SUPPORTING INFORMATION

Facile synthesis of pyrrolyl-containing semisquaraines in water as precursors for non-symmetric squaraines

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MATERIALS AND METHODS

All reagents and solvents were purchased from the commercial sources (Sigma-Aldrich, Acros, and TCI) and were used as received. NMR spectra were acquired on a Bruker Ascend 400 (400 MHz) spectrophotometer using DMSO-d6 as the solvent, and the chemical shifts are reported in ppm (δ) from the residual DMSO peak (2.51 ppm) or TMS (0.00 ppm). For the acquisition of the ^{13}C NMR spectra, D1 parameter was adjusted from 2.00 to 0.25 to better observe resonance of carbons that are associated with the squaraine core. For squaraines that showed low solubility in DMSO-d6, ^{13}C NMR spectra were acquired either: a) in CF_3CO_2H using DMSO-d6 as external standard (in the NMR description indicated as: CF_3CO_2H w/DMSO-d_6 as external standard) or b) a drop of CF_3CO_2H was added to the NMR tube that contained the squaraine solution in DMSO-d6 prior to acquisition of the ^{13}C NMR spectrum (in the NMR description indicated as: DMSO-d6/CF_3CO_2H). Multiplicities are reported as: s – singlet, bs – broad singlet, d – doublet, dd – doublet of doublets, ddd – doublet of doublet of doublets, t – triplet, q – quartet, p – pentet, sext – sextet, m – multiplet.

High-resolution mass spectra (HRMS-ESI) were acquired on an Agilent 6230 instrument at the Mass Spectrometry Facility, Louisiana State University.

Microwave facilitated reactions were conducted in CEM Discover or Discover-SP microwave synthesizers using 10 mL vials.

For spectroscopic measurements stock solutions of non-symmetric squaraine dyes were prepared in dimethyl sulfoxide (DMSO, 1 mM solution), and used within 48 hours. The UV-Vis absorption spectra were measured with a Cary 60 UV-Vis spectrophotometer (Agilent Technologies), with a resolution of 1 nm using 1 cm quartz cell. The fluorescence spectra were measured with a Cary Eclipse fluorescence spectrometer (Varian Inc.) using 1 cm quartz cell with a resolution of 2 nm. Fluorescence measurements were carried out as follows: excitation and emission width slits were 5 mm and 5 mm; samples were excited at the wavelength 20-40 nm below the absorption maxima; PMT detection voltage set to high.

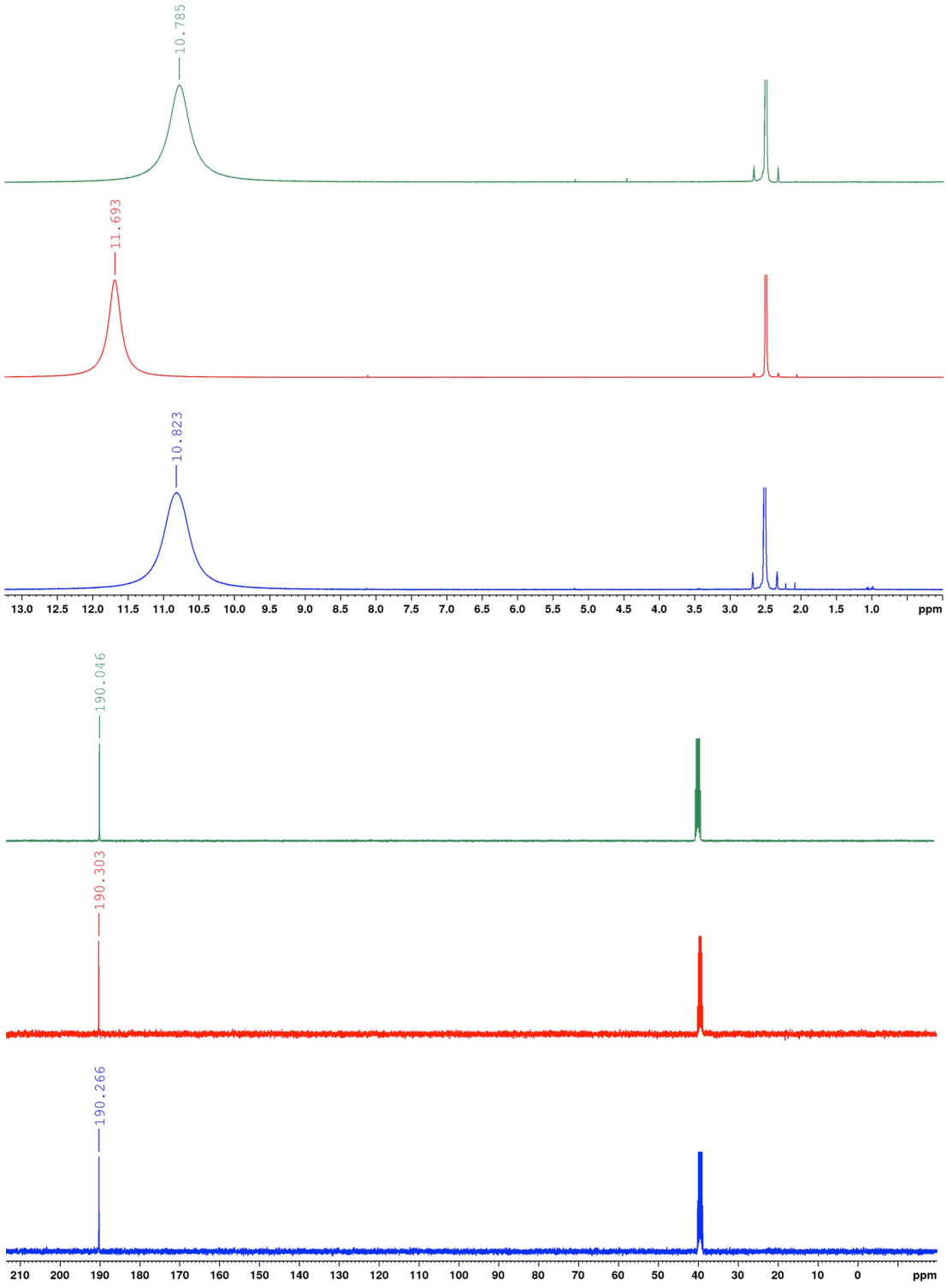


Figure S1. ^{1}H NMR (top) and ^{13}C NMR (bottom) of recovered and authentic squaric acid samples in DMSO-d6.

Blue: recovered squaric acid without charcoal treatment; **Red**: recovered squaric acid after charcoal treatment; **Green**: authentic squaric acid sample.

All samples showed the same decomposition point of >300 ^{o}C.

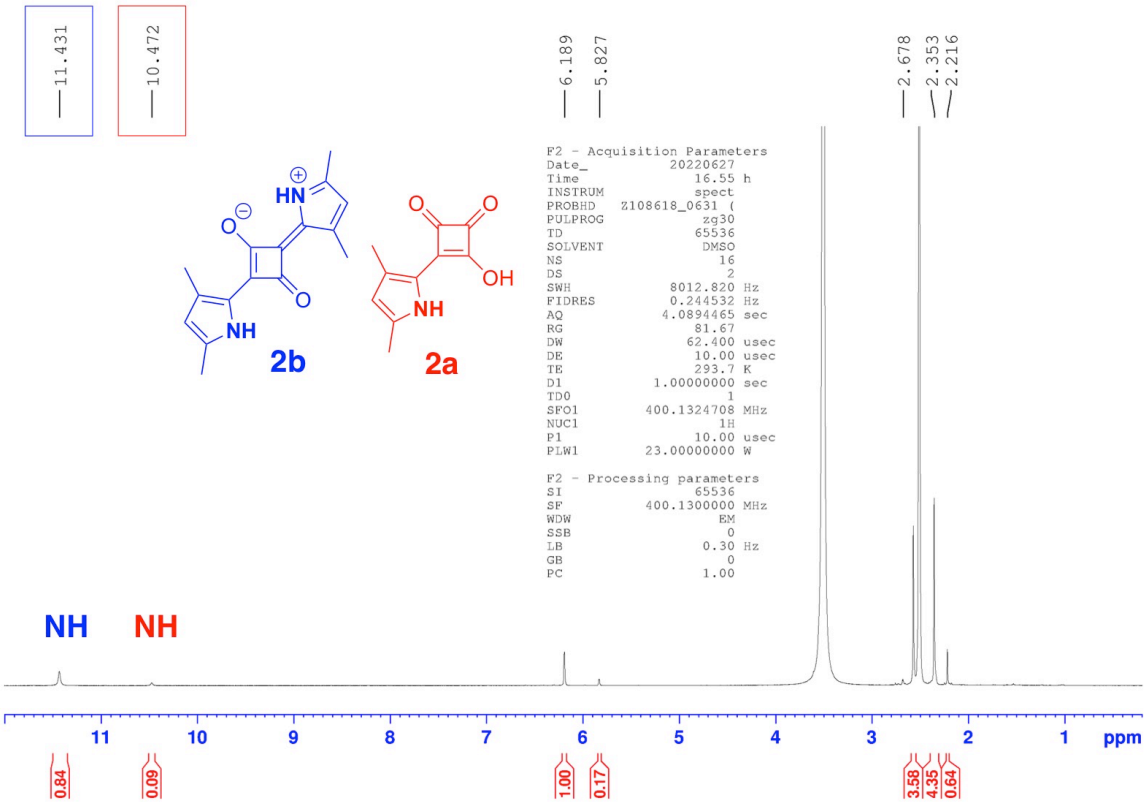
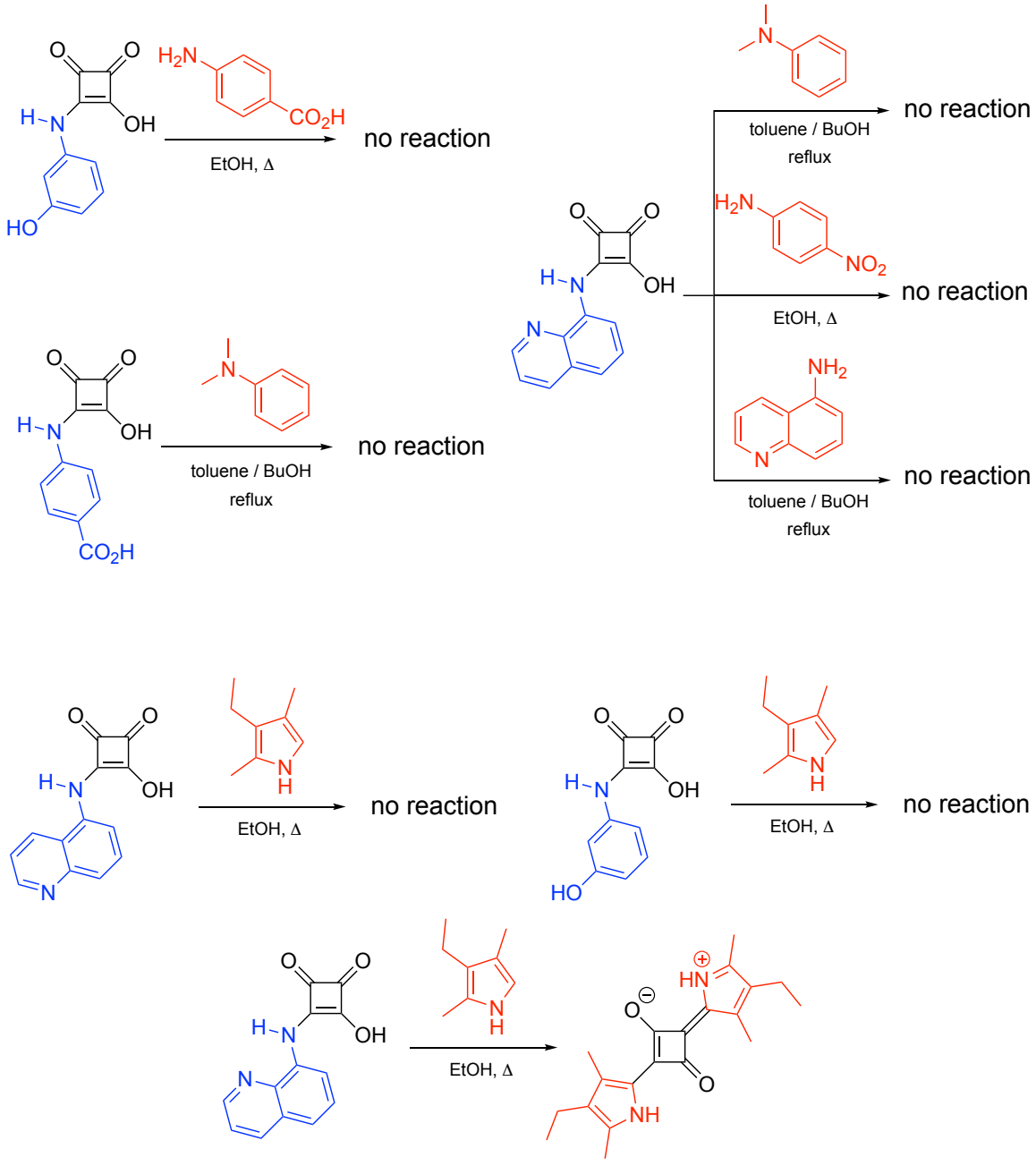


Figure S2. ^{1}H NMR (400 MHz, DMSO-d6) of the mixture **2a** and **2b**. See Scheme 3B and related text for details.



Scheme S1. Attempted syntheses of non-symmetric squaraines using squaric acids monoamines as precursors.

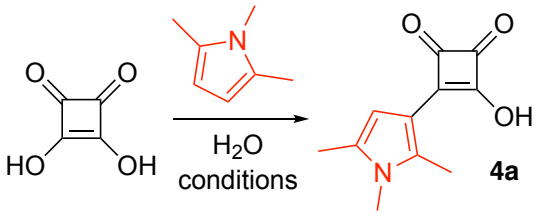
"No reaction" means starting semisquaraine was recovered.

Table S1. Cost of alkyl substituted pyrroles used in this work.[a]

pyrrole	Catalog #	quantity	unit price/ $	price: $/mol
3-ethyl-2,4-dimethylpyrrole	Acros 116210250	25g	379.00	1,868
2,4-dimethylpyrrole	TCI E1075	25g	400.00	1,522
2-ethylpyrrole	TCI E0713	5g	183.00	3,452
2,5-dimethylpyrrole	TCI D0260	25ml	105.00	427
1,2,5-trimethylpyrrole	TCI T1939	25ml	132.00	714

[a] – largest quantity available for sale was considered. Vendors' websites (Acros and TCI-America) accessed September 2022.

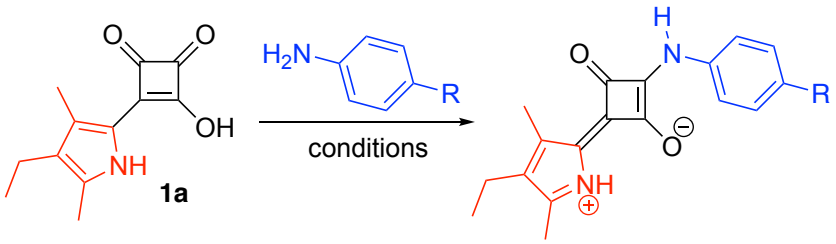
Table S2. Synthesis of **4a**: optimization studies.[a]



Entry	Squaric acid / eq.	Time / min	Temperature	Yield / %
1	5	15	reflux	51
2	1.5	15	reflux	76
3	1	5	reflux	14
4	1	5	70	14
5	1	5	80	45
6	1	15	80	50
7	1	30	80	60
8	**1**	**60**	**80**	**79**
9[b]	**1**	**60**	**80**	**84**
10[c]	**1**	**60**	**80**	**77**
11	1	150	r.t.	50

[a] – 1,2,5-trimethylpyrrole 1.5 mmol scale, [**pyrrole**] = 0.37 M; [b] – 1mL (1,2,5-trimethylpyrrole: 0.81 g, 9.1 mmol) scale; [c] – 5 mL (1,2,5-trimethylpyrrole: 4.05 g, 45.5 mmol) scale.

Table S3. Synthesis of non-symmetric squaraines; optimization studies.[a]



Entry	R / eq.	Time / min	Solvent	Heating[b]	Yield / %
1	CH_3 / 1.0	160	H_2O	conventional	55
2	CH_3 / 1.1	950	H_2O	conventional	83
3	CH_3 / 1.0	5	H_2O	MW	0
4	CH_3 / 1.0	30	H_2O	MW	64
5	CH_3 / 1.0	45	H_2O	MW	63
6	CH_3 / 1.0	60	H_2O	MW	62
7	CH_3 / 1.5	30	H_2O	MW	78
8	NO_2 / 1.5	30	H_2O	MW	15
9	NO_2 / 1.5	30	EtOH	MW	60
10	NO_2 / 1.5	30	1-BuOH	MW	75
11	NO_2 / 1.5	30	DMF	MW	30

[a] – **1a** 0.23mmol scale, [**1a**] = 0.37 M; [b] **–** Conventional heating – heating mantle or beads bath, reflux; MW – microwave heating, 90°C.

Table S4. Photophysical properties of non-symmetric squaraines.[a]

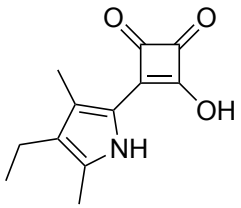
Squaraine	λ_{ab}^{max} / nm	λ_{em}^{max} / nm	ε / M^{-1} cm^{-1}
6	490	510	69,300
7	493	516	41,100
8	530	NF[b]	21,200
9	505	518	74,000
10	467	505	27,400
11	505	NF[b]	28,500
12	513	526	68,200
13	490	510	45,400
14	433	450	63,200
15	412	473	37,400
16	614	625	1,499,000
17	610	616	2,005,000
18	598	612	56,000

[a] – Solvent DMSO; for ε determination, absorption was determined at several concentrations of the dye to assure a linear dependence is observed between absorption (A) and dye's concentration; see individual spectra for specific conditions ([**dye**], λex). [b] – NF = no fluorescence signal was detected over a range of dye's concentrations.

SYNTHESIS AND CHARACTERIZATION OF PYRROLYL-SEMISQUARAINES

Synthesis of semisquaraine **1a**:

A round bottom flask was charged with a stirring bar, water (100 mL), and squaric acid (21.16 g, 185.5 mmol). Once the solution was brought to reflux, 2,4-dimethyl-3-ethyl-pyrrole (5.00 mL, 37.1 mmol) was added in one portion, and the reaction mixture was refluxed for 15 min. The reaction mixture was removed from heat, water (150 mL) was added, and the mixture brought to reflux. Next, while hot, the supernatant was decanted, and water (250 mL) was added to the residue, and the mixture was brought to reflux, followed by decantation. This sequence was repeated yep more times (total volume of water used 1000 mL for reaction and washings). After the final wash, the solid was filtered, dried under vacuum to give **1a** as a green solid (7.90 g, 97 %).



^{1}H NMR (DMSO-d_6): δ 10.57 (s, 1H), 2.33 (q, *J*= 7.5 Hz, 2H) 2.33 (s, 3H), 0.99 (t, *J* = 7.5 Hz, 3H).

^{13}C (DMSO-d_6): δ 191.41, 190.18, 166.26, 134.85, 126.03, 124.76, 117.20, 16.72, 15.07, 11.05, 10.29.

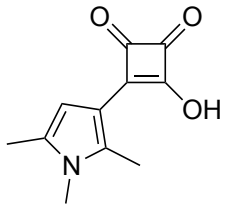
HRMS (ESI; $C_{12}H_{13}NO_3$) m/z: calc. for $[M+H]^+$ 220.0974; found: 220.0973.

Recovery of squaric acid: the water washes were combined, and charcoal (10.0 g) was added. The mixture was brought to reflux, filtered while hot, and water removed in vacuo, to give white squaric acid (16.07 g, 95 % recovery).

It should also be noted that unreacted squaric acid was also recovered according to the following procedure: after the final wash with water, **4a** was washed with $NaHCO_3$ (sat.), followed by HCl (1 M). All aqueous fractions were combined, nd water removed in vacuo (using ca. 40-50^{o}C bath). Separation of squaric acid from NaCl was subsequently done by treating squaric acid/NaCl mixture with DMF, followed by filtration and removal of DMF in vacuo. The resulting squaric acid was subjected to the aforementioned charcoal treatment (i.e., charcoal/water/reflux) to get rid of light green color, to produce white solid, whose spectral characteristics matched those of the authentic/commercial squaric acid.

Semisquaraine **4a**:

A round bottom flask was charged with a stirring bar, water (20 mL), and squaric acid (846.5 mg, 7.42 mmol). Once the solution was brought to 80 °C, 1,2,5-trimethylpyrrole (1.00 mL, 7.39 mmol) was added in one portion, and the reaction mixture was stirred at 80 °C for 60 min. The reaction mixture was removed from heat, cooled to room temperature, and filtered. Next, the precipitate was washed with water (3 x 10 mL). Then, the precipitate was washed with diethyl ether (2 x 5 mL) and dried under vacuum to give **4a** as a maroon solid (1.27 g, 84 %).



^{1}H NMR (DMSO-d_6): δ 6.32 (d, *J* = 0.9 Hz, 1H), 3.42 (s, 3H), 2.62 (s, 3H), 2.18 (s, 3H).

^{13}C (DMSO-d_6): δ 193.77, 192.80, 172.92, 133.39, 130.60, 109.65, 104.41, 30.46, 12.23, 12.06.

HRMS (ESI; $C_{11}H_{11}NO_3$) m/z: calc. for $[M+H]^+$ 206.0817; found: 206.0818.

SYNTHESIS AND CHARACTERIZATION OF NON-SYMMETRIC SQUARAINE DYES

General procedure for the synthesis of 1-N-alkyl/aryl-3-pyrrolylsquaraines 6–15.

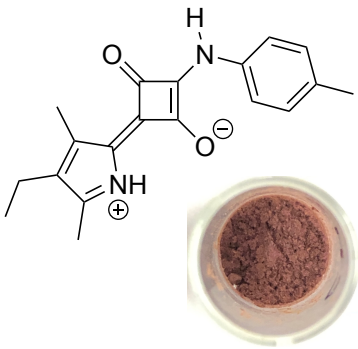
Squaraine **6**:

Microwave vial was charged with a stirring bar, semisquaraine **1a** (50.0 mg, 0.23 mmol), 1-BuOH (0.6 mL), and toluidine (0.34 mmol, 1.5 eq), sealed and subjected to MW irradiation at 90°C for 30 min. After the reaction was cooled to room temperature, the reaction mixture was sonicated (5 min), filtered and washed with acetone, and the red solid was dried under vacuum to give **6** (54.9 mg, 78 % yield).

^{1}H NMR (DMSO-d_6): δ 11.89 (s, 1H), 11.02 (s, 1H), 7.79 (d, *J* = 8.5 Hz, 2H), 7.21 (d, *J* = 8.2 Hz, 2H), 2.49 (s, 3H), 2.36 (q, *J* = 7.4 Hz, 2H), 2.30 (s, 3H), 2.29 (s, 3H), 1.01 (t, *J* = 7.5 Hz, 3H).

^{13}C (DMSO-d_6): δ 179.41, 173.47,171.51, 169.58, 140.12, 135.67, 135.21, 130.38, 130.16, 127.42, 121.55, 120.81, 21.01, 17.21, 15.23, 11.90, 11.21.

HRMS (ESI; $C_{19}H_{20}N_2O_2$) m/z: calc. for $[M+H]^+$ 309.1603; found: 309.1600.



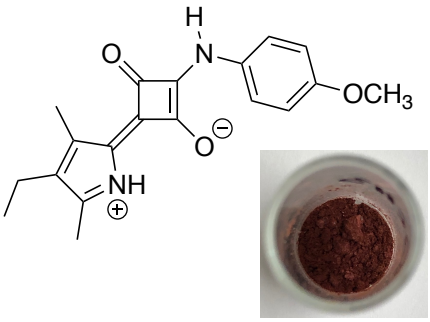
Squaraine **7**:

Obtained according to the general procedure; 90 % yield.

^{1}H NMR (DMSO-d_6): δ 11.89 (s, 1H), 10.94 (s, 1H), 7.85 (d, *J* = 9.0 Hz, 2H), 6.99 (d, *J* = 9.1 Hz, 2H), 3.77 (s, 3H), 2.49 (s, 3H), 2.36 (q, *J* = 7.4 Hz, 2H), 2.28 (s, 3H), 2.29 (s, 3H), 1.01 (t, *J* = 7.5 Hz, 3H).

^{13}C (DMSO-d_6): δ 178.60, 173.06, 171.21, 168.07, 157.11, 138.84, 130.87, 129.26, 126.62, 121.92, 120.96, 114.48, 55.40, 16.77, 14.82, 11.41, 10.73

HRMS (ESI; $C_{19}H_{20}N_2O_3$) m/z: calc. for $[M+H]^+$ 325.1552; found: 325.1582.



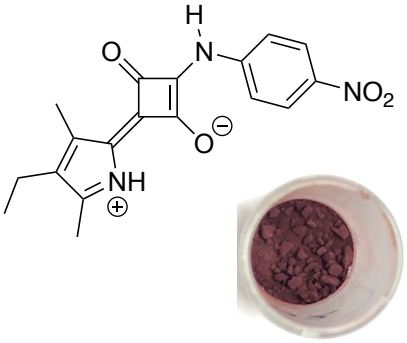
Squaraine **8**:

Obtained according to the general procedure; 75 % yield.

^{1}H NMR (DMSO-d_6): δ 12.07 (s, 1H), 11.45 (s, 1H), 8.26 (d, *J* = 9.2 Hz, 2H), 8.07 (d, *J* = 8.0 Hz, 2H), 2.54 (s, 3H), 2.39 (q, *J* = 7.6 Hz, 2H), 2.35 (s, 3H), 1.03 (t, *J* = 7.4 Hz, 3H).

^{13}C (CF_3CO_2H w/DMSO-d_6 as external standard): 172.96, 171.22, 159.36, 157.01, 155.29, 143.49, 141.94, 140.98, 134.29, 125.02, 122.34, 119.15, 15.71, 11.40, 10.45, 8.95

HRMS (ESI; $C_{18}H_{17}N_3O_4$) m/z: calc. for $[M+H]^+$ 340.1297; found: 340.1281.



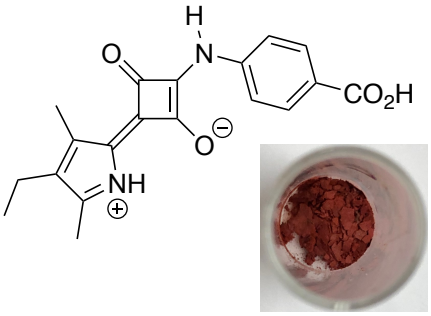
Squaraine **9**:

Obtained according to the general procedure; 50 % yield.

^{1}H NMR (DMSO-d_6): δ 12.91 (s, 1H), 11.98 (s, 1H), 11.30 (s, 1H), 8.00 (d, *J* = 8.8 Hz, 2H), 7.94 (d, *J* = 8.8 Hz, 2H), 2.53 (s, 3H), 2.38 (q, *J* = 7.4 Hz, 2H), 2.34 (s, 3H), 1.02 (t, *J* = 7.4 Hz, 3H).

^{13}C (DMSO-d_6): δ 177.90, 171.41, 166.77, 142.74, 141.60, 132.09, 130.63, 128.23, 126.53, 121.74, 119.85, 30.71, 16.72, 14.60, 11.61, 10.82.

HRMS (ESI; $C_{19}H_{18}N_2O_4$) m/z: calc. for $[M+H]^+$ 339.1345; found: 339.1348.



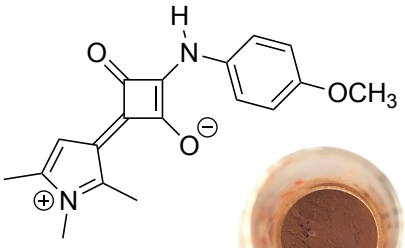
Squaraine **10**:

Obtained according to the general procedure, except for the purification step. Purification was done as follows: the crude solid was dissolved in minimum amount of DMSO, followed by trituration with acetone, and the product was collected by filtration; 57 % yield.

^{1}H NMR (DMSO-d_6): δ 12.11 (bs, 1H), 7.87 (d, *J* = 9.2 Hz, 2H), 7.00 (d, *J* = 9.2 Hz, 2H), 6.53 (d, *J* = 0.8 Hz, 1H), 3.78 (s, 3H), 3.44 (s, 3H), 2.79 (s, 3H), 2.20 (s, 3H).

^{13}C (DMSO-d_6): δ 185.00, 176.64, 175.85, 173.64, 157.97, 136.45, 131.72, 130.97, 122.88, 114.96, 113.51, 105.45, 55.88, 31.07, 13.19, 12.57.

HRMS (ESI; $C_{18}H_{18}N_2O_3$) m/z: calc. for $[M+H]^+$ 311.1396; found: 311.1395.



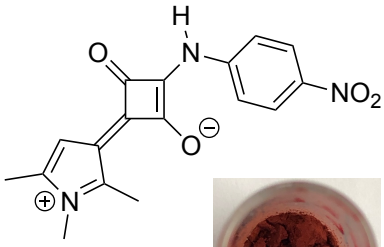
Squaraine **11**:

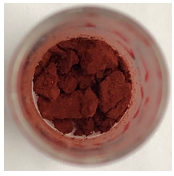
Obtained according to the general procedure, except for the purification step. Purification was done as follows: the crude solid was dissolved in minimum amount of DMF, followed by trituration with diethyl ether, and the product was collected by filtration; 71 % yield.

^{1}H NMR (DMSO-d_6): δ 12.47 (bs, 1H), 8.29 (dt, *J* = 9.2, 2.5 Hz, 2H), 8.14 (dt, *J* = 9.2, 2.5 Hz, 2H), 6.63 (d, *J* = 1.0 Hz, 1H), 3.48 (s, 3H), 2.85 (s, 3H), 2.22 (s, 3H).

^{13}C (DMSO-d_6): δ 189.19, 175.54, 175.26, 149.53, 137.70, 134.70, 130.95, 129.70, 115.56, 113.76, 113.37, 112.77, 105.21, 48.38, 30.92, 13.04, 12.56.

HRMS (ESI; $C_{17}H_{15}N_3O_4$) m/z: calc. for $[M+H]^+$ 326.1141; found: 326.1128.

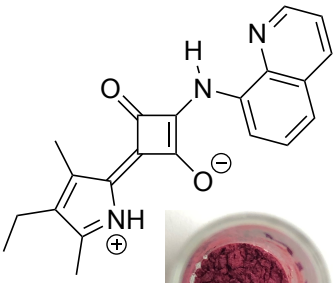




Squaraine **12**:

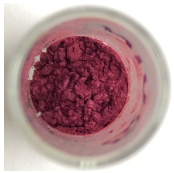
Obtained according to the general procedure; 80 % yield.

^{1}H NMR (DMSO-d_6): δ 11.28 (s, 1H), 10.89 (bs, 1H), 8.99 (dd, *J* = 4.2, 1.4 Hz, 1H), 8.56 (bs, 1H), 8.48 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.69 (m, 2H), 2.53 (s, 3H), 2.38 (q, *J* = 7.4 Hz, 2H), 2.33 (s, 3H), 1.03 (t, *J* = 7.4 Hz, 3H).



^{13}C (CF_3CO_2H w/DMSO-d_6 as external standard): δ 178.00, 176.07, 164.32, 162.81, 161.87, 153.08, 148.40, 146.54, 139.64, 136.10, 134.90, 134.79, 134.40, 132.24, 130.85, 127.45, 126.30, 20.32, 15.94, 15.27, 13.57.

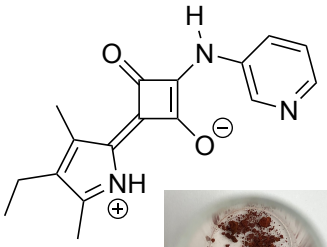
HRMS (ESI; $C_{21}H_{19}N_3O_2$) m/z: calc. for $[M+H]^+$ 346.1556; found: 346.1558.



Squaraine **13**:

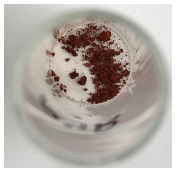
Obtained according to the general procedure; 52 % yield.

^{1}H NMR (DMSO-d_6): δ 12.00 (bs, 1H), 11.23 (s, 1H), 8.97 (bs, 1H), 8.37 (dd, *J* = 4.7, 1.4 Hz, 1H), 8.34 (ddd, *J* = 8.4, 2.7, 1.4 Hz, 1H), 7.45 (ddd, *J* = 8.3, 4.7, 0.6 Hz, 1H), 2.52 (s, 3H), 2.38 (q, *J* = 7.5 Hz, 2H), 2.32 (s, 3H), 1.02 (t, *J* = 7.5 Hz, 3H).



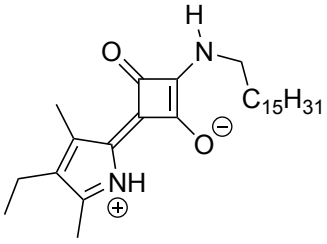
^{13}C (DMSO-d_6): δ 179.02, 173.10, 171.57, 146.13, 142.47, 142.25, 135.04, 132.09, 128.37, 127.74, 124.39, 121.93, 17.18, 15.10, 12.02, 11.25.

HRMS (ESI; $C_{17}H_{17}N_3O_2$) m/z: calc. for $[M+H]^+$ 296.1399; found: 296.1408.



Squaraine **14**:

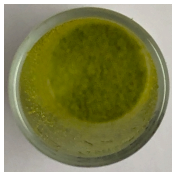
Obtained according to the general procedure; 47 % yield.



^{1}H NMR (DMSO-d_6): δ 10.61 (s, 1H), 10.27 (bs, 1H), 3.69 (t, *J* = 6.9 Hz, 2H), 2.41 (s, 3H), 2.32 (q, *J* = 7.5 Hz, 2H), 2.22 (s, 3H), 1.59 (m, 2H), 1.25 (m, 26H), 0.99 (t, *J* = 7.5 Hz, 3H), 0.86 (t, *J* = 6.8 Hz, 3H).

^{13}C (DMSO-d_6/CF_3CO_2H): δ 185.29, 167.58, 158.90, 136.05, 127.50, 125.56, 120.45, 44.62, 31.77, 30.02, 29.51 (overlap), 29.37, 29.33, 29.18, 26.17, 22.57, 17.25, 15.44, 14.44, 11.64, 11.03.

HRMS (ESI; $C_{28}H_{46}N_2O_2$) m/z: calc. for $[M+H]^+$ 443.3638; found: 443.3619.



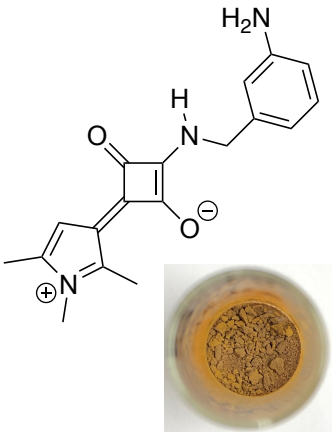
Squaraine **15**:

Obtained according to the general procedure; 25 % yield.

^{1}H NMR (DMSO-d_6): δ 10.88 (bs, 1H), 7.01 (t, *J* = 7.6 Hz, 1H), 6.49 (m, 4H), 5.17 (s, 2H), 4.76 (s, 2H), 3.42 (s, 3H), 2.73 (s, 3H), 2.18 (s, 3H).

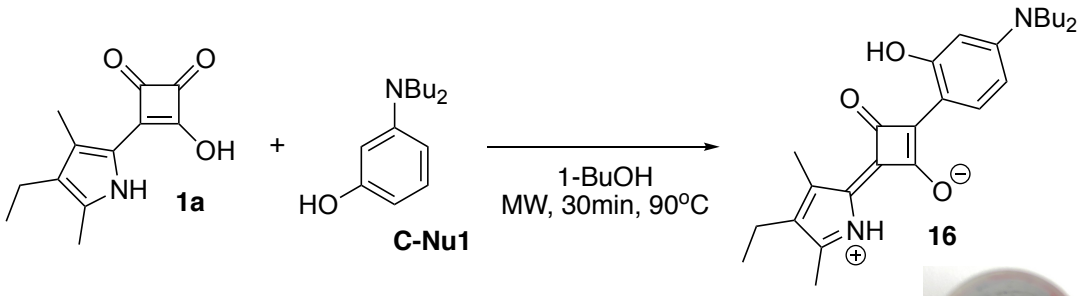
^{13}C (DMSO-d_6): δ 188.73, 175.03, 174.88, 174.74, 149.03, 137.21, 134.25, 130.48, 129.21, 115.11, 113.31, 112.92, 112.29, 104.75, 47.89, 30.42, 12.54, 12.05.

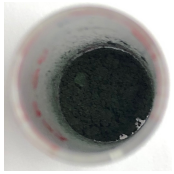
HRMS (ESI; $C_{18}H_{19}N_3O_2$) m/z: calc. for $[M+H]^+$ 310.1556; found: 310.1558.



Synthesis of 1-C-aryl/indolyn-3-pyrrolylsquaraines 16-18

Squaraine **16**:





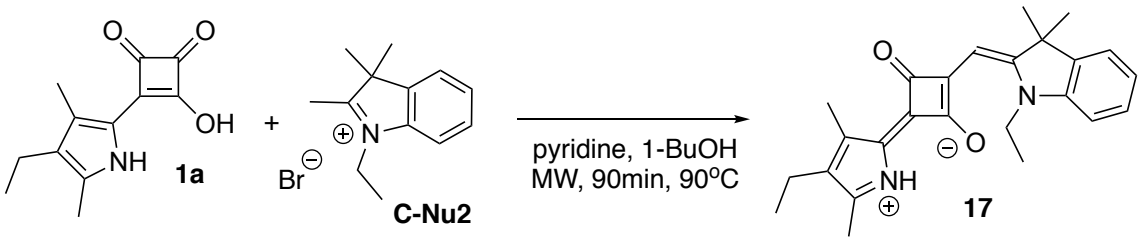
Microwave vial was charged with a stirring bar, semisquaraine **1a** (50.0 mg, 0.228 mmol), 1-BuOH (0.6 mL), and **C-Nu1** (78.1 mg, 0.353 mmol, 1.5 eq.; **C-Nu1** was prepared according to literature procedure [S1]), sealed and subjected to MW irradiation at 90°C for 30 min. After the reaction was cooled to room temperature, the reaction mixture was filtered, washed with EtOH, and the solid was recrystallized from EtOH/H_2O to give **16** (36.9 mg, 38 % yield).

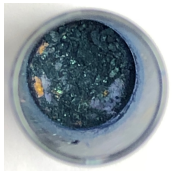
^{1}H NMR (DMSO-d_6): δ 12.42 (s, 1H), 11.78 (s, 1H), 7.92 (d, *J* = 9.1 Hz, 1H), 6.60 (dd, *J* = 9.2, 2.4 Hz, 1H), 6.17 (d, *J* = 2.4 Hz, 1H), 3.51 (m, 4H), 2.49 (q, *J* = 7.5 Hz, 2H), 2.48 (s, 3H), 1.65 (m, 4H), 1.44 (sext, *J* = 7.4 Hz, 4H), 1.12 (t. *J* = 7.5 Hz, 3H), 1.03 (t, *J* = 7.3 Hz, 6H).

^{13}C (DMSO-d_6): δ 196.14, 188.98, 173.28, 162.58, 155.19, 148.47, 135.24, 130.66, 122.61, 109.10, 107.32, 97.97, 50.32, 29.39, 19.54, 16.71, 14.33, 13.92, 13.81, 12.04, 10.82.

HRMS (ESI; $C_{26}H_{34}N_2O_3$) m/z: calc. for $[M+H]^+$ 423.2648; found: 423.2660.

Squaraine **17**:





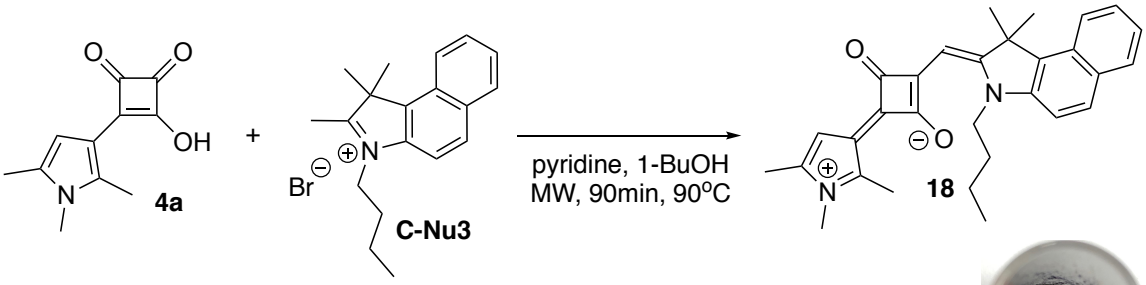
Microwave vial was charged with a stirring bar, semisquaraine **1a** (50.0 mg, 0.228 mmol), 1-BuOH (0.6 mL), **C-Nu2** (94.7 mmol, 0.353 mmol, 1.5 eq.; **C-Nu2** was prepared according to literature procedure [S2]), and pyridine (0.046 ml, 0.570 mmol, 2.5 eq.) sealed and subjected to MW irradiation at 90°C for 90 min. After the reaction was cooled to room temperature, the reaction mixture was filtered, washed with EtOH, and the solid was recrystallized from EtOH/H_2O to give **17** (40.7 mg, 46 % yield).

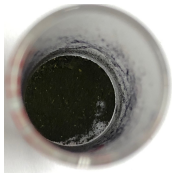
^{1}H NMR (DMSO-d_6): δ 11.11 (s, 1H), 7.56 (dd, *J* = 7.2, 1.1 Hz, 1H), 7.40 (m, 2H), 7.22 (td, *J* = 7.1, 1.6 Hz, 1H), 5.84 (s, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 3.35 (s, 1H), 2.51 (s, 3H; overlap with DMSO), 2.37 (q, *J* = 7.6 Hz, 2H), 2.32 (s, 3H), 1.71 (s, 6H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.02 (t, *J* = 7.5 Hz, 3H).

^{13}C (DMSO-d_6): δ 181.58, 170.81, 167.38, 142.31, 141.90, 141.89, 131.16, 128.58, 128.39, 124.85, 122.82, 122.33, 111.19, 87.49, 49.67, 38.78, 26.63, 17.28, 15.15, 12.37, 12.09, 11.27.

HRMS (ESI; $C_{25}H_{28}N_2O_2$) m/z: calc. for $[M+H]^+$ 389.2229; found: 389.2242.

Squaraine **18**:





Microwave vial was charged with a stirring bar, semisquaraine **4a** (50.0 mg, 0.244 mmol), 1-BuOH (0.6 mL), **C-Nu3** (126.7 mg, 0.366 mmol, 1.5 eq; **C-Nu3** was prepared according to literature procedure [S2]), and pyridine (0.049 mL, 0.610 mmol, 2.5 eq.) sealed and subjected to MW irradiation at 90°C for 90 min. After the reaction was cooled to room temperature, the reaction mixture was filtered, washed with EtOH, and the residue was subjected to column chromatography (SiO_2, $CHCl_3$/ethyl acetate – 2/1) to give **18** (33.4 mg, 30% yield).

^{1}H NMR (DMSO-d_6): δ 8.30 (d, J = 8.5 Hz, 1H), 8.09 (m, 2H), 7.85 (d, *J* = 8.9 Hz, 1H), 7.68 (ddd, *J* = 8.4, 6.9, 1.3 Hz, 1H), 7.54 (ddd, *J* = 8.1, 6.8, 1.1 Hz, 1H), 6.58 (d, J = 1.2 Hz, 1H), 6.01 (s, 1H), 4.36 (t, *J* = 7.3 Hz, 2H), 3.46 (s, 3H), 2.85 (s, 3H), 2.21 (d, *J* = 1.1 Hz, 3H), 1.97 (s, 6H), 1.78 (pent, *J* = 7.3 Hz, 3H), 1.43 (sext, *J* = 7.4 Hz, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

^{13}C (DMSO-d_6): δ 187.68, 175.45, 174.35, 139.61, 137.08, 135.18, 132.16, 131.97, 130.49, 130.24, 128.19, 128.05, 125.56, 123.00, 114.31, 112.39, 105.72, 87.87, 52.12, 44.14, 31.12, 29.78, 26.33, 20.02, 14.21, 13.26, 12.65.

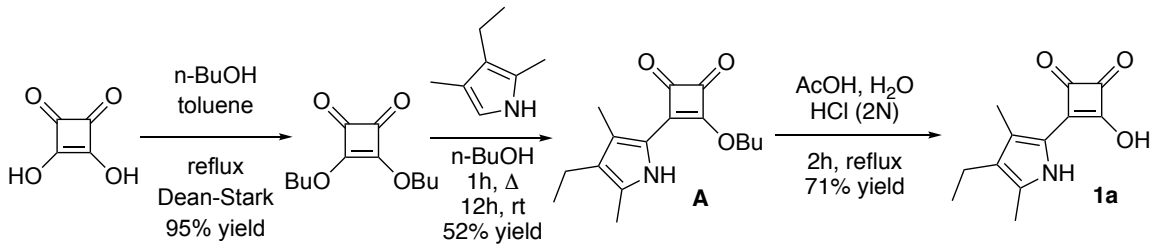
HRMS (ESI; $C_{30}H_{32}N_2O_2$) m/z: calc. for $[M+H]^+$ 453.2532; found: 453.2554.

GREEN CHEMISTRY CONSIDERATIONS, METRICS, AND CALCULATIONS

General considerations

It could be assumed that processes that require fewer synthetic steps and fewer reagents would be more attractive from the green chemistry/sustainability points of view. However, to gain a quantitative assessment of green chemistry metrics, literature preparation of **1a** [S3] was taken as a reference for comparison. It should be noted, however, that it was not the goal of the authors [S3] to prepare **1a** in a more environmentally benign manner. Thus, the assessments/calculations that follow should not be viewed as green chemistry shortcomings of the published procedure.

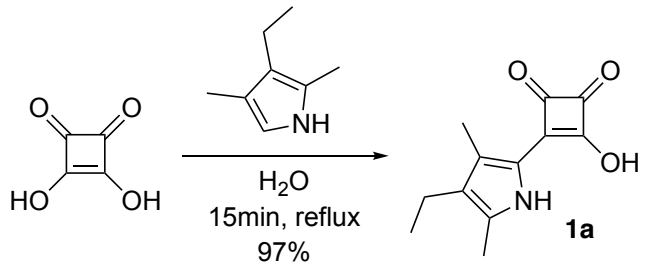
*Literature procedure for the synthesis of **1a** [S3]:*



Step 1 was accomplished according to literature procedure [S4].

Overall yield: 35%
Number of step from squaric acid: 3
Intermediate **A** was recrystallized from AcOH, quantities of AcOH were not specified.

*Our synthesis of **1a**:*



Overall yield: 97%
Number of steps from squaric acid: 1

HOW SYNTHESIS OF **1a** RELATES TO 12 PRINCIPLES OF GREEN CHEMISTRY [5]

	Green Chemistry Principle	**Features of 1a synthesis**
1	Waste prevention	H_2O is the only by-product
2	Atom efficiency	92.4 % is only 7.6 % lower than the optimal AE
3	Less hazardous/toxic materials	avoids the use of $SOCl_2$, HCl, LiOH, toluene
4	Safer product by design	–
5	Innocuous solvents and auxiliaries	H_2O is the only solvent used for the reaction
6	Energy efficient by design	shortened reaction times from hours to minutes; reactions could be done at room temperature
7	Renewable rather than depleting raw material	–
8	Shorter synthesis (avoid derivatization)	1 step synthesis from commercially available compounds
9	Catalytic rather than stoichiometric reagents	–
10	Design products for degradation	–
11	Analytical methods for pollution prevention	–
12	Inherently safer processes	simple reaction set-up, standard isolation and purification processes

E-FACTOR (E)

$$\text{E-factor} = \frac{\text{total mass of waste}}{\text{mass of product}}$$

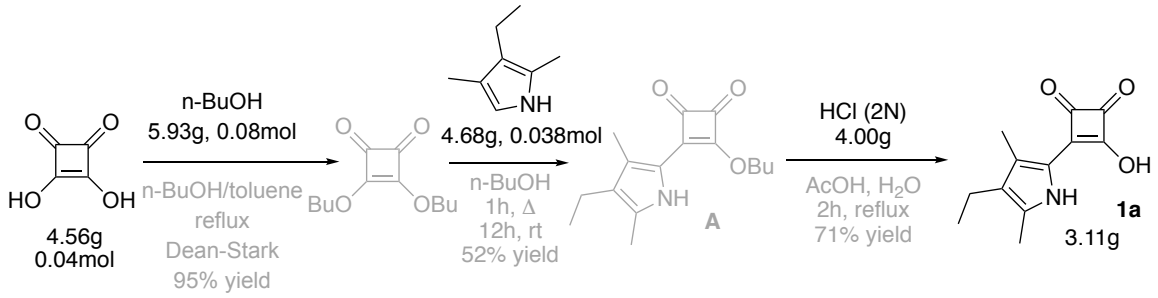
Key features: relies on the quantity of waste produced [S6].

However, E-factor may not be very informative, due to a somewhat ambiguous definition of what "waste" is, especially when "waste" is related to solvents used. Since many solvents could, in principle, be recovered, it could be argued that solvents should not be used in the calculation. Yet, it is not always obvious that the recovered solvent is of sufficient quality to be reused (or at least the quality of the recovered solvent should be established along with the efficiency of the solvent recovery).

Here, for simplicity, solvents used for reactions, isolation and purification of the products are not considered in calculation of E-factors for both the literature and our syntheses of **1a**.

*Literature procedure for the synthesis of **1a** [S3]:*

Step 1 was accomplished according to literature procedure [S4]. Step 3 leading to **1a** was done on 0.02mol scale; thus, the amounts of the reagents, solvents, etc used in steps 1 and 2 were recalculated to this scale, with considerations of the yields in each step.



Amounts used to produce 3.11g of **1a**:

Step 1: squaric acid (**4.56g**, 0.04mol), n-BuOH (**5.93g**, 0.08mol; only 2 equivalents of n-BuOH are considered as reagent here, the rest is considered as solvent; total volume of n-BuOH is 60mL. Notably, using 2 equivalents of n-BuOH to obtain the corresponding dialkoxy-squarate is just an assumption; literature accounts do indicate that large excess of alcohols is required to obtain dialkoxy-squarate[S#, S#]).

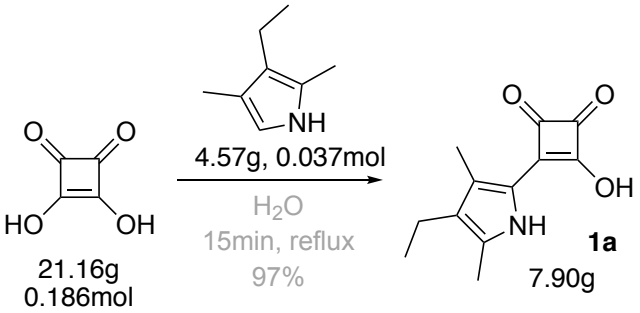
Step 2: pyrrole (**4.68g**, 0.038mol).

Step 3: HCl (**4.00g**; 4mL of 2N HCl), AcOH is considered a solvent.

Amount of waste: (4.56 + 5.93 + 4.68 + 4.00) – 3.11g = 19.17g

$$\text{E-factor} = \frac{19.17}{3.11} = 6.16$$

*Our procedure for the synthesis of **1a**:*



a) E-factor calculations without accounting for recovered squaric acid:
Amounts used to produce 7.90g of **1a**: squaric acid (21.16g), pyrrole (4.57g)
Amount of waste: (21.16 + 4.57) – 7.90 = 17.83g

$$\text{E-factor} = \frac{17.83}{7.90} = 2.26$$

b) E-factor calculations accounting for recovered squaric acid:
Amounts used to produce 7.90g of **1a** and with recovered 16.07g of squaric acid: squaric acid 21.16g, pyrrole 4.57g
Amount of waste: (21.16 + 4.57) – (7.90 + 16.07) = 1.76g

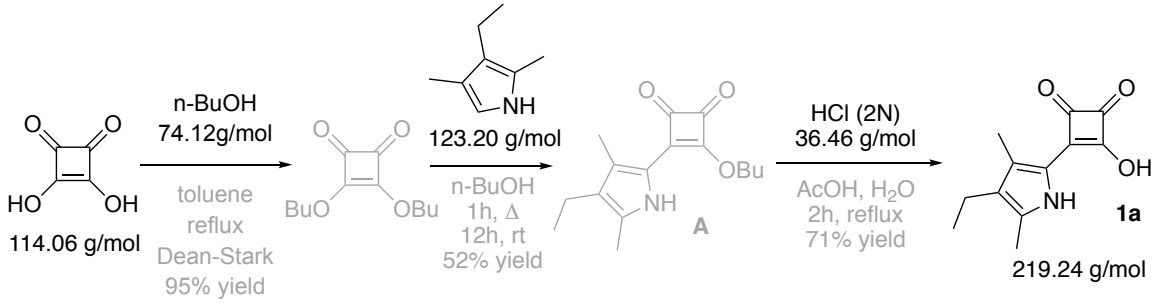
$$\text{E-factor} = \frac{1.76}{7.90} = 0.22$$

ATOM ECONOMY (AE)

$$AE = \frac{\text{MW of product}}{\text{sum of MWs of reactants/reagents}} \times 100\ \%$$

Key features: reaction yield, excess of reactants and reagents as well as solvents are not considered [S6].

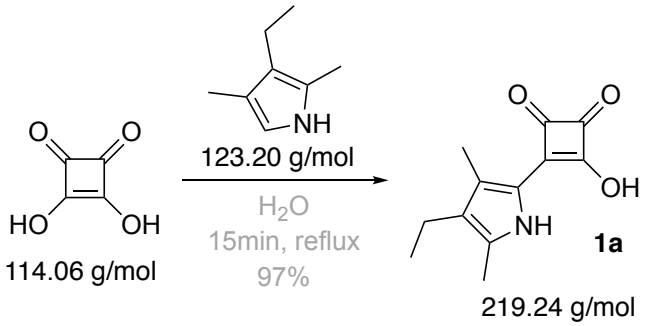
*Literature procedure for the synthesis of **1a** [S3]:*



$$AE = \frac{219.24}{114.06 + 74.12 + 123.20 + 36.46} \times 100\% = 63.0\%$$

Step 1 was accomplished according to literature procedure [S4].

*Our procedure for synthesis of **1a**:*



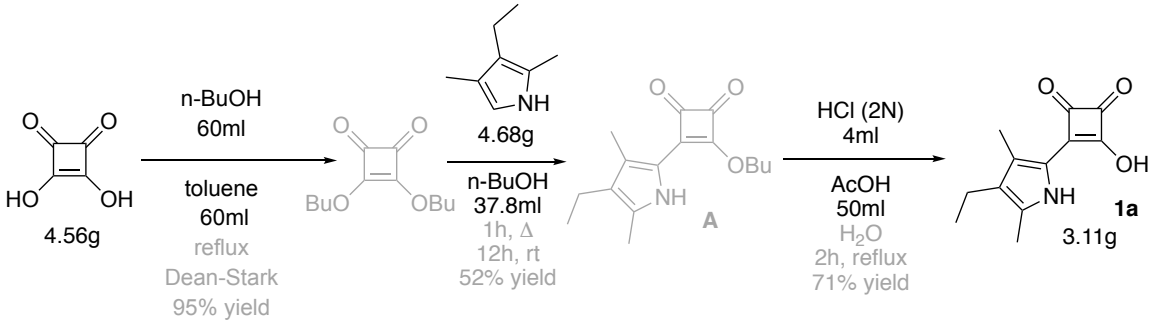
$$AE = \frac{219.24}{114.06 + 123.20} \times 100\% = 92.4\%$$

MASS INTENSITY (MI)

$$MI = \frac{\text{total mass used in the process}}{\text{mass of product}}$$

Key features: yields, mass of all reagents and solvents (including "acid, base, salt and organic solvent washes, and organic solvents used for extractions, crystallizations, or for solvent switching") are taken into consideration.[S6] However, water is excluded from mass calculations due to somewhat insignificant impact on the environment and also because "it skews mass data in many processes".[S6] Process MI (PMI) accounts for all solvents, including water, see next subsection.

*Literature procedure for **1a** [S3]:*



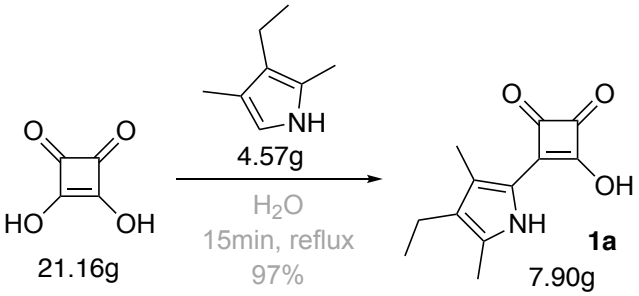
Step 1 was accomplished according to literature procedure [S4]. Step 3 leading to **1a** was done on 0.02mol scale; thus, the amounts of the reagents, solvents, etc used in steps 1 and 2 were recalculated to this scale, with considerations of the yields in each step.

Step 1: squaric acid (**4.56g**, 0.040mol), n-BuOH (**48.60g**, 60mL), toluene (**52.02g**, 60mL)
Step 2: kryptopyrrole (**4.68g**, 0.038mol), n-BuOH (**30.65g**, 37.80mL); AcOH used for crystallization is not accounted for, because the amounts were not reported.
Step 3: AcOH (**52.50g**, 50mL), HCl (**4.00g**, 4mL of 2N solution)

Amount of product **1a**: 3.11g

$$MI = \frac{4.56 + 86.6 + 52.02 + 4.68 + 30.65 + 52.50 + 4.00}{3.11} = 75.6$$

*Our procedure for **1a**:*



a) MI calculations without accounting for recovered squaric acid:
Step 1: squaric acid (**21.16g**, 0.186mol), kryptopyrrole (**4.57g**, 0.037mol).
Amount of product **1a**: 7.90g

$$MI = \frac{21.16 + 4.75}{7.90} = 3.3$$

b) MI calculations accounting for recovered squaric acid:
Step 1: squaric acid (**21.16g**, 0.186mol), kryptopyrrole (**4.57g**, 0.037mol).
Recovery step: charcoal (**10.00g**), acetone (**39.20g**, 50mL)
Amount of product **1a**: 7.90g; amount of recovered squaric acid: 16.07g

$$MI = \frac{21.16 + 4.75 + 10.00 + 39.2}{7.90 + 16.07} = 3.1$$

It is of interest to point out that the use of acetone and charcoal during the recovery step of squaric acid, indicates that recovery of squaric acid may not be beneficial, if judged strictly by the numerical value of MI. Arguably, other factors should be taken into account, especially in regard to costs of squaric acid, charcoal and acetone (for illustrative purposes only, see the table below).

	Squaric acid	**Acetone**	**Charcoal**
Vendor	TCI (America)	Fisher Scientific	Fisher Scientific
Catalog #	D1399	AC423240250	AC124370250
Quantity[a]	25g	25L	25kg
Price of the unit / $	165.00	746.50	873.50
Price: $/mol	**753.00**	**2.00**	**0.42**

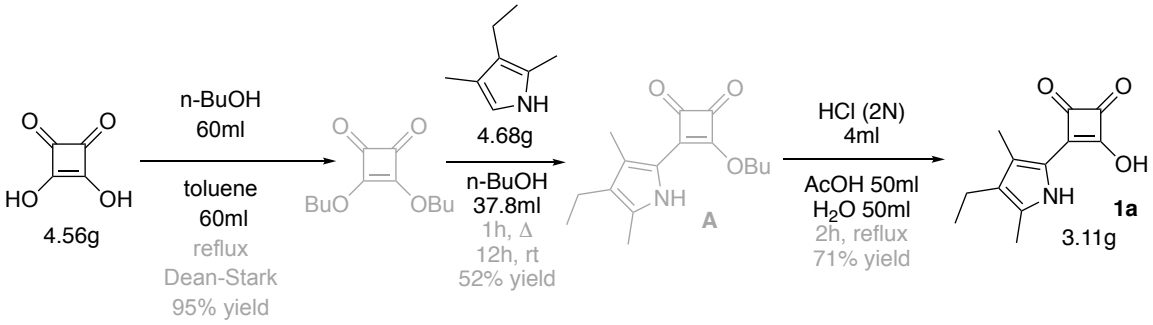
[a] – largest quantity available for sale was considered.

PROCESS MASS INSTENSITY (PMI)

$$PMI = \frac{\text{total mass used in the process (incl. } H_2O)}{\text{mass of product}}$$

Key features: yields, mass of all reagents and solvents, including water (!), is taken into consideration.[S6]

*Literature procedure for **1a** [#]:*



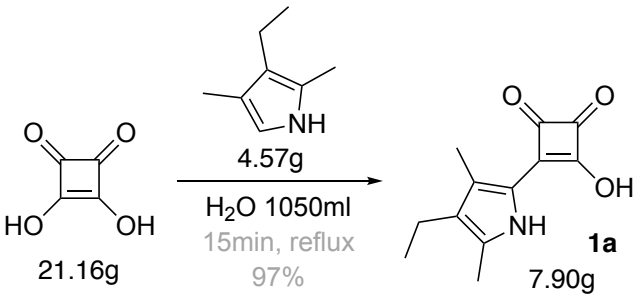
Step 1 was accomplished according to literature procedure [S4].

Step 1: squaric acid (**4.56g**, 0.040mol), n-BuOH (**48.60g**, 60mL), toluene (**52.02g**, 60mL)
Step 2: kryptopyrrole (**4.68g**, 0.038mol), n-BuOH (**30.65g**, 37.80mL); AcOH used for crystallization is not accounted for, because the amounts were not reported.
Step 3: AcOH (**52.50g**, 50mL), H_2O (**50.00g**, 50mL), HCl (**4.00g**, 4mL of 2N solution). **1a** was isolated by filtration from the reaction mixture, and washed with ether, yet the volume of ether was not reported; thus, it was not included in the calculation of PMI.

Amount of product **1a**: 3.11g

$$PMI = \frac{4.56 + 86.6 + 52.02 + 4.68 + 30.65 + 52.45 + 50.00 + 4.00}{3.11} = 82.3$$

*Our procedure for **1a**:*



a) PMI calculations without accounting for recovered squaric acid:

Step 1: squaric acid (**21.16g**, 0.186mol), kryptopyrrole (**4.57g**, 0.037mol), H_2O (**1000.00g**, 100mL were used in the synthesis, 900mL were used during the isolation of **1a**).

Amount of product **1a**: 7.90g

$$\text{PMI} = \frac{21.16 + 4.57 + 1000.00}{7.90} = 129.8$$

b) PMI calculations accounting for recovered squaric acid:

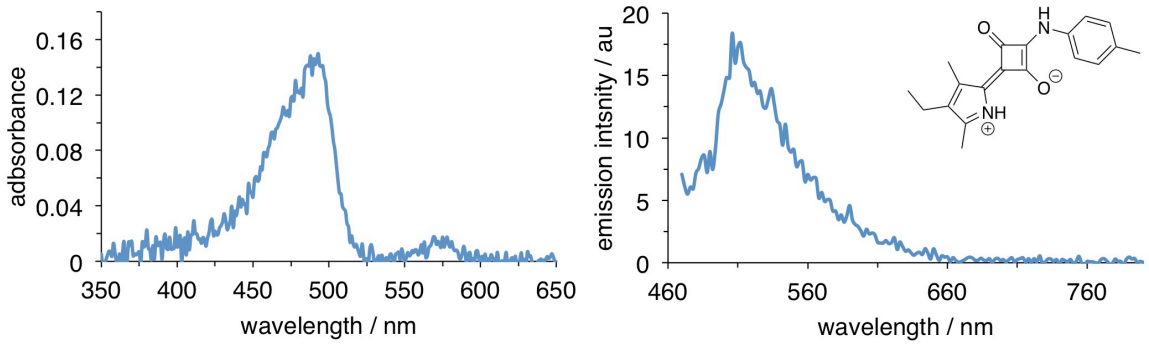
Step 1: squaric acid (**21.16g**, 0.186mol), kryptopyrrole (**4.57g**, 0.037mol), H_2O (**1000.00g**, 100mL were used in the synthesis, 900mL were used during the isolation of **1a**).

Recovery step: charcoal (**10.00g**), acetone (**39.20g**, 50mL)

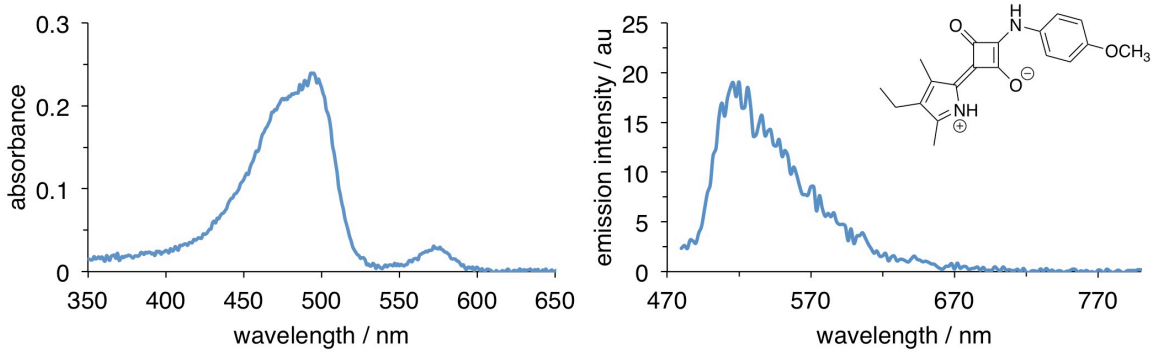
Amount of product **1a**: 7.90g; amount of recovered squaric acid: 16.07g

$$\text{PMI} = \frac{21.16 + 4.57 + 1000.00 + 39.20 + 10.00}{7.90 + 16.07} = 44.8$$

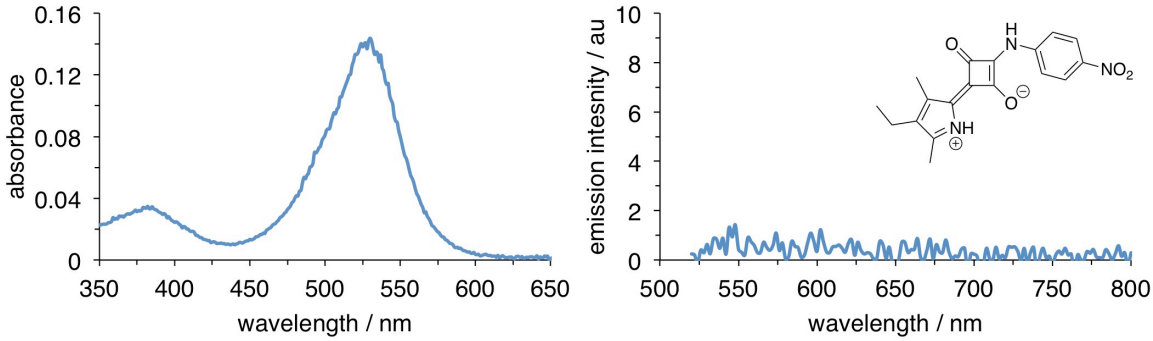
UV-VIS AND FLUORESCENCE EMISSION SPECTRA OF DYES 6 – 18 in DMSO



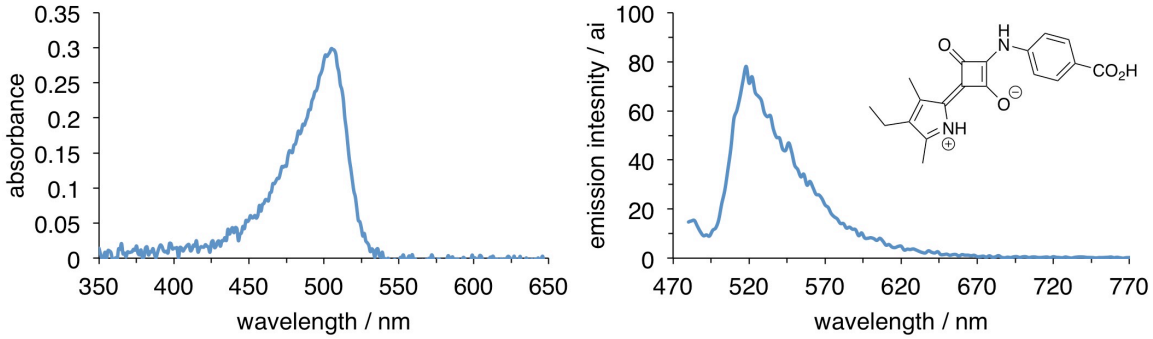
UV-Vis (left) and fluorescence emission (right) spectra of dye **6**.
Conditions: [**6**] = 2 μM, λex = 450 nm.



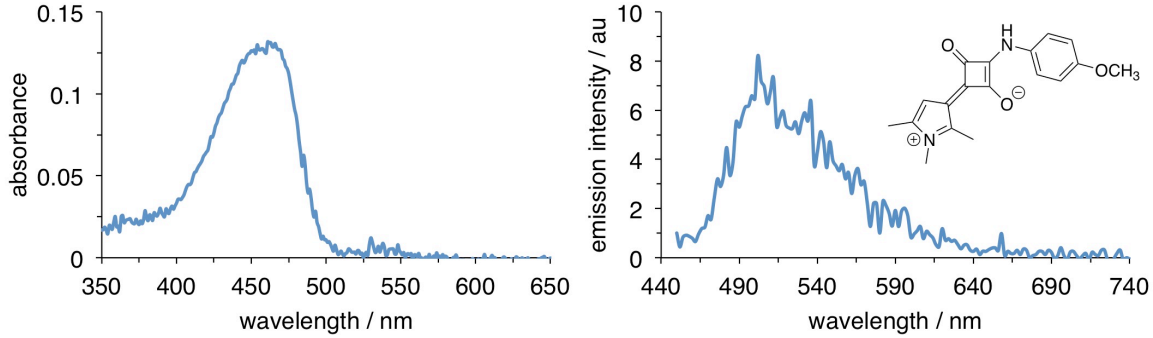
UV-Vis (left) and fluorescence emission (right) spectra of dye **7**.
Conditions: [**7**] = 6 μM, λex = 470 nm.



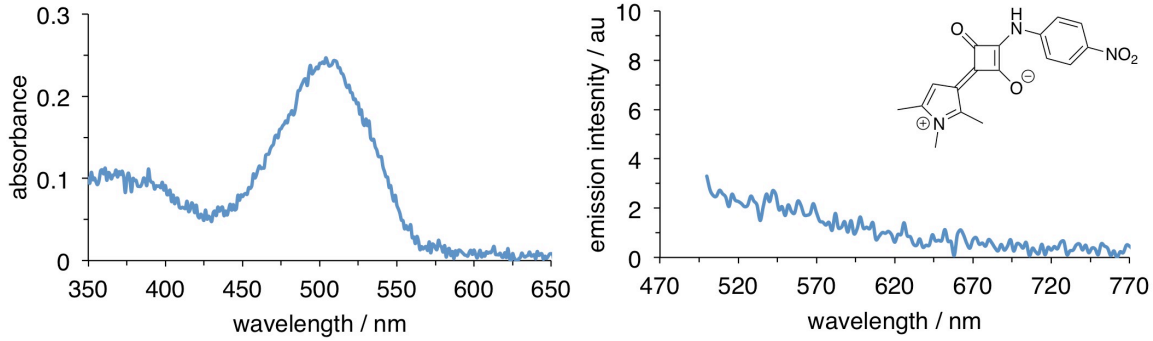
UV-Vis (left) and fluorescence emission (right) spectra of dye **8**.
Conditions: [**8**] = 10 μM, λex = 510 nm.



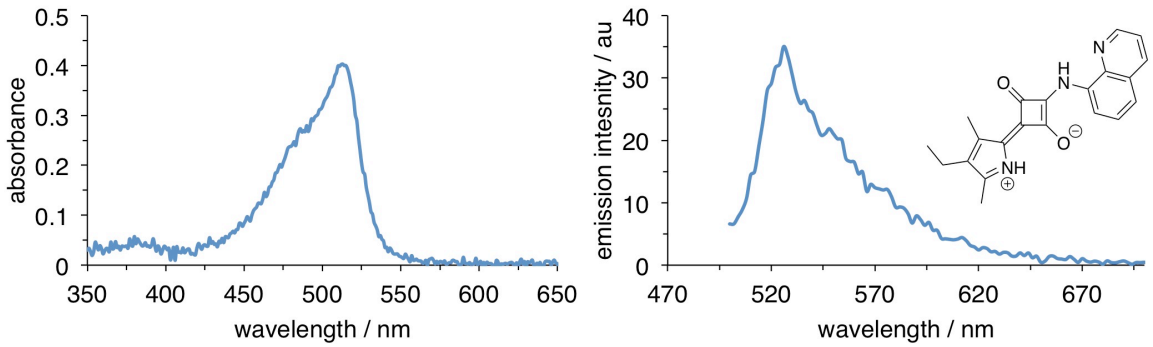
UV-Vis (left) and fluorescence emission (right) spectra of dye **9**.
Conditions: [**9**] = 4 μM, λex = 470 nm.



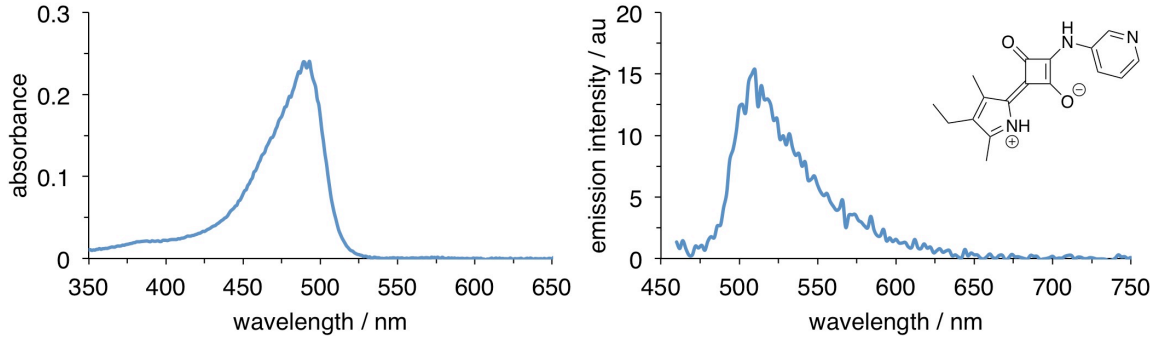
UV-Vis (left) and fluorescence emission (right) spectra of dye **10**.
Conditions: [**10**] = 4 μM, λex = 440 nm.



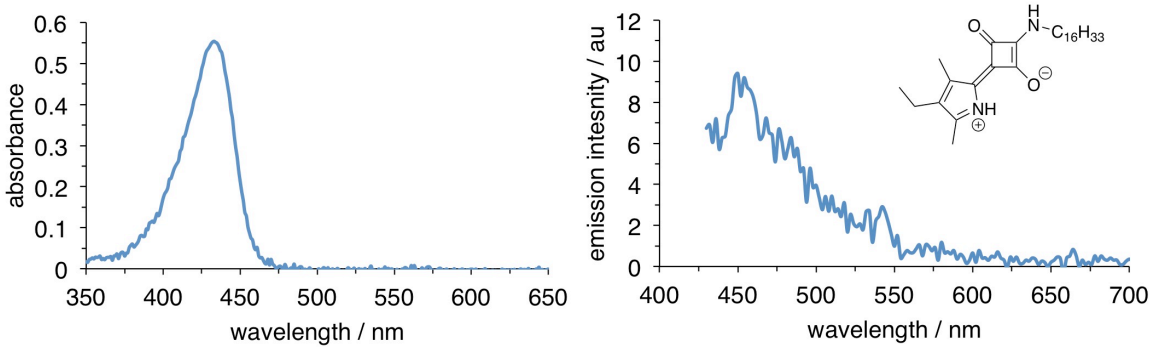
UV-Vis (left) and fluorescence emission (right) spectra of dye **11**.
Conditions: [**11**] = 8 μM, λex = 470 nm.



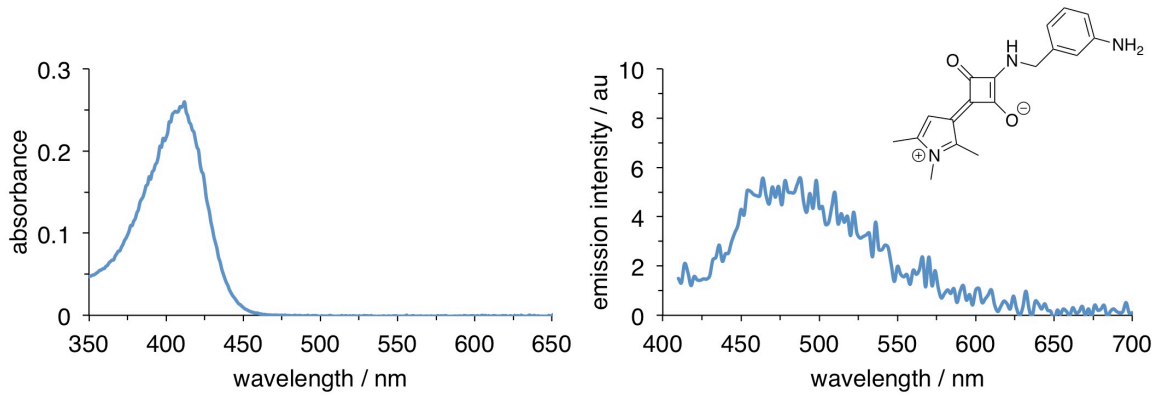
UV-Vis (left) and fluorescence emission (right) spectra of dye **12**.
Conditions: [**12**] = 6 μM, λex = 470 nm.



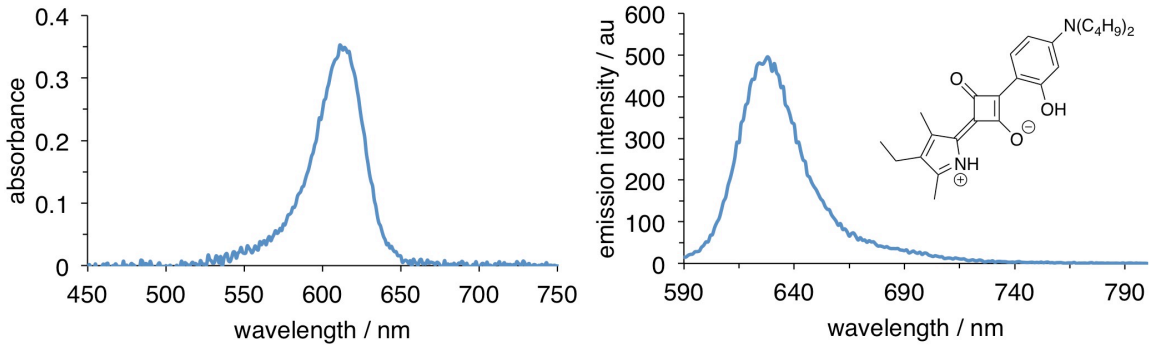
UV-Vis (left) and fluorescence emission (right) spectra of dye **13**.
Conditions: [**13**] = 4 μM, λex = 450 nm.



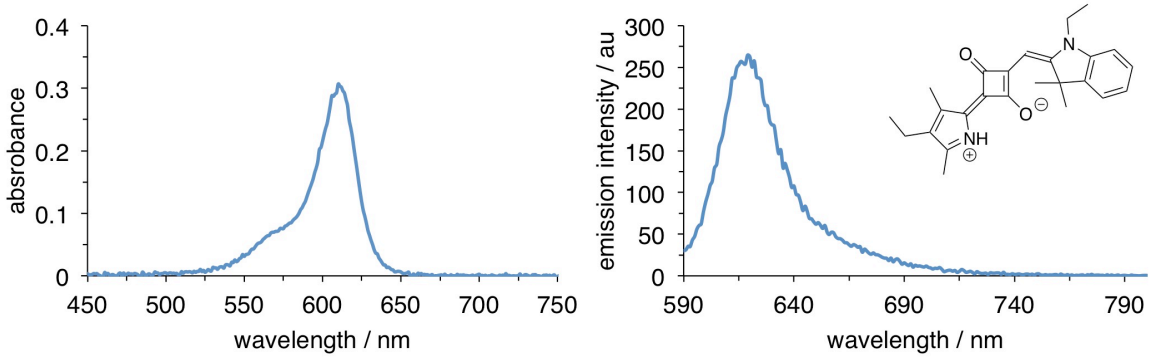
UV-Vis (left) and fluorescence emission (right) spectra of dye **14**.
Conditions: [**14**] = 8 μM, λex = 400 nm.



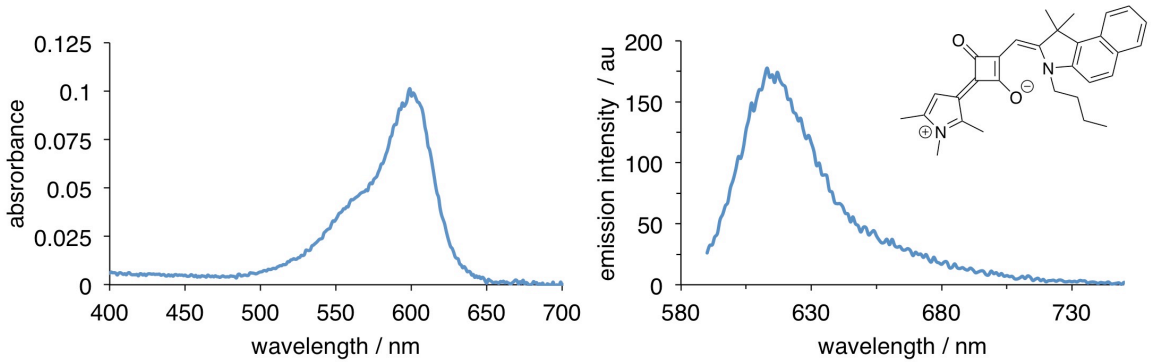
UV-Vis (left) and fluorescence emission (right) spectra of dye **15**.
Conditions: [**15**] = 8 μM, λex = 400 nm.



UV-Vis (left) and fluorescence emission (right) spectra of dye **16**.
Conditions: [**16**] = 1 μM, λex = 580 nm.

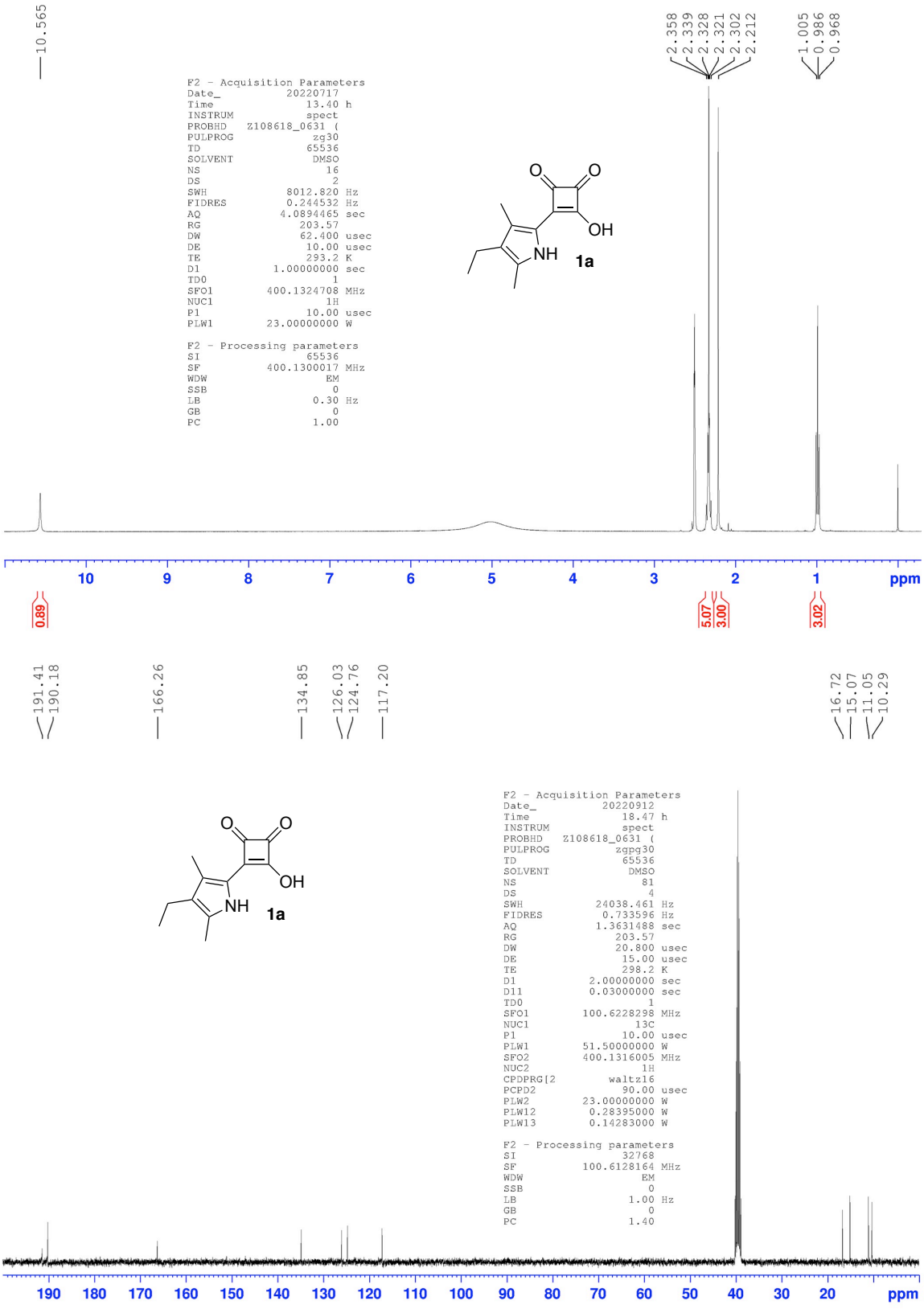


UV-Vis (left) and fluorescence emission (right) spectra of dye **17**.
Conditions: [**17**] = 3 μM, λex = 580 nm.



UV-Vis (left) and fluorescence emission (right) spectra of dye **18**.
Conditions: [**18**] = 2 μM, λex = 580 nm.

^{1}H (top) and ^{13}C (bottom) NMR SPECTRA
OF COMPOUNDS
1a, 4a, 6–18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2.339
2.328
2.321
2.302
2.212
1.005
0.986
0.968
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Date_ 20220717
Time 13.40 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300017 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
OH
NH
1a
10
9
8
7
6
5
4
3
2
1
ppm
0.89
5.07
3.00
3.02
191.41
190.18
166.26
134.85
126.03
124.76
117.20
16.72
15.07
11.05
10.29
F2 - Acquisition Parameters
Date_ 20220912
Time 18.47 h
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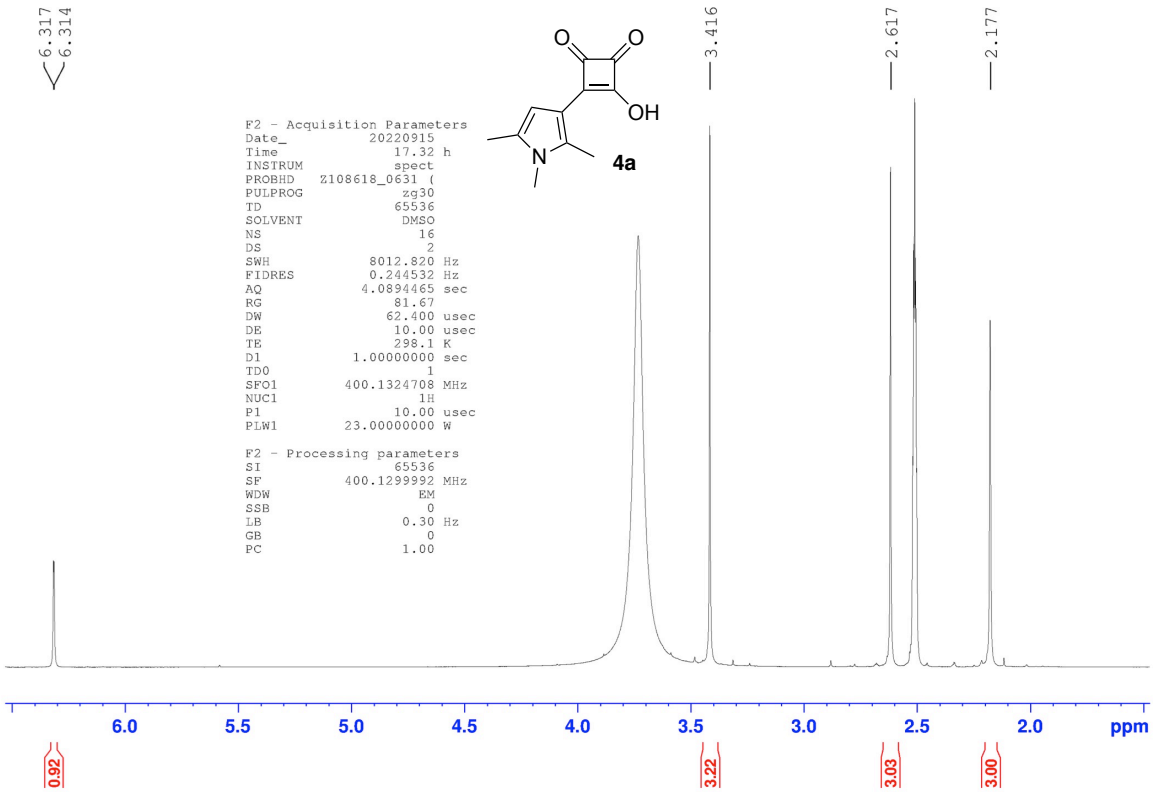
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 81
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128164 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
OH
NH
1a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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6.314
3.416
2.617
2.177
O
O
OH
N
4a
F2 - Acquisition Parameters
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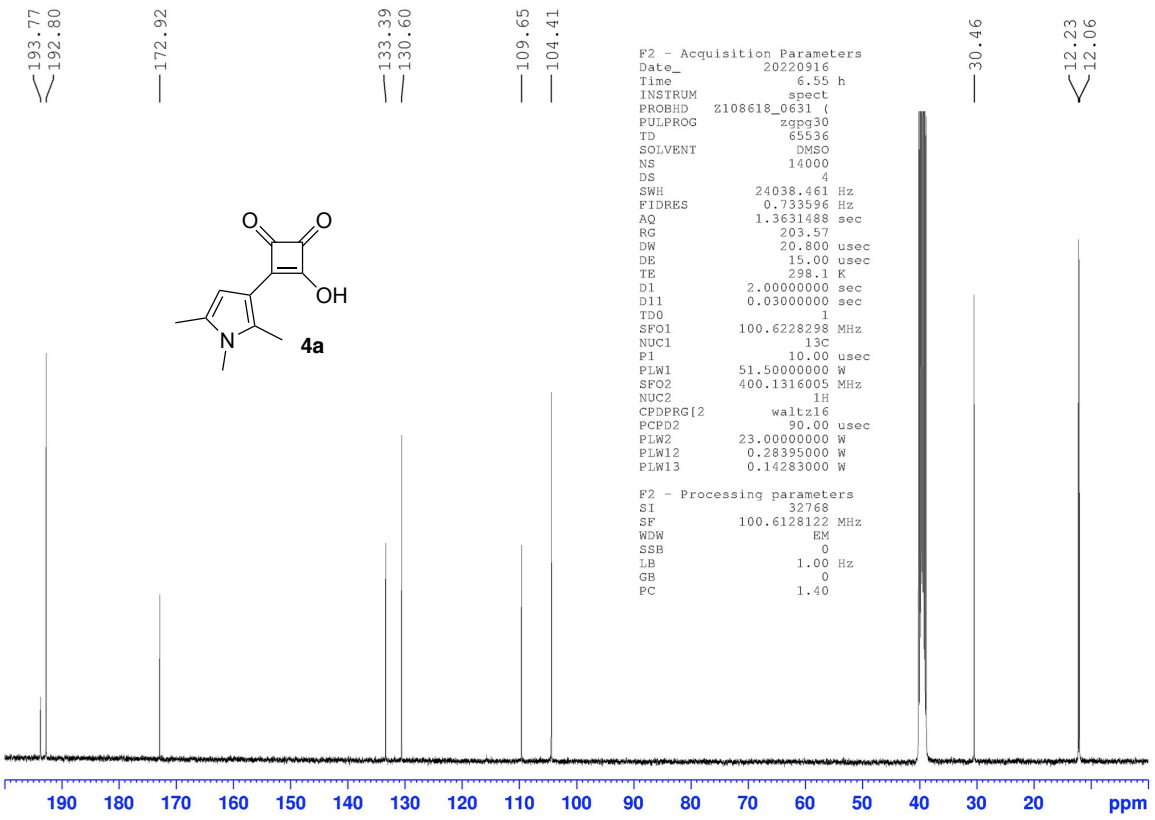
Time 17.32 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 81.67
DW 62.400 usec
DE 10.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299992 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
0.92
3.22
3.03
3.00


193.77
192.80
172.92
133.39
130.60
109.65
104.41
30.46
12.23
12.06
O
O
OH
N
4a
F2 - Acquisition Parameters
Date_ 20220916
Time 6.55 h
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PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 14000
DS 4
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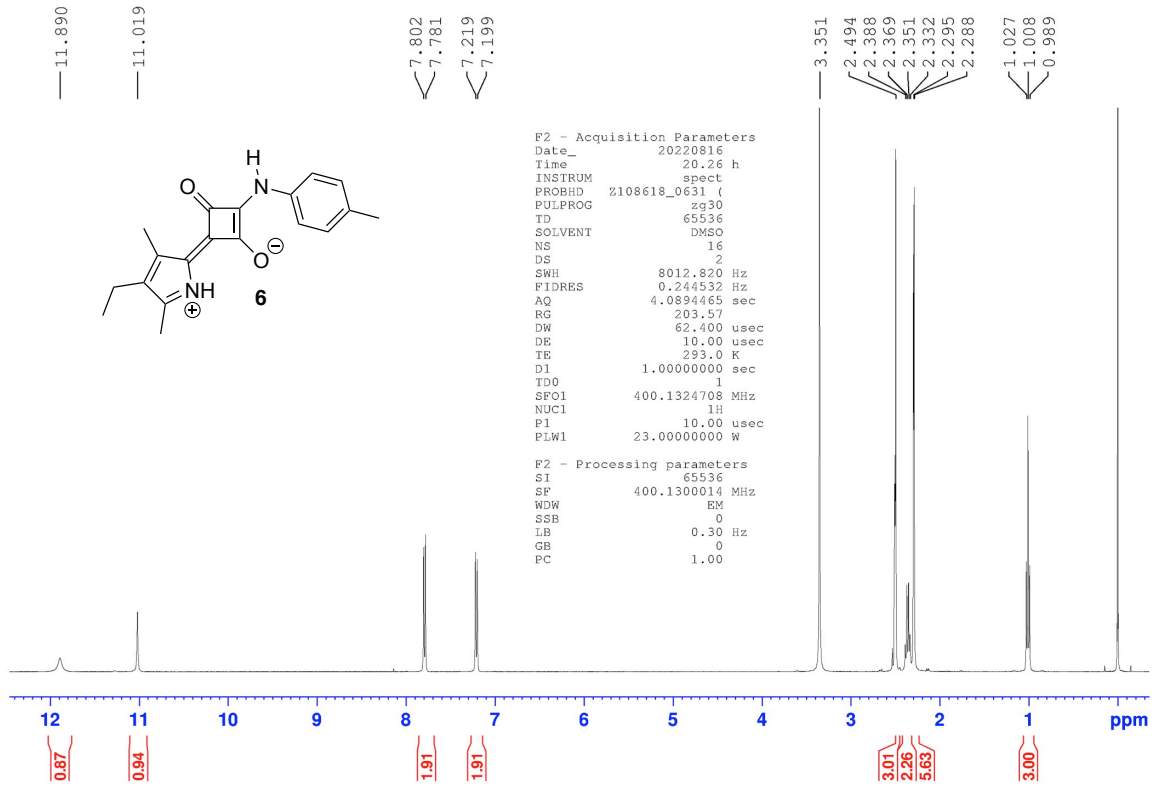
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128122 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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11.019
7.802
7.781
7.219
7.199
3.351
2.494
2.388
2.369
2.351
2.332
2.295
2.288
1.027
1.008
0.989
6
F2 - Acquisition Parameters
Date_ 20220816
Time 20.26 h
INSTRUM spect
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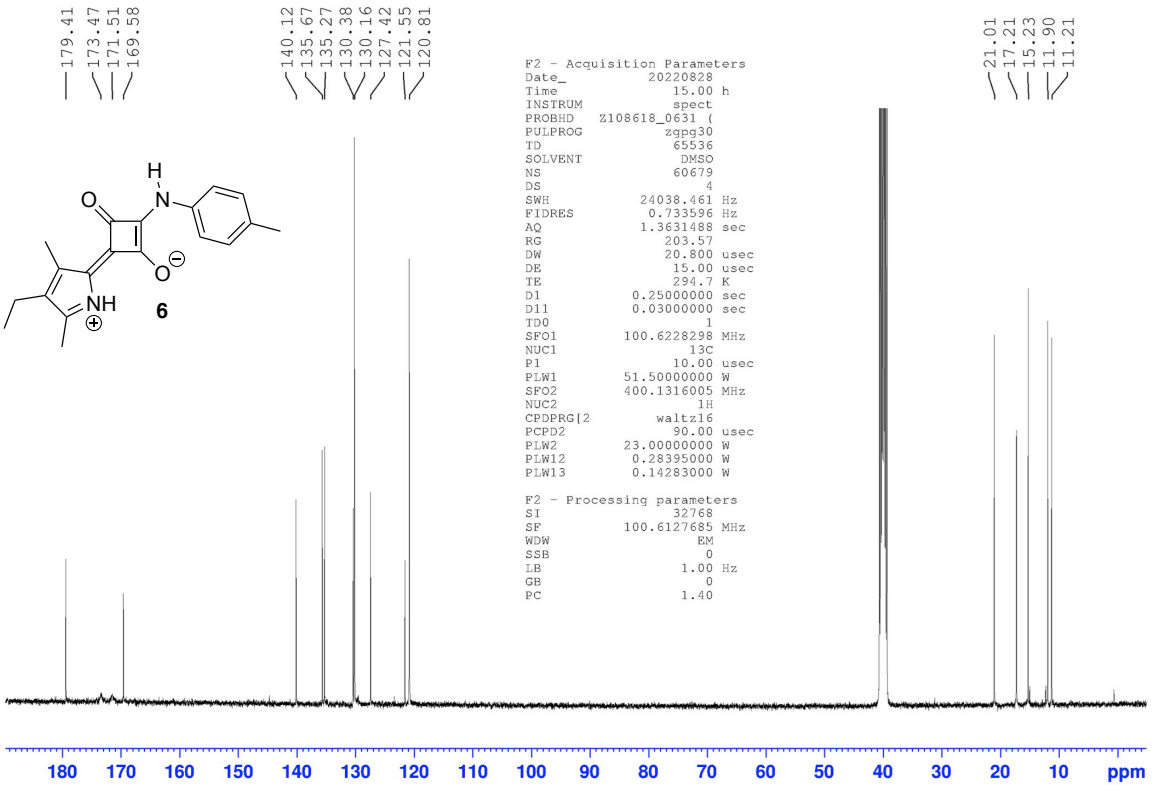
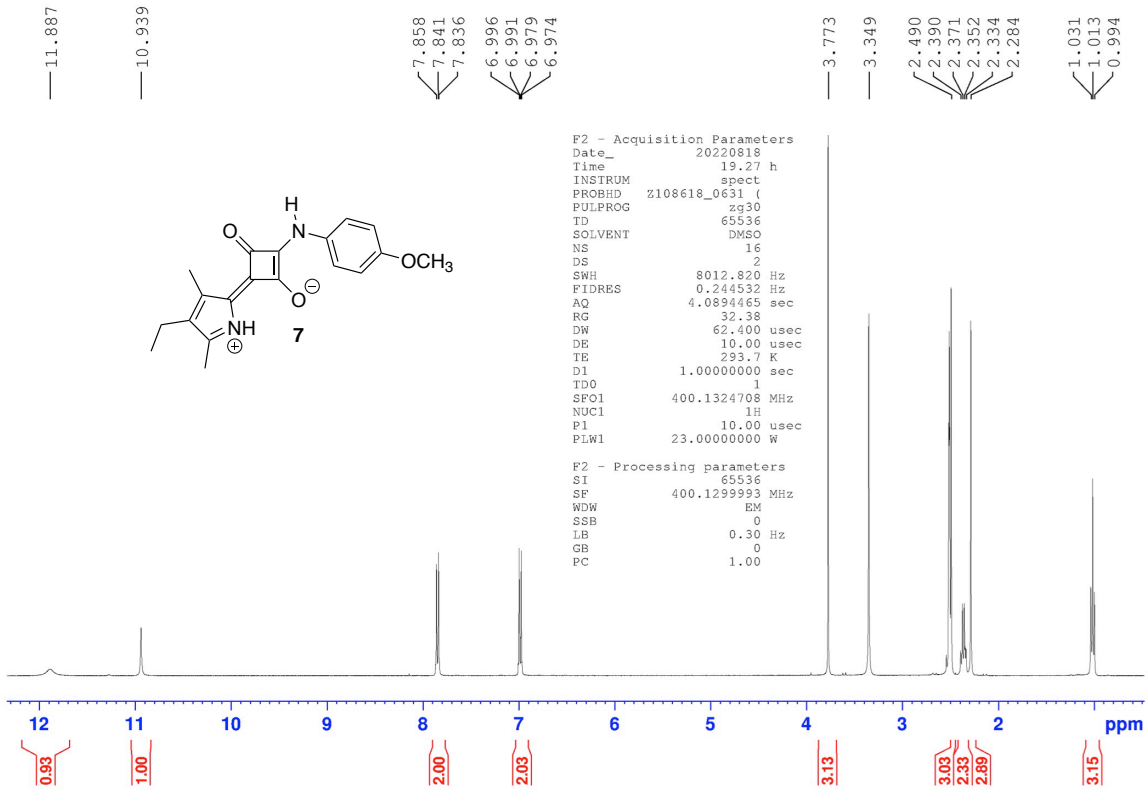
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300014 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.87
0.94
1.91
1.91
3.01
2.26
5.63
3.00


179.41
173.47
171.51
169.58
140.12
135.67
135.27
130.38
130.16
127.42
121.55
120.81
21.01
17.21
15.23
11.90
11.21
6
F2 - Acquisition Parameters
Date_ 20220828
Time 15.00 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 60679
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 294.7 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.858
7.841
7.836
6.996
6.991
6.979
6.974
3.773
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2.490
2.390
2.371
2.352
2.334
2.284
1.031
1.013
0.994
F2 - Acquisition Parameters
Date_ 20220818
Time 19.27 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 32.38
DW 62.400 usec
DE 10.00 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
N
O
OCH3
O
NH
7
12
11
10
9
8
7
6
5
4
3
2
ppm
0.93
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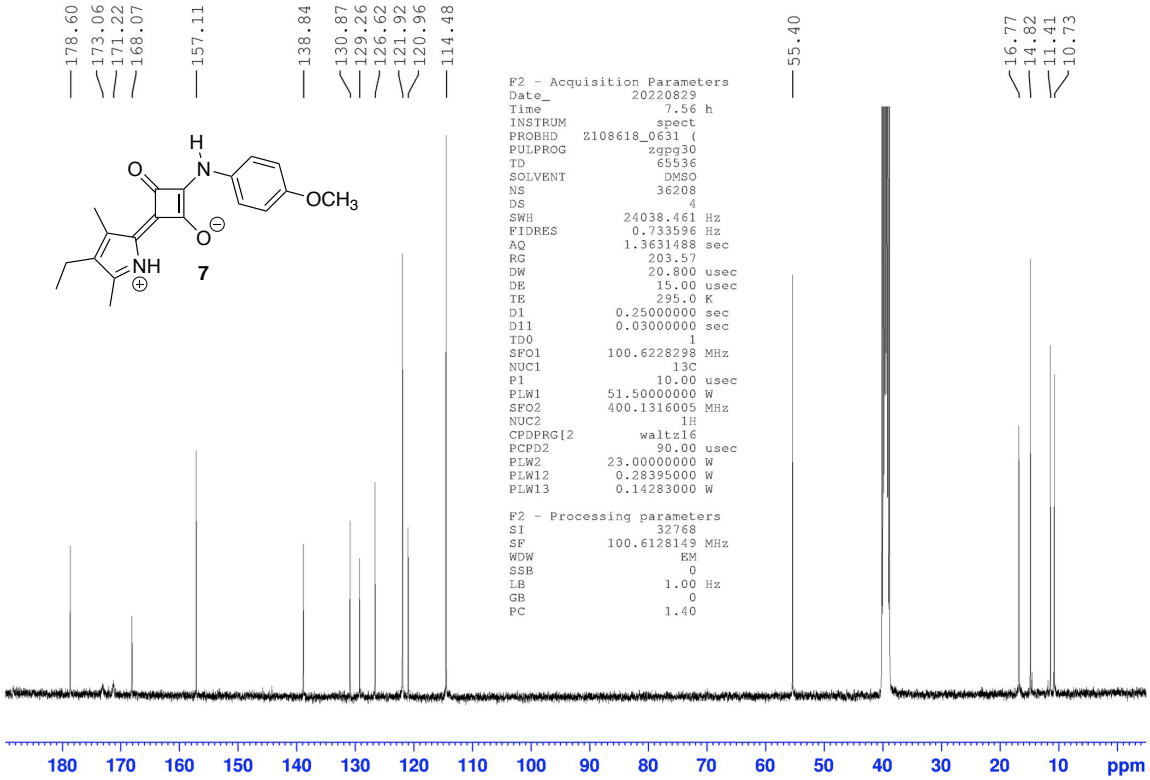
2.00
2.03
3.13
3.03
2.33
2.89
3.15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173.06
171.22
168.07
157.11
138.84
130.87
129.26
126.62
121.92
120.96
114.48
55.40
16.77
14.82
11.41
10.73
F2 - Acquisition Parameters
Date_ 20220829
Time 7.56 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 36208
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 295.0 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128149 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H
N
O
OCH3
O
NH
7
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150
140
130
120
110
100
90
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70
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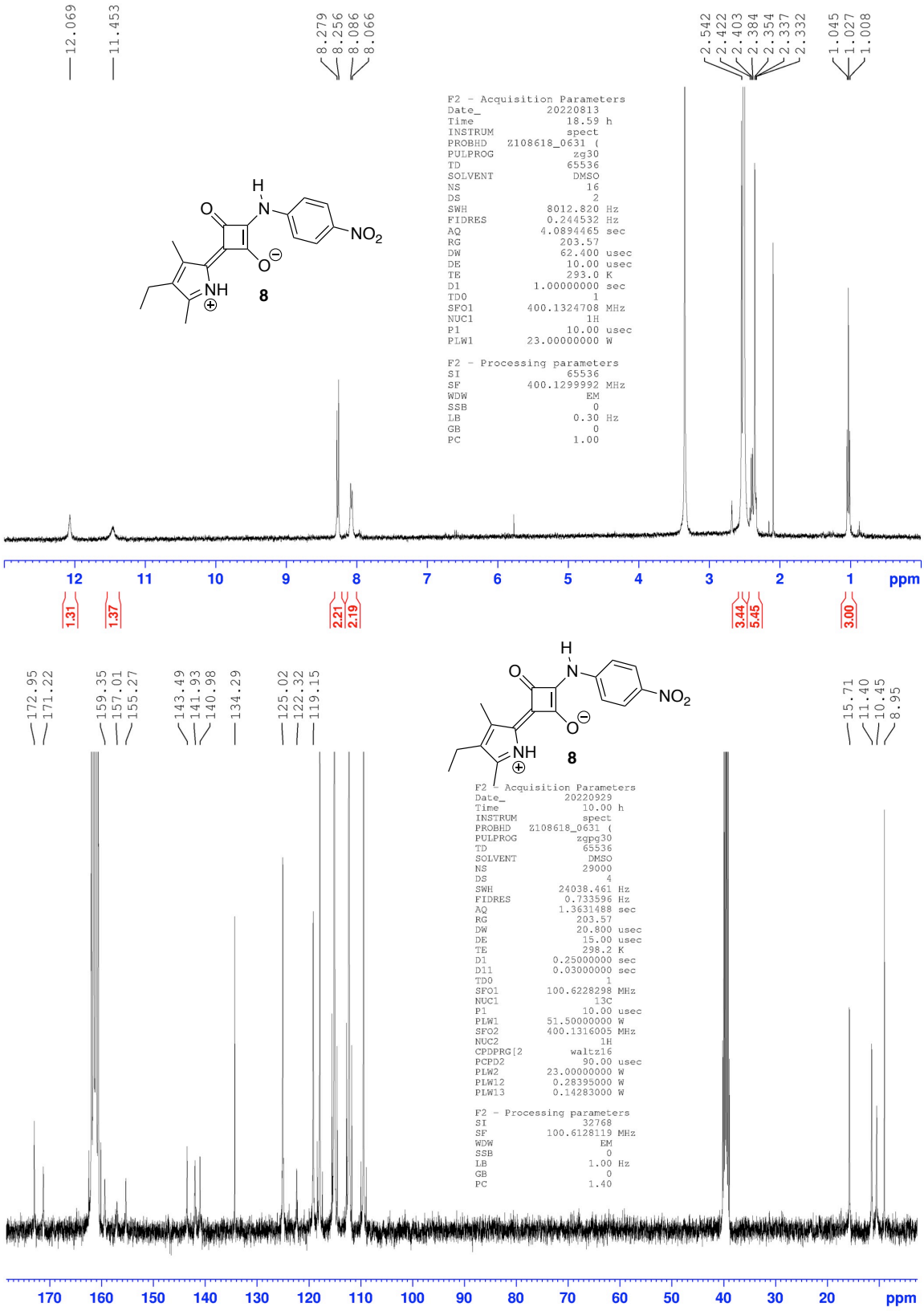
50
40
30
20
10
ppm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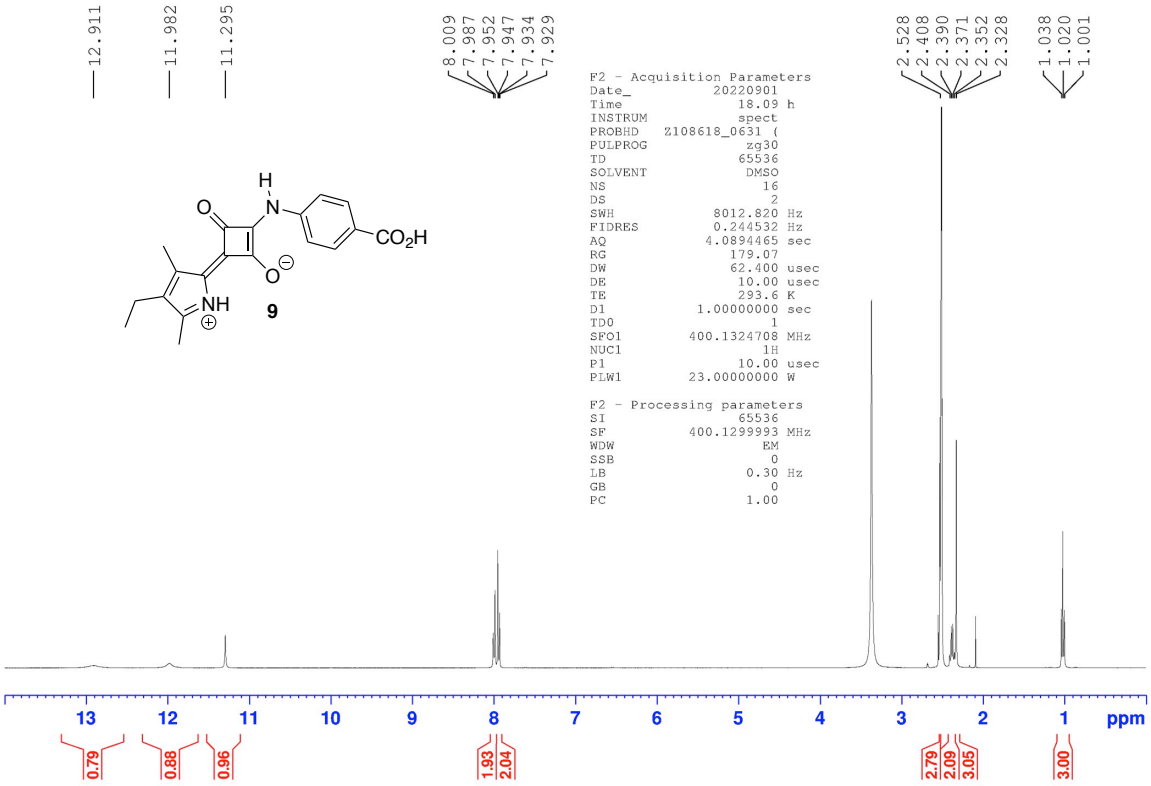
8.279
8.256
8.086
8.066
2.542
2.422
2.403
2.384
2.354
2.337
2.332
1.045
1.027
1.008
F2 - Acquisition Parameters
Date_ 20220813
Time 18.59 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299992 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.31
1.37
2.21
2.19
3.44
5.45
3.00
172.95
171.22
159.35
157.01
155.27
143.49
141.93
140.98
134.29
125.02
122.32
119.15
15.71
11.40
10.45
8.95
F2 - Acquisition Parameters
Date_ 20220929
Time 10.00 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 29000
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 298.2 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128119 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
8
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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11.982
11.295
8.009
7.987
7.952
7.947
7.934
7.929
2.528
2.408
2.390
2.371
2.352
2.328
1.038
1.020
1.001
F2 - Acquisition Parameters
Date_ 20220901
Time 18.09 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 179.07
DW 62.400 usec
DE 10.00 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
CO2H
NH
9
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
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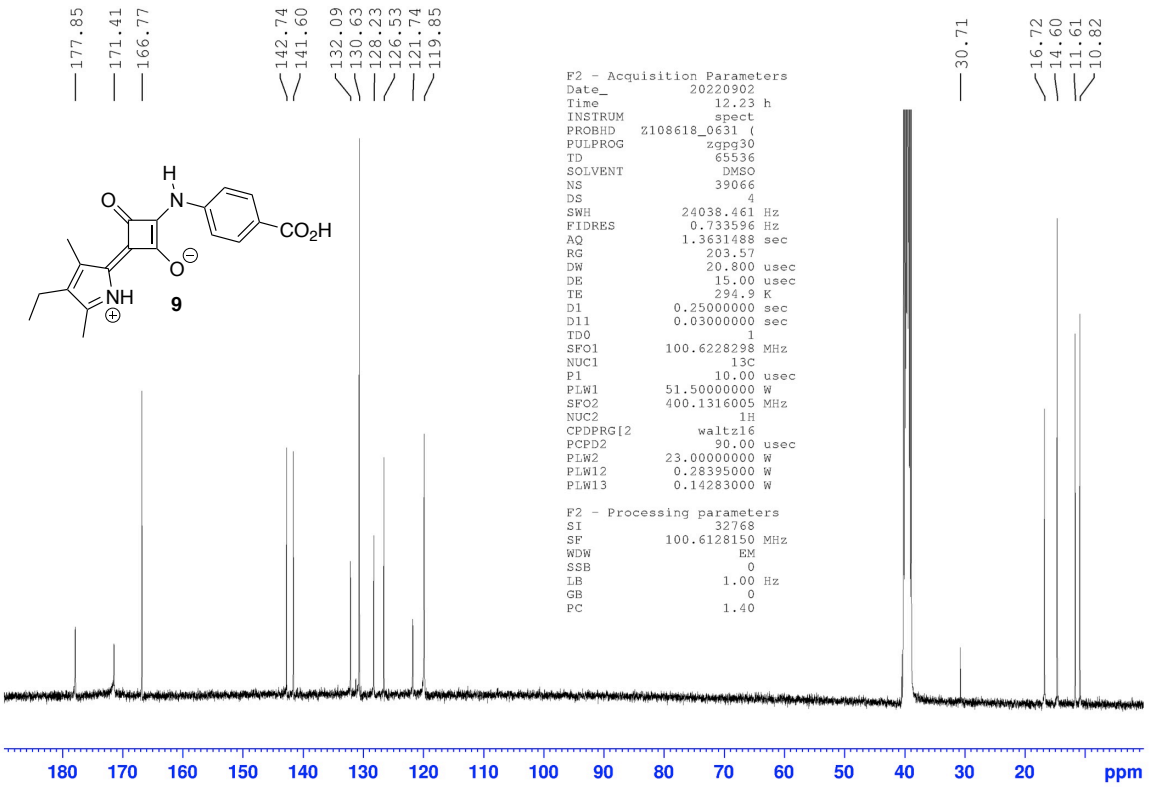
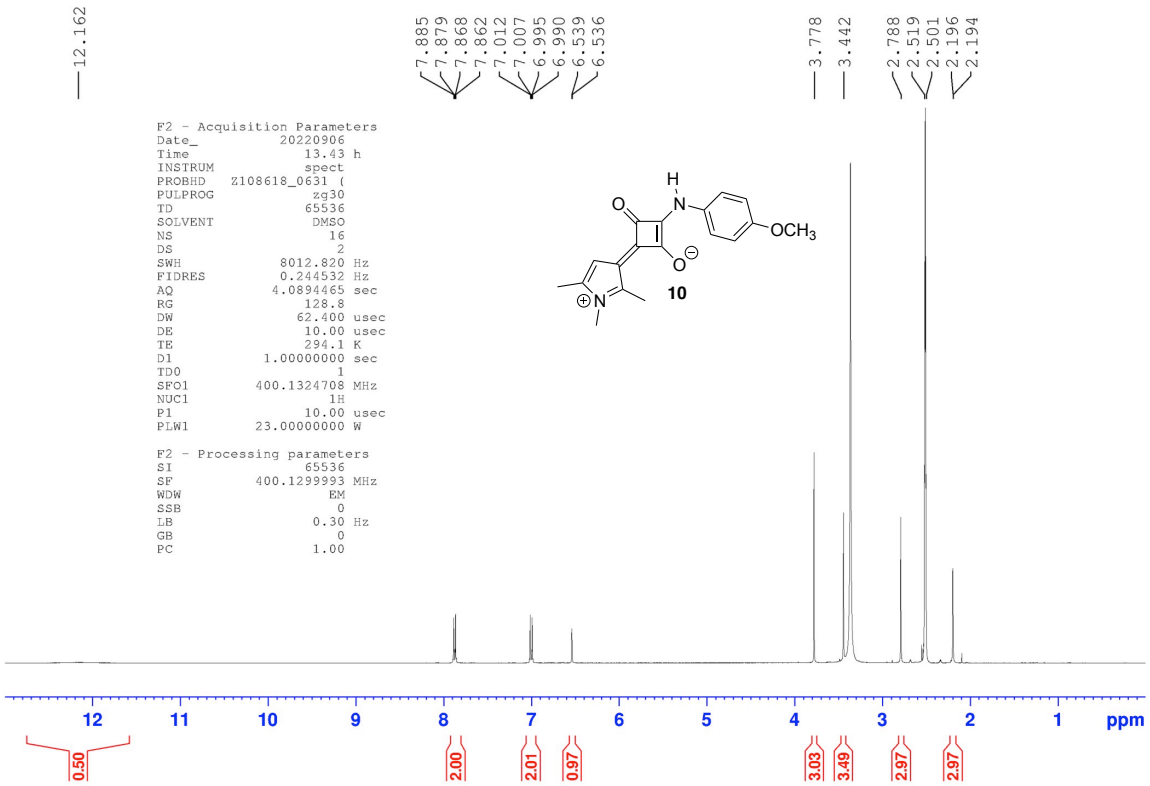
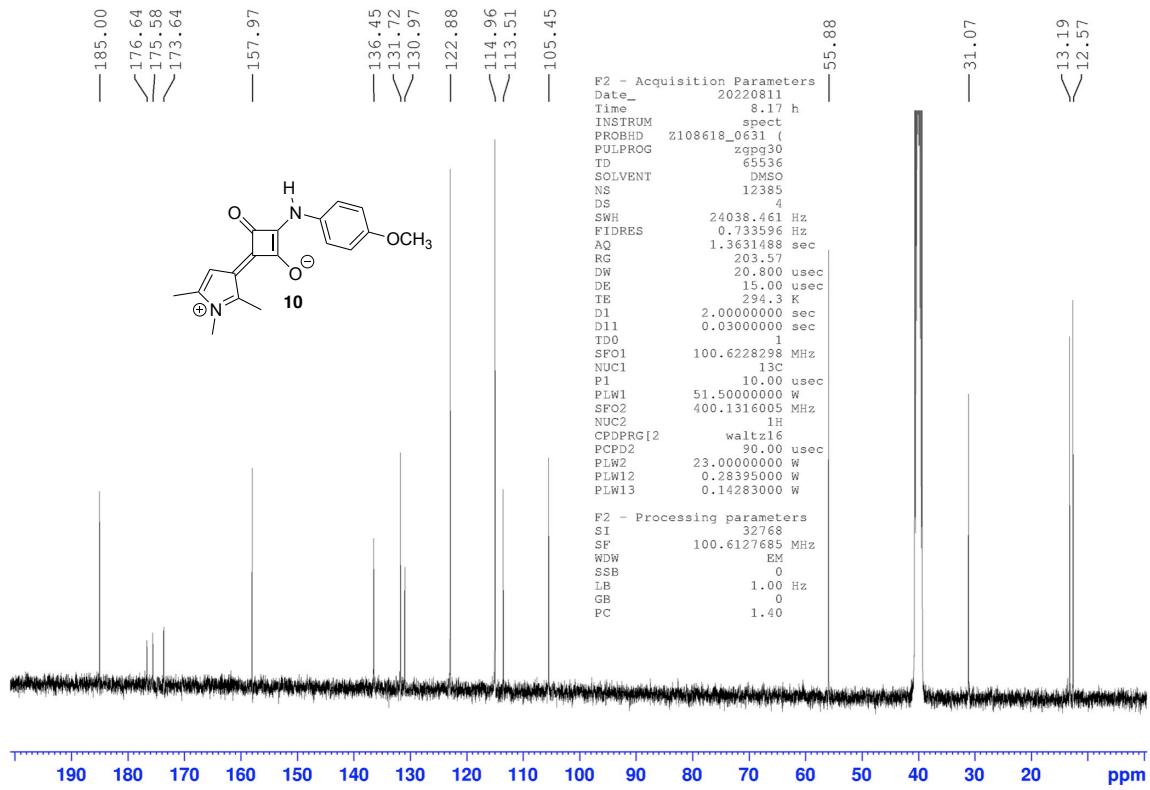
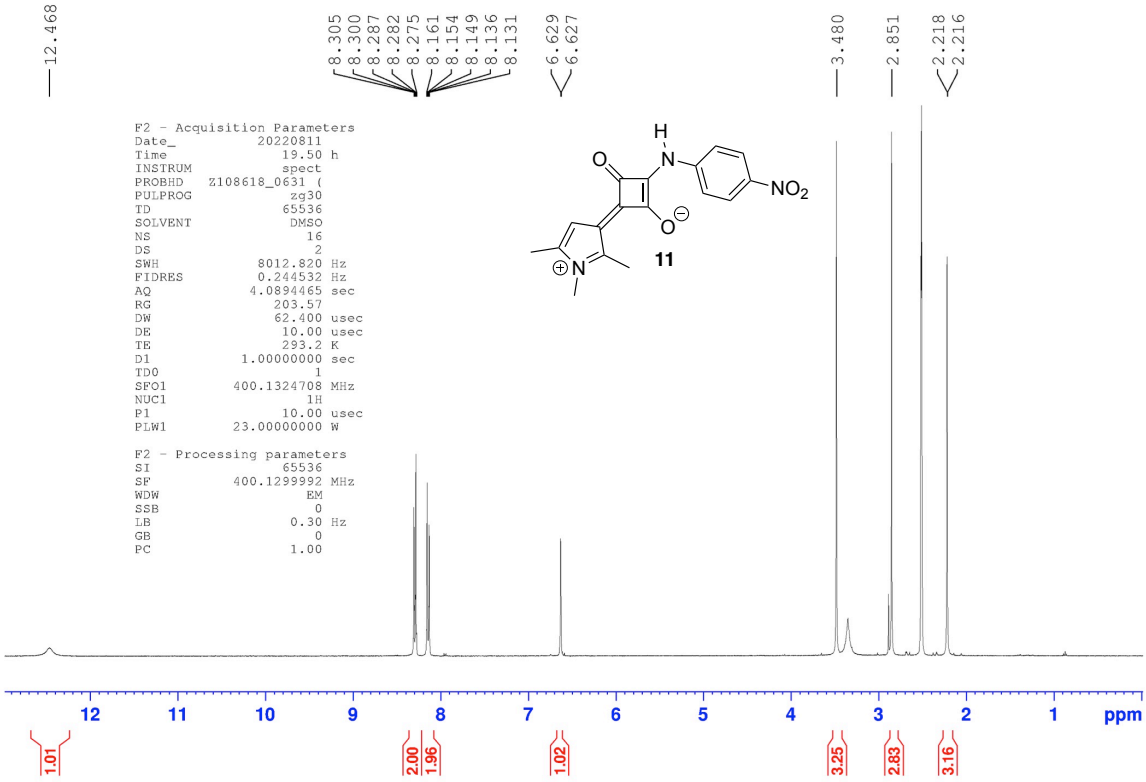
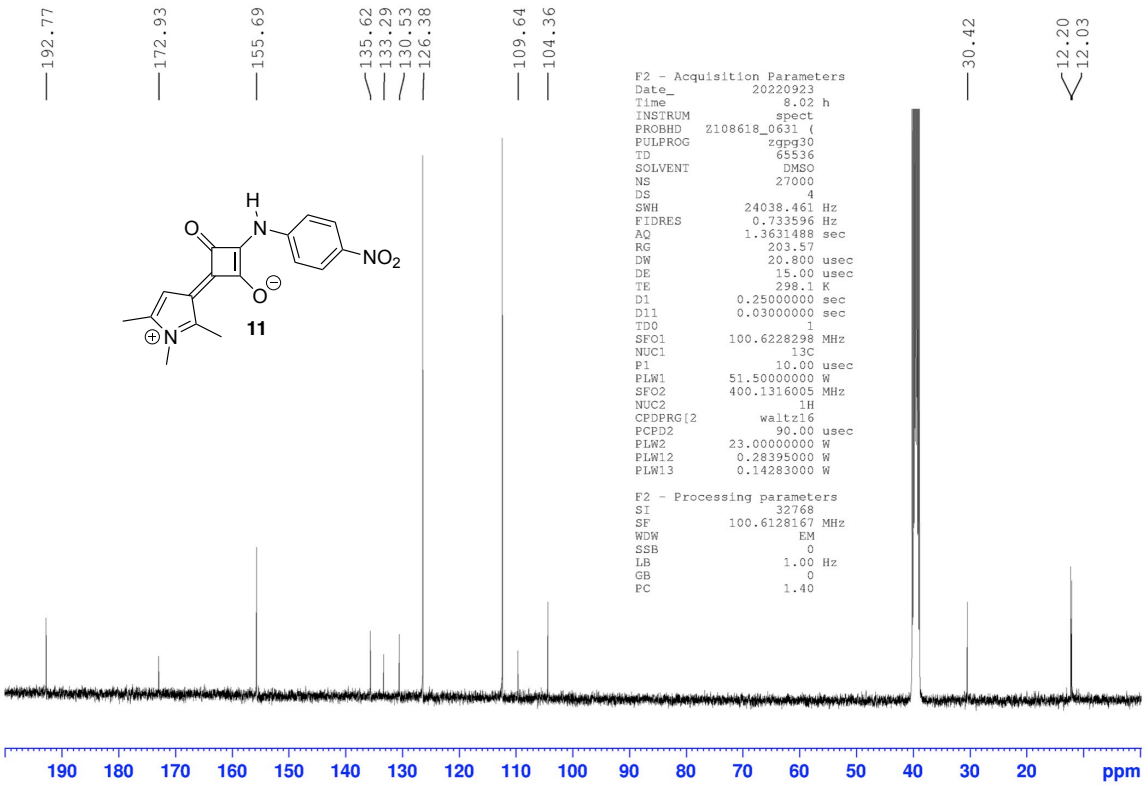
2.04
2.79
2.09
3.05
3.00

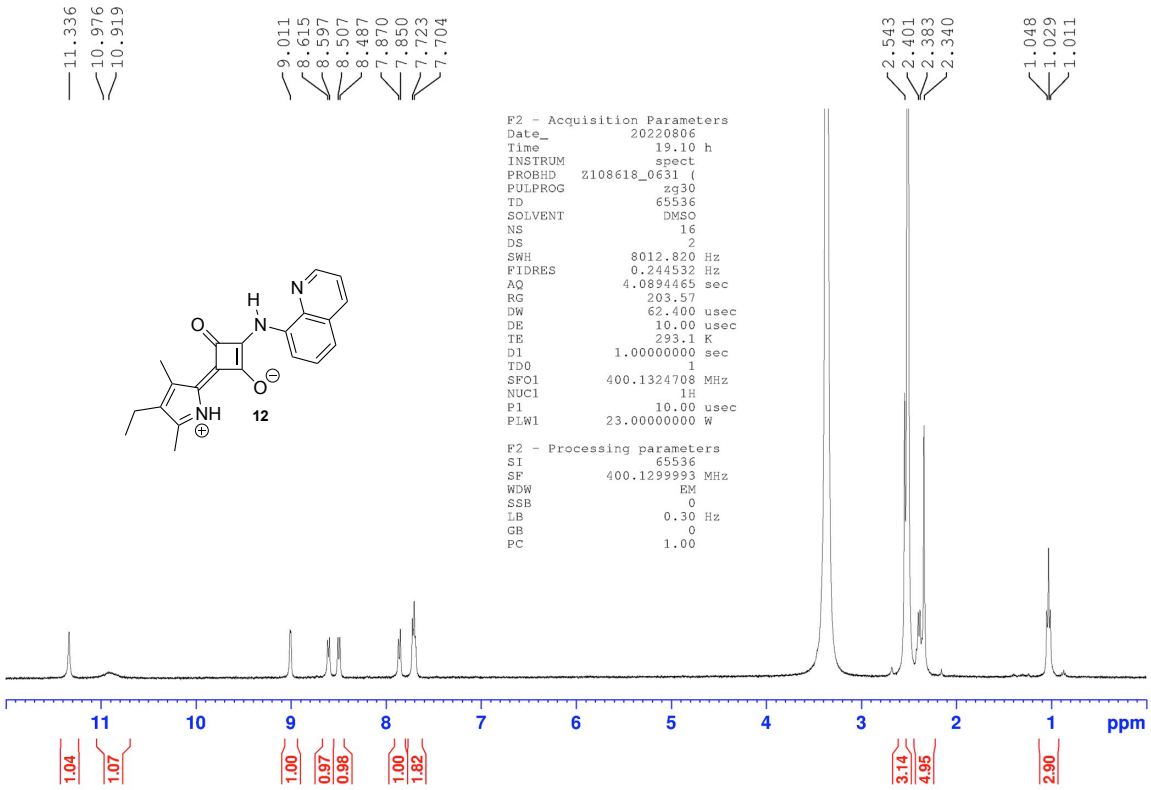

177.85
171.41
166.77
142.74
141.60
132.09
130.63
128.23
126.53
121.74
119.85
30.71
16.72
14.60
11.61
10.82
F2 - Acquisition Parameters
Date_ 20220902
Time 12.23 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 39066
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 294.9 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128150 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
CO2H
NH
9
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

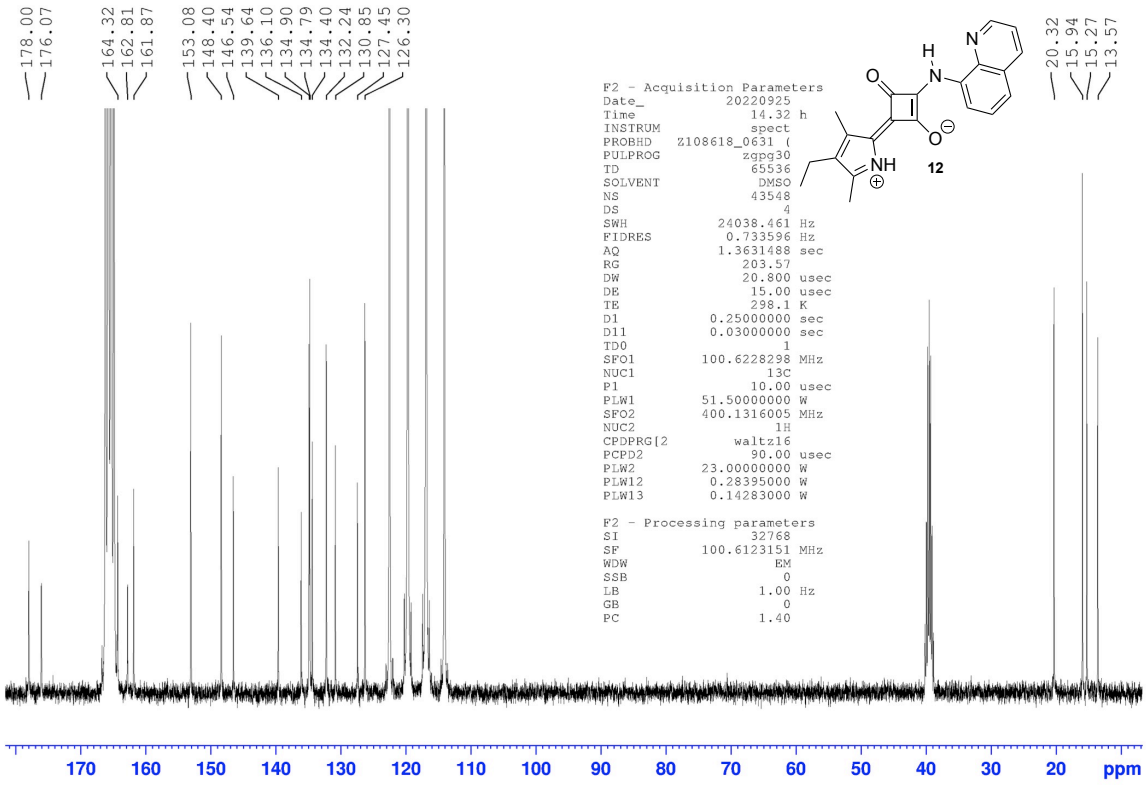

12.162
7.885
7.879
7.868
7.862
7.012
7.007
6.995
6.990
6.539
6.536
3.778
3.442
2.788
2.519
2.501
2.196
2.194
F2 - Acquisition Parameters
Date_ 20220906
Time 13.43 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 128.8
DW 62.400 usec
DE 10.00 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.50 2.00 2.01 0.97 3.03 3.49 2.97 2.97

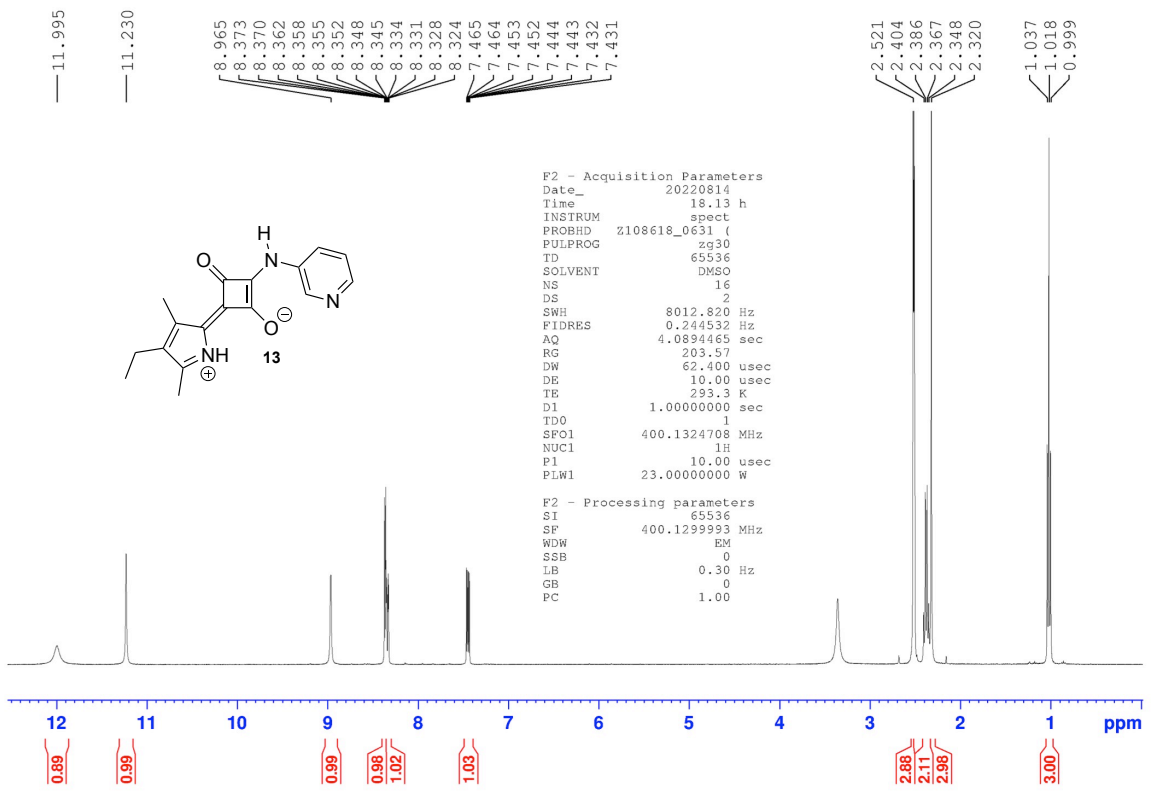

185.00
176.64
175.58
173.64
157.97
136.45
131.72
130.97
122.88
114.96
113.51
105.45
55.88
31.07
13.19
12.57
F2 - Acquisition Parameters
Date_ 20220811
Time 8.17 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 12385
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 294.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

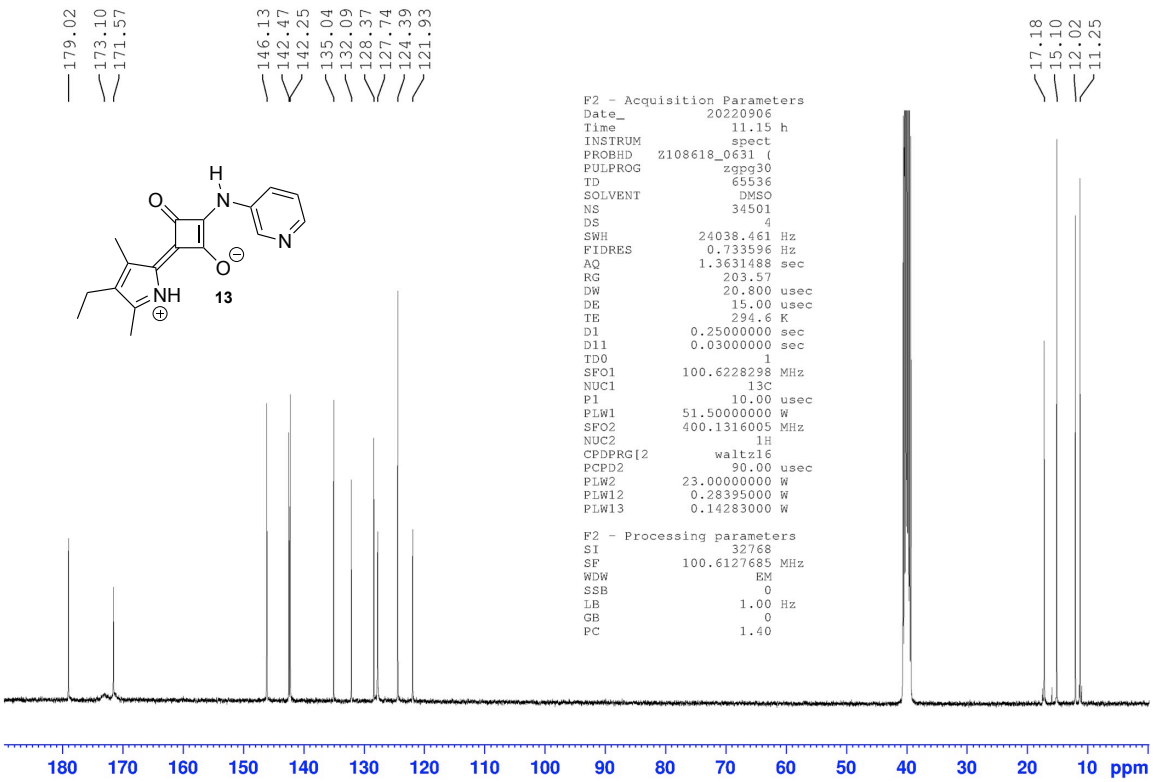

12.468
8.305
8.300
8.287
8.282
8.275
8.161
8.154
8.149
8.136
8.131
6.629
6.627
3.480
2.851
2.218
2.216
F2 - Acquisition Parameters
Date_ 20220811
Time 19.50 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299992 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
N
O
NO2
O
N
11
12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.01
2.00
1.96
1.02
3.25
2.83
3.16

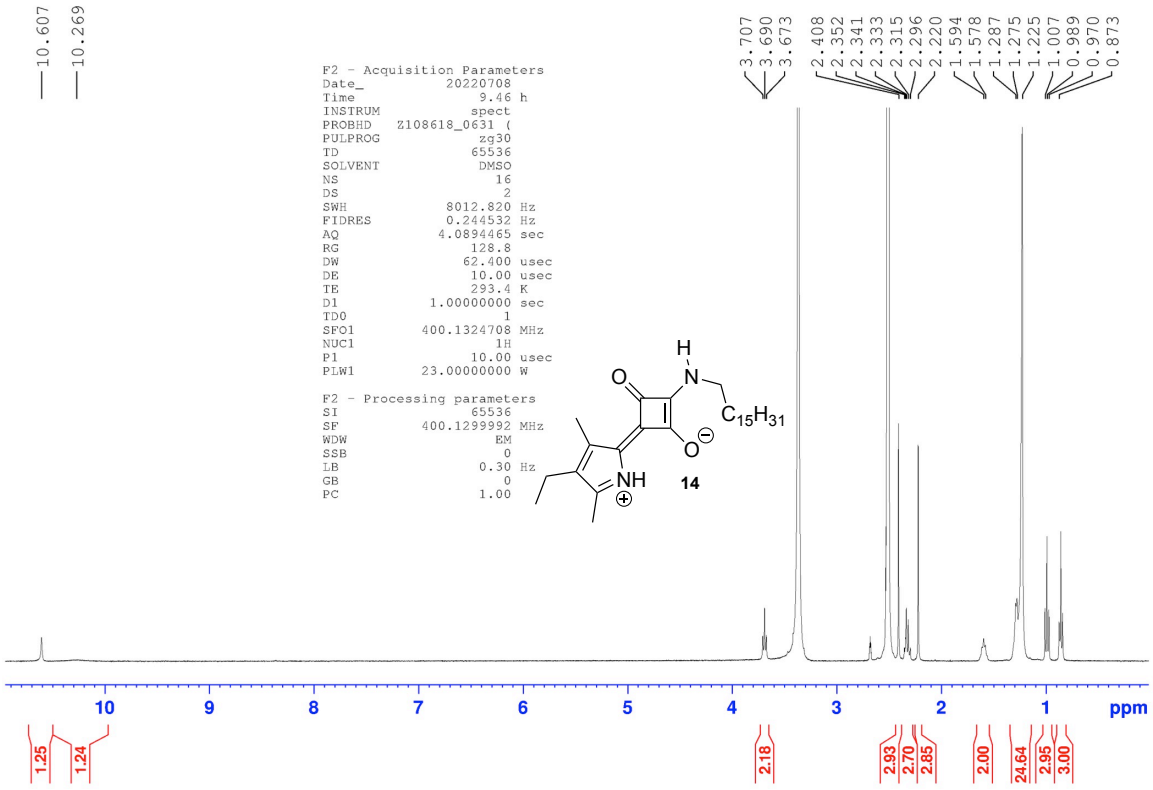

192.77
172.93
155.69
135.62
133.29
130.53
126.38
109.64
104.36
30.42
12.20
12.03
F2 - Acquisition Parameters
Date_ 20220923
Time 8.02 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 27000
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 298.1 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128167 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H
N
O
NO2
O
N
11
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

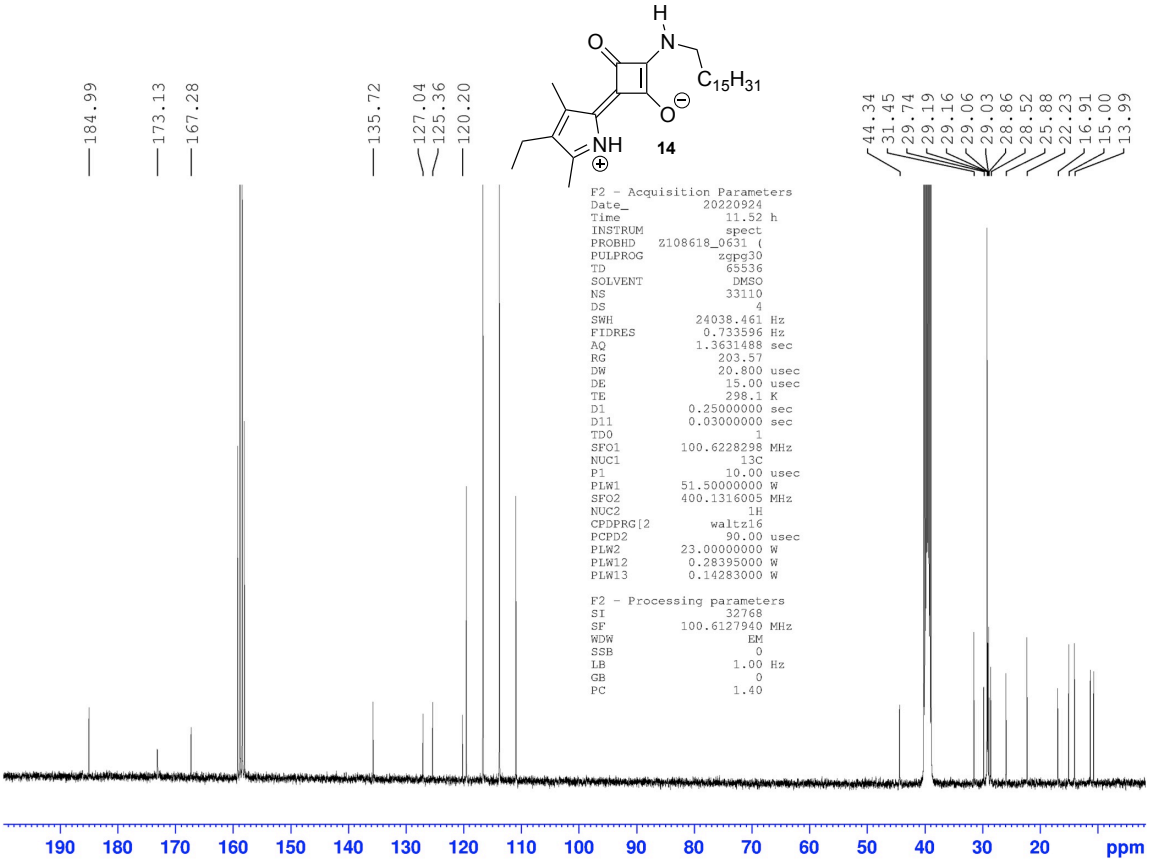

11.336
10.976
10.919
9.011
8.615
8.597
8.507
8.487
7.870
7.850
7.723
7.704
2.543
2.401
2.383
2.340
1.048
1.029
1.011
F2 - Acquisition Parameters
Date_ 20220806
Time 19.10 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.04
1.07
1.00
0.97
0.98
1.00
1.82
3.14
4.95
2.90

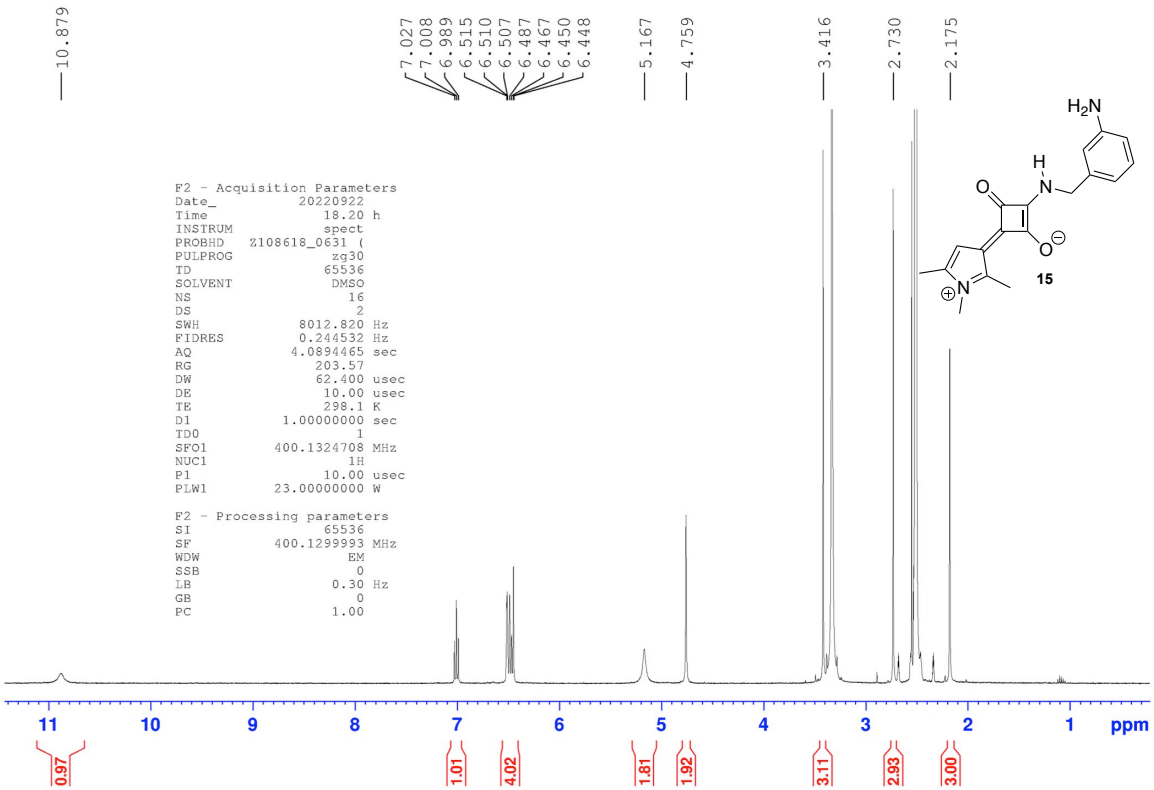

178.00
176.07
164.32
162.81
161.87
153.08
148.40
146.54
139.64
136.10
134.90
134.79
134.40
132.24
130.85
127.45
126.30
20.32
15.94
15.27
13.57
F2 - Acquisition Parameters
Date_ 20220925
Time 14.32 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 43548
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 298.1 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6123151 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
12
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

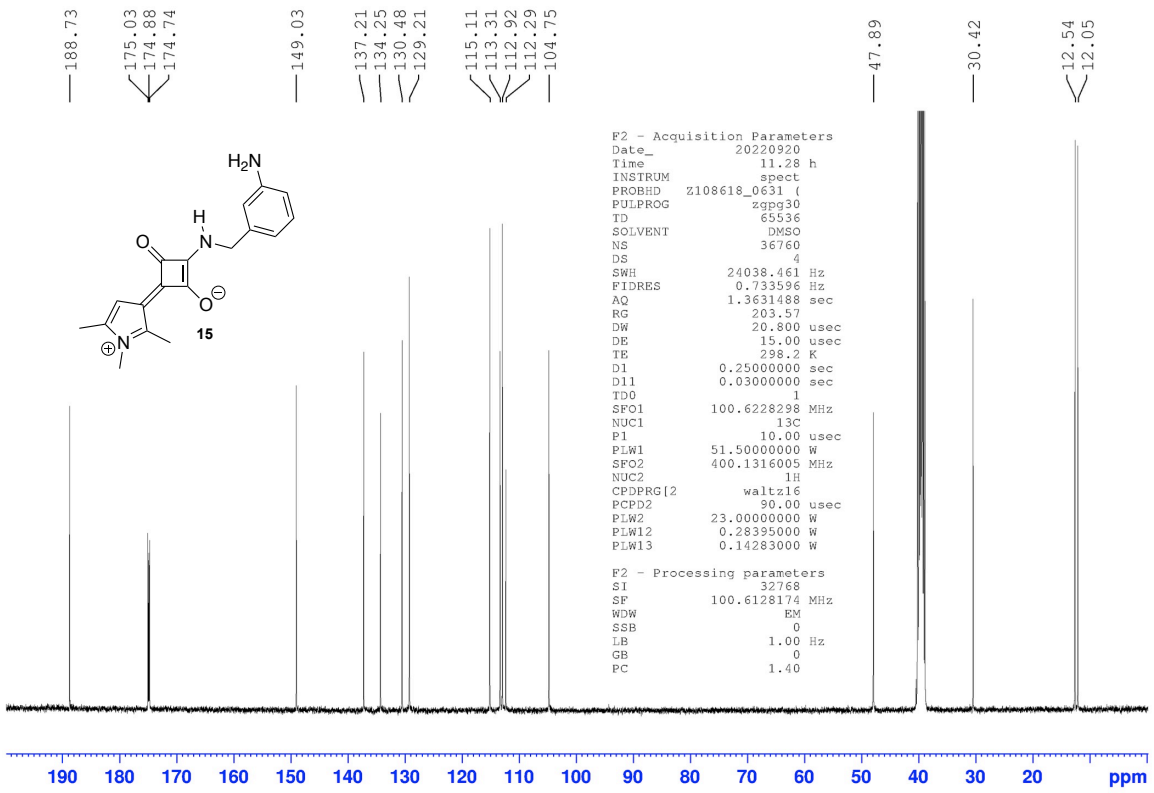

11.995
11.230
8.965
8.373
8.370
8.362
8.358
8.355
8.352
8.348
8.345
8.334
8.331
8.328
8.324
7.465
7.464
7.453
7.452
7.444
7.443
7.432
7.431
2.521
2.404
2.386
2.367
2.348
2.320
1.037
1.018
0.999
13
F2 - Acquisition Parameters
Date_ 20220814
Time 18.13 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.89
0.99
0.99
0.98
1.02
1.03
2.88
2.11
2.98
3.00

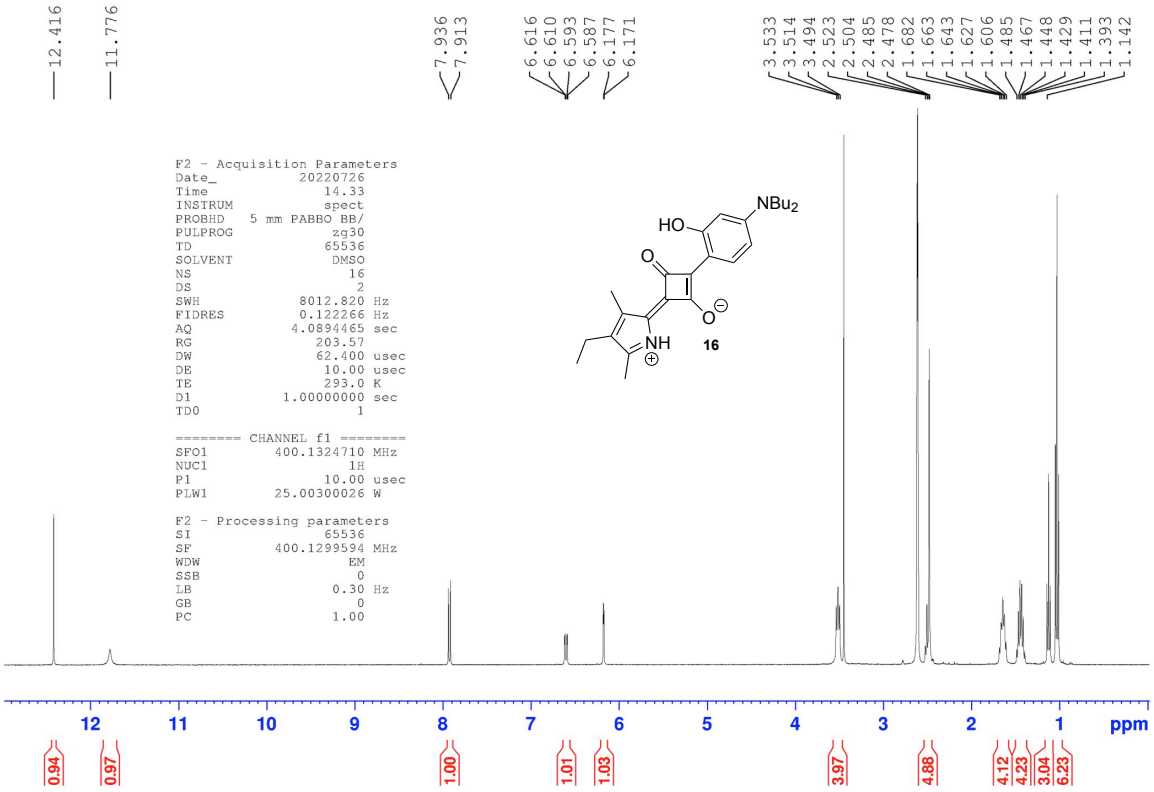

179.02
173.10
171.57
146.13
142.47
142.25
135.04
132.09
128.37
127.74
124.39
121.93
17.18
15.10
12.02
11.25
13
F2 - Acquisition Parameters
Date_ 20220906
Time 11.15 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 34501
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 294.6 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

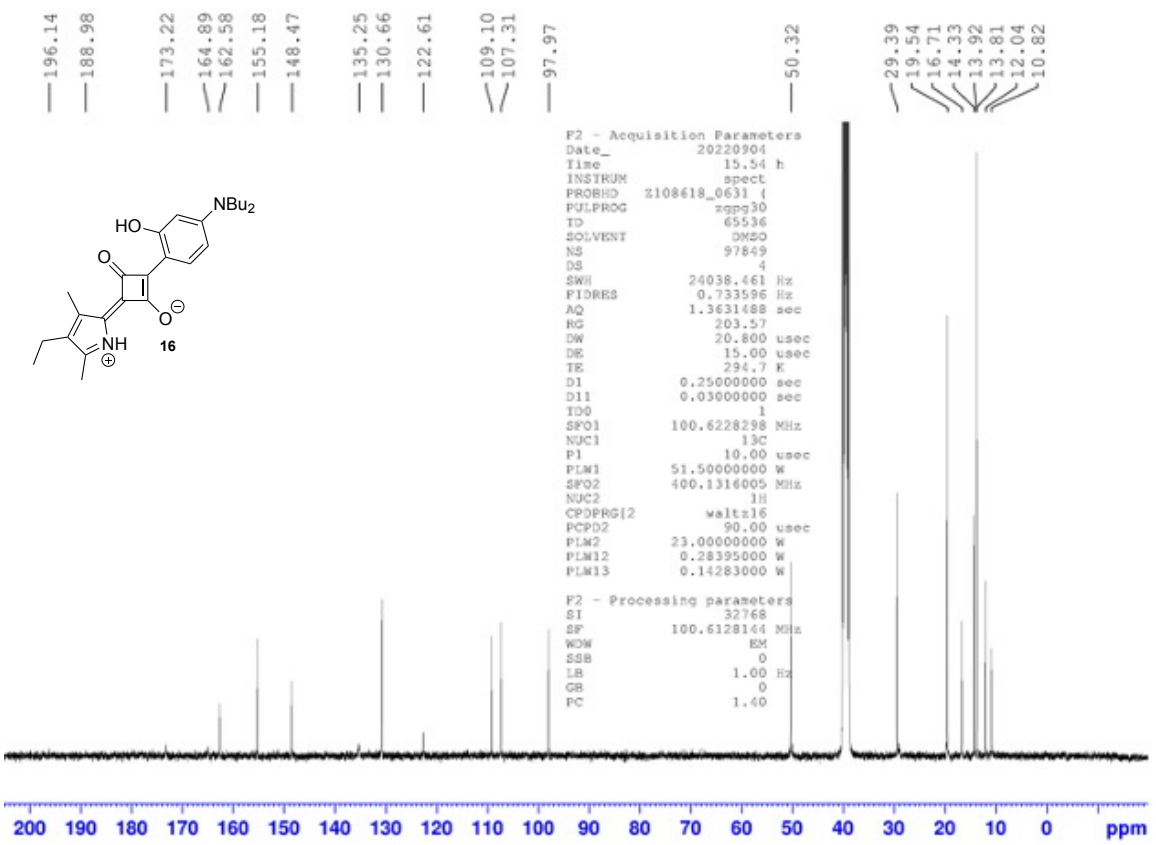

10.607
10.269
3.707
3.690
3.673
2.408
2.352
2.341
2.333
2.315
2.296
2.220
1.594
1.578
1.287
1.275
1.225
1.007
0.989
0.970
0.873
F2 - Acquisition Parameters
Date_ 20220708
Time 9.46 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 128.8
DW 62.400 usec
DE 10.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299992 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
N
O
C15H31
O
NH
14
10
9
8
7
6
5
4
3
2
1
ppm
1.25
1.24
2.18
2.93
2.70
2.85
2.00
24.64
2.95
3.00

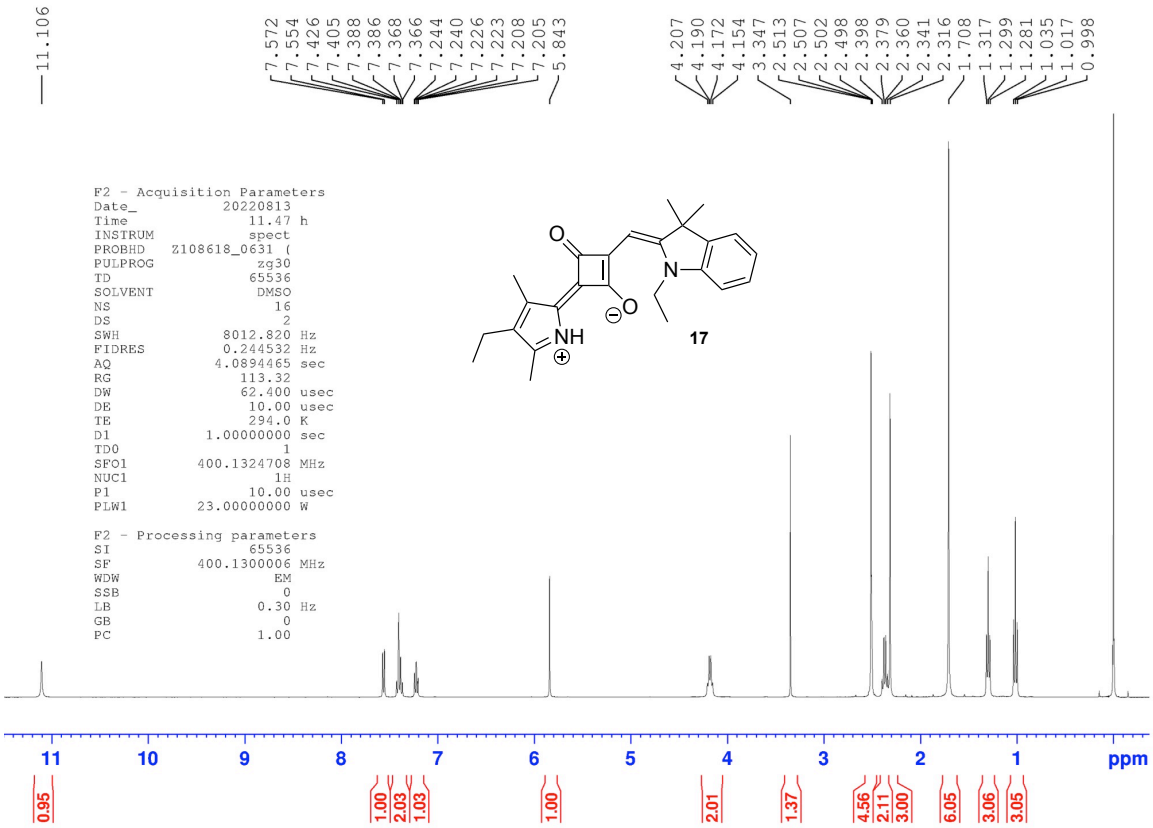

184.99
173.13
167.28
135.72
127.04
125.36
120.20
44.34
31.45
29.74
29.19
29.16
29.06
29.03
28.86
28.52
25.88
22.23
16.91
15.00
13.99
H
N
O
C15H31
O
NH
14
F2 - Acquisition Parameters
Date_ 20220924
Time 11.52 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 33110
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 298.1 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6127940 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

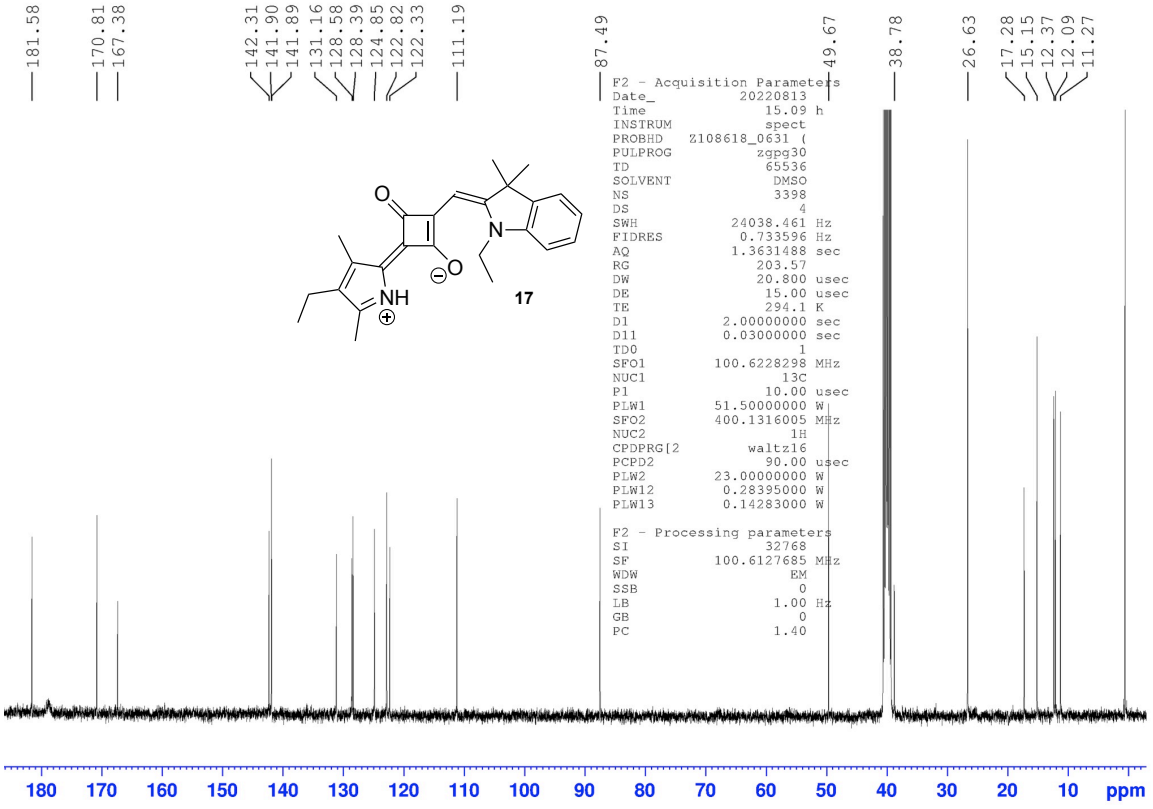

10.879
7.027
7.008
6.989
6.515
6.510
6.507
6.487
6.467
6.450
6.448
5.167
4.759
3.416
2.730
2.175
F2 - Acquisition Parameters
Date_ 20220922
Time 18.20 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H2N
H
N
O
O
N
15
11
10
9
8
7
6
5
4
3
2
1
ppm
0.97
1.01
4.02
1.81
1.92
3.11
2.93
3.00

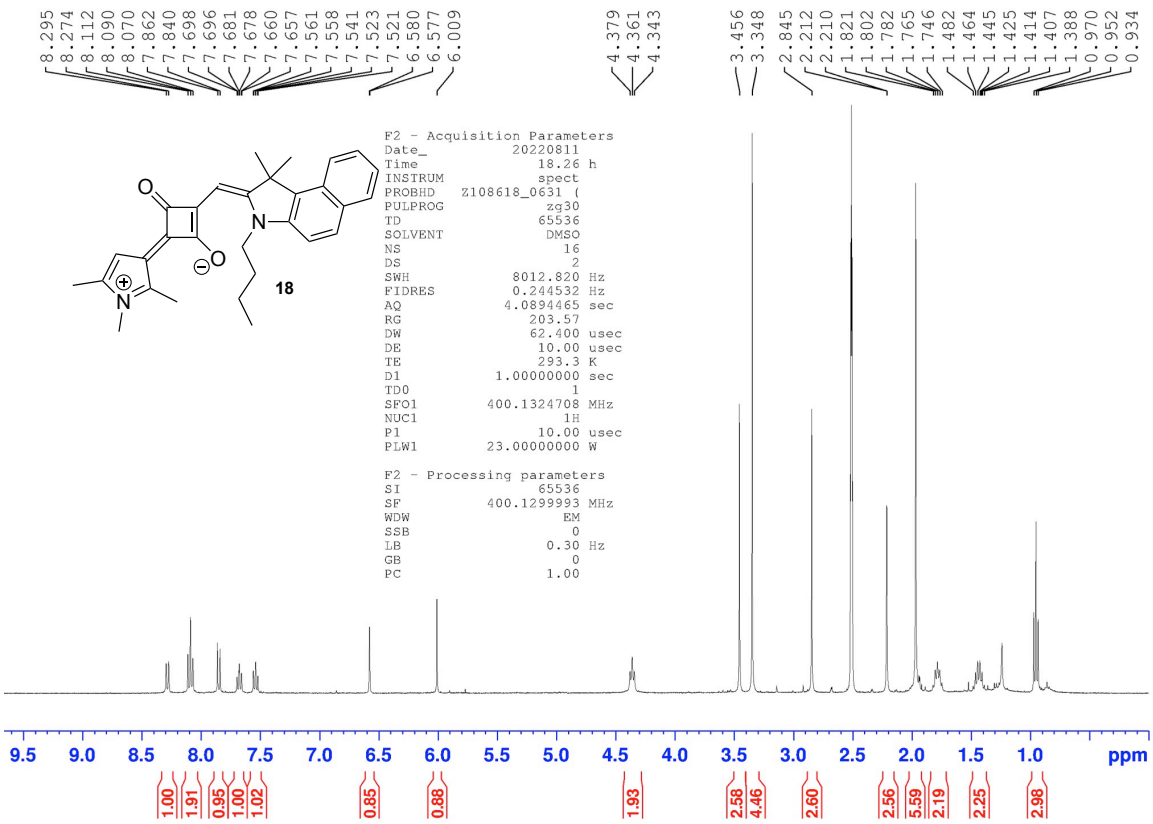

188.73
175.03
174.88
174.74
149.03
137.21
134.25
130.48
129.21
115.11
113.31
112.92
112.29
104.75
47.89
30.42
12.54
12.05
F2 - Acquisition Parameters
Date_ 20220920
Time 11.28 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 36760
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 298.2 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128174 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H2N
H
N
O
O
N
15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

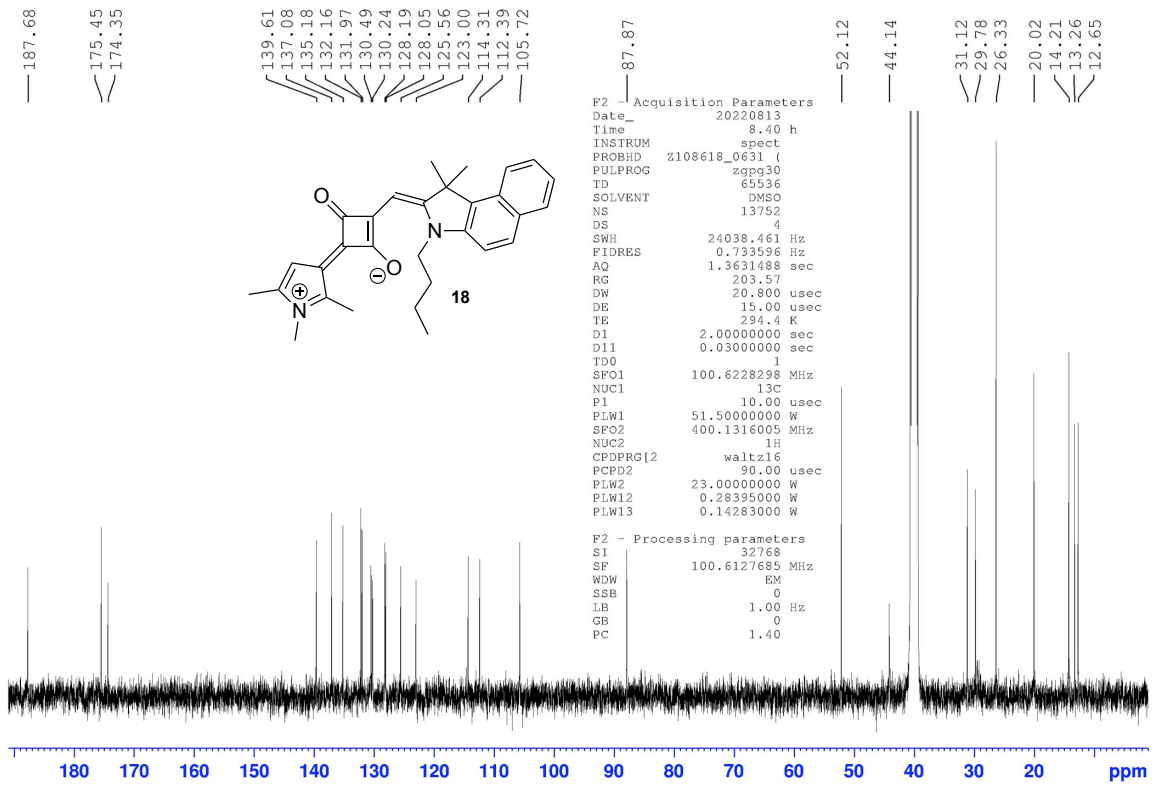

12.416
11.776
7.936
7.913
6.616
6.610
6.593
6.587
6.177
6.171
3.533
3.514
3.494
2.523
2.504
2.485
2.478
1.682
1.663
1.643
1.627
1.606
1.485
1.467
1.448
1.429
1.411
1.393
1.142
F2 - Acquisition Parameters
Date_ 20220726
Time 14.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.00300026 W
F2 - Processing parameters
SI 65536
SF 400.1299594 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NBu2
HO
O
O
NH
16
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.94
0.97
1.00
1.01
1.03
3.97
4.88
4.12
4.23
3.04
6.23


196.14
188.98
173.22
164.89
162.58
155.18
148.47
135.25
130.66
122.61
109.10
107.31
97.97
50.32
29.39
19.54
16.71
14.33
13.92
13.81
12.04
10.82
F2 - Acquisition Parameters
Date_ 20220904
Time 15.54 h
INSTRUM spect
PROBHD %108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 97849
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 294.7 K
D1 0.25000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6128144 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NBu2
HO
O
O
NH
16
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.106
7.572
7.554
7.426
7.405
7.388
7.386
7.368
7.366
7.244
7.240
7.226
7.223
7.208
7.205
5.843
4.207
4.190
4.172
4.154
3.347
2.513
2.507
2.502
2.498
2.398
2.379
2.360
2.341
2.316
1.708
1.317
1.299
1.281
1.035
1.017
0.998
F2 - Acquisition Parameters
Date_ 20220813
Time 11.47 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 113.32
DW 62.400 usec
DE 10.00 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300006 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
O
NH
17
11
10
9
8
7
6
5
4
3
2
1
ppm
0.95
1.00
2.03
1.03
1.00
2.01
1.37
4.56
2.11
3.00
6.05
3.06
3.05


181.58
170.81
167.38
142.31
141.90
141.89
131.16
128.58
128.39
124.85
122.82
122.33
111.19
87.49
49.67
38.78
26.63
17.28
15.15
12.37
12.09
11.27
F2 - Acquisition Parameters
Date_ 20220813
Time 15.09 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3398
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
N
O
NH
17
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.295
8.274
8.112
8.090
8.070
7.862
7.840
7.698
7.696
7.681
7.678
7.660
7.657
7.561
7.558
7.541
7.523
7.521
6.580
6.577
6.009
4.379
4.361
4.343
3.456
3.348
2.845
2.212
2.210
1.821
1.802
1.782
1.765
1.746
1.482
1.464
1.445
1.425
1.414
1.407
1.388
0.970
0.952
0.934
18
F2 - Acquisition Parameters
Date_ 20220811
Time 18.26 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 203.57
DW 62.400 usec
DE 10.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324708 MHz
NUC1 1H
P1 10.00 usec
PLW1 23.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1299993 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 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 ppm
1.00 1.91 0.95 1.00 1.02 0.85 0.88 1.93 2.58 4.46 2.60 2.56 5.59 2.19 2.25 2.98


187.68
175.45
174.35
139.61
137.08
135.18
132.16
131.97
130.49
130.24
128.19
128.05
125.56
123.00
114.31
112.39
105.72
87.87
52.12
44.14
31.12
29.78
26.33
20.02
14.21
13.26
12.65
18
F2 - Acquisition Parameters
Date_ 20220813
Time 8.40 h
INSTRUM spect
PROBHD Z108618_0631 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 13752
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 15.00 usec
TE 294.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 51.50000000 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 23.00000000 W
PLW12 0.28395000 W
PLW13 0.14283000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

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