Electronic Supplementary Material (ESI) for Catalysis Science & Technology.

Supporting information

Alumina Grafted SBA-15 Sustainable Bifunctional Catalysts for Direct Cross-Coupling of Benzylic Alcohols to Diarylmethanes

C. Rajendran[a], G. Satishkumar[a*], Charlotte Lang[b] and Eric M. Gaigneaux[b]

[a]Advanced Materials and Catalysis Lab, Department of Chemistry, School of Advanced Sciences, Vellore Institute of Technology, Vellore-632014, Tamilnadu, India
[b]Institute of Condensed Matter and Nanosciences, UCLouvain, 1348 Louvain-la-Neuve, Belgium.

Table of Contents

1. Materials and Methods

1.1 Materials

All chemicals were purchased from commercial suppliers and used without further purification. TEOS, HCl, Sodium aluminate ($NaAlO_2$) and P_{123} were purchased from Sigma Aldrich. Aluminium sec-tri butoxide ($Al(O\text{-}sec\text{-}Bu)_3$, Cerium nitrate ($Ce(NO_3)_3 \cdot 6H_2O$), and di-chloro-ethane (DCE) bought from Alfa Aesar. Thin layer chromatography (TLC) plates (60 F254, 0.2 mm) and all other reagent grade chemicals, solvents were used as received from Avra synthesis Pvt. Ltd.

1.2. Characterization Methods

Powder X-ray diffraction (XRD) patterns were obtained with a D8 Advance (BRUKER), diffractometer with a Cu K alpha ($\lambda = 1.5418$ Å) radiation source 2.2 kW and a Lynx Eye detector (Silicon strip detector technology). XRD scans were recorded between $2\theta = 0.75–5°$ with a step size of 0.05 and step time of 1s for low angle and between $2\theta = 10–90°$ for wide angle analysis. The Scherrer equation is applied to determine crystal size $h = K\lambda/[(B^2 - \beta^2)^{0.5} \cos(2\theta/2)]$. N_2 adsorption-desorption isotherms were performed at liquid nitrogen temperature (-196°C) using an Autosorb iQ and ASiQWin Version 5 model 6 analysers from Quantachrome. Before each measurement, samples were outgassed 5h at 300°C under Helium. Surface area, pore volume, and pore size distribution were obtained from N_2 adsorption-desorption isotherms by using the conventional BET and BJH methods. High Resolution Transmission electron microscopy (HR-TEM) images were taken using a Model FEI-TECNAI G2-20 TWIN 200kV instrument, filament LaB6. The temperature-programmed desorption of ammonia (NH_3-TPD) was conducted on Autosorb iQ and ASiQWin Version 5 model 6 analysers from Quantachrome. Typically, 0.100 g

of the dried sample was placed within a linear quartz tube (i.d. 12 mm) and pretreated in a helium flow at 400 °C for 1 h. Then, the sample was cooled down to 100°C and 20%NH_3/He mixture gas was allowed to flow for 0.5 h. The sample was flushed with helium for 1 h to remove the physisorbed ammonia. NH_3-TPD profile was recorded from 100 °C to 450 °C with a ramping rate of 10 °C min^{-1}. CO_2-TPD analysis was carried out using BELCAT II equipment. 50-100mg of the dried sample was placed within a linear quartz tube (i.d. 12 mm) and pretreatment heated up to 450°C at 10°C min^{-1}, then held at maximum temperature for 30 min in flowing He (50 ml/min). Analysis conditions are then 10% CO_2 + He gas mixture flow for 1h at 50°C, then the physisorbed probe molecule was removed by flowing He (50 $mL.min^{-1}$) for 1 h at 50°C, finally. CO_2-TPD profile was recorded from 50 to 600°C at a heating rate of 10°C.min^{-1} under the flow of helium to desorb the chemisorbed probe molecules. Pyridine chemisorption measurements were performed using a high vacuum setup and Fourier transform infrared (FT-IR) spectra were recorded in transmission mode using an IFS55 Equinox spectrometer (Bruker) equipped with a DTGS detector. 100 scans per spectrum were recorded between 400 and 4000 cm^{-1} with a resolution of 4 cm^{-1}. The pure catalyst powder was pressed into wafers (around 25mg) which were first degassed in the IR cell under vacuum (about 10^{-7} Torr) at 250 °C for 2h to remove water and adsorbed species. The pyridine adsorption took place at room temperature for 10 minutes. The samples were then evacuated for 1h from room temperature to 200 °C (each 100 °C) to progressively desorb the pyridine. The IR spectra were measured after each desorption temperature and the IR spectra recorded after desorption at 100 °C were used for the calculation of the distribution between Brønsted and Lewis acid sites. The distribution between Lewis and Brønsted acid sites was calculated thanks to the integrated absorbance of bands due to pyridine absorption on Brønsted

(around 1550 cm^{-1}) and Lewis (around 1450 cm^{-1}) acid sites. The number of acid sites was calculated using equation 1 [1]:

$$n_T = \frac{A_L C_d}{\varepsilon_L m} + \frac{A_B C_d}{\varepsilon_B m} \quad \text{Equation 1}$$

Where n_T is the total number of pyridine adsorbed at each temperature, in $\mu mol.g^{-1}$

A_L and A_B are the integrated absorbances of FTIR bands relative to Lewis or Brønsted sites respectively (cm^{-1})

C_d is the cross sectional area of the wafer in cm^2

ε_L = 1.44 and ε_B = 0.59 are the molar extinction coefficients in $cm.\mu mol^{-1}$, respectively for Lewis or Brønsted bands [1] and m is the mass of the water in g.

Magic angle spinning (MAS) ^{27}Al NMR spectra were recorded on ECX 400 - Jeol 400 MHz High-Resolution Multinuclear FT-NMR Spectrometer. NMR spectra were recorded on Bruker FT-NMR-400 MHz instrument for ^{1}H and ^{13}C NMR, respectively. All ^{1}H NMR spectra were reported in ppm unit (parts per million) and were measured relative to residual chloroform signal (7.26 ppm or 7.18 ppm) in the deuterated solvent. ^{13}C NMR spectra were reported in ppm relative to deuterated chloroform (77.23 ppm). The SEM analysis was carried out in SEM-Carl Zeiss with energy dispersive spectrometer (EDS) detector. XPS has been recorded using PHI 5000 Versa Probe II, FEI Inc. The products obtained has been confirmed through GC-MS Perkin Elmer, Clarus 680 model. The product yield was determined using Gas Chromatograph (Shimadzu 2014).

2. Characterisation results

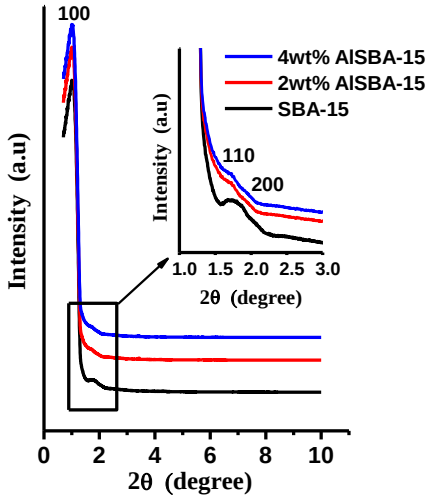


Fig. S1. Low angle XRD pattern of synthesized catalysts and support.

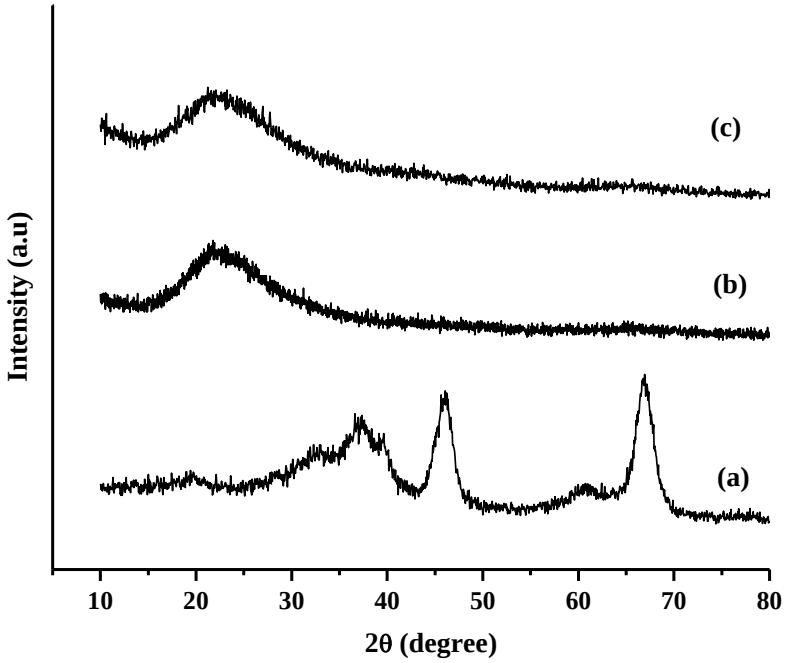


Fig. S2. Wide angle XRD pattern of synthesized catalysts (a) γ-Al_2O_3; (b) 2wt%AlSBA-15; (c) 4wt%AlSBA-15.

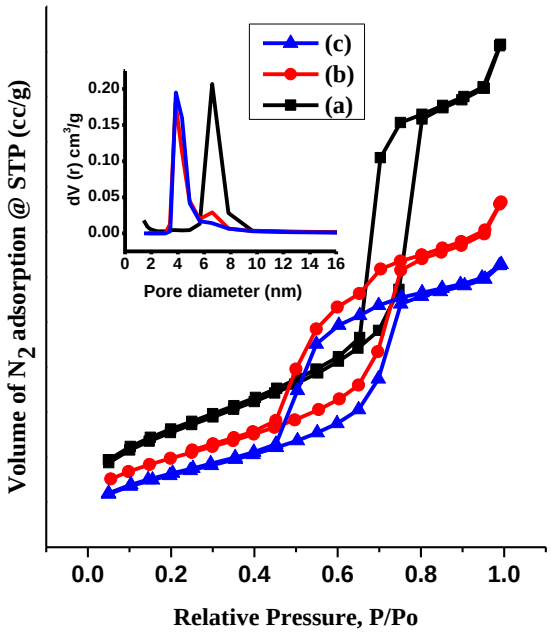


Fig. S3. N_2 adsorption and desorption isotherm of synthesised catalysts and support (a) SBA-15, (b) 2wt%AlSBA-15 and (c) 4wt%AlSBA-15.

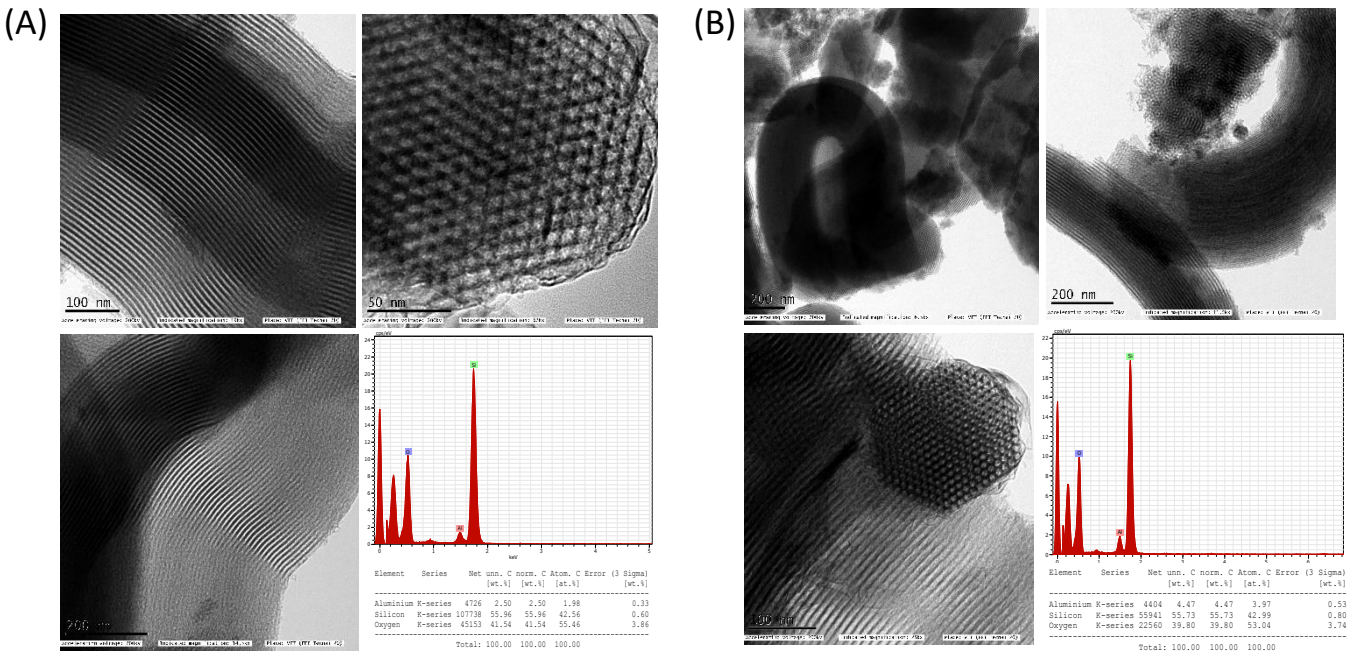


Fig. S4. HR-TEM images of (A) 2wt%AlSBA-15 and (B) 4wt%AlSBA-15

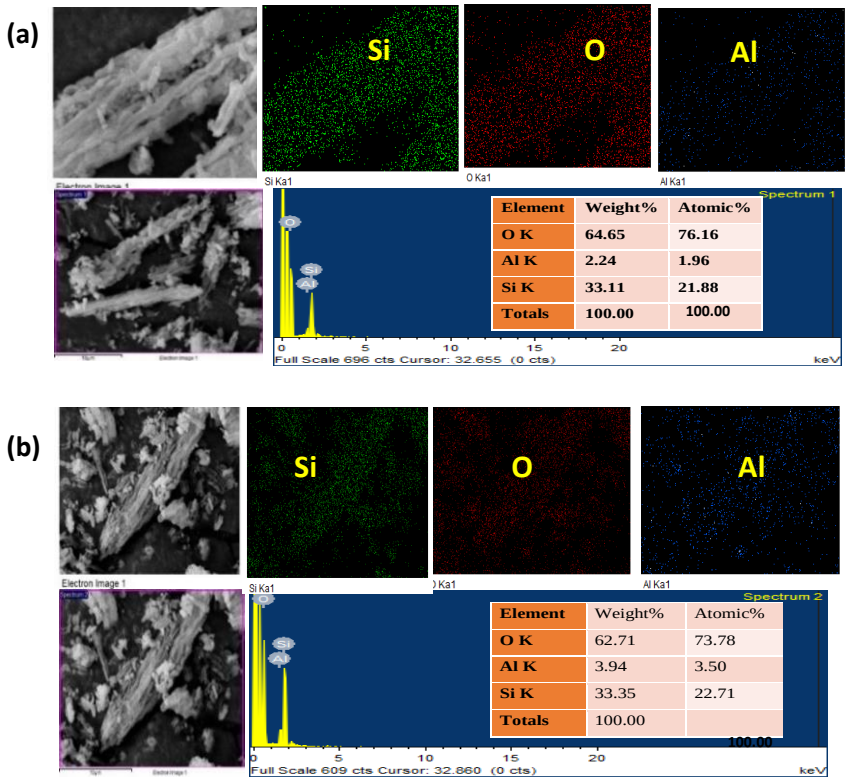


Fig. S5. SEM- EDAX elemental mapping of synthesised catalysts (a) 2wt%AlSBA-15 (b) 4wt%AlSBA-15.

Table S1: Acidity Measurements of 2, 4wt%AlSBA-15 and γ-Al_2O_3

S. No	Catalyst	Temperature T (°C)	Amount of desorbed NH_3 (mmol g^{-1})	Temperature T(°C)	Amount of desorbed NH_3 (mmol g^{-1})
1	γ-Al_2O_3	250	0.170	417	0.015
2	2wt%AlSBA-15	228	0.438	419	0.066
3	4wt%AlSBA-15	252	1.327	447	0.296

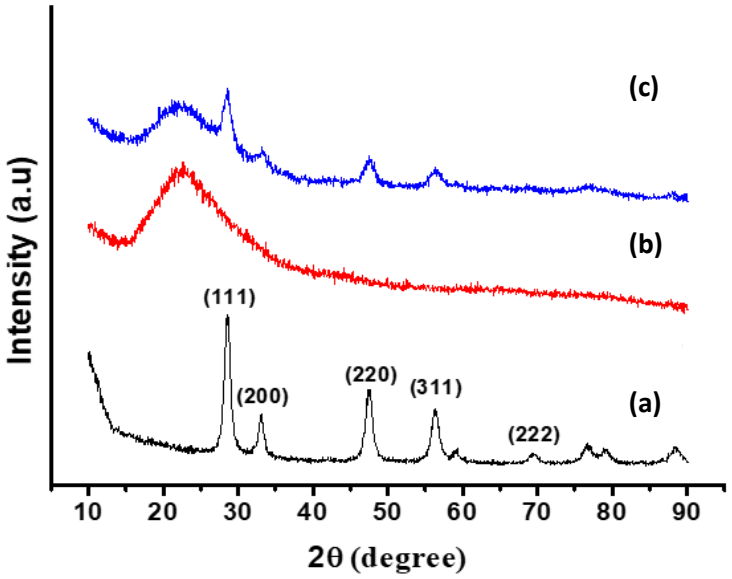
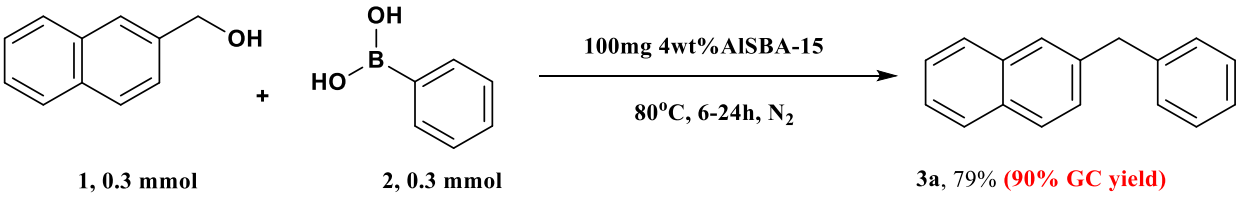


Fig. S6. Wide angel XRD pattern of synthesised catalysts and support. (a) CeO_2 **(JCPDS Card No. 65-5923)**, (b) IE4wt%AlSBA-15M, (c) CeO_2/IE4wt%AlSBA-15M.

3. General procedure for 4wt%AlSBA-15 catalysed construction of diarylmethane using 2-naphthylmethanol and phenylboronic acid.

The reaction was carried out by addition of 100mg AlSBA-15 catalyst to the mixture of 1 equiv. of 2-napthalenemethanol (0.3 mmol), 1 equiv. of phenylboronic acid (0.3 mmol), in 2-3ml of dichloroethane solvent. The reaction mixture was stirred at 80°C for 6 h in N_2 atmosphere. After cooling, the reaction mixture was stirred with 10ml of ethyl acetate and the catalyst was separated from the reaction mixture by filtration. The filtrate was further concentrated to remove the solvent and used for GC-MS analysis. For column chromatography, 1.5 g of silica gel (100-200 mesh) was added to the residue and purified by eluting with hexane to get the desired product white solid (79%).



1, 0.3 mmol **2, 0.3 mmol** **3a**, 79% **(90% GC yield)**

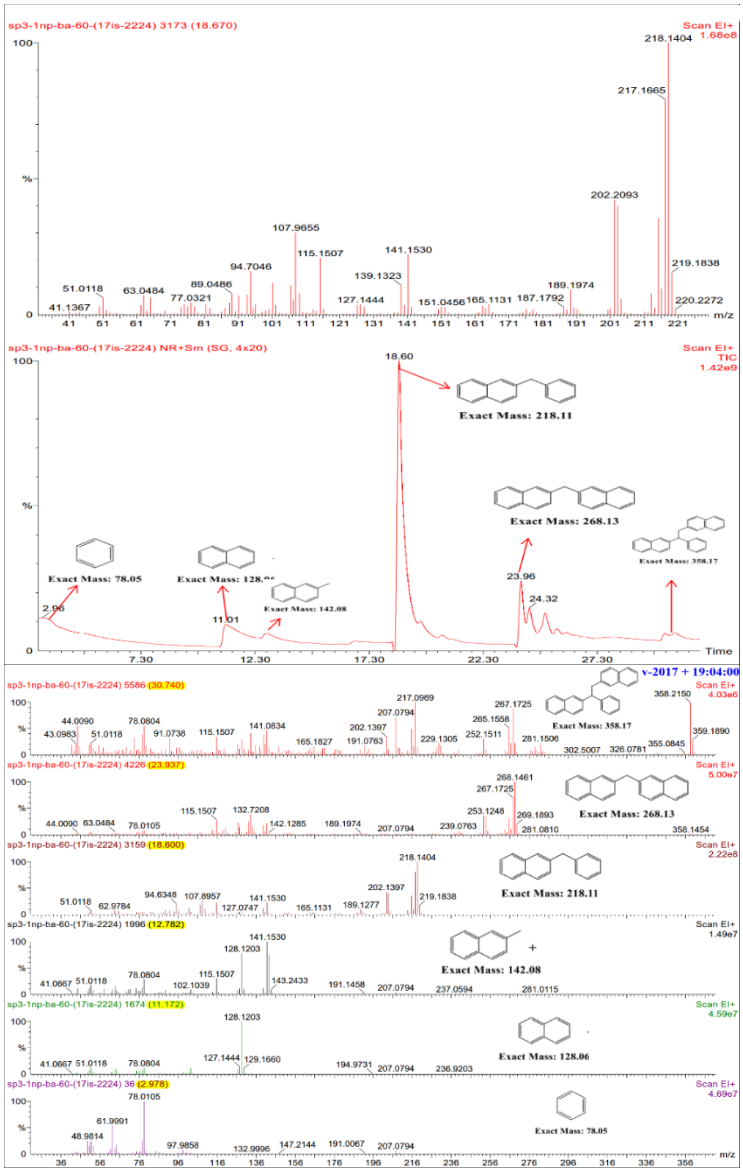


Figure S7 GC-MS result of 4wt%AlSBA-15 catalyzed 2-naphthalenemethanol with phenylboronic acid at 60 °C (67% yield)

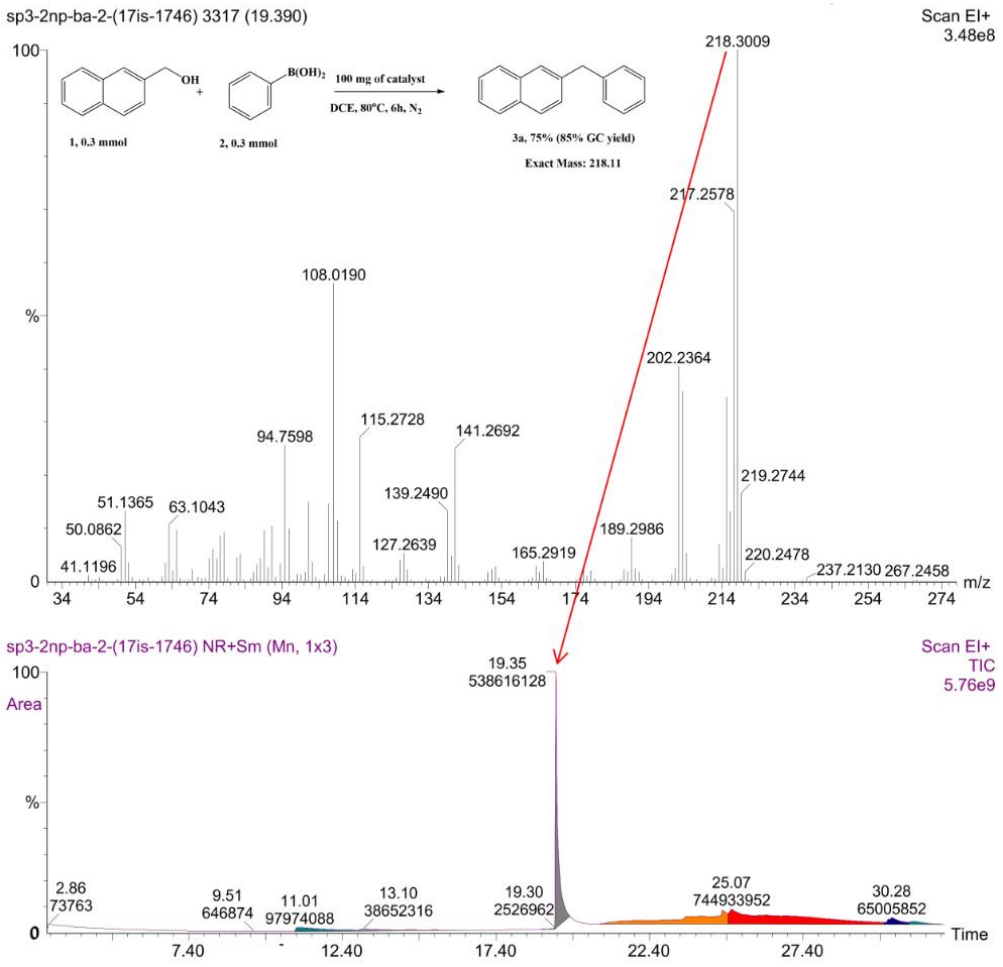


Figure S8 GC-MS result of 4wt%AlSBA-15 catalyzed 2-naphthalenemethanol with phenylboronic acid at 80 °C (85% yield)

4. Spent catalyst characterization results

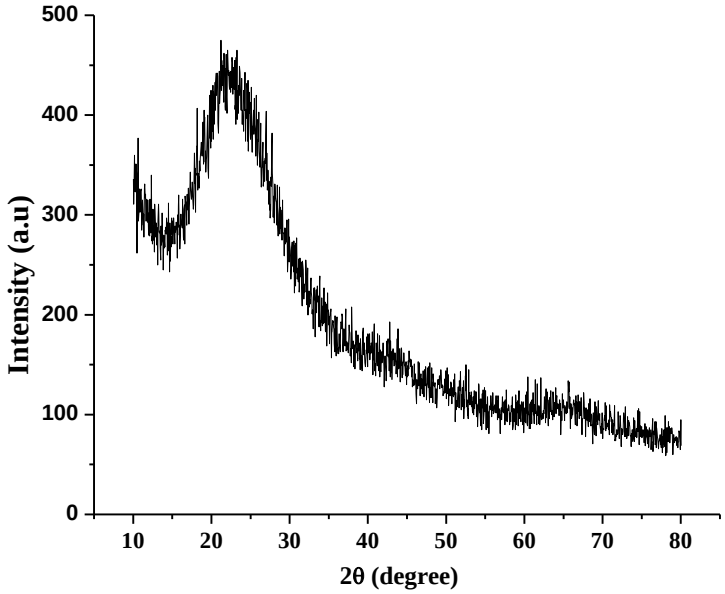


Fig. S9. Wide angle XRD pattern of spent catalyst 4wt%AlSBA-15.

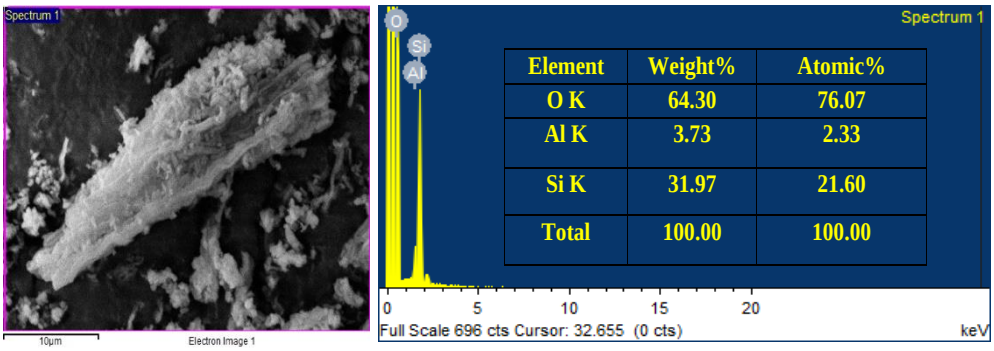
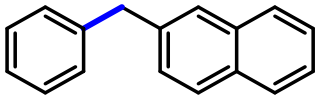


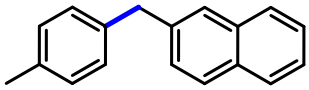
Fig. S10. SEM-EDAX pattern of spent catalyst 4wt%AlSBA-15

5. ^{1}H & ^{13}C NMR and GC-MS data of the obtained products



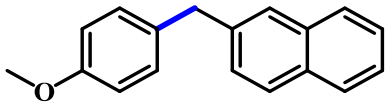
2-(4-bromobenzyl)naphthalene [2] (**3a**)

Followed the general procedure **1**, white solid (79% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 – 7.74 (m, 3H), 7.62 (s, 1H), 7.46–7.39 (m, 2H), 7.32–7.27 (m, 3H), 7.23 – 7.20 (m, 3H), 4.13 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.00, 138.62, 133.63, 132.10, 129.09, 128.51, 128.09, 127.66, 127.64, 127.57, 127.11, 126.17, 125.99, 125.36, 42.13. GC-MS: Calc. for $C_{17}H_{14}$ Exact Mass: 218.1096, Found Mass: 218.0726.



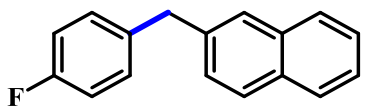
2-(4-methylbenzyl)naphthalene [2] (**3b**)

Followed the general procedure **1**, white solid (58% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 – 7.72 (m, 3H), 7.61 (s, 1H), 7.43 – 7.39 (m, 2H), 7.31 (d, J = 1.6 Hz, 1H), 7.10 – 7.09 (m, 4H), 4.09 (s, 2H), 2.3 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.93, 137.96, 135.67, 133.66, 132.11, 129.22, 128.94, 128.07, 127.67, 127.64, 127.58, 127.03, 125.96, 125.31, 41.72, 21.05. GC-MS: Calc. for $C_{18}H_{16}$ Exact Mass: 232.1252 Found Mass: 232.0254.



2-(4-methoxybenzyl)naphthalene [2] (**3c**)

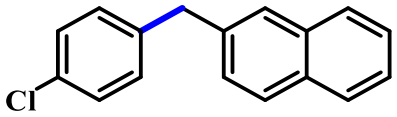
Followed the general procedure **1**, Colorless oil (67% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.65 (m, 3H), 7.53 (s, 1H), 7.36 – 7.33 (m, 2H), 7.22 (d, J = 7.6 Hz, 1H), 7.06 (d, J = 8.48 Hz, 2H), 6.75 (d, J = 8.52 Hz, 2H), 4.00 (s, 2H), 3.70 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.05, 139.09, 133.64, 133.12, 132.08, 129.98, 128.06, 127.63, 127.61, 127.56, 126.93, 125.97, 125.31, 113.94, 55.28, 41.23. GC-MS: Calc. for $C_{18}H_{16}O$ Exact Mass: 248.1201 Found Mass: 248.0522.



2-(4-fluorobenzyl)naphthalene [2] (**3d**)

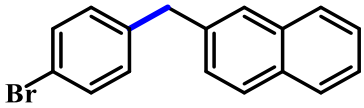
Followed the general procedure **1**, white solid (70% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 – 7.75 (m, 3H), 7.60 (s, 1H), 7.43 – 7.42 (m, 2H), 7.29 (t, J = 8.4 Hz, 1H), 7.18 (t, J = 8.0 Hz, 1H), 6.97 (t, J = 8.4 Hz, 2H), 4.10 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.83 (d, $J_{C1\text{-}F}$ = 252 Hz), 138.43, 136.66 (d, $J_{C4\text{-}F}$ = 3.2 Hz), 133.60, 132.12, 130.44

(d, $J_{C3\text{-}F}$ = 7.6 Hz), 128.21, 127.66, 127.56, 127.00, 126.67, 126.21, 125.21, 115.41(d, $J_{C2\text{-}F}$ = 21.2 Hz), 41.26. GC-MS: Calc. for $C_{17}H_{13}F$ Exact Mass: 236.1001 Found Mass: 236.1233.



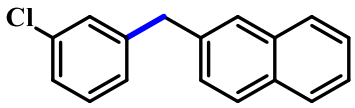
2-(4-chlorobenzyl)naphthalene [2] (**3e)**

Followed the general procedure **1**, white solid (70% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.75 (m, 3H), 7.60 (s, 1H), 7.45 – 7.42 (m, 2H), 7.22 – 7.19 (m, 3H), 7.09 (d, *J* = 8.32 Hz, 2H), 4.04 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.45, 138.03, 133.58, 132.14, 131.99, 130.37, 128.62, 128.26, 127.66, 127.56, 127.43, 127.13, 126.13, 125.53, 41.42. GC-MS: Calc. for $C_{17}H_{13}Cl$ Exact Mass: 252.0706, Found Mass: 252.0514.



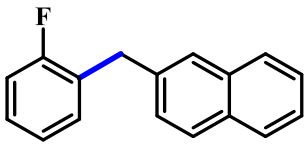
2-(4-bromobenzyl)naphthalene [3] (**3f**)

Followed the general procedure **1**, white solid (65% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.75 (m, 3H), 7.60 (s, 1H), 7.45 – 7.39 (m, 4H), 7.28 (s, 1H), 7.10 (d, *J* = 8 Hz, 2H), 4.08 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.97, 139.31, 137.92, 133.58, 132.14, 131.57, 130.78, 128.26, 127.66, 127.55, 127.42, 127.14, 126.13, 125.54, 120.05, 41.48. GC-MS: Calc. for $C_{17}H_{13}Br$ Exact Mass: 296.0201 Found Mass: 296.0501.



2-(3-chlorobenzyl)naphthalene [2] (**3g**)

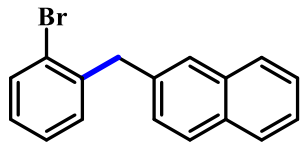
Followed the general procedure **1**, white solid (65% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.54 (m, 3H), 7.40 (s, 1H), 7.39 – 7.37 (m, 2H), 7.22 – 7.19 (m, 3H), 7.09 (d, *J* = 8 Hz, 2H), 4.04 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.42, 138.0, 133.55, 132.11, 131.96, 130.34, 128.58, 128.22, 127.63, 127.52, 127.40, 127.10, 126.09, 125.49, 41.38. GC-MS: Calc. for $C_{17}H_{13}Cl$ Exact Mass: 252.0706, Found Mass: 252.1511.



2-(2-fluorobenzyl)naphthalene [2] (**3h**)

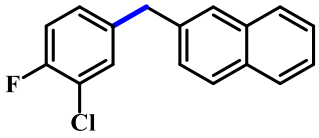
Followed the general procedure **1**, white solid (63% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.75 (m, 3H), 7.65 (s, 1H), 7.44 – 7.41 (m, 2H), 7.37 (d, *J* = 7.32 Hz, 1H), 7.19 – 7.16 (m, 2H), 7.07 – 6.97 (m, 2H), 4.16 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.03 (d, $J_{C1\text{-}F}$ = 244 Hz) 159.51, 137.34, 133.60, 132.14, 131.14 (d, $J_{C4\text{-}F}$ = 5 Hz), 128.20 (d, $J_{C3\text{-}F}$ = 8 Hz), 128.07, 127.99, 127.63 (d, $J_{C2\text{-}F}$ = 6 Hz), 127.42, 126.09

(d, $J_{C6\text{-}F}$ = 7 Hz), 125.43, 124.09, 115.47 (d, $J_{C6\text{-}F}$ = 22 Hz), 34.94 (d, $J_{C7\text{-}F}$ = 3 Hz). GC-MS: Calc. for $C_{17}H_{13}F$ Exact Mass: 236.1001 Found Mass: 236.0925.



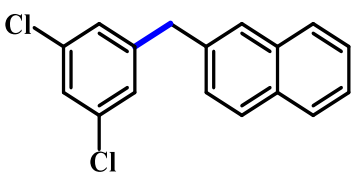
2-(2-bromobenzyl)naphthalene [4] (**3i**)

Followed the general procedure **1**, colorless oil (60% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.75 (m, 3H), 7.59 (d, *J* = 8.88 Hz, 2H), 7.45 – 7.42 (m, 2H), 7.34 (d, *J* = 8.36 Hz, 1H), 7.21 (d, *J* = 7.48 Hz, 1H), 7.16 – 7.10 (m, 2H), 4.27 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.27, 137.02, 133.59, 132.91, 131.19, 128.11, 127.99, 127.64, 127.61, 127.56, 127.51, 127.37, 126.02, 125.46, 124.97, 41.91. GC-MS: Calc. for $C_{17}H_{13}Br$ Exact Mass: 296.0201 Found Mass: 296.0363.



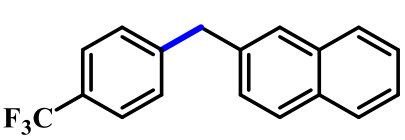
2-(3-chloro-4-fluorobenzyl) naphthalene **(3j)**

Followed the general procedure **1**, colorless oil (60% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.76 (m, 3H), 7.60 (s, 1H), 7.49 – 7.42 (m, 2H), 7.27 – 7.23 (m, 2H), 7.08 – 7.02 (m, 2H), 4.07 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.98 (d, $J_{C1\text{-}F}$ = 245.5 Hz), 138.04 (d, $J_{C4\text{-}F}$ = 4.05 Hz), 137.53, 133.57, 132.21, 130.92, 128.60 (d, $J_{C3\text{-}F}$ = 7.07 Hz), 128.40, 127.69 (d, $J_{C2\text{-}Cl\text{-}F}$ = 11.09 Hz), 127.30, 127.20, 126.23, 125.66, 120.91, 120.73, 116.59 (d, $J_{C2\text{-}F}$ = 20.9 Hz), 41.04. GC-MS: Calc. for $C_{17}H_{12}ClF$ Exact Mass: 270.0612, Found Mass: 270.0588.



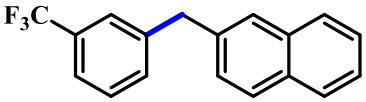
2-(3,5-dichlorobenzyl)naphthalene (**3k**)

Followed the general procedure **1**, white solid (55% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) : δ (ppm) 7.81 – 7.76 (m, 3H), 7.60 (s, 1H), 7.47 – 7.44 (m, 2H), 7.25 (d, *J* = 8.44 Hz, 1H), 7.20 (s, 1H), 7.09 (d, *J* = 1.56 Hz, 2H), 4.05 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 144.40, 136.76, 134.96, 133.58, 132.29, 128.54, 127.73, 127.63, 127.50, 127.41, 127.28, 126.52, 126.31, 125.79, 41.5. GC-MS: Calc. for $C_{17}H_{12}Cl_2$ Exact Mass: 286.0316, Found Mass: 286.0924.



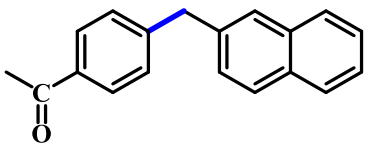
2-(4-(trifluoromethyl)benzyl)naphthalene [2] (**3l**)

Followed the general procedure **1**, white solid (60% isolated yield).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.53 (m, 3H), 7.46 (s, 1H), 7.45 (d, *J* = 8 Hz, 2H), 7.39 – 7.36 (m, 2H), 7.34 (d, *J* = 7.46 Hz, 2H), 7.23 (d, *J* = 8 Hz, 1H), 4.09 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.11, 137.46, 133.61, 132.22, 128.73, 128.40, 127.71, 127.59, 127.41, 127.32, 126.24, 125.68, 125.52, 125.49, 125.45, 122.99, 41.89. GC-MS: Calc. for $C_{18}H_{13}F_3$ Exact Mass: 286.0969, Found Mass: 286.0929



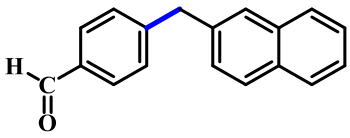
2-(3-(trifluoromethyl)benzyl)naphthalene (**3m**)

Followed the general procedure **1**, white solid (60% isolated yield).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.77 (m, 3H), 7.63 (s, 1H), 7.54 (d, *J* = 8 Hz, 2H), 7.47 – 7.44 (m, 2H), 7.33 – 7.27 (m, 3H), 4.19 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.58, 132.42, 131.96, 131.64, 131.32, 131.00, 130.53, 129.55, 128.94, 128.39, 128.09, 127.68, 127.59, 127.36, 126.19, 125.63, 125.38, 124.77, 124.01, 123.15, 122.67, 41.86. GC-MS: Calc. for $C_{18}H_{13}F_3$ Exact Mass: 286.0969, Found Mass: 286.0950.



1-(4-(naphthalen-2-ylmethyl)phenyl)ethan-1-one [2] (**3n**)

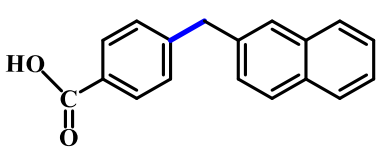
Followed the general procedure **1**, white solid (60% isolated yield).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.76 (m, 4H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.39 – 7.33 (m, 3H), 7.23 – 7.17 (m, 3H), 4.41 (s, 2H), 2.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.75, 145.44, 134.53, 134.25, 132.97, 130.94, 127.82, 127.75, 127.59, 126.51, 126.50, 125.12, 124.68, 124.52, 123.02, 38.10, 26.51. GC-MS: Calc. for $C_{19}H_{16}O$ Exact Mass: 260.1201, Found Mass: 260.0920.



4-(naphthalen-2-ylmethyl)benzaldehyde [2] (**3o**)

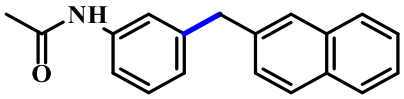
Followed the general procedure **1**, white solid (60% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.80 – 7.76 (m, 5H), 7.62 (s, 1H), 7.46 – 7.43 (m, 2H), 7.37 (d, *J* = 7.92 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 1H), 4.19 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 192.02, 148.31, 137.25, 133.61, 132.22, 130.11, 129.70,

128.44, 127.72, 127.59, 127.41, 127.39, 126.27, 125.71, 42.27. GC-MS: Calc. for $C_{18}H_{14}O$ Exact Mass: 246.1045, Found Mass: 246.0955.



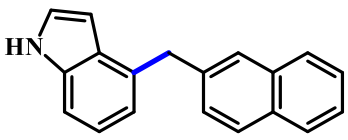
4-(naphthalen-2-ylmethyl)benzoic acid [5] (**3p**)

Followed the general procedure **1**, white solid (55% isolated yield). ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.81 (s, 1H), 8.01 (d, *J* = 9.28 Hz, 1H), 7.93 (d, *J* = 5.36 Hz, 1H), 7.85 (m, 3H), 7.49 (m, 3H), 7.44 (d, *J* = 6.8 Hz, 1H), 7.35 – 7.33 (d, *J* = 7.96 Hz, 2H), 4.51 (s, 2H).^{13}C NMR (100 MHz, DMSO-d_6): δ 167.63, 146.60, 136.55, 134.06, 131.89, 129.93, 129.10, 129.08, 129.04, 127.94, 127.61, 126.60, 126.18, 126.16, 124.64, 38.51. GC-MS: Calc. for $C_{18}H_{14}O_2$ Exact Mass: 262.0994 Found Mass: 262.1025.



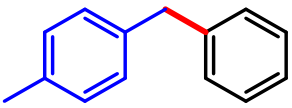
N-(3-(naphthalen-2-ylmethyl)phenyl)acetamide (**3q**)

Followed the general procedure **1**, white solid (45% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 8.05 – 7.98 (m, 1H), 7.87 – 7.77 (m, 2H), 7.75 – 7.68 (d, *J* = 7.7 Hz, 1H), 7.61 – 7.55 (d, *J* = 1.3 Hz, 1H), 7.51 – 7.41 (m, 3H), 7.38 – 7.26 (m, 2H), 7.07 – 7.00 (d, *J* = 8.0 Hz, 1H), 4.19 (s, 2H), 2.18 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 170.02, 138.70, 135.70, 133.59, 133.37, 132.78, 128.56, 127.71, 126.43, 126.32, 125.94, 125.39, 125.07, 122.91, 43.88, 23.30. GC-MS: Calc. for $C_{19}H_{17}NO$ Exact Mass: 275.1310, Found Mass: 275.1315.



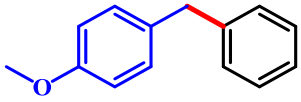
4-(naphthalen-2-ylmethyl)-1H-indole (**3r**)

Followed the general procedure **1**, white solid (50% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.73 – 7.62 (m, 3H), 7.58 (d, *J* = 7.8 Hz, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.37 – 7.33 (m, 3H), 7.14 – 7.12 (m, 2H), 7.06 – 6.97 (m, 2H), 6.85 (s, 1H), 6.48 (s, 1H), 4.20 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.92, 135.82, 133.16, 132.82, 131.98, 128.44, 127.92, 127.84, 127.61, 127.38, 126.63, 126.14, 126.13, 123.94, 121.61, 121.32, 111.95, 101.29, 38.04. GC-MS: Calc. for $C_{19}H_{15}N$ Exact Mass: 257.1204 Found Mass: 257.1656.



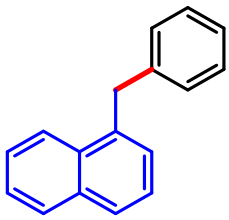
1-benzyl-4-methylbenzene [6] (**4a**)

Followed the general procedure **1**, white solid (65% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 2H), 7.22 – 7.16 (m, 3H), 7.10 – 7.05 (m, 4H), 3.93 (s, 2H), 2.33 – 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.46, 138.12, 135.58, 129.19, 128.91, 128.85, 128.47, 126.02, 41.55, 21.05. GC-MS: Calc. For $C_{14}H_{14}$ Exact Mass: 182.1096 Found Mass: 182.0126.



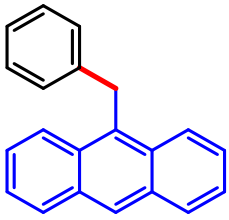
1-benzyl-4-methoxybenzene [6] (**4b**)

Followed the general procedure **1**, colorless oil (60% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.20 (m, 6H), 7.05 (s, 1H), 6.86 (s, 2H), 3.97 (s, 2H), 3.81 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.33, 141.02, 130.31, 129.66, 128.97, 128.25, 127.40, 125.77, 120.46, 110.39, 55.35, 35.85. GC-MS: Calc. for $C_{14}H_{14}O$ Exact Mass: 198.1045, Found Mass: 198.0984.



2-(benzyl)naphthalene [2] (**4c**)

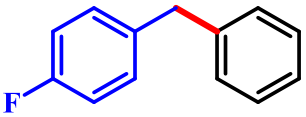
Followed the general procedure **1**, white solid (55% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (t, *J* = 6.4 Hz, 1H), 7.86 – 7.80 (t, *J* = 6.4 Hz, 1H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.46 – 7.40 (m, 3H), 7.29 – 7.24 (m, 3H), 7.20 - 7.18 (m, 3H) 4.45 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 140.65, 136.64, 133.95, 132.15, 128.76, 128.68, 128.47, 127.35, 127.17, 126.07, 125.99, 125.57, 124.29, 39.06. GC-MS: Calc. for $C_{17}H_{14}$ Exact Mass: 218.1096, Found Mass: 218.1096.



9-benzylanthracene [7] (**4d**)

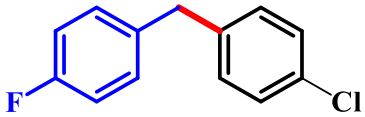
Followed the general procedure **1**, white solid (65% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (s, 1H), 8.16 – 8.13 (m, 2H), 7.98 – 7.95 (m, 2H), 7.40 – 7.37 (m, 4H), 7.12 – 7.04 (m, 5H), 4.95 (s, 2H).^{13}C NMR (100 MHz,

$CDCl_3$) δ 140.84, 131.67, 130.55, 129.13, 128.47, 128.13, 126.57, 125.96, 125.90, 124.93, 124.81, 33.55. GC-MS: Calc. for $C_{22}H_{17}ClO$ Exact Mass: 268.1252, Found Mass: 268.1240.



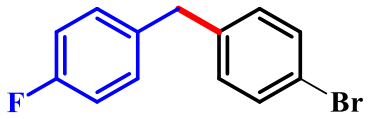
1-benzyl-4-fluorobenzene [8] (**4e**)

Followed the general procedure **1**, white solid (70% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.17 (m, 2H), 7.14 – 7.04 (m, 5H), 6.89 (t, *J* = 8.1 Hz, 2H), 3.87 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 162.64 (d, $J_{C1\text{-}F}$ = 242.3 Hz), 140.95, 136.79 (d, $J_{C4\text{-}F}$ = 3.1 Hz), 130.33, 128.84 (d, $J_{C3\text{-}F}$ = 29.5 Hz), 126.21, 115.32 (d, $J_{C2\text{-}F}$ = 20.9 Hz), 41.09. GC-MS: CALC. For $C_{13}H_{11}F$ Exact Mass: 186.0845 Found Mass: 186.0226.



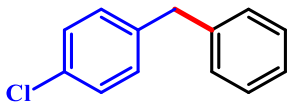
1-chloro-4-(4-fluorobenzyl)benzene [9] (**4f**)

Followed the general procedure **1**, Colorless oil (56% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 – 7.03 (m, 2H), 7.02 – 6.91(m, 4H), 6.89 – 6.87 (d, *J* = 8.3 Hz, 2H), 3.83 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 162.75 (d, $J_{C1\text{-}F}$ = 243 Hz), 139.41, 136.24 (d, $J_{C4\text{-}F}$ = 3 Hz), 132.06, 130.30 (d, $J_{C3\text{-}F}$ = 8 Hz), 128.66, 115.47 (d, $J_{C2\text{-}F}$ = 21 Hz), 40.40. GC-MS: Calc. For $C_{13}H_{10}ClF$ Exact Mass: 220.0455 Found Mass: 220.0485.



1-bromo-4-(4-fluorobenzyl)benzene [10] (**4g**)

Followed the general procedure **1,** Colorless oil (62% isolated yield). 1H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.4 Hz, 2H), 7.01 – 6.98 (m, 2H), 6.92 (d, *J* = 8 Hz, 2H), 6.85 (t, *J* = 8.1 Hz, 2H), 3.78 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 162.77 (d, $J_{C1\text{-}F}$ = 243 Hz), 139.95, 136.15 (d, $J_{C4\text{-}F}$ = 3 Hz), 131.63, 130.58, 130.31 (d, $J_{C3\text{-}F}$ = 7.67 Hz), 120.12, 115.49 (d, $J_{C2\text{-}F}$ = 21.1 Hz), 40.47. GC-MS: Calc. For $C_{13}H_{10}BrF$: 263.9950, Found Mass: 263.9825.



1-benzyl-4-chlorobenzene [6] (**4h**)

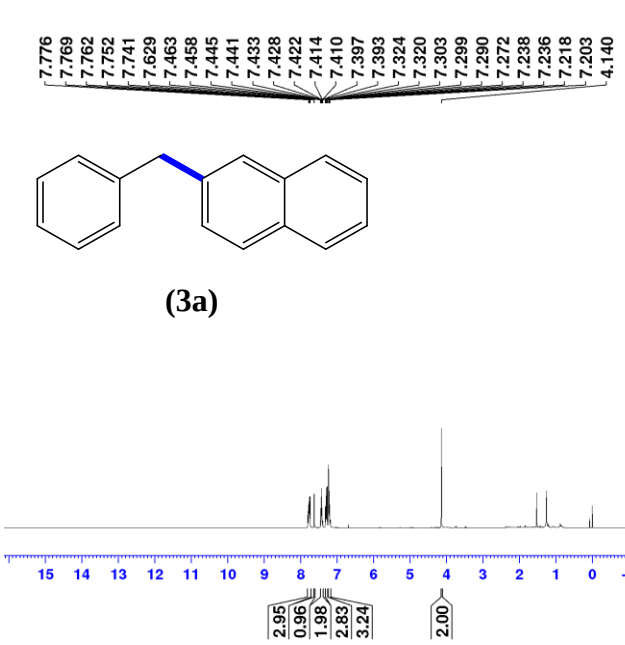
Followed the general procedure **1**, white solid (55% isolated yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.23 (m, 4H), 7.20 (s, 1H), 7.15 (d, *J* = 7.44 Hz, 2H), 7.10 (d, *J* = 8.2 Hz, 2H), 3.94 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 140.57, 139.60, 131.90, 130.27,

128.87, 128.57, 126.30, 41.25. GC-MS: Calc. For $C_{13}H_{11}Cl$ Exact Mass: 202.0549, Found Mass: 202.0858

6. Reference

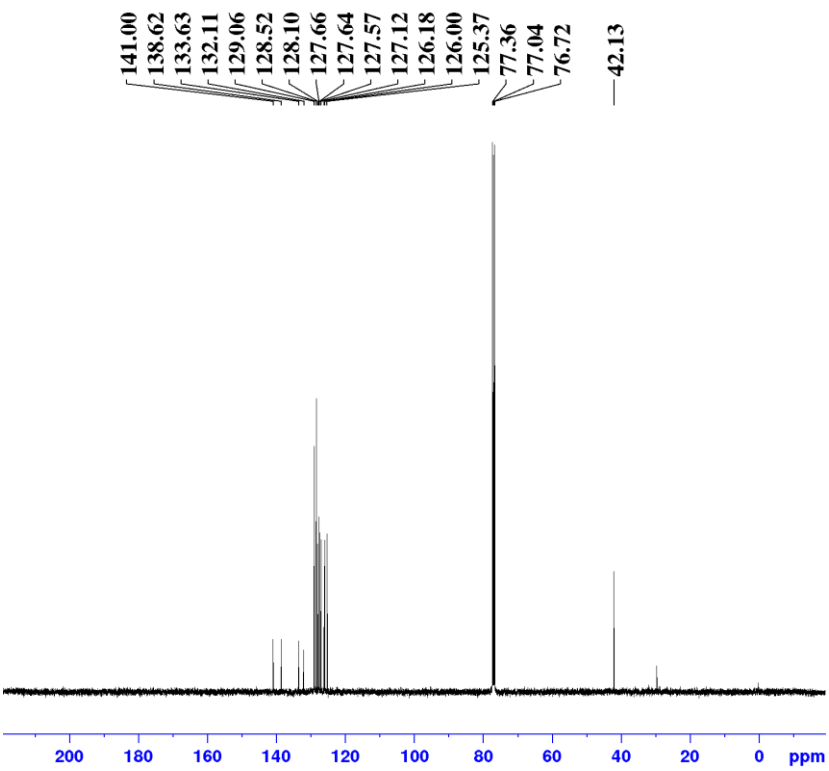
1. I. S. Pieta, M. Ishaq, R. P. K. Wells, J. A. Anderson, *Applied Catalysis A: General*, **2010**, *390*, 127-134.
2. Z. C. Cao, D.G. Yu, R.Y. Zhu, J. B. Wei, Z. J. Shi, *Chem. Commun.*, **2015**, *51*, 2683-2686.
3. X. Mo, J. Yakiwchuk, J. Dansereau, J. A. McCubbin, D. G. Hall, *J. Am. Chem. Soc.*, **2015,** *137* (30), 9694-9703.
4. C. Ramesh Kumar, K. T. V. Rao, P. S. Sai Prasad, N. Lingaiah, *J. Mol., Catal., A: Chemical* **2011,** *337* (1), 17-24.
5. M. Hasegawa, K. Takenouchi, K. Takahashi, T. Takeuchi, K. Komoriya, Y. Uejima, T. Kamimura, *J. Med., chemistry* **1997,** *40* (4), 395-407.
6. M. McLaughlin, *Org. Lett.*, **2005**, *7*, 4875-4878.
7. D. G. Yu, X. Wang, R. Y. Zhu, S. Luo, X. B. Zhang, B. Q. Wang, Z. J. Shi, *J. Am. Chem. Soc.*, **2012**, *134*, 14638-14641.
8. T. Suga, Y. Ukaji, *Org. Lett.*, **2018,** *20* (24), 7846-7850.
9. J. R. Schmink, N. E. Leadbeater, *Org. Lett.*, **2009,** *11* (12), 2575-2578.
10. L. Bering, K. Jeyakumar, A. P. Antonchick, *Org. Lett.*, **2018,** *20* (13), 3911-3914.

7. H^1 and C^{13} NMR Spectra of all compounds

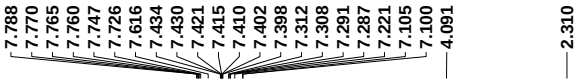


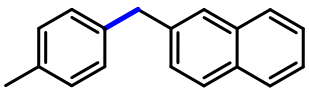
(3a)

```
Current Data Parameters
NAME          Dr.GSK280418
EXPNO                   30
PROCNO                   1

F2 - Acquisition Parameters
Date_              20180428
Time                 20.27 h
INSTRUM              spect
PROBHD    Z108618_0505 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                4.0894465 sec
RG                   127.79
DW                   62.400 usec
DE                    6.50 usec
TE                   300.5 K
D1             1.00000000 sec
TD0                     1
SFO1           400.2604716 MHz
NUC1                   1H
P1                   14.27 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                  65536
SF             400.2580183 MHz
WDW                    EM
SSB         0
LB                   0.30 Hz
GB          0
PC                   1.00
```

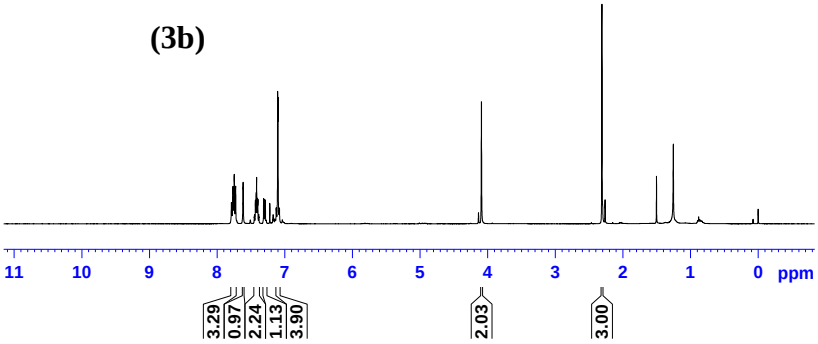


```
Current Data Parameters
NAME          Dr.GSK280418
EXPNO                   31
PROCNO                   1

F2 - Acquisition Parameters
Date_              20180428
Time                 20.57 h
INSTRUM              spect
PROBHD    Z108618_0505 (
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH              24038.461 Hz
FIDRES               0.733596 Hz
AQ                1.3631488 sec
RG                   112.69
DW                   20.800 usec
DE                    6.50 usec
TE                   301.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1           100.6550186 MHz
NUC1                   13C
P1                    9.80 usec
PLW1          58.00000000 W
SFO2           400.2596010 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          15.00000000 W
PLW12          0.37709999 W
PLW13          0.18968000 W

F2 - Processing parameters
SI                  32768
SF             100.6449542 MHz
WDW                    EM
SSB         0
LB                   1.00 Hz
GB          0
PC                   1.40
```





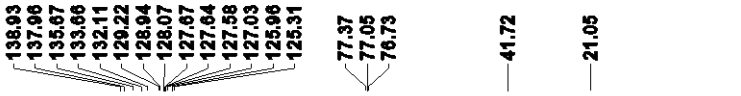
(3b)

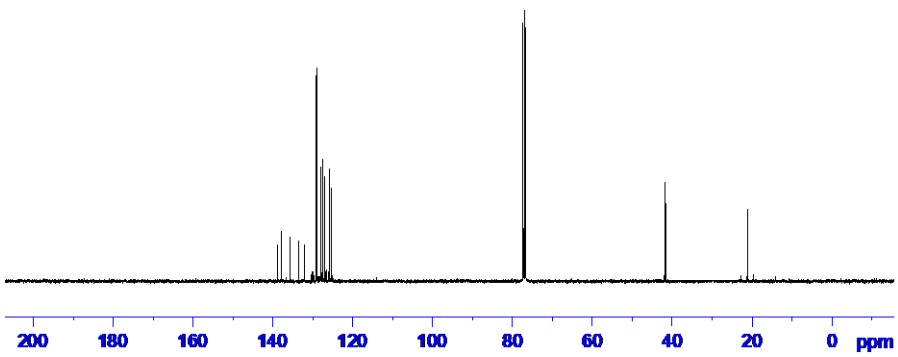


Current Data Parameters
NAME Dr.GSK050418
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180405
Time 23.00 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 88.69
DW 62.400 usec
DE 6.50 usec
TE 301.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.27 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2580253 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

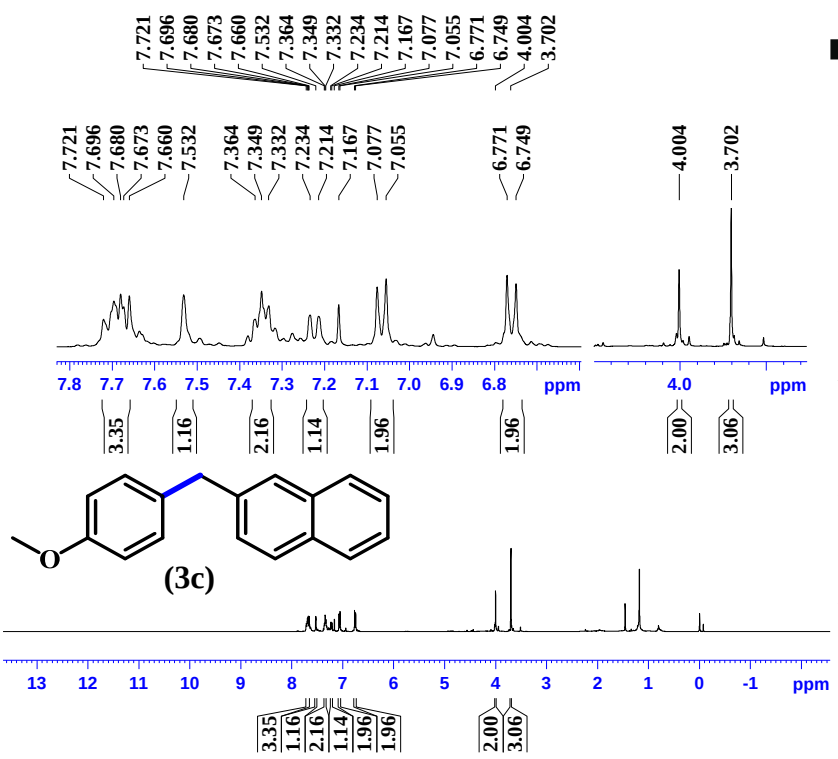




Current Data Parameters
NAME Dr.GSK050418
EXPNO 2
PROCNO 1

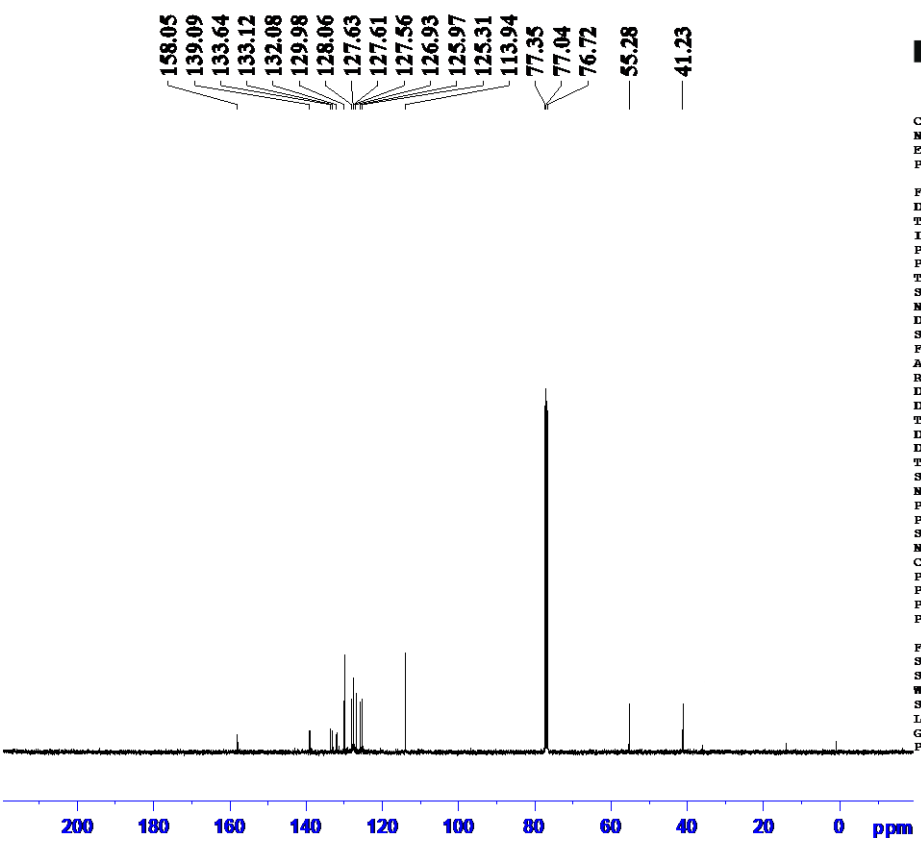
F2 - Acquisition Parameters
Date_ 20180405
Time 23.30 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 199.6
DW 20.800 usec
DE 6.50 usec
TE 302.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.37709999 W
PLW13 0.18968000 W

F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



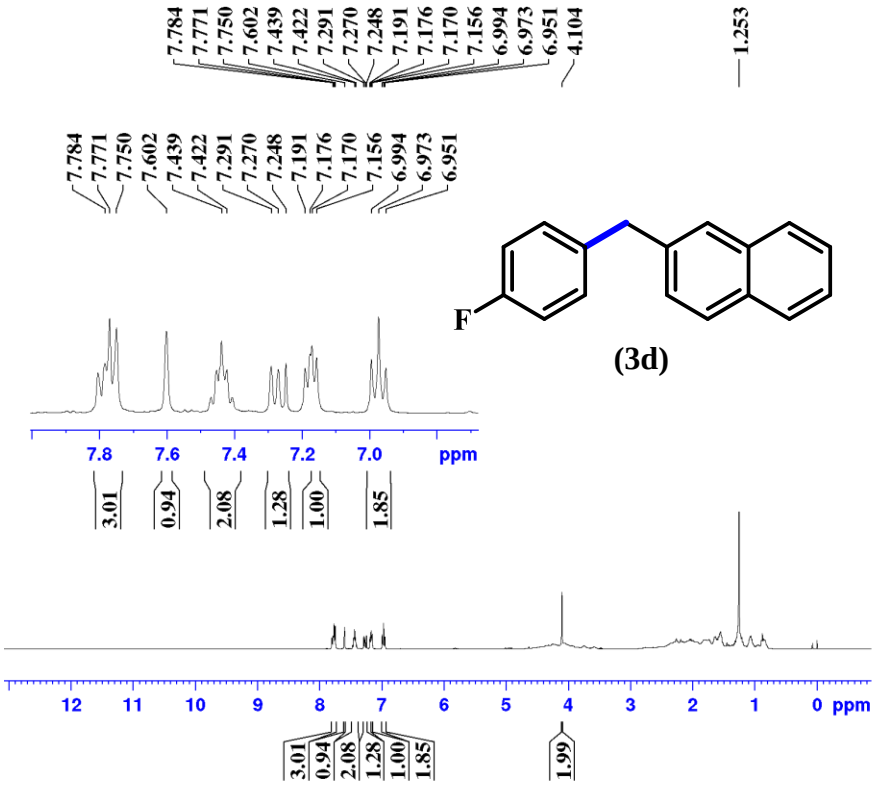
BRUKER

```
Current Data Parameters
NAME        Dr.GSK110418
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180411
Time              14.29 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              4.0894465 sec
RG                 112.69
DW                 62.400 usec
DE                  6.50 usec
TE                 301.5 K
D1              1.00000000 sec
TD0                    1
SFO1           400.2604716 MHz
NUC1                   1H
P1                 14.27 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                 65536
SF             400.2580473 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



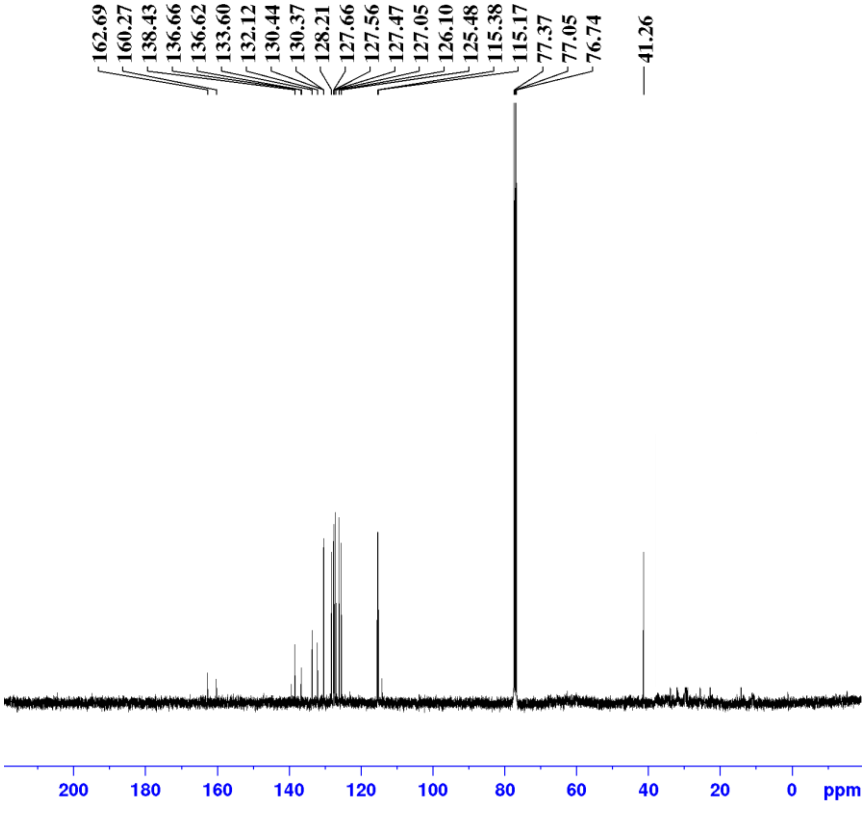
BRUKER

```
Current Data Parameters
NAME        Dr.GSK110418
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180411
Time              14.58 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES             0.733596 Hz
AQ              1.3631488 sec
RG                 199.6
DW                 20.800 usec
DE                  6.50 usec
TE                 301.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1           100.6550186 MHz
NUC1                   13C
P1                  9.80 usec
PLW1           58.00000000 W
SFO2           400.2596010 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2              90.00 usec
PLW2           15.00000000 W
PLW12           0.37709999 W
PLW13           0.18968000 W

F2 - Processing parameters
SI                 32768
SF             100.6449542 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



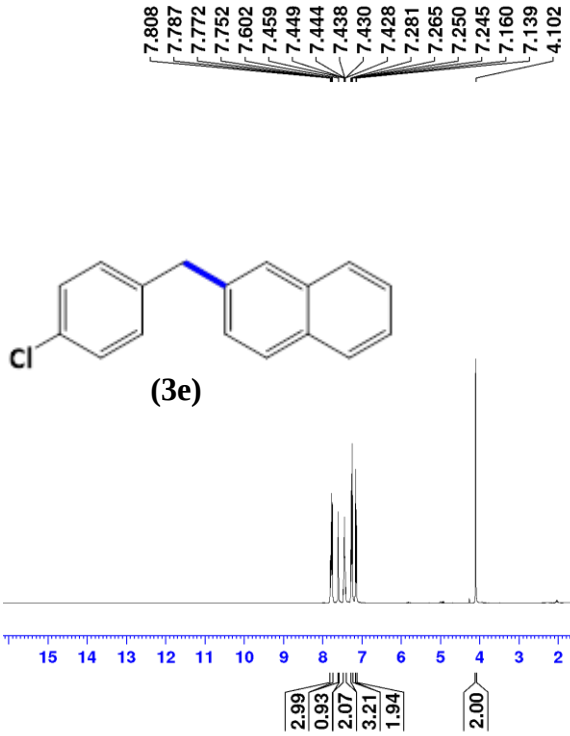
BRUKER

```
Current Data Parameters
NAME        Dr.GSK040318
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180304
Time              20.48 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              4.0894465 sec
RG                 63.11
DW                62.400 usec
DE                 6.50 usec
TE                297.2 K
D1             1.00000000 sec
TD0                    1
SF01          400.2604716 MHz
NUC1                  1H
P1                14.25 usec
PLW1          14.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.2580148 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



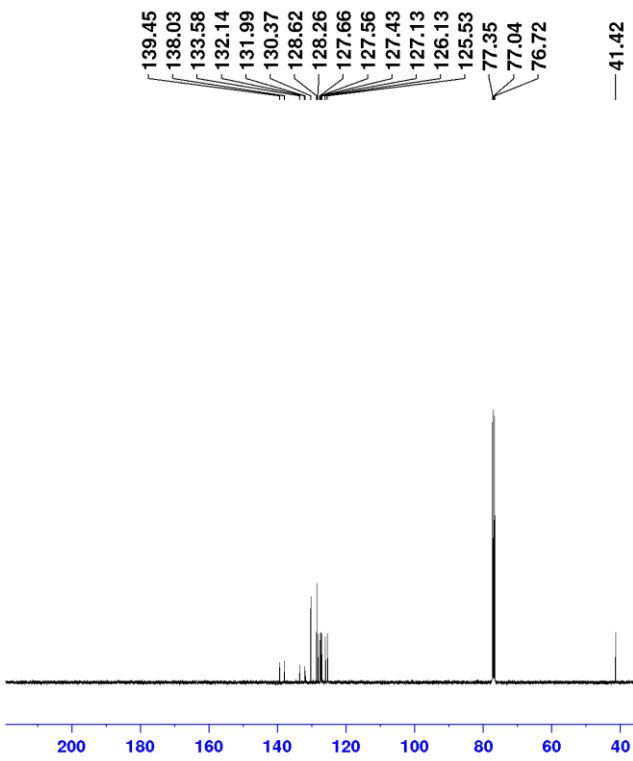
BRUKER

```
Curre
NAME        Dr.GSK040318
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180304
Time              21.19 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES             0.733596 Hz
AQ              1.3631488 sec
RG                 88.69
DW                20.800 usec
DE                 6.50 usec
TE                297.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SF01          100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1          58.00000000 W
SF02          400.2596010 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          14.00000000 W
PLW12          0.35097000 W
PLW13          0.17654000 W

F2 - Processing parameters
SI                 32768
SF            100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



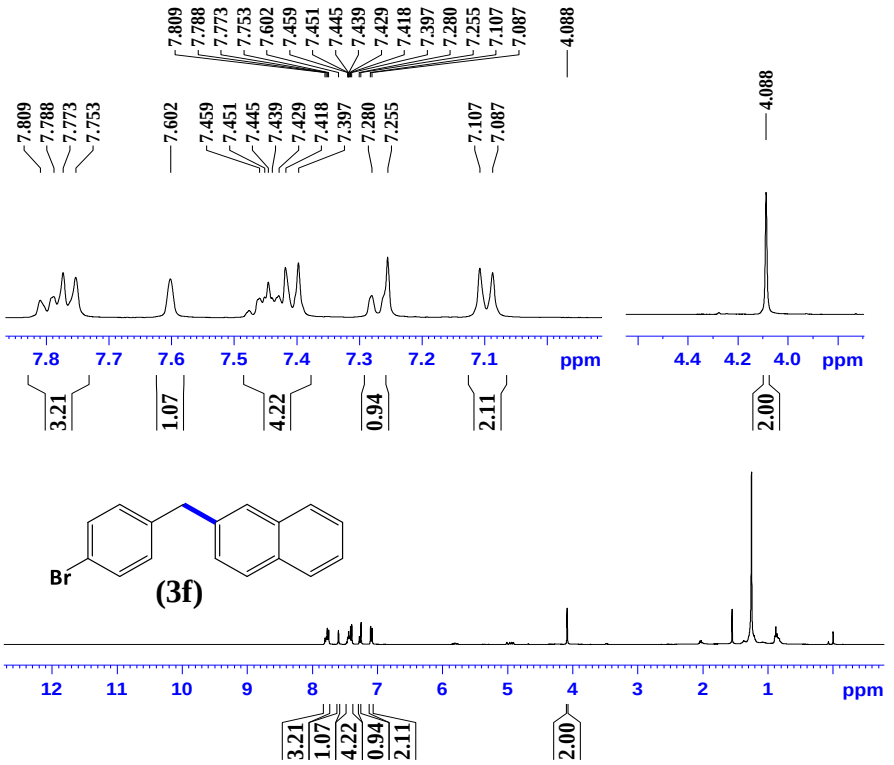
BRUKER

```
Current Data Parameters
NAME        Dr.GSK120317
EXPNO                 18
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180313
Time              11.08 h
INSTRUM             spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                156.91
DW                62.400 usec
DE                 6.50 usec
TE                297.9 K
D1             1.00000000 sec
TD0                    1
SFO1          400.2604716 MHz
NUC1                  1H
P1                14.25 usec
PLW1          14.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.2580136 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

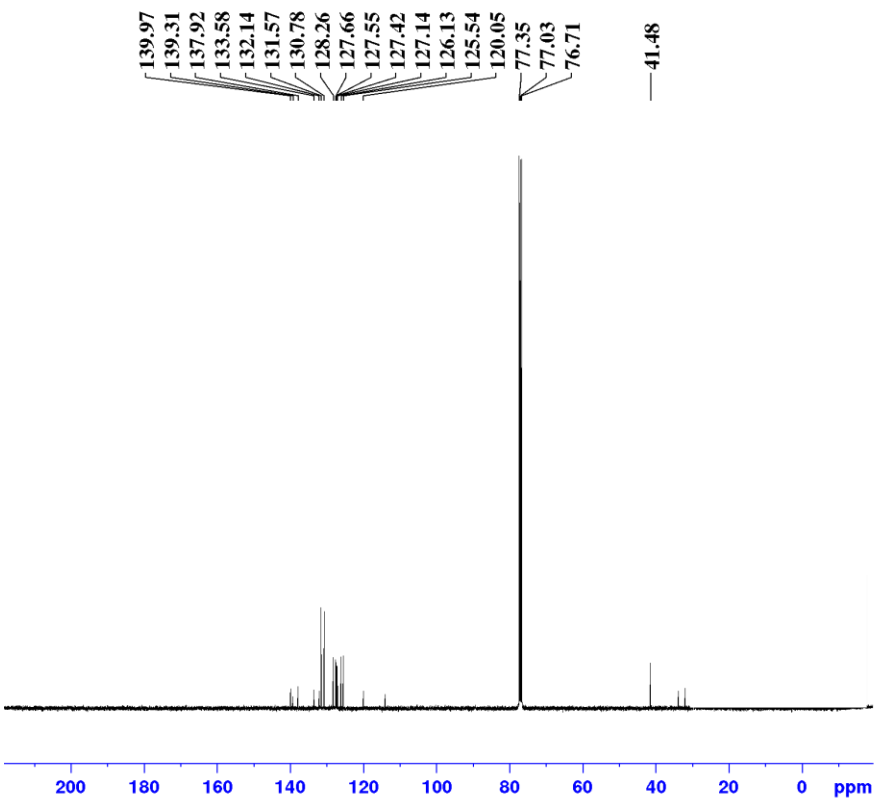


BRUKER

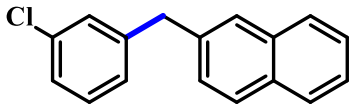
```
Current Data Parameters
NAME        Dr.GSK120317
EXPNO                 19
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180313
Time              11.39 h
INSTRUM             spect
PROBHD   Z108618_0505 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                156.91
DW                20.800 usec
DE                 6.50 usec
TE                298.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1          58.00000000 W
SFO2          400.2596010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          14.00000000 W
PLW12          0.35097000 W
PLW13          0.17654000 W

F2 - Processing parameters
SI                 32768
SF            100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



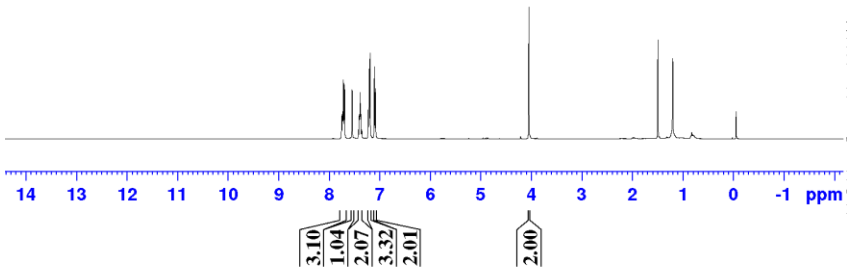

```
Current Data Parameters
NAME        Dr.GSK180218
EXPNO                 19
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180218
Time              23.18 h
INSTRUM             spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                143.73
DW                62.400 usec
DE                 6.50 usec
TE                297.5 K
D1            1.00000000 sec
TD0                    1
SFO1         400.2604716 MHz
NUC1                  1H
P1                 14.25 usec
PLW1         14.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2580121 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME        Dr.GSK180218
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180219
Time              0.17 h
INSTRUM             spect
PROBHD   Z108618_0505 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                127.79
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1         100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1         58.00000000 W
SFO2         400.2596010 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.35097000 W
PLW13         0.17654000 W

F2 - Processing parameters
SI                 32768
SF           100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



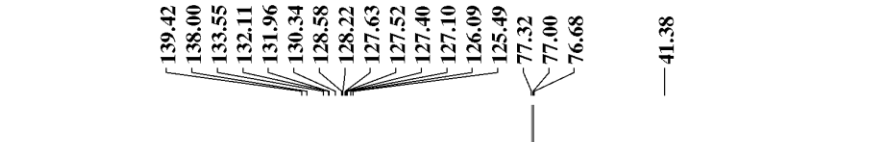
(3g)

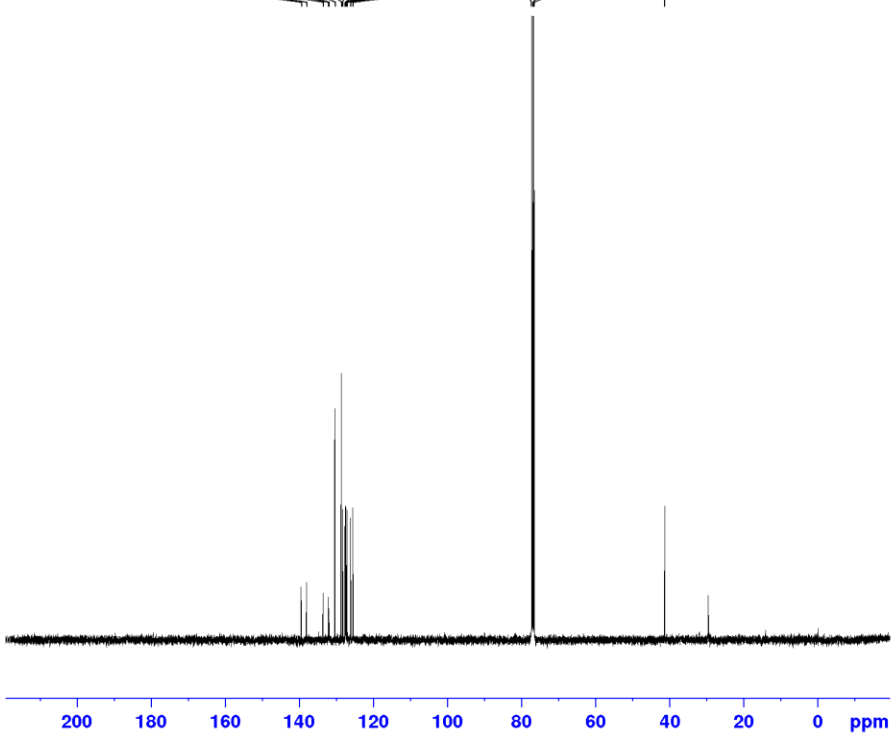


Current Data Parameters
NAME Dr.GSK120317
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180313
Time 11.08 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 156.91
DW 62.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2580352 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

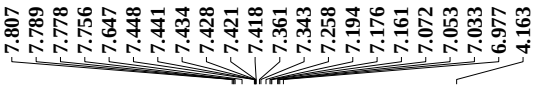


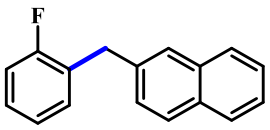


Current Data Parameters
NAME Dr.GSK120317
EXPNO 19
PROCNO 1

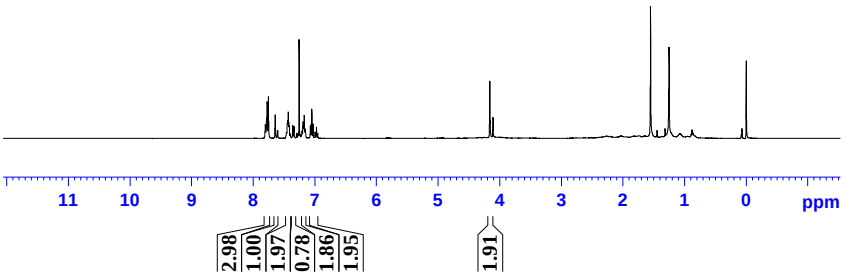
F2 - Acquisition Parameters
Date_ 20180313
Time 11.39 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 156.91
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.17654000 W

F2 - Processing parameters
SI 32768
SF 100.6449577 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

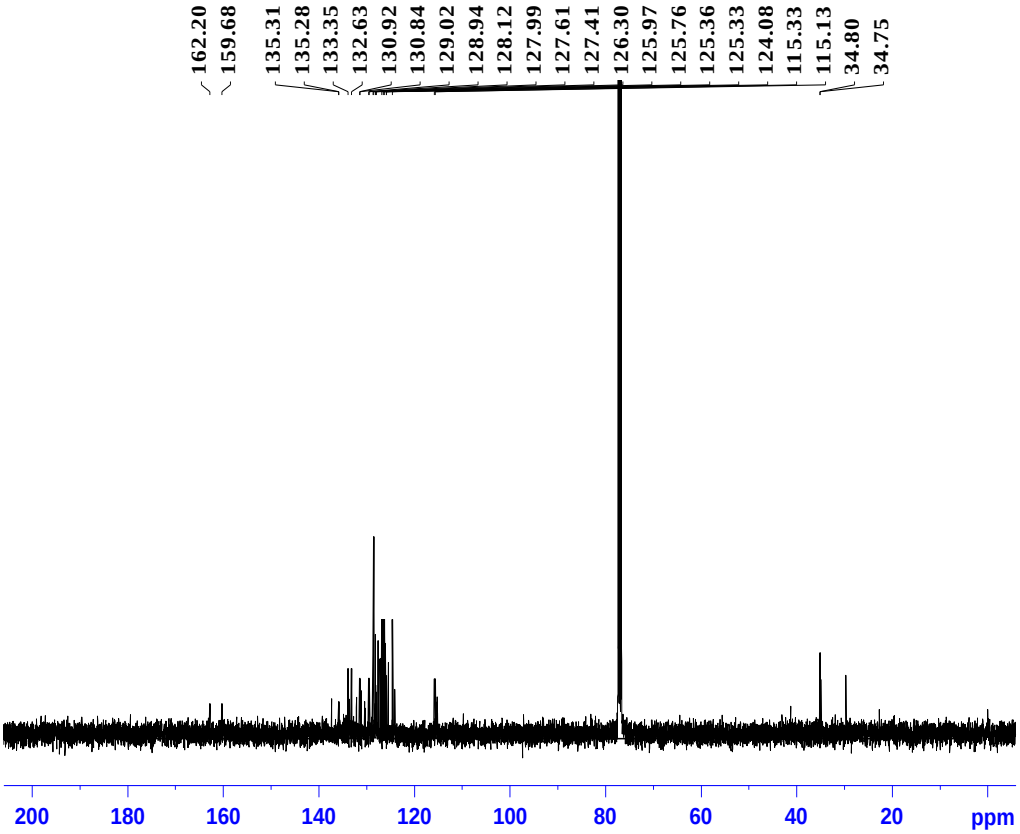




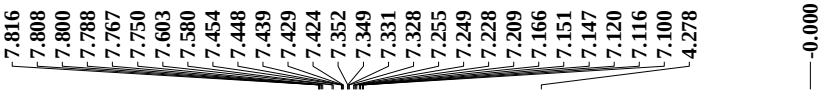
(3h)

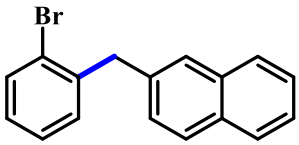



```
Current Data Parameters
NAME        Dr.GSK291118
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181130
Time               9.14 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             4.0894465 sec
RG                175.97
DW                62.400 usec
DE                 6.50 usec
TE                297.7 K
D1             1.00000000 sec
TD0                    1
SFO1         400.2604716 MHz
NUC1                  1H
P1                14.00 usec
PLW1         16.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2580105 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

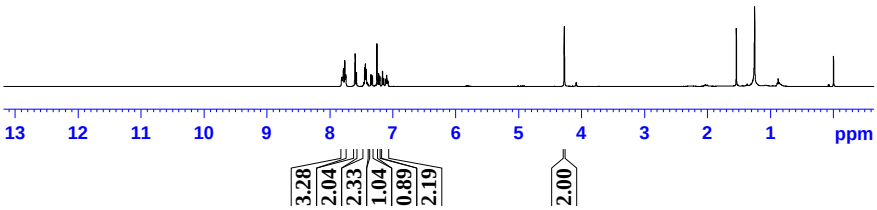



```
Current Data Parameters
NAME      Dr.GSK291118
EXPNO             14
PROCNO             1

F2 - Acquisition Parameters
Date_          20181130
Time              9.44 h
INSTRUM           spect
PROBHD   Z108618_0505 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 512
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ           1.3631488 sec
RG                77.73
DW              20.800 usec
DE               6.50 usec
TE               297.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1       100.6550186 MHz
NUC1               13C
P1               9.80 usec
PLW1        58.00000000 W
SFO2       400.2596010 MHz
NUC2               1H
CPDPRG[2       waltz16
PCPD2           90.00 usec
PLW2        16.00000000 W
PLW12        0.38716000 W
PLW13        0.19474000 W

F2 - Processing parameters
SI                 32768
SF         100.6449542 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

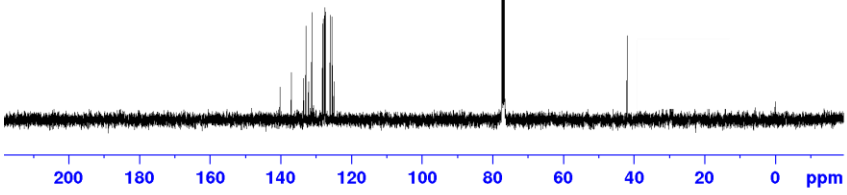




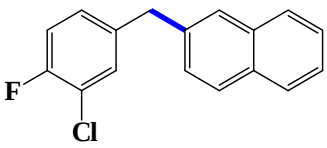
(3i)



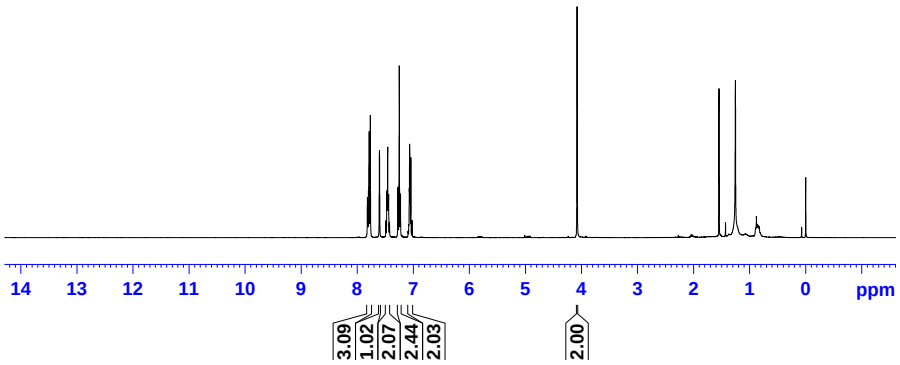

```
Current Data Parameters
NAME        Dr.GSK060818
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180806
Time              19.48 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                175.97
DW                62.400 usec
DE                 6.50 usec
TE                299.4 K
D1             1.00000000 sec
TD0                    1
SFO1          400.2604716 MHz
NUC1                  1H
P1                14.27 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.2580117 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME        Dr.GSK060818
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180806
Time              20.18 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                156.91
DW                20.800 usec
DE                 6.50 usec
TE                299.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1          58.00000000 W
SFO2          400.2596010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          15.00000000 W
PLW12          0.37709999 W
PLW13          0.18968000 W

F2 - Processing parameters
SI                 32768
SF            100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



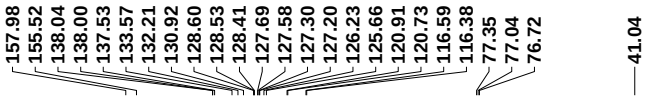
(3j)

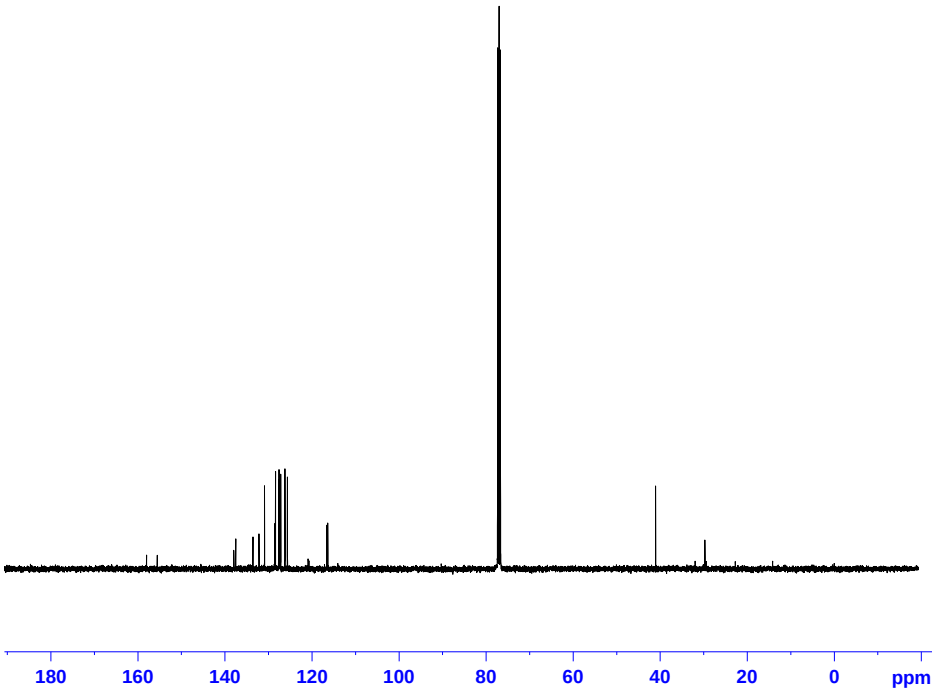


Current Data Parameters
NAME Dr.GSK030219
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190203
Time 21.36 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 143.73
DW 62.400 usec
DE 6.50 usec
TE 306.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.00 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2580133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

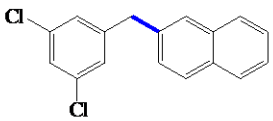




Current Data Parameters
NAME Dr.GSK030219
EXPNO 33
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190203
Time 22.06 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 77.73
DW 20.800 usec
DE 6.50 usec
TE 307.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.38716000 W
PLW13 0.19474000 W

F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

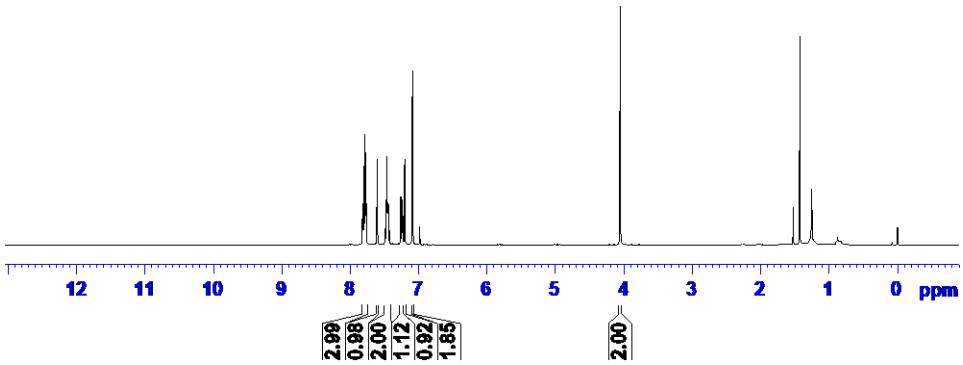


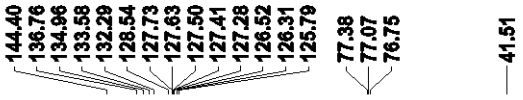
(3k)

Current Data Parameters
NAME Dr.GSK030219
EXPNO 28
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190203
Time 20.27 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 77.73
DW 62.400 usec
DE 6.50 usec
TE 306.8 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.00 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2580224 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

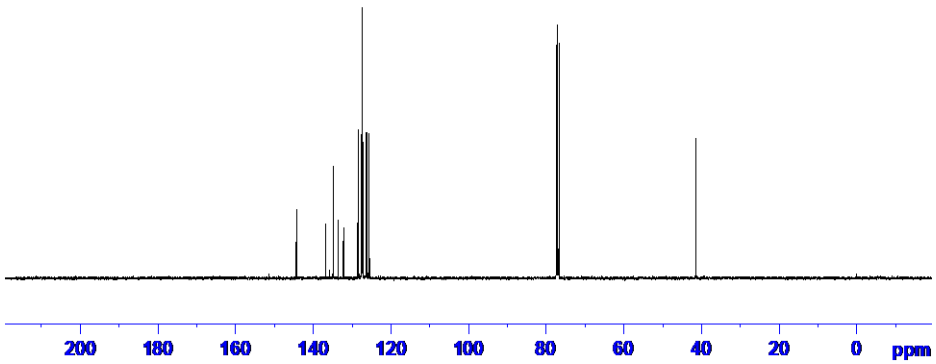


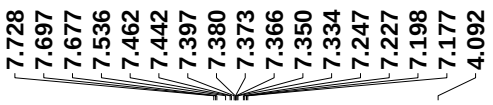


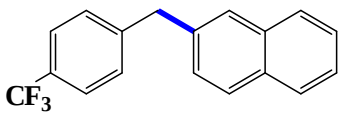
Current Data Parameters
NAME Dr.GSK030219
EXPNO 29
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190203
Time 20.57 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 143.73
DW 20.800 usec
DE 6.50 usec
TE 307.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.38716000 W
PLW13 0.19474000 W

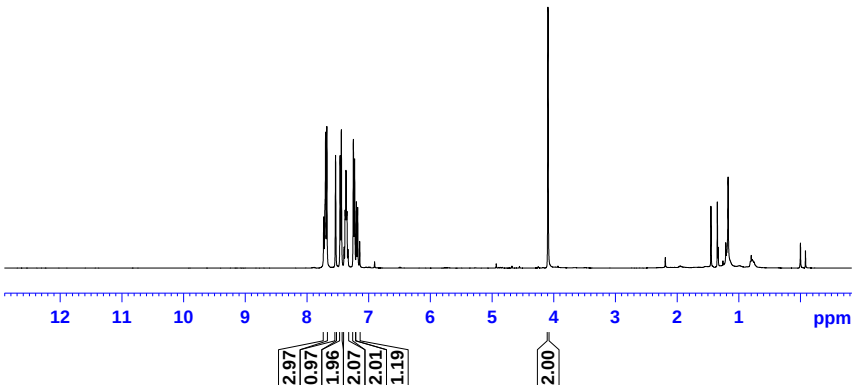
F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







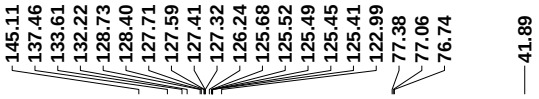
(3l)

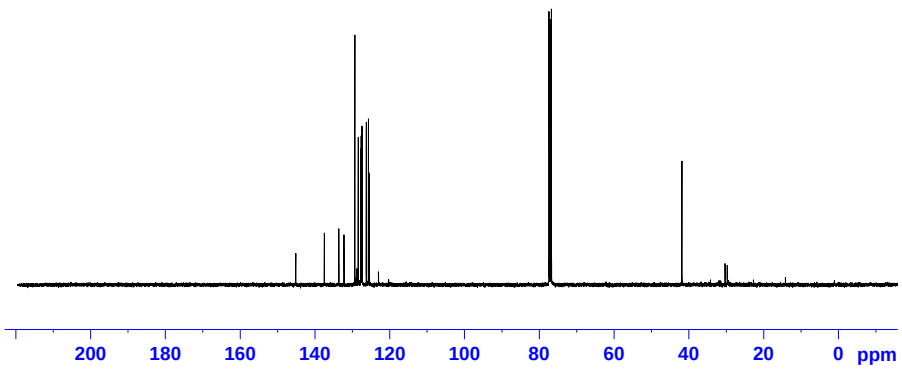


Current Data Parameters
NAME Dr.GSK280119
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190129
Time 0.04 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 77.73
DW 62.400 usec
DE 6.50 usec
TE 307.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.00 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2580559 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

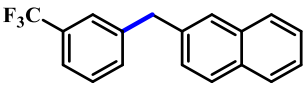




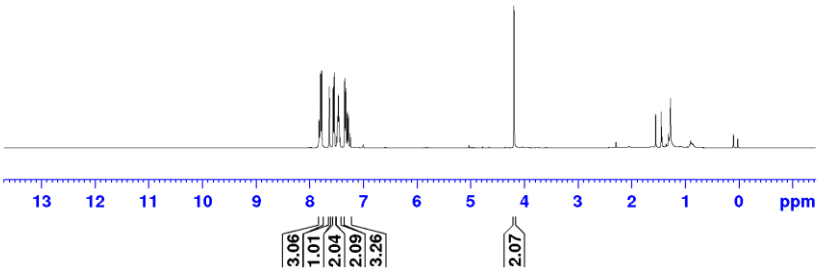
Current Data Parameters
NAME Dr.GSK280119
EXPNO 22
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190129
Time 0.34 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 127.79
DW 20.800 usec
DE 6.50 usec
TE 308.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.38716000 W
PLW13 0.19474000 W

F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



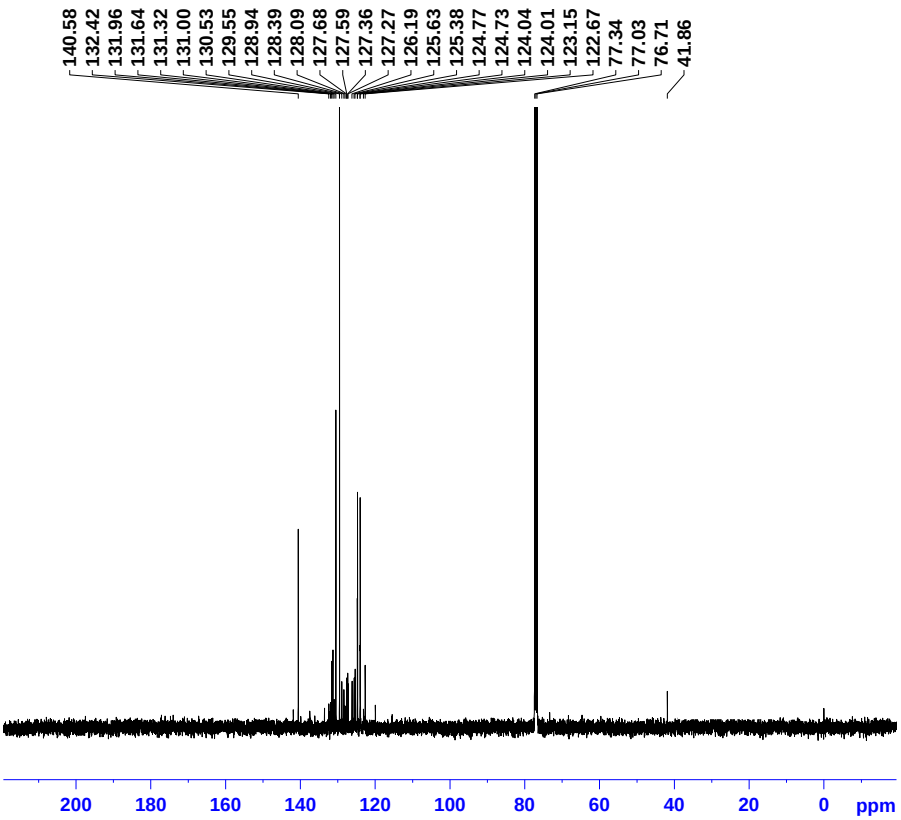
(3m)



Current Data Parameters
NAME Dr.GSK280119
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190129
Time 0.04 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 77.73
DW 62.400 usec
DE 6.50 usec
TE 307.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.00 usec
PLW1 16.00000000 W

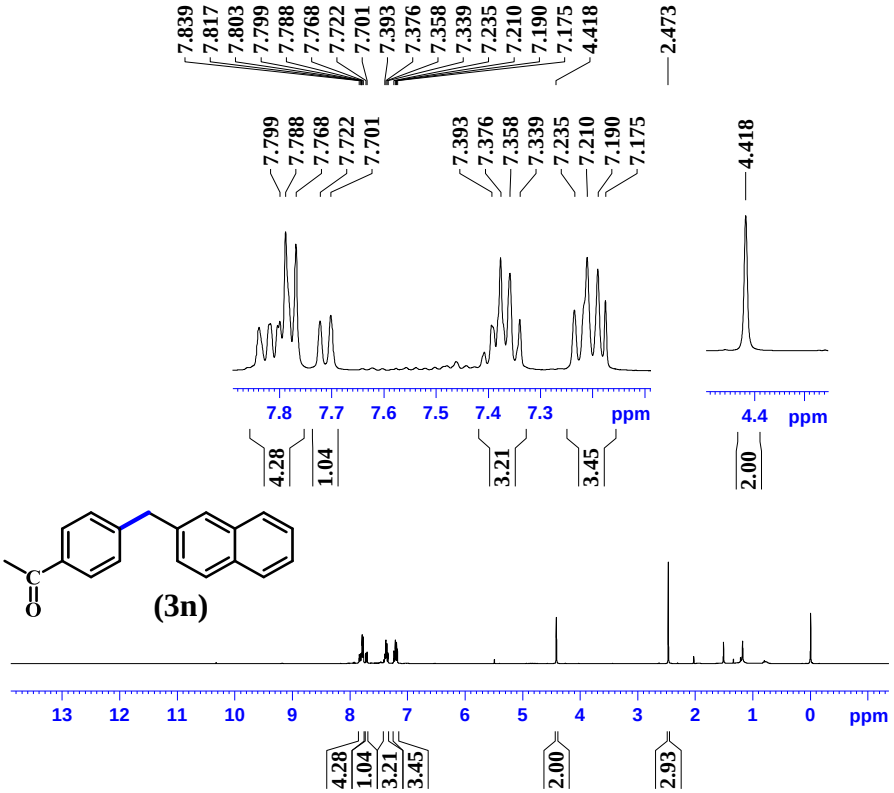
F2 - Processing parameters
SI 65536
SF 400.2580169 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME Dr.GSK220119
EXPNO 16
PROCNO 1

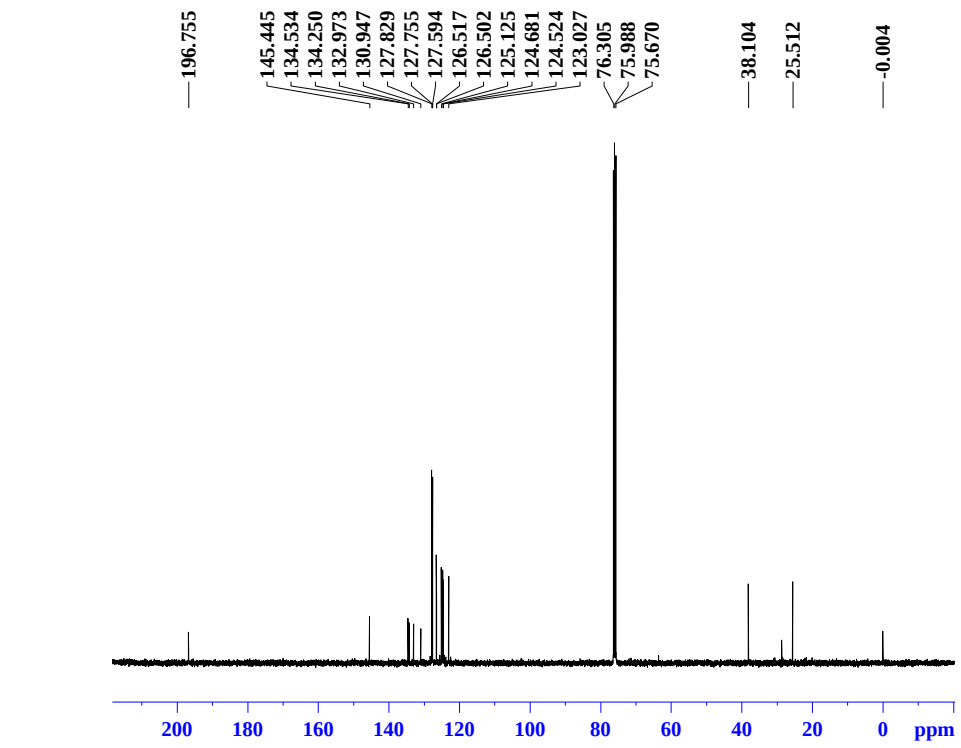
F2 - Acquisition Parameters
Date_ 20190123
Time 10.16 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 143.73
DW 20.800 usec
DE 6.50 usec
TE 311.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.38716000 W
PLW13 0.19474000 W

F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



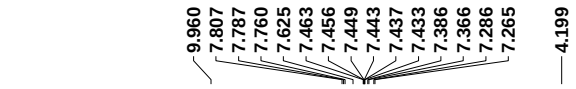
BRUKER

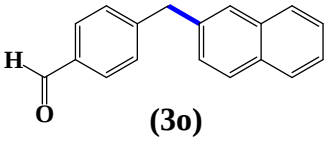
```
Current Data Parameters
NAME        Dr.GSK020719
EXPNO                 55
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190702
Time              23.57 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              4.0894465 sec
RG                 127.79
DW                 62.400 usec
DE                   6.50 usec
TE                 300.5 K
D1              1.00000000 sec
TD0                    1
SFO1           400.2604716 MHz
NUC1                  1H
P1                 14.00 usec
PLW1           16.00000000 W

F2 - Processing parameters
SI                 65536
SF             400.2580436 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                 1.00
```



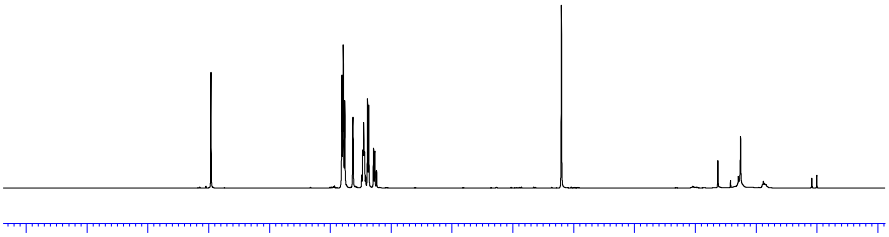
BRUKER

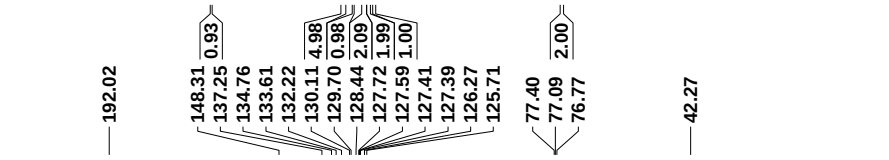
```
Current Data Parameters
NAME        Dr.GSK020719
EXPNO                 56
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190703
Time               0.29 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES             0.733596 Hz
AQ              1.3631488 sec
RG                 175.97
DW                 20.800 usec
DE                   6.50 usec
TE                 301.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1           100.6550186 MHz
NUC1                  13C
P1                   9.80 usec
PLW1           58.00000000 W
SFO2           400.2596010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2           16.00000000 W
PLW12           0.38716000 W
PLW13           0.19474000 W

F2 - Processing parameters
SI                 32768
SF             100.6450595 MHz
WDW                   EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```

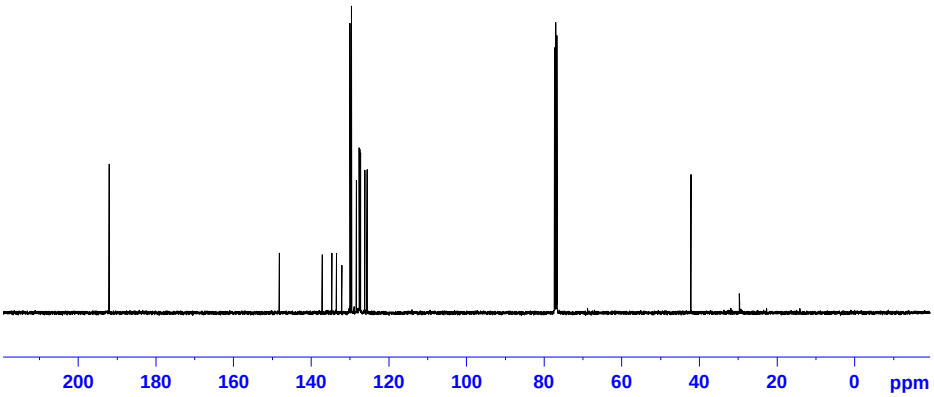




(3o)







Current Data Parameters
NAME Dr.GSK100219
EXPNO 7
PROCNO 1

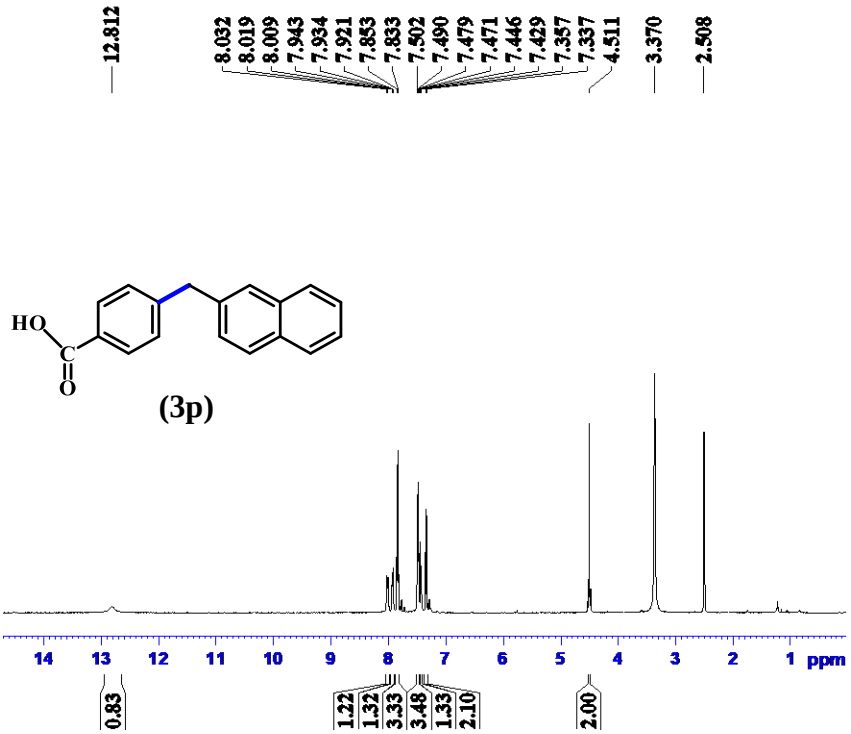
F2 - Acquisition Parameters
Date_ 20190211
Time 6.38 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 77.73
DW 62.400 usec
DE 6.50 usec
TE 303.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.00 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2580192 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME Dr.GSK100219
EXPNO 8
PROCNO 1

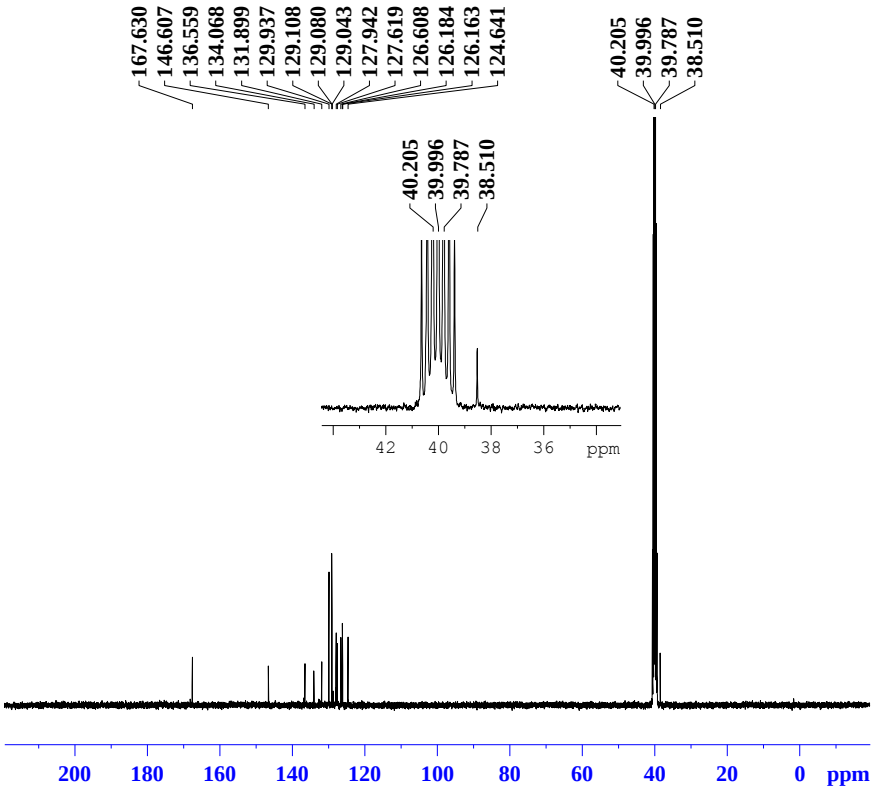
F2 - Acquisition Parameters
Date_ 20190211
Time 7.08 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 112.69
DW 20.800 usec
DE 6.50 usec
TE 303.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.38716000 W
PLW13 0.19474000 W

F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



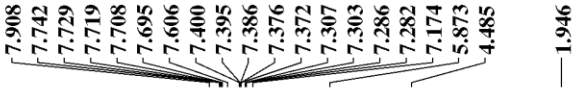
BRUKER

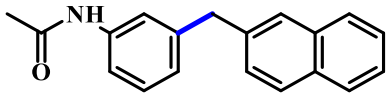
```
Current Data Parameters
NAME        Dr.GSK040719
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190705
Time                0.44 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                 127.79
DW                 62.400 usec
DE                  6.50 usec
TE                 301.0 K
D1              1.00000000 sec
TD0                    1
SFO1           400.2604716 MHz
NUC1                  1H
P1                 14.00 usec
PLW1           16.00000000 W

F2 - Processing parameters
SI                 65536
SF             400.2580000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



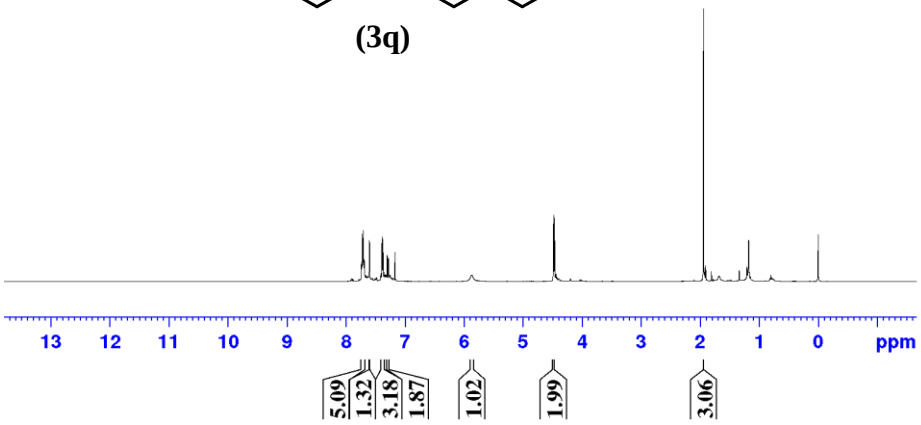
BRUKER

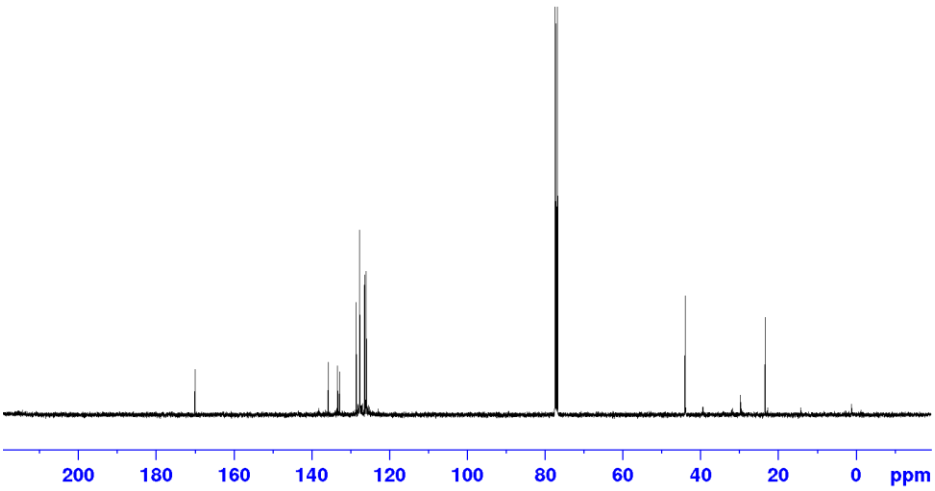
```
Current Data Parameters
NAME        Dr.GSK040719
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190705
Time                1.15 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                    512
DS                      4
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               1.3631488 sec
RG                 199.6
DW                 20.800 usec
DE                  6.50 usec
TE                 301.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1           100.6550186 MHz
NUC1                  13C
P1                  9.80 usec
PLW1           58.00000000 W
SFO2           400.2596010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           16.00000000 W
PLW12           0.38716000 W
PLW13           0.19474000 W

F2 - Processing parameters
SI                 32768
SF             100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





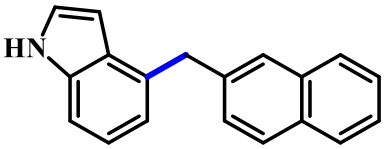
(3q)



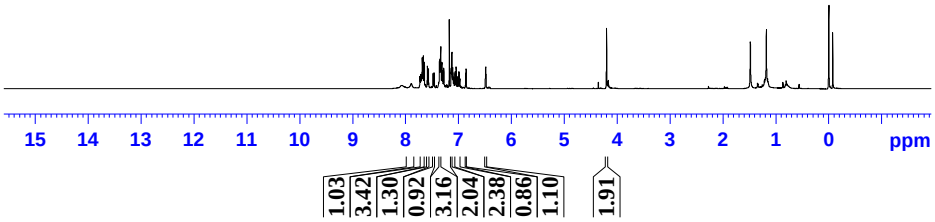


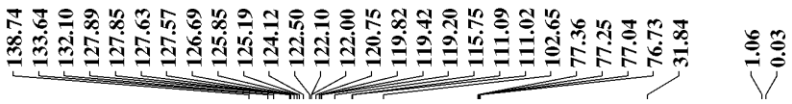

```
Current Data Parameters
NAME        Dr.GSK240617
EXPNO                 25
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190625
Time               2.13 h
INSTRUM             spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 88.69
DW                62.400 usec
DE                 6.50 usec
TE                301.3 K
D1             1.00000000 sec
TD0                    1
SFO1          400.2604716 MHz
NUC1                  1H
P1                 14.00 usec
PLW1          16.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.2580439 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

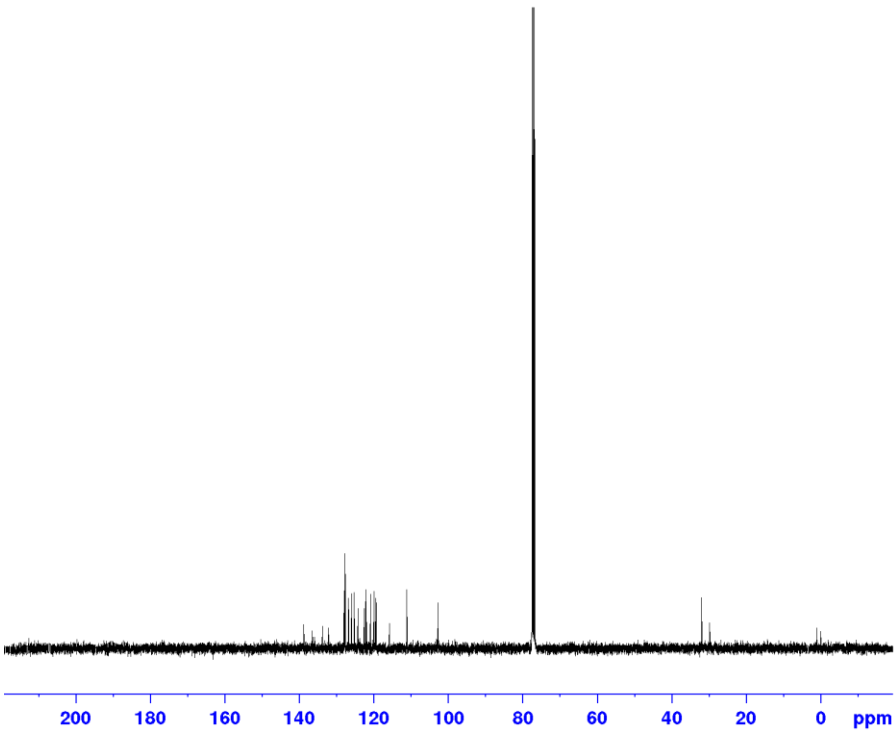

```
Current Data Parameters
NAME        Dr.GSK240617
EXPNO                 26
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190625
Time               2.44 h
INSTRUM             spect
PROBHD   Z108618_0505 (
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                175.97
DW                20.800 usec
DE                 6.50 usec
TE                301.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1          58.00000000 W
SFO2          400.2596010 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          16.00000000 W
PLW12          0.38716000 W
PLW13          0.19474000 W

F2 - Processing parameters
SI                 32768
SF            100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



(3r)

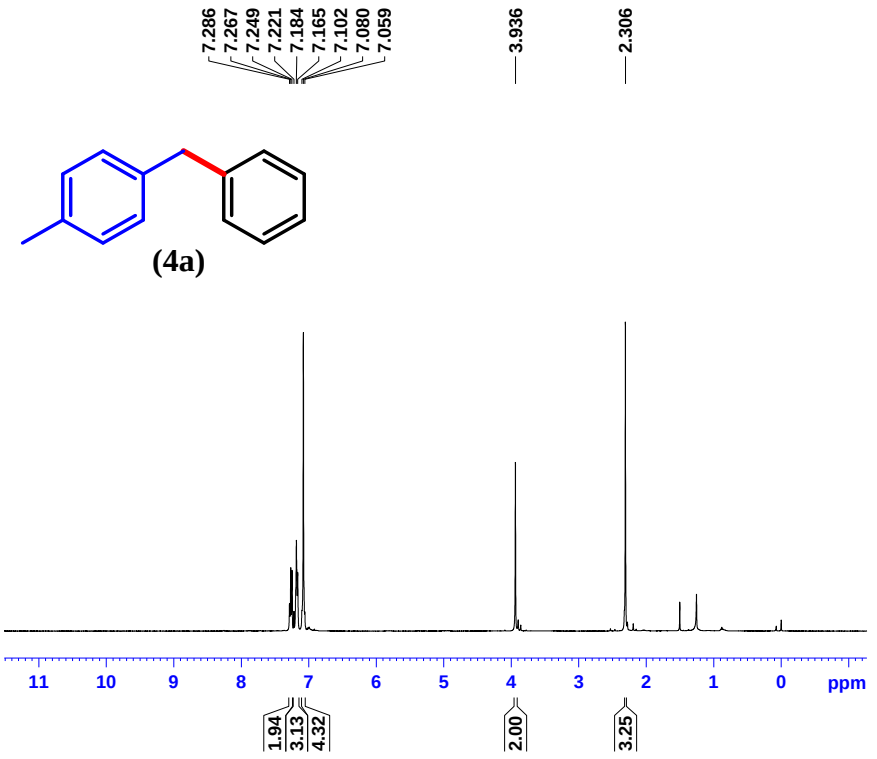




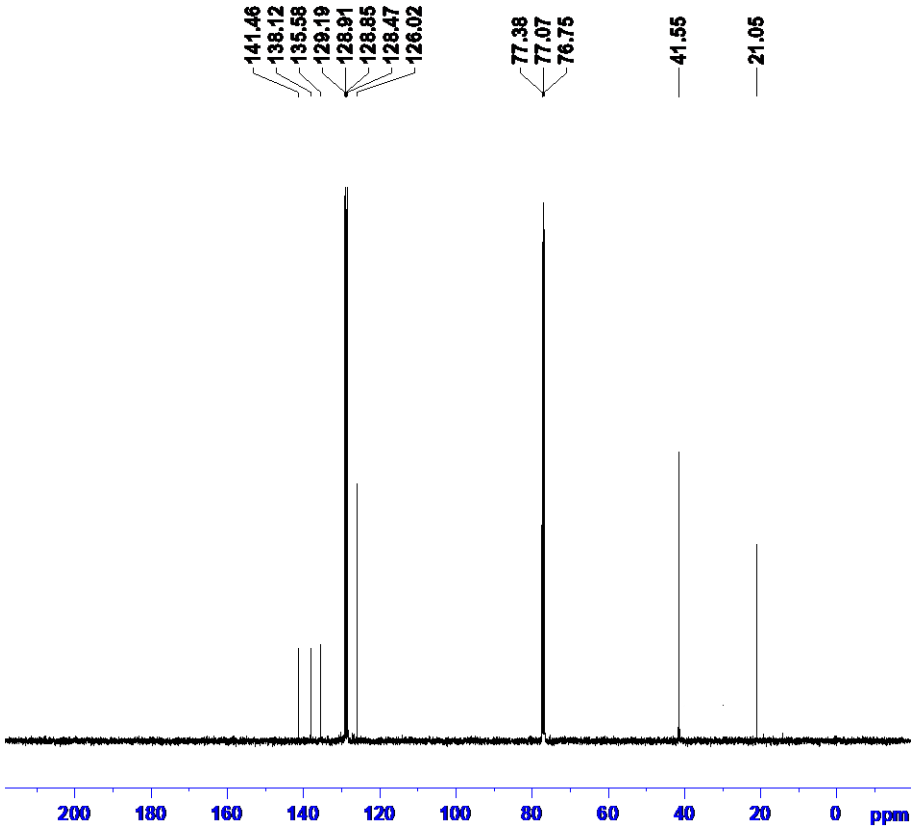



```
Current Data Parameters
NAME        Dr.GSK140818
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180817
Time              14.43 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                156.91
DW                62.400 usec
DE                 6.50 usec
TE                296.9 K
D1           1.00000000 sec
TD0                    1
SFO1         400.2604716 MHz
NUC1                  1H
P1                14.27 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.2580428 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

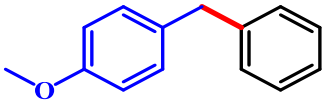

```
Current Data Parameters
NAME        Dr.GSK140818
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180817
Time              15.13 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                143.73
DW                20.800 usec
DE                 6.50 usec
TE                297.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1         58.00000000 W
SFO2         400.2596010 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         15.00000000 W
PLW12         0.37709999 W
PLW13         0.18968000 W

F2 - Processing parameters
SI                 32768
SF          100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



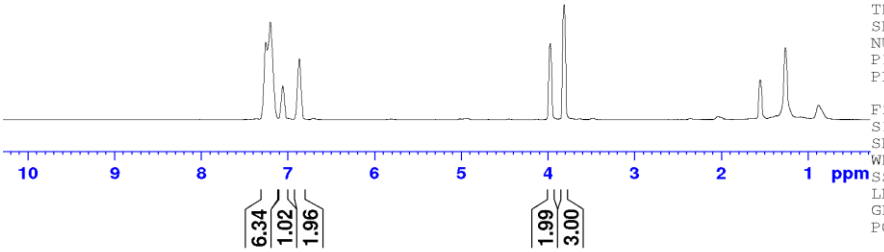

```
Current Data Parameters
NAME        Dr.GSK290618
EXPNO                 17
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180701
Time_               3.30 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 77.73
DW                 62.400 usec
DE                  6.50 usec
TE                299.0 K
D1             1.00000000 sec
TD0                    1
SFO1         400.2604716 MHz
NUC1                  1H
P1                 14.27 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2580251 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



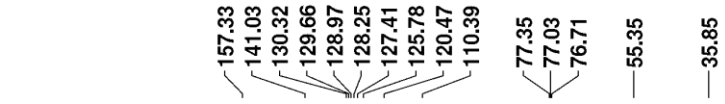

```
Current Data Parameters
NAME        Dr.GSK290618
EXPNO                 18
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180701
Time                4.00 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 143.73
DW                 20.800 usec
DE                  6.50 usec
TE                299.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1         100.6550186 MHz
NUC1                  13C
P1                  9.80 usec
PLW1          58.00000000 W
SFO2         400.2596010 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          15.00000000 W
PLW12          0.37709999 W
PLW13          0.18968000 W

F2 - Processing parameters
SI                 32768
SF           100.6449542 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

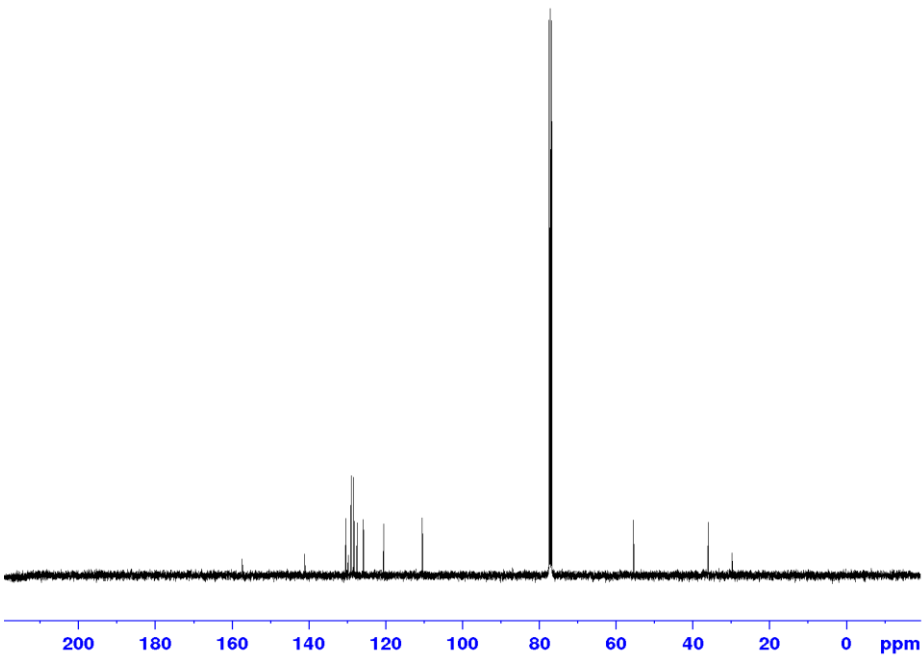


(4b)

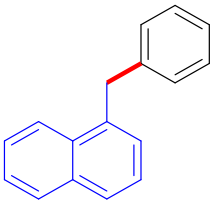


```
Current Data Parameters
NAME         Dr.GSK101117
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20171111
Time                0.20 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              4.0894465 sec
RG                156.91
DW                62.400 usec
DE                 6.50 usec
TE                297.7 K
D1             1.00000000 sec
TD0                    1
SFO1          400.2604716 MHz
NUC1                  1H
P1                 14.25 usec
PLW1          14.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.2580123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

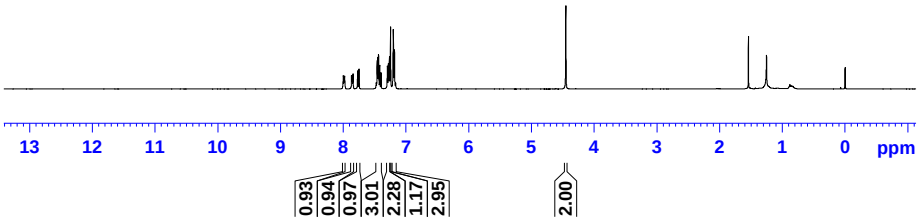




```
Current Data Parameters
NAME         Dr.GSK101117
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20171111
Time                0.50 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES             0.733596 Hz
AQ              1.3631488 sec
RG                143.73
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1          58.00000000 W
SFO2          400.2596010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          14.00000000 W
PLW12          0.35097000 W
PLW13          0.17654000 W

F2 - Processing parameters
SI                 32768
SF            100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



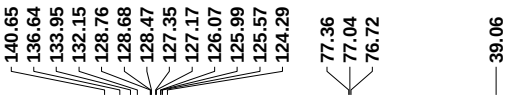
(4c)

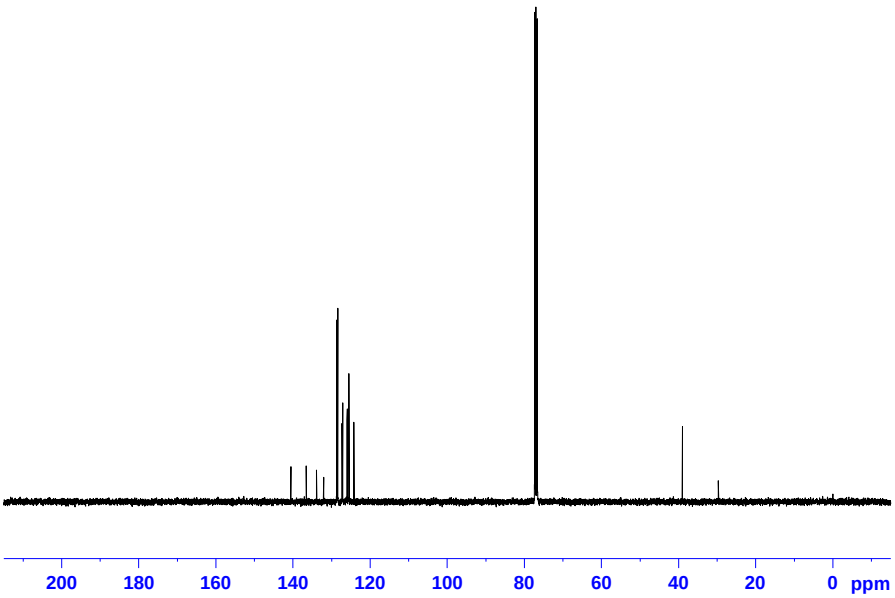


Current Data Parameters
NAME Dr.GSK040219
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190205
Time 11.43 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 143.73
DW 62.400 usec
DE 6.50 usec
TE 305.5 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.00 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2580144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

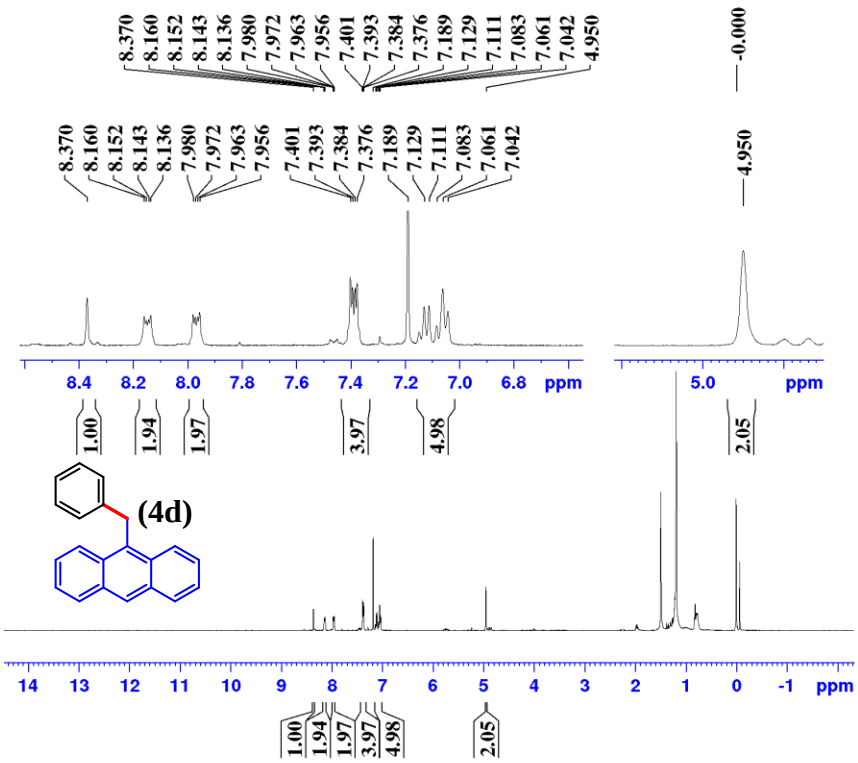




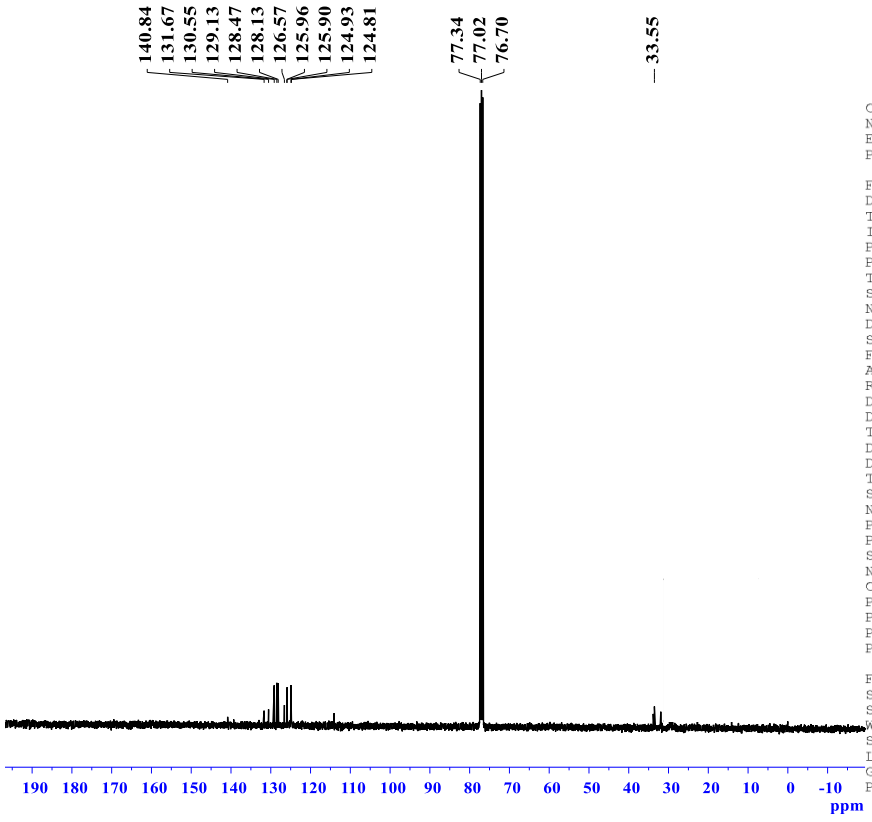
Current Data Parameters
NAME Dr.GSK060219
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190205
Time 21.51 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 88.69
DW 20.800 usec
DE 6.50 usec
TE 306.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.38716000 W
PLW13 0.19474000 W

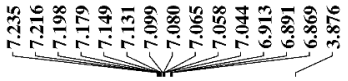
F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

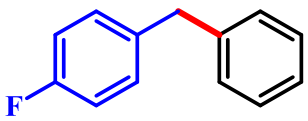


```
Current Data Parameters
NAME        Dr.GSK250217
EXPNO                 38
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170225
Time              10.27 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES            0.244532 Hz
AQ             4.0894465 sec
RG                 199.6
DW                62.400 usec
DE                 6.50 usec
TE                297.9 K
D1             1.00000000 sec
TD0                   1
SFO1          400.2604716 MHz
NUC1                 1H
P1                14.25 usec
PLW1          14.00000000 W

F2 - Processing parameters
SI                65536
SF            400.2580381 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

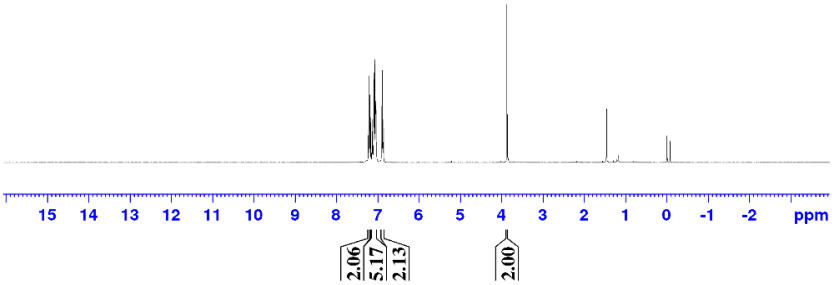


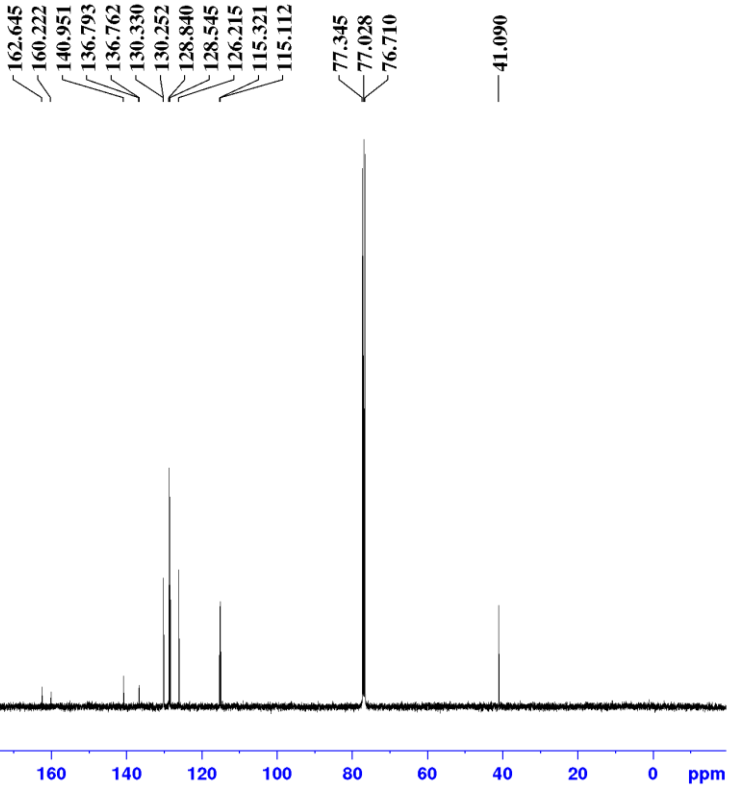
```
Current Data Parameters
NAME        Dr.GSK210518
EXPNO                16
PROCNO                1

F2 - Acquisition Parameters
Date_           20180521
Time              21.38 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES            0.733596 Hz
AQ             1.3631488 sec
RG                 88.69
DW                20.800 usec
DE                 6.50 usec
TE                298.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1          100.6550186 MHz
NUC1                 13C
P1                 9.80 usec
PLW1          58.00000000 W
SFO2          400.2596010 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          15.00000000 W
PLW12          0.37709999 W
PLW13          0.18968000 W

F2 - Processing parameters
SI                32768
SF            100.6449542 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```





(4e)





BRUKER

Current Data Parameters
NAME Dr.GSK230317
EXPNO 17
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170324
Time 9.44 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 156.91
DW 62.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.2604716 MHz
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W

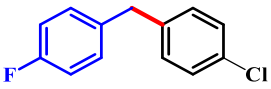
F2 - Processing parameters
SI 65536
SF 400.2580426 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

BRUKER

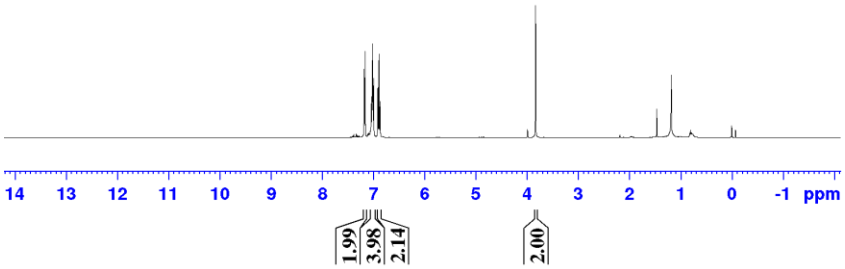
Current Data Parameters
NAME Dr.GSK240317
EXPNO 18
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170324
Time 12.54 h
INSTRUM spect
PROBHD Z108618_0505 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 143.73
DW 20.800 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6550186 MHz
NUC1 13C
P1 9.80 usec
PLW1 58.00000000 W
SFO2 400.2596010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.35097000 W
PLW13 0.17654000 W

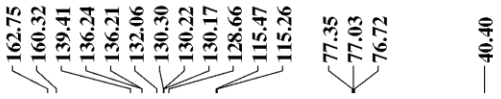
F2 - Processing parameters
SI 32768
SF 100.6449542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

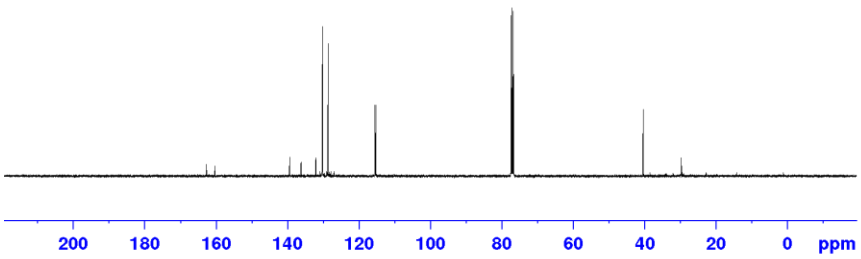


(4f)

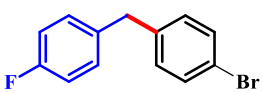



```
Current Data Parameters
NAME         Dr.GSK40419
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190404
Time               8.10 h
INSTRUM           spect
PROBHD   Z108618_0505 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ             4.0894465 sec
RG                98.85
DW               62.400 usec
DE                6.50 usec
TE               299.7 K
D1            1.00000000 sec
TD0                   1
SFO1         400.2604716 MHz
NUC1                 1H
P1                14.00 usec
PLW1         16.00000000 W

F2 - Processing parameters
SI                65536
SF           400.2580451 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

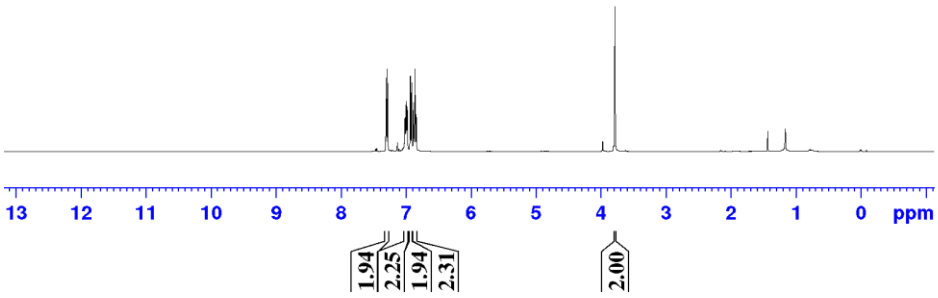




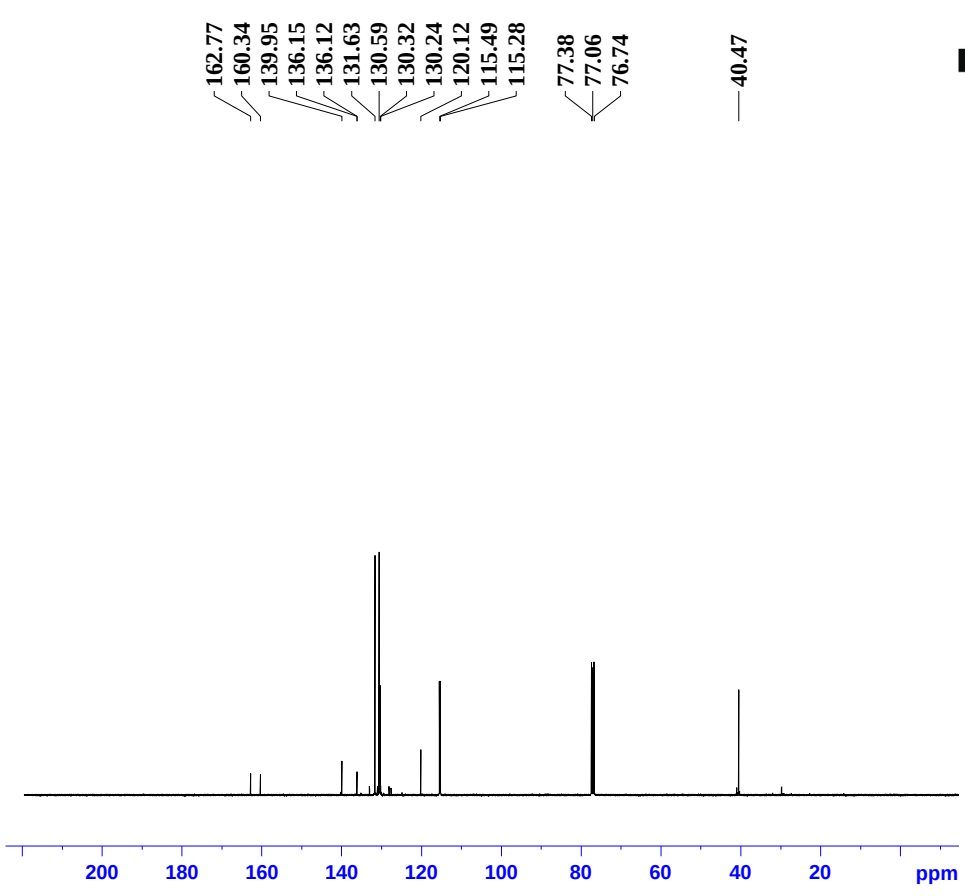

```
Current Data Parameters
NAME         Dr.GSK40419
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190404
Time               8.40 h
INSTRUM           spect
PROBHD   Z108618_0505 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                143.73
DW               20.800 usec
DE                6.50 usec
TE               300.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6550186 MHz
NUC1                 13C
P1                9.80 usec
PLW1         58.00000000 W
SFO2         400.2596010 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2         16.00000000 W
PLW12         0.38716000 W
PLW13         0.19474000 W

F2 - Processing parameters
SI                32768
SF           100.6449542 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



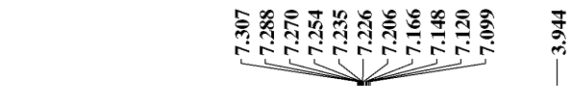
(4g)

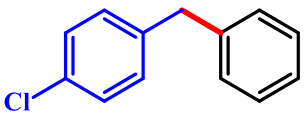



```
Current Data Parameters
NAME        Dr.GSK190419
EXPNO                 16
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190419
Time              10.09 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 71.13
DW                62.400 usec
DE                 6.50 usec
TE                300.5 K
D1             1.00000000 sec
TD0                    1
SFO1          400.2604716 MHz
NUC1                  1H
P1                 14.00 usec
PLW1          16.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2580585 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

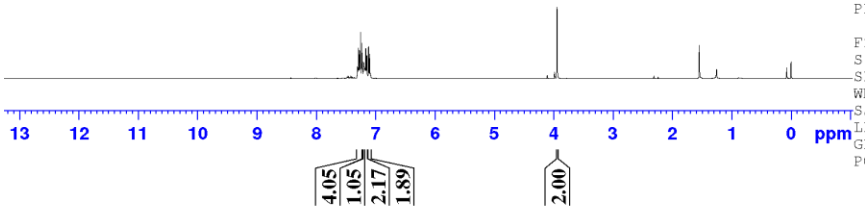



```
Current Data Parameters
NAME        Dr.GSK190419
EXPNO                 17
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190419
Time              14.05 h
INSTRUM            spect
PROBHD   Z108618_0505 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 199.6
DW                20.800 usec
DE                 6.50 usec
TE                301.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6550186 MHz
NUC1                  13C
P1                  9.80 usec
PLW1          58.00000000 W
SFO2          400.2596010 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          16.00000000 W
PLW12          0.38716000 W
PLW13          0.19474000 W

F2 - Processing parameters
SI                 32768
SF           100.6449542 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

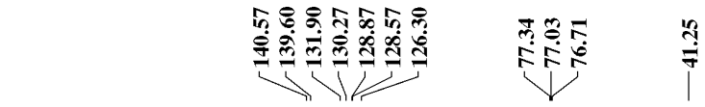


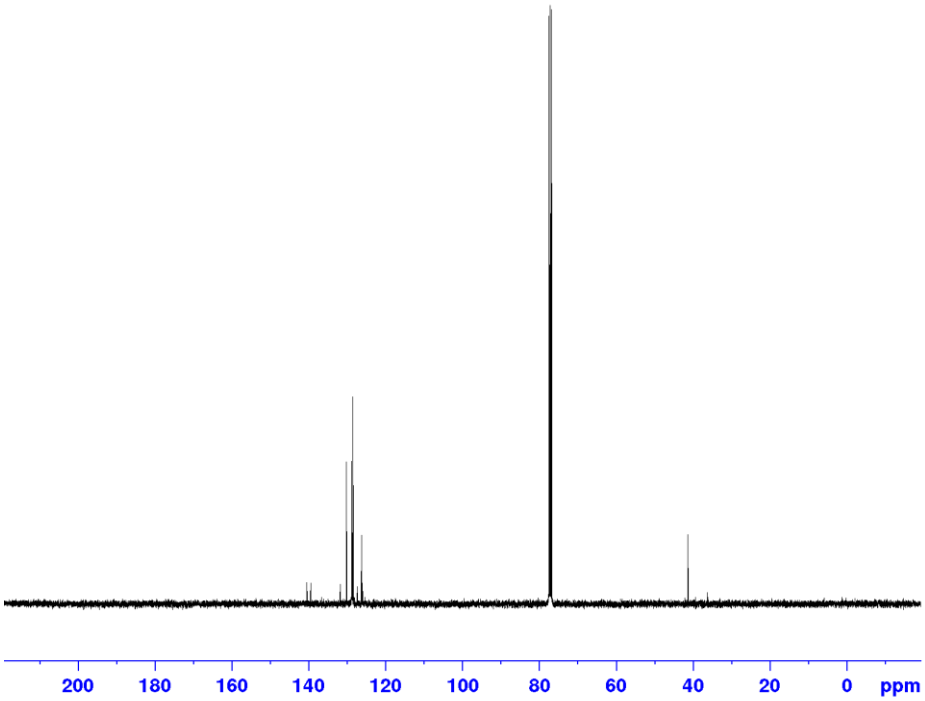


(4h)




```
Current Data Parameters
NAME        Dr.GSK100217
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170211
Time                3.17 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                175.97
DW                62.400 usec
DE                 6.50 usec
TE                298.3 K
D1           1.00000000 sec
TD0                    1
SFO1         400.2604716 MHz
NUC1                  1H
P1                 14.25 usec
PLW1         14.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.2580126 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```






```
Current Data Parameters
NAME        Dr.GSK100217
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170211
Time                3.48 h
INSTRUM            spect
PROBHD    Z108618_0505 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                143.73
DW                20.800 usec
DE                 6.50 usec
TE                298.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1         100.6550186 MHz
NUC1                  13C
P1                 9.80 usec
PLW1         58.00000000 W
SFO2         400.2596010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         14.00000000 W
PLW12         0.35097000 W
PLW13         0.17654000 W

F2 - Processing parameters
SI                 32768
SF          100.6449542 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```