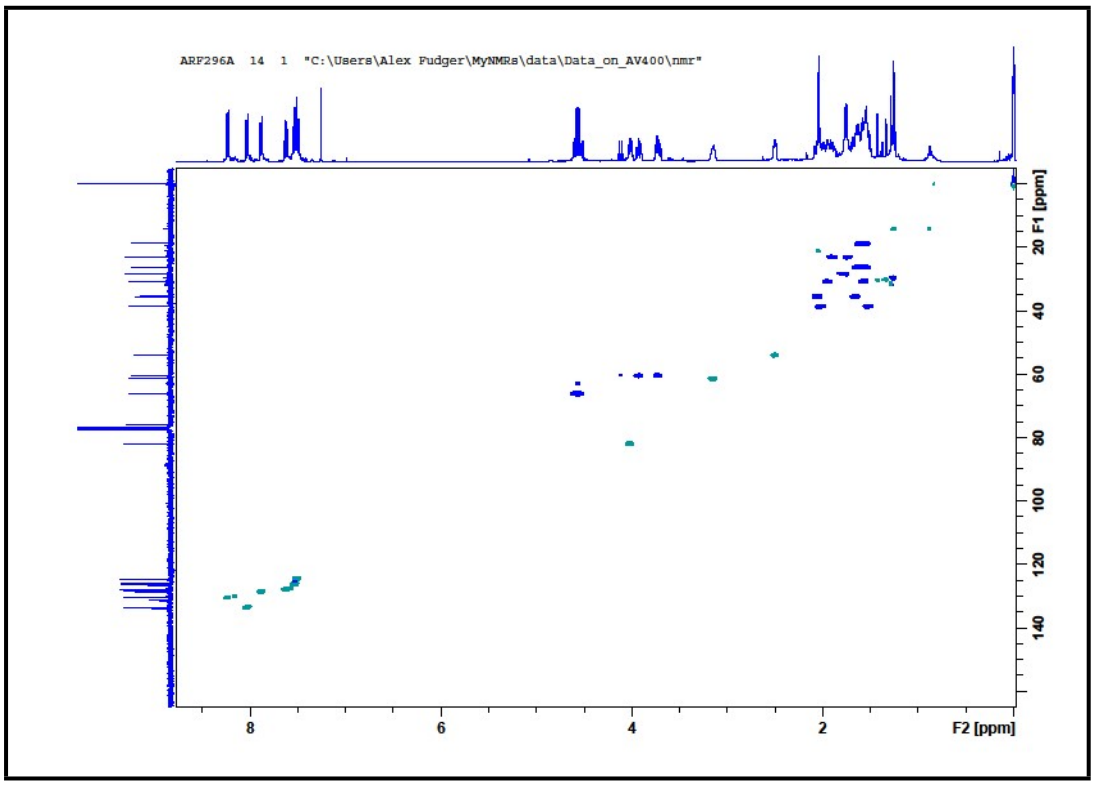
8.9207
8.2463
8.2423
8.2271
8.2241
8.0433
8.0228
7.8963
7.8760
7.6298
7.6259
7.6220
7.6082
7.6047
7.5575
7.5547
7.5373
7.5345
7.5309
7.5200
7.5172
7.5107
7.4920
7.2600
4.5861
4.5757
4.5592
4.5425
4.0184
3.7338
2.0473
2.0422
2.0358
2.0231
1.7685
1.7569
1.7619
1.7440
1.6827
1.6748
1.6606
1.6522
1.6458
1.6373
1.6277
1.6165
1.6113
1.5831
1.5701
1.5617
1.5564
1.5470
1.5378
1.5241
1.5054
1.4245
1.3328
1.2820
1.2727
1.2646
1.2531
1.2517
1.2369
1.0000
1.0108
1.1132
1.1988
1.1445
2.1116
2.2261
1.0822
1.1497
1.0538
1.1708
17.6015
8
6
4
2
[ppm]
[*1e6]


ARF296A 11 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
167.3048
133.8288
133.6416
131.3710
130.5116
128.5719
127.8659
126.6778
126.2501
125.7908
124.5439
81.9422
77.3519
77.2369
76.7158
75.9790
66.1755
61.3526
60.5334
60.4110
53.9707
38.6594
35.5070
31.9261
31.4324
30.7057
29.7027
28.3937
26.2155
23.1020
21.0650
18.7551
14.1972
0.0018
150
100
50
[ppm]
[*1e6]

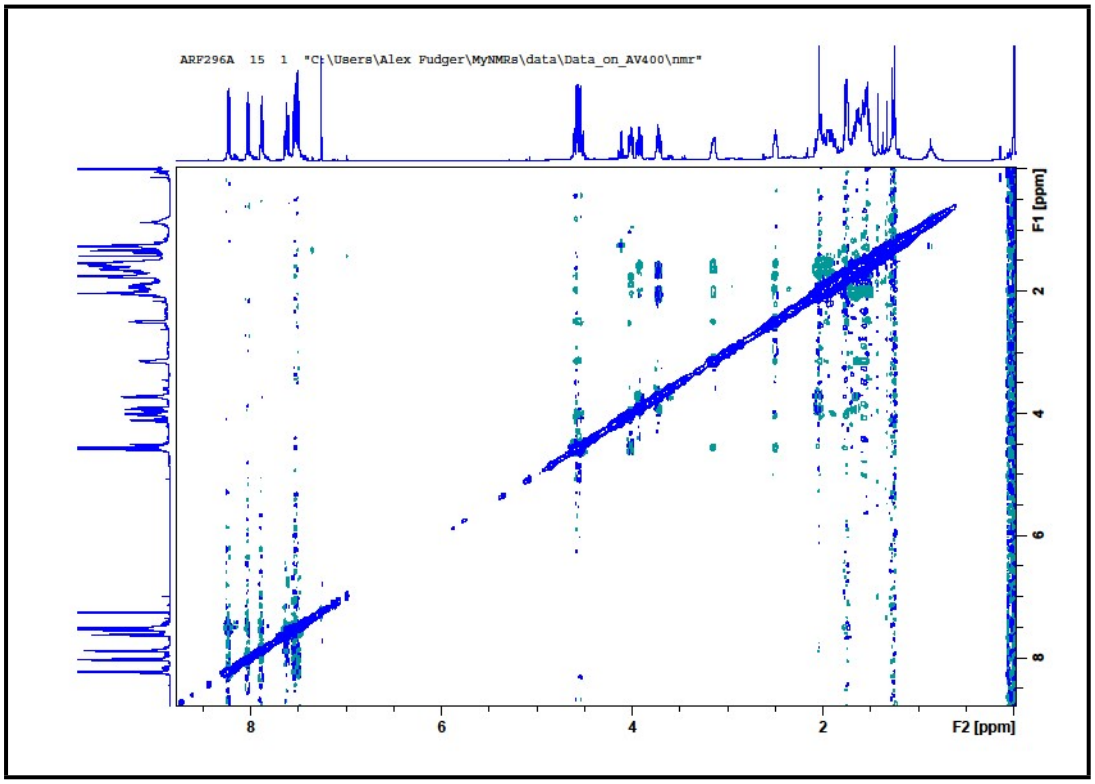
COSY NMR spectrum for compound **17ka**

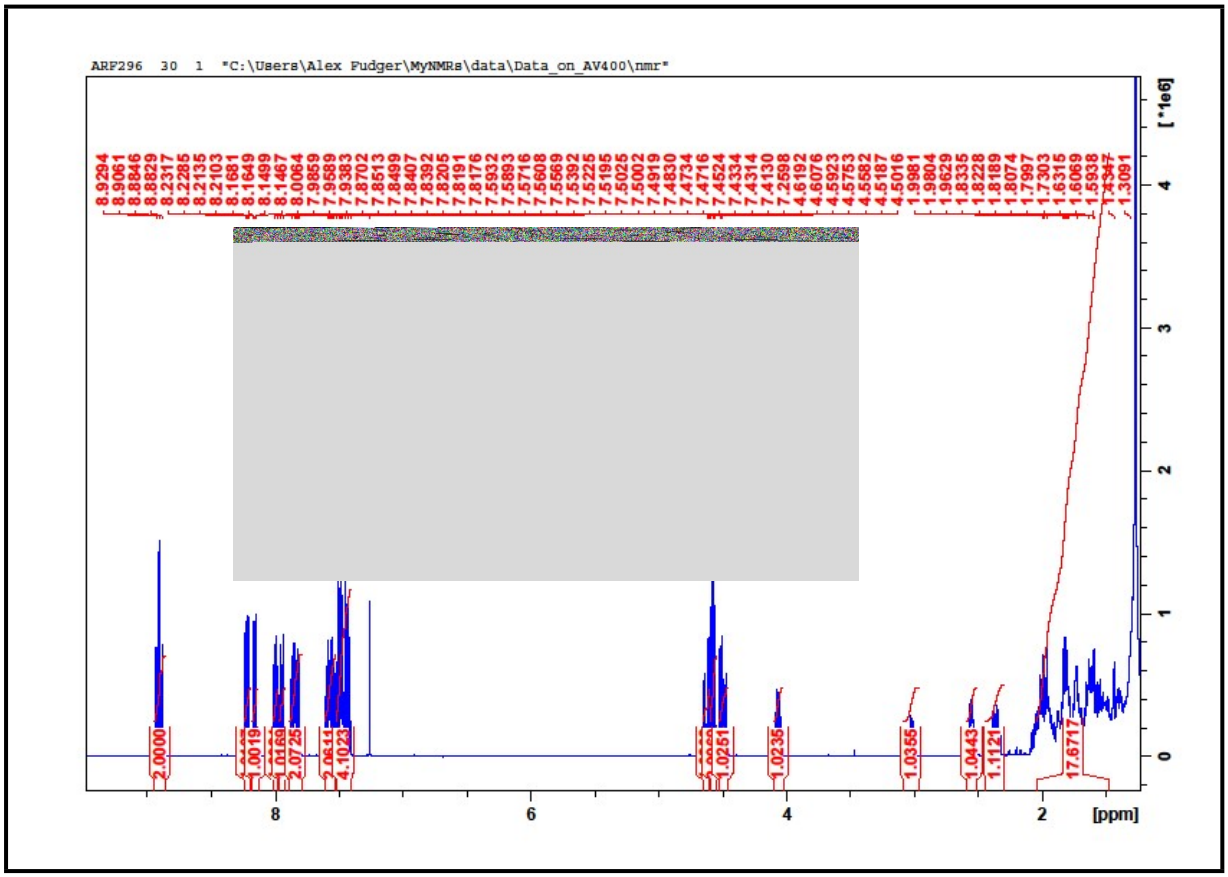


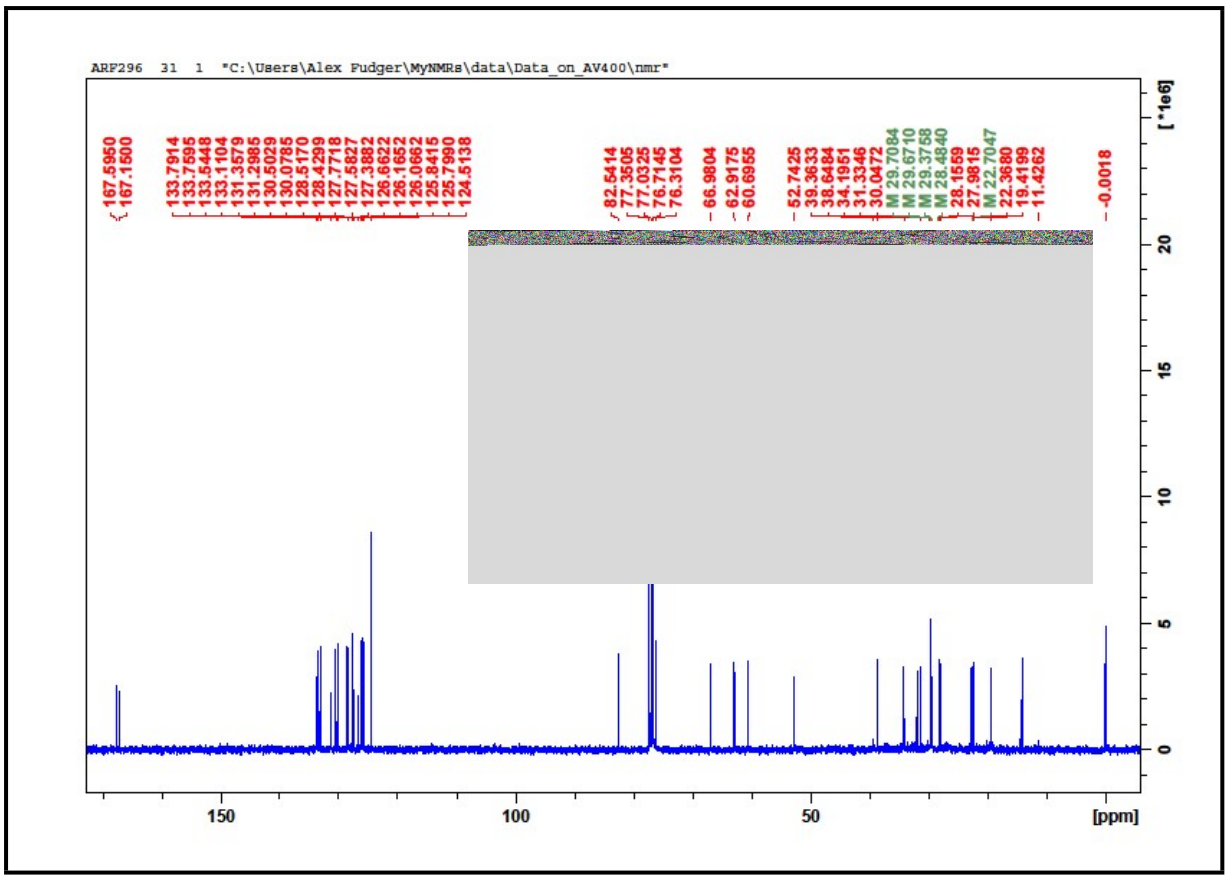

HSQC NMR spectrum for compound **17ka**



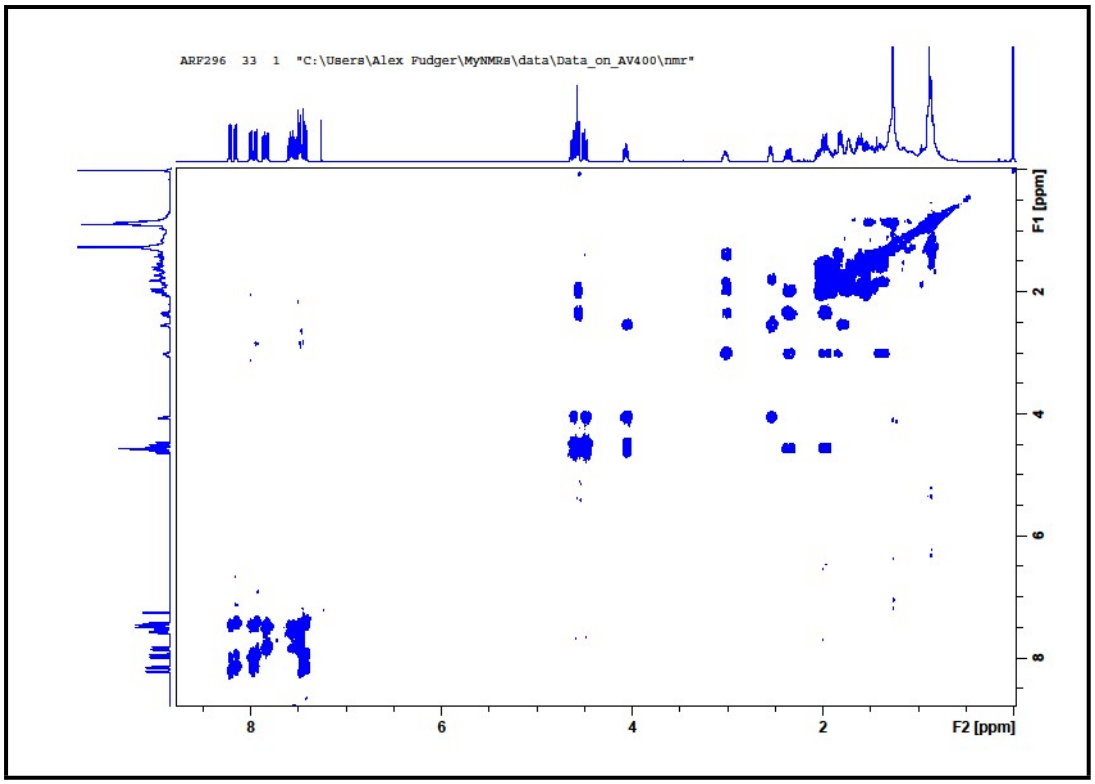
NOESY NMR spectrum for compound **17ka**



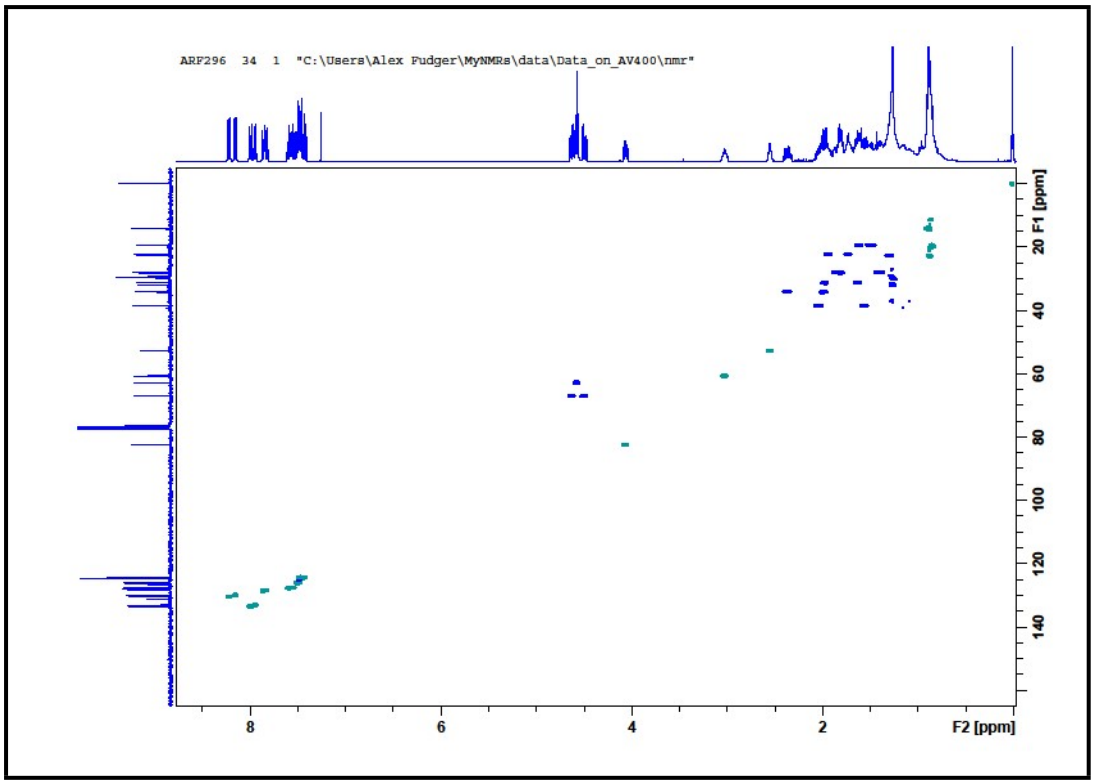

ARF296 30 1 "C:\Users\Alex Fudger\MyNMRs\data\Data_on_AV400\nmr"
8.9294
8.9061
8.8846
8.8829
8.2317
8.2285
8.2135
8.2103
8.1681
8.1649
8.1499
8.1467
8.0064
7.9859
7.9589
7.9383
7.8702
7.8513
7.8499
7.8407
7.8392
7.8205
7.8191
7.8176
7.5932
7.5893
7.5716
7.5608
7.5569
7.5392
7.5225
7.5195
7.5025
7.5002
7.4919
7.4830
7.4734
7.4716
7.4524
7.4334
7.4314
7.4130
7.2598
4.6192
4.6076
4.5923
4.5753
4.5682
4.5187
4.5016
1.9981
1.9804
1.9629
1.8335
1.8228
1.8189
1.8074
1.7997
1.7303
1.6315
1.6069
1.5938
1.4347
1.3091
2.0000
1.0019
1.0169
2.0725
2.0611
4.1023
1.0235
1.0261
1.0355
1.0443
1.1121
17.6717
8
6
4
2
[ppm]
[*1e6]
0
1
2
3
4



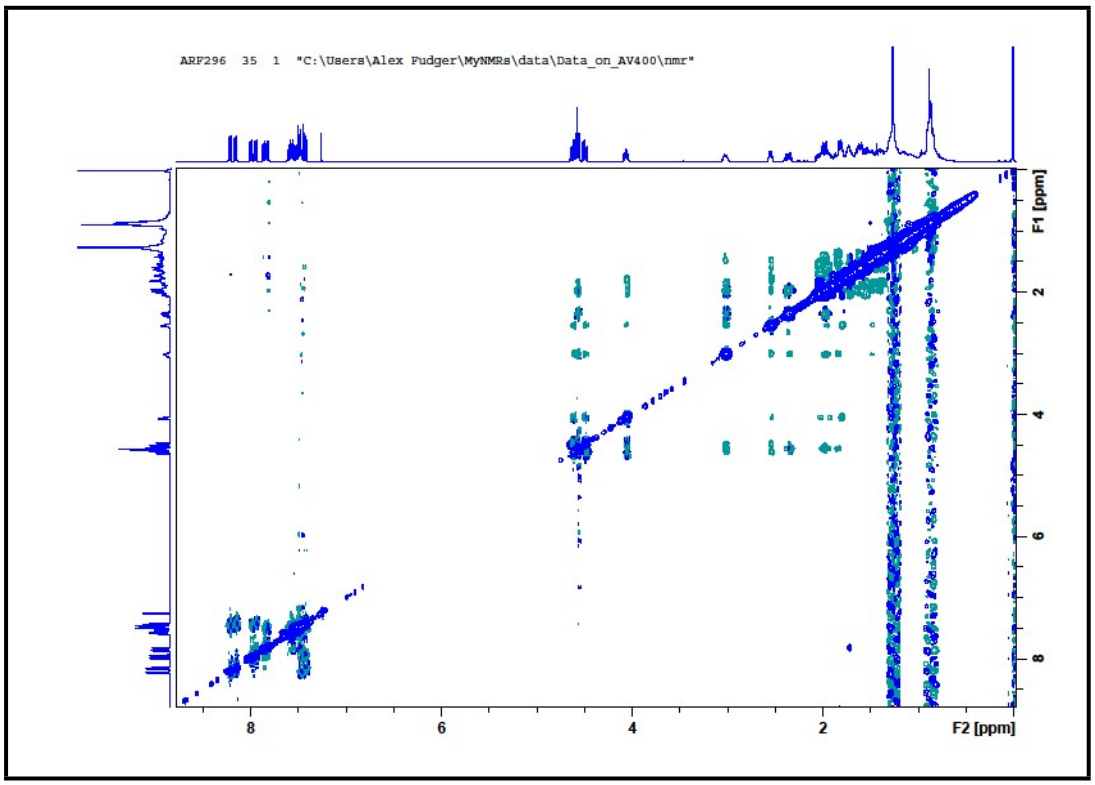

COSY NMR spectrum for compound **17kb**

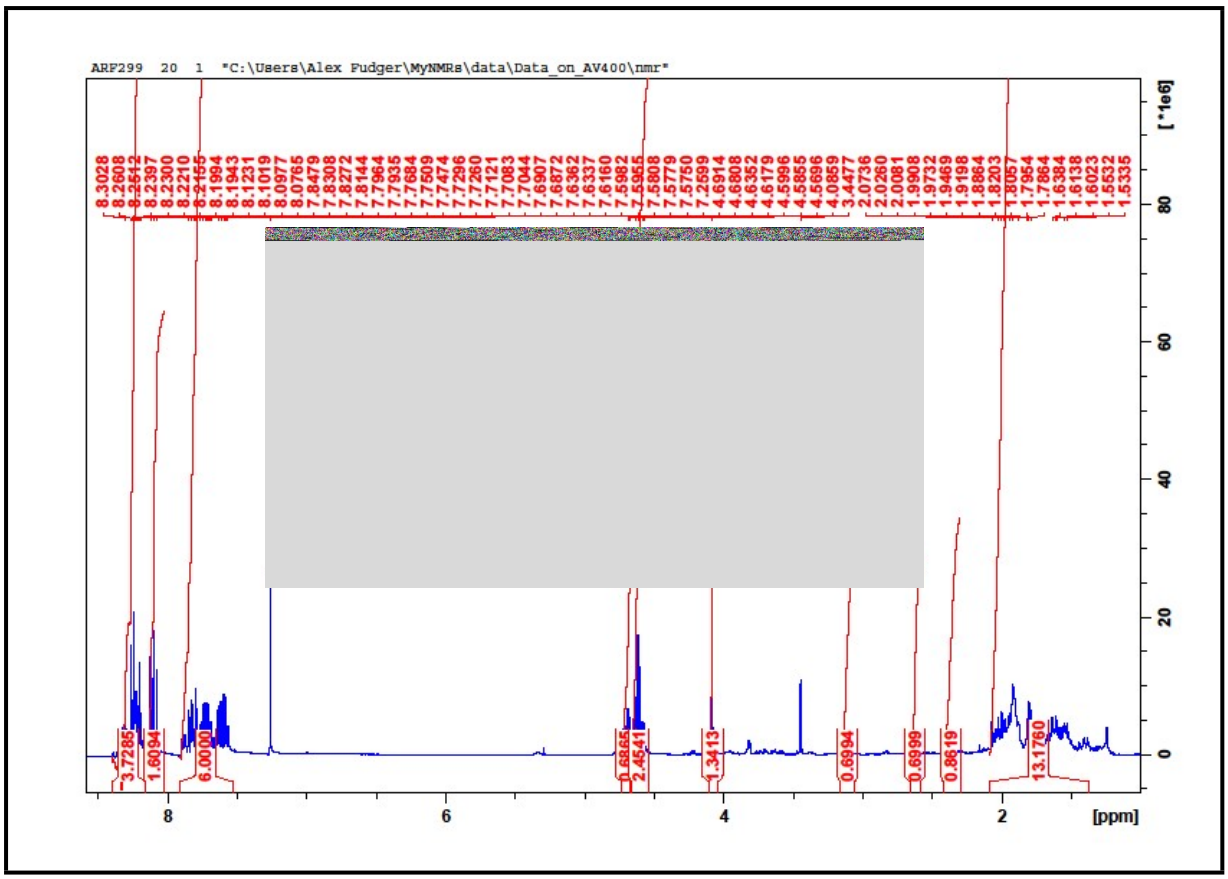


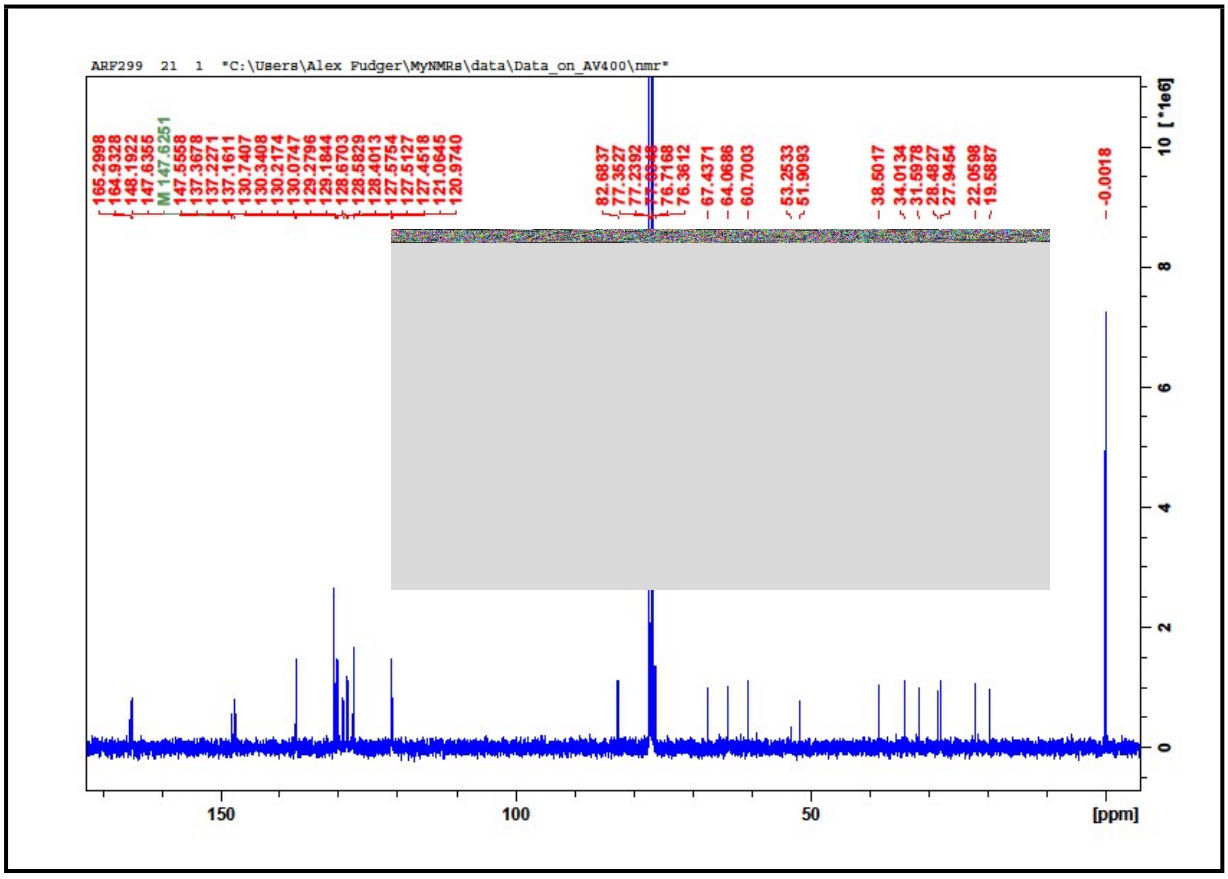
HSQC NMR spectrum for compound **17kb**



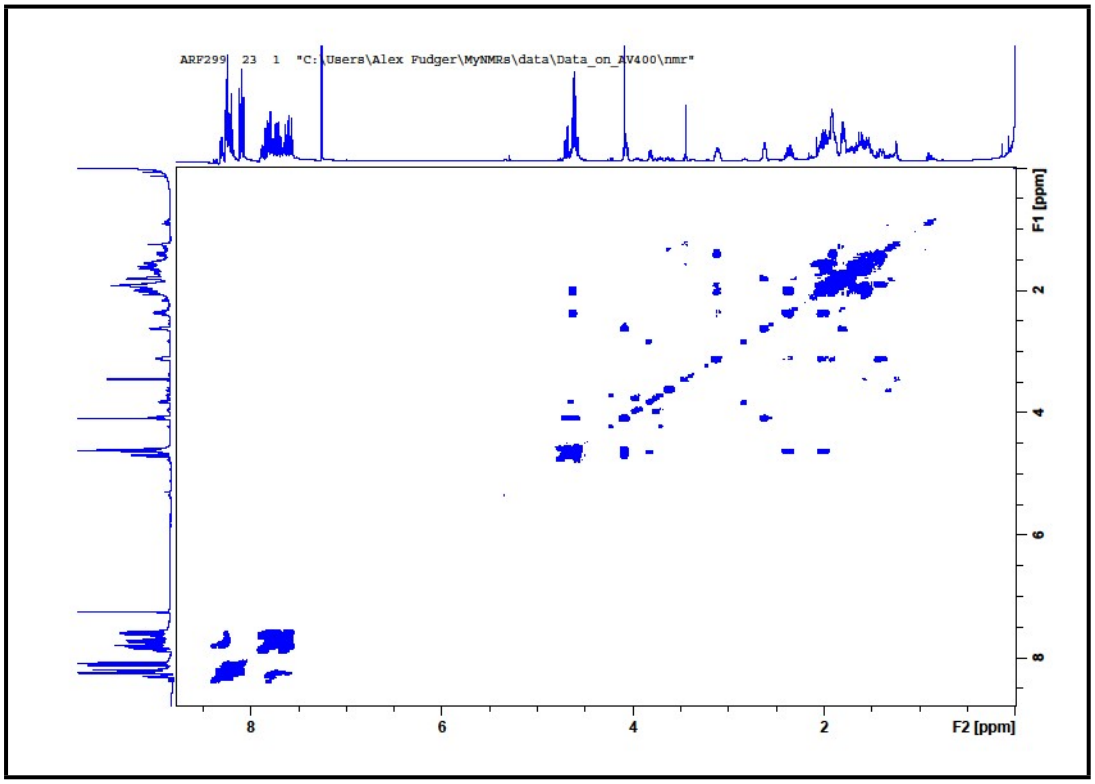

NOESY NMR spectrum for compound **17kb**



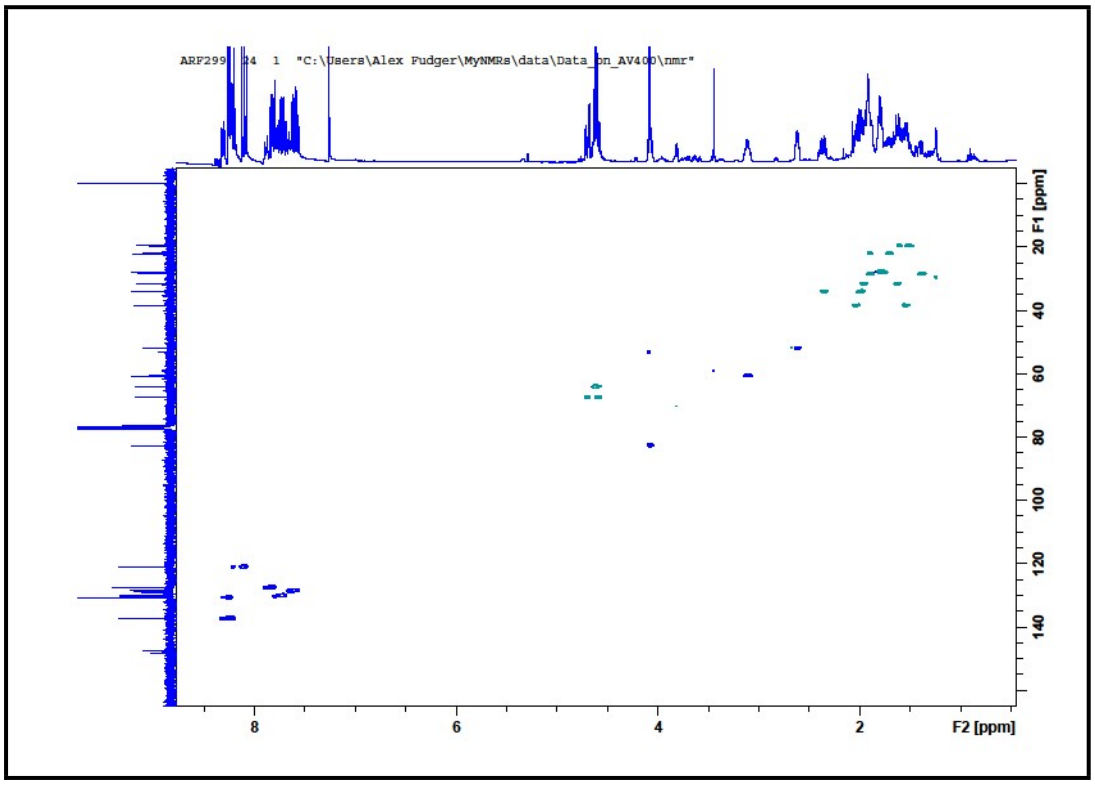

ARF299 20 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
[*1e6]
[ppm]


ARF299 21 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
[*1e6]
[ppm]

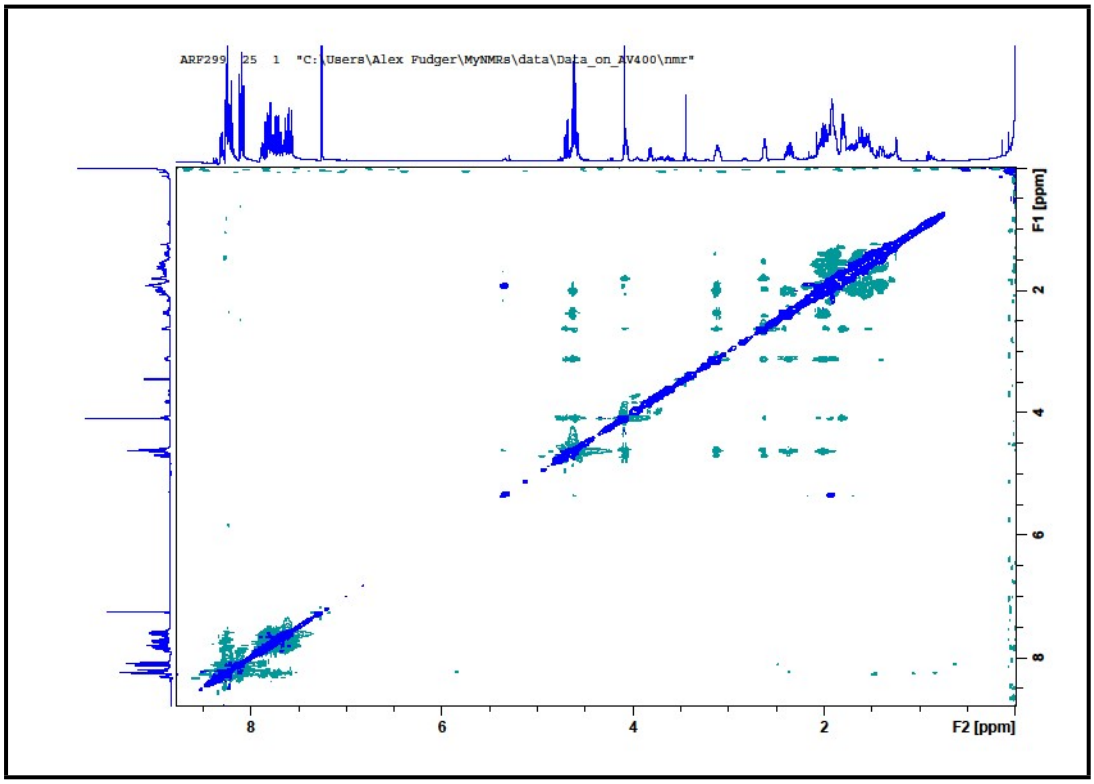
COSY NMR spectrum for compound **17la**

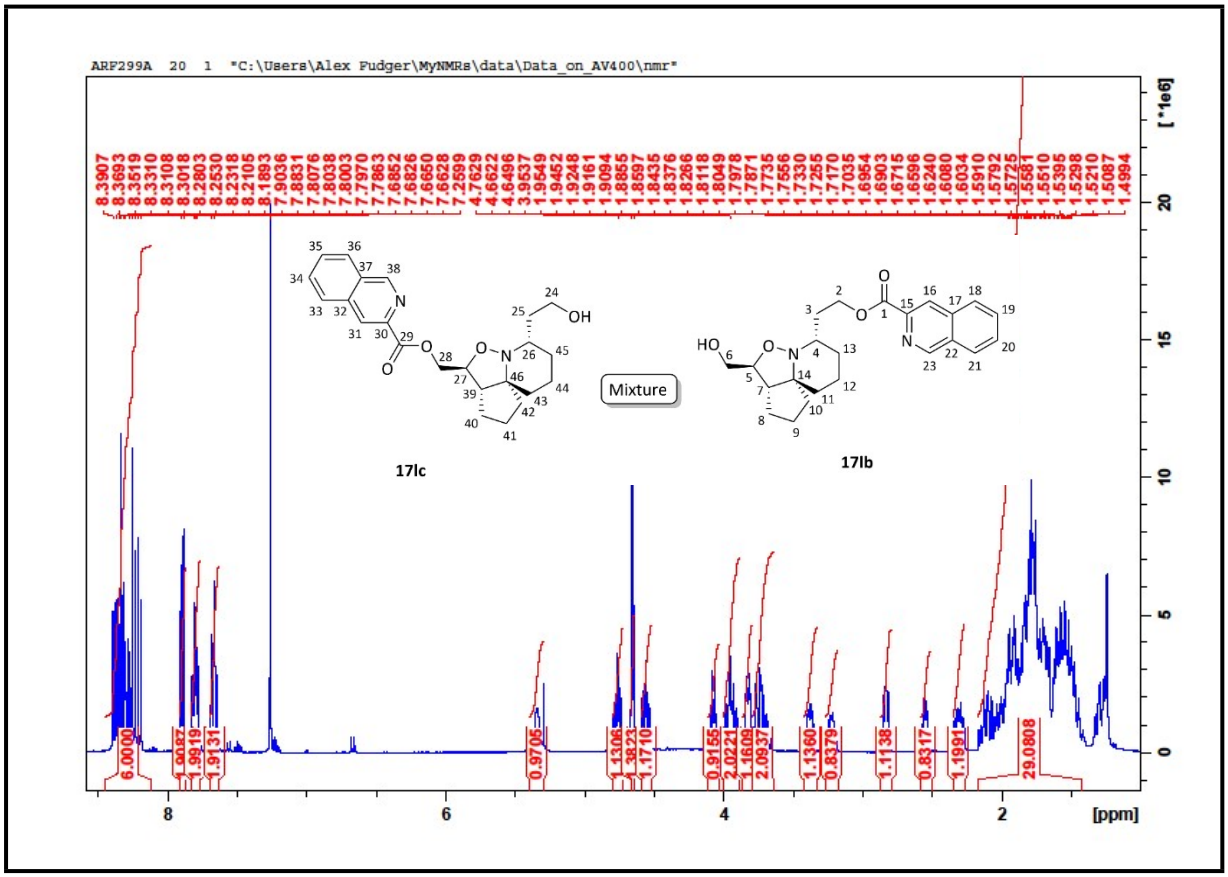


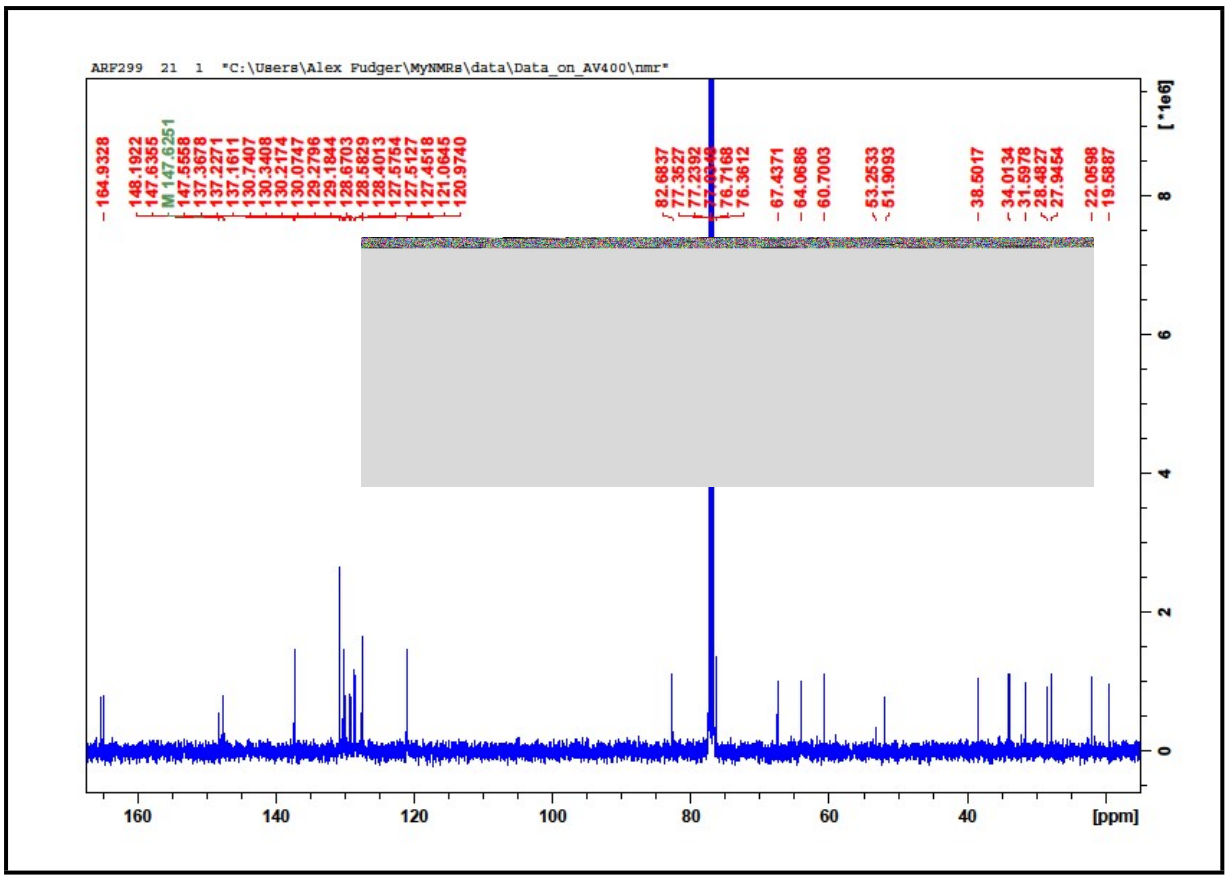

HSQC NMR spectrum for compound **17la**



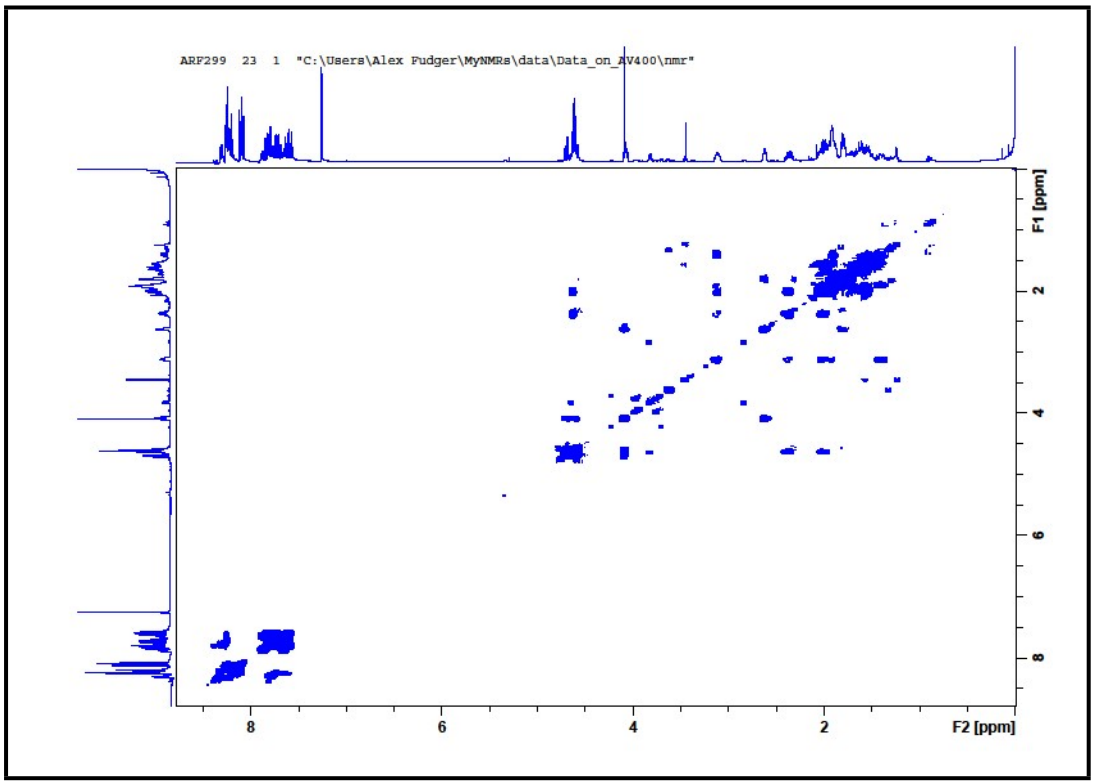
NOESY NMR spectrum for compound **17la**



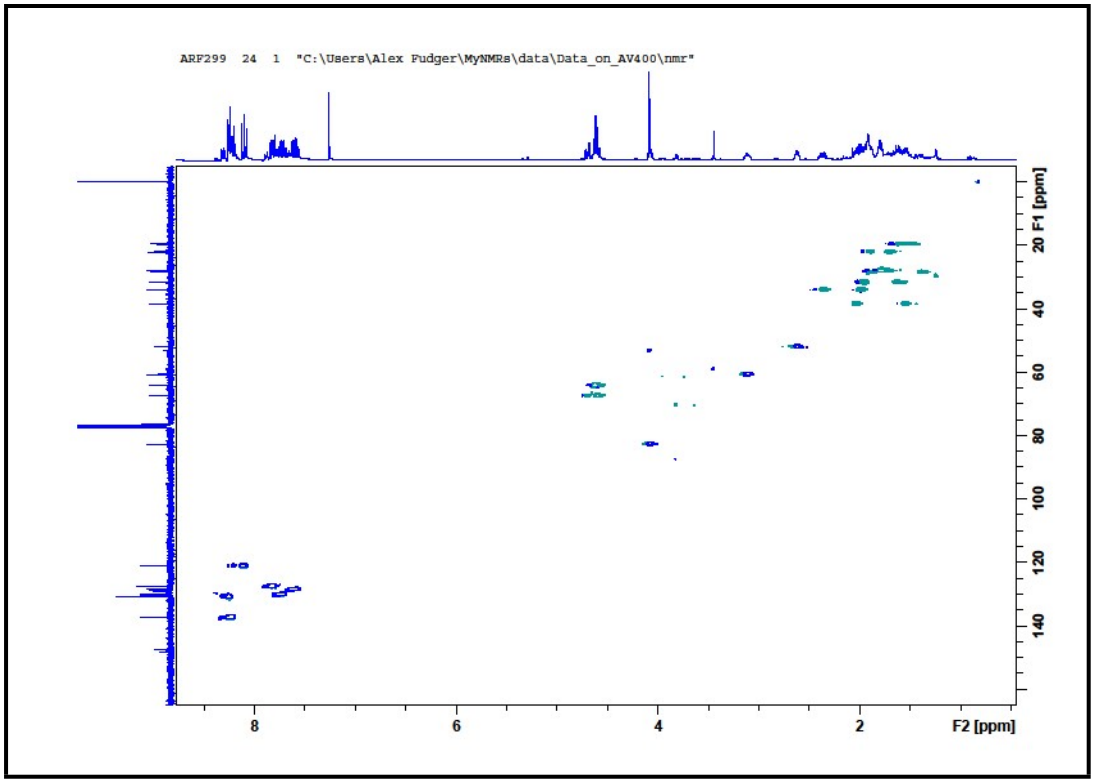

ARF299A 20 1 "C:\Users\Alex Pudger\MyNMRs\data\Data_on_AV400\nmr"
Mixture
17lc
17lb
[ppm]




COSY NMR spectrum for mixture of compounds **17lc** and **17lb**



HSQC NMR spectrum for mixture of compounds **17lc** and **17lb**




NOESY NMR spectrum for mixture of compounds **17lc** and **17lb**