# Supplementary Material

# Synthesis, solid-state structure, and electrochemical properties of thienodipyrimidine-2,4,5,7-tetra(thi)ones

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## Table of Contents



## 1. General Methods and Instrumentation

**UV-Vis spectroscopy.** Solution phase absorption spectra were recorded on an Agilent Cary 5000 UV-Vis-NIR spectrophotometer, using a quartz cell with a path length of 1 cm.

**Cyclic voltammetry.** Cyclic voltammetry was performed in $CH_2Cl_2$ under nitrogen with a three-electrode PGSTAT204 potentiostat from Metrohm Autolab. Glassy carbon disk was used as the working electrode (diameter = 5 mm), platinum wire as the counter electrode, and a silver wire as the pseudo reference electrode. An analyte of concentration of 1 mM was used with $n$Bu$_4$NPF$_6$ (100 mM) as the supporting electrolyte. Redox potentials relative to saturated calomel electrode (SCE) are reported, using ferrocene/ferrocenium ($E_{1/2}$ = 0.46 V) as an internal standard.[1]

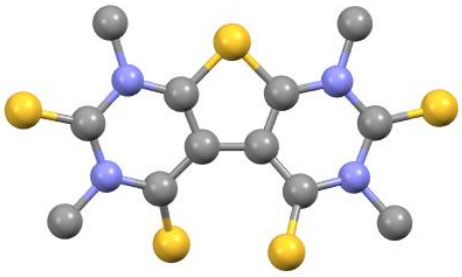
**Single-crystal X-ray diffraction.** Single crystals were grown; crystallization details for each sample can be found in the synthetic details. X-ray diffraction data were collected on a Rigaku Oxford Diffraction SuperNova dual source diffractometer with a mirror monochromator using Mo or Cu Kα radiation. The crystal was kept at 296(3) K during data collection. The structures were solved with the ShelXT[2] structure solution program using Intrinsic Phasing and refined with the ShelXL[3] refinement package using least squares minimization using Olex2.[4]

**Irradiation Details.** Photocyclization reactions were irradiated with EvoluChem 6200K ($\lambda_{irrad}$ = 415–725 nm; white light) or EvoluChem 365 PF ($\lambda_{rrad}$ = 355–385 nm; ultraviolet) LEDs.

## 2. Single-Crystal Structural Characterization

**Table S1.** Crystal data and structure refinement for **1a-S$_4$**.[a]

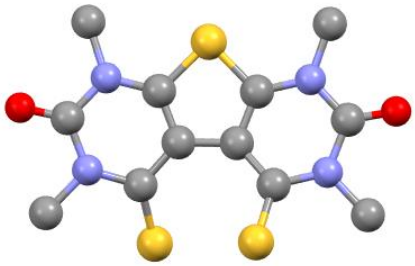


| | | | | | |
|---|---|---|---|---|---|
| Empirical formula | $C_{12}H_{12}N_4S_5$ | a | 8.5159(5) Å | α | 90 ° |
| Formula weight | 372.56 | b | 11.3808(8) Å | β | 96.173(7) ° |
| Temperature | 296(2) K | c | 16.2386(12) Å | γ | 90 ° |
| Crystal system | monoclinic | | | | |
| Space group | Pc | | | | |

| | |
|---|---|
| Volume | 1564.68(19) Å$^3$ |
| Z | 4 |
| $\rho_{calc}$ | 1.582 g cm$^{-3}$ |
| μ | 0.737 mm$^{-1}$ |
| F(000) | 768.0 |
| Crystal size | 0.216 mm × 0.179 mm × 0.038 mm |
| Radiation | Mo Kα (λ = 0.71073) |
| 2Θ range for data collection | 6.588 ° to 59.164 ° |
| Index ranges | -11 ≤ h ≤ 11, -15 ≤ k ≤ 15, -22 ≤ l ≤ 22 |
| Reflections collected | 14228 |
| Independent reflections | 6668 [$R_{int}$ = 0.0615, $R_{sigma}$ = 0.1782] |
| Data/restraints/parameters | 6668/2/387 |
| Goodness-of-fit on F$^2$ | 1.012 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0661, w$R_2$ = 0.1646 |
| Final R indexes [all data] | $R_1$ = 0.1121, w$R_2$ = 0.1729 |
| Largest diff. peak/hole | 0.78/-1.30 e Å$^{-3}$ |
| CCDC Number | 2244092 |

[a] Hydrogen atoms and one **1a-S$_4$** molecule omitted for clarity.

**Table S2.** Crystal data and structure refinement for **2a-$O_2S_2$**.[a]



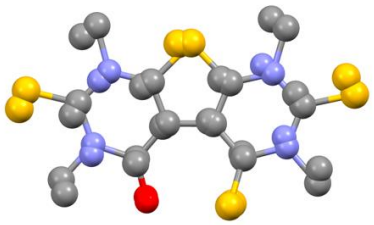
| | | | | | |
|---|---|---|---|---|---|
| Empirical formula | $C_{12}H_{12}N_4O_2S_3$ | a | 10.8682(5) Å | α | 90 ° |
| Formula weight | 340.453 | b | 8.5926(4) Å | β | 90 ° |
| Temperature | 298 K | c | 15.0316(10) Å | γ | 90 ° |
| Crystal system | orthorhombic | | | | |
| Space group | $Pca2_1$ | | | | |

| | |
|---|---|
| Volume | 1403.74(13) $Å^3$ |
| Z | 4 |
| $\rho_{calc}$ | 1.611 g $cm^{-3}$ |
| μ | 0.537 $mm^{-1}$ |
| F(000) | 705.7 |
| Crystal size | 0.35 mm × 0.26 mm × 0.16 mm |
| Radiation | Mo Kα (λ = 0.71073) |
| 2Θ range for data collection | 6.62 º to 59.34 º |
| Index ranges | 0 ≤ h ≤ 15, 0 ≤ k ≤ 11, -20 ≤ l ≤ 15 |
| Reflections collected | 13559 |
| Independent reflections | 3041 [$R_{int}$ = 0.0460, $R_{sigma}$ = 0.0339] |
| Data/restraints/parameters | 3041/1/230 |
| Goodness-of-fit on F2 | 1.121 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0439, $wR_2$ = 0.1005 |
| Final R indexes [all data] | $R_1$ = 0.0652, $wR_2$ = 0.1192 |
| Largest diff. peak/hole | 0.25/-0.34 e $Å^{-3}$ |
| CCDC Number | 2244099 |

[a] Hydrogen atoms are omitted for clarity.

**Table S3.** Crystal data and structure refinement for **3a-OS$_3$**.[a]



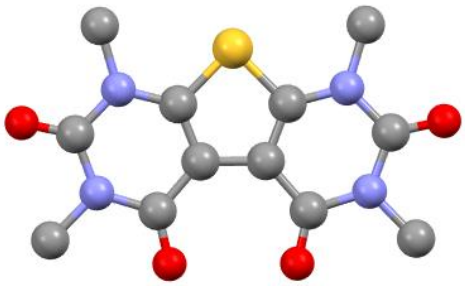
| | | | | | |
|---|---|---|---|---|---|
| Empirical formula | $C_{12}H_{12}N_4OS_4$ | a | 9.5240(4) Å | α | 90 ° |
| Formula weight | 356.50 | b | 8.4921(3) Å | β | 98.988(4) ° |
| Temperature | 293(2) K | c | 18.4678(8) Å | γ | 90 ° |
| Crystal system | monoclinic | | | | |
| Space group | $P2_1/n$ | | | | |

| | |
|---|---|
| Volume | 1475.31(10) Å$^3$ |
| Z | 4 |
| $\rho_{calc}$ | 1.605 g cm$^{-3}$ |
| μ | 5.955 mm$^{-1}$ |
| F(000) | 736.0 |
| Crystal size | 0.16 mm × 0.07 mm × 0.04 mm |
| Radiation | Cu Kα (λ = 1.54184) |
| 2Θ range for data collection | 9.698 ° to 144.82 ° |
| Index ranges | -11 ≤ h ≤ 11, -10 ≤ k ≤ 10, -21 ≤ l ≤ 22 |
| Reflections collected | 5911 |
| Independent reflections | 2869 [$R_{int}$ = 0.0283, $R_{sigma}$ = 0.0366] |
| Data/restraints/parameters | 2869/313/274 |
| Goodness-of-fit on F$^2$ | 1.082 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0709, w$R_2$ = 0.2023 |
| Final R indexes [all data] | $R_1$ = 0.0892, w$R_2$ = 0.2227 |
| Largest diff. peak/hole | 0.87/-0.77 e Å$^{-3}$ |
| CCDC Number | 2251143 |

[a] Hydrogen atoms are omitted for clarity.

**Table S4.** Crystal data and structure refinement for **4a-O$_4$**.[a]



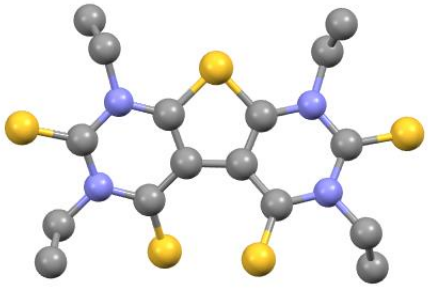
| | | | | | |
|---|---|---|---|---|---|
| Empirical formula | $C_{12}H_{12}N_4O_4S$ | a | 6.7836(2) Å | α | 90 ° |
| Formula weight | 308.32 | b | 8.4663(2) Å | β | 98.143(3) ° |
| Temperature | 296(2) K | c | 21.9949(6) Å | γ | 90 ° |
| Crystal system | monoclinic | | | | |
| Space group | $P2_1/n$ | | | | |

| | |
|---|---|
| Volume | 1250.47(6) Å$^3$ |
| Z | 4 |
| $\rho_{calc}$ | 1.638 g cm$^{-3}$ |
| μ | 2.548 mm$^{-1}$ |
| F(000) | 640.0 |
| Crystal size | 0.22 mm × 0.06 mm × 0.05 mm |
| Radiation | Cu Kα (λ = 1.54184) |
| 2Θ range for data collection | 8.122 ° to 145.972 ° |
| Index ranges | -8 ≤ h ≤ 7, -10 ≤ k ≤ 10, -24 ≤ l ≤ 27 |
| Reflections collected | 8484 |
| Independent reflections | 2481 [$R_{int}$ = 0.0299, $R_{sigma}$ = 0.0248] |
| Data/restraints/parameters | 2481/1107/384 |
| Goodness-of-fit on F$^2$ | 1.105 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0530, $wR_2$ = 0.1447 |
| Final R indexes [all data] | $R_1$ = 0.0589, $wR_2$ = 0.1492 |
| Largest diff. peak/hole | 0.44/-0.31 e Å$^{-3}$ |
| CCDC Number | 2247147 |

[a] Hydrogen atoms and one **4a-O$_4$** unit are omitted for clarity.

**Table S5.** Crystal data and structure refinement for **1b-$S_4$**.[a]



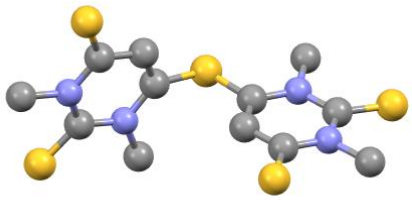
| | | | | | |
|---|---|---|---|---|---|
| Empirical formula | $C_{16}H_{20}N_4S_5$ | a | 11.8534(2) Å | α | 90 ° |
| Formula weight | 428.66 | b | 17.1645(3) Å | β | 90 ° |
| Temperature | 293(2) K | c | 18.8475(3) Å | γ | 90 ° |
| Crystal system | orthorhombic | | | | |
| Space group | Pbca | | | | |

| | |
|---|---|
| Volume | 3834.67(11) $Å^3$ |
| Z | 8 |
| $\rho_{calc}$ | 1.485 g $cm^{-3}$ |
| μ | 5.631 $mm^{-1}$ |
| F(000) | 1792.0 |
| Crystal size | 0.14 mm × 0.11 mm × 0.04 mm |
| Radiation | Cu Kα (λ = 1.54184) |
| 2Θ range for data collection | 9.384 ° to 145.956 ° |
| Index ranges | -14 ≤ h ≤ 12, -21 ≤ k ≤ 19, -23 ≤ l ≤ 18 |
| Reflections collected | 26895 |
| Independent reflections | 3813 [$R_{int}$ = 0.0470, $R_{sigma}$ = 0.0246] |
| Data/restraints/parameters | 3813/0/230 |
| Goodness-of-fit on $F^2$ | 1.046 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0394, $wR_2$ = 0.1034 |
| Final R indexes [all data] | $R_1$ = 0.0482, $wR_2$ = 0.1149 |
| Largest diff. peak/hole | 0.37/-0.29 e $Å^{-3}$ |
| CCDC Number | 2246037 |

[a] Hydrogen atoms are omitted for clarity.

**Table S6.** Crystal data and structure refinement for **5**.[a]



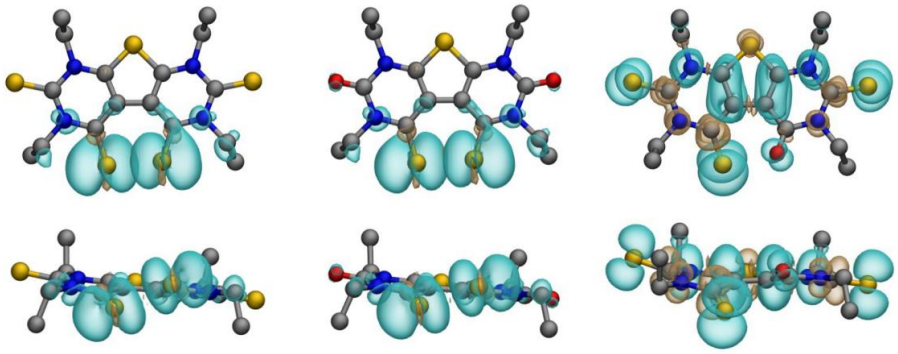
| | | | | | |
|---|---|---|---|---|---|
| Empirical formula | $C_{12}H_{12}N_4O_3S_3$ | a | 5.6075(4) Å | α | 113.098(8) ° |
| Formula weight | 374.56 | b | 11.9838(10) Å | β | 99.865(6) ° |
| Temperature | 296 K | c | 13.2276(11) Å | γ | 93.093(6) ° |
| Crystal system | triclinic | | | | |
| Space group | P $\bar{1}$ | | | | |

| | |
|---|---|
| Volume | 798.30(12) |
| Z | 2 |
| $\rho_{calc}$ | 1.558 g cm$^{-3}$ |
| μ | 0.723 mm$^{-1}$ |
| F(000) | 368.9 |
| Crystal size | 0.56 mm× 0.12 mm × 0.04 mm |
| Radiation | Mo Kα (λ = 0.71073) |
| 2Θ range for data collection | 6.84 º to 59.14 º |
| Index ranges | -7 ≤ h ≤ 7, -16 ≤ k ≤ 15, -14 ≤ l ≤ 18 |
| Reflections collected | 6731 |
| Independent reflections | 3709 [$R_{int}$ = 0.0246, $R_{sigma}$ = 0.0481] |
| Data/restraints/parameters | 3709/0/194 |
| Goodness-of-fit on F2 | 1.063 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0477, w$R_2$ = 0.1057 |
| Final R indexes [all data] | $R_1$ = 0.0759, w$R_2$ = 0.1238 |
| Largest diff. peak/hole | 0.43/-0.40 e Å$^{-3}$ |
| CCDC Number | 2244101 |

[a] Hydrogen atoms are omitted for clarity.

## 3. Electron Spin Density Plots



**1b-$S_4^{\bullet+}$** **2b-$O_2S_2^{\bullet+}$** **3b-$OS_3^{\bullet+}$**

**Figure S1.** Electron spin density distributions (isosurface value 0.001 a.u.) for radical cations **1b-$S_4^{\bullet+}$**, **2b-$O_2S_2^{\bullet+}$**, and **3b-$OS_3^{\bullet+}$** (top view above and side view below). Atoms color code: C grey, N blue, O red, S yellow; geometry optimized at the UωB97X-D/6-31G(d,p) level.

# 4. Cartesian Coordinates for Optimized Geometries

## 1a-$S_4$

| | | | |
|---|---|---|---|
| C | 3.49020 | 0.38533 | -0.06100 |
| N | 2.99912 | -0.88500 | -0.02451 |
| C | 1.64075 | -1.20565 | 0.32301 |
| C | 0.72568 | -0.17050 | 0.01002 |
| C | 1.22550 | 1.12299 | -0.07051 |
| N | 2.57775 | 1.40663 | 0.05451 |
| S | 0.00000 | 2.33743 | 0.00000 |
| C | -1.22550 | 1.12299 | 0.07052 |
| C | -0.72568 | -0.17050 | -0.01001 |
| C | -1.64075 | -1.20565 | -0.32301 |
| N | -2.99912 | -0.88499 | 0.02451 |
| C | -3.49020 | 0.38533 | 0.06100 |
| N | -2.57775 | 1.40663 | -0.05451 |
| C | -2.98906 | 2.80549 | -0.09636 |
| C | -3.92578 | -1.99815 | 0.23281 |
| C | 2.98906 | 2.80549 | 0.09635 |
| C | 3.92578 | -1.99815 | -0.23280 |
| S | 1.27231 | -2.62361 | 1.15259 |
| S | -1.27231 | -2.62361 | -1.15259 |
| S | -5.14643 | 0.70932 | 0.23352 |
| S | 5.14643 | 0.70932 | -0.23352 |
| H | -3.99832 | 2.86838 | -0.49239 |
| H | -2.31052 | 3.35025 | -0.75660 |
| H | -2.96776 | 3.24538 | 0.90570 |
| H | -3.33741 | -2.88739 | 0.44374 |
| H | -4.53590 | -2.17387 | -0.65691 |
| H | -4.57989 | -1.76203 | 1.07065 |
| H | 2.31053 | 3.35025 | 0.75661 |
| H | 2.96774 | 3.24539 | -0.90570 |
| H | 3.99833 | 2.86838 | 0.49237 |
| H | 4.57989 | -1.76203 | -1.07065 |
| H | 3.33741 | -2.88739 | -0.44374 |
| H | 4.53590 | -2.17387 | 0.65691 |

## [1a-$S_4$]$^{+1}$

| | | | |
|---|---|---|---|
| C | -3.49386 | 0.42363 | 0.06219 |
| N | -2.99113 | -0.88057 | 0.00990 |
| C | -1.66442 | -1.19916 | -0.12720 |
| C | -0.72614 | -0.14163 | 0.01849 |
| C | -1.22694 | 1.14284 | 0.05815 |
| N | -2.54939 | 1.43395 | 0.06366 |
| S | 0.00000 | 2.37652 | -0.00000 |
| C | 1.22694 | 1.14284 | -0.05815 |
| C | 0.72614 | -0.14163 | -0.01849 |
| C | 1.66442 | -1.19916 | 0.12720 |
| N | 2.99113 | -0.88057 | -0.00990 |
| C | 3.49386 | 0.42363 | -0.06219 |
| N | 2.54939 | 1.43395 | -0.06366 |
| C | 3.01984 | 2.82683 | -0.06714 |
| C | 3.98178 | -1.97111 | -0.00995 |
| C | -3.01984 | 2.82683 | 0.06714 |
| C | -3.98179 | -1.97111 | 0.00995 |
| S | -1.21261 | -2.77471 | -0.55617 |
| S | 1.21261 | -2.77471 | 0.55617 |
| S | 5.11535 | 0.76293 | -0.08143 |
| S | -5.11535 | 0.76293 | 0.08143 |
| H | 3.62228 | 3.00595 | 0.82259 |
| H | 2.16637 | 3.50104 | -0.06972 |
| H | 3.62130 | 3.00130 | -0.95826 |
| H | 3.50079 | -2.8738 | -0.37736 |
| H | 4.35951 | -2.12996 | 1.00166 |
| H | 4.80121 | -1.69002 | -0.66454 |
| H | -2.16637 | 3.5010 | 0.06971 |
| H | -3.62129 | 3.00131 | 0.95826 |
| H | -3.62229 | 3.00595 | -0.82259 |
| H | -4.80121 | -1.69003 | 0.66454 |
| H | -3.50079 | -2.87383 | 0.37735 |
| H | -4.35951 | -2.12996 | -1.00166 |

## 2a-$O_2S_2$

| | | | |
|---|---|---|---|
| C | 1.22009 | 1.16933 | 0.09117 |
| N | 2.54478 | 1.47729 | 0.15528 |
| C | 3.48667 | 0.45927 | 0.21970 |
| N | 2.99866 | -0.83108 | 0.00040 |
| C | 1.68191 | -1.14454 | -0.32060 |
| C | 0.72693 | -0.10880 | 0.00765 |
| S | -0.00000 | 2.41314 | -0.00000 |
| C | -1.22009 | 1.16933 | -0.09117 |
| C | -0.72693 | -0.10880 | -0.00765 |
| N | -2.54478 | 1.47729 | -0.15528 |
| C | -3.48667 | 0.45927 | -0.21970 |
| N | -2.99866. | -0.83108 | -0.00040 |
| C | -1.68191. | -1.14454 | 0.32060 |
| S | -1.30121 | -2.54772 | 1.12479 |
| O | -4.66673 | 0.69033 | -0.41047 |
| O | 4.66673 | 0.69033 | 0.41047 |
| S | 1.30121 | -2.54772 | -1.12479 |
| C | 2.99431 | 2.86491 | 0.23749 |
| C | 4.02280 | -1.87500 | -0.08621 |
| C | -4.02280 | -1.87501 | 0.08622 |
| C | -2.99431 | 2.86491 | -0.23749 |
| H | 4.07858 | 2.85697 | 0.30199 |
| H | 2.58158 | 3.34119 | 1.13008 |
| H | 4.89913 | -1.52700 | 0.45233 |
| H | 4.27986 | -2.07110 | -1.12942 |
| H | -4.89913 | -1.52700 | -0.45232 |
| H | -4.27985 | -2.07110 | 1.12942 |
| H | -2.68429 | 3.40917 | 0.65768 |
| H | -4.07858 | 2.85696 | -0.30199 |
| H | 2.68429 | 3.40917 | -0.65768 |
| H | 3.63147 | -2.78784 | 0.35947 |
| H | -3.63147 | -2.78784 | -0.35947 |
| H | -2.58159 | 3.34119 | -1.13008 |

## [2a-$O_2S_2$]$^{+1}$

| | | | |
|---|---|---|---|
| C | 1.22044 | 1.20783 | 0.05454 |
| N | 2.53478 | 1.52584 | 0.08113 |
| C | 3.49972 | 0.52862 | 0.08008 |
| N | 3.00618 | -0.78816 | -0.01169 |
| C | 1.68643 | -1.13167 | -0.08874 |
| C | 0.72907 | -0.08115 | 0.01882 |
| S | -0.00000 | 2.44473 | -0.00000 |
| C | -1.22044 | 1.20783 | -0.05454 |
| C | -0.72907 | -0.08115 | -0.01883 |
| N | -2.53478 | 1.52584 | -0.08113 |
| C | -3.49972 | 0.52862 | -0.08008 |
| N | -3.00618 | -0.78816 | 0.01169 |
| C | -1.68643 | -1.13167 | 0.08874 |
| S | -1.29470 | -2.75508 | 0.37192 |
| O | -4.68341 | 0.77320 | -0.13704 |
| O | 4.68341 | 0.77320 | 0.13704 |
| S | 1.29470 | -2.75508 | -0.37192 |
| C | 2.97266 | 2.92503 | 0.11069 |
| C | 4.03297 | -1.83901 | -0.07566 |
| C | -4.03297 | -1.83901 | 0.07566 |
| C | -2.97266 | 2.92503 | -0.11069 |
| H | 4.05771 | 2.93131 | 0.14925 |
| H | 2.57343 | 3.41595 | 1.00033 |
| H | 4.99501 | -1.37433 | 0.11218 |
| H | 4.02772 | -2.29883 | -1.06536 |
| H | -4.99501 | -1.37433 | -0.11218 |
| H | -4.02771 | -2.29884 | 1.06536 |
| H | -2.63565 | 3.43413 | 0.79481 |
| H | -4.05771 | 2.93131 | -0.14924 |
| H | 2.63565 | 3.43413 | -0.79481 |
| H | 3.82131 | -2.59219 | 0.68380 |
| H | -3.82131 | -2.59219 | -0.68380 |
| H | -2.57343 | 3.41595 | -1.00033 |

## 3a-$OS_3$

| | | | |
|---|---|---|---|
| C | -3.42200 | -0.35370 | -0.11812 |
| N | -2.96069 | 0.94957 | -0.08835 |
| C | -1.64539 | 1.32032 | 0.22922 |
| C | -0.68390 | 0.26735 | 0.01633 |
| C | -1.15343 | -1.02034 | 0.05168 |
| N | -2.48252 | -1.33873 | 0.08655 |
| S | 0.10466 | -2.22557 | 0.09599 |
| C | 1.30071 | -0.95665 | 0.00996 |
| C | 0.76000 | 0.29793 | -0.04972 |
| C | 1.65586 | 1.38713 | -0.39564 |
| N | 3.03042 | 1.07542 | -0.19620 |
| C | 3.54851 | -0.18285 | -0.02120 |
| N | 2.64281 | -1.22419 | -0.02746 |
| C | 3.10021 | -2.61469 | 0.03605 |
| C | 3.95965 | 2.20463 | -0.29236 |

| | | | |
|---|---|---|---|
| C | -2.89743 | -2.73808 | 0.21569 |
| C | -3.95345 | 2.01829 | -0.25344 |
| S | -1.27665 | 2.80118 | 0.87065 |
| O | 1.32677 | 2.46973 | -0.83368 |
| S | 5.19298 | -0.45516 | 0.16762 |
| S | -5.03666 | -0.73990 | -0.34603 |
| H | 4.06512 | -2.69044 | -0.45704 |
| H | 2.38416 | -3.24587 | -0.49157 |
| H | 3.19835 | -2.93821 | 1.07447 |
| H | 3.36379 | 3.10644 | -0.39530 |
| H | 4.60875 | 2.08389 | -1.16101 |
| H | 4.57341 | 2.24485 | 0.60695 |
| H | -2.11467 | -3.29071 | 0.73476 |
| H | -3.08080 | -3.17414 | -0.76808 |
| H | -3.81005 | -2.78298 | 0.80474 |
| H | -4.64304 | 1.72837 | -1.04198 |
| H | -3.42630 | 2.93100 | -0.51540 |
| H | -4.50832 | 2.17068 | 0.67476 |

## [3a-OS$_3$]$^{+1}$

| | | | |
|---|---|---|---|
| C | -3.40430 | -0.32594 | -0.13558 |
| N | -2.93594 | 0.96752 | -0.09830 |
| C | -1.63034 | 1.32674 | 0.25973 |
| C | -0.65979 | 0.26714 | 0.05605 |
| C | -1.14870 | -1.05588 | 0.10117 |
| N | -2.44842 | -1.34258 | 0.10622 |
| S | 0.10237 | -2.26641 | 0.13864 |
| C | 1.29255 | -0.99192 | 0.00092 |
| C | 0.73732 | 0.29084 | -0.05895 |
| C | 1.63181 | 1.40179 | -0.39488 |
| N | 3.00164 | 1.09514 | -0.24038 |
| C | 3.52632 | -0.15662 | -0.04364 |
| N | 2.60609 | -1.23049 | -0.03946 |
| C | 3.11578 | -2.61082 | 0.01311 |
| C | 3.93548 | 2.22177 | -0.36727 |
| C | -2.89813 | -2.73878 | 0.23463 |
| C | -3.91987 | 2.04246 | -0.29545 |
| S | -1.25252 | 2.76744 | 0.94840 |
| O | 1.26536 | 2.48353 | -0.79063 |
| S | 5.13928 | -0.44006 | 0.16430 |
| S | -4.98002 | -0.72571 | -0.41564 |
| H | 3.97384 | -2.69397 | -0.64792 |
| H | 2.33863 | -3.28845 | -0.33685 |
| H | 3.40916 | -2.85820 | 1.03341 |
| H | 3.34505 | 3.11754 | -0.53178 |
| H | 4.60449 | 2.05117 | -1.21138 |
| H | 4.52141 | 2.30906 | 0.54735 |
| H | -2.12716 | -3.30873 | 0.75069 |
| H | -3.09091 | -3.15642 | -0.75385 |
| H | -3.80798 | -2.75962 | 0.82769 |
| H | -4.55604 | 1.77578 | -1.13608 |
| H | -3.38176 | 2.96202 | -0.50428 |
| H | -4.52980 | 2.16567 | 0.60150 |

## 4a-O$_4$

| | | | |
|---|---|---|---|
| C | -1.22726 | 0.88920 | 0.00833 |
| N | -2.55589 | 1.21563 | 0.01284 |
| C | -3.52041 | 0.21651 | 0.01431 |
| N | -3.03365 | -1.08214 | -0.00200 |
| C | -1.68301 | -1.48635 | -0.03510 |
| C | -0.72589 | -0.38474 | 0.00172 |
| S | 0.00000 | 2.12531 | -0.00000 |
| C | 1.22726 | 0.88920 | -0.00833 |
| C | 0.72589 | -0.38474 | -0.00172 |
| N | 2.55589 | 1.21563 | -0.01284 |
| C | 3.52041 | 0.21651 | -0.01431 |
| N | 3.03365 | -1.08214 | 0.00200 |
| C | 1.68300 | -1.48635 | 0.03510 |
| O | 1.42586 | -2.67370 | 0.09320 |
| O | 4.71187 | 0.47950 | -0.02661 |
| C | 2.98201 | 2.61116 | -0.02091 |
| C | 4.01309 | -2.16854 | 0.01831 |
| C | -2.98201 | 2.61116 | 0.02091 |
| C | -4.01309 | -2.16854 | -0.01830 |
| O | -4.71187 | 0.47950 | 0.02661 |
| O | -1.42586 | -2.67370 | -0.09320 |
| H | 2.62017 | 3.12106 | 0.87577 |
| H | 2.60585 | 3.11485 | -0.91522 |
| H | 4.06825 | 2.62305 | -0.02978 |
| H | 5.00405 | -1.73130 | -0.05593 |
| H | 3.82826 | -2.83757 | -0.82260 |
| H | 3.91928 | -2.73695 | 0.94487 |
| H | -2.62017 | 3.12107 | -0.87577 |
| H | -2.60585 | 3.11485 | 0.91522 |
| H | -4.06825 | 2.62305 | 0.02978 |
| H | -5.00406 | -1.73130 | 0.05593 |
| H | -3.82826 | -2.83757 | 0.82261 |
| H | -3.91928 | -2.73695 | -0.94487 |

## [4a-O$_4$]$^{+1}$

| | | | |
|---|---|---|---|
| C | -1.21760 | 0.93198 | 0.00172 |
| N | -2.51396 | 1.22260 | 0.00977 |
| C | -3.49655 | 0.18301 | 0.01863 |
| N | -3.01003 | -1.10494 | -0.00022 |
| C | -1.66361 | -1.49779 | -0.05060 |
| C | -0.69956 | -0.38014 | -0.00497 |
| S | 0.00000 | 2.17684 | 0.00000 |
| C | 1.21760 | 0.93198 | -0.00172 |
| C | 0.69956 | -0.38014 | 0.00497 |
| N | 2.51396 | 1.22260 | -0.00977 |
| C | 3.49655 | 0.18301 | -0.01863 |
| N | 3.01003 | -1.10494 | 0.00022 |
| C | 1.66361 | -1.49779 | 0.05060 |
| O | 1.37627 | -2.66843 | 0.12932 |
| O | 4.66845 | 0.47044 | -0.03925 |
| C | 2.98132 | 2.61430 | -0.01711 |
| C | 3.98857 | -2.20045 | 0.01465 |
| C | 2.98132 | 2.61430 | 0.01711 |
| C | -3.98857 | -2.20045 | -0.01465 |
| O | -4.66845 | 0.47044 | 0.03925 |
| O | -1.37627 | -2.66843 | -0.12932 |
| H | 2.62517 | 3.12101 | 0.88198 |
| H | 2.61561 | 3.11498 | -0.91585 |
| H | 4.06675 | 2.59869 | -0.02318 |
| H | 4.97969 | -1.77274 | -0.09790 |
| H | 3.77232 | -2.88087 | -0.80811 |
| H | 3.91485 | -2.74091 | 0.95851 |
| H | -2.62517 | 3.12101 | -0.88198 |
| H | -2.61561 | 3.11498 | 0.91585 |
| H | -4.06675 | 2.59869 | 0.02318 |
| H | -4.97969 | -1.77274 | 0.09790 |
| H | -3.77232 | -2.88087 | 0.80811 |
| H | -3.91485 | -2.74091 | -0.95851 |

## 1b-S$_4$

| | | | |
|---|---|---|---|
| C | -1.21349 | 1.02754 | -0.16050 |
| N | -2.54677 | 1.31372 | -0.19042 |
| C | -3.46196 | 0.31491 | 0.07337 |
| N | -2.97551 | -0.97958 | 0.04626 |
| C | -1.63519 | -1.32270 | -0.17788 |
| C | -0.70860 | -0.23478 | 0.01317 |
| S | 0.01118 | 2.24537 | -0.42608 |
| C | 1.23959 | 1.01827 | -0.23743 |
| C | 0.73943 | -0.22201 | 0.06550 |
| N | 2.56932 | 1.29644 | -0.33419 |
| C | 3.49173 | 0.27672 | -0.19866 |
| N | 3.01140 | -0.91645 | 0.30475 |
| C | 1.66799 | -1.16523 | 0.63171 |
| S | 1.20114 | -2.37328 | 1.66814 |
| S | 5.10420 | 0.51938 | -0.59760 |
| S | -5.07296 | 0.68044 | 0.36414 |
| S | -1.17668 | -2.81882 | -0.72685 |
| C | -2.97706 | 2.71684 | -0.36364 |
| C | -2.98811 | 3.49360 | 0.94571 |
| C | -3.95623 | -2.08444 | 0.16672 |
| C | -4.63625 | -2.41784 | -1.15364 |
| C | 3.98159 | -2.00795 | 0.55261 |
| C | 4.03745 | -2.96240 | -0.63094 |
| C | 3.00309 | 2.68373 | -0.59814 |
| C | 3.29944 | 3.43098 | 0.69334 |
| H | -3.96848 | 2.68996 | -0.81061 |
| H | -2.29990 | 3.16512 | -1.09564 |
| H | -3.70277 | 3.05254 | 1.64266 |
| H | -3.28850 | 4.52442 | 0.74450 |
| H | -2.00136 | 3.51251 | 1.41573 |
| H | -4.68326 | -1.77468 | 0.91513 |
| H | -3.40089 | -2.94181 | 0.54212 |
| H | -5.18627 | -1.55761 | -1.54067 |
| H | -5.34704 | -3.23172 | -0.98861 |
| H | -3.90395 | -2.74295 | -1.89483 |
| H | 3.65025 | -2.51710 | 1.45448 |
| H | 4.94778 | -1.54778 | 0.73876 |
| H | 3.05881 | -3.41423 | -0.81404 |
| H | 4.74916 | -3.76297 | -0.41495 |
| H | 4.36522 | -2.44361 | -1.53511 |
| H | 2.21243 | 3.16794 | -1.17565 |

| | | | |
|---|---|---|---|
| H | 3.88194 | 2.62899 | -1.23666 |
| H | 2.42271 | 3.46582 | 1.34547 |
| H | 3.59356 | 4.45599 | 0.45686 |
| H | 4.12066 | 2.95140 | 1.23071 |

## [1b-$S_4$]$^{+1}$

| | | | |
|---|---|---|---|
| C | -1.23250 | 1.04632 | -0.12360 |
| N | -2.55377 | 1.33874 | -0.14739 |
| C | -3.49576 | 0.34404 | 0.04355 |
| N | -2.99459 | -0.96354 | 0.08407 |
| C | -1.66475 | -1.29253 | 0.02694 |
| C | -0.72647 | -0.22450 | 0.04392 |
| S | -0.01321 | 2.26372 | -0.38004 |
| C | 1.21823 | 1.04063 | -0.23787 |
| C | 0.72640 | -0.22091 | 0.02269 |
| N | 2.53440 | 1.33497 | -0.33513 |
| C | 3.49012 | 0.35270 | -0.14231 |
| N | 3.00252 | -0.89336 | 0.26716 |
| C | 1.67424 | -1.21867 | 0.37630 |
| S | 1.21052 | -2.72874 | 0.99460 |
| S | 5.10675 | 0.65573 | -0.36566 |
| S | -5.11224 | 0.69361 | 0.18222 |
| S | -1.18956 | -2.90967 | -0.16109 |
| C | -2.97782 | 2.75225 | -0.29656 |
| C | -3.05426 | 3.47114 | 1.04214 |
| C | -3.99168 | -2.06634 | 0.13717 |
| C | -4.46132 | -2.47755 | -1.25075 |
| C | 4.00826 | -1.94254 | 0.58776 |
| C | 4.33335 | -2.79895 | -0.62652 |
| C | 2.94735 | 2.72874 | -0.63101 |
| C | 3.20695 | 3.52232 | 0.64035 |
| H | -3.94399 | 2.73861 | -0.79569 |
| H | -2.26726 | 3.22653 | -0.97768 |
| H | -3.79509 | 2.99880 | 1.68960 |
| H | -3.35681 | 4.50559 | 0.86722 |
| H | -2.08896 | 3.48074 | 1.55472 |
| H | -4.81881 | -1.70717 | 0.74376 |
| H | -3.51423 | -2.89300 | 0.66079 |
| H | -4.95425 | -1.64614 | -1.75866 |
| H | -5.18175 | -3.29196 | -1.14773 |
| H | -3.63148 | -2.83180 | -1.86651 |
| H | 3.59779 | -2.53691 | 1.40292 |
| H | 4.89042 | -1.42675 | 0.95737 |
| H | 3.44428 | -3.30809 | -1.00507 |
| H | 5.06306 | -3.55716 | -0.33402 |
| H | 4.76762 | -2.19272 | -1.42379 |
| H | 2.15857 | 3.17393 | -1.24109 |
| H | 3.83817 | 2.66754 | -1.25216 |
| H | 2.31701 | 3.57676 | 1.27238 |
| H | 3.49508 | 4.53936 | 0.36677 |
| H | 4.02221 | 3.07507 | 1.21226 |

## 2b-$O_2S_2$

| | | | |
|---|---|---|---|
| C | -1.21278 | 1.07804 | -0.11288 |
| N | -2.54064 | 1.37877 | -0.10137 |
| C | -3.46178 | 0.37656 | 0.16755 |
| N | -2.97592 | -0.92961 | 0.12347 |
| C | -1.65459 | -1.27830 | -0.13382 |
| C | -0.71122 | -0.19194 | 0.02868 |
| S | 0.00345 | 2.29052 | -0.43257 |
| C | 1.23230 | 1.05597 | -0.30678 |
| C | 0.74475 | -0.18660 | 0.01695 |
| N | 2.55302 | 1.34056 | -0.45489 |
| C | 3.49263 | 0.32764 | -0.30931 |
| N | 3.02146 | -0.87432 | 0.21644 |
| C | 1.69462 | -1.13860 | 0.54714 |
| S | 1.27047 | -2.37906 | 1.57329 |
| O | 4.66609 | 0.51247 | -0.58025 |
| O | -4.63571 | 0.63201 | 0.37187 |
| S | -1.23897 | -2.78488 | -0.70417 |
| C | -3.03712 | 2.76089 | -0.17843 |
| C | -3.07572 | 3.43725 | 1.18541 |
| C | -4.02271 | -1.96638 | 0.21443 |
| C | -4.74618 | -2.18618 | -1.10722 |
| C | 4.06694 | -1.88011 | 0.48738 |
| C | 4.18367 | -2.87164 | -0.66170 |
| C | 3.04329 | 2.68788 | -0.78189 |
| C | 3.37843 | 3.49035 | 0.46772 |
| H | -4.03379 | 2.70730 | -0.61347 |
| H | -2.39894 | 3.30098 | -0.88294 |
| H | -3.74839 | 2.89628 | 1.85405 |
| H | -3.44411 | 4.45975 | 1.07488 |
| H | -2.08267 | 3.47854 | 1.64114 |
| H | -4.71788 | -1.63399 | 0.98462 |
| H | -3.53021 | -2.87784 | 0.54821 |
| H | -5.24756 | -1.27102 | -1.43007 |
| H | -5.50330 | -2.96418 | -0.97946 |
| H | -4.04724 | -2.50776 | -1.88211 |
| H | 3.79587 | -2.38029 | 1.41515 |
| H | 4.99790 | -1.33702 | 0.63218 |
| H | 3.23995 | -3.40144 | -0.81613 |
| H | 4.95764 | -3.60846 | -0.43248 |
| H | 4.45814 | -2.35959 | -1.58743 |
| H | 2.28119 | 3.18163 | -1.39007 |
| H | 3.92424 | 2.55190 | -1.40722 |
| H | 2.50531 | 3.60562 | 1.11555 |
| H | 3.72686 | 4.48502 | 0.18014 |
| H | 4.17232 | 2.99671 | 1.03281 |

## [2b-$O_2S_2$]$^{+1}$

| | | | |
|---|---|---|---|
| C | -1.22802 | 1.10536 | -0.12105 |
| N | -2.54463 | 1.40841 | -0.13779 |
| C | -3.49050 | 0.41339 | 0.06271 |
| N | -2.99339 | -0.90020 | 0.13837 |
| C | -1.67288 | -1.23800 | 0.06844 |
| C | -0.72816 | -0.17096 | 0.04439 |
| S | -0.00977 | 2.31898 | -0.39384 |
| C | 1.21839 | 1.09225 | -0.26344 |
| C | 0.73019 | -0.17269 | 0.00099 |
| N | 2.53044 | 1.39116 | -0.37188 |
| C | 3.48853 | 0.40562 | -0.17628 |
| N | 3.00258 | -0.85507 | 0.21067 |
| C | 1.68194 | -1.18307 | 0.32253 |
| S | 1.25031 | -2.71879 | 0.90530 |
| O | 4.67189 | 0.62779 | -0.30713 |
| O | -4.67420 | 0.65866 | 0.13704 |
| S | -1.23627 | -2.87198 | -0.08029 |
| C | -3.03978 | 2.79378 | -0.27291 |
| C | -3.15508 | 3.48889 | 1.07584 |
| C | -4.05014 | -1.93650 | 0.22605 |
| C | -4.57101 | -2.33472 | -1.14755 |
| C | 4.06578 | -1.85445 | 0.47746 |
| C | 4.43173 | -2.62424 | -0.78272 |
| C | 3.01653 | 2.75698 | -0.65824 |
| C | 3.30038 | 3.53740 | 0.61684 |
| H | -4.00876 | 2.72406 | -0.76368 |
| H | -2.36384 | 3.31871 | -0.95222 |
| H | -3.86394 | 2.96032 | 1.71628 |
| H | -3.52007 | 4.50658 | 0.92298 |
| H | -2.18959 | 3.54494 | 1.58541 |
| H | -4.84338 | -1.50807 | 0.83494 |
| H | -3.62146 | -2.78472 | 0.75827 |
| H | -5.01910 | -1.47685 | -1.65339 |
| H | -5.33937 | -3.10145 | -1.02676 |
| H | -3.77457 | -2.74374 | -1.77374 |
| H | 3.70395 | -2.51295 | 1.26603 |
| H | 4.91877 | -1.29899 | 0.86051 |
| H | 3.56892 | -3.16067 | -1.18461 |
| H | 5.20782 | -3.35394 | -0.54182 |
| H | 4.82097 | -1.94745 | -1.54628 |
| H | 2.26767 | 3.24683 | -1.28476 |
| H | 3.91820 | 2.63962 | -1.25683 |
| H | 2.40569 | 3.63921 | 1.23625 |
| H | 3.64917 | 4.53770 | 0.35209 |
| H | 4.08071 | 3.04371 | 1.19945 |

## 3b-$OS_3$

| | | | |
|---|---|---|---|
| C | -1.17697 | 0.95038 | -0.30130 |
| N | -2.50308 | 1.26892 | -0.36563 |
| C | -3.44764 | 0.28511 | -0.17168 |
| N | -2.99081 | -1.02080 | -0.21486 |
| C | -1.64614 | -1.40189 | -0.34824 |
| C | -0.70473 | -0.32856 | -0.15376 |
| S | 0.07942 | 2.15045 | -0.44225 |
| C | 1.27923 | 0.89876 | -0.22649 |
| C | 0.73904 | -0.34350 | -0.04137 |
| N | 2.61891 | 1.17717 | -0.21068 |
| C | 3.53099 | 0.16423 | 0.01134 |
| N | 3.00925 | -1.05658 | 0.35626 |
| C | 1.62520 | -1.37216 | 0.47293 |
| O | 1.28157 | -2.41103 | 0.99833 |
| S | 5.18501 | 0.44393 | -0.11634 |
| S | -5.06063 | 0.68262 | 0.07137 |
| S | -1.18754 | -2.93702 | -0.78289 |
| C | -2.89435 | 2.68842 | -0.49054 |
| C | -2.93711 | 3.40615 | 0.85160 |

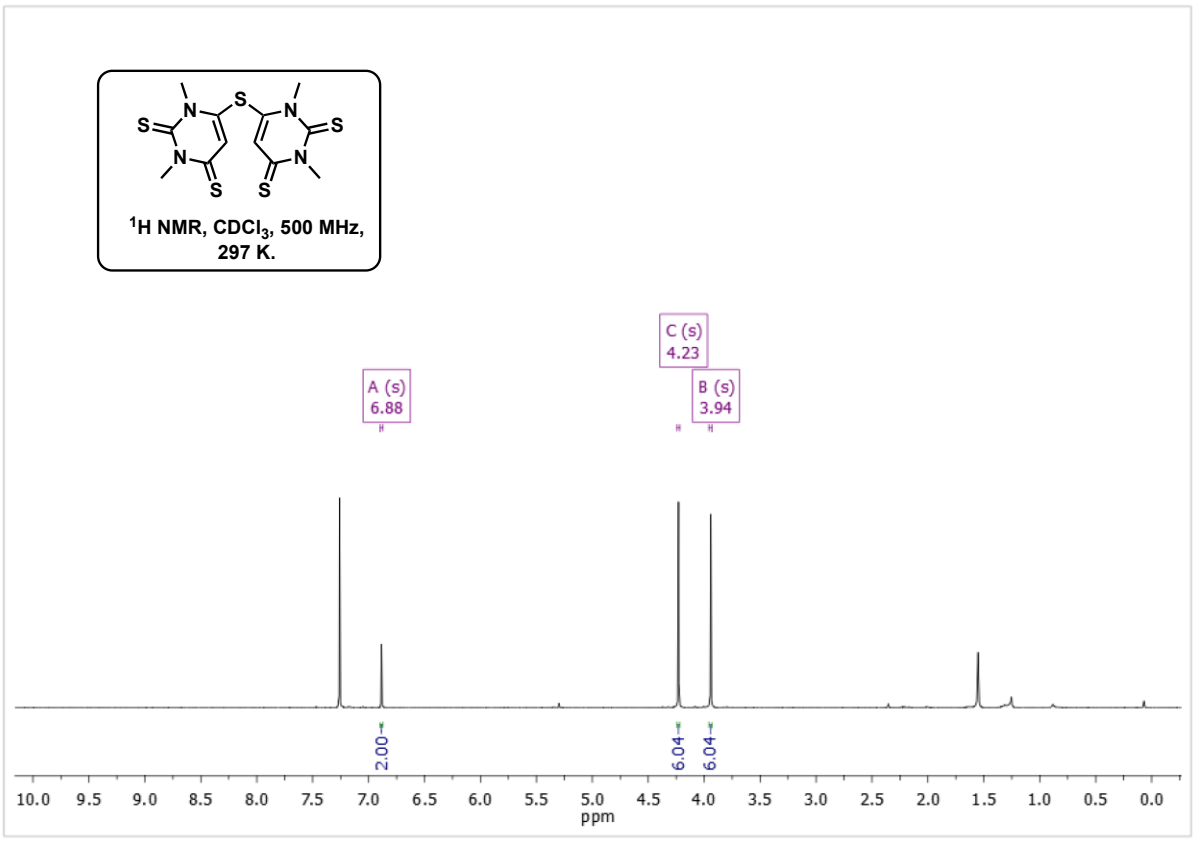
| | | | |
|---|---|---|---|
| C | -3.99914 | -2.10314 | -0.13633 |
| C | -4.17214 | -2.59125 | 1.29428 |
| C | 3.91657 | -2.17320 | 0.69161 |
| C | 4.20396 | -3.03835 | -0.52722 |
| C | 3.06610 | 2.57052 | -0.40792 |
| C | 3.20369 | 3.31566 | 0.91166 |
| H | -3.86905 | 2.70508 | -0.97344 |
| H | -2.18032 | 3.15172 | -1.17664 |
| H | -3.68942 | 2.95379 | 1.50014 |
| H | -3.20382 | 4.45250 | 0.68641 |
| H | -1.96936 | 3.37776 | 1.35908 |
| H | -3.64415 | -2.90165 | -0.78345 |
| H | -4.92912 | -1.70773 | -0.53490 |
| H | -3.23226 | -2.98595 | 1.68876 |
| H | -4.91428 | -3.39317 | 1.31327 |
| H | -4.52063 | -1.78323 | 1.94202 |
| H | 3.41571 | -2.74859 | 1.46696 |
| H | 4.82888 | -1.74234 | 1.09708 |
| H | 3.28019 | -3.46819 | -0.92297 |
| H | 4.86871 | -3.85805 | -0.24328 |
| H | 4.69107 | -2.45508 | -1.31250 |
| H | 2.34628 | 3.05218 | -1.07404 |
| H | 4.01572 | 2.52874 | -0.93687 |
| H | 2.25782 | 3.34332 | 1.45933 |
| H | 3.51537 | 4.34390 | 0.71427 |
| H | 3.96056 | 2.83946 | 1.53885 |

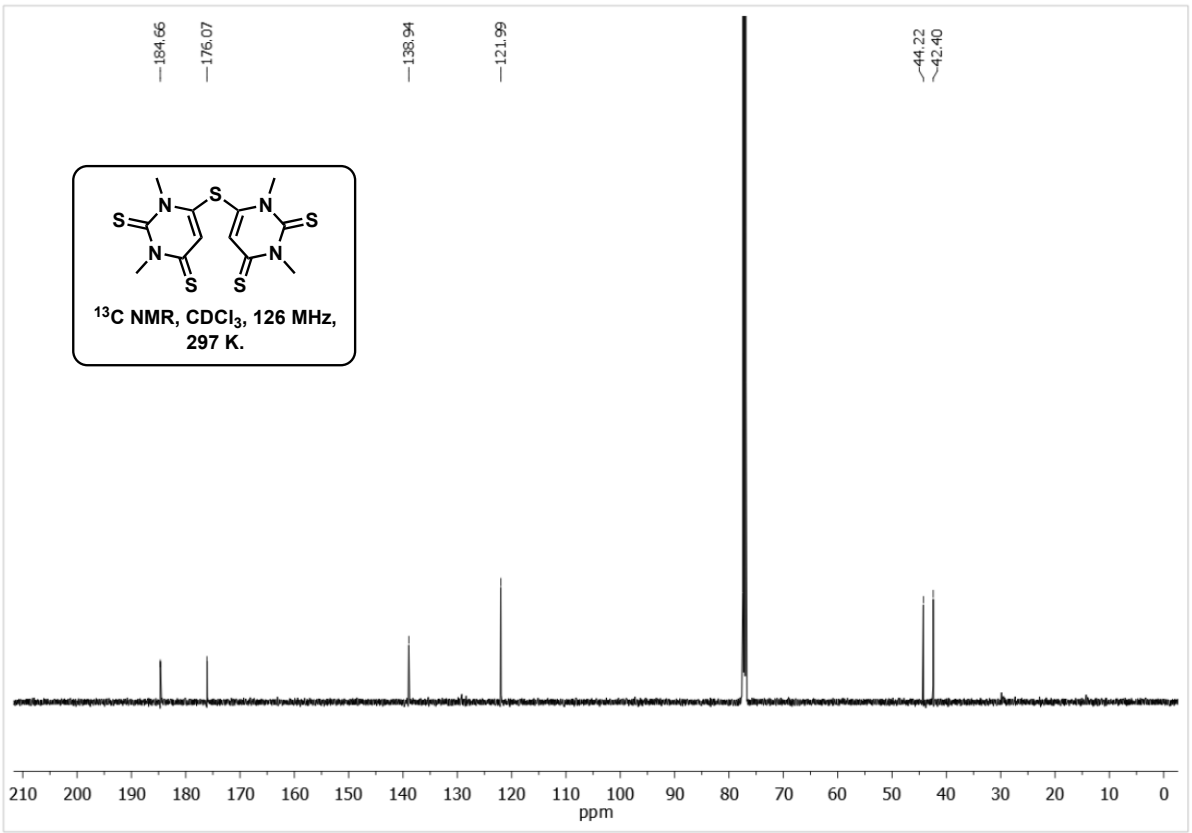
## [3b-OS$_3$]$^{+1}$

| | | | |
|---|---|---|---|
| C | -1.17468 | 0.98795 | -0.35002 |
| N | -2.47144 | 1.27873 | -0.36540 |
| C | -3.42943 | 0.26343 | -0.12555 |
| N | -2.97531 | -1.03279 | -0.23493 |
| C | -1.63467 | -1.40394 | -0.39344 |
| C | -0.68447 | -0.32701 | -0.20224 |
| S | 0.07552 | 2.19409 | -0.47654 |
| C | 1.26632 | 0.93701 | -0.22015 |
| C | 0.71126 | -0.33289 | -0.03760 |
| N | 2.57733 | 1.18922 | -0.20738 |
| C | 3.50496 | 0.14461 | 0.01976 |
| N | 2.97407 | -1.06133 | 0.39938 |
| C | 1.59943 | -1.38106 | 0.47034 |
| O | 1.22853 | -2.42516 | 0.95518 |
| S | 5.12843 | 0.41718 | -0.14989 |
| S | -4.98858 | 0.67352 | 0.23348 |
| S | -1.15802 | -2.91042 | -0.86052 |
| C | -2.90065 | 2.69550 | -0.51357 |
| C | -2.92296 | 3.44172 | 0.81335 |
| C | -3.98551 | -2.12041 | -0.18088 |
| C | -4.12836 | -2.67617 | 1.22734 |
| C | 3.88067 | -2.17154 | 0.77670 |
| C | 4.20003 | -3.06032 | -0.41617 |
| C | 3.05672 | 2.58166 | -0.39925 |
| C | 3.20905 | 3.31043 | 0.92693 |
| H | -3.88567 | 2.67545 | -0.97359 |
| H | -2.21278 | 3.14942 | -1.23106 |
| H | -3.66273 | 3.00739 | 1.48669 |
| H | -3.19788 | 4.47988 | 0.61674 |
| H | -1.94774 | 3.43661 | 1.30578 |
| H | -3.64994 | -2.88554 | -0.87727 |
| H | -4.92223 | -1.70643 | -0.54427 |
| H | -3.18397 | -3.09575 | 1.58303 |
| H | -4.87398 | -3.47428 | 1.21868 |
| H | -4.46019 | -1.90217 | 1.92312 |
| H | 3.36359 | -2.73032 | 1.55368 |
| H | 4.77894 | -1.72683 | 1.19870 |
| H | 3.28985 | -3.51002 | -0.82018 |
| H | 4.86502 | -3.86520 | -0.09479 |
| H | 4.70072 | -2.49495 | -1.20555 |
| H | 2.34624 | 3.07672 | -1.06460 |
| H | 4.00294 | 2.51733 | -0.93041 |
| H | 2.26297 | 3.36626 | 1.47113 |
| H | 3.55017 | 4.32842 | 0.72835 |
| H | 3.95199 | 2.81484 | 1.55494 |

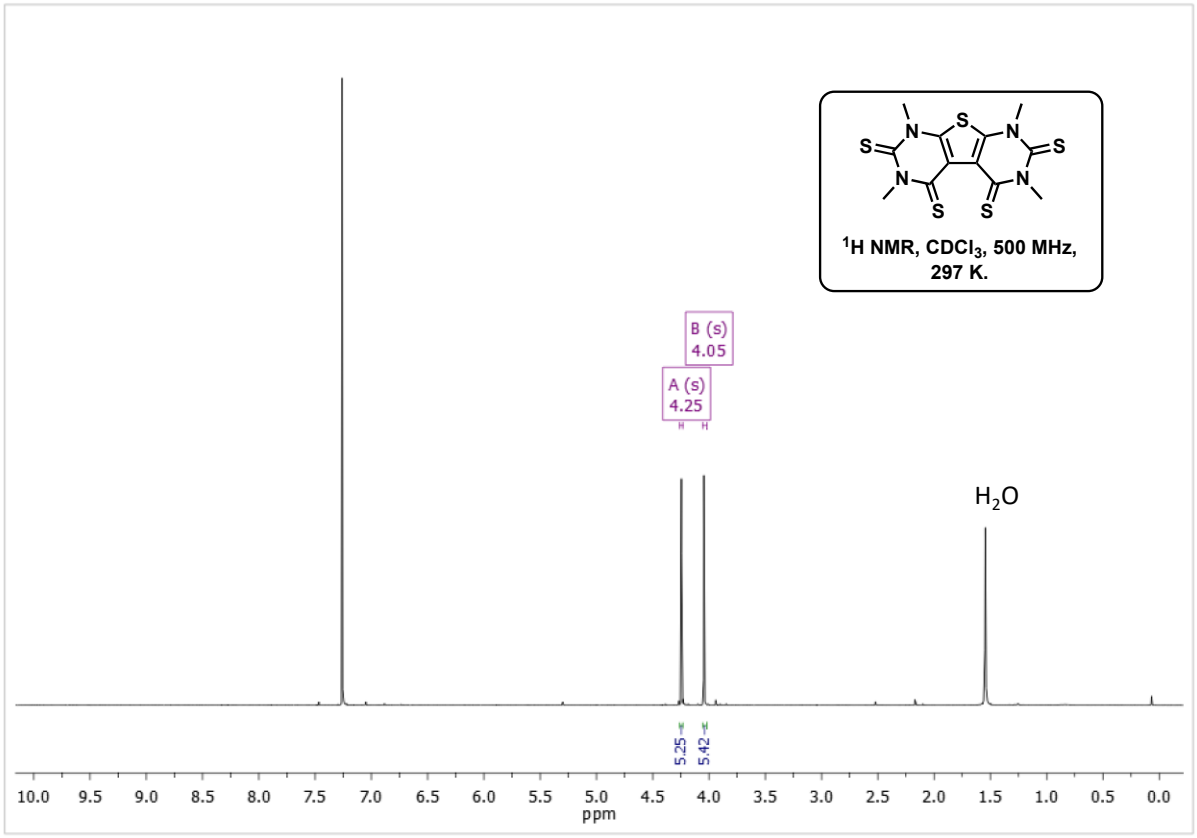
 

# 5. $^{1}$H and $^{13}$C NMR Spectra

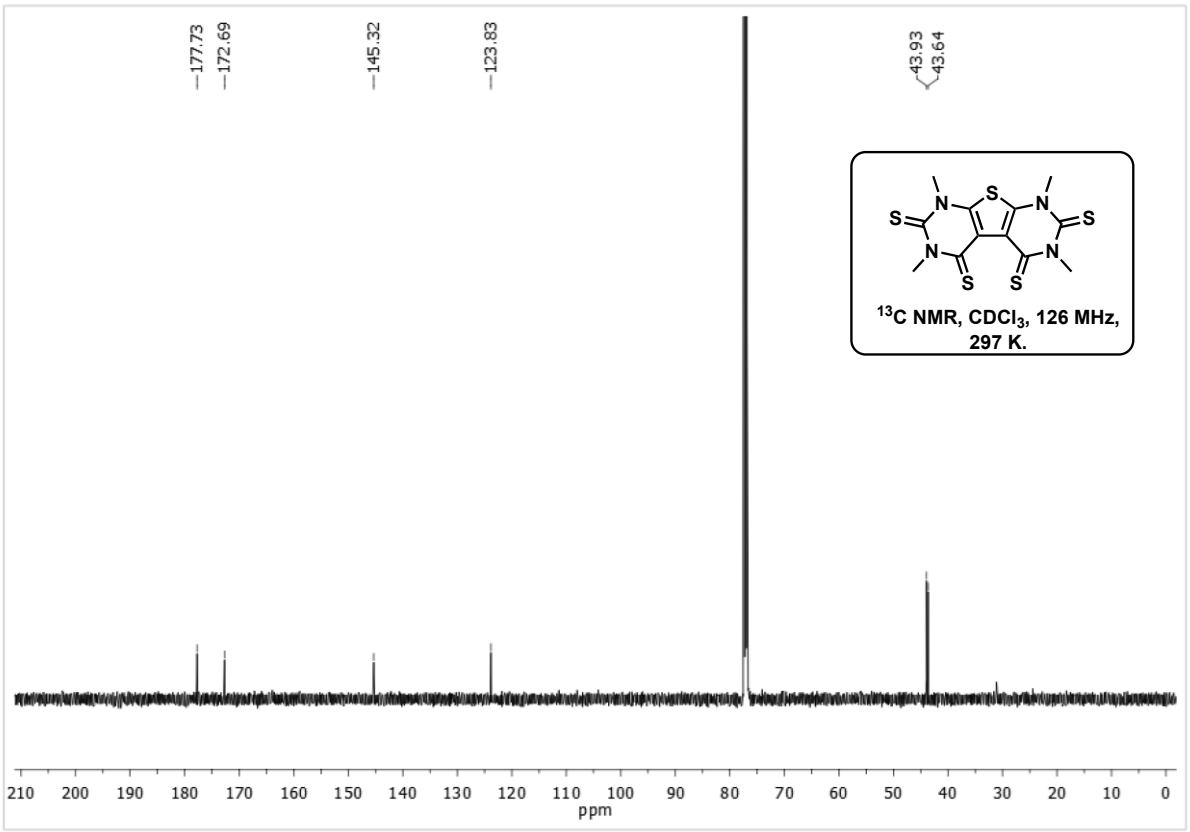


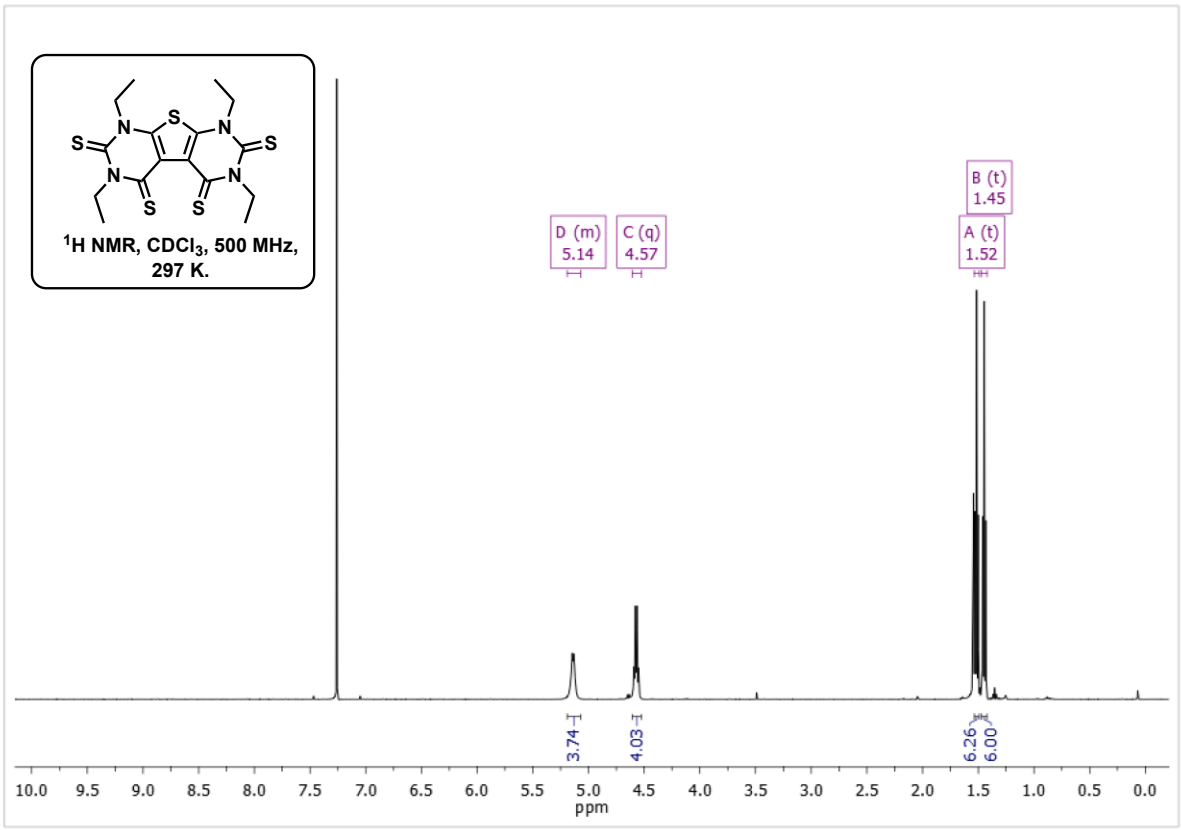

 

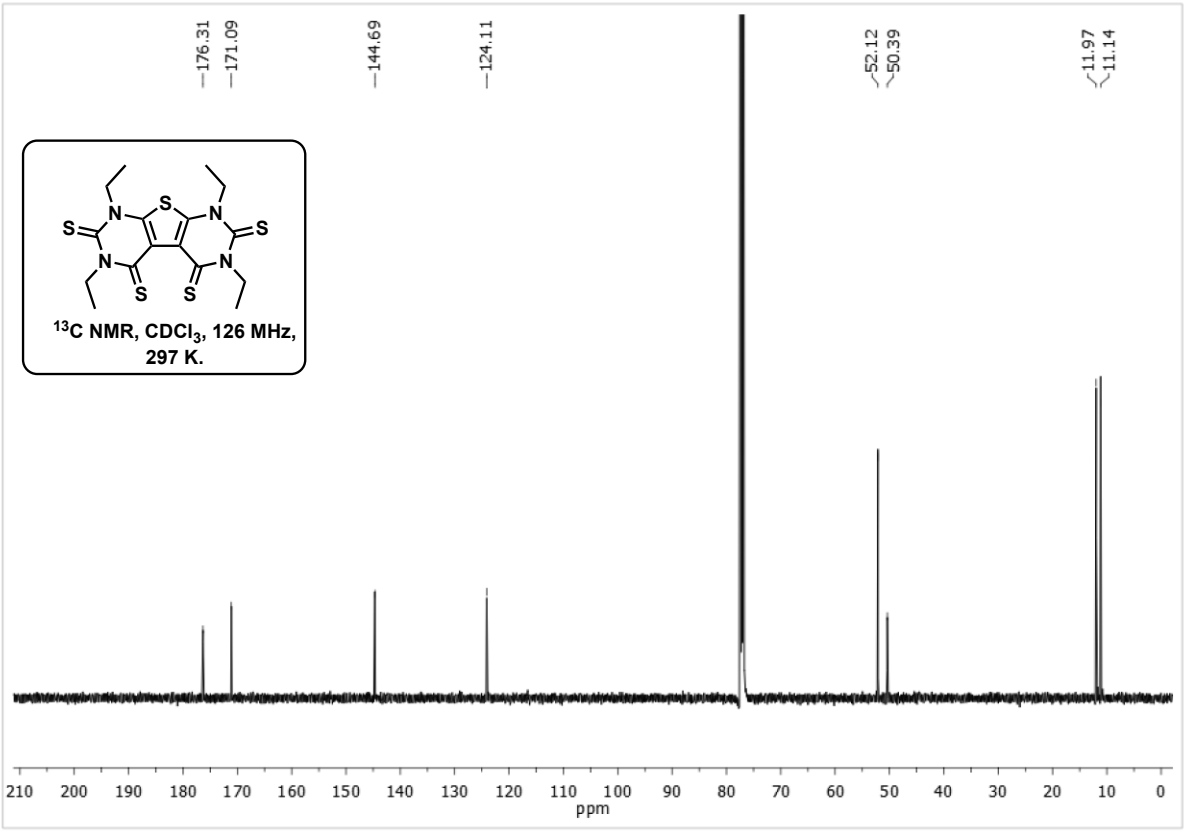



$^{1}$H NMR, $CDCl_3$, 500 MHz, 297 K.

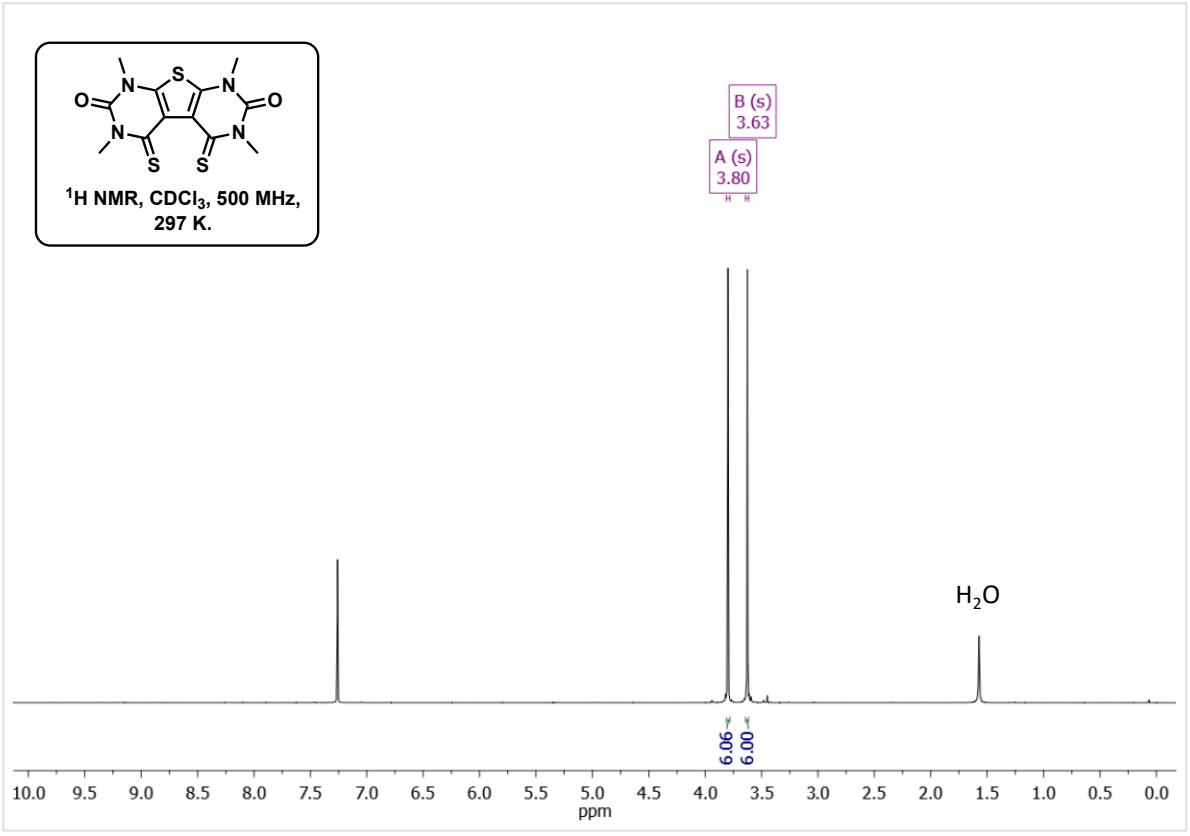



$^{13}$C NMR, $CDCl_3$, 126 MHz, 297 K.

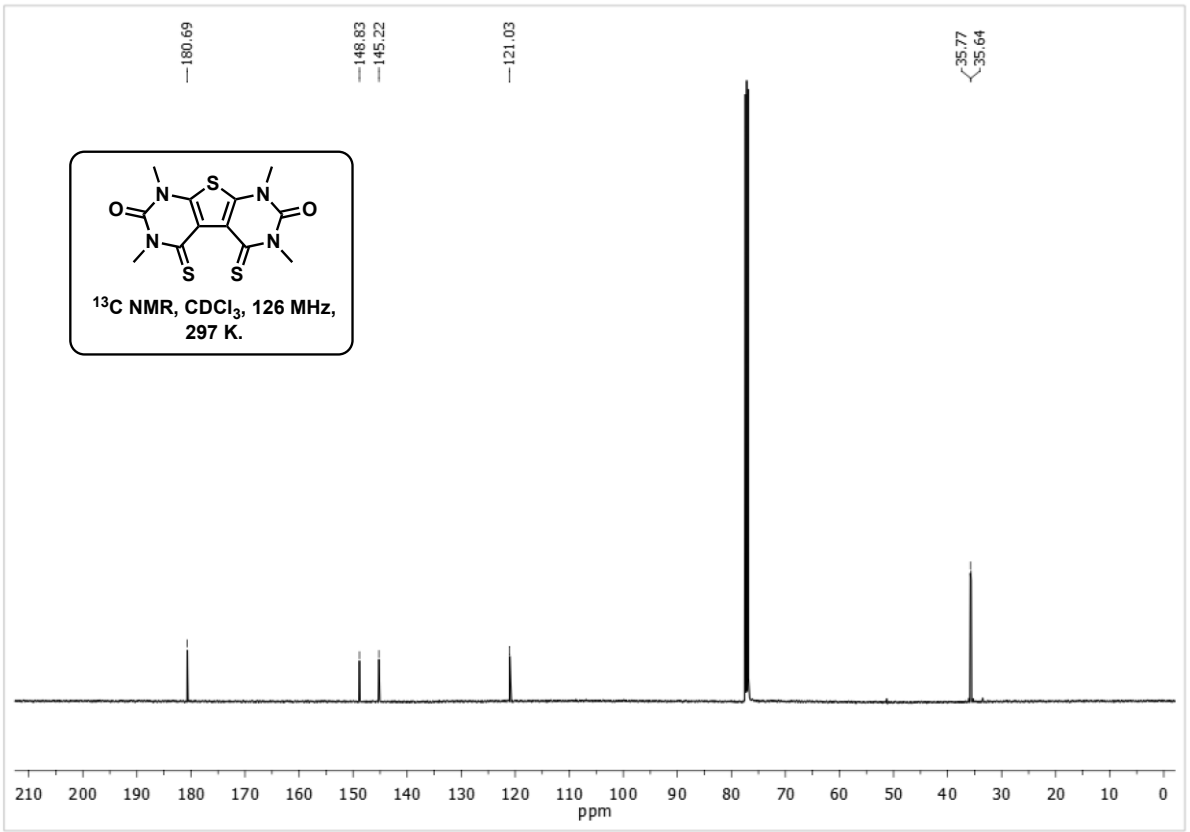

$^1$H NMR, $CDCl_3$, 500 MHz, 297 K.
D (m) 5.14
C (q) 4.57
B (t) 1.45
A (t) 1.52
3.74
4.03
6.26
6.00
ppm


176.31
171.09
144.69
124.11
52.12
50.39
11.97
11.14
$^{13}$C NMR, $CDCl_3$, 126 MHz, 297 K.
ppm

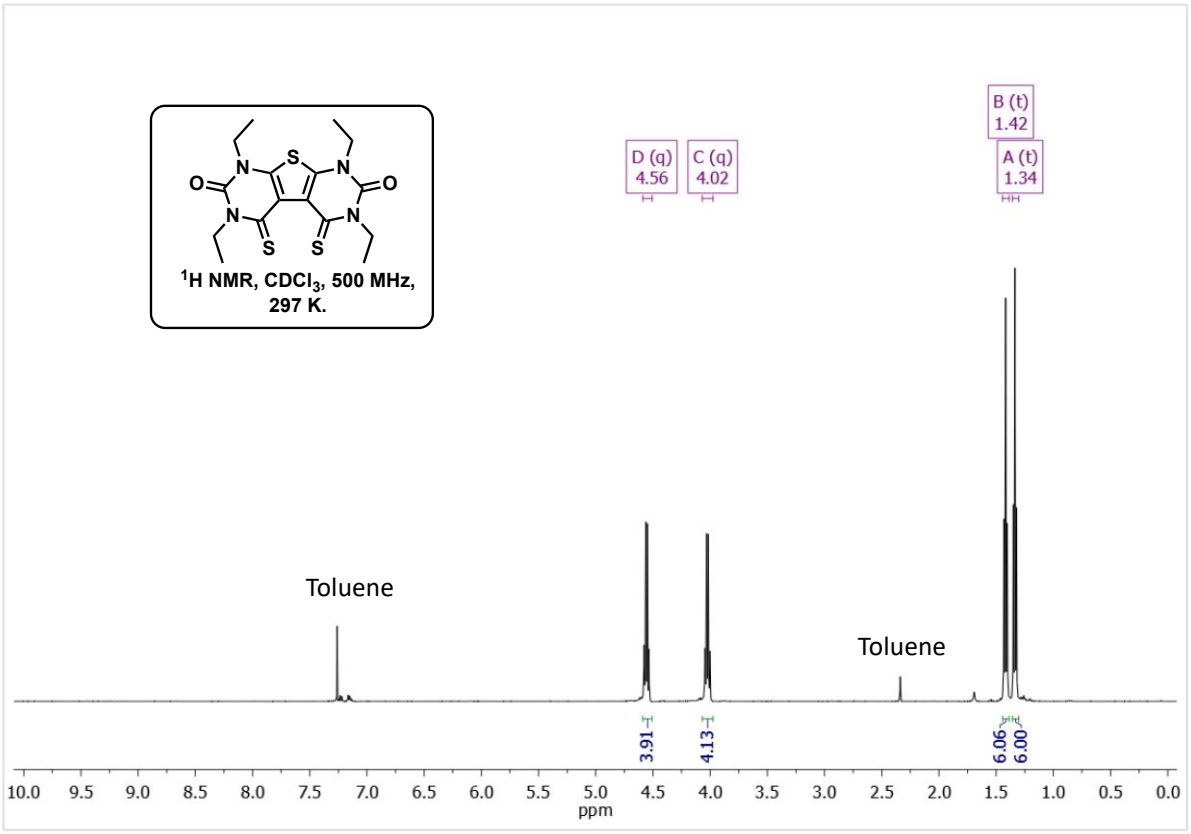
 

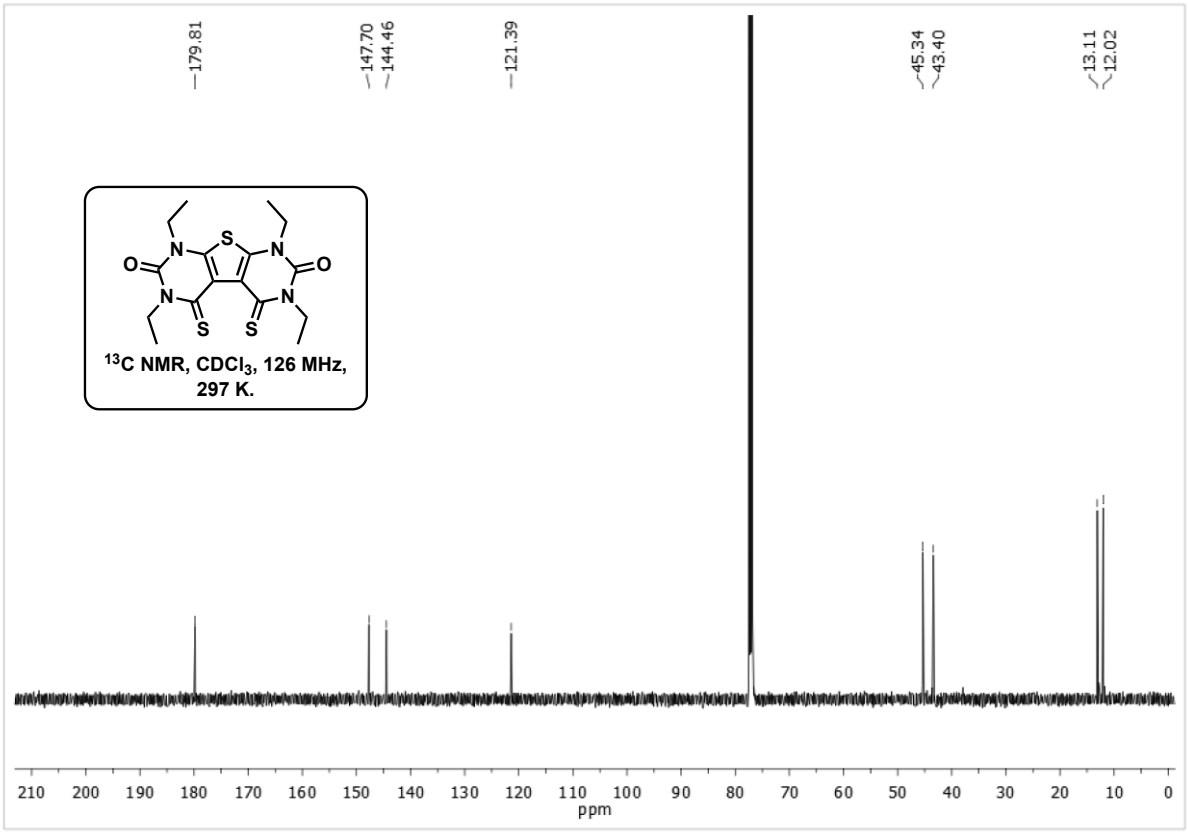



$^{1}$H NMR, $CDCl_3$, 500 MHz, 297 K.

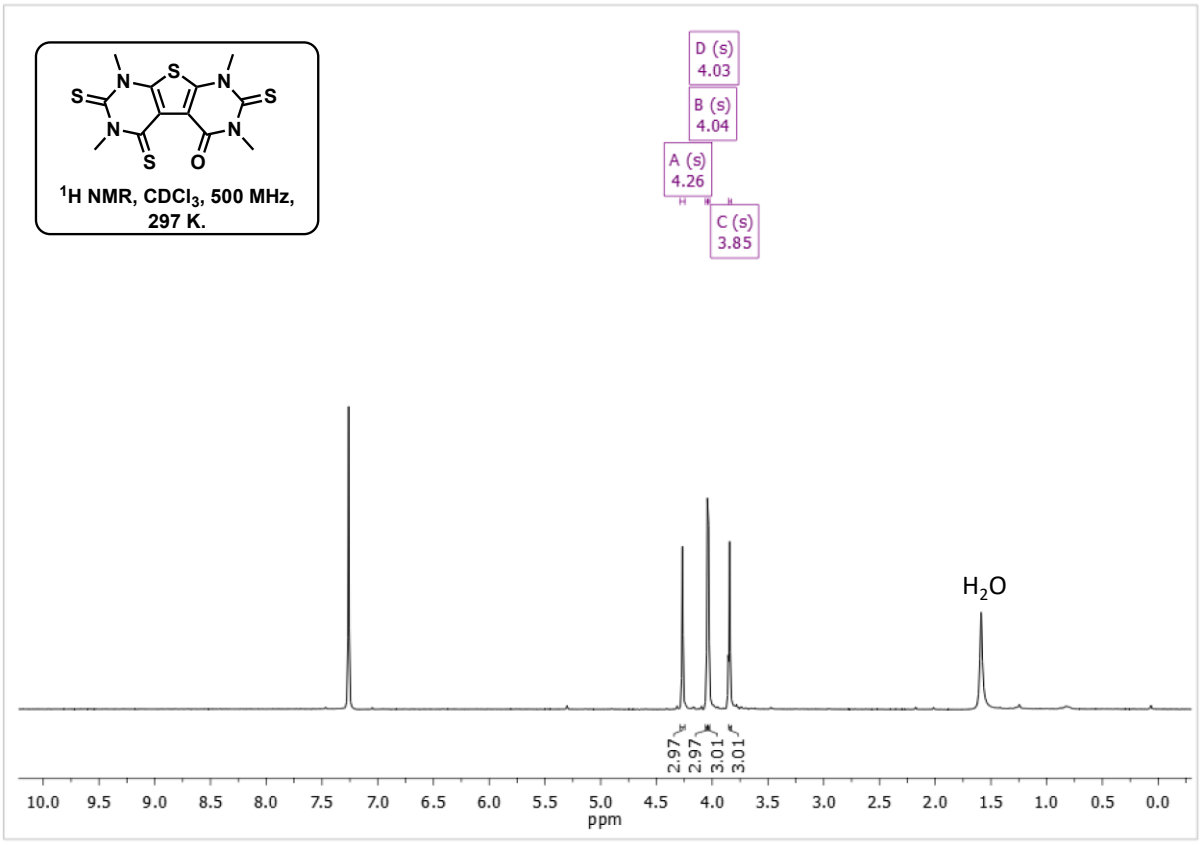


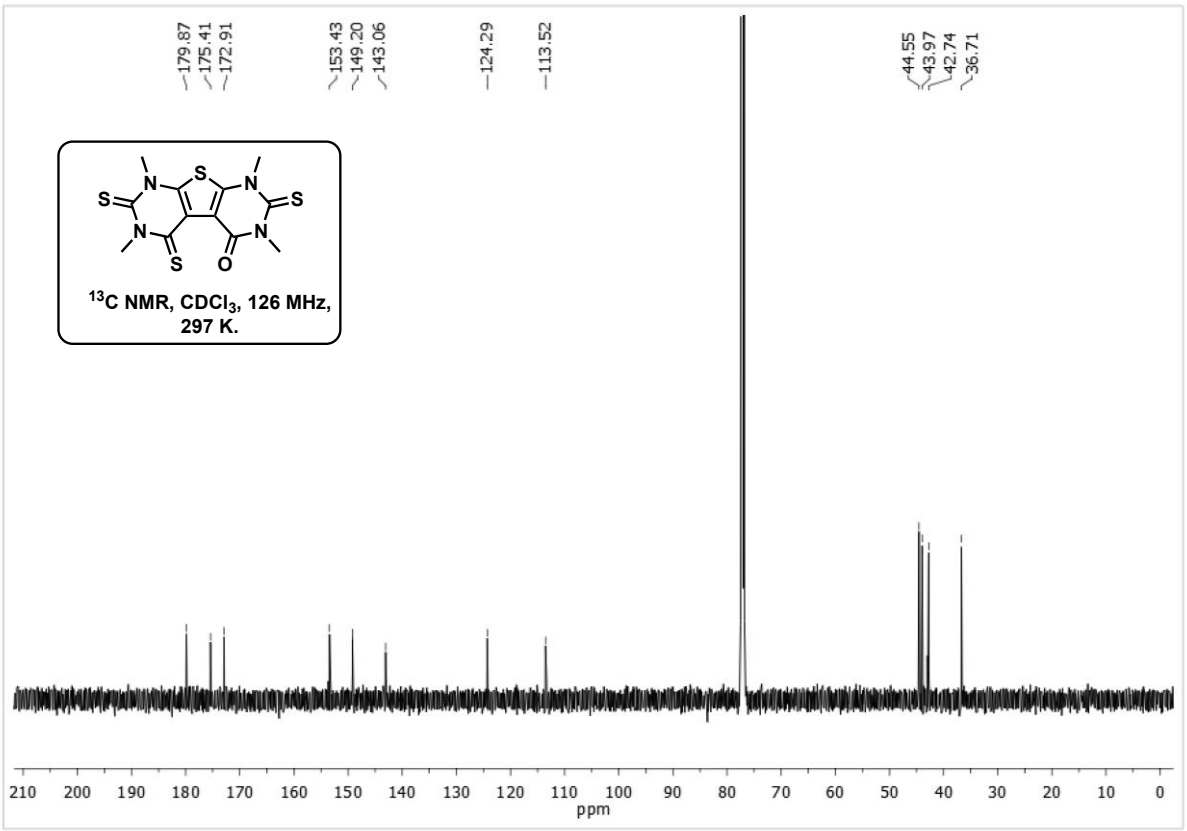

$^{13}$C NMR, $CDCl_3$, 126 MHz, 297 K.


$^1$H NMR, CDCl$_3$, 500 MHz, 297 K.
D (q) 4.56
C (q) 4.02
B (t) 1.42
A (t) 1.34
Toluene
Toluene
3.91
4.13
6.06
6.00
ppm

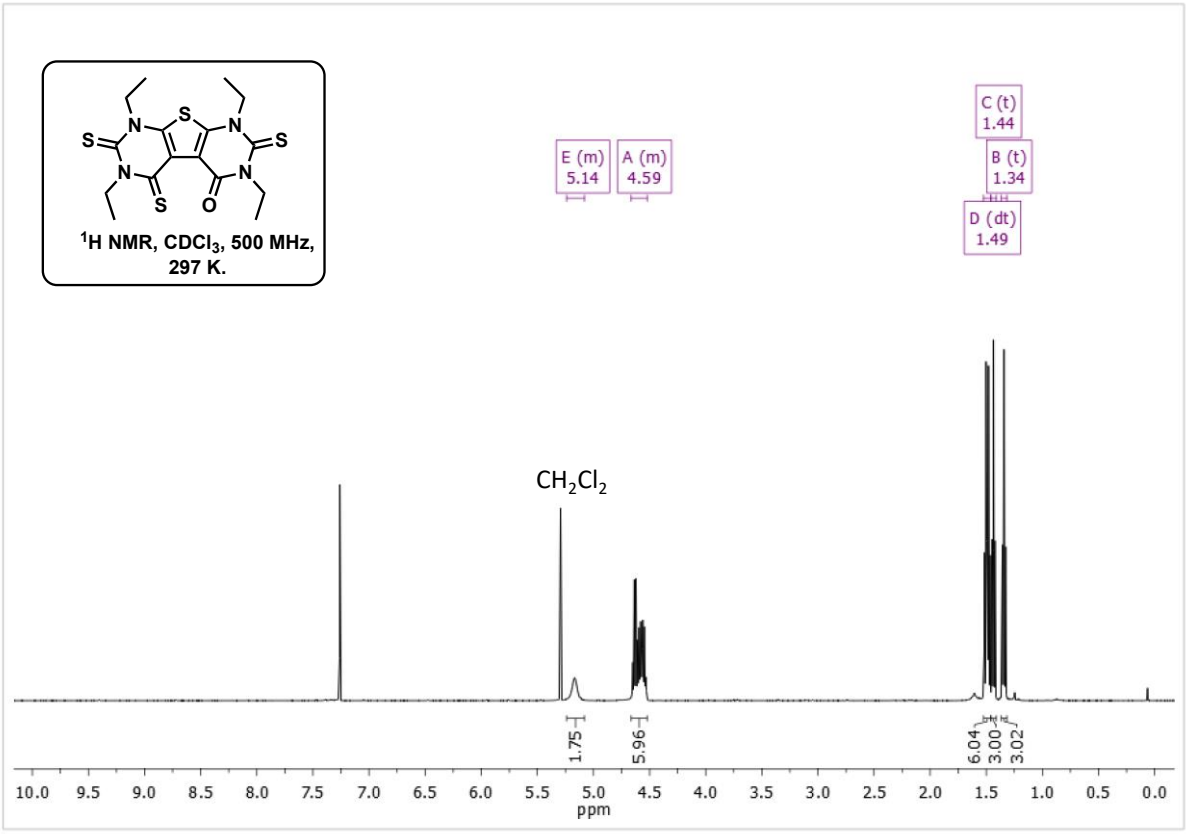

179.81
147.70
144.46
121.39
45.34
43.40
13.11
12.02
$^{13}$C NMR, CDCl$_3$, 126 MHz, 297 K.
ppm

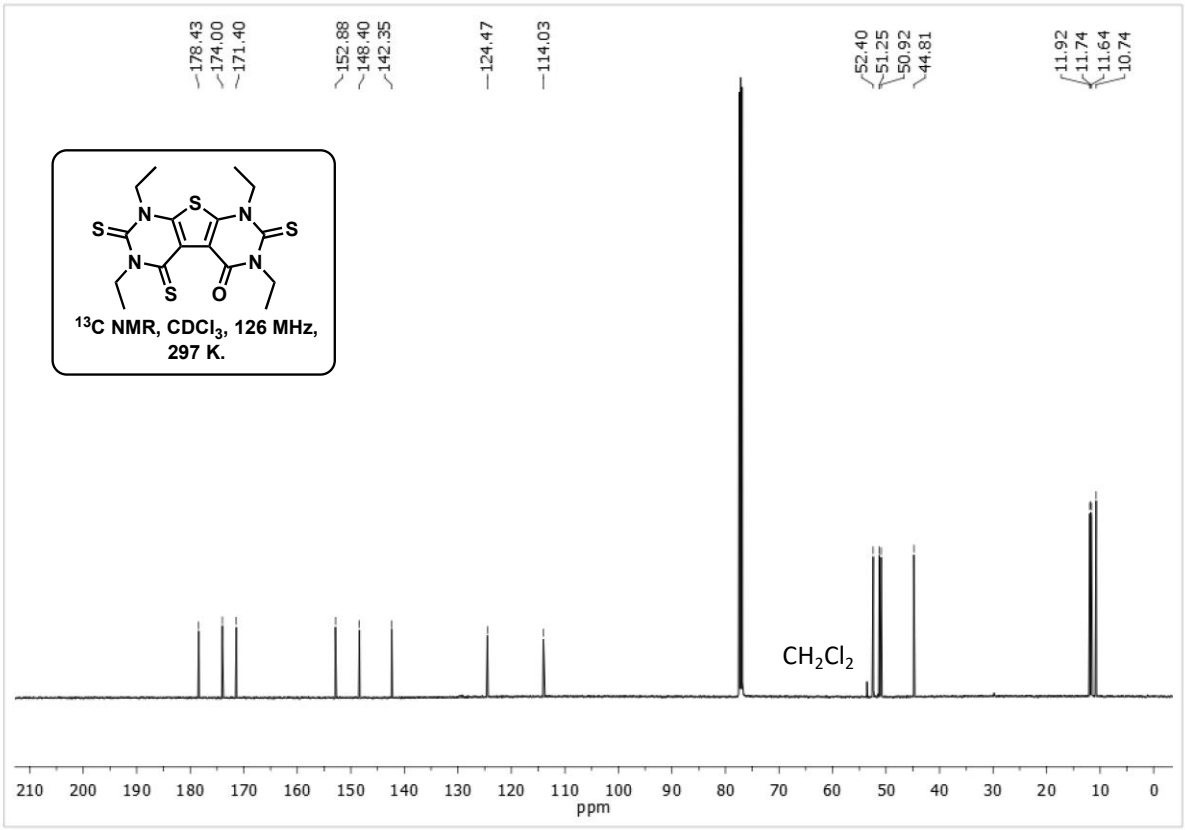
 


$^1$H NMR, $CDCl_3$, 500 MHz, 297 K.
D (s) 4.03
B (s) 4.04
A (s) 4.26
C (s) 3.85
$H_2O$
2.97
2.97
3.01
3.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm

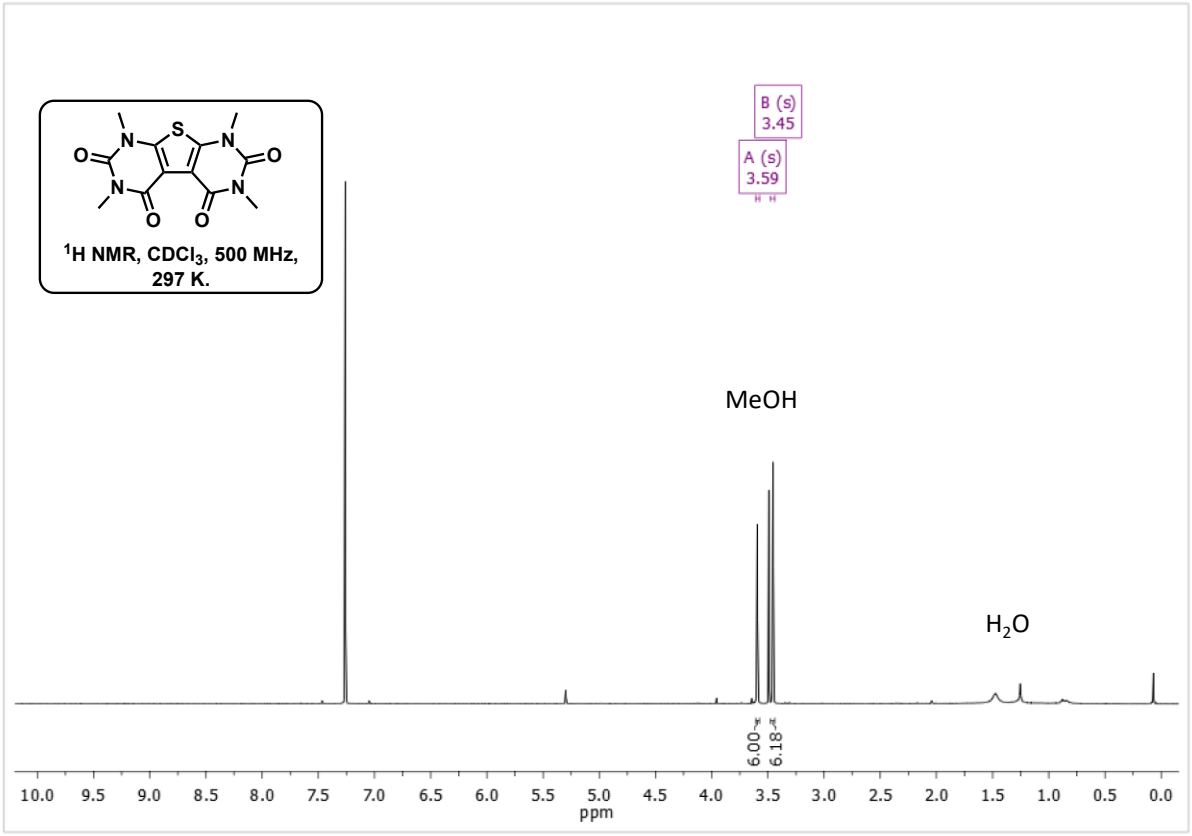

179.87
175.41
172.91
153.43
149.20
143.06
124.29
113.52
44.55
43.97
42.74
36.71
$^{13}$C NMR, $CDCl_3$, 126 MHz, 297 K.
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

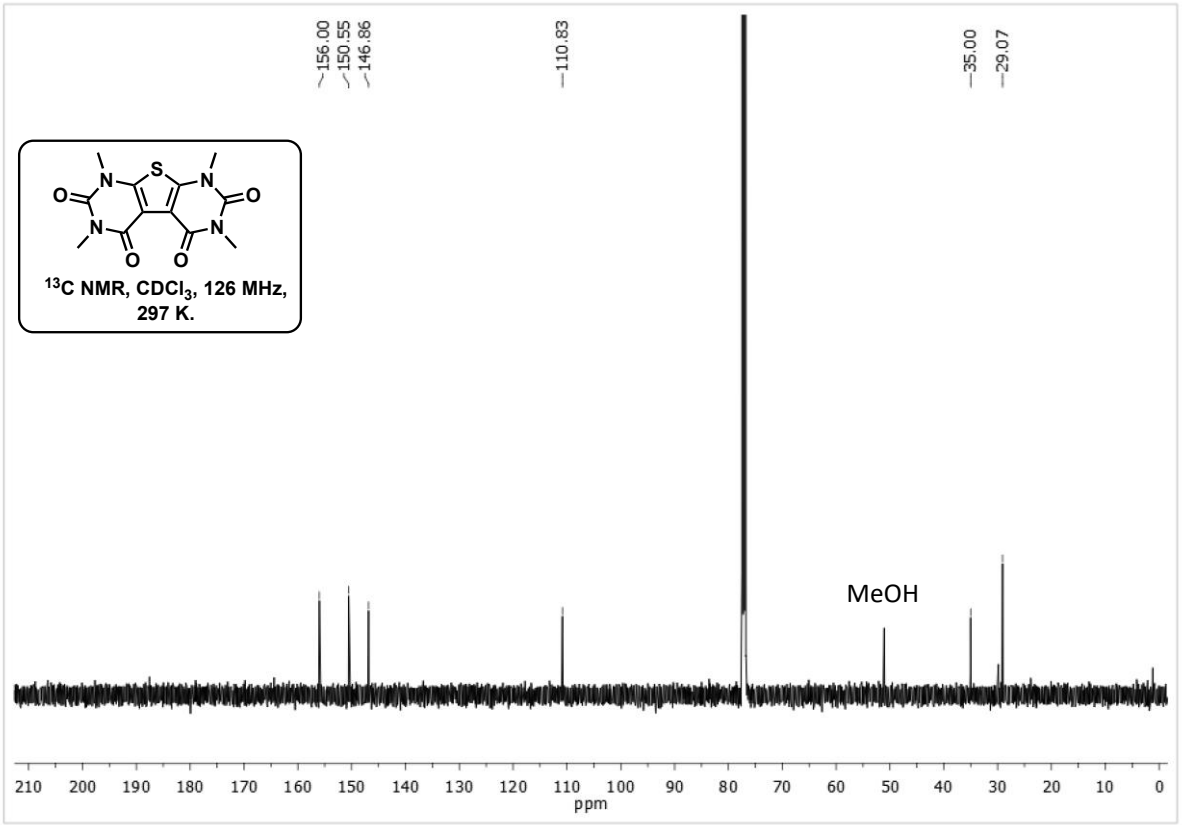

$^1$H NMR, $CDCl_3$, 500 MHz, 297 K.
E (m) 5.14
A (m) 4.59
C (t) 1.44
B (t) 1.34
D (dt) 1.49
$CH_2Cl_2$
1.75
5.96
6.04
3.00
3.02
ppm


178.43
174.00
171.40
152.88
148.40
142.35
124.47
114.03
52.40
51.25
50.92
44.81
11.92
11.74
11.64
10.74
$^{13}$C NMR, $CDCl_3$, 126 MHz, 297 K.
$CH_2Cl_2$
ppm



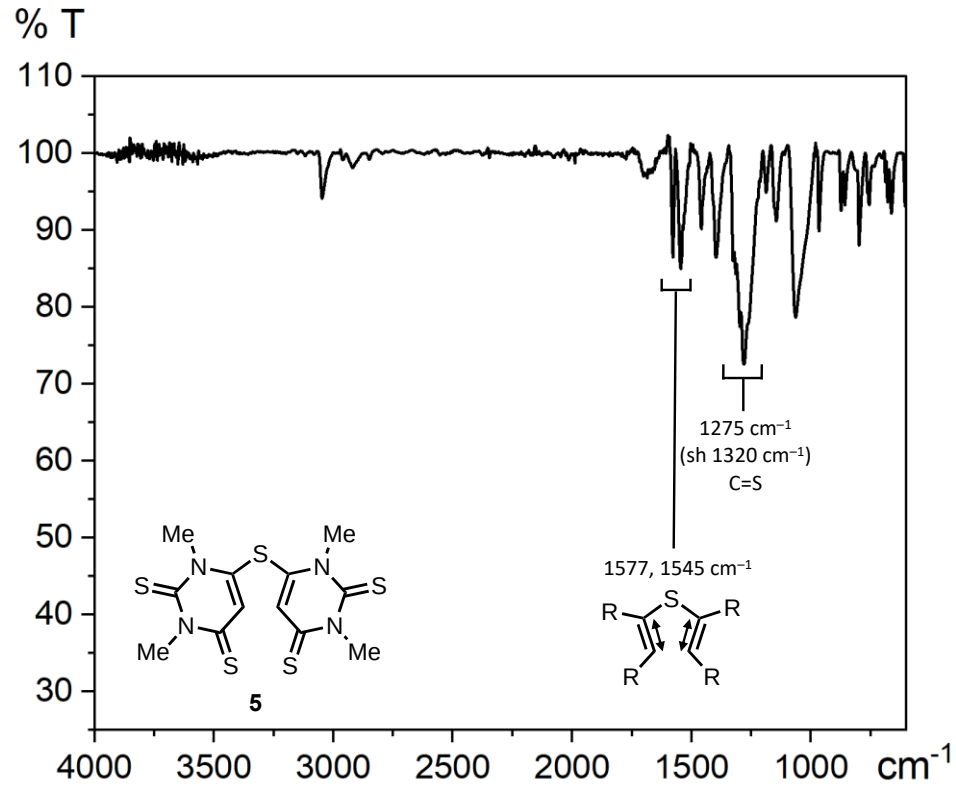

$^1$H NMR, $CDCl_3$, 500 MHz, 297 K.

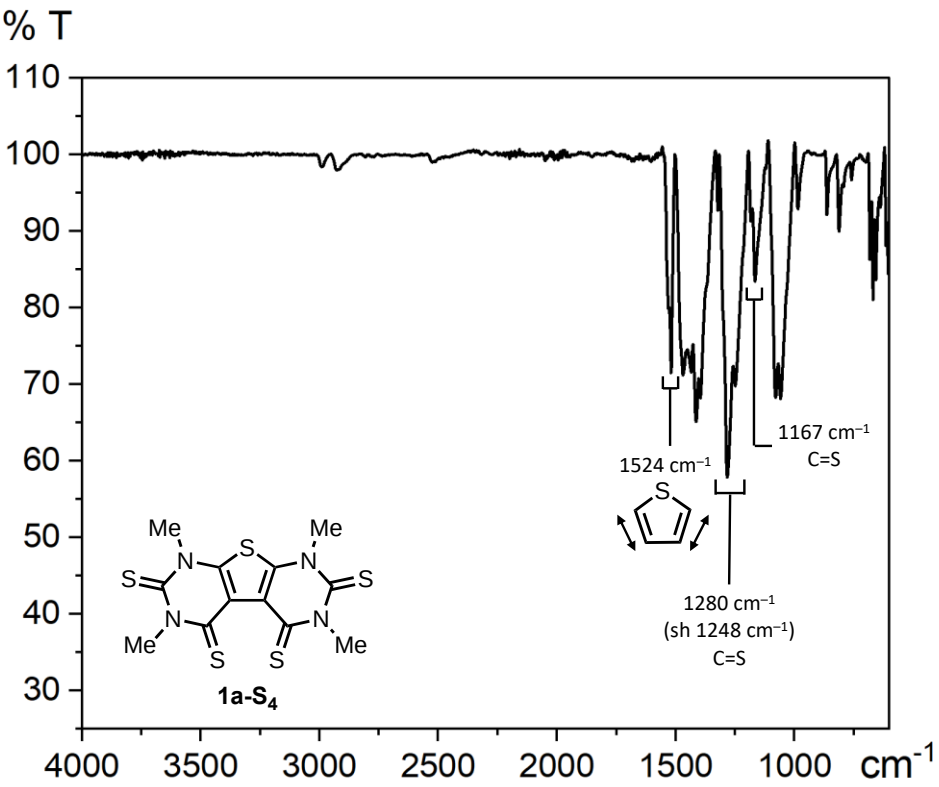



$^{13}$C NMR, $CDCl_3$, 126 MHz, 297 K.

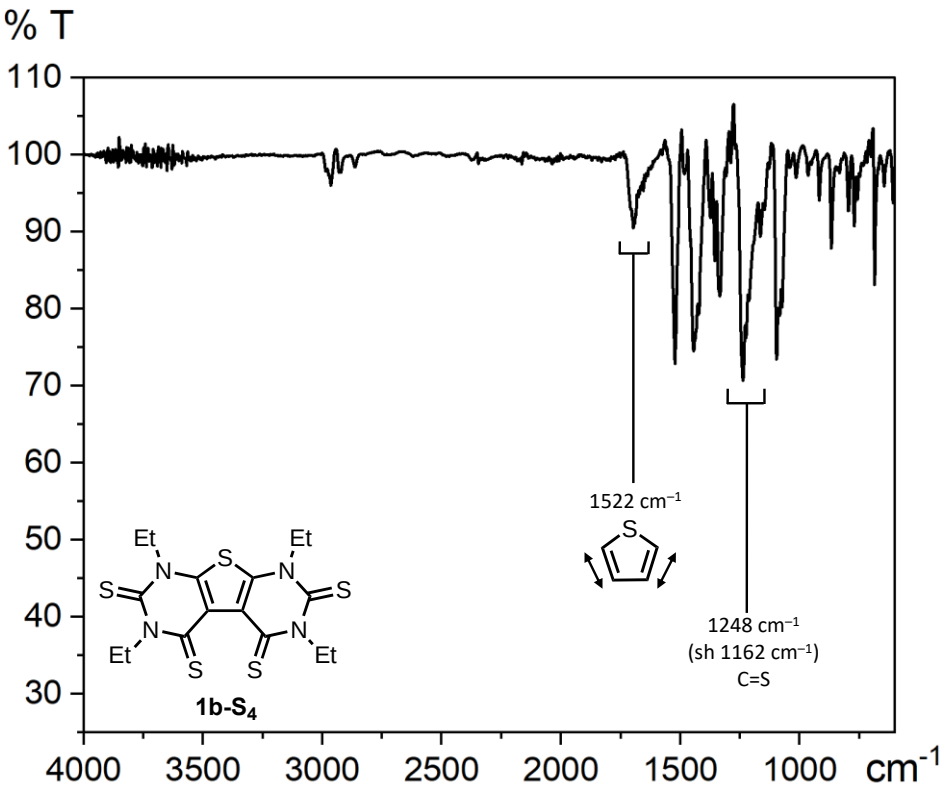
 

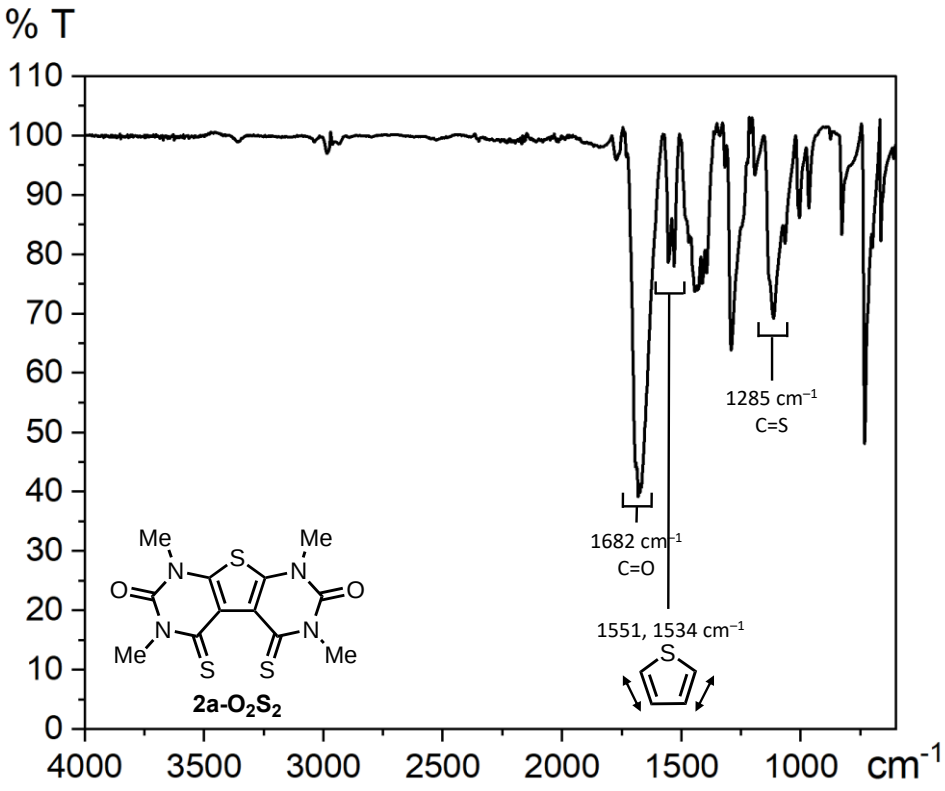
## 6. Infrared Spectra



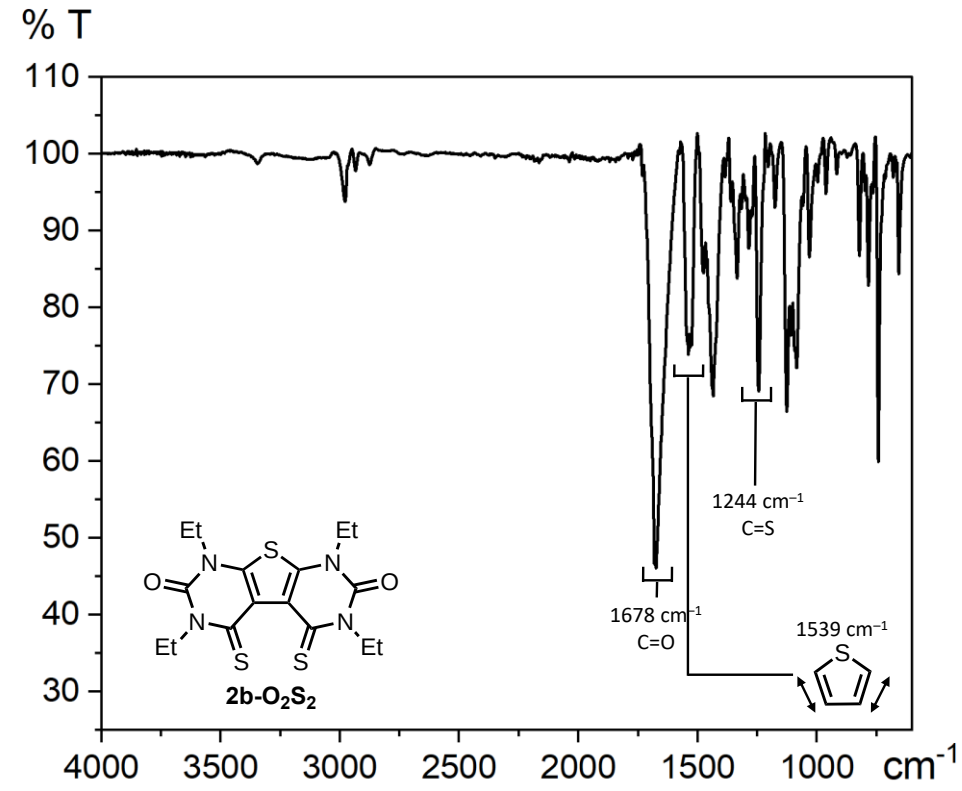



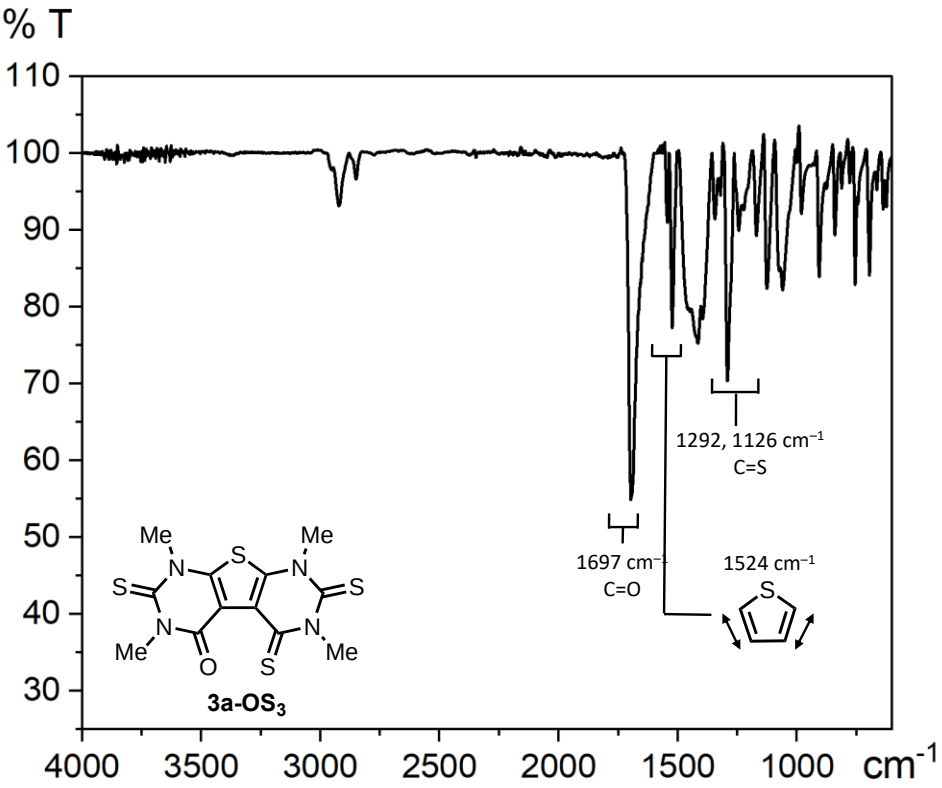

 


% T
110
100
90
80
70
60
50
40
30
4000
3500
3000
2500
2000
1500
1000
cm-1
1522 cm–1
1248 cm–1
(sh 1162 cm–1)
C=S
Et
N
S
S
Et
1b-S4

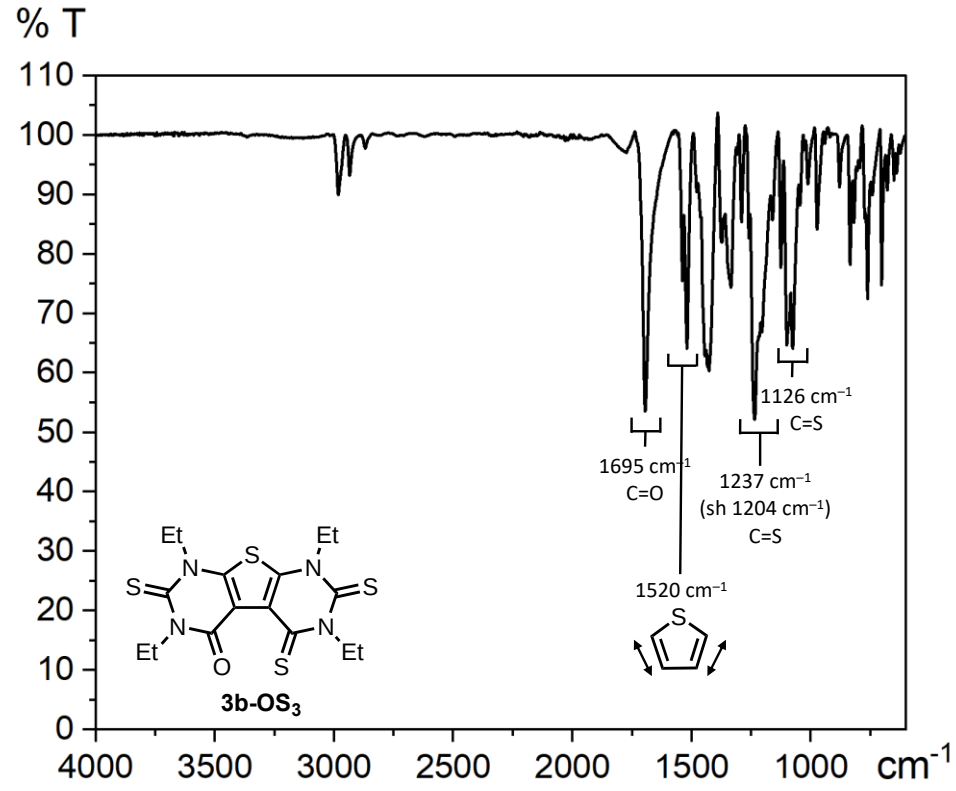

% T
110
100
90
80
70
60
50
40
30
20
10
0
4000
3500
3000
2500
2000
1500
1000
cm-1
1285 cm–1
C=S
1682 cm–1
C=O
1551, 1534 cm–1
Me
N
O
S
2a-O2S2

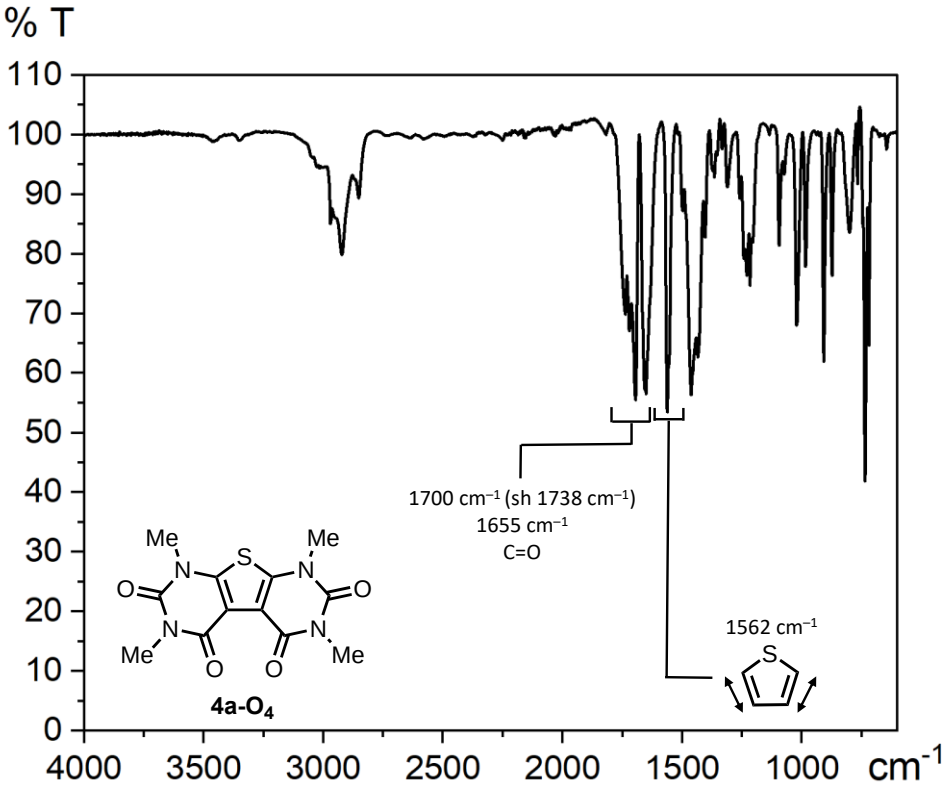
 


% T
110
100
90
80
70
60
50
40
30
4000
3500
3000
2500
2000
1500
1000
cm-1
1244 cm−1
C=S
1678 cm−1
C=O
1539 cm−1
Et
N
S
O
2b-O2S2


% T
110
100
90
80
70
60
50
40
30
4000
3500
3000
2500
2000
1500
1000
cm-1
1292, 1126 cm−1
C=S
1697 cm−1
C=O
1524 cm−1
Me
N
S
3a-OS3


% T
110
100
90
80
70
60
50
40
30
20
10
0
4000
3500
3000
2500
2000
1500
1000
cm-1
1695 cm-1
C=O
1520 cm-1
1126 cm-1
C=S
1237 cm-1
(sh 1204 cm-1)
C=S
Et
N
S
S
N
Et
Et
N
S
S
N
Et
O
S
3b-OS3


% T
110
100
90
80
70
60
50
40
30
20
10
0
4000
3500
3000
2500
2000
1500
1000
cm-1
1700 cm-1 (sh 1738 cm-1)
1655 cm-1
C=O
1562 cm-1
Me
N
S
N
Me
O
O
N
N
Me
O
O
4a-O4

## 7. References

1. Connelly, N. G.; Geiger, W. E. *Chem. Rev.* **1996**, *96*, 877.
2. Sheldrick, G. M. *Acta Crystallogr. Sect. C Struct. Chem.* **2015**, *71*, 3.
3. Sheldrick, G. M. *Acta Crystallogr. Sect. A Found. Crystallogr.* **2015**, *71*, 3.
4. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, A. K.; Puschmann, J. H. *Appl. Crystallogr.* **2009**, *42*, 339.