Electronic Supplementary Information:

Uranyl ion coordination with rigid aromatic carboxylates and structural characterization of their complexes

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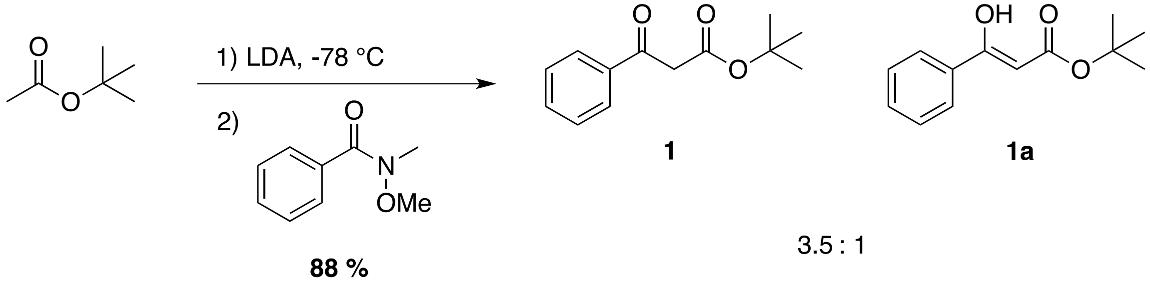
Experimental Details for the Synthesis of Reported Compounds

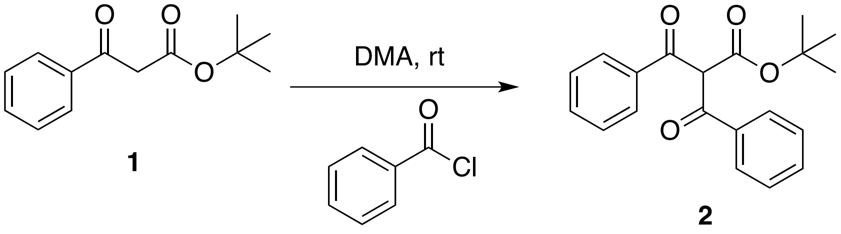
All NMR spectra were taken on a Bruker 600 MHz instrument using Top Spin software to acquire the data. After acquisition spectra were prepared using the MestReNova software package for Mac. The references to all known compounds can be found at the end of the ESI.

Beta ketoester (1)[1]



Tert-Butyl acetate (2 g, 17.2 mmol) was added to a flame dried round bottom flask and dissolved in dry THF (20 mL). The reaction mixture was cooled to -78 °C and 2 M LDA (8.6 mL) was added dropwise. The reaction mixture was stirred for 15 min before a solution of *N*-methoxy-*N*-methylbenzamide (2.58 g, 15.64 mmol) in dry THF (5 mL) was added dropwise. The reaction mixture was stirred at -78 °C for 2 h and warmed to room temperature. The pale yellow solution was poured into 1 M HCl (20 mL), extracted with Et_2O (3x50 mL), dried over $MgSO_4$, and concentrated to give a red/orange oil. The crude material was purified by silica gel column chromatography using 9:1 hexanes:EtOAc as the eluent to give **1** as a yellow oil. In solution, the compound is a mixture of tautomers (3.5:1) and the peaks of the major isomer are reported. Yield 3.021 g (88 %). 1H NMR (600 MHz, CDCl3) δ 7.99 – 7.95 (m, 2H), 7.61 (t, J = 7.4, 1H), 7.53 – 7.48 (m, 2H), 3.92 (s, 2H), 1.46 (s, J = 3.9, 9H). ESI-TOF high-acc: m/z: 221.1170 ([MH]+, $C_{13}H_{16}O_3$H+, calc. 221.1172).

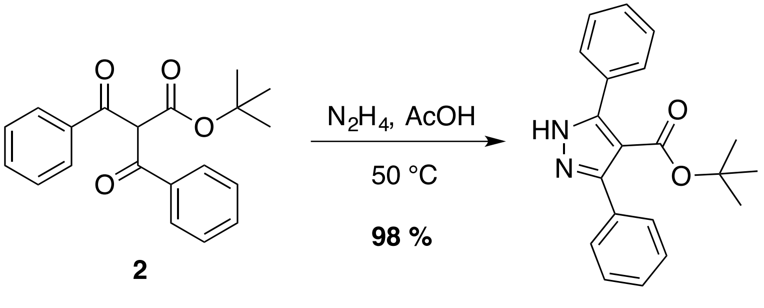
1,3-diketone (2)



86 %

Beta ketoester **1** (2.83 g, 12.8 mmol) and benzoyl chloride (1.8 g, 12.8 mmol) was dissolved in 5 mL *N,N*-Dimethylaniline (DMA). The orange solution was stirred at room temperature overnight (~20 h) giving a dark blue solution. The reaction mixture was poured into Et_2O (100 mL), washed with ice cold 10 % HCl (3x75 mL), dried over Na_2SO_4, and evaporated to dryness to give **2** as a white crystalline solid. Yield 2.904 g (86 %). 1H NMR (600 MHz, CDCl3) δ 7.92 (d, J = 8.2, 4H), 7.67 – 7.54 (m, 2H), 7.53 – 7.42 (m, 4H), 6.06 (s, 1H), 1.43 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.26, 164.90, 135.94, 133.98, 129.02, 128.69, 83.66, 65.52, 27.89. ESI-TOF high-acc: m/z: 325.1426 ([MH]+, $C_{20}H_{20}O_4H$+, calc. 325.1434).

Tert-Butyl-3,5-diphenyl-1H-pyrazole-4-carboxylate (3)

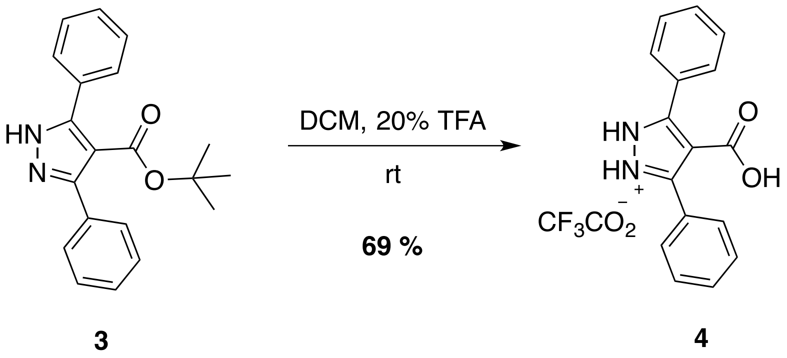


2

3

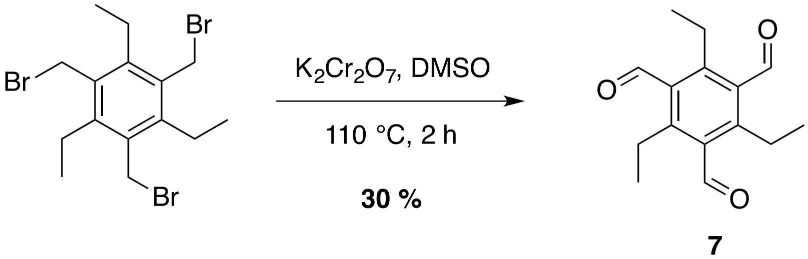
1,3-diketone **2** (0.500 g, 1.54 mmol) was dissolved in glacial acetic acid (5 mL). Anhydrous hydrazine (0.109 g, 3.4 mmol) was added dropwise and the reaction mixture was heated to 50 °C overnight (~20 h). The reaction mixture was evaporated to dryness to give a solid. The crude material was purified by silica gel column chromatography using 5 % EtOAc in DCM as the eluent to give **3** as a white solid. Yield 0.485 g (98 %). 1H NMR (600 MHz, CDCl3) δ 7.61 (m, 7.6, 4H), 7.47 – 7.39 (m, 6H), 1.28 (s, 9H). ^{13}C NMR (151 MHz, CDCl3) δ 162.99, 150.21, 130.59, 129.22, 129.16, 128.35, 111.24, 81.27, 27.94. ESI-TOF high-acc: m/z: 321.1597 ([MH]+, $C_{20}H_{20}N_2O_2H+$, calc. 321.1597).

3,5-diphenyl-1H-pyrazole-4-carboxylic acid (4)



Tert-Butyl-3,5-diphenyl-1H-pyrazole-4-carboxylate, **3** (0.250 g, 0.78 mmol) was dissolved in 26 mL DCM. TFA (5 mL) was added dropwise and the reaction mixture was stirred overnight. The reaction mixture was evaporated to dryness, suspended in 5 mL DCM, and sonicated for 1 min. The suspension was filtered and washed with DCM to give **4** as a white free flowing solid. Yield 0.203 g (69 %). 1H NMR (600 MHz, DMSO) δ 7.63 (d, J = 7.0, 4H), 7.44 (m, 23.8, 6H). ^{13}C NMR (151 MHz, DMSO) δ 165.57, 128.60, 128.08, 109.21, 99.13, 97.54. ESI-TOF high-acc: m/z: 265.0981 ([MH]+, $C_{16}H_{12}N_2O_2H^+$, calc. 265.0977).

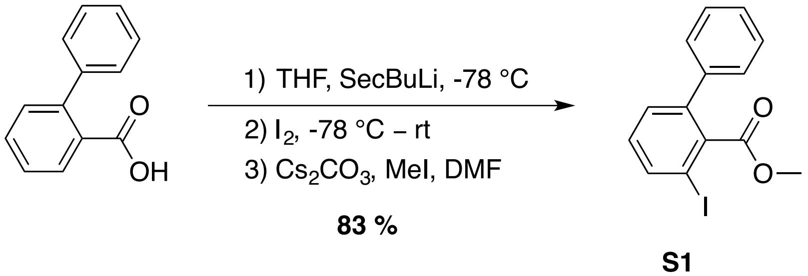
Trialdehyde (3)[2]



7

Potassium dichromate (6.0 g, 20.4 mmol) was suspended in dry DMSO (44 mL) and heated under vigorous stirring at 30 °C until dissolution. Under a nitrogen atmosphere 1,3,5-tris(bromomethyl)-2,4,6-triethylbenzene (2.00g, 4.5 mmol) was added and the reaction mixture was heated to 110 °C for 2 h. The dark mixture was poured into 175 mL of 10 M NaOH and extracted with CH_2Cl_2 (3x50 mL). The combined organic layers were washed with water (3x50 mL), dried over Na_2SO_4 and concentrated to give a pale yellow oil. The crude product was purified by silica gel column chromatography using 2:1 DCM:Hex as the eluent to give **7** as a white waxy solid. Yield 0.315 g (30 %). ^{1}H NMR (600 MHz, Chloroform-d) δ 10.61 (s, 1H), 3.00 (q, *J* = 7.4 Hz, 2H), 1.27 (t, *J* = 7.6 Hz, 3H).

Methyl 3-Iodobiphenyl-2-carboxylate (S1)[3,4]

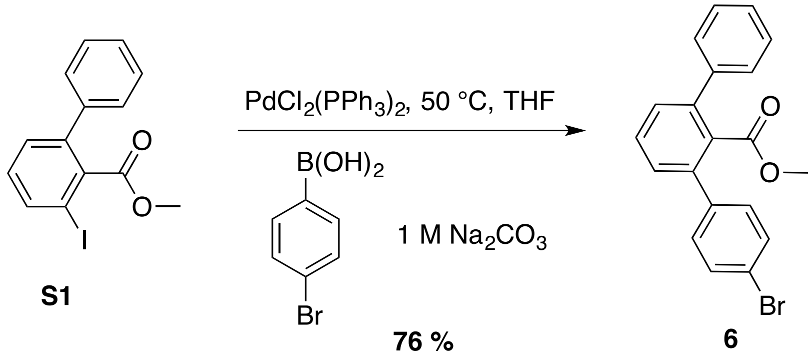


S1

*s*BuLi (1.3 M in cylohexane, 44 mL, 57.2 mmol) was added dropwise under argon at -78 °C, over a period of 30 min, to a vigorously stirred solution of biphenyl-2-carboxylic acid (5 g, 25.22 mmol) in anhydrous THF (180 mL). A deep red-orange coloration appeared during addition of the organolithium. After 2.5 h stirring at -78 °C, a solution of I_2 (19.19 g, 75.2 mmol) in anhydrous THF (100 mL) was added dropwise. The solution was then allowed to warm to room temperature over night. The mixture was transferred to a 1000 mL round bottom flask and evaporated to dryness. The crude mixture was dissolved in Et_2O (500 mL) and transferred to a sepratory funnel. The organic layer was washed with a saturated sodium sulfite solution (250 mL). The aqueous layer was removed and the organic solution was washed with a saturated sodium bicarbonate solution (300 mL). The aqueous layers were combined and washed with Et_2O (200 mL). The aqueous layer was acidified with concentrated HCl to give a bright yellow mixture. The yellow aquesous mixture was extracted with Et_2O (3 x275 mL). The combined organic layers were dried over Na_2SO_4 and evaporated to dryness to give an off white solid. Yield 7.226 g (88 %). This crude product was used without further purification.

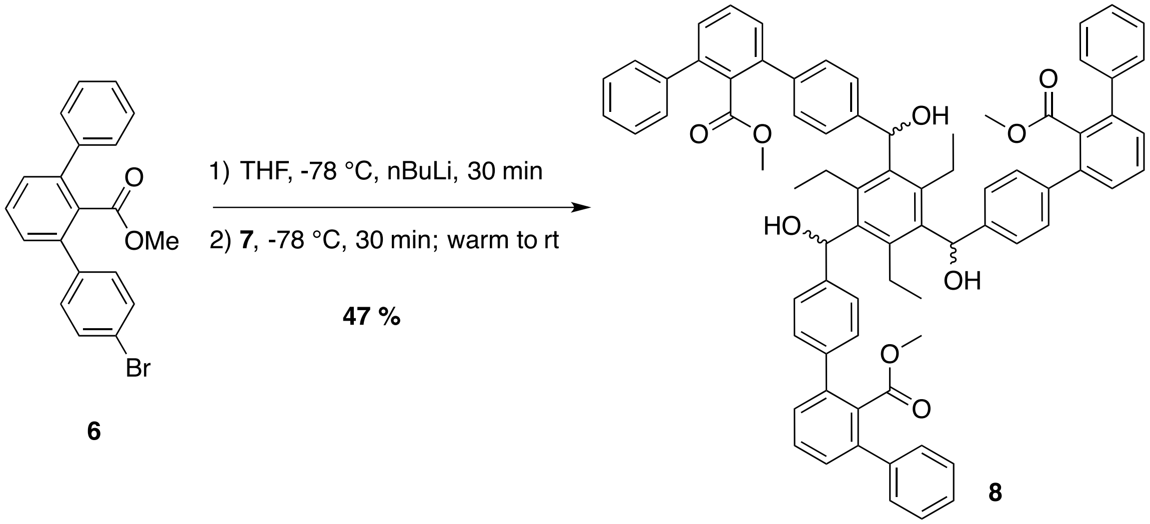
Cs_2CO_3 (8.76 g, 26.9 mmol) and methyl iodide (2.08 mL, 33.60 mmol) were added to a solution of the above crude product (7.226 g) in DMF (30 mL). The suspension was stirred for 0.5 h at room temperature, after which the solvents were removed by rotary evaporation. The remaining solids were extracted with DCM (250 mL) and washed with a saturated solution of sodium bicarbonate (2 x250 mL) and brine (150 mL). The organic layer was separated, dried over $MgSO_4$, and concentrated to dryness to give a white waxy solid, **S1**. Yield 7.54 g (95 %). ^{1}H NMR (600 MHz, Chloroform-d) δ 7.84 (d, *J* = 7.6 Hz, 1H), 7.41 – 7.34 (m, 6H), 7.18 – 7.14 (m, 1H), 3.67 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.47, 141.70, 139.70, 139.49, 138.09, 130.85, 129.61, 128.56, 128.35, 128.14, 92.68, 52.62. ESI-TOF high-acc: m/z: 338.9883 ([MH]+, $C_{14}H_{11}IO_2H^+$, calc. 338.9877).

Methyl 4-Bromo-1,1':3',1''-terphenyl-2'-carboxylate (6)[5]



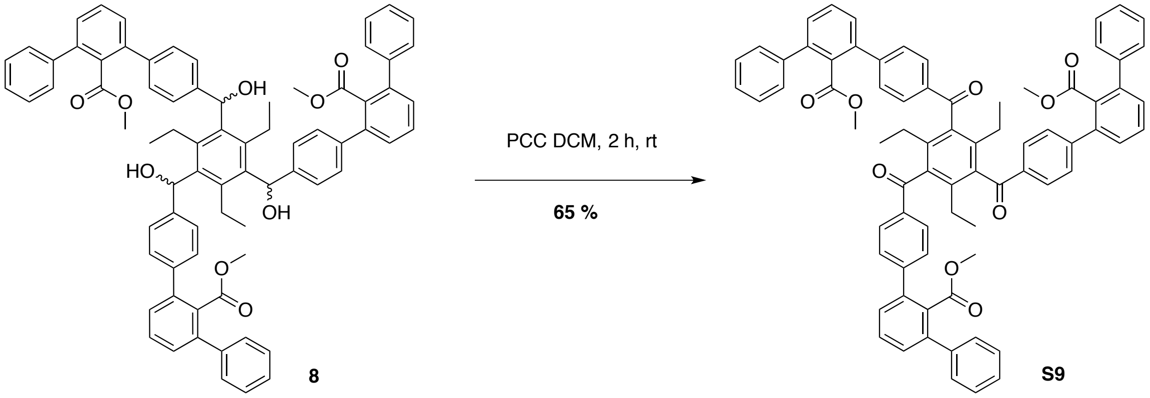
4-Bromophenylboronic acid (1.78 g, 8.86 mmol) was added to methyl 3-iodobiphenyl-2-carboxylate **S1** (2.00 g, 5.91 mmol) in THF (20 mL) and 1 M aqueous Na_2CO_3 (15 mL). The reaction mixture was deoxygenated by bubbling argon through it for 20 min, after which $[PdCl_2(PPh_3)_2]$ (0.210 g, 5 mol%) was added. The reaction mixture was heated to 50 °C, and the brownish orange-red emulsion turned yellow after 1 h. After 6 h, the reaction mixture was cooled to room temperature. Water (50 mL) was added and the product extracted with CH_2Cl_2 (2x50 mL). The combined organic phases were dried with $MgSO_4$, filtered, and concentrated to dryness. The crude material was dissolved in CH_2Cl_2, added to a sample of silica gel and evaporated to dryness. The material was then purified with silica gel column chromatography using 95:5 hexanes:diethyl ether as the eluent to give **6** as a white solid. Yield 1.65 g (76 %). ^1H NMR (600 MHz, Chloroform-d) δ 7.56 – 7.49 (m, 3H), 7.42 – 7.33 (m, 7H), 7.28 (d, *J* = 8.6 Hz, 2H), 3.40 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.80, 140.67, 140.44, 139.55, 139.21, 132.80, 131.62, 130.21, 129.66, 129.35, 128.79, 128.50, 128.47, 127.79, 122.10, 52.06. ESI-TOF high-acc: m/z: 367.0321 ([MH]+, $C_{20}H_{15}BrO_2H^+$, calc. 367.0328).

Terphenyl benzylic alcohol (8)



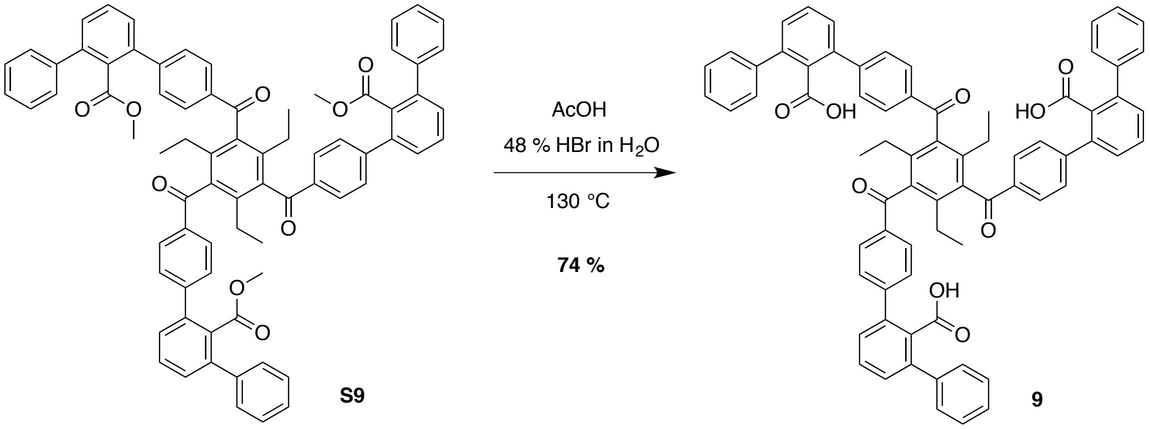
Methyl 4-Bromo-1,1’:3’,1’’-terphenyl-2’-carboxylate **6** (1.267 g, 3.45 mmol) was added to a flame dried two-neck round bottom flask and placed under an N_2 atmosphere. Dry THF (50 mL) was added to the flask and the reaction mixture was cooled to -78 °C. Once cool, a 1.6 M soln of nBuLi (1.97 mL, 3.16 mmol) was added dropwise giving a yellow solution. After stirring the reaction mixture at -78 °C for 0.5 h, a solution of **7** (0.236 g, 0.958 mmol) in THF (8 mL) was added dropwise. The reaction mixture was stirred at -78 °C for 1 h and then warmed to room temperature, giving an orange solution. The reaction mixture was quenched with water (5 mL) and washed with 10 % HCl (2x50 mL) and brine (50 mL). The organic phase was dried over Na_2SO_4 and concentrated to dryness. The crude solid was purified with silica gel column chromatography using 7:4 Hex:EtOAc as the eluent to give a white foam which is a mixture of diastereomers that was used without further purification. Yield 0.501 g (47 %). ESI-TOF high-acc: m/z: 1111.4772 ([MH]+, $C_{75}H_{66}O_9H^+$, calc. 1111.4779).

Terphenyl triethylbenzene triketone alcohol methylester (S9)



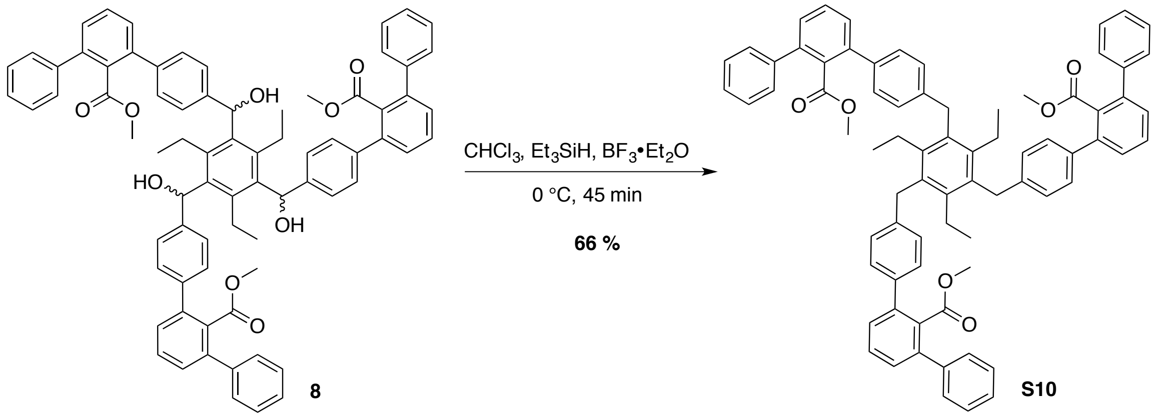
PCC (0.305 g, 1.42 mmol) was suspended in DCM (50 mL) and stirred. To the stirring mixture a solution of **8** (0.350 g, 0.315 mmol) in DCM (5 mL) was added dropwise. The reaction was stirred for 2 hours at room temperature and 0.300 g celite was added. The suspension was filtered and the filtrate was evaporated to dryness to give a brown solid. The crude material was dissolved in CH_2Cl_2, added to a sample of silica gel and evaporated to dryness. The resulting solid was purified with silica gel column chromatography using 5:1 Hex:EtOAc as the eluent to give **S9** as an off white foam. Yield 0.225 g (65 %). 1H NMR (600 MHz, CDCl3) δ 7.94 (bs, 5H), 7.53 (m, 9H), 7.39 (bs, 22H), 3.33 (bs, 9H), 2.53 (bs, 2H), 2.28 (bs, 4H), 1.07 (bs, 3H), 0.84 (bs, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.23, 169.44, 146.60, 146.44, 140.76, 140.19, 139.07, 138.19, 138.14, 136.36, 132.67, 129.77, 129.64, 129.03, 128.59, 128.40, 128.33, 127.73, 51.89, 25.39, 24.36, 16.34, 15.46. ESI-TOF high-acc: m/z: 1127.4121 ([MNa]+, $C_{75}H_{60}O_9Na^+$, calc. 1127.4129).

Terphenyl triethylbenzene triketone carboxylic acid (9)



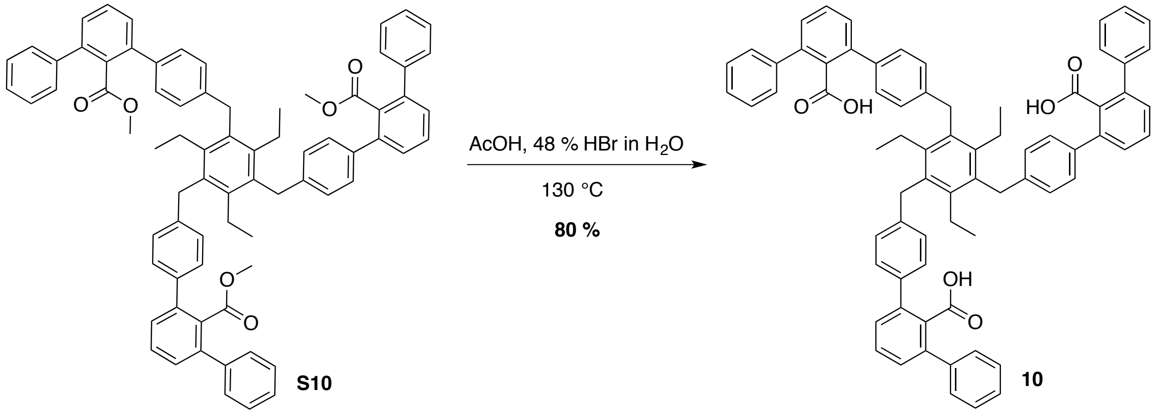
S9 (0.075, 0.0068 mmol) was dissolved in glacial acetic acid (3 mL). The mixture was stirred and 48 % HBr in H_2O was added dropwise (0.5 mL) causing a precipitate to form. The suspension was heated to 130 °C overnight, forming a solution upon warming. After stirring at 130 °C for 17 h an off white suspension forms. The reaction mixture is cooled to room temperature and the mixture is sonicated to break up the precipitate into a fine powder. The reaction mixture was filtered and washed with water. The off white solid **9** was dried overnight under vacuum. Yield 0.054 g (74 %). ^{1}H NMR (600 MHz, Methanol-d4) δ 8.02 – 7.89 (m, 6H), 7.67 (d, *J* = 7.7 Hz, 5H), 7.60 – 7.52 (m, 4H), 7.44 – 7.34 (m, 21H), 2.50 (s, 2H), 2.27 (s, 4H), 1.04 (s, 6H), 0.84 (s, 3H). ^{13}C NMR (151 MHz, MeOD) δ 148.40, 141.78, 141.37, 139.71, 139.43, 137.58, 135.28, 131.01, 130.89, 130.75, 130.66, 130.39, 129.75, 129.66, 129.45, 129.41, 129.32, 128.80, 128.68, 26.26, 25.30, 16.56, 15.85. ESI-TOF high-acc: m/z: 1085.3646 ([MNa]+, $C_{72}H_{54}O_9$+, calc. 1085.366).

Terphenyl triethylbenzene methylester (S10)



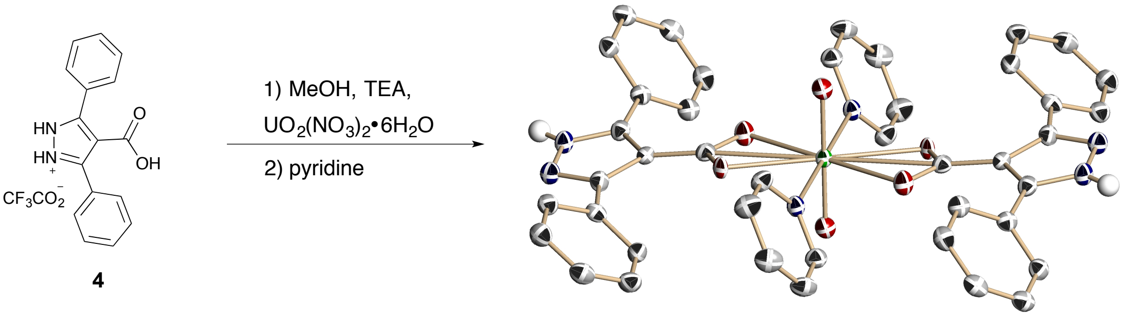
8 (0.300 g, 0.27 mmol) was dissolved in dry CHCl$_3$ (30 mL) and cooled to 0 °C under a N$_2$ atmosphere. Triethylsilane (1.08 mL, 6.75 mmol) was added dropwise to the reaction mixture followed by the addition of BF$_3$•Et$_2$O (0.5 mL, 4.05 mmol). The reaction mixture was stirred at 0 °C for 1 h and then warmed to room temperature. The reaction was quenched by the addition of 10 % HCl (30 mL) and extracted with DCM. The combined organic layers were washed with brine, dried over MgSO$_4$, and concentrated to dryness. The crude material was purified with silica gel column chromatography starting with toluene as the eluent and gradually changing to 2:1 toluene:DCM to give an off white foam. Yield 0.190 g (66 %). 1H NMR (600 MHz, CDCl3) δ 7.46 (t, J = 7.7, 3H), 7.41 – 7.31 (m, 21H), 7.28 (d, J = 8.2, 6H), 7.05 (d, J = 8.1, 6H), 4.19 (s, 6H), 3.29 (s, 9H), 2.53 (q, J = 7.1, 6H), 1.07 (t, J = 7.4, 9H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 170.02, 141.48, 140.96, 140.68, 140.37, 140.27, 138.07, 134.09, 132.84, 129.44, 128.97, 128.79, 128.51, 128.48, 128.39, 127.88, 127.60, 51.81, 34.57, 23.89, 15.28. ESI-TOF high-acc: m/z: 1063.4932 ([MH]+, C$_{75}$H$_{66}$O$_6$H+, calc. 1063.4932).

Terphenyl triethylbenzene carboxylic acid (10)



S10 (0.142 g, 0.13 mmol) was dissolved in glacial acetic acid (5 mL). The mixture was stirred and 48 % HBr in H_2O was added dropwise (1 mL) causing a precipitate to form. The suspension was heated to 130 °C overnight, forming a solution upon warming. After stirring at 130 °C for 17 h an off white suspension formed. The reaction mixture was cooled to room temperature and the mixture was sonicated to break up the precipitate into a fine powder. The reaction mixture was filtered and washed with water. The off white solid **10** was dried overnight under vacuum. Yield 0.109 g (80 %). 1H NMR (600 MHz, MeOD) δ 7.53 – 7.23 (m, 30H), 7.08 (d, J = 7.8, 6H), 4.23 (s, 6H), 2.54 (q, J = 7.4, 6H), 1.09 (t, J = 7.4, 9H). ^{13}C NMR (151 MHz, MeOD) δ 173.73, 142.63, 142.14, 142.00, 140.97, 140.83, 139.52, 139.37, 135.09, 130.15, 130.07, 129.76, 129.70, 129.35, 129.24, 128.75, 128.53, 35.30, 24.73, 15.65. ESI-TOF high-acc: m/z: 1021.4453 ([MH]+, $C_{72}H_{60}O_6H^+$, calc. 1021.4462).

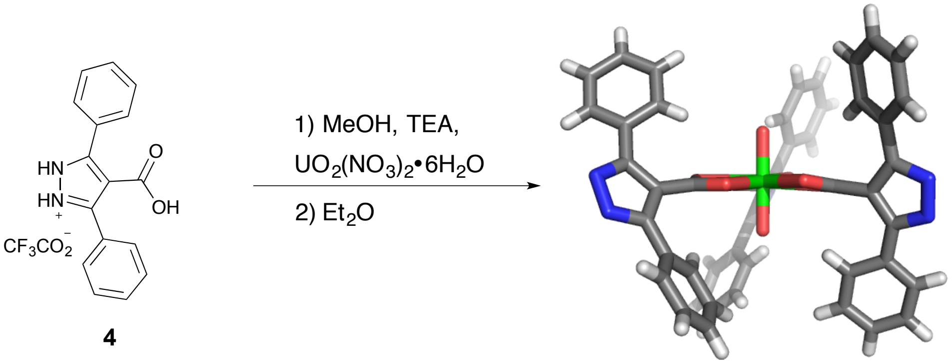
Synthesis of uranyl complexes with pyrazole ligand 4



4

$UO_2(\mathbf{4})_2(pyr)_2$

Pyrazole **4** (0.075 g, 0.198 mmol) was added to methanol (30 mL) and stirred. Triethylamine (0.060 g, 0.595 mmol) was added to the stirring mixture followed by the addition of $UO_2(NO_3)_2$•$6H_2O$ (0.048 g, 0.095 mmol) to give a yellow solution. The solution was evaporated to dryness under reduced pressure. The resulting residue was dissolved in pyridine to give a yellow solution. Upon slow diffusion of pentane at room temperature, yellow prism-like crystals suitable for X-ray diffraction formed. ^{1}H NMR (600 MHz, DMF-*d*$_7$) δ 8.65 – 8.58 (m, 4H), 8.06 (bs, 4H), 7.84 – 7.80 (m, 2H), 7.77 (d, *J* = 7.3 Hz, 4H), 7.58 (s, 4H), 7.46 (bd, *J* = 24.5 Hz, 2H), 7.42 – 7.38 (m, 4H). ^{13}C NMR (151 MHz, DMF-*d*$_7$) δ 166.94, 150.91, 137.14, 130.70, 130.03, 129.92, 129.31, 129.09, 125.02, 110.48.
Mass: This complex was not dectectable by mass spectroscopy.



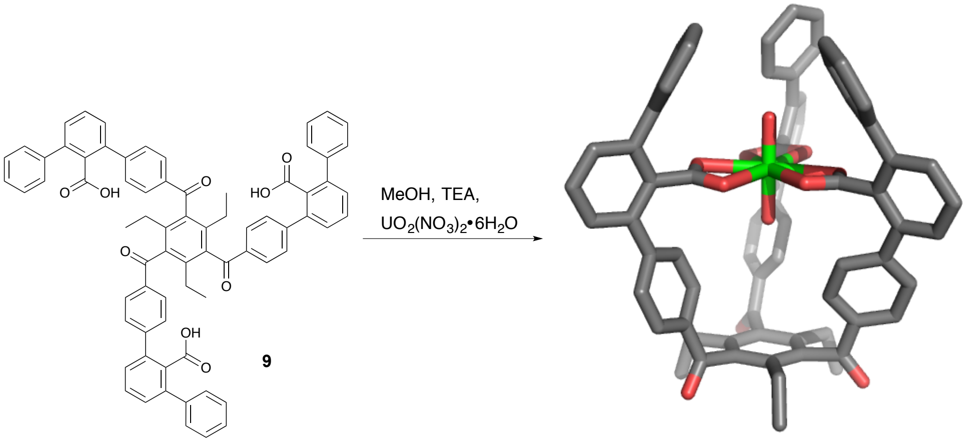
4

$UO_2(\mathbf{4})_3$

Pyrazole **4** (0.030 g, 0.08 mmol) was added to methanol (3 mL) and stirred. Triethylamine (0.025 g, 0.250 mmol) was added to the stirring mixture followed by the addition of $UO_2(NO_3)_2$•$6H_2O$ (0.014 g, 0.027 mmol) to give a yellow solution. Upon slow diffusion of Et_2O into the reaction mixture, pale yellow plate-like crystals suitable for X-ray diffraction formed. ^{1}H NMR (600 MHz, DMF-*d*$_7$) δ 8.12 (d, J = 7.3 Hz, 6H), 8.10 – 8.05 (m, 4H), 7.78 (d, J = 7.6 Hz, 2H), 7.51 –

7.42 (m, 6H), 7.37 – 7.17 (m, 12H), 3.05 (s, 6H), 1.20 (t, J = 7.2 Hz, 9H). ^{13}C NMR (151 MHz, DMF-d_7) δ 183.08, 129.99, 129.78, 129.21, 129.10, 128.94, 117.05, 47.48, 10.40.
Mass: This complex was not dectectable by mass spectroscopy.

Synthesis of uranyl complex $UO_2(9)$• Et_3NH



9

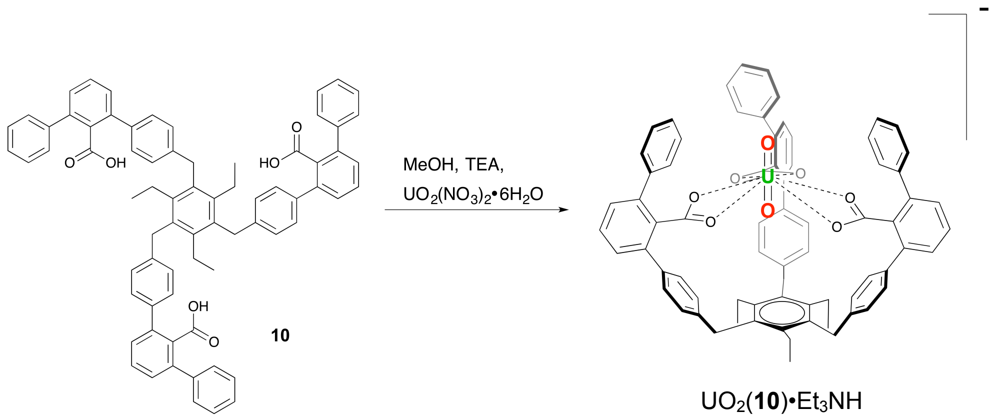
UO_2(**9**)•Et_3NH

Ligand **9** (0.030 g, 0.028 mmol) was suspended in MeOH (50 mL). Triethylamine (0.009 g, 0.088 mmol) was added to give a solution. $UO_2(NO_3)_2$•$6H_2O$ (0.014 g, 0.028 mmol) was dissolved in 10 mL MeOH and added dropwise to the ligand solution giving a clear, pale yellow solution. The mixture was stirred for an additional 30 minutes, evaporated to dryness, suspended in H_2O (5 mL), and triturated. The resulting suspension was filtered and washed with H_2O (20 mL) to give a pale yellow solid (0.038 g). The solid was purified by triturating with MeOH (5 mL). The suspension was filtered and the solid was collected to give UO_2(**9**)•Et_3NH as an off white solid (0.015 g, 38 % yield). Single crystals suitable for X-ray diffraction were grown by layering cyclopentane onto a dillute DCM solution of the crude material (without MeOH trituration) at room temperature.

^{1}H NMR (600 MHz, DMF-d_7) δ 8.39 (s, 3H), 7.94 (d, *J* = 7.6 Hz, 4H), 7.79 (d, *J* = 7.6 Hz, 4H), 7.76 – 7.66 (m, 9H), 7.63 (d, *J* = 7.6 Hz, 3H), 7.25 (s, 3H), 7.16 (t, *J* = 7.5 Hz, 3H), 7.11 (s, 3H), 6.97 (t, *J* = 7.7 Hz, 4H), 3.21 (s, 6H), 2.14 (q, *J* = 6.7 Hz, 6H), 1.27 (s, 9H), 1.06 (t, *J* = 7.1 Hz, 9H).

^{13}C NMR (151 MHz, DMF-d_7) δ 198.19, 145.72, 141.34, 140.42, 140.38, 138.65, 138.62, 138.26, 136.50, 135.41, 131.30, 129.95, 129.93, 129.71, 129.55, 129.02, 128.98, 128.02, 127.57, 47.29, 26.99, 17.46. ESI-TOF high-acc: m/z: 1329.4002 ([M-H]-, $C_{72}H_{51}O_{11}U$-, calc. 1329.3945).

Synthesis of uranyl complex UO$_2$(10)• Et$_3$NH



10

UO$_2$(**10**)•Et$_3$NH

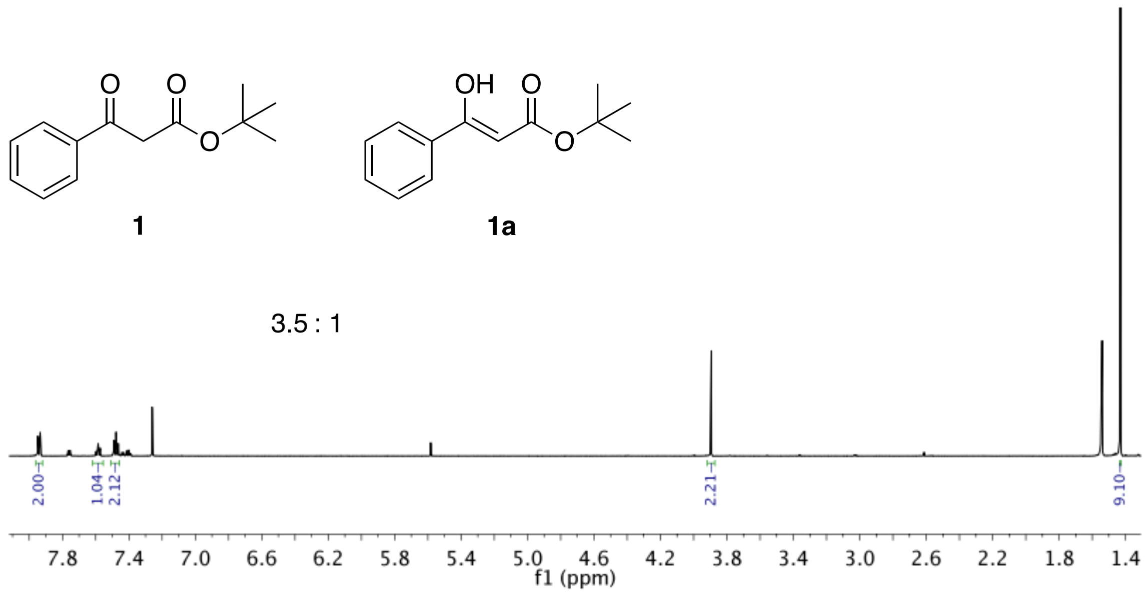
Ligand **10** (0.020 g, 0.019 mmol) was suspended in MeOH (50 mL). Triethylamine (0.006 g, 0.06 mmol) was added to give a solution. UO$_2$(NO$_3$)$_2$•6H$_2$O (0.010 g, 0.019 mmol) was dissolved in 10 mL MeOH and added dropwise to the ligand solution giving a clear, pale yellow solution. After 10 minutes stirring at room temperature a fine white precipitate forms. The mixture was stirred for an additional 30 minutes, evaporated to dryness, suspended in H$_2$O (5 mL), and triturated. The resulting suspension was filtered and washed with H$_2$O (20 mL) to give a pale yellow solid (0.026 g). The solid was purified by triturating with MeOH (5 mL). The suspension was filtered and the solid was collected to give UO$_2$(**10**)•Et$_3$NH as an off white solid (0.012 g, 44 % yield).

^{1}H NMR (600 MHz, DMF-*d*$_7$) δ 8.20 (s, 6H), 8.00 – 7.89 (m, 9H), 7.79 (t, *J* = 7.9 Hz, 3H), 7.70 (d, *J* = 7.7 Hz, 3H), 7.29 (t, *J* = 7.4 Hz, 3H), 7.08 (t, *J* = 7.8 Hz, 6H), 6.88 (d, *J* = 8.4 Hz, 6H), 4.12 (s, 6H), 3.44 (s, 6H), 2.62 (q, *J* = 7.4 Hz, 6H), 1.40 (t, *J* = 7.2 Hz, 9H), 1.35 (t, *J* = 7.3 Hz, 9H).

^{13}C NMR (151 MHz, DMF-*d*$_7$) δ 186.97, 141.68, 141.20, 140.36, 140.00, 139.91, 138.46, 137.48, 135.26, 130.04, 129.91, 129.72, 129.50, 129.47, 129.18, 127.52, 47.60, 34.52, 24.81, 16.37.

ESI-TOF high-acc: m/z: 1287.4613 ([M-H]-, $C_{72}H_{57}O_8U^-$, calc. 1287.4567).

NMR Spectra: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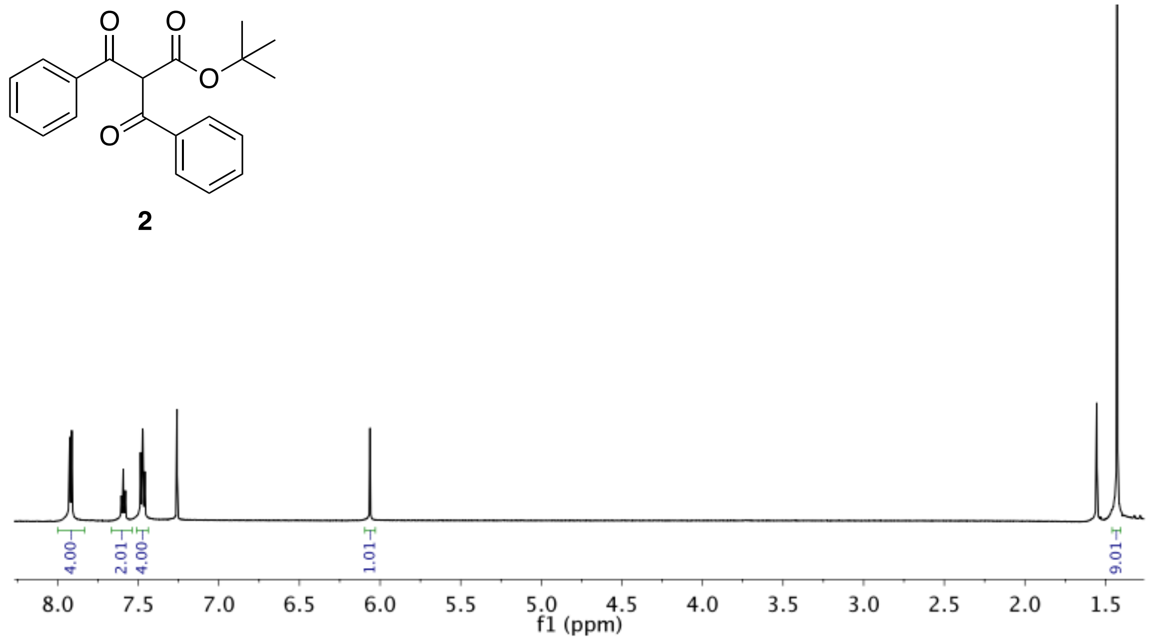
O
O
1
OH
O
O
1a
3.5 : 1
2.00
1.04
2.12
2.21
9.10
7.8
7.4
7.0
6.6
6.2
5.8
5.4
5.0
4.6
4.2
3.8
3.4
3.0
2.6
2.2
1.8
1.4
f1 (ppm)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O
O
O
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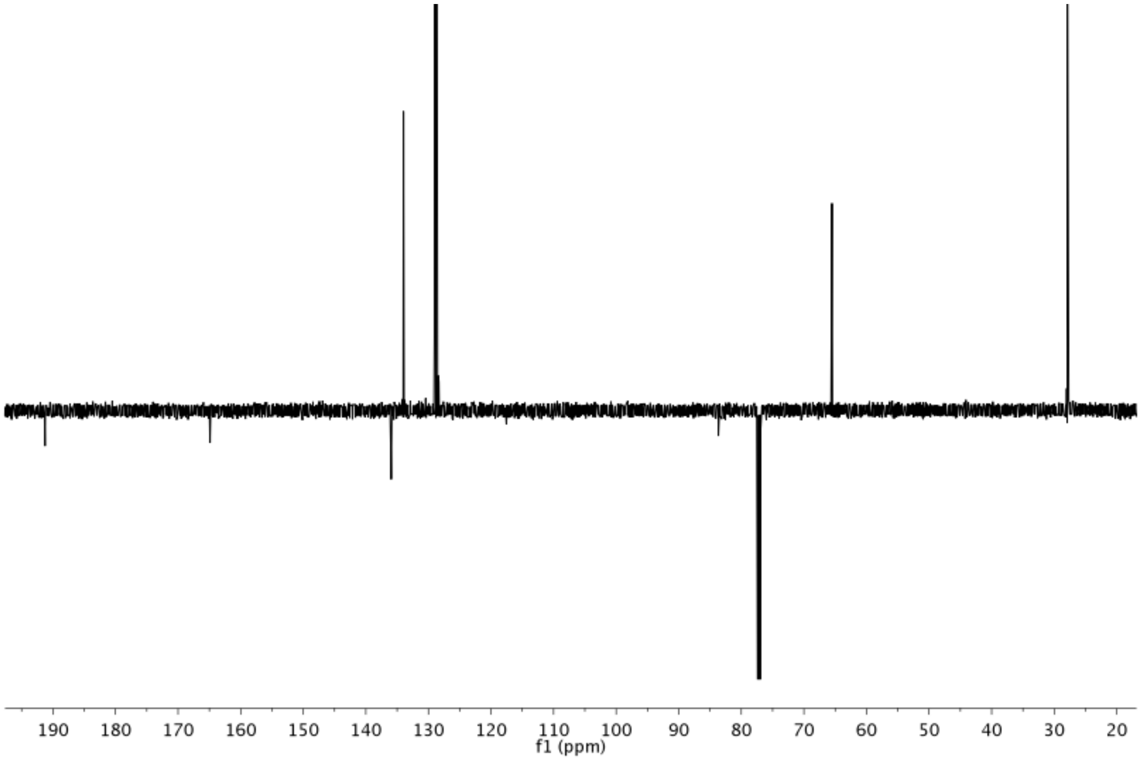
2.01
4.00
1.01
9.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)

2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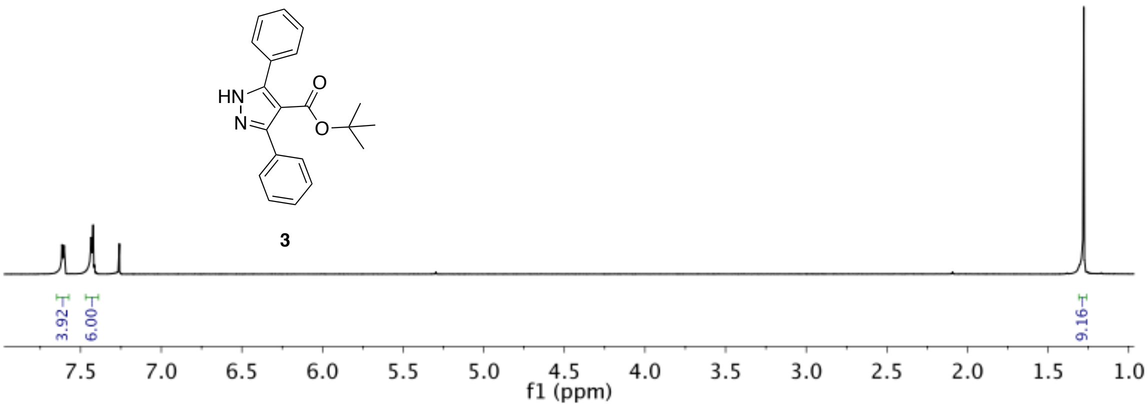
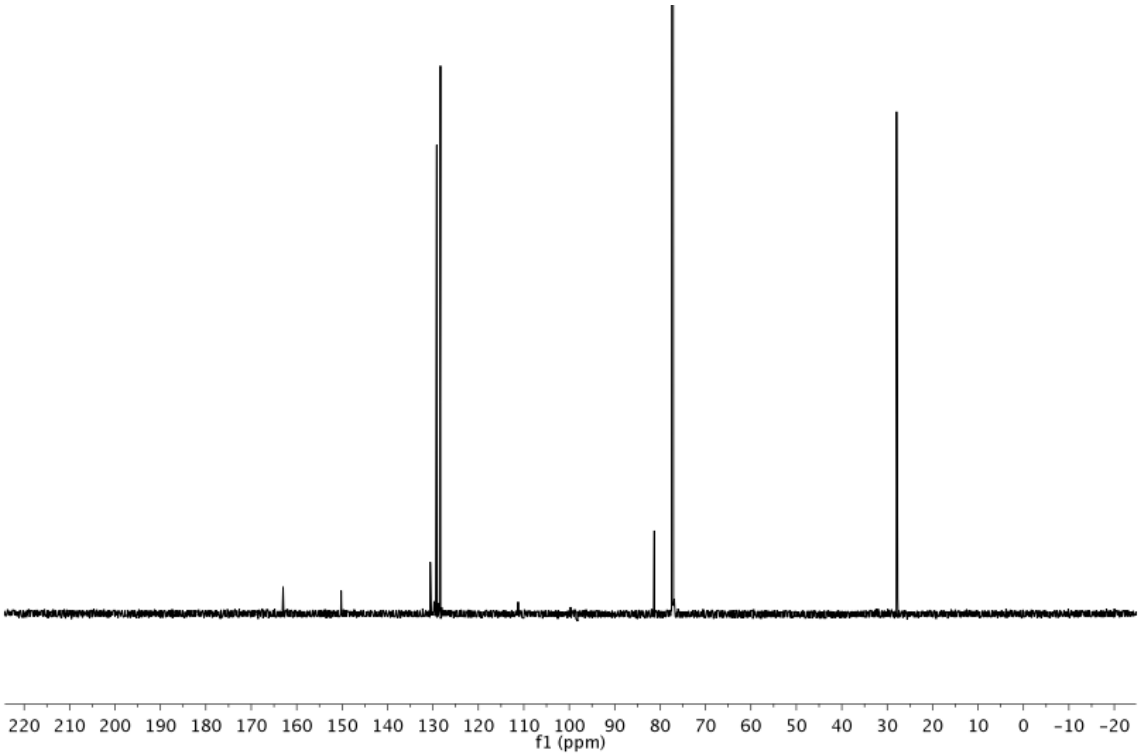
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

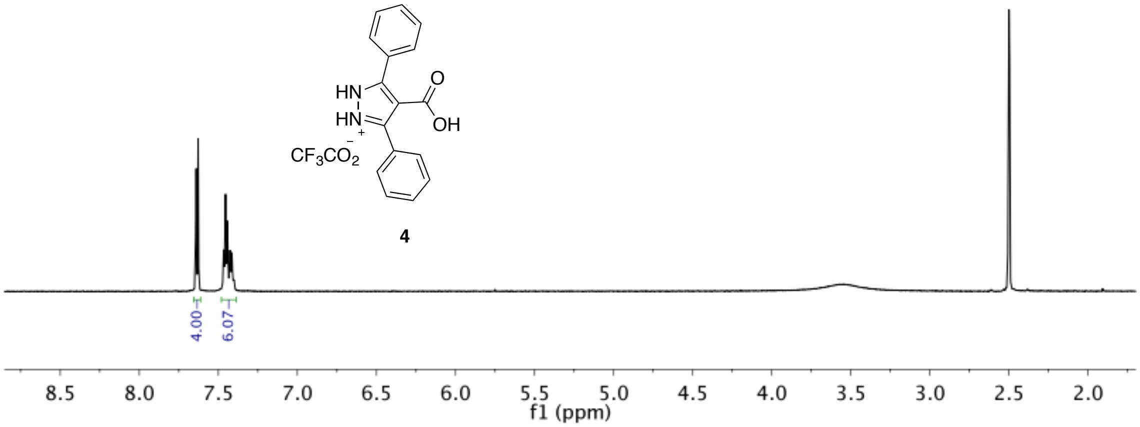


3

HN
N
O
O
3.92
6.00
9.16
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)



220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
−20
f1 (ppm)


HN
HN
+
O
OH
CF3CO2−
4
4.00
6.07
8.5
8.0
7.5
7.0
6.5
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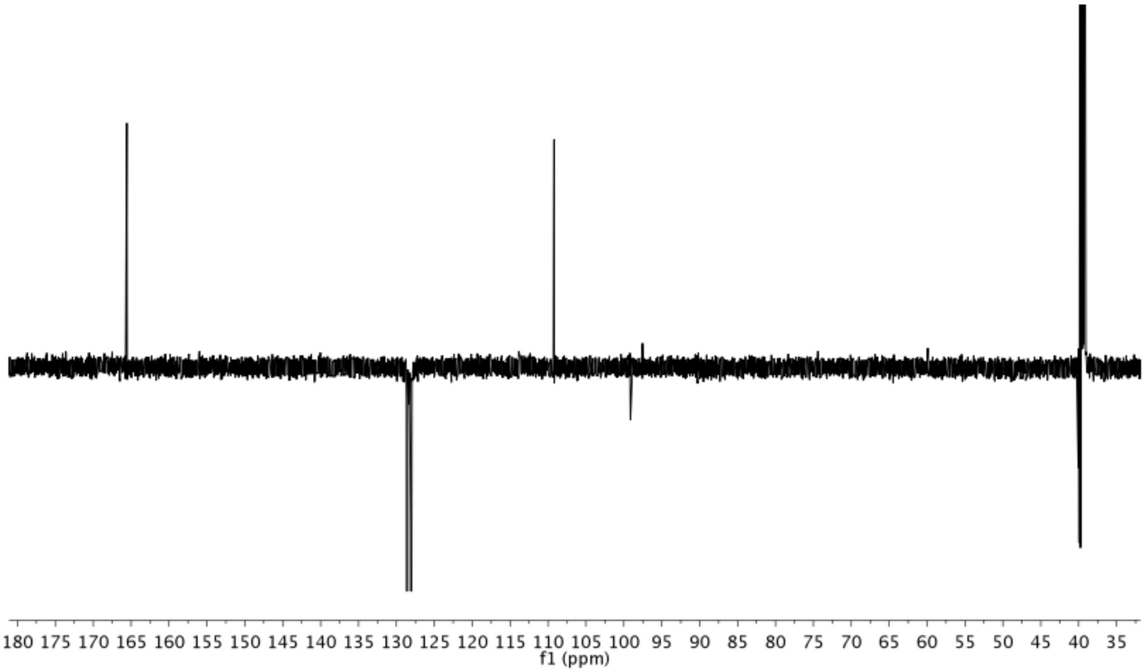
2.5
2.0
f1 (ppm)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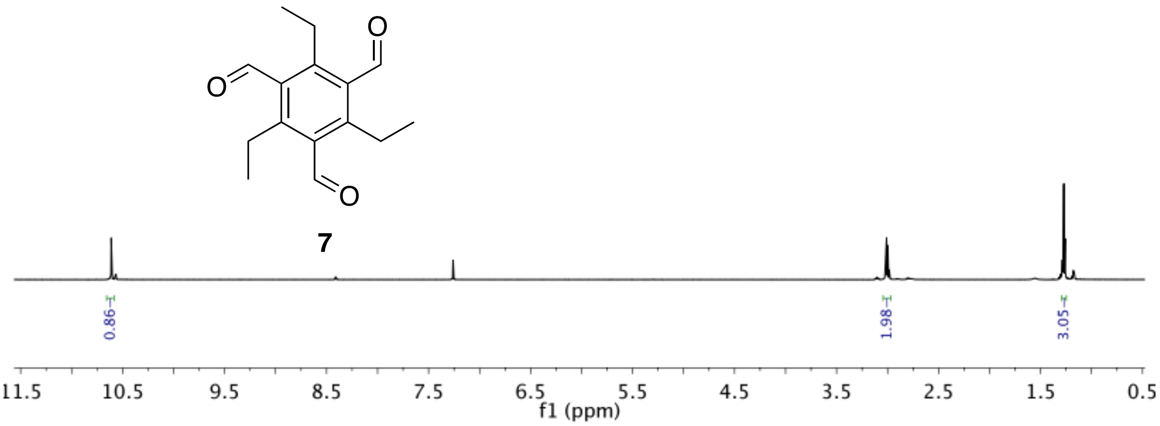
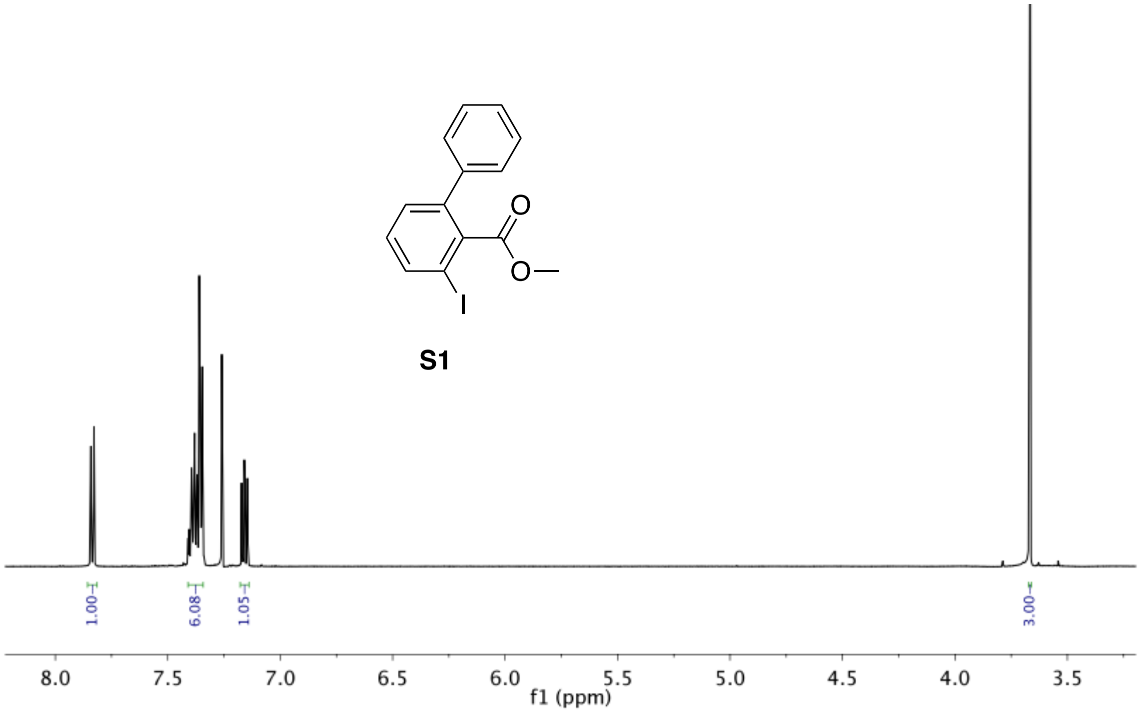
f1 (ppm)



7


O
O
I
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6.08
1.05
3.00
8.0
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6.5
6.0
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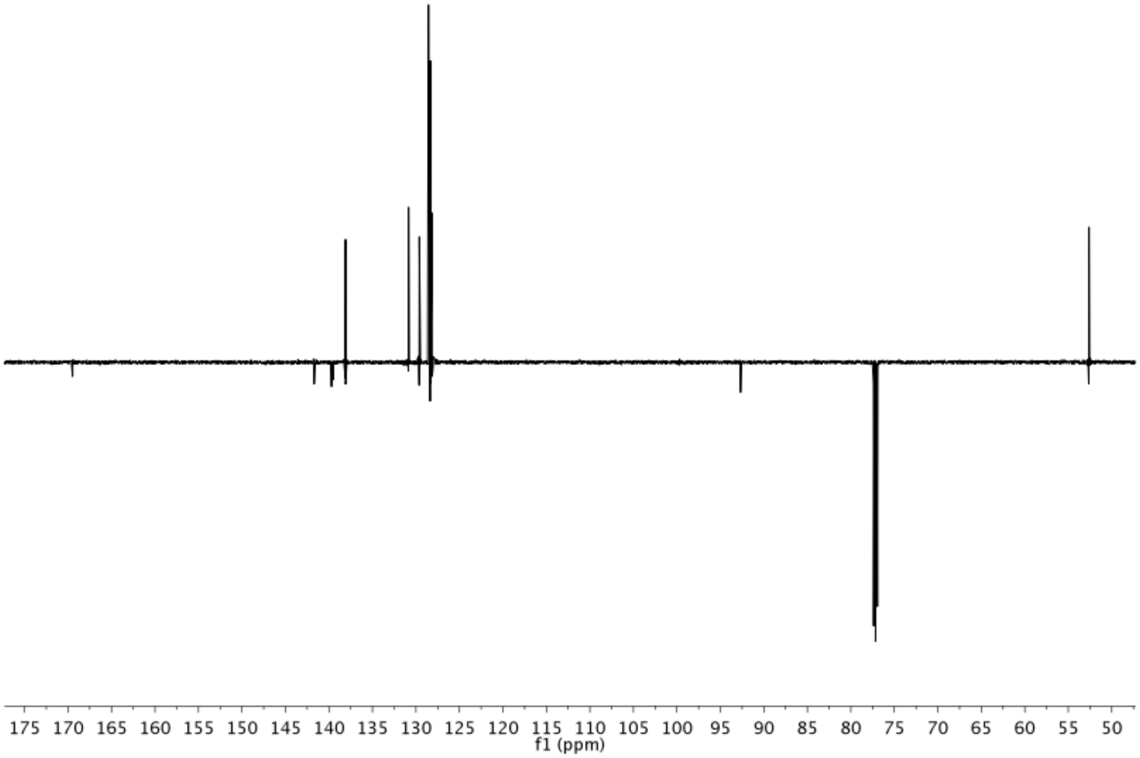
5.0
4.5
4.0
3.5
f1 (ppm)

S1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170
165
160
155
150
145
140
135
130
125
120
115
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105
100
95
90
85
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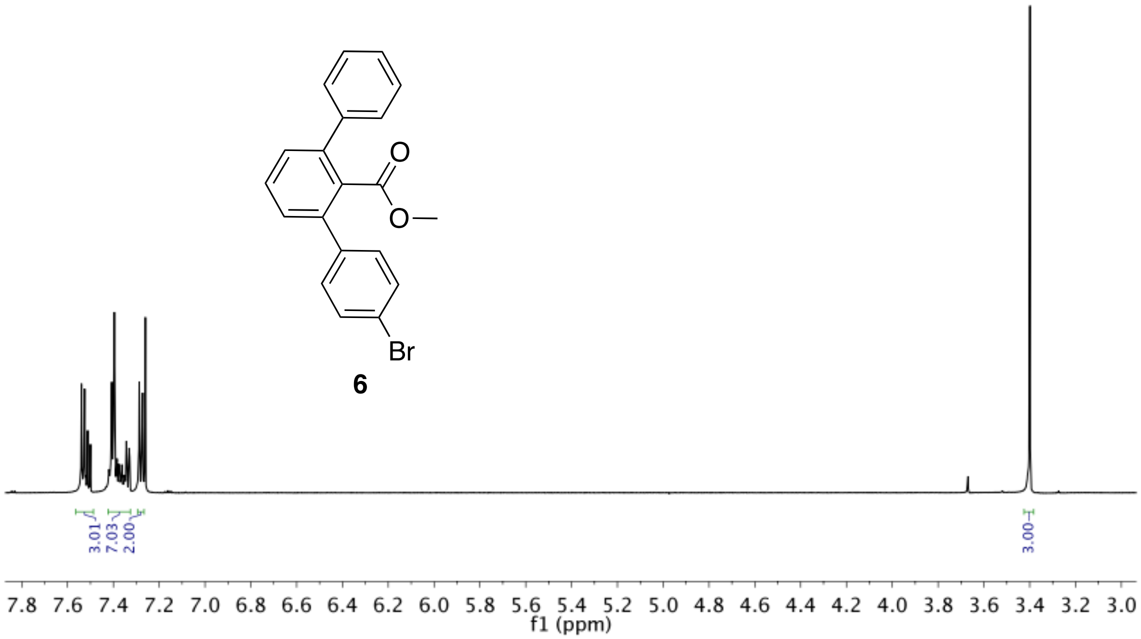
65
60
55
50
f1 (ppm)


6
O
O
Br
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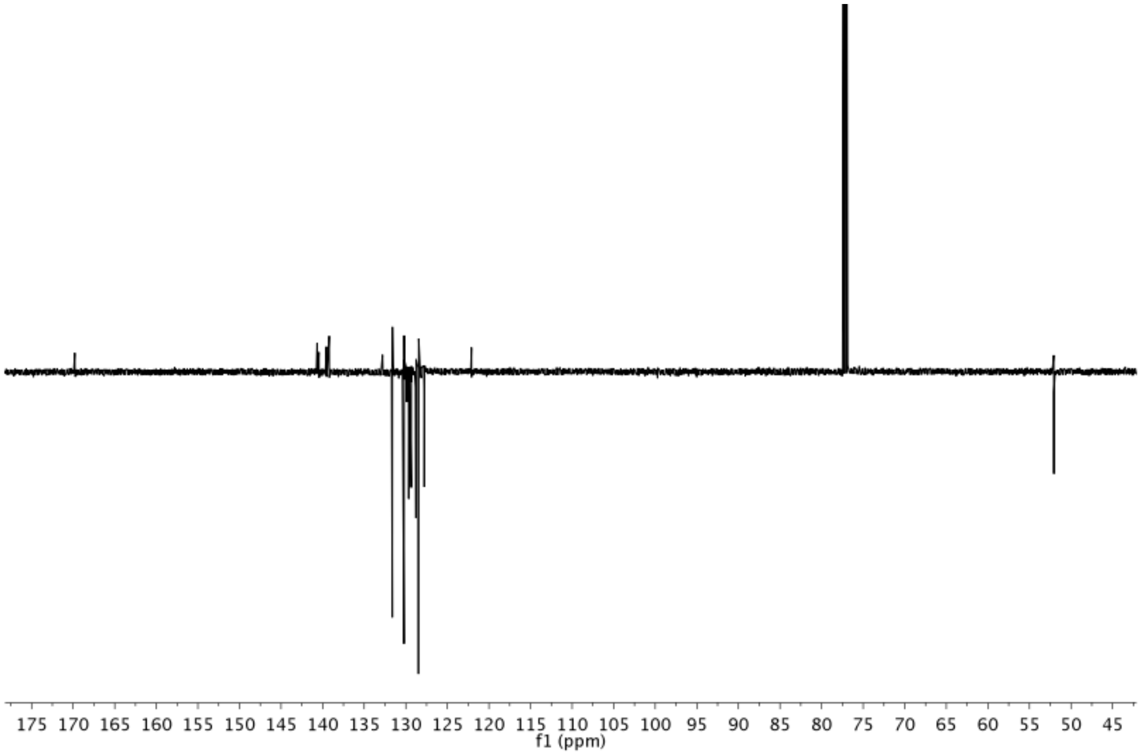
7.03
2.00
3.00
7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0
f1 (ppm)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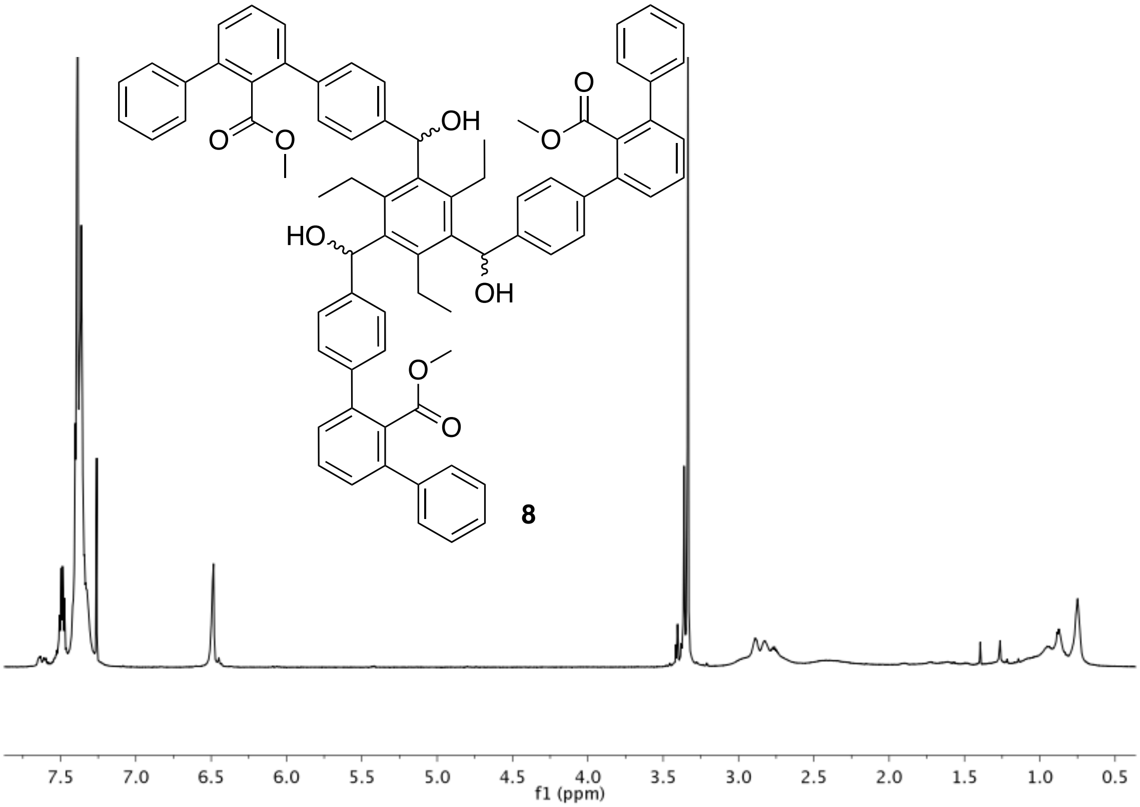
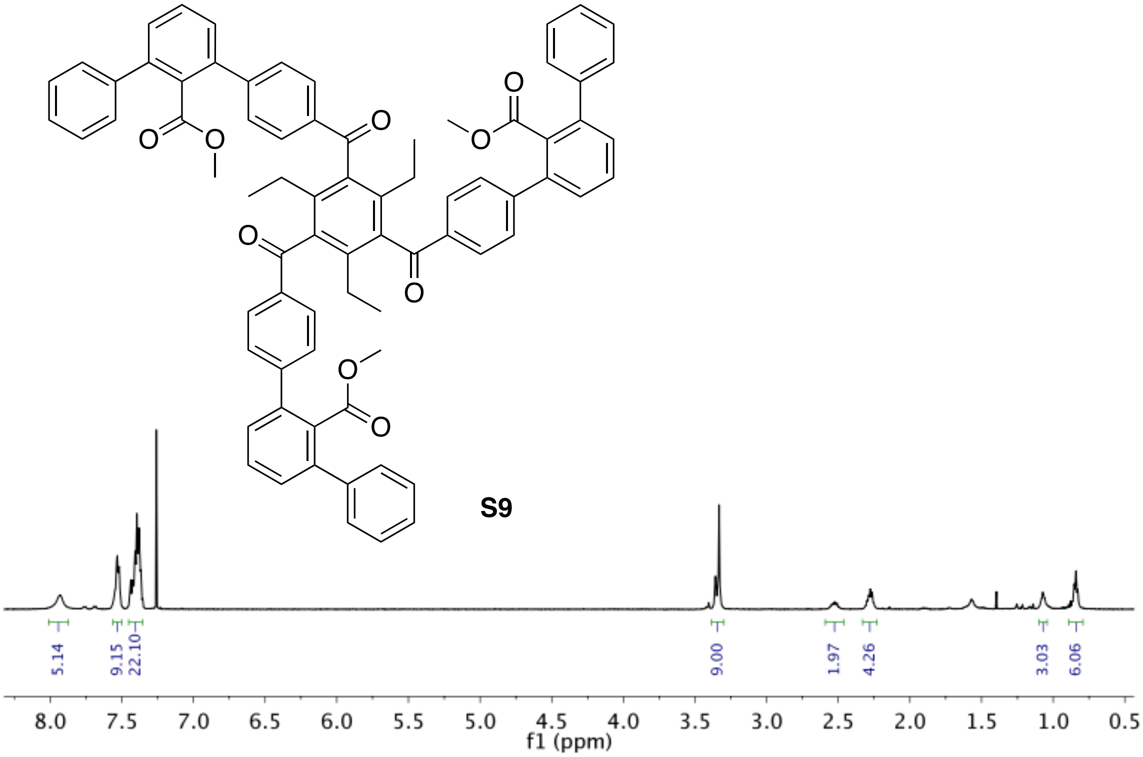
f1 (ppm)



8


O
O
O
O
O
O
O
O
O
S9
5.14
9.15
22.10
9.00
1.97
4.26
3.03
6.06
8.0
7.5
7.0
6.5
6.0
5.5
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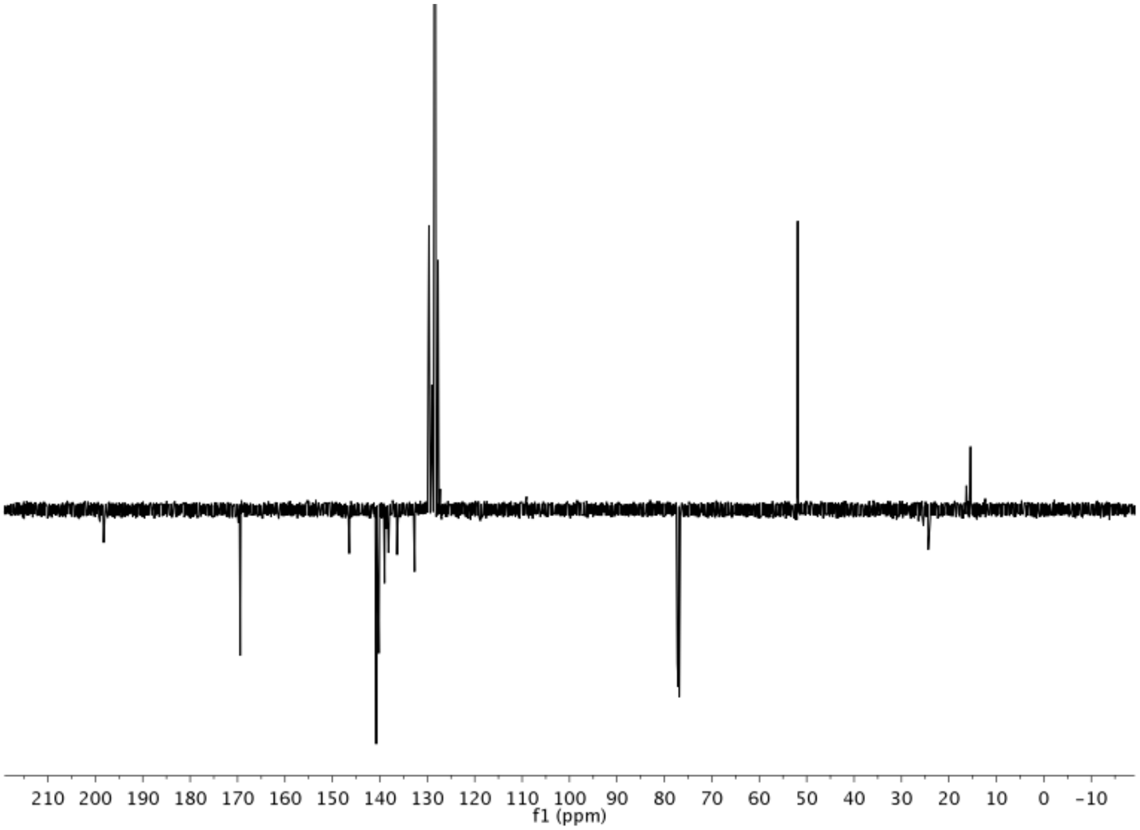
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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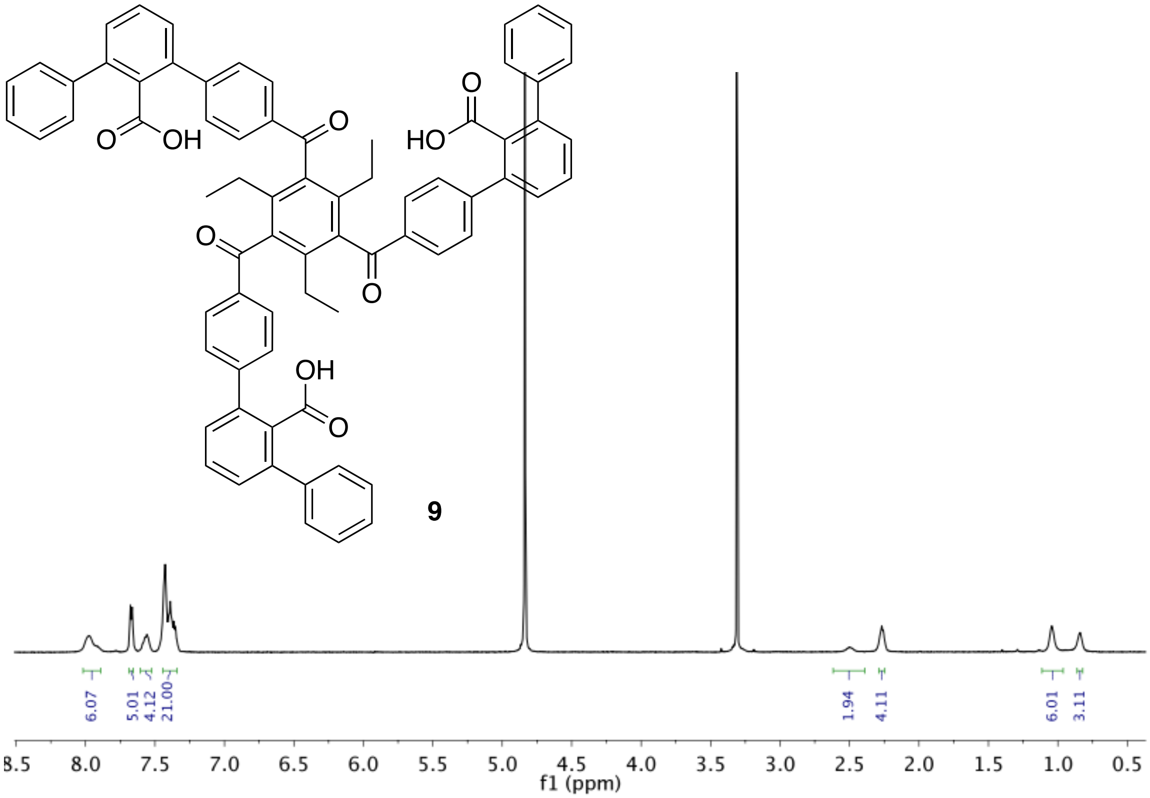
f1 (ppm)


O
OH
O
HO
O
O
O
OH
O
9
6.07
5.01
4.12
21.00
1.94
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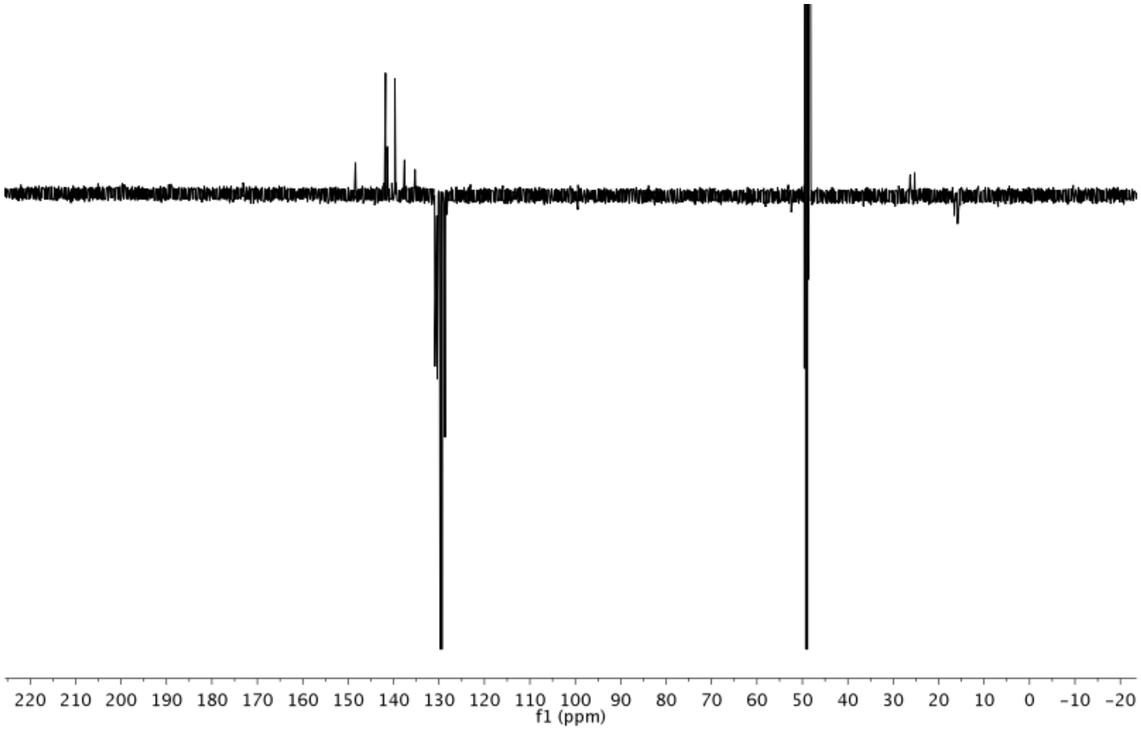
6.01
3.11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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180
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90
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50
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30
20
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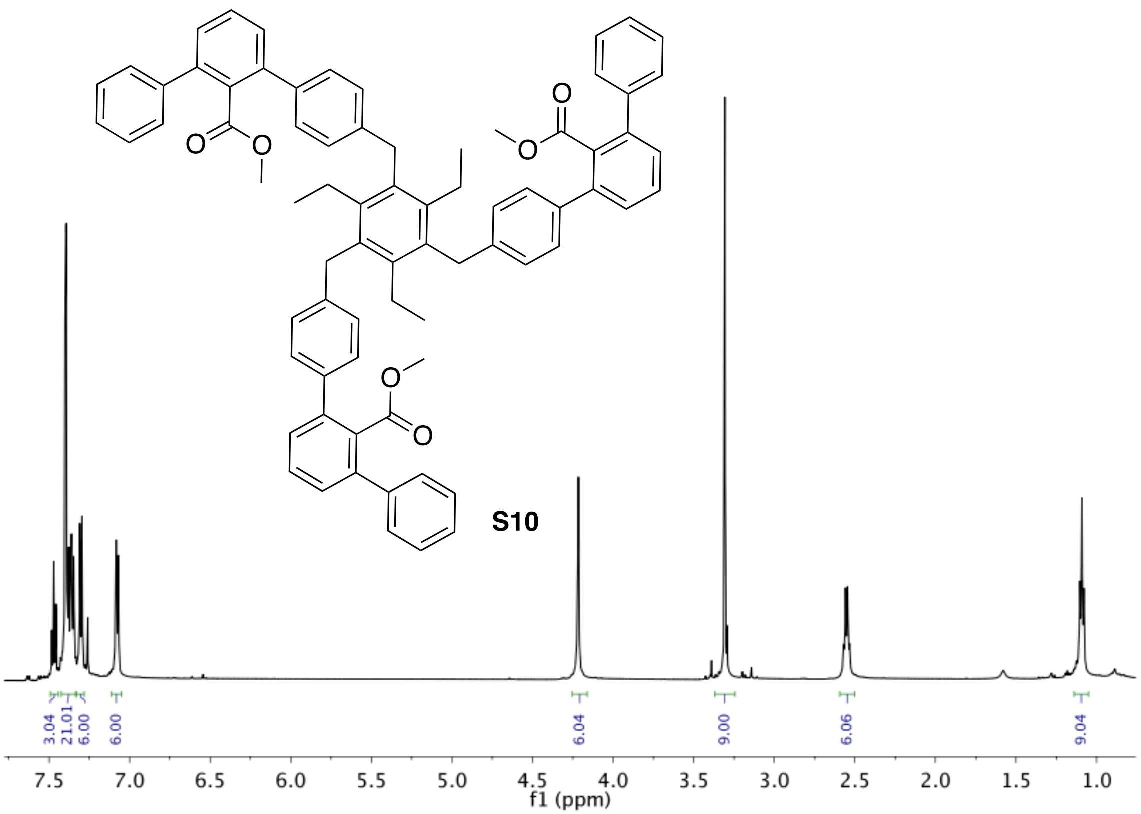
0
-10
-20
f1 (ppm)


S10
O
O
O
O
O
O
3.04
21.01
6.00
6.00
6.04
9.00
6.06
9.04
7.5
7.0
6.5
6.0
5.5
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4.5
4.0
3.5
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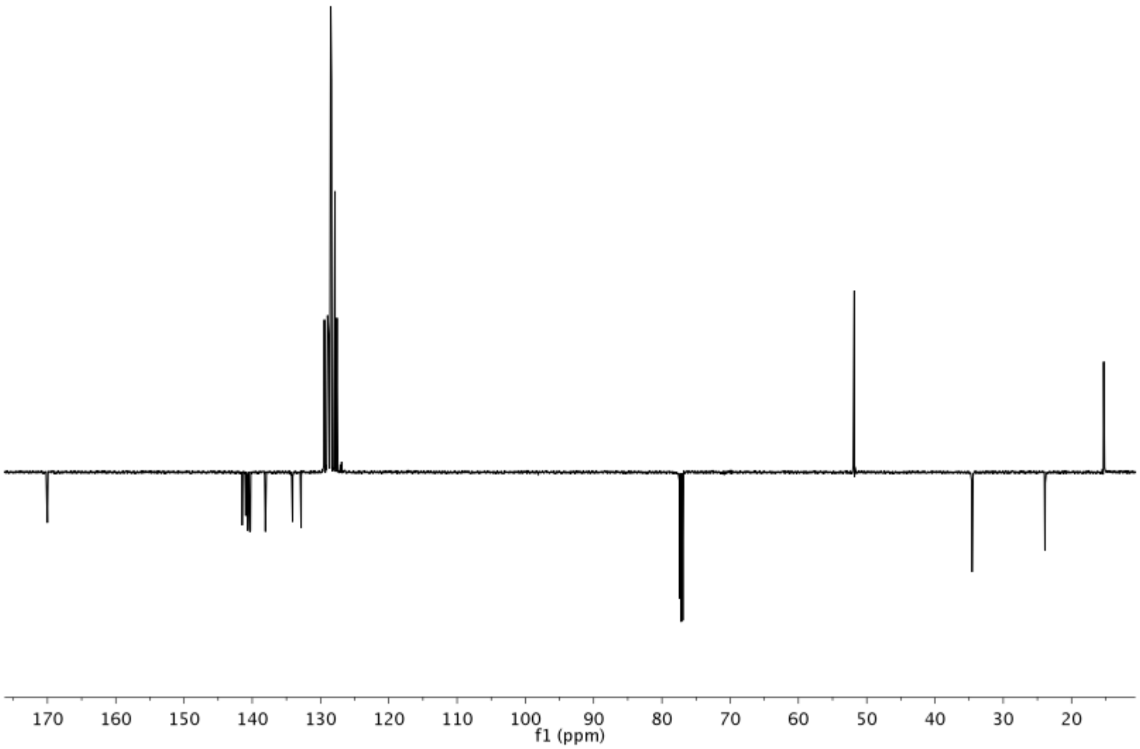
2.5
2.0
1.5
1.0
f1 (ppm)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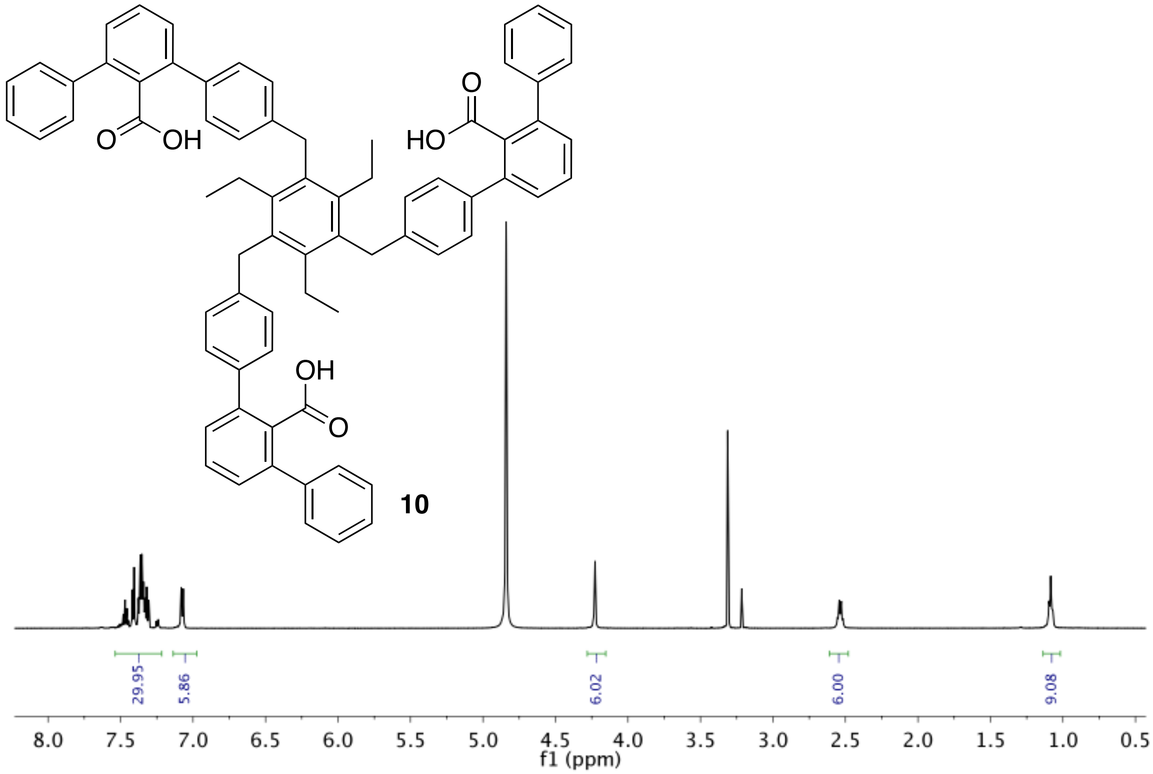
f1 (ppm)


O
OH
HO
O
OH
O
29.95
5.86
6.02
6.00
9.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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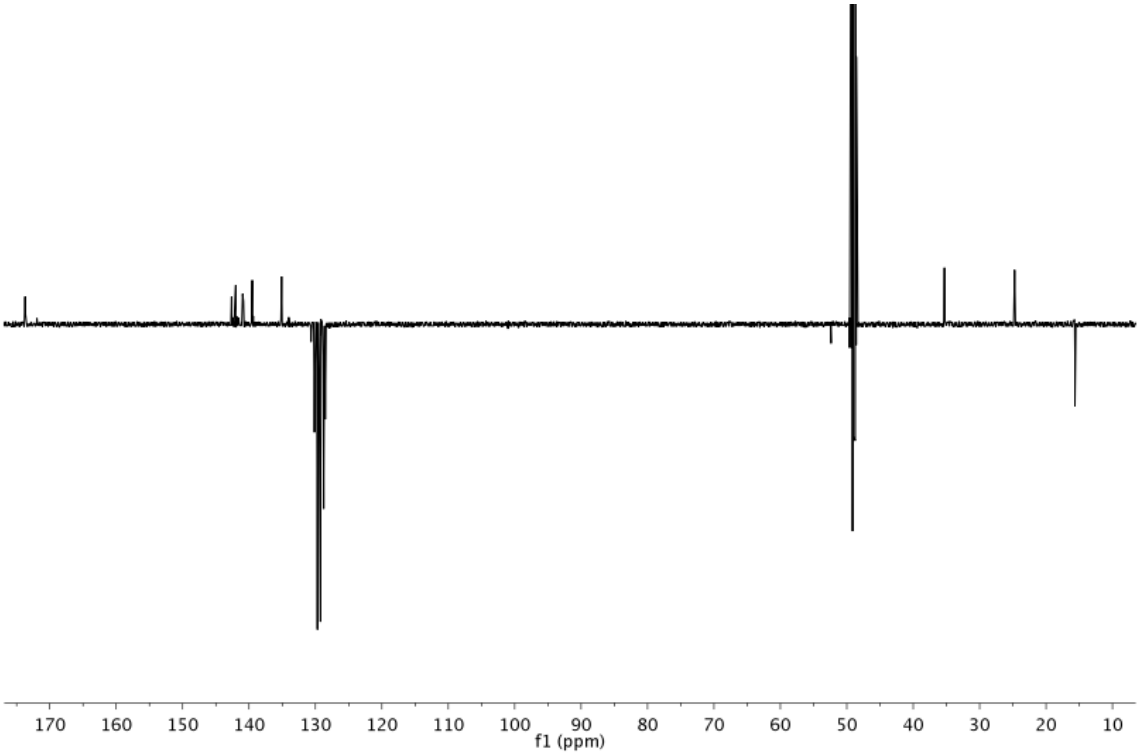
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

10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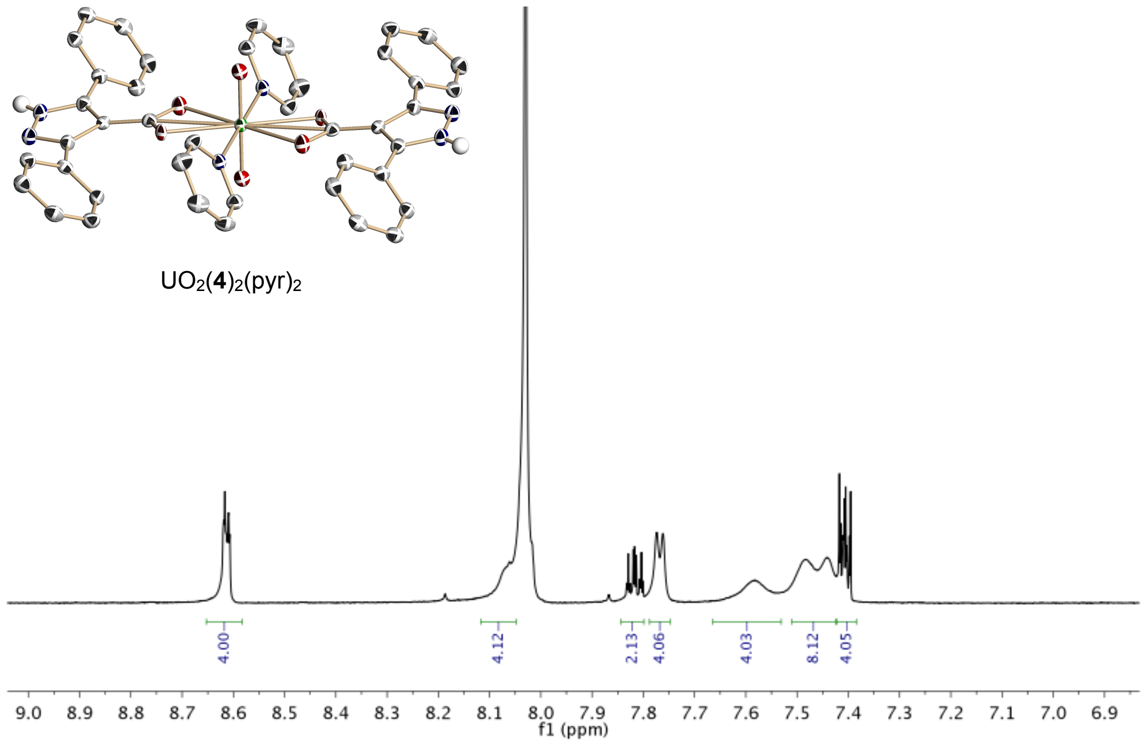
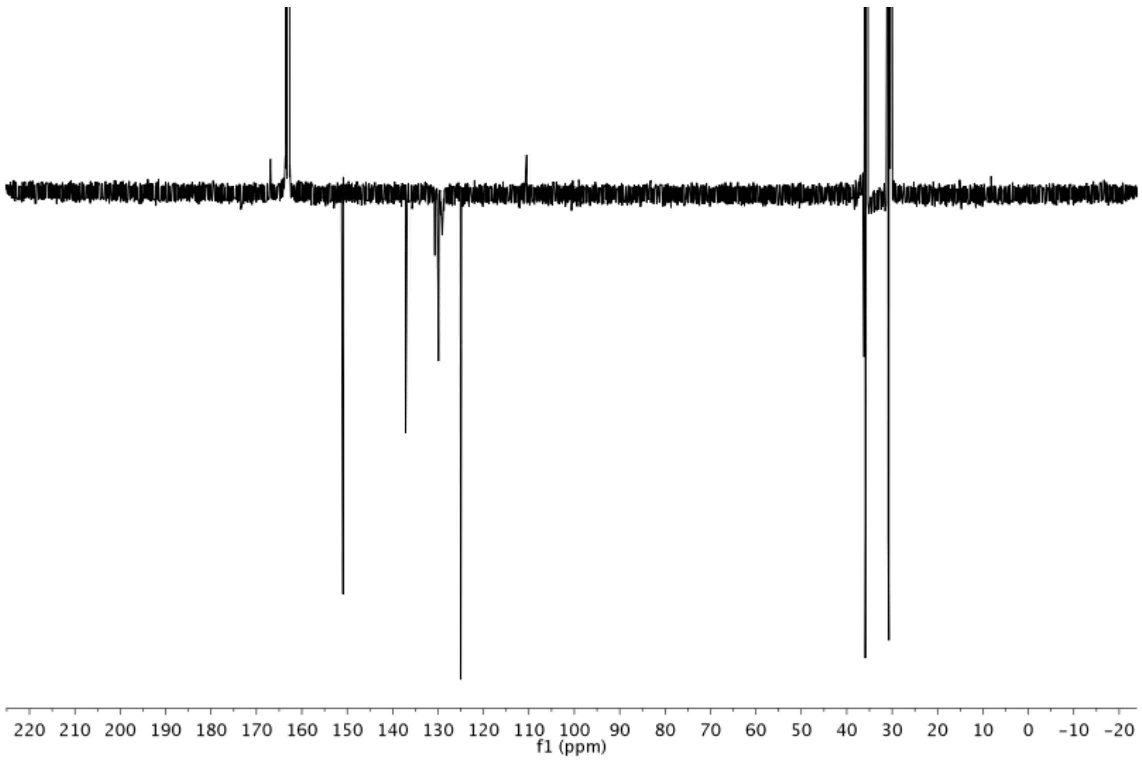
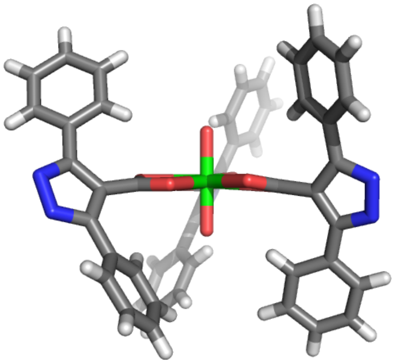
f1 (ppm)

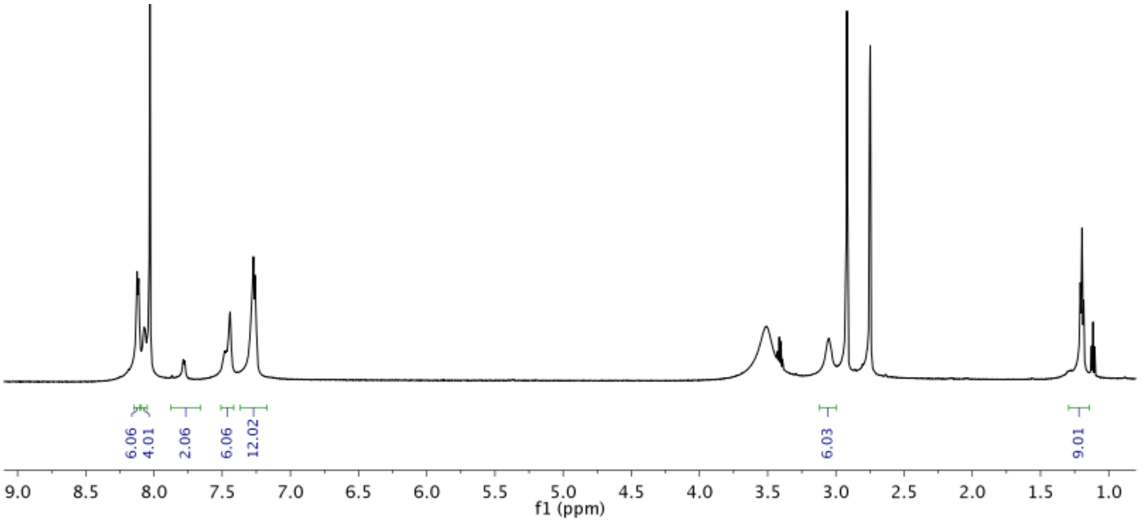


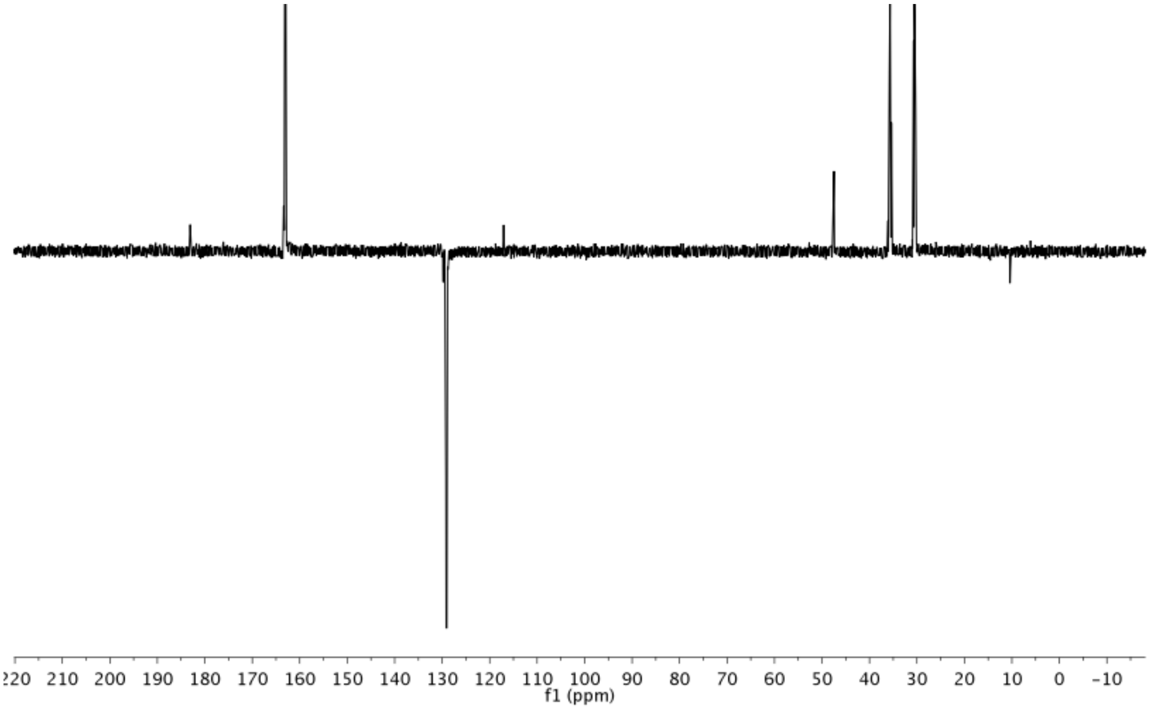
$UO_2(\mathbf{4})_2(pyr)_2$

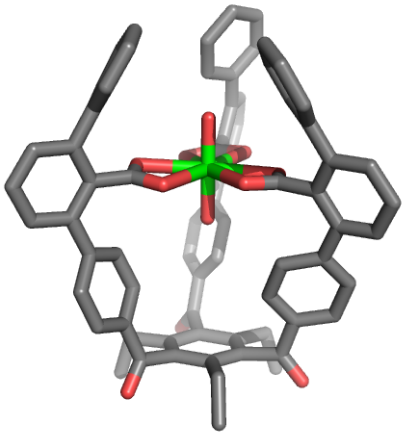




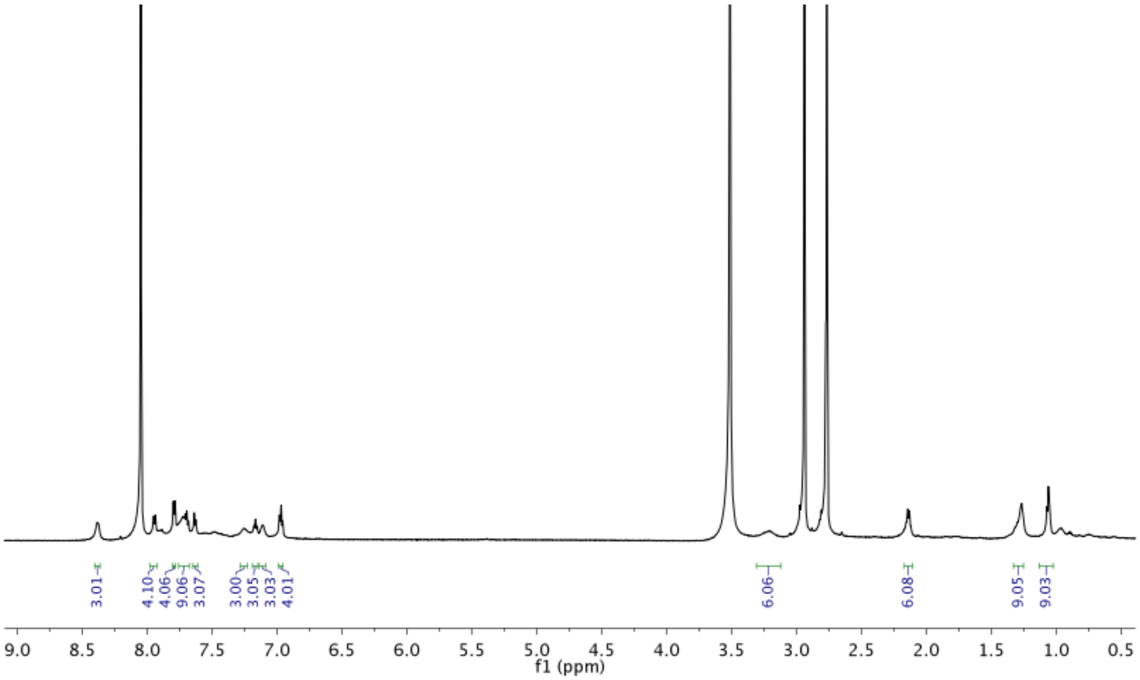
$UO_2(\mathbf{4})_3$•Et_3NH







UO_2(**9**)•Et_3NH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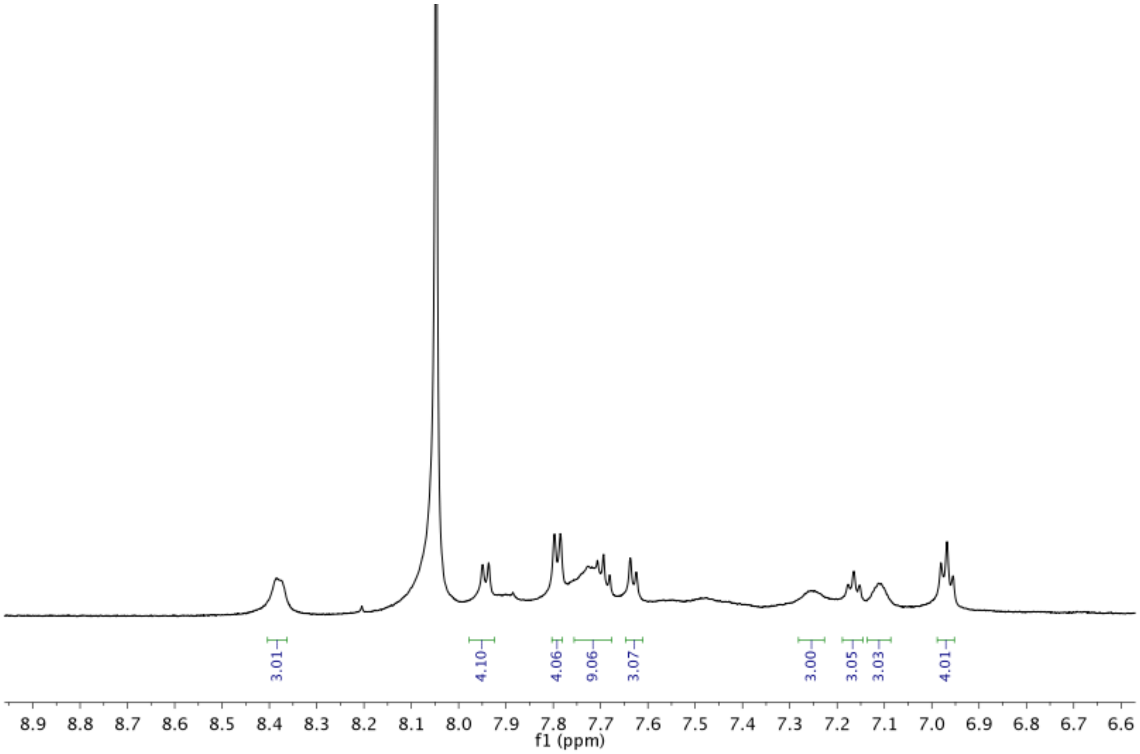
4.10
4.06
9.06
3.07
3.00
3.05
3.03
4.01
6.06
6.08
9.05
9.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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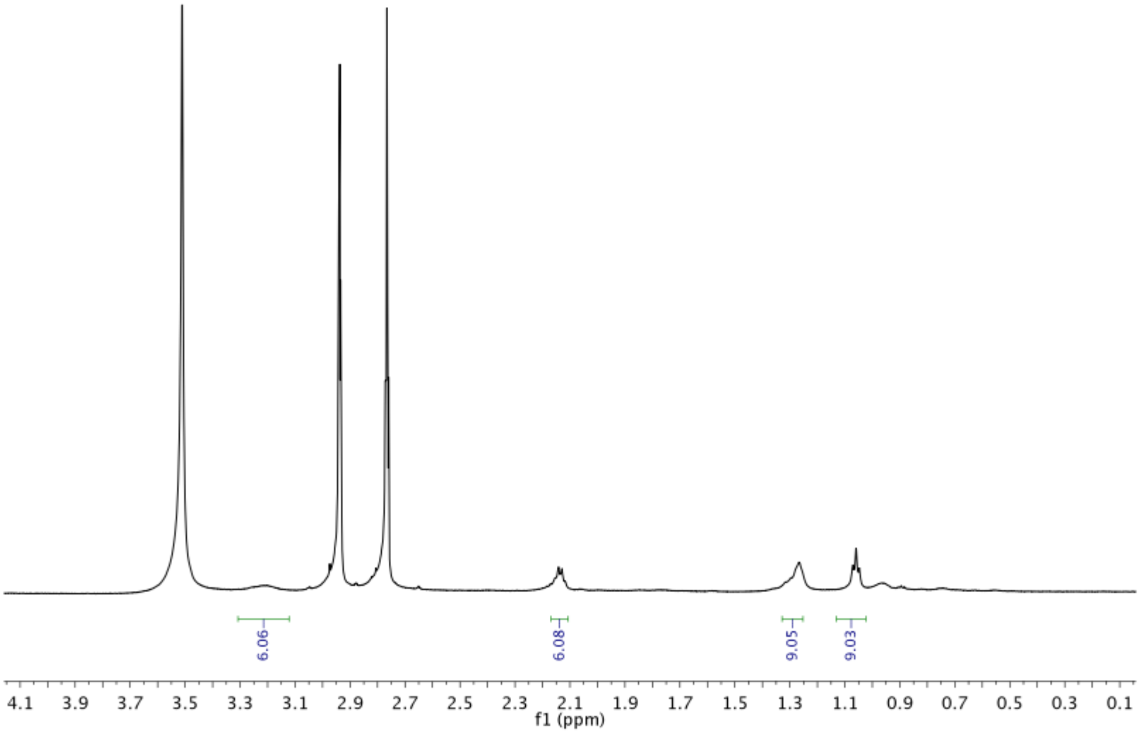
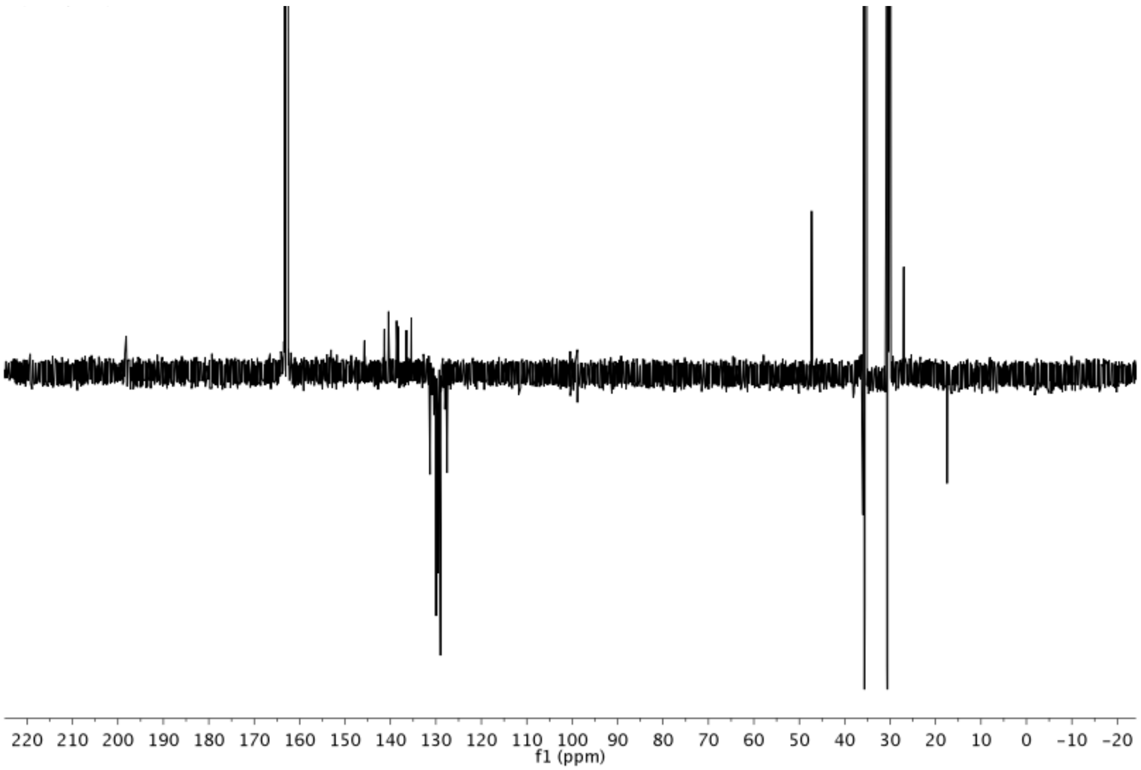
4.10
4.06
9.06
3.07
3.00
3.05
3.03
4.01
8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6
f1 (ppm)


6.06
6.08
9.05
9.03
4.1 3.9 3.7 3.5 3.3 3.1 2.9 2.7 2.5 2.3 2.1 1.9 1.7 1.5 1.3 1.1 0.9 0.7 0.5 0.3 0.1
f1 (ppm)


220
210
200
190
180
170
160
150
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130
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110
100
90
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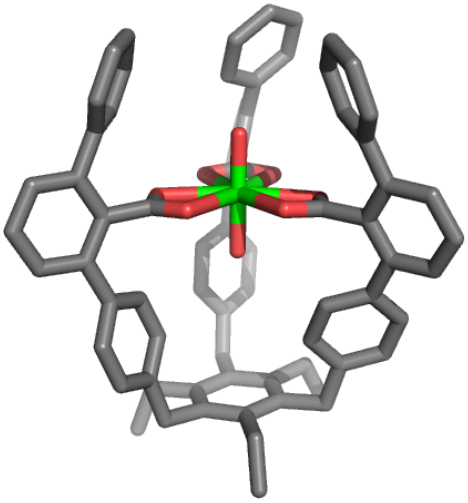
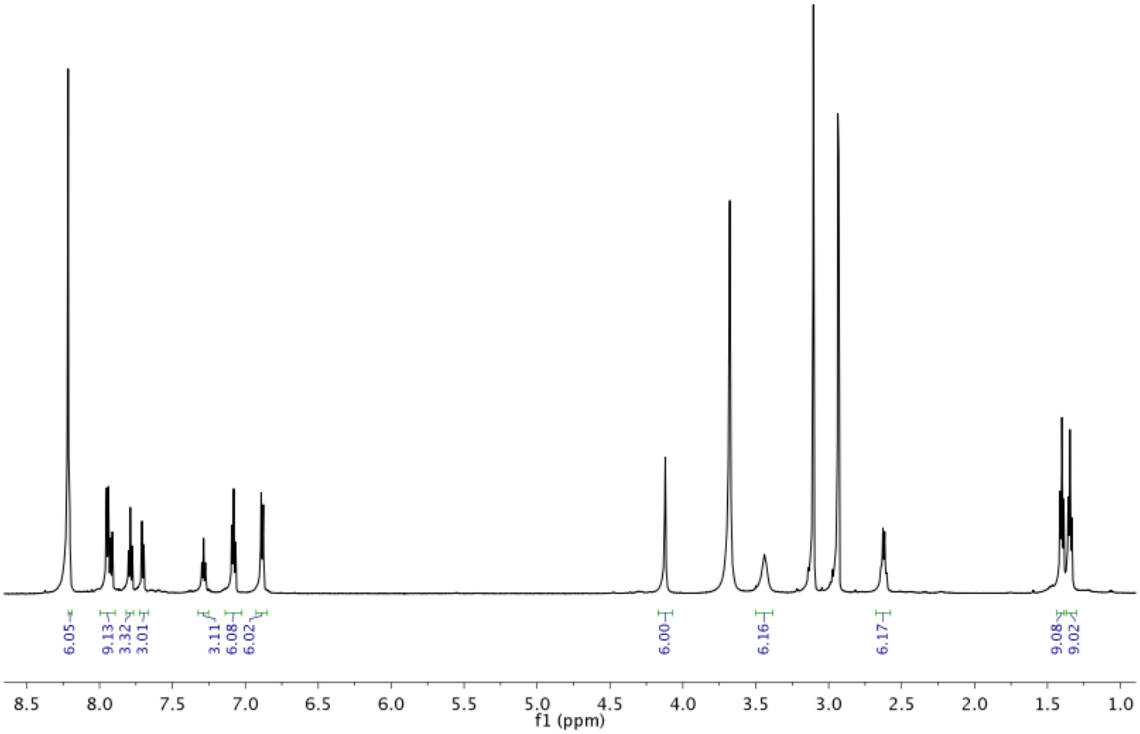
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)



UO_2(**10**)•Et_3NH


6.05
9.13
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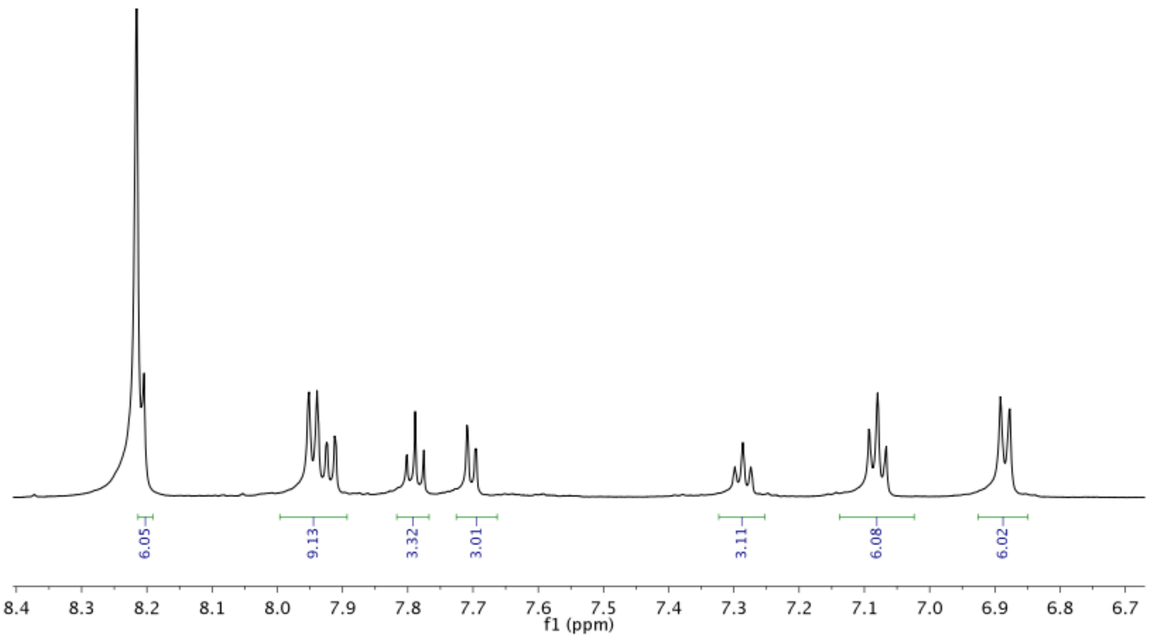
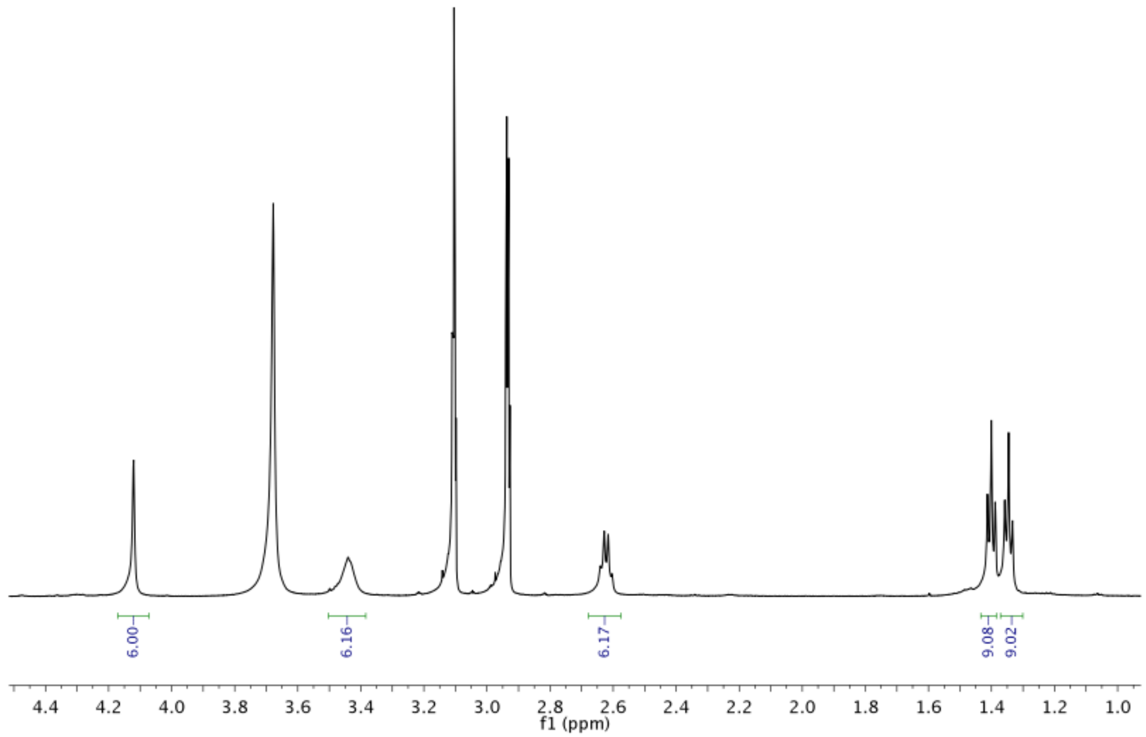
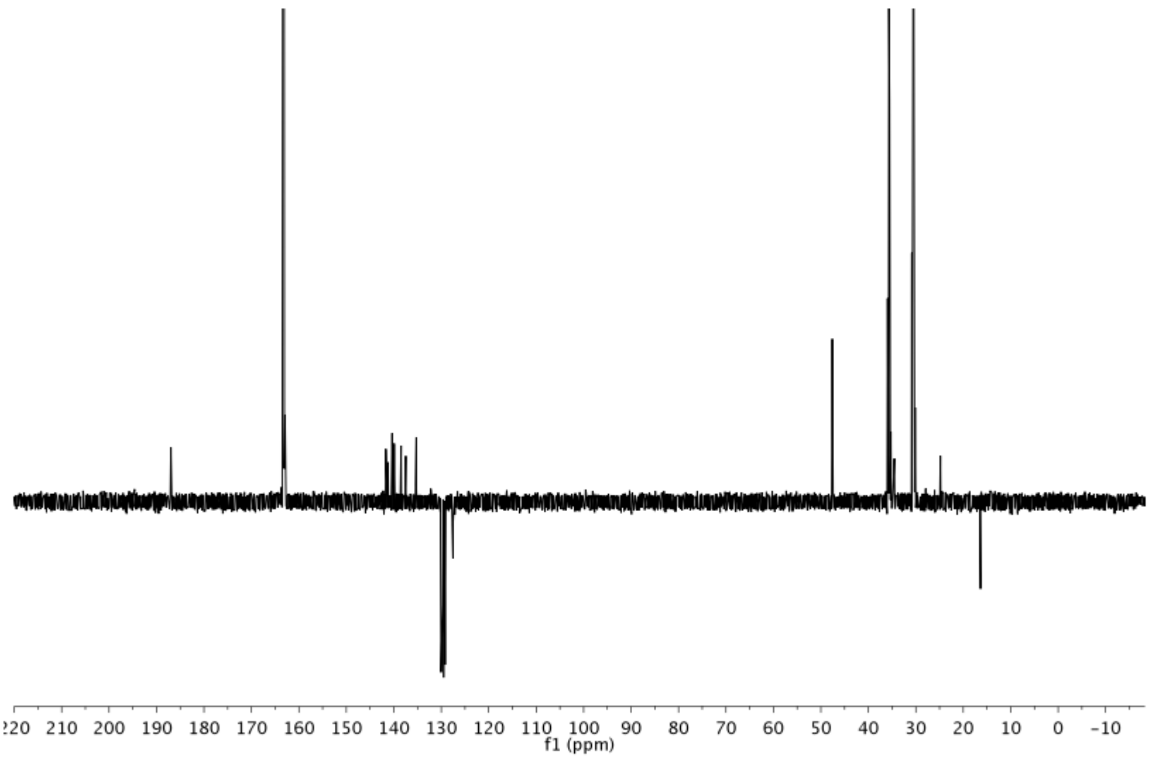
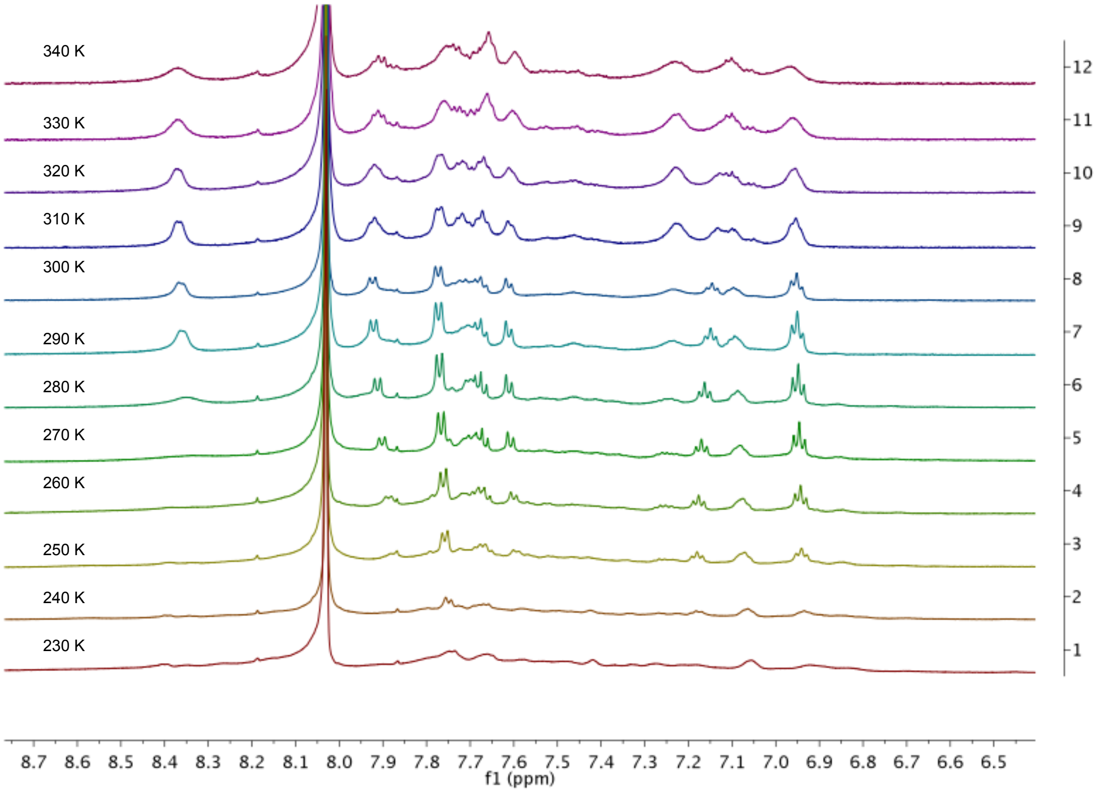
6.02
6.00
6.16
6.17
9.08
9.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

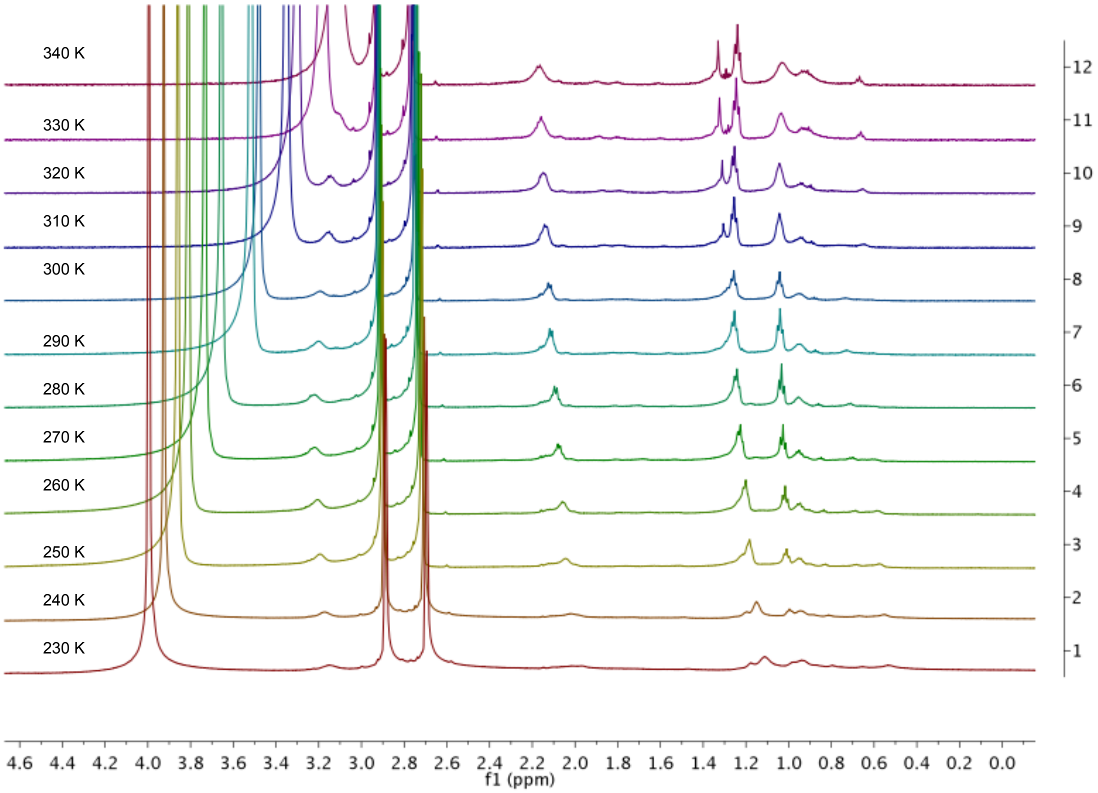

6.05
9.13
3.32
3.01
3.11
6.08
6.02
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
f1 (ppm)


6.00
6.16
6.17
9.08
9.02
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
f1 (ppm)

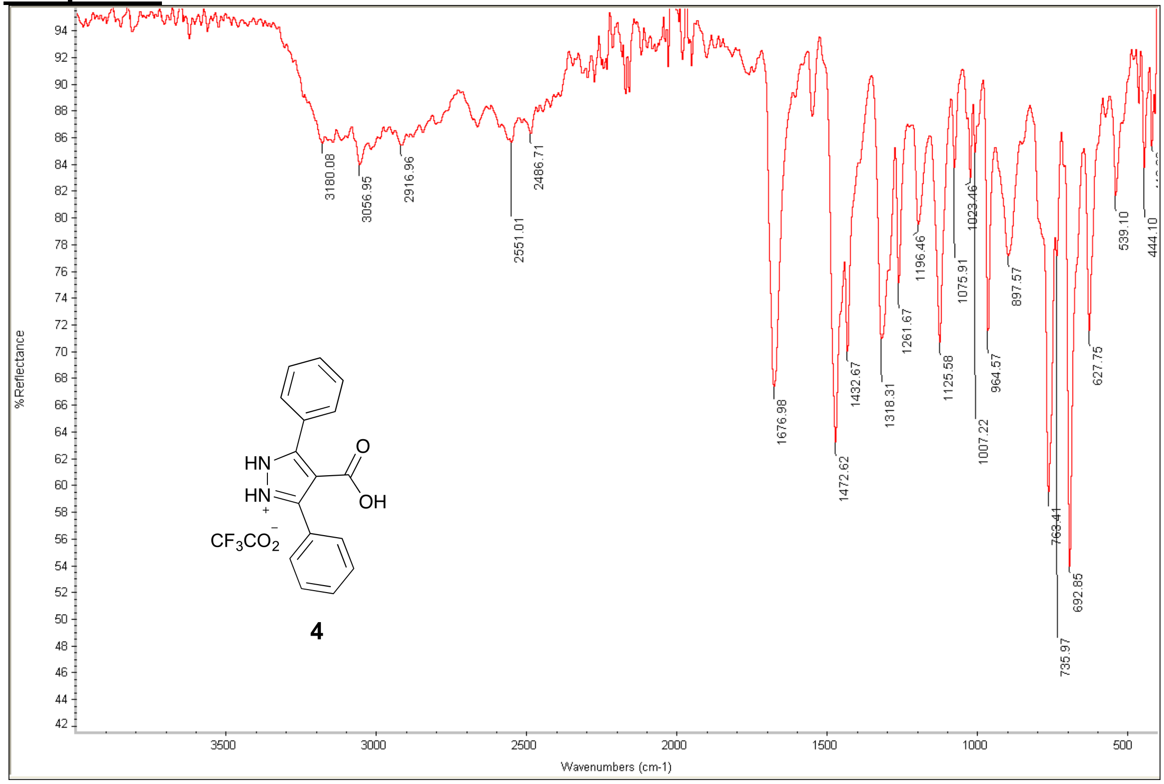

220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
–10
f1 (ppm)

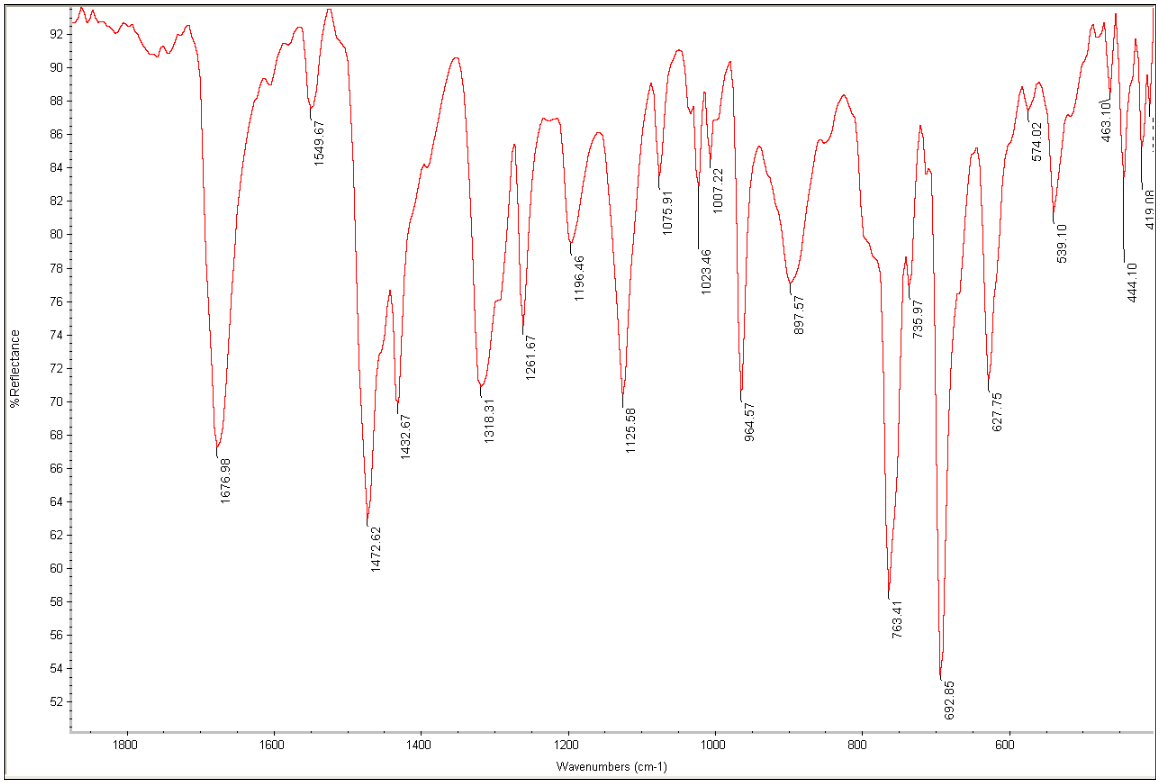
Variable Temperature ^{1}H NMR for UO_2(9)•Et_3NH

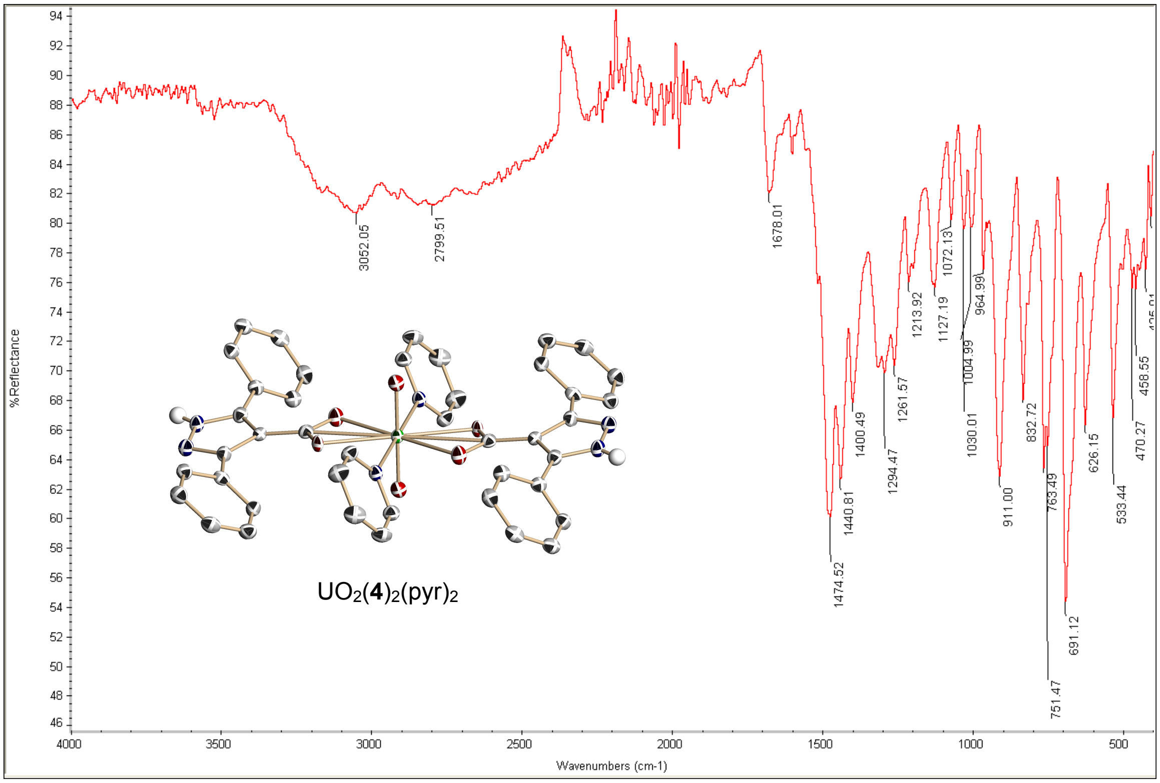


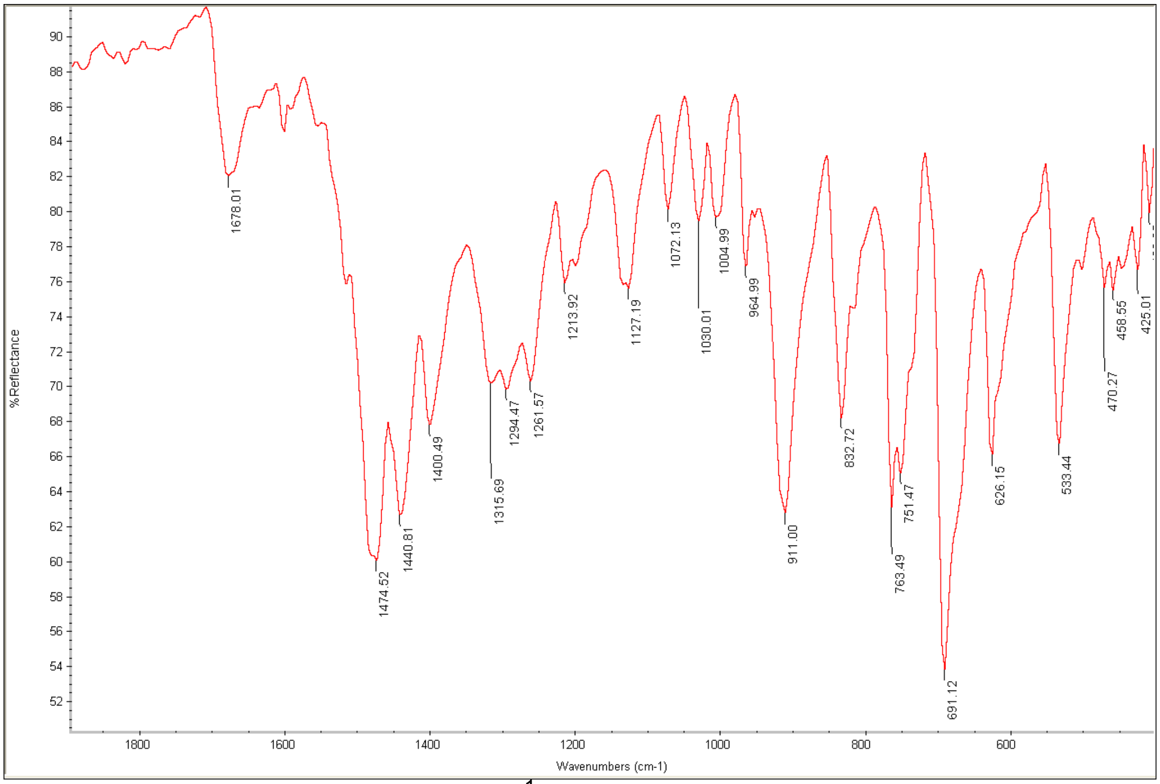

340 K
330 K
320 K
310 K
300 K
290 K
280 K
270 K
260 K
250 K
240 K
230 K
12
11
10
9
8
7
6
5
4
3
2
1
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
f1 (ppm)

IR Spectra


94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
46
44
42
%Reflectance
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
3180.08
3056.95
2916.96
2551.01
2486.71
1676.98
1472.62
1432.67
1318.31
1261.67
1196.46
1125.58
1075.91
1023.46
1007.22
964.57
897.57
763.41
735.97
692.85
627.75
539.10
444.10
HN
HN
+
$CF_3CO_2^-$
O
OH
4

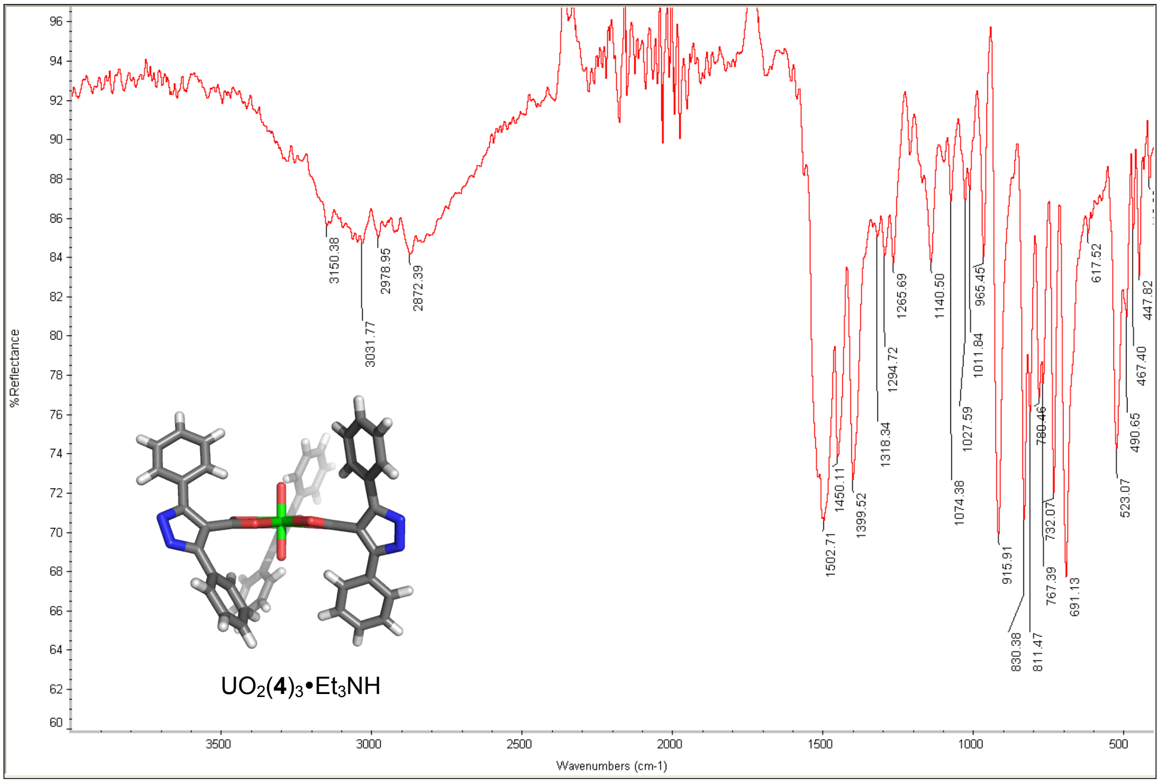

92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
%Reflectance
1800
1600
1400
1200
1000
800
600
Wavenumbers (cm-1)
1676.98
1549.67
1472.62
1432.67
1318.31
1261.67
1196.46
1125.58
1075.91
1023.46
1007.22
964.57
897.57
763.41
735.97
692.85
627.75
574.02
539.10
463.10
444.10
419.08

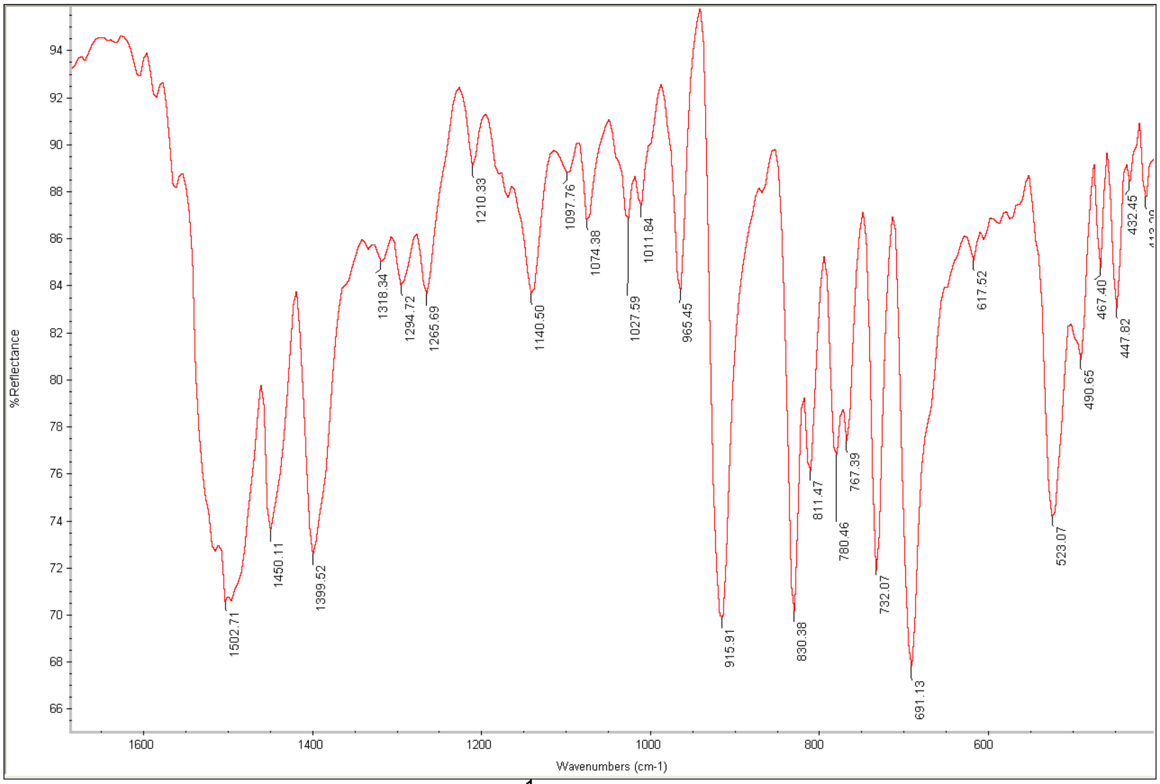




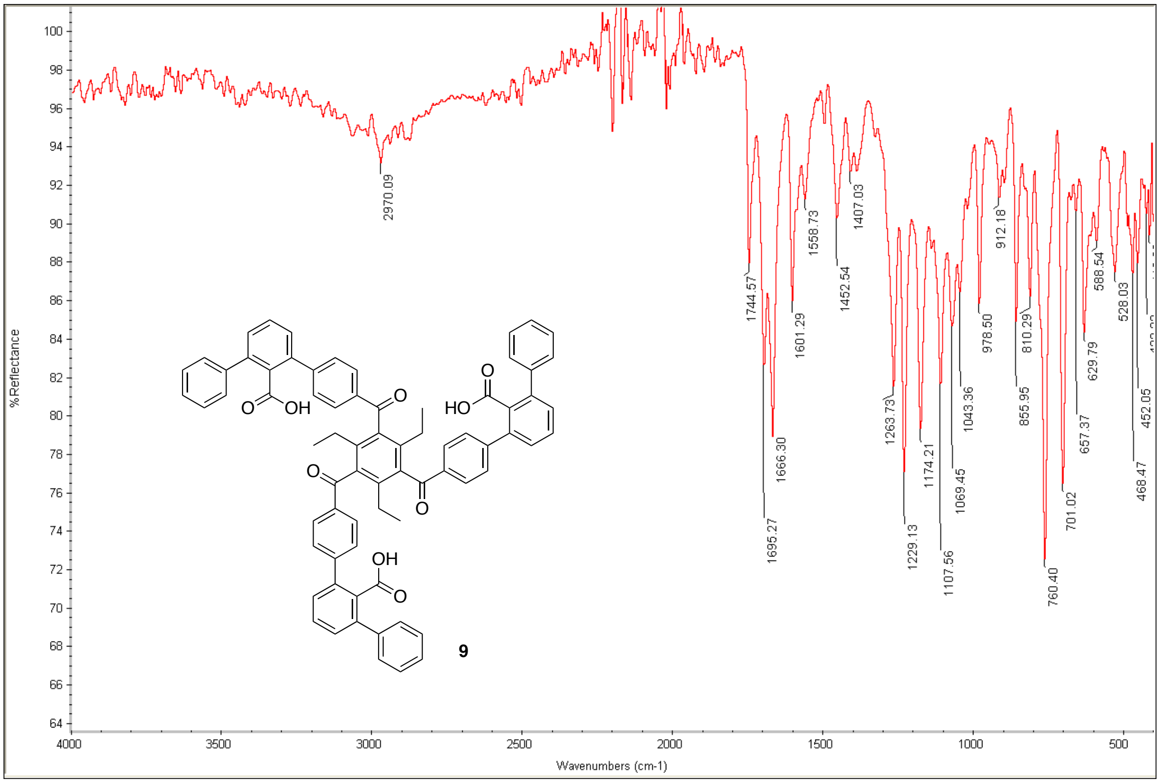
UO$_2$ asymmetric stretch (v_3): 911 cm^{-1}

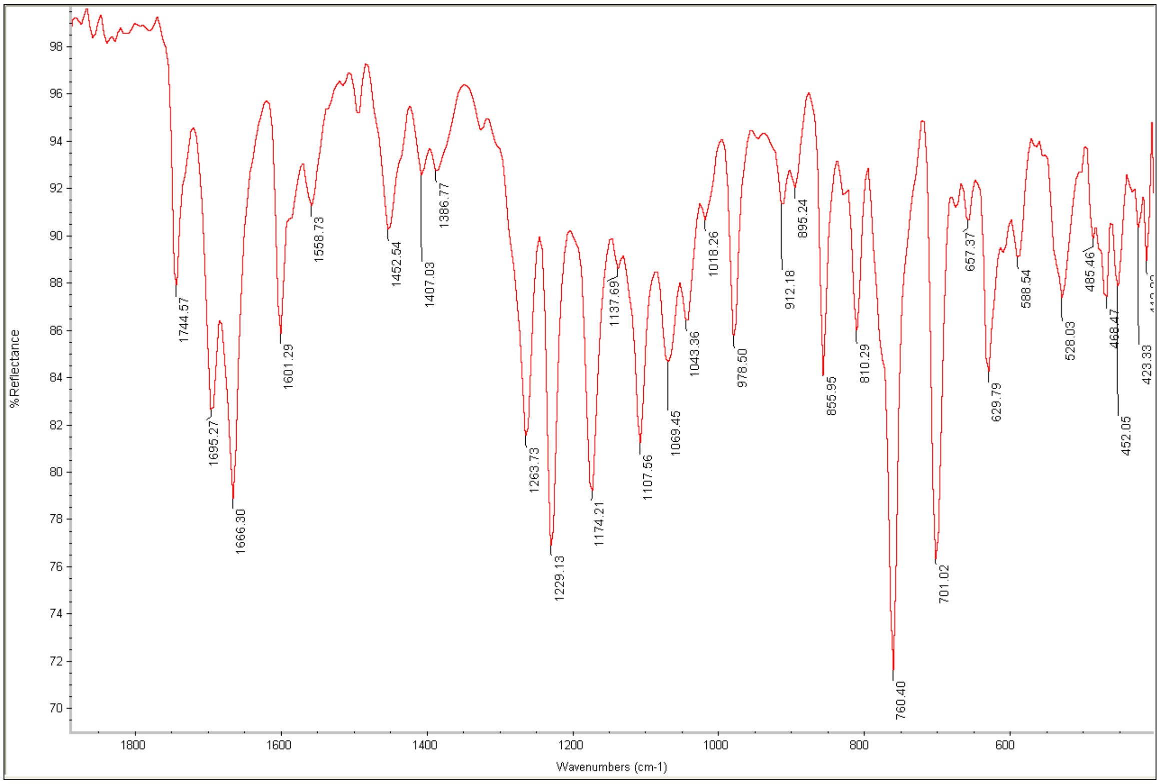
Electronic Supplementary Material (ESI) for Chemical Communications

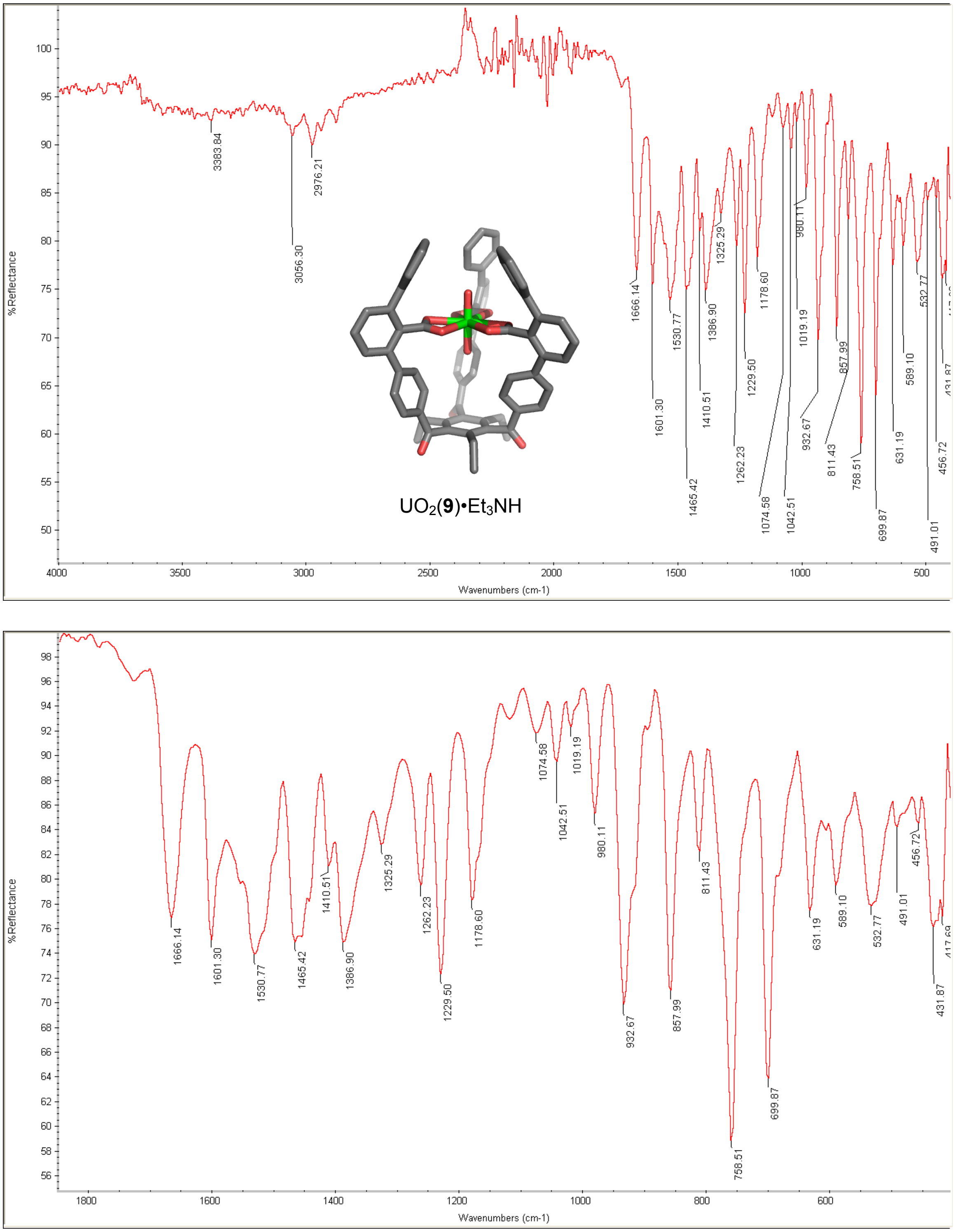




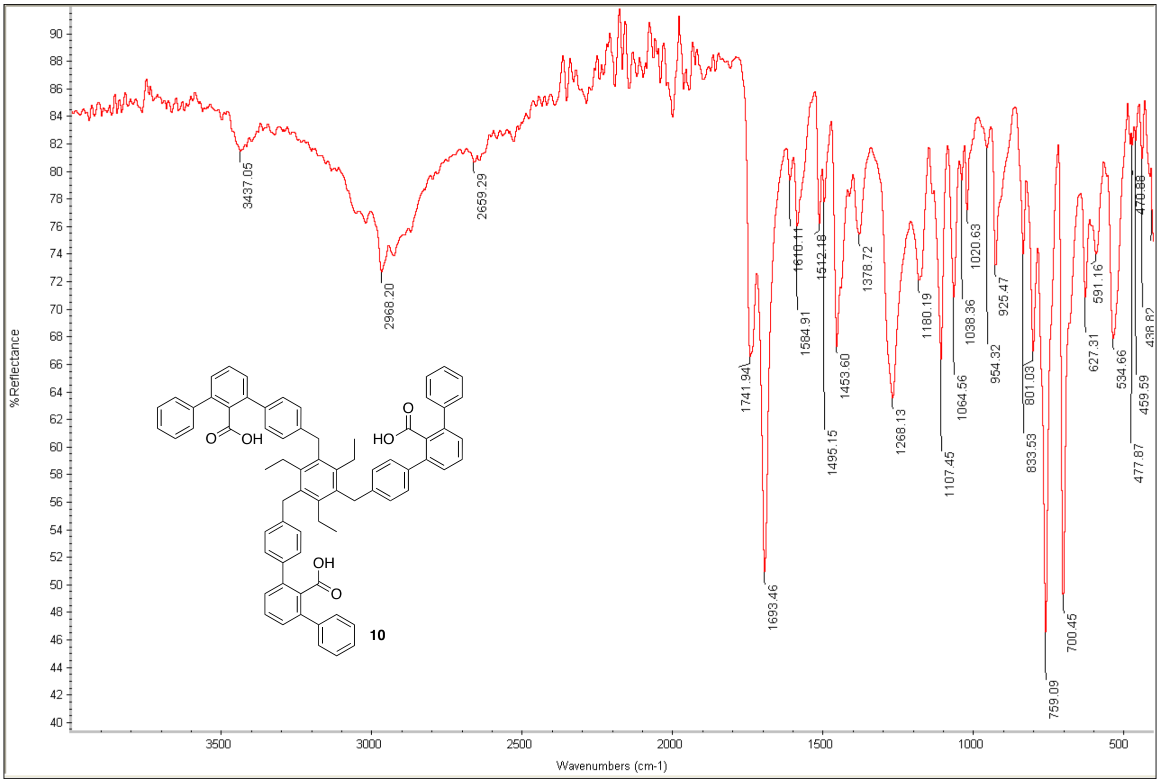
UO$_2$ asymmetric stretch (v_3): 915 cm^{-1}

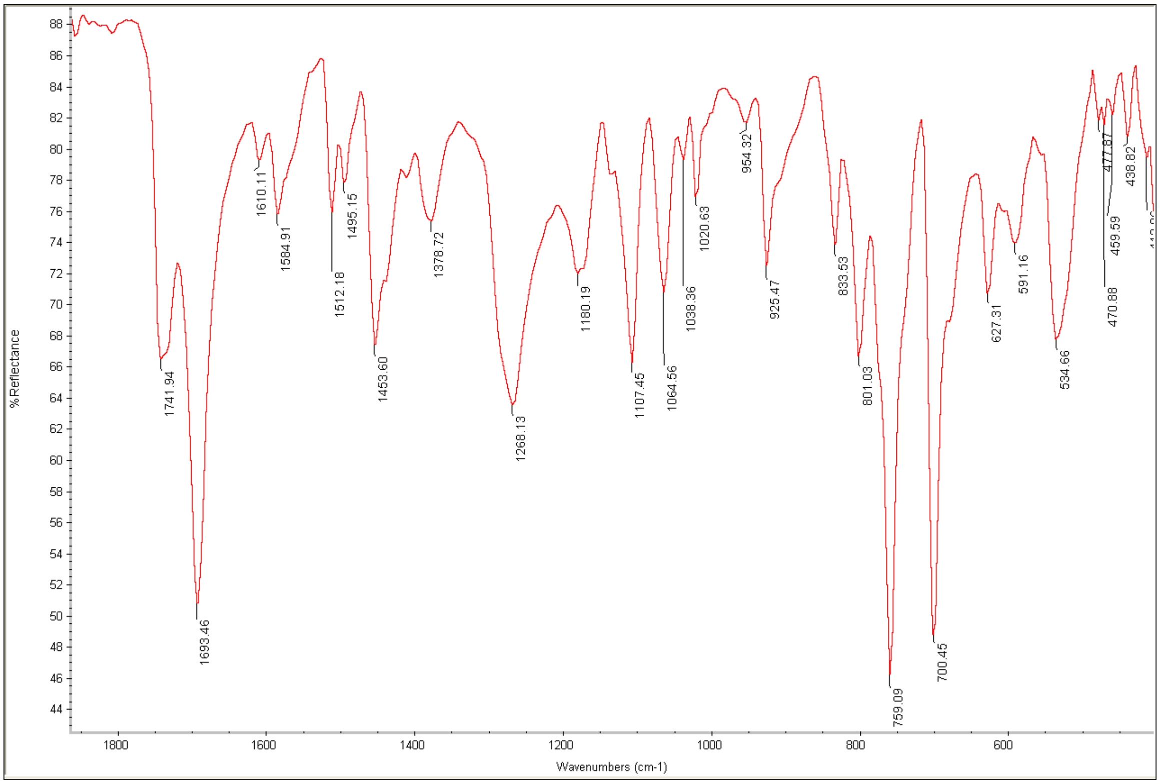

%Reflectance
Wavenumbers (cm-1)
100
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
4000
3500
3000
2500
2000
1500
1000
500
2970.09
1744.57
1695.27
1666.30
1601.29
1558.73
1452.54
1407.03
1263.73
1229.13
1174.21
1107.56
1069.45
1043.36
978.50
912.18
855.95
810.29
760.40
701.02
657.37
629.79
588.54
528.03
468.47
452.05
O
OH
HO
OH
9


%Reflectance
Wavenumbers (cm-1)
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
1800
1600
1400
1200
1000
800
600
1744.57
1695.27
1666.30
1601.29
1558.73
1452.54
1407.03
1386.77
1263.73
1229.13
1174.21
1137.69
1107.56
1069.45
1043.36
1018.26
978.50
912.18
895.24
855.95
810.29
760.40
701.02
657.37
629.79
588.54
528.03
485.46
468.47
452.05
423.33



(a) UO_2 asymmetric stretch (ν_3): 932 cm^{-1} (b) UO_2 symmetric stretch (ν_1): 857 cm^{-1}


%Reflectance
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
46
44
42
40
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
3437.05
2968.20
2659.29
1741.94
1693.46
1610.11
1584.91
1512.18
1495.15
1453.60
1378.72
1268.13
1180.19
1107.45
1064.56
1038.36
1020.63
954.32
925.47
833.53
801.03
759.09
700.45
627.31
591.16
534.66
477.87
470.88
459.59
438.82
O
OH
HO
O
OH
O
10


%Reflectance
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
46
44
1800
1600
1400
1200
1000
800
600
Wavenumbers (cm-1)
1741.94
1693.46
1610.11
1584.91
1512.18
1495.15
1453.60
1378.72
1268.13
1180.19
1107.45
1064.56
1038.36
1020.63
954.32
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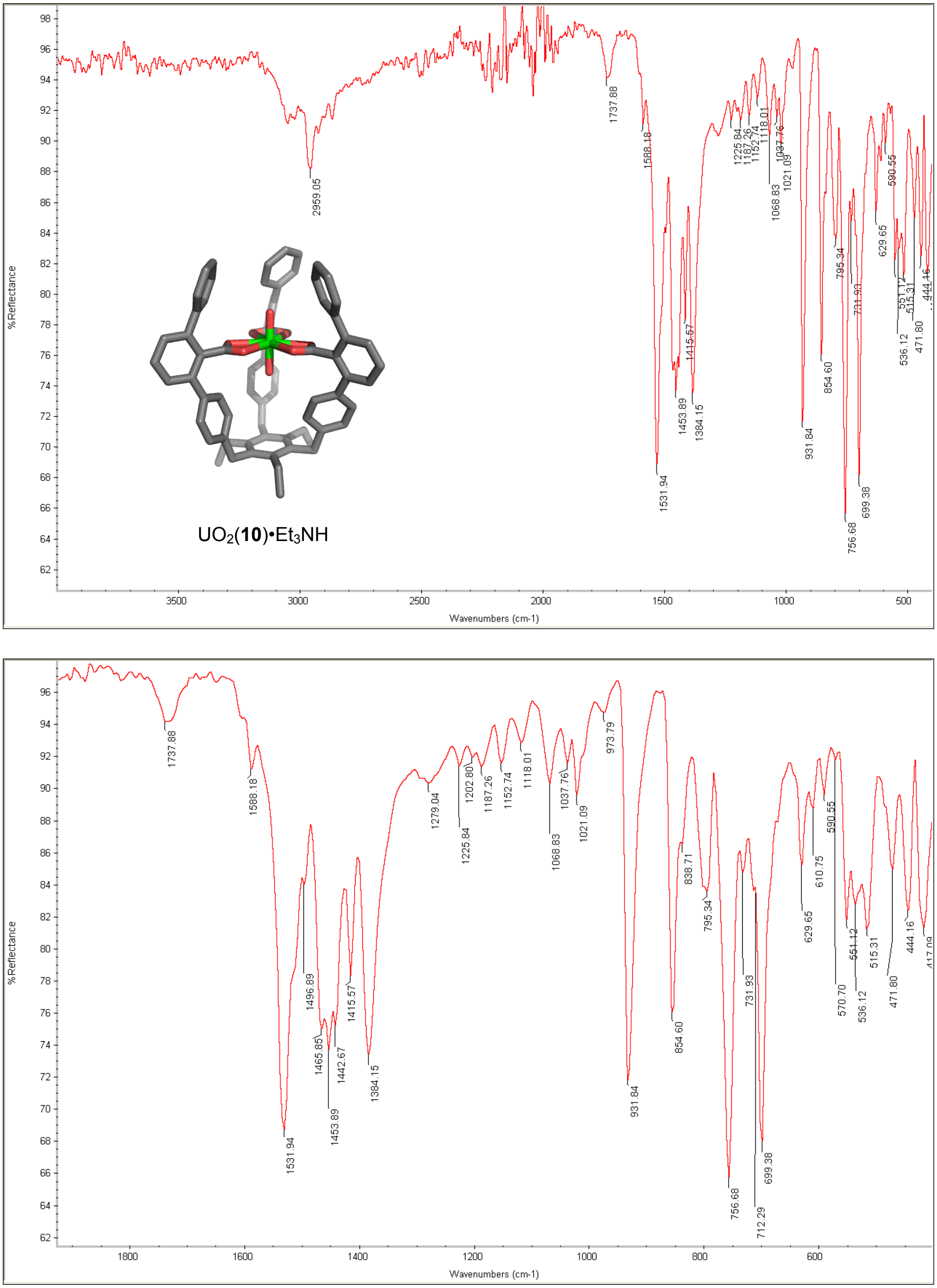
627.31
591.16
534.66
477.87
470.88
459.59
438.82



(a) UO$_2$ asymmetric stretch (ν_3): 931 cm^{-1} (b) UO$_2$ symmetric stretch (ν_1): 854 cm^{-1}

Extraction of a 400 ppm UO_2 solution in a pH 5 acetate buffer with 9 and 10

Preparation of Uranyl Stock Solutions:

A 2000 ppm uranium stock solution was prepared by dissolving 422 mg $UO_2(NO_3)_2{\bullet}6H_2O$ in 100 mL aqueous acetate buffer with the pH adjusted to 5. This solution was used to prepare further solutions at lower concentrations (400 ppm).

A 400 ppm uranium stock solution (or a 1.68 mM UO_2 solution) was prepared by adding 10 mL of the 2000ppm stock solution to a 50 mL volumetric flask and filling to the final volume with aqueous acetate buffer adjusted to pH 5. All measurements measured with Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES)

Preparation of U standards to Calibrate the ICP-AES:

Blank: To a 25 mL volumetric flask was added 250 µL of a 1000 ppm Yttrium standard (Inorganic Ventures, catalog #CGY1-1) and the remainder of the volume was filled with 2% HNO_3.

100 ppb: To a 25 mL volumetric flask was added 250 µL of a 10 ppm uranium standard (Inorganic Ventures, catalog #MSU-10PPM), 250 µL of a 1000 ppm yttrium standard, and the remainder of the volume was filled with 2% HNO_3.

200 ppb: To a 25 mL volumetric flask was added 500 µL of a 10 ppm uranium standard, 250 µL of a 1000 ppm yttrium standard, and the remainder of the volume was filled with 2% HNO_3.

400 ppb: To a 50 mL volumetric flask was added 2 mL of a 10 ppm uranium standard, 500 µL of a 1000 ppm yttrium standard, and the remainder of the volume was filled with 2% HNO_3.

600 ppb: To a 50 mL volumetric flask was added 3 mL of a 10 ppm uranium standard, 500 µL of a 1000 ppm yttrium standard, and the remainder of the volume was filled with 2% HNO_3.

800 ppb: To a 25 mL volumetric flask was added 2 mL of a 10 ppm uranium standard, 250 µL of a 1000 ppm yttrium standard, and the remainder of the volume was filled with 2% HNO_3.

All analysis was completed on a Varian Vista AX CCD Simultaneous ICP-AES.

Preparation of Ligand (**9** and **10**) solutions for extraction:

A 1 mM solution was prepared by dissolving **9** (0.011 g, 0.01 mmol) in 10 mL chloroform.

A 1 mM solution was prepared by dissolving **10** (0.010 g, 0.01 mmol) in 10 mL chloroform.

General Liquid-Liquid Extraction of a pH 5 400 ppm UO_2 Solution:

The extractions were performed by mixing an organic phase (1 mL ligand solution) with an aqueous phase (1 mL 400 ppm UO_2) overnight. After vigorous mixing, 0.010 mL was taken from the aqueous layer and placed in a 10 mL volumetric flask that has been charged with 100 μL of a 1000 ppm yttrium internal standard. The mixture was diluted to 10 mL with 2% HNO_3 and the concentration of U in the aqueous phase was determined by ICP-AES.

To determine how much UO_2 is extracted into pure chloroform, a blank was prepared by mixing 1 mL chloroform with 1 mL 400 ppm UO_2 overnight. After vigorous mixing, 0.010 mL was taken from the aqueous layer and placed in a 10 mL volumetric flask that has been charged with 100 μL of a 1000 ppm yttrium internal standard. The mixture was diluted to 10 mL with 2% HNO_3 and the concentration of U in the aqueous phase was determined by ICP-AES.

The concentration of U in the 400 ppm stock solution was also measured prior to mixing. 0.010 mL of the stock solution was placed in a 10 mL volumetric flask that has been charged with 100 μL of a 1000 ppm yttrium internal standard. The mixture was diluted to 10 mL with 2% HNO_3 and the concentration of U in the aqueous phase was determined by ICP-AES.

For 1 mL of a 400 ppm U solution, if every ligand (1×10^{-6} mol) in the organic phase were to extract a uranyl ion (1.68×10^{-6} mol), there would be 0.68×10^{-6} mol (or ~160 ppm U) uranyl ion remaining in the aqueous phase.

Percent U extracted by either ligand (**9** or **10**) was calculated by treating the blank U concentration as the total U available in the aqueous phase to correct for the U extracted into the organic phase without any ligand present.

For example, for 1 mL of a 383 ppm U solution, if every ligand (1×10^{-6} mol) in the organic phase were to extract a uranyl ion (1.61×10^{-6} mol), there would be 0.61×10^{-6} mol (or ~145 ppm) uranyl ion remaining in the aqueous phase (see Table 6-1). 145 ppm U was then subtracted from the concentration of U in the blank (383 ppm = $U_{initial}$) to give the amount of total U (U_{total}) available for the ligands to extract: 383−145 = 238 ppm = U_{total}. $U_{initial}$ was then subtracted from the average extraction value of either ligand.

As an example for **9**: 383−330 = 53 ppm U extracted into the organic phase by **9** (U_{9ext}). Finally the percentage of U extracted was calculated by: U_{9ext}/ U_{total})*100 = ~ 22 %. Error for identical liquid-liquid extraction experiments at this concentration and volume was previously found to be 9% by taking the standard deviation of three separate extractions.

Table S1. Extraction of a pH 5 400 ppm UO_2 solution with **9** and **10**.

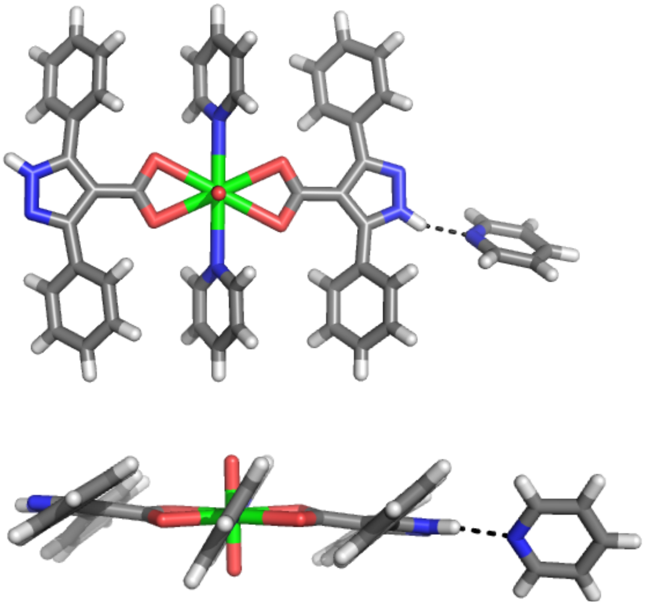
Sample	**U conc. (ppm)**	**avg**	**% U Extracted**
400ppb stock	405	403	
	393		
	411		
Blank	385	383	
	380		
Ligand **9** ext	328	330	22 ± 9 %
	344		
	319		
Ligand **10** ext	330	328	23 ± 9 %
	331		
	323		

Crystallographic Information

$UO_2(\mathbf{4})_2(pyr)_2$

Table S2. Crystal data and structure refinement for $UO_2(\mathbf{4})_2(pyr)_2$.

Identification code	CCDC #930587	
Empirical formula	C52 H42 N8 O6 U	
Molecular formula	C42 H32 N6 O6 U, 2(C5 H5 N)	
Formula weight	1112.96	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 16.8356(11) Å	α= 90°.
	b = 9.0753(6) Å	β= 93.824(2)°.
	c = 14.9922(10) Å	γ = 90°.
Volume	2285.5(3) $Å^3$	
Z	2	
Density (calculated)	1.617 Mg/m^3	
Absorption coefficient	10.493 mm^{-1}	
F(000)	1100	
Crystal size	0.287 x 0.116 x 0.106 mm^3	
Crystal color, habit	Colorless Block	
Theta range for data collection	2.630 to 57.703°.	
Index ranges	-18<=h<=16, -9<=k<=9, -8<=l<=16	
Reflections collected	17768	
Independent reflections	3065 [R(int) = 0.0391]	
Completeness to theta = 67.679°	74.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7451 and 0.5243	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3065 / 0 / 308	
Goodness-of-fit on F^2	1.087	
Final R indices [I>2sigma(I)]	R1 = 0.0218, wR2 = 0.0613	
R indices (all data)	R1 = 0.0241, wR2 = 0.0648	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.860 and -0.679 e.$Å^{-3}$	

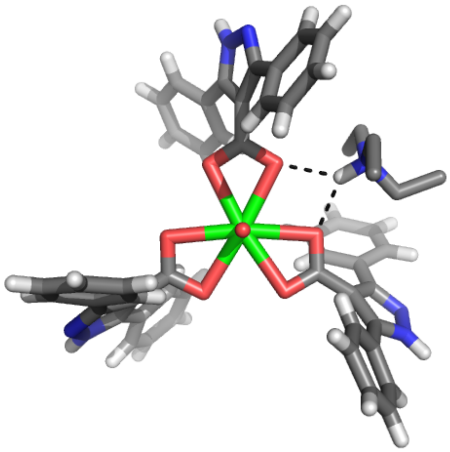


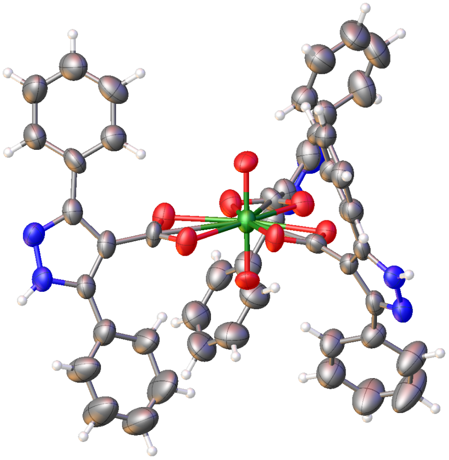
(pyr)$_2$**4**$_2$UO$_2$: C42 H32 N6 O6 U, 2(C5 H5 N), M = 1112.96, Monoclinic, P2$_1$/c, a = 16.8356(11), b = 9.0754(6), c = 14.9921(10) Å, α = 90.00°, β = 93.824(2) °, γ = 90.00°, V = 2285.5(3) Å^3, Z = 2, ρ_{calcd} = 1.617 g mL^{-1}, μ(Cu-Kα) = 10.49 mm^{-1}, θ_{max} = 57.7°, T = 100(2) K, 17762 reflections collected, $R1$ = 0.0218 for 2709 reflections, R_{int} = 0.039, (304 parameters) with $I > 2\sigma(I)$, and $R1$ = 0.0242, w$R2$ = 0.0648, GooF = 1.09 for all 3067 data, CCDC #930587.

$UO_2(\mathbf{4})_3$

Table S3. Crystal data and structure refinement for $UO_2(\mathbf{4})_3$.

Identification code	CCDC #930586	
Empirical formula	C58.50 H62 N7 O10 U	
Molecular formula	C48 H33 N6 O8 U, C6 H16 N, C4 H10 O, 0.5(C H4 O), 0.5(H2 O)	
Formula weight	1261.18	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 14.4680(11) Å	α= 90°.
	b = 31.719(2) Å	β= 103.416(3)°.
	c = 25.7639(18) Å	γ = 90°.
Volume	11500.6(15) $Å^3$	
Z	8	
Density (calculated)	1.457 Mg/m^3	
Absorption coefficient	8.455 mm^{-1}	
F(000)	5072	
Crystal size	0.151 x 0.093 x 0.055 mm^3	
Crystal color, habit	Colorless Block	
Theta range for data collection	3.225 to 67.774°.	
Index ranges	-17<=h<=17, -36<=k<=38, -30<=l<=25	
Reflections collected	92864	
Independent reflections	20520 [R(int) = 0.0363]	
Completeness to theta = 68.000°	97.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.0933 and 0.0140	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	20520 / 30 / 1303	
Goodness-of-fit on F^2	1.205	
Final R indices [I>2sigma(I)]	R1 = 0.0762, wR2 = 0.1705	
R indices (all data)	R1 = 0.0773, wR2 = 0.1709	
Extinction coefficient	n/a	
Largest diff. peak and hole	2.824 and -2.735 e.$Å^{-3}$	



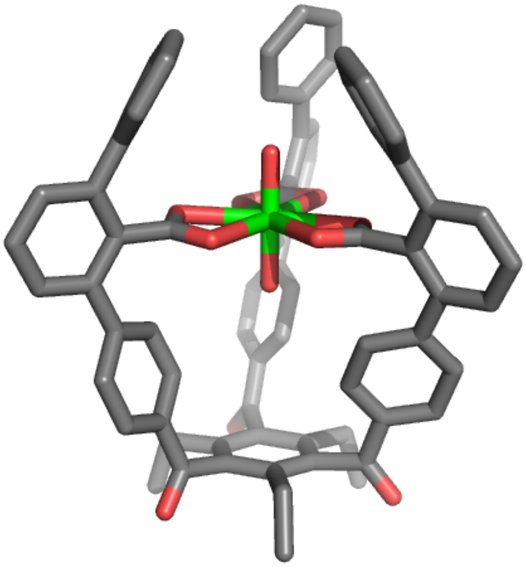


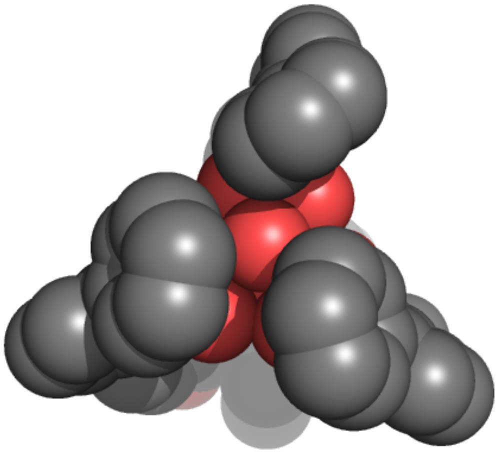
Et_3NH^+•$\mathbf{4_3}UO_2^-$: C48 H33 N6 O8 U, C6 H16 N, C4 H10 O, 0.5(C H4 O), 0.5(H2 O), M = 1261.18, Monoclinic, $P2_1/n$, a = 14.4680(11), b = 31.719(2), c = 25.7639(18) Å, α = 90.00°, β = 103.416(3)°, γ = 90.00°, V = 11500.6(15) Å^3, Z = 8, ρ_{calcd} = 1.453g mL^{-1}, μ(Cu-Kα) = 8.46 mm^{-1}, θ_{max} = 68.00°, T = 100(2) K, 92864 reflections collected, $R1$ = 0.0762 for 19904 reflections, R_{int} = 0.036 (1303 parameters) with $I > 2\sigma(I)$, and $R1$ = 0.0773, $wR2$ = 0.1709, GooF = 1.205 for all 20520 data, CCDC #930586.

UO_2(**9**)$_3$

Table S4. Crystal data and structure refinement for UO_2(**9**).

Identification code	CCDC #930588	
Empirical formula	C88 H87 Cl5 N O11 U	
Molecular formula	C72 H51 O11 U, 1.5(C5 H10), 2.5(C H2 Cl2), C6 H16 N	
Formula weight	1749.86	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 19.6677(9) Å	α= 90°.
	b = 18.8428(9) Å	β= 109.593(2)°.
	c = 22.6324(10) Å	γ = 90°.
Volume	7901.8(6) Å^3	
Z	4	
Density (calculated)	1.471 Mg/m^3	
Absorption coefficient	2.285 mm^{-1}	
F(000)	3548	
Crystal size	0.153 x 0.147 x 0.112 mm^3	
Crystal color, habit	Colorless Block	
Theta range for data collection	2.738 to 26.422°.	
Index ranges	-24<=h<=24, -23<=k<=22, -26<=l<=28	
Reflections collected	64835	
Independent reflections	16177 [R(int) = 0.0846]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7454 and 0.6506	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	16177 / 28 / 949	
Goodness-of-fit on F^2	1.010	
Final R indices [I>2sigma(I)]	R1 = 0.0472, wR2 = 0.0900	
R indices (all data)	R1 = 0.0915, wR2 = 0.1028	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.222 and -1.235 e.Å^{-3}	



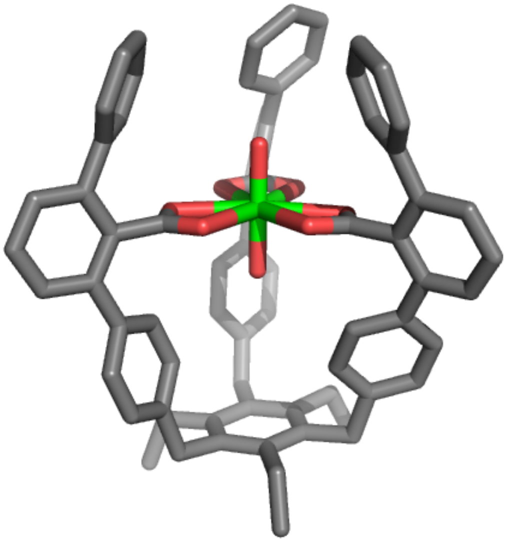


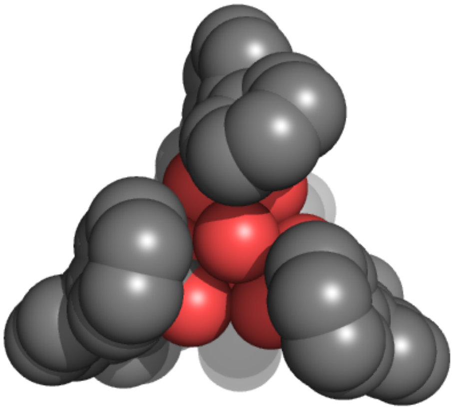
Et$_3$NH$^+$•**9**UO$_2^-$: C72 H51 O11 U, 1.5(C5 H10), 2.5(C H2 Cl2), C6 H16 N, M = 1749.86, Monoclinic, $P2_1/n$, a = 19.6677(9), b = 18.8428(9), c = 22.6324(10) Å, α = 90.00°, β = 109.593(2)°, γ = 90.00°, V = 7901.8(6) Å^3, Z = 4, ρ_{calcd} = 1.471 g mL^{-1}, μ(Mo-Kα) = 2.286 mm^{-1}, θ_{max} = 25.242°, T = 100(2) K, 64835 reflections collected, $R1$ = 0.0472 for 10896 reflections, R$_{\text{int}}$ = 0.0846, (949 parameters) with $I > 2\sigma(I)$, and $R1$ = 0.0915, $wR2$ = 0.1028, GooF = 1.010 for all 16177 data, CCDC #930588.

UO_2(**10**)$_3$

Table S5. Crystal data and structure refinement for UO_2(**10**).

Identification code	CCDC #940964	
Empirical formula	C82 H89 N O12 U	
Molecular formula	C72 H57 O8 U, C6 H16 N, 4(C H4 O)	
Formula weight	1518.57	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 14.2622(12) Å	α= 90°.
	b = 18.6958(15) Å	β= 93.484(3)°.
	c = 26.384(2) Å	γ = 90°.
Volume	7022.1(10) Å^3	
Z	4	
Density (calculated)	1.436 Mg/m^3	
Absorption coefficient	7.021 mm^{-1}	
F(000)	3104	
Crystal size	0.3 x 0.05 x 0.05 mm^3	
Crystal color, habit	Colorless Block	
Theta range for data collection	2.90 to 68.00°.	
Index ranges	-15<=h<=16, -22<=k<=15, -31<=l<=31	
Reflections collected	56122	
Independent reflections	12372 [R(int) = 0.0302]	
Completeness to theta = 67.17°	98.5 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.7529 and 0.4520	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12372 / 1 / 900	
Goodness-of-fit on F^2	1.062	
Final R indices [I>2sigma(I)]	R1 = 0.0228, wR2 = 0.0559	
R indices (all data)	R1 = 0.0246, wR2 = 0.0568	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.848 and -0.825 e.Å^{-3}	





Et_3NH^+•**10**UO_2^-: C72 H57 O8 U, C6 H16 N, 4(C H4 O), M = 1518.57, Monoclinic, $P2_1/n$, a = 14.2622(12), b = 18.6958(15), c = 26.384(2) Å, α = 90.00°, β = 93.484(3)°, γ = 90.00°, V = 7022.2(10) $Å^3$, Z = 4, ρ_{calcd} = 1.436g mL^{-1}, μ(Cu-Kα) = 7.02 mm^{-1}, θ_{max} = 68.00°, T = 100(2) K, 56112 reflections collected, $R1$ = 0.0228 for 11660 reflections, R_{int} = 0.030 (900 parameters) with $I > 2\sigma(I)$, and $R1$ = 0.0246, $wR2$ = 0.0568, GooF = 1.062 for all 12372 data, CCDC #940964.

References

1. J. A. Turner and W. S. Jacks, *J. Org. Chem.*, 1989, **54**, 4229.
2. A. Ana Ardá, C. Venturi, C. Nativi, O. Francesconi, G. Gabrielli, F. J. Cañada, J. Jiménez-Barbero and S. Roelens, *Chem.–Eur. J.*, 2010, **16**, 414.
3. D. Tilly, S. S. Samanta, A.-S. Castanet, A. De and J. Mortier, *Eur. J. Org. Chem.* 2006, 174.
4. E. Reisner, T. C. Abikoff and S. J. Lippard, *Inorg. Chem.* 2007, **46**, 10229.
5. E. Reisner and S. J. Lippard, *Eur. J. Org. Chem.*, 2008, 156.
6. Sather, A. C.; Berryman, O. B.; Rebek, J., Jr. *J. Am. Chem. Soc.* **2010**, *132*, 13572.