Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supplementary information: "*Click-fluors*": Triazole-linked saccharide sensors

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1. General information

Commercially available solvents and reagents were purchased and used without further purification. ^{1}H NMR spectra were recorded at 300 MHz on a Bruker AVIII300 NMR spectrometer, ^{11}B NMR spectra at 128 MHz on a Varian DirectDrive NMR spectrometer with AUTOXPFG and TRIPLE RESONANCE PFG probes, and also at 128 MHz on a Bruker AVIII400 NMR spectrometer. ^{13}C NMR spectra at 101 MHz on a Bruker AVIII400 NMR spectrometer, ^{19}F NMR spectra at 282 MHz on a Bruker AVIII300 NMR spectrometer were proton decoupled and were recorded at room temperature unless otherwise stated. Data was processed with Mestrec version 5.2.5-4731 and Topspin 2.0 (Version of: Nov 9th 2006). Chemical shifts (δ) are reported in ppm relative to TMS (δ 0.00) for the ^{1}H NMR and to chloroform (δ 77.0) for the ^{13}C NMR spectroscopy, coupling constant *J* are expressed in Hertz, pendant technique was used for some ^{13}C NMR spectral assignment, spectrums thus obtained are shown later. Mass spectra were recorded with electrospray MS Waters LCT Time of Flight Mass Spectrometer and with EI (GC/MS) Waters GCT Premier Time of Flight Mass Spectrometer. Infrared Spectra were recorded on a PerkinElmer 100FT-IR spectrometer at room temperature.

2. Fluorescence studies

Fluorescence spectra were recorded on both Shimadzu RF-5301PC (Shimadzu) and FluoroSENS (Gilden Photonics) fluorimeters. All the measurements were carried out in pH 8.21 methanolic buffer. The buffer was prepared in a 1 L volumetric flask according to a literature procedure[1] and consisted of: 52.1 *wt*% HPLC grade methanol, in deionised water with KCl (0.7456 g, 10.00 mM), KH_2PO_4 (0.3745 g, 2.752 mM) and Na_2HPO_4 (0.3914g, 2.757 mM).

A quartz cuvette with 10 mm path lengths, with four faces polished, was used for fluorescence test. All pH measurements taken during fluorescence experiments were on a pH meter which was calibrated using standard buffer solutions. All solvents used in fluorescence measurements were HPLC or fluorescent grade. All saccharides used in fluorescent measurements were certified as ≥99% pure.

A 1 mM stock solution of the synthesised compound **1a** was prepared in methanol and stored protected from light before spectroscopy measurements. An aliquot of **1a** (50 µL of a 100 µM solution) was added to 2.95 mL of methanolic buffer to give a concentration of 1.67 µM. A known amount of saccharides were added to this solution, ensuring complete dissolution before the fluorescence spectrum was recorded. The saccharide solution was stirred at room temperature for no more than 10 min before each measurement.[2]

2.1 Fluorescence of first generation "*click-fluors*" with monosaccharides

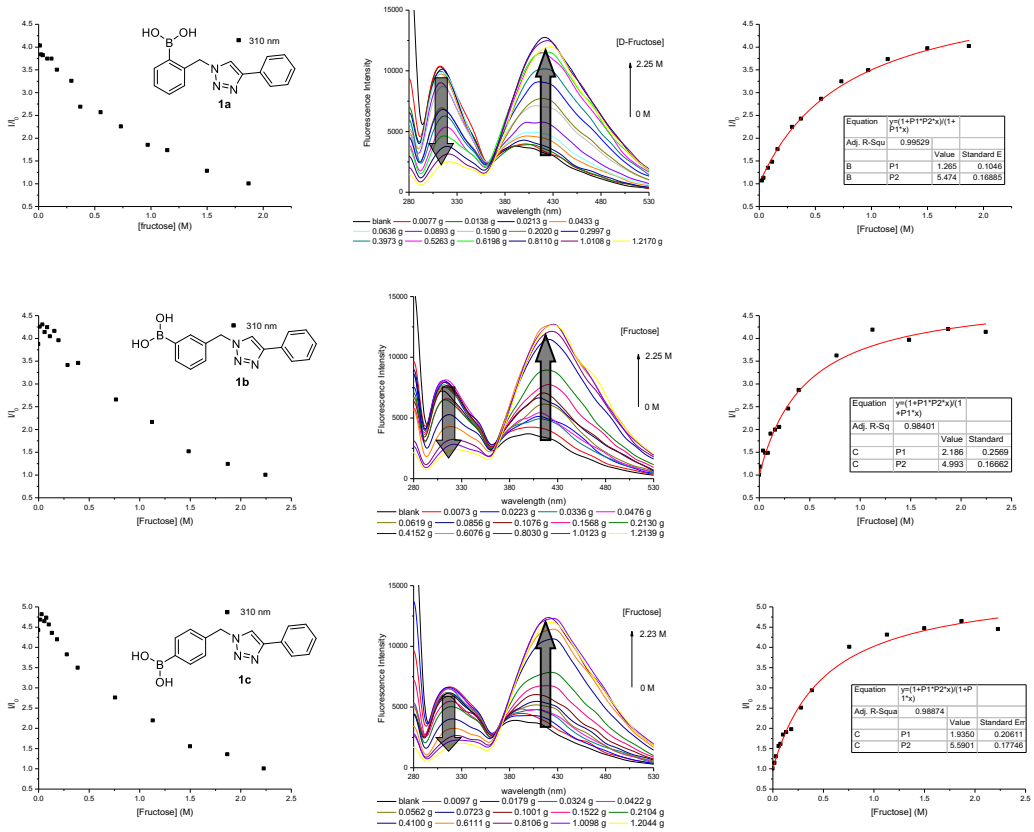


Figure S1. Fluorescence study of "*click-fluors*" **1a-c** binding with D-fructose in pH 8.21 methanolic buffer. Excitation wavelength: 276 nm.

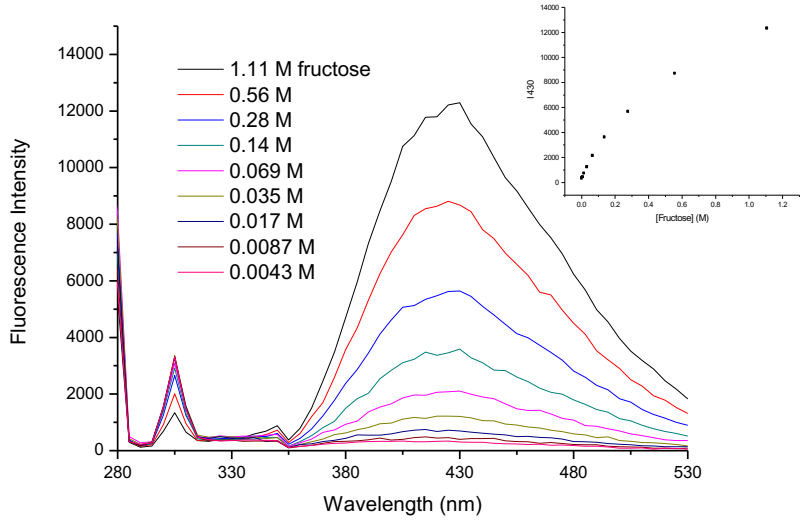


Figure S2. Fluorescence spectra of different concentrations of D-fructose in deionised water. Excitation wavelength: 276 nm.

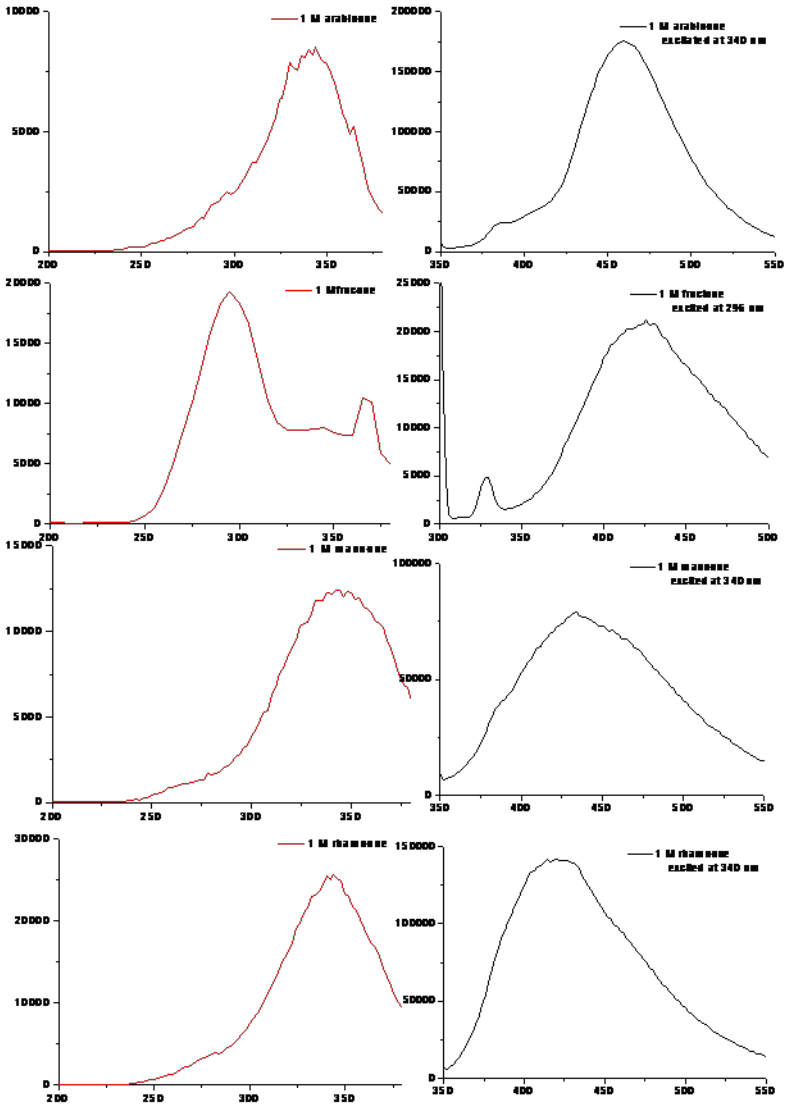


Figure S3. Excitation (left) and emission (right) spectra of 1 M L-arabinose, D-fructose, D-mannose and L-rhamnose in deionised water.

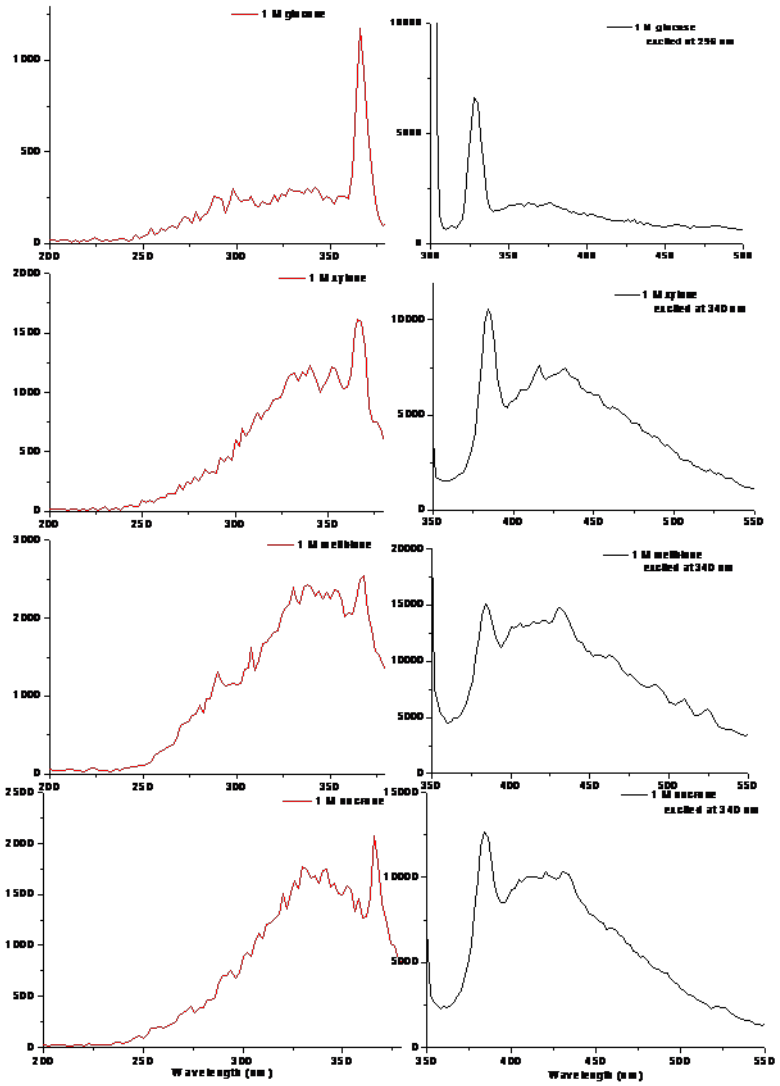


Figure S4. Excitation (left) and emission (right) spectra of 1 M D-glucose, D-xylose, D-melibiose and sucrose in deionised water.

Table S1. Excitation and emission wavelengths of recorded saccharide samples.

Saccharide	Excitation wavelength (nm)	Emission wavelength (nm)
L-arabinose	342	460
D-fructose	295	425
D-mannose	344	434
L-rhamnose	343	421
D-glucose	-	-
D-xylose	-	-
D-melibiose	-	-
sucrose	-	-

2.2 Fluorescence of second generation "*click-fluors*"

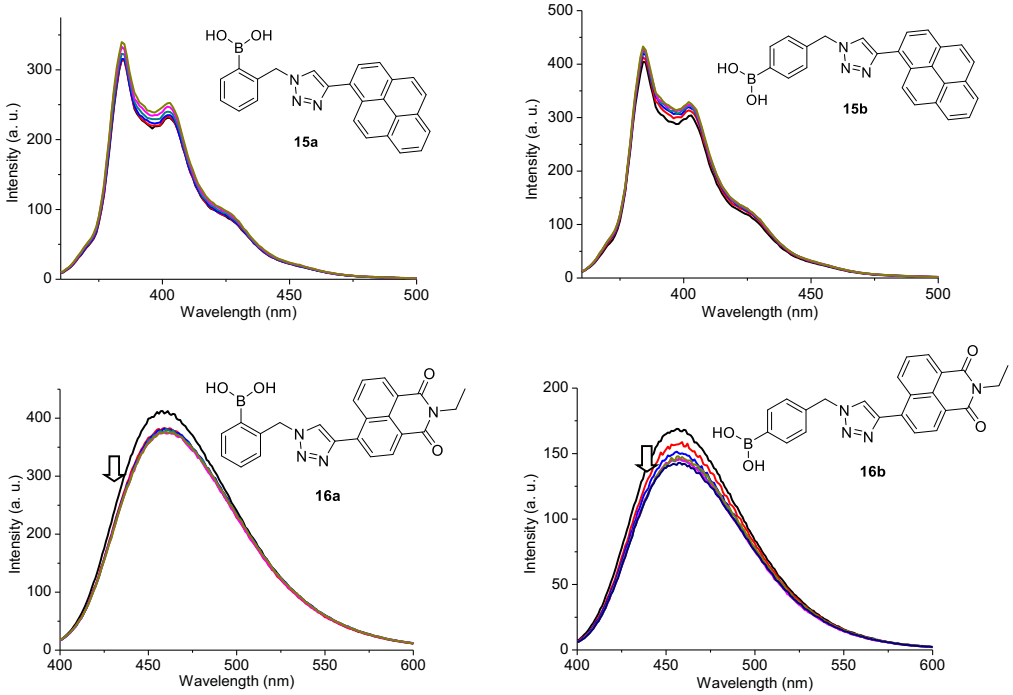


Figure S5. Fluorescence spectra of **15a,b** (top) and **16a,b** (bottom) in the presence of increasing concentration of D-fructose (0-6 mM; black curve: 0 mM, dark yellow curve: 6mM). Excitation wavelength: 339 nm (**15a,b**); 358 nm (**16a,b**).

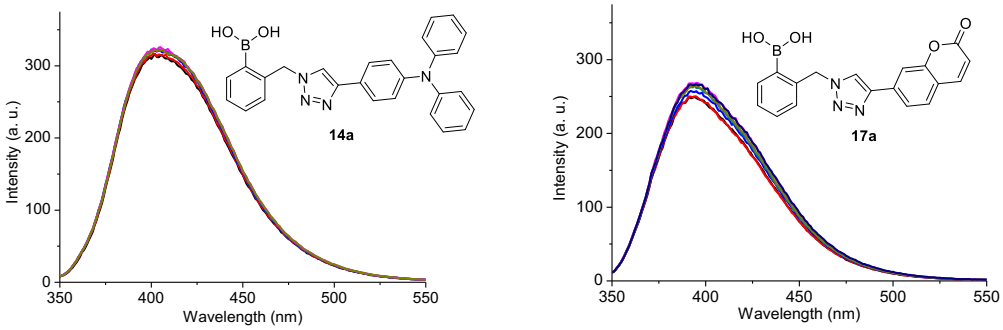


Figure S6. Fluorescence spectra of **14a** (left) and **17a** (right) in the presence of increasing concentration of D-glucose (0-6 mM; black curve: 0 mM, dark yellow curve: 6mM). Excitation wavelength: 290 nm (**14a**); 330 nm (**17a**).

3. Isothermal titration calorimetry (ITC) study

ITC study was carried out with a MicroCal VP-ITC instrument (MicroCal). All the experiments were conducted in pH 8.21 methanolic buffer. The same batch of buffer stock solution was used for preparing all boronic acids and D-fructose samples in all experiments. The concentrations of each boronic acid compound and fructose were optimised for each individual experiment. The ITC cell volume was 1.474 mL. Experiments were performed at a constant cell temperature of 25 °C. The reference power was set to 30 $\mu Cal \cdot s^{-1}$. Each titration comprised 49 separate injections of concentrated D-fructose solution at time intervals of 180 s. Data was analysed using Origin 7E (OriginLab). The first injection point was routinely discarded. Data were fit using "One Sites" model.

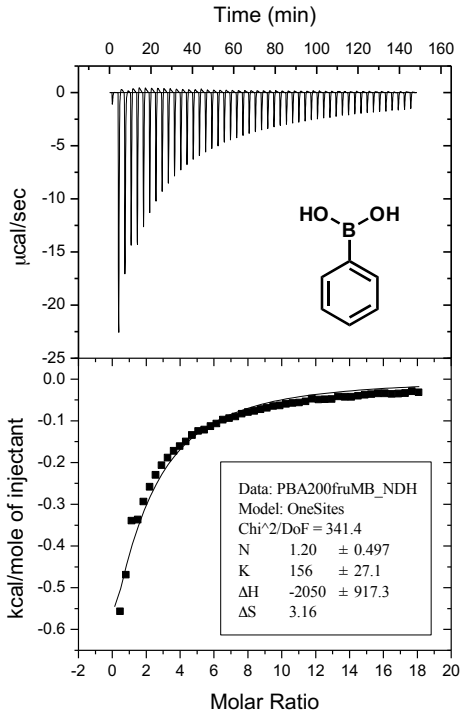


Figure S7. ITC study of phenylboronic acid (PBA) and D-fructose in pH 8.21 methanolic buffer.

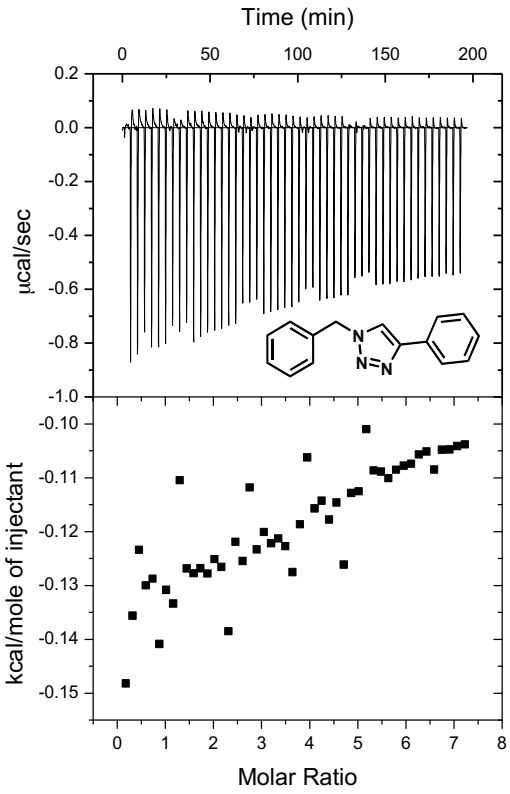


Figure S8. ITC data of 1-benzyl-4-phenyl-1*H*-1,2,3-triazole titrating with D-fructose in pH 8.21 methanolic buffer as a negative control.

4. ^{1}H and ^{11}B NMR spectroscopy titrations

^{1}H NMR titration was carried out by the following the procedures, introduced by Mulla *et al*.[3] In detail, 3.8 mg compound **1a** (or **1c**) was dissolved in 500 µL DMSO-*d6*. D_2O buffer was prepared following literature and the pD was carefully adjusted to 8.21 using a pH meter. A 20 mM fructose solution was made as the stock solution in the prepared buffer. The stock solution was stored at room temperature overnight to make sure equilibrium is reached.

During the titration, a ^{1}H NMR spectrum of **1a** in DMSO-*d6* was recorded first (see Figure S9, 0 equiv. fructose). After that, 5 µL of the fructose stock solution was added, the sample was kept at room temperature for at least 30 mins before the next spectrum was collected. The procedure was repeated until the end of the experiment.

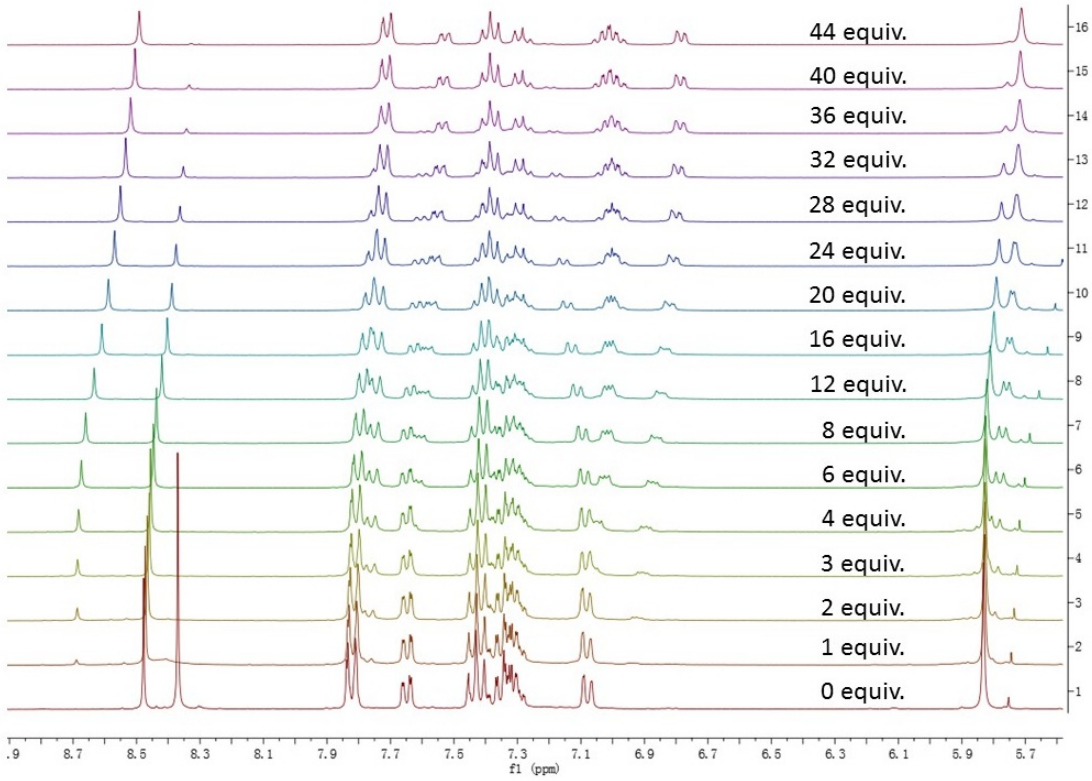


Figure S9. ^{1}H NMR titration of compound **1a** and fructose in DMSO-*d6*/D_2O buffer (pD = 8.21). The amount of fructose increased from 0 to 44 equivalents.

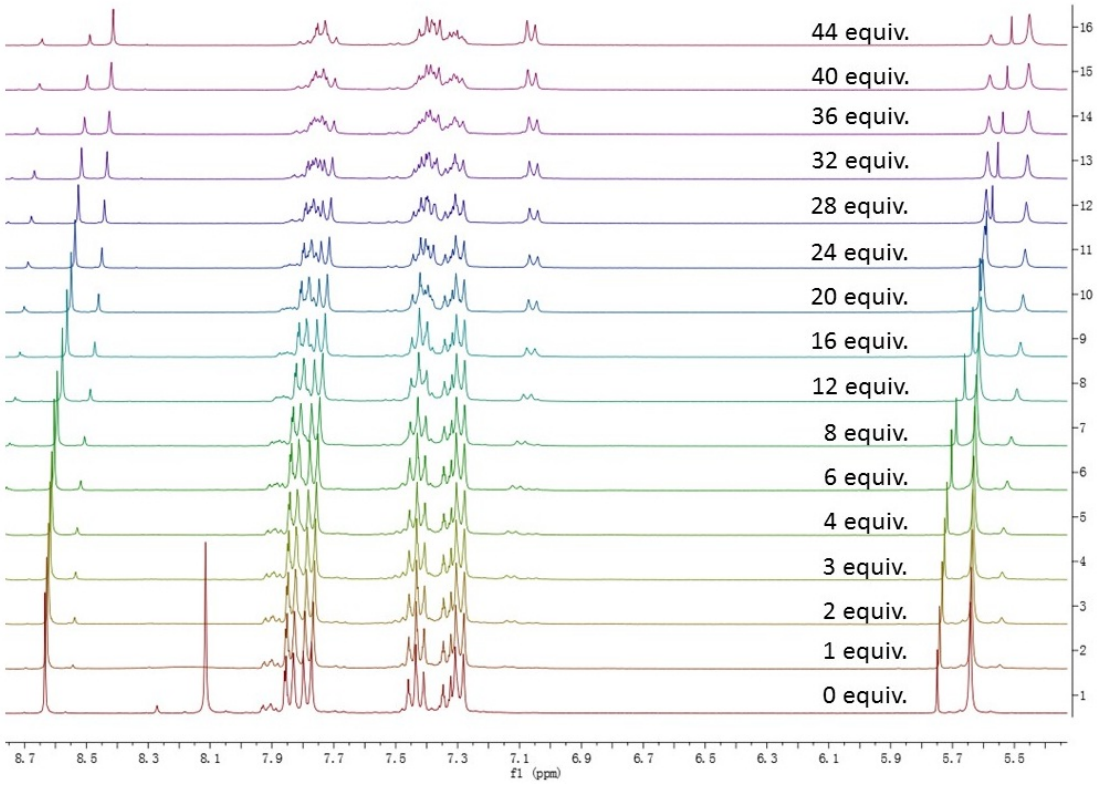


Figure S10. ^{1}H NMR titration of compound **1c** and fructose in DMSO-*d6*/D_2O buffer (pD = 8.21). The amount of fructose increased from 0 to 44 equivalents.

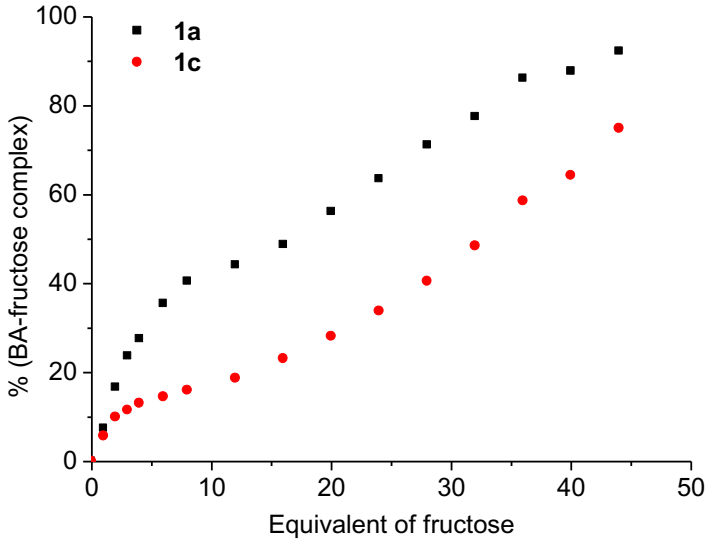


Figure S11. The number of equivalents of fructose plotted against the percentage of boronic acid-fructose complex (calculated based on the integration of ^{1}H NMR spectra).

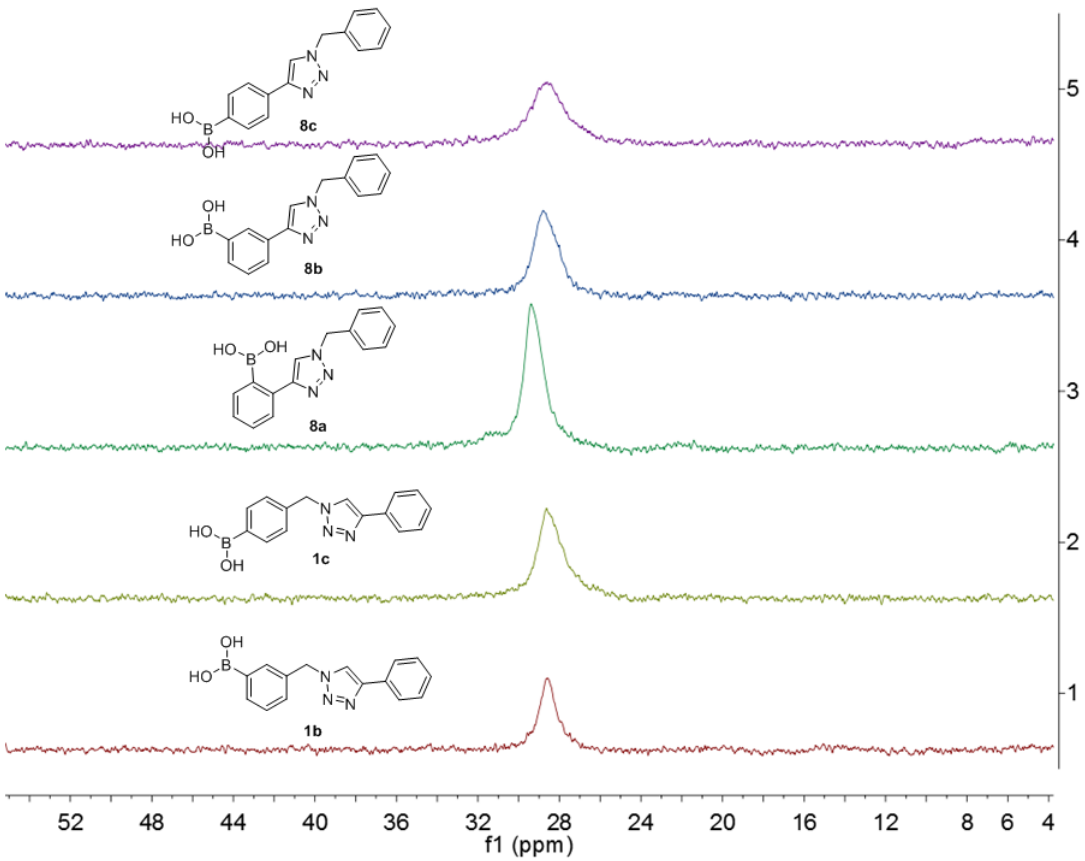


Figure S12. ^{11}B NMR spectra of compound **1b-c** and **8a-c** in CD_3CN (spectrum of **1a** was not recorded due to low solubility).

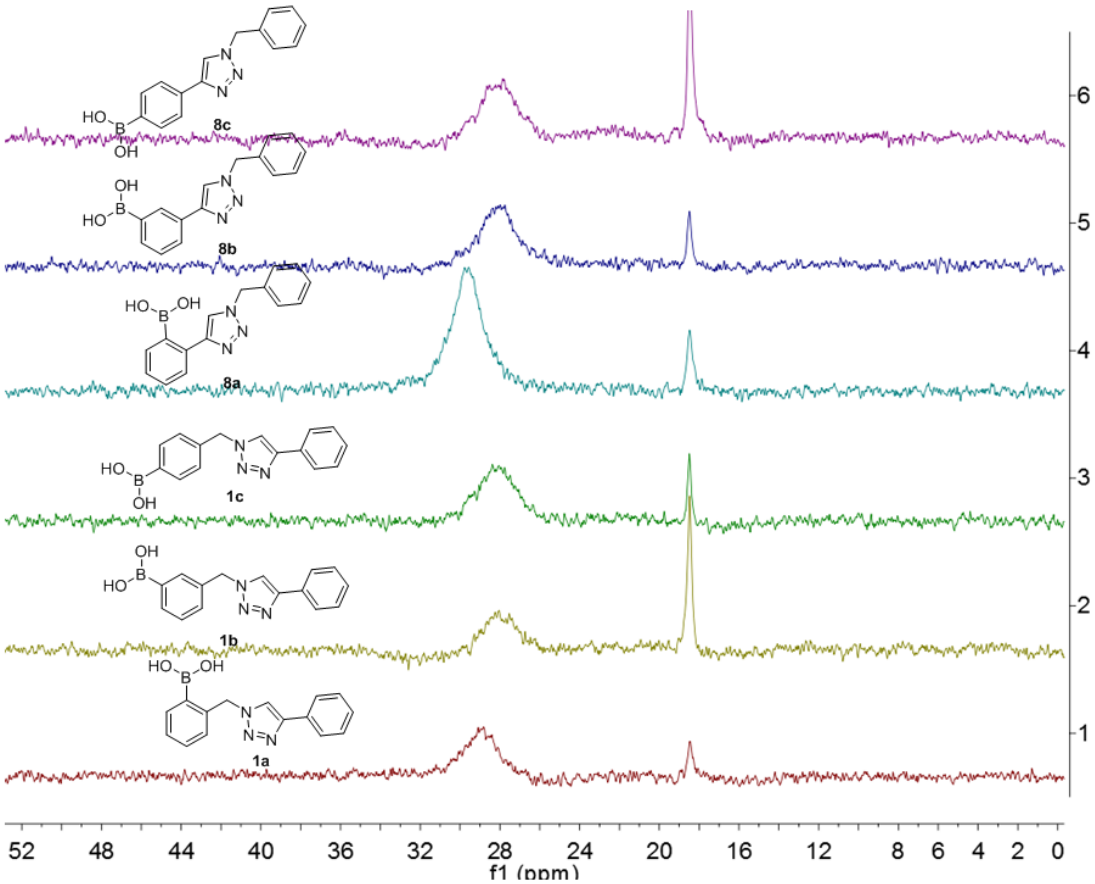


Figure S13. ^{11}B NMR spectra of compound **1a-c** and **8a-c** in CD_3OD. The peak at 19 ppm is boric acid, $B(OH)_3$.

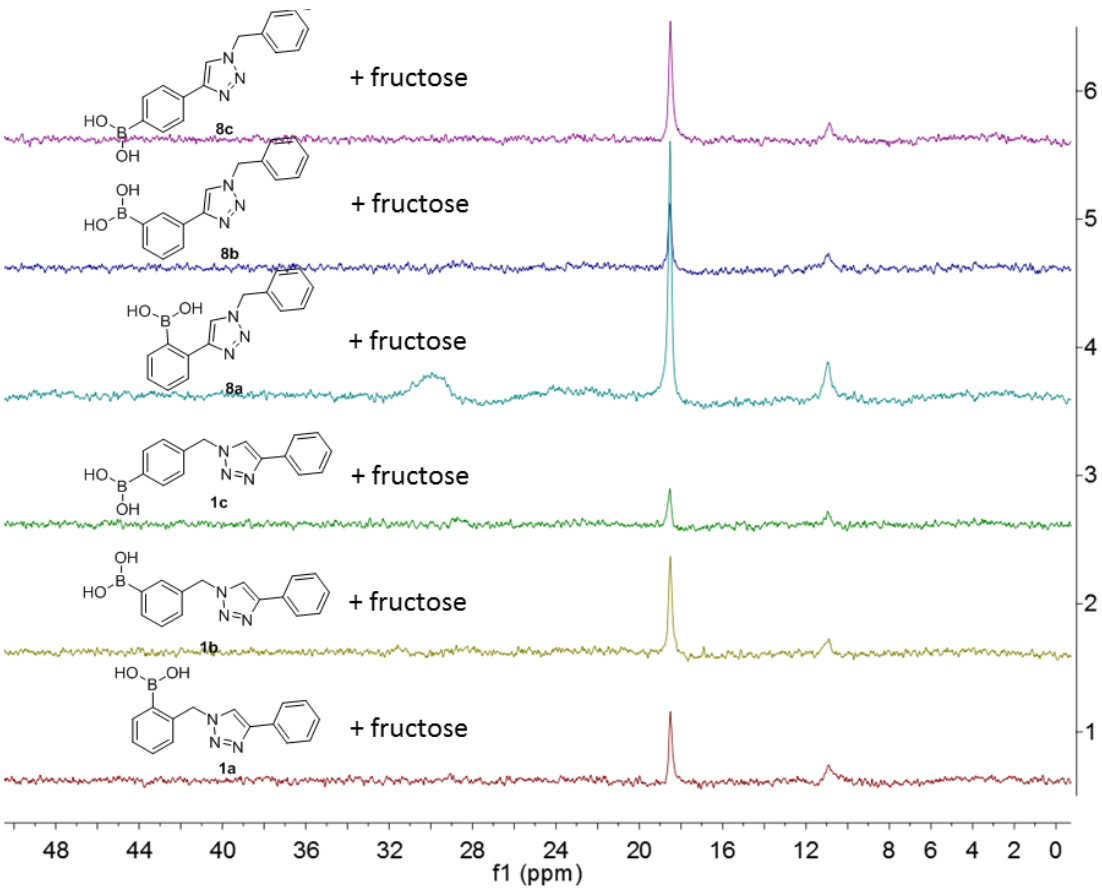
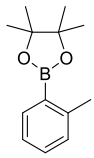


Figure S14. ^{11}B NMR spectra of compound **1a-c** and **8a-c** in CD_3OD in the presence of fructose.[4]

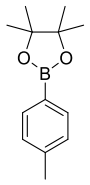
5. Synthetic procedures and characterisation data: First generation "*click-fluors*"

5.1 Pinacol protection

Tolylboronic acid (1.09 g, 8.0 mmol) and pinacol (1.12 g, 9.5 mmol) were mixed in toluene (100 mL). The reaction mixture was heated under reflux for 2 h, water was removed using Dean-Stark apparatus. The reaction solution was cooled to room temperature and toluene was removed by rotary evaporator. The crude product was dissolved in ethyl acetate (50 mL), and washed with deionised water (2 × 50 mL), dried over magnesium sulfate and filtered. The solvent was removed *in vacuo* to obtain boronic acid pinacol ester.



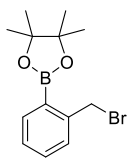
4,4,5,5-Tetramethyl-2-(*o*-tolyl)-1,3,2-dioxaborolane $C_{13}H_{19}BO_2$, oil (92% yield), R_f = 0.90 (PE/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.80 (dd, *J* = 7.5, 1.6, 1H) , 7.35 (dd, *J* = 7.5, 1.6, 1H), 7.25 - 7.16 (m, 2H), 2.58 (s, 2H), 1.38 (s, 12H); δ_C (101 MHz, $CDCl_3$) 144.8, 135.9, 130.8, 129.8, 124.7, 83.4, 24.9, 22.3.



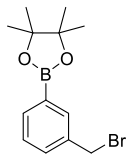
4,4,5,5-Tetramethyl-2-(*p*-tolyl)-1,3,2-dioxaborolane $C_{13}H_{19}BO_2$ white solid (86% yield), R_f = 0.89 (hexane/EtOAc, 6:1) δ_H (300 MHz, $CDCl_3$) 7.74 (d, *J* = 7.6, 2H), 7.22 (d, *J* = 7.6, 2H), 2.40 (s, 3H), 1.37 (s, 12H). δ_C (101 MHz, $CDCl_3$) 141.4, 134.8, 128.5, 83.6, 77.3, 77.0, 76.7, 24.9, 21.7.

5.2 Bromination

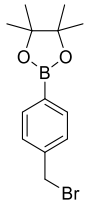
4,4,5,5-Tetramethyl-2-*o*-tolyl-1,3,2-dioxaborolane (1.54 g, 7.0 mmol), *N*-bromosuccinimide (1.87 g, 10.5 mmol), and azobisisobutylonitrile (12 mg, 1 mol%) were added in 100 mL of acetonitrile and refluxed at 90 °C for 2 h. After the reaction was completed, the mixture was allowed to cool at room temperature and the solvent was removed by rotary evaporator. Hexane was added to dissolve the product and the remaining solid was removed after filtration. The filtrate was concentrated by rotary evaporator and dried *in vacuo* to obtain the bromination product.



2-(2-(Bromomethyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3a) $C_{13}H_{18}BBrO_2$, white solid (99% yield), R_f = 0.81 (PE/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.86 (d, *J* = 7.2, 1H), 7.46 – 7.39 (m, 2H), 7.36 – 7.27 (m, 1H), 4.96 (s, 2H), 1.41 (s, 12H). δ_C (101 MHz, $CDCl_3$) 144.3, 136.4, 131.3, 130.1, 127.6, 83.9, 34.0, 24.9.



2-(3-(Bromomethyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3b) $C_{13}H_{18}BBrO_2$, white solid (89% yield), R_f = 0.79 (PE/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.85 (s, 1H), 7.76 (dt, *J* = 7.2, 1.2, 1H), 7.53 (dt, *J* = 7.6, 1.8, 1H), 7.38 (t, *J* = 7.6, 1H), 4.53 (s, 2H), 1.37 (s, 12H); δ_C (101 MHz, $CDCl_3$) 137.0, 135.2, 134.8, 132.0, 128.3, 84.0, 33.5, 24.9.

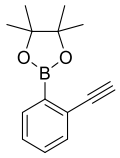


2-(4-(Bromomethyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c) $C_{13}H_{18}BBrO_2$, white solid (99% yield), R_f = 0.82 (hexane/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.81 (d, *J* = 8.1, 2H), 7.42 (d, *J* = 8.1, 2H), 4.51 (s, 2H), 1.36 (s, 12H). δ_C (101 MHz, $CDCl_3$) 140.7, 135.2, 128.3, 125.7, 84.0, 33.3, 24.9.

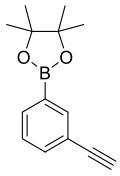
5.3 TMS deprotection

Starting material (153 mg, 0.7 mmol) was dissolved in THF (10 mL). The flask was cooled to 0 °C and a 1 M solution of tetrabutylammonium fluoride (TBAF) in THF (1.34 mL, 1.3 mmol) was slowly added *via* syringe. The reaction mixture was stirred for 2 h at room temperature, washed three times with water and brine, dried over magnesium sulfate, filtered and concentrated by rotary evaporator. The crude product was purified by flash chromatography on silica gel ((hexane/EtOAc) 95:5, *v*/*v*).

2-(2-Ethynylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (6a) $C_{14}H_{17}BO_2$, brown solid (97% yield), R_f = 0.91 (PE/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.69 – 7.64 (m, 1H), 7.51 – 7.38 (m, 1H), 7.36 – 7.20 (m, 2H), 3.19 (s, 1H), 1.27 (s, 12H). δ_C (101 MHz, $CDCl_3$) 134.9, 133.5, 130.1, 127.9, 126.3, 84.3, 83.9, 79.2, 24.9.



2-(3-Ethynylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (6b) $C_{14}H_{17}BO_2$, brown solid, (92% yield), R_f = 0.67 (PE/DCM, 1:1), δ_H (300 MHz, $CDCl_3$) 7.99 (s, 1H), 7.80 (d, *J* = 7.5, 1H), 7.60 (dt, *J* = 7.5, 1.2, 1H), 7.35 (t, *J* = 7.5, 1H), 3.08 (s, 1H), 1.37 (s, 12H). δ_C (101 MHz, $CDCl_3$) 137.4, 133.3, 130.4, 127.7, 126.5, 83.9, 83.6, 79.3, 24.7.

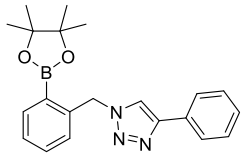


5.4 CuAAC reaction (1st generation "*click-fluors*")

Brominated boronic acid pinacol ester (0.59 g, 2.0 mmol) and sodium azide (0.16 g, 2.4 mmol) were dissolved in dimethyl sulfoxide (10 mL) and heated at 60 ^{0}C for 30 min. Then phenylacetylene (0.22 g, 2.2 mmol), copper(I) iodide (38 mg, 0.2 mmol) and sodium ascorbate (0.20 g, 1.0 mmol) were added. The reaction mixture was heated at 80 ^{0}C for 2 h. After cooling to room temperature, deionised water (50 mL) was added into the reaction flask. Ethyl acetate was used to extract the crude product (3 × 30 mL). After removing water using magnesium sulfate, the product was concentrated by rotary evaporator. Flash chromatography was employed to purify the crude product (hexane/EtOAc, 6:1, *v*/*v*). The appropriate

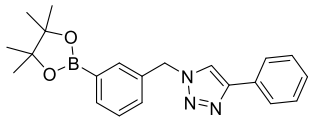
fractions were combined together, and the solvents were removed under reduced pressure to give the corresponding triazole compounds.

4-Phenyl-1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4a)



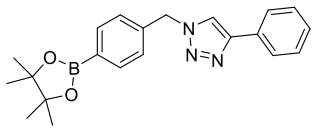
$C_{21}H_{24}BN_3O_2$, white solid (43% yield), R_f = 0.29 (hexane/EtOAc, 5:1), mp 100 – 102 °C, δ_H (300 MHz, $CDCl_3$) 7.96 (dd, *J* = 7.3, 1.4, 1H), 7.86 – 7.75 (m, 3H), 7.51 – 7.22 (m, 6H), 5.92 (s, 2H), 1.38 (s, 12H); δ_C (101 MHz, $CDCl_3$) 147.6, 140.9, 136.7, 131.9, 130.9, 129.2, 128.8, 128.0, 127.9, 125.6, 119.9, 84.2, 53.4, 24.9; δ_B (128 MHz, $CDCl_3$) 31.8. *v*/cm^{-1} 2975, 1601, 1442, 1344, 1321, 1143, 1109, 1067, 966, 764, 721, 696.

4-Phenyl-1-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4b)



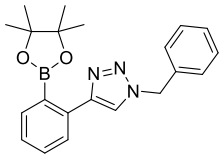
$C_{21}H_{24}BN_3O_2$, white solid (52% yield) R_f = 0.37 (hexane/EtOAc, 6:1), mp 136 – 138 °C, δ_H (300 MHz, $CDCl_3$) 7.76 – 7.58 (m, 4H), 7.54 (s, 1H), 7.31 – 7.17 (m, 4H), 7.17 – 7.08 (m, 1H), 5.37 (s, 2H), 1.19 (s, 12H); δ_C (101 MHz, $CDCl_3$) 148.0, 135.2, 134.5, 134.0, 131.0, 130.6, 128.8, 128.6, 128.1, 125.7, 119.7, 84.1, 54.2, 24.9; δ_B (128 MHz, $CDCl_3$) 13.0. M/z: (ES^+) 362.2 $[M + H]^+$. High resolution MS calc. for formula $C_{21}H_{25}BN_3O_2$: 362.2040; found: 362.2034. *v*/cm^{-1} 2979, 2934, 1611, 1467, 1361, 1329, 1142, 1090, 859, 767, 694.

4-Phenyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4c)



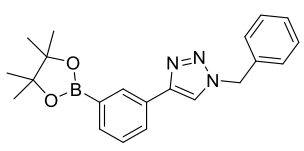
$C_{21}H_{24}BN_3O_2$, white solid (65% yield), R_f = 0.21 (PE/EtOAc, 5:1), mp 175 – 177 °C, δ_H (300 MHz, $CDCl_3$) 7.90 – 7.76 (m, 4H), 7.66 (s, 1H), 7.46 – 7.38 (m, 2H), 7.38 – 7.29 (m, 3H), 5.61 (s, 2H), 1.36 (s, 12H); δ_C (101 MHz, $CDCl_3$) 148.2, 137.4, 135.6, 130.5, 128.8, 128.2, 127.3, 125.7, 119.5, 84.0, 77.3, 77.0, 76.7, 54.3, 24.9; δ_B (128 MHz, $CDCl_3$) 13.0. M/z: (ES^+) 362.2 $[M + H]^+$. High resolution MS calc. for formula $C_{21}H_{25}BN_3O_2$: 362.2040; found: 362.2032. *v*/cm^{-1} 2980, 2933, 1612, 1411, 1360, 1329, 1271, 1142, 1090, 967, 860, 768, 694.

1-Benzyl-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7a)



$C_{21}H_{24}BN_3O_2$, white solid (33% yield) R_f = 0.21 (PE/EtOAc, 6:1), mp 76 – 79 °C, δ_H (300 MHz, $CDCl_3$) 7.77 (s, 1H), 7.71 (dd, *J* = 7.4, 1.1, 1H), 7.65 (dd, *J* = 7.7, 0.6, 1H), 7.46 – 7.27 (m, 7H), 5.54 (s, 2H), 1.27 (s, 12H); δ_C (101 MHz, $CDCl_3$) 148.5, 135.0, 134.9, 134.7, 130.2, 129.1, 128.6, 128.1, 127.7, 127.3, 121.5, 83.9, 54.0, 24.8; δ_B (128 MHz, $CDCl_3$) 13.8. M/z: (ES^+) 362.2 $[M + H]^+$. High resolution MS calc. for formula $C_{21}H_{25}BN_3O_2$: 362.2040; found: 362.2049. *v*/cm^{-1} 2979, 2933, 1610, 1426, 1349, 1320, 1141, 858, 767, 724, 694.

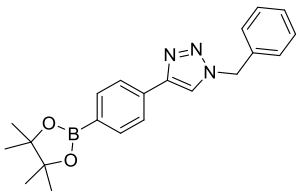
1-Benzyl-4-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7b)



$C_{21}H_{24}BN_3O_2$, white solid (44% yield), R_f = 0.26 (PE/EtOAc, 5:1), mp 177 – 179 °C, δ_H (300 MHz, $CDCl_3$) 8.11 (s, 1H), 8.09 – 8.03 (m, 1H), 7.78 (dt, *J* = 7.4, 1.2, 1H), 7.74 (s, 1H), 7.52 – 7.36 (m, 4H), 7.36 – 7.26 (m, 2H), 5.58 (s, 2H), 1.35 (s, 12H); δ_C (101 MHz, $CDCl_3$) 148.2, 134.7, 134.5, 131.9, 129.9, 129.2, 128.8, 128.6, 128.3, 128.1, 119.7, 83.9, 54.2, 24.9; δ_B (128 MHz, $CDCl_3$) 13.4. M/z: (ES^+) 362.2 $[M + H]^+$. High resolution MS calc. for formula $C_{21}H_{25}BN_3O_2$: 362.2040; found:

362.2048. *v*/cm^{-1} 2978, 2925, 1609, 1436, 1356, 1337, 1141, 1078, 963, 861, 794, 724, 707.

1-Benzyl-4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7c)

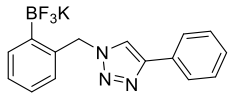


$C_{21}H_{24}BN_3O_2$, white solid (61% yield), Rf = (0.21 PE/EtOAc, 5:1), mp 160 – 162 °C, δ_H (300 MHz, $CDCl_3$) 7.90 – 7.79 (m, 4H), 7.73 (s, 1H), 7.42 – 7.34 (m, 3H), 7.34 – 7.25 (m, 2H), 5.53 (s, 2H), 1.35 (s, 12H); δ_C (101 MHz, $CDCl_3$) 148.0, 135.3, 134.6, 133.1, 129.1, 128.8, 128.1, 124.8, 120.1, 83.9, 54.2, 24.9. δ_B (128 MHz, $CDCl_3$) 13.4. M/z: (ES^+) 362.2 $[M + H]^+$. High resolution MS calc. for formula $C_{21}H_{25}BN_3O_2$: 362.2040; found: 362.2046. *v*/cm^{-1} 2987, 1615, 1459, 1393, 1352, 1330, 1267, 1135, 1094, 962, 848, 808, 655.

5.5 Pinacol deprotection step 1: Trifluoroborate salts

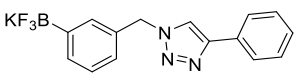
The triazole intermediate (0.18 g, 0.5 mmol) was dissolved in 7 mL of methanol and added to aqueous potassium hydrogen difluoride (0.55 g, 7.0 mmol) in a plastic container. The resulting white slurry was stirred at room temperature for 15 min, concentrated by rotary evaporator, and then dissolved in hot acetone (60 mL). The mixture was filtered and the filtrate was concentrated *in vacuo*. The solid was dissolved in minimum amount of acetone, and then diethyl ether (20 mL) was added resulting in precipitation of the potassium trifluoroborate salt.

Potassium (2-((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)trifluoroborate $C_{15}H_{12}BF_3KN_3$, white



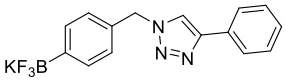
solid (98% yield), mp 243 – 245 °C, δ_H (300 MHz, $(CD_3)_2CO$) 8.27 (s, 1H), 7.90 – 7.79 (m, 2 H), 7.65 (d, *J* = 6.6, 1H), 7.46 – 7.33 (m, 2H), 7.33 – 7.23 (m, 1H), 7.14 – 6.96 (m, 3H), 5.80 (s, 2H); δ_C (101 MHz, CD_3OD) 147.3, 137.7, 132.3, 130.5, 128.5, 127.7, 127.5, 126.6, 126.5, 125.2, 120.9, 53.6. M/z: (ES^-) 302.1 $[M - K]^+$. High resolution MS calc. for formula $C_{15}H_{12}BN_3F_3$: 302.1082; found: 302.1077.

Potassium (3-((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)trifluoroborate $C_{15}H_{12}BF_3KN_3$, white



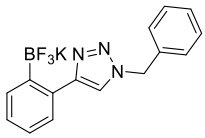
solid (95% yield), mp 289 – 291 °C, δ_H (300 MHz, CD_3OD) 7.97 (s, 1H), 7.80 – 7.62 (m, 3H), 7.59 (d, *J* = 6.9, 1H), 7.43 – 7.18 (m, 3H), 7.18 – 7.00 (m, 2H), 5.32 (s, 2H); δ_C (101 MHz, CD_3OD) 147.5, 133.4, 131.6, 131.4, 130.2, 128.6, 127.9, 127.4, 126.0, 125.3, 120.8, 54.3. δ_F (282 MHz, CD_3OD) -142.6. M/z: (ES^-) 302.1 $[M - K]^+$. High resolution MS calc. for formula $C_{15}H_{12}BN_3F_3$: 302.1082; found: 302.1079.

Potassium (4-((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)trifluoroborate $C_{15}H_{12}BF_3KN_3$, white

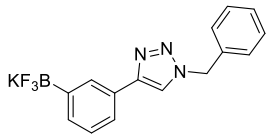


solid (93% yield), mp > 300 °C, δ_H (300 MHz, $(CD_3)_2CO$) 8.27 (s, 1H), 7.93 –7.86 (m, 2H), 7.51 (d, *J* = 7.6, 2H), 7.45 – 7.35 (m, 2H), 7.34 – 7.25 (m, 1H), 7.16 (d, *J* = 7.6, 2H), 5.55 (s, 2H); δ_F (282 MHz, $(CD_3)_2CO$) -142.7. M/z: (ES^-) 302.1 $[M - K]^+$. High resolution MS calc. for formula $C_{15}H_{12}BN_3F_3$: 302.1082; found: 302.1086.

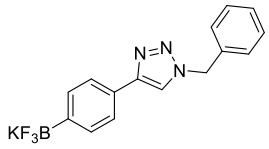
Potassium (2-(1-benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)trifluoroborate $C_{15}H_{12}BF_3KN_3$, white solid (99% yield), mp 213 – 215 mp 289 – 291 °C, δ_H (300 MHz, CD_3OD) 8.31 (s, 1H), 7.82 – 7.65 (m, 2H), 7.42 – 7.18 (m, 7H), 5.56 (s, 2H); δ_C (101 MHz, CD_3OD) 149.9, 135.7, 133.2, 132.7, 128.5, 127.9, 127.5, 127.5, 126.5, 126.2, 124.0, 123.9, 53.3. M/z: (ES^-) 302.1 $[M - K]^+$. High resolution MS calc. for formula $C_{15}H_{12}BN_3F_3$: 302.1082; found: 302.1081.



Potassium (3-(1-benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)trifluoroborate $C_{15}H_{12}BF_3KN_3$, white solid (98% yield), mp 256 – 259 mp 289 – 291 °C, δ_H (300 MHz, CD_3OD) 8.08 (s, 1H), 8.00 (s, 1H), 7.62 (dt, *J* = 7.9, 1.7, 1H), 7.56 (d, *J* = 7.3, 1H), 7.41 – 7.17 (m, 6H), 5.45 (s, 2H); δ_C (101 MHz, CD_3OD) 149.0, 135.4, 131.5, 128.7, 128.6, 128.4, 128.1, 127.7, 127.2, 123.3, 120.5, 53.5. M/z: (ES^-) 302.1 $[M - K]^+$. High resolution MS calc. for formula $C_{15}H_{12}BN_3F_3$: 302.1082; found: 302.1075.



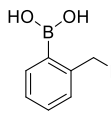
Potassium (4-(1-benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)trifluoroborate $C_{15}H_{12}BF_3KN_3$, white solid (98% yield), mp 243 – 245 mp 289 – 291 °C, δ_H (300 MHz, CD_3OD) 8.22 (s, 1H), 7.64 (d, *J* = 8.0, 2H), 7.58 (d, *J* = 8.0, 2H), 7.49 – 7.26 (m, 5H), 5.62 (s, 2H); δ_C (101 MHz, CD_3OD) 148.9, 135.5, 131.6, 128.6, 128.1, 127.6, 123.8, 120.2, 53.6. δ_F (282 MHz, CD_3OD) -142.7. M/z: (ES^+) 302.1 [M - K]. High resolution MS calc. for formula $C_{15}H_{12}BF_3N_3$: 302.1076; found: 302.1065. M/z: (ES^-) 302.1 $[M - K]^+$. High resolution MS calc. for formula $C_{15}H_{12}BN_3F_3$: 302.1082; found: 302.1089.



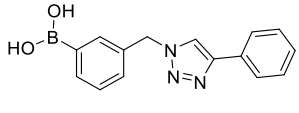
5.6 Pinacol deprotection step 2: Boronic acids

The obtained potassium trifluoroborate salt (180 mg, 0.5 mmol) and lithium hydroxide (42 mg, 1.8 mmol) were dissolved in acetonitrile (10 mL) and deionised water (5 mL), respectively. Then the solutions were mixed together and stirred at room temperature for 24 h. The solution was acidified using saturated ammonium chloride (8 mL) and hydrochloric acid solution (1 mL, 3 M). After that, the resulting solution was extracted with ethyl acetate (3 × 20 mL). The combined extracts were washed with hydrochloric acid solution (20 mL, 0.5 M), dried over magnesium sulfate, and filtered. The crude product was by flash chromatography (DCM/methanol, 95:5, *v*/*v*).

(2-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1a) $C_{15}H_{14}BN_3O_2$, white solid (97% yield), R_f = 0.73 (DCM/MeOH, 95:5), mp 135 – 136 °C, δ_H (300 MHz, $CDCl_3$) 8.20 - 7.92 (m, 1H), 7.85 - 7.00 (m, 9H), 5.88 (s, 2H); δ_H (300 MHz, DMSO-*d6*) 8.47 (s, 1H), 8.36 (s, 2H) , 7.85 – 7.76 (m, 2H), 7.64 (dd, *J* = 7.2, 1.5, 1H), 7.45 – 7.40 (m, 2H), 7.37 – 7.25 (m, 3H), 7.11 – 7.03 (m, 1H), 5.82 (s, 2H); δ_C (101 MHz, CD_3OD) 149.5, 140.0, 136.1, 134.0, 132.0, 131.6, 131.0, 130.6, 130.4, 129.8, 129.3, 127.1, 122.7, 55.8; δ_B (128 MHz, CD_3OD) 28.8. M/z: (ES^+) 280.1 $[M + H]^+$. High resolution MS calc. for formula $C_{15}H_{15}BN_3O_2$: 280.1257; found: 280.1253. ν/cm^{-1} 3420, 3209, 3145, 2938, 1607, 1435, 1399, 1153, 766, 717.

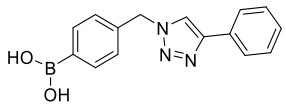


(3-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1b) $C_{15}H_{14}BN_3O_2$, white solid (95% yield), R_f = 0.76 (DCM/MeOH, 95:5), mp 218 – 220 °C, δ_H (300 MHz, CD_3OD) 8.29 (s, 1H), 7.95 – 7.70 (m, 3H), 7.64 (s, 1H), 7.53 – 7.20 (m,

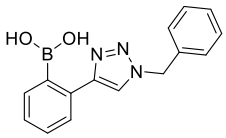


5H), 5.63 (s, 2H); δ_C (101 MHz, CD_3OD) 149.2, 135.1, 134.7, 134.2, 131.7, 131.0, 130.4, 130.0, 129.4, 126.7, 122.2, 55.2; δ_B (128 MHz, CD_3CN) 28.6. M/z: (ES^+) 280.1 $[M + H]^+$. High resolution MS calc. for formula $C_{15}H_{15}BN_3O_2$: 280.1257; found: 280.1250. ν/cm^{-1} 3418, 3210, 3141, 2934, 1607, 1433, 1381, 1359, 1333, 1151, 1092, 1049, 982, 764, 703.

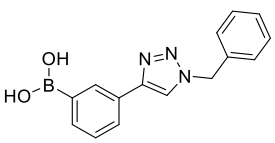
(4-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1c) $C_{15}H_{14}BN_3O_2$, white solid (89% yield), R_f = 0.73 (DCM/MeOH, 95:5), mp 234 – 235 °C, δ_H (300 MHz, CD_3OD) 8.30 (s, 1H), 7.93 – 7.70 (m, 3H), 7.63 (d, *J* = 7.1, 1H), 7.50 – 7.37 (m, 2H), 7.37 – 7.23 (m, 3H), 5.62 (s, 2H); δ_C (101 MHz, CD_3OD) 147.8, 134.2, 133.8, 130.2, 128.6, 128.0, 126.8, 125.3, 120.9, 53.6; δ_B (128 MHz, CD_3CN) 28.6. M/z: (ES^+) 280.1 $[M + H]^+$. High resolution MS calc. for formula $C_{15}H_{15}BN_3O_2$: 280.1257; found: 280.1253. ν/cm^{-1} 3420, 3217, 3143, 2950, 1608, 1435, 1390, 1344, 1157, 1050, 998, 762.



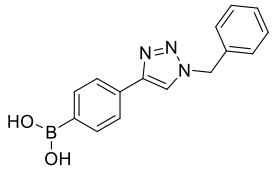
(2-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8a) $C_{15}H_{14}BN_3O_2$, with solid (90%), Rf = 0.77 (DCM/MeOH, 95:5), mp 137 – 139 °C, δ_H (300 MHz, CD_3OD) 8.20 (s, 1H), 7.67 – 7.58 (m, 1H), 7.47 – 7.28 (m, 8H), 5.62 (s, 2H); δ_C (101 MHz, CD_3OD) 148.7, 135.5, 132.5, 131.1, 128.7, 128.5, 128.2, 127.7, 127.4, 125.3, 120.4, 53.6; δ_B (128 MHz, CD_3CN) 29.4. M/z: (ES^+) 280.1 $[M + H]^+$. High resolution MS calc. for formula $C_{15}H_{15}BN_3O_2$: 280.1257; found: 280.1249. ν/cm^{-1} 3306, 3149, 2922, 1598, 1444, 1379, 1246, 1131, 1076, 768, 714.



(3-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8b) $C_{15}H_{14}BN_3O_2$, white solid (95% yield), R_f = 0.76 (DCM/MeOH, 95:5), mp 237 – 238 °C, δ_H (300 MHz, CD_3OD) 8.42 – 7.99 (m, 2H), 7.84 (d, *J* = 7.7, 1H), 7.79 – 7.51 (m, 1H), 7.51 – 7.19 (m, 6H), 5.61 (s, 2H); δ_C (101 MHz, CD_3OD) 148.1, 135.4, 133.5, 133.0, 130.8, 130.3, 129.3, 128.7, 128.2, 127.7, 127.1, 126.5, 120.7, 53.6; δ_B (128 MHz, CD_3CN) 28.8. M/z: (ES^+) 280.1 $[M + H]^+$. High resolution MS calc. for formula $C_{15}H_{15}BN_3O_2$: 280.1257; found: 280.1250. ν/cm^{-1} 3236, 3135, 2926, 1609, 1496, 1347, 1120, 1045, 799, 703.



(4-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8c) $C_{15}H_{14}BN_3O_2$, white solid (92% yield), R_f = 0.81 (DCM/MeOH), mp 169 – 171 °C, δ_H (300 MHz, CD_3OD) 7.89 – 7.57 (m, 4H), 7.50 – 7.21 (m, 5H), 5.60 (s, 2H); δ_C (101 MHz, CD_3OD) 147.8, 135.3, 134.1, 133.9, 131.7, 128.6, 128.2, 127.7, 124.3, 121.1, 53.6; δ_B (128 MHz, CD_3CN) 28.5. M/z: (ES^+) 280.1 $[M + H]^+$. High resolution MS calc. for formula $C_{15}H_{15}BN_3O_2$: 280.1257; found: 280.1258. ν/cm^{-1} 3352, 3136, 2924, 1615, 1497, 1334, 1193, 972, 822, 713.



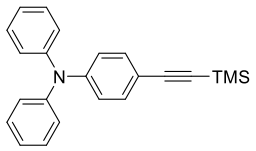
6. Synthetic procedures and characterisation data: Second generation "*click-fluors*"

6.1 Synthesis of acetylene-modified fluorophores

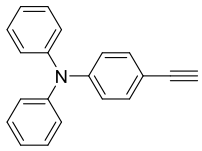
Synthesis of *N,N*-diphenyl-4-((trimethylsilyl)ethynyl)aniline

A three-neck round-bottom flask was dried in oven overnight and charged with 4-bromo-*N,N*-diphenylaniline (1.50 g, 4.6 mmol). 20 mL dry TEA was added *via* syringe. After bubbling with argon gas for 15 min, $Pd(PPh_3)_2Cl_2$ (325 mg, 0.5 mmol), and CuI (88 mg, 0.5 mmol) was added to the flask. After bubbling with argon gas for another 15 min, trimethylsilylacetylene (1.0 mL, 7.0 mmol) was injected *via* syringe, and the reaction mixture turned black. The mixture was refluxed for 20 h, diluted with DCM, filtered, and concentrated by rotary evaporator. The crude product was purified by chromatographyon silica gel using hexane as eluent.[5]

In a one-neck round-bottom flask, *N,N*-diphenyl-4-((trimethylsilyl)ethynyl)aniline (223 mg, 1.1 mmol) was dissolved in THF (10 mL). The flask was cooled to 0 °C and a 1 M solution of TBAF in THF (1.34 mL, 1.3 mmol) was slowly added *via* syringe. The reaction mixture was stirred for 2 h at room temperature, washed three times with water and brine, dried over $MgSO_4$, filtered and concentrated by rotary evaporator. The crude product was purified by flash chromatography on silica gel ((hexane/EtOAc) 95:5, *v*/*v*).



***N,N*-Diphenyl-4-((trimethylsilyl)ethynyl)aniline** $C_{23}H_{23}NSi$ Brown solid (93% yield), R_f = 0.81 (PE/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.37 (dd, *J* = 7.3, 7.2, 4H), 7.30 (d, *J* = 8.6, 2H), 7.12 (t, *J* = 7.3, 2H), 7.06 (d, *J* = 7.2, 4H), 6.85 (d, *J*= 8.6, 2H), 0.23 (s, 9H); δ_C (101 MHz, CD_3OD) 147.7, 146.3, 132.7, 129.6, 125.0, 124.0, 120.8, 114.5, 105.5, 92.7, 0.1. M/z: (ES^+) 341.3 $[M]^+$. *v*/cm^{-1} 3597, 2924, 2153, 1641, 1591, 1486, 1273, 1206, 948, 930, 834, 750, 694.



4-Ethynyl-*N,N*-diphenylaniline $C_{20}H_{15}N$ brown solid (84% yield), R_f = 0.77 (PE/EtOAc, 6:1). δ_H (300 MHz, $CDCl_3$) 7.33 (m, 6H), 7.10 (t, *J* = 7.2, 2H), 7.05 (d, *J* = 7.2, 4H), 6.86 (d, *J* = 8.5, 2H), 4.05 (s, 1H). δ_C (300 MHz, $CDCl_3$) 148.3; 146.9; 133.3; 130.2; 125.4; 124.5; 121.7; 114.7; 84.1; 80.1.

Synthesis of 4-ethynyl-1,8-naphthalimide

4-bromo-1,8-naphthalic anhydride (1.00 g, 3.6 mmol) and ethylamine (70% solution in water) (0.35 mL, 4.3 mmol) were refluxed in 1,4-dioxane (50 mL) for 16 h. The crude mixture was then poured onto ice-cold water. The precipitate was dissolved in minimum amount of hot dichloromethane first, then precipitated in large amount of methanol. After filtration, the product was dried *in vacuo* to yield 4-bromo-*N*-ethyl-1,8-naphthalimide as a light yellow solid.[6]

For Sonogashira cross-coupling reactions and TMS deprotections, the same procedures were followed as previously described. The product was purified by flash chromatography on silica gel ((hexane/EtOAc, 9:1, *v*/*v*) to give 4-

ethynyl-*N*-ethyl-1,8-naphthalimide as a yellow-brown solid.

6-Bromo-2-ethyl-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione $C_{14}H_{10}BrNO_2$ light yellow solid (85% yield), R_f = 0.76 (hexane/EtOAc, 5:1), δ_H (300 MHz, $CDCl_3$) 8.61 (dd, *J* = 7.3, 1.1, 1H), 8.50 (dd, *J* = 8.5, 1.1, 1H), 8.36 (d, *J* = 7.9, 1H), 7.99 (d, *J* = 7.9, 1H), 7.81 (dd, *J* = 8.5, 7.3, 1H), 4.23 (q, *J* = 7.1, 2H), 1.34 (t, *J* = 7.1, 3H). δ_C (101 MHz, $CDCl_3$) δ 163.3, 133.1, 131.9, 131.1, 131.0, 130.5, 130.1, 128.8, 128.0, 123.1, 122.2, 35.6, 13.3. M/z: (LD^+) 304.0 $[M]^+$. ν/cm^{-1} 2980, 1693, 1659, 1587, 1568, 1339, 1242, 1060, 961, 775, 747.

2-Ethyl-6-((trimethylsilyl)ethynyl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione $C_{19}H_{19}NO_2Si$ yellow-brown solid (92% yield), R_f = 0.83 (hexane/EtOAc, 5:1), δ_H (300 MHz, $CDCl_3$) 8.61 (d, *J* = 7.9, 2H), 8.51 (dd, *J* = 7.6, 4.0, 1H), 7.88 (dd, *J* = 8.6, 4.8, 1H), 7.82 (dd, *J* = 9.8, 5.9, 1H), 4.23 (q, *J* = 7.1, 2H), 1.32 (t, *J* = 7.1, 3H), 0.38 (s, 9H),. δ_C (101 MHz, $CDCl_3$) 163.7, 163.5, 132.3, 131.7, 131.5, 131.1, 130.1, 127.8, 127.4, 127.2, 122.9, 122.3, 105.2, 101.2, 35.6, 13.3, -0.1. ν/cm^{-1} 2980, 1922, 1692, 1652, 1587, 1567, 1338, 1241, 1059, 961, 774, 747.

2-Ethyl-6-ethynyl-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione $C_{16}H_{11}NO_2$ yellow solid (76% yield), R_f = 0.75 (hexane/EtOAc, 5:1), δ_H (400 MHz, $CDCl_3$) 8.74 – 8.59 (m, 1H), 8.54 (d, *J* = 7.6, 1H), 7.95 (d, *J* = 7.6, 1H), 7.84 (dd, *J* = 8.2, 7.6, 1H), 4.26 (q, *J* = 7.1, 1H), 3.76 (s, 1H), 1.36 (t, *J* = 7.1, 2H). δ_C (101 MHz, $CDCl_3$) 163.7, 163.4, 132.1, 131.9, 131.6, 130.1, 127.9, 127.7, 126.1, 123.0, 122.8, 86.5, 80.3, 35.6, 13.3. M/z: (ES^+) 250.1 $[M+H]^+$. ν/cm^{-1} 3224, 2980, 2099, 1690, 1649, 1588, 1342, 1238, 1059, 869, 781, 687.

Synthesis of 7-ethynylcoumarin

7-Hydroxycoumarin (1.00 g, 6.2 mmol) was initially combined with anhydrous pyridine (20 mL). The reaction mixture was placed on ice and allowed to cool prior to the addition of trifluoromethanesulfonic anhydride (1.00 mL, 5.9 mmol). The mixture was stirred in an ice bath for 4 h, after which diethyl ether (100 mL) was added. The first precipitate was separated by filtration and discarded. Following a second precipitation after the addition of 1 M HCl (5 mL), the product was collected by filtration, and dried under vacuum to give 1.66 g of 2-oxo-2*H*-chromen-7-yl trifluoromethanesulfonate.[7]

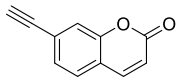
For Sonogashira cross-coupling reaction and TMS deprotection, the same procedures was followed as previously described. The final product was purified using chromatography on silica gel ((hexane/EtOAc, 5:1, *v*/*v*).

2-Oxo-2*H*-chromen-7-yl trifluoromethanesulfonate $C_{10}H_5F_3O_5S$, light yellow solid (91% yield), R_f = 0.24 (hexane/EtOAc, 3:1), δ_H (300 MHz, $CDCl_3$) 7.76 (d, *J* = 9.6, 1H), 7.62 (d, *J* = 8.5, 1H), 7.32 - 7.20 (m, 2H), 6.50 (d, *J* = 9.6, 1H). δ_C (101 MHz, $CDCl_3$) 159.3, 154.5, 150.8, 142.2, 129.5, 120.2, 118.8, 117.7, 117.0, 110.5. δ_F (282 MHz, $CDCl_3$) -72.6, M/z: (AP^+) 295.0 $[M+H]^+$. ν/cm^{-1} 3092, 3058, 1719, 1608, 1421, 1400, 1221, 1196, 1131, 1103, 980, 851, 747.

7-((Trimethylsilyl)ethynyl)-2*H*-chromen-2-one $C_{14}H_{14}O_2Si$, yellow-brown solid (95% yield), R_f = 0.83 (hexane/EtOAc, 2:1) δ_H (300 MHz, $CDCl_3$) 7.69 (d, *J* = 9.5, 1H,), 7.38 (m, 3H), 6.43 (d, *J* = 9.5, 1H), 0.29 (s, 9H). δ_C (101 MHz, $CDCl_3$) 160.4, 153.6,

142.8, 134.1, 134.0, 129.8, 128.5, 128.0, 127.6, 126.8, 119.9, 118.8, 117.0, 103.2, 98.6, -0.2. M/z: (ES^+) 265.2 $[M + Na]^+$. ν/cm^{-1} 3059, 2963, 2156, 1721, 1609, 1396, 1243, 1134, 985, 838, 765.

7-Ethynyl-2*H*-chromen-2-one $C_{11}H_6O_2$, yellow solid (83% yield), R_f = 0.74 (hexane/EtOAc, 2:1), δ_H

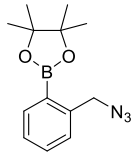


(300 MHz, $CDCl_3$) 7.68 (d, *J* = 9.5, 1H), 7.44 - 7.36 (m, 3H), 6.43 (d, *J* = 9.5, 1H), 3.27 (s, 1H). δ_C (101 MHz, $CDCl_3$) 160.1, 153.5, 142.6, 127.9, 127.6, 125.5, 120.1, 119.0, 117.1, 82.0, 80.6. M/z: (ES^+) 193.2 $[M+Na]^+$. ν/cm^{-1} 3226, 2980, 2099, 1690, 1649, 1588, 1342, 1237, 1058, 869, 781.

6.2 Azido substitution

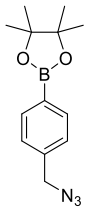
Bromo-substitued starting material (1.00 g, 3.4 mmol) and sodium azide (0.30 g, 5.1 mmol) were charged into a round-bottom flask. 5 mL DMSO was added and the reaction mixture was heated at 80 ^{0}C for 30 minutes. After cooling to room temperature, water was added and the reaction mixture was extracted with diethyl ether three times. The product was concentrated *in vacuo*.

2-(2-(Azidomethyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (9a) $C_{13}H_{18}BN_3O_2$, liquid (99%



yield), R_f = 0.78 (PE/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.93 (dd, *J* = 7.6, 1.4, 1H), 7.54 – 7.46 (m, 1H), 7.42 – 7.34 (m, 2H), 4.70 (s, 2H), 1.40 (s, 12H), ν/cm^{-1} 3597, 3039, 2979, 2092, 1601, 1345, 1210, 1143, 961, 848, 752, 656.

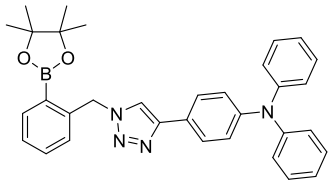
2-(4-(Azidomethyl)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (9b) $C_{13}H_{18}BN_3O_2$, colorless



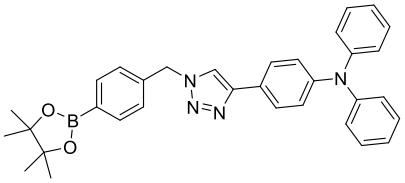
crystalline (99% yield), R_f = 0.79 (PE/EtOAc, 6:1), δ_H (300 MHz, $CDCl_3$) 7.87 (d, *J* = 8.0, 2H), 7.34 (d, *J* = 8.0, 2H), 4.35 (s, 2H), 1.37 (s, 12H),. ν/cm^{-1} 2981, 2935, 2095, 1612, 1390, 1356, 1337, 1269, 1140, 1086, 857, 819, 735, 655.

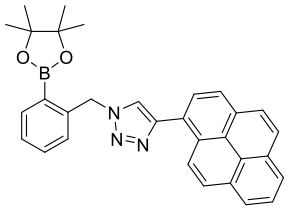
6.3 CuAAC reaction (second generation "*click-fluors*")

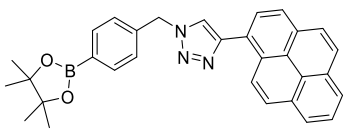
***N,N*-Diphenyl-4-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-**

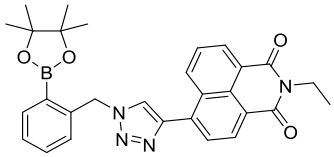


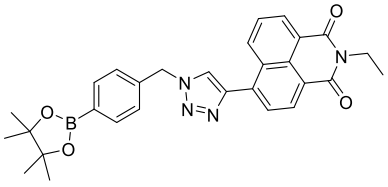
yl)aniline (10a) $C_{33}H_{33}BN_4O_2$ yellow solid (46% yield), R_f = 0.23 (hexane/EtOAc, 5:1), mp 113 – 115 °C, δ_H (300 MHz, $CDCl_3$) 7.92 (dd, *J* = 7.3, 1.4, 1H), 7.69 (s, 1H), 7.67 – 7.60 (m, 2H), 7.42 (td, *J* = 7.5, 1.6, 1H), 7.33 (td, *J* = 7.4, 1.3, 1H), 7.28 – 7.18 (m, 6H), 7.13 – 7.05 (m, 6H), 7.04 – 6.96 (m, 2H), 5.89 (s, 2H), 1.36 (s, 12H); δ_C (101 MHz, $CDCl_3$) 147.6, 141.0, 136.7, 131.9, 129.3, 129.1, 127.9, 126.5, 124.9, 124.4, 123.8, 123.0, 119.3, 84.2, 53.4, 24.9; δ_B (128 MHz, $CDCl_3$) 31.2. M/z: (ES^+) 529.4 $[M + H]^+$. High resolution MS calc. for formula $C_{33}H_{34}BN_4O_2$: 529.2775; found: 529.2780. ν/cm^{-1} 3599, 3033, 2976, 2097, 1592, 1503, 1291, 956, 831, 744.

***N,N*-Diphenyl-4-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline (10b)**  $C_{33}H_{33}BN_4O_2$ yellow solid (41% yield), R_f = 0.22 (hexane/EtOAc, 5:1), mp 173 – 175 °C, δ_H (300 MHz, $CDCl_3$) 7.82 (d, *J* = 8.1, 2H), 7.68 – 7.60 (m, 2H), 7.56 (s, 1H), 7.32 – 7.19 (m, 6H), 7.12 – 7.05 (m, 6H), 7.05 – 6.98 (m, 2H), 5.55 (s, 2H), 1.33 (s, 12H); δ_C (101 MHz, $CDCl_3$) 148.1, 147.8, 147.5, 137.6, 135.5, 129.3, 127.3, 126.6, 124.7, 124.5, 123.7, 123.0, 118.9, 84.0, 54.2,24.9; δ_B (128 MHz, $CDCl_3$) 30.9. M/z: (ES^+) 529.5 $[M + H]^+$. High resolution MS calc. for formula $C_{33}H_{34}BN_4O_2$: 529.2775; found: 529.2772. ν/cm^{-1} 3596, 3040, 2977, 2102, 1588, 1487, 1360, 1273, 1209, 961, 834, 752, 694.

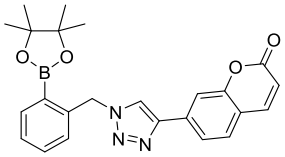
4-(Pyren-1-yl)-1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1H-1,2,3-triazole (11a)  $C_{31}H_{28}BN_3O_2$ brown solid (70% yield), R_f = 0.22 (hexane/EtOAc, 6:1), mp 92 – 93 °C, δ_H (300 MHz, $CDCl_3$) 7.91 (dd, J = 7.3, 1.4, 1H), 7.69 (s, 1H), 7.67 – 7.60 (m, 2H), 7.44 (td, *J* = 7.4, 1.5, 1H), 7.35 (td, *J* = 7.4, 1.5, 1H), 7.31 – 7.20 (m, 6H), 7.14 – 7.05 (m, 5H), 7.05 – 6.97 (m, 2H), 5.90 (s, 2H), 1.36 (s, 12H); δ_C (101 MHz, $CDCl_3$) 147.2, 140.9, 136.8, 132.0, 131.4, 131.2, 130.9, 129.6, 128.4, 128.0, 127.7, 127.4, 127.1, 126.0, 125.6, 125.3, 125.0, 124.9, 124.8, 123.1, 84.3, 53.6,25.0; δ_B (128 MHz, $CDCl_3$) 31.1. M/z: (ES^+) 486.2 $[M + H]^+$. High resolution MS calc. for formula $C_{31}H_{29}BN_3O_2$: 486.2353; found: 486.2361. ν/cm^{-1} 3044, 2977, 1601, 1443, 1345, 1318, 1142, 1051, 963, 844, 760, 711, 655.

4-(Pyren-1-yl)-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (11b)  $C_{31}H_{28}BN_3O_2$, Brown solid (58% yield), R_f = 0.26 (hexane/EtOAc, 5:1), mp 212 – 213, δ_H (400 MHz, $CDCl_3$) 8.68 (d, J = 9.3, 1H), 8.24 – 8.15 (m, 4H), 8.14 – 7.98 (m, 4H), 7.91 (d, *J* = 8.0, 2H), 7.84 (s, 1H), 7.43 (d, *J* = 8.0, 2H), 5.71 (s, 2H), 1.38 (s, 12H); δ_C (101 MHz, $CDCl_3$) 148.0, 137.5, 135.6, 131.3, 131.3, 130.9, 128.5, 128.2, 127.8, 127.5, 127.3, 127.2, 126.1, 125.4, 125.1, 125.0, 124.8, 124.8, 122.8, 84.1, 54.4, 24.9; δ_B (128 MHz, $CDCl_3$) 30.9. M/z: (ES^+) 486.2 $[M + H]^+$. High resolution MS calc. for formula $C_{31}H_{29}BN_3O_2$: 486.2353; found: 486.2363. ν/cm^{-1} 3043, 2977, 1614, 1401, 1362, 1142, 1092, 964, 848, 716.

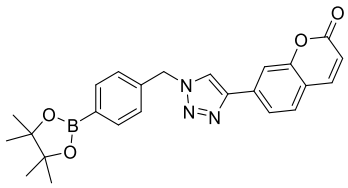
2-Ethyl-6-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1H-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2H)-dione (12a)  $C_{29}H_{29}BN_4O_4$ white solid (65% yield), R_f = 0.41 (hexane/EtOAc, 3:1), mp 201 – 203 °C, δ_H (300 MHz, $CDCl_3$) 9.01 (dd, *J* = 8.6, 1.1, 1H), 8.62 (dd, *J* = 7.3, 1.1, 1H), 8.58 (d, *J* = 7.6, 1H), 8.00 (s, 1H), 7.98 – 7.92 (m, 1H), 7.88 (d, *J* = 7.6, 1H), 7.75 (dd, *J* = 8.6, 7.3, 1H), 7.56 – 7.46 (m, 1H), 7.46 – 7.36 (m, 2H), 6.00 (s, 2H), 4.24 (q, *J* = 7.1, 2H), 1.38 (s, 12H), 1.34 (t, *J* = 7.1, 3H); δ_C (101 MHz, $CDCl_3$) 164.0, 163.7, 145.6, 140.3, 137.0, 134.5, 132.7, 132.1, 131.3, 130.7, 129.7, 129.2, 128.8, 128.3, 127.2, 127.0, 123.6, 122.8, 122.3, 84.3, 53.7, 35.5, 25.0, 13.4; δ_B (128 MHz, $CDCl_3$) 31.1. M/z: (AP^+) 531.2 $[M + Na]^+$. High resolution MS calc. for formula $C_{29}H_{29}BN_4O_4Na$: 531.2180; found: 531.2189. ν/cm^{-1} 2981, 1723, 1616, 1357, 1141, 1088, 931, 796, 703.

2-Ethyl-6-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2H)-dione (12b) 

$C_{29}H_{29}BN_4O_4$ white solid (56% yield), R_f = 0.36 (hexane/EtOAc, 3:1), mp 218 – 220 °C, δ_H (300 MHz, $CDCl_3$) 7.94 (dd, *J* = 7.4, 1.3, 1H), 7.90 (s, 1H), 7.80 (dd, *J* = 8.1, 1.5, 1H), 7.69 (d, *J* = 9.5, 1H), 7.64 (d, *J* = 1.3, 1H), 7.51 (d, *J* = 8.1, 1H), 7.47 (td, *J* = 7.5, 1.5, 1H), 7.38 (td, *J* = 7.6, 1.2, 1H), 7.35 (d, *J* = 7.7, 1H), 6.40 (d, *J* = 9.5, 1H), 5.92 (s, 2H), 1.38 (s, 12H); δ_C (101 MHz, $CDCl_3$) 160.8, 154.5, 145.9, 143.1, 140.4, 136.9, 134.6, 132.0, 129.6, 128.4, 128.2, 121.7, 120.9, 118.3, 116.3, 113.3, 84.3, 53.6, 25.0; δ_B (128 MHz, $CDCl_3$) 30.7. M/z: (AP^+) 509.2 $[M + H]^+$. High resolution MS calc. for formula $C_{29}H_{30}BN_4O_4$: 509.2360; found: 509.2357. ν/cm^{-1} 2978, 1716, 1615, 1359, 1143, 1090, 937, 846, 794, 711.

7-(1-(2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13a) 

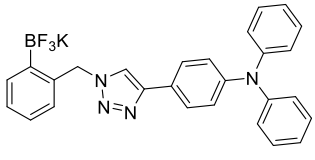
$C_{24}H_{24}BN_3O_4$, light brown solid (63% yield), R_f = 0.35 (hexane/EtOAc, 2:1), mp 89 – 91 °C, δ_H (400 MHz, $CDCl_3$) 7.94 (dd, *J* = 7.4, 1.3, 1H), 7.90 (s, 1H), 7.80 (dd, *J* = 8.1, 1.5, 1H), 7.69 (d, *J* = 9.5, 1H), 7.64 (d, *J* = 1.3, 1H), 7.51 (d, *J* = 8.1, 1H), 7.47 (td, *J* = 7.6 & 1.2, 1H), 7.38 (td, *J* = 7.6, 1.2, 1H), 7.35 (d, *J* = 7.6, 1H), 6.40 (d, *J* = 9.5, 1H), 5.92 (s, 2H), 1.38 (s, 12H); δ_H (300 MHz, DMSO-d_6) 8.68 (s, 1H), 8.35 (s, 2H), 8.07 (d, *J* = 9.5, 1H), 7.89 – 7.81 (m, 2H), 7.78 (d, *J* = 7.9, 1H), 7.70 – 7.62 (m, 1H), 7.42 – 7.28 (m, 2H), 7.11 (d, *J* = 6.5, 1H), 6.48 (d, *J* = 9.5, 1H), 5.87 (s, 2H). δ_C (101 MHz, $CDCl_3$) 160.8, 154.5, 145.9, 143.1, 140.4, 136.9, 134.6, 132.0, 129.6, 128.4, 128.2, 121.7, 120.9, 118.3, 116.3, 113.3, 84.3, 53.6, 25.0; δ_B (128 MHz, $CDCl_3$) 31.0. M/z: (ES^+) 452.2 $[M + Na]^+$. High resolution MS calc. for formula $C_{24}H_{24}BN_3O_4Na$: 452.1758; found: 452.1754. ν/cm^{-1} 2947, 1735, 1612, 1221, 1206, 843, 759, 663.

7-(1-(2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13b) 

$C_{24}H_{24}BN_3O_4$, light brown solid (45% yield), R_f = 0.38 (hexane/EtOAc, 2:1), mp 221 – 223 °C, δ_H (300 MHz, $CDCl_3$) 7.84 (d, *J* = 8.1, 2H), 7.78 (s, 1H), 7.77 (dd, *J* = 8.1, 1.6, 1H), 7.69 (d, *J* = 9.5, 1H), 7.67 (s, 1H), 7.50 (d, *J* = 8.1, 1H), 7.34 (d, *J* = 8.1, 2H), 6.39 (d, *J* = 9.5, 1H), 5.62 (s, 2H), 1.34 (s, 12H); δ_C (101 MHz, $CDCl_3$) 160.7, 154.4, 146.5, 143.1, 137.1, 135.6, 134.2, 128.4, 127.4, 121.8, 120.7, 118.4, 116.4, 113.4, 84.1, 54.4, 24.9; δ_B (128 MHz, $CDCl_3$) 30.7. M/z: (ES^+) 452.2 $[M + Na]^+$. High resolution MS calc. for formula $C_{24}H_{24}BN_3O_4Na$: 452.1758; found: 452.1763. ν/cm^{-1} 2953, 1726, 1615, 1233, 1198, 861, 665.

6.4 Pinacol deprotection step 1: Trifluoroborate salts

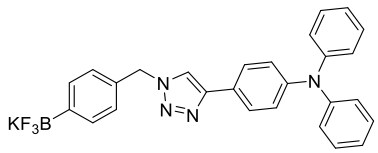
The procedure for the synthesis of the trifluoroborate salts is the same as previously described.

Potassium (2-((4-(4-(diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)trifluoroborate 

$C_{27}H_{21}BF_3KN_4$, mp 238 – 239 °C, δ_H (300 MHz, CD_3OD) 8.09 (s, 1H), 7.66 – 7.60 (m, 3H), 7.30 – 7.25 (m, 4H), 7.18 – 7.01 (m, 11H), 5.78 (s, 2H); δ_C (101 MHz, CD_3OD) 147.8, 147.6, 137.7, 132.3, 129.0, 127.4, 126.5, 126.4, 126.1, 124.5, 124.3, 123.1, 122.9, 120.4, 53.5. δ_F (282

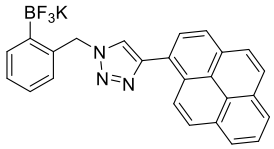
MHz, CD_3OD) -138.3 ν/cm^{-1} 3038, 1589, 1487, 1274, 1200, 955, 803, 752, 694. MS was not readily obtained, this intermediate was used without MS analysis.

Potassium (4-((4-(4-(diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)trifluoroborate



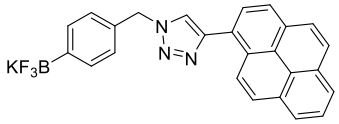
$C_{27}H_{21}BF_3KN_4$, mp 289 – 291 °C, δ_H (300 MHz, CD_3OD) 8.12 (s, 1H), 7.65 (dt, *J* = 8.7, 2.1, 2H), 7.54 (d, *J* = 7.8, 2H), 7.31 – 7.20 (m, 6H), 7.08 – 7.02 (m, 8H) 5.55 (s, 2H); δ_C (101 MHz, CD_3OD) 147.9, 147.5, 132.4, 131.8, 129.1, 126.3, 126.2, 124.3, 124.2, 123.0, 120.0, 54.2. ν/cm^{-1} 2987, 2100, 1588, 1487, 1279, 1220, 962, 844, 753, 695. MS was not readily obtained, this intermediate was used without MS analysis.

4-(Pyren-1-yl)-1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt $C_{25}H_{16}BN_3KF_3$,



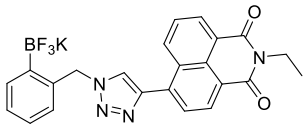
mp 176 – 177 , δ_H (300 MHz, $(CD_3)_2CO$) 8.99 (d, *J* = 9.4, 1H), 8.52 (s, 1H), 8.34 – 8.24 (m, 4H), 8.24 – 8.13 (m, 3H), 8.11 – 8.02 (m, 1H), 7.74 – 7.64 (m, 1H), 7.27 – 7.04 (m, 3H), 5.95 (s, 2H); δ_C (101 MHz, $(CD_3)_2CO$) 132.7, 127.6, 127.5, 127.4, 127.3, 126.9, 126.2, 126.1, 125.9, 125.4, 125.2, 124.9, 123.9, 53.5. δ_F (300 MHz, $(CD_3)_2CO$) -138.1 ν/cm^{-1} 3595, 3139, 3039, 1641, 1602, 1433, 1206, 949, 847, 751. MS was not readily obtained, this intermediate was used without MS analysis.

4-(Pyren-1-yl)-1-(4-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt $C_{25}H_{16}BN_3KF_3$,



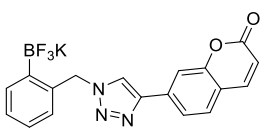
mp 221 – 224 °C, δ_H (300 MHz, CD_3OD) 8.48 (d, *J* = 9.3, 1H), 8.35 (s, 1H), 8.23 (t, *J* = 6.9, 3H), 8.19 – 8.09 (m, 4H), 8.03 (t, *J* = 7.7, 1H), 7.61 (d, *J* = 7.7, 2H), 7.34 (d, *J* = 7.7, 2H), 5.72 (s, 2H); δ_C (101 MHz, CD_3OD) 132.5, 131.9, 131.3, 130.8, 128.4, 127.8, 127.5, 126.9, 126.3, 125.9, 125.1, 124.8, 124.6, 124.5, 124.1, 54.3. δ_F (282 MHz, CD_3OD) -142.5 ν/cm^{-1} 3593, 3137, 3039, 1641, 1603, 1433, 1207, 949, 847, 751. MS was not readily obtained, this intermediate was used without MS analysis.

2-Ethyl-6-(1-(2-(trifluoro-λ^4-boranyl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-



1,3(2*H*)-dione, potassium salt $C_{23}H_{17}BF_3KN_4O_2$, mp 279 – 281 °C, δ_H (300 MHz, $(CD_3)_2CO$) 9.04 (1H, dd, *J* = 8.6, 1.1), 8.46 (1H, s), 8.38 (1H, dd, *J* = 7.2, 1.1), 8.29 (1H, d, *J* = 7.5), 7.81 (1H, d, *J* = 7.5), 7.72 - 7.67 (1H, m), 7.63 (1H, dd, *J* = 8.6, 7.3), 7.21 - 7.10 (3H, m), 5.92 (2H, s), 4.11 (2H, q, *J* = 7.1), 1.26 (3H, t, *J* = 7.1); δ_C (101 MHz, $(CD_3)_2CO$) 163.3, 162.9, 145.0, 138.3, 134.6, 132.7, 130.4, 130.1, 128.6, 128.3, 127.8, 126.9, 126.6, 126.5, 126.2, 124.9, 122.6, 121.6, 53.7, 34.8, 12.7. M/z: (ES$^+$) 527.1 $[M + K]^+$. High resolution MS calc. for formula $C_{23}H_{17}BN_4O_2F_3K_2$: 527.0671; found: 527.0679.

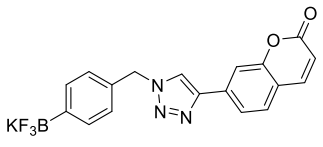
7-(1-(2-(trifluoro-λ^4-boranyl)benzyl)-1H-1,2,3-triazol-4-yl)-2H-chromen-2-one potassium salt



$C_{18}H_{12}BF_3KN_3O_2$, δ_H (300 MHz, DMSO-*d6*) 8.59 (s, 1H), 8.07 (d, *J* = 9.6, 1H), 7.88 – 7.80 (m, 2H), 7.77 (dd, *J* = 9.6, 3.6, 1H), 7.49 (dd, *J* = 7.0, 1.7, 1H), 7.14 – 7.01 (m, 2H), 6.79 (d, *J* = 6.9, 1H), 6.49 (dd, *J* = 9.6, 3.6, 1H), 5.75 (s, 2H); δ_C (101 MHz, DMSO-*d6*) 160.5, 154.6, 145.4, 144.5, 138.5, 134.9, 132.6, 129.6, 126.7, 126.6, 126.4, 123.4, 121.7, 118.6, 116.2, 112.6, 53.6; δ_F (282 MHz, DMSO-*d6*)

-135.8. M/z: (ES$^+$) 432.1 [M + Na]$^+$. High resolution MS calc. for formula $C_{18}H_{12}BN_3O_2F_3KNa$: 432.0509; found: 432.0502.

7-(1-(4-(trifluoro-λ^4-boranyl)benzyl)-1H-1,2,3-triazol-4-yl)-2H-chromen-2-one potassium salt

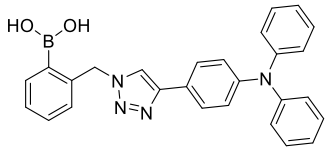


$C_{18}H_{12}BF_3KN_3O_2$, δ_H (300 MHz, DMSO-*d6*) 9.17 (dd, *J* = 8.6, 1.0, 1H), 8.91 (s, 1H), 8.62 – 8.48 (m, 2H), 8.13 (d, *J* = 7.7, 1H), 7.93 (dd, *J* = 8.6, 7.4, 1H), 7.36 (d, *J* = 7.6, 2H), 7.20 (d, *J* = 7.6, 2H), 5.64 (s, 2H). δ_C (101 MHz, DMSO-*d6*) 163.7, 145.3, 134.5, 133.1, 132.2, 131.3, 130.9, 129.9, 128.7, 128.1, 127.6, 126.7, 125.8, 122.9, 122.1, 54.2; δ_F (282 MHz, DMSO-*d6*) 139.1. MS was not readily obtained, this intermediate was used without MS analysis.

6.5 Pinacol deprotection step 2: Boronic acids

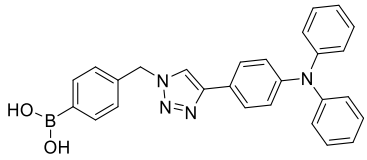
The procedure for the final step is the same as before.

(2-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14a)



$C_{27}H_{23}BN_4O_2$, yellow solid (82% yield), R_f = 0.76 (DCM/MeOH, 95:5), mp 156 – 158 °C, δ_H (300 MHz, $CDCl_3$) 8.07 (s, 1H), 7.60 (d, *J* = 8.4, 2H), 7.34 – 7.20 (m, 7H), 7.03 – 6.97 (m, 8H), 5.81 (s, 2H); δ_C (101 MHz, $CDCl_3$) 148.1, 147.4, 129.1, 128.7, 127.5, 126.6, 126.3, 124.7, 124.4, 123.1, 122.9, 120.4, 54.0; δ_B (128 MHz, CD_3OD) 29.3. M/z: (MALDI) 447.3 [M + H]$^+$. ν/cm^{-1} 3036, 2926, 1589, 1487, 1273, 974, 809, 751, 693.

(4-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14b)



$C_{27}H_{23}BN_4O_2$, brown solid (85% yield), R_f = 0.79 (DCM/MeOH, 95:5), mp 158 – 161 °C, δ_H (300 MHz, CD_3OD) 8.20 (s, 1H), 7.77 – 7.64 (m, 4H), 7.30 – 7.24 (m, 6H), 7.07 – 7.01 (m, 8H), 5.62 (s, 2H); δ_C (101 MHz, CD_3OD) 148.0, 147.6, 147.5, 134.2, 133.8, 129.1, 126.7, 126.2, 124.4, 124.0, 123.0, 122.9, 120.3, 53.6; δ_B (128 MHz, CD_3OD) 28.6. M/z: (MALDI) 447.3 [M + H]$^+$. ν/cm^{-1} 3314, 3035, 1588, 1487, 1329, 1273, 1180, 1019, 752, 693.

(2-((4-(Pyren-1-yl)-1H-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15a) $C_{25}H_{18}BN_3O_2$, light yellow solid (79% yield), R_f = 0.82 (DCM/MeOH, 9:1), mp 214 – 216 °C, δ_H NMR (300 MHz, $CDCl_3$) 8.43 – 8.36 (m, 1H), 7.80 (d, *J* = 6.8, 1H), 7.73 (d, *J* = 9.3, 1H), 7.67 (d, *J* = 7.5, 1H), 7.57 – 7.63 (m, 4H), 7.52 (d, *J* = 8.9, 1H), 7.38 (s, 1H), 7.23 – 7.12 (m, 2H), 7.09 (d, *J* = 7.9, 1H), 6.97 (d, *J* = 7.9, 1H), 6.04 (s, 2H). δ_C NMR (101 MHz, DMSO-*d6*) 146.9, 138.2, 135.1, 131.4, 131.1, 130.8, 128.5, 128.2, 127.8, 127.5, 126.9, 126.0, 125.7, 125.6, 125.3, 125.2, 124.8, 124.4, 53.7, 31.2; δ_B (128 MHz, DMSO-*d6*) 29.1. M/z: (MALDI) 404.2 [M + H]$^+$. ν/cm^{-1} 3284, 3149, 2987, 1716, 1611, 1411, 1354, 1182, 1050, 905, 839, 724.

(4-((4-(Pyren-1-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15b) $C_{25}H_{18}BN_3O_2$, light yellow solid (82% yield), R_f = 0.76 (DCM/MeOH, 95:5), mp 229 – 231 °C, δ_H (300 MHz, $CDCl_3$) 8.56 (d, *J* = 9.3, 1H), 8.15 (t, *J* = 3.9, 5H), 8.11 – 8.03 (m, 3H), 8.00 (d, *J* = 7.6, 2H), 7.80 (s, 1H), 7.23 (d, *J* = 8.6, 2H), 6.89 (d, *J* = 8.6, 2H), 5.56 (s, 2H); δ_C (101 MHz, $CDCl_3$) 147.81, 135.67, 135.26, 134.31, 131.30, 130.83, 128.55, 128.18, 127.85, 127.33, 127.17, 126.08, 125.40, 125.15, 124.96, 124.80, 123.55, 122.90, 54.31; δ_B (128 MHz, DMSO-*d6*) 27.9. M/z: (MALDI) 404.2 $[M + H]^+$. ν/cm^{-1} 3045, 1730, 1600, 1443, 1377, 1311, 1079, 844, 707.

(2-((4-(2-Ethyl-1,3-dioxo-2,3-dihydro-1*H*-benzo[de]isoquinolin-6-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (16a) $C_{23}H_{19}BN_4O_4$, light green solid (91% yield), R_f = 0.75 (DCM/MeOH, 95:5), mp 187 – 189 °C, δ_H (300 MHz, CD_3OD) 8.80 - 8.74 (m, 1H), 8.45 - 8.34 (m, 3H), 7.83 (d, *J* = 7.6, 1H), 7.66 (m, 1H), 7.56 - 7.39 (m, 4H), 5.79 (s, 2H), 4.13 (q, *J* = 7.0, 2H), 1.27 (t, *J* = 7.0, 3H); δ_C (101 MHz, $CDCl_3$) 167.6, 167.3, 149.1, 142.0, 136.3, 136.0, 134.6, 134.0, 133.2, 133.0, 132.7, 132.1, 131.6, 131.0, 130.9, 128.7, 126.3, 125.9, 58.1, 38.9, 16.0; δ_B (128 MHz, CD_3OD) 28.8. M/z: (MALDI) 427.2 $[M + H]^+$. ν/cm^{-1} 3344, 2924, 1694, 1646, 1587, 1337, 1244, 1064, 781, 756.

(4-((4-(2-Ethyl-1,3-dioxo-2,3-dihydro-1H-benzo[de]isoquinolin-6-yl)-1H-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (16b) $C_{23}H_{19}BN_4O_4$, light green solid (87% yield), R_f = 0.71 (DCM/MeOH, 95:5), mp 218 – 220 °C, δ_H NMR (300 MHz, $CDCl_3$) 8.89 (d, *J* = 8.6, 1H), 8.53 (dd, *J* = 17.3, 6.9, 2H), 7.66 – 8.16 (m, 5H), 7.29 – 7.51 (m, 2H), 5.68 (s, 2H), 4.21 (q, *J* = 7.2, 2H), 3.67 (s, 2H), 3.67 (s, 2H), 1.32 (t, *J* = 7.2, 3H). δ_C NMR (101 MHz, $CDCl_3$) 164.1, 163.8, 146.3, 134.5, 134.0, 132.7, 131.4, 130.6, 129.2, 128.8, 127.6, 127.4, 127.3, 123.3, 122.8, 122.6, 54.5, 35.6, 13.4. M/z: (MALDI) 427.2 $[M + H]^+$. ν/cm^{-1} 3357, 3127, 2966, 1684, 1641, 1589, 1333, 1245, 1064, 781, 700.

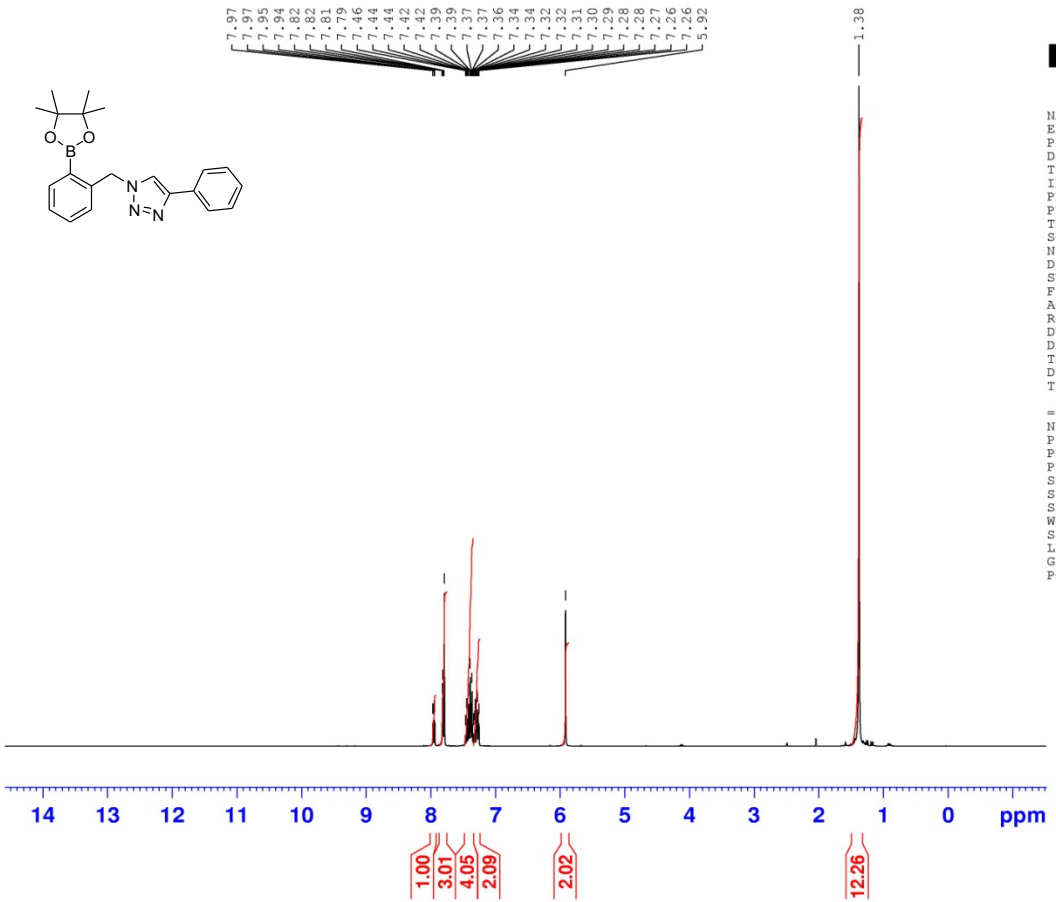
(2-((4-(2-Oxo-2H-chromen-7-yl)-1H-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17a) $C_{18}H_{14}BN_3O_4$, white solid (93% yield), R_f = 0.79 (DCM/MeOH, 95:5), mp 198 – 201 °C, δ_H NMR (300 MHz, CD_3OD) 8.42 (s, 1H), 7.96 (d, *J* = 9.5, 1H), 7.84 – 7.77 (m, 2H), 7.69 (d, *J* = 8.6, 1H), 7.50 – 7.37 (m, 4H), 6.44 (d, *J* = 9.5, 1H), 5.72 (s, 2H). δ_C NMR (101 MHz, DMSO-*d6*) 160.4, 154.6, 145.5, 144.4, 139.9, 135.0, 134.7, 130.2, 129.6, 128.6, 127.7, 123.6, 121.7, 118.7, 116.3, 112.7, 53.6, 51.1, 49.1; δ_B (128 MHz, CD_3OD) 28.8. M/z: (MALDI) 348.1 $[M + H]^+$. ν/cm^{-1} 3134, 1715, 1617, 1321, 1217, 943, 843, 754, 715.

(4-((4-(2-Oxo-2H-chromen-7-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17b) $C_{18}H_{14}BN_3O_4$, white solid (95% yield), R_f = 0.81 (DCM/MeOH, 95:5), mp 261 – 263 °C, δ_H NMR (300 MHz, DMSO-*d6*) 8.61 (s, 1H), 8.00 (d, *J* = 9.2, 1H), 7.92 (d, *J* = 8.2, 3H), 7.85 (s, 1H), 7.74 (d, *J* = 8.1, 1H), 7.41 (d, *J* = 8.4, 2H), 6.42 (d, *J* = 9.2, 1H), 5.75 (s, 2H); δ_C NMR (101 MHz, DMSO-*d6*) 160.4, 154.6, 145.8, 144.4, 137.9, 135.1,

134.9, 134.6, 129.6, 127.4, 123.6, 121.7, 118.7, 116.3, 112.7, 53.7, 50.7, 49.1. M/z: (MALDI) 348.1 $[M + H]^+$. ν/cm^{-1} 3332, 3098, 1710, 1614, 1567, 1340, 1153, 940, 844, 784, 713.

7. NMR spectra of the synthesised compounds

4-Phenyl-1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4a) ^{1}H NMR spectrum

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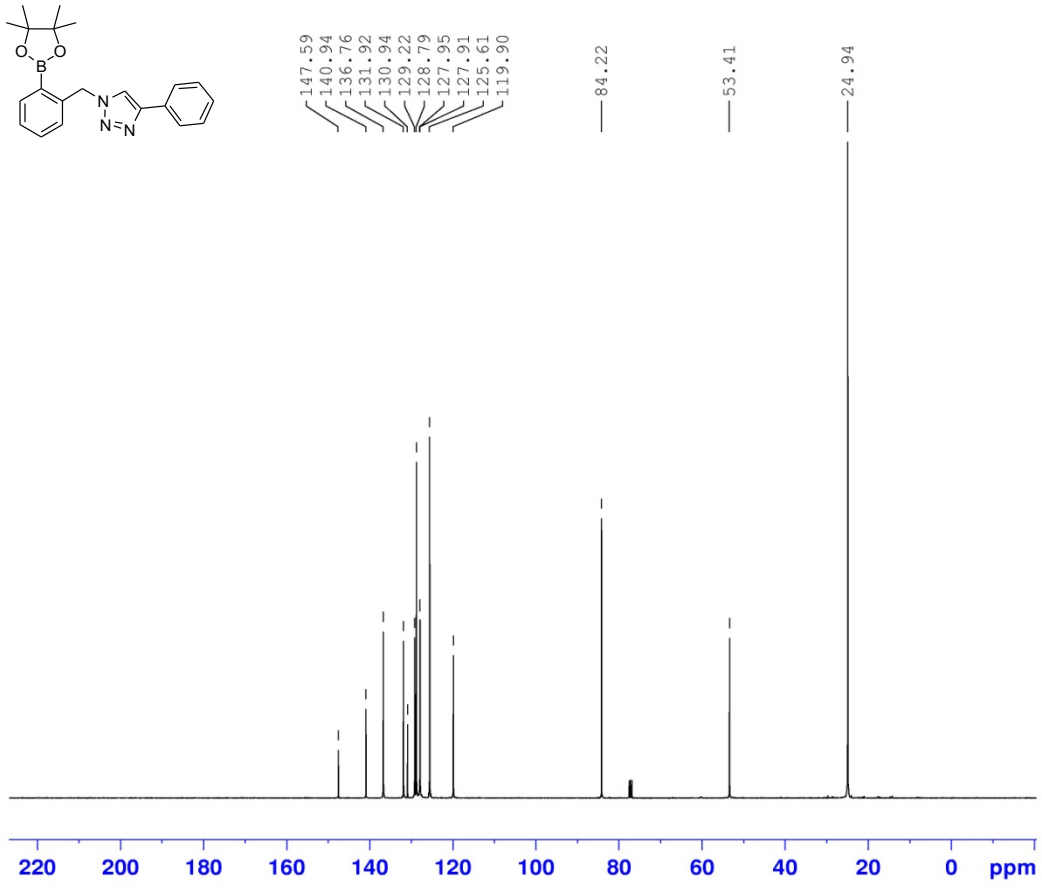
NAME          04-29-Fossey-35 WZ02-03
EXPNO                 10
PROCNO                 1
Date_           20130429
Time               23.15
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             2.7263477 sec
RG                    18
DW                83.200 usec
DE                12.89 usec
TE                290.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.80 usec
PL1                1.00 dB
PL1W          9.57725906 W
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

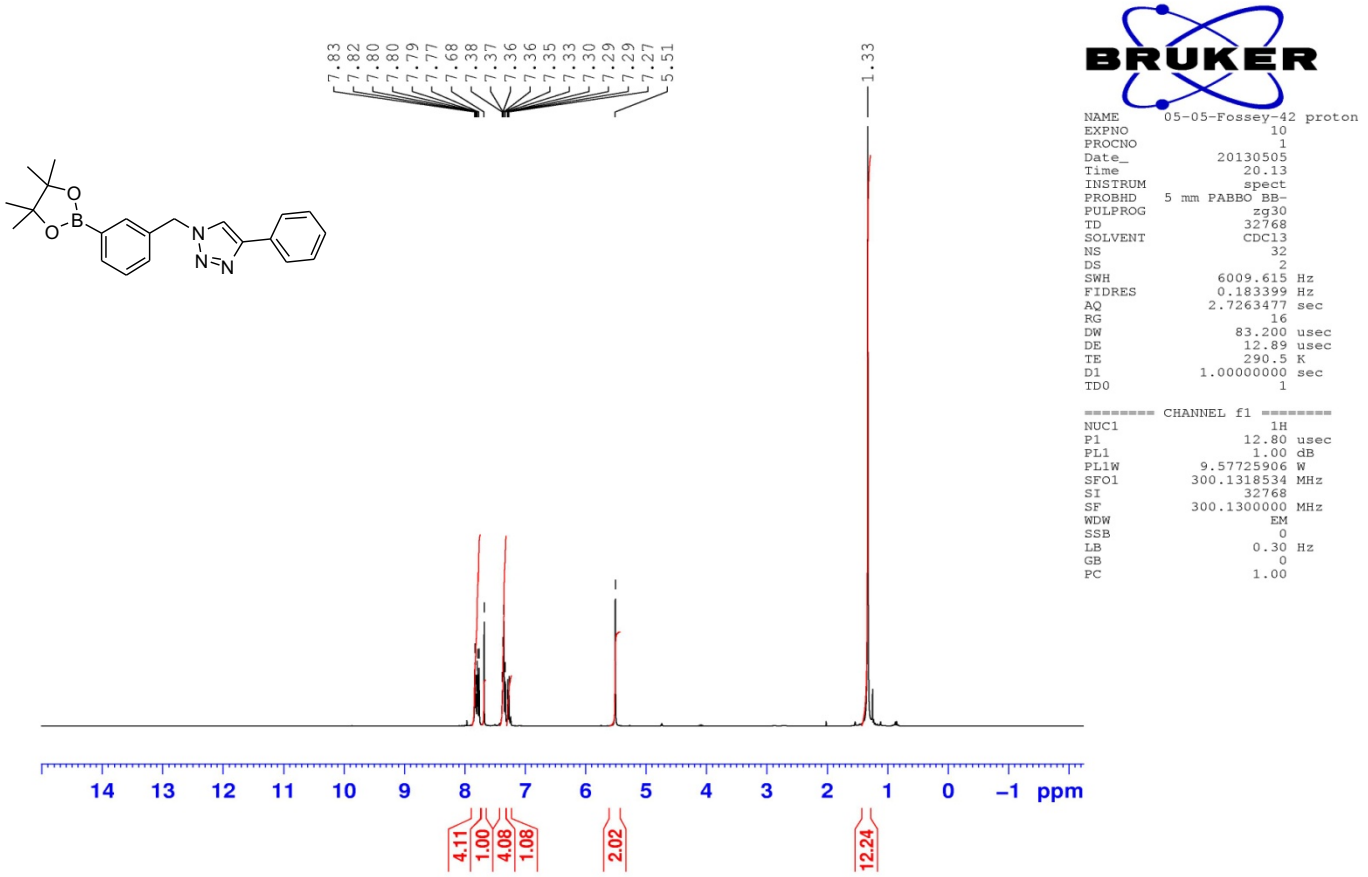
4-Phenyl-1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4a) ^{13}C NMR spectrum



BRUKER

NAME	04-29-Fossey-9 WZ02-03 C
EXPNO	10
PROCNO	1
Date_	20130429
Time	23.40
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	udeft
TD	18178
SOLVENT	CDC13
NS	380
DS	0
SWH	25252.525 Hz
FIDRES	1.389181 Hz
AQ	0.3599744 sec
RG	2050
DW	19.800 usec
DE	8.20 usec
TE	294.3 K
D1	3.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
D20	200.00000000 sec
TD0	380
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P12	2000.00 usec
P26	500.00 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
SP2	6.27 dB
SP8	6.27 dB
SPNAM2	Crp60comp.4
SPNAM8	Crp60,0.5,20.1
SPOAL2	0.500
SPOAL8	0.500
SPOFFS2	0.00 Hz
SPOFFS8	0.00 Hz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.00

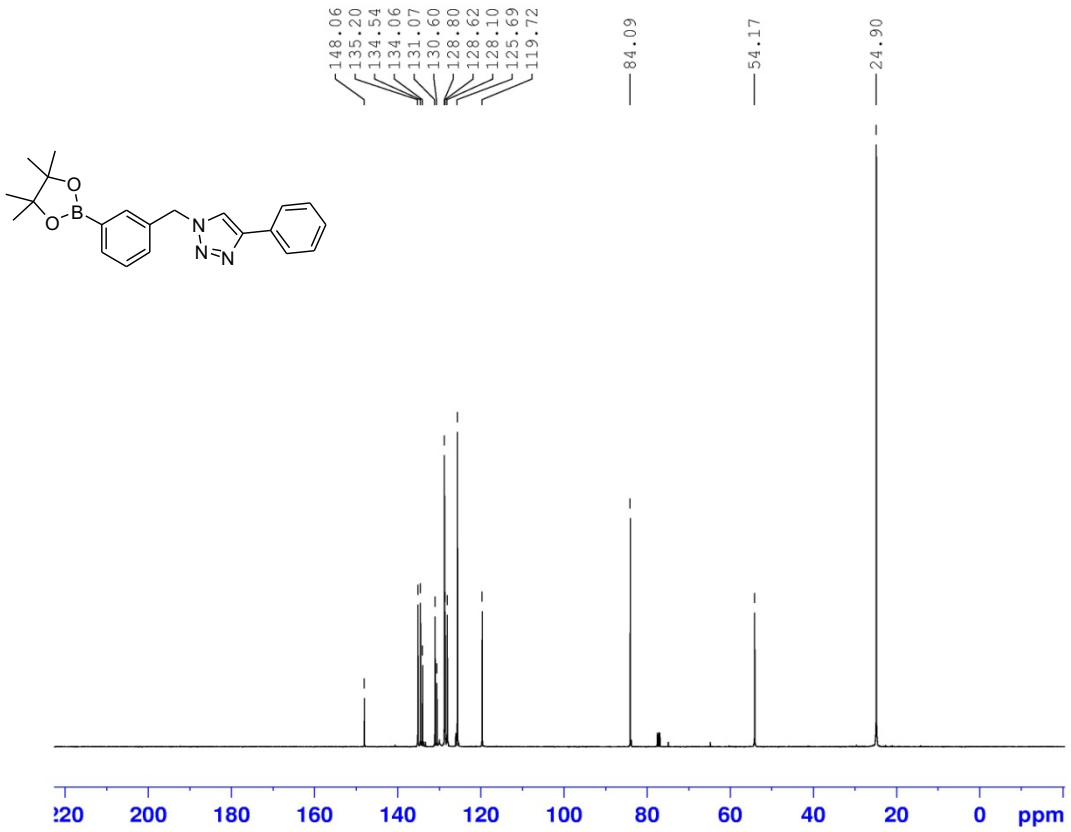
4-Phenyl-1-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4b) ^{1}H NMR spectrum



BRUKER

NAME	05-05-Fossey-42 proton
EXPNO	10
PROCNO	1
Date_	20130505
Time	20.13
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	16
DW	83.200 usec
DE	12.89 usec
TE	290.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

4-Phenyl-1-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4b) ^{13}C NMR spectrum



BRUKER

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NAME          05-05-Fossey-10 carbon
EXPNO                      10
PROCNO                      1
Date_                20130505
Time                    20.27
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                 udeft
TD                      18178
SOLVENT                 CDCl3
NS                        380
DS                          0
SWH                 25252.525 Hz
FIDRES              1.389181 Hz
AQ                  0.3599744 sec
RG                       2050
DW                     19.800 usec
DE                      8.20 usec
TE                     294.2 K
D1                3.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec
D20             200.00000000 sec
TD0                       380

======== CHANNEL f1 ========
NUC1                      13C
P1                       8.80 usec
P12                   2000.00 usec
P26                    500.00 usec
PL1                     -3.00 dB
PL1W             58.63890457 W
SFO1             100.6233333 MHz
SP2                      6.27 dB
SP8                      6.27 dB
SPNAM2          Crp60comp.4
SPNAM8        Crp60,0.5,20.1
SPOAL2                  0.500
SPOAL8                  0.500
SPOFFS2                 0.00 Hz
SPOFFS8                 0.00 Hz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                   90.00 usec
PL2                     -4.00 dB
PL12                    15.35 dB
PL2W             24.29185867 W
PL12W             0.28213742 W
SFO2             400.1316005 MHz
SI                        65536
SF               100.6127690 MHz
WDW                        EM
SSB                         0
LB                      2.00 Hz
GB                          0
PC                      1.00
```
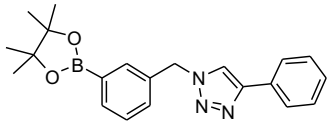
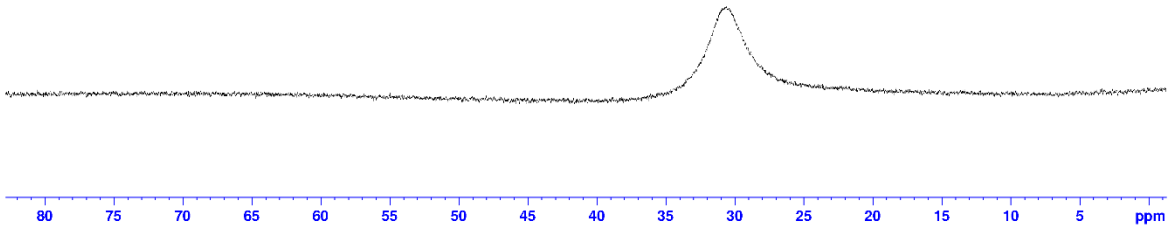

4-Phenyl-1-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4b) ^{11}B NMR spectrum





BRUKER

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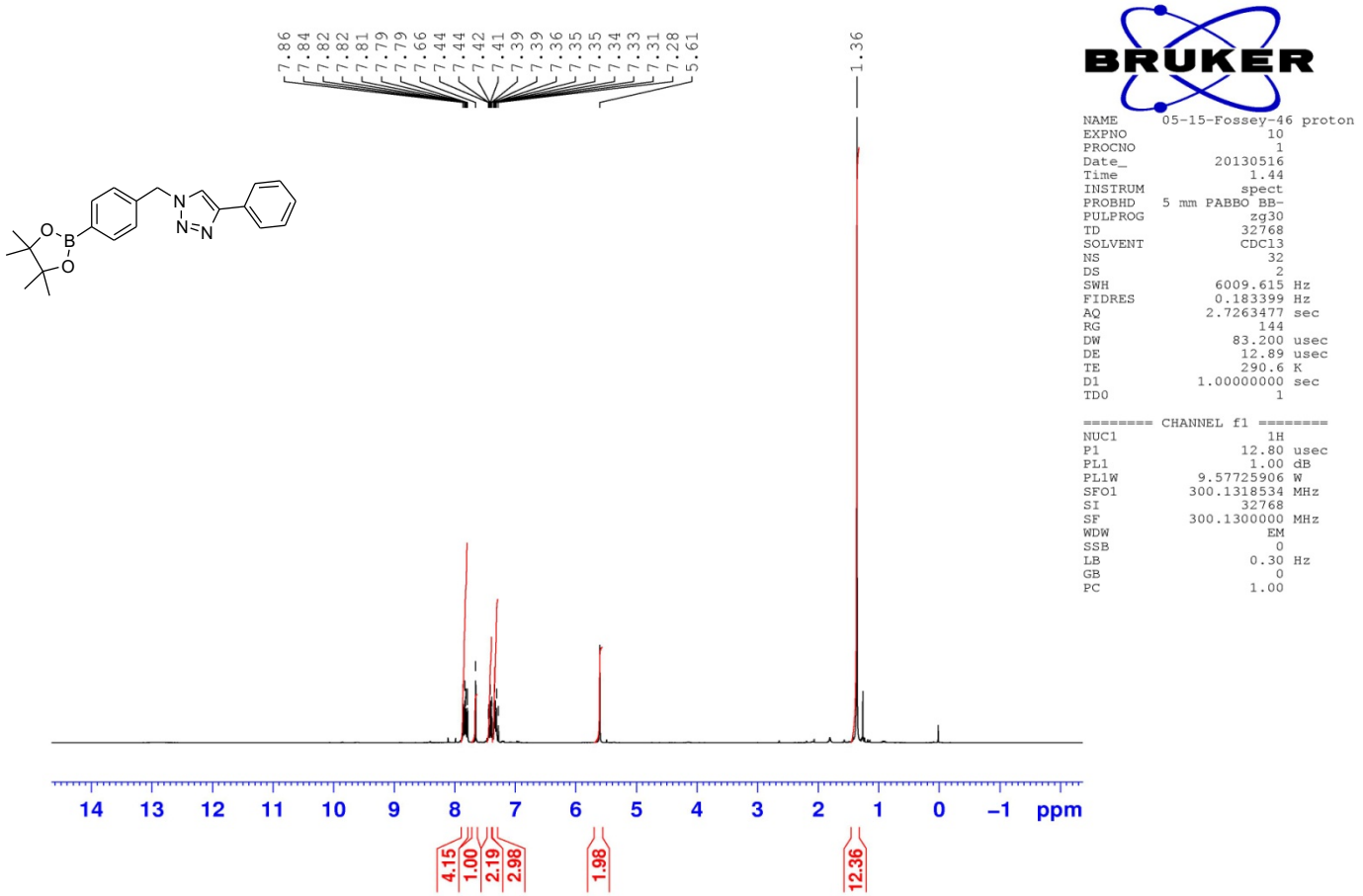
Current Data Parameters
NAME      04-14.4.Fossey
EXPNO               10
PROCNO             999

F2 - Acquisition Parameters
Date_          20140415
Time               0.06
INSTRUM           av400
PROBHD   5 mm BBO BB-1H
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                3072
DS                   4
SWH          25641.025 Hz
FIDRES        0.391251 Hz
AQ           1.2779520 sec
RG               812.7
DW              19.500 use
DE               6.50 use
TE              296.3 K
D1           1.00000000 sec
TD0                  6

======== CHANNEL f1 ========
NUC1               11B
P1               10.50 use
PL1              12.00 dB
SFO1        128.3583548 MHz

F2 - Processing parameters
SI                32768
SF          128.3583560 MHz
WDW                  EM
SSB       0
LB               2.00 Hz
GB        0
PC               1.00
```

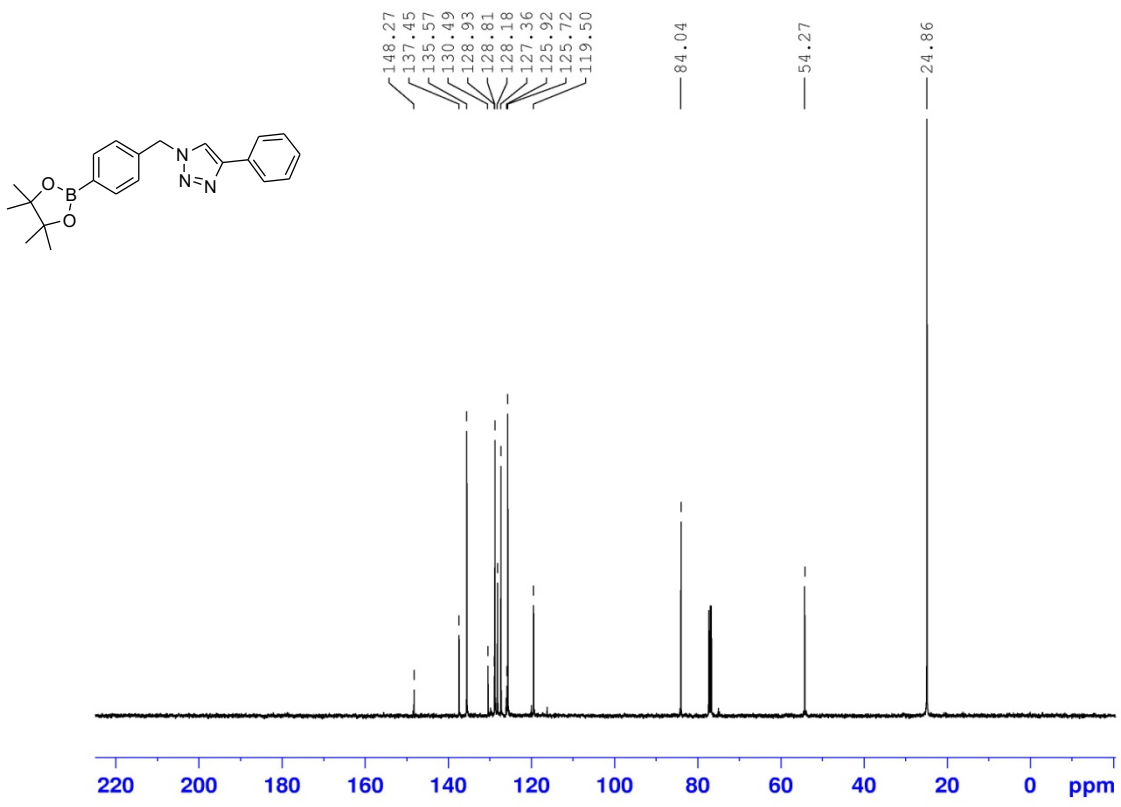
4-Phenyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4c) ^{1}H NMR spectrum




```
NAME          05-15-Fossey-46 proton
EXPNO                            10
PROCNO                            1
Date_                      20130516
Time                           1.44
INSTRUM                       spect
PROBHD    5 mm PABBO BB-
PULPROG                       zg30
TD                           32768
SOLVENT                      CDCl3
NS                              32
DS                               2
SWH                       6009.615 Hz
FIDRES                    0.183399 Hz
AQ                        2.7263477 sec
RG                             144
DW                          83.200 usec
DE                          12.89 usec
TE                          290.6 K
D1                    1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                           1H
P1                          12.80 usec
PL1                          1.00 dB
PL1W                 9.57725906 W
SFO1                300.1318534 MHz
SI                           32768
SF                  300.1300000 MHz
WDW                             EM
SSB                              0
LB                           0.30 Hz
GB                               0
PC                           1.00
```

4-Phenyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4c) ^{13}C NMR spectrum

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NAME          05-15-Fossey-30 carbon
EXPNO                 10
PROCNO                1
Date_           20130516
Time                2.13
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG            udeft
TD                 18178
SOLVENT            CDCl3
NS                   380
DS                     0
SWH            25252.525 Hz
FIDRES          1.389181 Hz
AQ              0.3599744 sec
RG                  2050
DW                19.800 usec
DE                 8.20 usec
TE                294.2 K
D1           3.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
D20        200.00000000 sec
TD0                  380

======== CHANNEL f1 ========
NUC1                13C
P1                 8.80 usec
P12             2000.00 usec
P26              500.00 usec
PL1               -3.00 dB
PL1W         58.63890457 W
SFO1         100.6233333 MHz
SP2                6.27 dB
SP8                6.27 dB
SPNAM2       Crp60comp.4
SPNAM8     Crp60,0.5,20.1
SPOAL2             0.500
SPOAL8             0.500
SPOFFS2            0.00 Hz
SPOFFS8            0.00 Hz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -4.00 dB
PL12              15.35 dB
PL2W         24.29185867 W
PL12W         0.28213742 W
SFO2         400.1316005 MHz
SI                65536
SF           100.6127690 MHz
WDW                  EM
SSB                   0
LB                 2.00 Hz
GB                    0
PC                 1.00
```
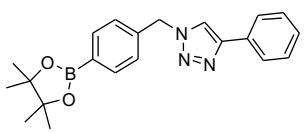

4-Phenyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (4c) ^{11}B NMR spectrum

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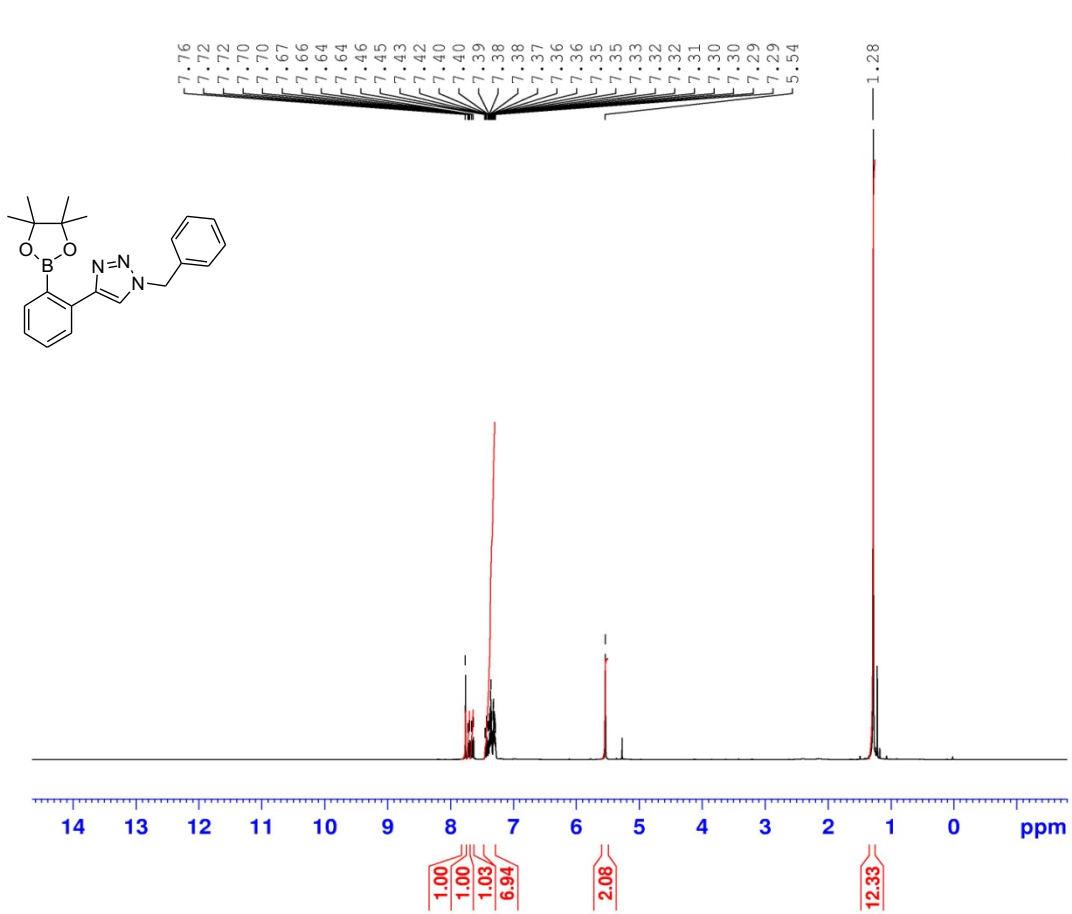
Current Data Parameters
NAME      04-14.5.Possey
EXPNO                 10
PROCNO               999

F2 - Acquisition Parameters
Date_           20140415
Time              2.10
INSTRUM           av400
PROBHD   5 mm BBO BB-1H
PULPROG           zg30
TD               65536
SOLVENT           CDCl3
NS                3072
DS                   4
SWH          25641.025 Hz
FIDRES        0.391251 Hz
AQ           1.2779520 sec
RG               812.7
DW              19.500 usec
DE               6.50 usec
TE              296.4 K
D1           1.00000000 sec
TD0                  6

======== CHANNEL f1 ========
NUC1               11B
P1               10.50 usec
PL1              12.00 dB
SFO1        128.3583548 MHz

F2 - Processing parameters
SI                32768
SF          128.3583560 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

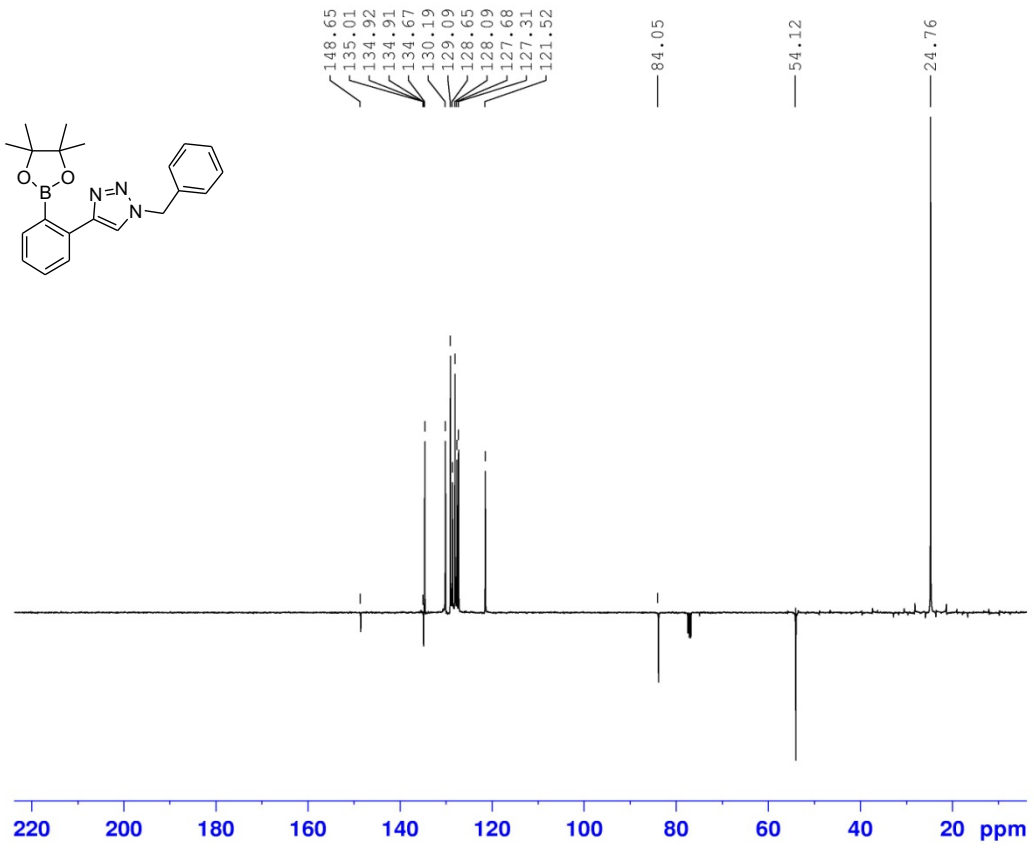
1-Benzyl-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7a) ^{1}H NMR spectrum



BRUKER

NAME	08-13-Fossey-33
EXPNO	10
PROCNO	1
Date_	20130813
Time	22.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	36
DW	83.200 usec
DE	12.89 usec
TE	290.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

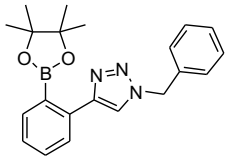
1-Benzyl-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7a) ^{13}C NMR spectrum



BRUKER

NAME	08-13-Fossey-26
EXPNO	10
PROCNO	1
Date_	20130814
Time	4.34
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	294.9 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

1-Benzyl-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7a) ^{11}B NMR spectrum

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Current Data Parameters
NAME      04-14.6.Fossey
EXPNO                 10
PROCNO               999

P2 - Acquisition Parameters
Date_           20140415
Time               4.13
INSTRUM           av400
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           25641.025 Hz
FIDRES         0.391251 Hz
AQ             1.2779520 sec
RG                812.7
DW               19.500 use
DE                6.50 use
TE              296.3 K
D1             1.00000000 sec
TD0                   6

======== CHANNEL f1 ========
NUC1                 11B
P1                10.50 use
PL1               12.00 dB
SFO1        128.3583548 MHz

P2 - Processing parameters
SI                32768
SF          128.3583560 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```
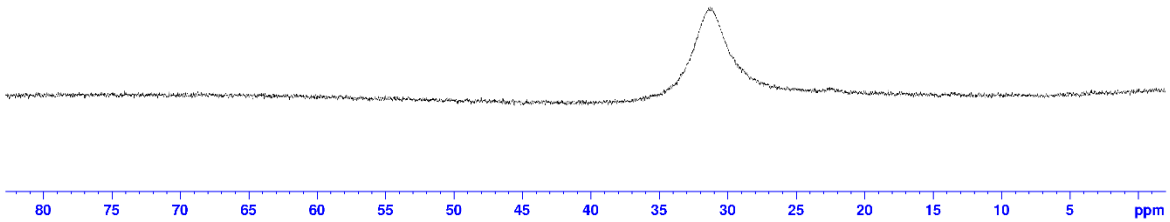
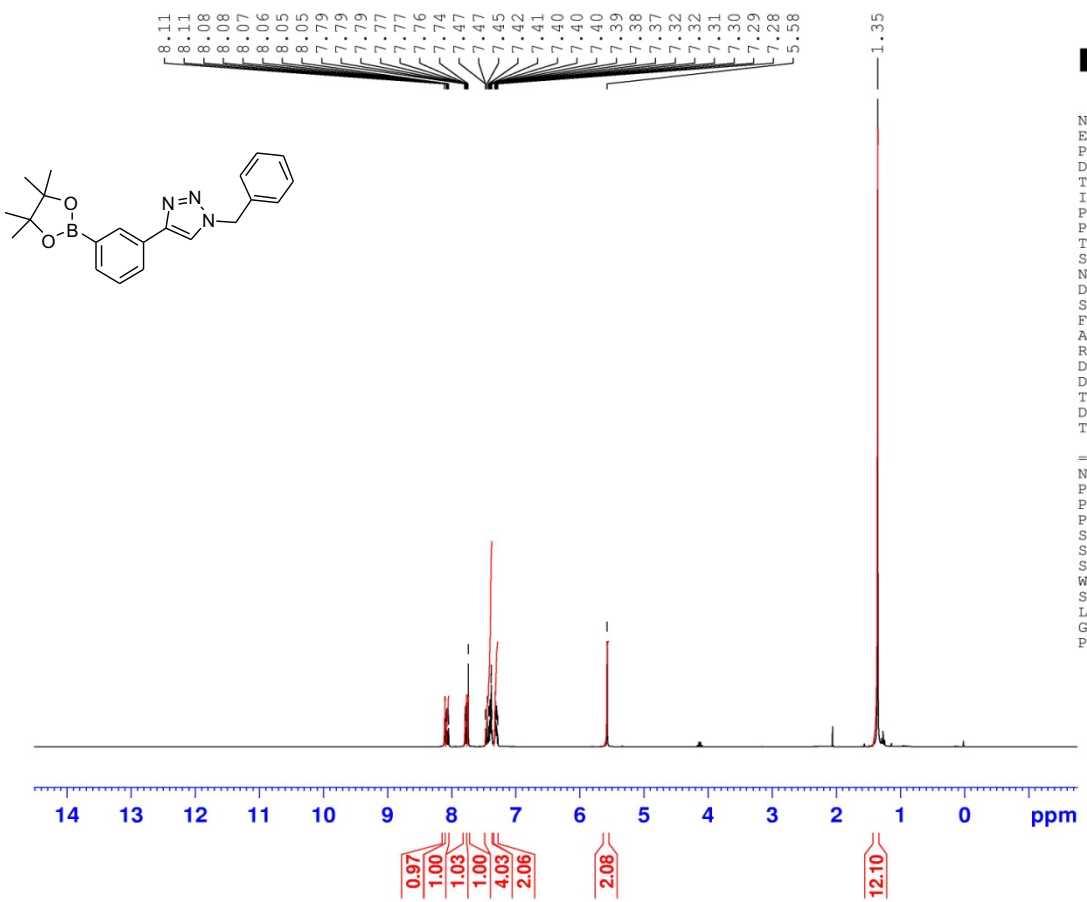



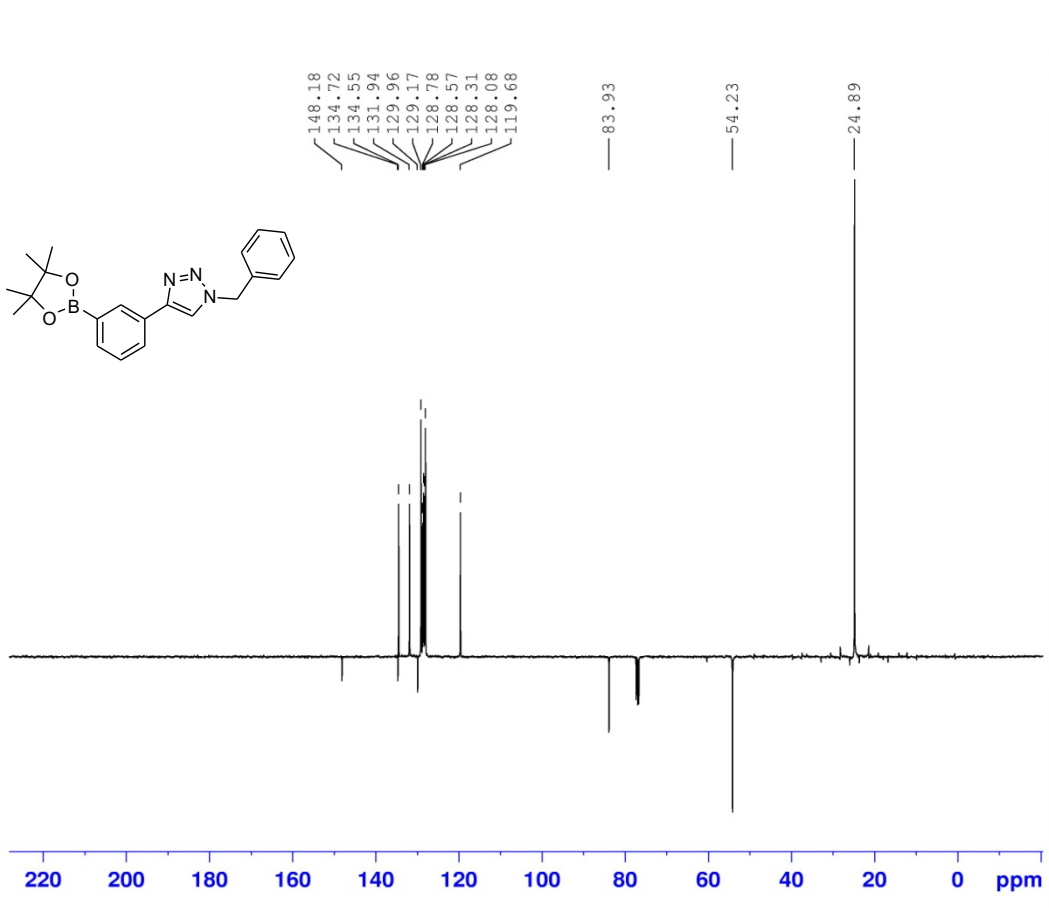
1-Benzyl-4-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7b) ^{1}H NMR spectrum



BRUKER

NAME	08-11-Fossey-39
EXPNO	10
PROCNO	1
Date_	20130811
Time	22.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	71.8
DW	83.200 usec
DE	12.89 usec
TE	289.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

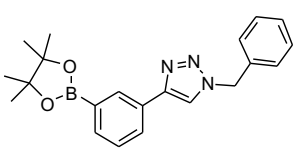
1-Benzyl-4-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7b) ^{13}C NMR spectrum

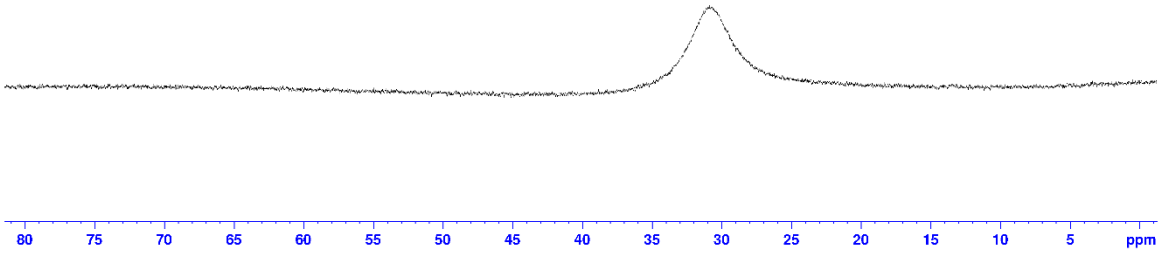


BRUKER

NAME	08-11-Fossey-4
EXPNO	10
PROCNO	1
Date_	20130811
Time	23.05
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	294.9 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

1-Benzyl-4-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7b) ^{11}B NMR spectrum



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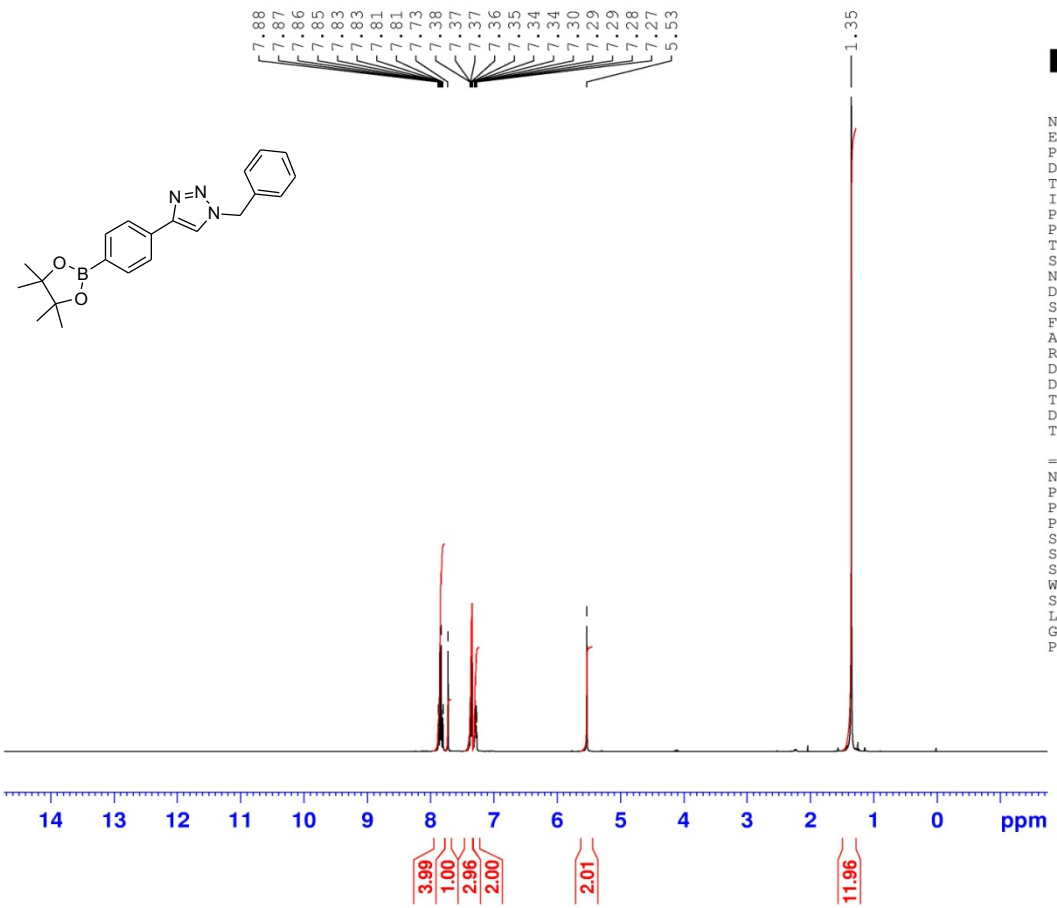
Current Data Parameters
NAME      04-14.2.fossey
EXPNO                 10
PROCNO               999

F2 - Acquisition Parameters
Date_           20140414
Time              19.58
INSTRUM           av400
PROBHD   5 mm BBO BB-1H
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                3072
DS                   4
SWH          25641.025 Hz
FIDRES        0.391251 Hz
AQ            1.2779520 sec
RG               812.7
DW              19.500 usec
DE               6.50 usec
TE              296.2 K
D1           1.00000000 sec
TD0                  6

======== CHANNEL f1 ========
NUC1               11B
P1               10.50 usec
PL1              12.00 dB
SFO1       128.3583548 MHz

F2 - Processing parameters
SI                32768
SF         128.3583560 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

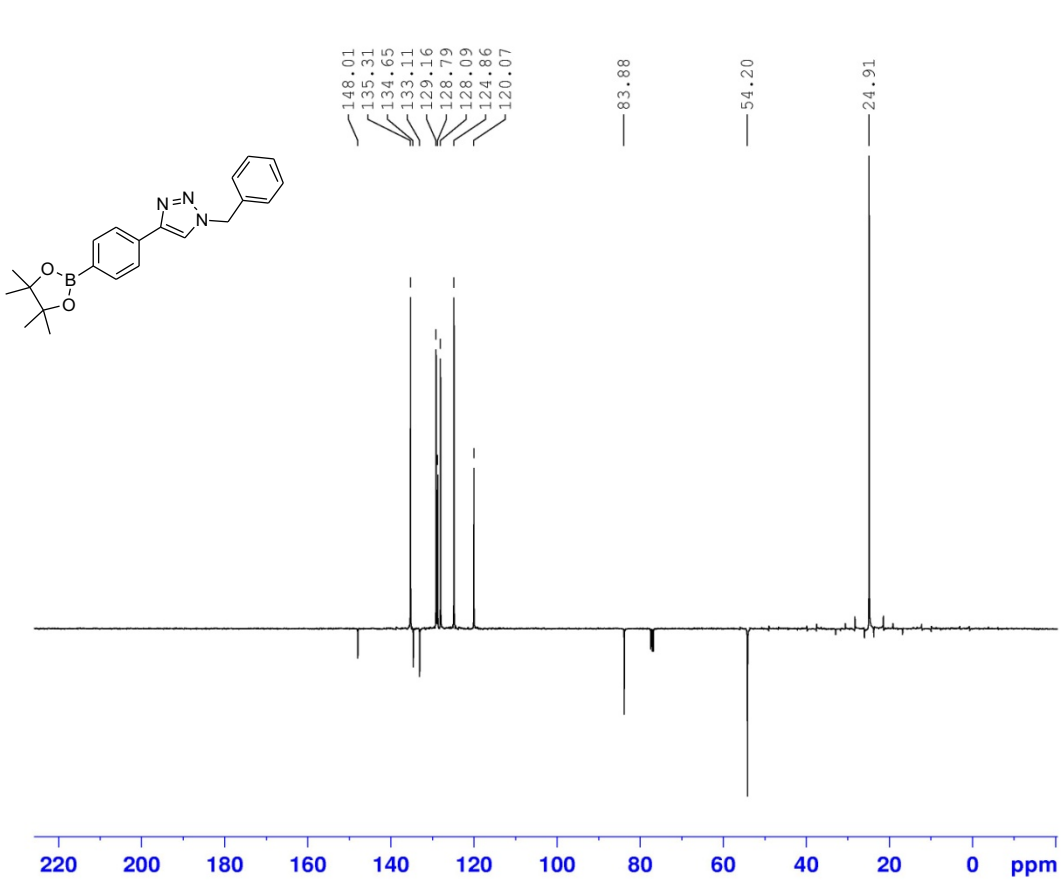
1-Benzyl-4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7c) ^{1}H NMR spectrum



BRUKER

NAME	07-26-Fossey-17
EXPNO	10
PROCNO	1
Date_	20130726
Time	19.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	36
DW	83.200 usec
DE	12.89 usec
TE	290.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

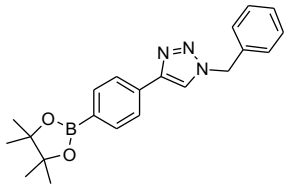
1-Benzyl-4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7c) ^{13}C NMR spectrum



BRUKER

NAME	07-26-Fossey-9
EXPNO	10
PROCNO	1
Date_	20130727
Time	3.46
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	295.1 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

1-Benzyl-4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-1,2,3-triazole (7c) ^{11}B NMR spectrum



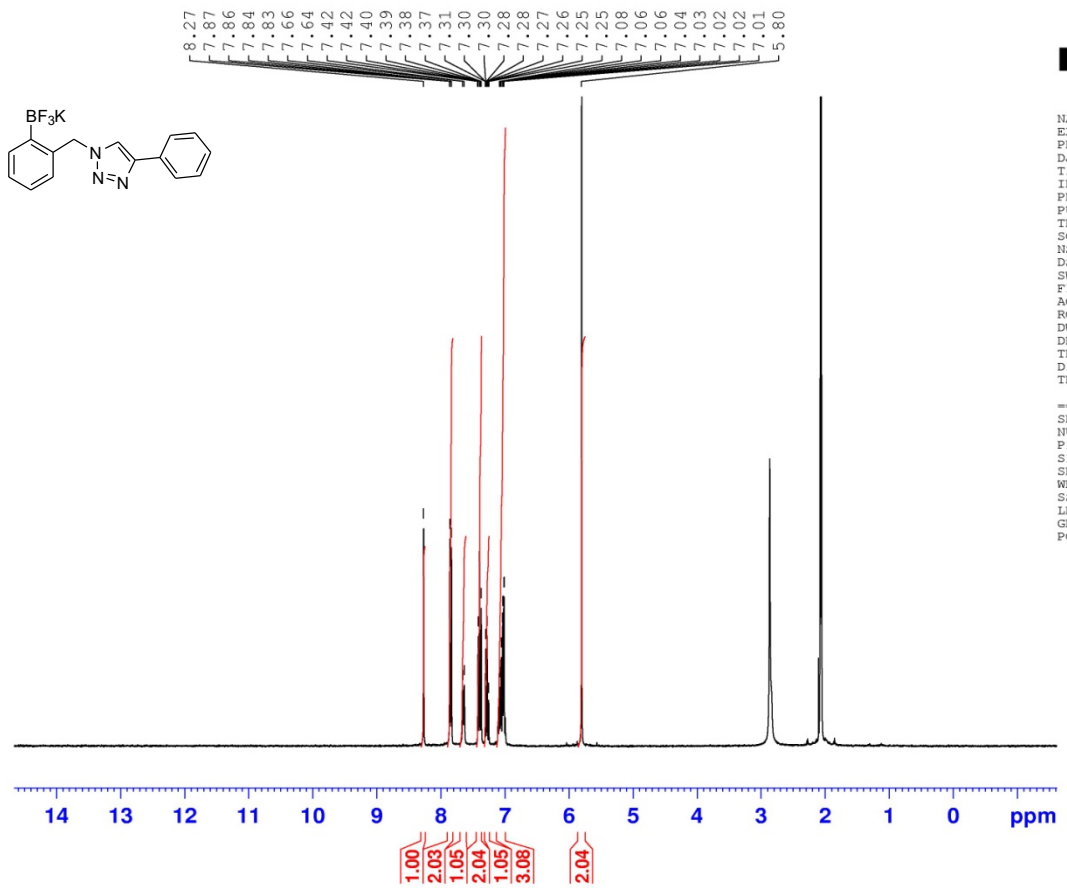

```
Current Data Parameters
NAME      04-14.3.Fossey
EXPNO                 10
PROCNO               999

F2 - Acquisition Parameters
Date            20140414
Time                22.02
INSTRUM             av400
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH           25641.025 Hz
FIDRES         0.391251 Hz
AQ             1.2779520 sec
RG                812.7
DW               19.500 usec
DE                6.50 usec
TE                296.2 K
D1           1.00000000 sec
TD0                    6

======== CHANNEL f1 ========
NUC1                  11B
P1                 10.50 usec
PL1                12.00 dB
SFO1         128.3583548 MHz

F2 - Processing parameters
SI                 32768
SF           128.3583560 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

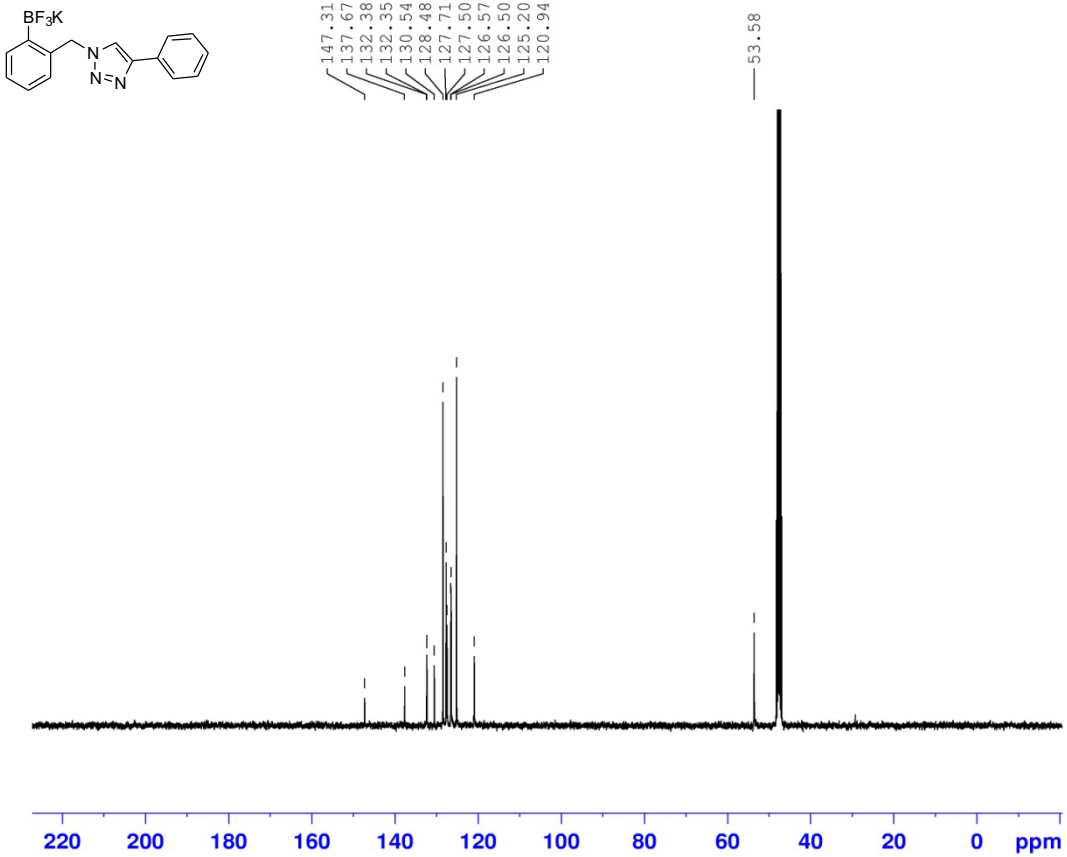
4-Phenyl-1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum



BRUKER

NAME	10-20-Fossey-52 lit BF3K
EXPNO	10
PROCNO	1
Date_	20141020
Time	23.46
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	Acetone
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	1030
DW	83.200 usec
DE	12.89 usec
TE	294.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

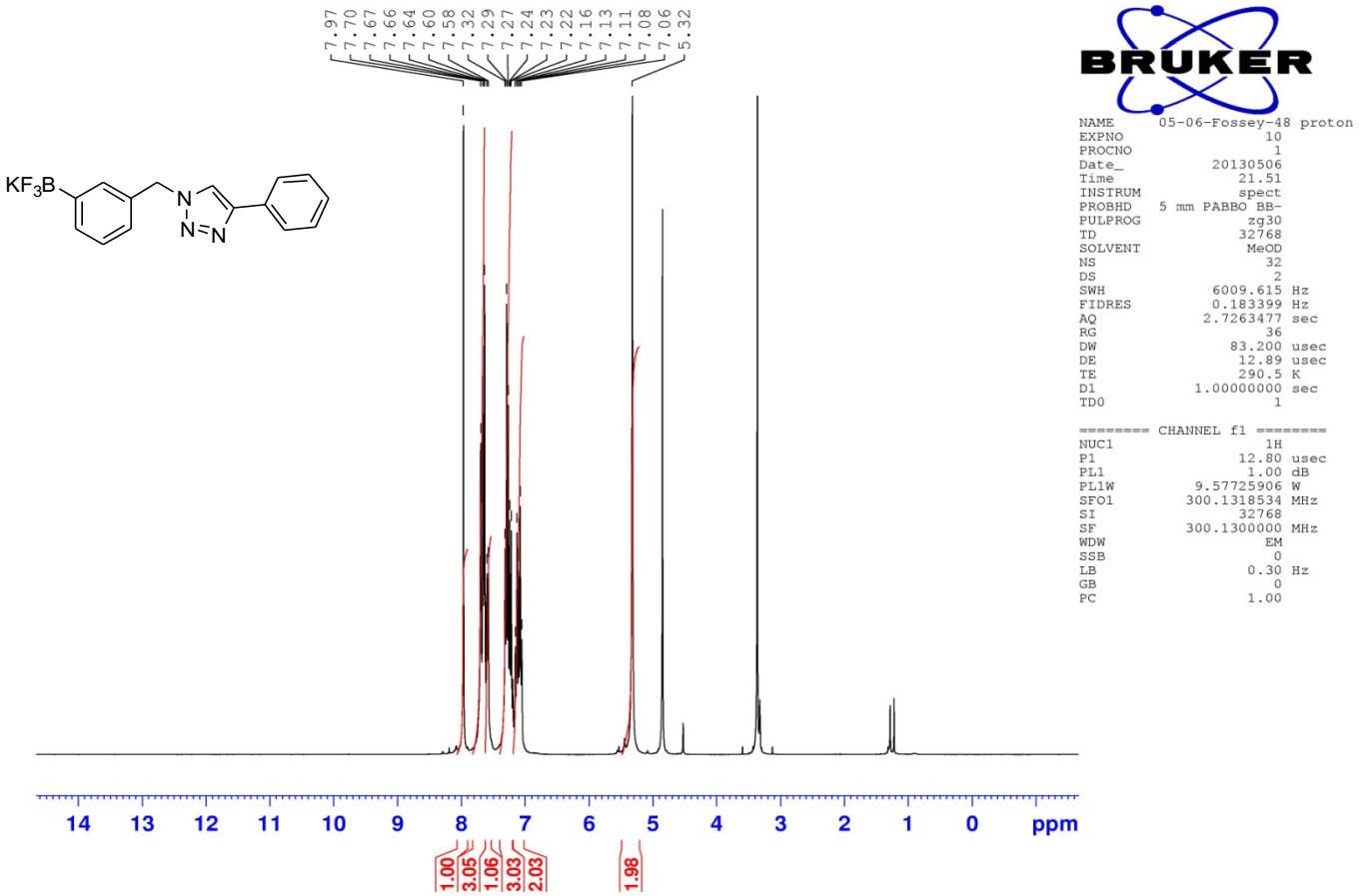
4-Phenyl-1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{13}C NMR spectrum



BRUKER

NAME	04-30-Fossey-4 WZ02-04 C
EXPNO	10
PROCNO	1
Date_	20130430
Time	19.45
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	udeft
TD	18178
SOLVENT	MeOD
NS	380
DS	0
SWH	25252.525 Hz
FIDRES	1.389181 Hz
AQ	0.3599744 sec
RG	2050
DW	19.800 usec
DE	8.20 usec
TE	294.3 K
D1	3.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
D20	200.00000000 sec
TD0	380
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P12	2000.00 usec
P26	500.00 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
SP2	6.27 dB
SP8	6.27 dB
SPNAM2	Crp60comp.4
SPNAM8	Crp60,0.5,20.1
SPOAL2	0.500
SPOAL8	0.500
SPOFFS2	0.00 Hz
SPOFFS8	0.00 Hz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.00

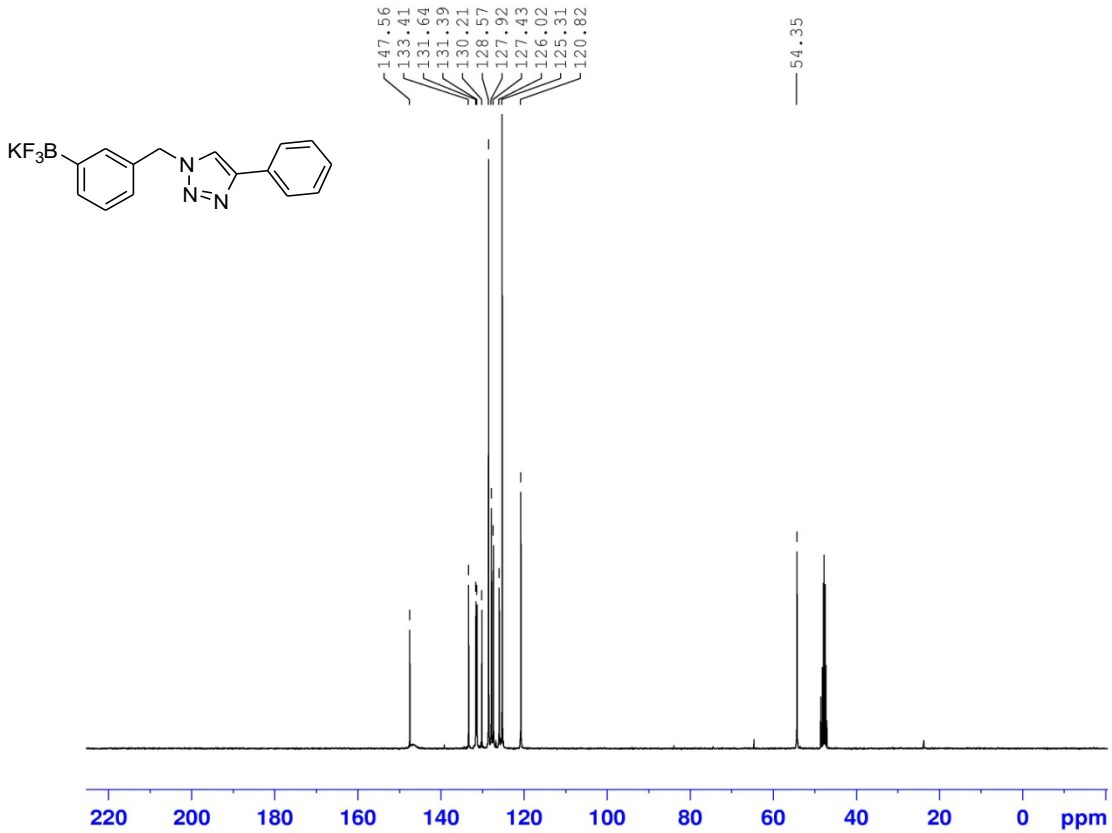
4-Phenyl-1-(3-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum



BRUKER

NAME	05-06-Fossey-48 proton
EXPNO	10
PROCNO	1
Date_	20130506
Time	21.51
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	36
DW	83.200 usec
DE	12.89 usec
TE	290.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

4-Phenyl-1-(3-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{13}C NMR spectrum

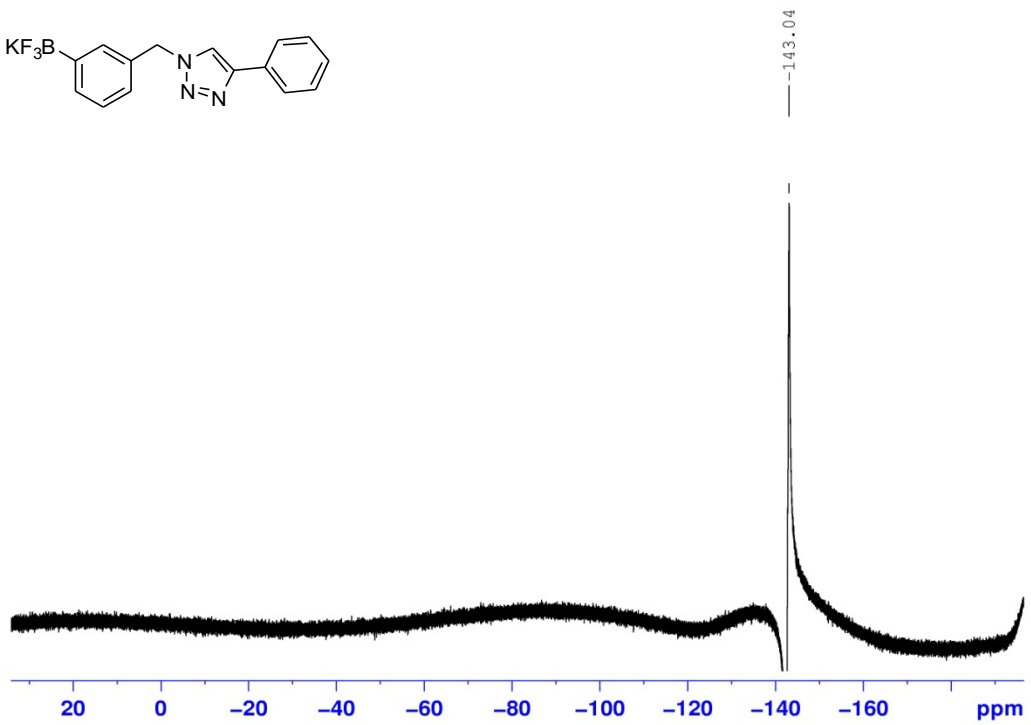



```
NAME          05-06-Fossey-15 carbon
EXPNO                            10
PROCNO                            1
Date_                      20130506
Time                          22.05
INSTRUM                       spect
PROBHD    5 mm PADUL 13C
PULPROG                       udeft
TD                            18178
SOLVENT                        MeOD
NS                              380
DS                                0
SWH                       25252.525 Hz
FIDRES                     1.389181 Hz
AQ                        0.3599744 sec
RG                             2050
DW                           19.800 usec
DE                            8.20 usec
TE                           294.2 K
D1                    3.00000000 sec
D11                   0.03000000 sec
D12                   0.00002000 sec
D20                 200.00000000 sec
TD0                             380

======== CHANNEL f1 ========
NUC1                           13C
P1                            8.80 usec
P12                        2000.00 usec
P26                         500.00 usec
PL1                           -3.00 dB
PL1W                 58.63890457 W
SFO1                100.6233333 MHz
SP2                           6.27 dB
SP8                           6.27 dB
SPNAM2               Crp60comp.4
SPNAM8            Crp60,0.5,20.1
SPOAL2                        0.500
SPOAL8                        0.500
SPOFFS2                       0.00 Hz
SPOFFS8                       0.00 Hz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                           1H
PCPD2                        90.00 usec
PL2                           -4.00 dB
PL12                          15.35 dB
PL2W                24.29185867 W
PL12W                0.28213742 W
SFO2                400.1316005 MHz
SI                           65536
SF                  100.6127690 MHz
WDW                            EM
SSB                             0
LB                            2.00 Hz
GB                              0
PC                            1.00
```

4-Phenyl-1-(3-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{19}F NMR spectrum



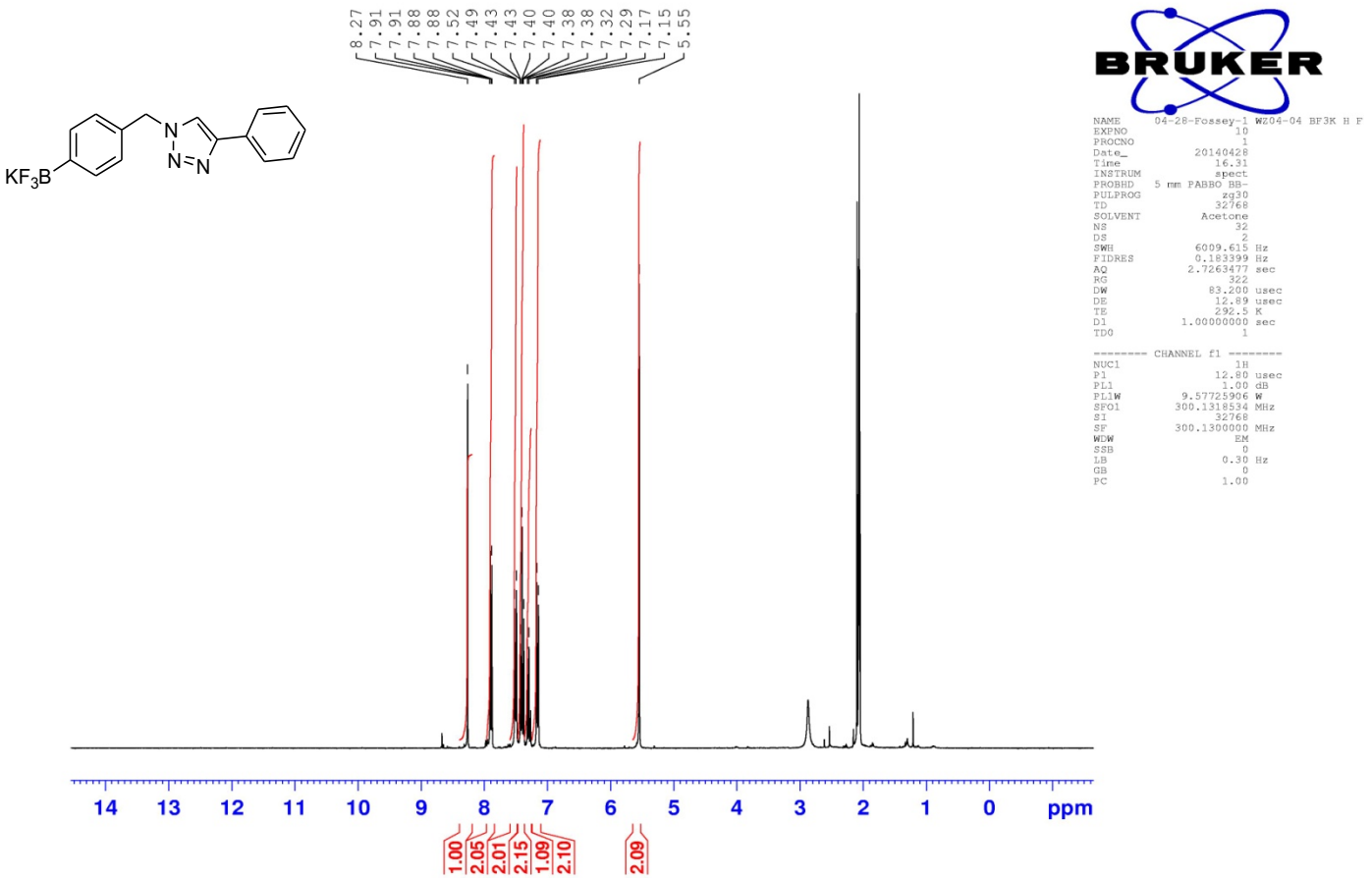
BRUKER

```
NAME          04-09-Fossey-44 WZ03-04 H F
EXPNO                      11
PROCNO                      1
Date_                20140410
Time                     1.12
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zqig
TD                    131072
SOLVENT              Acetone
NS                         32
DS                          4
SWH                66964.289 Hz
FIDRES             0.510897 Hz
AQ                 0.9787210 sec
RG                        322
DW                    7.467 usec
DE                     7.27 usec
TE                    291.7 K
D1                 3.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      19F
P1                       8.70 usec
PL1                     -5.00 dB
PL1W              30.58163643 W
SFO1              282.3823550 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL2W               9.57725906 W
PL12W              0.24056987 W
SFO2              300.1312005 MHz
SI                        131072
SF                282.4043550 MHz
WDW                         EM
SSB                          0
LB                        0.50 Hz
GB                           0
PC                        1.00
```

4-Phenyl-1-(4-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum

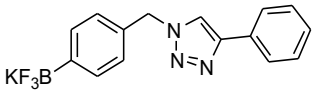


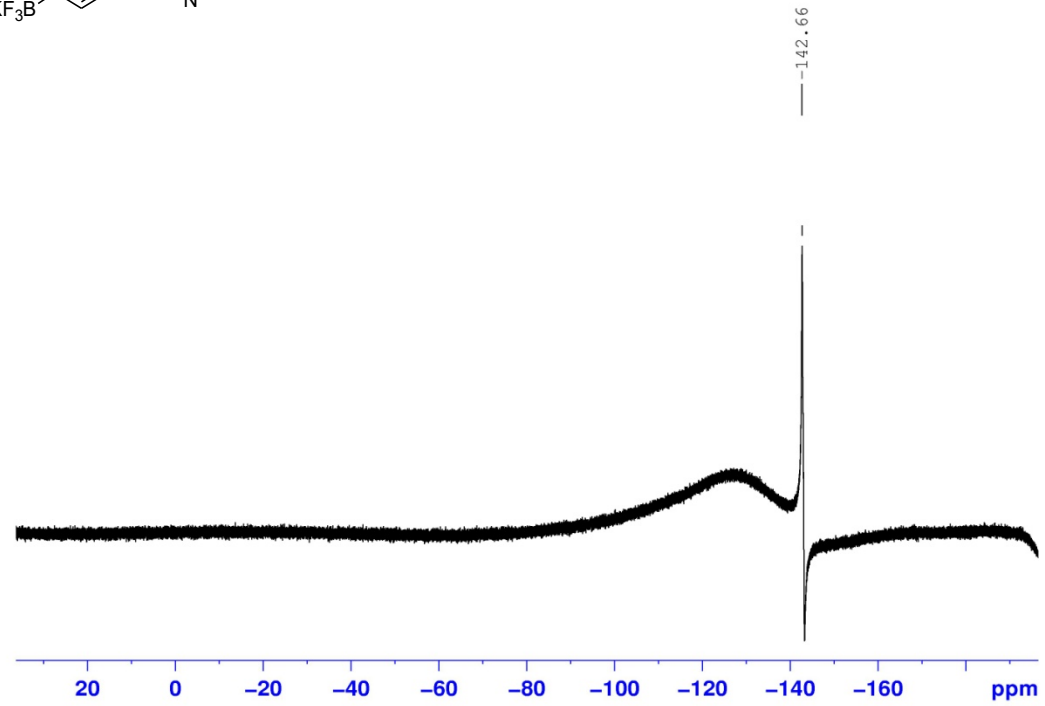
BRUKER

```
NAME          04-28-Fossey-1 WZ04-04 BF3K H F
EXPNO                    10
PROCNO                    1
Date_              20140428
Time                  16.31
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT             Acetone
NS                       32
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7263477 sec
RG                      322
DW                   83.200 usec
DE                   12.89 usec
TE                   292.5 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    12.80 usec
PL1                    1.00 dB
PL1W             9.57725906 W
SFO1           300.1318534 MHz
SI                    32768
SF             300.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

4-Phenyl-1-(4-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{19}F NMR spectrum





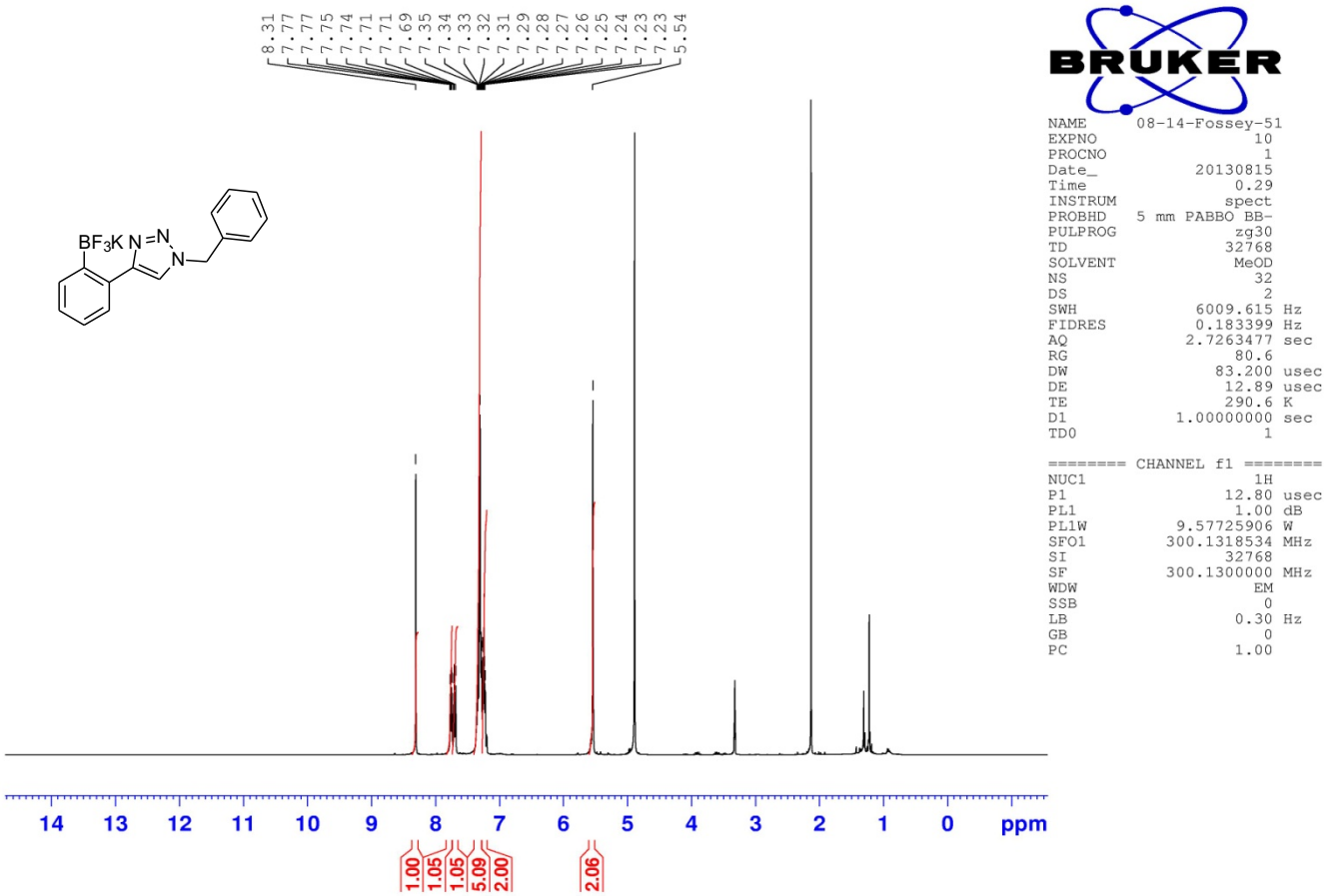
BRUKER

```
NAME          04-28-Fossey-1 WZ04-04 BF3K H F
EXPNO                    11
PROCNO                    1
Date_              20140428
Time                  16.36
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zgig
TD                   131072
SOLVENT             Acetone
NS                        32
DS                         4
SWH                66964.289 Hz
FIDRES              0.510897 Hz
AQ                  0.9787210 sec
RG                       512
DW                     7.467 usec
DE                     7.27 usec
TE                    292.6 K
D1                 3.00000000 sec
D11                0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     19F
P1                     8.70 usec
PL1                   -5.00 dB
PL1W            30.58163643 W
SFO1           282.3823550 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    1.00 dB
PL12                  17.00 dB
PL2W            9.57725906 W
PL12W           0.24056987 W
SFO2           300.1312005 MHz
SI                   131072
SF             282.4043550 MHz
WDW                      EM
SSB                       0
LB                     0.50 Hz
GB                        0
PC                     1.00
```

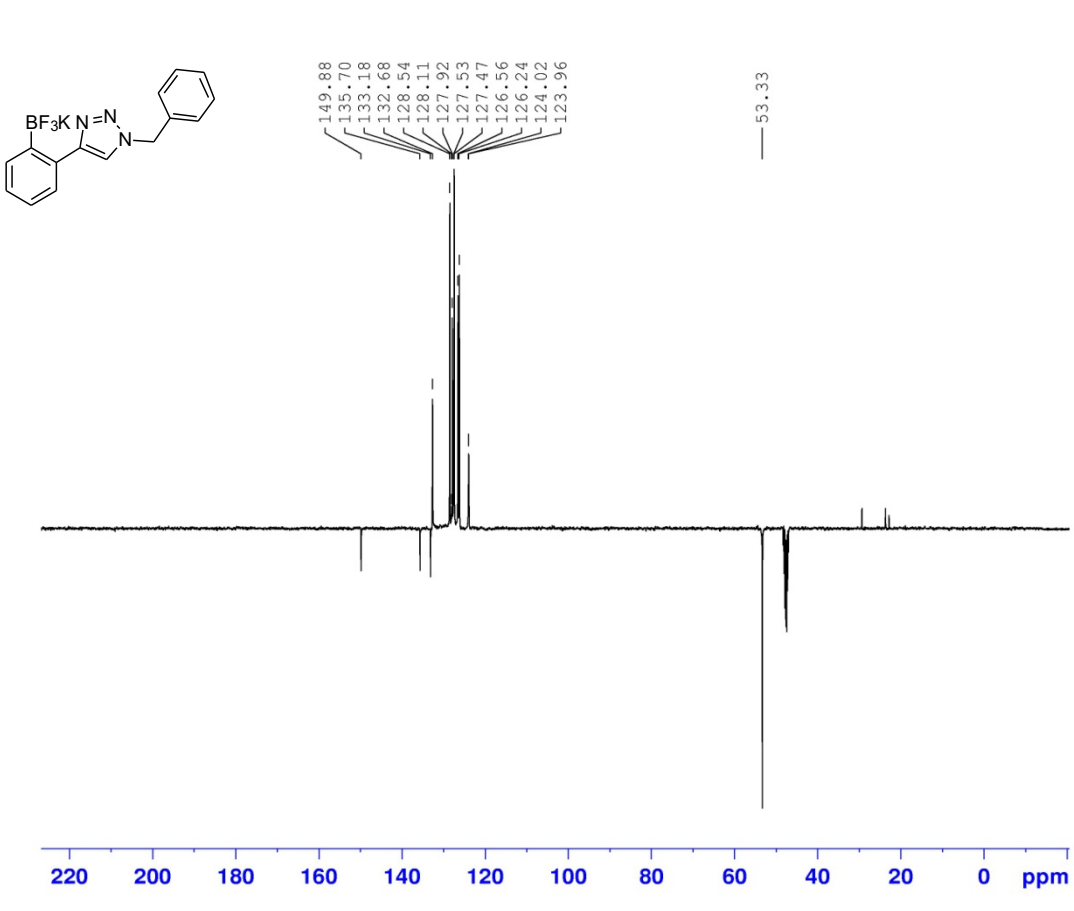
1-Benzyl-4-(2-(trifluoro-l4-boranyl)phenyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum



BRUKER

NAME	08-14-Fossey-51
EXPNO	10
PROCNO	1
Date_	20130815
Time	0.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	80.6
DW	83.200 usec
DE	12.89 usec
TE	290.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

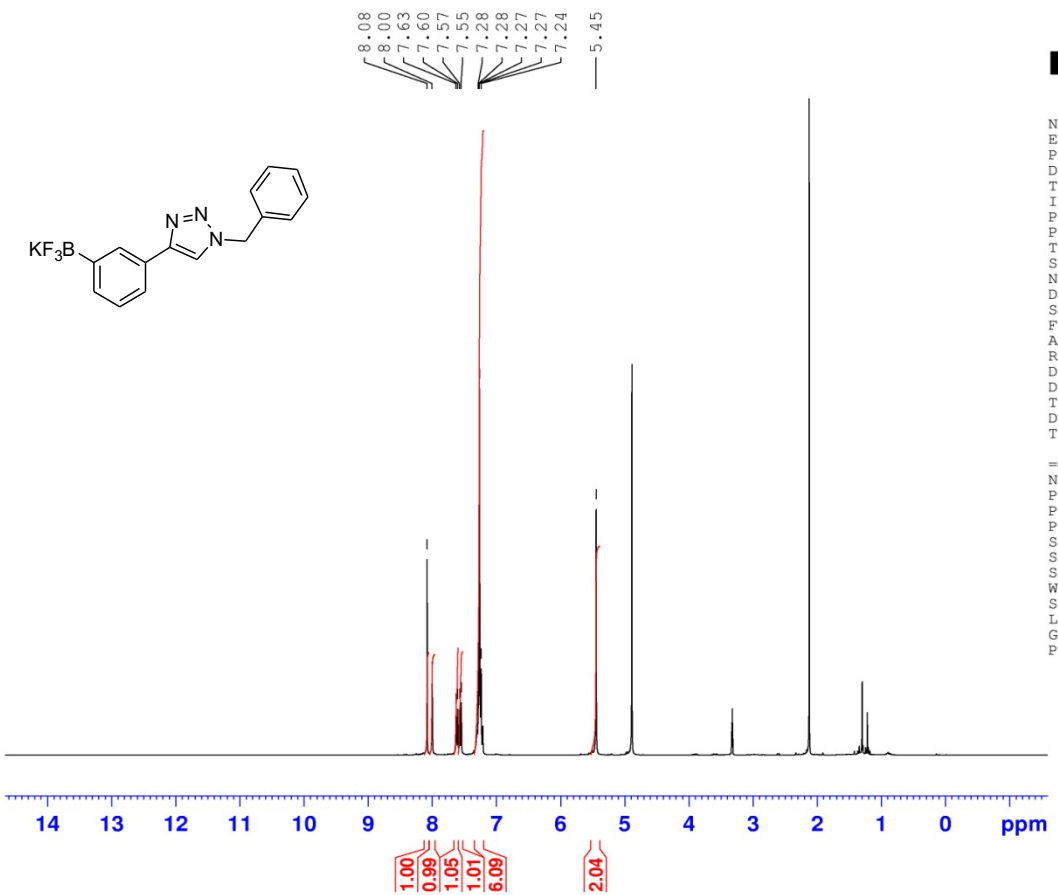
1-Benzyl-4-(2-(trifluoro-l4-boranyl)phenyl)-1*H*-1,2,3-triazole, potassium salt ^{13}C NMR spectrum



BRUKER

NAME	08-14-Fossey-42
EXPNO	10
PROCNO	1
Date_	20130815
Time	9.55
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	MeOD
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	294.7 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

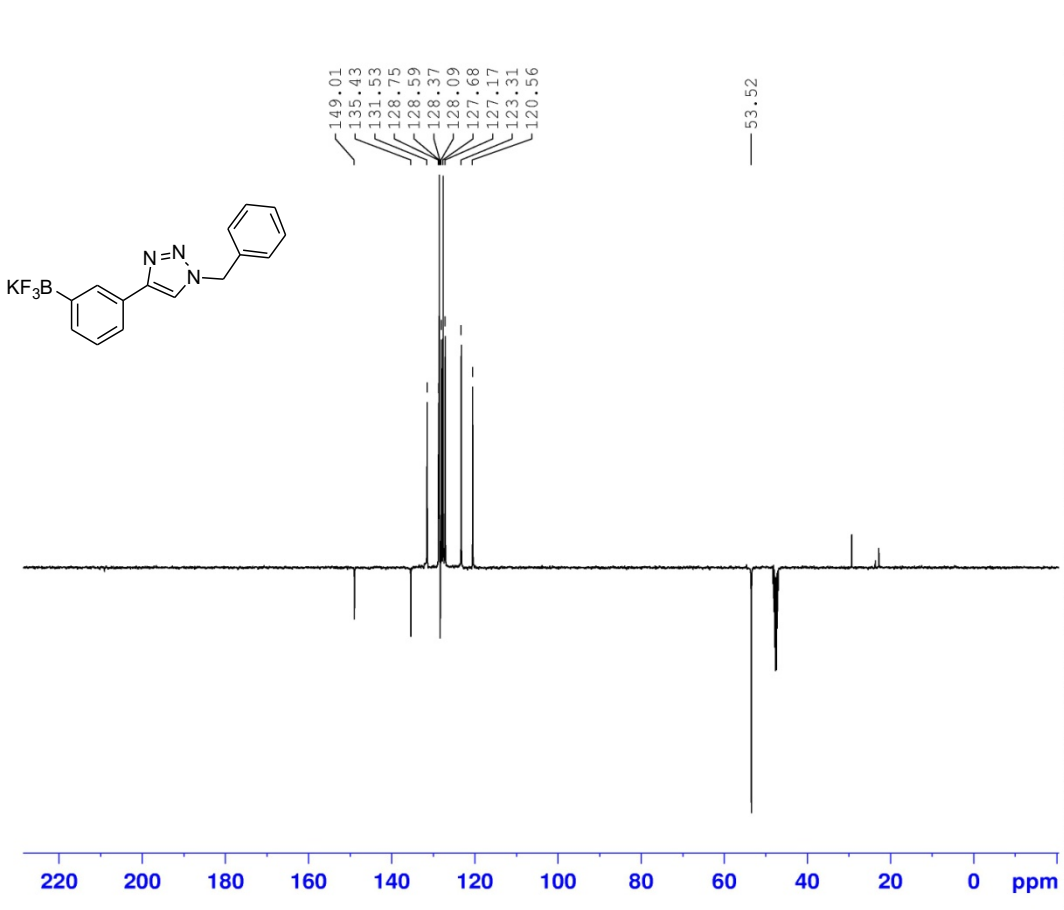
1-Benzyl-4-(3-(trifluoro-l4-boranyl)phenyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum



BRUKER

NAME	08-12-Fossey-13
EXPNO	10
PROCNO	1
Date_	20130812
Time	18.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	71.8
DW	83.200 usec
DE	12.89 usec
TE	290.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

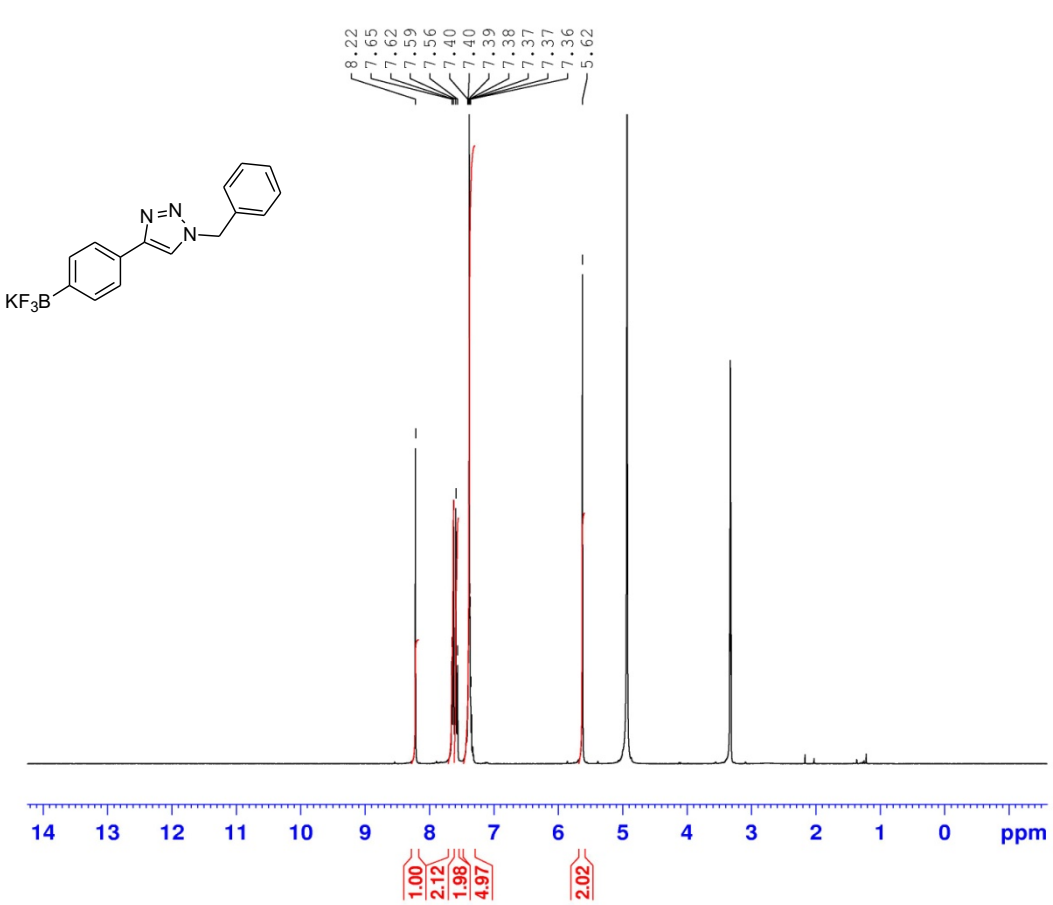
1-Benzyl-4-(3-(trifluoro-l4-boranyl)phenyl)-1*H*-1,2,3-triazole, potassium salt ^{13}C NMR spectrum



BRUKER

NAME	08-12-Fossey-7	
EXPNO	10	
PROCNO	1	
Date_	20130812	
Time	21.08	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	pendantns	
TD	65536	
SOLVENT	MeOD	
NS	512	
DS	4	
SWH	25252.525	Hz
FIDRES	0.385323	Hz
AQ	1.2976629	sec
RG	2050	
DW	19.800	usec
DE	6.50	usec
TE	295.1	K
CNST2	145.0000000	
CNST3	1.0000000	
CNST4	5.0000000	
D1	1.50000000	sec
D2	0.00172414	sec
D3	0.00431034	sec
D12	0.00002000	sec
D13	0.00000400	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	8.80	usec
P2	17.60	usec
PL1	-3.00	dB
PL1W	58.63890457	W
SFO1	100.6233333	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
P3	9.70	usec
P4	19.40	usec
PCPD2	90.00	usec
PL2	-4.00	dB
PL12	15.35	dB
PL2W	24.29185867	W
PL12W	0.28213742	W
SFO2	400.1316005	MHz
SI	65536	
SF	100.6127690	MHz
WDW	EM	
SSB	0	
LB	4.00	Hz
GB	0	
PC	1.00	

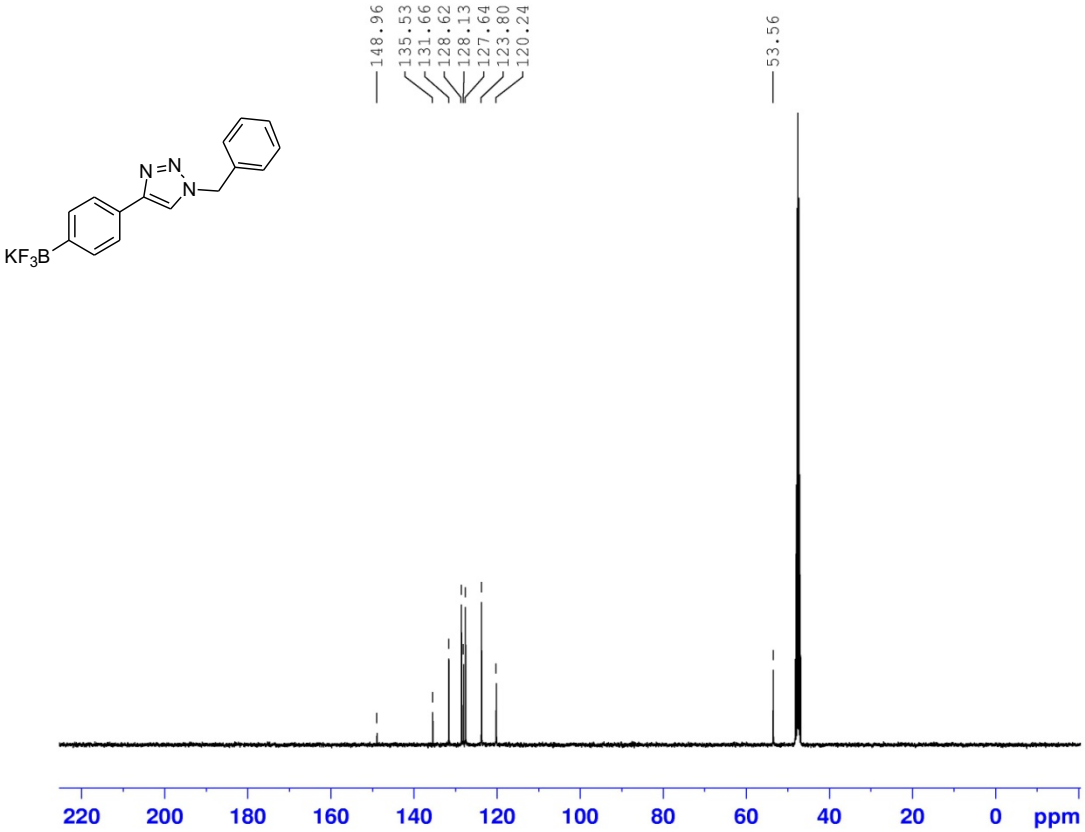
1-Benzyl-4-(4-(trifluoro-l4-boranyl)phenyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum




```
NAME        07-30-Fossey-1
EXPNO                   10
PROCNO                   1
Date_             20130730
Time                 16.46
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   32768
SOLVENT               MeOD
NS                      32
DS                       2
SWH               6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7263477 sec
RG                     203
DW                  83.200 usec
DE                  12.89 usec
TE                  290.1 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   12.80 usec
PL1                   1.00 dB
PL1W           9.57725906 W
SFO1          300.1318534 MHz
SI                   32768
SF            300.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

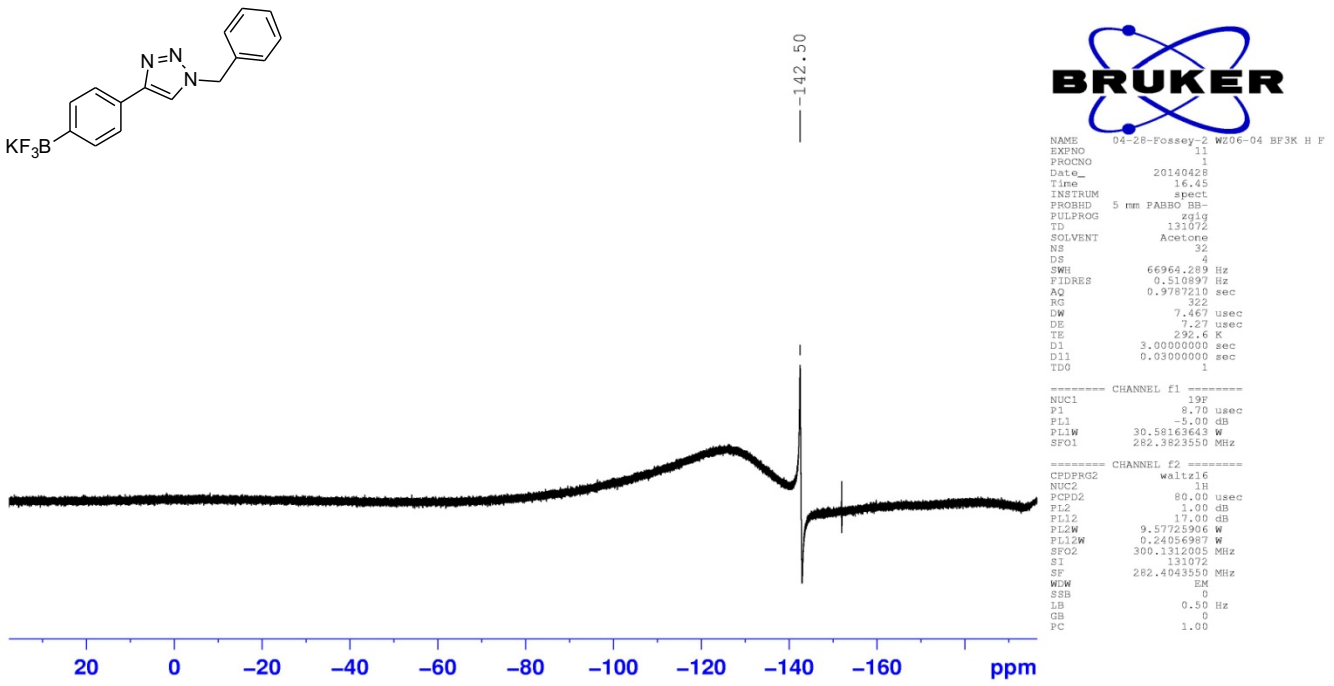
1-Benzyl-4-(4-(trifluoro-l4-boranyl)phenyl)-1*H*-1,2,3-triazole, potassium salt ^{13}C NMR spectrum



BRUKER

NAME	07-30-Fossey-2
EXPNO	10
PROCNO	1
Date_	20130730
Time	17.18
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	udeft
TD	18178
SOLVENT	MeOD
NS	380
DS	0
SWH	25252.525 Hz
FIDRES	1.389181 Hz
AQ	0.3599744 sec
RG	2050
DW	19.800 usec
DE	8.20 usec
TE	294.5 K
D1	3.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
D20	200.00000000 sec
TD0	380
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P12	2000.00 usec
P26	500.00 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
SP2	6.27 dB
SP8	6.27 dB
SPNAM2	Crp60comp.4
SPNAM8	Crp60,0.5,20.1
SPOAL2	0.500
SPOAL8	0.500
SPOFFS2	0.00 Hz
SPOFFS8	0.00 Hz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.00

1-Benzyl-4-(4-(trifluoro-l4-boranyl)phenyl)-1*H*-1,2,3-triazole, potassium salt ^{19}F NMR spectrum

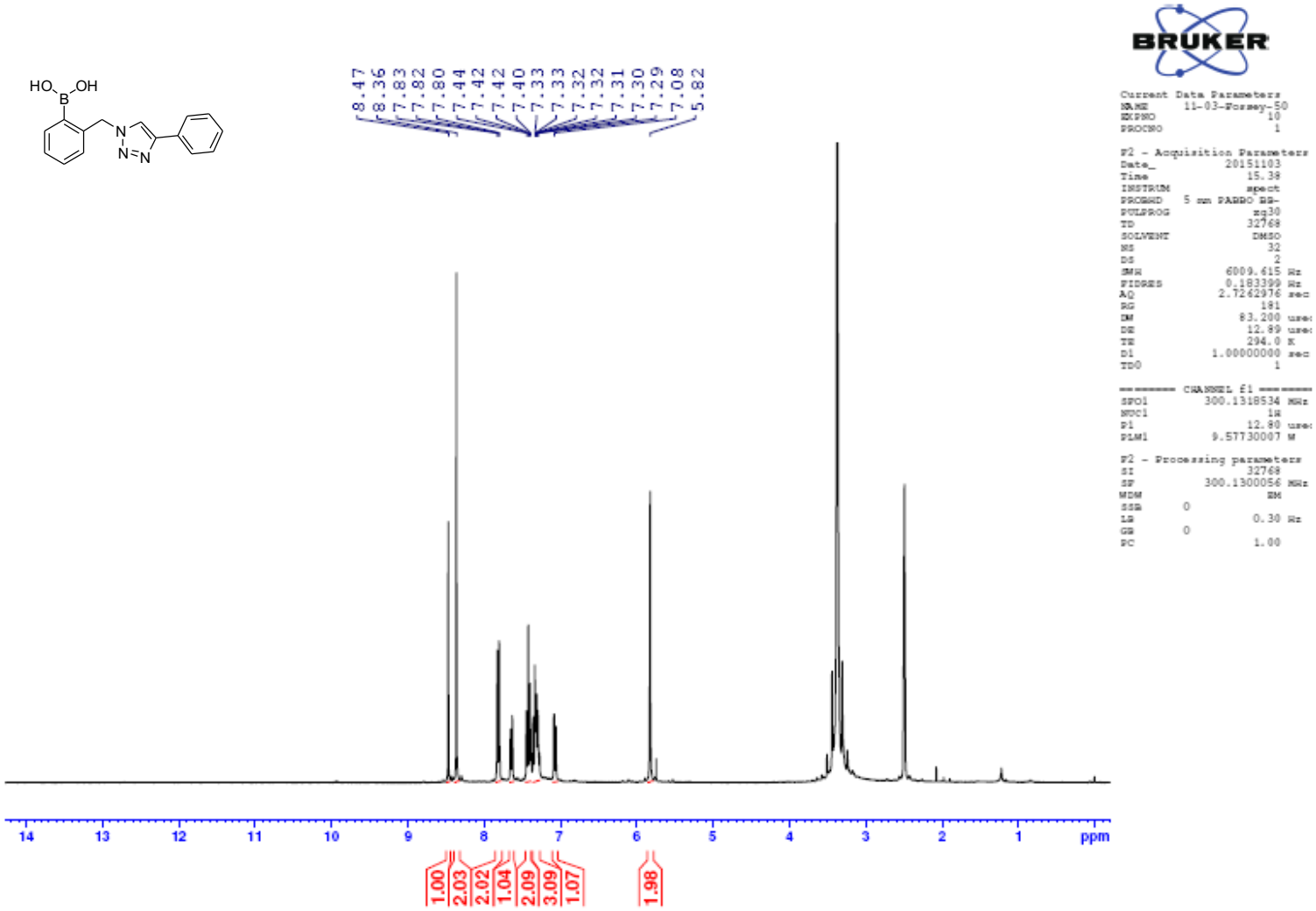



```
NAME          04-28-Fossey-2 WZ06-04 BF3K H F
EXPNO                    11
PROCNO                    1
Date_               20140428
Time                   16.45
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgig
TD                   131072
SOLVENT              Acetone
NS                        32
DS                         4
SWH                66964.289 Hz
FIDRES              0.510897 Hz
AQ                  0.9787210 sec
RG                       322
DW                     7.467 usec
DE                     7.27 usec
TE                     292.6 K
D1                 3.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      19F
P1                       8.70 usec
PL1                     -5.00 dB
PL1W              30.58163643 W
SFO1              282.3823550 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      1.00 dB
PL12                    17.00 dB
PL2W               9.57725906 W
PL12W              0.24056987 W
SFO2              300.1312005 MHz
SI                       131072
SF                282.4043550 MHz
WDW                        EM
SSB                         0
LB                       0.50 Hz
GB                          0
PC                       1.00
```

(2-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1a) ^{1}H NMR spectrum



BRUKER

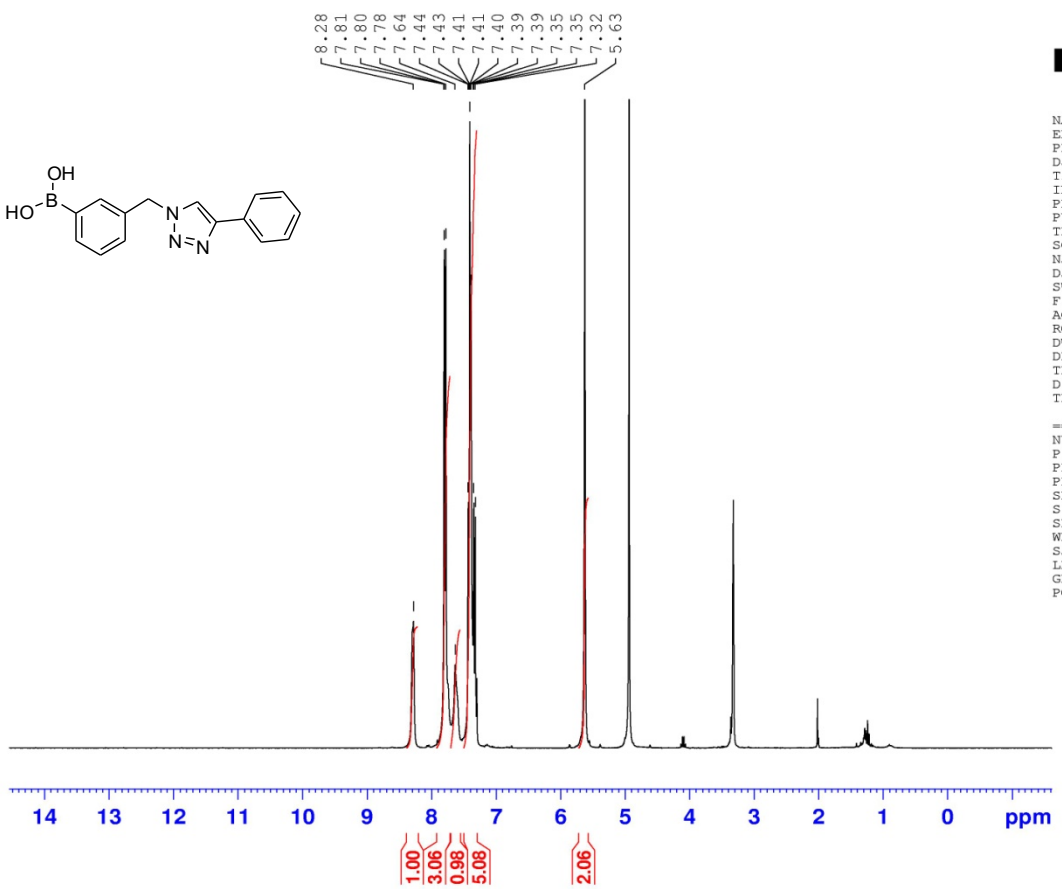
```
Current Data Parameters
NAME      11-03-Forsey-50
EXPNO              10
PROCNO              1

F2 - Acquisition Parameters
Date_          20151103
Time              15.38
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               32768
SOLVENT           DMSO
NS                   32
DS                    2
SWH            6009.615 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                 181
DW              83.200 use
DE              12.89 use
TE              294.0 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         300.1318534 MHz
NUC1              1H
P1               12.80 use
PLW1          9.57730007 W

F2 - Processing parameters
SI               32768
SF          300.1300056 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

(3-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1b) ^{1}H NMR spectrum

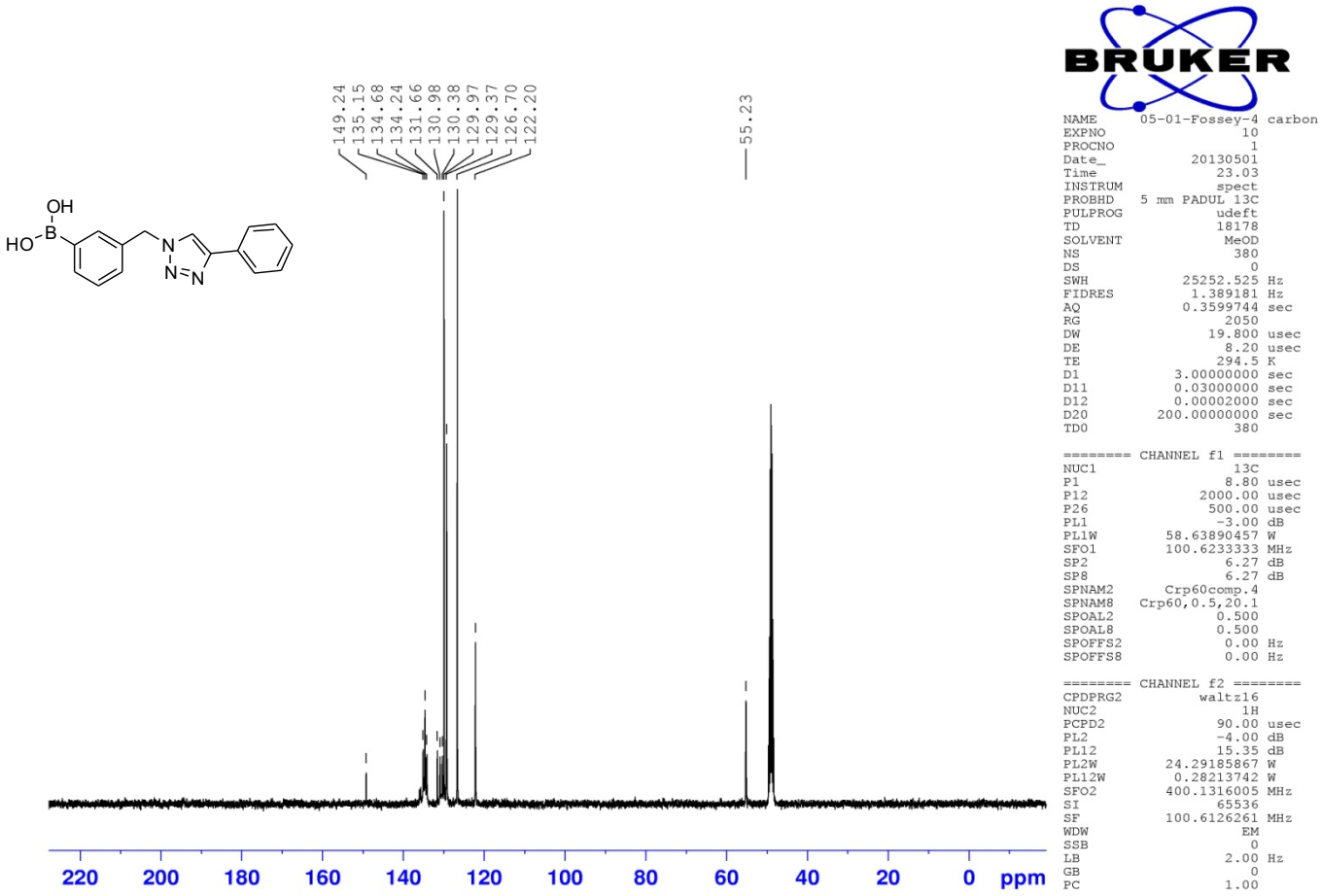


BRUKER

```
NAME          05-01-Fossey-28 proton
EXPNO                            10
PROCNO                            1
Date_                      20130501
Time                          22.08
INSTRUM                       spect
PROBHD    5 mm PABBO BB-
PULPROG                        zg30
TD                            32768
SOLVENT                        MeOD
NS                               32
DS                                2
SWH                      6009.615 Hz
FIDRES                   0.183399 Hz
AQ                       2.7263477 sec
RG                             161
DW                          83.200 usec
DE                          12.89 usec
TE                            290.6 K
D1                    1.00000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                             1H
P1                          12.80 usec
PL1                          1.00 dB
PL1W                 9.57725906 W
SFO1                300.1318534 MHz
SI                            32768
SF                  300.1300000 MHz
WDW                             EM
SSB                              0
LB                           0.30 Hz
GB                               0
PC                           1.00
```

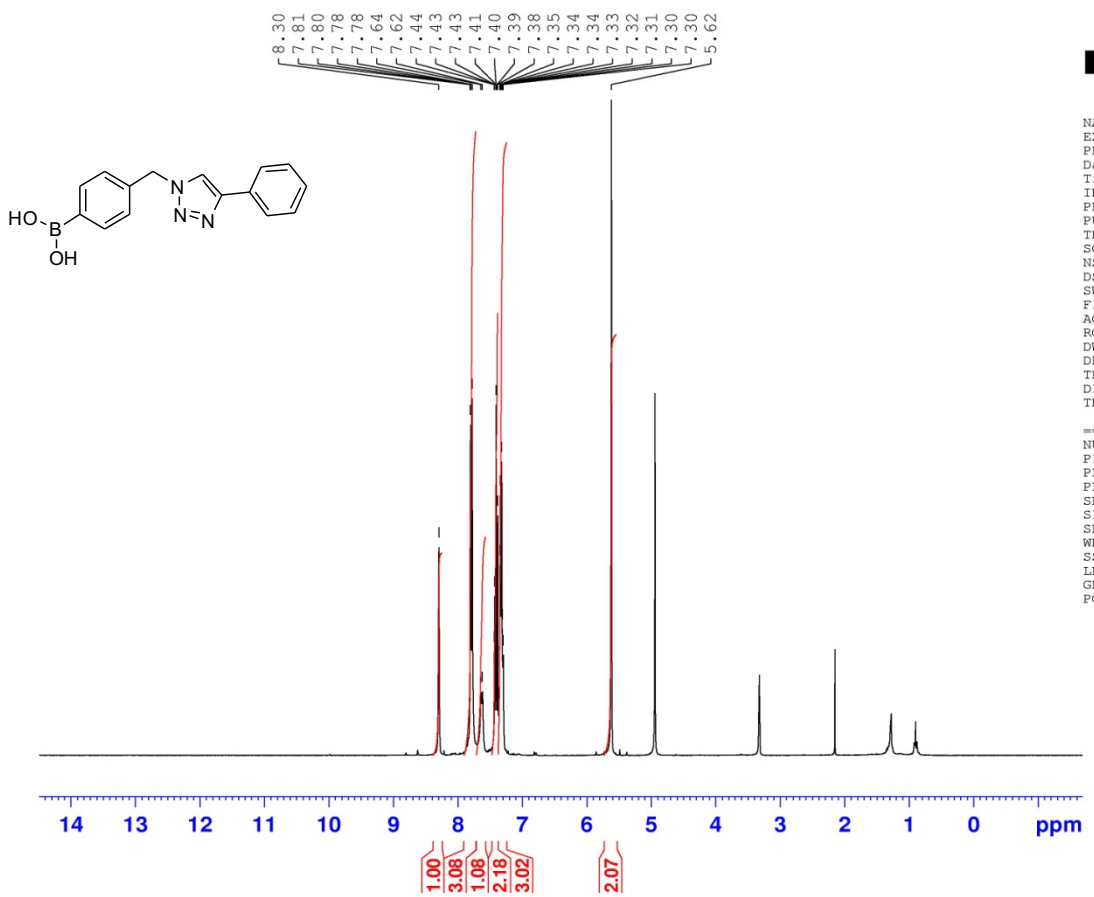
(3-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1b) ^{13}C NMR spectrum



BRUKER

NAME	05-01-Fossey-4 carbon
EXPNO	10
PROCNO	1
Date_	20130501
Time	23.03
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	udeft
TD	18178
SOLVENT	MeOD
NS	380
DS	0
SWH	25252.525 Hz
FIDRES	1.389181 Hz
AQ	0.3599744 sec
RG	2050
DW	19.800 usec
DE	8.20 usec
TE	294.5 K
D1	3.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
D20	200.00000000 sec
TD0	380
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P12	2000.00 usec
P26	500.00 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
SP2	6.27 dB
SP8	6.27 dB
SPNAM2	Crp60comp.4
SPNAM8	Crp60,0.5,20.1
SPOAL2	0.500
SPOAL8	0.500
SPOFFS2	0.00 Hz
SPOFFS8	0.00 Hz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6126261 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.00

(4-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1c) ^{1}H NMR spectrum

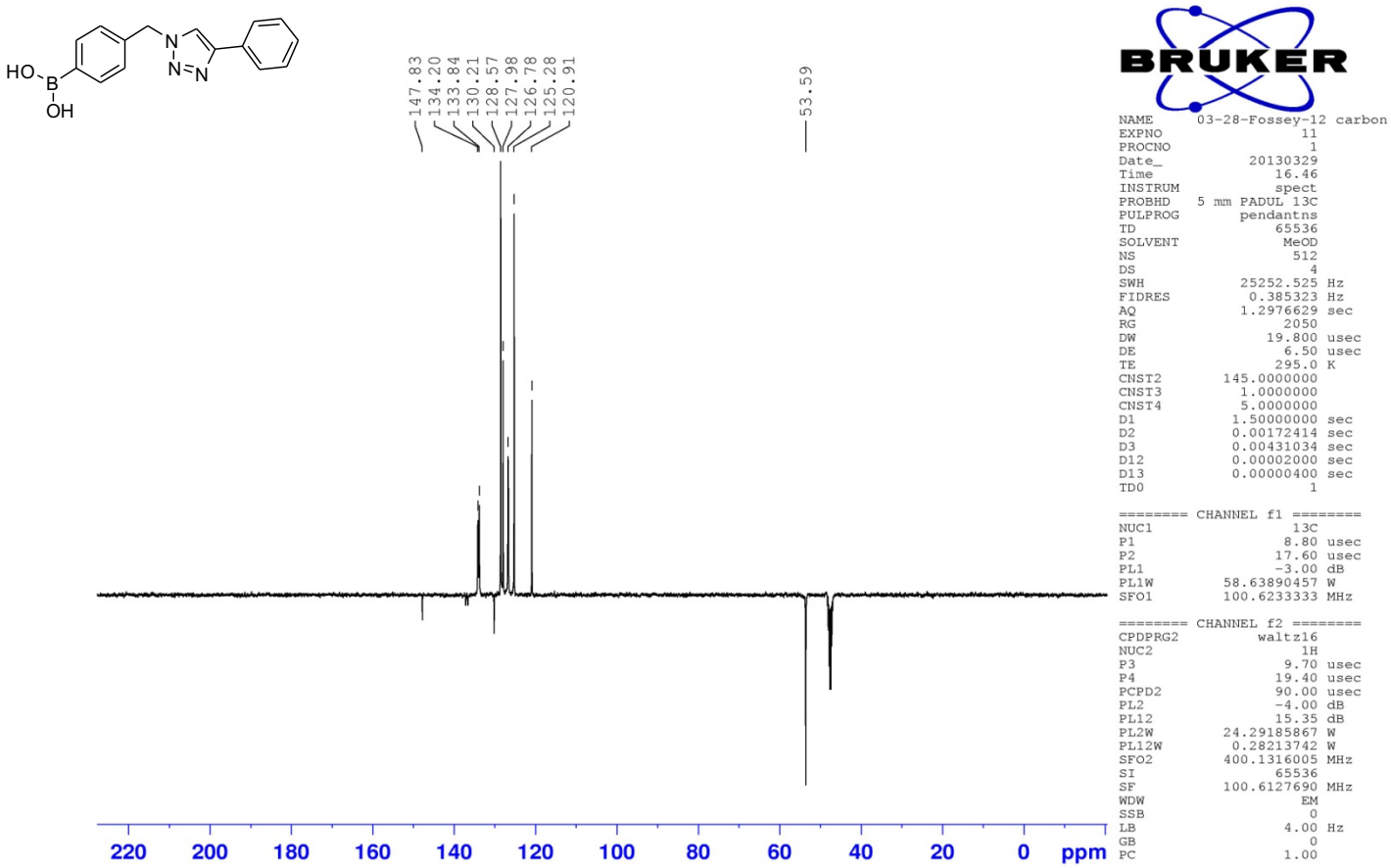


BRUKER

```
NAME          03-28-Fossey-34 proton
EXPNO                 10
PROCNO                 1
Date_           20130329
Time                2.57
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT             MeOD
NS                    32
DS                     2
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ              2.7263477 sec
RG                   144
DW                83.200 usec
DE                12.89 usec
TE                290.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.80 usec
PL1                1.00 dB
PL1W          9.57725906 W
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

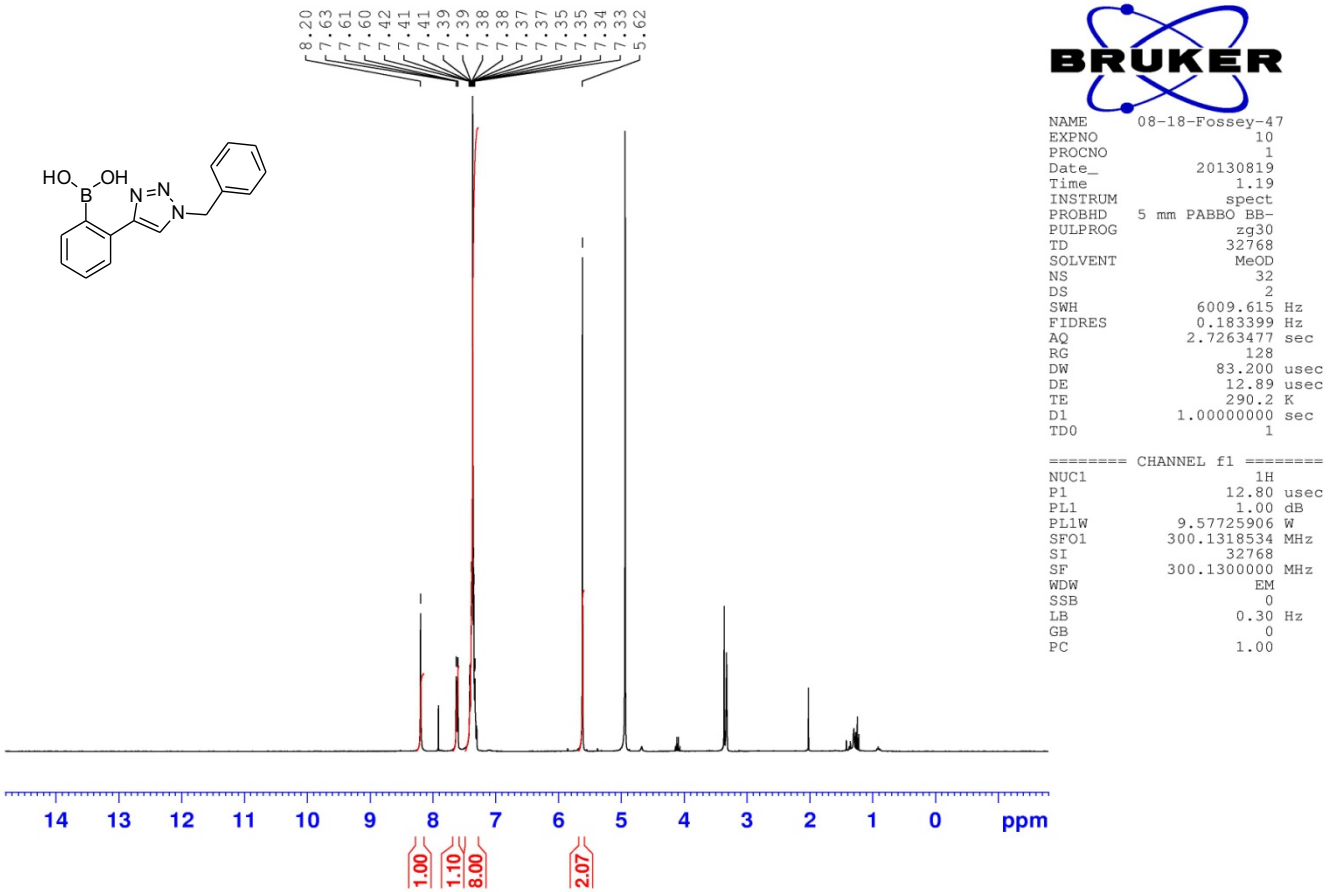
(4-((4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (1c) ^{13}C NMR spectrum



BRUKER

NAME	03-28-Fossey-12 carbon
EXPNO	11
PROCNO	1
Date_	20130329
Time	16.46
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	MeOD
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	295.0 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

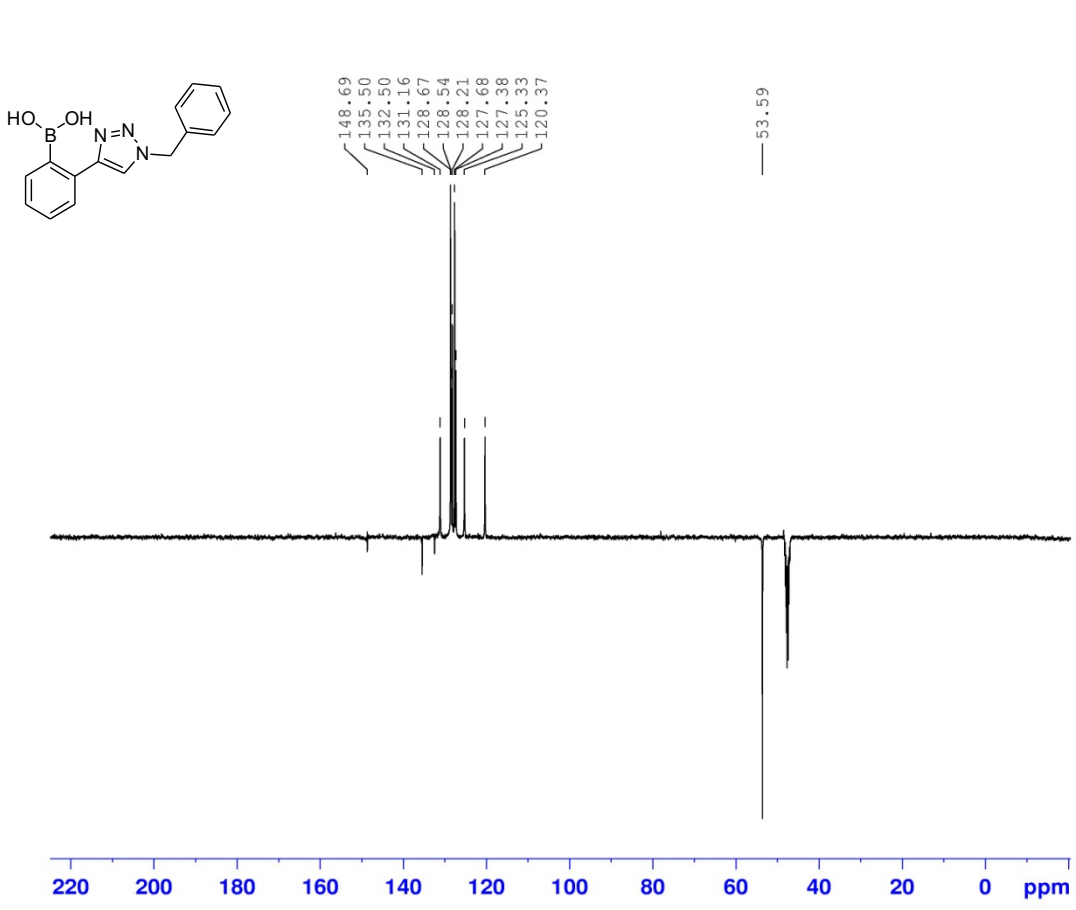
(2-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8a) ^{1}H NMR spectrum



BRUKER

NAME	08-18-Fossey-47
EXPNO	10
PROCNO	1
Date_	20130819
Time	1.19
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	128
DW	83.200 usec
DE	12.89 usec
TE	290.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

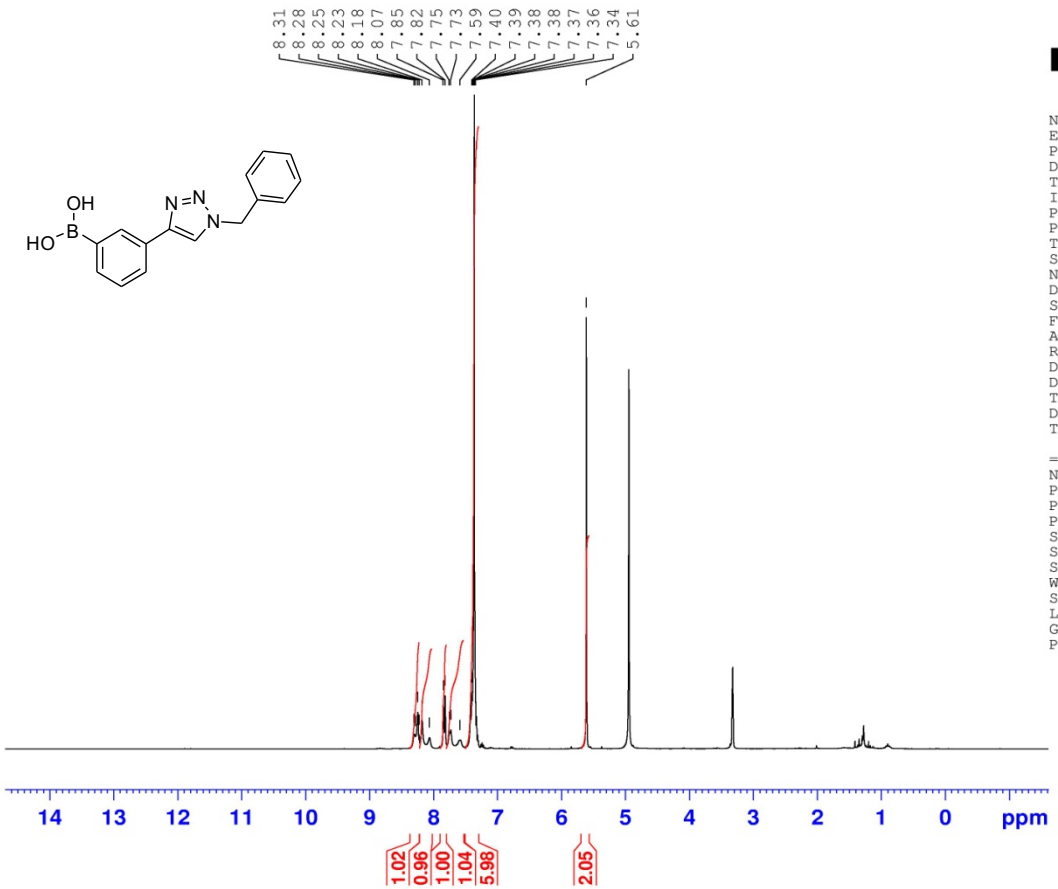
(2-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8a) ^{13}C NMR spectrum



BRUKER

NAME	08-18-Fossey-15
EXPNO	10
PROCNO	1
Date_	20130819
Time	2.07
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	MeOD
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	294.9 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

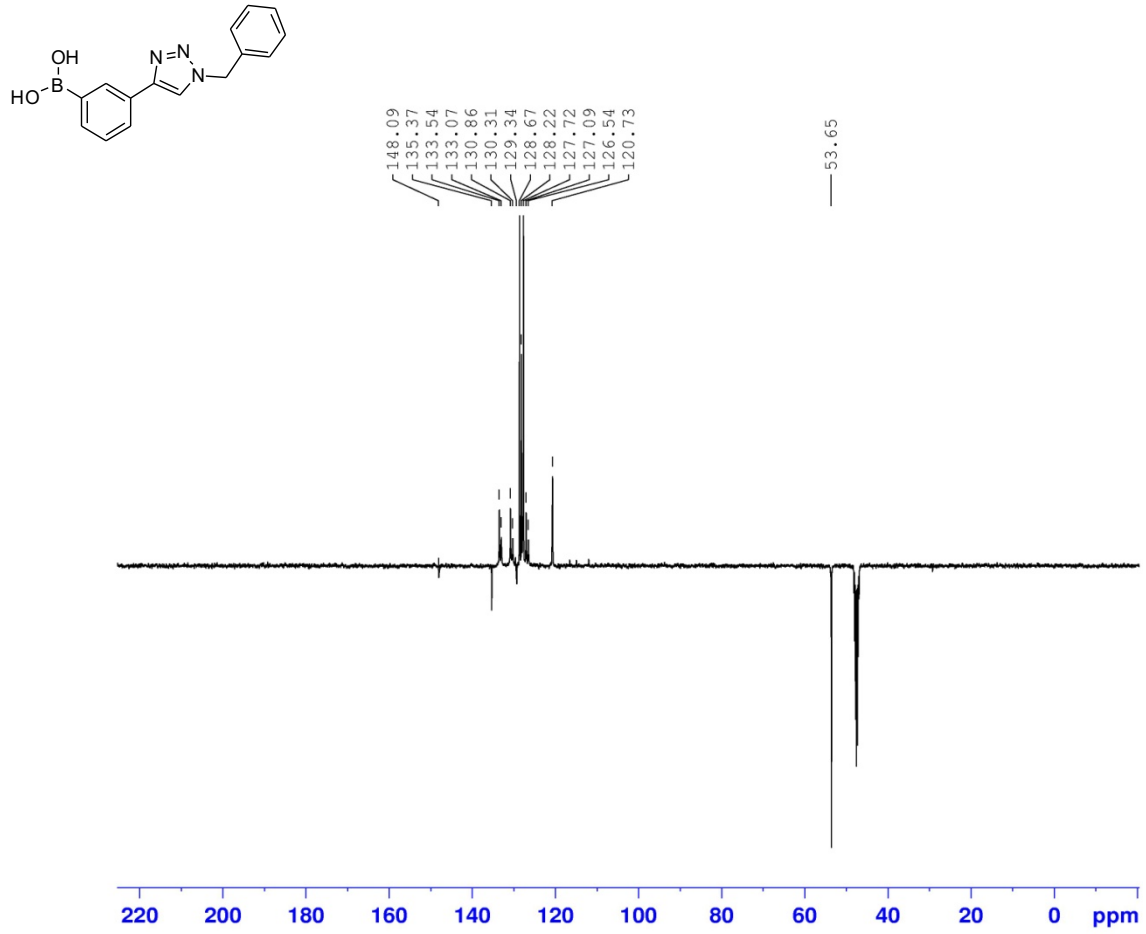
(3-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8b) ^{1}H NMR spectrum



BRUKER

NAME	08-20-Fossey-18
EXPNO	10
PROCNO	1
Date_	20130820
Time	20.02
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	128
DW	83.200 usec
DE	12.89 usec
TE	290.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

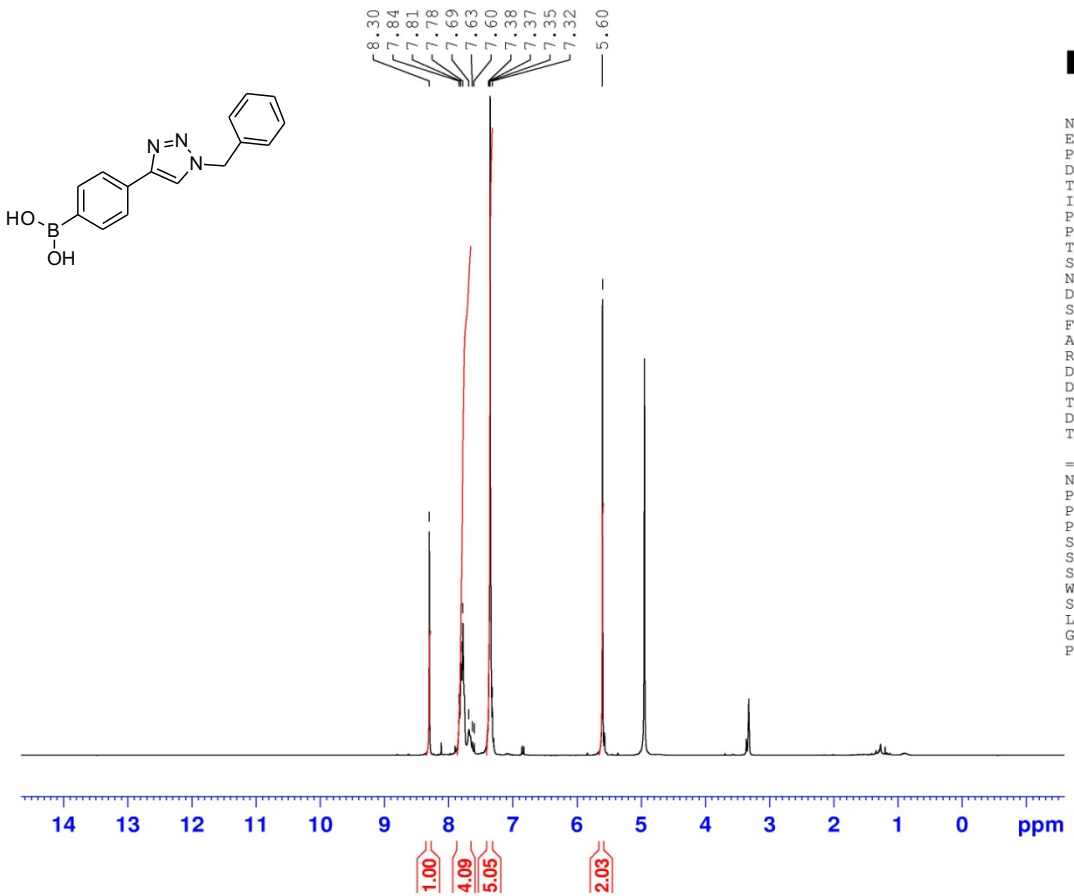
(3-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8b) ^{13}C NMR spectrum



BRUKER

NAME	08-20-Fossey-5
EXPNO	10
PROCNO	1
Date_	20130820
Time	20.56
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	MeOD
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	295.2 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

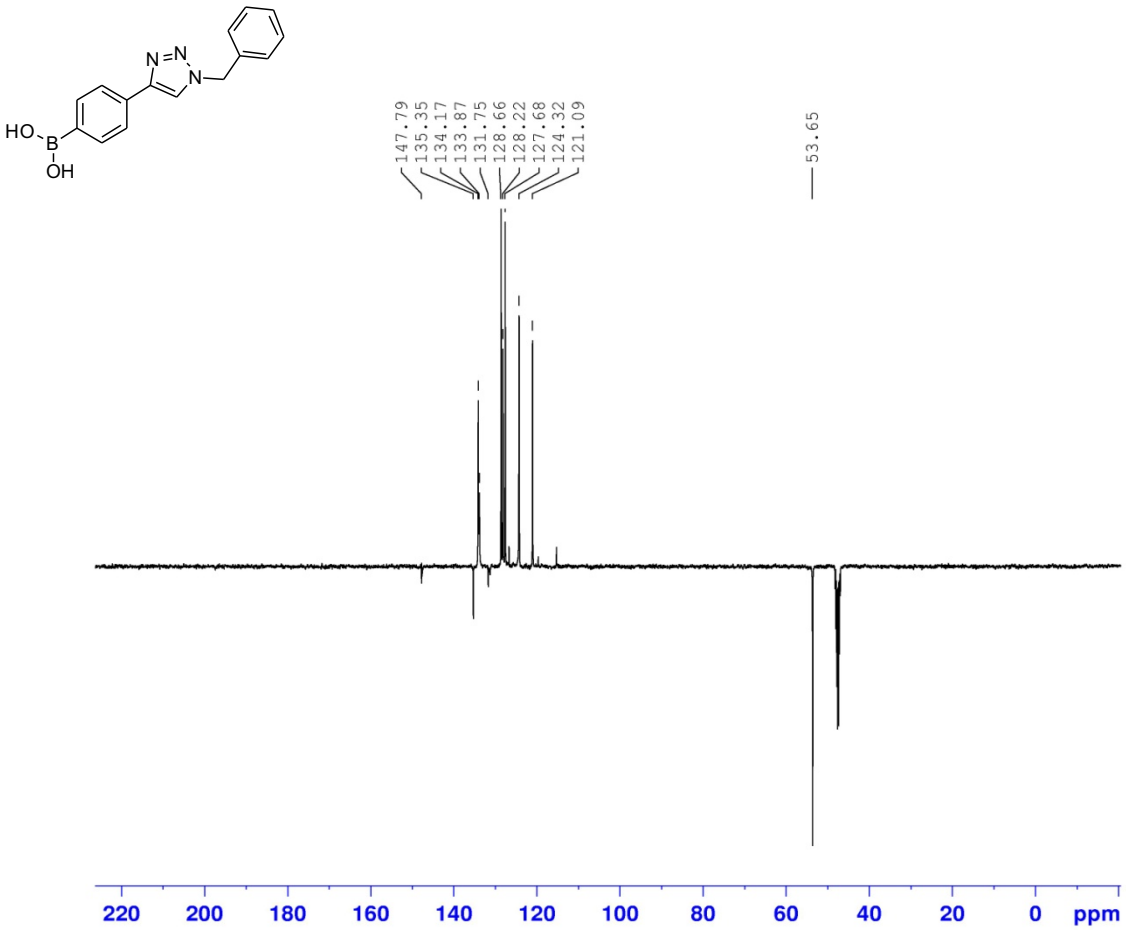
(4-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8c) ^{1}H NMR spectrum



BRUKER

NAME	06-20-Fossey-40
EXPNO	10
PROCNO	1
Date_	20130620
Time	23.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	114
DW	83.200 usec
DE	12.89 usec
TE	290.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

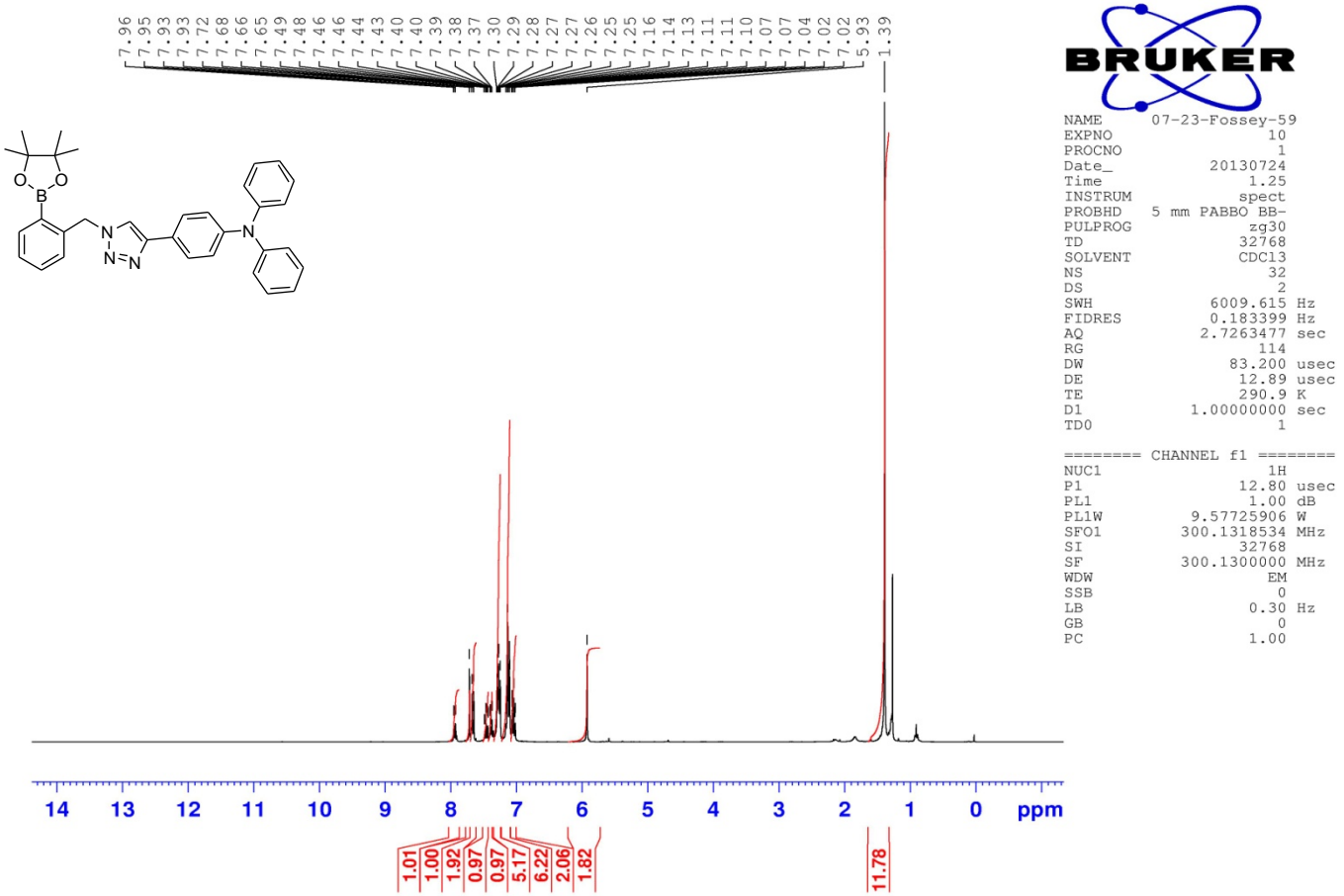
(4-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl)boronic acid (8c) ^{13}C NMR spectrum



BRUKER

NAME	06-20-Fossey-9
EXPNO	11
PROCNO	1
Date_	20130621
Time	2.55
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	MeOD
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	295.1 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

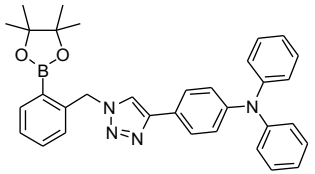
***N,N*-Diphenyl-4-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline (10a) ^{1}H NMR spectrum**

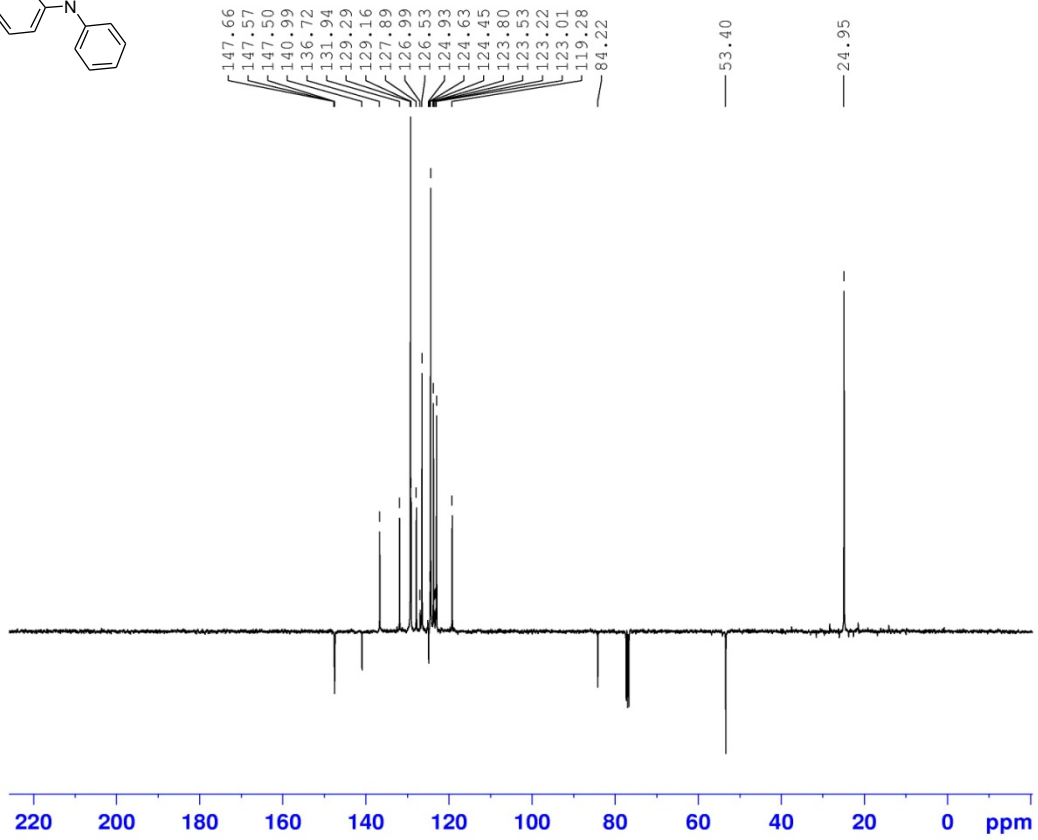



```
NAME          07-23-Fossey-59
EXPNO                      10
PROCNO                      1
Date_                20130724
Time                     1.25
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        32
DS                         2
SWH                 6009.615 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                       114
DW                    83.200 usec
DE                    12.89 usec
TE                    290.9 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     12.80 usec
PL1                     1.00 dB
PL1W              9.57725906 W
SFO1            300.1318534 MHz
SI                     32768
SF              300.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

***N,N*-Diphenyl-4-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline (10a) ^{13}C NMR spectrum**

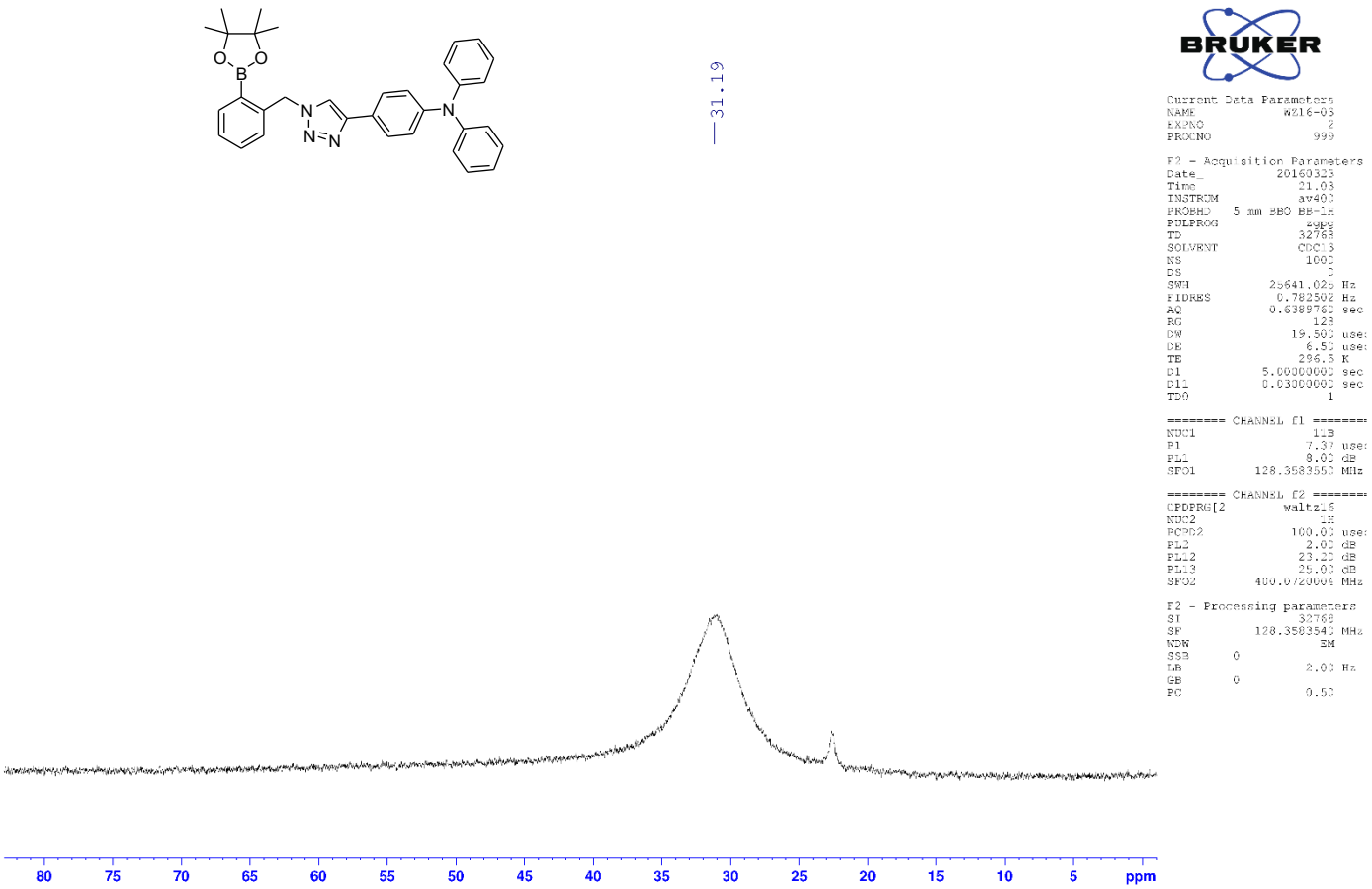




BRUKER

NAME	07-24-Fossey-1	
EXPNO	10	
PROCNO	1	
Date_	20130724	
Time	17.54	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	pendantns	
TD	65536	
SOLVENT	CDCl3	
NS	512	
DS	4	
SWH	25252.525	Hz
FIDRES	0.385323	Hz
AQ	1.2976629	sec
RG	2050	
DW	19.800	usec
DE	6.50	usec
TE	295.0	K
CNST2	145.0000000	
CNST3	1.0000000	
CNST4	5.0000000	
D1	1.50000000	sec
D2	0.00172414	sec
D3	0.00431034	sec
D12	0.00002000	sec
D13	0.00000400	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	8.80	usec
P2	17.60	usec
PL1	-3.00	dB
PL1W	58.63890457	W
SFO1	100.6233333	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
P3	9.70	usec
P4	19.40	usec
PCPD2	90.00	usec
PL2	-4.00	dB
PL12	15.35	dB
PL2W	24.29185867	W
PL12W	0.28213742	W
SFO2	400.1316005	MHz
SI	65536	
SF	100.6127690	MHz
WDW	EM	
SSB	0	
LB	4.00	Hz
GB	0	
PC	1.00	

***N,N*-Diphenyl-4-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline (10a) ^{11}B NMR spectrum**




```
Current Data Parameters
NAME                  WZ16-03
EXPNO                       2
PROCNO                    999

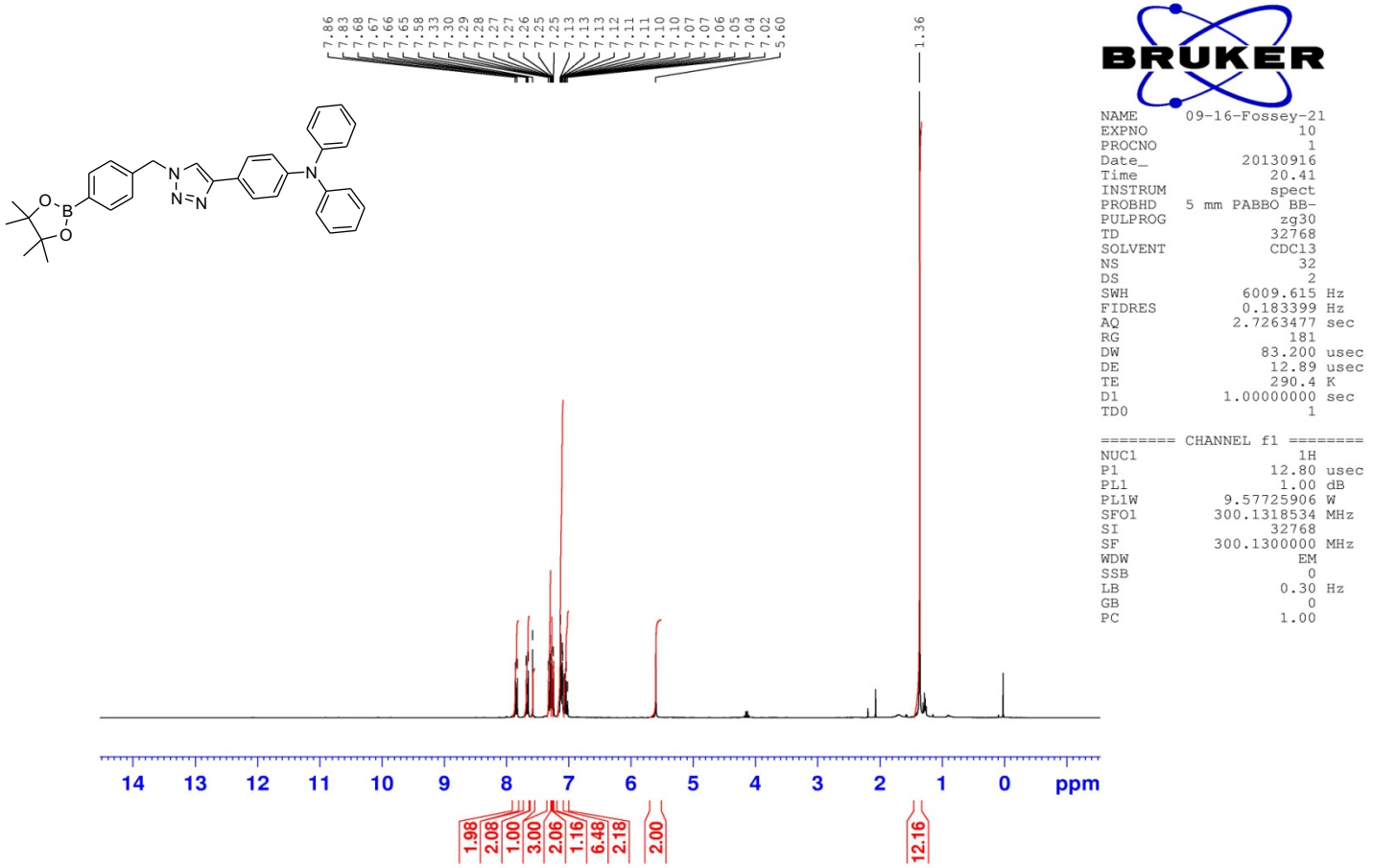
F2 - Acquisition Parameters
Date_              20160323
Time                   21.03
INSTRUM                av400
PROBHD    5 mm BBO BB-1H
PULPROG                zgBG
TD                      32768
SOLVENT                CDCl3
NS                       1000
DS                          0
SWH                25641.025 Hz
FIDRES             0.782502 Hz
AQ                 0.6389760 sec
RG                        128
DW                   19.500 use
DE                    6.50 use
TE                   296.5 K
D1                5.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      11B
P1                       7.37 use
PL1                      8.00 dB
SFO1             128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                      1H
PCPD2                  100.00 use
PL2                       2.00 dB
PL12                     23.20 dB
PL13                     25.00 dB
SFO2             400.0720004 MHz

F2 - Processing parameters
SI                       32768
SF               128.3583540 MHz
WDW                         EM
SSB          0
LB                        2.00 Hz
GB           0
PC                        0.50
```

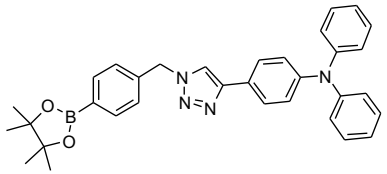
***N,N*-Diphenyl-4-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline (10b) ^{1}H NMR spectrum**

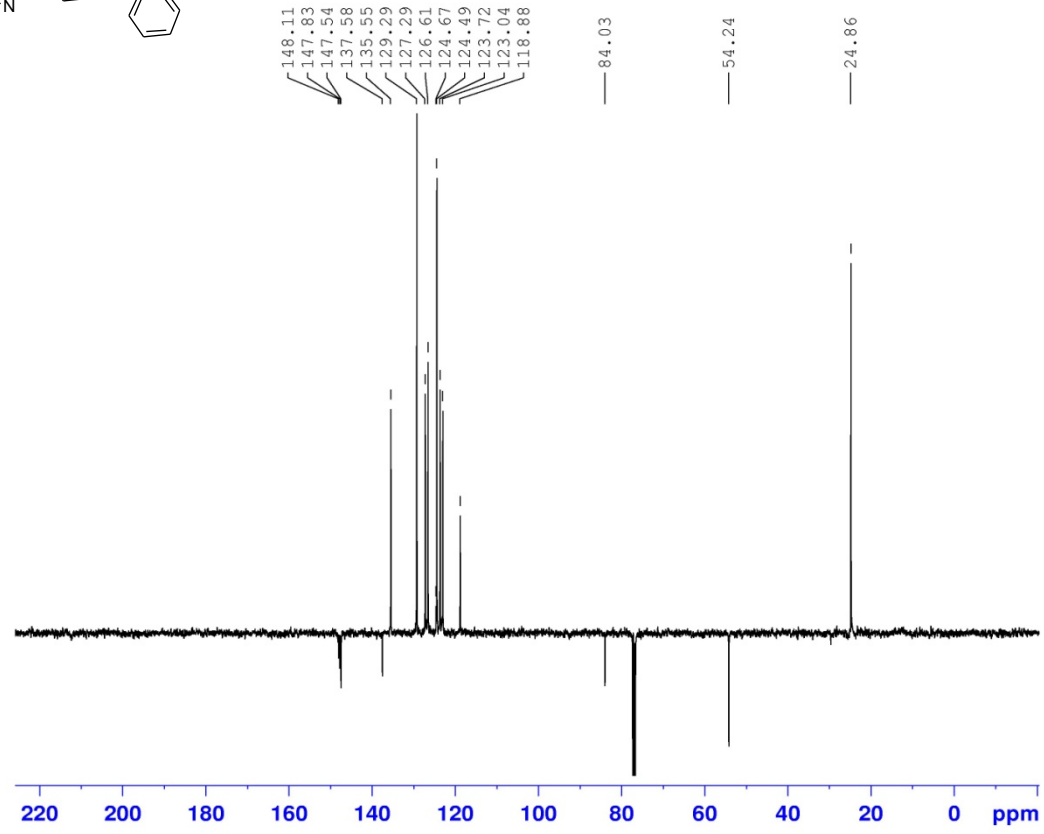


BRUKER

NAME	09-16-Fossey-21
EXPNO	10
PROCNO	1
Date_	20130916
Time	20.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	181
DW	83.200 usec
DE	12.89 usec
TE	290.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

***N,N*-Diphenyl-4-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline (10b) ^{13}C NMR spectrum**

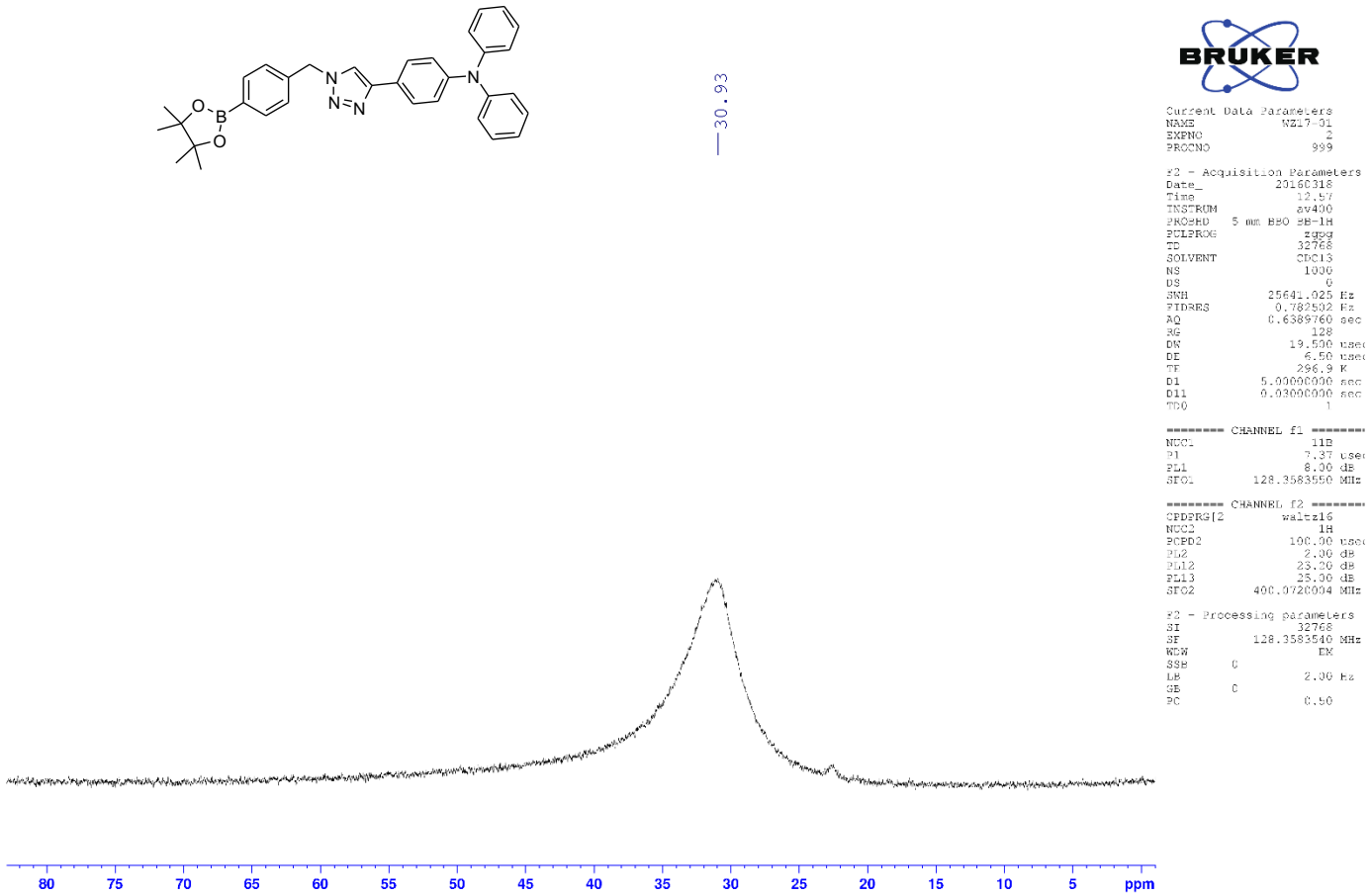




BRUKER

NAME	09-16-Fossey-9
EXPNO	10
PROCNO	1
Date_	20130916
Time	22.44
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	295.2 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

***N,N*-Diphenyl-4-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline (10b) ^{11}B NMR spectrum**




```
Current Data Parameters
NAME                  WZ17-01
EXPNO                       2
PROCNO                    999

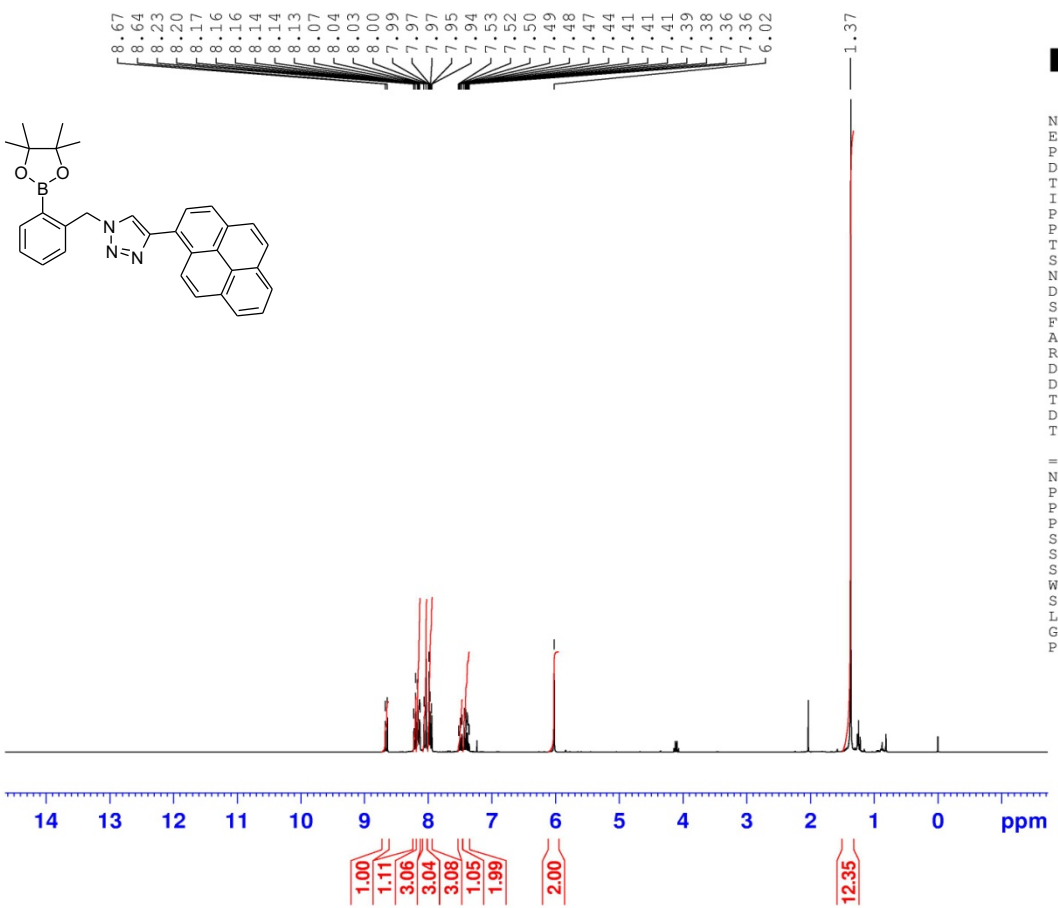
F2 - Acquisition Parameters
Date_              20160318
Time                   12.57
INSTRUM                av400
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                     1000
DS                        0
SWH               25641.025 Hz
FIDRES             0.782502 Hz
AQ                 0.6389760 sec
RG                      128
DW                 19.500 usec
DE                  6.50 usec
TE                  296.9 K
D1              5.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    11B
P1                   7.37 usec
PL1                  8.00 dB
SFO1          128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                  2.00 dB
PL12                23.20 dB
PL13                25.00 dB
SFO2          400.0720004 MHz

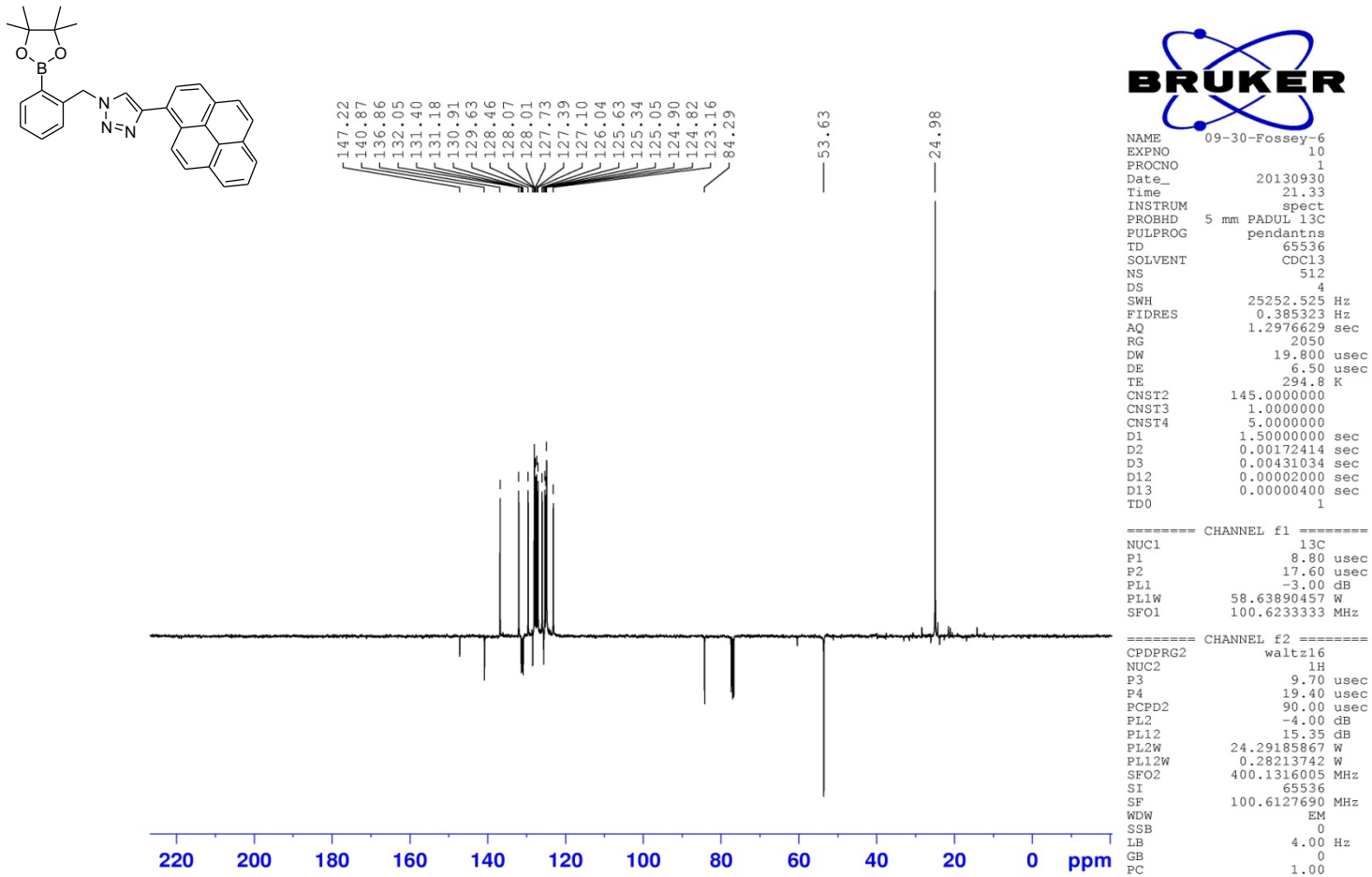
F2 - Processing parameters
SI                    32768
SF            128.3583540 MHz
WDW                      EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   0.50
```

4-(Pyren-1-yl)-1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (11a) ^{1}H NMR spectrum



NAME	09-30-Fossey-14
EXPNO	10
PROCNO	1
Date_	20130930
Time	20.22
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	90.5
DW	83.200 usec
DE	12.89 usec
TE	290.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300135 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

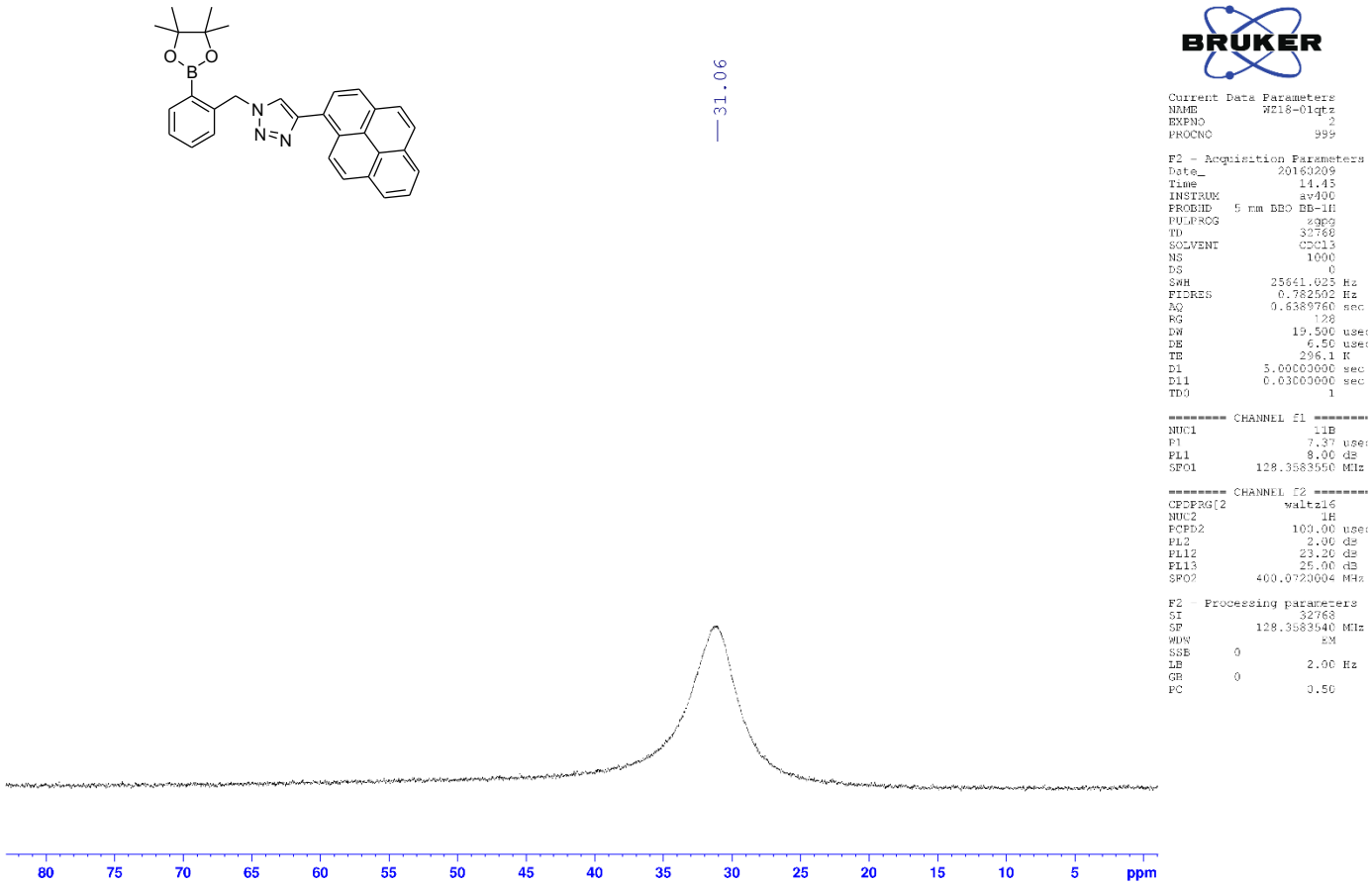
4-(Pyren-1-yl)-1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (11a) ^{13}C NMR spectrum



BRUKER

NAME	09-30-Fossey-6
EXPNO	10
PROCNO	1
Date_	20130930
Time	21.33
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	294.8 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
P2	17.60 usec
PL1	-3.00 dB
PL1W	58.63890457 W
SFO1	100.6233333 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
P3	9.70 usec
P4	19.40 usec
PCPD2	90.00 usec
PL2	-4.00 dB
PL12	15.35 dB
PL2W	24.29185867 W
PL12W	0.28213742 W
SFO2	400.1316005 MHz
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

4-(Pyren-1-yl)-1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (11a) ^{11}B NMR spectrum




```
Current Data Parameters
NAME            WZ18-01qtz
EXPNO                    2
PROCNO                 999

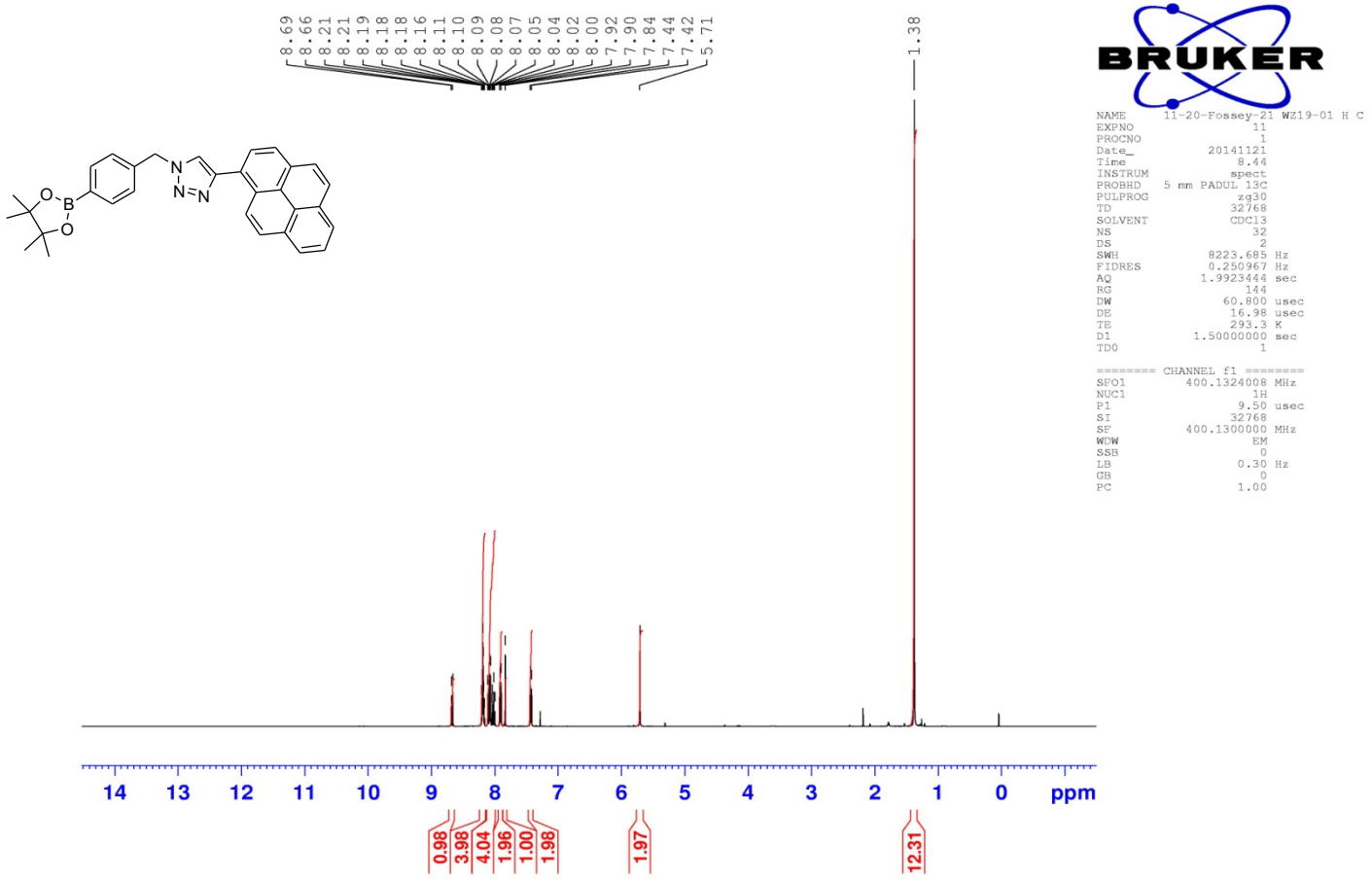
F2 - Acquisition Parameters
Date_           20160209
Time                14.45
INSTRUM             av400
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg
TD                 32768
SOLVENT            CDCl3
NS                  1000
DS                     0
SWH           25641.025 Hz
FIDRES        0.782502 Hz
AQ            0.6389760 sec
RG                   128
DW               19.500 use
DE                6.50 use
TE              296.1 K
D1            5.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                11B
P1                 7.37 use
PL1                8.00 dB
SFO1        128.3583550 MHz

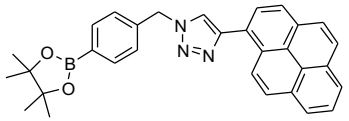
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2            100.00 use
PL2                2.00 dB
PL12              23.20 dB
PL13              25.00 dB
SFO2        400.0720004 MHz

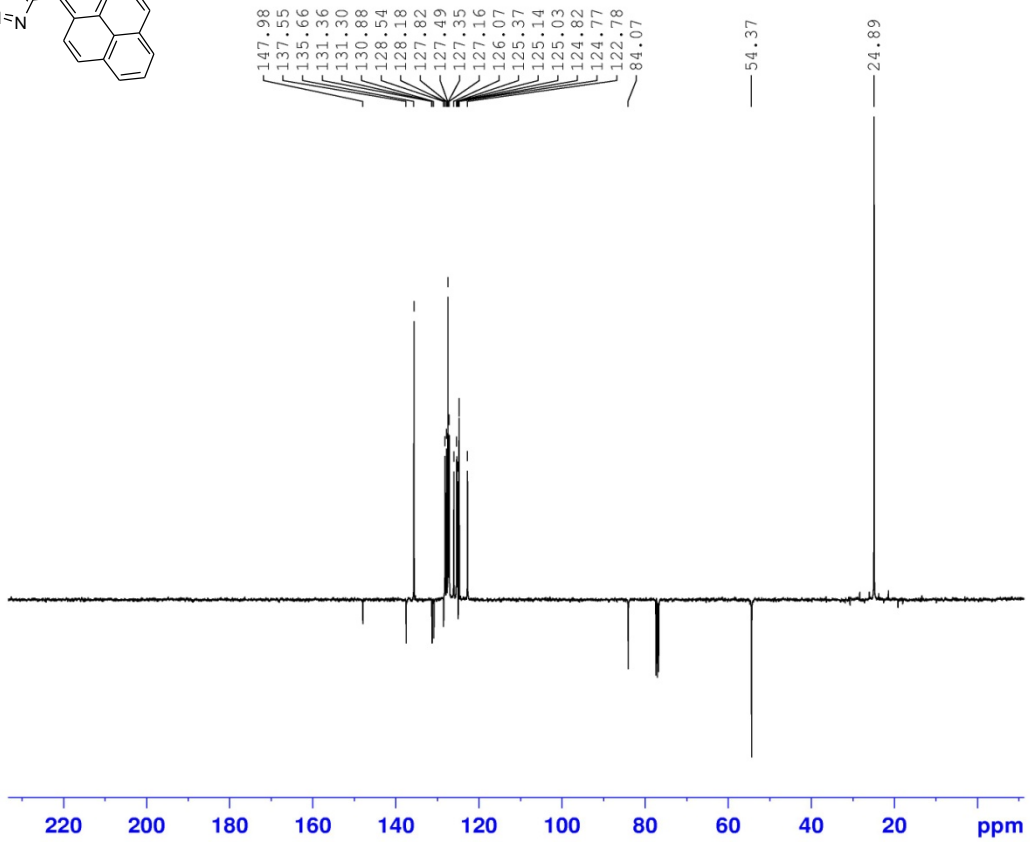
F2 - Processing parameters
SI                 32768
SF          128.3583540 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 0.50
```

4-(Pyren-1-yl)-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (11b) ^{1}H NMR spectrum



4-(Pyren-1-yl)-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (11b) ^{13}C NMR spectrum



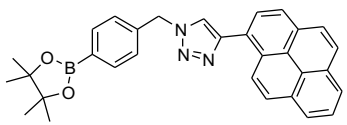


BRUKER

```
NAME          11-20-Fossey-21 WZ19-01 H C
EXPNO                      10
PROCNO                      1
Date_                20141121
Time                     8.41
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG             pendantns
TD                      65536
SOLVENT                 CDCl3
NS                        512
DS                          4
SWH                 25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                         2050
DW                       19.800 usec
DE                        6.50 usec
TE                       293.7 K
CNST2               145.0000000
CNST3                 1.0000000
CNST4                 5.0000000
D1                    1.50000000 sec
D2                    0.00172414 sec
D3                    0.00431034 sec
D12                   0.00002000 sec
D13                   0.00000400 sec
TD0                         1

======== CHANNEL f1 ========
SFO1                100.6242690 MHz
NUC1                        13C
P1                        8.80 usec
P2                       17.60 usec
SI                       65536
SF                  100.6127690 MHz
WDW                         EM
SSB                          0
LB                        4.00 Hz
GB                           0
PC                        1.00
```

4-(Pyren-1-yl)-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole (11b) ^{11}B NMR spectrum




```
Current Data Parameters
NAME                  WZ19-01
EXPNO                       2
PROCNO                    999

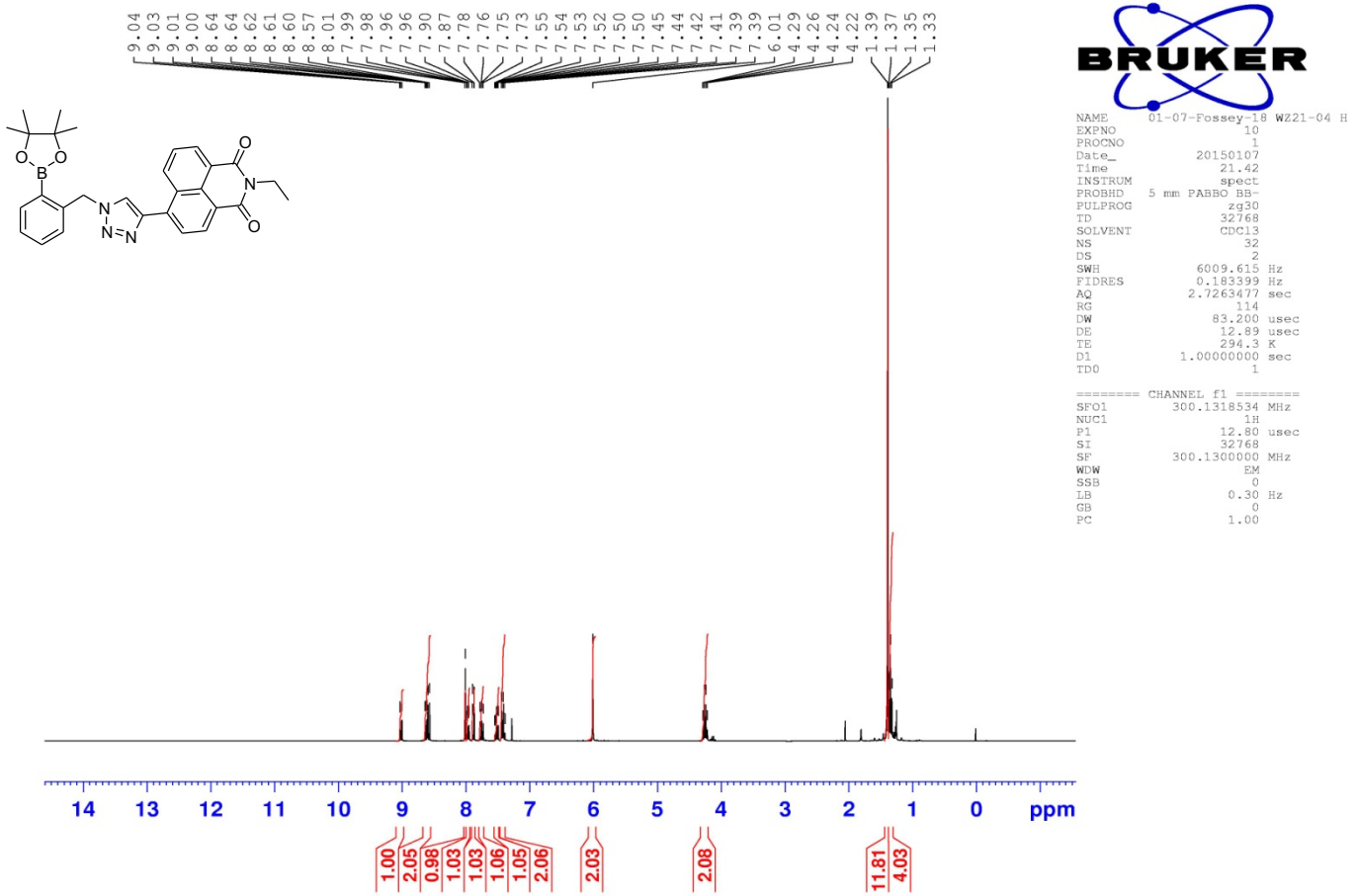
F2 - Acquisition Parameters
Date_              20160202
Time                  18.33
INSTRM                av400
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                     1000
DS                        0
SWH               25641.025 Hz
FIDRES            0.782502 Hz
AQ                1.6389760 sec
RG                      128
DW                 19.500 usec
DE                  6.50 usec
TE                  296.8 K
D1              5.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    11B
P1                   7.37 usec
PL1                  8.00 dB
SFO1          128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  2.00 dB
PL12                23.20 dB
PL13                25.00 dB
SFO2          400.0720004 MHz

F2 - Processing parameters
SI                    32768
SF            128.3583540 MHz
WDW                      EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   0.50
```

2-Ethyl-6-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione (12a) ^{1}H NMR spectrum

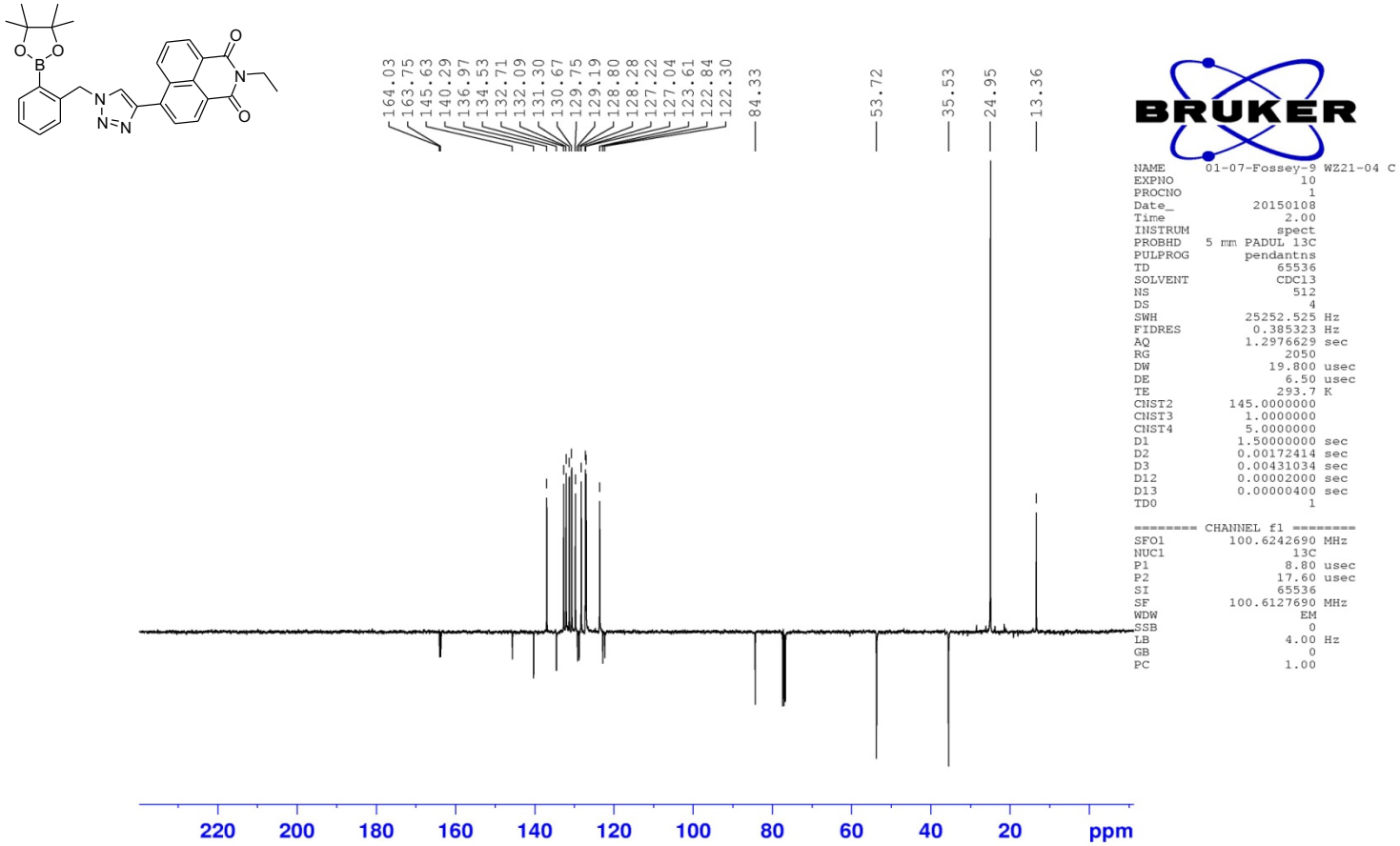


BRUKER

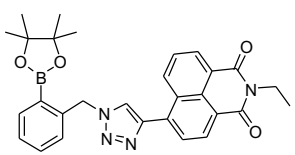
```
NAME          01-07-Fossey-18 WZ21-04 H
EXPNO                      10
PROCNO                      1
Date_                20150107
Time                    21.42
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        32
DS                         2
SWH                 6009.615 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                       114
DW                    83.200 usec
DE                    12.89 usec
TE                    294.3 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               300.1318534 MHz
NUC1                       1H
P1                    12.80 usec
SI                     32768
SF                 300.1300000 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

2-Ethyl-6-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione (12a) ^{13}C NMR spectrum



2-Ethyl-6-(1-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione (12a) ^{11}B NMR spectrum




```
Current Data Parameters
NAME                  WZ21-04
EXPNO                       2
PROCNO                    999

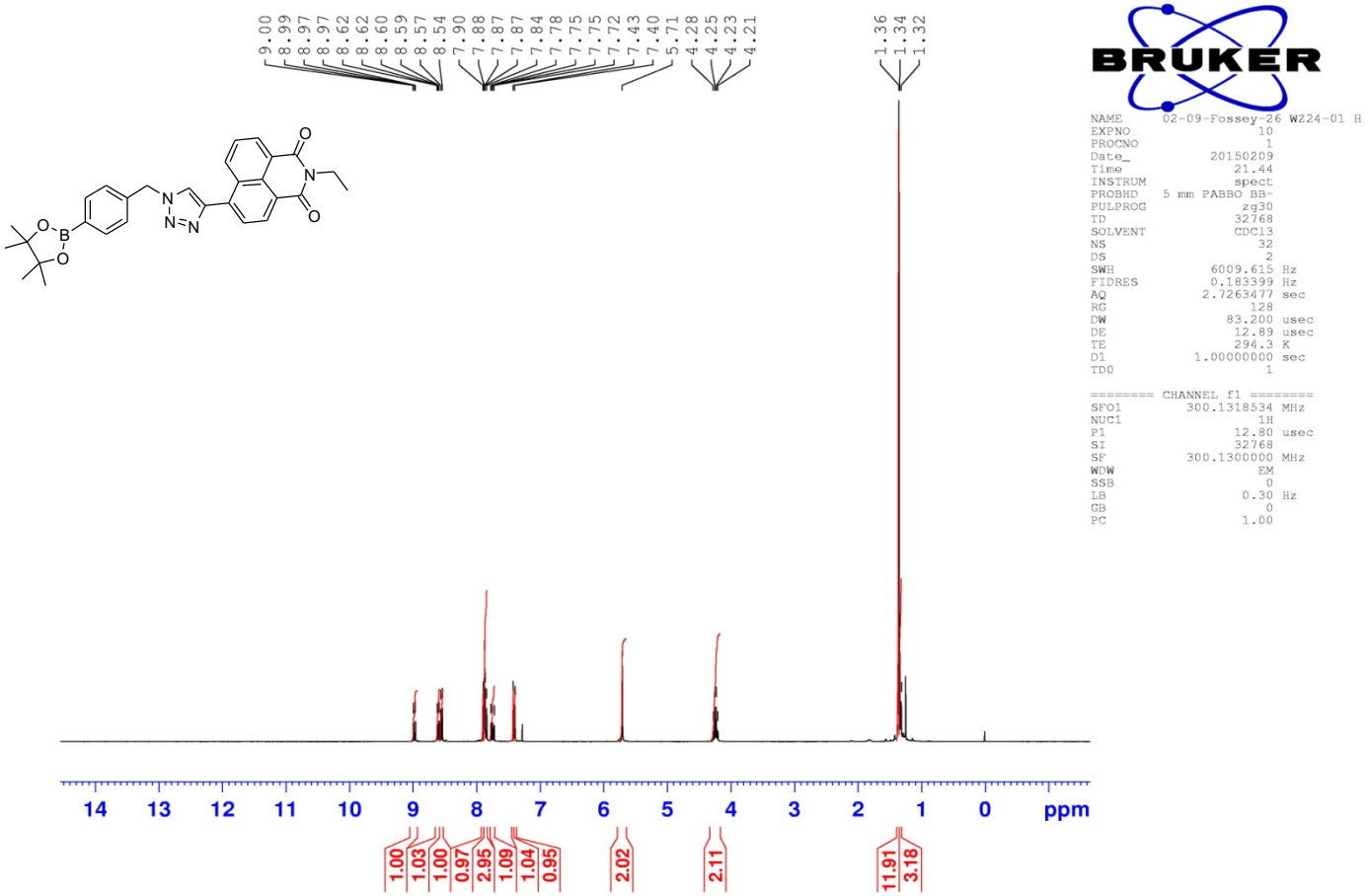
F2 - Acquisition Parameters
Date_              20160205
Time                   14.06
INSTRUM                av400
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                     1000
DS                        0
SWH               25641.025 Hz
FIDRES            0.782532 Hz
AQ                0.6389760 sec
RG                      128
DW                 19.500 usec
DE                  6.50 usec
TE                  296.7 K
D1                5.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    11B
P1                   7.37 usec
PL1                  8.00 dB
SFO1           128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  2.00 dB
PL12                23.20 dB
PL13                25.00 dB
SFO2           400.0730004 MHz

F2 - Processing parameters
SI                    32768
SF             128.3583540 MHz
WDW                      EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   0.50
```

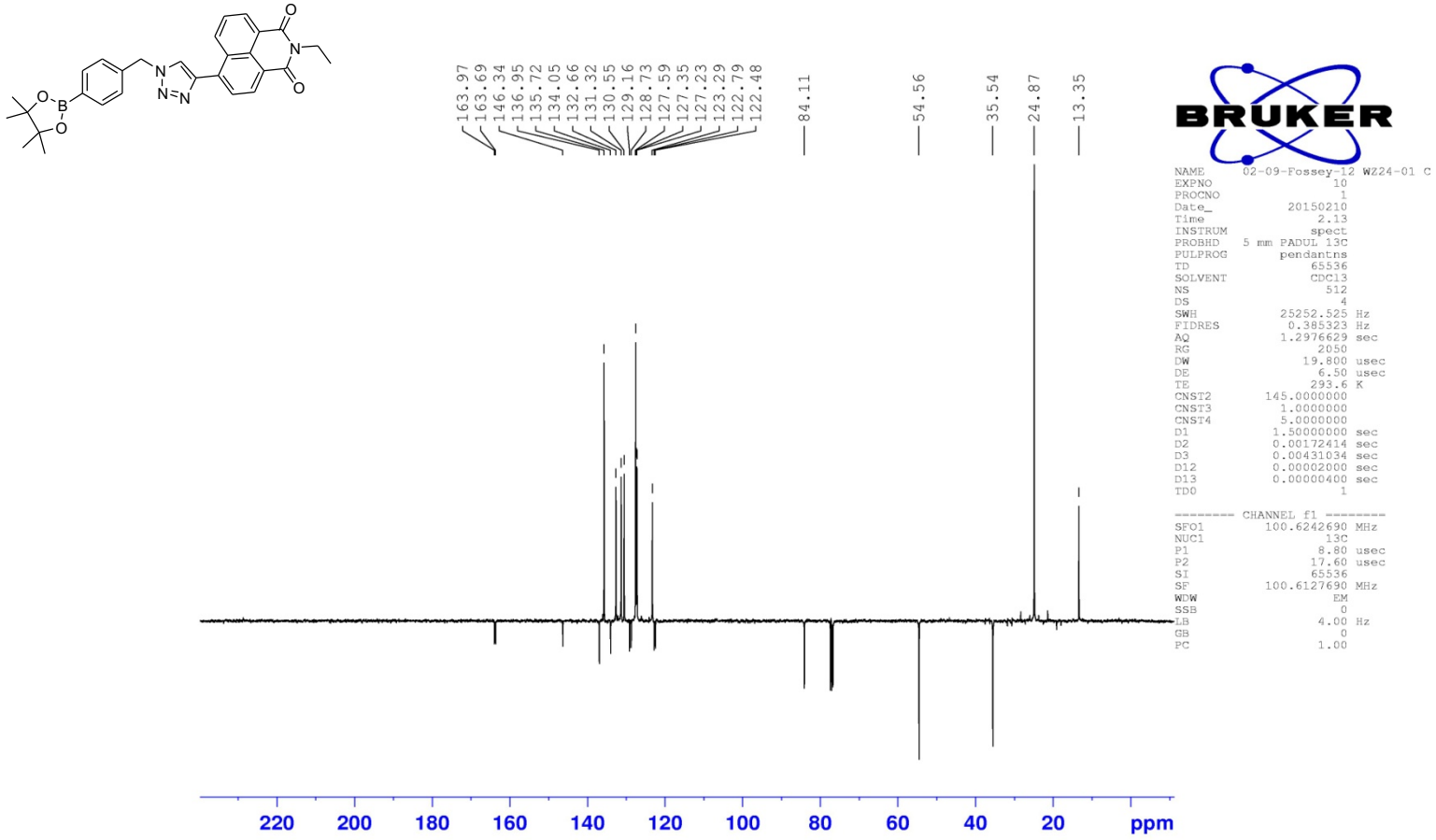
2-Ethyl-6-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione (12b) ^{1}H NMR spectrum



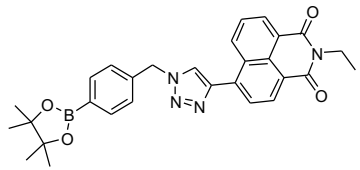

```
NAME          02-09-Fossey-26 WZ24-01 H
EXPNO                   10
PROCNO                   1
Date_              20150209
Time                 21.44
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        2
SWH                6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7263477 sec
RG                      128
DW                   83.200 usec
DE                   12.89 usec
TE                   294.3 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1             300.1318534 MHz
NUC1                      1H
P1                   12.80 usec
SI                    32768
SF               300.1300000 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                     1.00
```

2-Ethyl-6-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione (12b) ^{13}C NMR spectrum



2-Ethyl-6-(1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione (12b) ^{11}B NMR spectrum




```
Current Data Parameters
NAME                  WZ24-01
EXPNO                       2
PROCNO                    999

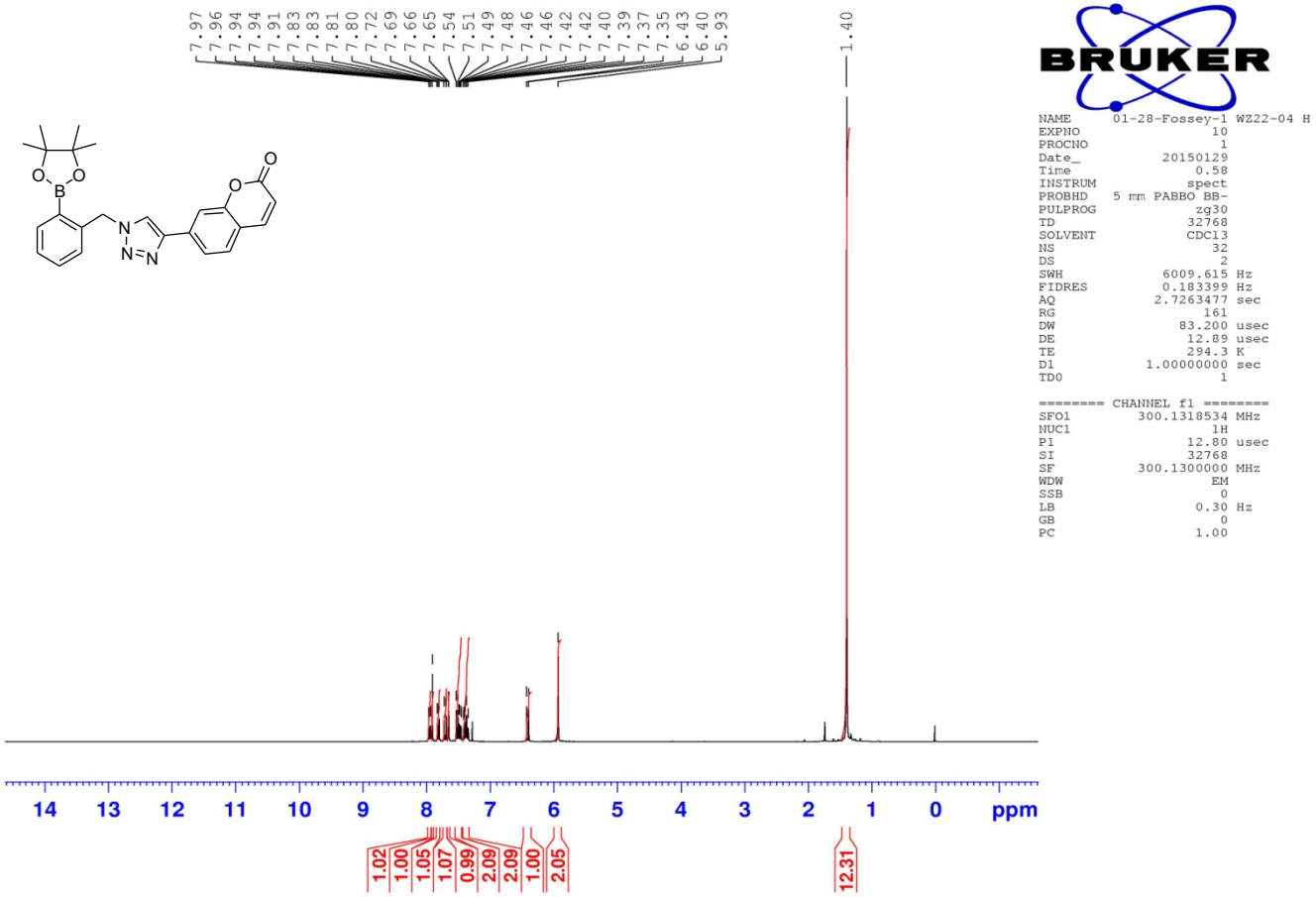
F2 - Acquisition Parameters
Date_              20160209
Time                   18.50
INSTRUM                av400
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg
TD                     32768
SOLVENT                CDCl3
NS                      1000
DS                         0
SWH               25641.025 Hz
FIDRES            0.782502 Hz
AQ                0.6389760 sec
RG                       128
DW                  19.500 use
DE                   6.50 use
TE                   296.2 K
D1              5.00000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     11B
P1                      7.37 use
PL1                     8.00 dB
SFO1           128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                     1H
PCPD2                100.00 use
PL2                     2.00 dB
PL12                   23.20 dB
PL13                   25.00 dB
SFO2           400.0720004 MHz

F2 - Processing parameters
SI                       32768
SF             128.3583540 MHz
WDW                         EM
SSB          0
LB                      2.00 Hz
GB           0
PC                      0.50
```

7-(1-(2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13a) ^{1}H NMR spectrum

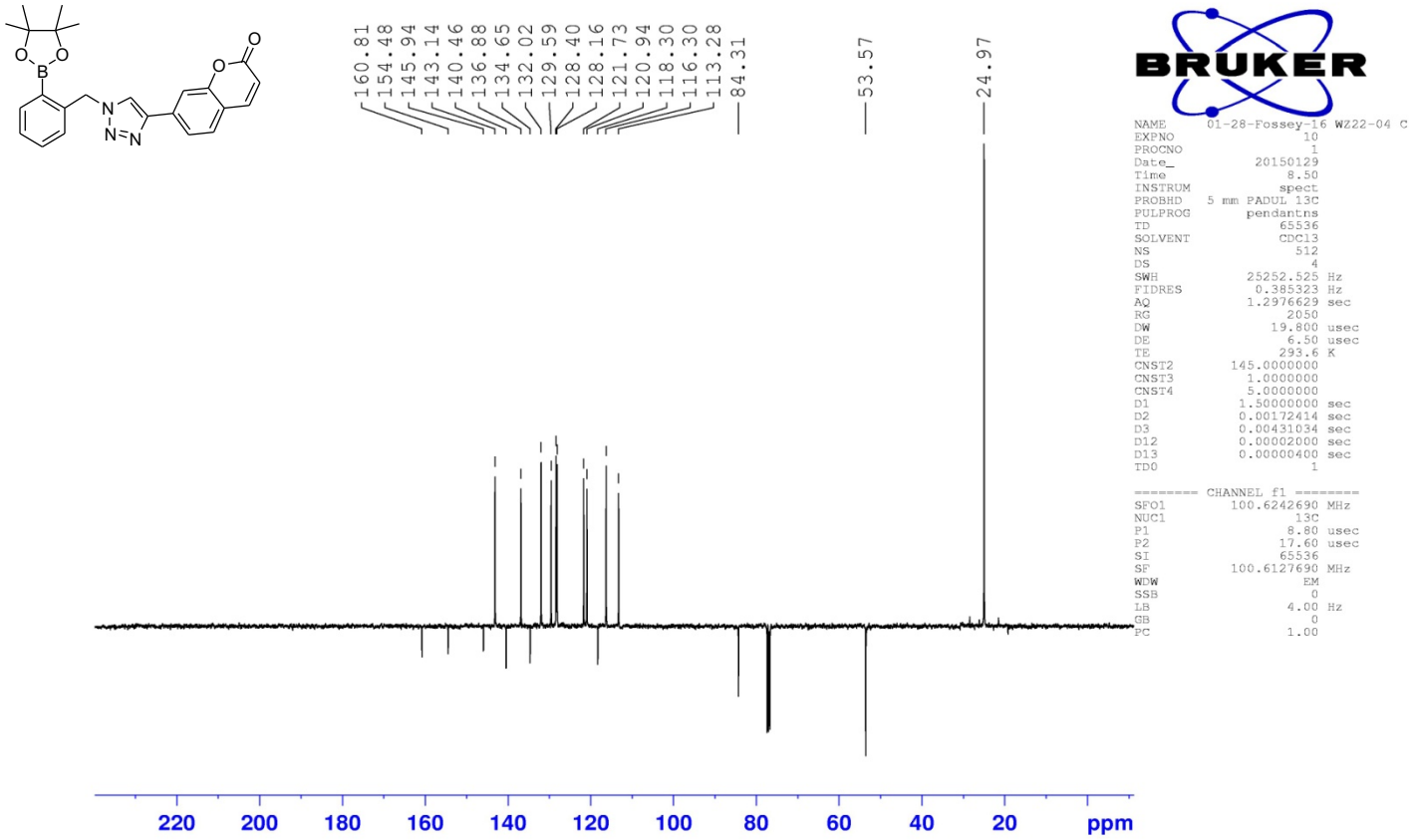


BRUKER

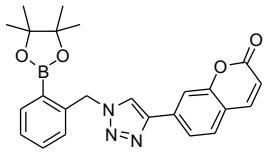
```
NAME          01-28-Fossey-1 WZ22-04 H
EXPNO                      10
PROCNO                      1
Date_                20150129
Time                     0.58
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        32
DS                         2
SWH                 6009.615 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                       161
DW                    83.200 usec
DE                    12.89 usec
TE                    294.3 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             300.1318534 MHz
NUC1                      1H
P1                    12.80 usec
SI                     32768
SF               300.1300000 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                    1.00
```

7-(1-(2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13a) ^{13}C NMR spectrum



7-(1-(2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13a) ^{11}B NMR spectrum




```
Current Data Parameters
NAME                  WZ22-04
EXPNO                       2
PROCNO                    999

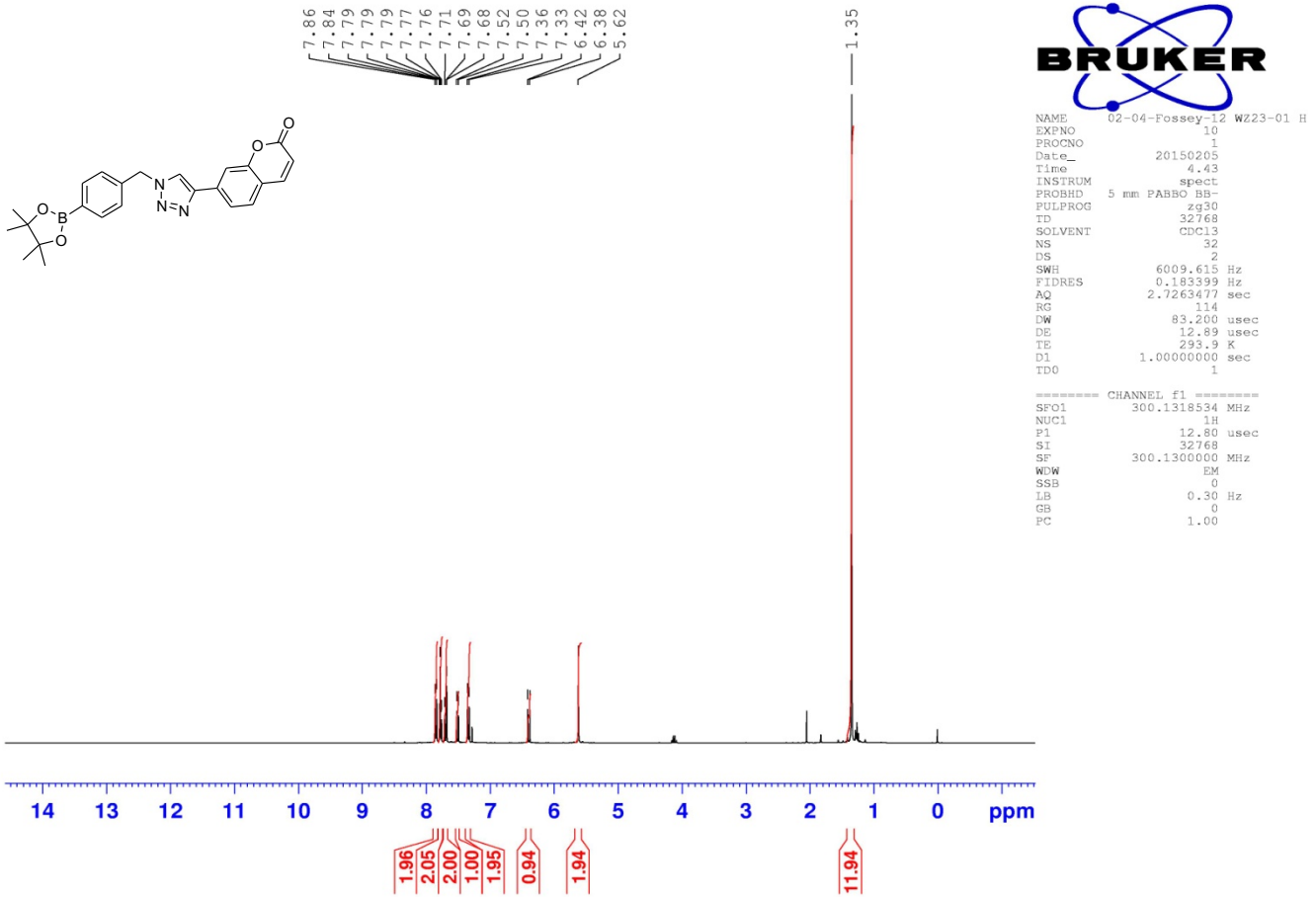
F2 - Acquisition Parameters
Date_              20160205
Time                   19.21
INSTRUM                av400
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                     1000
DS                        0
SWH               25641.025 Hz
FIDRES             0.782502 Hz
AQ                 0.6389760 sec
RG                      128
DW                 19.500 usec
DE                  6.50 usec
TE                  296.4 K
D1                5.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    11B
P1                   7.37 usec
PL1                  8.00 dB
SFO1          128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                  2.00 dB
PL12                23.20 dB
PL13                25.20 dB
SFO2          400.0720004 MHz

F2 - Processing parameters
SI                    32768
SF            128.3583540 MHz
WDW                      EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   0.50
```

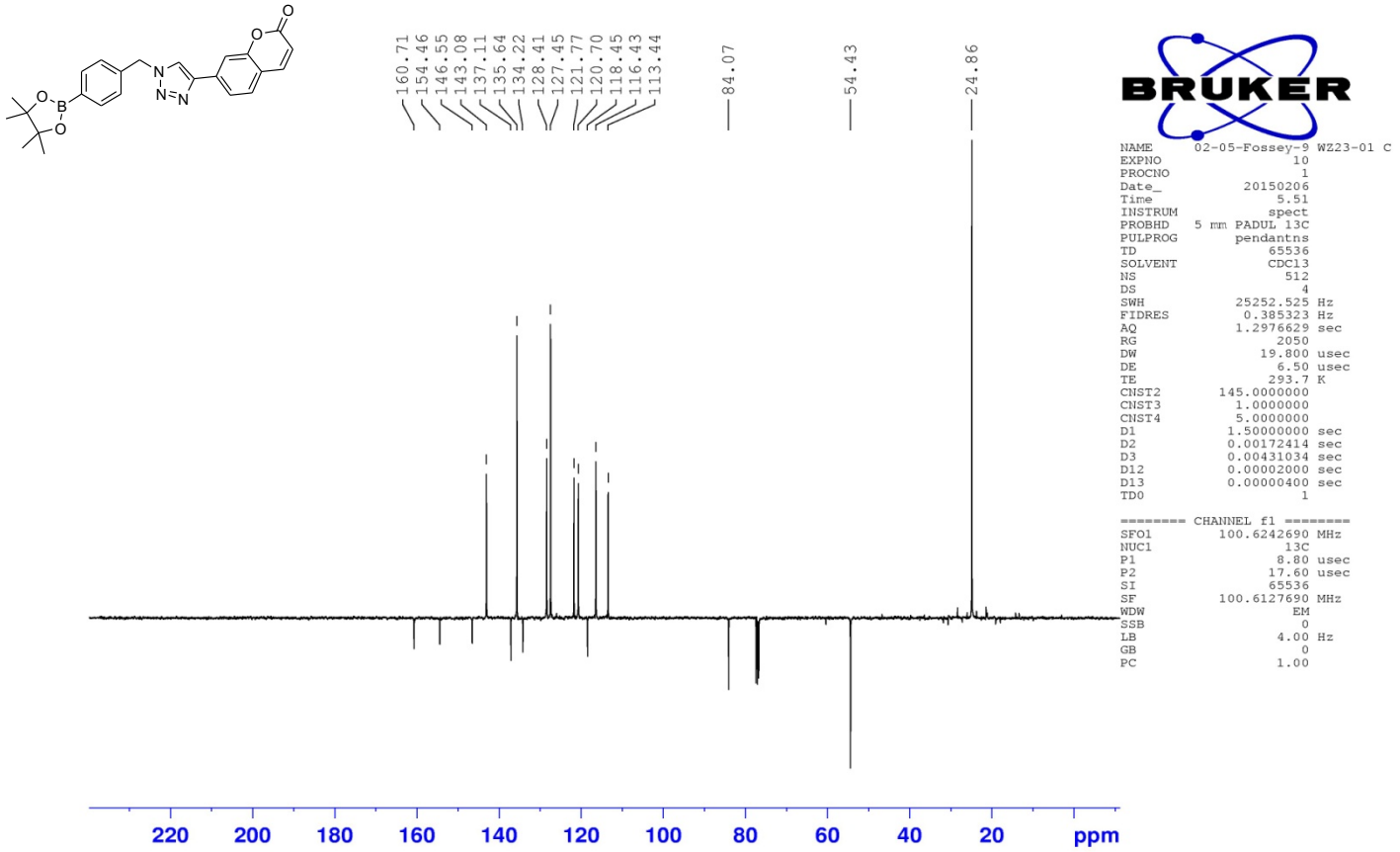
7-(1-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13b) ^{1}H NMR spectrum



BRUKER

NAME	02-04-Fossey-12 WZ23-01 H
EXPNO	10
PROCNO	1
Date_	20150205
Time	4.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	114
DW	83.200 usec
DE	12.89 usec
TE	293.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

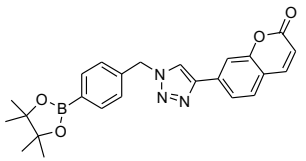
7-(1-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13b) ^{13}C NMR spectrum




```
NAME          02-05-Fossey-9 WZ23-01 C
EXPNO                    10
PROCNO                    1
Date_              20150206
Time                   5.51
INSTRUM               spect
PROBHD     5 mm PADUL 13C
PULPROG           pendantns
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        4
SWH               25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                     2050
DW                   19.800 usec
DE                    6.50 usec
TE                   293.7 K
CNST2           145.0000000
CNST3             1.0000000
CNST4             5.0000000
D1                1.50000000 sec
D2                0.00172414 sec
D3                0.00431034 sec
D12               0.00002000 sec
D13               0.00000400 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6242690 MHz
NUC1                    13C
P1                    8.80 usec
P2                   17.60 usec
SI                    65536
SF              100.6127690 MHz
WDW                     EM
SSB                      0
LB                    4.00 Hz
GB                       0
PC                    1.00
```

7-(1-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazol-4-yl)-2*H*-chromen-2-one (13b) ^{11}B NMR spectrum




```
Current Data Parameters
NAME                  WZ23-01
EXPNO                       2
PROCNO                    999

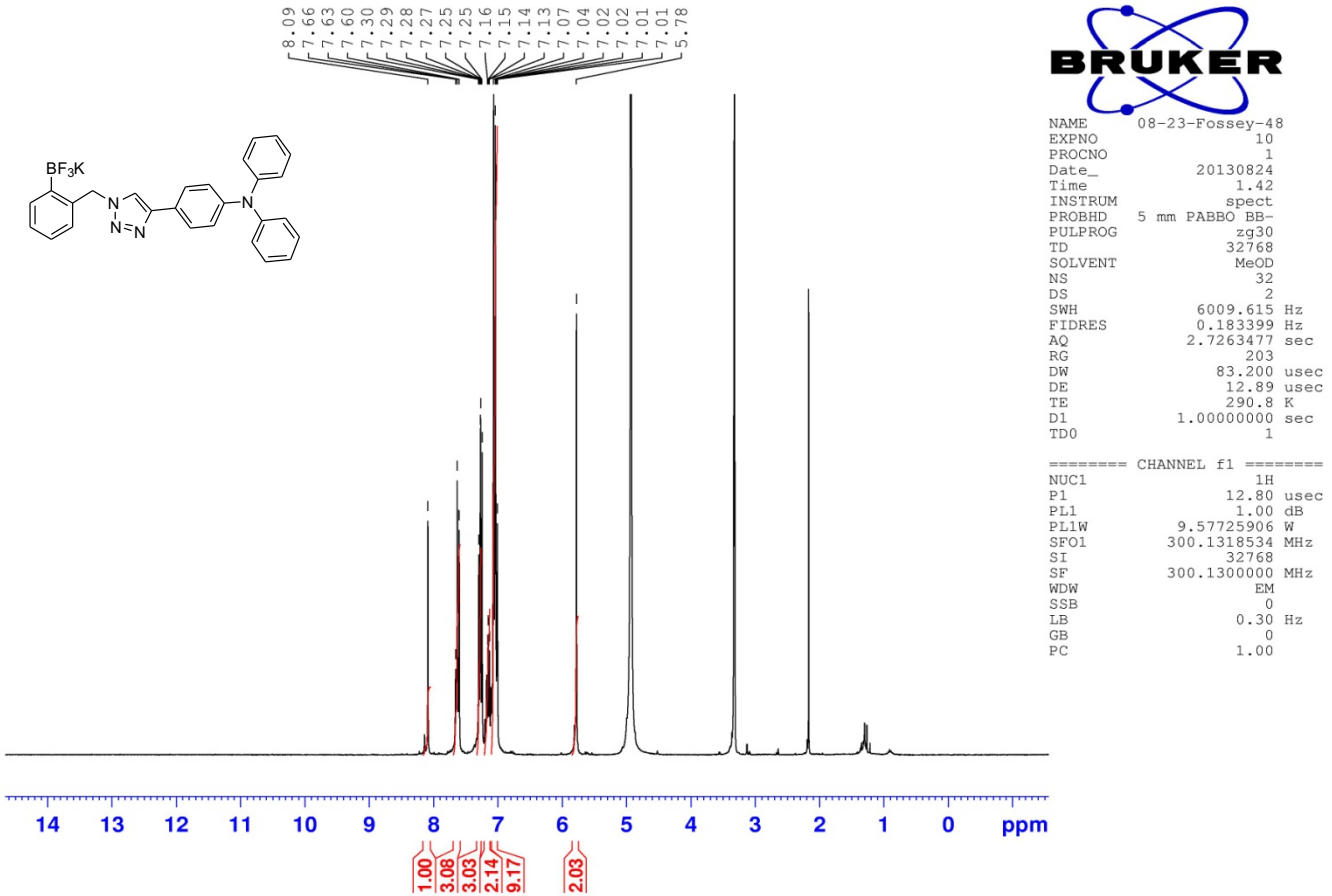
F2 - Acquisition Parameters
Date_              20160208
Time                   14.26
INSTRUM                av400
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                     1000
DS                        0
SWH               25641.025 Hz
FIDRES            0.782502 Hz
AQ                0.6389760 sec
RG                      128
DW                 19.500 use
DE                  6.50 use
TE                  296.9 K
D1              5.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    11B
P1                   7.37 use
PL1                  8.00 dB
SFO1          128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2            waltz16
NUC2                    1H
PCPD2               100.00 use
PL2                  2.00 dB
PL12                23.20 dB
PL13                25.00 dB
SFO2          400.0720004 MHz

F2 - Processing parameters
SI                     32768
SF            128.3583940 MHz
WDW                       EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   0.50
```

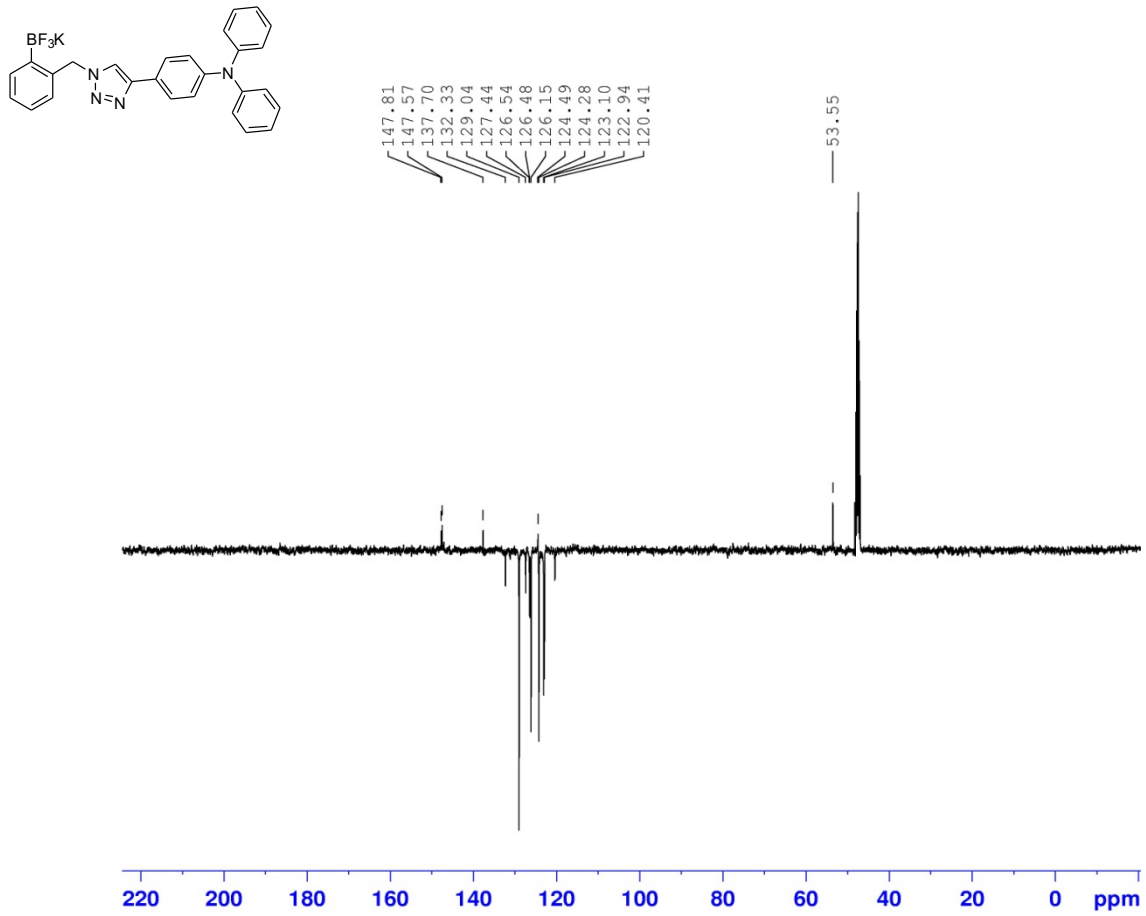
***N,N*-Diphenyl-4-(1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline, potassium salt ^{1}H NMR spectrum**



BRUKER

NAME	08-23-Fossey-48
EXPNO	10
PROCNO	1
Date_	20130824
Time	1.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	203
DW	83.200 usec
DE	12.89 usec
TE	290.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

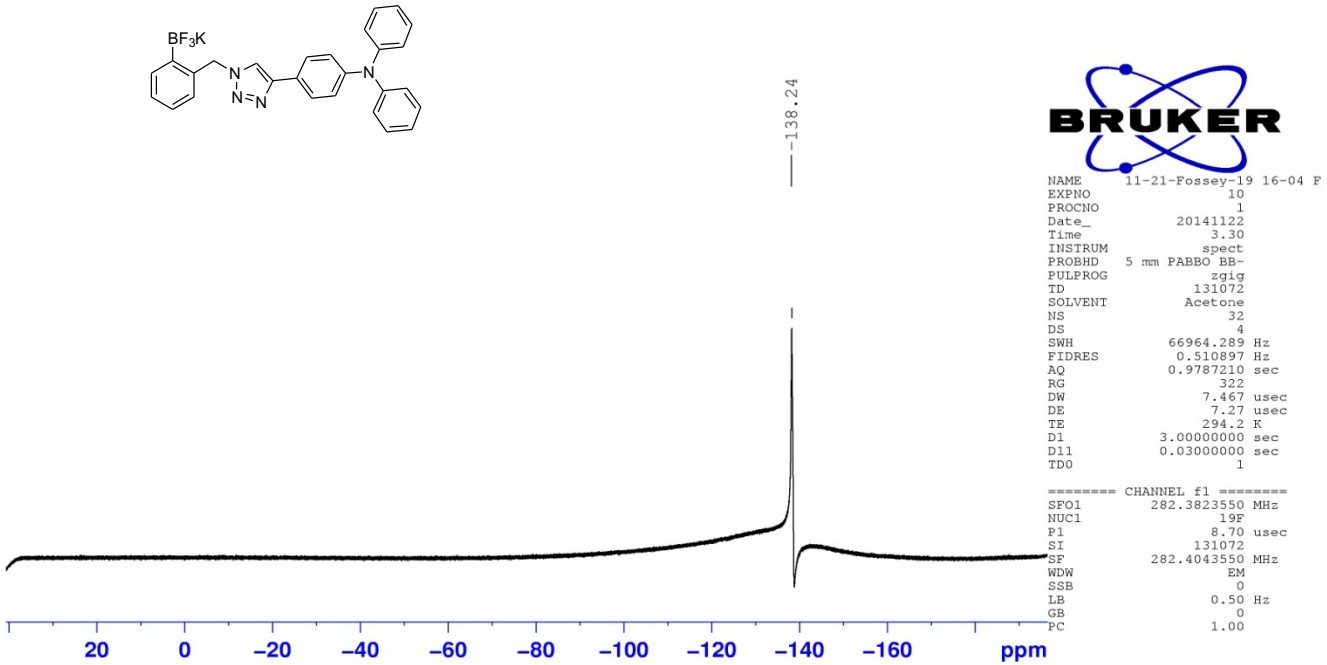
***N,N*-Diphenyl-4-(1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline, potassium salt ^{13}C NMR spectrum**



BRUKER

NAME	08-27-Fossey-1	
EXPNO	10	
PROCNO	1	
Date_	20130827	
Time	17.03	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	pendantns	
TD	65536	
SOLVENT	MeOD	
NS	512	
DS	4	
SWH	25252.525	Hz
FIDRES	0.385323	Hz
AQ	1.2976629	sec
RG	2050	
DW	19.800	usec
DE	6.50	usec
TE	294.8	K
CNST2	145.0000000	
CNST3	1.0000000	
CNST4	5.0000000	
D1	1.50000000	sec
D2	0.00172414	sec
D3	0.00431034	sec
D12	0.00002000	sec
D13	0.00000400	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	8.80	usec
P2	17.60	usec
PL1	-3.00	dB
PL1W	58.63890457	W
SFO1	100.6233333	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
P3	9.70	usec
P4	19.40	usec
PCPD2	90.00	usec
PL2	-4.00	dB
PL12	15.35	dB
PL2W	24.29185867	W
PL12W	0.28213742	W
SFO2	400.1316005	MHz
SI	65536	
SF	100.6127690	MHz
WDW	EM	
SSB	0	
LB	4.00	Hz
GB	0	
PC	1.00	

***N,N*-Diphenyl-4-(1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline, potassium salt ^{19}F NMR spectrum**

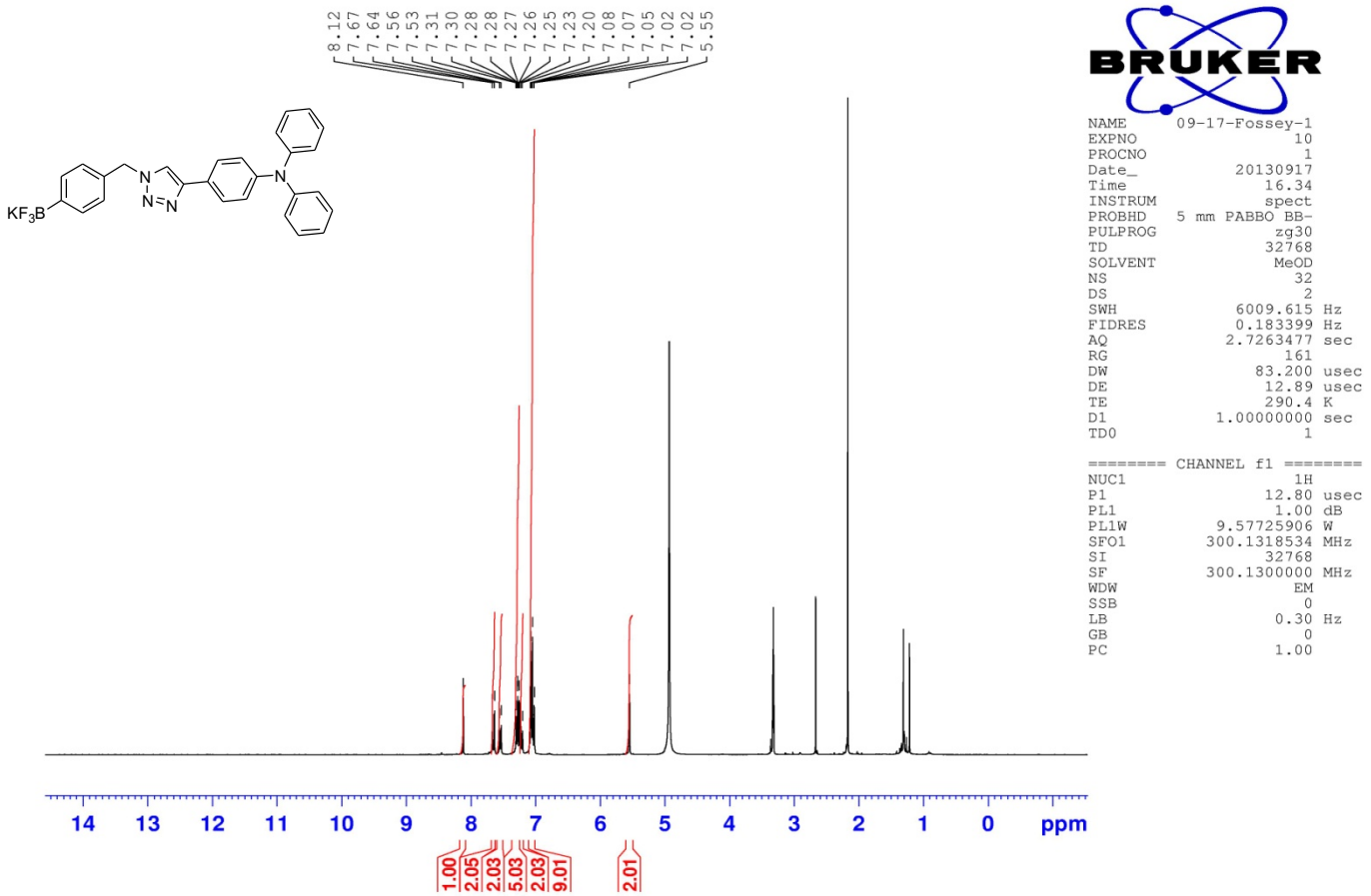


BRUKER

```
NAME          11-21-Fossey-19 16-04 F
EXPNO                    10
PROCNO                    1
Date_              20141122
Time                   3.30
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zgig
TD                   131072
SOLVENT             Acetone
NS                        32
DS                         4
SWH               66964.289 Hz
FIDRES             0.510897 Hz
AQ                0.9787210 sec
RG                       322
DW                   7.467 usec
DE                    7.27 usec
TE                   294.2 K
D1             3.00000000 sec
D11            0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          282.3823550 MHz
NUC1                   19F
P1                    8.70 usec
SI                  131072
SF            282.4043550 MHz
WDW                    EM
SSB                     0
LB                   0.50 Hz
GB                      0
PC                   1.00
```

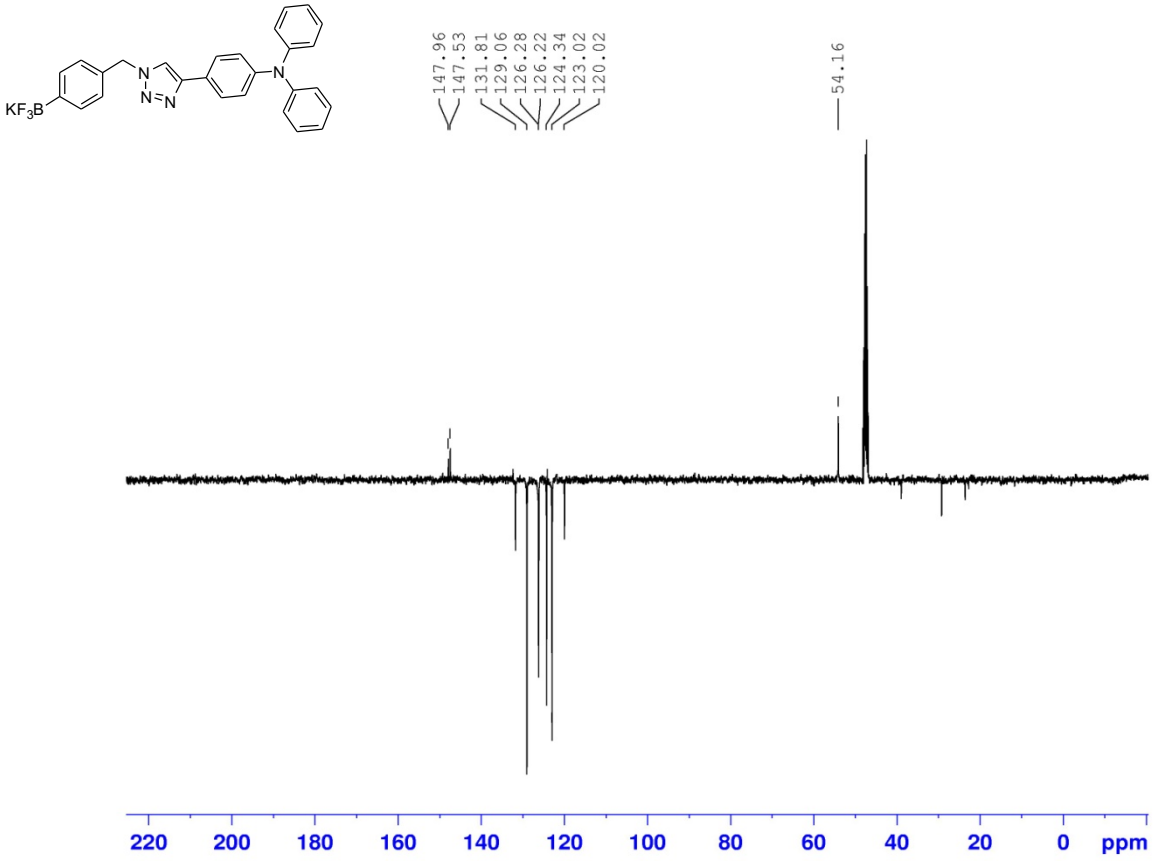
***N,N*-Diphenyl-4-(1-(4-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazol-4-yl)aniline, potassium salt ^{1}H NMR spectrum**



BRUKER

NAME	09-17-Fossey-1
EXPNO	10
PROCNO	1
Date_	20130917
Time	16.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	161
DW	83.200 usec
DE	12.89 usec
TE	290.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

_N,N_-Diphenyl-4-(1-(4-(trifluoro-l4-boranyl)benzyl)-1_H_-1,2,3-triazol-4-yl)aniline, potassium salt ^{13}C NMR spectrum

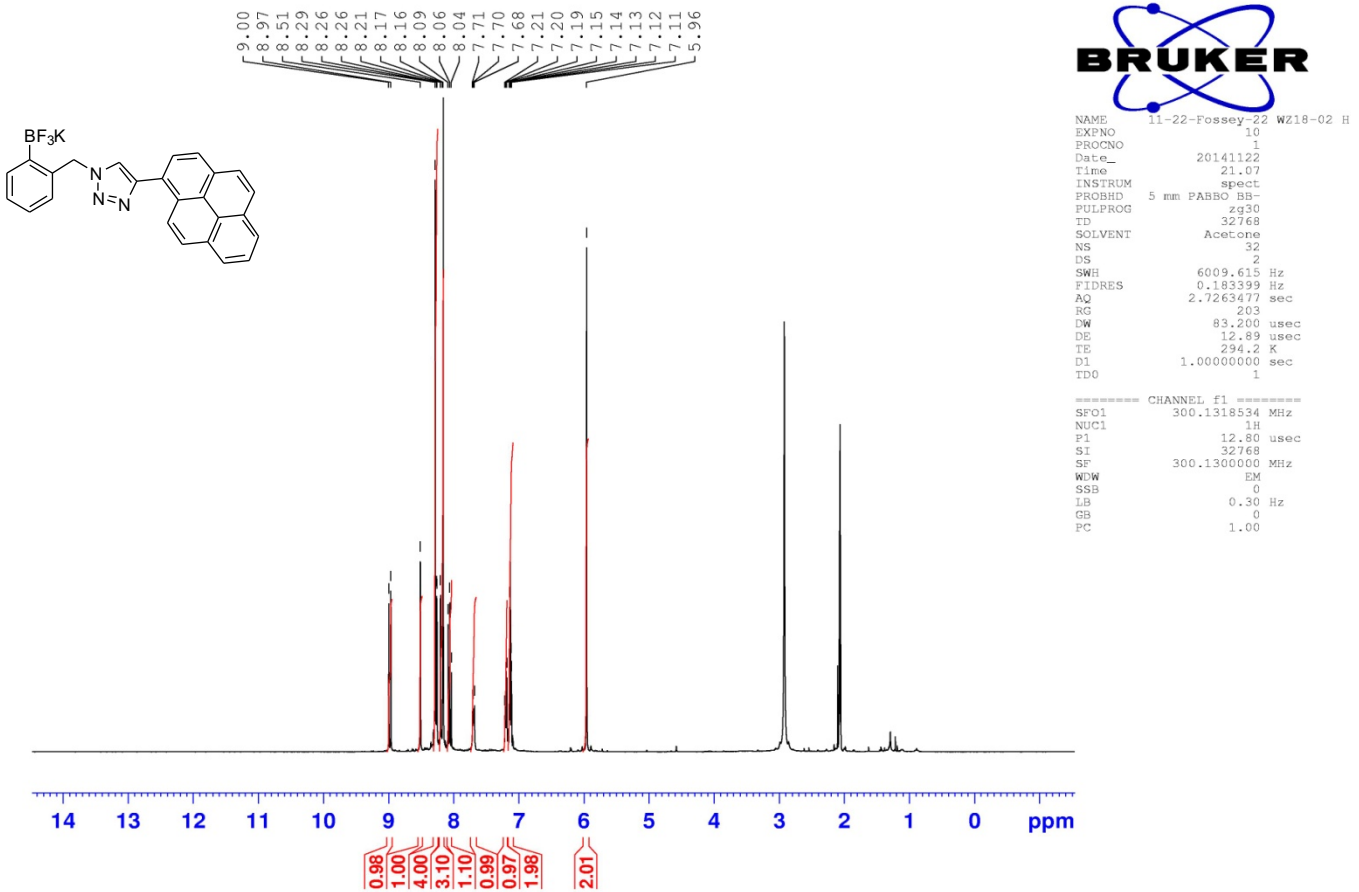



```
NAME          09-17-Fossey-1c
EXPNO                      10
PROCNO                      1
Date_                20130917
Time                    17.04
INSTRUM                  spect
PROBHD    5 mm PADUL 13C
PULPROG              pendantns
TD                       65536
SOLVENT                   MeOD
NS                         512
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       6.50 usec
TE                      294.7 K
CNST2               145.0000000
CNST3                 1.0000000
CNST4                 5.0000000
D1                   1.50000000 sec
D2                   0.00172414 sec
D3                   0.00431034 sec
D12                  0.00002000 sec
D13                  0.00000400 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       8.80 usec
P2                      17.60 usec
PL1                     -3.00 dB
PL1W                58.63890457 W
SFO1               100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
P3                       9.70 usec
P4                      19.40 usec
PCPD2                   90.00 usec
PL2                     -4.00 dB
PL12                    15.35 dB
PL2W                24.29185867 W
PL12W                0.28213742 W
SFO2               400.1316005 MHz
SI                        65536
SF                 100.6127690 MHz
WDW                          EM
SSB                           0
LB                       4.00 Hz
GB                            0
PC                         1.00
```

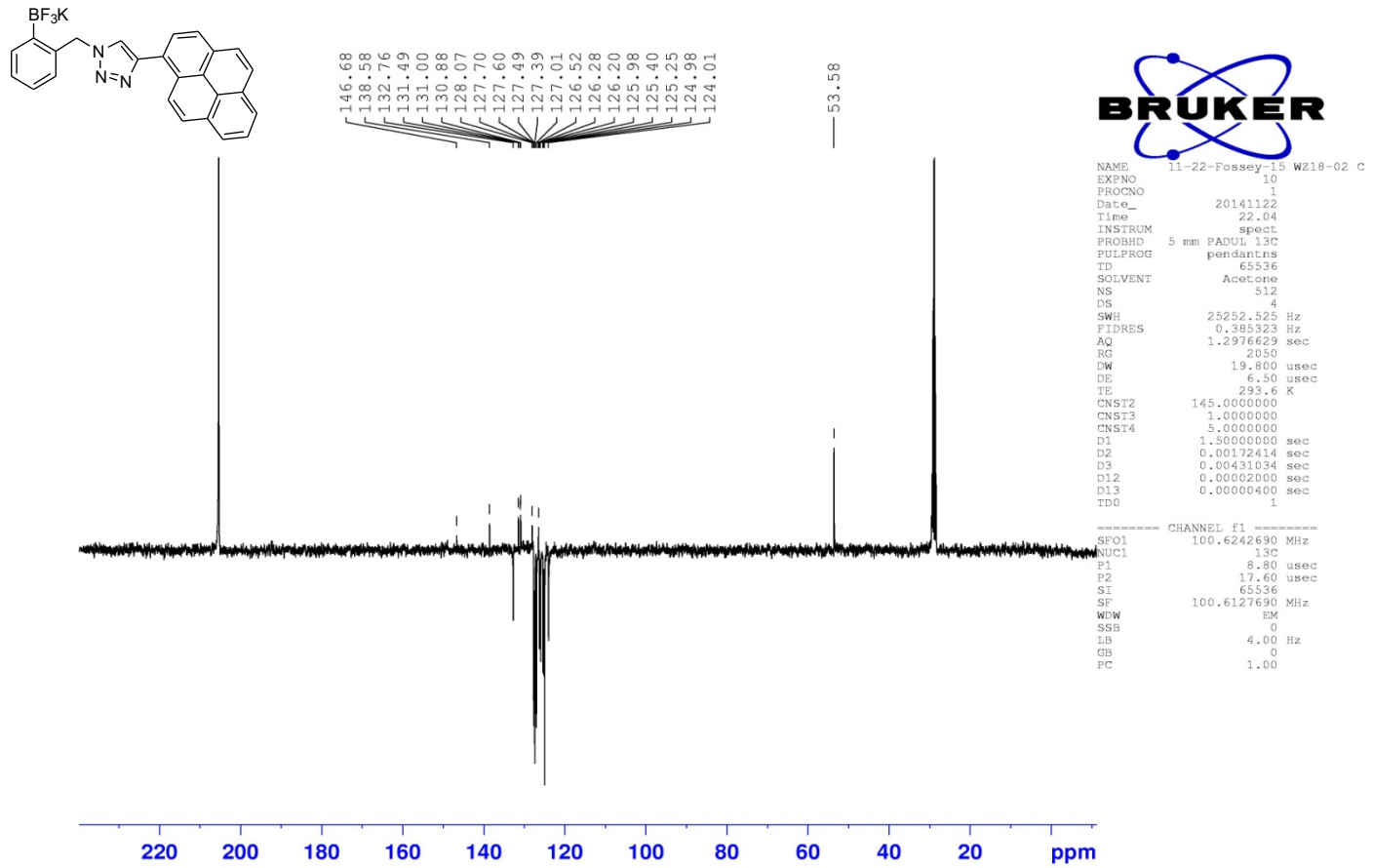
4-(Pyren-1-yl)-1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum



BRUKER

NAME	11-22-Fossey-22 WZ18-02 H
EXPNO	10
PROCNO	1
Date_	20141122
Time	21.07
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	Acetone
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	203
DW	83.200 usec
DE	12.89 usec
TE	294.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

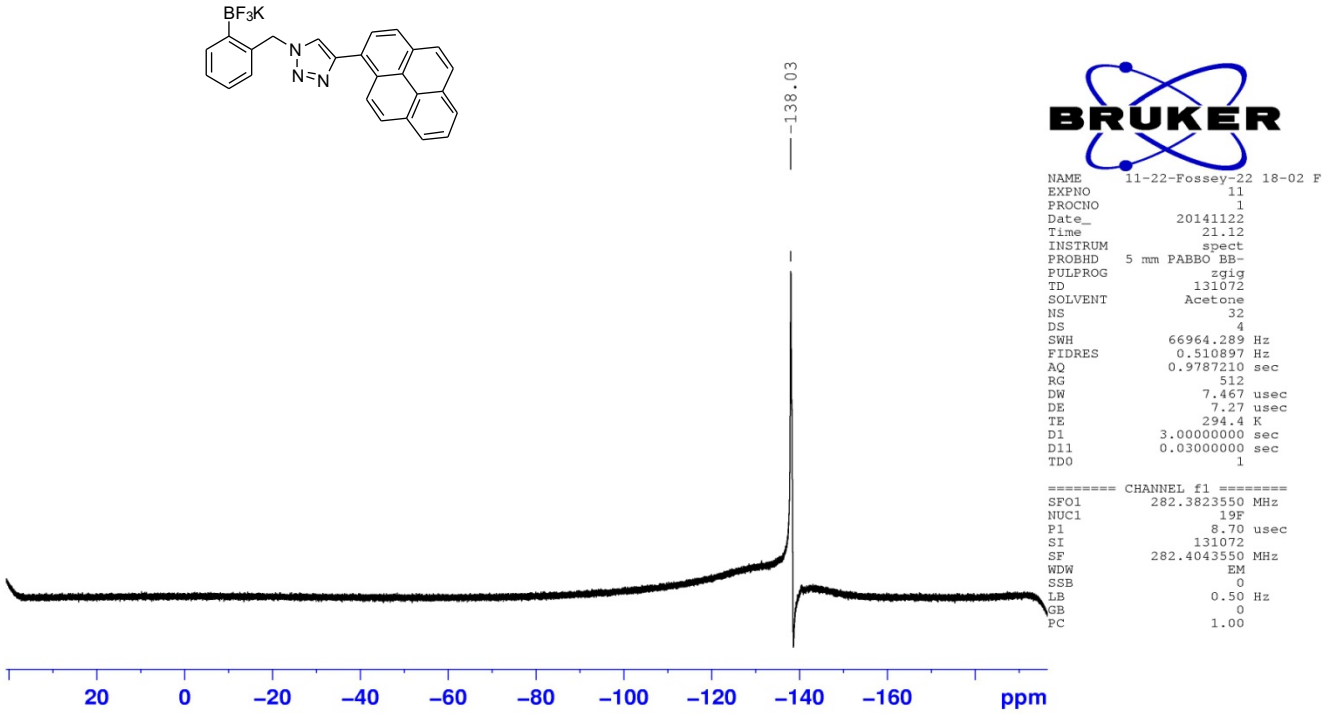
4-(Pyren-1-yl)-1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{13}C NMR spectrum



BRUKER

NAME	11-22-Fossey-15 WZ18-02 C
EXPNO	10
PROCNO	1
Date_	20141122
Time	22.04
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	Acetone
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.6 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

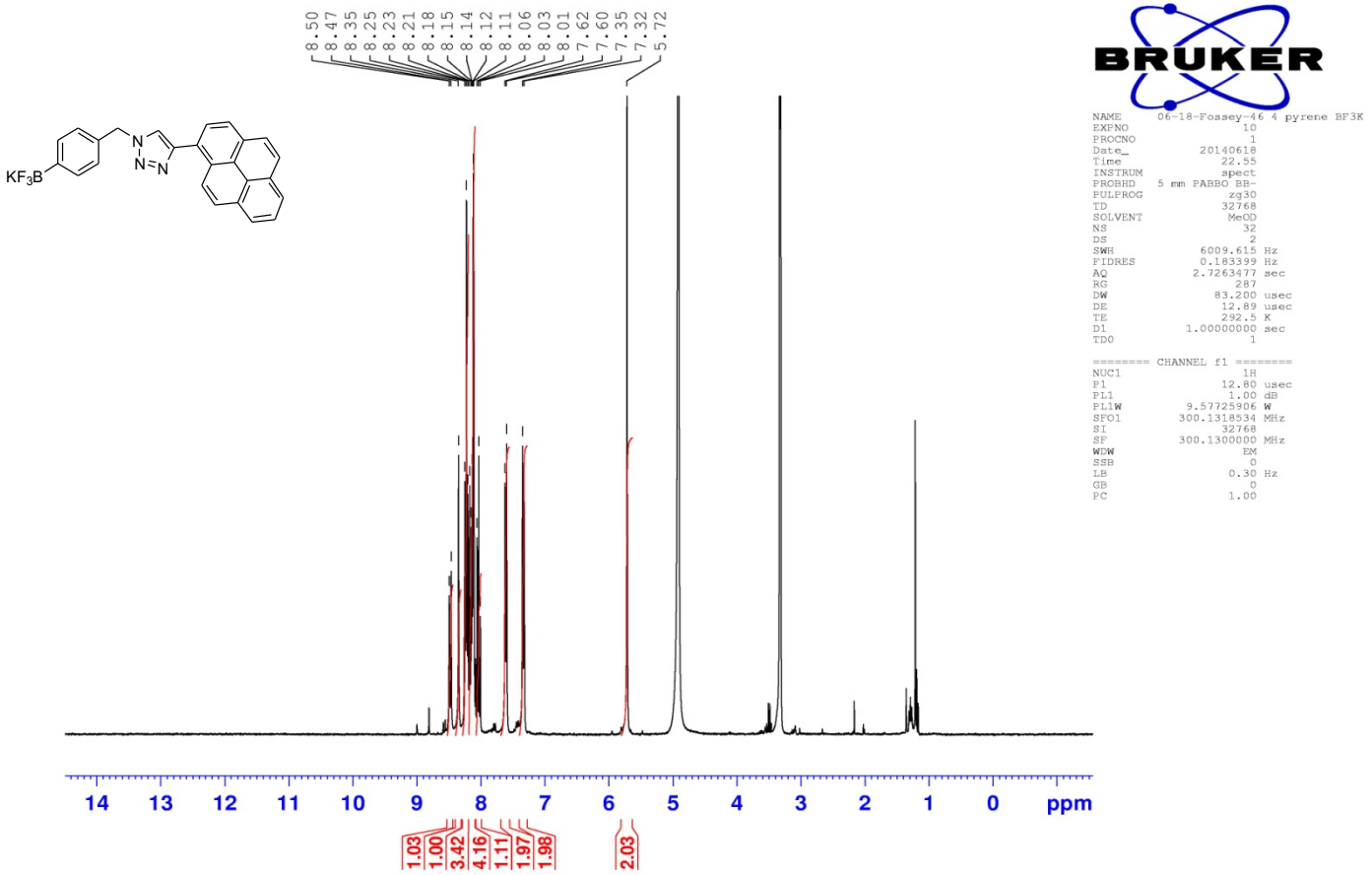
4-(Pyren-1-yl)-1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{19}F NMR spectrum




```
NAME          11-22-Fossey-22 18-02 F
EXPNO                    11
PROCNO                    1
Date_              20141122
Time                 21.12
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                zgig
TD                   131072
SOLVENT             Acetone
NS                       32
DS                        4
SWH               66964.289 Hz
FIDRES             0.510897 Hz
AQ                 0.9787210 sec
RG                       512
DW                     7.467 usec
DE                     7.27 usec
TE                     294.4 K
D1               3.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            282.3823550 MHz
NUC1                    19F
P1                     8.70 usec
SI                   131072
SF              282.4043550 MHz
WDW                      EM
SSB                       0
LB                     0.50 Hz
GB                        0
PC                     1.00
```

4-(Pyren-1-yl)-1-(4-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{1}H NMR spectrum

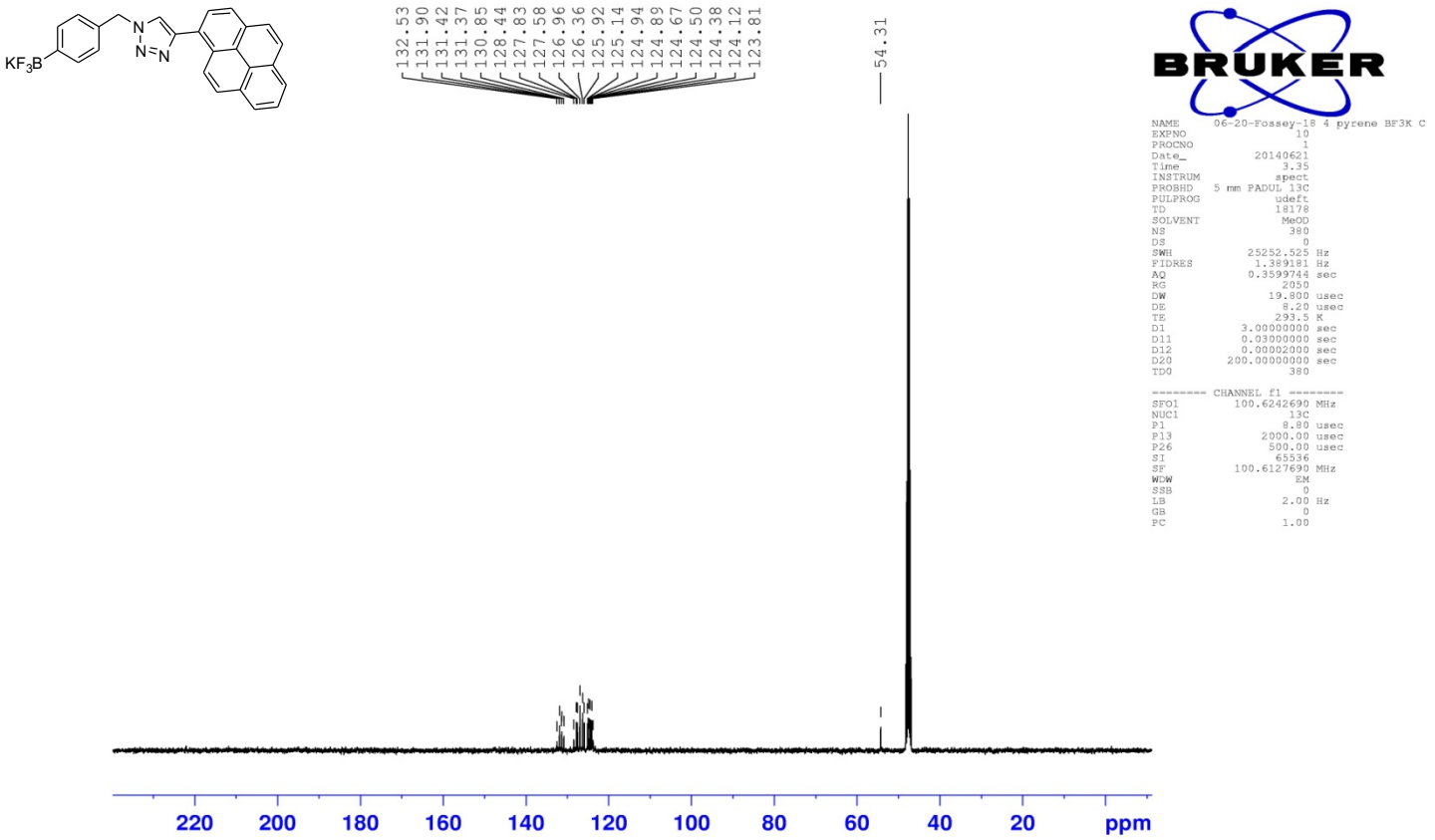


BRUKER

```
NAME          06-18-Fossey-46 4 pyrene BF3K
EXPNO                    10
PROCNO                    1
Date_              20140618
Time                  22.55
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT                MeOD
NS                       32
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7263477 sec
RG                      287
DW                   83.200 usec
DE                   12.89 usec
TE                   292.5 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    12.80 usec
PL1                    1.00 dB
PL1W           9.57725906 W
SFO1          300.1318534 MHz
SI                    32768
SF            300.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

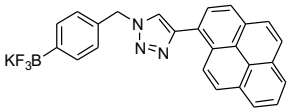
4-(Pyren-1-yl)-1-(4-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{13}C NMR spectrum

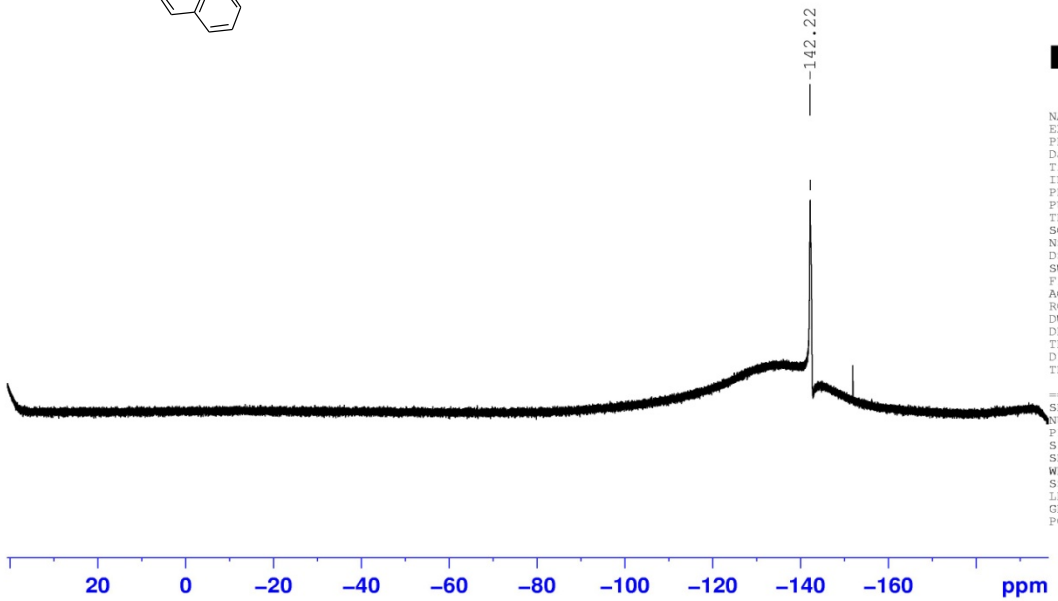


BRUKER

NAME	06-20-Fossey-18 4 pyrene BF3K C
EXPNO	10
PROCNO	1
Date_	20140621
Time	3.35
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	udeft
TD	18178
SOLVENT	MeOD
NS	380
DS	0
SWH	25252.525 Hz
FIDRES	1.389181 Hz
AQ	0.3599744 sec
RG	2050
DW	19.800 usec
DE	8.20 usec
TE	293.5 K
D1	3.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
D20	200.00000000 sec
TD0	380
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P13	2000.00 usec
P26	500.00 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.00

4-(Pyren-1-yl)-1-(4-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazole, potassium salt ^{19}F NMR spectrum



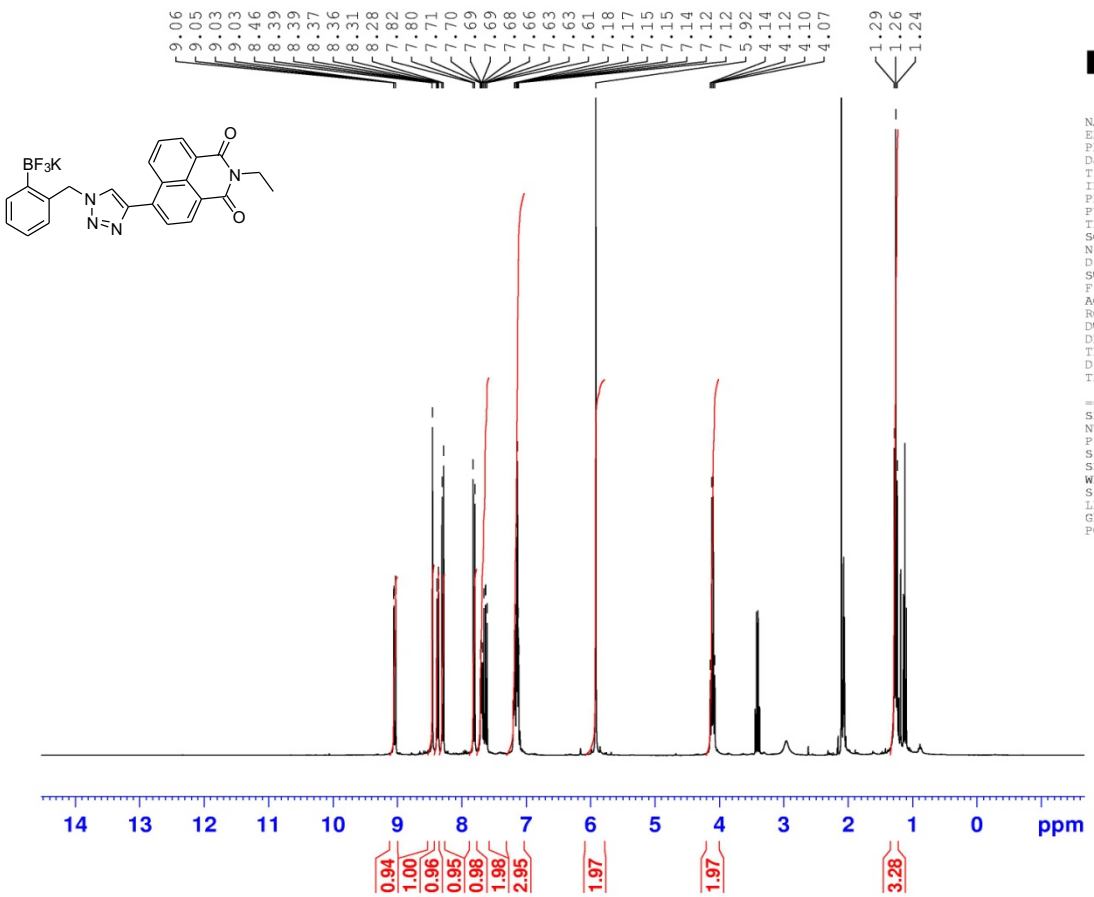


BRUKER

```
NAME          11-22-Fossey-27 WZ19-02 H
EXPNO                      11
PROCNO                      1
Date_                20141122
Time                    23.07
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                    zg
TD                     131072
SOLVENT                 CD3CN
NS                         32
DS                          4
SWH                66964.289 Hz
FIDRES             0.510897 Hz
AQ                 0.9787210 sec
RG                        322
DW                    7.467 usec
DE                     7.27 usec
TE                    294.2 K
D1                 3.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             282.3823550 MHz
NUC1                    19F
P1                     8.70 usec
SI                     131072
SF               282.4043550 MHz
WDW                       EM
SSB                        0
LB                      0.50 Hz
GB                         0
PC                      1.00
```

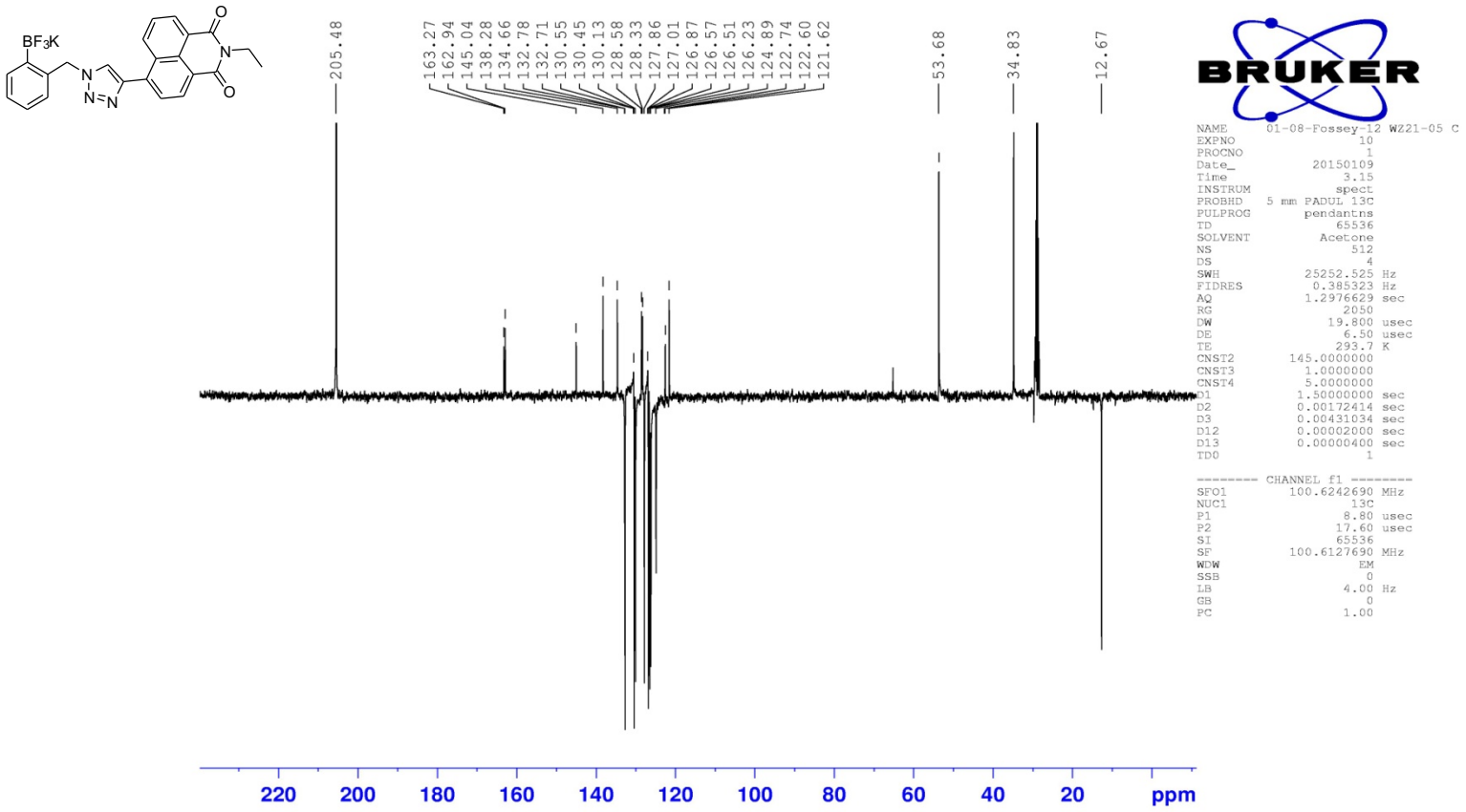
2-Ethyl-6-(1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione, potassium salt ^{1}H NMR spectrum



BRUKER

NAME	01-08-Fossey-40 WZ21-05 H
EXPNO	10
PROCNO	1
Date_	20150109
Time	1.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	Acetone
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	128
DW	83.200 usec
DE	12.89 usec
TE	294.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

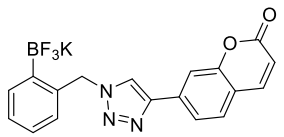
2-Ethyl-6-(1-(2-(trifluoro-l4-boranyl)benzyl)-1*H*-1,2,3-triazol-4-yl)-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione, potassium salt ^{13}C NMR spectrum

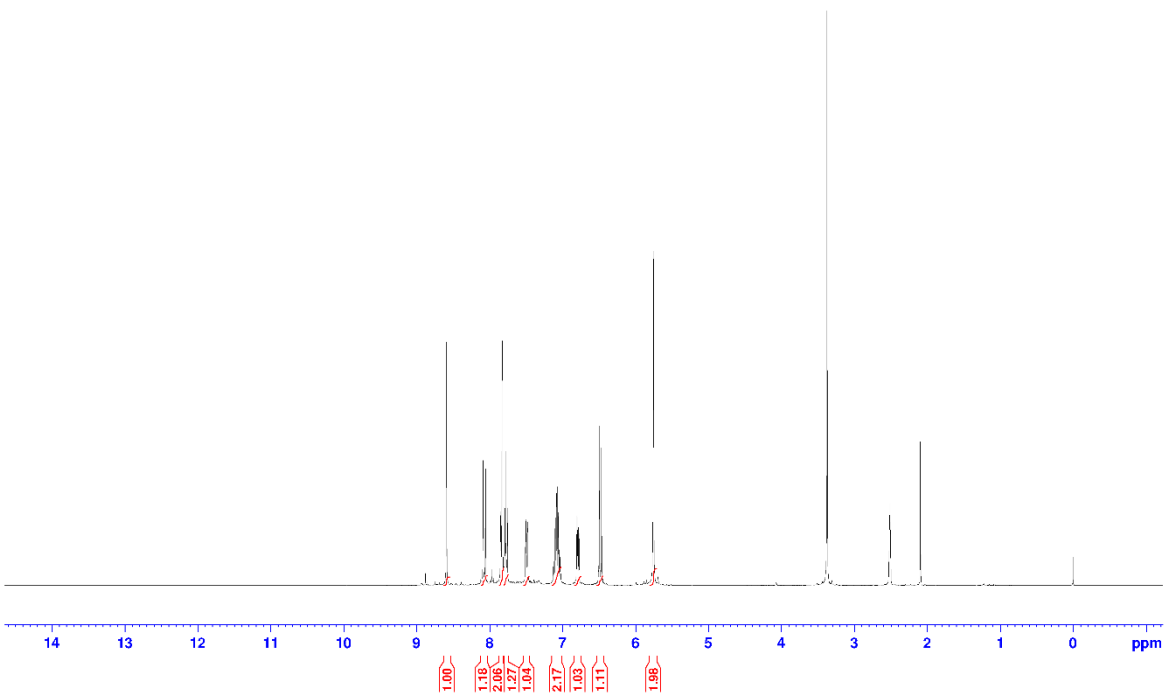


BRUKER

NAME	01-08-Fossey-12 WZ21-05 C
EXPNO	10
PROCNO	1
Date_	20150109
Time	3.15
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	Acetone
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.7 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

7-(1-(2-(Trifluoro- λ^4-boranyl)benzyl)-1H-1,2,3-triazol-4-yl)-2*H*-chromen-2-one, potassium salt, ^{1}H NMR spectrum



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Current Data Parameters
NAME      2016-Apr-15-Fossey-
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_              20160415
Time                  14.48
INSTRM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT                DMSO
NS                       32
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                       203
DW                  83.200 use
DE                  12.89 use
TE                    292.5 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             300.1318534 MHz
NUC1                       1H
P1                    12.80 use
PLW1             9.57730007 W

F2 - Processing parameters
SI                        32768
SF               300.1299998 MHz
WDW                        EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```
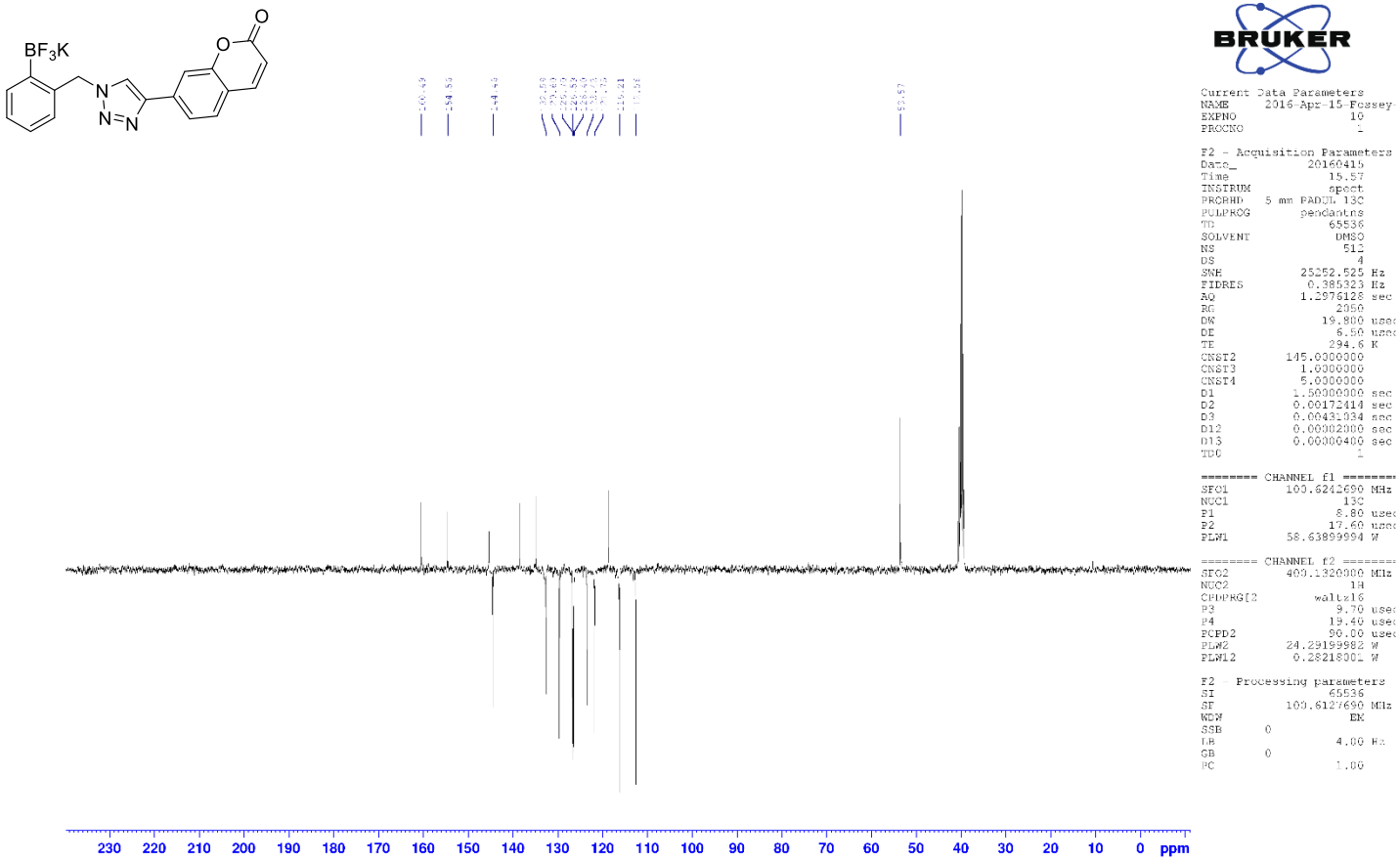
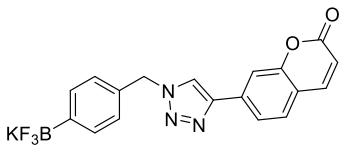

7-(1-(2-(Trifluoro- λ^4-boranyl)benzyl)-1H-1,2,3-triazol-4-yl)-2*H*-chromen-2-one, potassium salt, ^{13}C NMR spectrum

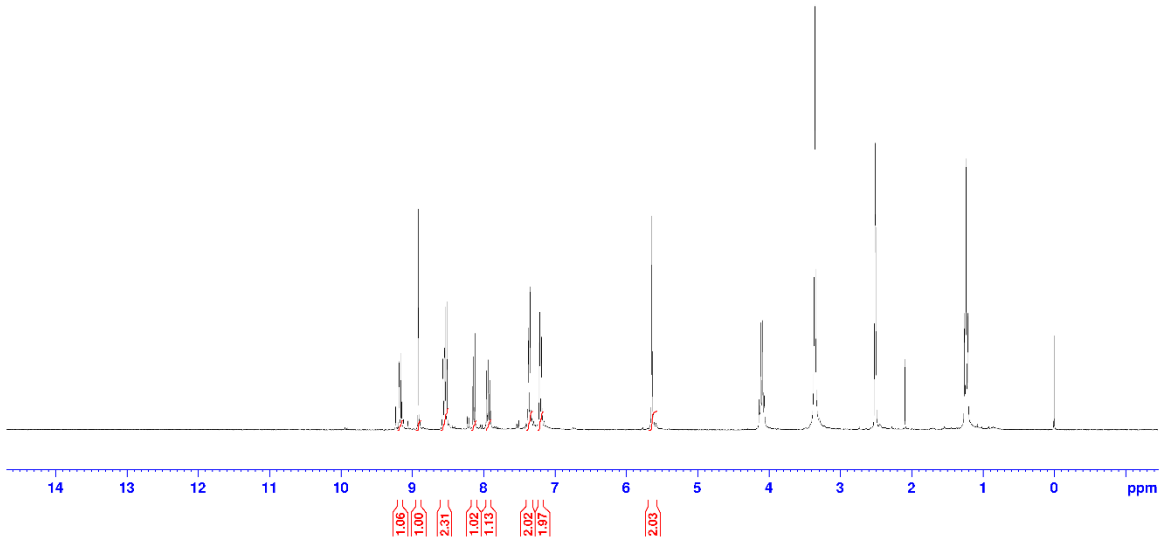


BRUKER

```
Current Data Parameters
NAME      2016-Apr-15-Fossey
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160415
Time                  15.57
INSTRUM               spect
PROBHD   5 mm PADUL 13C
PULPROG          pendantns
TD                   65536
SOLVENT               DMSO
NS                      512
DS                        4
SWH              25252.525 Hz
FIDRES           0.385323 Hz
AQ               1.2976128 sec
RG                     2050
DW                19.800 use
DE                 6.50 use
TE                294.6 K
CNST2          145.0000000
CNST3            1.0000000
CNST4            5.0000000
D1               1.50000000 sec
D2               0.00172414 sec
D3               0.00431034 sec
D12              0.00002000 sec
D13              0.00300400 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6242690 MHz
NUC1                    13C
P1                    8.80 use
P2                   17.60 use
PLW1           58.63899994 W

======== CHANNEL f2 ========
SFO2           400.1320000 MHz
NUC2                    1H
CPDPRG[2           waltz16
P3                    9.70 use
P4                   19.40 use
PCPD2                90.00 use
PLW2           24.29199982 W
PLW12           0.28218001 W

F2 - Processing parameters
SI                    65536
SF           100.6127690 MHz
WDW                     EM
SSB          0
LB                    4.00 Hz
GB           0
PC                    1.00
```

7-(1-(4-(Trifluoro- λ^4-boranyl)benzyl)-1H-1,2,3-triazol-4-yl)-2*H*-chromen-2-one, potassium salt, ^{1}H NMR spectrum

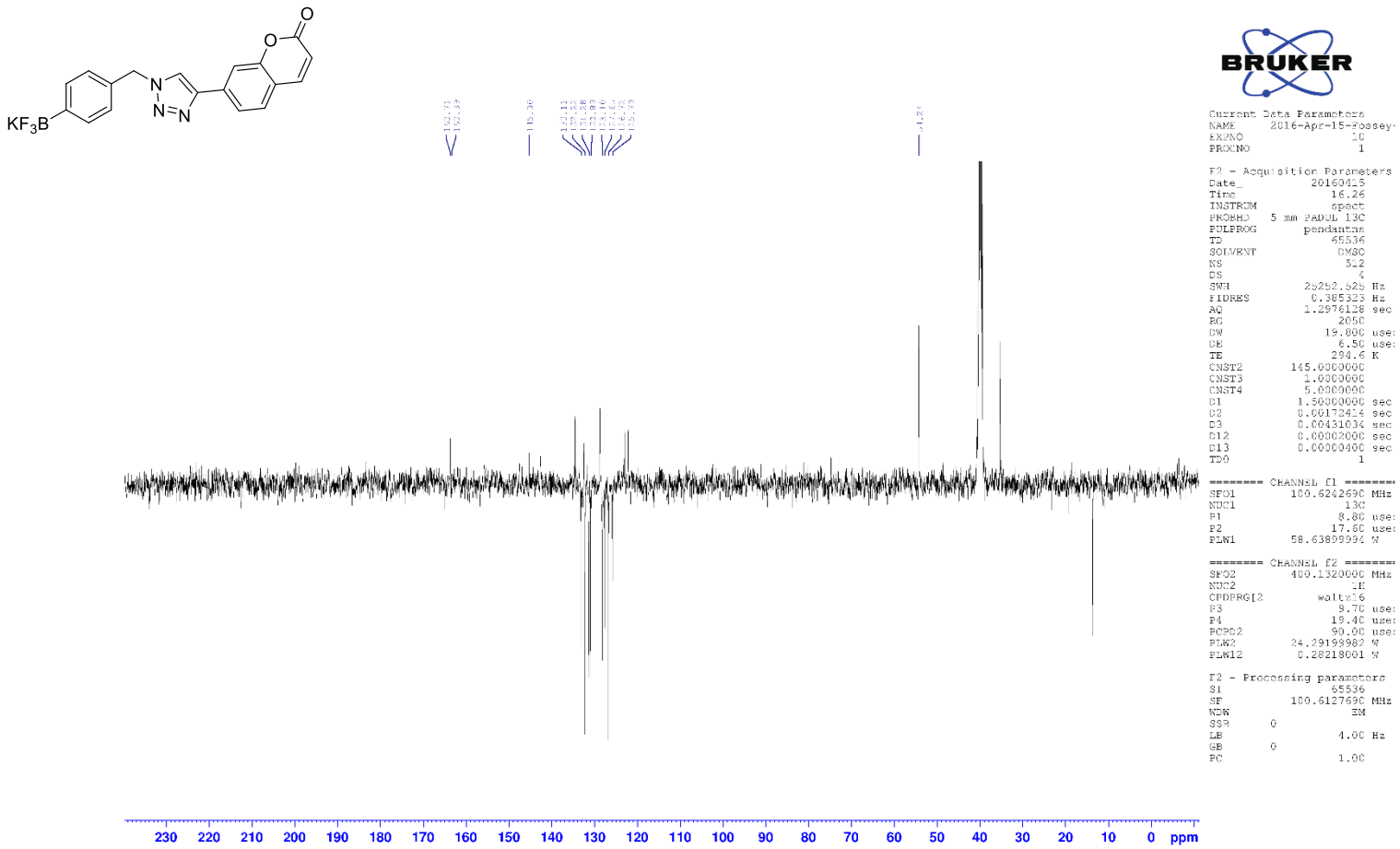




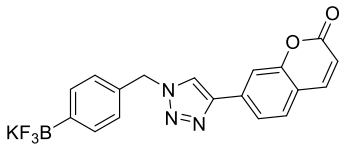
BRUKER

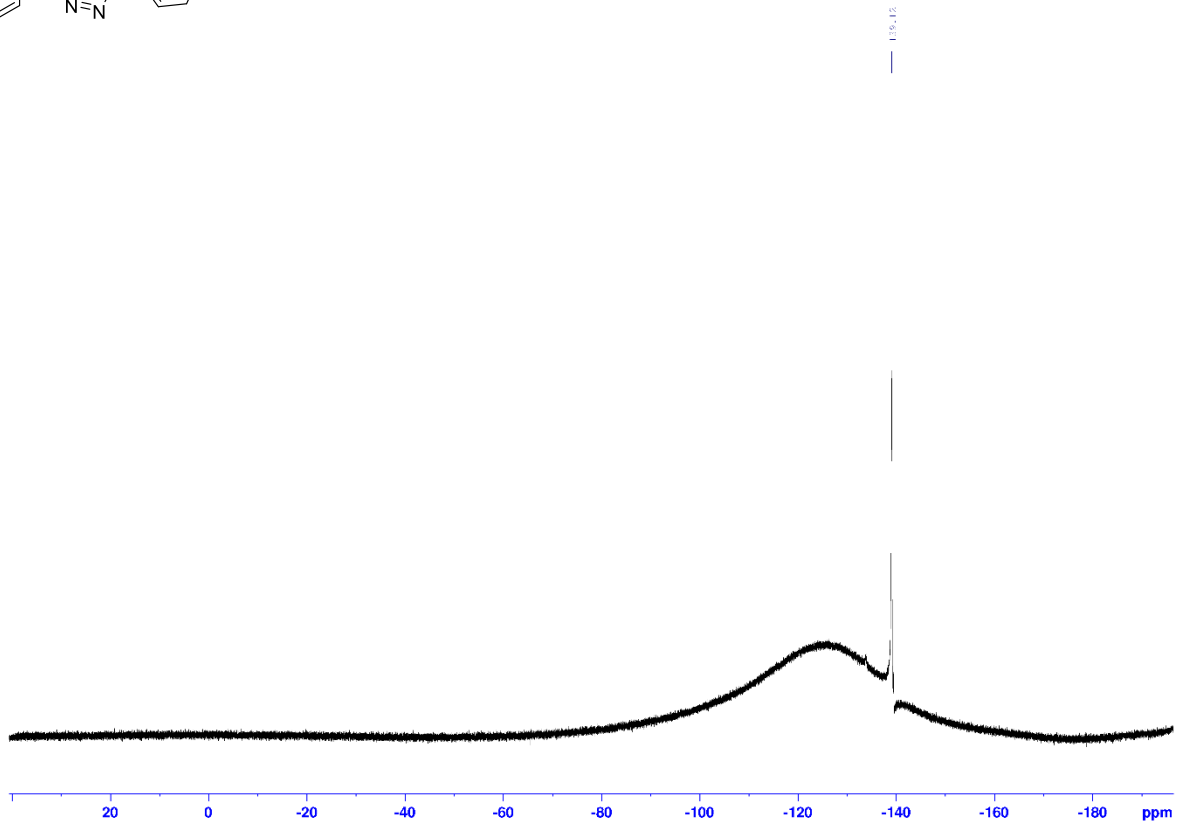
```
Current Data Parameters
NAME      2016-Apr-15-Tossy-
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20160415
Time                  14.59
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT                DMSO
NS                       32
DS                        2
SWH                6009.615 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                       575
DW                  83.200 usec
DE                  12.89 usec
TE                  292.5 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            300.1318534 MHz
NUC1                      1H
P1                   12.80 usec
PLW1             9.57730007 W

F2 - Processing parameters
SI                    32768
SF              300.1300010 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

7-(1-(4-(Trifluoro- λ^4-boranyl)benzyl)-1H-1,2,3-triazol-4-yl)-2*H*-chromen-2-one, potassium salt, ^{13}C NMR spectrum



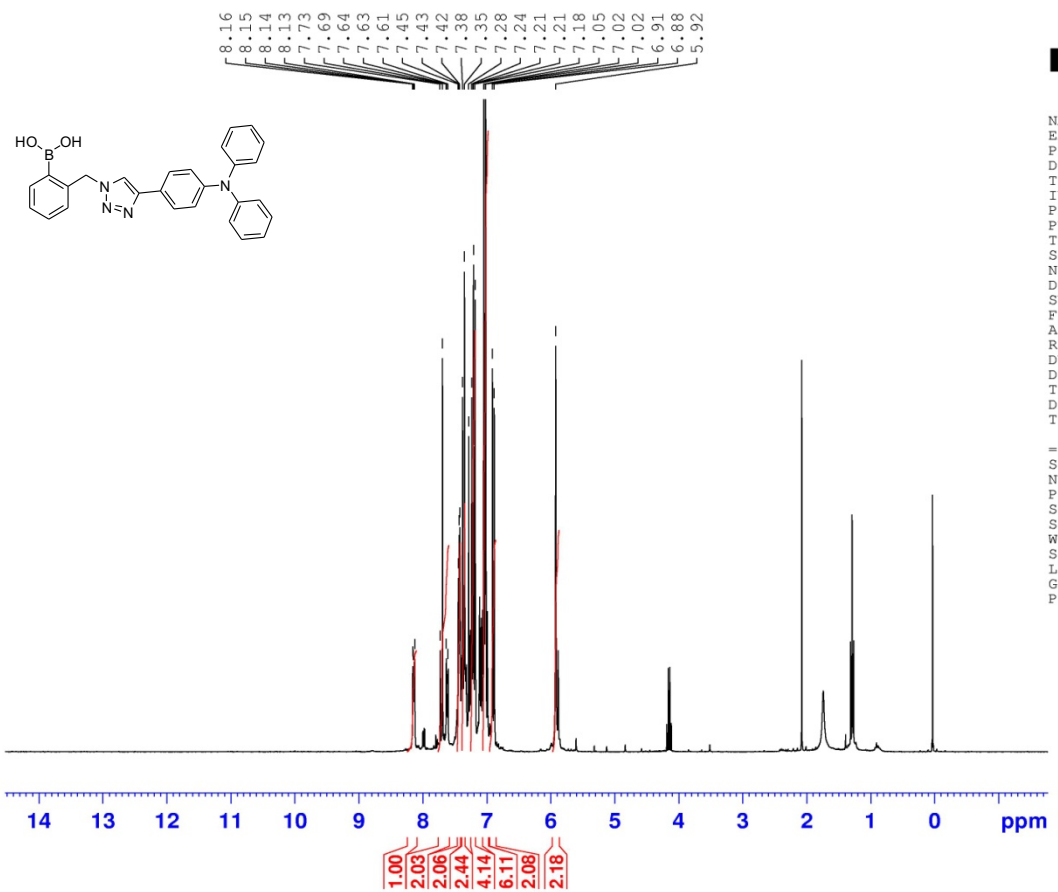
7-(1-(4-(Trifluoro- λ^4-boranyl)benzyl)-1H-1,2,3-triazol-4-yl)-2*H*-chromen-2-one, potassium salt, ^{19}F NMR spectrum






```
Current Data Parameters
NAME      2016-Apr-15-Fossey-
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_              20160415
Time                  15.04
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg1g
TD                   131072
SOLVENT                DMSO
NS                       32
DS                        4
SWH                66964.289 Hz
FIDRES              0.510897 Hz
AQ                  0.9786710 sec
RG                        406
DW                    7.467 use
DE                    7.27 use
TE                    292.6 K
D1                 3.00000000 sec
D11                0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             282.3823550 MHz
NUC1                      19F
P1                       8.70 use
PLW1            30.58203073 W

======== CHANNEL f2 ========
SFO2             300.1312003 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                   90.00 use
PLW2             9.57733007 W
PLW12            0.19372000 W

F2 - Processing parameters
SI                      131072
SF                282.4043950 MHz
WDW                        EM
SSB          0
LB                        0.50 Hz
GB           0
PC                        1.00
```

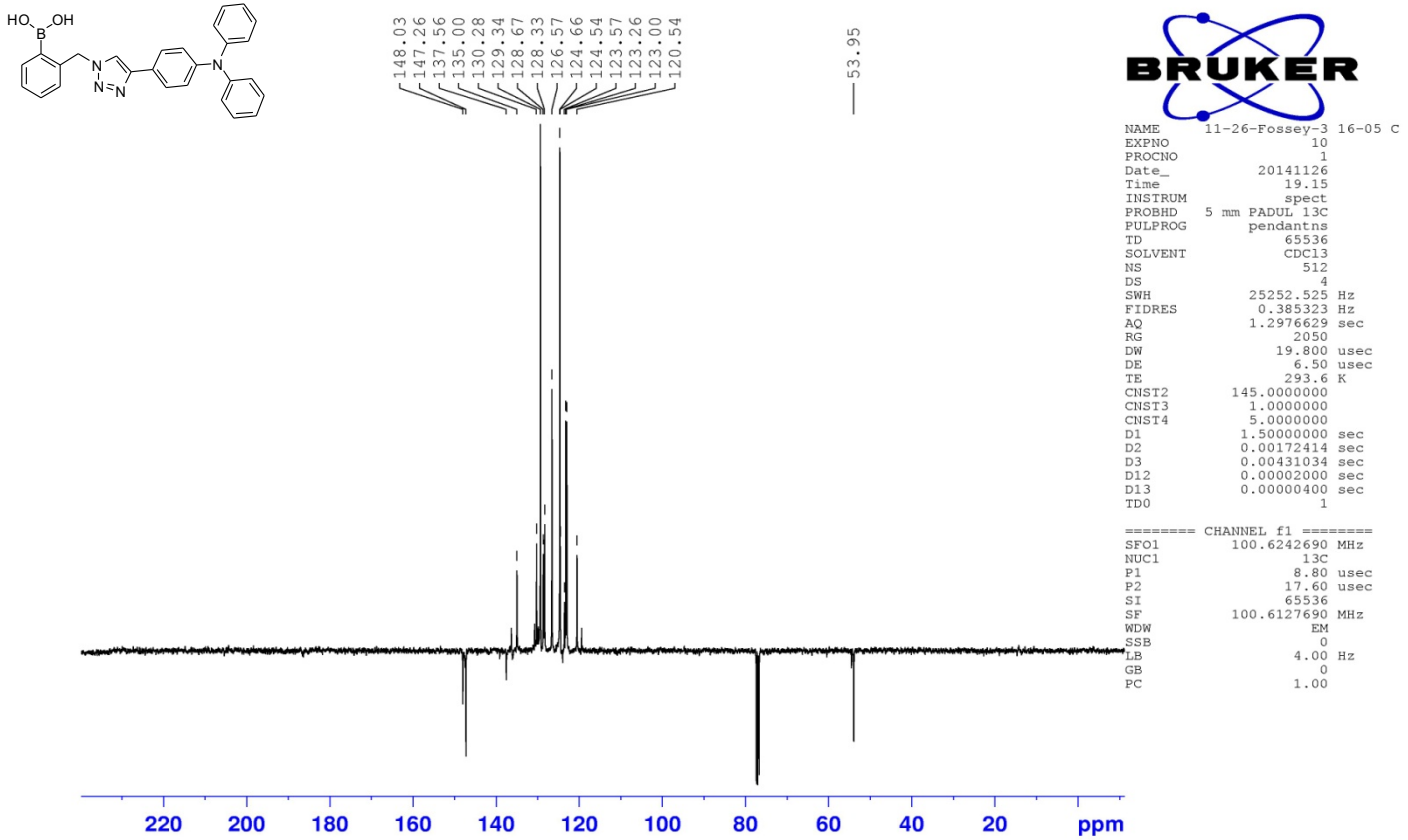
(2-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14a) ^{1}H NMR spectrum



BRUKER

NAME	11-26-Fossey-5 16-05
EXPNO	10
PROCNO	1
Date_	20141126
Time	17.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	181
DW	83.200 usec
DE	12.89 usec
TE	294.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

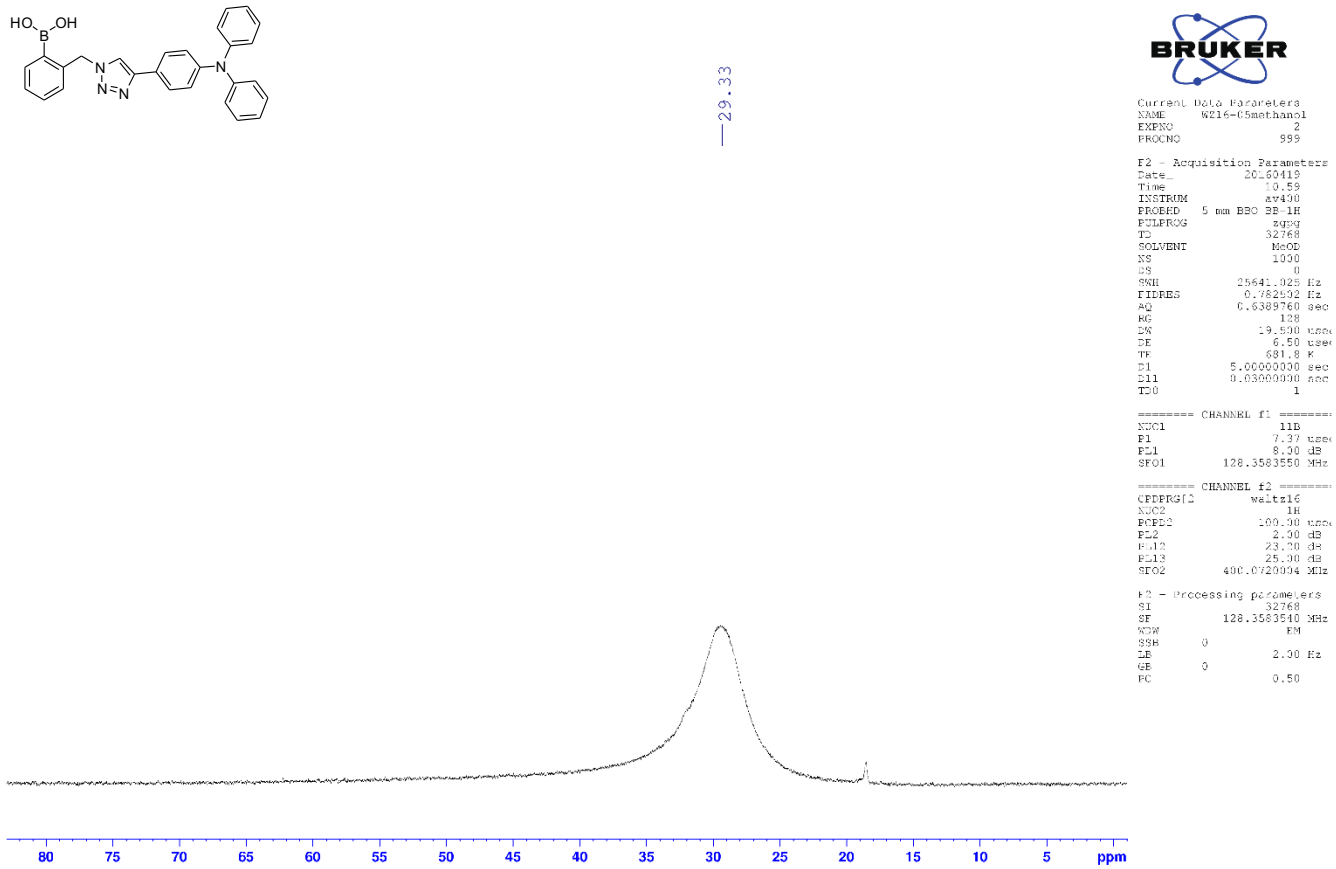
(2-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14a) ^{13}C NMR spectrum



BRUKER

```
NAME          11-26-Fossey-3 16-05 C
EXPNO                      10
PROCNO                      1
Date_                20141126
Time                    19.15
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG              pendantns
TD                       65536
SOLVENT                  CDCl3
NS                         512
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       6.50 usec
TE                      293.6 K
CNST2               145.0000000
CNST3                 1.0000000
CNST4                 5.0000000
D1                    1.50000000 sec
D2                    0.00172414 sec
D3                    0.00431034 sec
D12                   0.00002000 sec
D13                   0.00000400 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6242690 MHz
NUC1                       13C
P1                        8.80 usec
P2                       17.60 usec
SI                       65536
SF                  100.6127690 MHz
WDW                         EM
SSB                          0
LB                        4.00 Hz
GB                           0
PC                        1.00
```

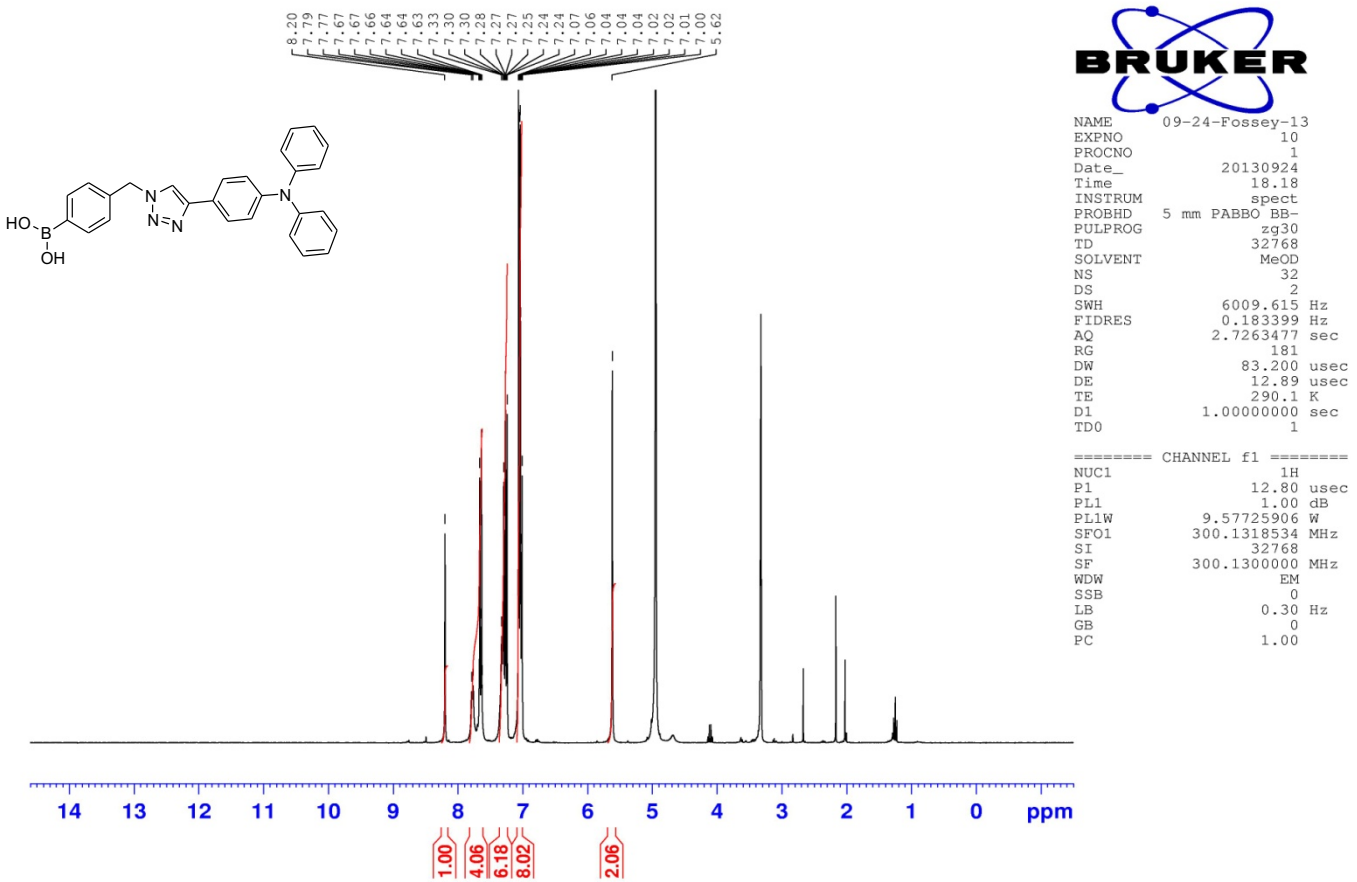
(2-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14a) ^{11}B NMR spectrum



BRUKER

```
Current Data Parameters
NAME      W216-05methanol
EXPNO                   2
PROCNO                999

F2 - Acquisition Parameters
Date_          20160419
Time               10.59
INSTRUM           av400
PROBHD  5 mm BBO BB-1H
PULPROG           zgpg
TD                32768
SOLVENT            MeOD
NS                 1000
DS                    0
SWH           25641.025 Hz
FIDRES        0.782502 Hz
AQ            0.6389760 sec
RG                  128
DW               19.500 usec
DE                6.50 usec
TE               281.8 K
D1            5.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 11B
P1                  7.37 usec
PL1                 8.00 dB
SFO1         128.3583560 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                 2.00 dB
PL12               23.70 dB
PL13               25.00 dB
SFO2         400.0720094 MHz

F2 - Processing parameters
SI                  32768
SF           128.3583540 MHz
WDW                    EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  0.50
```

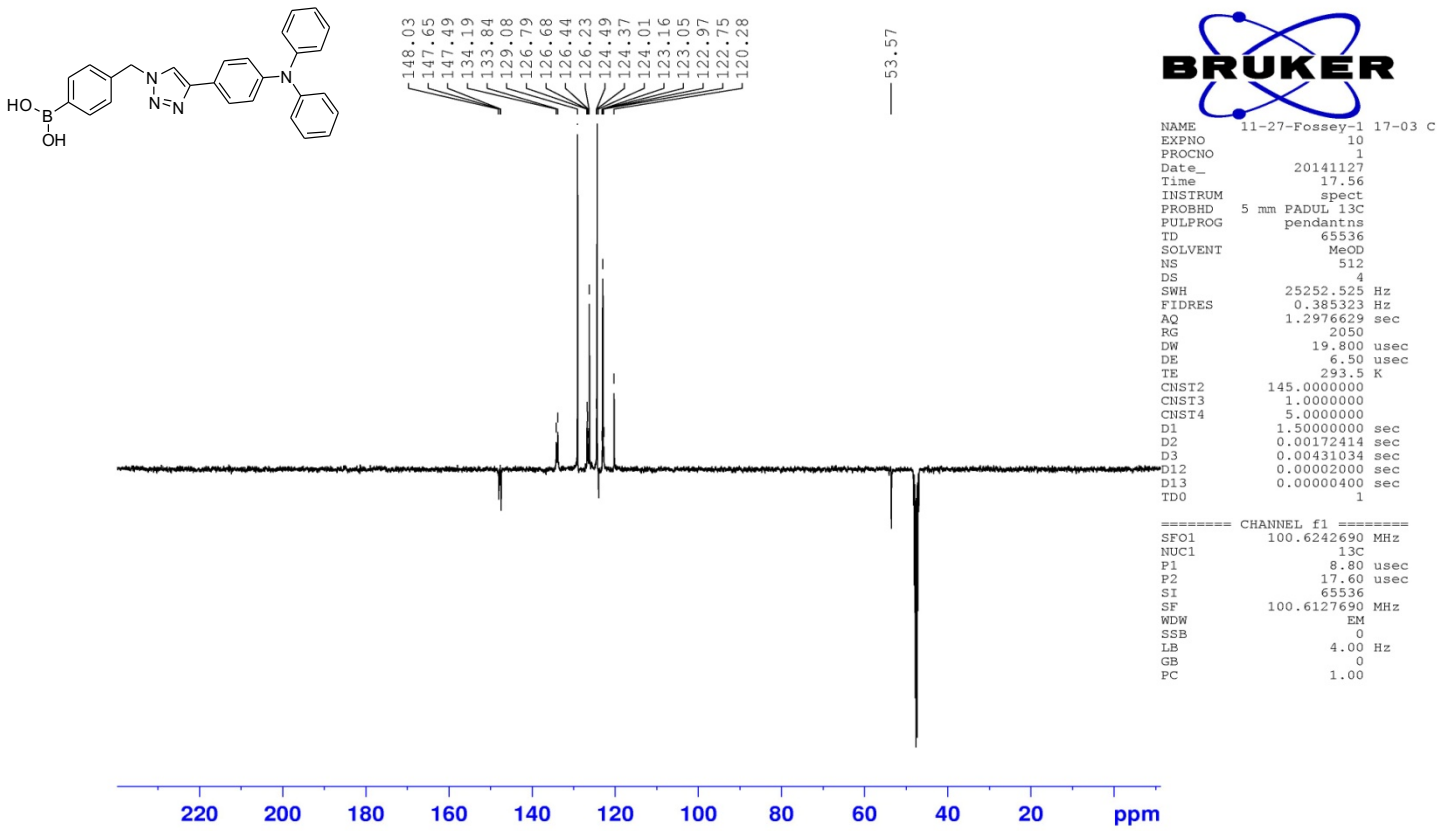
(4-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14b) ^{1}H NMR spectrum



BRUKER

NAME	09-24-Fossey-13
EXPNO	10
PROCNO	1
Date_	20130924
Time	18.18
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	181
DW	83.200 usec
DE	12.89 usec
TE	290.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

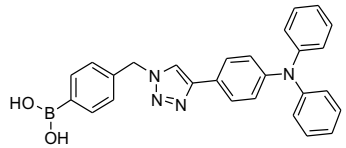
(4-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14b) ^{13}C NMR spectrum

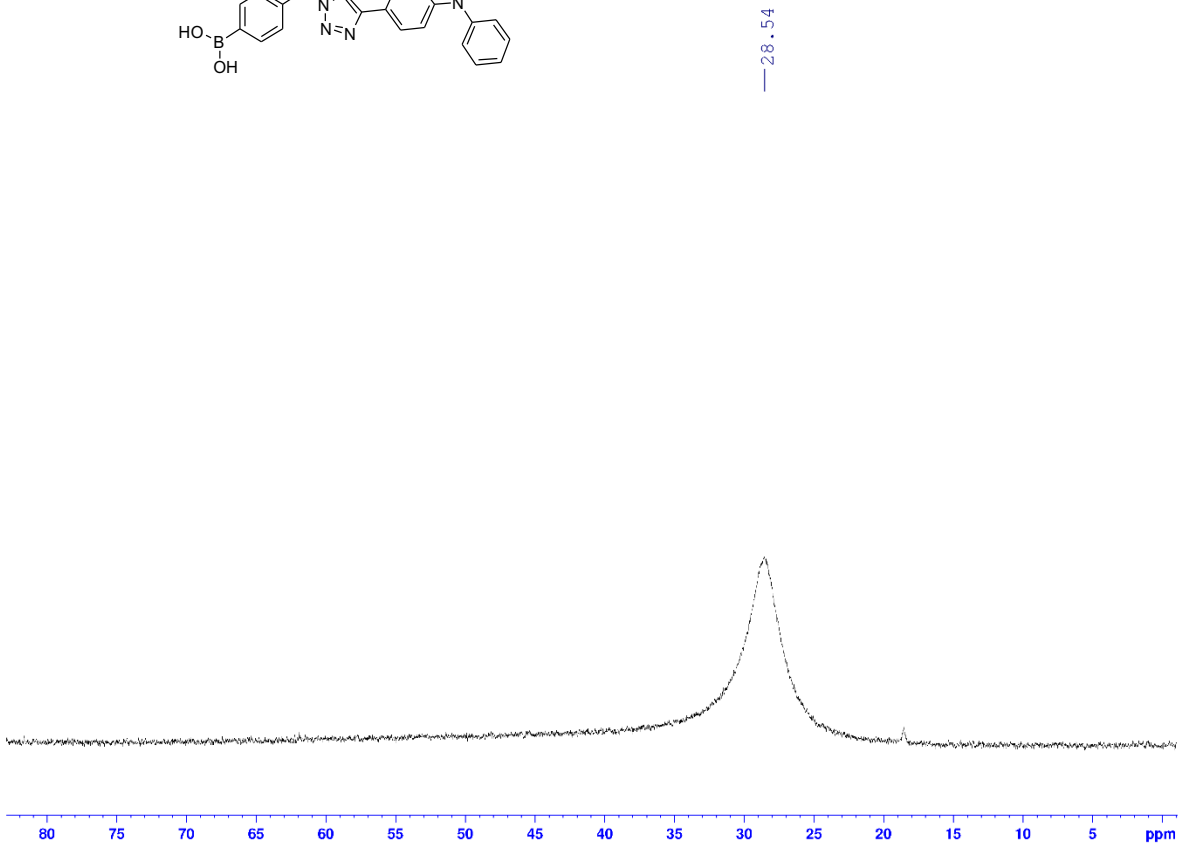


BRUKER

NAME	11-27-Fossey-1 17-03 C
EXPNO	10
PROCNO	1
Date_	20141127
Time	17.56
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	MeOD
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.5 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

(4-((4-(4-(Diphenylamino)phenyl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (14b) ^{11}B NMR spectrum

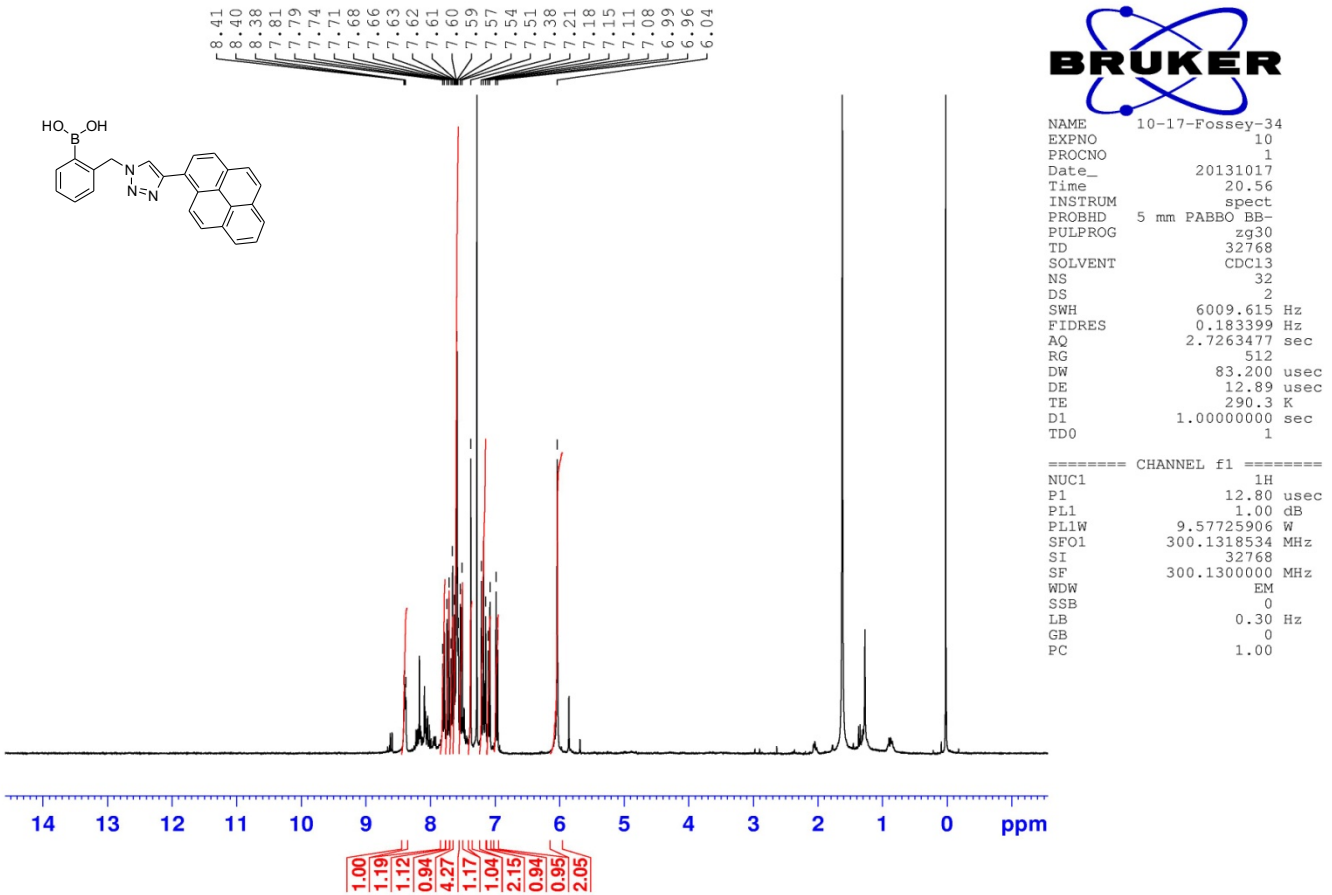




BRUKER

```
Current Data Parameters
NAME      WX17-03methanol
EXPNO                   2
PROCNO                999

F2 - Acquisition Parameters
Date_           20150219
Time               9.23
INSTRUM           av400
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg
TD                32768
SOLVENT             MeOD
NS                  1000
DS                     0
SWH            25641.025 Hz
FIDRES          0.782502 Hz
AQ             0.6389760 sec
RG                   128
DW                19.500 usec
DE                 6.50 usec
TE                297.0 K
D1             5.00000000 sec
D11            0.03000000 sec
ID0                    1

======== CHANNEL f1 ========
NUC1                  11B
P1                   7.37 usec
PL1                  8.00 dB
SFO1          128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2               100.00 usec
PL2                  2.00 dB
PL12                23.20 dB
PL13                25.00 dB
SFO2          400.0720084 MHz

F2 - Processing parameters
SI                   32768
SF            128.3583540 MHz
WDW                     EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   0.50
```

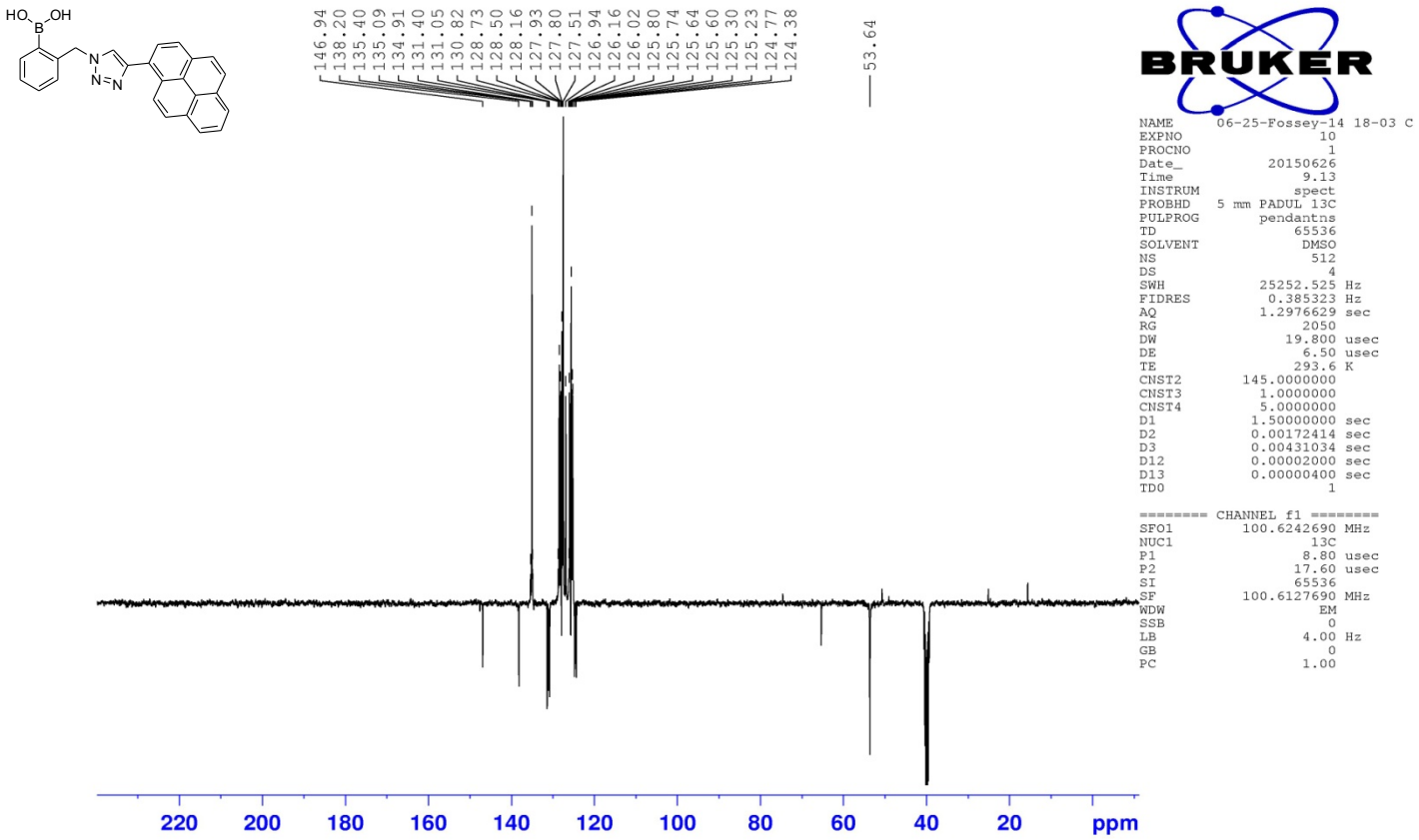
(2-((4-(Pyren-1-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15a) ^{1}H NMR spectrum



BRUKER

NAME	10-17-Fossey-34
EXPNO	10
PROCNO	1
Date_	20131017
Time	20.56
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	512
DW	83.200 usec
DE	12.89 usec
TE	290.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.80 usec
PL1	1.00 dB
PL1W	9.57725906 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

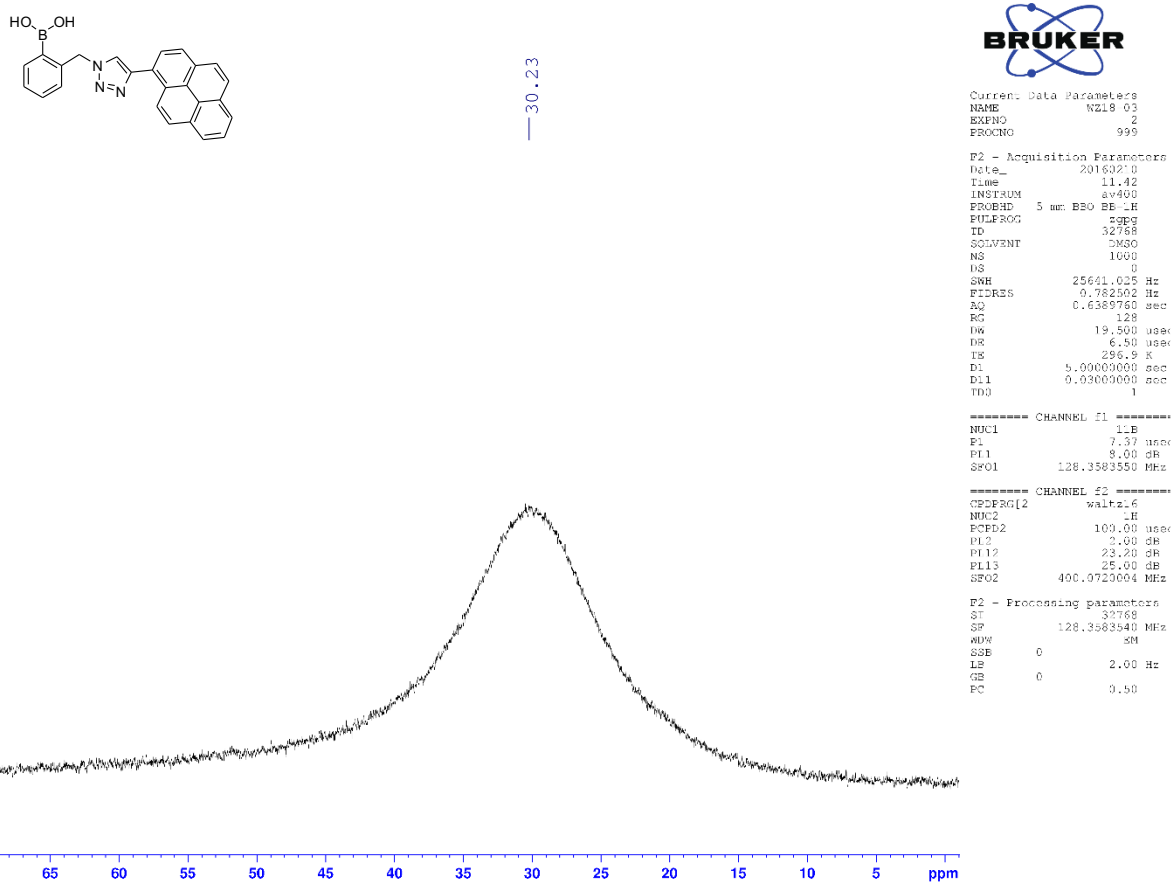
(2-((4-(Pyren-1-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15a) ^{13}C NMR spectrum



BRUKER

NAME	06-25-Fossey-14 18-03 C
EXPNO	10
PROCNO	1
Date_	20150626
Time	9.13
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	DMSO
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.6 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

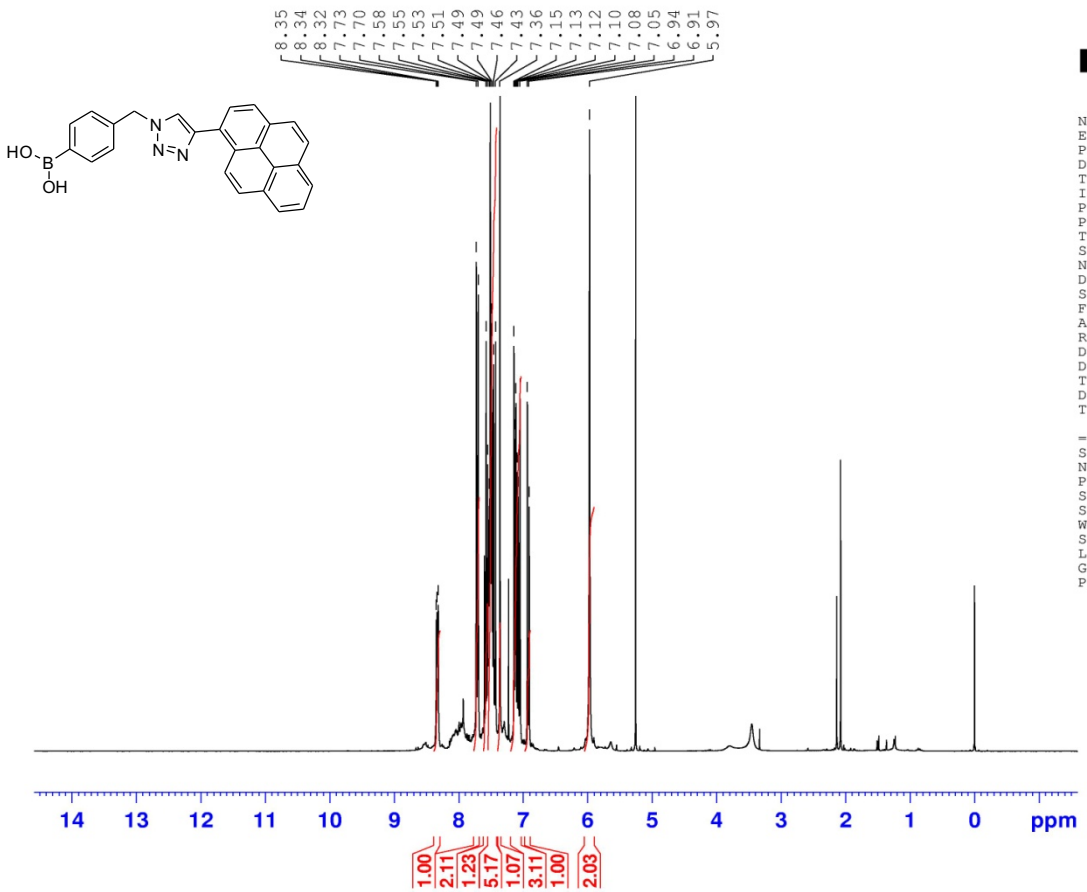
(2-((4-(Pyren-1-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15a) ^{11}B NMR spectrum



BRUKER

```
Current Data Parameters
NAME                  NZ18_03
EXPNO                       2
PROCNO                    999

F2 - Acquisition Parameters
Date_              20160210
Time                  11.42
INSTRUM                av400
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT                DMSO
NS                     1000
DS                        0
SWH               25641.025 Hz
FIDRES            0.782502 Hz
AQ                0.6389760 sec
RG                      128
DW                 19.500 use
DR                  6.50 use
TE                 296.9 K
D1              5.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    11B
P1                   7.37 use
PL1                  9.00 dB
SFO1          128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2             waltz16
NUC2                    1H
PCPD2               100.00 use
PL2                  2.00 dB
PL12                23.20 dB
PL13                25.00 dB
SFO2          400.0723004 MHz

F2 - Processing parameters
SI                    32768
SF            128.3583540 MHz
WDW                      EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   3.50
```

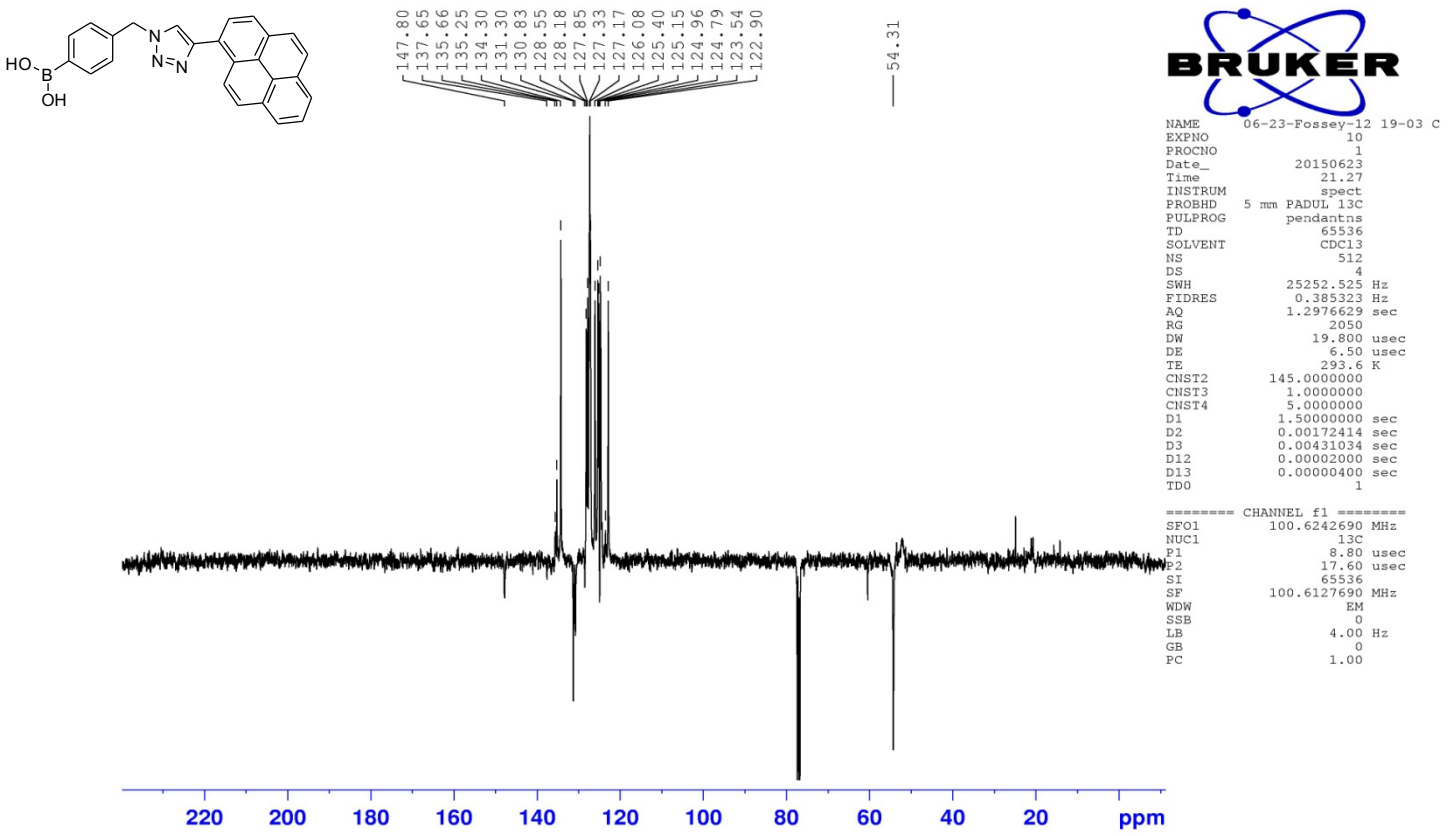
(4-((4-(Pyren-1-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15b) ^{1}H NMR spectrum



BRUKER

NAME	06-22-Fossey-36 19-03
EXPNO	10
PROCNO	1
Date_	20150623
Time	6.28
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	128
DW	83.200 usec
DE	12.89 usec
TE	294.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300167 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

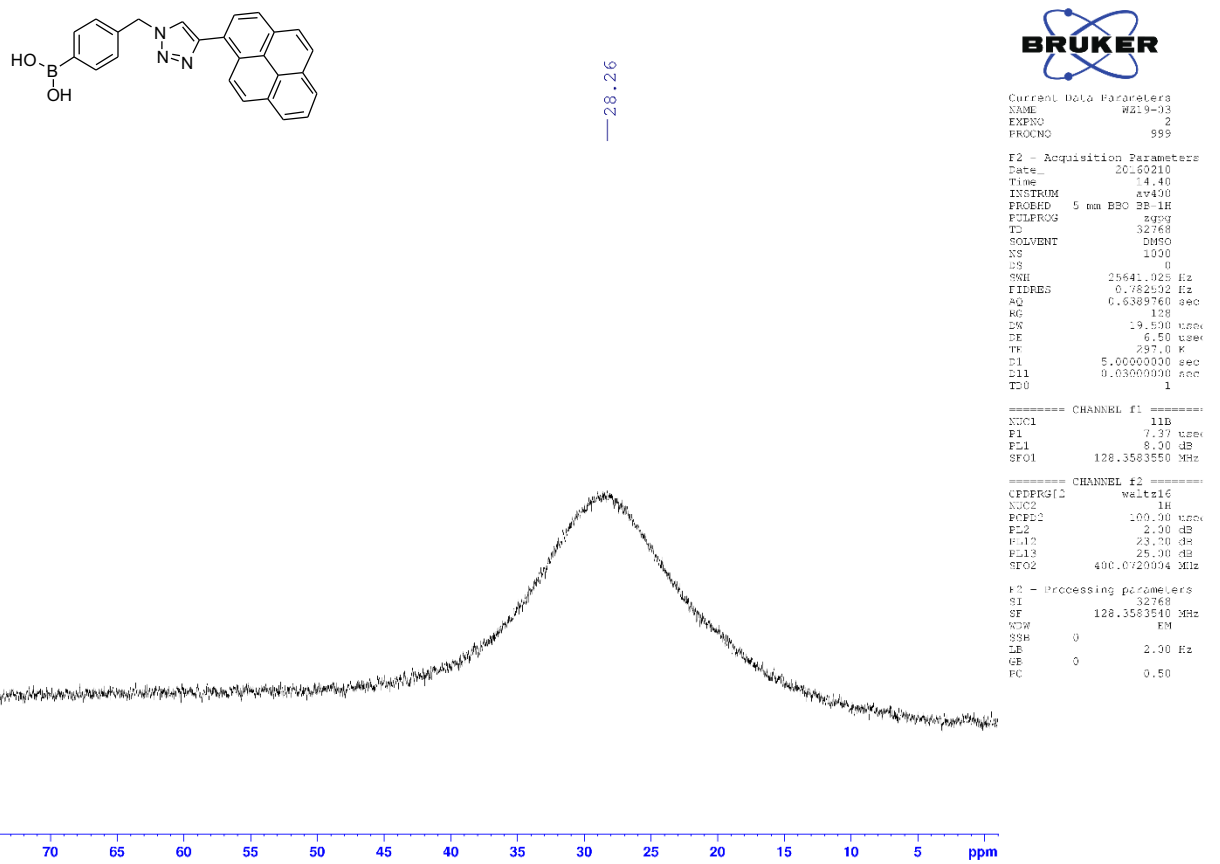
(4-((4-(Pyren-1-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15b) ^{13}C NMR spectrum



BRUKER

NAME	06-23-Fossey-12 19-03 C
EXPNO	10
PROCNO	1
Date_	20150623
Time	21.27
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.6 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

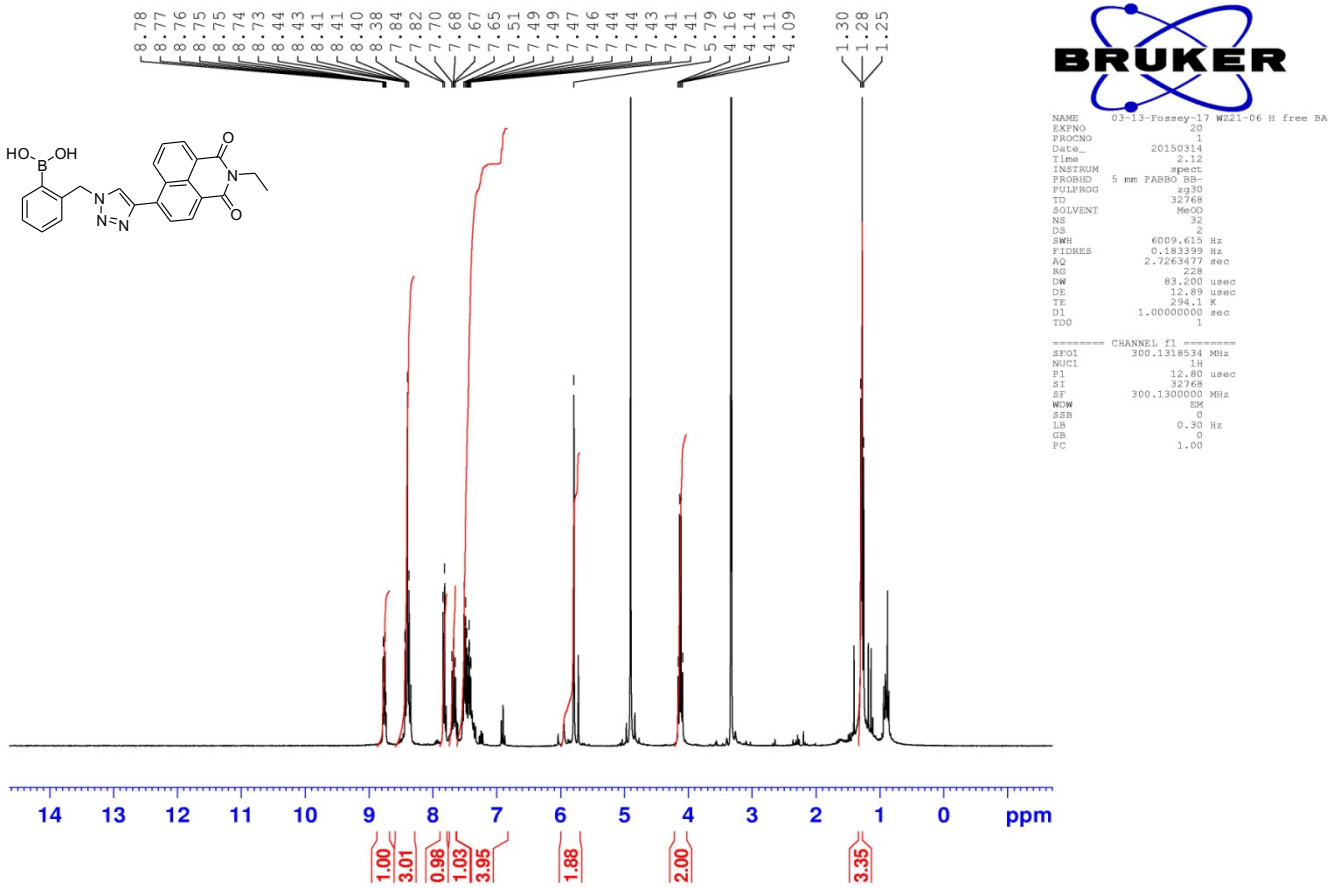
(4-((4-(Pyren-1-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (15b) ^{11}B NMR spectrum



BRUKER

```
Current Data Parameters
NAME                  WZ19-03
EXPNO                       2
PROCNO                    999

F2 - Acquisition Parameters
Date_              20160210
Time                   14.40
INSTRUM                av400
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT                DMSO
NS                     1000
DS                        0
SWH               25641.025 Hz
FIDRES             0.782502 Hz
AQ                 0.6389760 sec
RG                      128
DW                 19.500 usec
DE                  6.50 usec
TE                  297.0 K
D1                5.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    11B
P1                   7.37 usec
PL1                  8.00 dB
SFO1           128.3583560 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                  2.00 dB
PL12                23.70 dB
PL13                25.00 dB
SFO2           400.0720094 MHz

F2 - Processing parameters
SI                    32768
SF             128.3583540 MHz
WDW                      EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   0.50
```

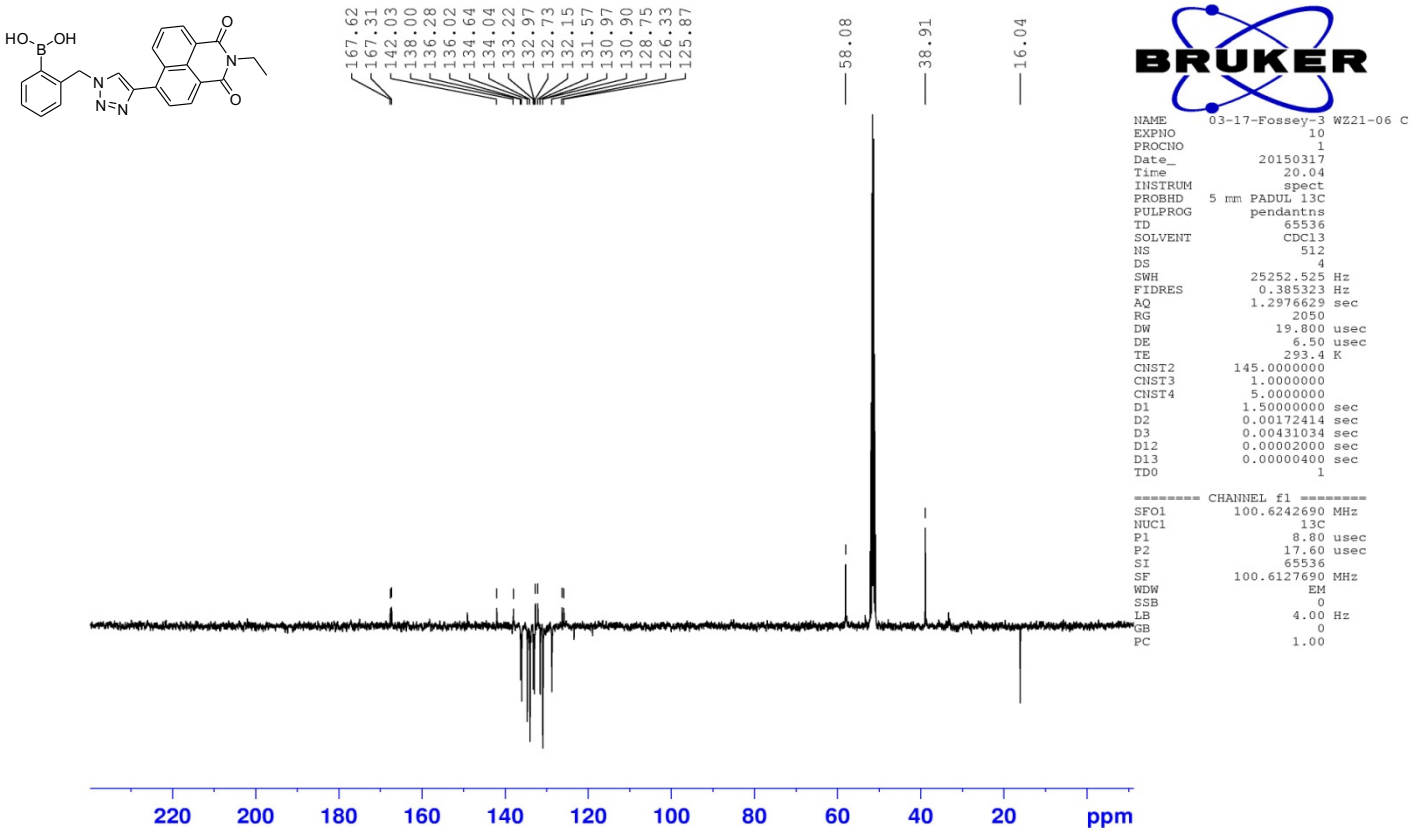
(2-((4-(2-Ethyl-1,3-dioxo-2,3-dihydro-1*H*-benzo[de]isoquinolin-6-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (16a) ^{1}H NMR spectrum



BRUKER

```
NAME          03-13-Fossey-17 WZ21-06 H free BA
EXPNO                     20
PROCNO                     1
Date_               20150314
Time                    2.12
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT                MeOD
NS                       32
DS                        2
SWH                6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7263477 sec
RG                      228
DW                  83.200 usec
DE                  12.89 usec
TE                  294.1 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            300.1318534 MHz
NUC1                      1H
P1                  12.80 usec
SI                    32768
SF              300.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

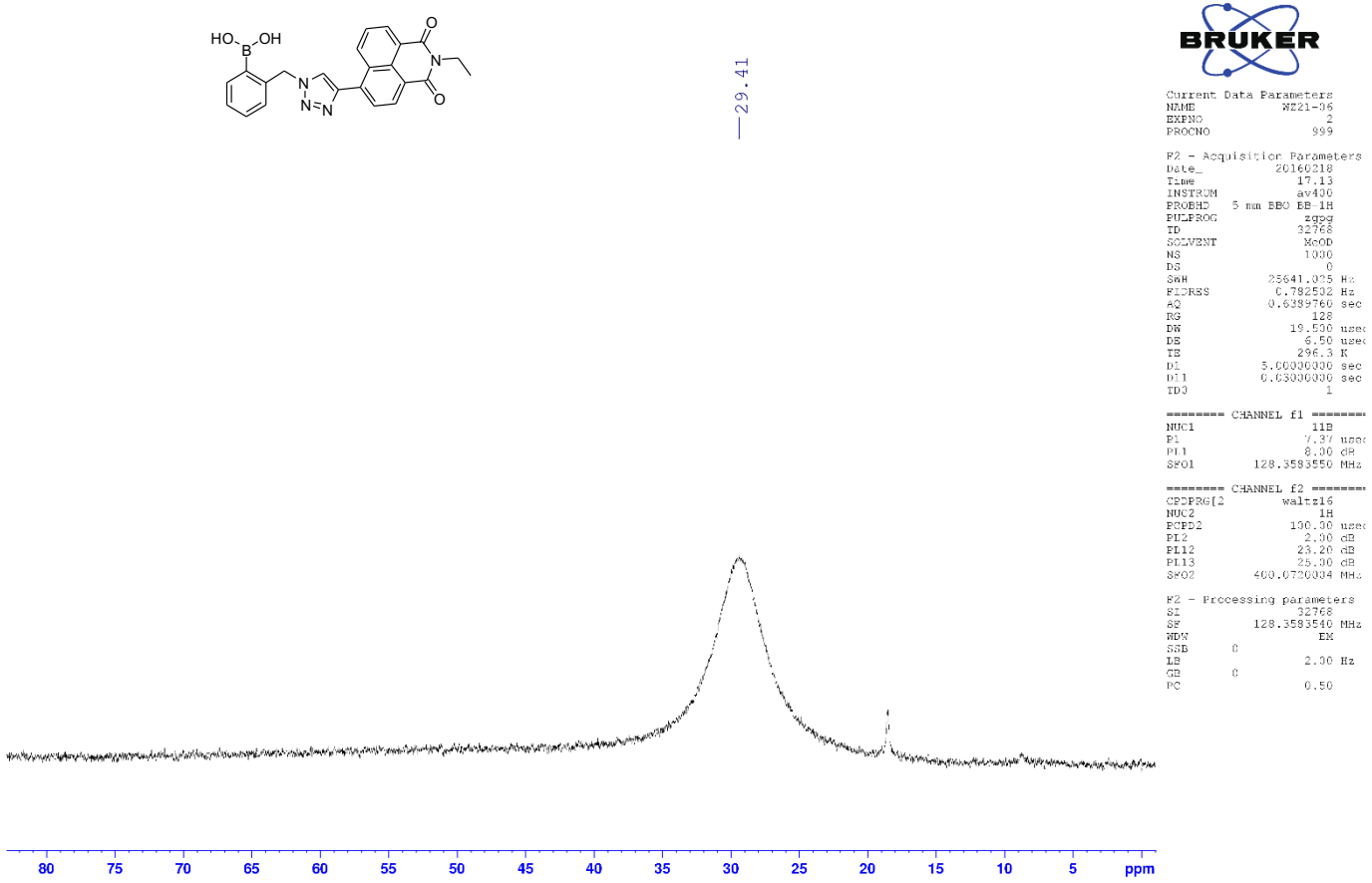
(2-((4-(2-Ethyl-1,3-dioxo-2,3-dihydro-1*H*-benzo[de]isoquinolin-6-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (16a) ^{13}C NMR spectrum



BRUKER

NAME	03-17-Fossey-3 WZ21-06 C
EXPNO	10
PROCNO	1
Date_	20150317
Time	20.04
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.4 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

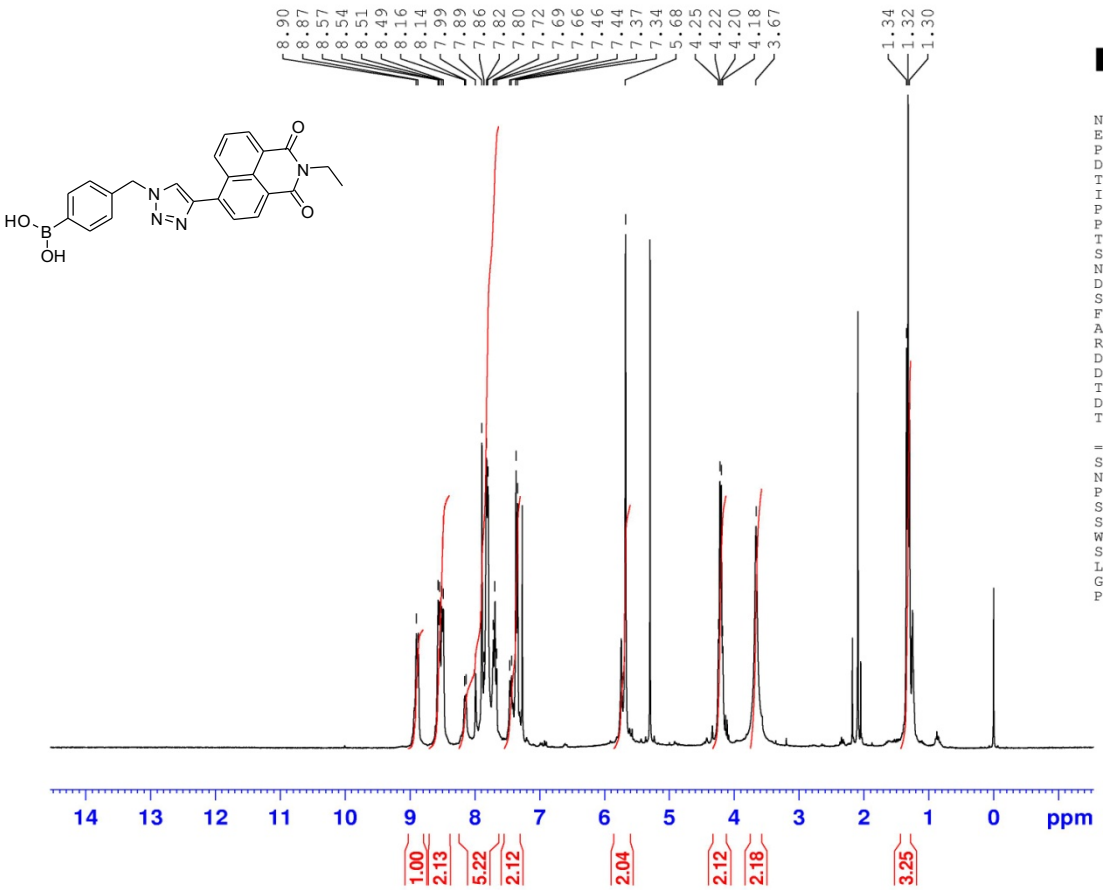
(2-((4-(2-Ethyl-1,3-dioxo-2,3-dihydro-1*H*-benzo[de]isoquinolin-6-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (16a) ^{11}B NMR spectrum



BRUKER

```
Current Data Parameters
NAME                  WZ21-06
EXPNO                       2
PROCNO                    999

F2 - Acquisition Parameters
Date_               20160218
Time                   17.13
INSTRUM                av400
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg
TD                    32768
SOLVENT                MeOD
NS                      1000
DS                         0
SWH               25641.035 Hz
FIDRES             0.782502 Hz
AQ                 0.6389760 sec
RG                       128
DW                  19.500 usec
DE                   6.50 usec
TE                  296.3 K
D1                5.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     11B
P1                    7.37 usec
PL1                   8.00 dB
SFO1           128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   2.00 dB
PL12                 23.20 dB
PL13                 25.00 dB
SFO2           400.0730004 MHz

F2 - Processing parameters
SI                    32768
SF             128.3583540 MHz
WDW                      EM
SSB       0
LB                    2.00 Hz
GB        0
PC                    0.50
```

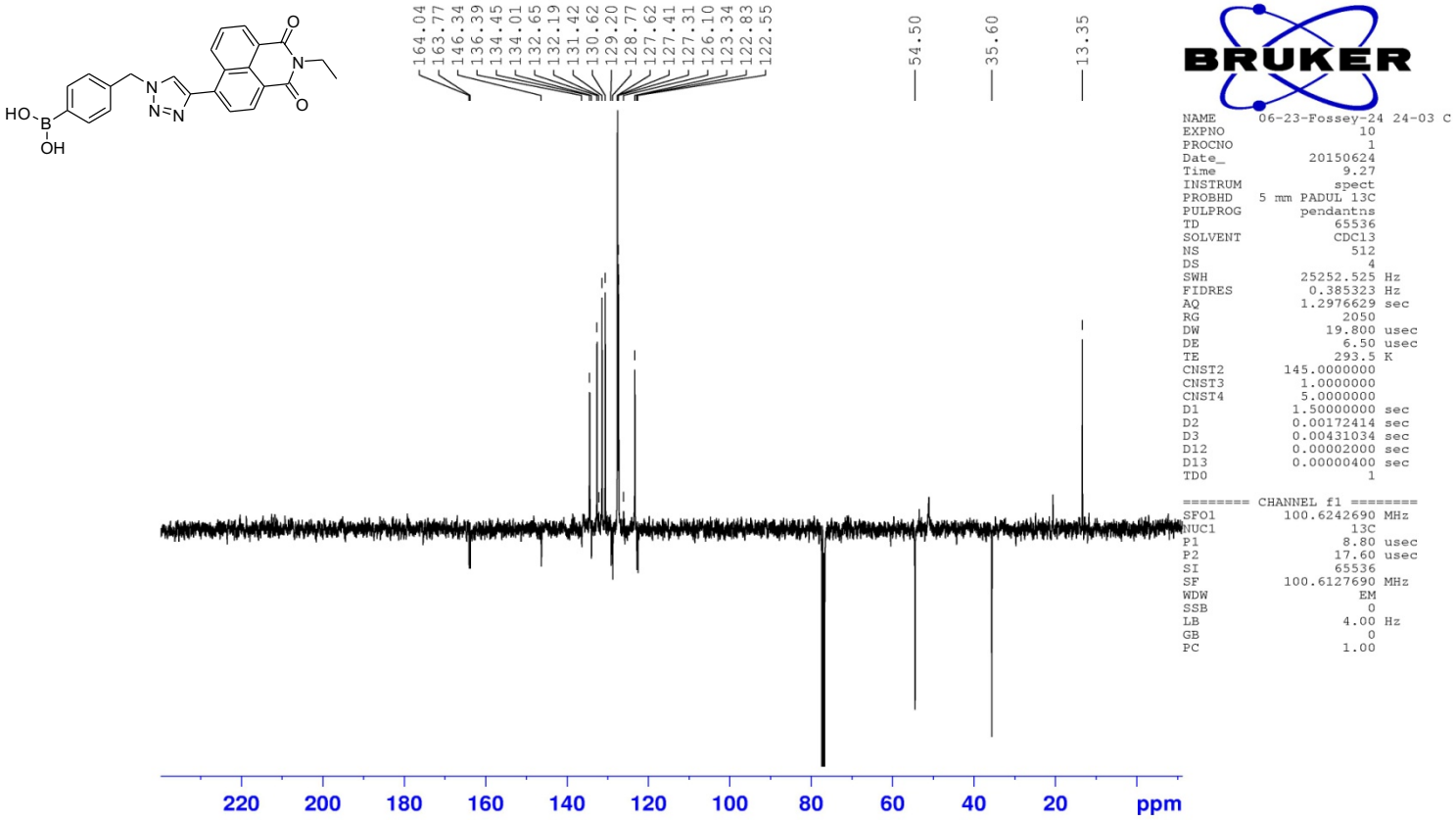
(4-((4-(2-Ethyl-1,3-dioxo-2,3-dihydro-1*H*-benzo[de]isoquinolin-6-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (16b) ^{1}H NMR spectrum



BRUKER

NAME	06-23-Fossey-3 24-03
EXPNO	10
PROCNO	1
Date_	20150624
Time	5.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	161
DW	83.200 usec
DE	12.89 usec
TE	294.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300047 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

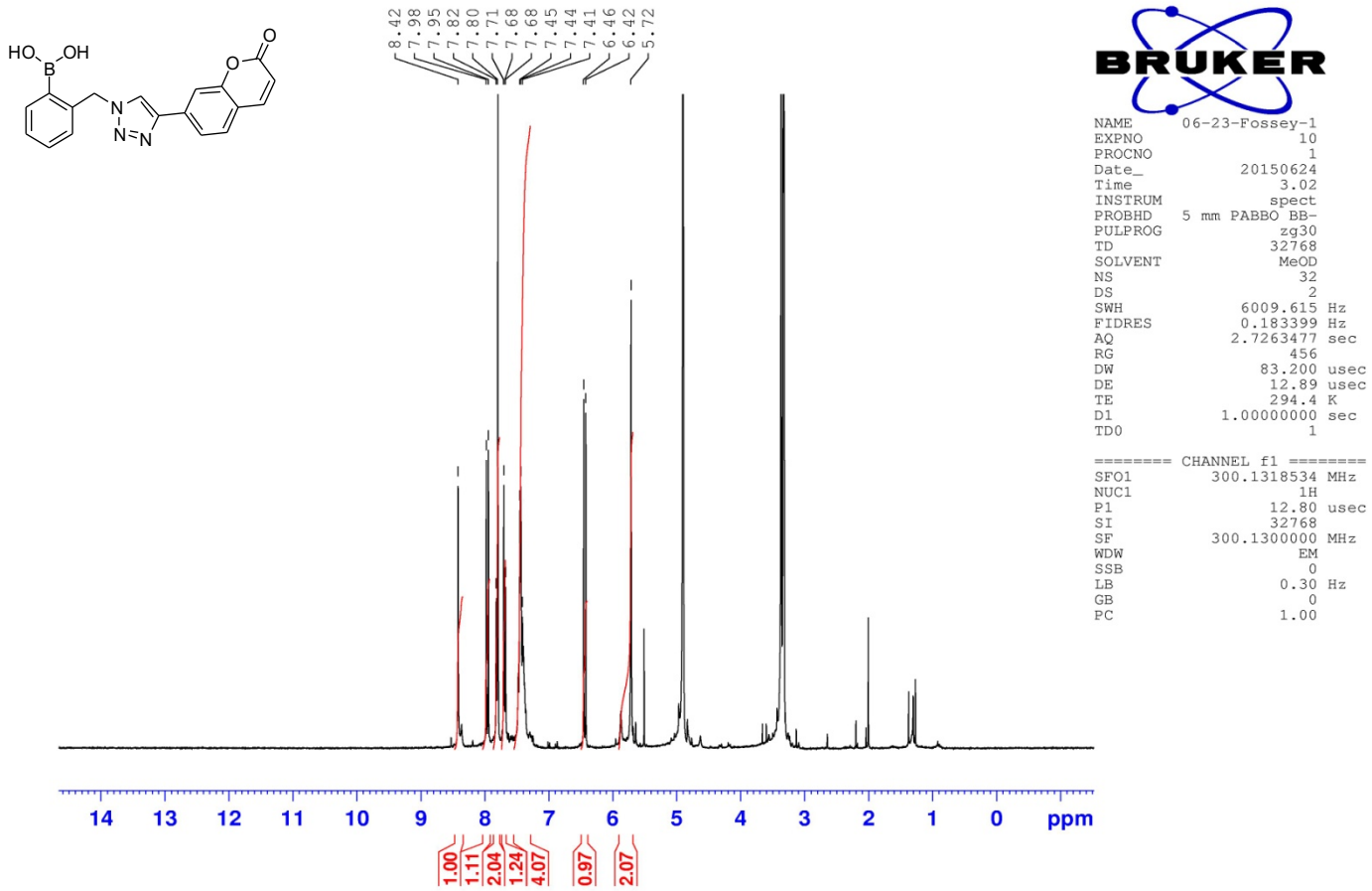
(4-((4-(2-Ethyl-1,3-dioxo-2,3-dihydro-1*H*-benzo[de]isoquinolin-6-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (16b) ^{13}C NMR spectrum



BRUKER

NAME	06-23-Fossey-24 24-03 C
EXPNO	10
PROCNO	1
Date_	20150624
Time	9.27
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.5 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

4(2-((4-(2-Oxo-2H-chromen-7-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17a) ^{1}H NMR spectrum (d_4-methanol)



BRUKER

NAME	06-23-Fossey-1
EXPNO	10
PROCNO	1
Date_	20150624
Time	3.02
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PULPROG	zg30
TD	32768
SOLVENT	MeOD
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	456
DW	83.200 usec
DE	12.89 usec
TE	294.4 K
D1	1.00000000 sec
TD0	1
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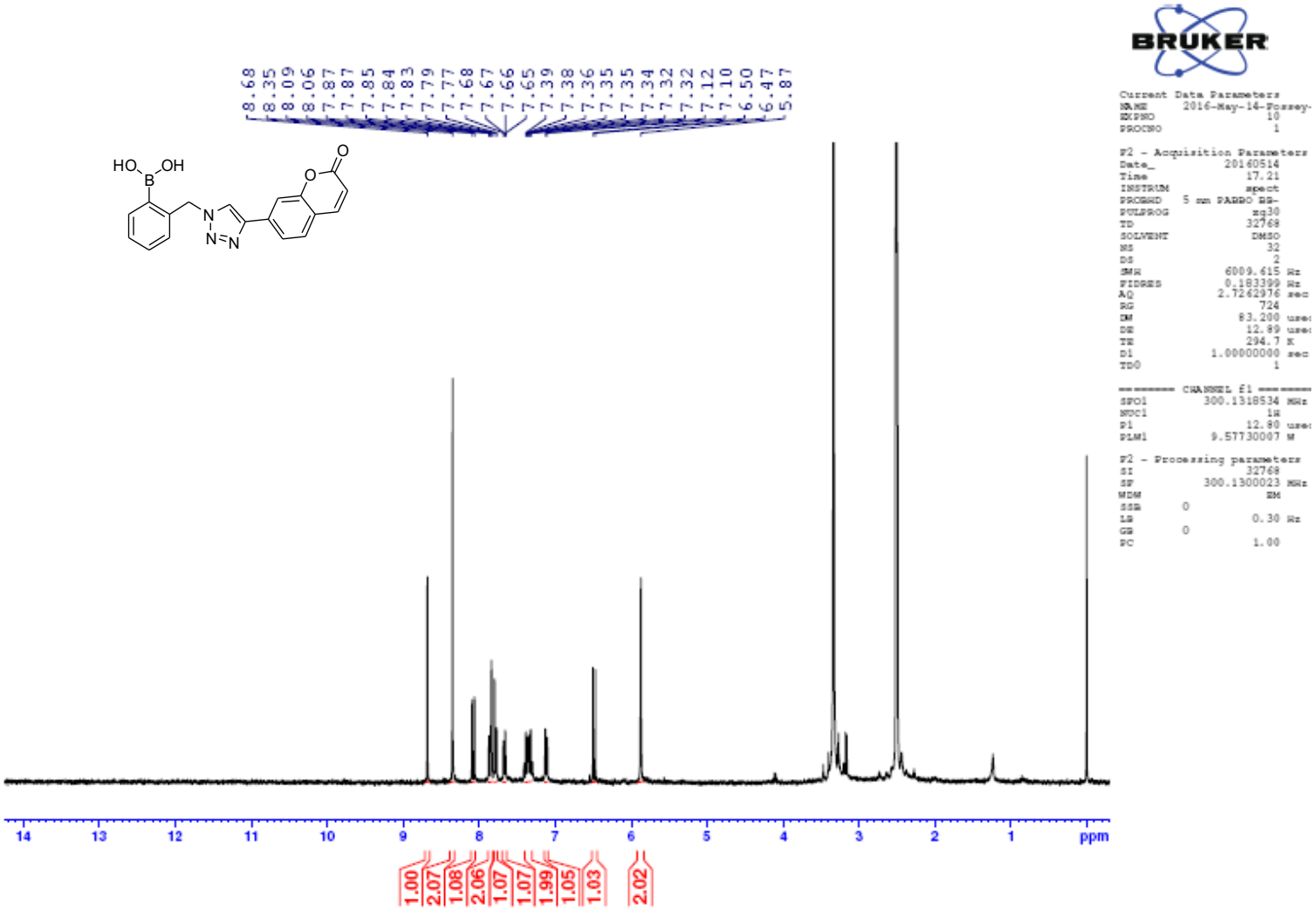
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

4(2-((4-(2-Oxo-2H-chromen-7-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17a) ^{1}H NMR spectrum (d_6-DMSO)



BRUKER

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PROCNO                         1

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PULPROG                    zg30
TD                        32768
SOLVENT                    DMSO
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DE                       12.89 use
TE                       294.7 K
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PLW1                 9.57730007 W

F2 - Processing parameters
SI                        32768
SF                   300.1300023 MHz
WDW                          EM
SSB                   0
LB                            0.30 Hz
GB                    0
PC                            1.00
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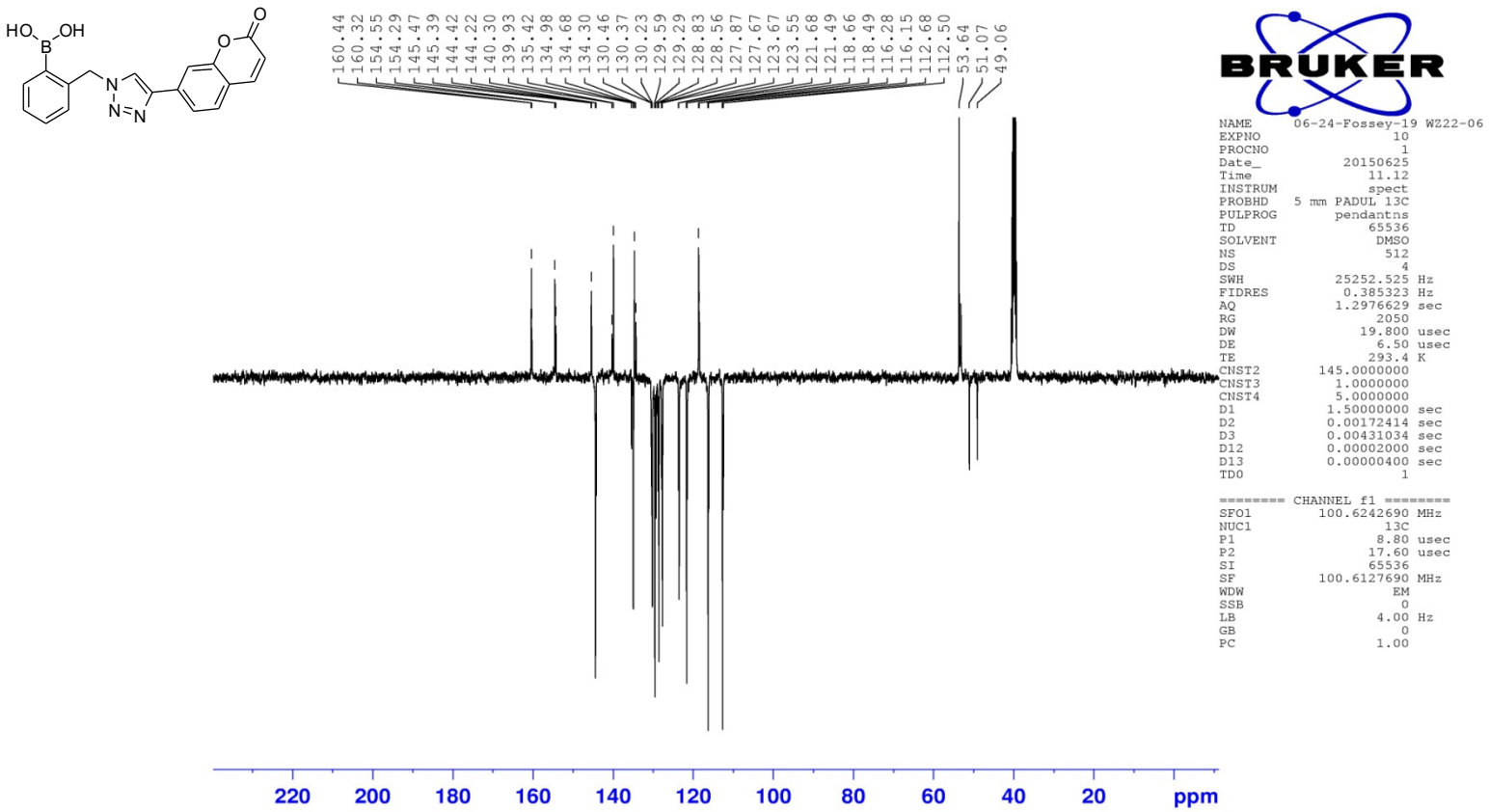
4(2-((4-(2-Oxo-2H-chromen-7-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17a) ^{13}C NMR spectrum



BRUKER

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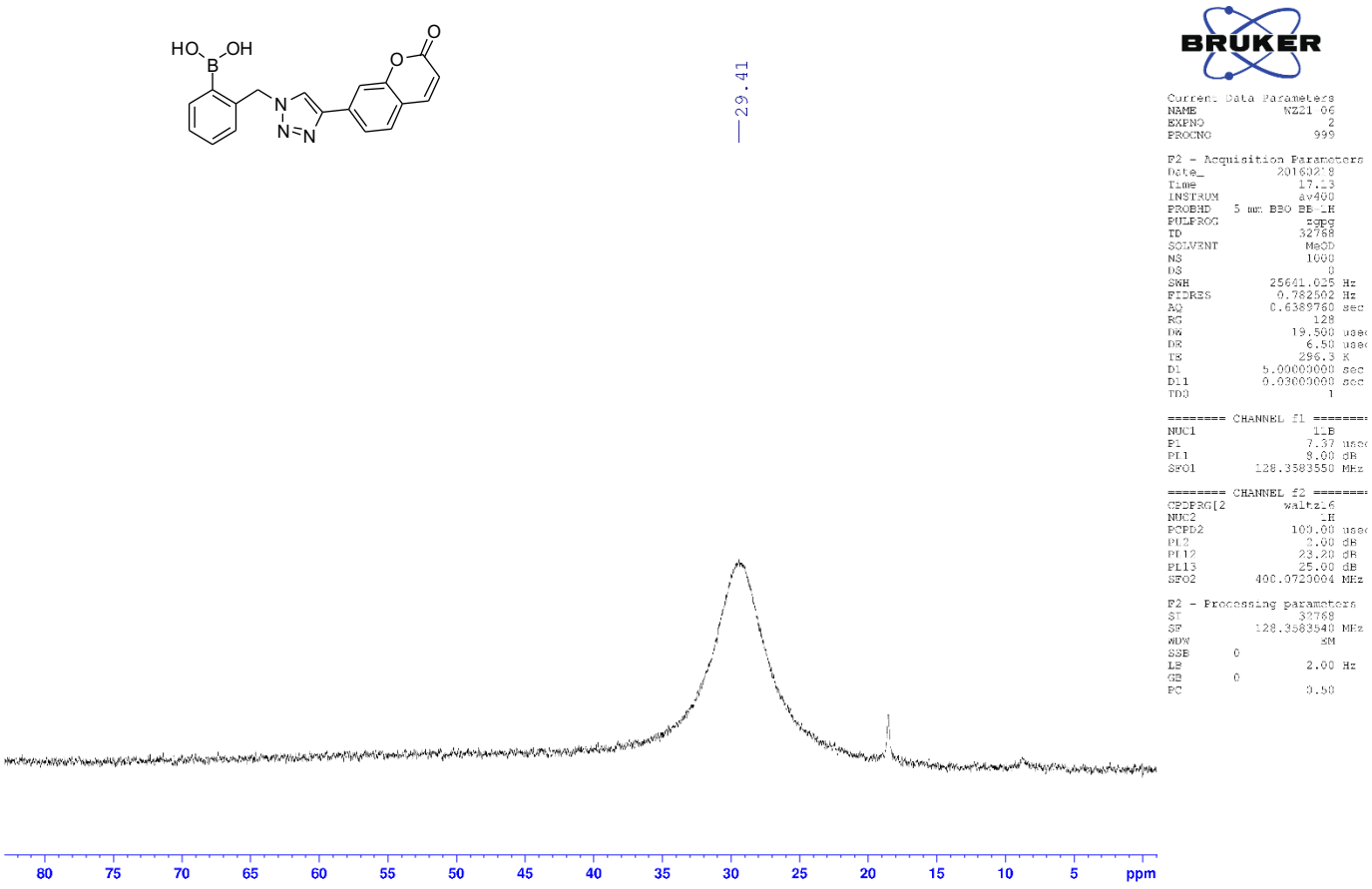
EXPNO	10
PROCNO	1
Date_	20150625
Time	11.12
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	pendantns
TD	65536
SOLVENT	DMSO
NS	512
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	6.50 usec
TE	293.4 K
CNST2	145.0000000
CNST3	1.0000000
CNST4	5.0000000
D1	1.50000000 sec
D2	0.00172414 sec
D3	0.00431034 sec
D12	0.00002000 sec
D13	0.00000400 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6242690 MHz
NUC1	13C
P1	8.80 usec
P2	17.60 usec
SI	65536
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	4.00 Hz
GB	0
PC	1.00

4(2-((4-(2-Oxo-2H-chromen-7-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17a) ^{11}B NMR spectrum



BRUKER

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Current Data Parameters
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EXPNO                       2
PROCNO                    999

F2 - Acquisition Parameters
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PULPROG                zgpg
TD                     32768
SOLVENT                MeOD
NS                      1000
DS                         0
SWH                25641.025 Hz
FIDRES             0.782502 Hz
AQ                 0.6389760 sec
RG                       128
DW                    19.500 usec
DE                     6.50 usec
TE                    296.3 K
D1                 5.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     11B
P1                      7.37 usec
PL1                     9.00 dB
SFO1             128.3583550 MHz

======== CHANNEL f2 ========
CPDPRG[2              waltz16
NUC2                     1H
PCPD2                 100.00 usec
PL2                     2.00 dB
PL12                   23.20 dB
PL13                   25.00 dB
SFO2             400.0720004 MHz

F2 - Processing parameters
SI                       32768
SF                128.3583540 MHz
WDW                         EM
SSB          0
LB                      2.00 Hz
GB           0
PC                      3.50
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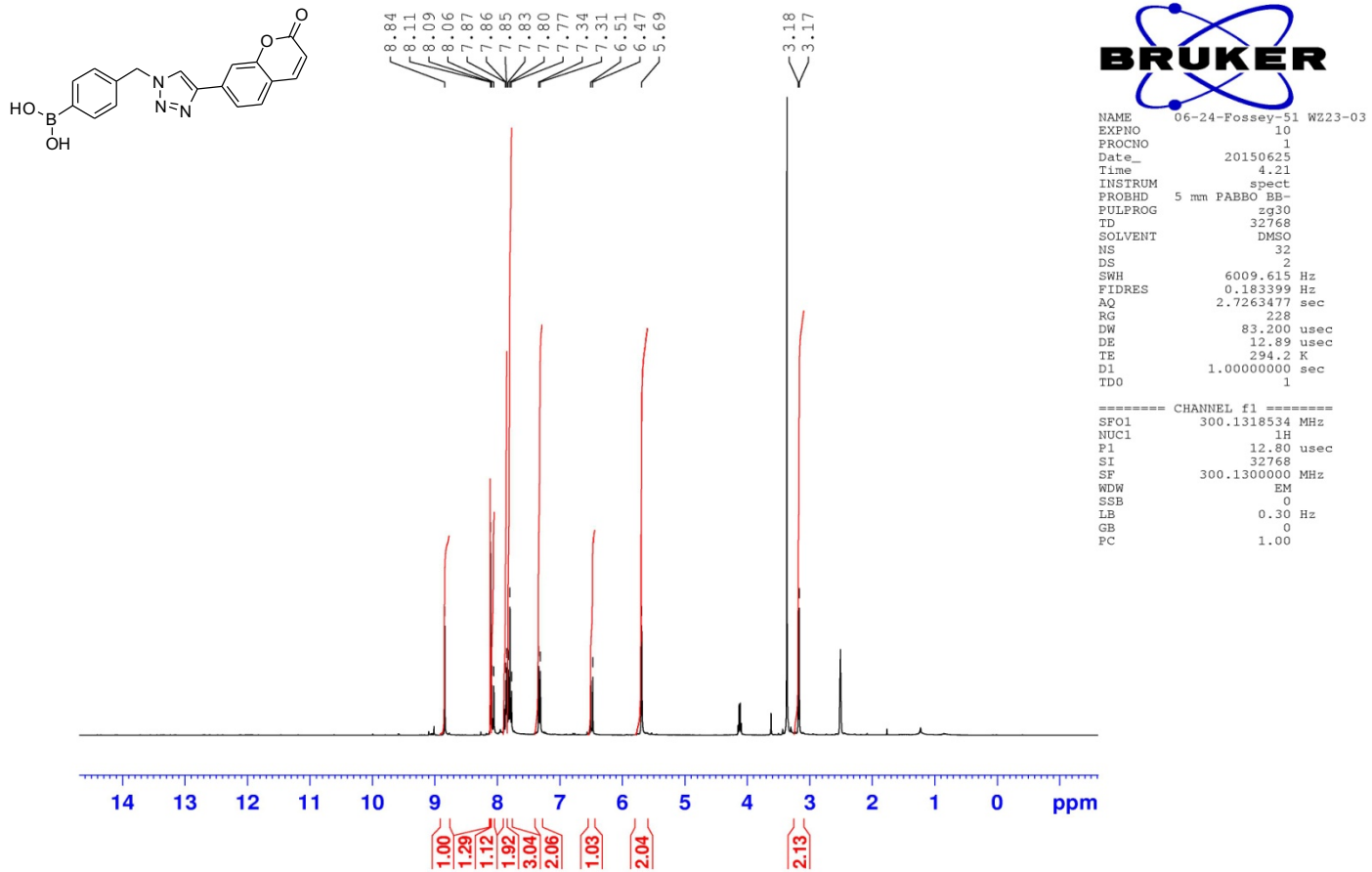
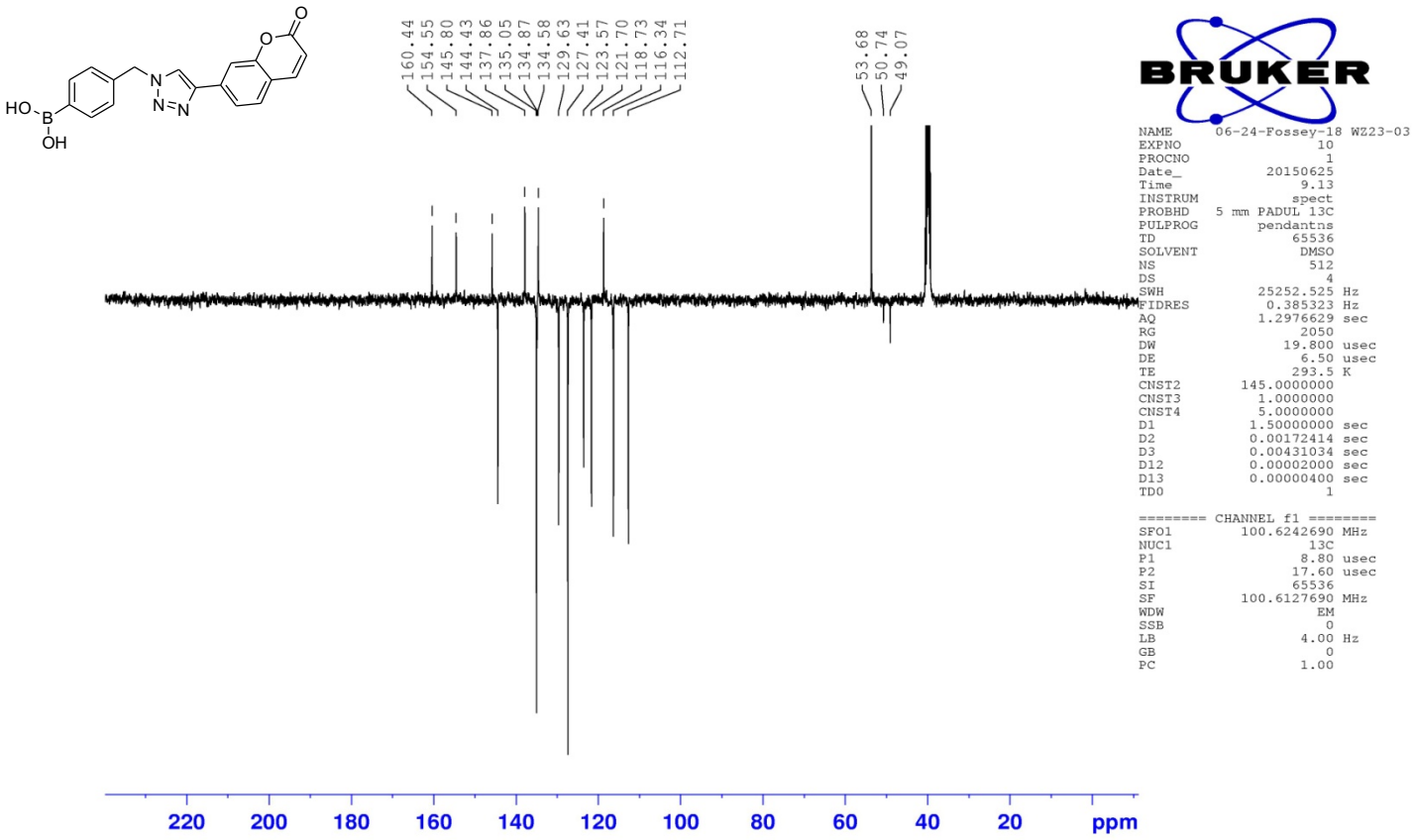
(4-((4-(2-Oxo-2H-chromen-7-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17b) ^{1}H NMR spectrum



BRUKER

NAME	06-24-Fossey-51 WZ23-03
EXPNO	10
PROCNO	1
Date_	20150625
Time	4.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	DMSO
NS	32
DS	2
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	228
DW	83.200 usec
DE	12.89 usec
TE	294.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	300.1318534 MHz
NUC1	1H
P1	12.80 usec
SI	32768
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

(4-((4-(2-Oxo-2H-chromen-7-yl)-1*H*-1,2,3-triazol-1-yl)methyl)phenyl)boronic acid (17b) ^{13}C NMR spectrum

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EXPNO                 10
PROCNO                 1
Date_           20150625
Time                9.13
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG         pendantns
TD                 65536
SOLVENT             DMSO
NS                   512
DS                     4
SWH            25252.525 Hz
FIDRES         0.385323 Hz
AQ             1.2976629 sec
RG                  2050
DW                19.800 usec
DE                 6.50 usec
TE                293.5 K
CNST2         145.0000000
CNST3           1.0000000
CNST4           5.0000000
D1              1.50000000 sec
D2              0.00172414 sec
D3              0.00431034 sec
D12             0.00002000 sec
D13             0.00000400 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6242690 MHz
NUC1                 13C
P1                  8.80 usec
P2                 17.60 usec
SI                 65536
SF            100.6127690 MHz
WDW                  EM
SSB                   0
LB                 4.00 Hz
GB                    0
PC                 1.00
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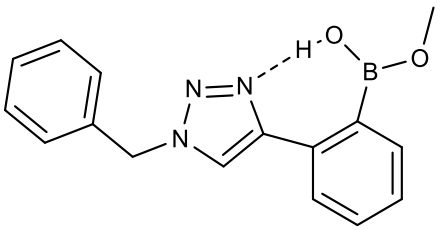
8. X-Ray crystallographic information

8.1 Compound 8a



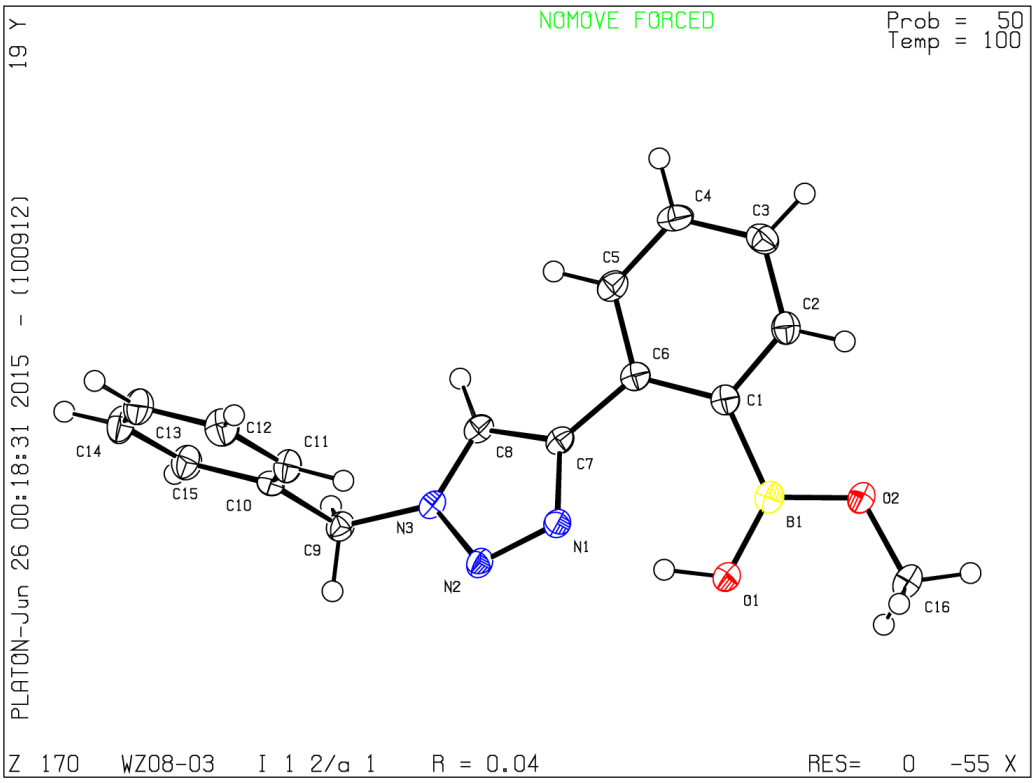


Table 1 Crystal data and structure refinement for COMPOUND 8A.

Identification code	COMPOUND 8A
Empirical formula	$C_{16}H_{16}BN_3O_2$
Formula weight	293.13
Temperature/K	99.98(11)
Crystal system	monoclinic
Space group	I2/a
a/Å	25.8560(9)
b/Å	5.37038(17)
c/Å	21.5154(7)
α/°	90

β/°	101.646(3)
γ/°	90
Volume/Å^3	2926.05(18)
Z	8
ρ_{calc}g/cm^3	1.331
μ/mm^{-1}	0.089
F(000)	1232.0
Crystal size/mm^3	0.2588 × 0.1815 × 0.1003
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.506 to 52.742
Index ranges	-32 ≤ h ≤ 25, -5 ≤ k ≤ 6, -22 ≤ l ≤ 26
Reflections collected	5839
Independent reflections	2976 [R_{int} = 0.0188, R_{sigma} = 0.0283]
Data/restraints/parameters	2976/0/204
Goodness-of-fit on F^2	1.090
Final R indexes [I>=2σ (I)]	R_1 = 0.0418, wR_2 = 0.1011
Final R indexes [all data]	R_1 = 0.0491, wR_2 = 0.1059
Largest diff. peak/hole / e Å^{-3}	0.24/-0.19

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for COMPOUND 8A. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
B_1	3830.3(7)	5355(3)	6078.7(8)	19.3(4)
C_1	4041.9(6)	3315(3)	6607.2(7)	17.7(3)
C_2	3750.6(6)	3155(3)	7091.9(7)	21.6(3)
C_3	3844.1(6)	1372(3)	7565.2(7)	22.9(4)
C_4	4240.2(6)	−366(3)	7567.5(7)	23.2(4)
C_5	4548.5(6)	−228(3)	7112.9(7)	21.1(3)
C_6	4460.5(6)	1585(3)	6635.8(7)	16.8(3)
C_7	4835.3(6)	1532(3)	6199.4(7)	16.1(3)
C_8	5154.7(6)	−389(3)	6082.7(7)	17.8(3)
C_9	5853.9(6)	−687(3)	5417.7(7)	19.2(3)
C_{10}	6376.3(6)	−732(3)	5881.9(7)	18.0(3)
C_{11}	6517.3(6)	1119(3)	6333.2(8)	22.3(4)
C_{12}	7003.2(7)	1040(3)	6750.3(8)	27.3(4)
C_{13}	7353.5(7)	−870(4)	6712.6(8)	27.9(4)
C_{14}	7219.3(7)	−2706(4)	6258.9(9)	29.7(4)
C_{15}	6730.8(7)	−2654(3)	5846.2(8)	24.3(4)
C_{16}	3119.7(7)	8161(4)	5649.8(8)	29.0(4)
N_1	4949.3(5)	3528(2)	5859.4(6)	18.3(3)

N_2	5315.6(5)	2931(2)	5538.8(6)	19.1(3)
N_3	5437.9(5)	537(2)	5677.0(6)	16.6(3)
O_1	4071.8(5)	6200(2)	5620.2(5)	21.8(3)
O_2	3339.3(4)	6271(2)	6094.0(5)	24.7(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for COMPOUND 8A. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
B_1	17.0(8)	18.2(9)	21.4(9)	−2.8(7)	0.7(7)	−1.9(7)
C_1	15.8(7)	17.9(8)	18.3(7)	−2.6(6)	1.0(6)	−3.4(6)
C_2	17.9(8)	23.3(8)	23.3(8)	−1.2(7)	3.4(6)	−0.3(7)
C_3	20.1(8)	29.7(9)	18.9(8)	0.0(7)	4.1(6)	−4.2(7)
C_4	24.4(8)	24.4(9)	18.7(8)	6.1(7)	−0.6(6)	−3.9(7)
C_5	19.5(8)	19.8(8)	22.2(8)	1.1(7)	0.0(6)	−0.1(7)
C_6	15.7(7)	16.6(7)	16.4(7)	−1.7(6)	−0.9(6)	−4.3(6)
C_7	14.2(7)	15.8(8)	16.0(7)	−0.8(6)	−2.2(5)	−2.2(6)
C_8	17.8(8)	14.4(8)	19.6(7)	0.6(6)	0.1(6)	−1.4(6)
C_9	20.7(8)	17.8(8)	18.7(7)	−1.2(6)	3.2(6)	3.1(6)
C_{10}	17.7(8)	18.6(8)	18.7(7)	4.2(6)	6.2(6)	0.3(6)
C_{11}	20.4(8)	21.6(8)	25.1(8)	−0.9(7)	4.9(6)	3.6(7)
C_{12}	23.7(9)	30.0(9)	26.8(9)	−4.9(8)	1.9(7)	−2.7(8)
C_{13}	16.0(8)	36(1)	30.2(9)	3.7(8)	0.7(7)	0.8(7)
C_{14}	23.1(9)	28.9(10)	38.1(10)	4.2(8)	8.7(7)	9.4(8)
C_{15}	24.8(9)	21.7(8)	26.7(8)	−2.0(7)	5.8(7)	3.6(7)
C_{16}	23.0(9)	31.9(10)	31.1(9)	7.7(8)	3.3(7)	9.1(8)
N_1	16.6(6)	17.5(7)	20.4(6)	1.5(5)	3.1(5)	0.7(5)
N_2	18.1(7)	16.6(7)	22.8(7)	1.8(6)	4.1(5)	3.3(5)
N_3	15.9(6)	15.0(6)	17.6(6)	0.4(5)	0.1(5)	1.4(5)
O_1	19.9(6)	21.6(6)	24.4(6)	5.3(5)	5.6(5)	4.4(5)
O_2	19.1(6)	26.9(6)	28.4(6)	7.5(5)	5.2(5)	5.6(5)

Table 4 Bond Lengths for COMPOUND 8A.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
B_1	C_1	1.594(2)	C_8	N_3	1.343(2)
B_1	O_1	1.348(2)	C_9	C_{10}	1.509(2)
B_1	O_2	1.368(2)	C_9	N_3	1.4638(19)
C_1	C_2	1.407(2)	C_{10}	C_{11}	1.386(2)

C_1	C_6	1.418(2)	C_{10}	C_{15}	1.393(2)
C_2	C_3	1.383(2)	C_{11}	C_{12}	1.389(2)
C_3	C_4	1.385(2)	C_{12}	C_{13}	1.381(3)
C_4	C_5	1.383(2)	C_{13}	C_{14}	1.382(3)
C_5	C_6	1.400(2)	C_{14}	C_{15}	1.390(2)
C_6	C_7	1.480(2)	C_{16}	O_2	1.431(2)
C_7	C_8	1.376(2)	N_1	N_2	1.3189(18)
C_7	N_1	1.363(2)	N_2	N_3	1.3428(18)

Table 5 Bond Angles for COMPOUND 8A.

Atom	Atom	Atom	Angle/˚	Atom	Atom	Atom	Angle/˚
O_1	B_1	C_1	127.62(15)	N_3	C_8	C_7	105.48(14)
O_1	B_1	O_2	117.75(15)	N_3	C_9	C_{10}	112.54(12)
O_2	B_1	C_1	114.61(14)	C_{11}	C_{10}	C_9	121.72(14)
C_2	C_1	B_1	114.37(14)	C_{11}	C_{10}	C_{15}	119.16(15)
C_2	C_1	C_6	116.49(14)	C_{15}	C_{10}	C_9	119.11(14)
C_6	C_1	B_1	129.06(14)	C_{10}	C_{11}	C_{12}	120.42(15)
C_3	C_2	C_1	123.21(15)	C_{13}	C_{12}	C_{11}	120.18(16)
C_2	C_3	C_4	119.24(15)	C_{12}	C_{13}	C_{14}	119.82(16)
C_5	C_4	C_3	119.53(15)	C_{13}	C_{14}	C_{15}	120.20(16)
C_4	C_5	C_6	121.62(15)	C_{14}	C_{15}	C_{10}	120.20(16)
C_1	C_6	C_7	125.23(14)	N_2	N_1	C_7	110.19(13)
C_5	C_6	C_1	119.81(14)	N_1	N_2	N_3	106.19(12)
C_5	C_6	C_7	114.95(14)	C_8	N_3	C_9	128.37(13)
C_8	C_7	C_6	128.62(14)	N_2	N_3	C_8	111.32(13)
N_1	C_7	C_6	124.46(14)	N_2	N_3	C_9	120.23(13)
N_1	C_7	C_8	106.81(13)	B_1	O_2	C_{16}	118.96(13)

Table 6 Hydrogen Bonds for COMPOUND 8A.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
C_8	H_8	N_1^1	0.93	2.57	3.329(2)	138.5
C_9	H_{9A}	O_1^2	0.97	2.41	3.317(2)	154.8
O_1	H_1	N_1	0.94(3)	1.74(3)	2.6458(17)	161(2)

[1]+X,-1+Y,+Z; [2]1-X,1-Y,1-Z

Table 7 Torsion Angles for COMPOUND 8A.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
B_1	C_1	C_2	C_3	174.63(15)	C_8	C_7	N_1	N_2	0.52(17)
B_1	C_1	C_6	C_5	−173.55(15)	C_9	C_{10}	C_{11}	C_{12}	−179.37(15)
B_1	C_1	C_6	C_7	7.6(2)	C_9	C_{10}	C_{15}	C_{14}	178.54(15)
C_1	B_1	O_2	C_{16}	178.36(14)	C_{10}	C_9	N_3	C_8	−78.73(19)
C_1	C_2	C_3	C_4	−0.4(3)	C_{10}	C_9	N_3	N_2	97.80(16)
C_1	C_6	C_7	C_8	−161.18(15)	C_{10}	C_{11}	C_{12}	C_{13}	0.9(3)
C_1	C_6	C_7	N_1	23.2(2)	C_{11}	C_{10}	C_{15}	C_{14}	0.0(2)
C_2	C_1	C_6	C_5	2.9(2)	C_{11}	C_{12}	C_{13}	C_{14}	−0.1(3)
C_2	C_1	C_6	C_7	−175.91(14)	C_{12}	C_{13}	C_{14}	C_{15}	−0.8(3)
C_2	C_3	C_4	C_5	2.6(2)	C_{13}	C_{14}	C_{15}	C_{10}	0.8(3)
C_3	C_4	C_5	C_6	−2.0(2)	C_{15}	C_{10}	C_{11}	C_{12}	−0.9(2)
C_4	C_5	C_6	C_1	−0.8(2)	N_1	C_7	C_8	N_3	−0.59(16)
C_4	C_5	C_6	C_7	178.08(14)	N_1	N_2	N_3	C_8	−0.17(16)
C_5	C_6	C_7	C_8	20.0(2)	N_1	N_2	N_3	C_9	−177.25(12)
C_5	C_6	C_7	N_1	−155.67(14)	N_3	C_9	C_{10}	C_{11}	−29.9(2)
C_6	C_1	C_2	C_3	−2.3(2)	N_3	C_9	C_{10}	C_{15}	151.58(14)
C_6	C_7	C_8	N_3	−176.83(14)	O_1	B_1	C_1	C_2	168.61(16)
C_6	C_7	N_1	N_2	176.96(13)	O_1	B_1	C_1	C_6	−14.9(3)
C_7	C_8	N_3	C_9	177.26(14)	O_1	B_1	O_2	C_{16}	−3.4(2)
C_7	C_8	N_3	N_2	0.48(16)	O_2	B_1	C_1	C_2	−13.3(2)
C_7	N_1	N_2	N_3	−0.22(16)	O_2	B_1	C_1	C_6	163.20(15)

Table 8 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for COMPOUND 8A.

Atom	*x*	*y*	*z*	U(eq)
H_2	3482	4306	7094	26
H_3	3643	1340	7878	27
H_4	4298	−1617	7873	28
H_5	4821	−1369	7124	25
H_8	5171	−1991	6250	21
H_{9A}	5897	180	5036	23
H_{9B}	5747	−2382	5301	23
H_{11}	6285	2422	6357	27
H_{12}	7093	2276	7056	33
H_{13}	7679	−919	6992	34
H_{14}	7457	−3981	6230	36
H_{15}	6640	−3906	5545	29

H_{16A}	3137	7632	5228	43
H_{16B}	3317	9675	5747	43
H_{16C}	2758	8443	5676	43
H_1	4411(10)	5520(50)	5658(11)	54(7)

Experimental

Single crystals of $C_{16}H_{16}BN_3O_2$ **8a** were obtained by slow evaporation of a methanol solution. A **s**uitable crystal was selected and analysed on a SuperNova, Dual, Cu at zero, Atlas diffractometer. The crystal was kept at 99.98(11) K during data collection. Using Olex2,[8] the structure was solved with the Superflip[9-11] structure solution program using Charge Flipping and refined with the ShelXL[12] refinement package using Least Squares minimisation.

Crystal structure determination of 8a

Crystal Data for $C_{16}H_{16}BN_3O_2$ (M =293.13 g/mol): monoclinic, space group I2/a (no. 15), a = 25.8560(9) Å, b = 5.37038(17) Å, c = 21.5154(7) Å, β = 101.646(3)°, V = 2926.05(18) Å^3, Z = 8, T = 99.98(11) K, μ(MoKα) = 0.089 mm^{-1}, *Dcalc* = 1.331 g/cm^3, 5839 reflections measured (5.506° ≤ 2Θ ≤ 52.742°), 2976 unique (R_{int} = 0.0188, R_{sigma} = 0.0283) which were used in all calculations. The final R_1 was 0.0418 (I > 2σ(I)) and wR_2 was 0.1059 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso

 At 1.2 times of:

  All C(H) groups, All C(H,H) groups

 At 1.5 times of:

  All C(H,H,H) groups

2.a Secondary CH2 refined with riding coordinates:

 C9(H9A,H9B)

2.b Aromatic/amide H refined with riding coordinates:

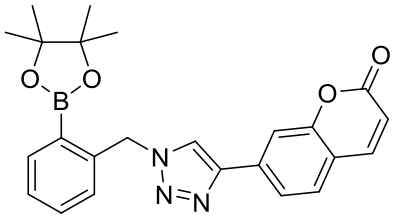
 C2(H2), C3(H3), C4(H4), C5(H5), C8(H8), C11(H11), C12(H12), C13(H13),

 C14(H14), C15(H15)

2.c Idealised Me refined as rotating group:

 C16(H16A,H16B,H16C)

8.2 Compound 13a



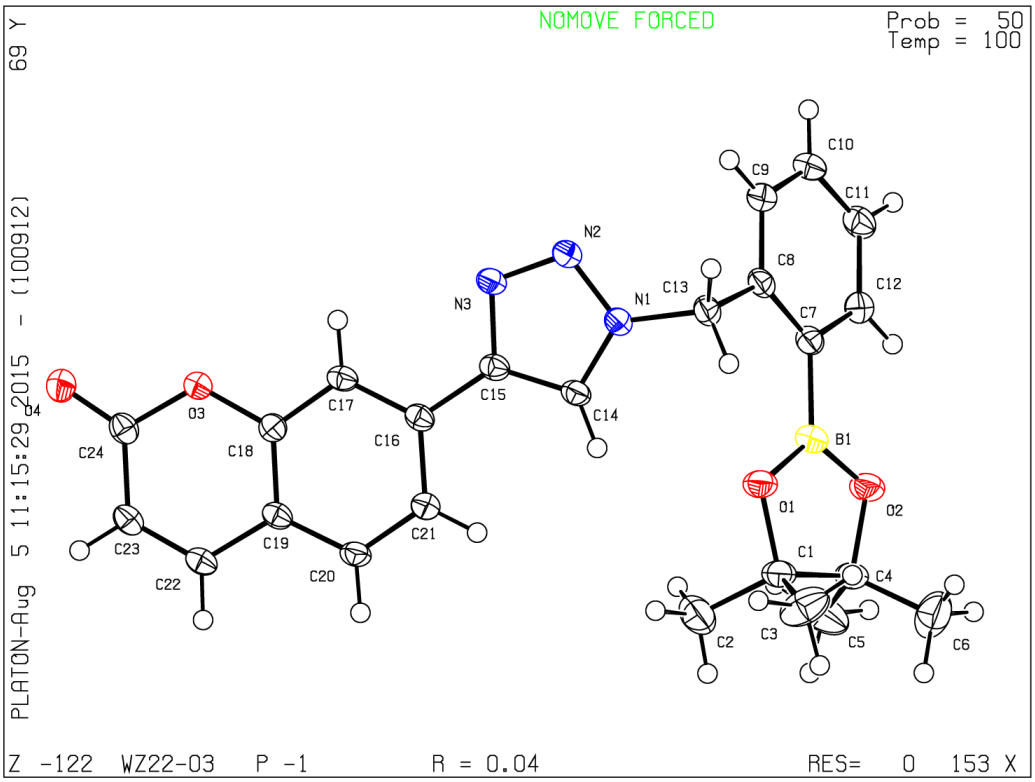


Table 1 Crystal data and structure refinement for COMPOUND 13A.

Identification code	COMPOUND 13A
Empirical formula	$C_{24}H_{24}BN_3O_4$
Formula weight	429.27
Temperature/K	99.9(2)
Crystal system	triclinic
Space group	P-1
a/Å	7.1065(4)
b/Å	9.0913(6)
c/Å	17.6850(11)
α/°	81.994(5)
β/°	84.409(5)

γ/°	68.790(6)
Volume/Å^3	1053.43(12)
Z	2
ρ_{calc}g/cm^3	1.353
μ/mm^{-1}	0.750
F(000)	452.0
Crystal size/mm^3	0.2165 × 0.173 × 0.0282
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	10.116 to 136.484
Index ranges	-8 ≤ h ≤ 7, -10 ≤ k ≤ 7, -21 ≤ l ≤ 21
Reflections collected	6836
Independent reflections	3847 [R_{int} = 0.0249, R_{sigma} = 0.0340]
Data/restraints/parameters	3847/0/293
Goodness-of-fit on F^2	1.042
Final R indexes [I>=2σ (I)]	R_1 = 0.0443, wR_2 = 0.1147
Final R indexes [all data]	R_1 = 0.0539, wR_2 = 0.1232
Largest diff. peak/hole / e Å^{-3}	0.43/-0.34

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for COMPOUND 13A. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O3	7946.2(17)	-1718.1(13)	11246.4(7)	22.4(3)
O4	8442.2(19)	-3233.6(15)	12349.9(7)	28.5(3)
O1	5207.9(18)	3151.8(14)	6411.5(7)	28.7(3)
O2	2202.6(19)	4573.0(14)	5875.3(8)	31.0(3)
C17	7580(2)	-30.3(19)	10090(1)	20.8(3)
N3	7313(2)	3000.6(16)	9242.3(8)	23.1(3)
N1	6923(2)	3814.2(16)	8049.2(8)	21.7(3)
C18	7725(2)	-1507.9(19)	10469.7(9)	20.0(3)
C19	7627(2)	-2726.4(19)	10091.2(10)	20.9(3)
C22	7812(2)	-4223.0(19)	10532.6(10)	22.5(3)
C15	7174(2)	1800.0(19)	8889.4(10)	20.7(3)
C21	7230(2)	-963.3(19)	8916.7(10)	22.7(3)
N2	7165(2)	4224.1(16)	8725.4(8)	23.9(3)
C16	7329(2)	252.2(18)	9308.2(10)	19.9(3)
C24	8190(2)	-3159(2)	11676.5(10)	23.1(3)
C23	8105(2)	-4431.9(19)	11283.8(10)	23.7(3)
C20	7378(2)	-2425.1(19)	9302.5(10)	22.4(3)
C9	4085(3)	7463(2)	7688.8(10)	26.9(4)

C14	6917(2)	2323.6(18)	8126(1)	21.5(3)
C12	1324(3)	7197(2)	6775.1(10)	26.1(4)
C7	3240(3)	5985.1(19)	6817.8(9)	22.8(3)
C11	805(3)	8495(2)	7181.4(11)	29.2(4)
C8	4636(2)	6153.4(19)	7278.6(9)	22.2(3)
C10	2176(3)	8626(2)	7649.6(11)	30.0(4)
C4	2805(3)	3053(2)	5575.9(11)	28.6(4)
C1	4926(3)	2077(2)	5928.8(11)	27.3(4)
B1	3576(3)	4535(2)	6363.8(12)	24.5(4)
C13	6760(2)	4942(2)	7355.6(10)	23.4(3)
C2	4934(3)	606(2)	6460.5(14)	44.2(5)
C6	2838(4)	3355(3)	4720.4(13)	54.7(7)
C3	6687(3)	1683(3)	5350.6(14)	47.1(6)
C5	1150(3)	2376(3)	5873.7(17)	51.7(6)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for COMPOUND 13A. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\cdots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O3	26.1(6)	18.7(6)	21.8(6)	-2.6(4)	-1.8(4)	-6.8(4)
O4	34.2(7)	26.7(6)	22.9(6)	-0.9(5)	-2.1(5)	-9.1(5)
O1	29.8(6)	21.9(6)	33.4(7)	-9.0(5)	-7.7(5)	-4.4(5)
O2	32.2(7)	20.8(6)	37.4(7)	-8.9(5)	-9.5(5)	-2.2(5)
C17	19.2(7)	16.7(8)	26.2(8)	-5.6(6)	-1.0(6)	-4.8(6)
N3	25.8(7)	16.9(7)	26.3(7)	-3.6(6)	-3.2(6)	-6.4(5)
N1	23.6(7)	17.5(7)	23.5(7)	-1.2(5)	-3.3(5)	-6.7(5)
C18	16.7(7)	19.2(8)	21.7(8)	-3.2(6)	-1.4(6)	-2.9(6)
C19	18.5(7)	16.0(7)	26.2(9)	-3.8(6)	-0.3(6)	-3.5(6)
C22	21.0(8)	16.3(7)	29.0(9)	-4.1(6)	-0.1(6)	-4.7(6)
C15	18.1(7)	17.6(8)	25.5(8)	-4.7(6)	-1.6(6)	-4.2(6)
C21	25.5(8)	18.5(8)	23.1(8)	-3.6(6)	-2.5(6)	-5.8(6)
N2	28.0(7)	18.4(7)	25.8(7)	-2.1(6)	-4.7(6)	-8.1(6)
C16	16.1(7)	16.4(8)	25.5(8)	-3.2(6)	-0.9(6)	-3.4(6)
C24	20.7(8)	20.1(8)	25.4(9)	-1.1(6)	0.7(6)	-4.7(6)
C23	22.4(8)	17.2(8)	28.3(9)	-1.3(6)	0.7(6)	-4.2(6)
C20	23.7(8)	16.3(8)	26.3(8)	-6.6(6)	-1.6(6)	-4.4(6)
C9	34.3(9)	21.4(8)	26.9(9)	-1.8(7)	-7.2(7)	-11.1(7)
C14	22.5(8)	15.4(8)	26.1(8)	-3.9(6)	-2.5(6)	-5.3(6)

C12	27.7(8)	23.7(8)	25.8(9)	-1.2(7)	-5.4(7)	-7.2(7)
C7	26.9(8)	18.8(8)	21.9(8)	-0.9(6)	-0.3(7)	-7.9(7)
C11	28.5(9)	21.0(8)	32.7(10)	-3.4(7)	-1.7(7)	-2.3(7)
C8	25.7(8)	17.2(8)	23.0(8)	1.3(6)	-0.7(6)	-8.1(6)
C10	40.5(10)	18.1(8)	30.0(9)	-5.8(7)	-2.2(8)	-7.6(7)
C4	33.2(9)	19.9(8)	32.2(10)	-7.9(7)	-7.7(7)	-5.4(7)
C1	28.7(9)	21.8(8)	32.3(9)	-8.3(7)	-5.5(7)	-7.0(7)
B1	25.1(9)	20.0(9)	28.7(10)	-5.3(8)	-0.4(8)	-7.7(7)
C13	26.7(8)	20.0(8)	23.9(8)	1.9(6)	-3.9(6)	-9.6(7)
C2	47.6(12)	28.2(10)	58.6(14)	5.8(9)	-23.4(10)	-14.1(9)
C6	56.2(14)	52.2(14)	33.3(12)	-11(1)	-11.5(10)	12.2(11)
C3	32(1)	63.5(15)	46.3(13)	-27.4(11)	-1.0(9)	-10.1(10)
C5	34.9(11)	29.5(11)	91(2)	-5.5(11)	-15.1(12)	-9.3(9)

Table 4 Bond Lengths for COMPOUND 13A.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O3	C18	1.377(2)	C15	C16	1.468(2)
O3	C24	1.381(2)	C15	C14	1.374(2)
O4	C24	1.211(2)	C21	C16	1.409(2)
O1	C1	1.460(2)	C21	C20	1.379(2)
O1	B1	1.367(2)	C24	C23	1.452(2)
O2	C4	1.450(2)	C9	C8	1.395(2)
O2	B1	1.354(2)	C9	C10	1.387(3)
C17	C18	1.389(2)	C12	C7	1.409(2)
C17	C16	1.388(2)	C12	C11	1.384(3)
N3	C15	1.368(2)	C7	C8	1.405(2)
N3	N2	1.317(2)	C7	B1	1.571(2)
N1	N2	1.345(2)	C11	C10	1.385(3)
N1	C14	1.345(2)	C8	C13	1.518(2)
N1	C13	1.470(2)	C4	C1	1.585(2)
C18	C19	1.397(2)	C4	C6	1.499(3)
C19	C22	1.440(2)	C4	C5	1.534(3)
C19	C20	1.399(2)	C1	C2	1.523(3)
C22	C23	1.341(2)	C1	C3	1.506(3)

Table 5 Bond Angles for COMPOUND 13A.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C18	O3	C24	121.73(13)	C10	C9	C8	121.38(16)
B1	O1	C1	108.90(13)	N1	C14	C15	104.95(14)
B1	O2	C4	109.75(13)	C11	C12	C7	121.71(16)
C16	C17	C18	119.15(15)	C12	C7	B1	116.19(15)
N2	N3	C15	108.58(14)	C8	C7	C12	117.56(15)
N2	N1	C13	119.84(13)	C8	C7	B1	126.22(15)
C14	N1	N2	110.90(14)	C12	C11	C10	120.16(16)
C14	N1	C13	129.24(14)	C9	C8	C7	120.03(15)
O3	C18	C17	116.77(14)	C9	C8	C13	117.70(15)
O3	C18	C19	121.00(14)	C7	C8	C13	122.27(15)
C17	C18	C19	122.23(15)	C11	C10	C9	119.13(16)
C18	C19	C22	118.17(15)	O2	C4	C1	103.69(13)
C18	C19	C20	117.97(15)	O2	C4	C6	107.95(16)
C20	C19	C22	123.85(15)	O2	C4	C5	105.33(16)
C23	C22	C19	120.22(15)	C6	C4	C1	115.43(17)
N3	C15	C16	122.05(15)	C6	C4	C5	110.1(2)
N3	C15	C14	108.10(14)	C5	C4	C1	113.49(16)
C14	C15	C16	129.84(15)	O1	C1	C4	103.83(13)
C20	C21	C16	120.69(16)	O1	C1	C2	105.92(15)
N3	N2	N1	107.46(13)	O1	C1	C3	107.31(15)
C17	C16	C15	120.53(14)	C2	C1	C4	113.37(15)
C17	C16	C21	119.43(15)	C3	C1	C4	114.52(17)
C21	C16	C15	120.04(15)	C3	C1	C2	111.06(18)
O3	C24	C23	117.34(15)	O1	B1	C7	125.34(16)
O4	C24	O3	116.49(15)	O2	B1	O1	113.68(15)
O4	C24	C23	126.16(16)	O2	B1	C7	120.98(16)
C22	C23	C24	121.44(15)	N1	C13	C8	112.10(13)
C21	C20	C19	120.54(15)				

Table 6 Torsion Angles for COMPOUND 13A.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O3	C18	C19	C22	-1.5(2)	C14	C15	C16	C17	-179.72(16)
O3	C18	C19	C20	179.28(14)	C14	C15	C16	C21	0.5(3)
O3	C24	C23	C22	0.3(2)	C12	C7	C8	C9	1.7(2)
O4	C24	C23	C22	179.84(16)	C12	C7	C8	C13	-178.69(15)
O2	C4	C1	O1	2.45(18)	C12	C7	B1	O1	-170.75(17)

O2 C4 C1 C2	116.91(17)	C12C7 B1 O2	9.2(2)
O2 C4 C1 C3	-114.23(18)	C12C11C10C9	1.7(3)
C17C18C19C22	179.38(14)	C7 C12C11C10	-0.4(3)
C17C18C19C20	0.2(2)	C7 C8 C13N1	-97.79(18)
N3 C15C16C17	1.5(2)	C11C12C7 C8	-1.3(3)
N3 C15C16C21	-178.33(14)	C11C12C7 B1	176.57(16)
N3 C15C14N1	0.32(17)	C8 C9 C10C11	-1.2(3)
C18O3 C24O4	177.49(14)	C8 C7 B1 O1	6.9(3)
C18O3 C24C23	-3.0(2)	C8 C7 B1 O2	-173.17(16)
C18C17C16C15	-179.91(14)	C10C9 C8 C7	-0.5(3)
C18C17C16C21	-0.1(2)	C10C9 C8 C13	179.89(16)
C18C19C22C23	-1.1(2)	C4 O2 B1 O1	4.0(2)
C18C19C20C21	-0.2(2)	C4 O2 B1 C7	-175.92(15)
C19C22C23C24	1.6(2)	C1 O1 B1 O2	-2.3(2)
C22C19C20C21	-179.38(15)	C1 O1 B1 C7	177.69(16)
C15N3 N2 N1	0.30(17)	B1 O1 C1 C4	-0.29(19)
N2 N3 C15C16	178.63(14)	B1 O1 C1 C2	-119.96(17)
N2 N3 C15C14	-0.39(18)	B1 O1 C1 C3	121.34(18)
N2 N1 C14C15	-0.15(18)	B1 O2 C4 C1	-3.85(19)
N2 N1 C13C8	-81.81(18)	B1 O2 C4 C6	-126.77(18)
C16C17C18O3	-179.16(13)	B1 O2 C4 C5	115.62(18)
C16C17C18C19	0.0(2)	B1 C7 C8 C9	-175.90(16)
C16C15C14N1	-178.60(15)	B1 C7 C8 C13	3.7(3)
C16C21C20C19	0.1(2)	C13N1 N2 N3	-178.61(13)
C24O3 C18C17	-177.25(13)	C13N1 C14C15	178.20(15)
C24O3 C18C19	3.6(2)	C6 C4 C1 O1	120.29(19)
C20C19C22C23	178.09(15)	C6 C4 C1 C2	-125.2(2)
C20C21C16C17	0.0(2)	C6 C4 C1 C3	3.6(2)
C20C21C16C15	179.86(14)	C5 C4 C1 O1	-111.28(18)
C9 C8 C13N1	81.84(18)	C5 C4 C1 C2	3.2(2)
C14N1 N2 N3	-0.09(18)	C5 C4 C1 C3	132.0(2)
C14N1 C13C8	99.97(19)		

Table 7 Hydrogen Atom Coordinates ($Å \times 10^4$) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for COMPOUND 13A.

Atom	*x*	*y*	*z*	U(eq)
H17	7651	760	10356	25
H22	7729	-5048	10296	27

H21	7063	-778	8393	27
H23	8257	-5414	11559	28
H20	7312	-3217	9036	27
H9	5017	7559	7995	32
H14	6771	1768	7745	26
H12	383	7123	6466	31
H11	-468	9281	7140	35
H10	1821	9482	7934	36
H13A	7139	4361	6911	28
H13B	7697	5491	7370	28
H2A	6245	81	6665	66
H2B	4626	-103	6180	66
H2C	3937	913	6872	66
H6A	1500	3975	4561	82
H6B	3302	2360	4508	82
H6C	3733	3922	4544	82
H3A	6744	2645	5063	71
H3B	6522	1009	5010	71
H3C	7919	1144	5610	71
H5A	1035	2303	6422	78
H5B	1495	1338	5715	78
H5C	-116	3062	5671	78

Experimental

Single crystals of $C_{24}H_{24}BN_3O_4$ **13a** were obtained from a solution of hexane and ethylacetate. A suitable crystal was selected and studied on a SuperNova, Dual, Cu at zero, Atlas diffractometer. The crystal was kept at 99.9(2) K during data collection. Using Olex2,[8] the structure was solved with the ShelXS[12] structure solution program using Direct Methods and refined with the ShelXL[12] refinement package using Least Squares minimisation.

Crystal structure determination of **13a**

Crystal Data for $C_{24}H_{24}BN_3O_4$ (M =429.27 g/mol): triclinic, space group P-1 (no. 2), a = 7.1065(4) Å, b = 9.0913(6) Å, c = 17.6850(11) Å, α = 81.994(5)°, β = 84.409(5)°, γ = 68.790(6)°, V = 1053.43(12) Å^3, Z = 2, T = 99.9(2) K, μ(CuKα) = 0.750 mm^{-1}, $Dcalc$ = 1.353 g/cm^3, 6836 reflections measured (10.116° ≤ 2Θ ≤ 136.484°), 3847 unique (R_{int} = 0.0249, R_{sigma} = 0.0340) which were used in all calculations. The final R_1 was 0.0443 (I > 2σ(I)) and wR_2 was 0.1232 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.
Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups

2.a Secondary CH2 refined with riding coordinates:

C13(H13A,H13B)

2.b Aromatic/amide H refined with riding coordinates:

C17(H17), C22(H22), C21(H21), C23(H23), C20(H20), C9(H9), C14(H14), C12(H12),

C11(H11), C10(H10)

2.c Idealised Me refined as rotating group:

C2(H2A,H2B,H2C), C6(H6A,H6B,H6C), C3(H3A,H3B,H3C), C5(H5A,H5B,H5C

9. References for ESI

1. D. D. Perrin and B. Dempsey, *Chapman and Hall: London*, 1974.
2. D. K. Scrafton, J. E. Taylor, M. F. Mahon, J. S. Fossey and T. D. James, *J. Org. Chem.*, 2008, **73**, 2871-2874.
3. K. Mulla, P. Dongare, N. Zhou, G. Chen, D. W. Thompson and Y. Zhao, *Org. Biomol. Chem.*, 2011, **9**, 1332-1336.
4. L. Zhu, S. H. Shabbir, M. Gray, V. M. Lynch, S. Sorey and E. V. Anslyn, *J. Am. Chem. Soc.*, 2006, **128**, 1222-1232.
5. F. Xu, L. Peng, A. Orita and J. Otera, *Org. Lett.*, 2012, **14**, 3970-3973.
6. T. Kaufmann, M. T. Gokmen, S. Rinnen, H. F. Arlinghaus, F. Du Prez and B. J. Ravoo, *J. Mater. Chem.*, 2012, **22**, 6190-6199.
7. M. Chtchigrovsky, A. Primo, P. Gonzalez, K. Molvinger, M. Robitzer, F. Quignard and F. Taran, *Angew. Chem.*, 2009, **121**, 6030-6034.
8. O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Crystallogr.*, 2009, **42**, 339-341.
9. L. Palatinus and G. Chapuis, *J. Appl. Crystallogr.*, 2007, **40**, 786-790.
10. L. Palatinus and A. van der Lee, *J. Appl. Crystallogr.*, 2008, **41**, 975-984.
11. L. Palatinus, S. J. Prathapa and S. van Smaalen, *J. Appl. Crystallogr.*, 2012, **45**, 575-580.
12. G. M. Sheldrick, *Acta Crystallogr., Sect. A*, 2008, **64**, 112-122.