Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

Synthesis of 1-perfluoroalkyl-3-heteroaryl Bicyclo[1.1.1]pentanes via Visible Light-Induced and Metal-free Perfluoroalkylation of [1.1.1]propellane

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Supporting Information

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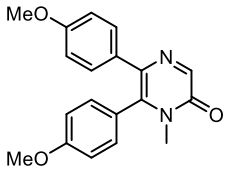
1. General information

Commercially available reagents and solvents were used without any purification. The purple LEDs light source (λ_{max} = 405 nm) was a commercial 1-meter LED strip. The progress of the reactions was monitored by TLC, and the visualization was carried out under UV light (254 nm). Melting points were determined using a Büchi B-540 capillary melting point apparatus. ^{1}H NMR spectra were recorded using a Bruker Avance III 400 MHz spectrometers. ^{13}C NMR spectra were recorded using a Bruker Advance III 100 MHz spectrometers. ^{19}F NMR spectra were recorded using a Bruker Advance III 376 MHz spectrometers. Chemical shifts of ^{1}H NMR were reported relative to the solvent signal ($CDCl_3$: δ = 7.26 ppm). Chemical shifts of ^{13}C NMR were reported relative to the solvent signal ($CDCl_3$: δ = 77.00 ppm). High-resolution mass spectra (HRMS) were recorded on Waters SYNAPT G2-S spectrometer. UV/vis studies were measured in a 1 cm quartz cuvette using a Shimadzu 2550 UV spectrophotometer. Column chromatography was performed on silica gel (300-400 mesh).

2. Synthesis of substrates 1

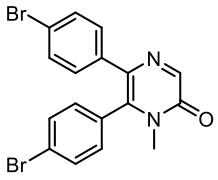
The substrates (**1a-1h, 1m, 1ae**, **1af**),[1] (**1i-1l**, **1n-1t**),[2] and (**1u-1ad**),[3] were prepared following the literature procedure, and the NMR data of all these compounds were compared with the corresponding reported data.

5,6-bis(4-methoxyphenyl)-1-methylpyrazin-2(1*H*)-one (1f)



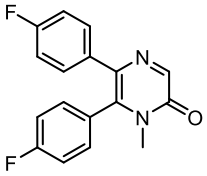
Eluent in chromatography: petroleum ether/ethyl acetate 10:1 to 5:1, **1f** was isolated as a yellow solid (437 mg, 21%); M.p.: 159-160 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.28 (s, 1H), 7.14 – 7.09 (m, 2H), 7.09 – 7.03 (m, 2H), 6.94 – 6.88 (m, 2H), 6.72 – 6.66 (m, 2H), 3.83 (s, 3H), 3.74 (s, 3H), 3.32 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 160.3, 158.5, 156.2, 146.2, 137.8, 133.9, 131.3, 130.3, 130.1, 124.4, 114.6, 113.3, 55.3, 55.1, 33.7. HRMS (ESI) m/z: calcd for $C_{19}H_{19}N_2O_3$ $[M+H]^+$ 323.1390, found: 323.1396.

5,6-bis(4-bromophenyl)-1-methylpyrazin-2(1*H*)-one (1g)



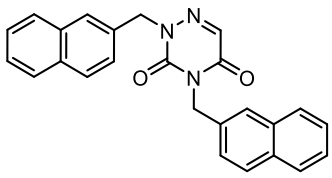
Eluent in chromatography: petroleum ether/ethyl acetate 8:1 to 3:1, **1g** was isolated as a yellow solid (882 mg, 36%); M.p.: 197-198 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (s, 1H), 7.60 – 7.55 (m, 2H), 7.34 – 7.27 (m, 2H), 7.12 – 7.07 (m, 2H), 7.01 – 6.96 (m, 2H), 3.30 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.8, 147.2, 137.4, 136.1, 132.7, 132.6, 131.4, 131.2, 130.7, 130.7, 124.5, 121.6, 33.8. HRMS (ESI) m/z: calcd for $C_{17}H_{13}Br_2N_2O$ $[M+H]^+$ 418.9389, found: 418.9394.

5,6-bis(4-fluorophenyl)-1-methylpyrazin-2(1*H*)-one (1h)



Eluent in chromatography: petroleum ether/ethyl acetate 20:1 to 10:1, **1h** was isolated as a yellow solid (1.125 g, 56%); M.p.: 143-144 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.28 (s, 1H), 7.23 – 7.16 (m, 3H), 7.15 – 7.03 (m, 6H), 6.89 – 6.80 (m, 3H), 3.31 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.15 (d, *J* = 250.3 Hz), 161.83 (d, *J* = 246.2 Hz), 155.9, 146.9, 137.4, 133.3 (d, *J* = 3.4 Hz), 133.1, 131.9 (d, *J* = 8.4 Hz), 130.9 (d, *J* = 8.2 Hz), 128.0 (d, *J* = 3.8 Hz), 116.6 (d, *J* = 22.0 Hz), 114.9 (d, *J* = 21.7 Hz), 33.7. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -109.43, 114.37. HRMS (ESI) m/z: calcd for $C_{17}H_{13}F_2N_2O$ $[M+H]^+$ 299.0990, found: 299.0997.

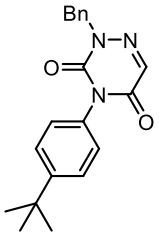
2,4-bis(naphthalen-2-ylmethyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (1x)



Eluent in chromatography: petroleum ether/ethyl acetate 15:1 to 5:1, **1x** was isolated as a yellow solid (2.649 g, 34%); M.p.: 136-137 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 (s, 1H), 7.87 – 7.76 (m, 7H), 7.61 – 7.56 (m, 1H), 7.52 – 7.43 (m, 6H), 5.27 (s, 2H), 5.25 (s, 2H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.9, 148.7, 134.6, 133.2 (d, *J* = 3.5 Hz), 133.1, 133.0, 132.7, 128.6, 128.6, 128.3, 128.0 (d, *J* = 1.5 Hz), 127.9, 127.7, 127.6, 126.9, 126.3 (d, *J* = 3.5 Hz), 126.2, 126.1, 55.6, 44.1. HRMS

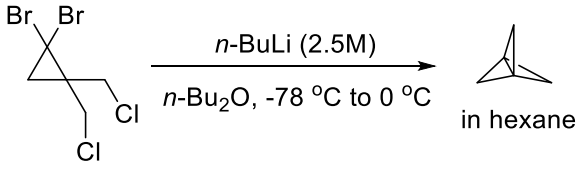
(ESI) m/z: calcd for $C_{25}H_{20}N_3O_2$ [M+H] $^+$ 394.1550, found: 394.1551.

2-benzyl-4-(4-(tert-butyl)phenyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (1ac)



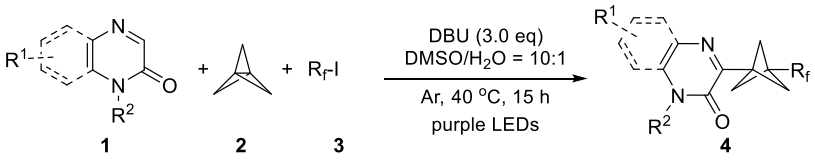
Eluent in chromatography: petroleum ether/ethyl acetate 20:1 to 10:1, **1ac** was isolated as a colourless oil (172 mg, 24%); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 – 7.52 (m, 3H), 7.50 – 7.44 (m, 2H), 7.42 – 7.37 (m, 2H), 7.36 – 7.28 (m, 3H), 5.16 (s, 2H), 1.34 (s, 9H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.7, 151.5, 148.3, 137.5, 135.3, 134.9, 129.8, 128.6, 128.2, 126.4, 125.9, 124.6, 114.7, 44.2, 34.7, 31.2. HRMS (ESI) m/z: calcd for $C_{20}H_{22}N_3O_2$ [M+H] $^+$ 336.1707, found: 336.1713.

3. Preparation of the solution of [1.1.1]propellane in hexane



A solution of *n*-BuLi (32 mL, 80 mmol, 2.0 equiv, 2.5M in hexane) was added dropwise to a suspension of 1,1-dibromo-2,2-bis(chloromethyl)cyclopropane (40 mmol) in anhydrous dibutyl ether (40 mL) under argon at –78 °C. After the addition was complete, the mixture was allowed to warm to 0 °C and stirred for 2 h before distillation under vacuum. The concentration can be measured by ^{1}H-NMR with 1,3,5-trimethoxybenzene as an internal standard (typically concentrations are 0.4-0.7 M)

4. General method



An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with **1** (0.2 mmol,

1.0 equiv.) and then evacuated and backfilled with Ar for three times. Afterwards, DMSO/H_2O = 10:1 (0.55 mL), DBU (0.6 mmol, 3.0 equiv.), perfluoroalkyl iodide (0.4 mmol, 2.0 equiv.), and **2** (0.3 mmol, 1.5 equiv.) were added under Ar atmosphere. The purple LEDs were turned on and the mixture was stirred under irradiation for 15 h at 40 °C. After the reaction was complete as monitored by TLC analysis, it was quenched with H_2O (3 mL) at room temperature, and then extracted with ethyl acetate (10 mL × 3). The organic layers were combined, washed with brine, dried over Na_2SO_4 filtered, and then concentrated in vacuum. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate to afford the desired product **4**.

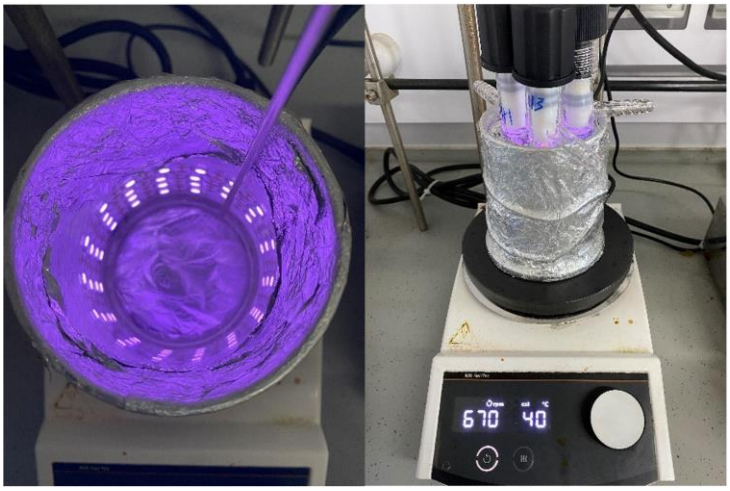


Figure **1** Reaction Set-Up

5. Gram-scale synthesis of compound 4u

An oven-dried 50 mL reaction tube equipped with a magnetic stir bar was charged with **1u** (3.0 mmol, 880 mg) and then evacuated and backfilled with Ar for three times. Afterwards, DMSO/H_2O = 10:1 (7.5 mL), DBU (9.0 mmol, 1.370 g), perfluoroalkyl iodide (6.0 mmol, 2.075 g.) and **2** (4.5 mmol, 1.5 equiv.) were added under Ar atmosphere. The purple LEDs were turned on and the mixture was stirred under irradiation for 15 h at 40 °C. After the reaction was complete as monitored by TLC analysis, it was quenched with H_2O (20 mL) at room temperature, and then extracted with ethyl acetate (30 mL × 3). The organic layers were combined, washed with brine, dried over Na_2SO_4 filtered, and then concentrated in vacuum. The residue was purified by flash chromatography on silica gel with petroleum ether/ethyl acetate to afford the desired product **4u** as a white solid (1.551 g, 89%).

6. Mechanistic experiments

6.1 UV/vis absorption spectra

UV/vis absorption spectra were measured in a 1 cm quartz cuvette using a Shimadzu 2550 UV spectrophotometer. Absorption spectra of individual reaction components and mixtures thereof were recorded. A bathochromic shift was observed for a mixture of **3a** (0.1 M, 1.0 equiv) and DBU (0.15 M, 1.5 equiv) in DMSO/H_2O = 10/1, which was a visibly intense yellow in color. This indicates the formation of an electron donor-acceptor (EDA) complex (Figure **2**).

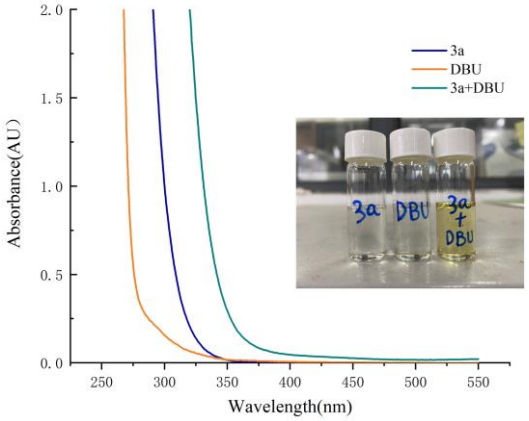


Figure **2.** UV/Vis absorption spectra of perfluorobutyl iodide **3a** (0.1 M), and DBU (0.15 M) in DMSO/H_2O = 10/1.

6.2 Job's analysis

The Job's plot of the EDA complex between **3a** and DBU was calculated by measuring the absorption of DMSO solutions at 405 nm with different donor/acceptor ratios with constant concentration (0.2 M) of the two components.[4] The absorbance values were plotted against the molar fraction (%) of DBU. The Job's plot analysis of the EDA complex between **3a** and DBU showed a maximal absorbance at 50% molar fraction of DBU indicated the 1:1 stoichiometry of the EDA complex in solution.

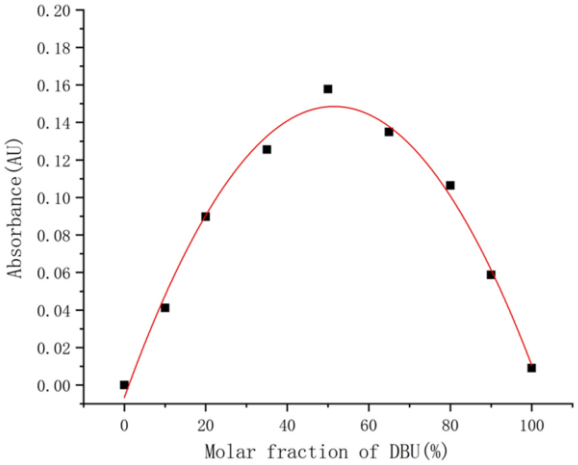
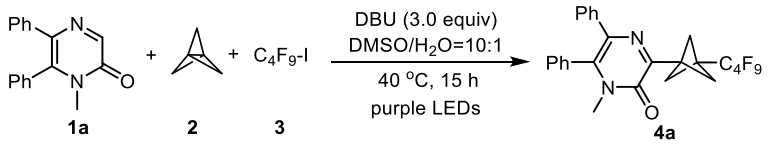
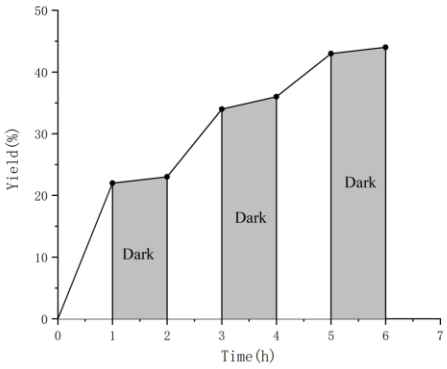


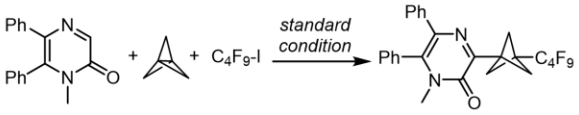
Figure **3.** Job plot for a mixture of perfluoroalkyl iodide **3a** and DBU in DMSO/H_2O = 10/1 (0.2 M).

6.3 Light dark experiment



Six oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with **1a** (0.2 mmol, 1.0 equiv.) and then evacuated and backfilled with Ar for three times. Afterwards, DMSO/H_2O = 10:1 (0.55 mL), DBU (0.6 mmol, 3.0 equiv.), perfluoroalkyl iodide (0.4 mmol, 2.0 equiv.), and **2** (0.3 mmol, 1.5 equiv.) were added under Ar atmosphere. The purple LEDs were turned on and the mixture was stirred under irradiation at 40 °C. After 1 h, the purple LEDs were turned off, and one vial was removed from the irradiation setup for analysis. The remaining five vials were stirred in the absence of light for an additional 1 h. Then, one vial was removed for analysis, and the purple LEDs were turned back on to irradiate the remaining four reaction mixtures. After an additional 1 h of irradiation, the purple LEDs were turned off, and one vial was removed for analysis. The remaining three vials were stirred in the absence of light for an additional 1 h. Then, one vial was removed for analysis, and the purple LEDs were turned back on to irradiate the remaining two reaction mixtures. After 1 h, the purple LEDs were turned off, and one vial was removed for analysis. The last vial was stirred in the absence of light for an additional 1 h, and then it was analyzed. The yield was determined by ^{1}H NMR analysis with dibromomethane as the internal standard.

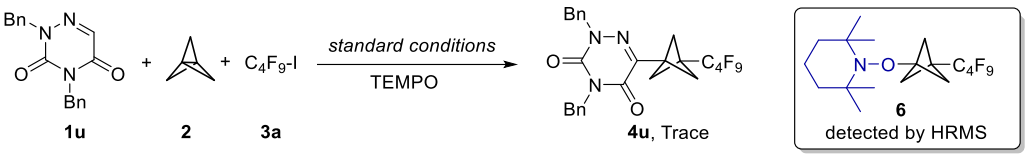




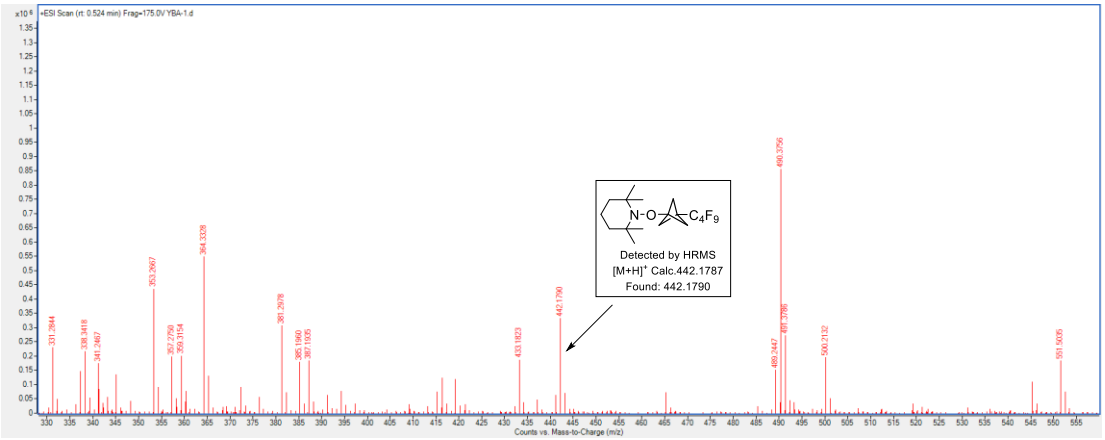
conditions	Yield (%)
w/o hv, 15 h	0
w/ hv (purple LED, 15 W), 1 h	22
w/ hv (purple LED, 15 W), 1 h Then w/o hv, 14 h	40

Figure **4.** The light/dark experiment was prepared according to substrate **1a**, with dibromomethane as ^{1}H NMR internal standard.

6.4 TEMPO trapping experiment



An oven-dried 10 mL reaction tube equipped with a magnetic stir bar was charged with **1u** (0.2 mmol, 1.0 equiv.), TEMPO (0.4 mmol, 2.0 equiv) and then evacuated and backfilled with Ar for three times. Afterwards, DMSO/H_2O = 10:1 (0.55 mL), DBU (0.6 mmol, 3.0 equiv.), perfluoroalkyl iodide **3** (0.4 mmol, 2.0 equiv.) and **2** (0.3 mmol, 1.5 equiv.) were added under Ar atmosphere. The purple LEDs were turned on and the mixture was stirred at 40 °C under irradiation for 15 h. Only trace amount of product was formed and the corresponding adducts **6** was detected by the HRMS. HRMS (ESI) m/z: calcd for $C_{18}H_{25}F_9NO$ $[M+H]^+$ 442.1787, found: 442.1790.



6.5 Quantum yield

The quantum yield of the reaction is defined as:

$$\Phi(\text{reaction at 405 nm}) = \frac{\text{mol of formed product}}{\text{mol of photon flux} \cdot t \cdot f} \qquad (1)$$

where Φ is the quantum yield of the reaction, t is the time of the reaction (s), and f is the incident light absorbed by the EDA complex at 405 nm. The photon flux is calculated by standard ferrioxalate actinometry[5] (see Section C).

A. Incident light absorbed by the EDA complex

The fraction of light, f, absorbed was determined according to equation 2:

$$F = 1 - 10^{-A} \qquad (2)$$

Where A is the absorbance of the EDA complex in DMSO at 405 nm. The wavelength of 405 nm was chosen based on the known absolute $\Phi(Fe^{2+})$ value. The absorbance of EDA complex was measured (0.1 M perfluorobutyl iodide **3a** and 0.15 M DBU) in DMSO (3 mL) to a cuvette equipped with a Teflon-coated magnetic stir bar and stirred for 30 seconds. The absorbance was recorded. The absorbance (A) at 405 nm was determined to be 0.056, thus indicating the fraction of light absorbed is ~0.122 according to equation 2.

B. Photoredox at 405 nm

Standard ferrioxalate actinometry was used to determine the photon flux of the spectrophotometer using equations 3 and 4. For the ferrioxalate actinometer ,the production of iron(II) ions proceeds by the following reactions:[6]

$$[Fe(C_2O_4)n]^{(3-2n)+} \xrightarrow{\text{light}} Fe^{2+} + (n-1)(C_2O_4)^{2-} + C_2O_4^{-}$$

$$[Fe(C_2O_4)n]^{(3-2n)+} + C_2O_4^{-} \longrightarrow Fe^{2+} + (n)(C_2O_4)^{2-} + 2\,CO_2$$

The moles of Fe^{2+} formed are determined spectrophotometrically by development with 1,10-phenanthroline (phen) to form the red $[Fe(phen)3]^{2+}$ moiety (λ = 510 nm).[6] The photon flux is defined as shown in equation 3:

$$\text{Photon flux} = \frac{\text{mol } Fe^{2+}}{\Phi(Fe^{2+}) \cdot t \cdot f} \qquad (3)$$

where Φ is the quantum yield for the ferrioxalate actinometer (1.188 at λ = 405 nm),[7] t is the time (s), f ~1, and the mol of Fe^{2+} are calculated according to equation 4.

$$\text{mol } (Fe^{2+}) = \frac{V \cdot \Delta A}{l \cdot \varepsilon} \qquad (4)$$

where V is the total volume of the solution, ΔA is the difference in absorbance between irradiated and nonirradiated solutions, l is the path length (1.0 cm), and ε is the molar absorptivity at 510 nm (11110 L mol^{-1} cm^{-1}).[6]

C. Experiment

The following solutions were prepared in the dark (flasks were wrapped in aluminum foil) and stored in the dark at room temperature:

− Ferrioxalate solution (0.15 M): Potassium ferrioxalate hydrate (2.211 g) was added to a flask wrapped in aluminum foil containing H_2SO_4 (30 mL, 0.05 M). The flask was stirred for complete solvation of the green solid in complete darkness. It is noteworthy that the solution should not be exposed to any incident light.

− Developer solution: 1,10-Phenanthroline (50 mg) and NaOAc (11.25 g) was added to a flask containing H_2SO_4 (50 mL, 0.5 M) and sonicated until completely solvated.

The absorbance of the non-irradiated sample. The buffered solution of phen (350 μL) was added to a ferrioxalate solution (2.0 mL) in a vial that had been covered with aluminum foil and with the lights of the laboratory switched off. The vial was capped and allowed to rest for 1 h and then transferred to a cuvette. The absorbance of the non-irradiated solution was measured at 510 nm to be 0.55 (average of two determinations).

The absorbance of the irradiated sample. In a cuvette equipped with a stir bar was added the ferrioxalate solution (2.0 mL), and the stirred solution was irradiated for 90 s at λ = 405 nm with an excitation slit width = 10.0 nm. After irradiation, the buffered phen solution (350 μL) was added to the cuvette and allowed to rest for 1 h in the dark to allow the ferrous ions to coordinate completely to phen. The absorbance was measured at 510 nm to be 1.09 (average of two determinations).

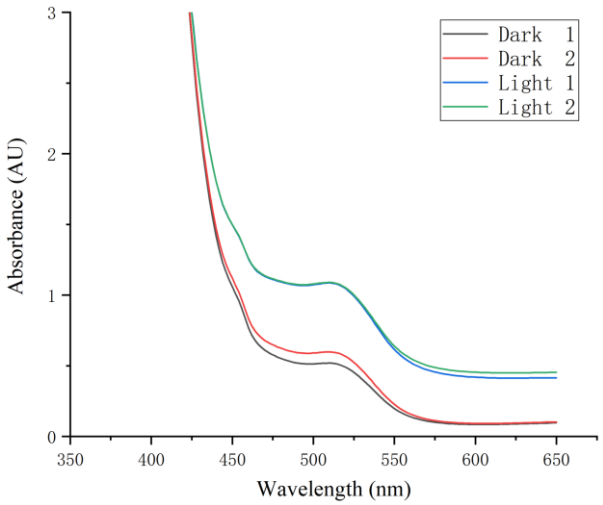


Figure **5**. Absorption spectra for irradiated and non-irradiated samples of red [Fe(phen)3] $^{2+}$

Photon flux sample calculation. Sample calculation:

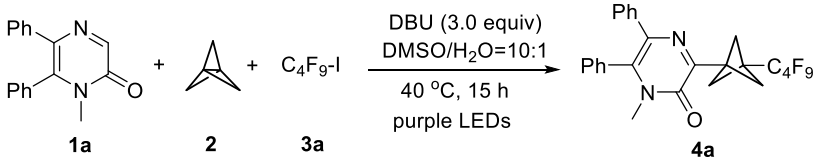
$$\text{mol } (Fe^{2+}) = \frac{V \cdot \Delta A}{1 \cdot \varepsilon} \qquad (4)$$

$$\text{mol } (Fe^{2+}) = \frac{0.00235 \text{ L} \cdot 0.54}{1.0 \text{ cm} \cdot 11100 \text{ L} \cdot \text{mol}^{-1}\text{cm}^{-1}} = 1.10 \times 10^{-7} \text{ mol}$$

$$\text{Photon flux} = \frac{\text{mol } Fe^{2+}}{\Phi\,(Fe^{2+}) \cdot t \cdot f} \qquad (3)$$

$$\text{Photon flux} = \frac{1.10 \times 10^{-7} \text{ mol}}{1.188 \cdot 90 \cdot 1} = 1.03 \times 10^{-9} \text{ einstein s}^{-1}$$

D. The photoredox reaction



The photoredox transformation was developed using the general procedure by purple LED (λ_{max} = 405 nm) for 1 h (3600 s). The yield of product was determined by ^{1}H NMR analysis using dibromomethane as an internal standard. The yield of **4a** was determined to be 22% (4.4×10^{-5} mol). The reaction quantum yield (Φ) was determined using equation 1 where the photon flux is 1.03×10^{-9} einsteins s^{-1} (see section C), t is the reaction time (1800 s) and f is the fraction of incident light absorbed by the EDA, determined using equation 2 (see Section A).

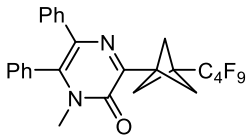
$$\Phi(\text{reaction at 405 nm}) = \frac{\text{mol of formed product}}{\text{mol of photon flux} \cdot t \cdot f} \quad (1)$$

$$\Phi(\text{reaction at 405 nm}) = \frac{4.4 \times 10^{-5}\ \text{mol}}{1.03 \times 10^{-9}\text{einstein s}^{-1} \cdot 3600 \cdot 0.122} = 97$$

The quantum yield studies indicate that this is a radical-chain process as evidenced by the Φ value. In other words, the quantum yield value indicated that 97 equivalents of product are formed for every photon absorbed, which is a result that could only be consistent with a radical chain mechanism.

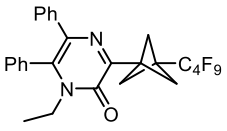
7. Characterization of products

1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4 λ ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5,6-diphenylpyrazin-2(1*H*)-one (**4a**)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4a** was isolated as a yellow solid (76 mg, 71%, **4a**:**4a-S** > 95:5); M.p.: 143-144 ºC; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.36 (m, 3H), 7.22 – 7.17 (m, 2H), 7.13 (s, 5H), 3.30 (s, 3H), 2.55 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 151.5, 138.0, 137.5, 132.6, 132.4, 129.9, 129.6, 129.2, 129.1, 127.7, 127.0, 50.9 (t, *J* = 3.6 Hz), 41.8, 37.8 (t, *J* = 30.0 Hz), 33.6. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.94 – -81.17 (m, 3F), -116.43 – -116.61 (m, 2F), -122.20 – -122.39 (m, 2F), -125.95 – -126.11 (m, 2F). HRMS (ESI) m/z: calcd for $C_{26}H_{20}F_9N_2O$ $[M+H]^+$ 547.1426, found: 547.1431. *Characteristic data for staffane* **4a-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.17 (s, 6H), 1.95 (s, 6H).

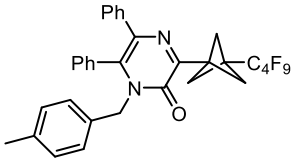
1-ethyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4 λ ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5,6-diphenylpyrazin-2(1*H*)-one (4b)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **4b** was isolated as a yellow solid (59 mg, 53%); M.p.: 65-66 ºC; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.35 (m, 3H), 7.25 – 7.21 (m, 2H), 7.12 (s, 5H), 3.87 (q, *J* = 7.0 Hz, 2H), 2.56 (s, 6H), 1.17 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100

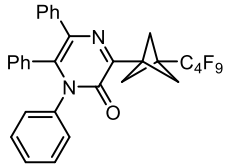
MHz, Chloroform-*d*) δ 154.6, 152.0, 137.8, 137.7, 132.8, 132.2, 130.1, 129.5, 129.2, 128.8, 127.6, 127.0, 51.0 (t, *J* = 3.4 Hz), 41.8, 41.2, 37.8 (t, *J* = 30.2 Hz), 13.6. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -81.07 (m, 3F), -116.56 (m, 2F), -122.32 (m, 2F), -126.03 (m, 2F). HRMS (ESI) m/z: calcd for $C_{27}H_{22}F_9N_2O$ $[M+H]^+$ 561.1583, found: 561.1588.

1-(4-methylbenzyl)-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5,6-diphenylpyrazin-2(1*H*)-one (4c)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4c** was isolated as a yellow solid (86 mg, 76%); M.p.: 116-117 ºC; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 – 7.33 (m, 1H), 7.30 – 7.22 (m, 2H), 7.16 – 7.08 (m, 5H), 7.06 – 6.98 (m, 4H), 6.76 (d, *J* = 8.1 Hz, 2H), 5.08 (s, 2H), 2.58 (s, 6H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.0, 152.4, 137.9, 137.6, 137.2, 133.0, 132.9, 132.0, 130.5, 129.5, 129.3, 129.1, 128.6, 127.6, 127.2, 127.0, 51.1 (t, *J* = 3.4 Hz), 48.5, 41.9, 37.9 (t, *J* = 30.0 Hz), 21.0. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.94 – -81.16 (m, 3F), -116.46 – -116.61 (m, 2F), -122.21 – -122.42 (m, 2F), -125.92 – -126.13 (m, 2F). HRMS (ESI) m/z: calcd for $C_{33}H_{26}F_9N_2O$ $[M+H]^+$ 637.1896, found: 637.1903.

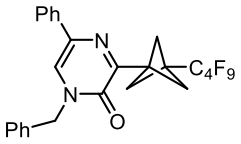
3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,5,6-triphenylpyrazin-2(1*H*)-one (4d)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **4d** was isolated as a colourless oil (34 mg, 28%, **4d**:**4d-S** > 95:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 – 7.40 (m, 4H), 7.33 – 7.16 (m, 11H), 2.61 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.8, 153.3, 147.5, 145.9, 139.5, 138.5, 137.7, 129.8, 129.7, 129.4, 128.5, 128.2, 128.2, 128.0, 124.8, 121.0, 51.3 (t, *J* = 2.9 Hz), 40.2, 38.1 (t, *J* = 30.3 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.93 – -81.14 (m, 3F), -116.36 – -116.64 (m, 2F), -122.10 – -122.42 (m, 2F), -125.92 – -126.14 (m, 2F). HRMS (ESI) m/z: calcd for $C_{31}H_{22}F_9N_2O$ $[M+H]^+$ 609.1583, found: 609.1591.

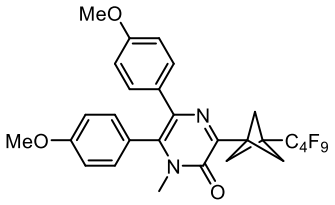
Characteristic data for staffane **4d-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.22 (s, 6H), 1.96 (s, 6H).

1-benzyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5-phenylpyrazin-2(1*H*)-one (4e)



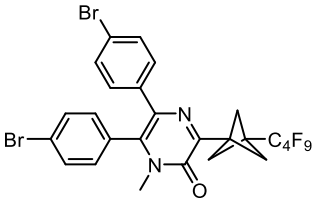
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4e** was isolated as a yellow oil (33 mg, 15%); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 – 7.67 (m, 2H), 7.45 (s, 1H), 7.43 – 7.29 (m, 8H), 5.14 (s, 2H), 2.55 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 154.6, 153.8, 135.6, 134.8, 132.7, 129.2, 128.8, 128.7, 128.5, 128.0, 124.9, 123.6, 52.1, 51.1 (t, *J* = 3.3 Hz), 41.9, 37.9 (t, *J* = 30.0 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.83 – -81.37 (m, 3F), -116.23 – -116.73 (m, 2F), -122.17 – -122.43 (m, 2F), -125.76 – -126.14 (m, 2F). HRMS (ESI) m/z: calcd for $C_{26}H_{20}F_9N_2O$ $[M+H]^+$ 547.1426, found: 547.1426.

5,6-bis(4-methoxyphenyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (4f)



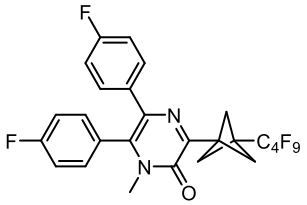
Eluent in chromatography: petroleum ether/ethyl acetate 20:1 to 10:1, **4f** was isolated as a yellow solid (52 mg, 43%); M.p.: 158-160 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.13 – 7.04 (m, 4H), 6.95 – 6.88 (m, 2H), 6.72 – 6.65 (m, 2H), 3.83 (s, 3H), 3.74 (s, 3H), 3.29 (s, 3H), 2.54 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 160.3, 158.5, 155.4, 151.1, 137.3, 132.7, 131.3, 130.4, 130.3, 124.8, 114.6, 113.2, 55.3, 55.1, 51.0 (t, *J* = 3.5 Hz), 41.8, 37.9 (t, *J* = 30.1 Hz), 33.5. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.95 – -81.22 (m, 3F), -116.35 – -116.62 (m, 2F), -122.11 – -122.47 (m, 2F), -125.85 – -126.13 (m, 2F). HRMS (ESI) m/z: calcd for $C_{28}H_{24}F_9N_2O_3$ $[M+H]^+$ 607.1638, found: 607.1638.

5,6-bis(4-bromophenyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (4g)



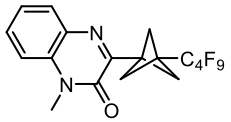
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4g** was isolated as a yellow solid (78 mg, 56%); M.p.: 173-174 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 8.0 Hz, 2H), 3.27 (s, 3H), 2.53 (s, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.0, 152.3, 136.8, 136.2, 132.7, 131.5, 131.4, 131.1, 131.0, 130.8, 124.4, 121.6, 50.9 (t, *J* = 3.2 Hz), 41.7, 37.9 (t, *J* = 30.0 Hz), 33.6. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.98 – -81.18 (m, 3F), -116.37 – -116.75 (m, 2F), -122.14 – -122.42 (m, 2F), -125.90 – -126.12 (m, 2F). HRMS (ESI) m/z: calcd for $C_{26}H_{18}Br_2F_9N_2O$ $[M+H]^+$ 702.9637, found: 702.9644.

5,6-bis(4-fluorophenyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (4h)



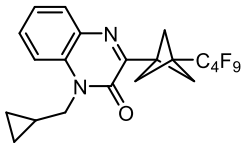
Eluent in chromatography: petroleum ether/ethyl acetate 20:1 to 15:1, **4h** was isolated as a yellow solid (74 mg, 65%); M.p.: 126-127 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.20 – 7.14 (m, 2H), 7.14 – 7.05 (m, 4H), 6.88 – 6.81 (m, 2H), 3.29 (s, 3H), 2.54 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.2 (d, *J* = 251.7 Hz), 161.9 (d, *J* = 247.6 Hz), 155.2, 152.1, 136.8, 133.6 (d, *J* = 3.4 Hz), 132.0 (d, *J* = 8.2 Hz), 131.0 (d, *J* = 8.2 Hz), 128.4 (d, *J* = 3.4 Hz), 116.6 (d, *J* = 22.0 Hz), 114.8 (d, *J* = 22.0 Hz), 51.0 (t, *J* = 3.4 Hz), 41.7, 38.0 (t, *J* = 30.0 Hz), 33.6. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.01 – -81.20 (m, 3F), -109.63 (s, 1F), -114.65 (s, 1F), -116.46 – -116.60 (m, 2F), -122.21 – -122.36 (m, 2F), -125.92 – -126.07 (m, 2F). HRMS (ESI) m/z: calcd for $C_{26}H_{18}F_{11}N_2O$ $[M+H]^+$ 583.1238, found: 583.1247.

1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4i)



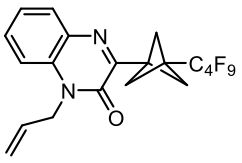
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4i** was isolated as a yellow solid (54 mg, 61%); M.p.: 77-78 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.85 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.59 – 7.52 (m, 1H), 7.38 – 7.27 (m, 2H), 3.67 (s, 3H), 2.55 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 154.8, 154.5, 133.5, 132.8, 130.4, 130.2, 123.7, 113.6, 51.2 (t, *J* = 3.5 Hz), 42.1, 37.9 (t, *J* = 30.0 Hz), 28.6. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -80.98 – -81.20 (m, 3F), -116.45 – -116.71 (m, 2F), -122.15 – -122.43 (m, 2F), -125.90 – -126.09 (m, 2F). HRMS (ESI) m/z: calcd for $C_{18}H_{14}F_9N_2O$ $[M+H]^+$ 445.0957, found: 445.0958.

1-(cyclopropylmethyl)-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4j)



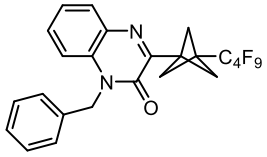
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4j** was isolated as a brown solid (55 mg, 57%); M.p.: 65-66 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.58 – 7.51 (m, 1H), 7.45 – 7.39 (m, 1H), 7.37 – 7.30 (m, 1H), 4.16 (d, *J* = 7.0 Hz, 2H), 2.55 (s, 6H), 1.34 – 1.20 (m, 1H), 0.61 – 0.51 (m, 4H). ^{13}C NMR (100 MHz, Chloroform-d) δ 155.0, 154.5, 133.0, 130.4, 130.3, 123.4, 113.9, 51.3 (t, *J* = 3.2 Hz), 45.8, 42.1, 37.9 (t, *J* = 30.2 Hz), 9.6, 4.2. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -81.05 – -81.17 (m, 3F), -116.50 – -116.69 (m, 2F), -122.21 – -122.41 (m, 2F), -125.90 – -126.08 (m, 2F). HRMS (ESI) m/z: calcd for $C_{21}H_{18}F_9N_2O$ $[M+H]^+$ 485.1270, found: 485.1273.

1-allyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4k)



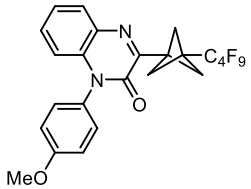
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4k** was isolated as a brown solid (49 mg, 52%); M.p.: 54-55 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.86 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.55 – 7.48 (m, 1H), 7.37 – 7.24 (m, 2H), 5.98 – 5.87 (m, 1H), 5.31 – 5.24 (m, 1H), 5.22 – 5.14 (m, 1H), 4.90 – 4.84 (m, 2H), 2.56 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-d) δ 154.9, 154.0, 132.9, 132.8, 130.5, 130.3, 130.3, 123.7, 118.3, 114.2, 51.3 (t, *J* = 3.4 Hz), 44.2, 42.0, 37.9 (t, *J* = 30.2 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -80.98 – -81.25 (m, 3F), -116.49 – -116.68 (m, 2F), -122.20 – -122.41 (m, 2F), -125.87 – -126.11 (m, 2F). HRMS (ESI) m/z: calcd for $C_{20}H_{16}F_9N_2O$ $[M+H]^+$ 471.1113, found: 471.1111.

1-benzyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4l)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4l** was isolated as a brown solid (59 mg, 58%); M.p.: 98-99 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.90 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.50 – 7.43 (m, 1H), 7.40 – 7.24 (m, 7H), 5.50 (s, 2H), 2.64 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-d) δ 155.0, 154.5, 135.1, 133.0, 132.9, 130.4, 130.3, 129.0, 127.7, 126.9, 123.7, 114.4, 51.3 (t, *J* = 3.4 Hz), 45.6, 42.1, 37.9 (t, *J* = 30.2 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -80.97 – -81.19 (m, 3F), -116.46 – -116.62 (m, 2F), -122.19 – -122.36 (m, 2F), -125.88 – -126.04 (m, 2F). HRMS (ESI) m/z: calcd for $C_{24}H_{18}F_9N_2O$ $[M+H]^+$ 521.1270, found: 521.1276.

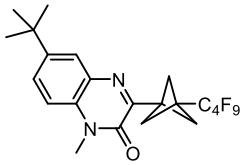
1-(4-methoxyphenyl)-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4m)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4m** was isolated as a brown solid (61 mg, 57%); M.p.: 109-110 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.91 – 7.85 (m, 1H), 7.39 – 7.28 (m, 2H), 7.23 – 7.16 (m, 2H), 7.15 – 7.08 (m, 2H), 6.77 – 6.72 (m, 1H), 3.89 (s, 3H), 2.57 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-d) δ 160.2, 155.6, 154.5, 134.7, 132.7, 130.0, 129.8, 129.2, 127.8, 123.8, 115.6, 115.6, 55.6, 51.4 (t, *J* = 3.5 Hz), 42.0, 37.9 (t, *J* = 30.2 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could

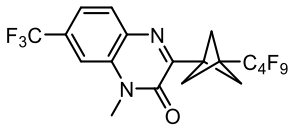
not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -81.01 – -81.18 (m, 3F), -116.48 – -116.64 (m, 2F), -122.24 – -122.38 (m, 2F), -125.89 – -126.05 (m, 2F). HRMS (ESI) m/z: calcd for $C_{24}H_{18}F_9N_2O_2$ $[M+H]^+$ 537.1219, found: 537.1220.

6-(tert-butyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4n)



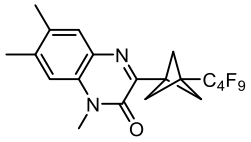
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4n** was isolated as a yellow solid (51 mg, 52%); M.p.: 107-108 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.85 (d, *J* = 2.3 Hz, 1H), 7.61 (dd, *J* = 8.8, 2.3 Hz, 1H), 7.24 (d, *J* = 8.8 Hz, 1H), 3.66 (s, 3H), 2.56 (s, 6H), 1.39 (s, 9H). ^{13}C NMR (100 MHz, Chloroform-d) δ 154.7, 154.5, 147.2, 132.5, 131.2, 128.2, 126.6, 113.3, 51.2 (t, *J* = 3.7 Hz), 42.1, 37.9 (t, *J* = 30.1 Hz), 34.5, 31.3, 28.6. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -81.00 – -81.20 (m, 3F), -116.46 – -116.67 (m, 2F), -122.19 – -122.40 (m, 2F), -125.90 – -126.07 (m, 2F). HRMS (ESI) m/z: calcd for $C_{22}H_{22}F_9N_2O$ $[M+H]^+$ 501.1583, found: 501.1593.

1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-7-(trifluoromethyl)quinoxalin-2(1*H*)-one (4o)



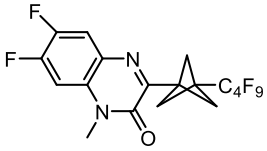
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4o** was isolated as a pink solid (34 mg, 33%); M.p.: 94-95 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.96 (d, *J* = 8.3 Hz, 1H), 7.61 – 7.55 (m, 1H), 7.53 (s, 1H), 3.70 (s, 3H), 2.56 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-d) δ 157.5, 154.1, 134.4, 133.6, 132.0 (q, *J* = 33.0 Hz), 131.0, 123.6 (q, *J* = 272.7 Hz), 120.2 (q, *J* = 3.6 Hz), 111.1 (q, *J* = 4.1 Hz), 51.2 (t, *J* = 3.3 Hz), 42.1, 38.0 (t, J = 30.3 Hz), 28.8. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -62.47 (s, 3F), -81.03 – -81.17 (m, 3F), -116.53 – -116.75 (m, 2F), -122.15 – -122.39 (m, 2F), -125.89 – -126.12 (m, 2F). HRMS (ESI) m/z: calcd for $C_{19}H_{13}F_{12}N_2O$ $[M+H]^+$ 513.0831, found: 513.0829.

1,6,7-trimethyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4p)



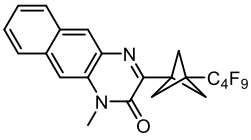
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4p** was isolated as a yellow solid (43 mg, 45%); M.p.: 109-110 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.61 (s, 1H), 7.05 (s, 1H), 3.64 (s, 3H), 2.54 (s, 6H), 2.41 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 154.6, 153.5, 140.4, 132.7, 131.5, 131.2, 130.3, 114.2, 51.2 (t, *J* = 3.6 Hz), 42.1, 37.8 (t, *J* = 30.0 Hz), 28.5, 20.5, 19.0. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.88 – -81.25 (m, 3F), -116.41 – -116.71 (m, 2F), -122.11 – -122.45 (m, 2F), -125.85 – -126.13 (m, 2F). HRMS (ESI) m/z: calcd for $C_{20}H_{18}F_9N_2O$ $[M+H]^+$ 473.1270, found: 473.1280.

6,7-difluoro-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4q)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4q** was isolated as a white solid (35 mg, 37%); M.p.: 112-113 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 7.66 (dd, *J* = 12.0, 8.0 Hz, 1H), 7.09 (dd, *J* = 12.0, 8.0 Hz, 1H), 3.62 (s, 3H), 2.53 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.4 (d, *J* = 3.5 Hz), 154.0, 151.6 (dd, *J* = 254.2, 14.4 Hz), 146.7 (dd, *J* = 247.4, 14.0 Hz), 130.8 (dd, *J* = 9.0, 1.8 Hz), 129.0 (dd, *J* = 9.2, 3.0 Hz), 117.8 (dd, *J* = 18.0, 2.2 Hz), 102.3 (d, *J* = 23.2 Hz), 51.2 (t, *J* = 3.4 Hz), 41.9, 38.0 (t, *J* = 30.2 Hz), 29.1. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -81.03 – -81.19 (m, 3F), -116.54 – -116.74 (m, 2F), -122.21 – -122.38 (m, 2F), -125.94 – -126.10 (m, 2F), -130.14 (d, *J* = 22.4 Hz), -141.87 (d, *J* = 22.4 Hz). HRMS (ESI) m/z: calcd for $C_{18}H_{12}F_{11}N_2O$ $[M+H]^+$ 481.0768, found: 481.0770.

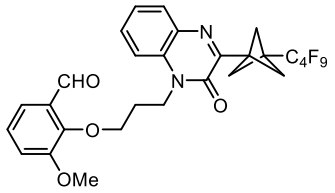
1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)benzo[g]quinoxalin-2(1*H*)-one (4r)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4r** was isolated as a yellow solid (43 mg, 43%, **4r**:**4r-S** > 95:5); M.p.: 105-107 ºC; ^{1}H NMR (400 MHz, Chloroform-d) δ 8.35 (s, 1H), 7.96 (d, *J* = 8.2 Hz, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.61 – 7.53 (m, 2H), 7.51 – 7.45 (m, 1H), 3.71 (s, 3H), 2.60 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.4, 154.3, 133.8, 132.0, 131.9, 129.8, 129.5, 128.5, 128.1, 127.2, 125.4, 110.0, 51.4 (t, *J* = 3.0 Hz), 42.2, 37.9 (t, *J* = 30.0 Hz), 28.6. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-d) δ -80.93 – -81.18 (m, 3F), -116.47 – -116.75 (m, 2F), -122.14 – -122.41 (m, 2F), -125.86 – -126.14 (m, 2F). HRMS (ESI) m/z: calcd for $C_{22}H_{16}F_9N_2O$ $[M+H]^+$ 495.1113, found: 495.1112.

Characteristic data for staffane **4r-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.21 (s, 6H), 1.97 (s, 6H).

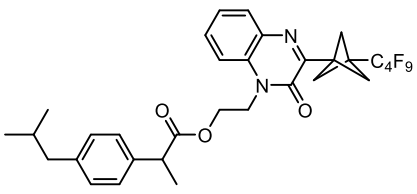
3-methoxy-2-(3-(3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-oxoquinoxalin-1(2*H*)-yl)propoxy)benzaldehyde (4s)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **4s** was isolated as a yellow oil (57 mg, 46%, **4s**:**4s-S** > 85:15); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.52 – 10.43 (m, 1H), 7.98 (d, *J* = 8.2 Hz, 1H), 7.86 – 7.77 (m, 1H), 7.68 – 7.49 (m, 2H), 7.47 – 7.39 (m, 1H), 7.19 – 7.09 (m, 2H), 4.84 – 4.71 (m, 2H), 4.44 – 4.31 (m, 2H), 3.84 (s, 3H), 2.54 (s, 6H), 2.44 – 2.35 (m, 2H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 189.9, 156.3, 152.9, 151.5, 145.1, 140.2, 138.5, 129.8, 129.7, 128.6, 126.8, 126.7, 124.2, 119.4, 118.0, 71.6, 63.3, 55.9, 51.2 (t, *J* = 3.6 Hz), 41.1, 38.0 (t, *J* = 30.1 Hz), 29.7. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.74 – -81.31 (m, 3F), -116.27 – -116.79 (m, 2F), -122.08 – -122.51 (m, 2F), -125.82 – -126.27 (m, 2F). HRMS (ESI) m/z: calcd for $C_{28}H_{24}F_9N_2O_4$ $[M+H]^+$ 623.1587, found: 623.1589.

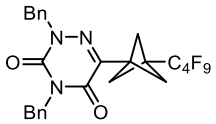
Characteristic data for staffane **4s-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.15 (s, 6H), 1.95 (s, 6H).

2-(3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-oxoquinoxalin-1(2*H*)-yl)ethyl 2-(4-isobutylphenyl)propanoate (4t)



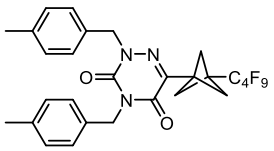
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4t** was isolated as a light brown oil (66 mg, 45%); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 7.9 Hz, 1H), 7.52 – 7.43 (m, 1H), 7.39 – 7.29 (m, 2H), 7.12 – 7.00 (m, 4H), 4.55 – 4.32 (m, 4H), 3.59 (q, *J* = 7.2 Hz, 1H), 2.54 (s, 6H), 2.43 (d, *J* = 7.1 Hz, 2H), 1.89 – 1.75 (m, 1H), 1.42 (d, *J* = 7.1 Hz, 3H), 0.89 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 174.7, 154.6, 154.2, 140.7, 137.1, 132.9, 132.8, 130.5, 130.4, 129.4, 127.0, 123.8, 113.8, 61.0, 51.2 (t, *J* = 3.5 Hz), 45.0 (d, *J* = 1.8 Hz), 41.9, 40.5, 37.8 (t, *J* = 30.2 Hz), 30.1, 22.4, 18.3. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.97 – -81.16 (m, 3F), -116.54 – -116.75 (m, 2F), -122.19 – -122.44 (m, 2F), -125.89 – -126.19 (m, 2F). HRMS (ESI) m/z: calcd for $C_{32}H_{32}F_9N_2O_3$ $[M+H]^+$ 663.2264, found: 663.2268.

2,4-dibenzyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4u)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **4u** was isolated as a white solid (97 mg, 83%); M.p.: 110-111 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.48 (m, 2H), 7.45 – 7.27 (m, 8H), 5.10 (s, 2H), 5.06 (s, 2H), 2.42 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.2, 149.0, 140.6, 135.5 (2C), 129.5, 128.8, 128.7, 128.6, 128.3, 128.1, 55.4, 51.0 (t, *J* = 3.3 Hz), 44.1, 38.8, 38.2 (t, *J* = 30.0 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -81.00 – -81.18 (m, 3F), -116.52 – -116.79 (m, 2F), -122.18 – -122.44 (m, 2F), -125.93 – -126.16 (m, 2F). HRMS (ESI) m/z: calcd for $C_{26}H_{21}F_9N_3O_2$ $[M+H]^+$ 578.1485, found: 578.1506.

2,4-bis(4-methylbenzyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4v)

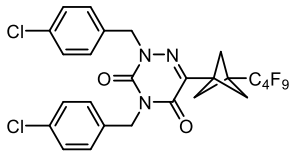


Eluent in chromatography: petroleum ether/ethyl acetate 30:1, **4v** was isolated as a yellow oil (110 mg, 91%, **4v**:**4v-S** > 95:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.07 (s, 2H), 5.03 (s, 2H), 2.43 (s, 6H), 2.37 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 148.9, 140.5, 138.1, 137.9, 132.6, 132.5, 129.6, 129.4, 129.2, 128.8, 55.2, 51.0 (t, *J* = 3.4 Hz), 43.8, 38.8, 38.2 (t, *J* = 30.2 Hz), 21.1. ^{13}C-

NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.93 – -81.23 (m, 3F), -116.44 – -116.72 (m, 2F), -122.15 – -122.42 (m, 2F), -125.90 – -126.10 (m, 2F). HRMS (ESI) m/z: calcd for $C_{28}H_{25}F_9N_3O_2$ $[M+H]^+$ 606.1798, found: 606.1805.

Characteristic data for staffane **4v-S:** ^1H NMR (400 MHz, Chloroform-*d*) δ 2.04 (s, 6H), 1.95 (s, 6H).

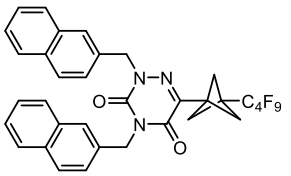
2,4-bis(4-chlorobenzyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4w)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4w** was isolated as a yellow solid (108 mg, 84%, **4w**:**4w-S** > 96:4); M.p.: 99-100 °C; ^1H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.38 (m, 2H), 7.32 (s, 4H), 7.30 – 7.26 (m, 2H), 5.04 (s, 2H), 5.00 (s, 2H), 2.40 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.1, 148.8, 140.9, 134.5, 134.2, 133.8 (2C), 131.1, 130.2, 129.0, 128.8, 54.8, 51.1 (t, *J* = 3.5 Hz), 43.4, 38.8, 38.2 (t, *J* = 30.4 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.93 – -81.23 (m, 3F), -116.54 – -116.69 (m, 2F), -122.19 – -122.38 (m, 2F), -125.93 – -126.10 (m, 2F). HRMS (ESI) m/z: calcd for $C_{26}H_{19}Cl_2F_9N_3O_2$ $[M+H]^+$ 646.0705, found: 646.0686.

Characteristic data for staffane **4w-S:** ^1H NMR (400 MHz, Chloroform-*d*) δ 2.00 (s, 6H), 1.92 (s, 6H).

2,4-bis(naphthalen-2-ylmethyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4x)

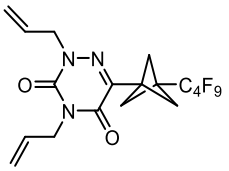


Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4x** was isolated as a yellow oil (82 mg, 62%, **4x**:**4x-S** > 96:4); ^1H NMR (400 MHz, Chloroform-*d*) δ 7.97 (s, 1H), 7.90 – 7.79 (m, 7H), 7.66 – 7.60 (m, 1H), 7.56 – 7.45 (m, 5H), 5.28 (s, 2H), 5.24 (s, 2H), 2.44 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 149.1, 140.7, 133.3, 133.2, 133.1, 133.0, 132.9, 132.8, 128.8, 128.6, 128.3, 128.0, 127.7, 127.6, 127.1, 126.4, 126.4, 126.2, 126.2, 55.6, 51.1 (t, *J* = 3.8 Hz), 44.3, 38.8, 38.2 (t, *J* = 30.4 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.96 – -81.16 (m, 3F), -116.46 – -116.63 (m, 2F), -122.15 – -122.35 (m, 2F), -125.88 – -126.06 (m, 2F). HRMS

(ESI) m/z: calcd for $C_{34}H_{25}F_9N_3O_2$ [M+H] $^+$ 678.1798, found: 678.1806.

Characteristic data for staffane **4x-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.05 (s, 6H), 1.94 (s, 6H).

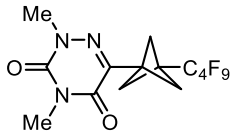
2,4-diallyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4y)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4y** was isolated as a brown solid (57 mg, 61%, **4y**:**4y-S** > 95:5); M.p.: 54-56 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.00 – 5.77 (m, 2H), 5.37 – 5.18 (m, 4H), 4.57 – 4.47 (m, 4H), 2.40 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.1, 148.5, 140.5, 131.2, 130.3, 119.5, 119.3, 54.2, 51.0, 42.8, 38.8, 38.2 (t, *J* = 30.4 Hz). ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -81.03 – -81.28 (m, 3F), -116.52 – -116.83 (m, 2F), -122.24 – -122.48 (m, 2F), -125.96 – -126.17 (m, 2F). HRMS (ESI) m/z: calcd for $C_{18}H_{17}F_9N_3O_2$ [M+H] $^+$ 478.1172, found: 478.1182.

Characteristic data for staffane **4y-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.00 (s, 6H), 1.91 (s, 6H).

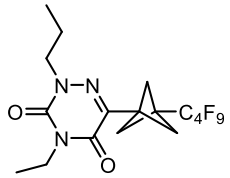
2,4-dimethyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4z)



Eluent in chromatography: petroleum ether/ethyl acetate 20:1 to 15:1, **4z** was isolated as a white solid (56 mg, 66%, **4z**:**4z-S** > 95:5); M.p.: 96-98 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 3.62 (s, 3H), 3.32 (s, 3H), 2.39 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.8, 149.2, 139.9, 50.9 (t, *J* = 3.4 Hz), 39.6, 38.8, 38.2 (t, *J* = 30.4 Hz), 26.9. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.95 – -81.18 (m, 3F), -116.53 – -116.83 (m, 2F), -122.17 – -122.48 (m, 2F), -125.94 – -126.21 (m, 2F). HRMS (ESI) m/z: calcd for $C_{14}H_{12}F_9N_3NaO_2$ [M+Na] $^+$ 448.0678, found: 448.0664.

Characteristic data for staffane **4z-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.00 (s, 6H), 1.91 (s, 6H).

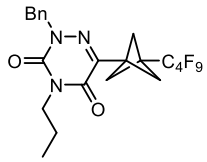
4-ethyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-propyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4aa)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1, **4aa** was isolated as a white solid (72 mg, 77%, **4aa**:**4aa-S** > 94:6); M.p.: 62-63 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.00 (q, *J* = 7.4 Hz, 2H), 3.85 (t, *J* = 7.8 Hz, 2H), 2.39 (s, 6H), 1.65 (q, *J* = 7.6 Hz, 2H), 1.31 (t, 3H), 0.94 (t, *J* = 8.2 Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.5, 148.6, 140.1, 51.0 (t, *J* = 3.6 Hz), 47.0, 42.3, 38.9, 38.1 (t, *J* = 30.6 Hz), 20.6, 13.3, 11.3. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -81.00 – -81.23 (m, 3F), -116.57 – -116.85 (m, 2F), -122.24 – -122.51 (m, 2F), -125.95 – -126.24 (m, 2F). HRMS (ESI) m/z: calcd for $C_{17}H_{19}F_9N_3O_2$ $[M+H]^+$ 468.1328, found: 468.1328.

Characteristic data for staffane **4aa-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.00 (s, 6H), 1.91 (s, 6H).

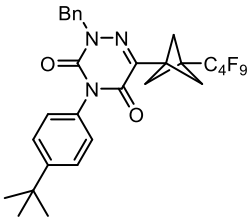
2-benzyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-4-propyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4ab)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4ab** was isolated as a yellow oil (100 mg, 78%, **4ab**:**4ab-S** > 96:4); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 – 7.46 (m, 2H), 7.36 – 7.28 (m, 3H), 5.07 (s, 2H), 3.96 – 3.85 (m, 2H), 2.40 (s, 6H), 1.81 – 1.70 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 149.0, 140.2, 135.6, 129.6, 128.6, 128.1, 53.4, 51.0 (t, *J* = 3.6 Hz), 44.0, 38.8, 38.2 (t, *J* = 30.2 Hz), 21.50, 10.91. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.73 – -81.30 (m, 3F), -116.51 – -116.71 (m, 2F), -122.20 – -122.42 (m, 2F), -125.93 – -126.11 (m, 2F). HRMS (ESI) m/z: calcd for $C_{22}H_{21}F_9N_3O_2$ $[M+H]^+$ 530.1485, found: 530.1495.

Characteristic data for staffane **4ab-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.00 (s, 6H), 1.92 (s, 6H).

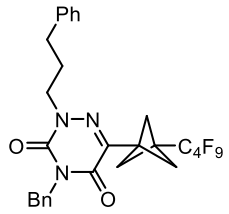
2-benzyl-4-(4-(tert-butyl)phenyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4ac)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **4ac** was isolated as a yellow solid (52 mg, 42%, **4ac**:**4ac-S** > 96:4); M.p.: 129-130 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 7.8 Hz, 2H), 7.48 (d, *J* = 7.6 Hz, 2H), 7.43 – 7.29 (m, 5H), 5.14 (s, 2H), 2.44 (s, 6H), 1.35 (s, 9H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.1, 151.4, 148.6, 141.0, 137.6, 135.4, 130.0, 128.6, 128.3, 125.9, 124.6, 51.1 (t, *J* = 3.2 Hz), 44.3, 38.9, 38.2 (t, *J* = 30.2 Hz), 34.7, 31.3. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.95 – -81.17 (m, 3F), -116.51 – -116.82 (m, 2F), -122.20 – -122.45 (m, 2F), -125.96 – -126.14 (m, 2F). HRMS (ESI) m/z: calcd for $C_{29}H_{27}F_9N_3O_2$ $[M+H]^+$ 620.1954, found: 620.1958.

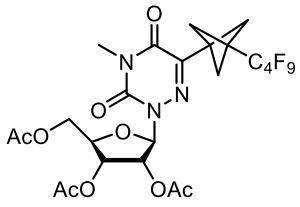
Characteristic data for staffane **4ac-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.05 (s, 6H), 1.92 (s, 6H).

4-benzyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-(3-phenylpropyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4ad)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **4ad** was isolated as a colorless liquid (92 mg, 76%); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.31 (m, 5H), 7.27 – 7.20 (m, 2H), 7.20 – 7.13 (m, 3H), 5.06 (s, 2H), 4.00 – 3.91 (m, 2H), 2.69 (t, *J* = 7.8 Hz, 2H), 2.40 (s, 6H), 2.04 – 1.96 (m, 2H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 148.8, 140.9, 140.3, 135.5, 128.8, 128.7, 128.4, 128.3, 128.2, 126.0, 55.4, 51.0 (t, *J* = 3.6 Hz), 40.8, 38.8, 38.2 (t, *J* = 30.4 Hz), 33.2, 28.1. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -81.00 – -81.17 (m, 3F), -116.52 – -116.78 (m, 2F), -122.20 – -122.43 (m, 2F), -125.94 – -126.14 (m, 2F). HRMS (ESI) m/z: calcd for $C_{28}H_{25}F_9N_3O_2$ $[M+H]^+$ 606.1798, found: 606.1805.

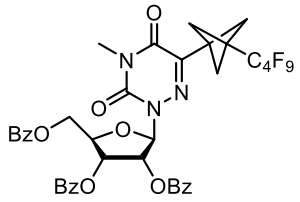
(2*R*,3*R*,4*R*,5*R*)-2-(acetoxymethyl)-5-(4-methyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)tetrahydrofuran-3,4-diyl diacetate (4ae)



Eluent in chromatography: petroleum ether/ethyl acetate 5:1 to 3:1, **4ae** was isolated as a colourless liquid (41 mg, 31%, **4ae**:**4ae-S** > 95:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.33 – 6.25 (m, 1H), 5.63 – 5.56 (m, 1H), 5.48 – 5.38 (m, 1H), 4.39 – 4.27 (m, 2H), 4.20 – 4.10 (m, 1H), 3.30 (s, 3H), 2.43 (s, 6H), 2.11 (s, 3H), 2.09 (s, 3H), 2.06 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.4, 169.6, 169.5, 154.9, 148.6, 141.7, 89.3, 78.8, 73.1, 70.5, 63.5, 51.0 (t, *J* = 3.6 Hz), 38.8, 38.3 (t, *J* = 30.6 Hz), 27.0, 20.7, 20.5, 20.4. ^{13}C-NMR for $CF_2CF_2CF_2CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.80 – -81.30 (m, 3F), -116.48 – -116.89 (m, 2F), -122.06 – -122.46 (m, 2F), -125.91 – -126.25 (m, 2F). HRMS (ESI) m/z: calcd for $C_{24}H_{25}F_9N_3O_9$ $[M+H]^+$ 670.1442, found: 670.1441.

Characteristic data for staffane **4ae-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.03 (s, 6H), 1.91 (s, 6H).

(2*R*,3*R*,4*R*,5*R*)-2-((benzoyloxy)methyl)-5-(4-methyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)tetrahydrofuran-3,4-diyl dibenzoate (4af)

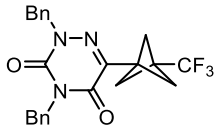


Eluent in chromatography: petroleum ether/ethyl acetate 15:1 to 8:1, **4af** was isolated as a colourless viscous liquid (83 mg, 49%, **4af**:**4af-S** > 95:5); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 (d, *J* = 7.7 Hz, 2H), 8.00 – 7.92 (m, 4H), 7.61 – 7.50 (m, 3H), 7.44 – 7.32 (m, 6H), 6.61 – 6.54 (m, 1H), 6.10 – 6.02 (m, 1H), 5.98 – 5.89 (m, 1H), 4.83 – 4.68 (m, 2H), 4.64 – 4.54 (m, 1H), 3.32 (s, 3H), 2.37 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 166.1, 165.2, 165.1, 154.9, 148.8, 141.8, 133.7, 133.6, 133.3, 129.8, 129.7, 129.7, 129.4, 128.7, 128.6, 128.5, 128.4 (2C), 89.4, 79.4, 73.5, 71.4, 63.8, 50.9 (t, *J* = 3.6 Hz), 38.7, 38.2 (t, *J* = 30.4 Hz), 27.0. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.90 – -81.19 (m, 3F), -116.37 – -116.80 (m, 2F), -122.00 – -122.42 (m, 2F), -125.88 – -126.24 (m, 2F). HRMS (ESI) m/z: calcd for $C_{39}H_{31}F_9N_3O_9$

[M+H] $^{+}$ 856.1911, found: 856.1902.

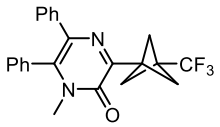
Characteristic data for staffane **4af-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.02 (s, 6H), 1.92 (s, 6H).

2,4-dibenzyl-6-(3-(trifluoromethyl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5a)



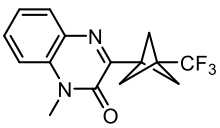
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **5a** was isolated as a white solid (48 mg, 55%); M.p.: 111-112 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 – 7.46 (m, 2H), 7.43 – 7.28 (m, 8H), 5.09 (s, 2H), 5.05 (s, 2H), 2.33 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 149.0, 140.8, 135.5, 135.5, 129.5, 128.8, 128.7, 128.6, 128.3, 128.1, 122.6 (q, *J* = 274.0 Hz), 55.4, 50.3 (q, *J* = 2.0 Hz), 44.1, 38.2 (q, *J* = 38.8 Hz), 38.2 – 38.1 (m). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -73.17. HRMS (ESI) m/z: calcd for $C_{23}H_{21}F_3N_3O_2$ [M+H] $^{+}$ 428.1580, found: 428.1579.

1-methyl-5,6-diphenyl-3-(3-(trifluoromethyl)bicyclo[1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (5b)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **5b** was isolated as a yellow solid (34 mg, 44%); M.p.: 160-162 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.35 (m, 3H), 7.22 – 7.17 (m, 2H), 7.13 (s, 5H), 3.30 (s, 3H), 2.48 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 151.7, 137.9, 137.5, 132.7, 132.4, 129.9, 129.6, 129.3, 129.1, 127.7, 127.0, 123.0 (q, *J* = 274.0 Hz), 50.2 (q, *J* = 2.3 Hz), 41.0, 37.8 (q, *J* = 38.2 Hz), 33.6. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -73.06. HRMS (ESI) m/z: calcd for $C_{23}H_{20}F_3N_2O$ [M+H] $^{+}$ 397.1522, found: 397.1527.

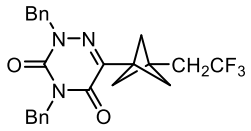
1-methyl-3-(3-(trifluoromethyl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (5c)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 20:1, **5c** was isolated as a yellow solid (35 mg, 39%); M.p.: 174-175 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.58 – 7.52 (m, 1H), 7.38 – 7.27 (m, 2H), 3.67 (s, 3H), 2.48 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.0, 154.5, 133.5, 132.8, 130.4, 130.2, 123.7, 122.9 (q, *J* = 274.0 Hz), 113.6, 50.4 (q, *J* = 2.0 Hz), 41.3, 37.9 (q, *J* = 38.3 Hz), 28.6. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -73.13. HRMS (ESI) m/z: calcd

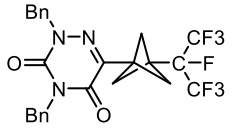
for $C_{15}H_{14}F_3N_2O$ $[M+H]^+$ 295.1053, found: 295.1060.

2,4-dibenzyl-6-(3-(2,2,2-trifluoroethyl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5d)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **5d** was isolated as a white solid (23 mg, 26%); M.p.: 86-88 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 – 7.45 (m, 2H), 7.42 – 7.27 (m, 8H), 5.07 (s, 2H), 5.04 (s, 2H), 2.36 (q, *J* = 11.0 Hz, 2H), 2.18 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.4, 149.0, 141.7, 135.6, 135.6, 129.5, 128.8, 128.7, 128.5, 128.2, 128.0, 126.2 (q, *J* = 277.0 Hz), 55.3, 53.0, 43.9, 39.3 (d, *J* = 1.9 Hz), 35.7 (q, *J* = 28.3 Hz), 34.7 (q, *J* = 2.8 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -64.29. HRMS (ESI) m/z: calcd for $C_{24}H_{23}F_3N_3O_2$ $[M+H]^+$ 442.1737, found: 442.1740.

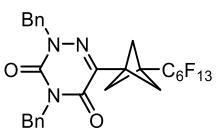
2,4-dibenzyl-6-(3-(perfluoropropan-2-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5e)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **5e** was isolated as a white solid (43 mg, 41%, **5e**:**5e-S** > 96:4); M.p.: 90-91 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 – 7.45 (m, 2H), 7.43 – 7.28 (m, 8H), 5.08 (s, 2H), 5.04 (s, 2H), 2.41 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 148.9, 140.5, 135.5, 135.4, 129.5, 128.8, 128.7, 128.6, 128.3, 128.1, 55.5, 52.0 (d, *J* = 4.0 Hz), 44.1, 38.7, 36.8 (d, *J* = 25.6 Hz). ^{13}C-NMR for $CFCF_3CF_3$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -74.66 (s, 3F), -74.69 (s, 3F), -183.16 – -183.32 (m, 1F). HRMS (ESI) m/z: calcd for $C_{25}H_{21}F_7N_3O_2$ $[M+H]^+$ 528.1517, found: 528.1522.

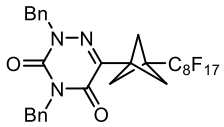
Characteristic data for staffane **5e-S:** ^{1}H NMR (400 MHz, Chloroform-*d*) δ 2.00 (s, 6H), 1.93 (s, 6H).

2,4-dibenzyl-6-(3-(6,6,6,6,6,6,6,6,6,6,6,6,6-tridecafluoro-6λ^{16}-hexa-1,3,5-triyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5f)



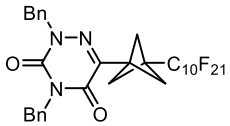
Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **5f** was isolated as a yellow solid (112 mg, 82%); M.p.: 98-99 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.49 (m, 2H), 7.46 – 7.28 (m, 8H), 5.11 (s, 2H), 5.07 (s, 2H), 2.43 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 149.0, 140.6, 135.5, 135.5, 129.6, 128.8, 128.7, 128.6, 128.3, 128.1, 55.5, 51.1 (t, *J* = 3.6 Hz), 44.1, 38.8, 38.3 (t, *J* = 30.4 Hz). ^{13}C-NMR for C_6F_{13} could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.75 – -81.13 (m, 3F), -116.32 – -116.51 (m, 2F), -121.19 – -121.40 (m, 2F), -121.73 – -122.05 (m, 2F), -122.72 – -123.05 (m, 2F), -126.01 – -126.20 (m, 2F). HRMS (ESI) m/z: calcd for $C_{28}H_{21}F_{13}N_3O_2$ $[M+H]^+$ 678.1421, found: 678.1404.

2,4-dibenzyl-6-(3-(8,8,8,8,8,8,8,8,8,8,8,8,8,8,8,8,8-heptadecafluoro-8λ^{20}-octa-1,3,5,7-tetrayn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5g)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **5g** was isolated as a white solid (53 mg, 34%); M.p.: 119-120 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 – 7.46 (m, 2H), 7.43 – 7.28 (m, 8H), 5.09 (s, 2H), 5.05 (s, 2H), 2.40 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 149.0, 140.7, 135.5, 135.5, 129.6, 128.8, 128.7, 128.6, 128.3, 128.1, 55.5, 51.1 (t, *J* = 3.2 Hz), 44.1, 38.8, 38.3 (t, *J* = 30.4 Hz). ^{13}C-NMR for C_8F_{17} could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.70 – -80.90 (m, 3F), -116.30 – -116.51 (m, 2F), -121.13 – -121.37 (m, 2F), -121.56 – -122.04 (m, 6F), -122.52 – -122.85 (m, 2F), -125.93 – -126.22 (m, 2F). HRMS (ESI) m/z: calcd for $C_{30}H_{21}F_{17}N_3O_2$ $[M+H]^+$ 778.1357, found: 778.1376.

2,4-dibenzyl-6-(3-(10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10-henicosafluoro-10λ^{24}-deca-1,3,5,7,9-pentayn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5h)



Eluent in chromatography: petroleum ether/ethyl acetate 30:1 to 25:1, **5h** was isolated as a white solid (26 mg, 15%); M.p.: 141-142 °C; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.51 – 7.45 (m, 2H), 7.42 – 7.28 (m, 8H), 5.08 (s, 2H), 5.05 (s, 2H), 2.40 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.3, 149.0, 140.6, 135.5, 135.5, 129.6, 128.8, 128.7, 128.6, 128.3, 128.1, 55.5, 51.1 (t *J* = 3.2 Hz), 44.1, 38.8, 38.3(t,

J = 31.2 Hz). ^{13}C-NMR for $C_{10}F_{21}$ could not be assigned. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -80.76 (t, J = 10.0 Hz, 3F), -116.25 – -116.53 (m, 2F), -121.09 – -121.37 (m, 2F), -121.53 – -121.97 (m, 10F), -122.49 – -122.81 (m, 2F), -125.91 – -126.19 (m, 2F). HRMS (ESI) m/z: calcd for $C_{32}H_{21}F_{21}N_3O_2$ $[M+H]^+$ 878.1293, found: 878.1301.

8. References

[1] P. Ghosh, N. Y. Kwon, S. Kim, S. Han, S. H. Lee, W. An, N. K. Mishra, S. B. Han, I. S. Kim, *Angew. Chem. Int. Ed. Engl.* **2021**, *60*, 191-196.

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[4] M. J. Cabrera-Afonso, A. Granados, G. A. Molander, *Angew. Chem. Int. Ed. Engl.* **2022**, *61*, e202202706.

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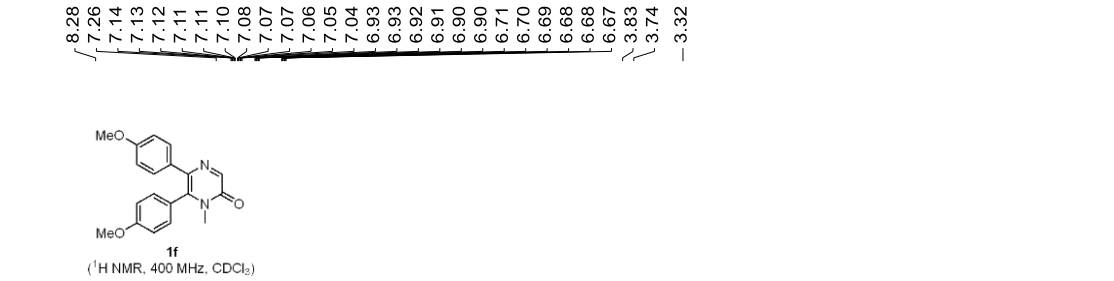
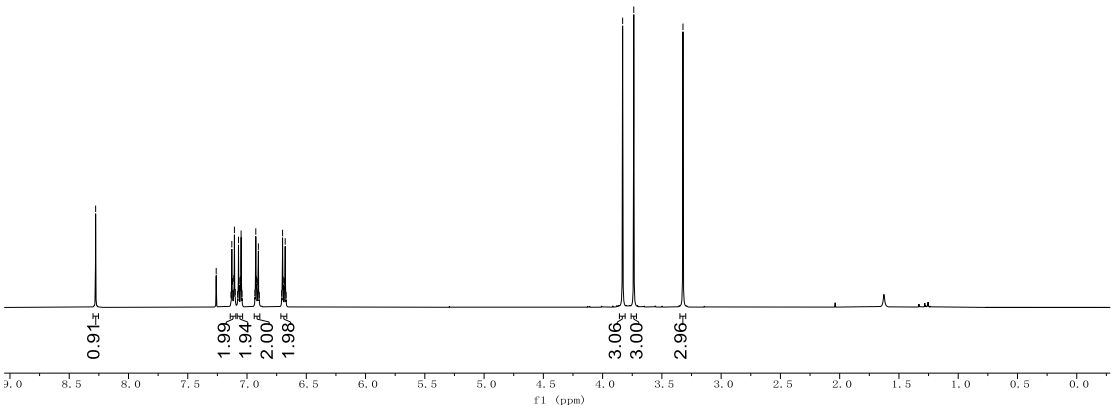
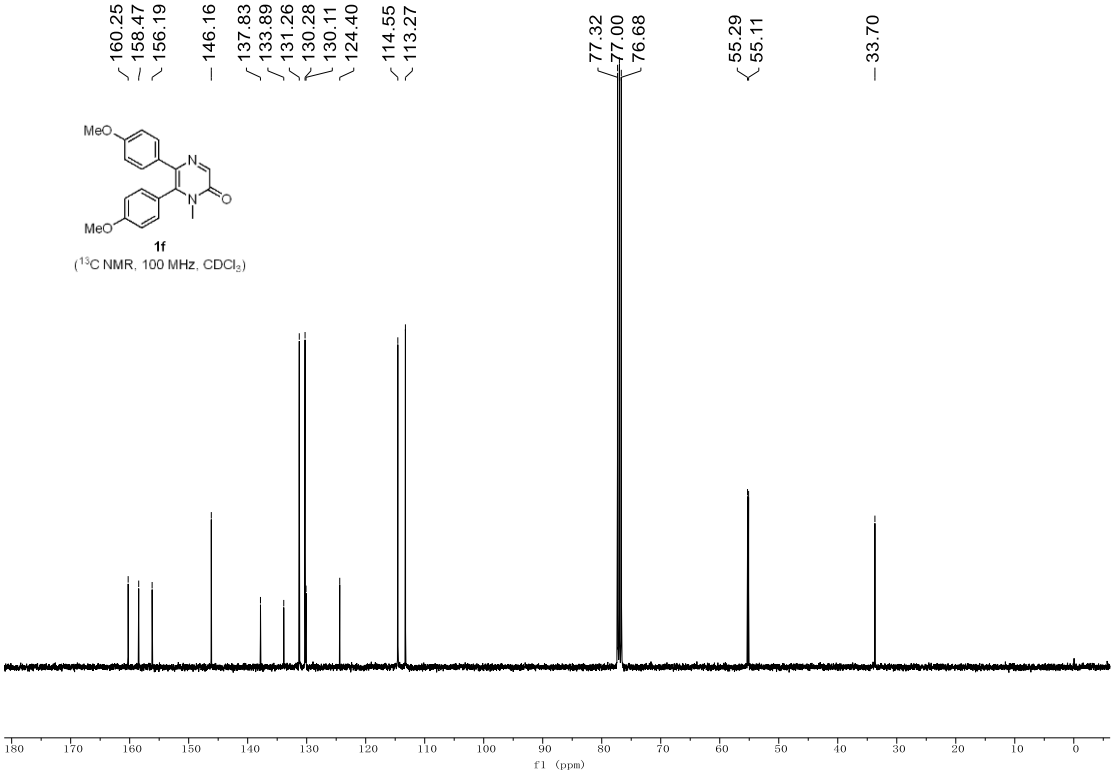
9. Copies of ^{1}H, ^{13}C NMR, and ^{19}F NMR spectra of all compounds

5,6-bis(4-methoxyphenyl)-1-methylpyrazin-2(1*H*)-one (1f)



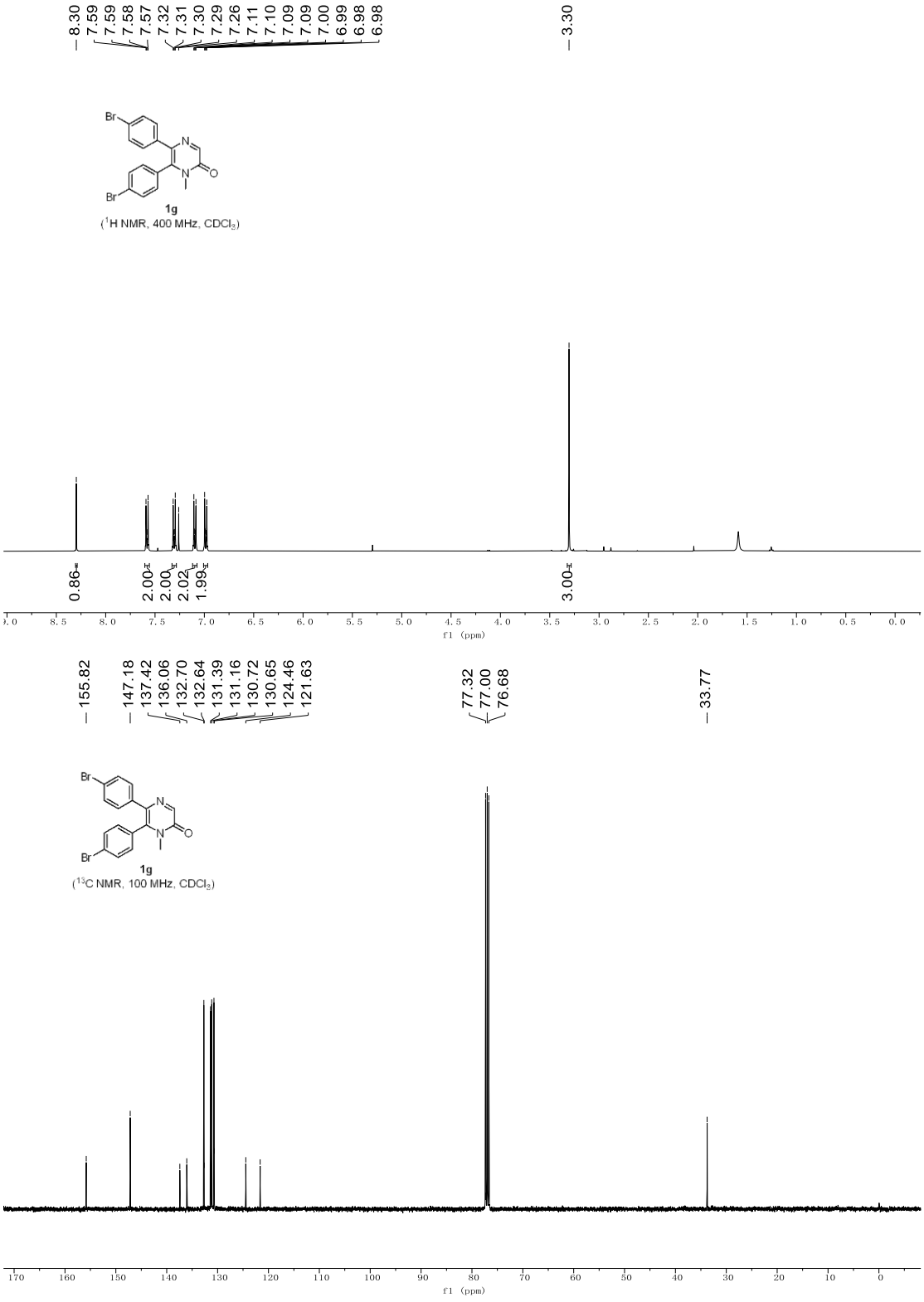
1f
(^{1}H NMR, 400 MHz, $CDCl_3$)



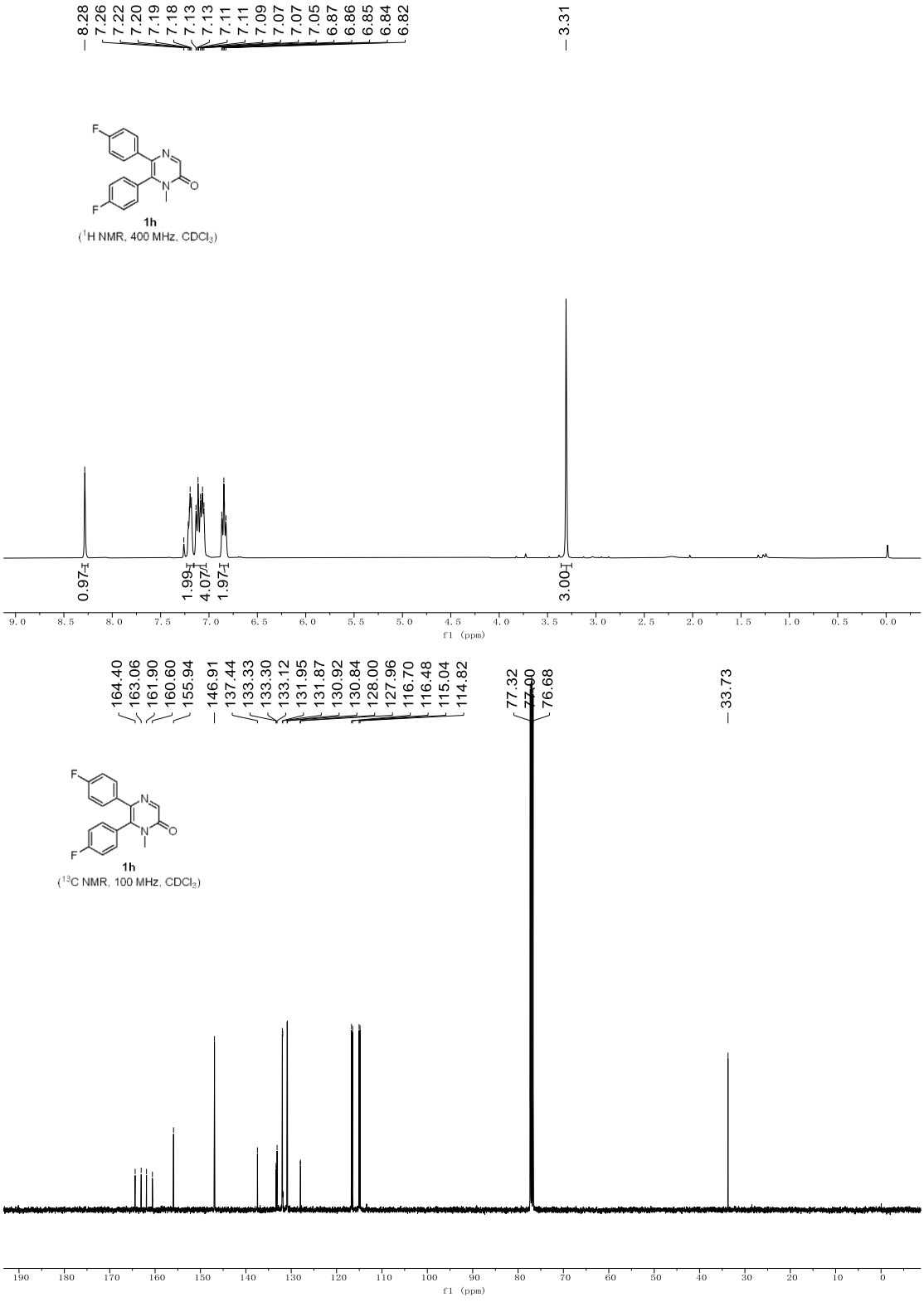


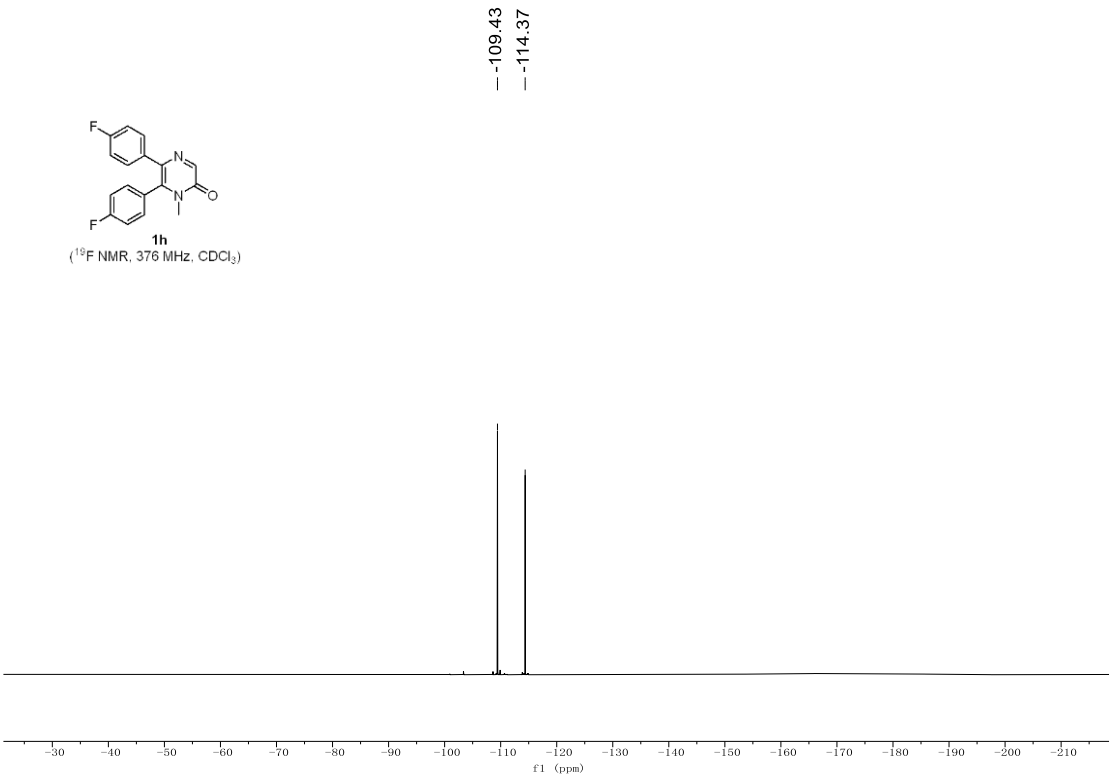
1f
(^{13}C NMR, 100 MHz, $CDCl_3$)

5,6-bis(4-bromophenyl)-1-methylpyrazin-2(1*H*)-one (1g)

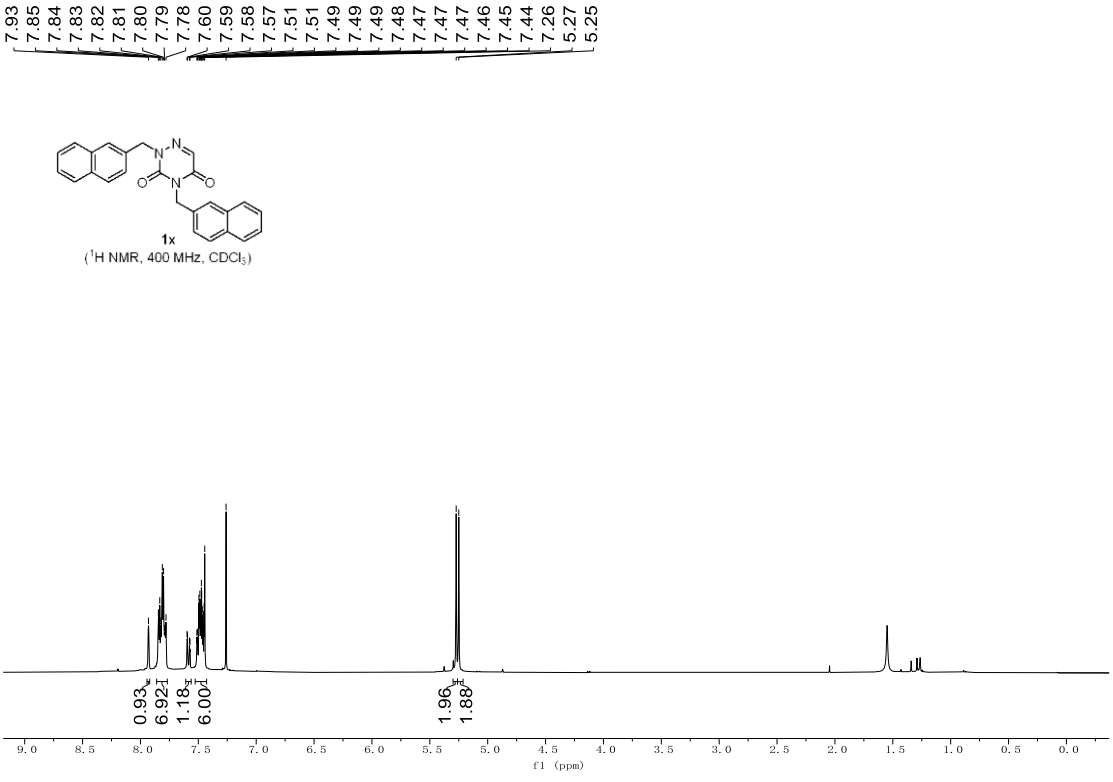


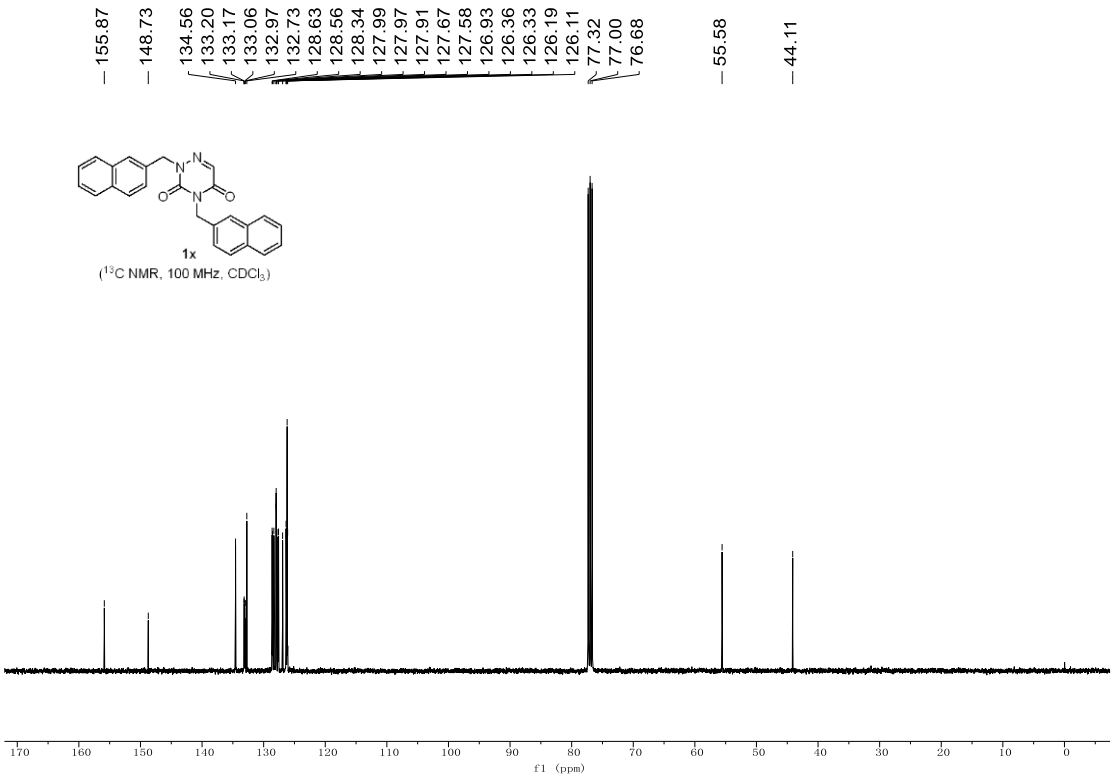
5,6-bis(4-fluorophenyl)-1-methylpyrazin-2(1*H*)-one (1h)



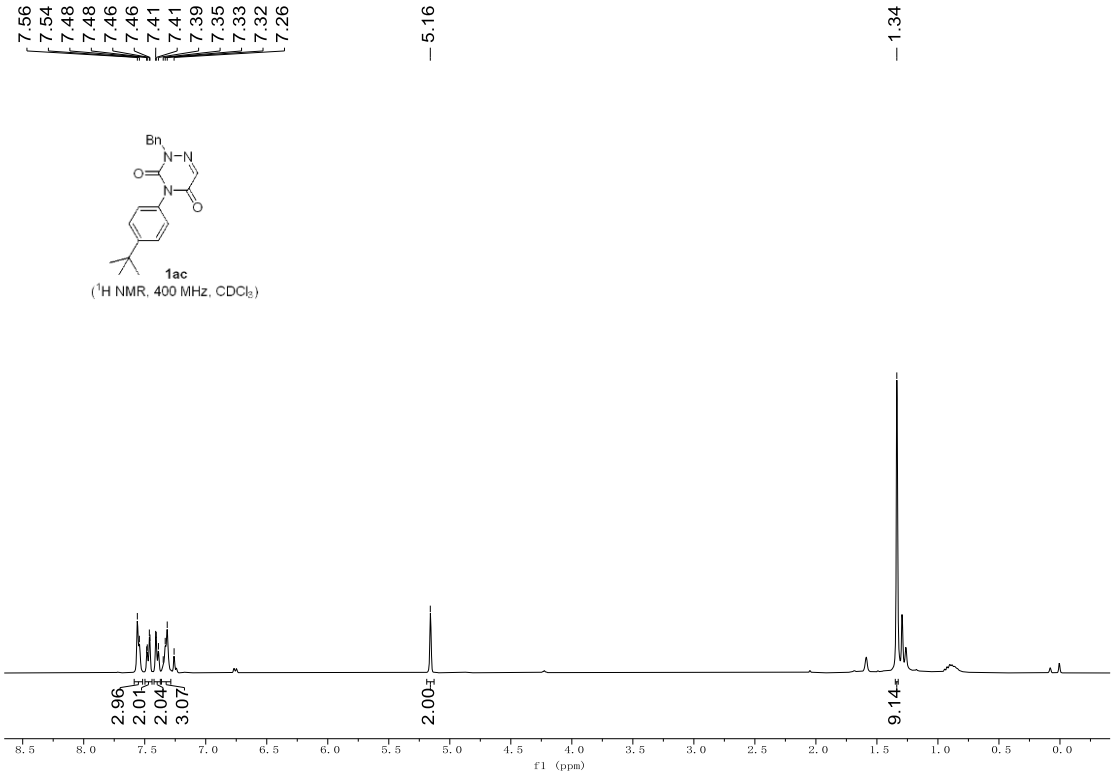


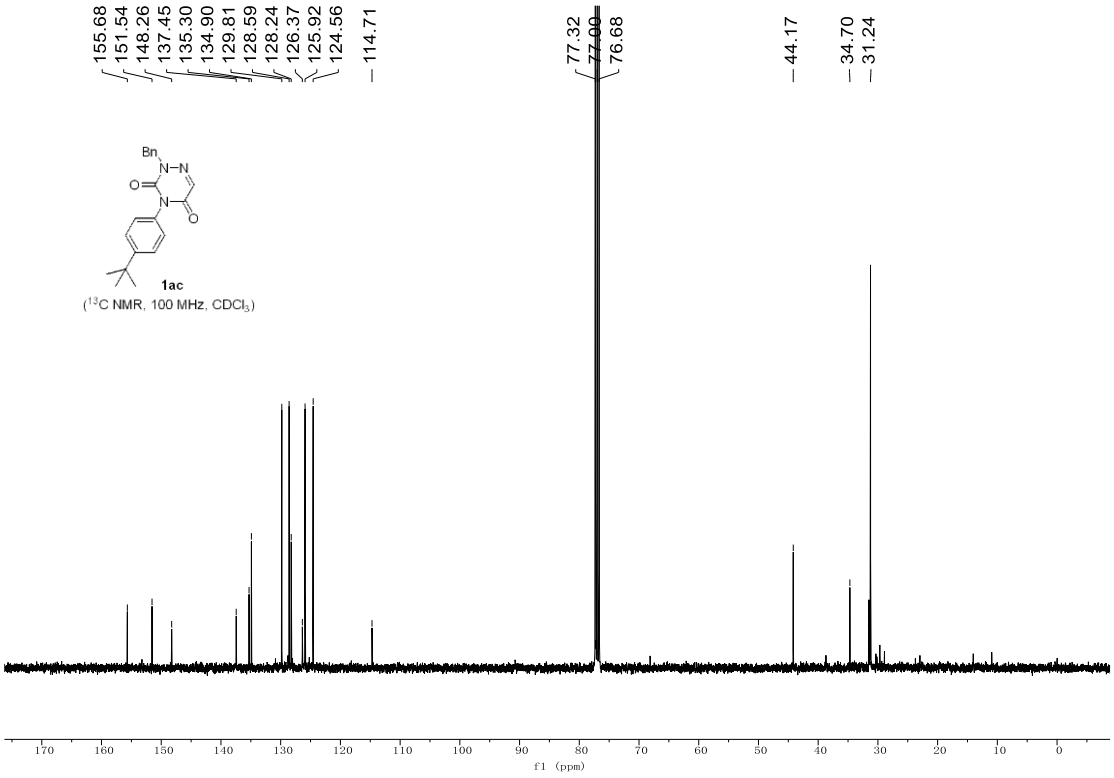
2,4-bis(naphthalen-2-ylmethyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (1x)



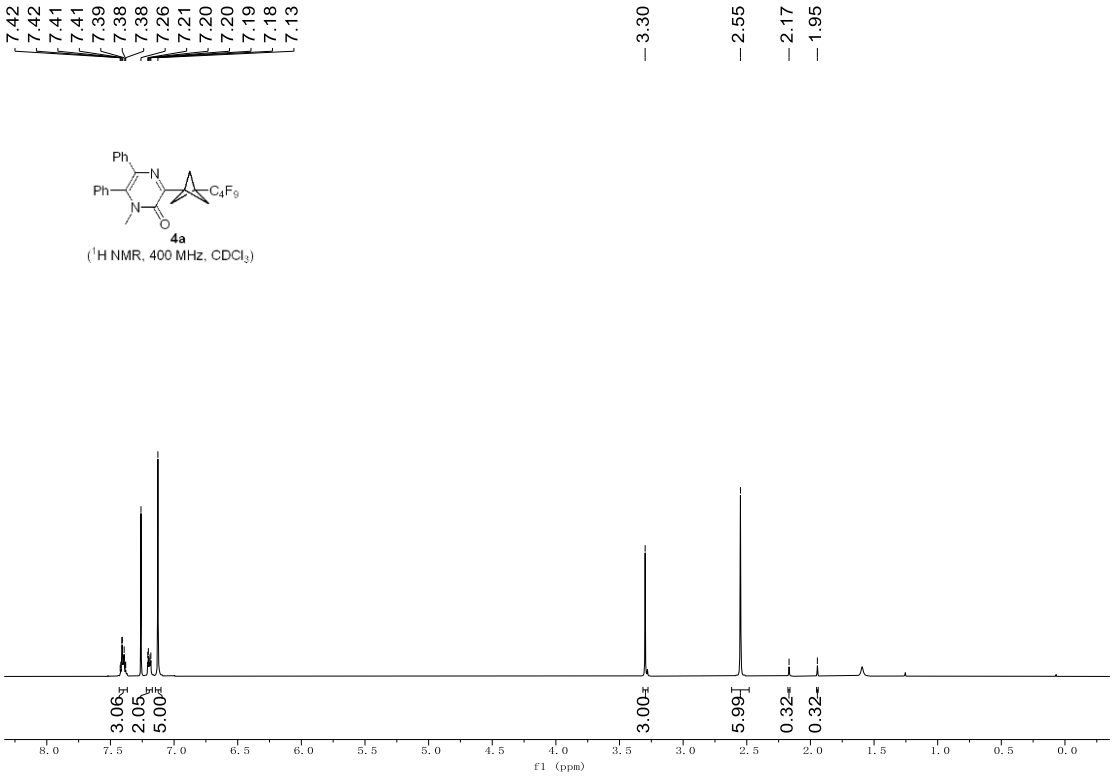


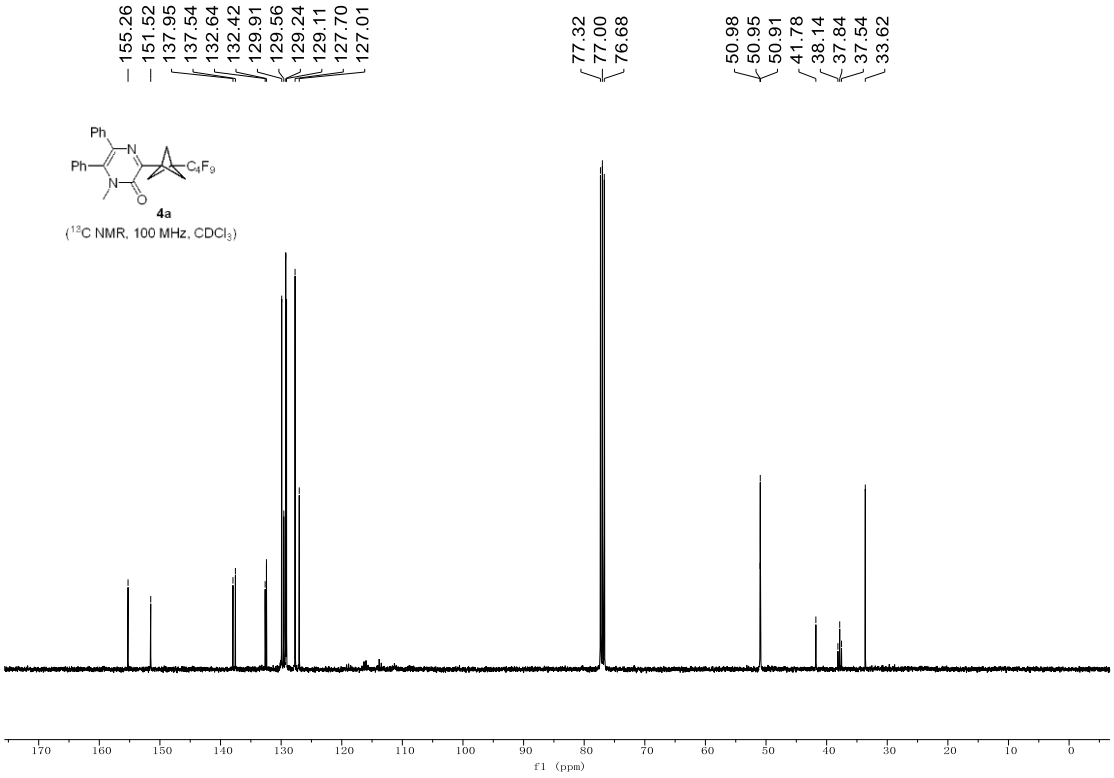
2-benzyl-4-(4-(tert-butyl)phenyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (1ac)





1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4 λ [12]-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5,6-diphenylpyrazin-2(1*H*)-one (4a)

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132.42
129.91
129.56
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129.11
127.70
127.01
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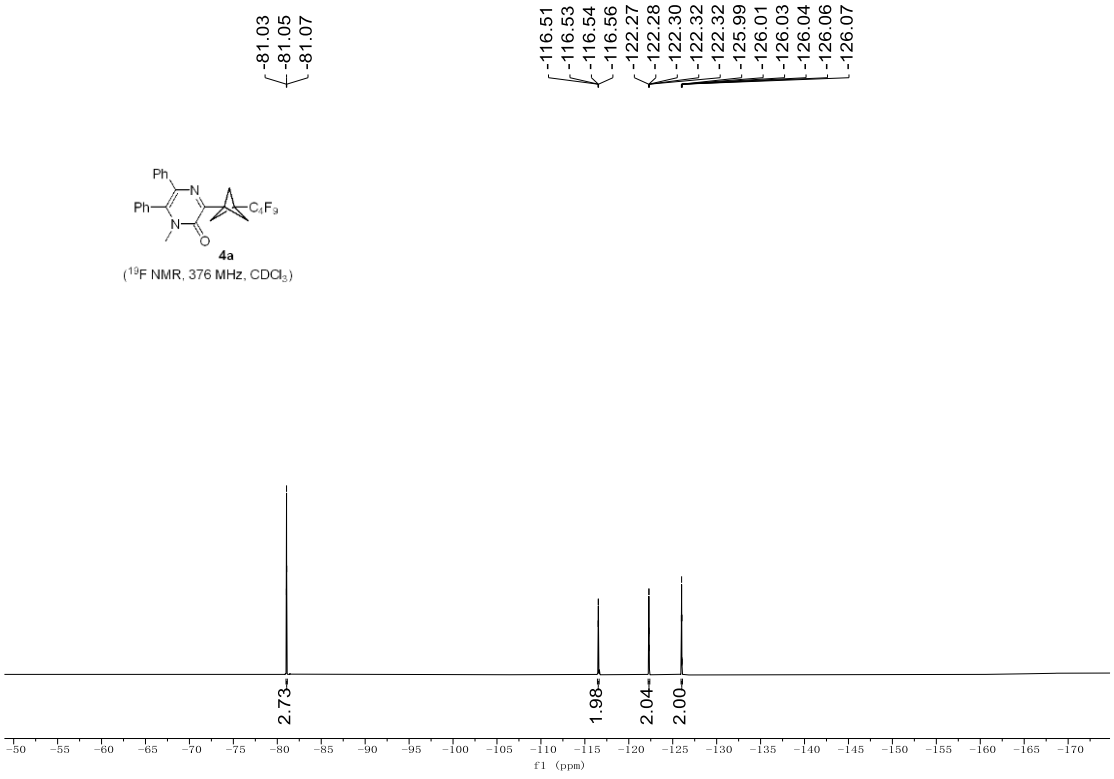
77.00
76.68
50.98
50.95
50.91
41.78
38.14
37.84
37.54
33.62
Ph
Ph
N
N
O
C4F9
4a
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)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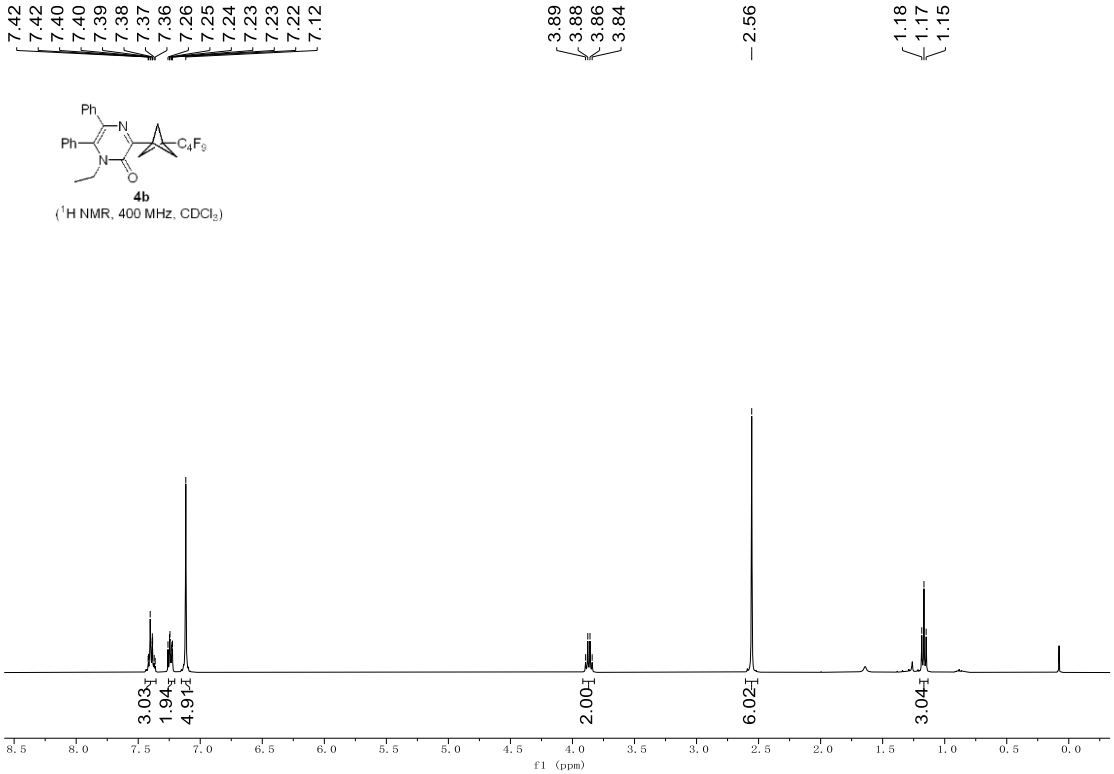
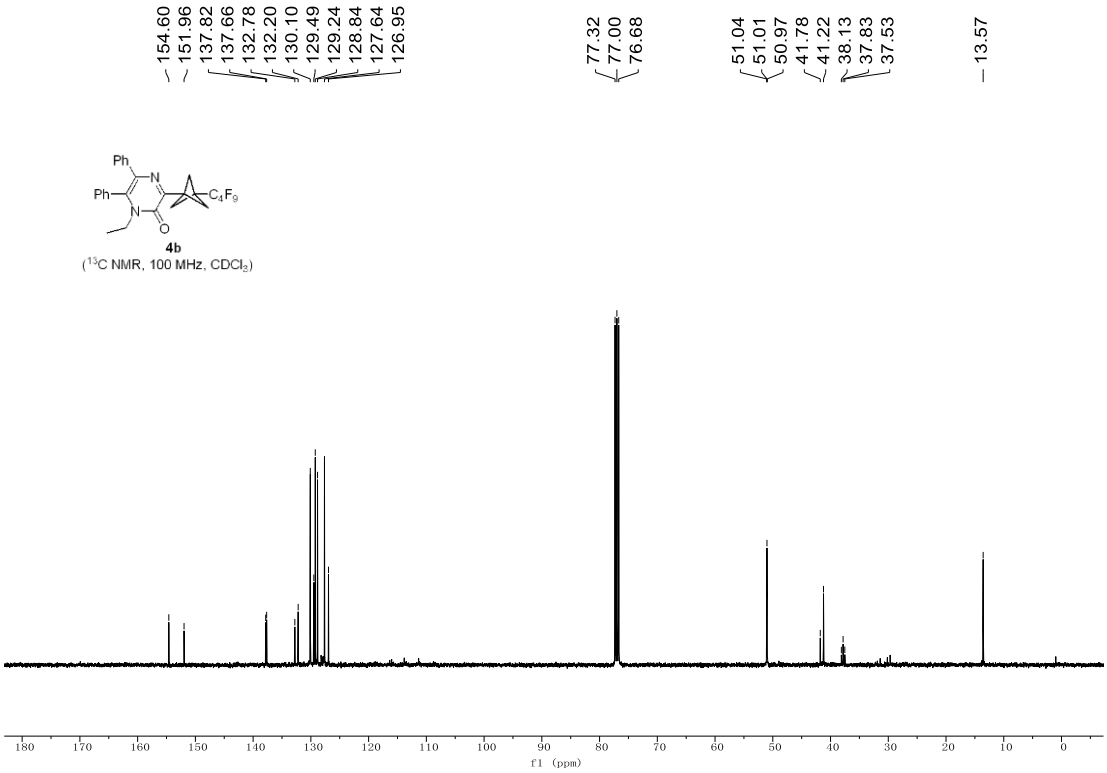
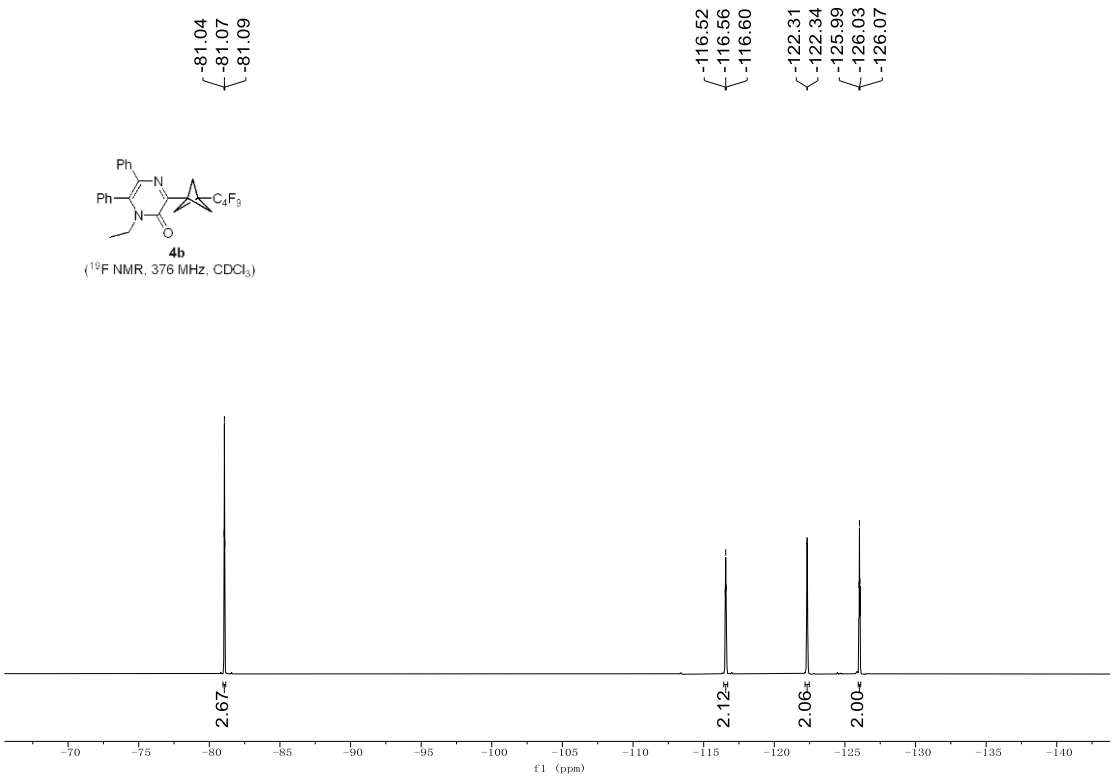
-122.32
-122.32
-125.99
-126.01
-126.03
-126.04
-126.06
-126.07
Ph
Ph
N
N
O
C4F9
4a
(19F NMR, 376 MHz, CDCl3)
2.73
1.98
2.04
2.00
f1 (ppm)

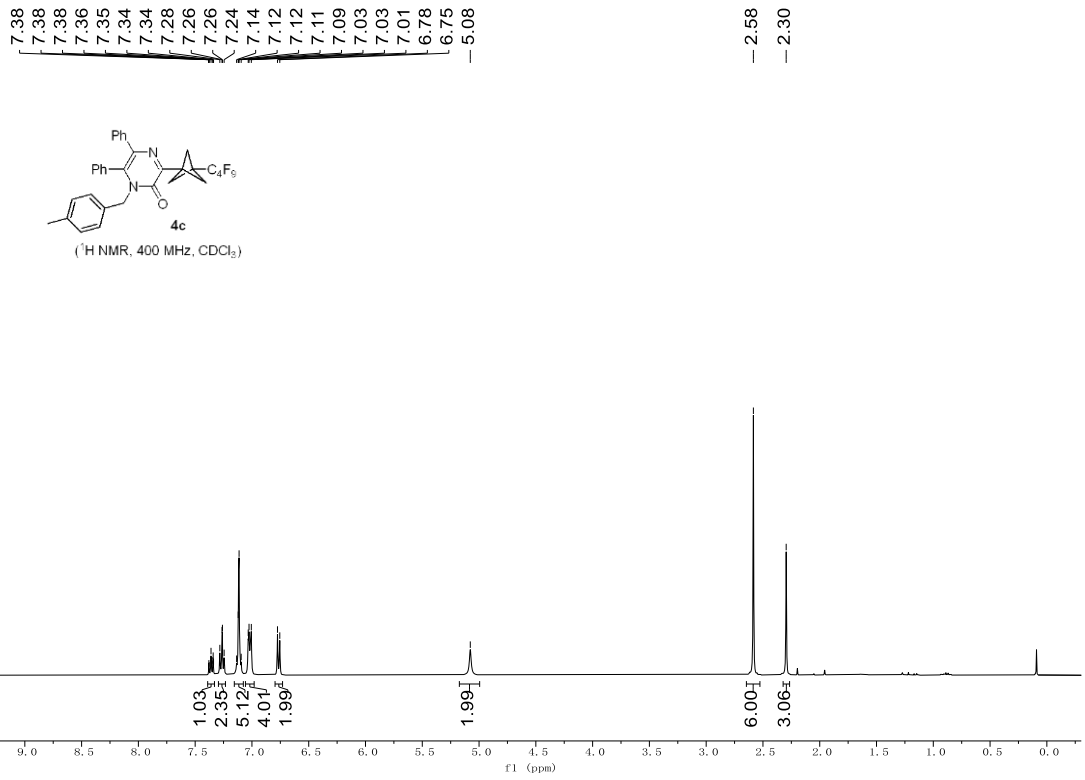
1-ethyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5,6-diphenylpyrazin-2(1*H*)-one (4b)

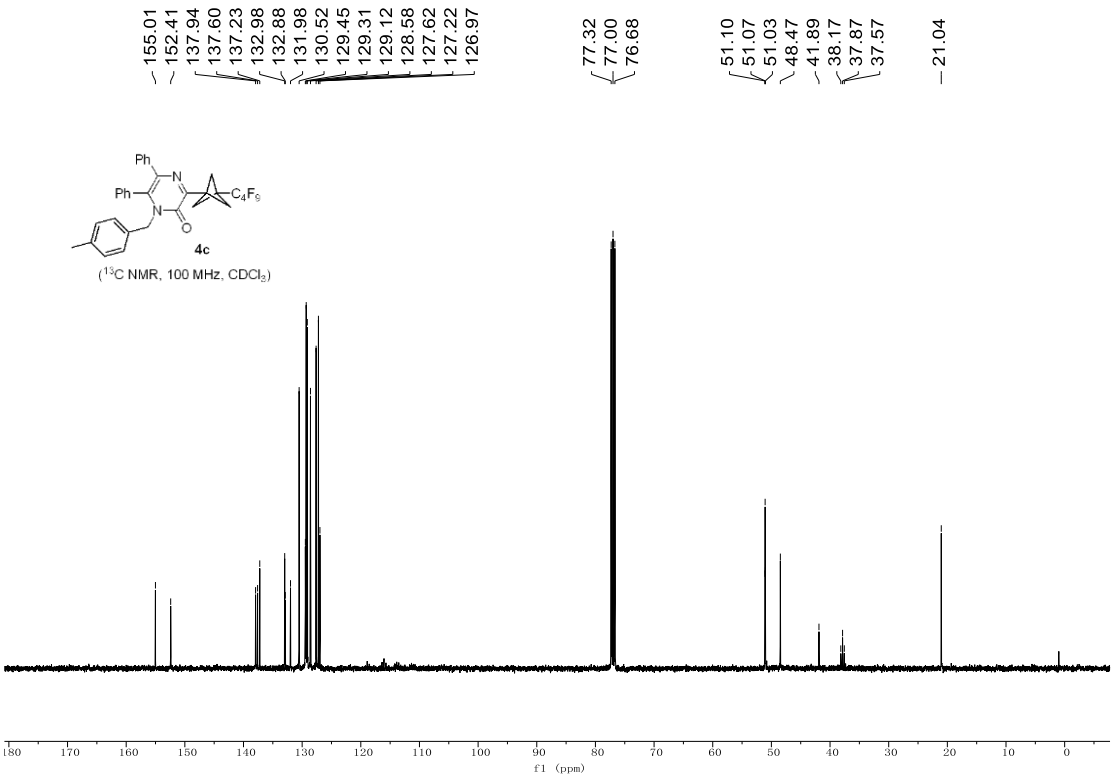






1-(4-methylbenzyl)-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5,6-diphenylpyrazin-2(1*H*)-one (4c)

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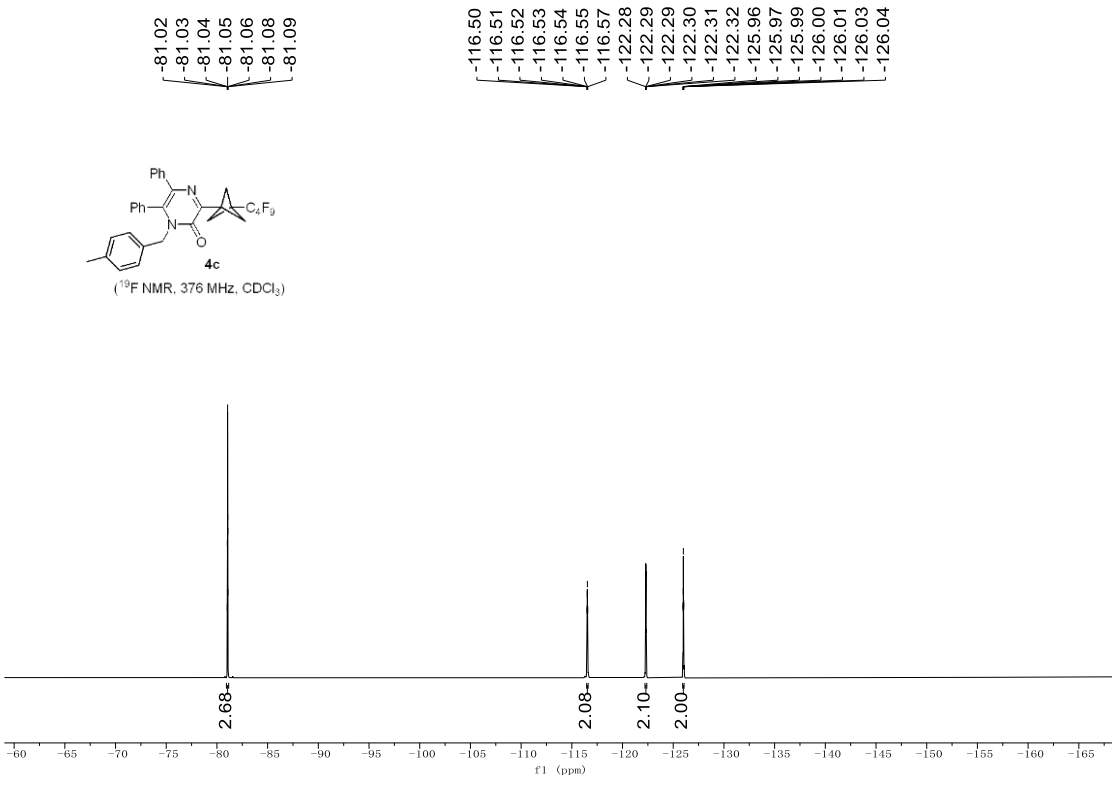
77.00
76.68
51.10
51.07
51.03
48.47
41.89
38.17
37.87
37.57
21.04
Ph
Ph
N
N
O
C_4F_9
4c
(^{13}C NMR, 100 MHz, $CDCl_3$)
f1 (ppm)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-116.57
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-122.29
-122.30
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-122.32
-125.96
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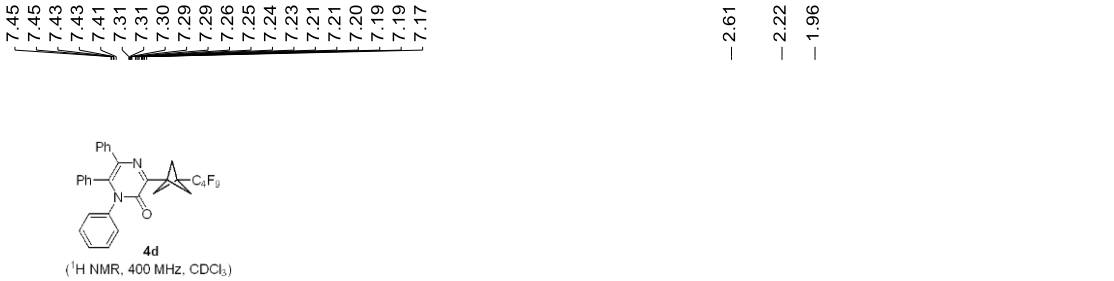
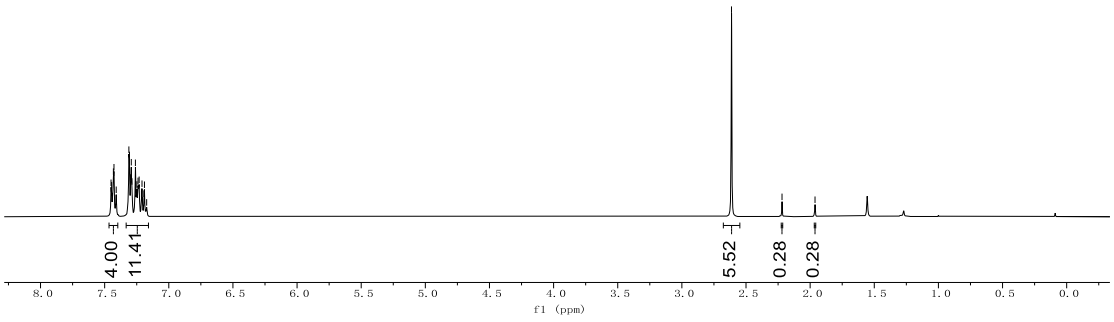
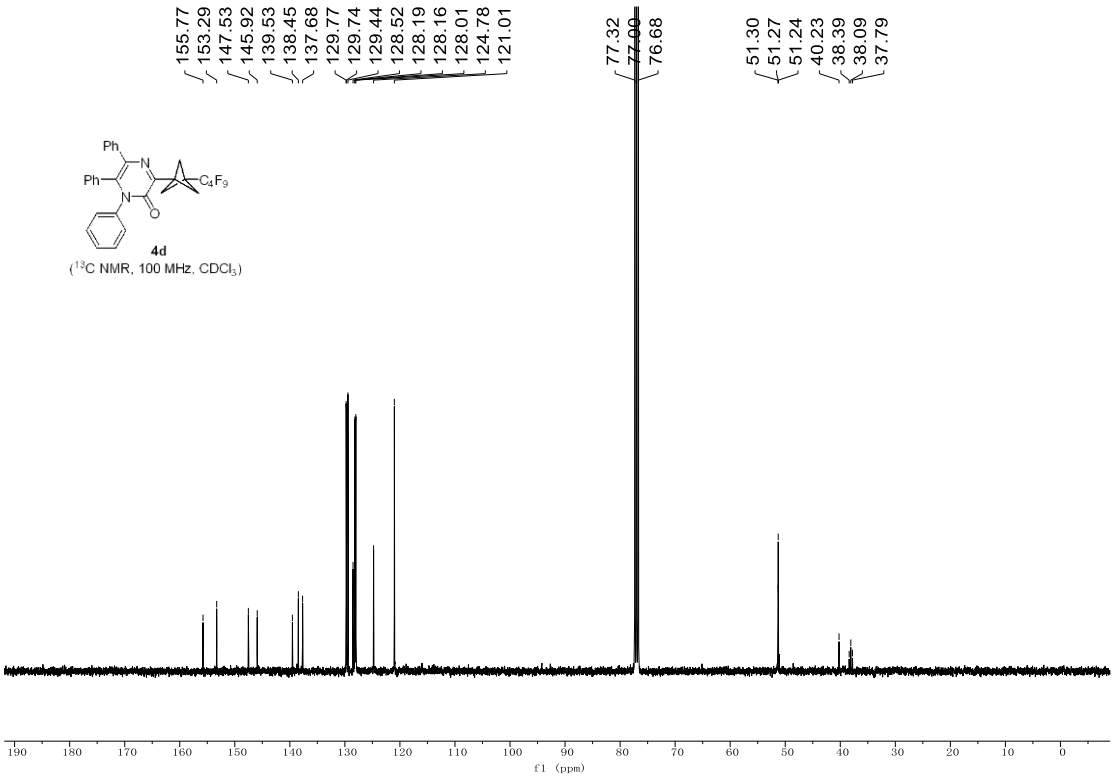
-125.99
-126.00
-126.01
-126.03
-126.04
Ph
Ph
N
N
O
C_4F_9
4c
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.68
2.08
2.10
2.00
f1 (ppm)

3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4 λ ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,5,6-triphenylpyrazin-2(1*H*)-one (4d)

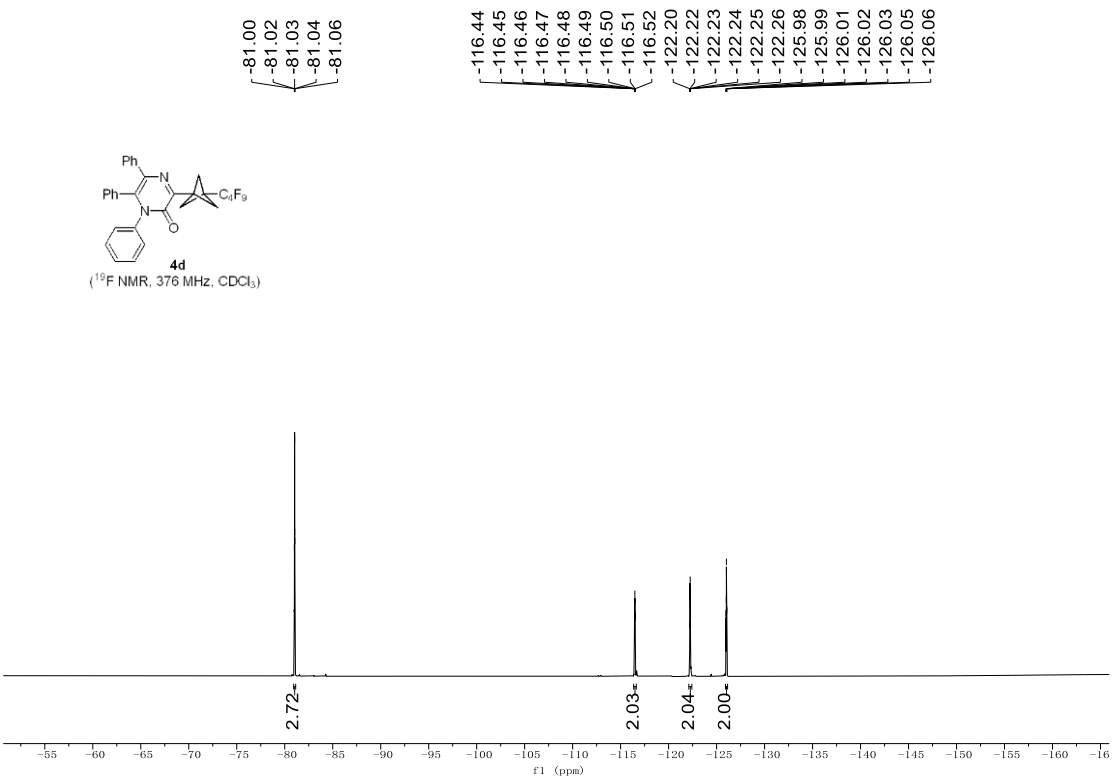


4d
(^{1}H NMR, 400 MHz, $CDCl_3$)

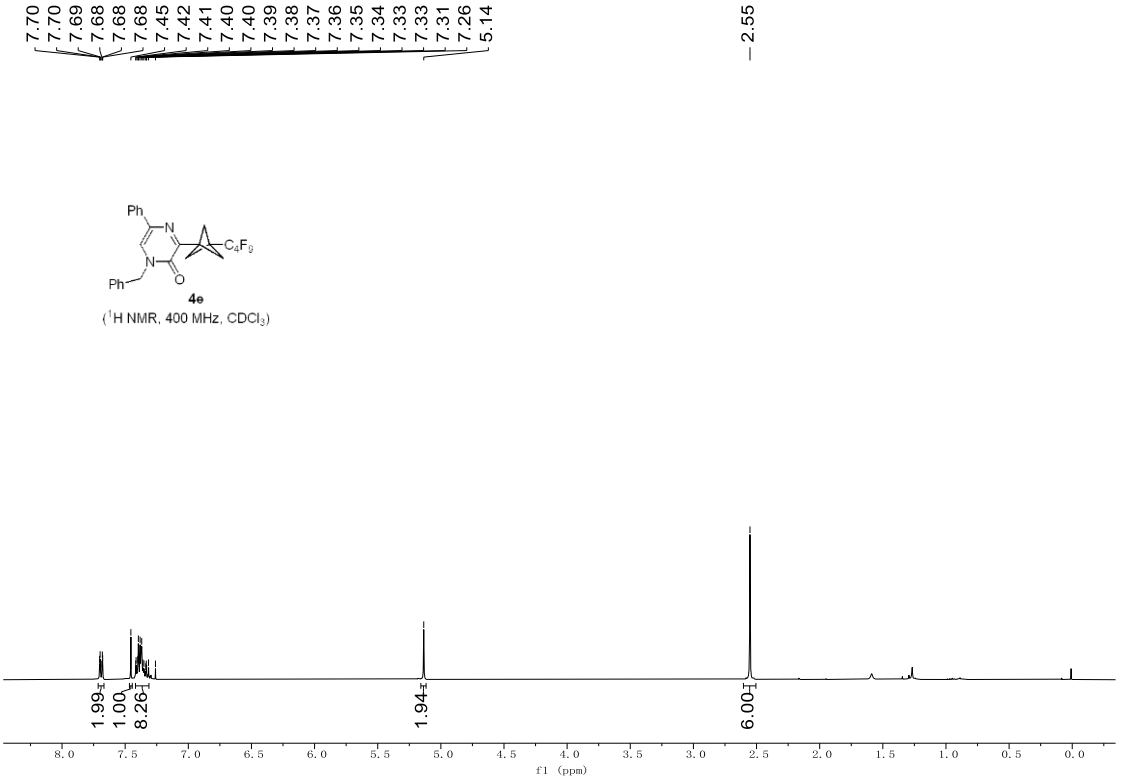


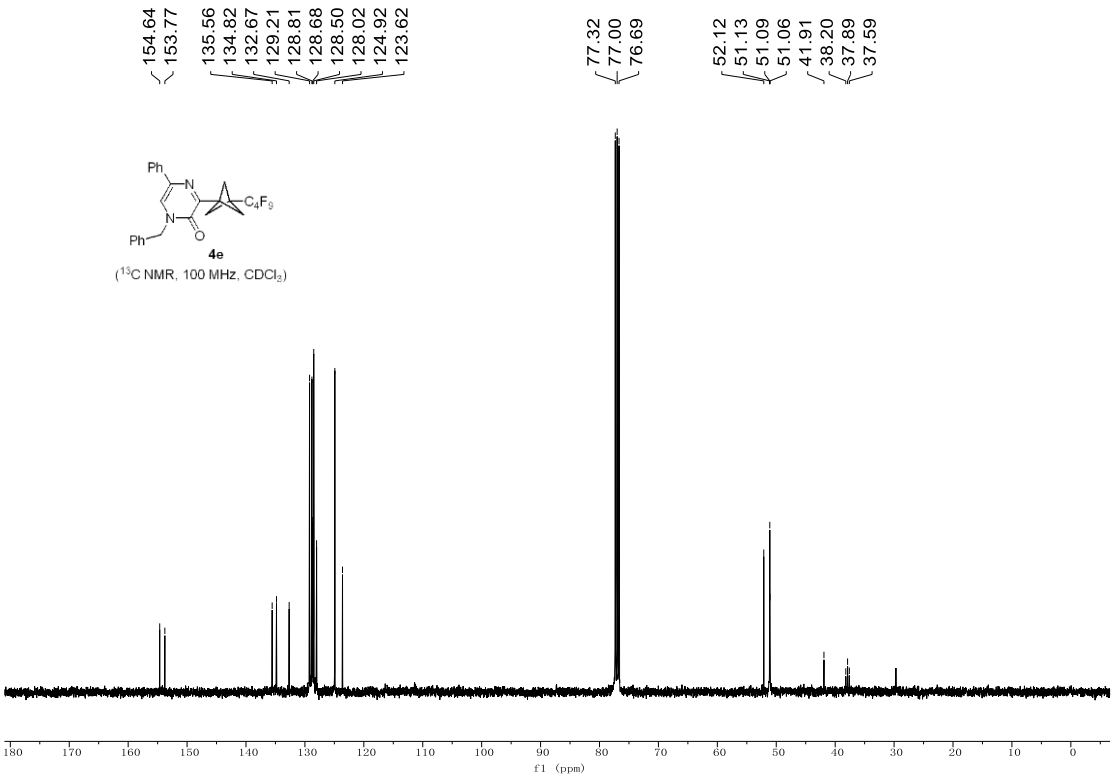


4d
(^{13}C NMR, 100 MHz, $CDCl_3$)



1-benzyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-5-phenylpyrazin-2(1*H*)-one (4e)

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77.00
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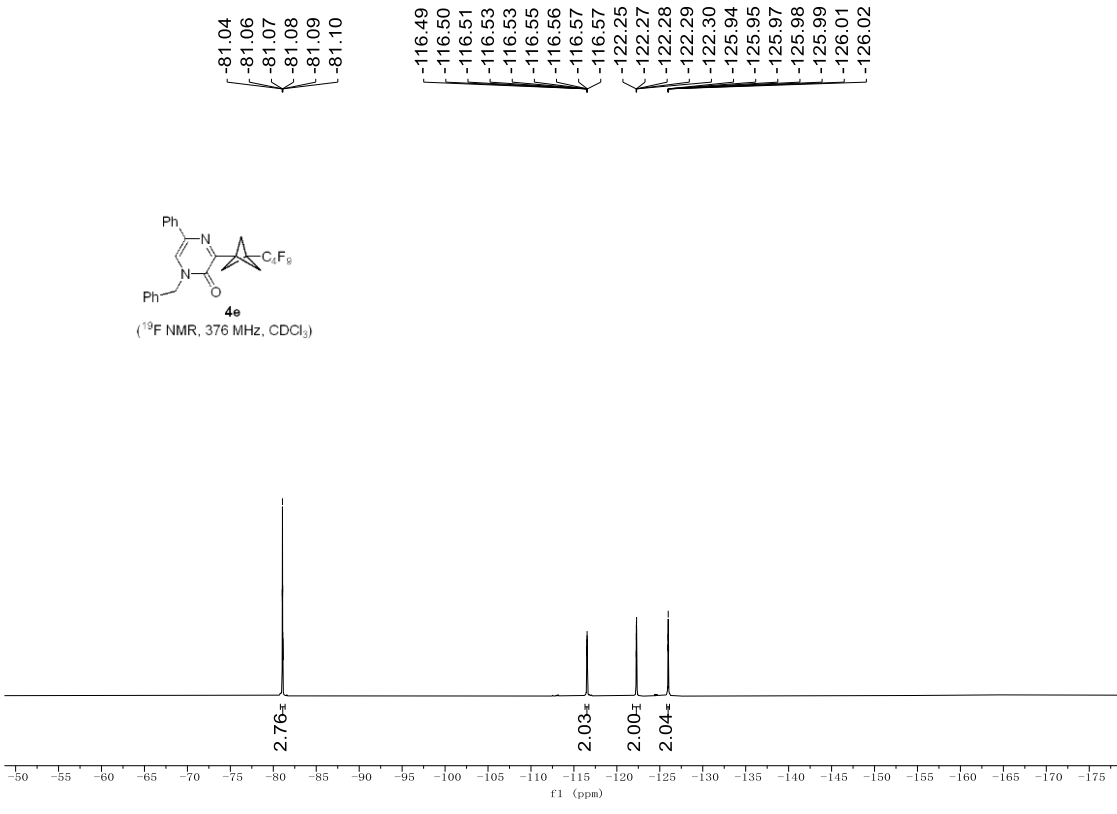
52.12
51.13
51.09
51.06
41.91
38.20
37.89
37.59
Ph
N
N
O
Ph
C_4F_9
4e
(^{13}C NMR, 100 MHz, $CDCl_3$)
f1 (ppm)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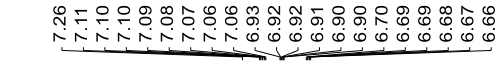
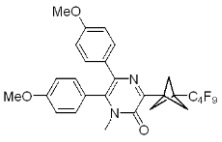
-125.95
-125.97
-125.98
-125.99
-126.01
-126.02
Ph
N
N
O
Ph
C_4F_9
4e
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.76
2.03
2.00
2.04
f1 (ppm)

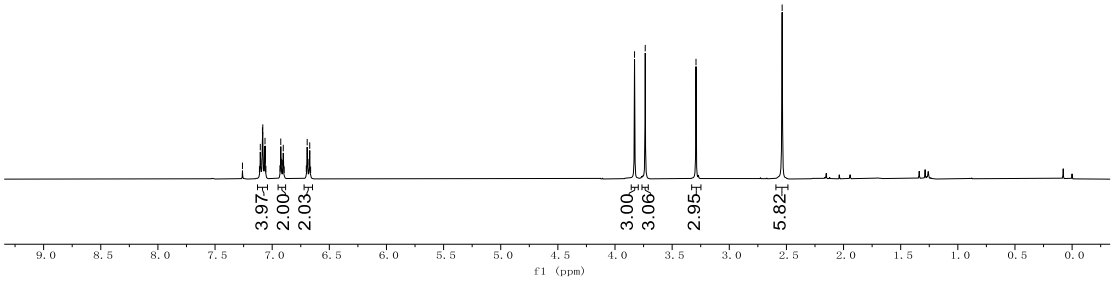
5,6-bis(4-methoxyphenyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (4f)

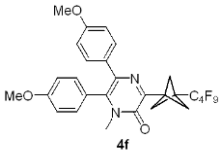




4f

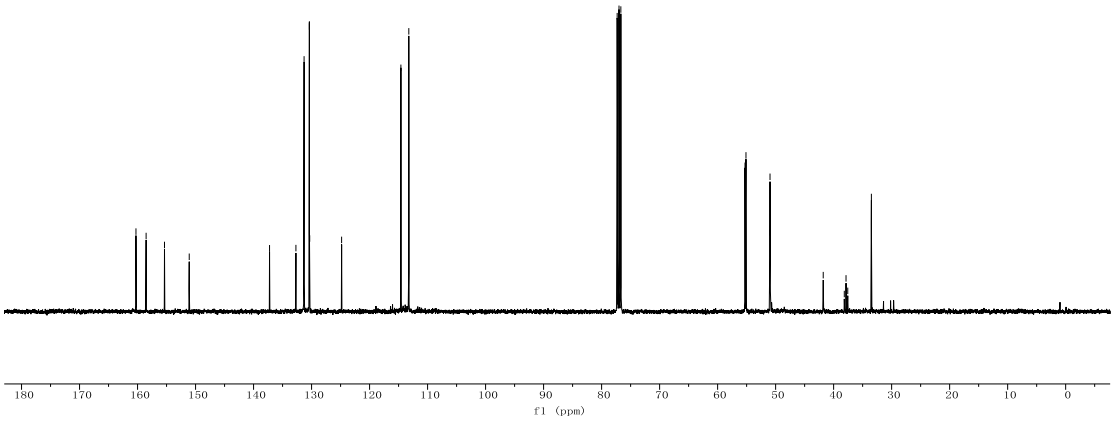
(^{1}H NMR, 400 MHz, $CDCl_3$)

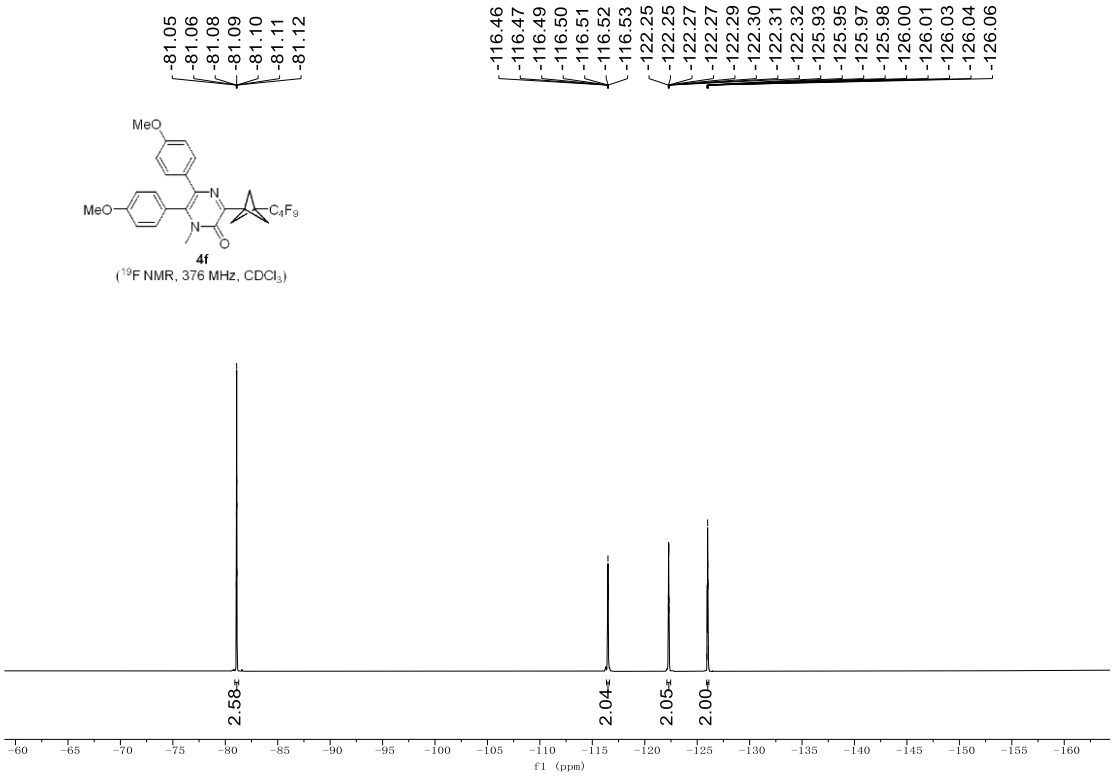




4f

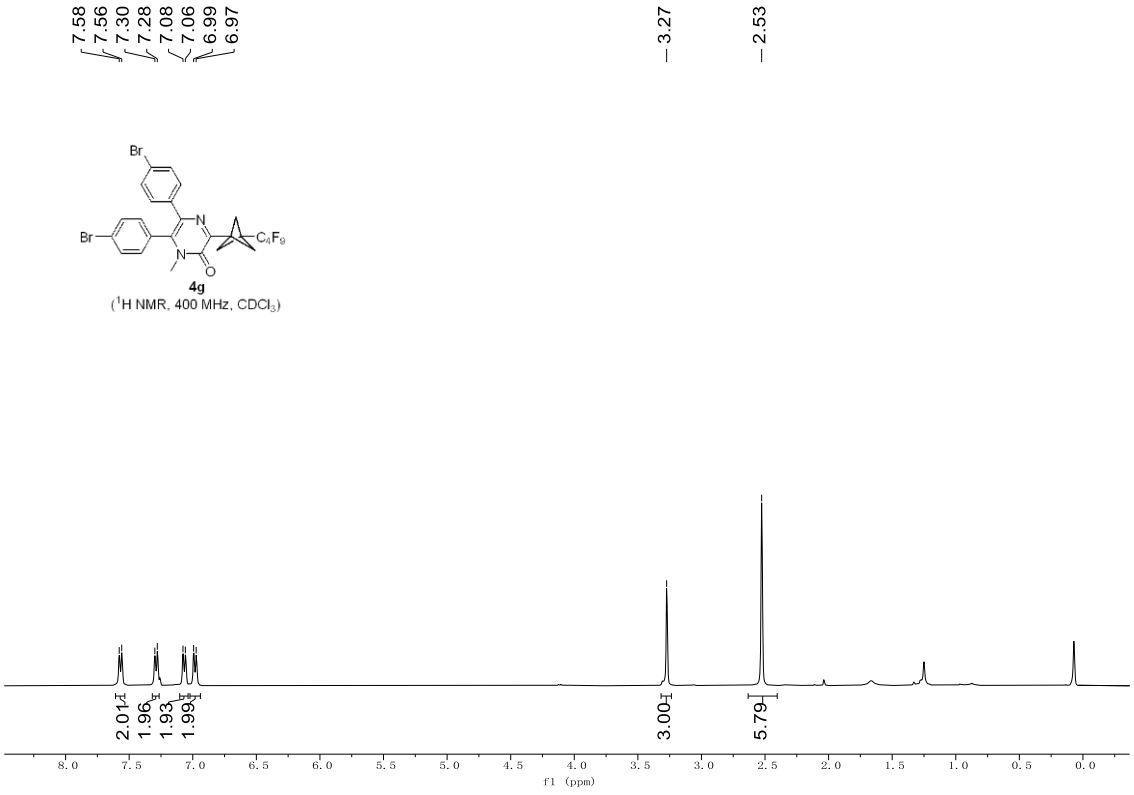
(^{13}C NMR, 100 MHz, $CDCl_3$)



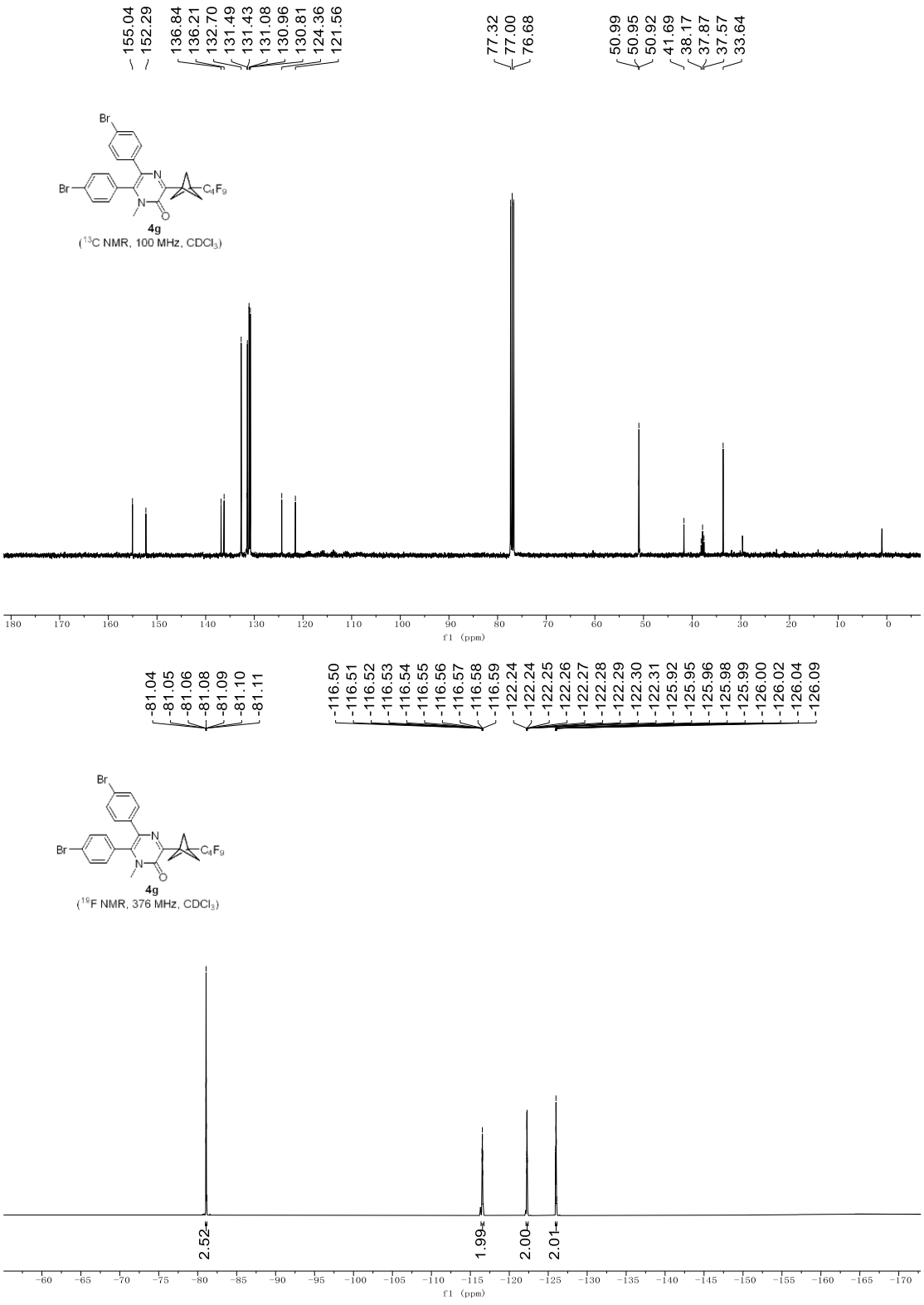


4f
(^{19}F NMR, 376 MHz, $CDCl_3$)

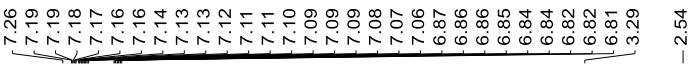
5,6-bis(4-bromophenyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (4g)

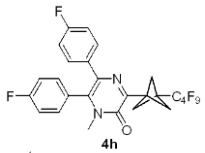


4g
(^{1}H NMR, 400 MHz, $CDCl_3$)



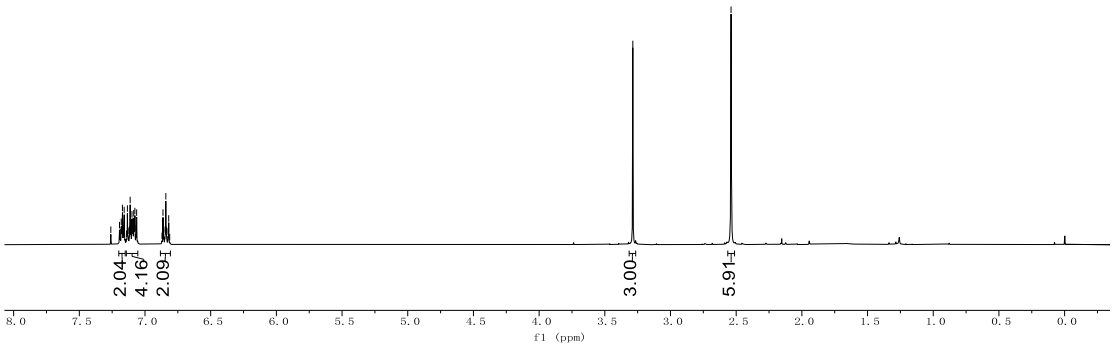
5,6-bis(4-fluorophenyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo [1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (4h)

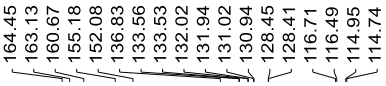


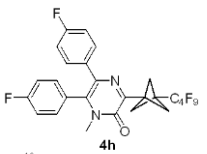


4h

(^{1}H NMR, 400 MHz, $CDCl_3$)

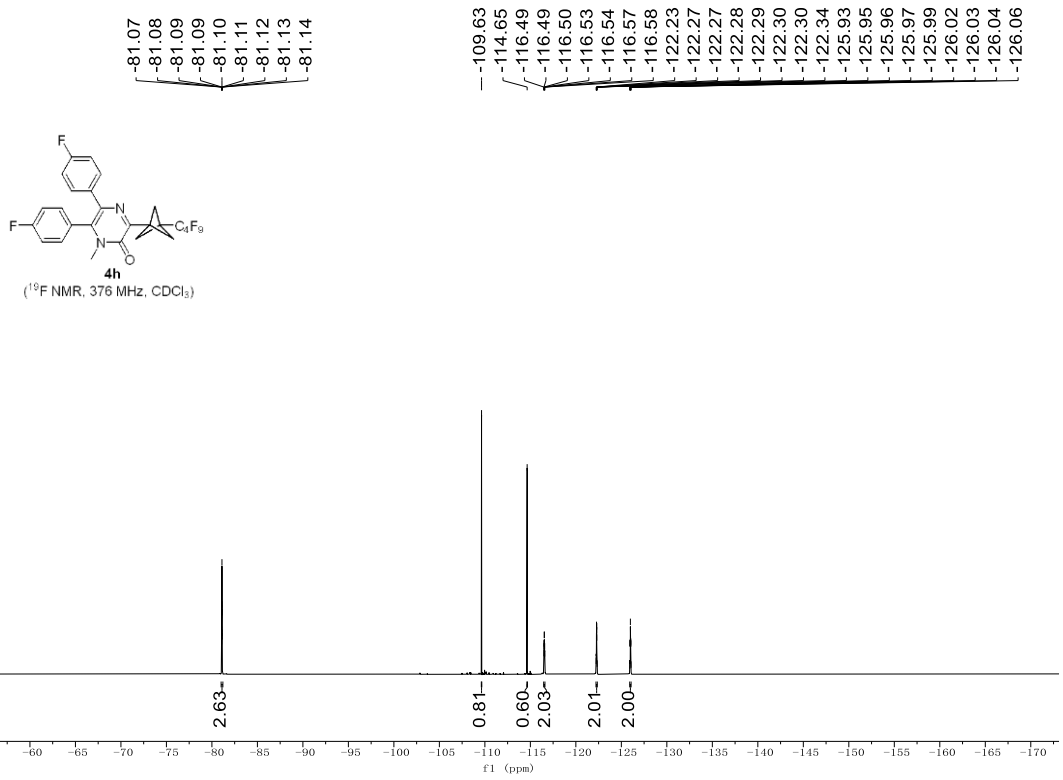




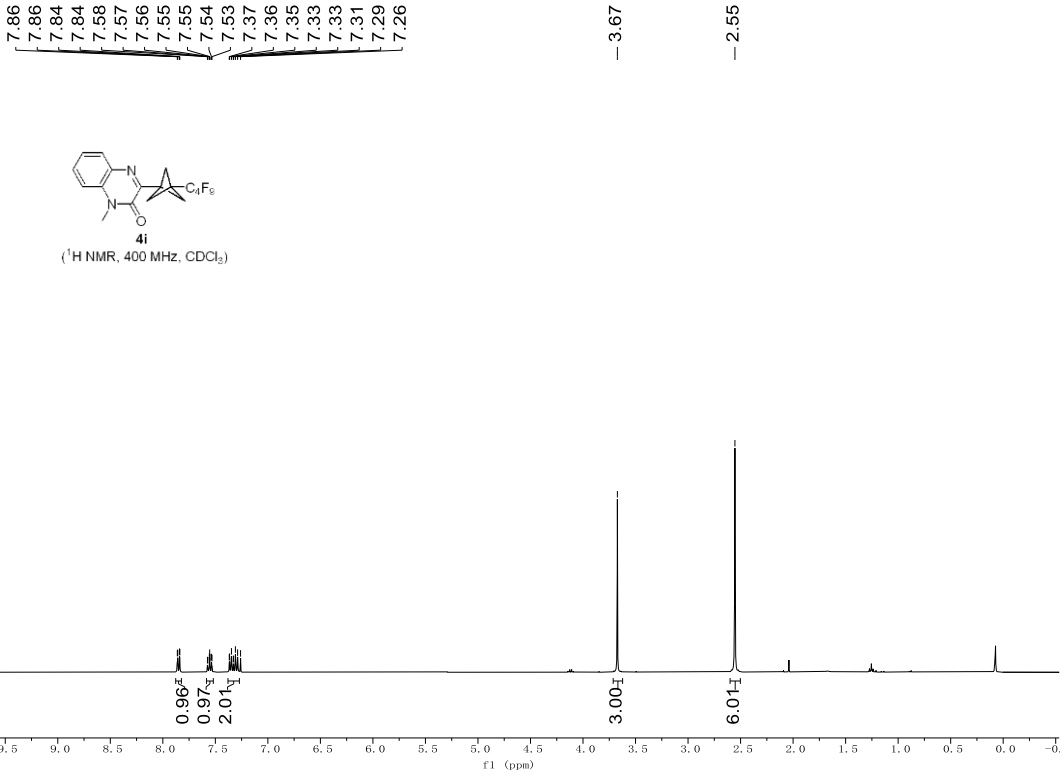


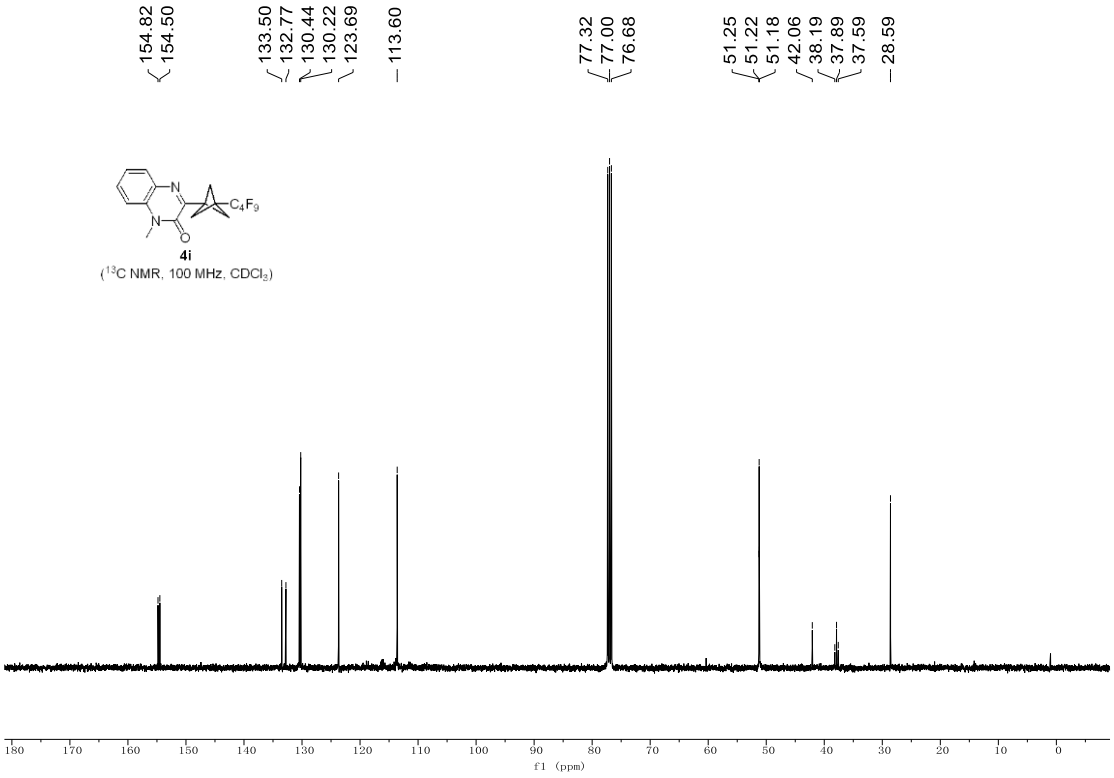
4h

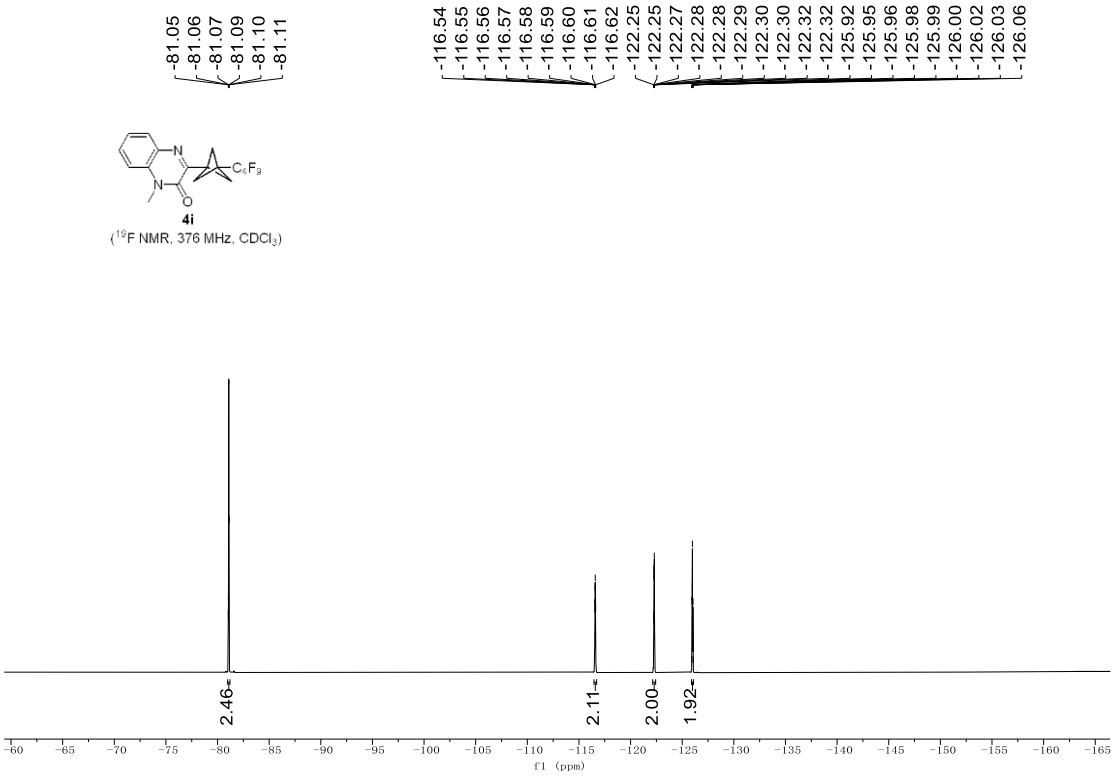
(^{13}C NMR, 100 MHz, $CDCl_3$)



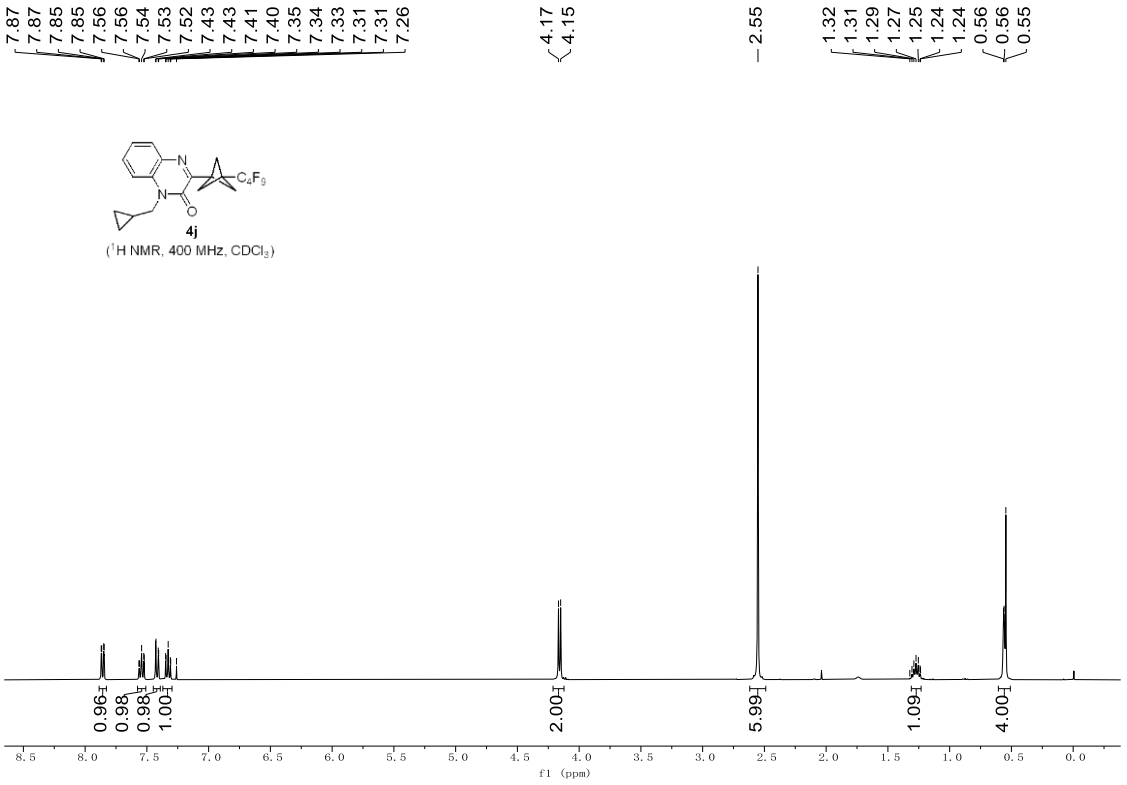
1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4i)

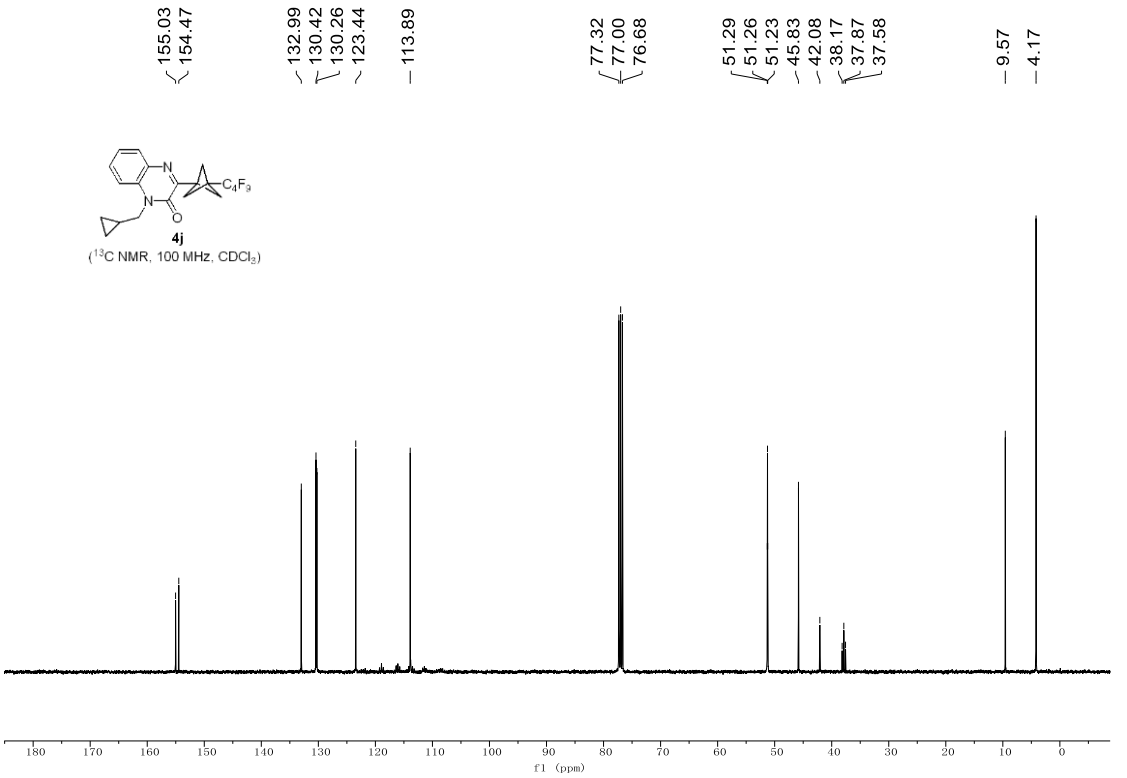


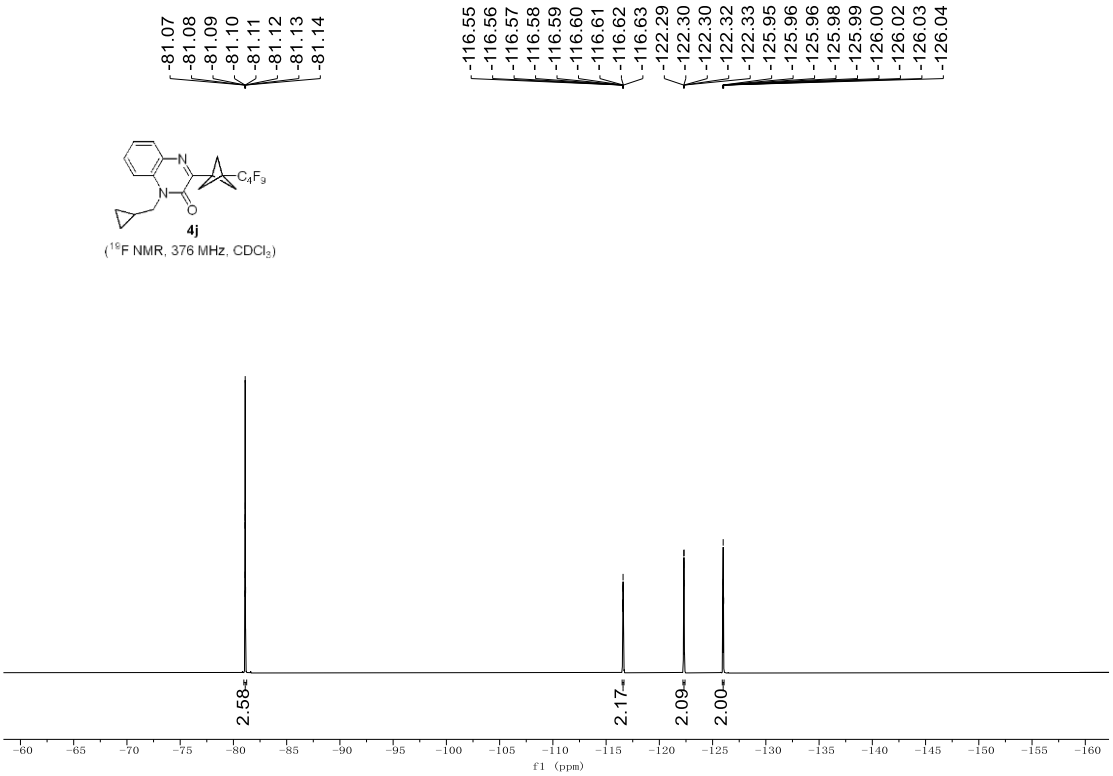

154.82
154.50
133.50
132.77
130.44
130.22
123.69
113.60
77.32
77.00
76.68
51.25
51.22
51.18
42.06
38.19
37.89
37.59
28.59
C4F9
4i
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)


-81.05
-81.06
-81.07
-81.09
-81.10
-81.11
-116.54
-116.55
-116.56
-116.57
-116.58
-116.59
-116.60
-116.61
-116.62
-122.25
-122.25
-122.27
-122.28
-122.28
-122.29
-122.30
-122.30
-122.32
-122.32
-125.92
-125.95
-125.96
-125.98
-125.99
-126.00
-126.02
-126.03
-126.06
C6F9
4i
(19F NMR, 376 MHz, CDCl3)
2.46
2.11
2.00
1.92
f1 (ppm)

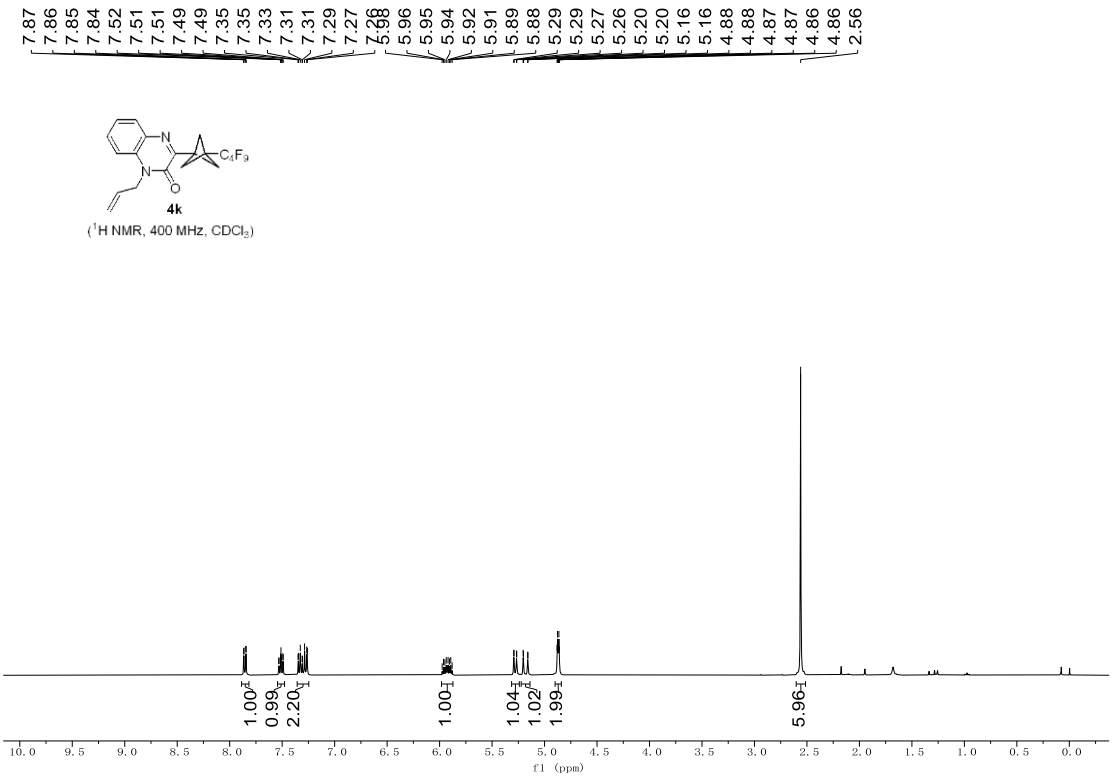
1-(cyclopropylmethyl)-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4j)

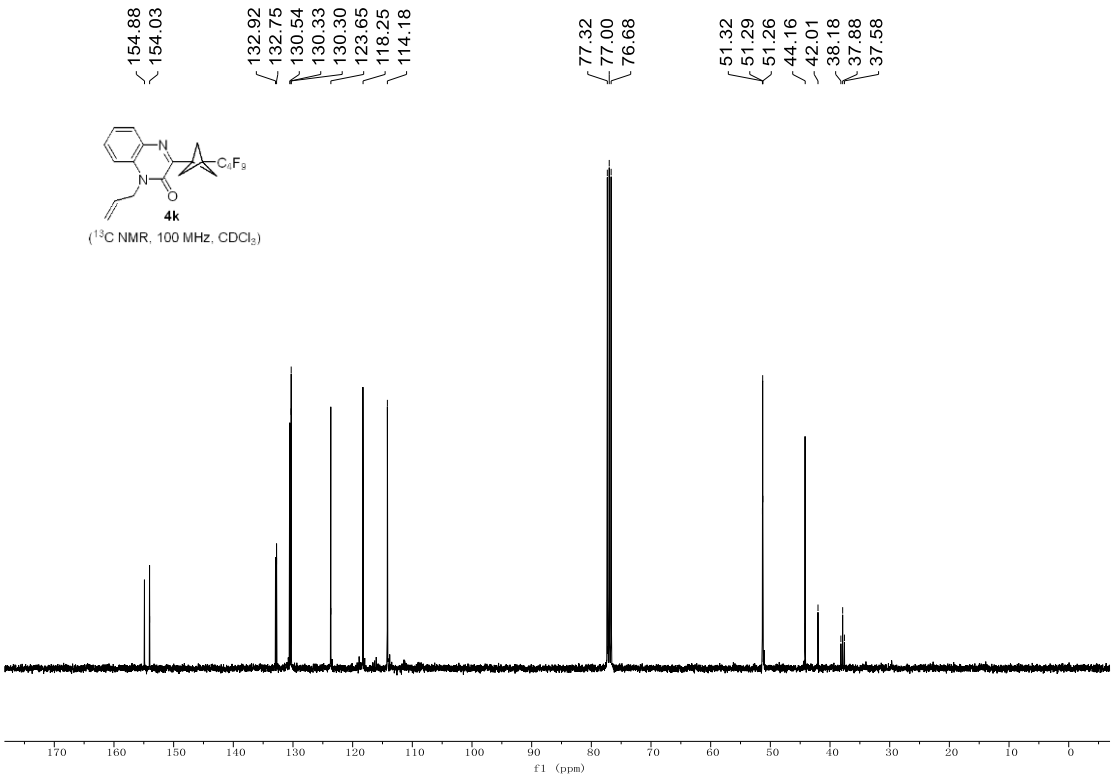


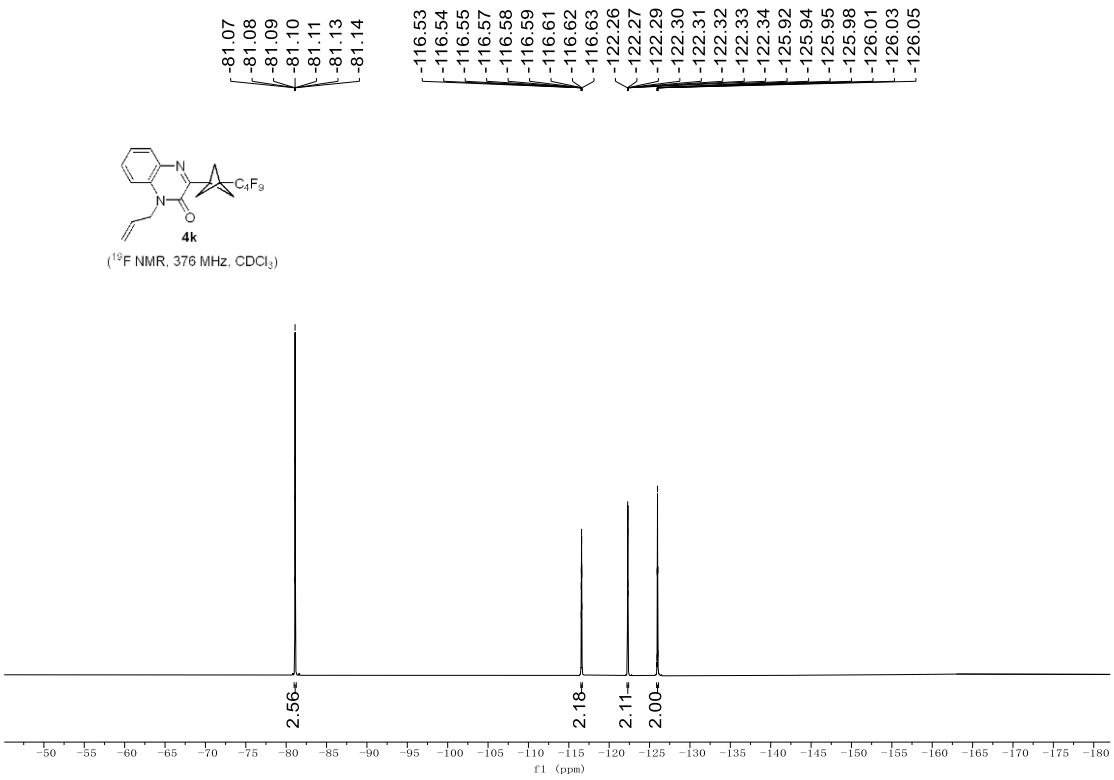




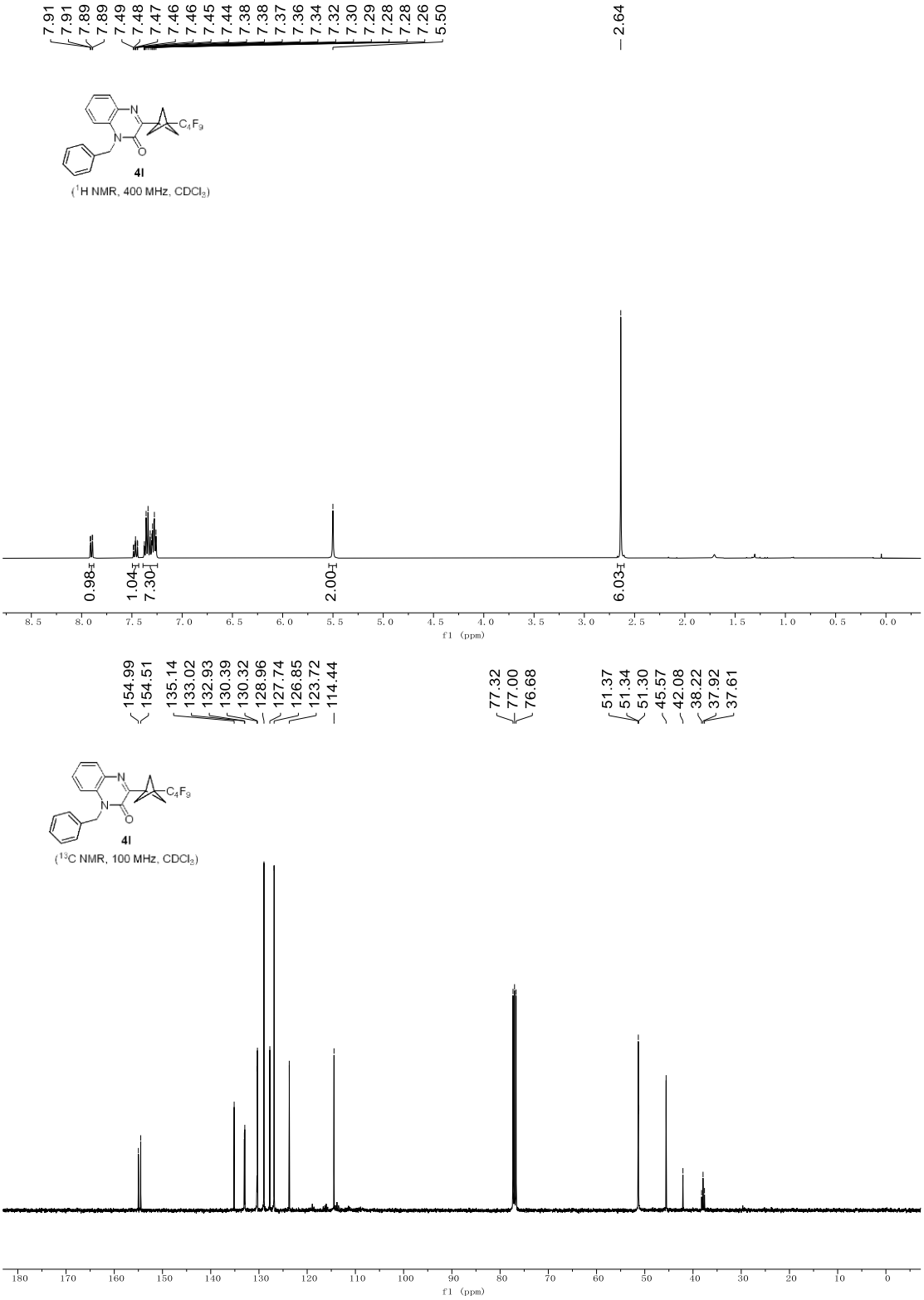
1-allyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4k)

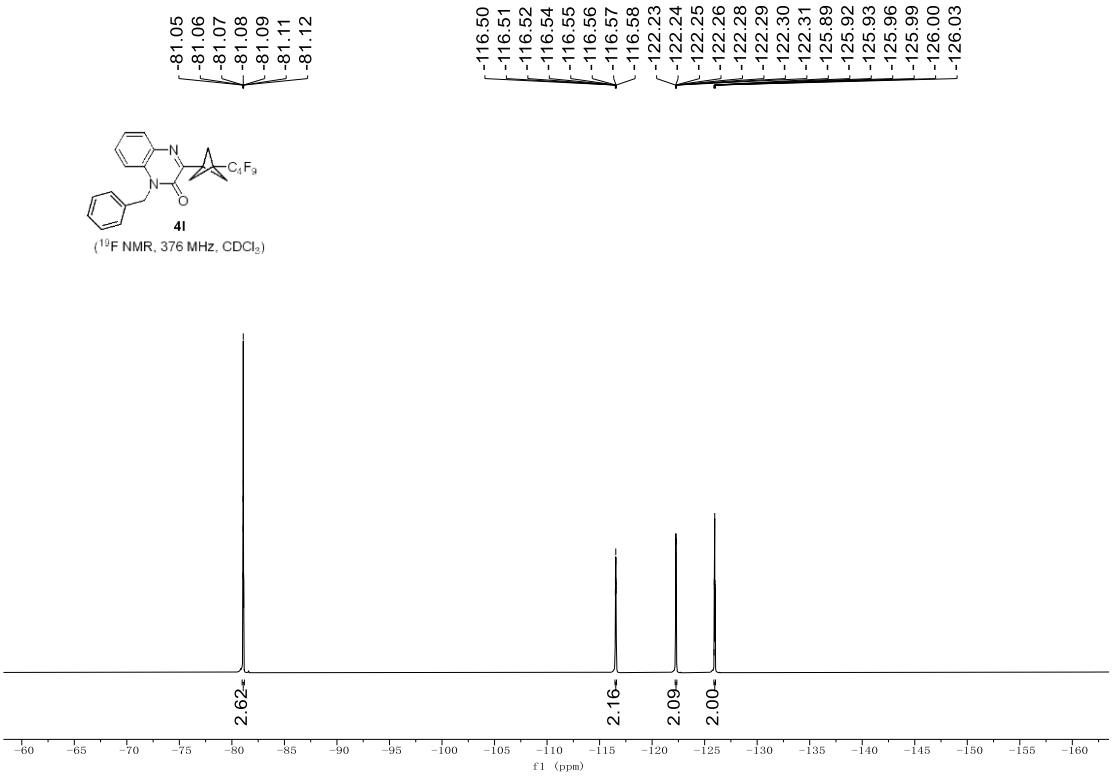



154.88
154.03
132.92
132.75
130.54
130.33
130.30
123.65
118.25
114.18
77.32
77.00
76.68
51.32
51.29
51.26
44.16
42.01
38.18
37.88
37.58
4k
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)

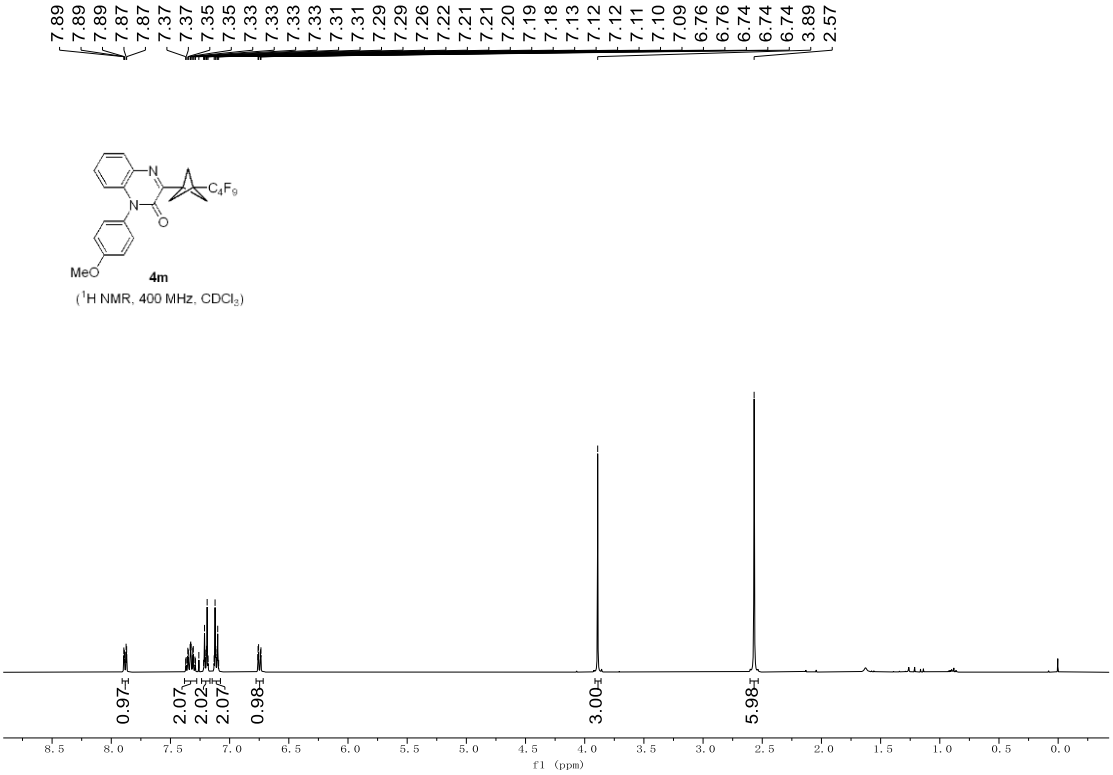

-81.07
-81.08
-81.09
-81.10
-81.11
-81.13
-81.14
-116.53
-116.54
-116.55
-116.57
-116.58
-116.59
-116.61
-116.62
-116.63
-122.26
-122.27
-122.29
-122.30
-122.31
-122.32
-122.33
-122.34
-125.92
-125.94
-125.95
-125.98
-126.01
-126.03
-126.05
4k
(19F NMR, 376 MHz, CDCl3)
2.56
2.18
2.11
2.00
f1 (ppm)

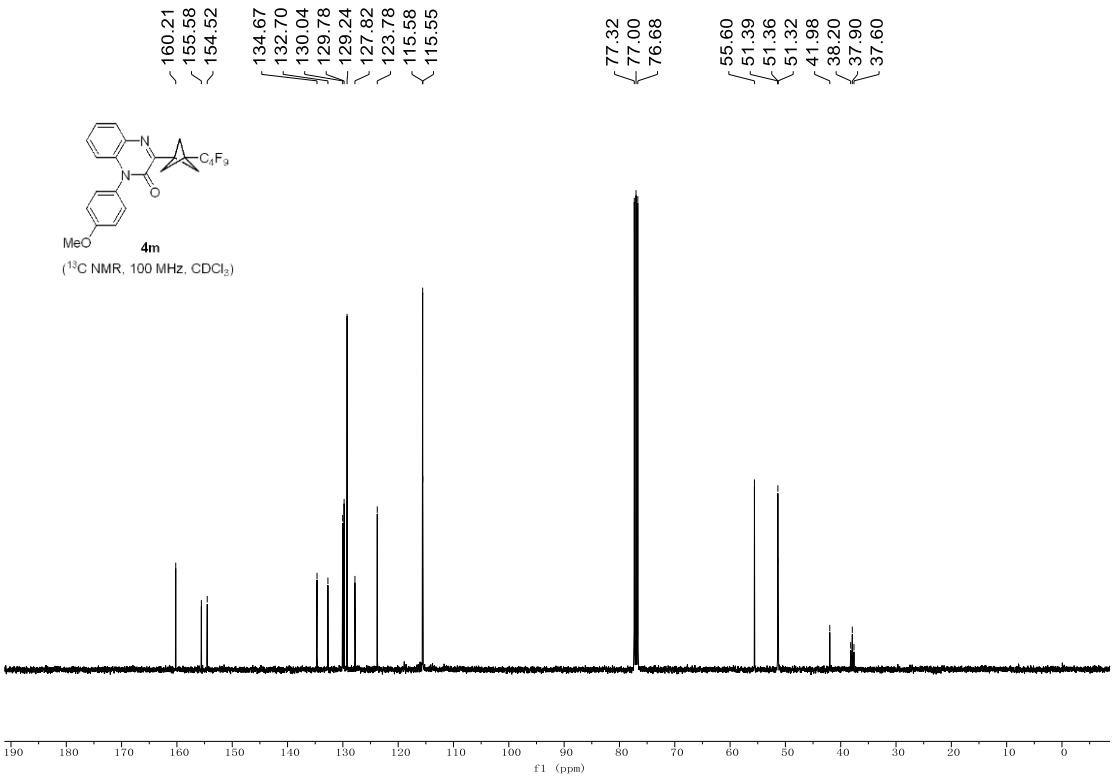
1-benzyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4l)

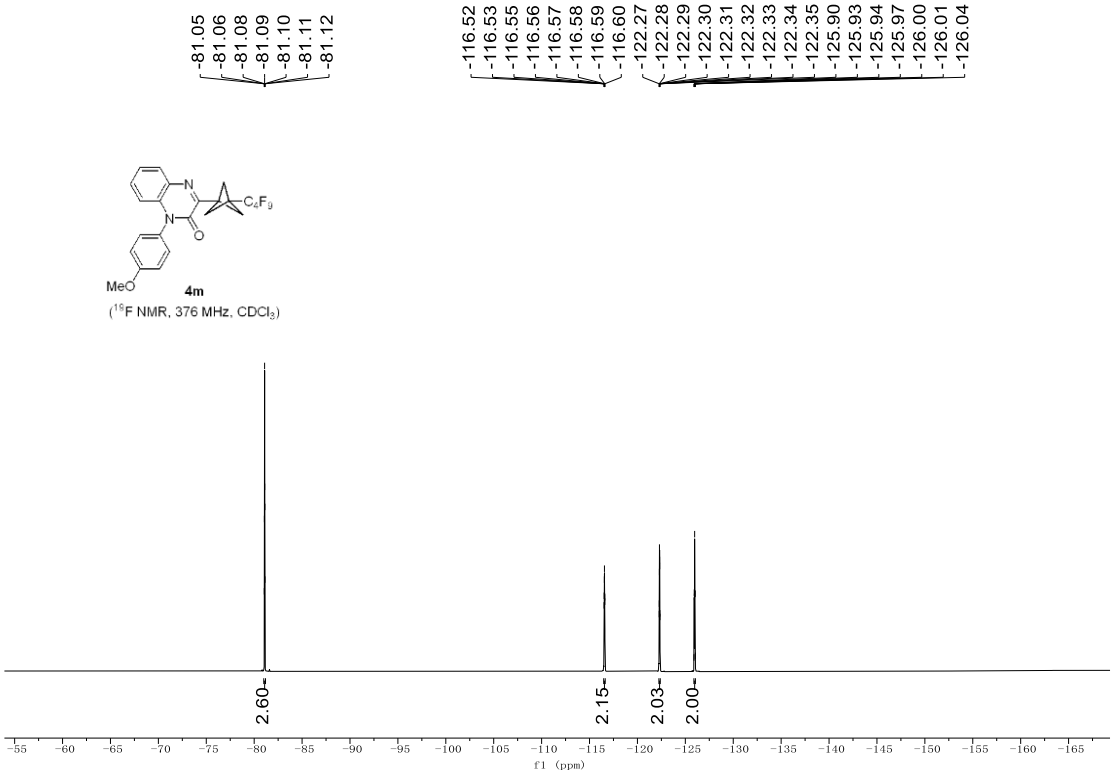




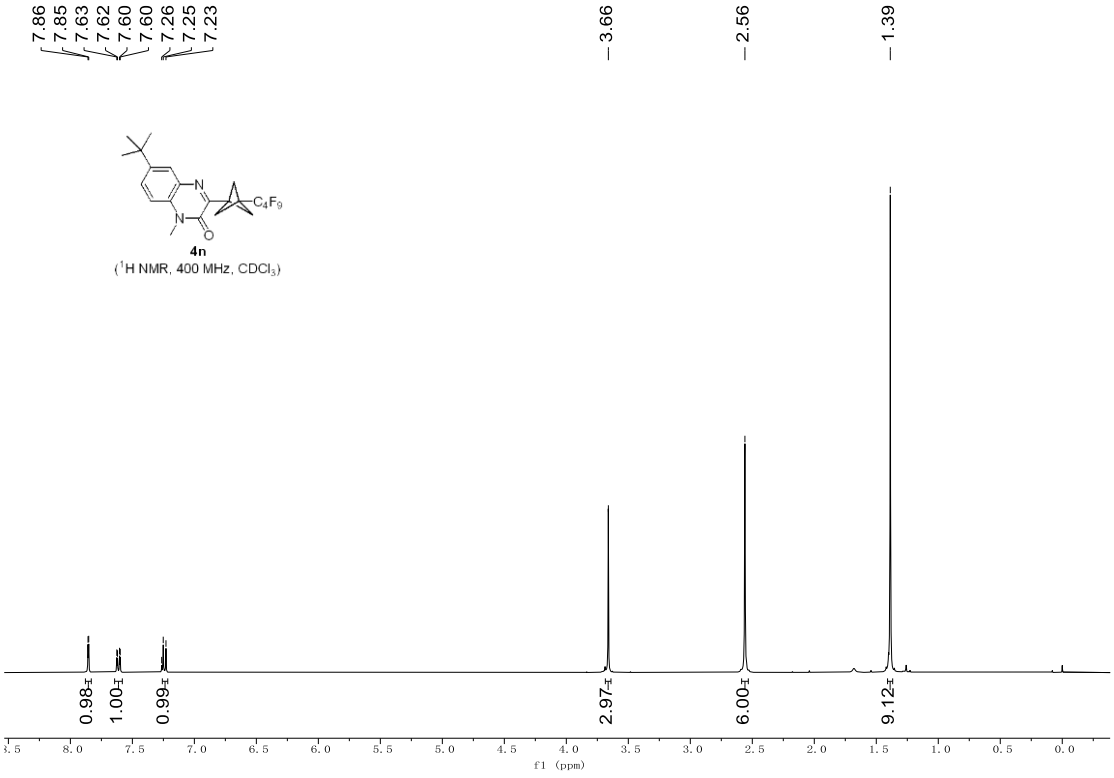
1-(4-methoxyphenyl)-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]penta n-1-yl)quinoxalin-2(1*H*)-one (4m)

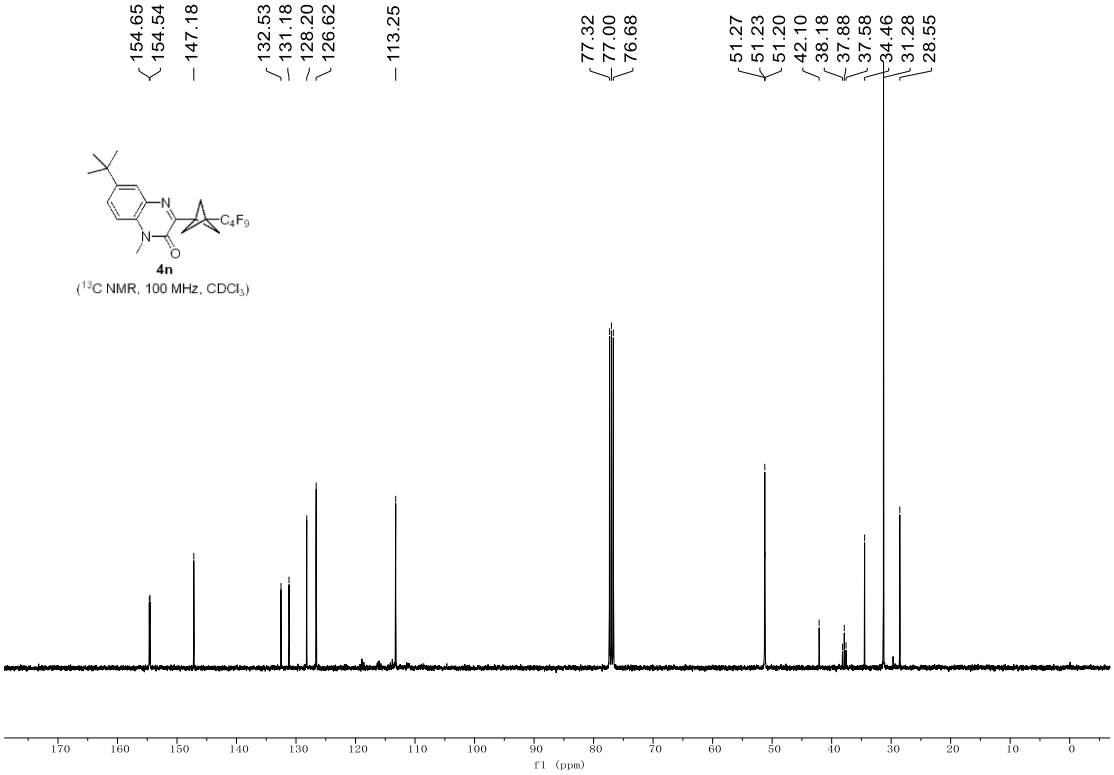


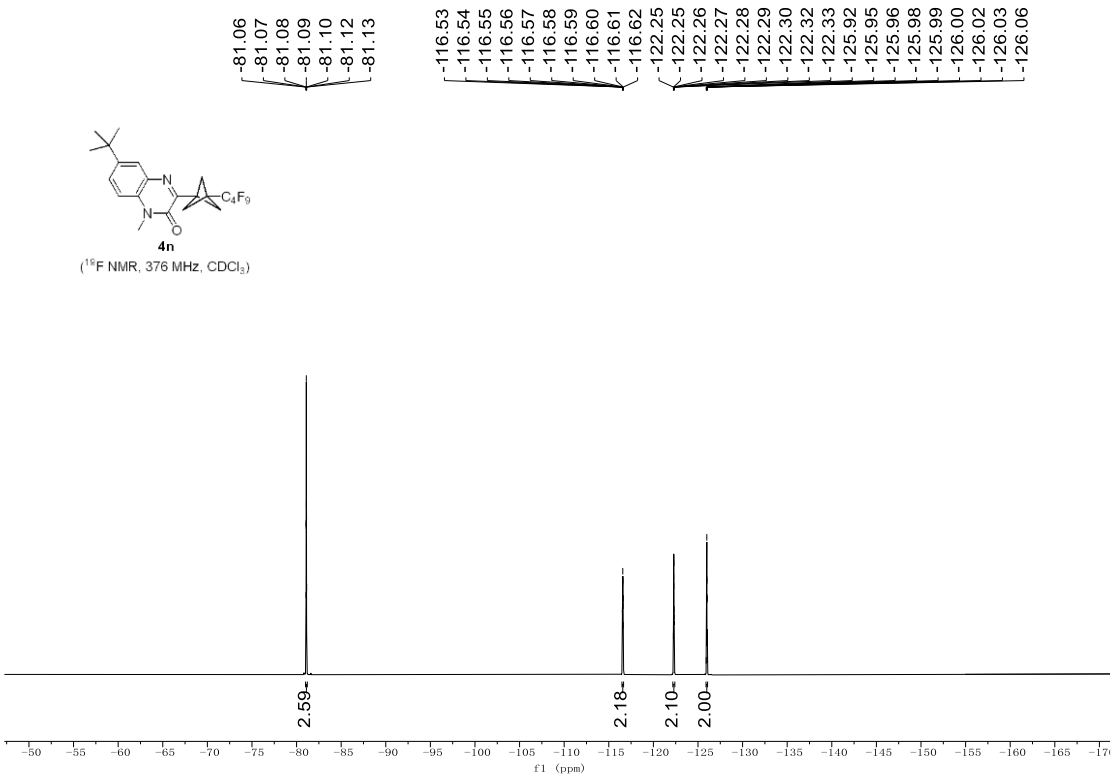

160.21
155.58
154.52
134.67
132.70
130.04
129.78
129.24
127.82
123.78
115.58
115.55
77.32
77.00
76.68
55.60
51.39
51.36
51.32
41.98
38.20
37.90
37.60
MeO
4m
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)


-81.05
-81.06
-81.08
-81.09
-81.10
-81.11
-81.12
-116.52
-116.53
-116.55
-116.56
-116.57
-116.58
-116.59
-116.60
-122.27
-122.28
-122.29
-122.30
-122.31
-122.32
-122.33
-122.34
-122.35
-125.90
-125.93
-125.94
-125.97
-126.00
-126.01
-126.04
MeO
4m
(19F NMR, 376 MHz, CDCl3)
2.60
2.15
2.03
2.00
f1 (ppm)

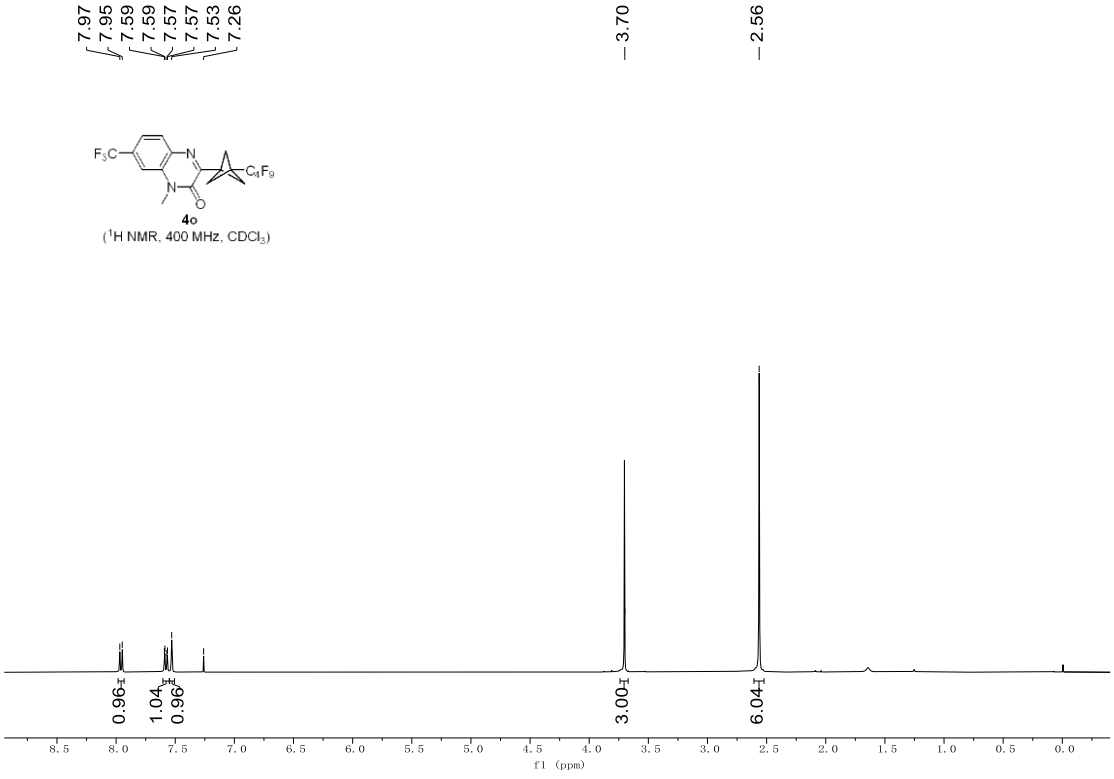
6-(tert-butyl)-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4n)

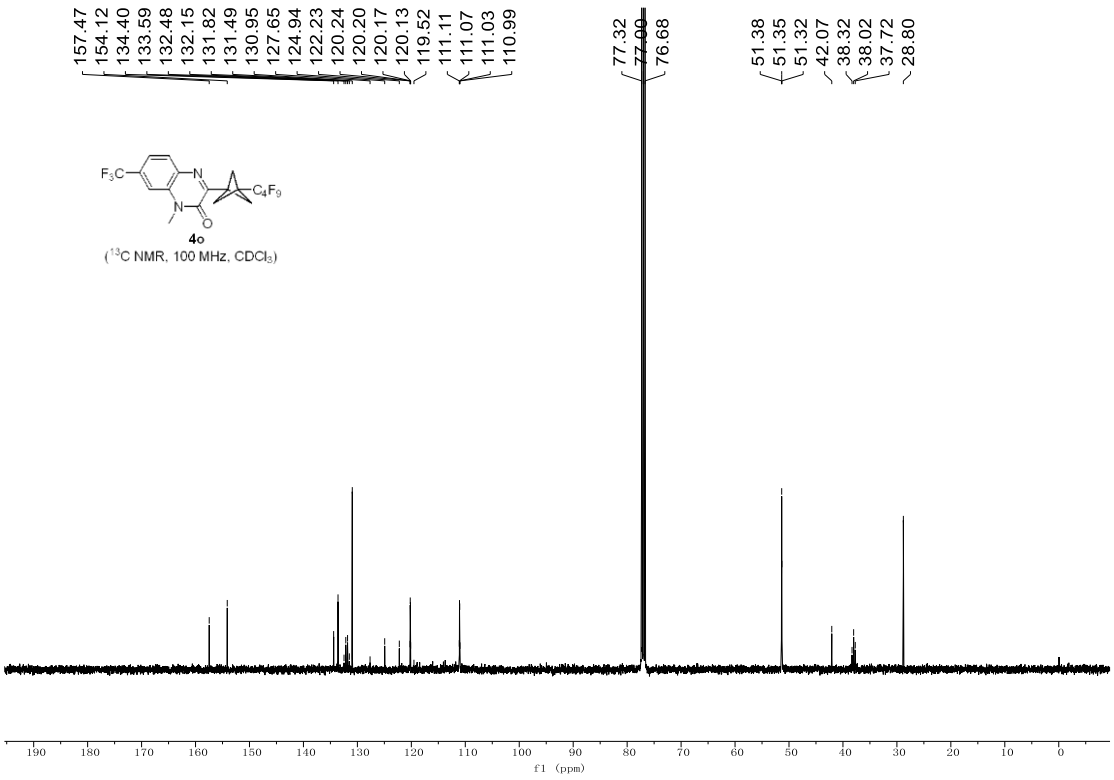


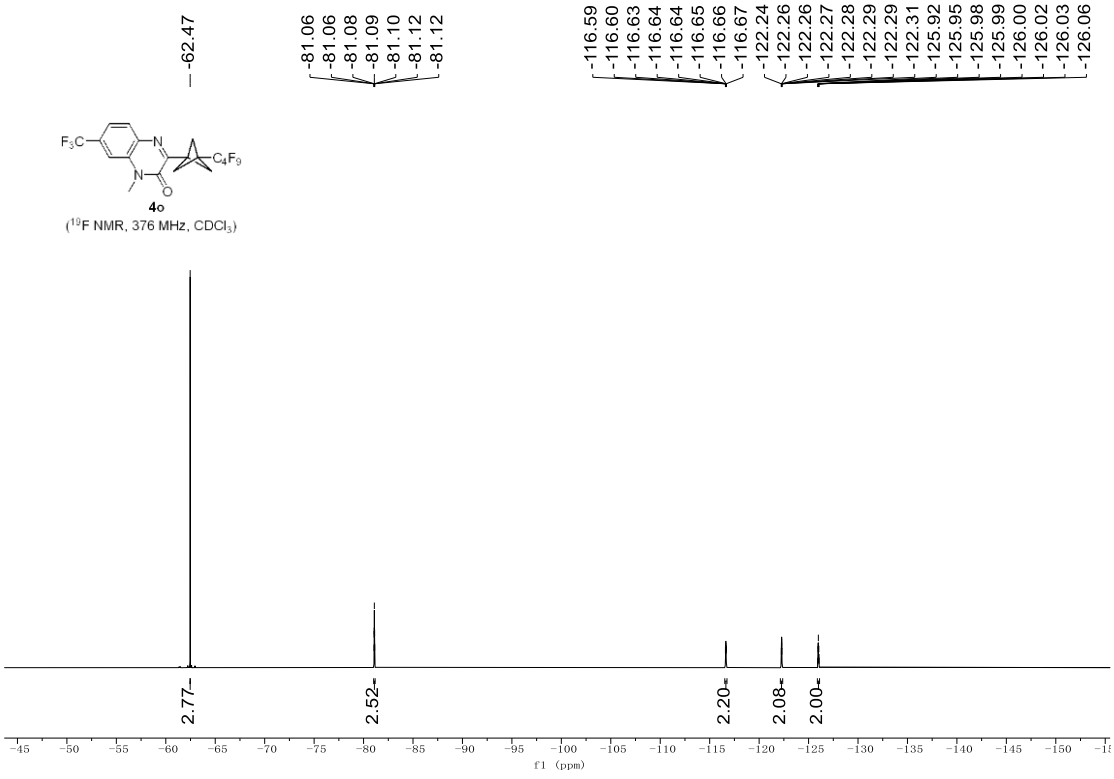




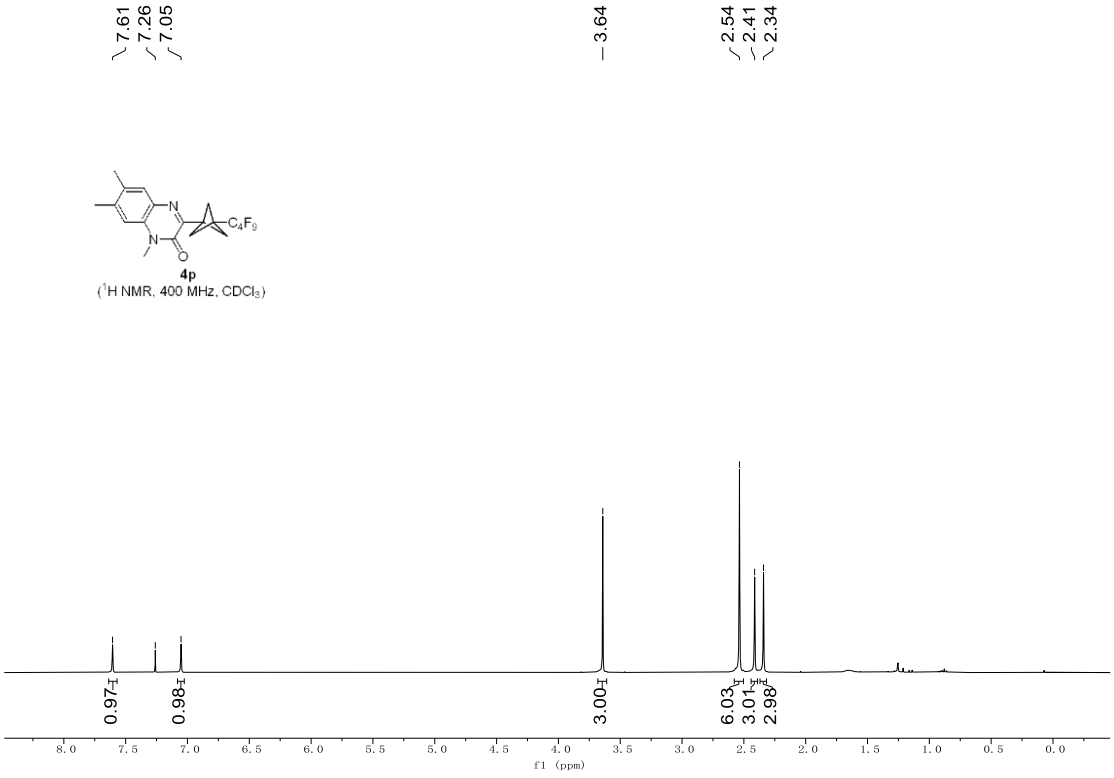
1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-7-(trifluoromethyl)quinoxalin-2(1*H*)-one (4o)

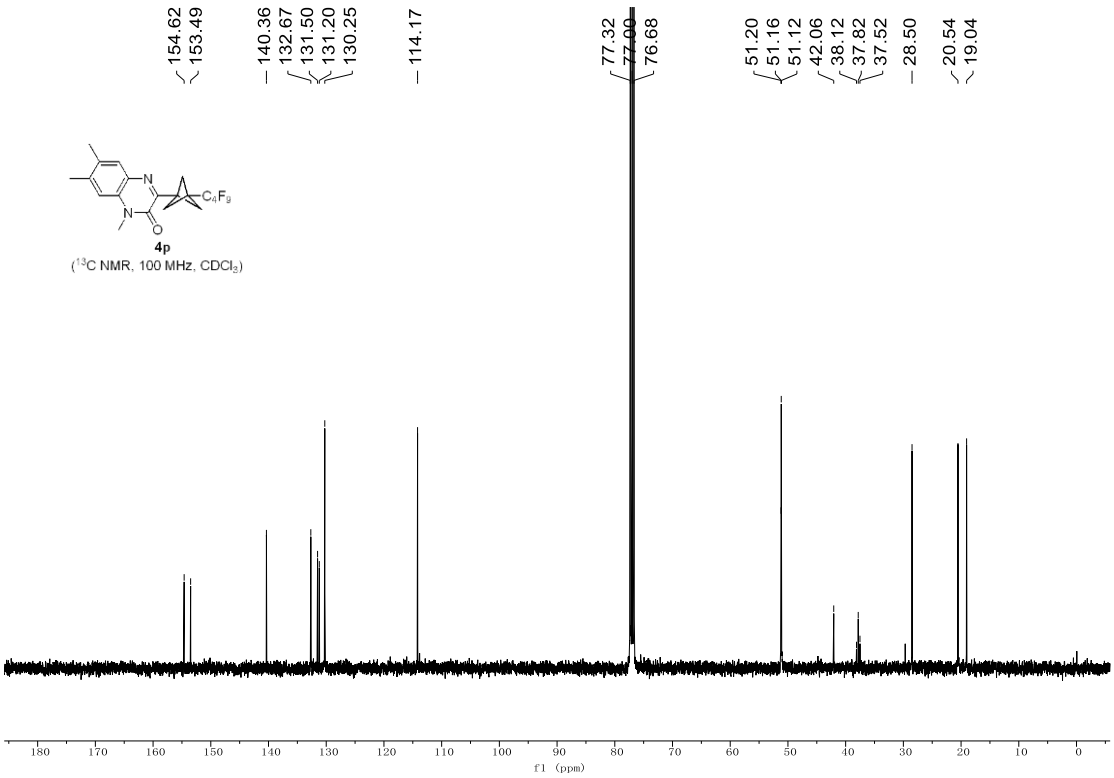


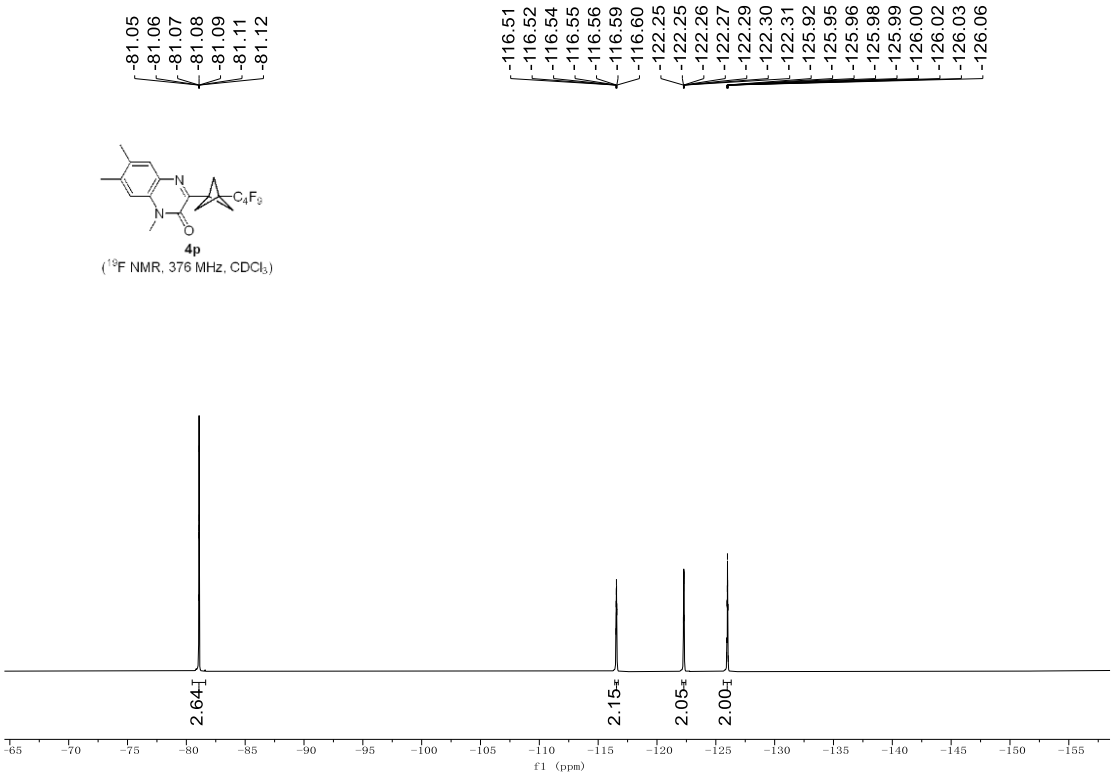

157.47
154.12
134.40
133.59
132.48
132.15
131.82
131.49
130.95
127.65
124.94
122.23
120.24
120.20
120.17
120.13
119.52
111.11
111.07
111.03
110.99
77.32
77.00
76.68
51.38
51.35
51.32
42.07
38.32
38.02
37.72
28.80
F3C
N
C4F9
N
O
4o
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)


-62.47
-81.06
-81.06
-81.08
-81.09
-81.10
-81.12
-81.12
-116.59
-116.60
-116.63
-116.64
-116.64
-116.65
-116.66
-116.67
-122.24
-122.26
-122.26
-122.27
-122.28
-122.29
-122.29
-122.31
-125.92
-125.95
-125.98
-125.99
-126.00
-126.02
-126.03
-126.06
F3C
N
C4F9
N
O
4o
(19F NMR, 376 MHz, CDCl3)
2.77
2.52
2.20
2.08
2.00
f1 (ppm)

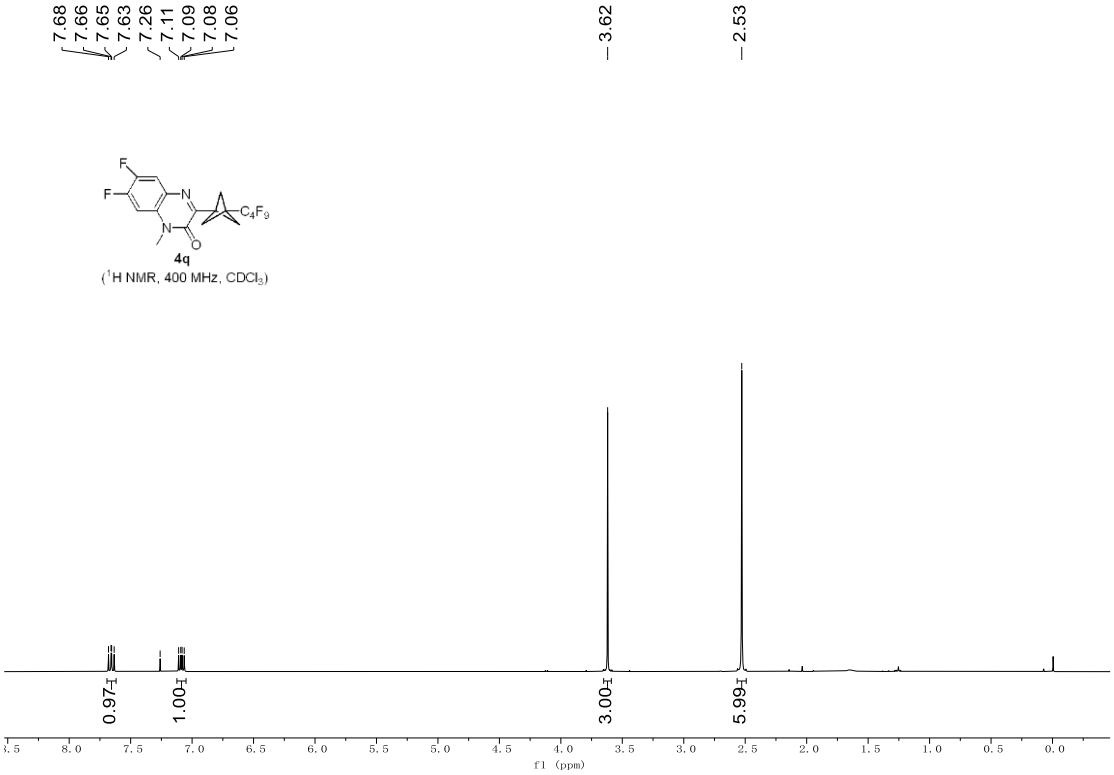
1,6,7-trimethyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4p)

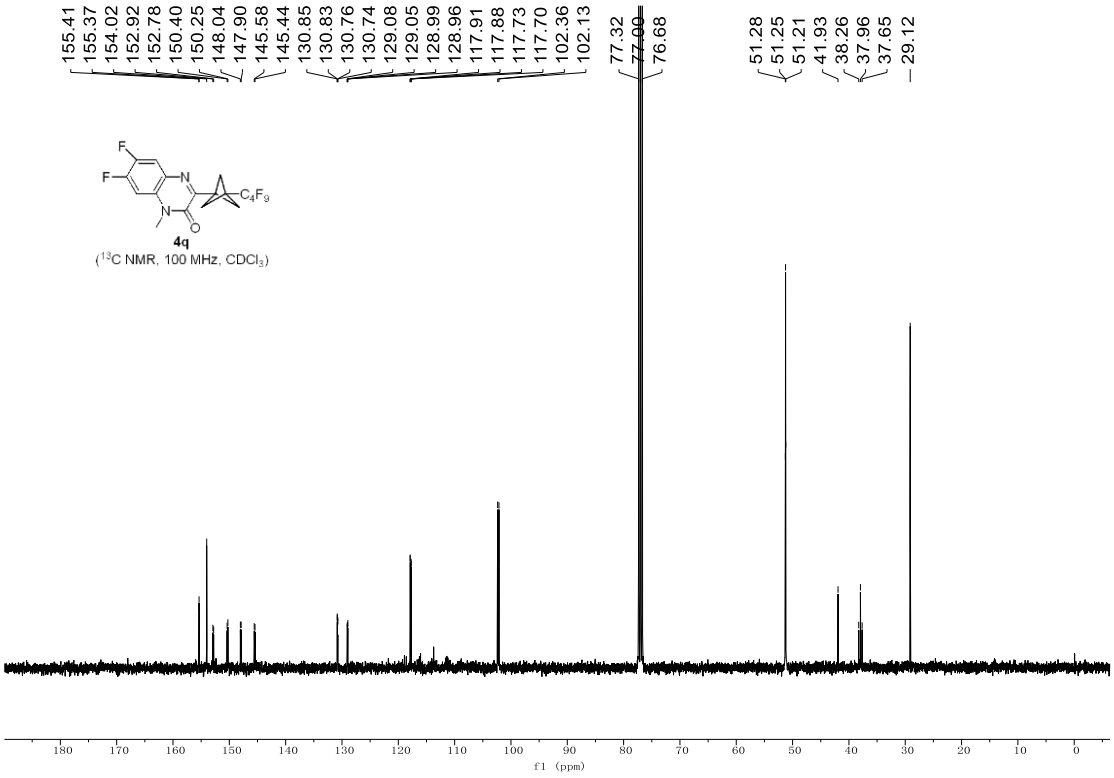


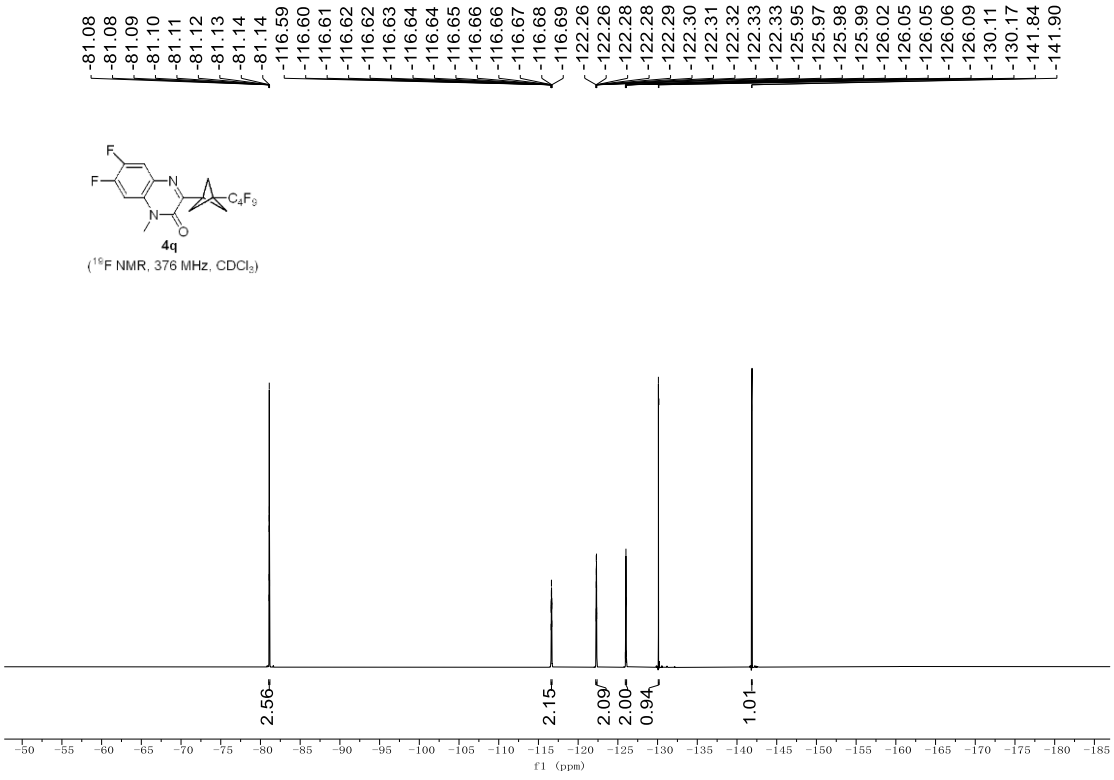




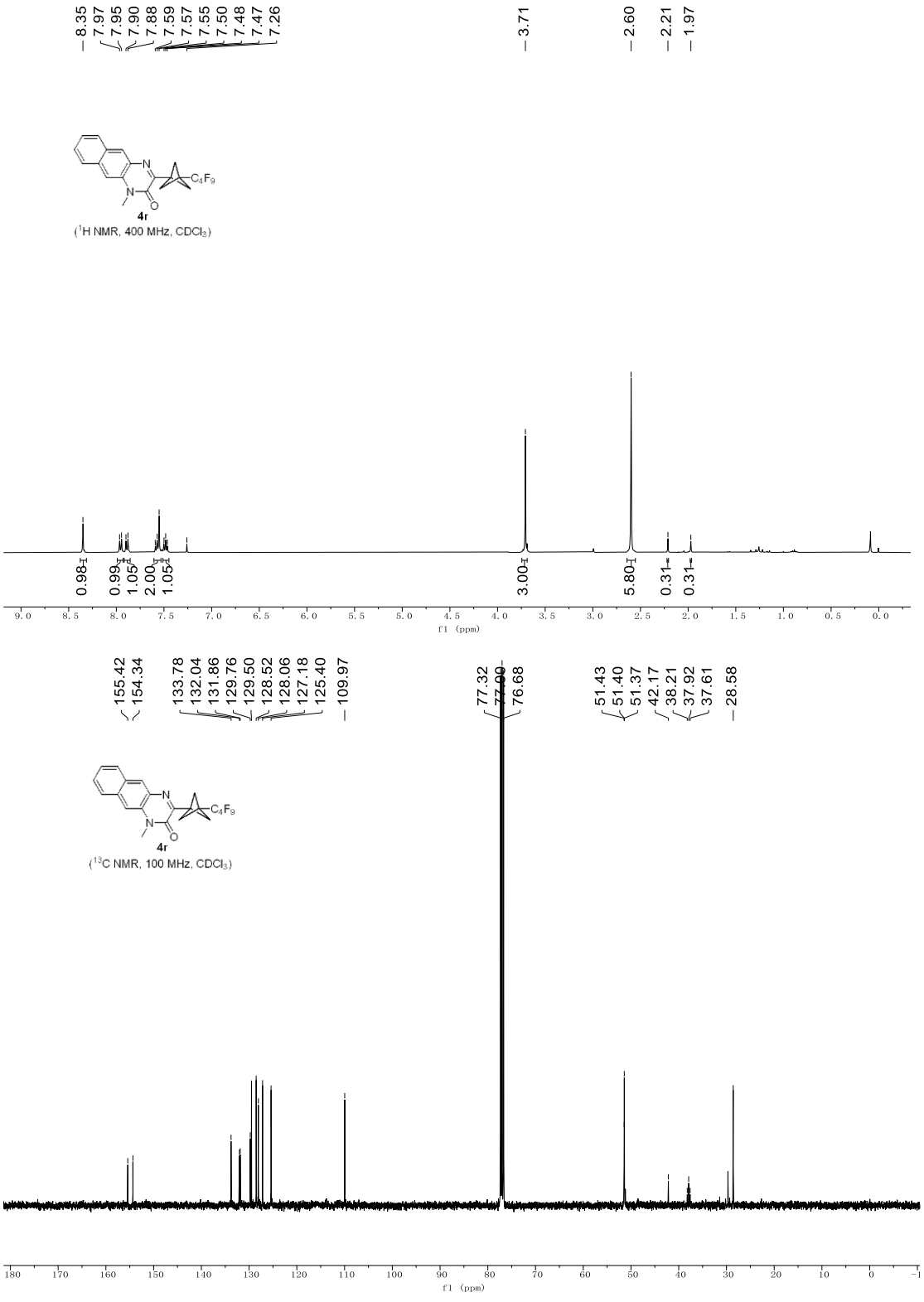
6,7-difluoro-1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (4q)

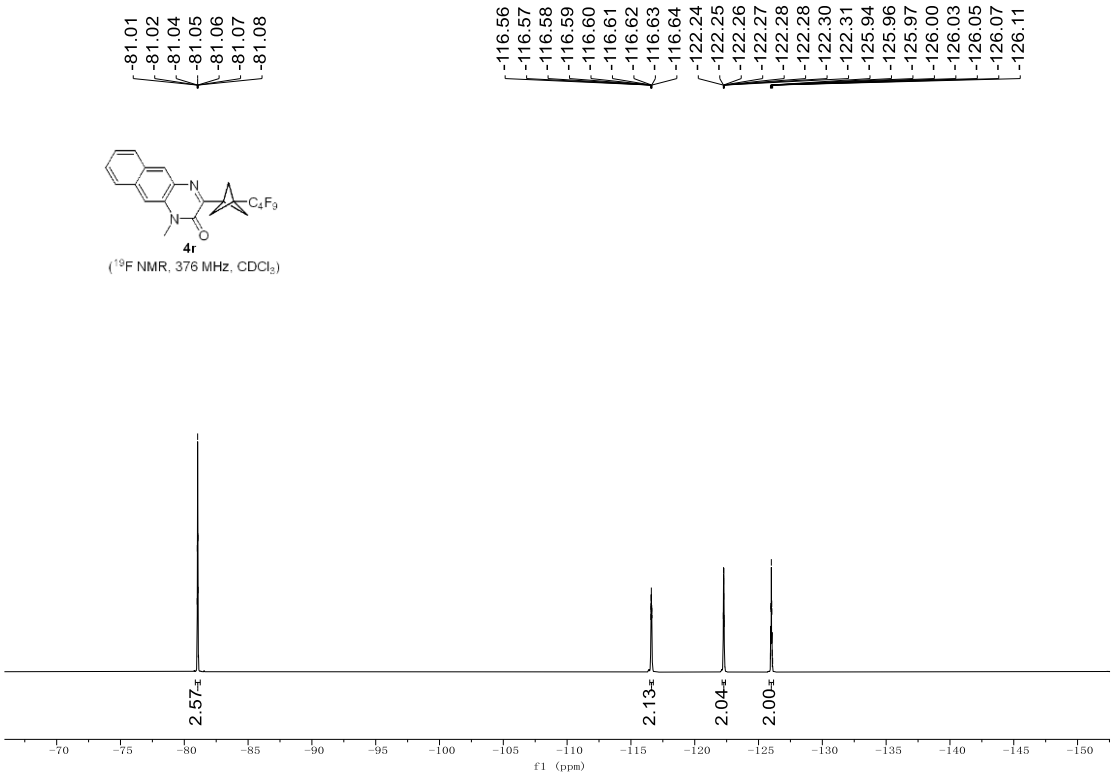



155.41
155.37
154.02
152.92
152.78
150.40
150.25
148.04
147.90
145.58
145.44
130.85
130.83
130.76
130.74
129.08
129.05
128.99
128.96
117.91
117.88
117.73
117.70
102.36
102.13
77.32
77.00
76.68
51.28
51.25
51.21
41.93
38.26
37.96
37.65
29.12
F
F
N
N
O
C4F9
4q
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)

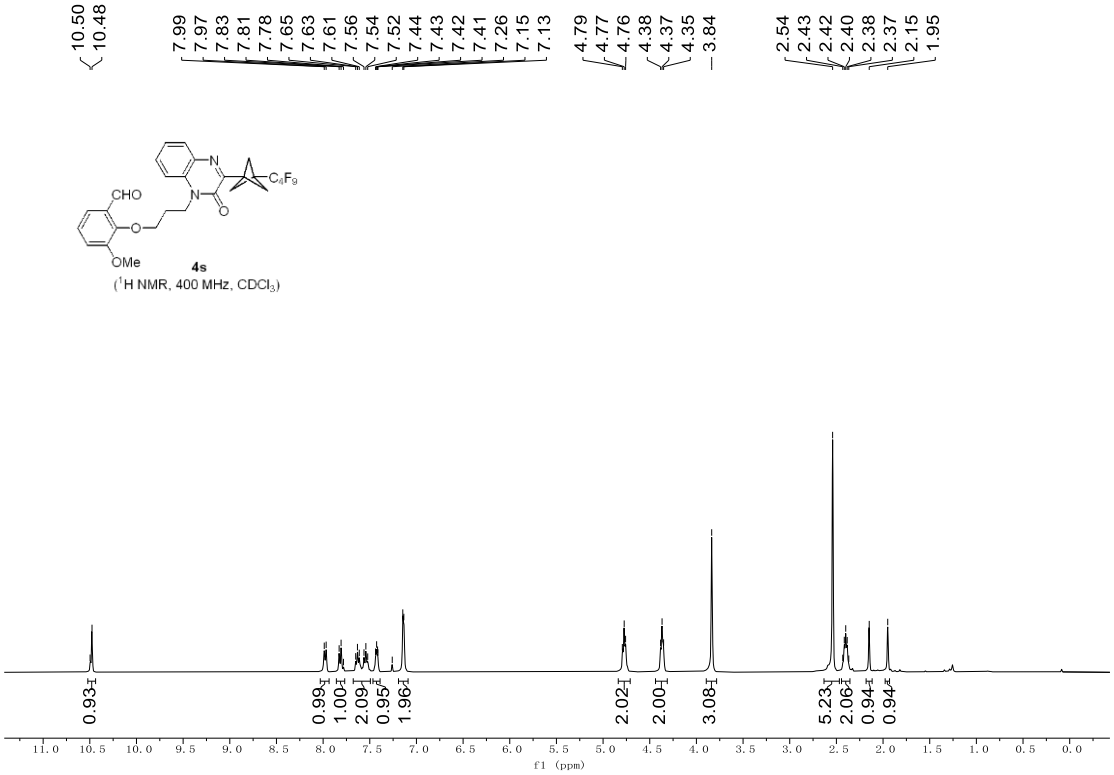

-81.08
-81.08
-81.09
-81.10
-81.11
-81.12
-81.13
-81.14
-81.14
-116.59
-116.60
-116.61
-116.62
-116.62
-116.63
-116.64
-116.64
-116.65
-116.66
-116.66
-116.67
-116.68
-116.69
-122.26
-122.26
-122.28
-122.28
-122.29
-122.30
-122.31
-122.32
-122.33
-122.33
-125.95
-125.97
-125.98
-125.99
-126.02
-126.05
-126.05
-126.06
-126.09
-130.11
-130.17
-141.84
-141.90
F
F
N
N
O
C4F9
4q
(19F NMR, 376 MHz, CDCl3)
2.56
2.15
2.09
2.00
0.94
1.01
f1 (ppm)

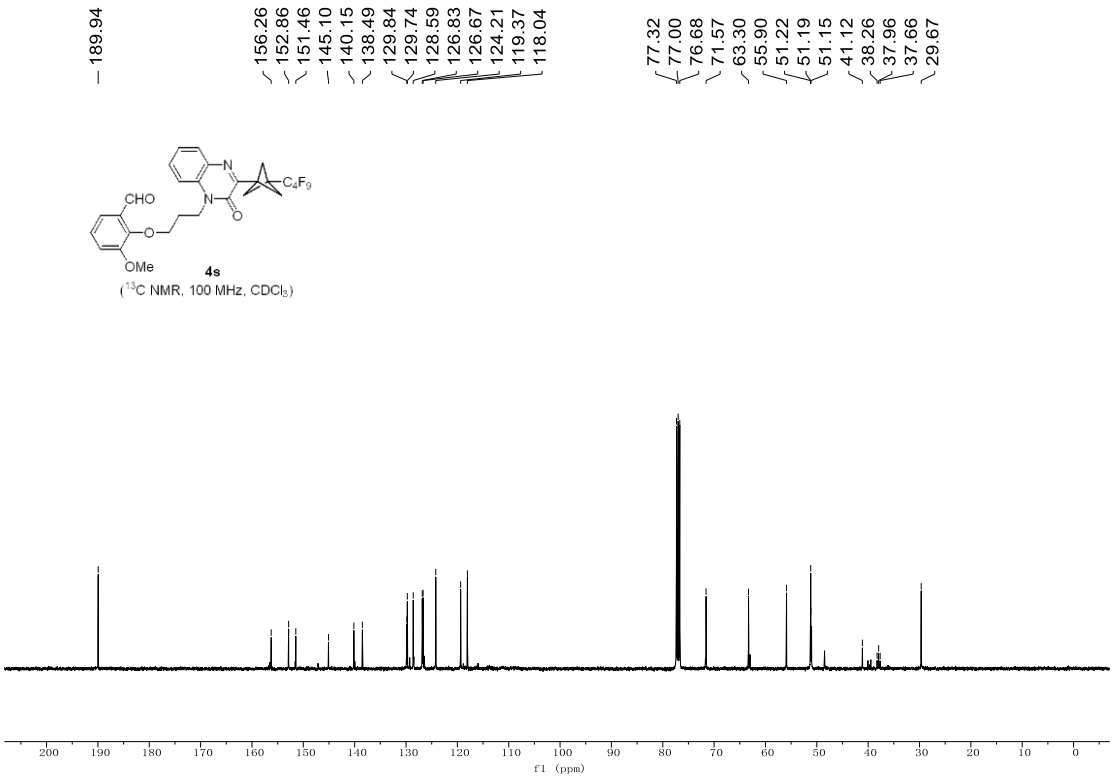
1-methyl-3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)benzo[g]quinoxalin-2(1*H*)-one (4r)

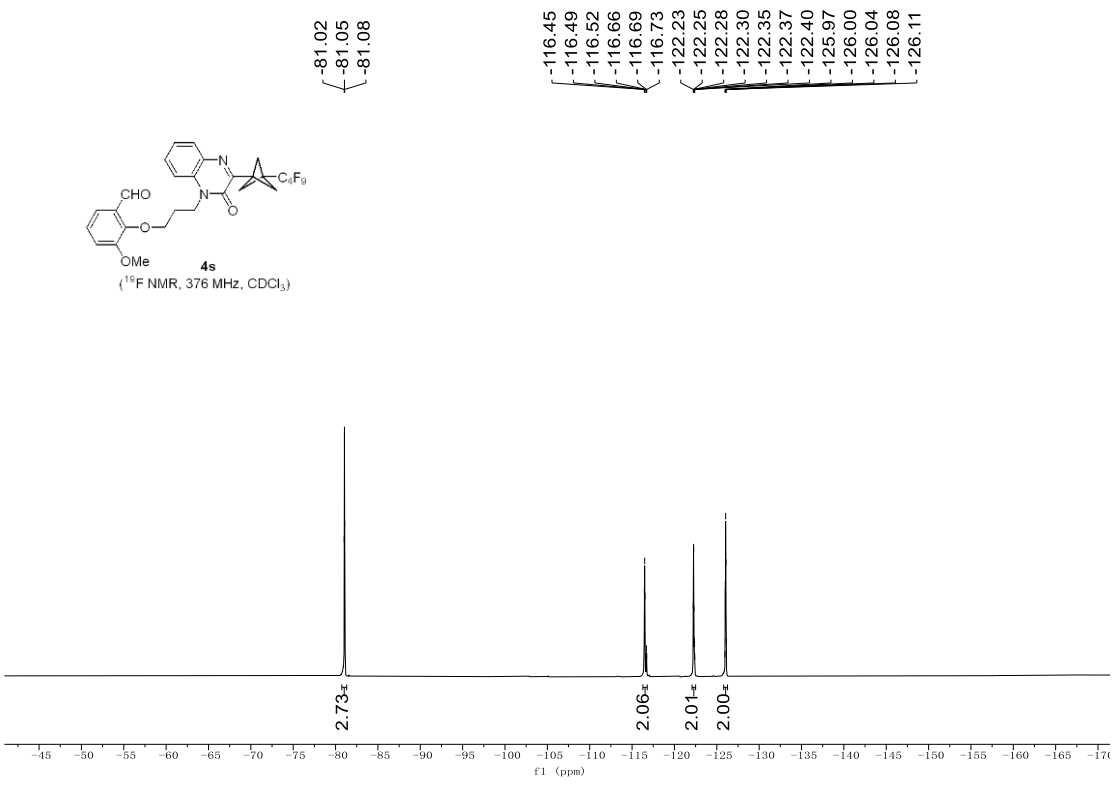




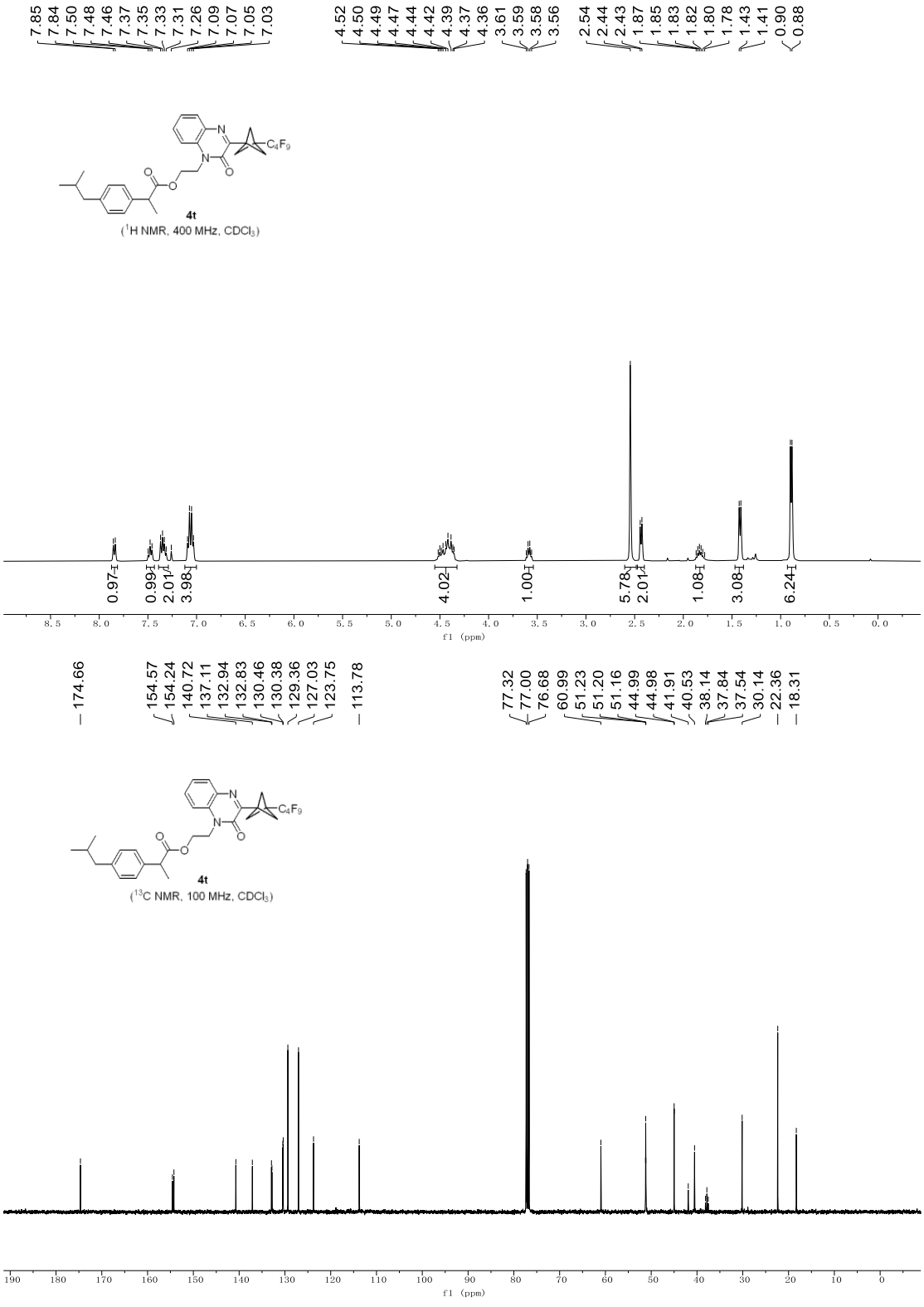
3-methoxy-2-(3-(3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-oxoquinoxalin-1(2*H*)-yl)propoxy)benzaldehyde (4s)

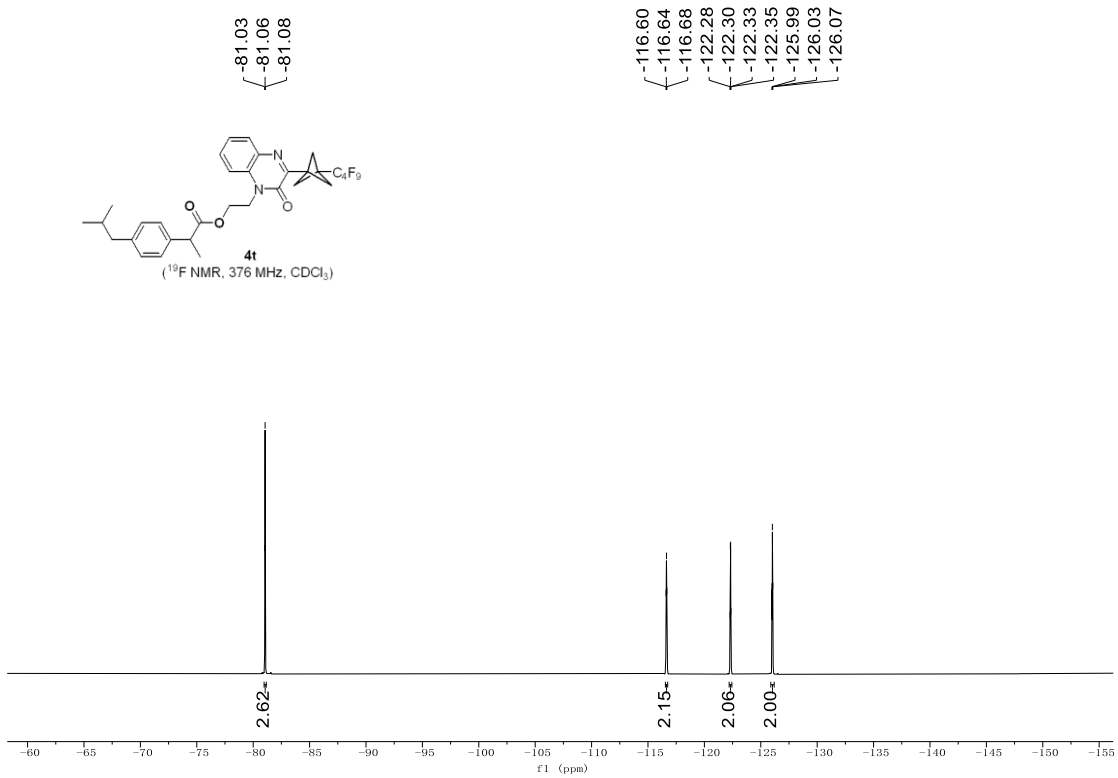



189.94
156.26
152.86
151.46
145.10
140.15
138.49
129.84
129.74
128.59
126.83
126.67
124.21
119.37
118.04
77.32
77.00
76.68
71.57
63.30
55.90
51.22
51.19
51.15
41.12
38.26
37.96
37.66
29.67
CHO
OMe
C_4F_9
4s
(^{13}C NMR, 100 MHz, $CDCl_3$)
f1 (ppm)

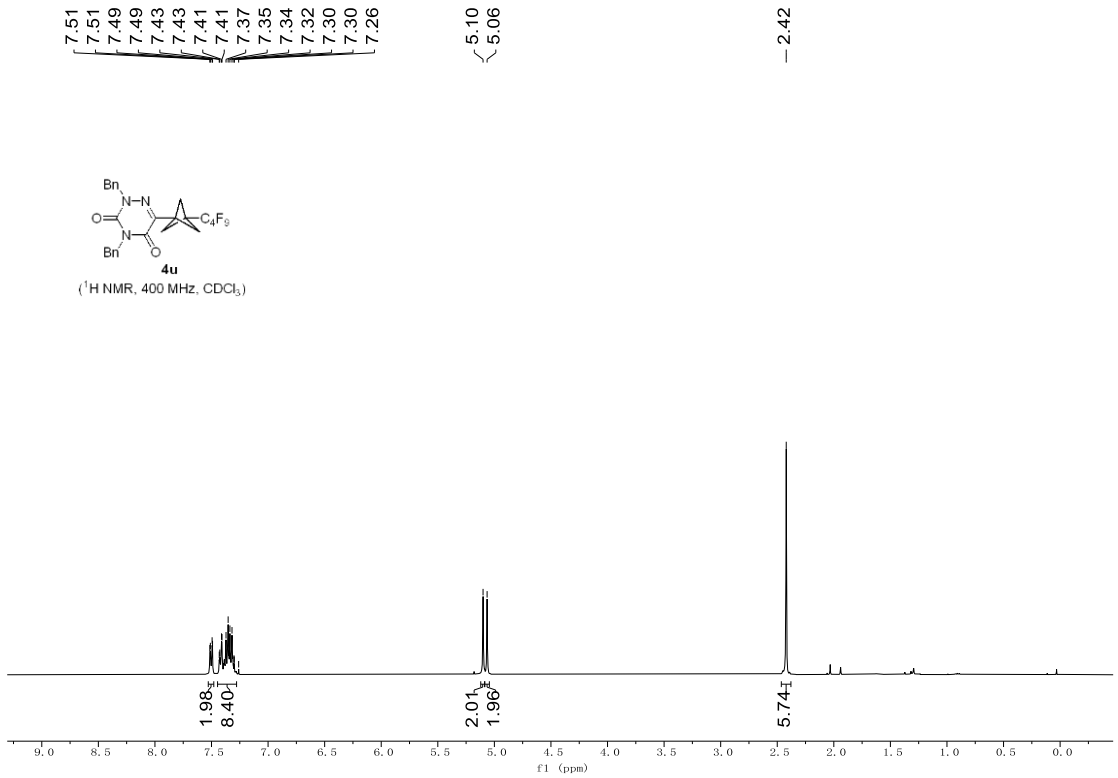

-81.02
-81.05
-81.08
-116.45
-116.49
-116.52
-116.66
-116.69
-116.73
-122.23
-122.25
-122.28
-122.30
-122.35
-122.37
-122.40
-125.97
-126.00
-126.04
-126.08
-126.11
CHO
OMe
C_4F_9
4s
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.73
2.06
2.01
2.00
f1 (ppm)

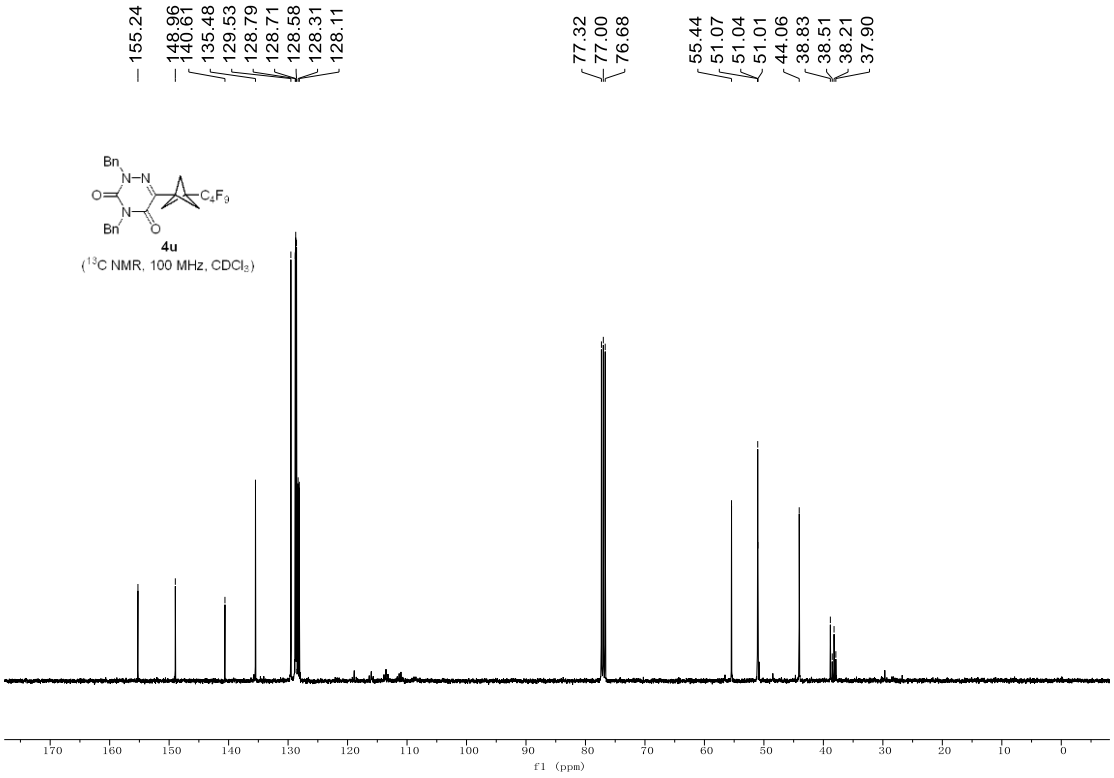
2-(3-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-oxoquinoxalin-1(2*H*)-yl)ethyl 2-(4-isobutylphenyl)propanoate (4t)

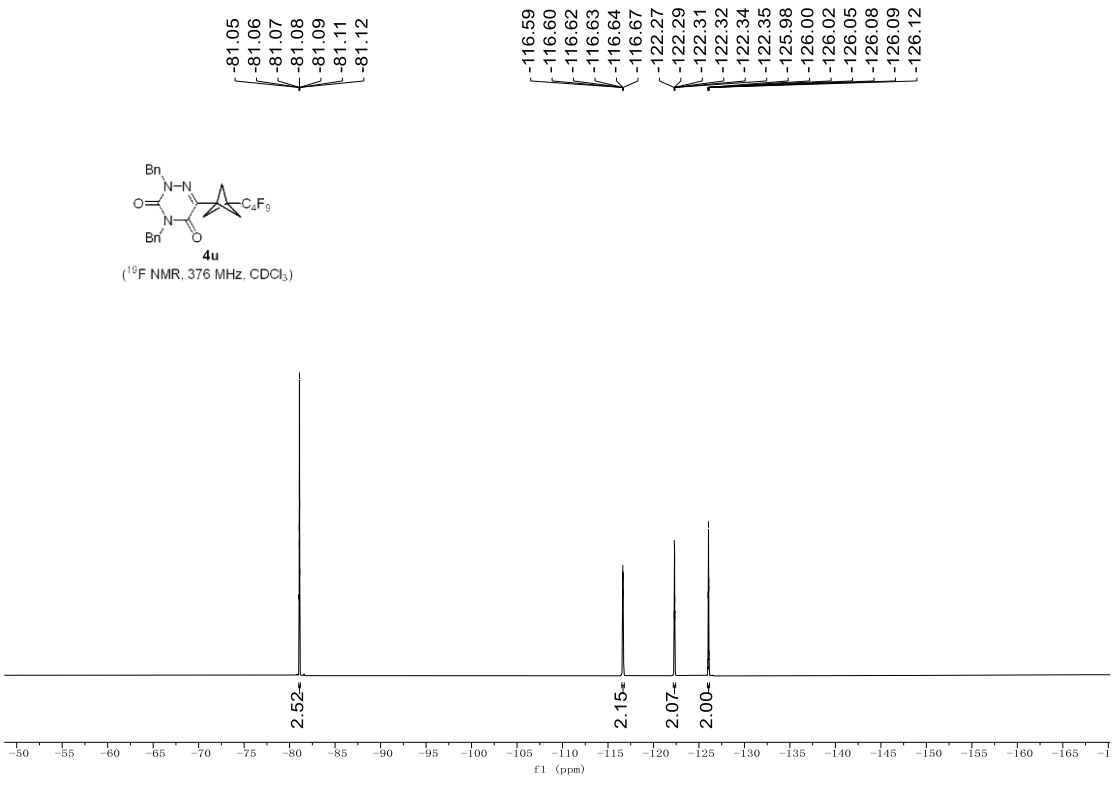




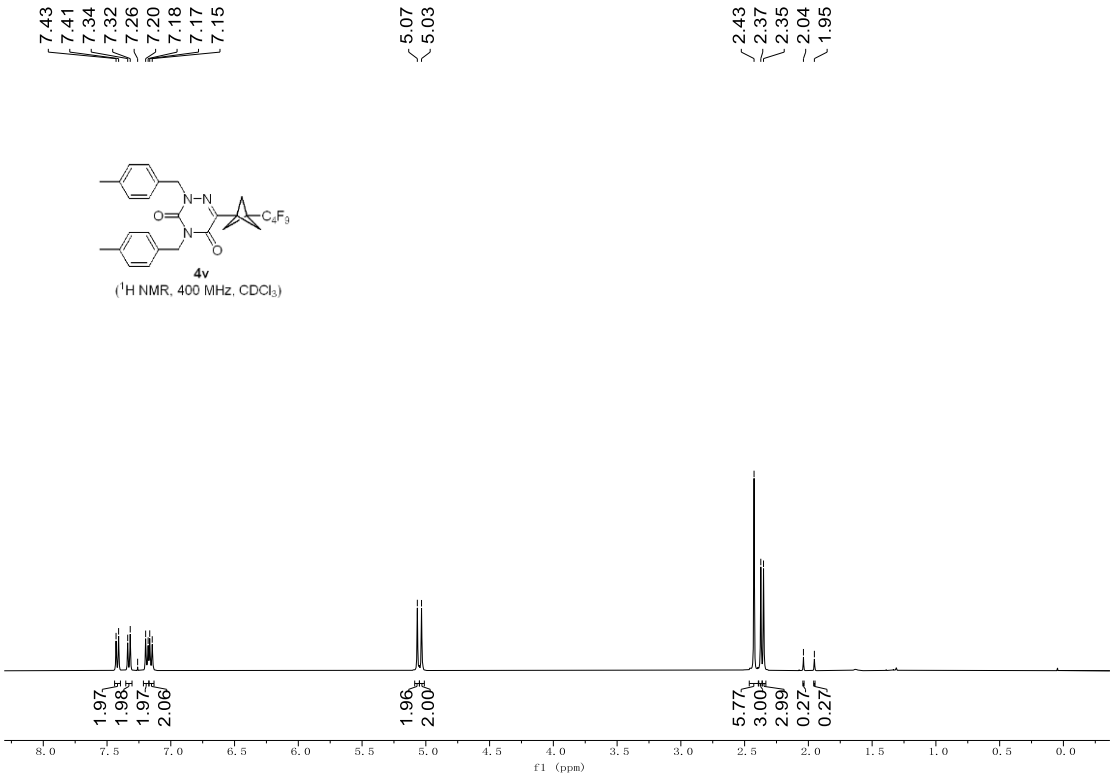
2,4-dibenzyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4u)

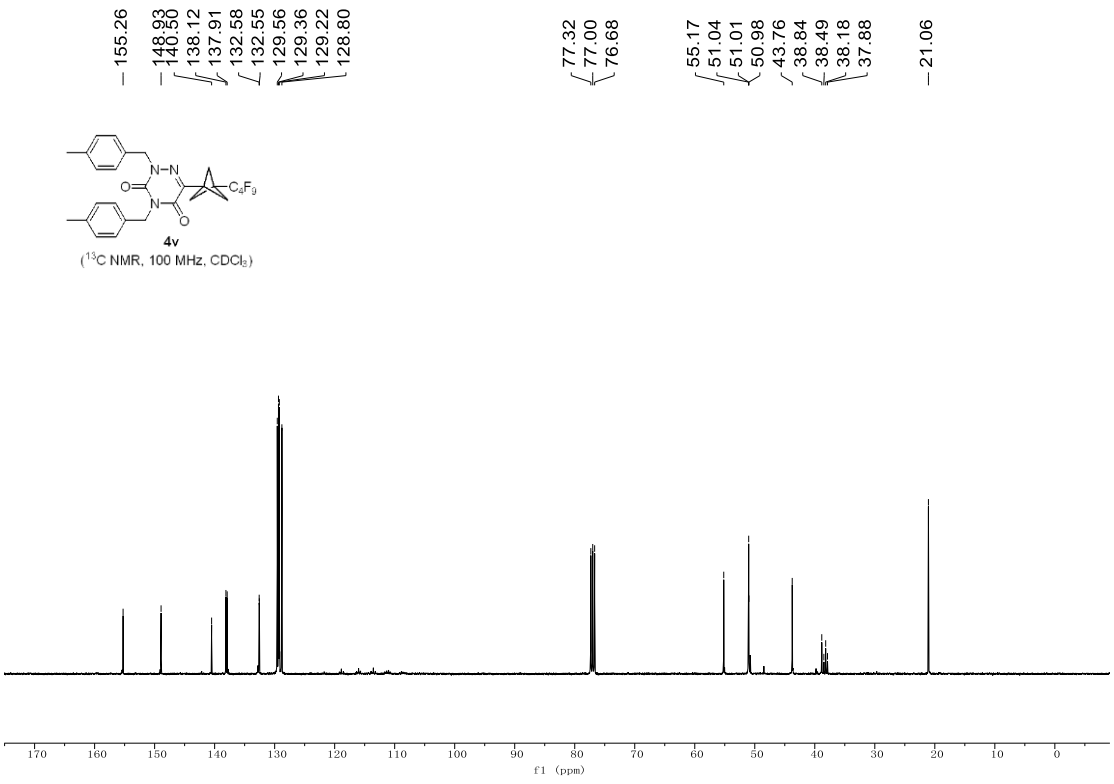


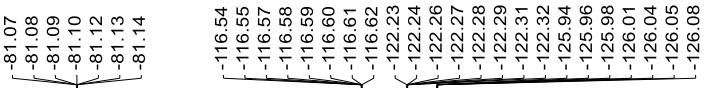

155.24
148.96
140.61
135.48
129.53
128.79
128.71
128.58
128.31
128.11
77.32
77.00
76.68
55.44
51.07
51.04
51.01
44.06
38.83
38.51
38.21
37.90
Bn
N–N
O
N
Bn
O
C_4F_9
4u
(^{13}C NMR, 100 MHz, $CDCl_3$)
f1 (ppm)

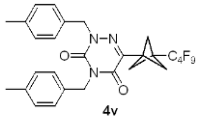

-81.05
-81.06
-81.07
-81.08
-81.09
-81.11
-81.12
-116.59
-116.60
-116.62
-116.63
-116.64
-116.67
-122.27
-122.29
-122.31
-122.32
-122.34
-122.35
-125.98
-126.00
-126.02
-126.05
-126.08
-126.09
-126.12
Bn
N–N
O
N
Bn
O
C_4F_9
4u
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.52
2.15
2.07
2.00
f1 (ppm)

2,4-bis(4-methylbenzyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4v)



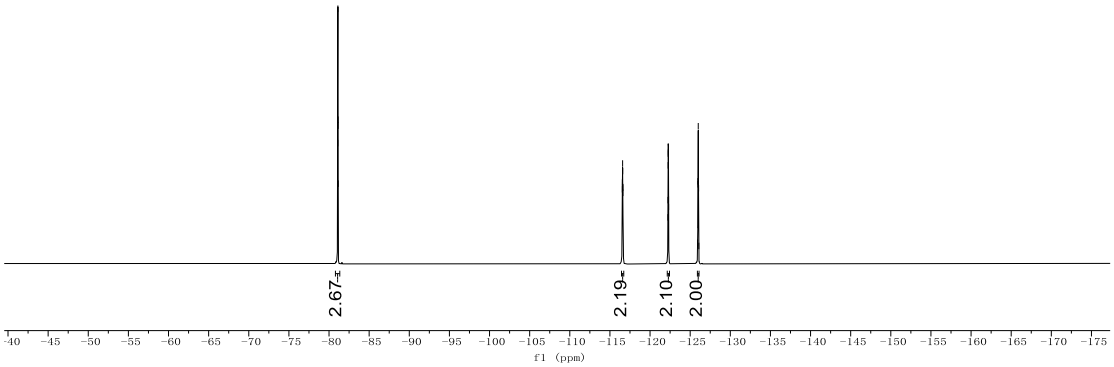




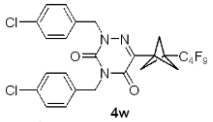


4v

(^{19}F NMR, 376 MHz, $CDCl_3$)

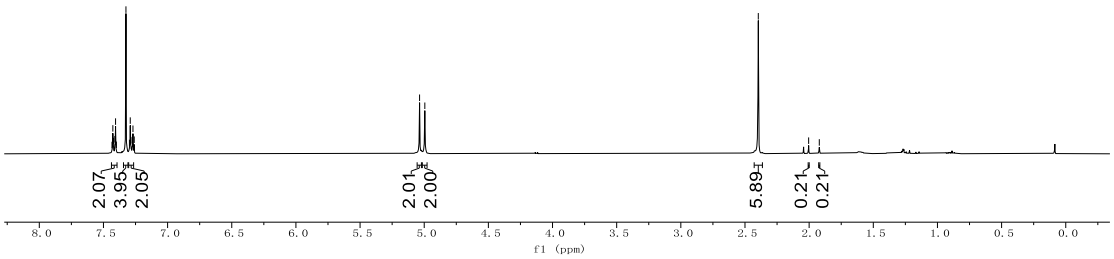


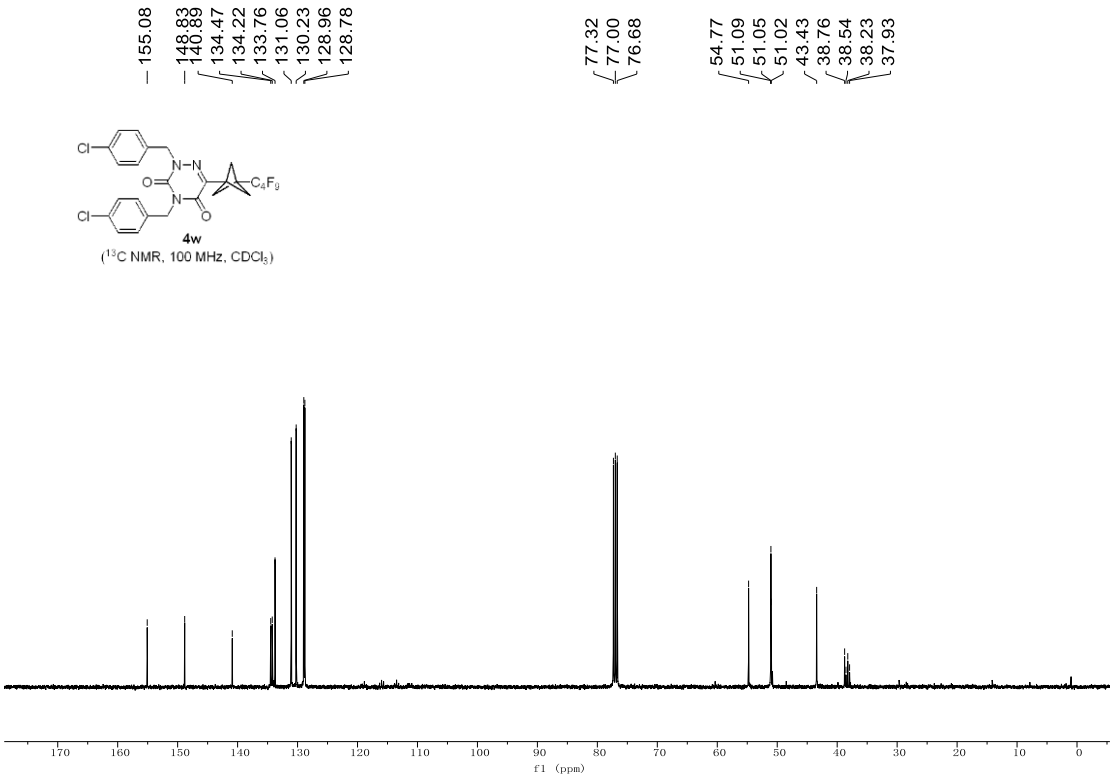
2,4-bis(4-chlorobenzyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4w)

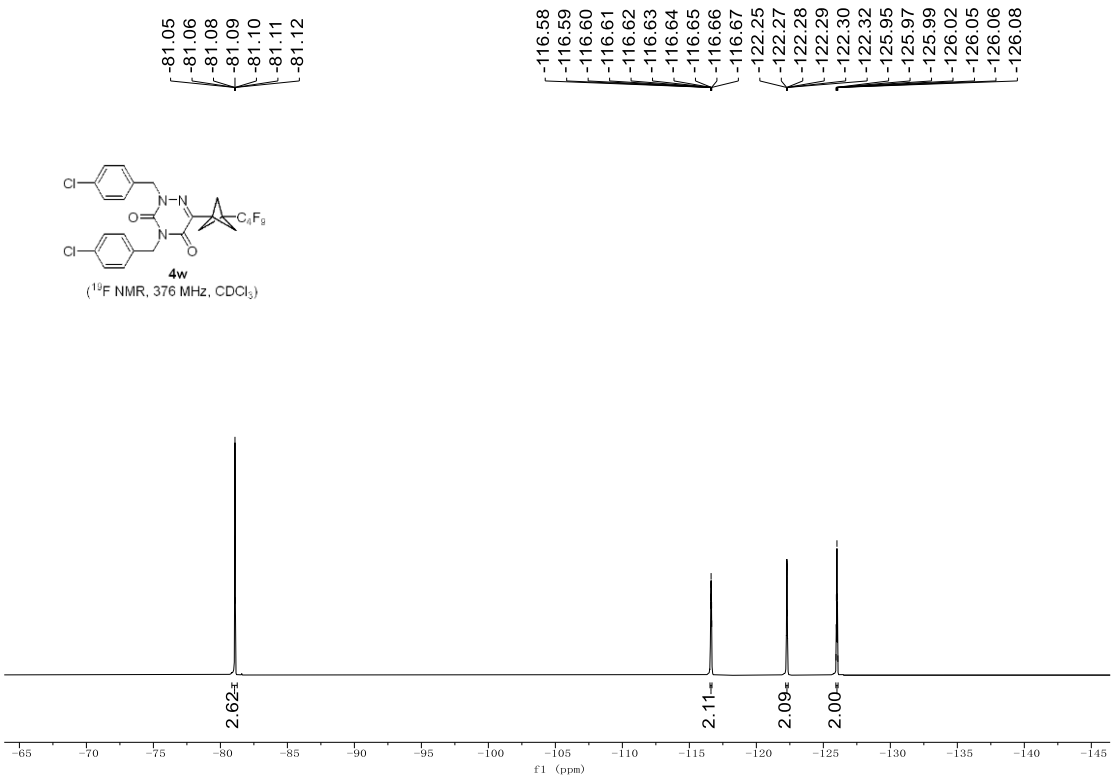


4w

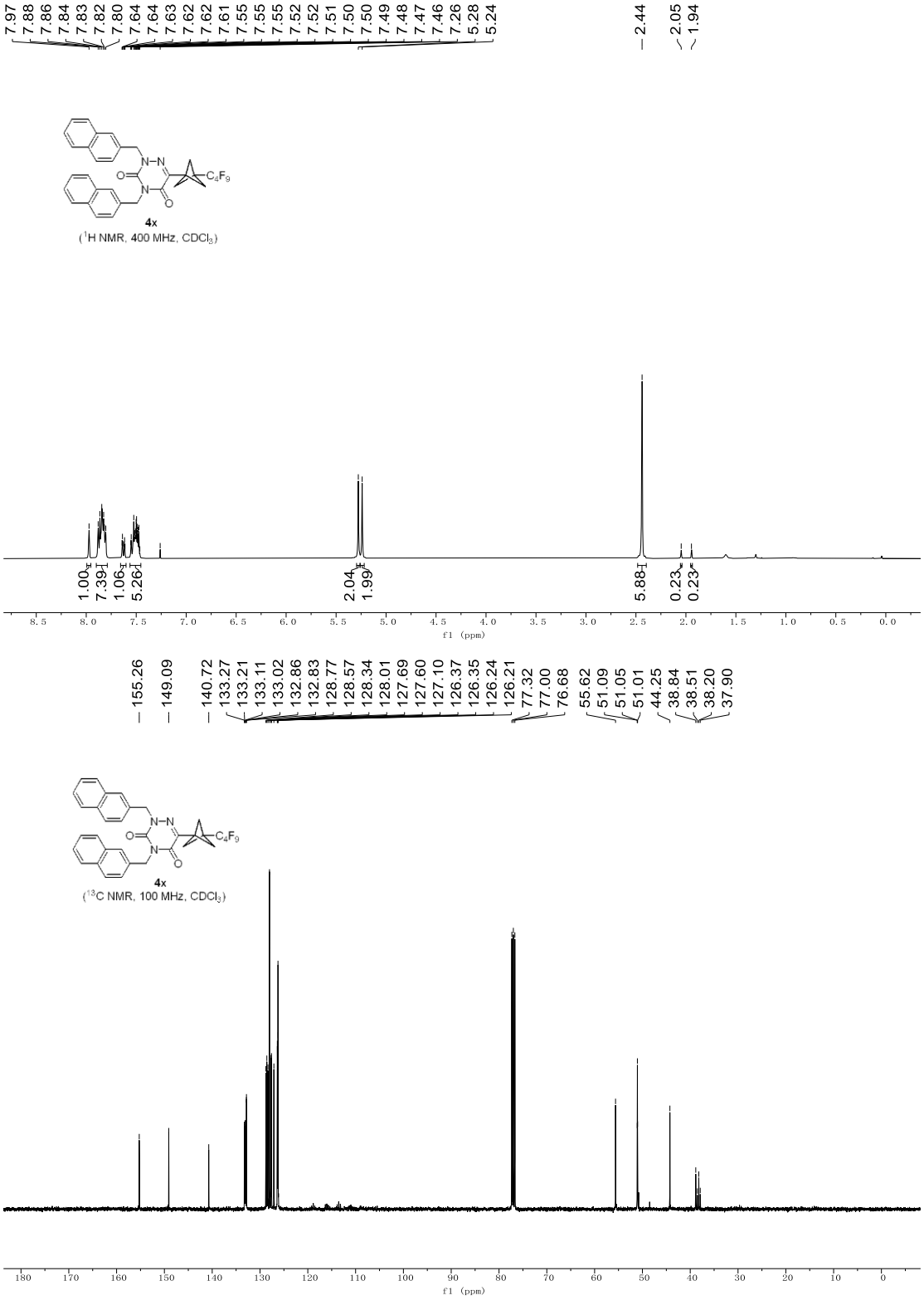
(^{1}H NMR, 400 MHz, $CDCl_3$)

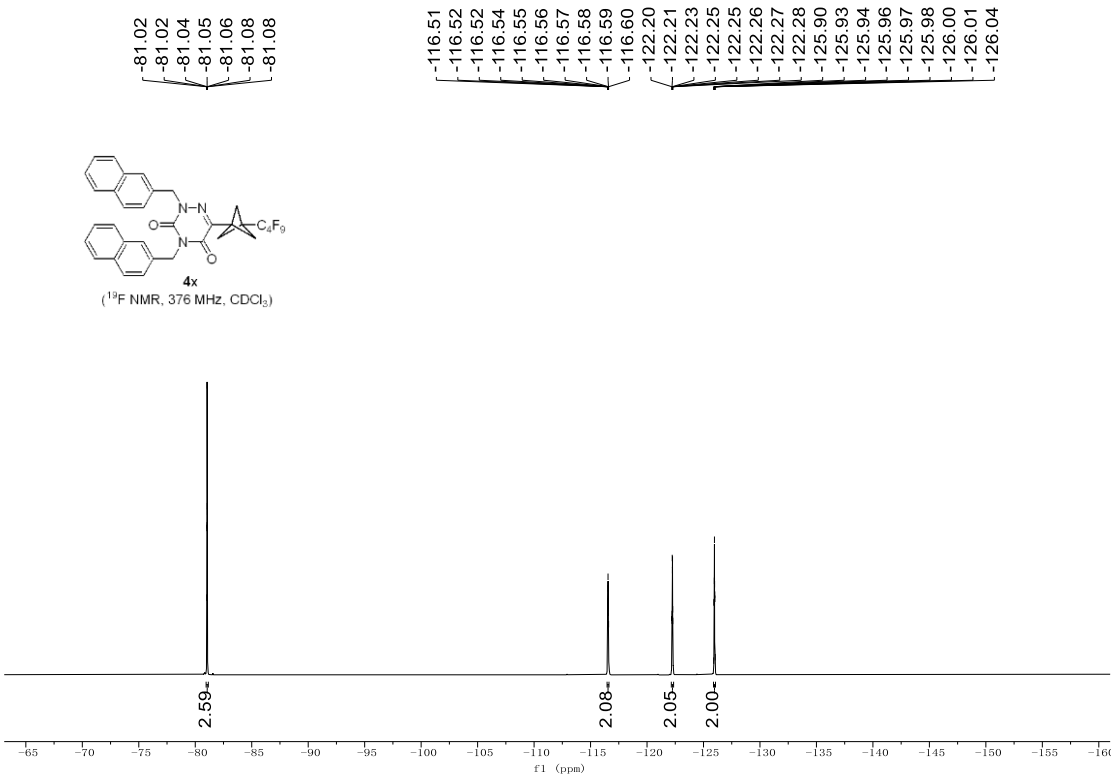



155.08
148.83
140.89
134.47
134.22
133.76
131.06
130.23
128.96
128.78
77.32
77.00
76.68
54.77
51.09
51.05
51.02
43.43
38.76
38.54
38.23
37.93
Cl
N–N
O
N
O
Cl
C_4F_9
4w
(^{13}C NMR, 100 MHz, $CDCl_3$)
f1 (ppm)

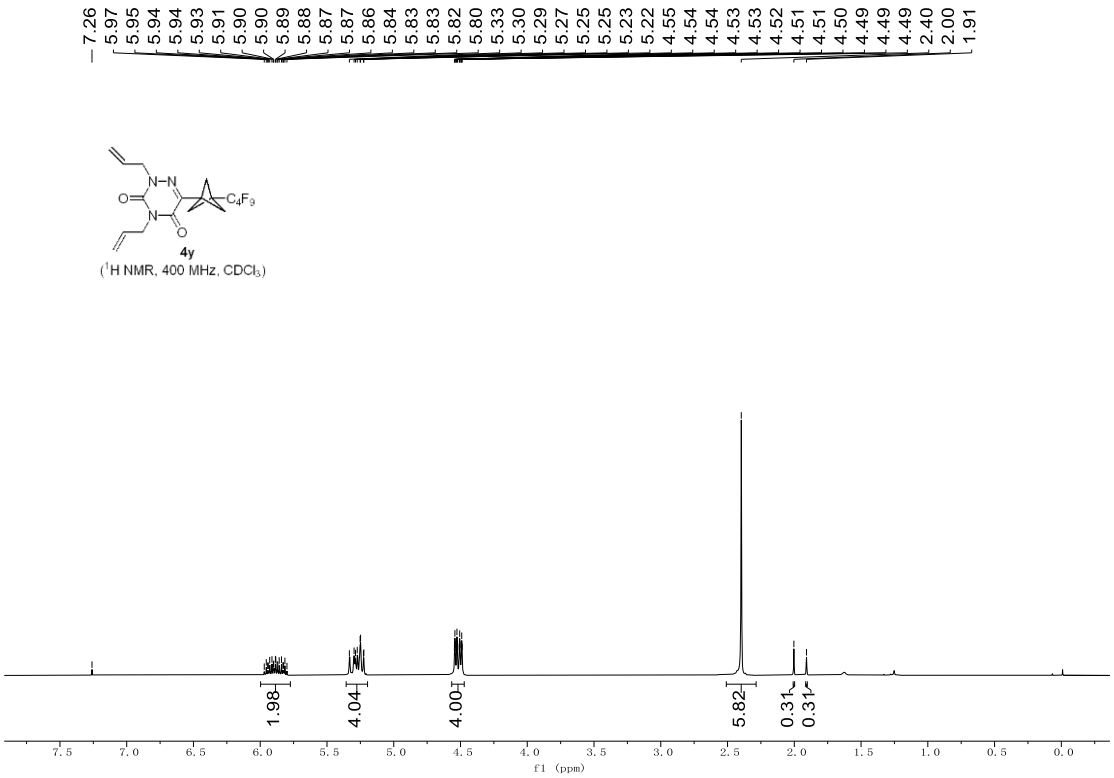

-81.05
-81.06
-81.08
-81.09
-81.10
-81.11
-81.12
-116.58
-116.59
-116.60
-116.61
-116.62
-116.63
-116.64
-116.65
-116.66
-116.67
-122.25
-122.27
-122.28
-122.29
-122.30
-122.32
-125.95
-125.97
-125.99
-126.02
-126.05
-126.06
-126.08
Cl
N–N
O
N
O
Cl
C_4F_9
4w
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.62
2.11
2.09
2.00
f1 (ppm)

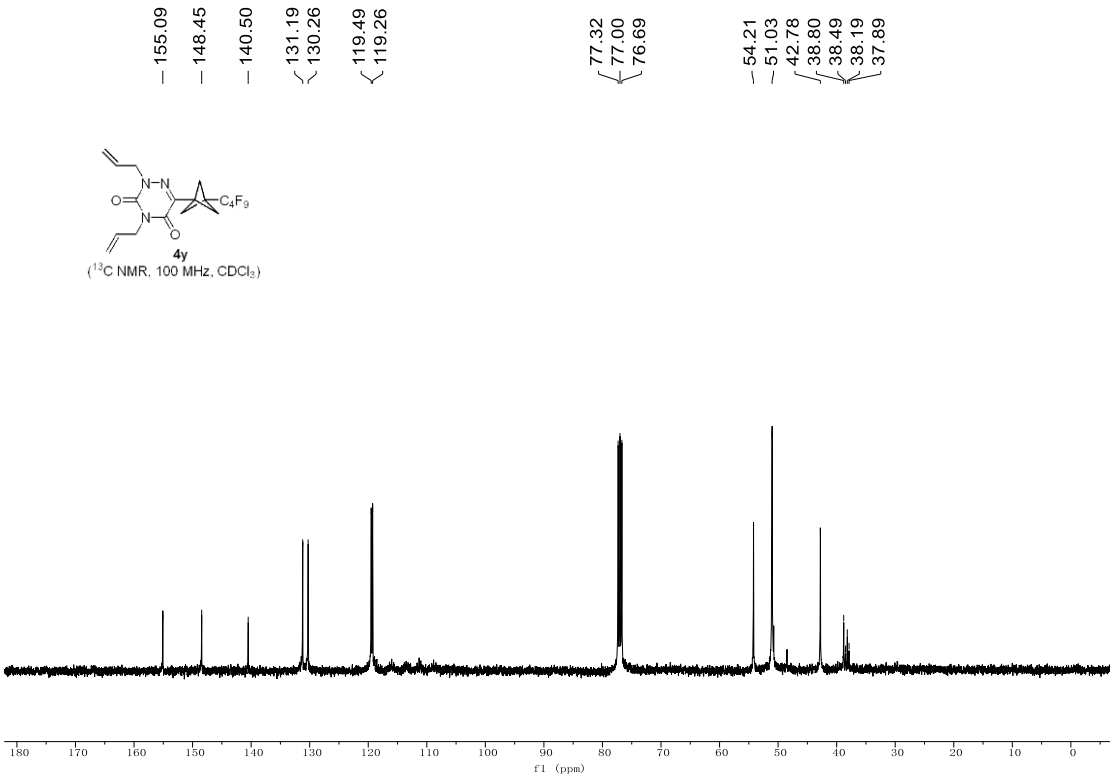
2,4-bis(naphthalen-2-ylmethyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo [1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4x)

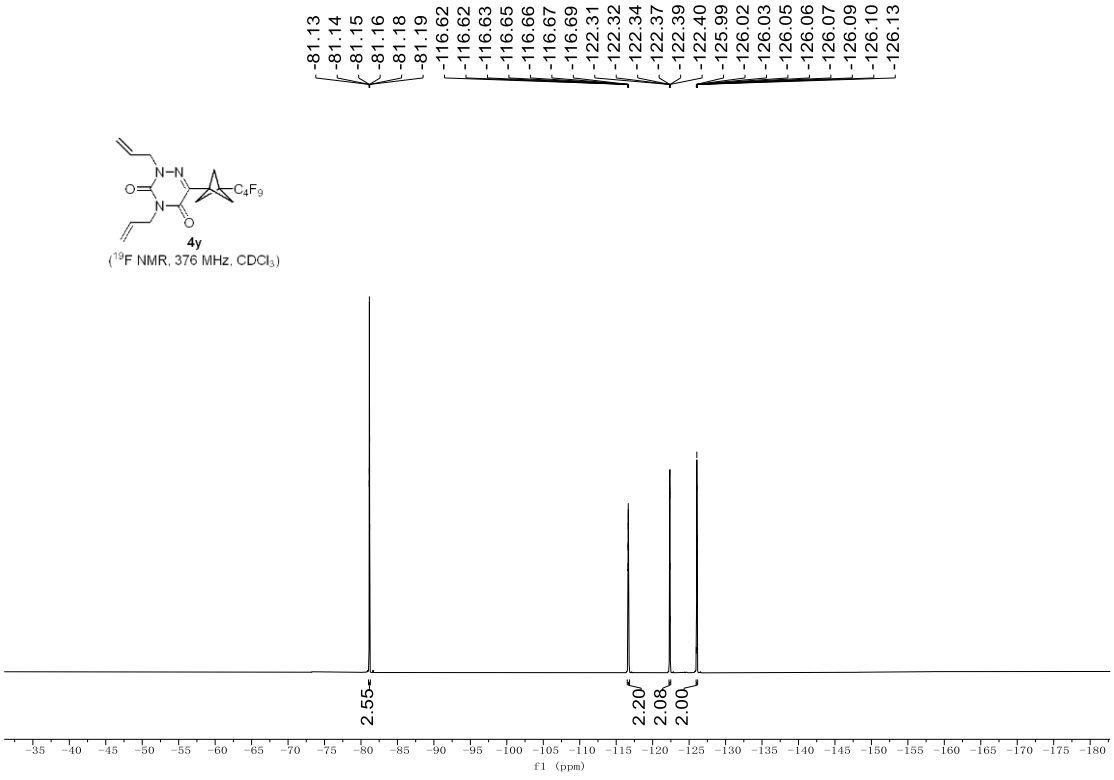




2,4-diallyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4l12-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4y)




155.09
148.45
140.50
131.19
130.26
119.49
119.26
77.32
77.00
76.69
54.21
51.03
42.78
38.80
38.49
38.19
37.89
C4F9
4y
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)


-81.13
-81.14
-81.15
-81.16
-81.18
-81.19
-116.62
-116.63
-116.65
-116.66
-116.67
-116.69
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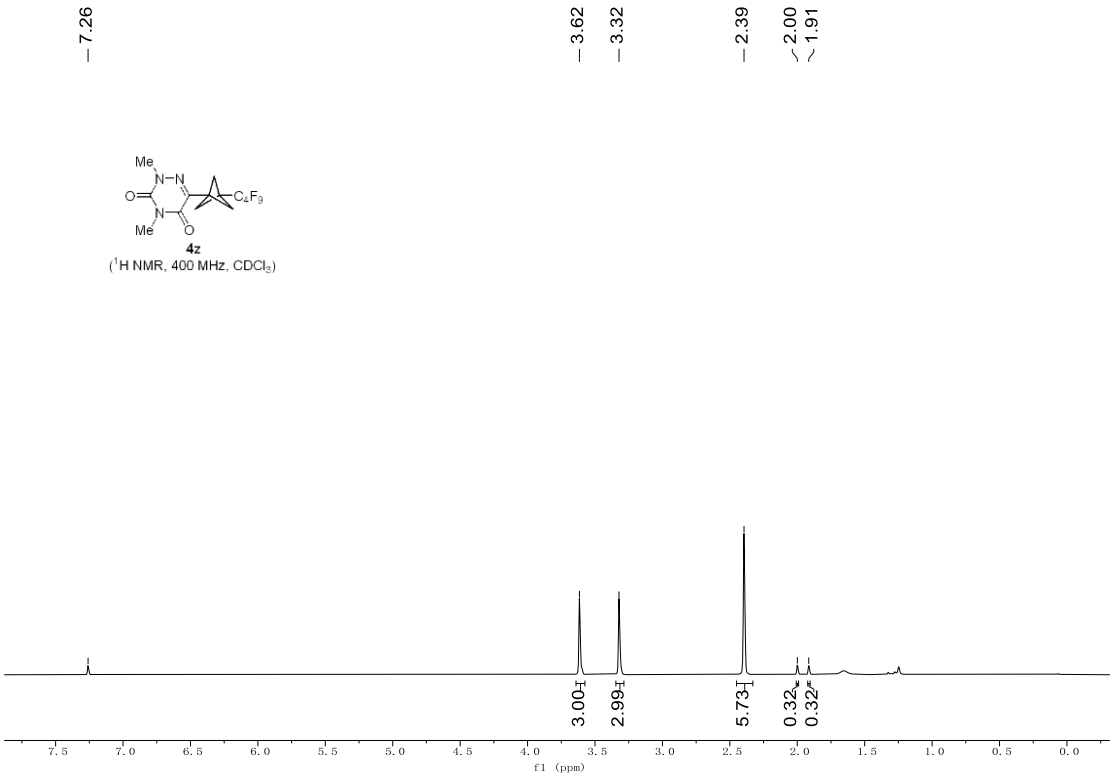
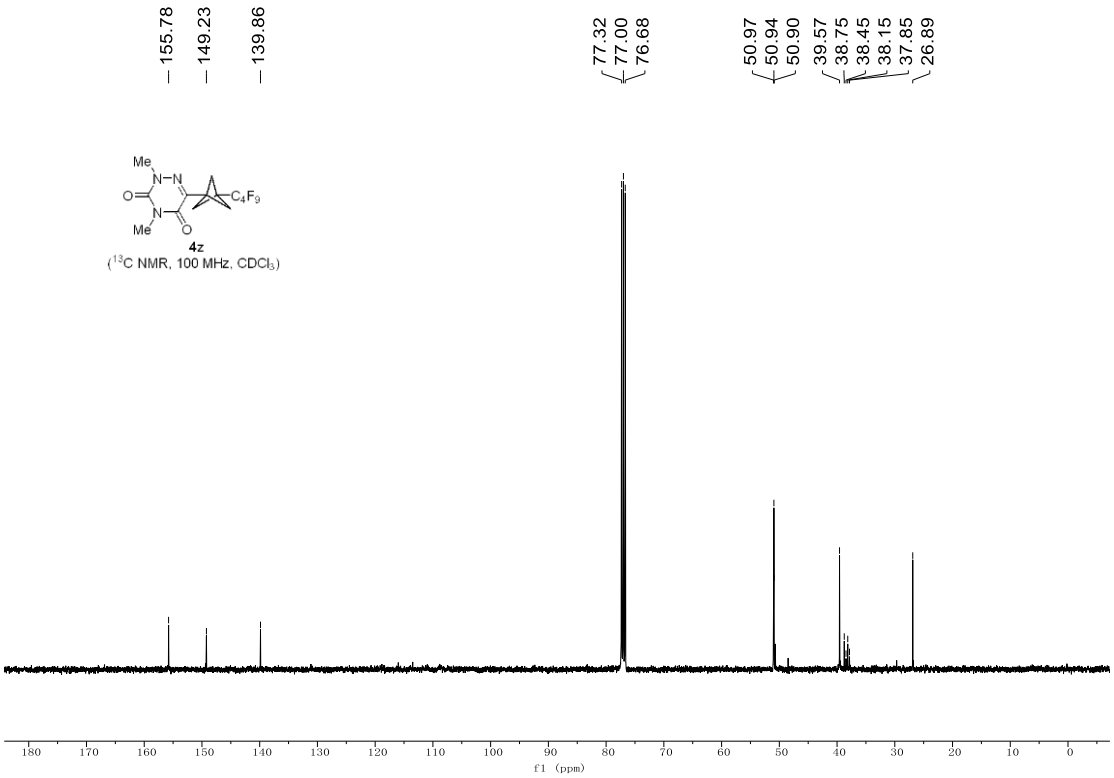
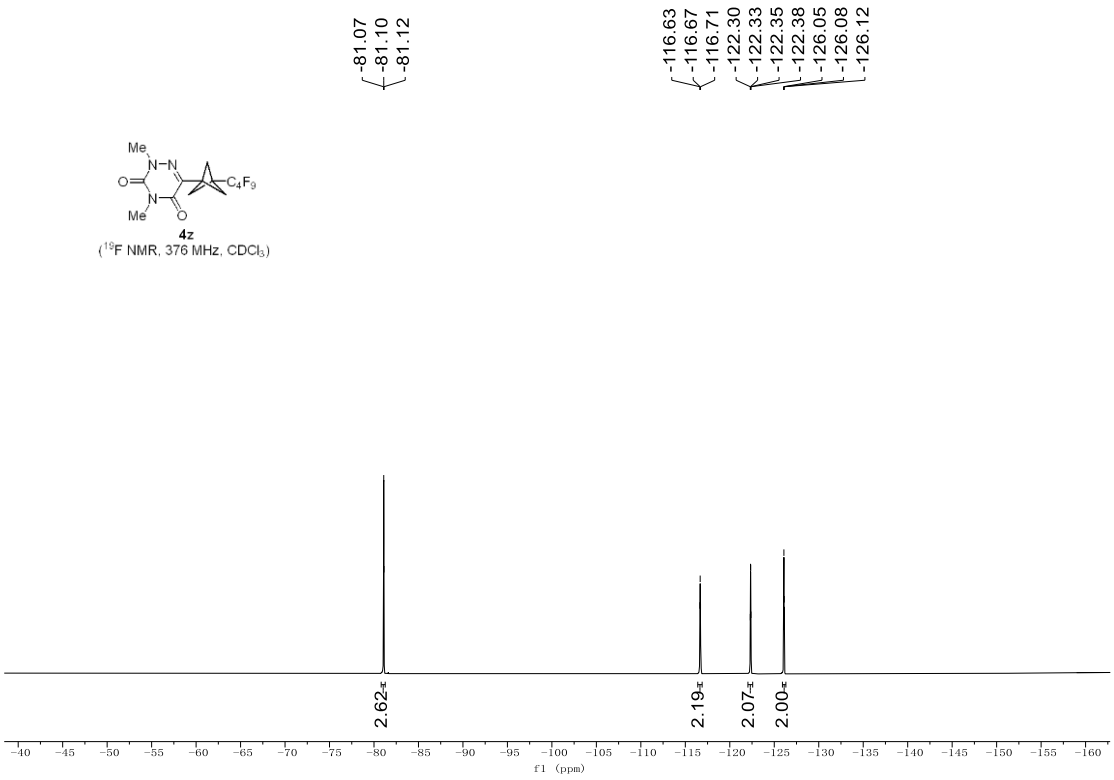
-122.32
-122.34
-122.37
-122.39
-122.40
-125.99
-126.02
-126.03
-126.05
-126.06
-126.07
-126.09
-126.10
-126.13
C4F9
4y
(19F NMR, 376 MHz, CDCl3)
2.55
2.20
2.08
2.00
f1 (ppm)

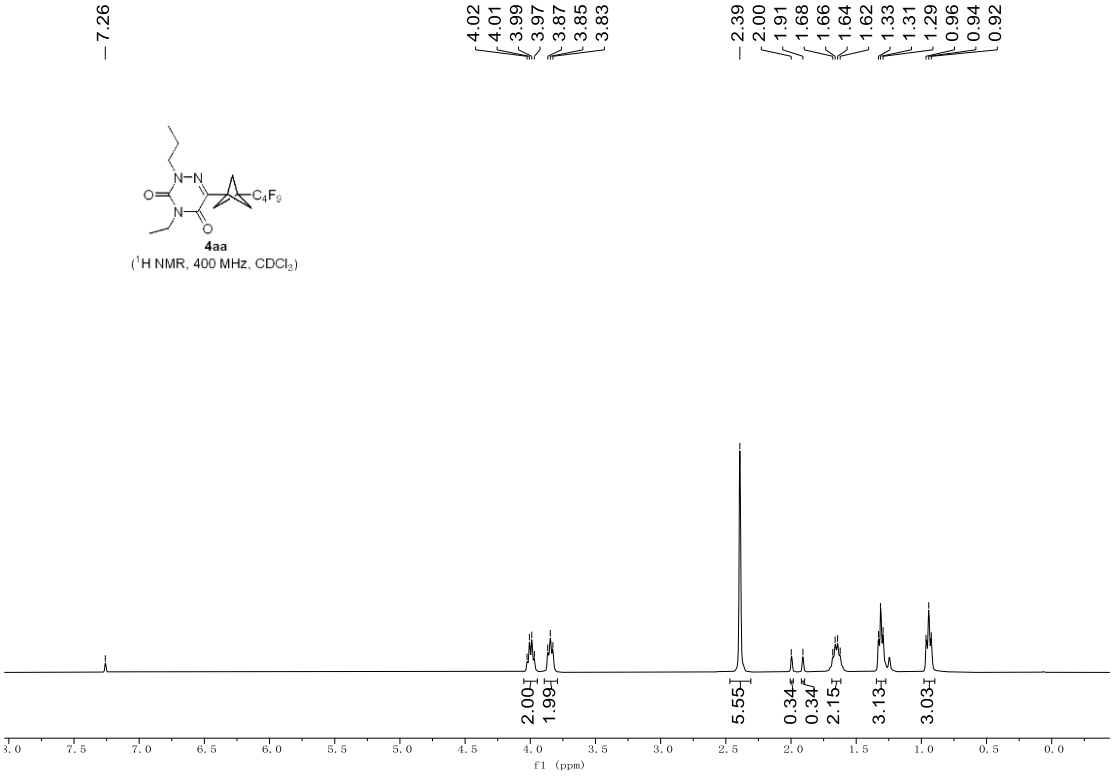
2,4-dimethyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4z)

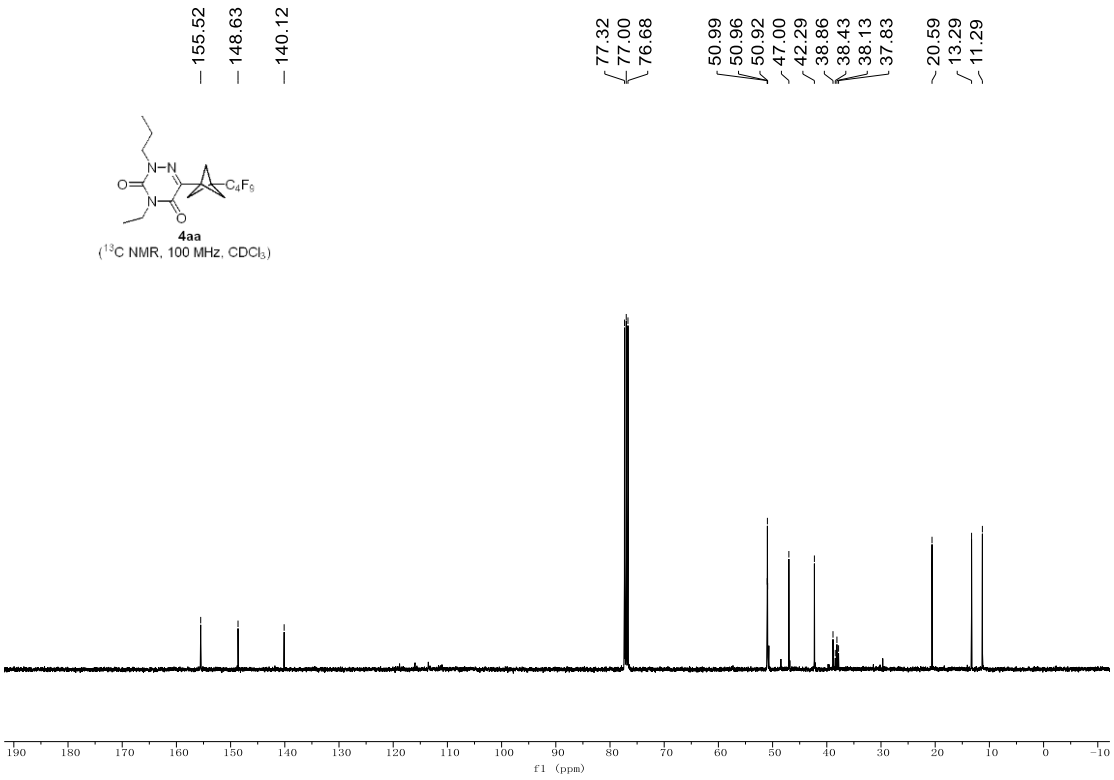


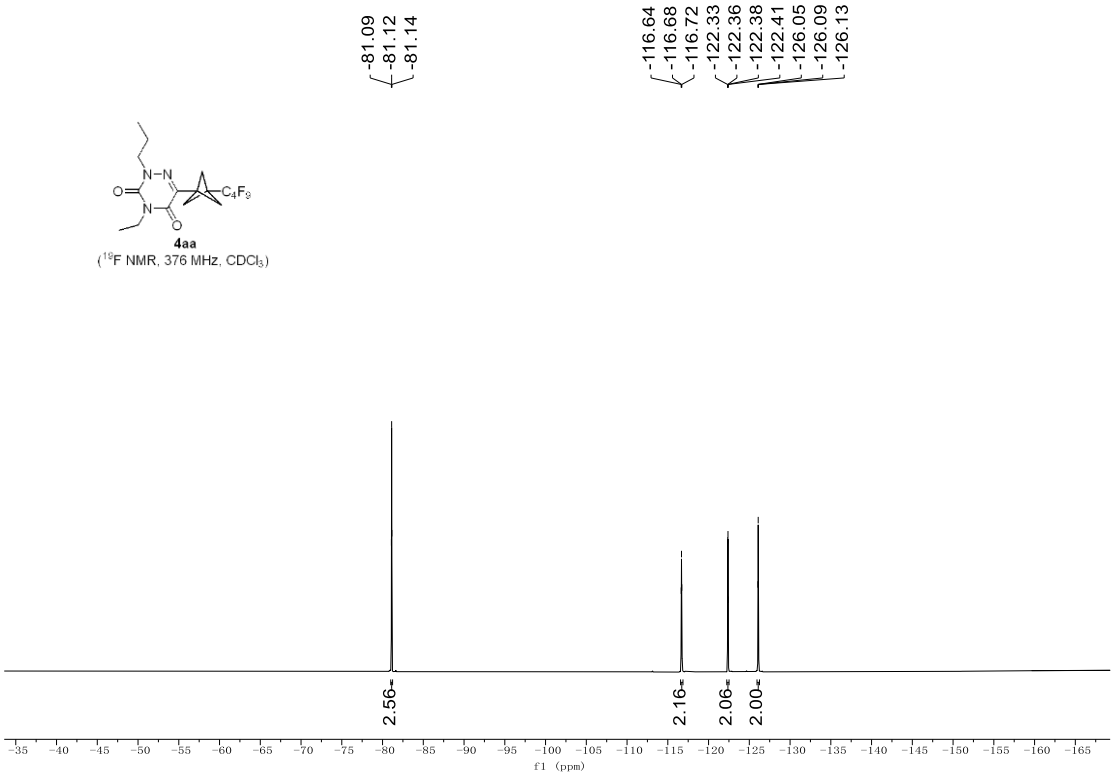




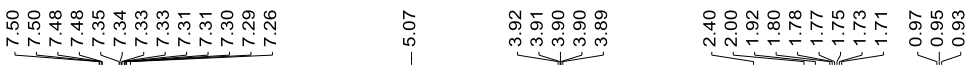
4-ethyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-propyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4aa)

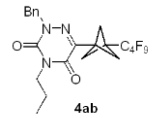



155.52
148.63
140.12
77.32
77.00
76.68
50.99
50.96
50.92
47.00
42.29
38.86
38.43
38.13
37.83
20.59
13.29
11.29
C4F9
4aa
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)

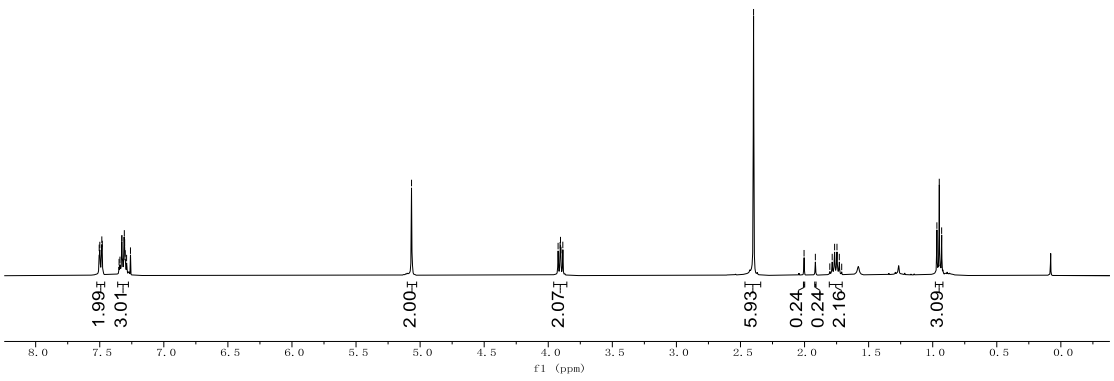

-81.09
-81.12
-81.14
-116.64
-116.68
-116.72
-122.33
-122.36
-122.38
-122.41
-126.05
-126.09
-126.13
C4F9
4aa
(19F NMR, 376 MHz, CDCl3)
2.56
2.16
2.06
2.00
f1 (ppm)

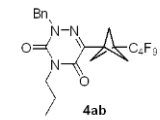
2-benzyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-4-propyl-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4ab)



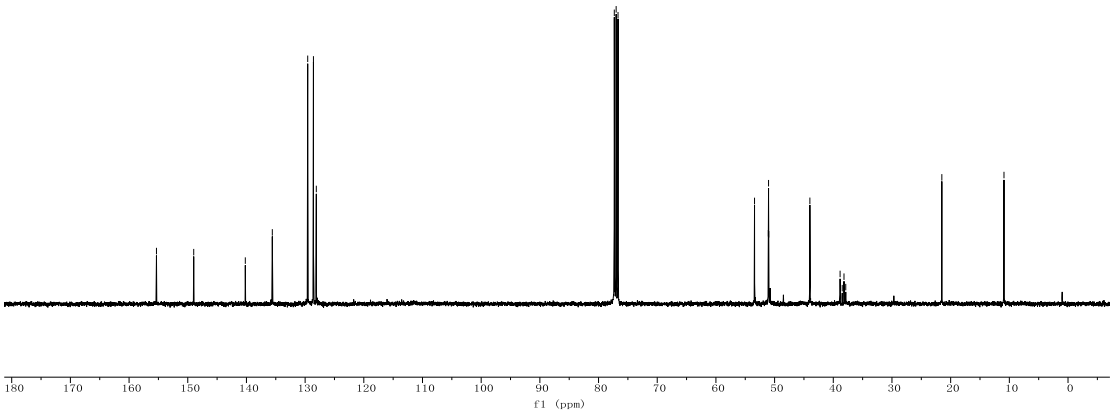


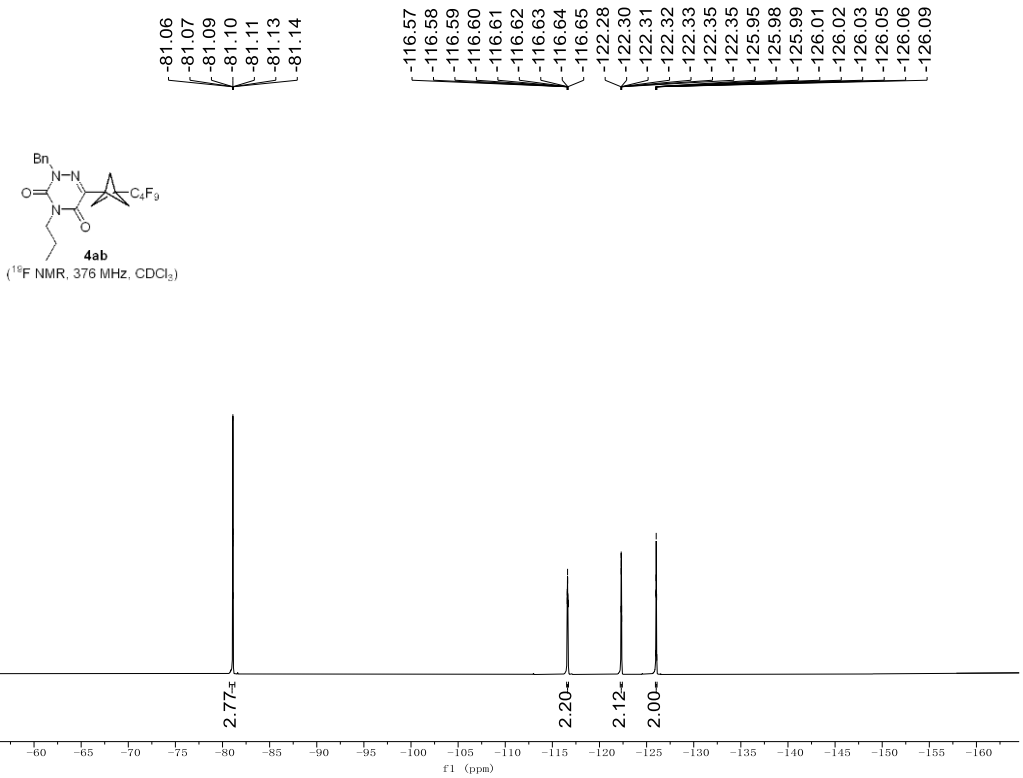
4ab
(^{1}H NMR, 400 MHz, $CDCl_3$)



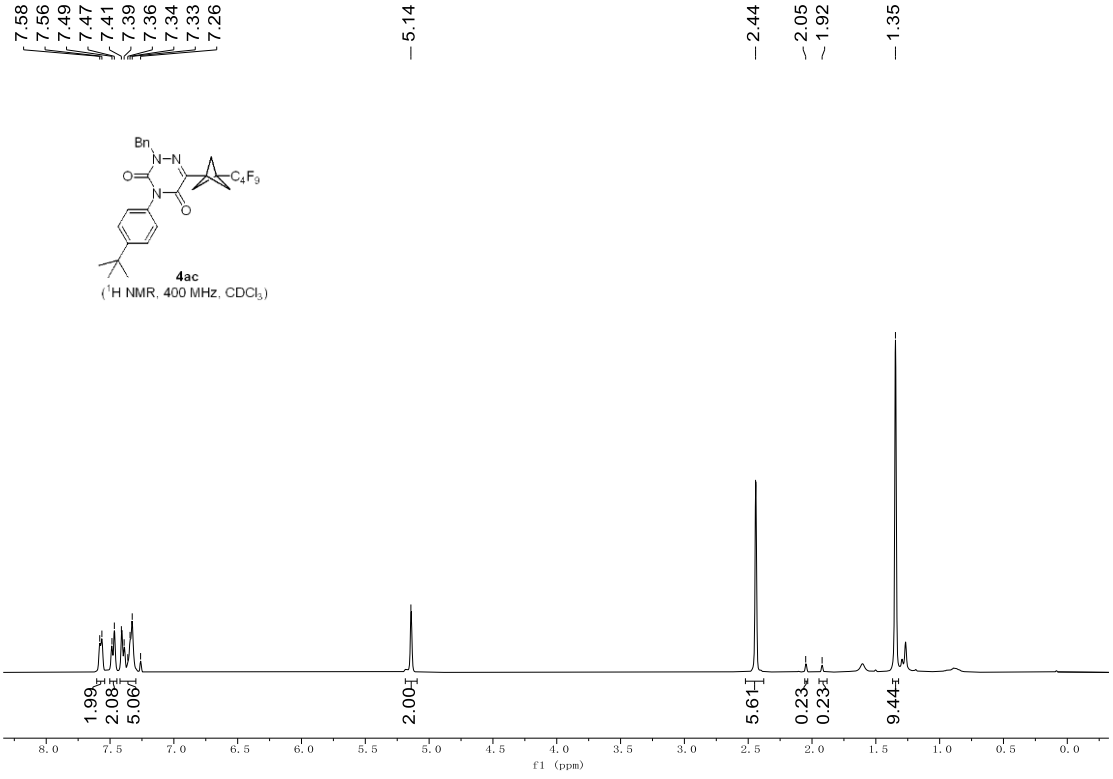


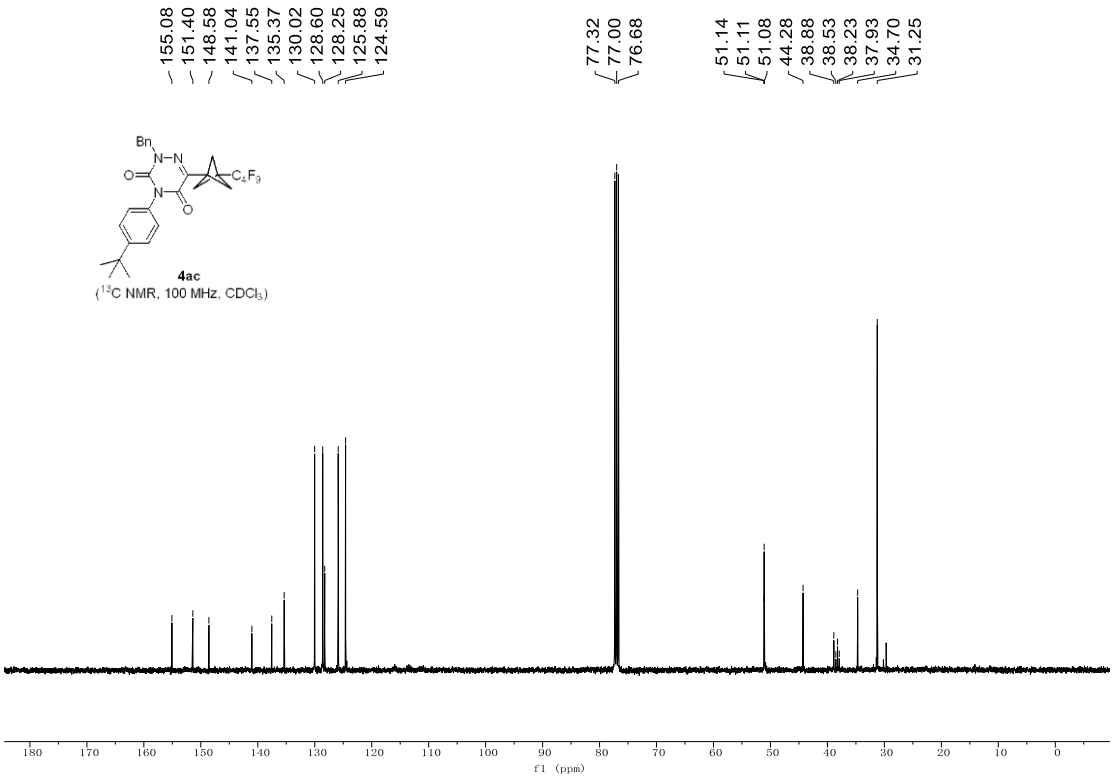
4ab
(^{13}C NMR, 100 MHz, $CDCl_3$)

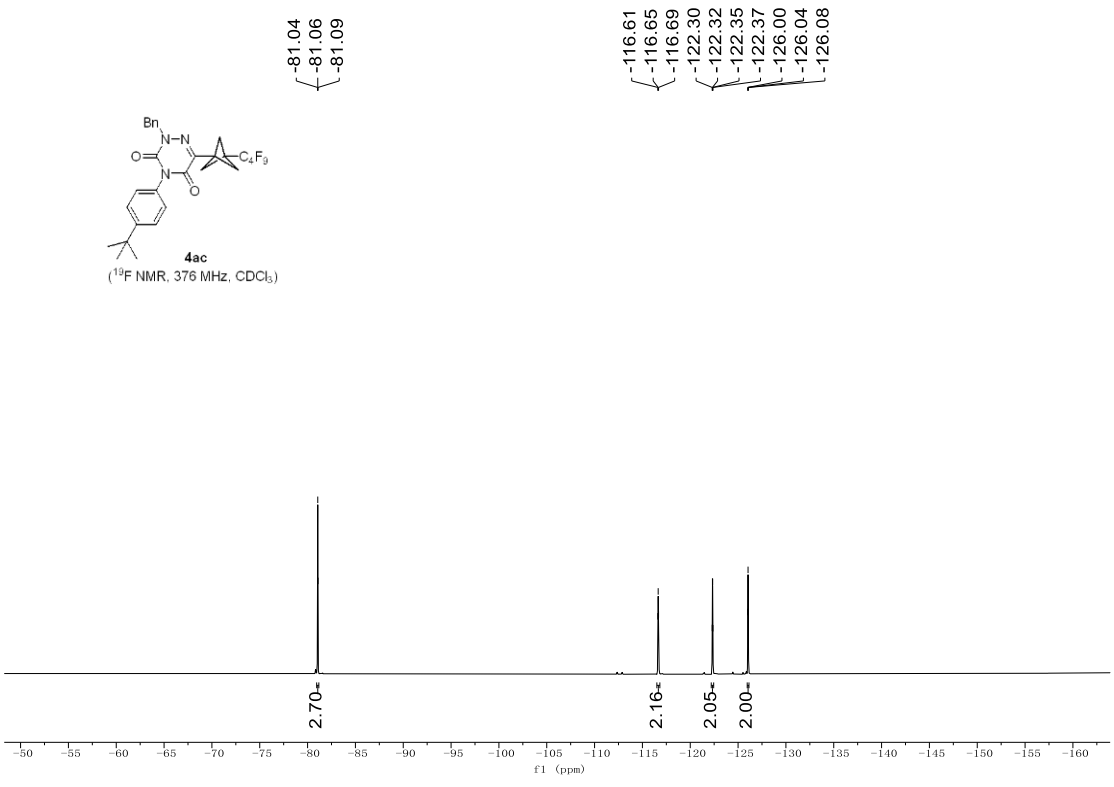




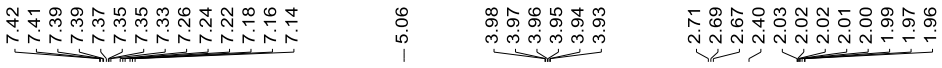
2-benzyl-4-(4-(tert-butyl)phenyl)-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4ac)

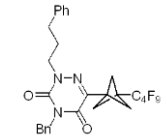



155.08
151.40
148.58
141.04
137.55
135.37
130.02
128.60
128.25
125.88
124.59
77.32
77.00
76.68
51.14
51.11
51.08
44.28
38.88
38.53
38.23
37.93
34.70
31.25
Bn
N–N
O
N
O
C_4F_9
4ac
(^{13}C NMR, 100 MHz, $CDCl_3$)
f1 (ppm)

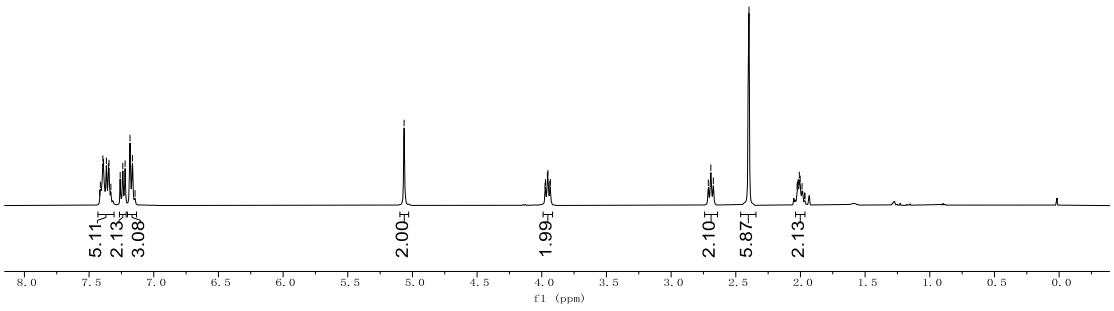

-81.04
-81.06
-81.09
-116.61
-116.65
-116.69
-122.30
-122.32
-122.35
-122.37
-126.00
-126.04
-126.08
Bn
N–N
O
N
O
C_4F_9
4ac
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.70
2.16
2.05
2.00
f1 (ppm)

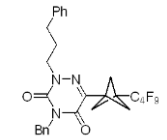
4-benzyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-2-(3-phenylpropyl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (4ad)



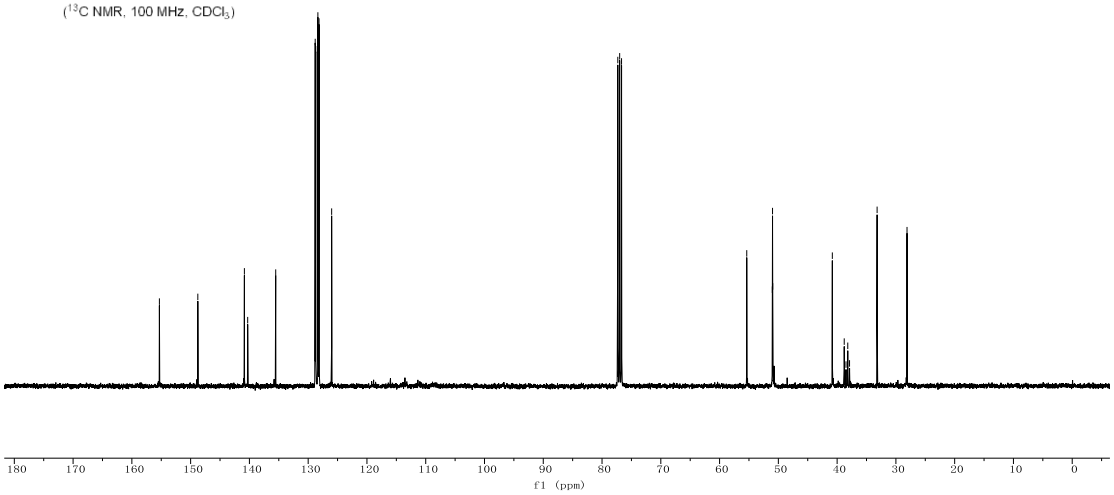


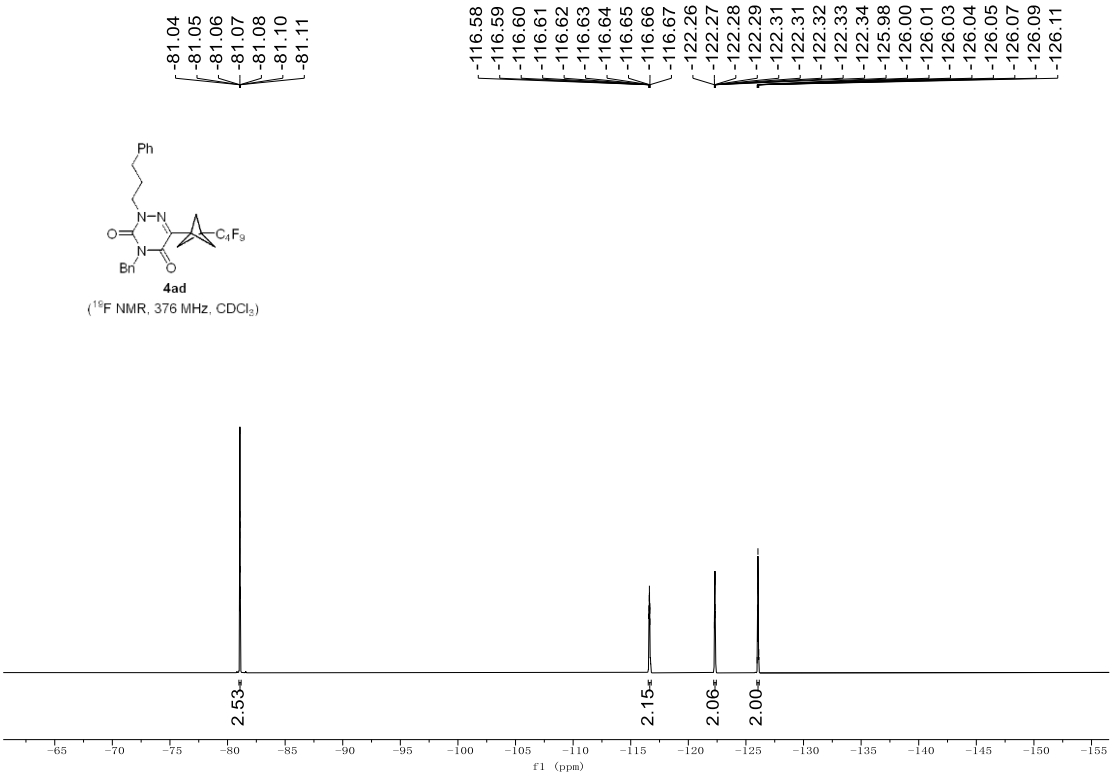
4ad
(^{1}H NMR, 400 MHz, $CDCl_3$)



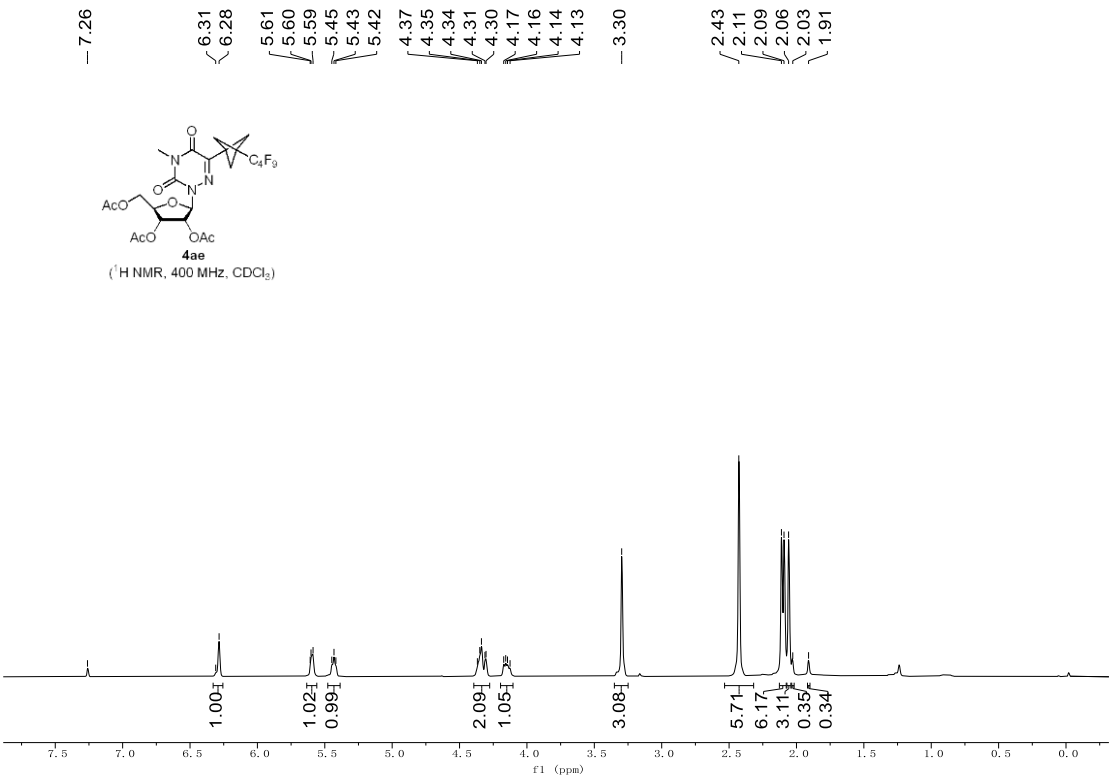


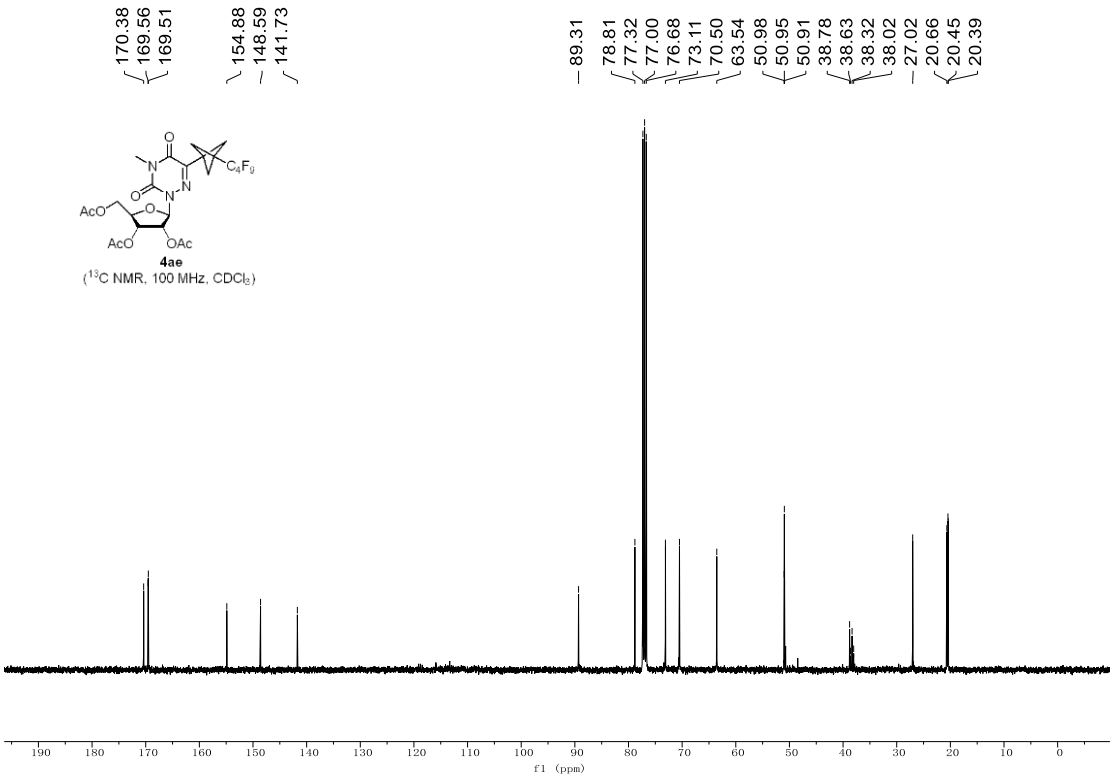
4ad
(^{13}C NMR, 100 MHz, $CDCl_3$)

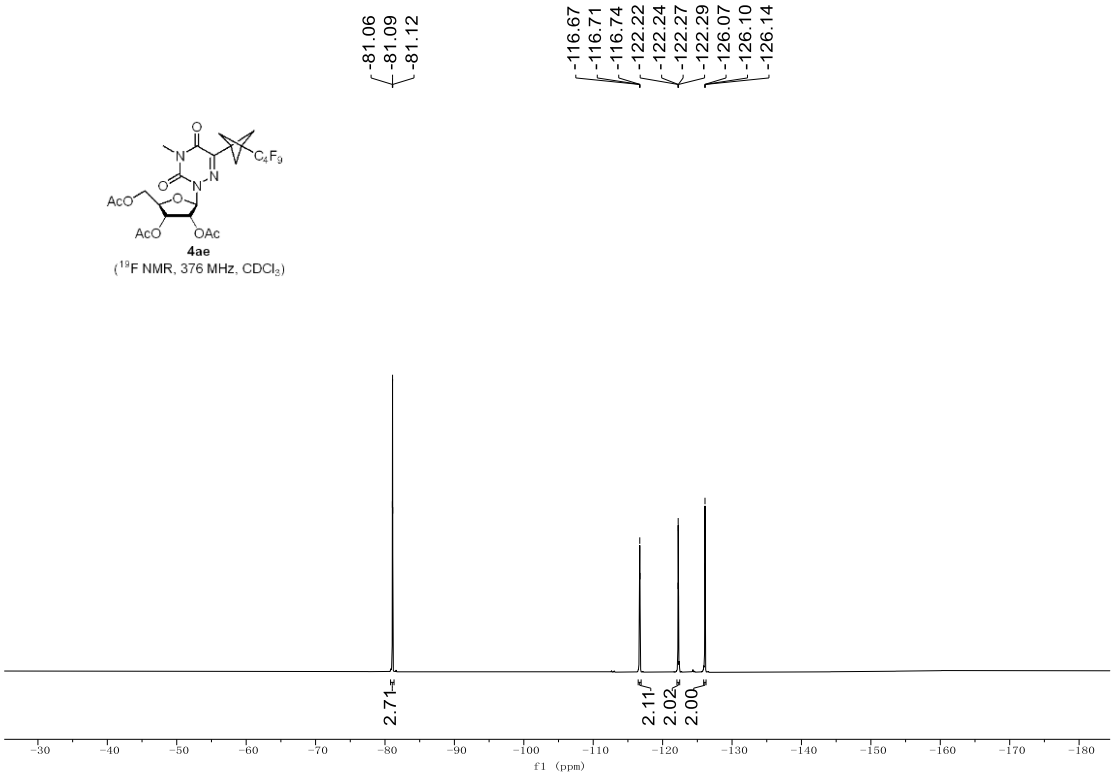




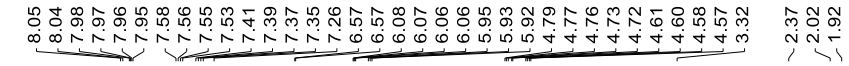
(2*R*,3*R*,4*R*,5*R*)-2-(acetoxymethyl)-5-(4-methyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)tetrahydrofuran-3,4-diyl diacetate (4ae)

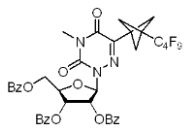



170.38
169.56
169.51
154.88
148.59
141.73
89.31
78.81
77.32
77.00
76.68
73.11
70.50
63.54
50.98
50.95
50.91
38.78
38.63
38.32
38.02
27.02
20.66
20.45
20.39
AcO
AcO
OAc
C_4F_9
4ae
(^{13}C NMR, 100 MHz, $CDCl_3$)
f1 (ppm)


-81.06
-81.09
-81.12
-116.67
-116.71
-116.74
-122.22
-122.24
-122.27
-122.29
-126.07
-126.10
-126.14
AcO
AcO
OAc
C_4F_9
4ae
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.71
2.11
2.02
2.00
f1 (ppm)

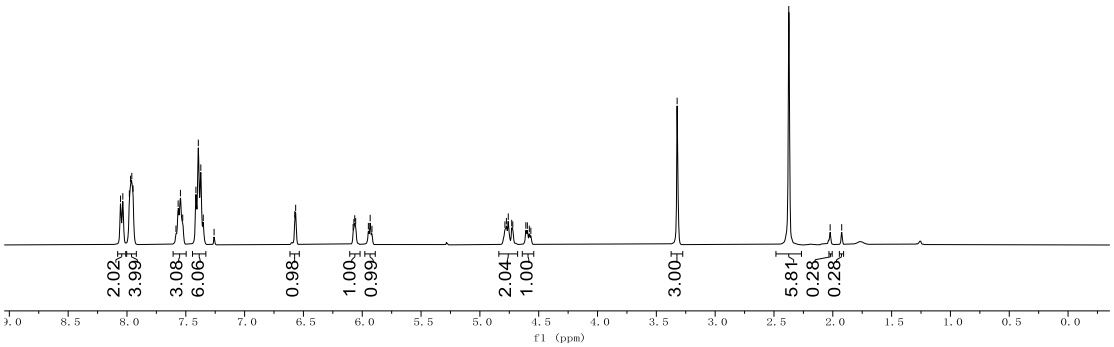
(2*R*,3*R*,4*R*,5*R*)-2-((benzoyloxy)methyl)-5-(4-methyl-6-(3-(4,4,4,4,4,4,4,4,4-nonafluoro-4λ^{12}-buta-1,3-diyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-3,5-dioxo-4,5-dihydro-1,2,4-triazin-2(3*H*)-yl)tetrahydrofuran-3,4-diyl dibenzoate (4af)

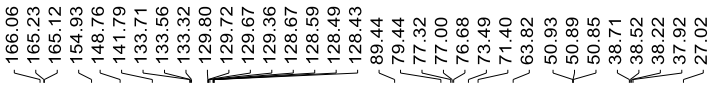


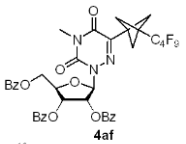


4af

(^{1}H NMR, 400 MHz, $CDCl_3$)

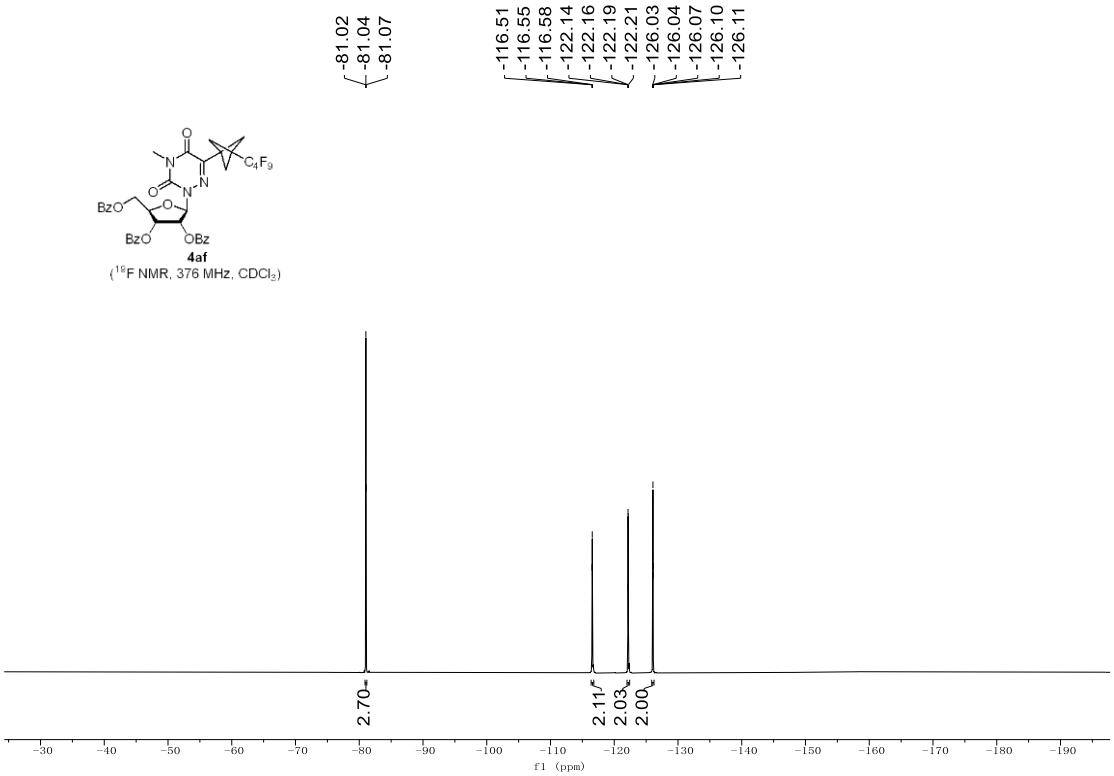




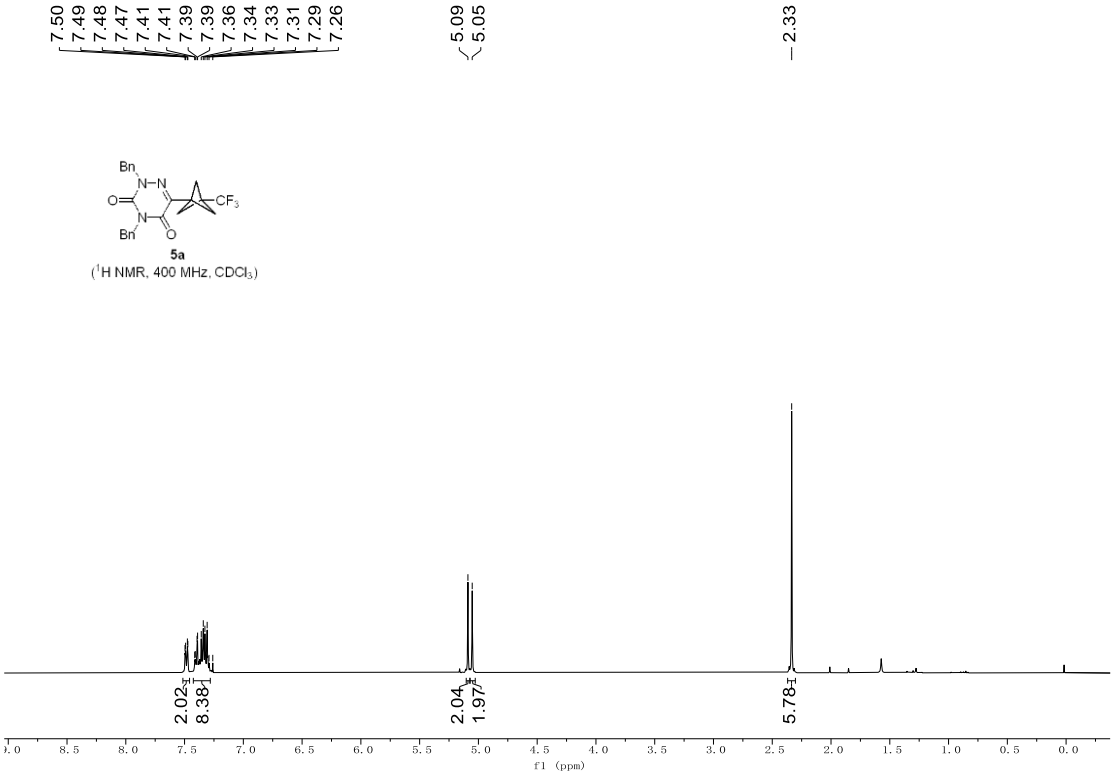


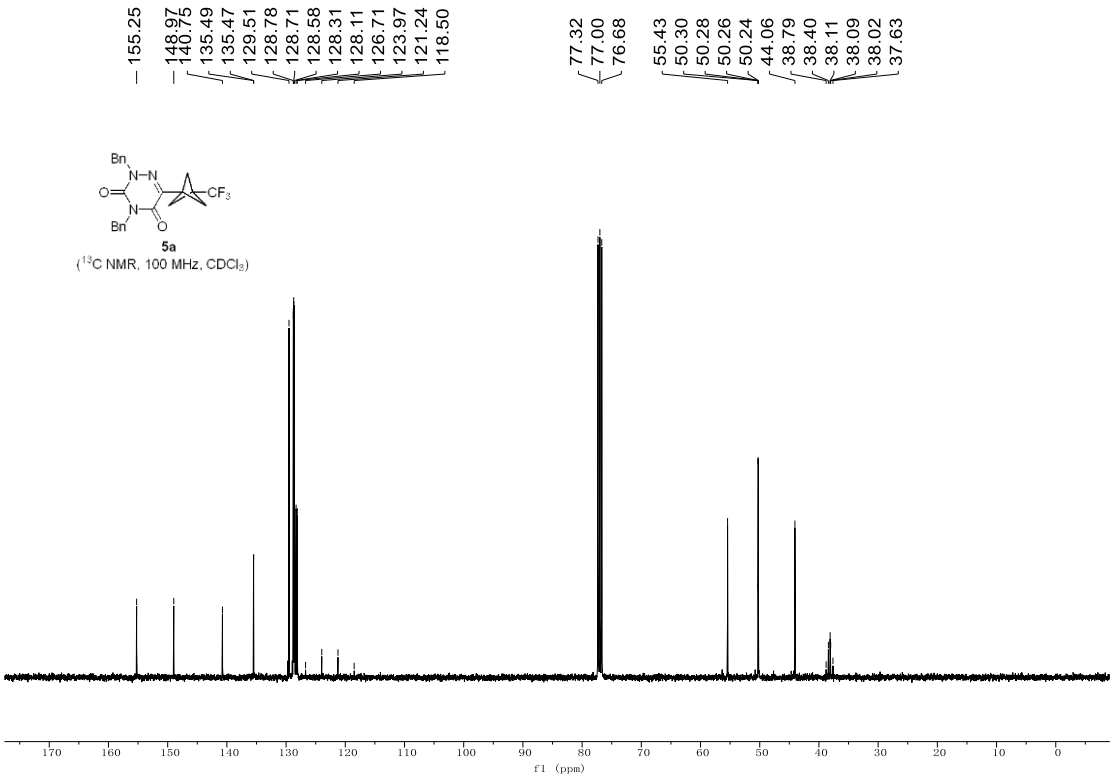
4af

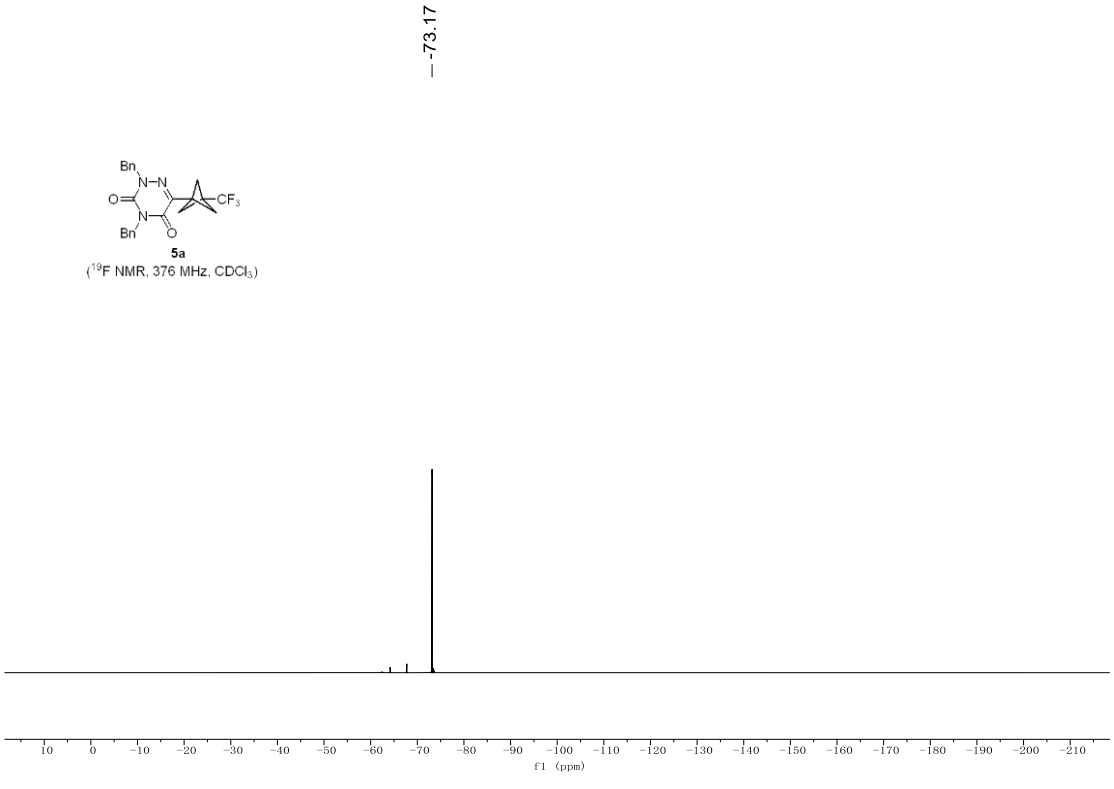
(^{13}C NMR, 100 MHz, $CDCl_3$)



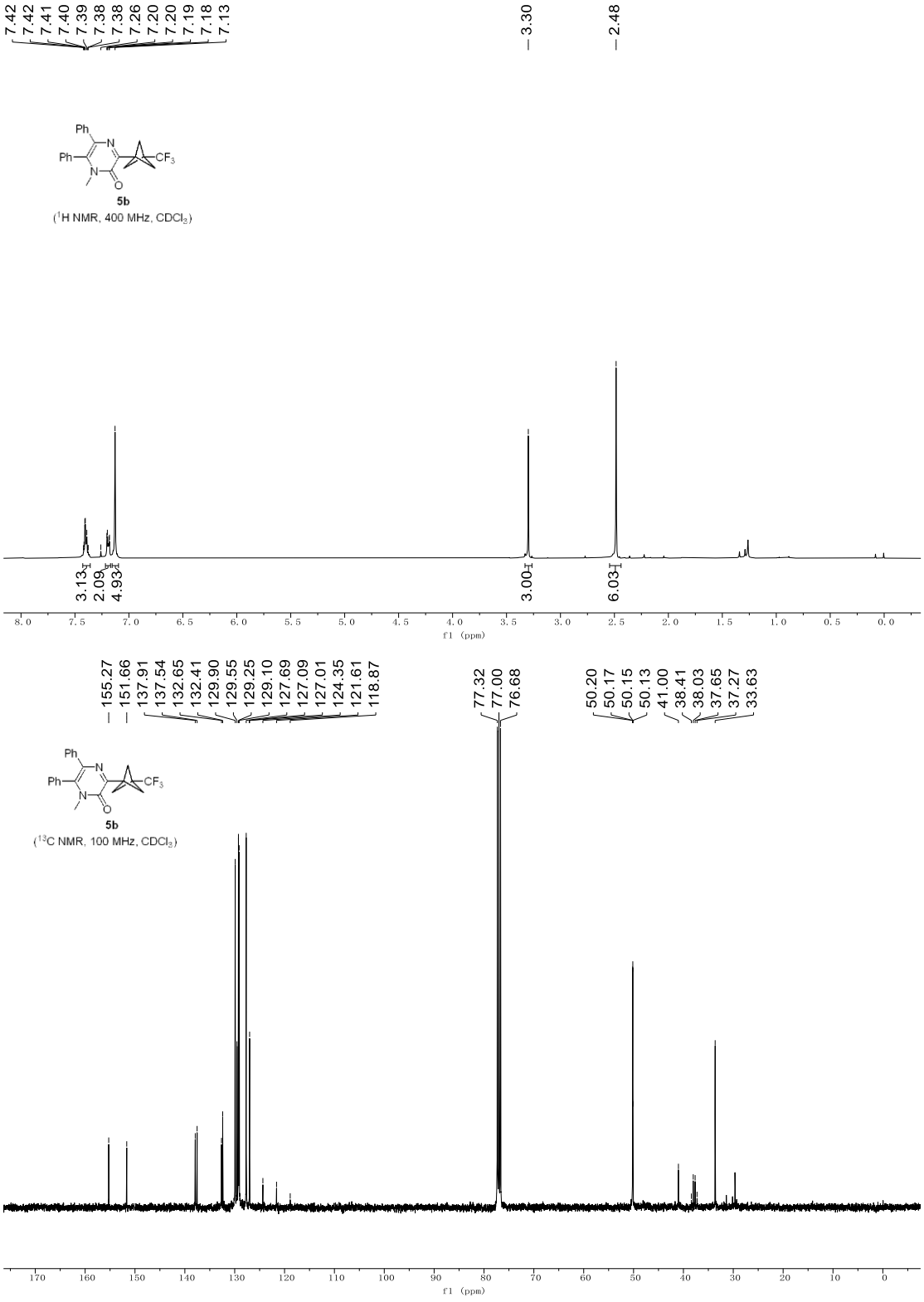
2,4-dibenzyl-6-(3-(trifluoromethyl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5a)

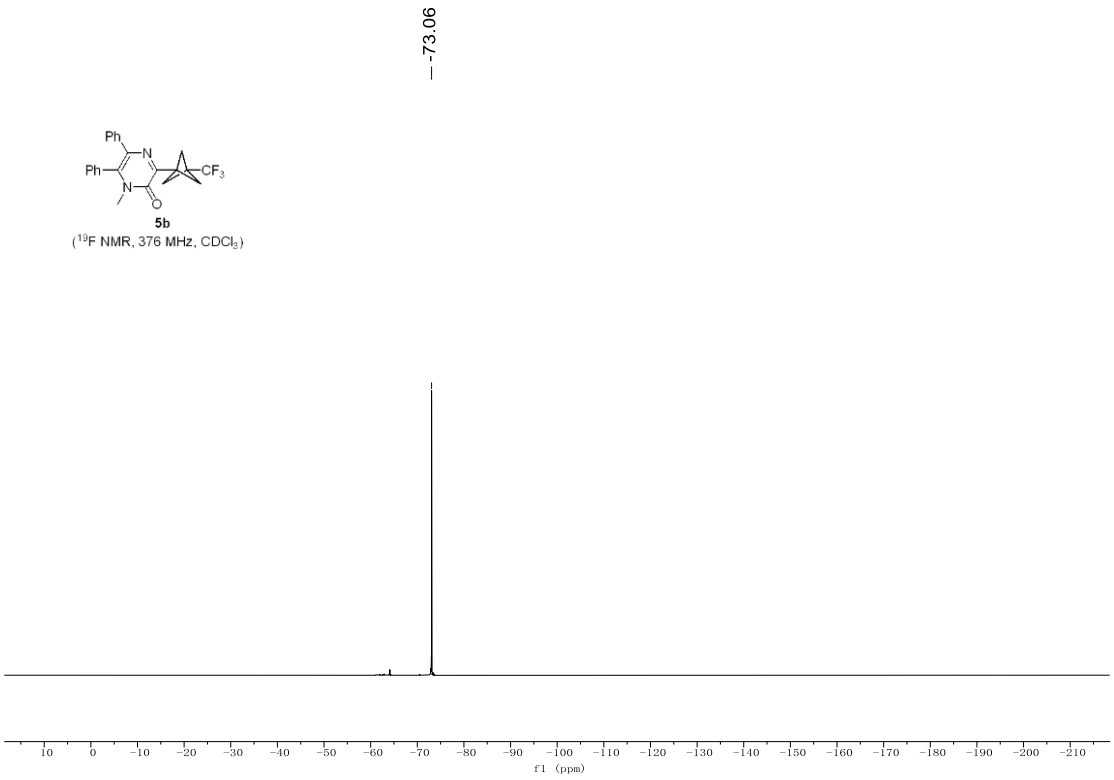



155.25
148.97
140.75
135.49
135.47
129.51
128.78
128.71
128.58
128.31
128.11
126.71
123.97
121.24
118.50
77.32
77.00
76.68
55.43
50.30
50.28
50.26
50.24
44.06
38.79
38.40
38.11
38.09
38.02
37.63
Bn
N–N
O
N
CF3
Bn
O
5a
(13C NMR, 100 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

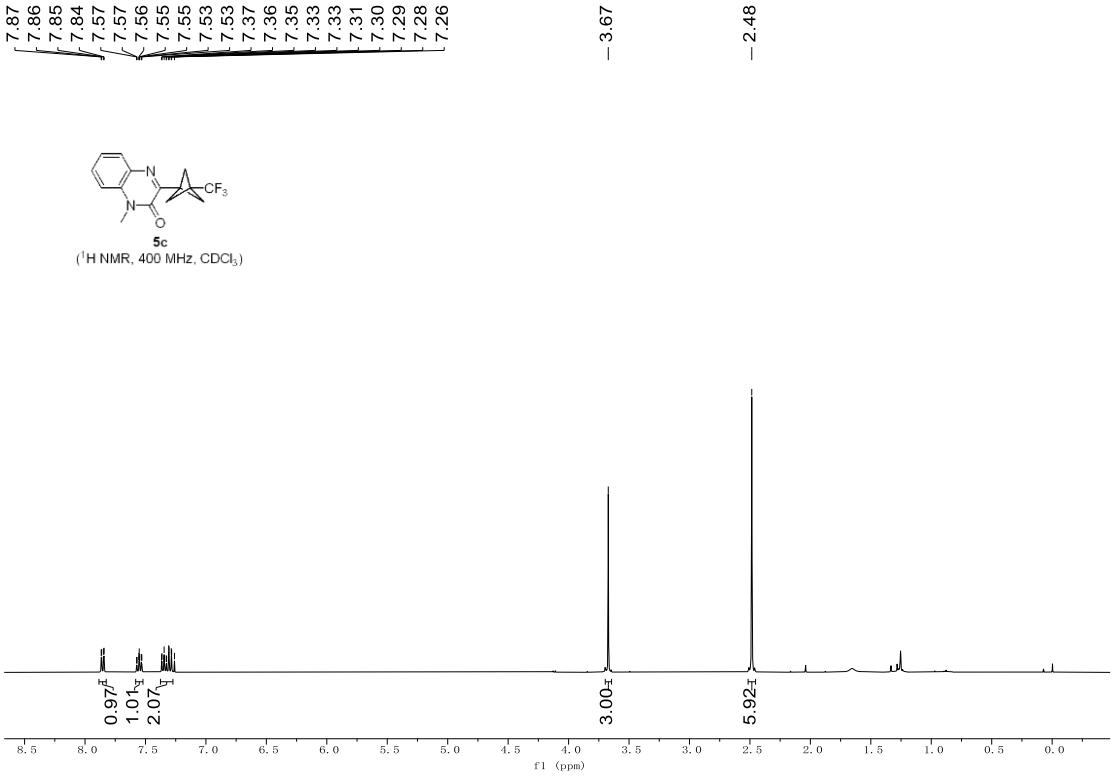

-73.17
Bn
N–N
O
N
CF3
Bn
O
5a
(19F NMR, 376 MHz, CDCl3)
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)

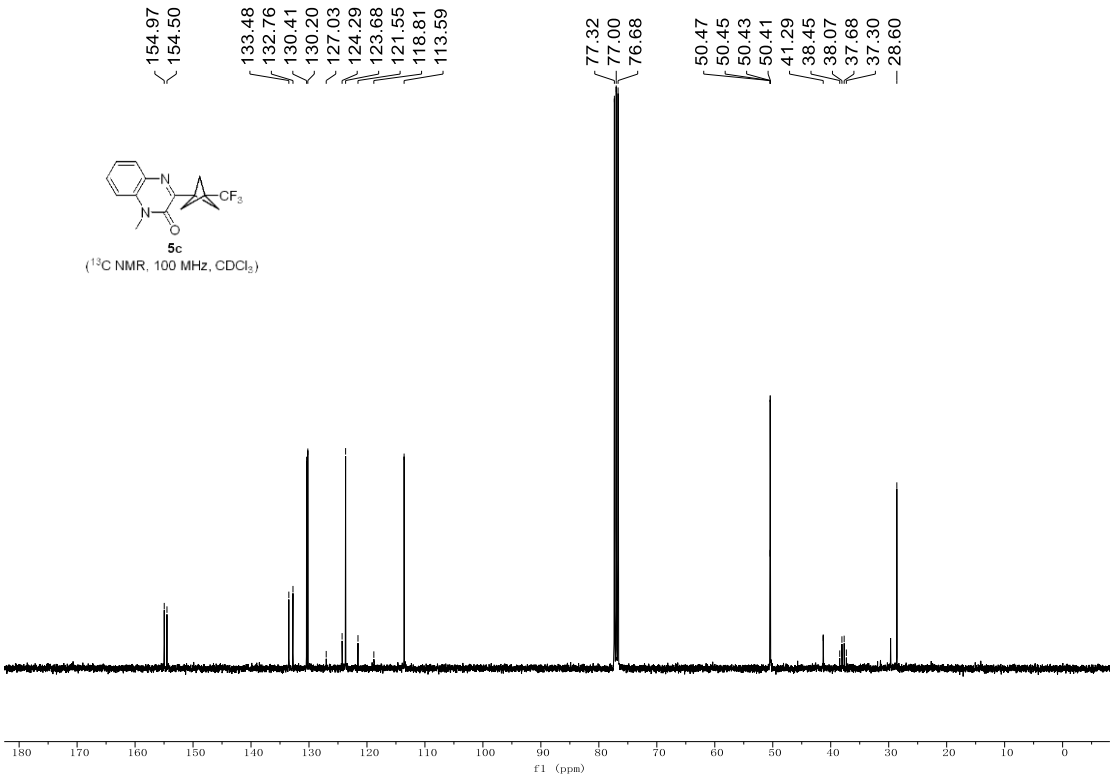
1-methyl-5,6-diphenyl-3-(3-(trifluoromethyl)bicyclo[1.1.1]pentan-1-yl)pyrazin-2(1*H*)-one (5b)





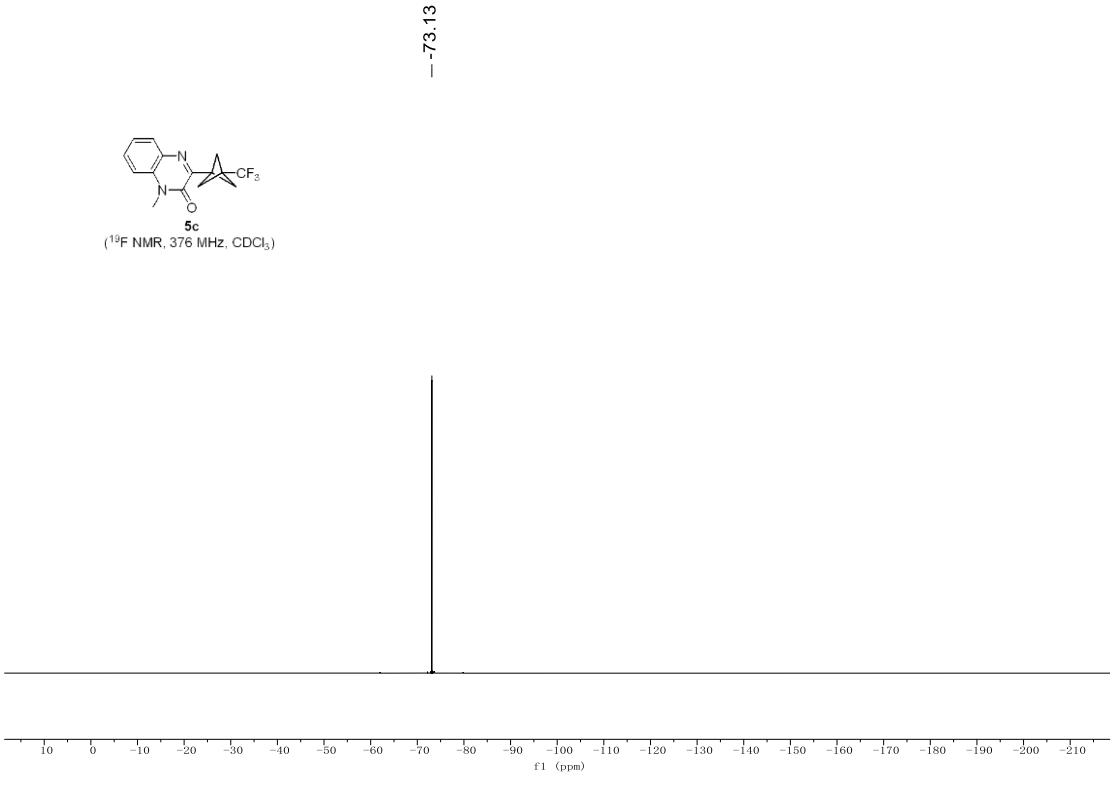
1-methyl-3-(3-(trifluoromethyl)bicyclo[1.1.1]pentan-1-yl)quinoxalin-2(1*H*)-one (5c)





5c

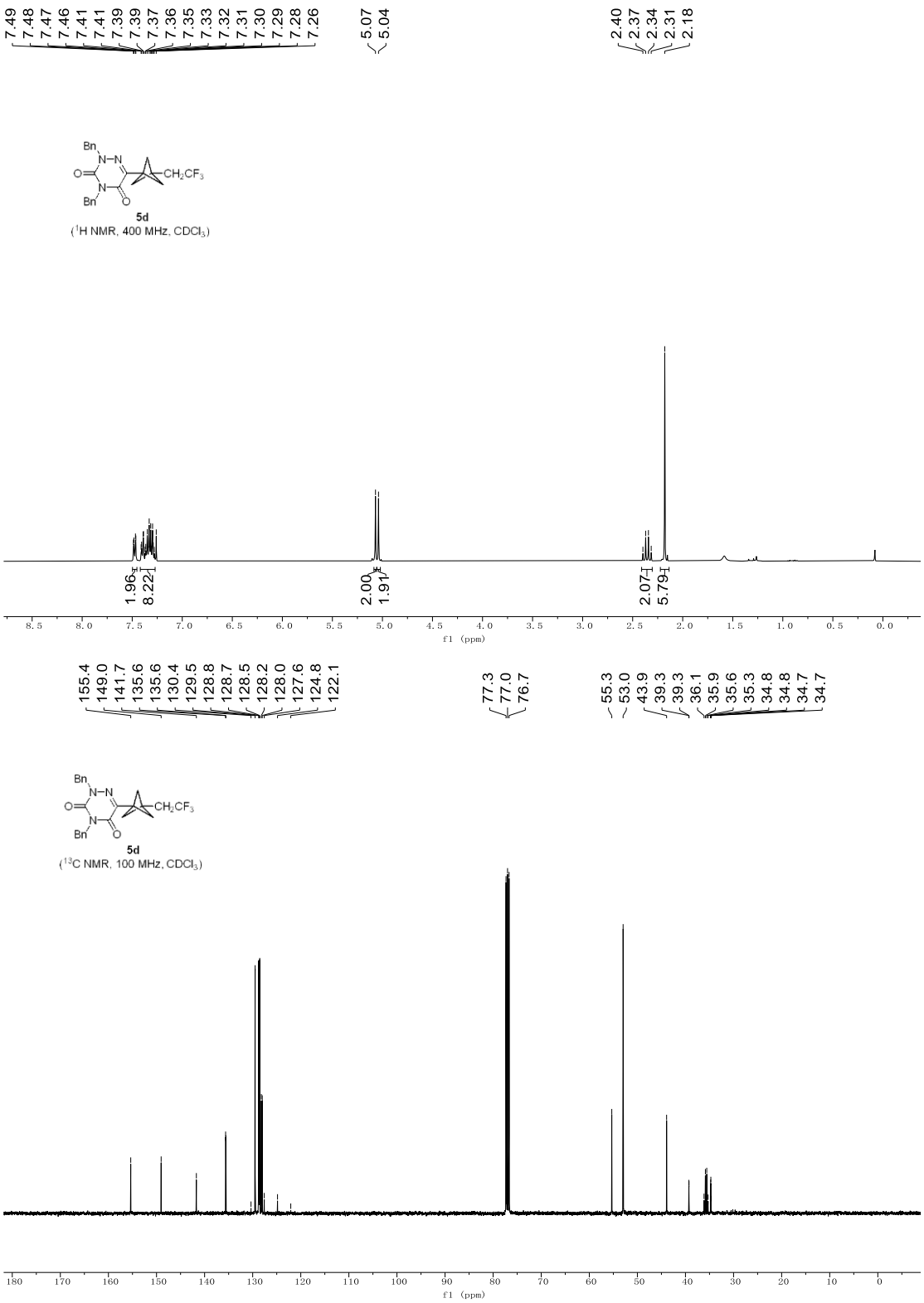
(^{13}C NMR, 100 MHz, $CDCl_3$)

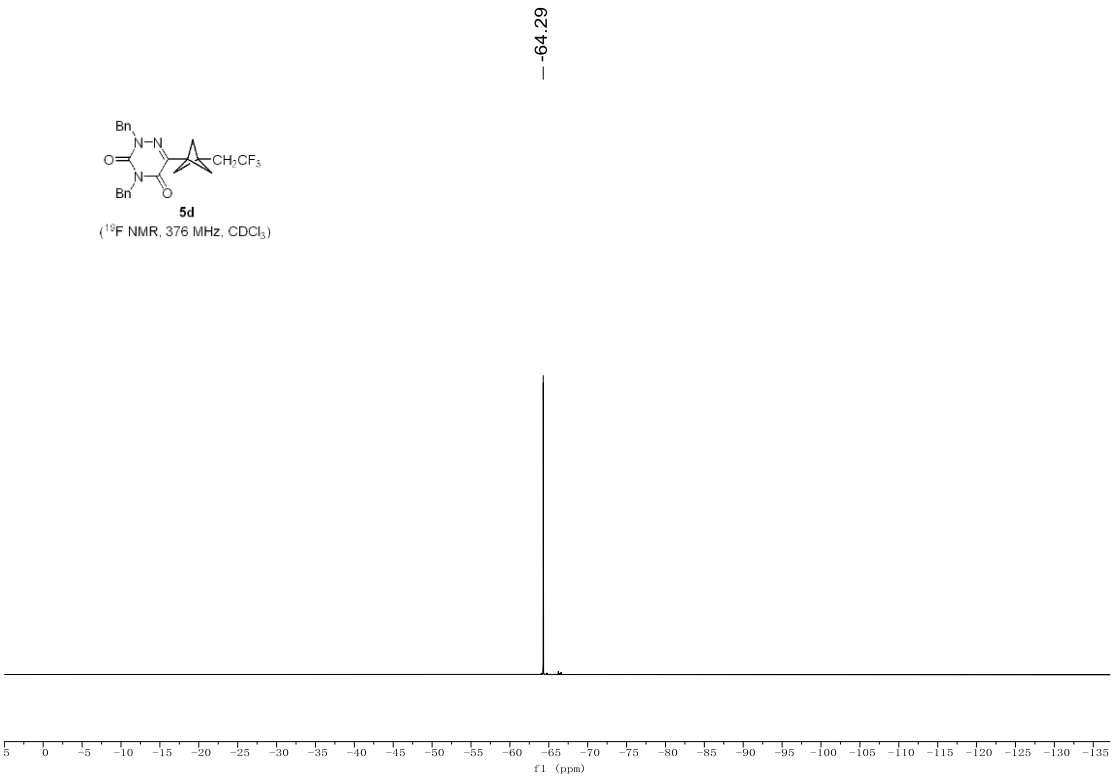


5c

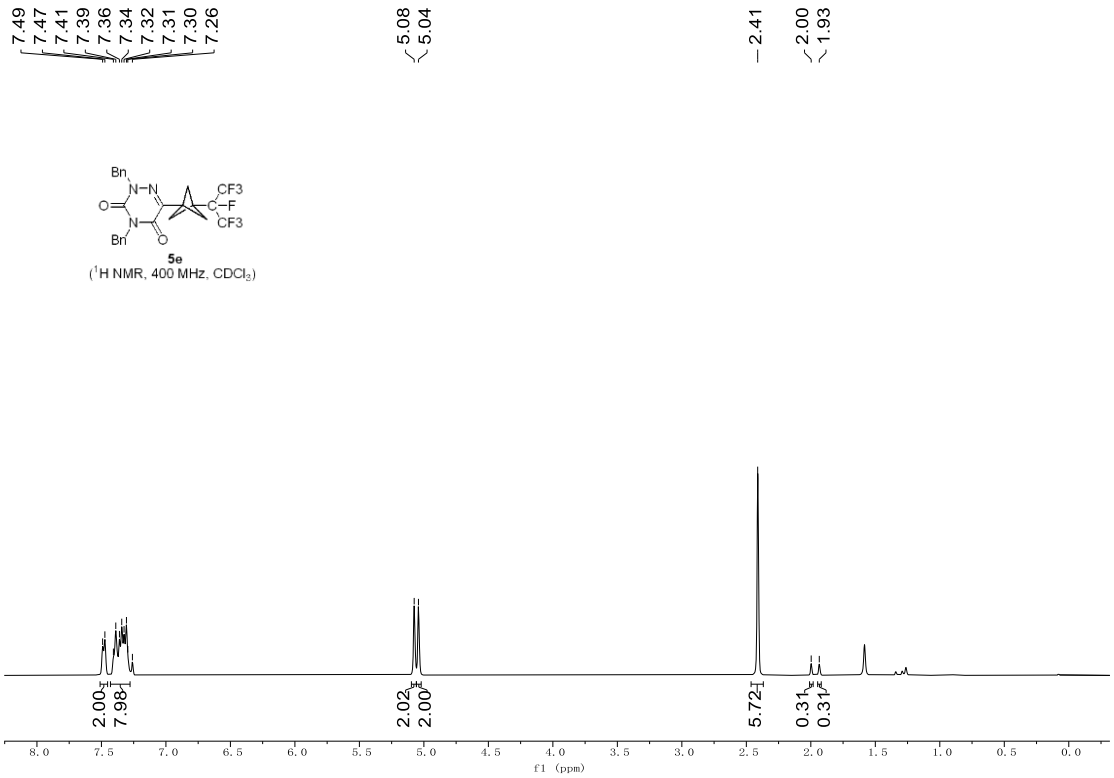
(^{19}F NMR, 376 MHz, $CDCl_3$)

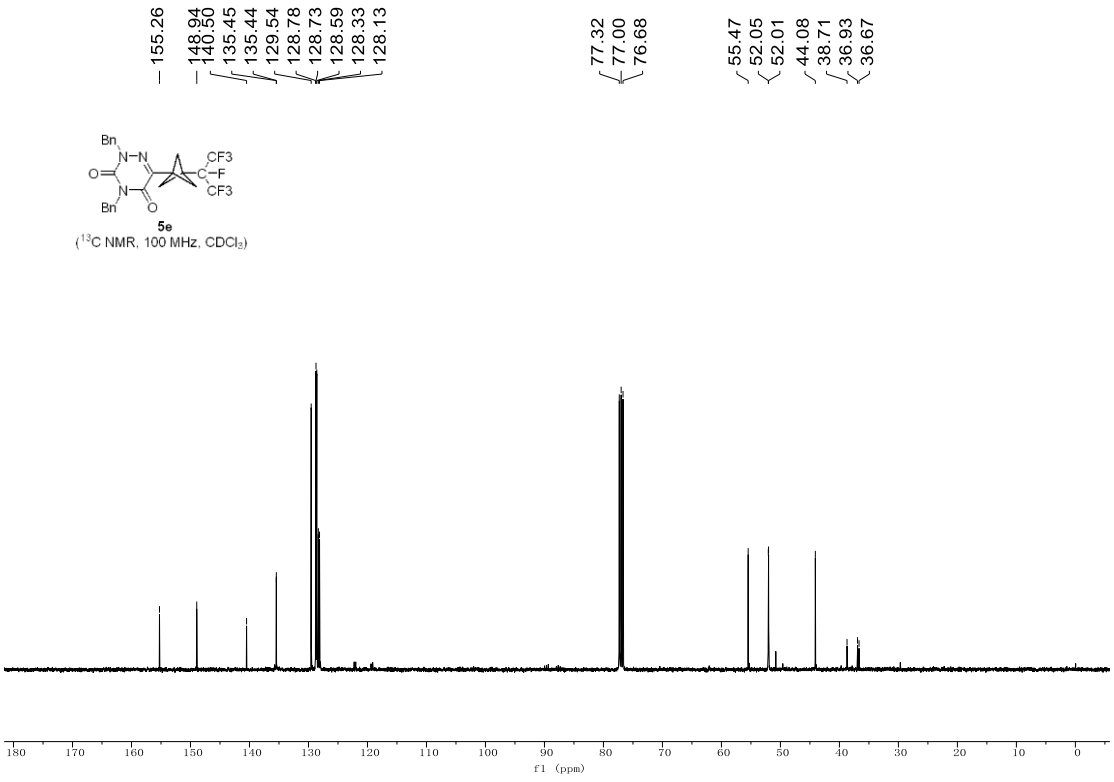
2,4-dibenzyl-6-(3-(2,2,2-trifluoroethyl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5d)



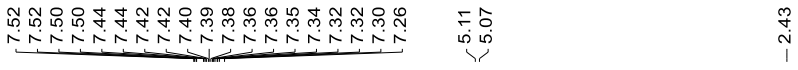


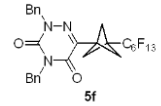
2,4-dibenzyl-6-(3-(perfluoropropan-2-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5e)




155.26
148.94
140.50
135.45
135.44
129.54
128.78
128.73
128.59
128.33
128.13
77.32
77.00
76.68
55.47
52.05
52.01
44.08
38.71
36.93
36.67
Bn
N–N
O
N
Bn
O
CF3
C–F
CF3
5e
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)
-74.66
-74.69
-183.17
-183.19
-183.22
-183.24
-183.26
-183.28
-183.30
Bn
N–N
O
N
Bn
O
CF3
C–F
CF3
5e
(19F NMR, 376 MHz, CDCl3)
3.20
3.24
1.00
f1 (ppm)

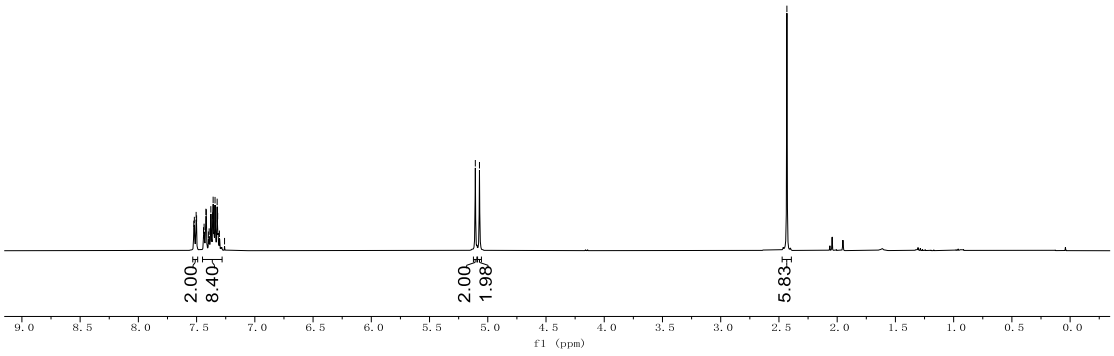
2,4-dibenzyl-6-(3-(6,6,6,6,6,6,6,6,6,6,6,6,6-tridecafluoro-6λ^{16}-hexa-1,3,5-triyn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5f)

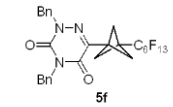




5f

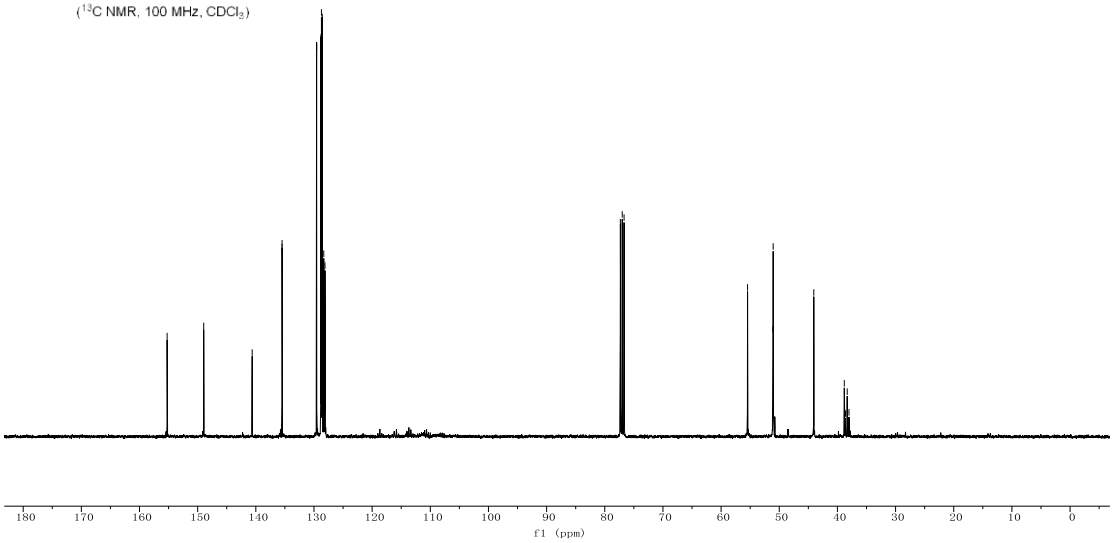
(^{1}H NMR, 400 MHz, $CDCl_3$)

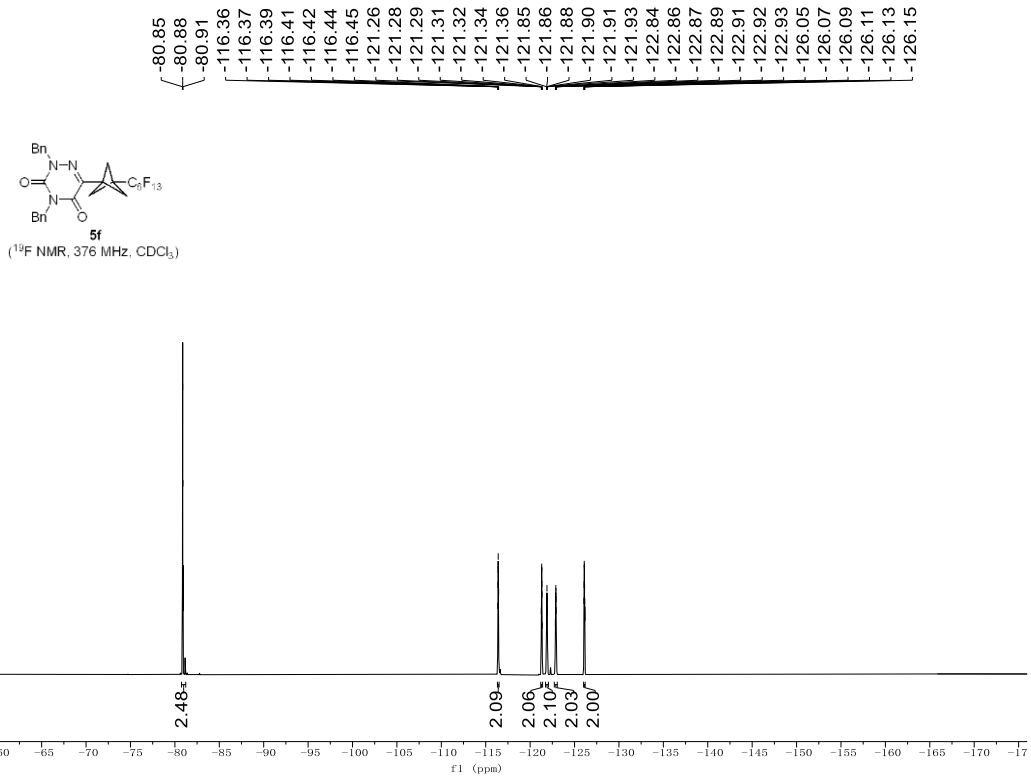




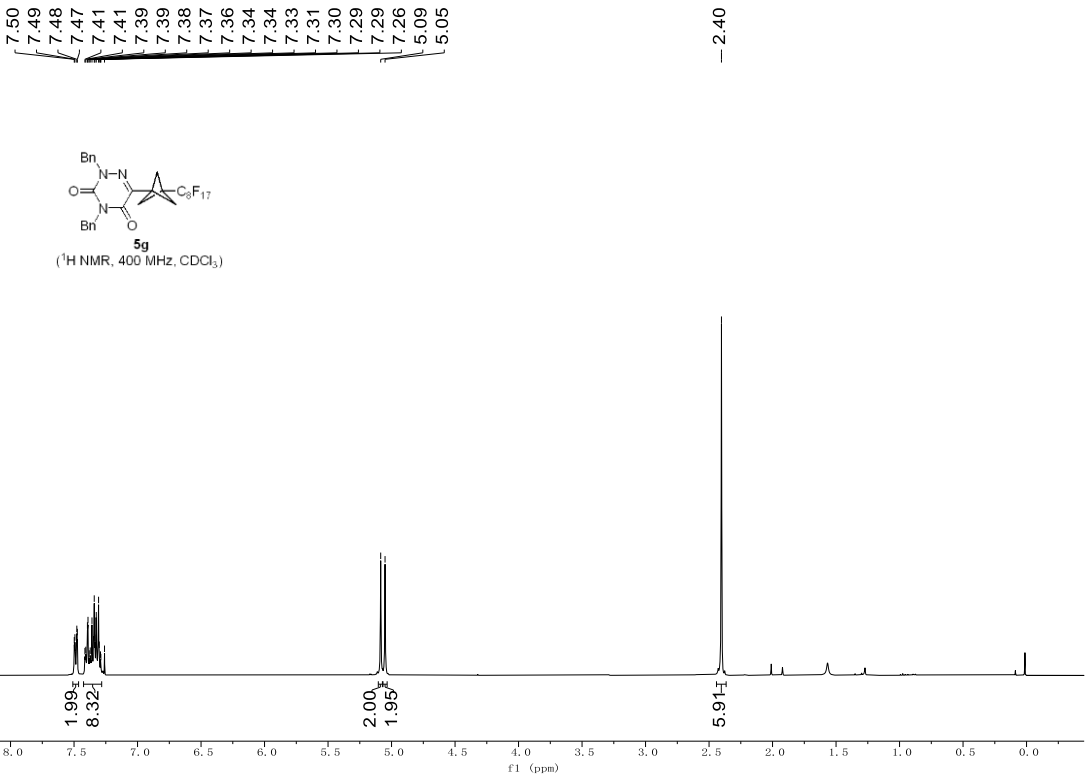
5f

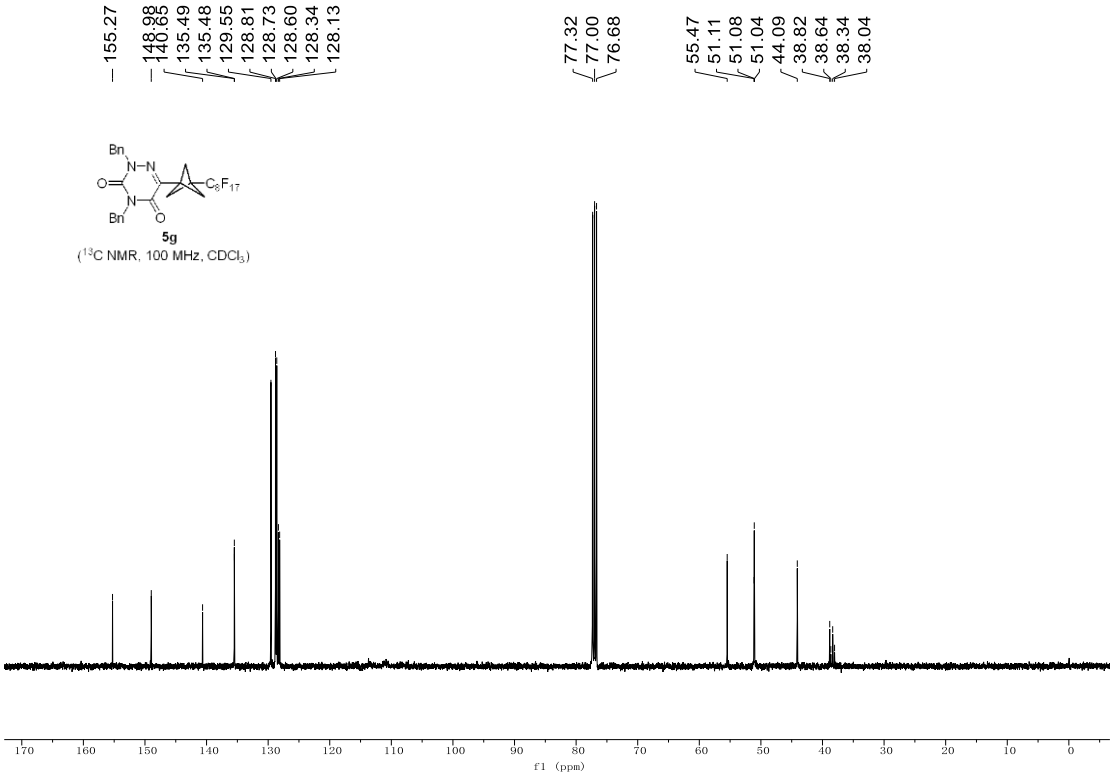
(^{13}C NMR, 100 MHz, $CDCl_3$)

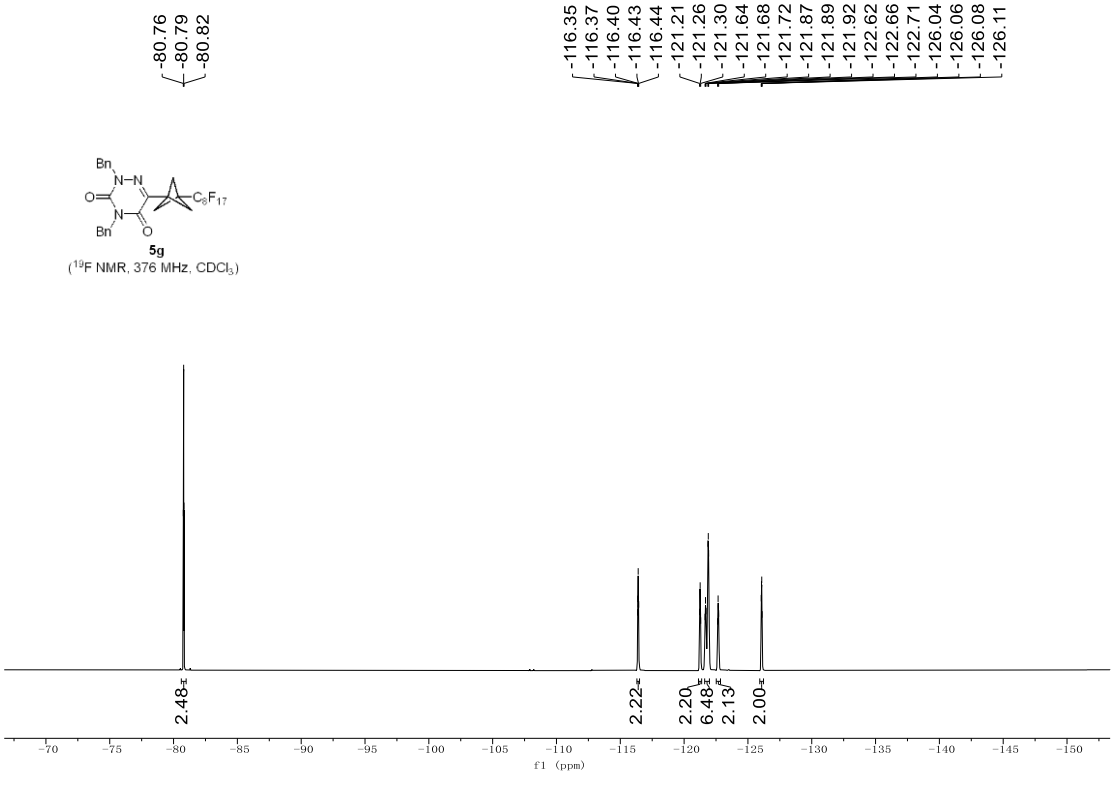




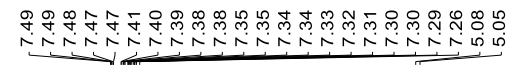
2,4-dibenzyl-6-(3-(8,8,8,8,8,8,8,8,8,8,8,8,8,8,8,8,8-heptadecafluoro-8λ^{20}-octa-1,3,5,7-tetrayn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5g)

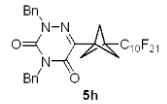



155.27
148.98
140.65
135.49
135.48
129.55
128.81
128.73
128.60
128.34
128.13
77.32
77.00
76.68
55.47
51.11
51.08
51.04
44.09
38.82
38.64
38.34
38.04
Bn
N–N
O
N
O
Bn
C8F17
5g
(13C NMR, 100 MHz, CDCl3)
f1 (ppm)


-80.76
-80.79
-80.82
-116.35
-116.37
-116.40
-116.43
-116.44
-121.21
-121.26
-121.30
-121.64
-121.68
-121.72
-121.87
-121.89
-121.92
-122.62
-122.66
-122.71
-126.04
-126.06
-126.08
-126.11
Bn
N–N
O
N
O
Bn
C8F17
5g
(19F NMR, 376 MHz, CDCl3)
2.48
2.22
2.20
6.48
2.13
2.00
f1 (ppm)

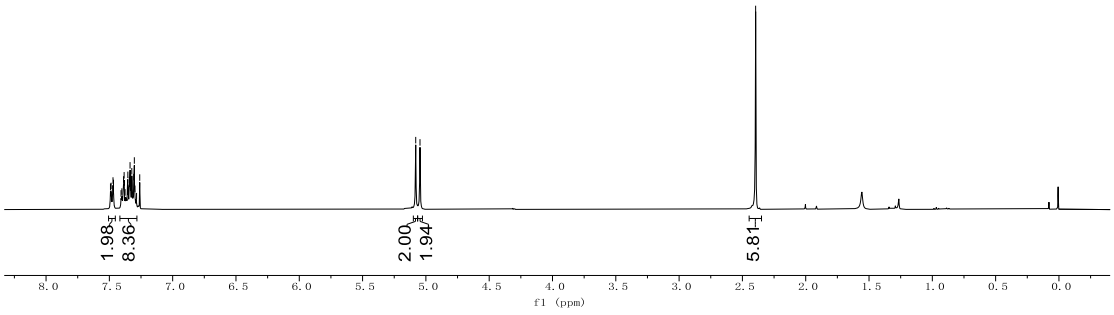
2,4-dibenzyl-6-(3-(10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10,10-henicosafluoro-10λ^{24}-deca-1,3,5,7,9-pentayn-1-yl)bicyclo[1.1.1]pentan-1-yl)-1,2,4-triazine-3,5(2*H*,4*H*)-dione (5h)

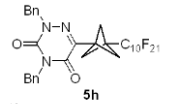




5h

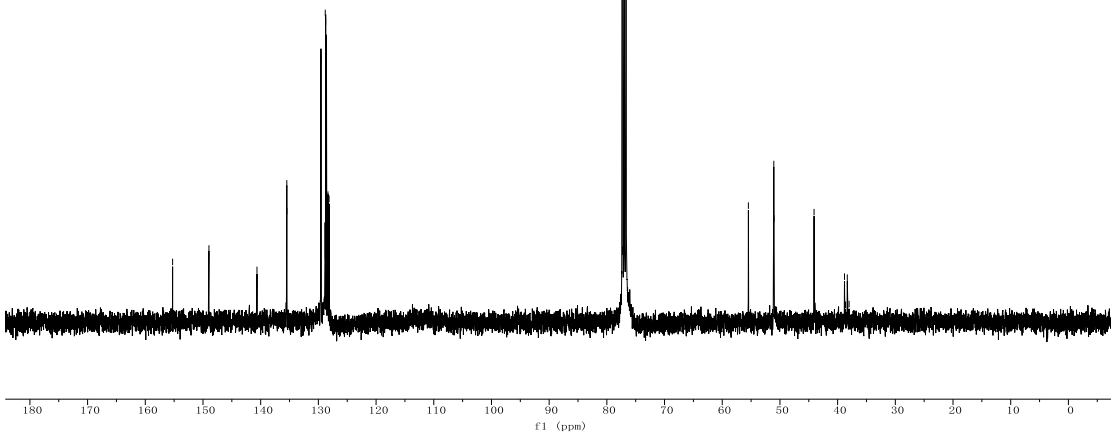
(^{1}H NMR, 400 MHz, $CDCl_3$)

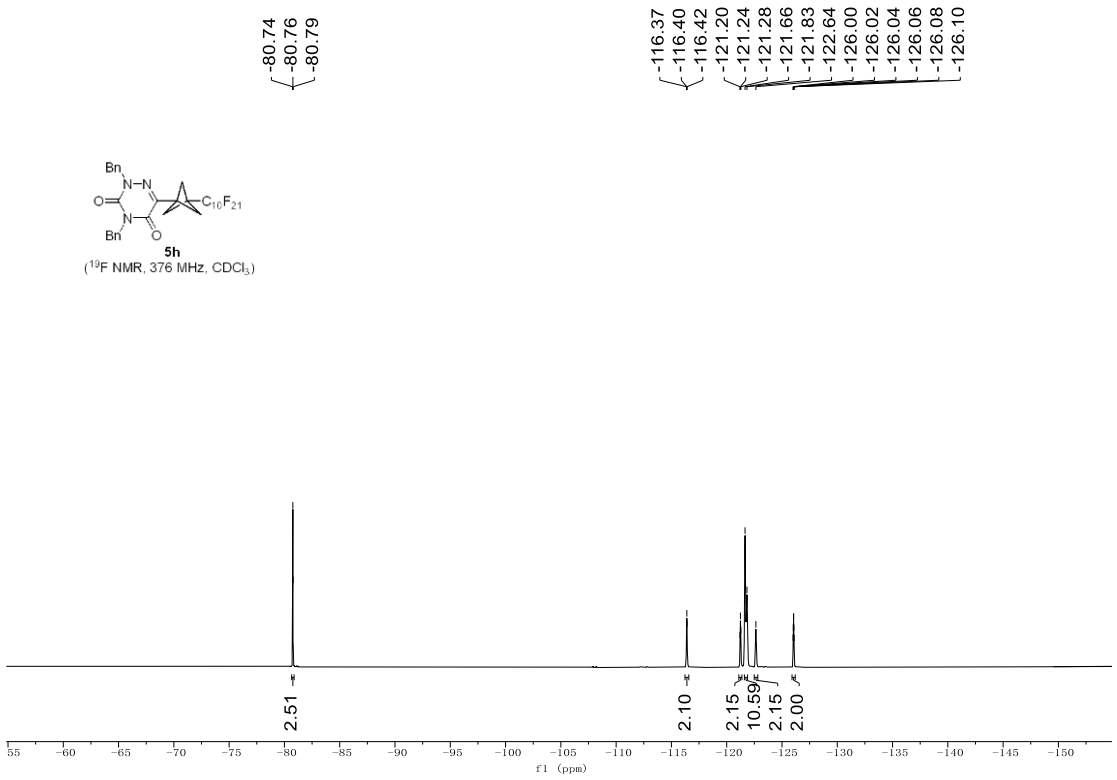




5h

(^{13}C NMR, 100 MHz, $CDCl_3$)




-80.74
-80.76
-80.79
-116.37
-116.40
-116.42
-121.20
-121.24
-121.28
-121.66
-121.83
-122.64
-126.00
-126.02
-126.04
-126.06
-126.08
-126.10
Bn
N
N
O
N
O
Bn
$C_{10}F_{21}$
5h
(^{19}F NMR, 376 MHz, $CDCl_3$)
2.51
2.10
2.15
10.59
2.15
2.00
f1 (ppm)