Supporting Information

for

Monothiatruxene: new versatile core for functional materials

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Experimental Section

General methods.

NMR spectra were recorded on a Bruker (400 and 500 MHz) spectrometers for solutions in $CDCl_3$. Chemical shifts are reported in parts per million. Chemical shifts multiplicities are reported as s: singlet, d: doublet, t: triplet, q: quartet, quint: quintet, and m: multiplet. Mass spectra (EI, ESI) were recorded with Xevo QTOF (Waters) high resolution, accurate mass tandem mass spectrometer equipped with Atmospheric Solids Analysis Probe (ASAP) and Bruker MicroToF 2. Elemental analysis was performed at the Institute of Organic Chemistry PAS with a Vario EL III (Elementar GmbH, Germany) and by Stephen Boyer of the Science Centre, London Metropolitan University using a Carlo Erba CE1108 Elemental Analyser.

Optical characterization

Solution UV-visible absorption spectra were recorded using a Jasco V-670 UV/vis/NIR spectrophotometer controlled with SpectraManager software. Photoluminescence (PL) spectra were recorded with a Fluoromax-3 fluorimeter controlled by the ISAMain software. All samples were measured in a 1 cm cell at room temperature with dichloromethane as solvent. Concentrations used were that to give absorption not higher 0.1 at λ_{max} for fluorescence measurements and 10^{-6} mol/l for measurements of absorption coefficient. A qunine sulfate in 0.1 N H_2SO_4 (Φ= 0.51) was used as a standard for the fluorescence quantum yield determination with solvent refractive index correction.

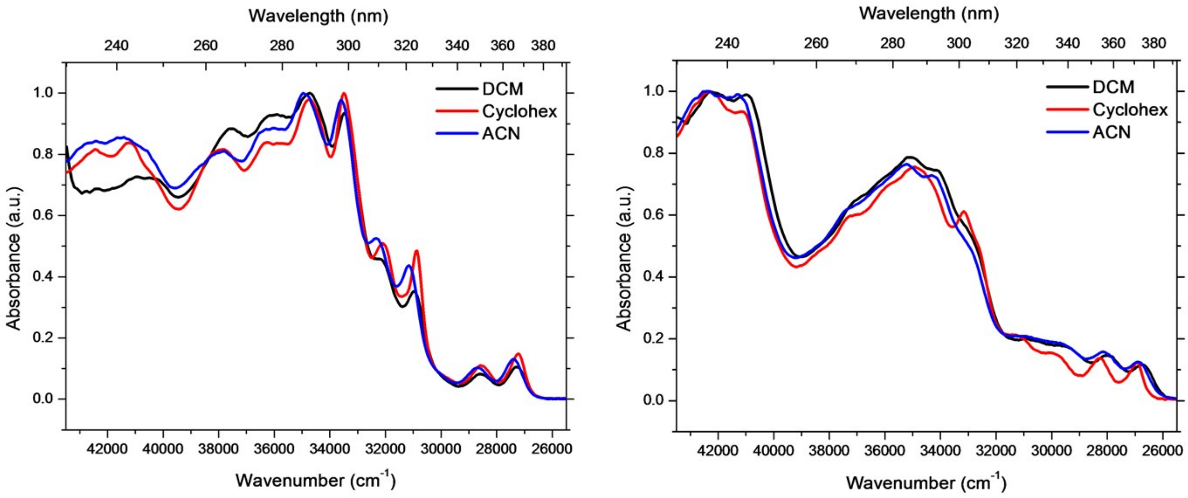


Figure S 1. Normalized absorbance spectra of TrxS (left) and $TrxSO_2$ (right) in dichloromethane, cyclohexane and acetonitrile.

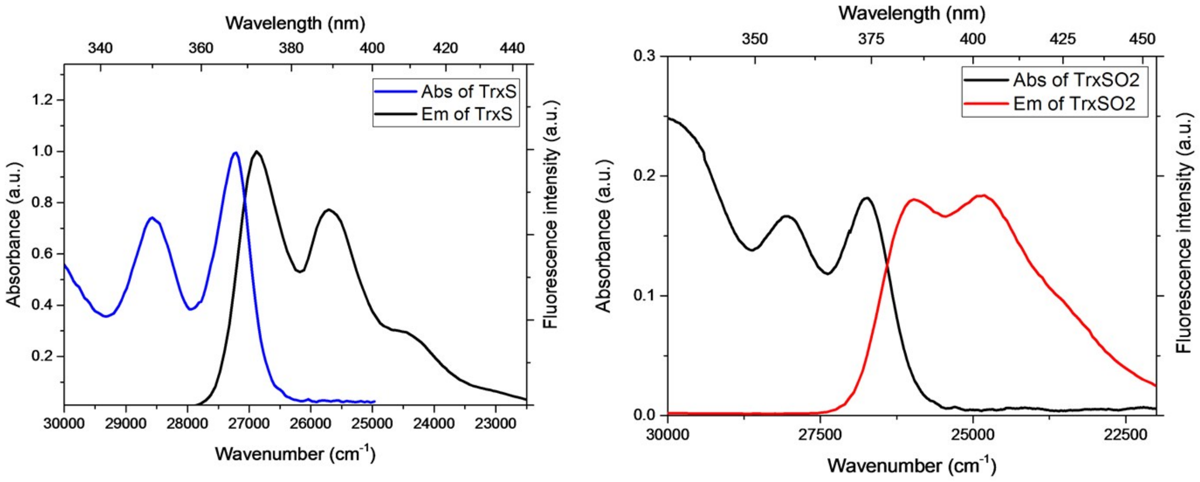


Figure S 2. Normalized absorption and emission spectra in dichloromethane of TrxS (left) and TrxSO$_2$ (right).

Electrochemical characterization

All cyclic voltammetry measurements were carried out in freshly distilled CH_2Cl_2 using 0.3 M [TBA][PF_6] electrolyte in a three- electrode system, with each solution being purged with nitrogen prior to measurement. The working electrode was a Pt disk. The reference electrode was Ag/AgCl and the counter electrode was a Pt rod. All measurements were made at room temperature using a mAUTOLAB Type III potentiostat, driven by the electrochemical software GPES. Cyclic voltammetry (CV) measurements used scan rates of 0.1 V/s to 0.5 V/s; square wave voltammetry (SWV) was carried out at a step potential of 0.004 V, square wave amplitude of 0.025 V, and a square wave frequency of 25 Hz. Ferrocene was used as the internal standard in each measurement.

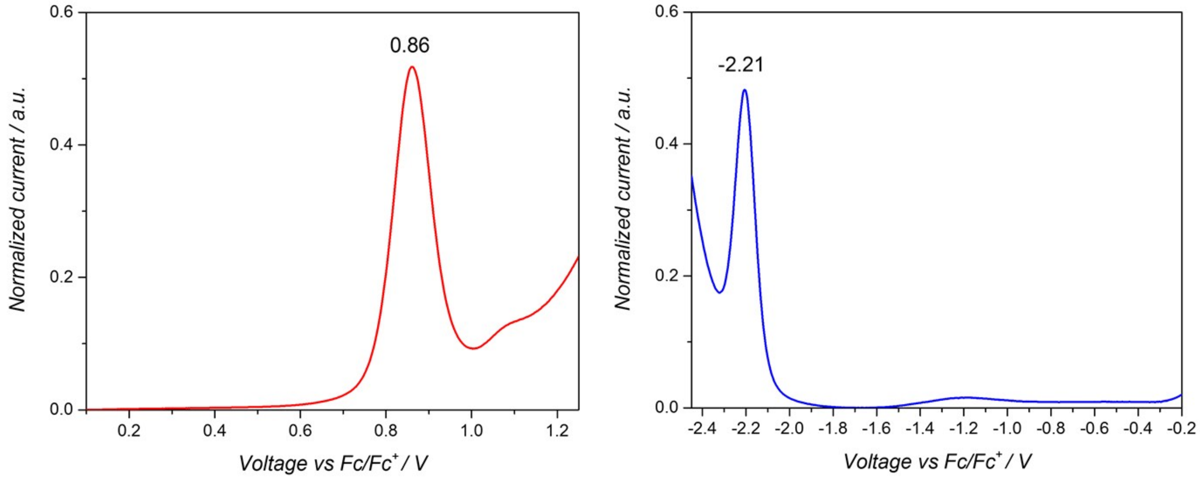


Figure S 3. Square-wave voltammetry (SWV) oxidation of TrxS (left) and reduction of TrxSO$_2$ (right).

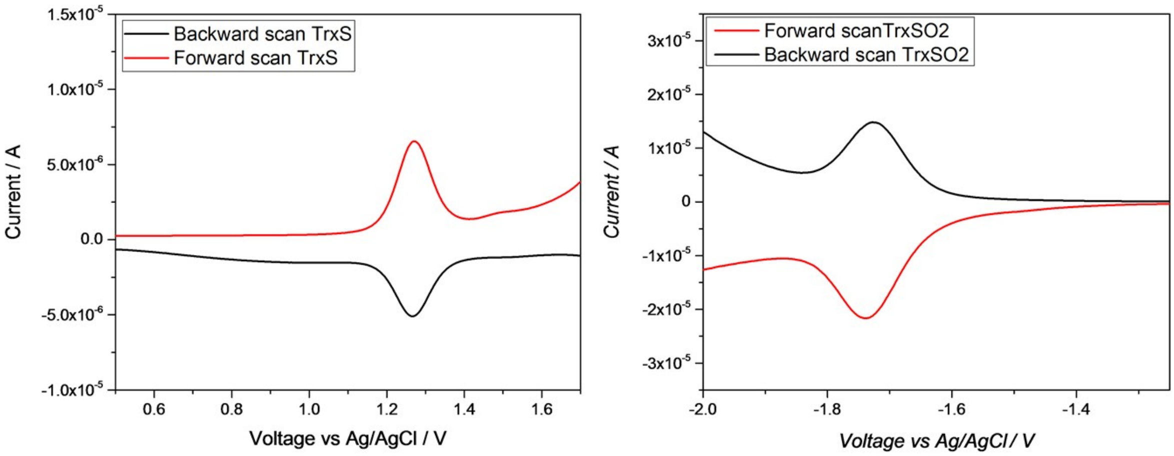


Figure S 4. Square-wave voltammetries of TrxS and TrxSO$_2$.

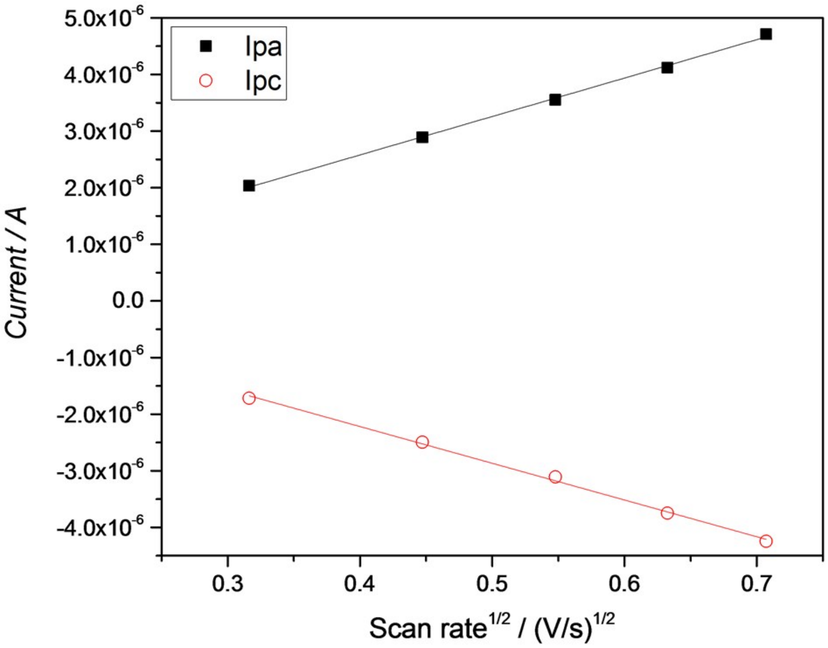


Figure S 5. Plot of ipa (second) and ipc (second) versus (scan rate)$^{-2}$ (from 0.1 V/s to 0.5 V/s) from cyclic voltammograms of TrxS.

Above plot for TrxS illustrates that oxidation peak (R^2= 0.998) and reduction peak (R^2= 0.997) are independent of scan rate. Scan rate plots are consistent with the Randles-Sevcik; the peak current increases with square root of the scan rate.

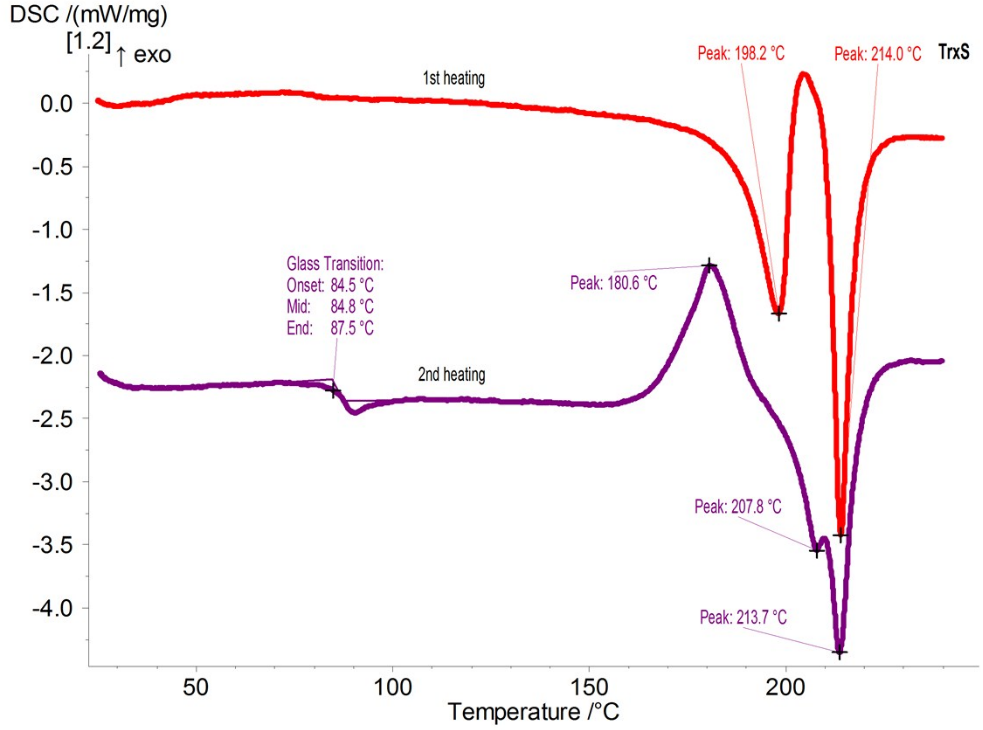
Table S 1. Cyclic voltammetry data for TrxS.

Scan rate V/s	(Scan rate)$^{1/2}$	I_{pa} /A	I_{pc} /A	E_{pa} /V	E_{pc} /V	ΔEp /V	I_{pa}/I_{pc}
0.1	0.32	2.03E-06	-1.72E-06	1.316	1.238	0.078	-1.2
0.2	0.45	2.89E-06	-2.50E-06	1.316	1.238	0.078	-1.2
0.3	0.55	3.55E-06	-3.11E-06	1.311	1.233	0.078	-1.1
0.4	0.63	4.12E-06	-3.75E-06	1.316	1.238	0.078	-1.1
0.5	0.71	4.71E-06	-4.24E-06	1.311	1.233	0.078	-1.1

The peak-to-peak separation (ΔEp) is constant 78 mV value when the scan rate is changed. The value is slightly larger than the Nernstian value of 0.059 mV (at 25 °C) for a one-electron reversible redox couple as expected since value for the ferrocene is 80 mV under these conditions. The current ratios (I_{pa}/I_{pc}) is approximately 1, proving stability of the oxidized species. Therefore it may be concluded that this redox couple is reversible.

Thermal characterization

Differential scanning calorimetry (DSC) and Thermogravimetric analysis (TGA) were performed on NETZSCH STA 449F1 under nitrogen atmosphere in DSC/TG aluminium pan at a scan rate of 20 K min^{-1} and 10 K min^{-1}, respectively.



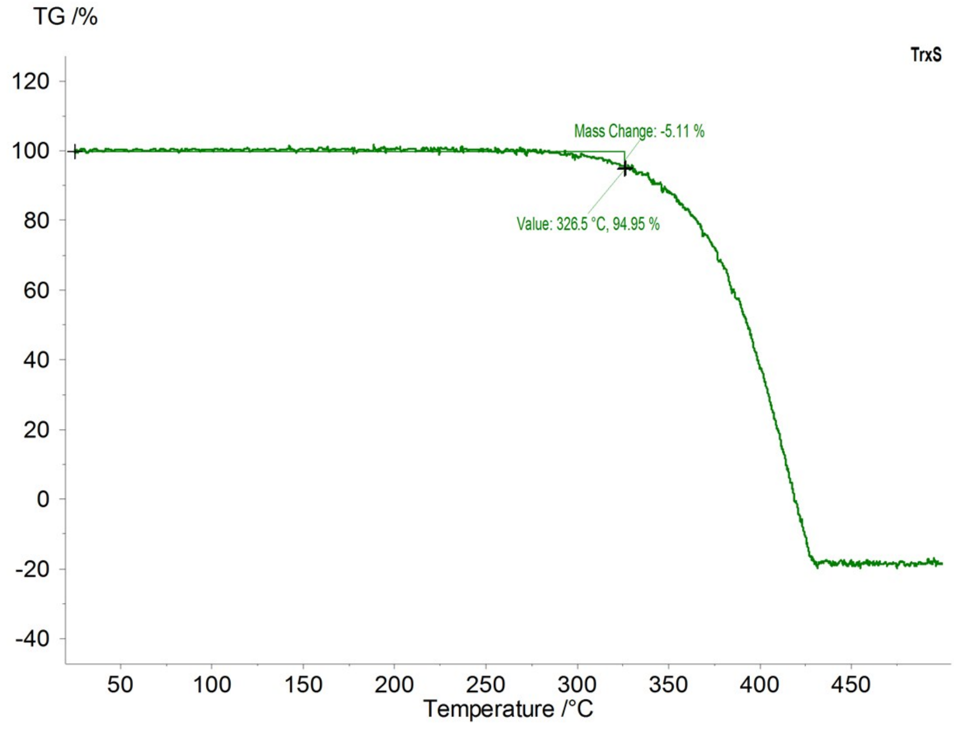
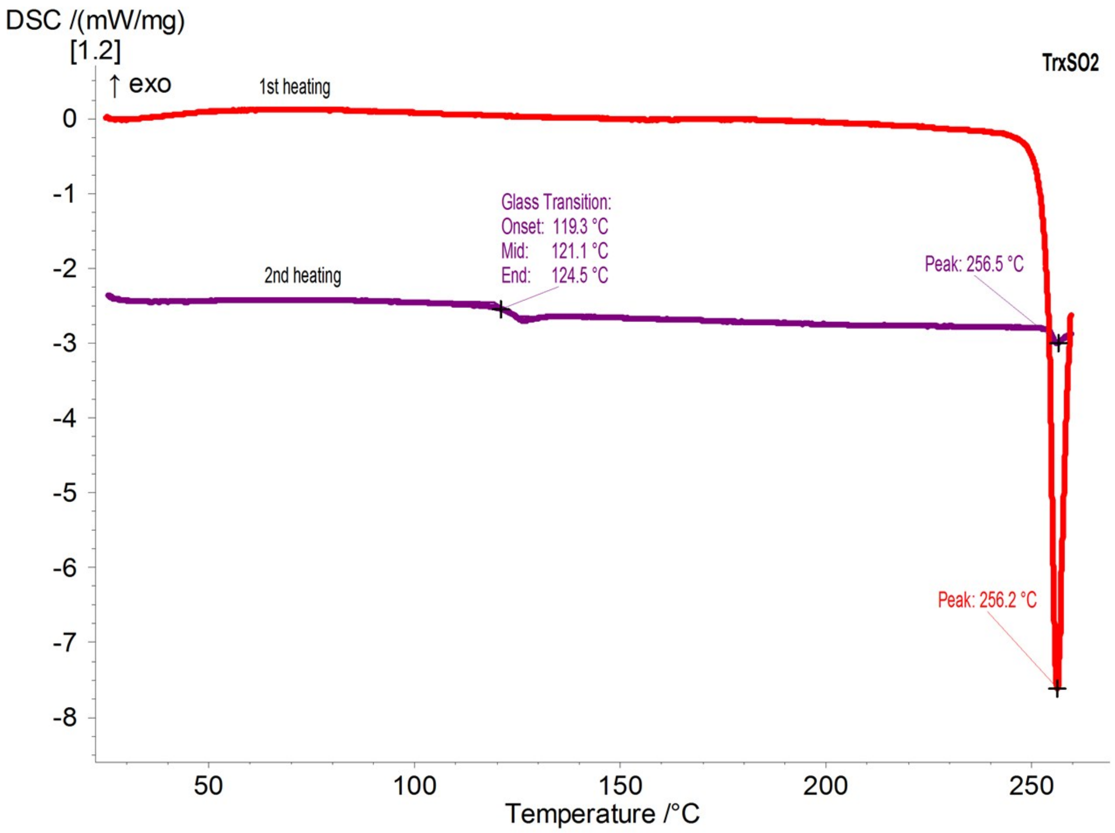


Figure S 6. Differential scanning calorimetry (top) and thermogravimetric analysis (bottom) of TrxS.



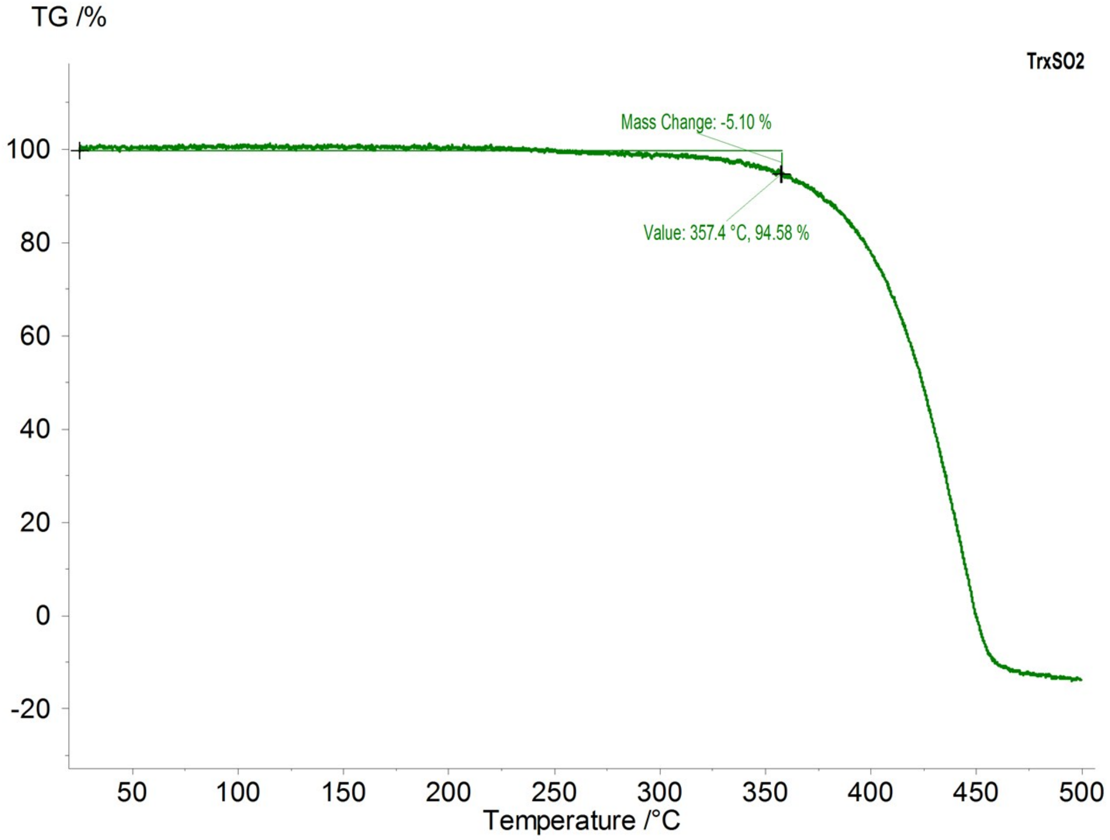


Figure S 7. Differential scanning calorimetry (top) and thermogravimetric analysis (bottom) of $TrxSO_2$.

X-ray Powder Diffraction (XRD)

Powder diffraction was performed on a Bruker Discover D8 with CuKa1/2 source and a scintillation detector with the powder grind after the reaction.

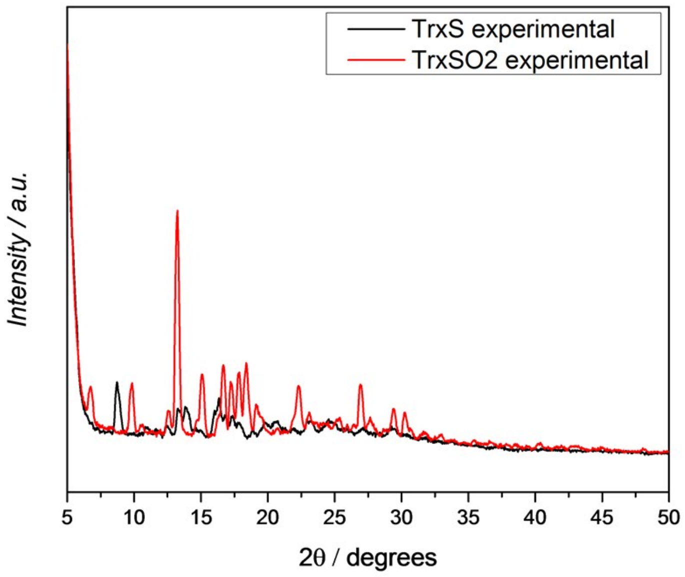


Figure S 8. Experimental XRD powder patterns of TrxS and TrxSO$_2$.

Materials and synthesis.

All preparations were carried out using standard Schlenk line and air-sensitive chemistry techniques under nitrogen atmosphere. Tetrahydrofuran and dichloromethane were dried using a solvent purification system. Column chromatography was carried out using Silica 60A (particle size 35-70 µm, Fisher, UK) as the stationary phase, and TLC was performed on pre-coated silica gel plates (0.25 mm thick, 60 F254, Merck, Germany) and observed under UV light. Boron trifluoride diethyl etherate was distilled from calcium hydride with excess of diethyl ether at reduced pressure. Diethyl ketone was dried over molecular sieves type 3A. 1,3,5-tris-2'-bromophenylbenzene was synthesized according to procedure describe earlier.[1] All other materials were purchased from commercial suppliers and used without further purification.

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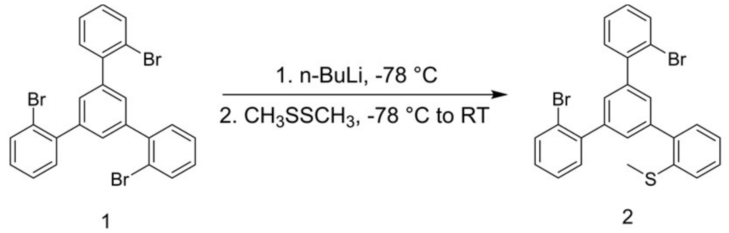
[1] Xinliang Feng, Jishan Wu, Volk Enkelmann, Klaus Müllen, *Org. Lett.*, **2006**, *8* (6),1145.

Synthetic procedure for 1,3-bis(2-bromophenyl)-5-(2-thiomethylphenyl)benzene



1 2

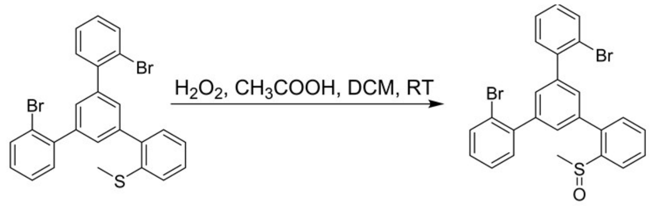
1,3,5-tris(2-bromophenyl)benzene (5 g, 9.208 mmol) was dissolved in THF (30 mL) under nitrogen atmosphere and then n-butyllithium (3.87 mL, 2.5 M, 9.675 mmol) was added dropwise to the solution at -78 °C. After 1 h at this temperature dimethyl disulphide (0.87 mL, 9.668 mmol) was added in 3 mL of THF. The reaction was slowly brought to room temperature and stirred overnight. Then it was quenched with saturated NH_4Cl solution and extracted with ethyl acetate. Organic layer was dried over anhydrous $MgSO_4$, filtered and solvent was removed on rotary evaporator to give yellow oil. Column chromatography of the crude product (SiO_2, eluent: diethyl ether/hexane 1/70) afforded **2** 3.413 g (73%) as colourless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 2.40 (s, 3 H, CH_3), 7.19-7.26 (m, 3 H, Ar-H), 7.31-7.39 (m, 5 H, Ar-H), 7.46-7.53 (m, 5 H, Ar-H), 7.69 (d, *J*=7.8 Hz, 2 H, Ar-H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 16.3 (CH_3), 122.8 (C_{Ar}), 125.0 (C_{Ar}), 125.8 (C_{Ar}), 127.5 (C_{Ar}), 128.21 (C_{Ar}), 128.97 (C_{Ar}), 129.7 (C_{Ar}), 130.5 (C_{Ar}), 131.7 (C_{Ar}), 133.3 (C_{Ar}), 137.3 (C_{Ar}), 140.1 (C_{Ar}), 140.5 (C_{Ar}), 140.6 (C_{Ar}), 142.2 (C_{Ar}).

MS (ESI): m/z (%)= 510.62 [M^+, 24]; 532.66 [$(M+Na)^+$, 92].

Synthetic procedure for 1,3-bis(2-bromophenyl)-5-(2-oxythiomethylphenyl)benzene



2 3

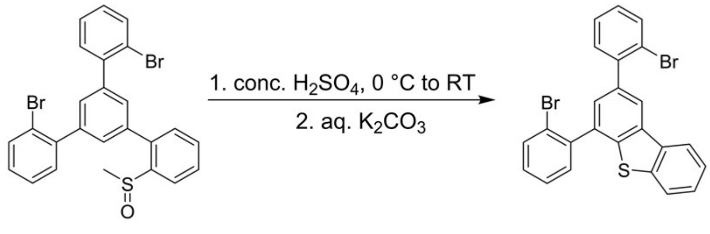
A flask was charged with **1** (3.413 g, 6.688 mmol), dichloromethane (2 mL), glacial acetic acid (30 mL) and hydrogen peroxide solution (1 mL, 30%). The reaction mixture was stirred at r.t. for two days. Then water was added and it was extracted with dichloromethane. Organic layer was washed with saturated sodium carbonate, dried over anhydrous $MgSO_4$, filtered and solvent was removed on rotary evaporator to give white foamy solid. Column chromatography of the crude product (SiO_2, eluent: acetone/hexane 1/5) afforded 2.830 g (80%) of **2** as a white solid.

^1H NMR (400 MHz, $CDCl_3$, δ ppm): 2.49 (s, 3 H, CH_3), 7.20-7.26 (m, 2 H, Ar-H), 7.36-7.49 (m, 8 H, Ar-H), 7.55 (t, *J*=7.3 Hz, 1 H, Ar-H), 7.63 (t, *J*=7.5 Hz, 1 H, Ar-H), 7.68 (d, *J*=7.9 Hz , 2 H, Ar-H), 8.15 (d, *J*=7.8 Hz , 1 H, Ar-H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 41.7 (CH_3), 122.6 (C_{Ar}), 123.5 (C_{Ar}), 127.6 (C_{Ar}), 129.0 (C_{Ar}), 129.3 (C_{Ar}), 130.3 (C_{Ar}), 130.5 (C_{Ar}), 130.8 (C_{Ar}), 131.3 (C_{Ar}), 133.3 (C_{Ar}), 137.4 (C_{Ar}), 138.8 (C_{Ar}), 141.49 (C_{Ar}), 141.54 (C_{Ar}), 144.2 (C_{Ar}).

MS (ESI): m/z (%)= 526.67 [M^+, 98]; 548.79 [$(M+Na)^+$, 80].

Synthetic procedure for 1,3-bis(2-bromophenyl)-dibenzothiophene[2]



3

4

Compound **3** (3.241 g, 6.158 mmol) was added in portions to stirred concentrated H_2SO_4 (15mL) in a one-necked, round-bottomed flask containing a guard tube at 0-5 °C. The reaction mixture was stirred at room temperature for 1 h. The reaction mixture was poured on ice-cold water and then made basic with aqueous potassium carbonate solution (pH 8). The aqueous layer was extracted with ethyl acetate (2 x 15mL). The organic layer was dried over anhydrous sodium sulfate, filtered and evaporated to give crude product as colourless oil. Column chromatography of the crude product (SiO_2, eluent: ethyl acetate/hexane 1/30) afforded 2.671 g (88%) of a **4** as a white solid.

^1H NMR (400 MHz, $CDCl_3$, δ ppm): 7.23 (t, *J*=7.19 Hz, 1 H, Ar-H), 7.31 (t, *J*=7.7 Hz, 1 H, Ar-H), 7.38-7.56 (m, 7H, Ar-H), 7.71-7.81 (m, 3 H, Ar-H), 8.18-8.19 (m , 1 H, Ar-H), 8.23 (s, 1 H, Ar-H).

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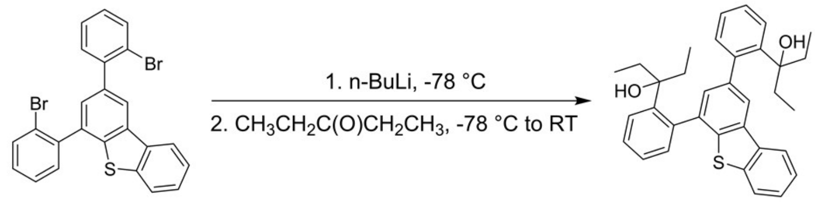
[2] Vrajesh B. Pandya et al, *Synth. Commun*. 42, 497, **2012**

^{13}C NMR (100 MHz, CDCl$_3$, δ ppm): 121.8 (C$_{Ar}$), 122.0 (C$_{Ar}$), 122.9 (C$_{Ar}$), 123.1 (C$_{Ar}$), 123.4 (C$_{Ar}$), 124.6 (C$_{Ar}$), 127.1 (C$_{Ar}$), 127.6 (C$_{Ar}$), 127.7 (C$_{Ar}$) 129.0 (C$_{Ar}$), 129.1 (C$_{Ar}$), 129.3 (C$_{Ar}$), 130.0 (C$_{Ar}$), 131.1 (C$_{Ar}$), 131.8 (C$_{Ar}$), 133.3 (C$_{Ar}$), 133.4 (C$_{Ar}$), 135.6 (C$_{Ar}$), 135.7 (C$_{Ar}$), 135.9 (C$_{Ar}$), 137.8 (C$_{Ar}$), 139.2 (C$_{Ar}$), 140.1 (C$_{Ar}$), 140.8 (C$_{Ar}$), 142.3 (C$_{Ar}$).

MS (ESI): m/z (%)= 414.87 [(M-Br)$^+$, 100]; 412.90 [(M-Br)$^+$, 87].

Synthetic procedure for 1,3-bis(2-diethylhydroxymethylphenyl)-dibenzothiophene



4 5

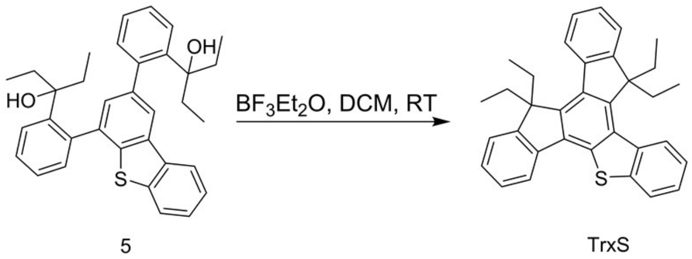
The substrate (**4**) (1.744 g, 3.529 mmol) was dissolved in THF (10 mL) under nitrogen atmosphere and then n-butyllithium (4.6 mL, 1.6 M, 7.411 mmol) was added dropwise to the solution at -78 °C. After 20 min. at this temperature diethyl ketone (0.75 mL, 7.057 mmol) was added in 2 mL of THF. The reaction was slowly brought to room temperature and stirred for 72 hours. Then it was quenched with saturated NH$_4$Cl solution and extracted with ethyl acetate. Organic layer was dried over anhydrous MgSO$_4$, filtered and solvent was removed on rotary evaporator to give yellowish oil. Column chromatography of the crude product (SiO$_2$, eluent: ethyl acetate/petroleum ether 1/30) afforded 1.102 g (61%) of **5** as a foamy white solid.

^{1}H NMR (400 MHz, CDCl$_3$, δ ppm): 0.77-0.87 (m, 12 H, CH$_3$), 1.70-2.00 (m, 10 H, CH$_2$, OH), 7.19-7.45 (m, 11 H, Ar-H), 7.78 - 7.80 (m, 1 H, Ar-H), 7.97-8.01 (m, 1 H, Ar-H), 8.11-8.13 (m, 1 H, Ar-H).

^{13}C NMR (100 MHz, CDCl$_3$, δ ppm): 8.3 (CH$_3$), 8.4 (CH$_3$), 8. 6 (CH$_3$), 35.1 (CH$_2$), 35.5 (CH$_2$), 36.1 (CH$_2$), 36.5 (CH$_2$), 79.9 ($\underline{C}$-(CH$_2$)$_2$), 80.2 ($\underline{C}$-(CH$_2$)$_2$), 119.6 (C$_{Ar}$), 120.4 (C$_{Ar}$), 121.9 (C$_{Ar}$), 122.9 (C$_{Ar}$), 124.5 (C$_{Ar}$), 125.6 (C$_{Ar}$), 126.0 (C$_{Ar}$), 126.2 (C$_{Ar}$), 126.8 (C$_{Ar}$), 127.4 (C$_{Ar}$), 128.1 (C$_{Ar}$), 132.2 (C$_{Ar}$), 132.6 (C$_{Ar}$), 134.7 (C$_{Ar}$), 135.9 (C$_{Ar}$), 138.4 (C$_{Ar}$), 138.7 (C$_{Ar}$), 140.6 (s, 1 C, C$_{Ar}$), 140.8 (C$_{Ar}$), 143.2 (C$_{Ar}$), 144.4 (C$_{Ar}$).

MS (ESI): m/z (%)= 530.96 [(M+Na)$^+$, 100], 472.97 [((M-2Et)+Na)$^+$, 100].

Synthetic procedure for monothiatruxene[3]



Diol (5) (1.102 g, 2.166 mmol) was dissolved in CH_2Cl_2 (40 mL), and then BF_3Et_2O (0.6 mL) was injected at room temperature. The reaction mixture was kept for about 10 min and then quenched with methanol. After concentration under vacuum the product TrxS (0.870 g, 85%) was obtained as a white solid.

Elemental analysis (%). Calculated for $C_{34}H_{32}S$: C 86.39, H 6.82. Result: C 86.42, H 6.58.

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 0.25 (m, 12 H, CH_3), 2.24-2.33 (m, 4 H, CH_2), 2.95-3.04 (m, 2 H, CH_2), 3.08-3.17 (m, 2 H, CH_2), 7.38-7.57 (m, 8 H, Ar-H), 8.03 (d, *J*=7.6 Hz, 1 H, Ar-H), 8.18 (d, *J*=7.4 Hz, 1 H, Ar-H), 8.36 (d, *J*=7.4 Hz, 1 H, Ar-H), 8.93 (d, *J*=7.8 Hz, 1 H, Ar-H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 8.89 (CH_3), 8.94 (CH_3), 29.6 (CH_2), 29.9 (CH_2), 57.9 ($\underline{C}$-$(CH_2)_2$), 58.9 ($\underline{C}$-$(CH_2)_2$), 121.8 (C_{Ar}), 122.2 (C_{Ar}), 122.5 (C_{Ar}), 123.3 (C_{Ar}), 124.0 (C_{Ar}), 124.2 (C_{Ar}), 125.9 (C_{Ar}), 126.5 (C_{Ar}), 126.7 (C_{Ar}), 127.11 (C_{Ar}), 127.15 (C_{Ar}), 127.2 (C_{Ar}), 132.8 (C_{Ar}), 133.0 (C_{Ar}), 134.6 (C_{Ar}), 135.9 (C_{Ar}), 137.7 (C_{Ar}), 140.5 (C_{Ar}), 140.7 (C_{Ar}), 140.9 (C_{Ar}), 142.6 (C_{Ar}), 146.0 (C_{Ar}), 152.1 (C_{Ar}), 152.2 (C_{Ar}).

MS (TOF MS AP+):m/z (%)= 473.2 [$(M+H)^+$, 100], 489.2 [$(M+O)^+$, 60)], 445.1 [$(M-Et)^+$, 50)].

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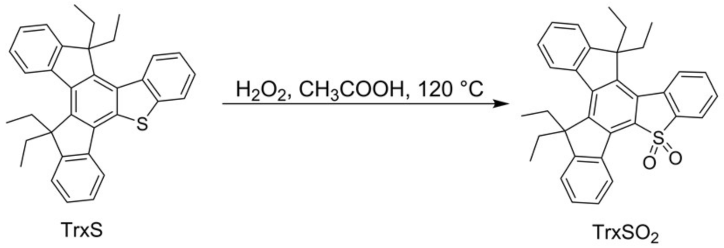
[3] Jian Pei et al. ; *J. Am. Chem. Soc.*, **2007**, 129 (37), 11314; Cho, Sung Yong et al., *J. Am. Chem. Soc.*, 129(39), 11910; **2007**

Synthetic procedure for monosulfonyltruxene.



Monothiatruxene (0.044 g, 0.093 mmol) has been suspended in acetic acid (5 mL) and hydrogen peroxide (0.045 mL) was added dropwise. This mixture was then refluxed for 4.5 hours and poured on water, extracted with DCM, dried over magnesium sulfate, filtrated and concentrated on rotavap. Purified by column chromatography (SiO_2, eluent: acetone/hexane 20/1) gave pure product **TrxSO2** 0.040 g (85 %) as a slightly yellow solid.

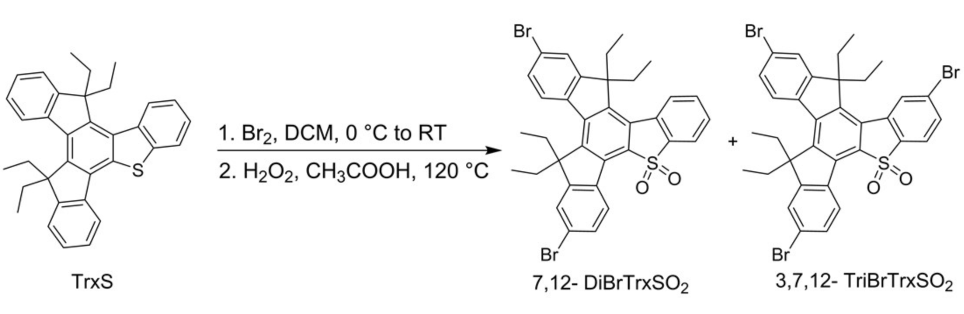
Elemental analysis (%). Calculated for $C_{34}H_{32}O_2S$: C 80.92, H 6.39. Result: C 81.1, H 6.5.

^{1}H NMR (601 MHz, Chloroform-*d*) δ 0.27 (td, *J* = 7.3, 1.4 Hz, 12H), 2.21 (m, *J* = 14.1, 10.4, 7.4 Hz, 4H), 2.89 (m, *J* = 14.0, 7.2, 4.6 Hz, 4H), 7.43 – 7.52 (m, 6H), 7.59 (td, *J* = 7.5, 0.7 Hz, 1H), 7.72 (ddd, *J* = 8.3, 7.4, 1.4 Hz, 1H), 8.00 (dd, *J* = 7.6, 1.3 Hz, 1H), 8.37 (d, *J* = 7.3 Hz, 1H), 8.45 (d, *J* = 8.1 Hz, 1H), 8.68 – 8.77 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 8.7 (CH_3), 8.8 (CH_3), 29.6 (CH_2), 29.7 (CH_2), 57.3 ($\underline{C}$-$(CH_2)_2$), 58.3 ($\underline{C}$-$(CH_2)_2$), 122.0 (C_{Ar}-H), 122.5 (C_{Ar}-H), 122.6 (C_{Ar}-H), 125.0 (C_{Ar}-H), 125.3 (C_{Ar}-H), 126.9 (C_{Ar}-H), 127.4 (C_{Ar}-H), 127.6 (C_{Ar}-H), 127.8 (C_{Ar}), 128.6 (C_{Ar}-H), 129.3 (C_{Ar}-H), 129.4 (C_{Ar}-H), 129.6 (C_{Ar}), 131.9 (C_{Ar}), 133.2 (C_{Ar}-H), 136.6 (C_{Ar}), 138.1 (C_{Ar}), 138.4 (C_{Ar}), 139.9 (C_{Ar}), 145.2 (C_{Ar}), 145.5 (C_{Ar}), 146.3 (C_{Ar}), 152.3 (C_{Ar}), 153.2 (C_{Ar}).

MS (EI): m/z (%)= 504.2 [$(M)^+$, 45]; 475.1 [$(M\text{-}Et)^+$, 100].

Synthetic procedure for brominated monosulfonyltruxenes.



A solution of monothiatruxene (0.660 g, 1.396 mmol) in DCM (30 mL) was cooled to 0°C. Bromine (0.15 mL, 2.862 mmol) was added to the reaction and then it was slowly brought to room temperature. After overnight stirring the reaction was stopped by the addition of sodium thiosulfate solution. It was extracted with DCM, organic layer was dried over anhydrous sodium sulfate, filtered and concentrated on rotary evaporator to give yellow oil. Then glacial acetic acid (45 mL) and hydrogen peroxide solution (0.65 mL, 30%) were added to the flask.

After 2.5 h of reflux the reaction was poured onto ice/water and extracted with DCM. Organic layer was dried over magnesium sulfate, filtered and solvent was removed on rotary evaporator to give a yellow solid. Column chromatography of the crude products (SiO_2, eluent: petroleum ether/chloroform 70/30) gave pure tribrominated truxene 0.103 g (10%) as a slightly yellow solid and dibrominated truxene was further purified by crystallization from acetone to give 0.600 g (65%) of a yellowish solid.

Dibrominated truxene

^{1}H NMR (500 MHz, $CDCl_3$, δ ppm): 0.28 (t, *J*=7.3 Hz, 6H), 0.284 (t, *J*=7.2 Hz, 6H) 2.11–2.23 (m, 4H), 2.76–2.92 (m, 4H), 7.56–7.64 (m, 5H), 7.73 (t, *J*=7.3 Hz, 1H), 7.99 (d, *J*=7.0 Hz, 1H), 8.18 (d, *J*=8.4 Hz, 1H), 8.41 (d, *J*=8.1 Hz, 1H), 8.60 (d, *J*=8.4 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$, δ ppm): 8.87 (CH_3), 8.93 (CH_3), 29.6 (CH_2), 29.8 (CH_2), 57.8 ($\underline{C}$-$(CH_2)_2$), 58.8 ($\underline{C}$-$(CH_2)_2$), 122.8 (C_{Ar}-H), 123.6 (C_{Ar}-Br), 124.1 (C_{Ar}-Br), 125.6 (C_{Ar}-H), 126.1 (C_{Ar}-H), 126.6 (C_{Ar}-H), 126.7 (C_{Ar}-H), 127.5 (C_{Ar}-H), 128.3 (C_{Ar}), 129.9 (C_{Ar}-H), 130.2 (C_{Ar}), 130.5 (C_{Ar}-H), 131.1 (C_{Ar}-H), 131.6 (C_{Ar}), 133.6 (C_{Ar}-H), 135.6 (C_{Ar}), 137.8 (C_{Ar}), 138.0 (C_{Ar}), 139.1 (C_{Ar}), 144.6 (C_{Ar}), 145.3 (C_{Ar}), 145.9 (C_{Ar}), 154.5 (C_{Ar}), 155.6 (C_{Ar}).

MS (ESI): m/z (%)= 685.02 [$(M+Na)^+$, 100]; 663.04 [$(M+H)^+$, 20].

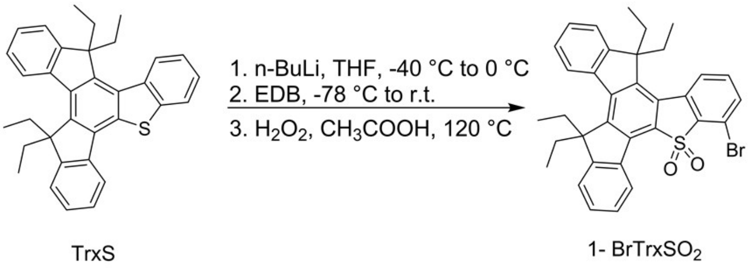
Tribrominated truxene

^{1}H NMR (500 MHz, $CDCl_3$, δ ppm): 0.27 (t, *J*=7.2 Hz, 6H), 0.31 (t, *J*=7.2 Hz, 6H), 2.12–2.25 (m, 4H), 2.70–2.86 (m, 4H), 7.58–7.64 (m, 4H), 7.75 (dd, *J*=8.1 Hz, 1.3 Hz, 1H), 7.85 (d, *J*=8.1 Hz, 1H), 8.18 (d, *J*=8.3 Hz, 1H), 8.55 (d, *J*=8.5 Hz,1H), 8.56 (d, *J*=1.1 Hz 1H).

^{13}C NMR (126 MHz, $CDCl_3$, δ ppm): 8.8 (CH_3), 8.9 (CH_3), 29.7 (CH_2), 29.8 (CH_2), 57.8 ($\underline{C}$-$(CH_2)_2$), 58.9 ($\underline{C}$-$(CH_2)_2$), 123.8 (C_{Ar}-Br), 123.9 (C_{Ar}-H), 124.3 (C_{Ar}-Br), 125.6 (C_{Ar}-H), 126.1 (C_{Ar}-H), 126.6 (C_{Ar}-H), 126.7 (C_{Ar}-H), 127.0 (C_{Ar}), 128.3 (C_{Ar}), 130.3 (C_{Ar}), 130.6 (C_{Ar}), 130.7 (C_{Ar}-H), 131.2 (C_{Ar}-H), 132.7 (C_{Ar}-H), 133.6 (C_{Ar}), 135.3 (C_{Ar}), 136.8 (C_{Ar}), 137.0 (C_{Ar}), 139.3 (C_{Ar}), 144.7 (C_{Ar}), 145.6 (C_{Ar}), 146.7 (C_{Ar}), 154.5 (C_{Ar}), 155.5 (C_{Ar}).

MS (EI): m/z (%)= 712.8 [$(M-Et)^+$, 100]; 741.9 [$(M)^+$, 50].

Synthetic procedure for monobrominated monosulfonyltruxenes



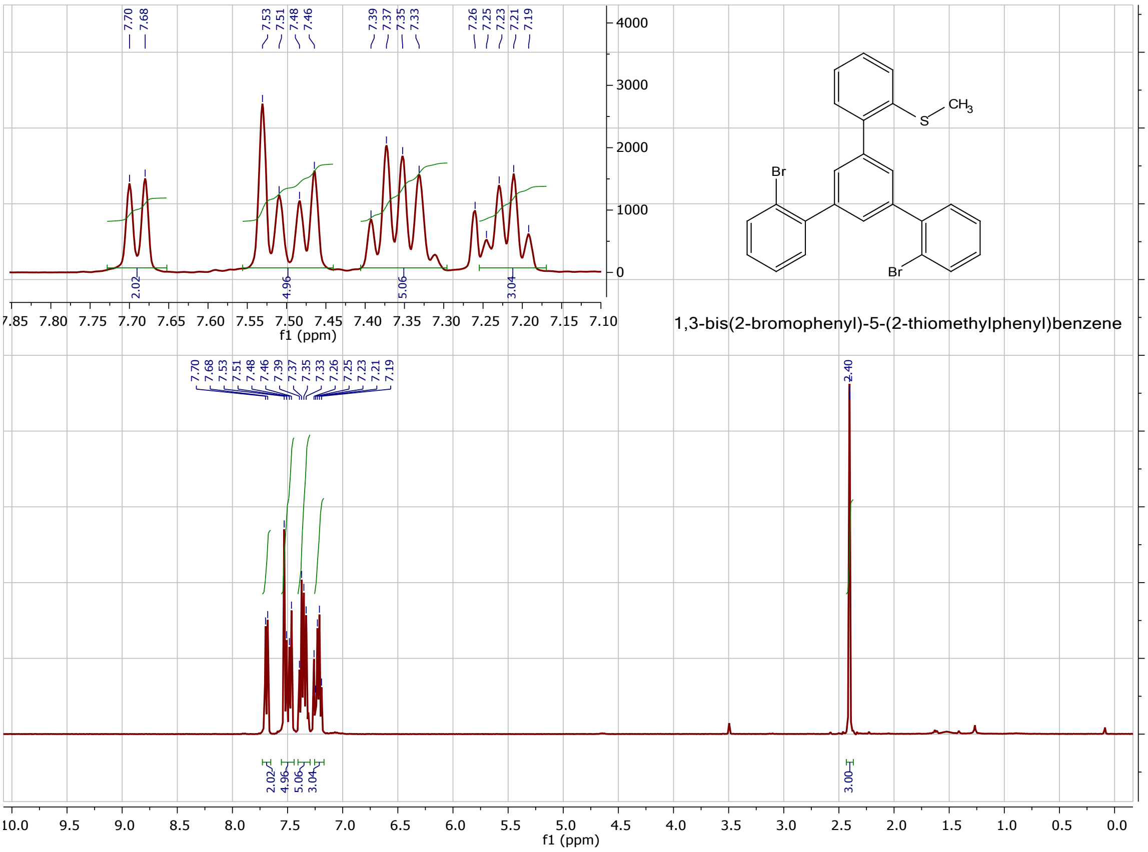
A solution of monothiatruxene (0.418 g, 0.884 mmol) in THF (5 mL) was cooled to -40°C. n-BuLi (0.61 mL, 0.972 mmol, 1.1 eq, 1.6 M) has been added dropwise and it was kept at this temperature for 0.5 h and then it was slowly brought 0°C to be stirred for additional 6 hours. After that the dark mixture was cooled to -78°C and ethylene dibromide (0.15 mL, 1.768 mmol, 2 eq) was added in 0.5 mL of THF. After 30 min. at this temperature the dark colour disappeared and it was stirred overnight at r.t. Then it was quenched with brine and extracted with ethyl acetate, dried over magnesium sulfate, filtrated and concentrated on rotavap to give 0.453 g of crude mixture with 60% of the expected product (according to NMR analysis). This mixture was taken to the next reaction by adding acetic acid (40 mL) and hydrogen peroxide (0.5 mL) dropwise. This cloudy mixture was then refluxed to yield clear yellow solution after 4 hours. It was brought to r.t. and poured on water, extracted with DCM, dried over magnesium sulfate, filtrated and concentrated on rotavap. Purified by column chromatography (SiO_2, eluent: petroleum ether/DCM 10/1) gave pure product 0.260 g (50 %) as a slightly yellow solid.

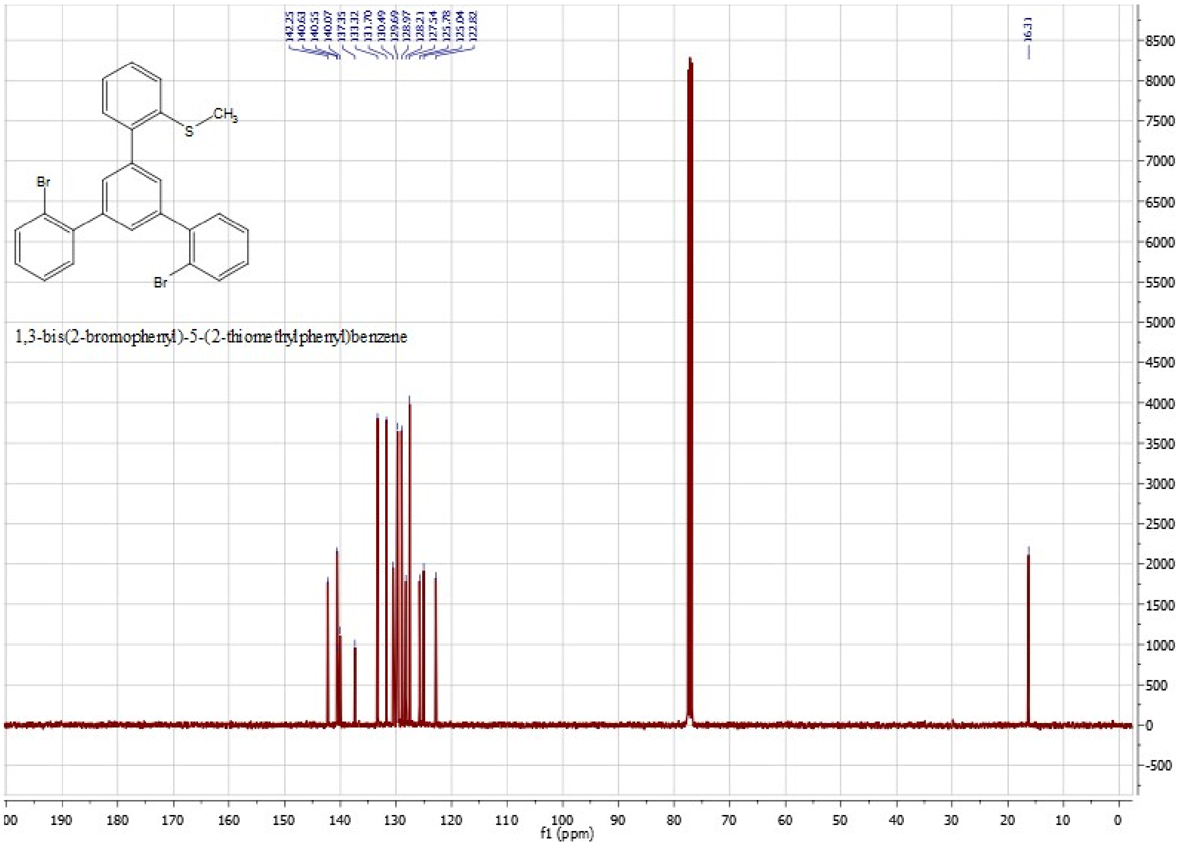
^{1}H NMR (500 MHz, Chloroform-*d*) δ 0.26 (q, *J* = 7.4 Hz, 12 H), 2.10 – 2.35 (m, 4H), 2.77 – 2.94 (m, 4H), 7.43 – 7.52 (m, 6H), 7.54 (t, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 7.9 Hz, 1H), 8.34 – 8.40 (m, 1H), 8.42 (d, *J* = 8.1 Hz, 1H), 8.77 (dd, *J* = 6.5, 1.4 Hz, 1H).

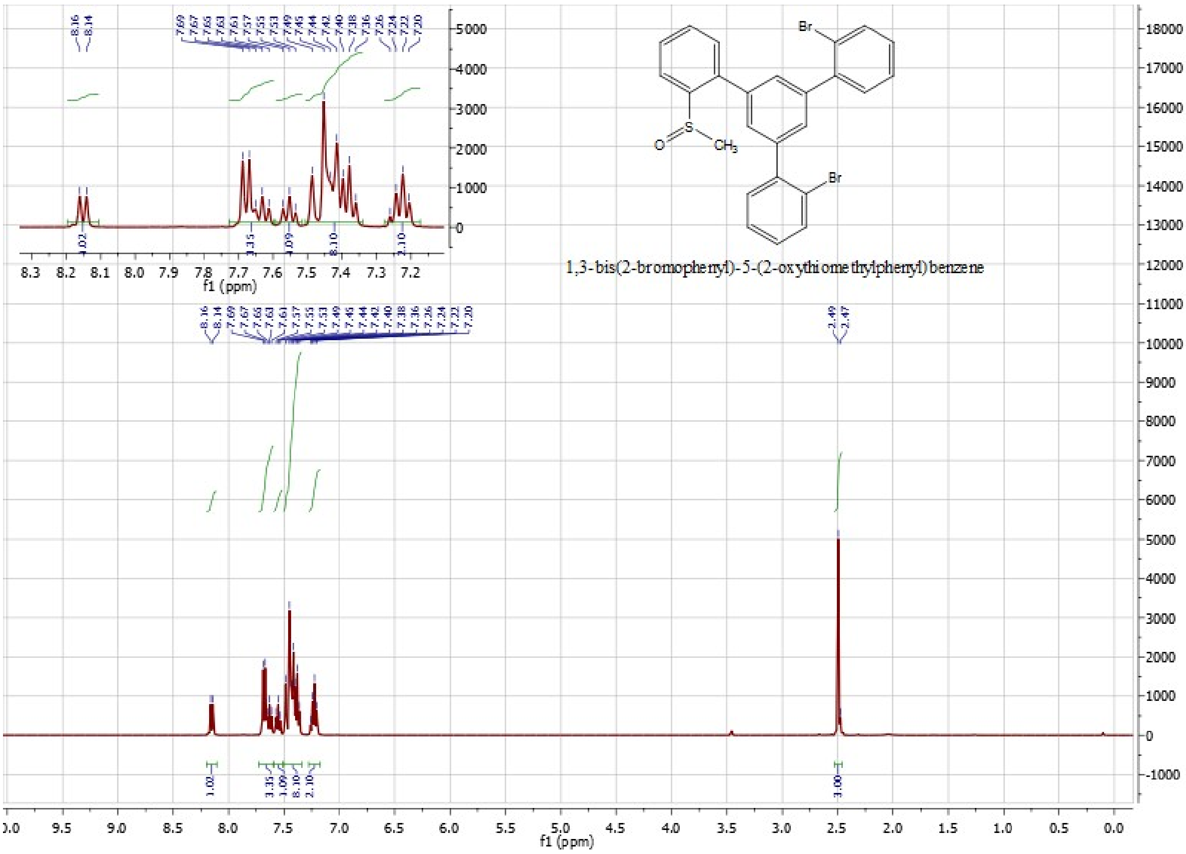
^{13}C NMR (126 MHz, Chloroform-*d*) δ 8.7 (CH_3), 8.8 (CH_3), 29.6 (CH_2), 29.7 (CH_2), 57.3 ($\underline{C}$-$(CH_2)_2$), 58.3 ($\underline{C}$-$(CH_2)_2$), 117.9 (C_{Ar}-Br), 122.0 (C_{Ar}-H), 122.5 (C_{Ar}-H), 125.2 (C_{Ar}-H), 125.3 (C_{Ar}-H), 126.0 (C_{Ar}-H), 126.2 (C_{Ar}), 127.0 (C_{Ar}-H), 127.6 (C_{Ar}-H), 128.8 (C_{Ar}-H), 129.4 (C_{Ar}-H), 129.7 (C_{Ar}), 133.6 (C_{Ar}-H), 133.8 (C_{Ar}-H), 135.2 (C_{Ar}), 136.3 (C_{Ar}), 136.4 (C_{Ar}), 138.2 (C_{Ar}), 139.9 (C_{Ar}), 145.1 (C_{Ar}), 145.7 (C_{Ar}), 146.9 (C_{Ar}), 152.4 (C_{Ar}), 153.2 (C_{Ar}).

MS (ESI): m/z (%)= 607.11 [$(M+Na)^+$, 100]; 605.11 [$(M+Na)^+$, 90]; 585.13 [$(M+H)^+$, 50]; 583.11 [$(M+H)^+$, 40].

Copies of NMR and MS spectra



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7.65
7.63
7.61
7.57
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7.53
7.49
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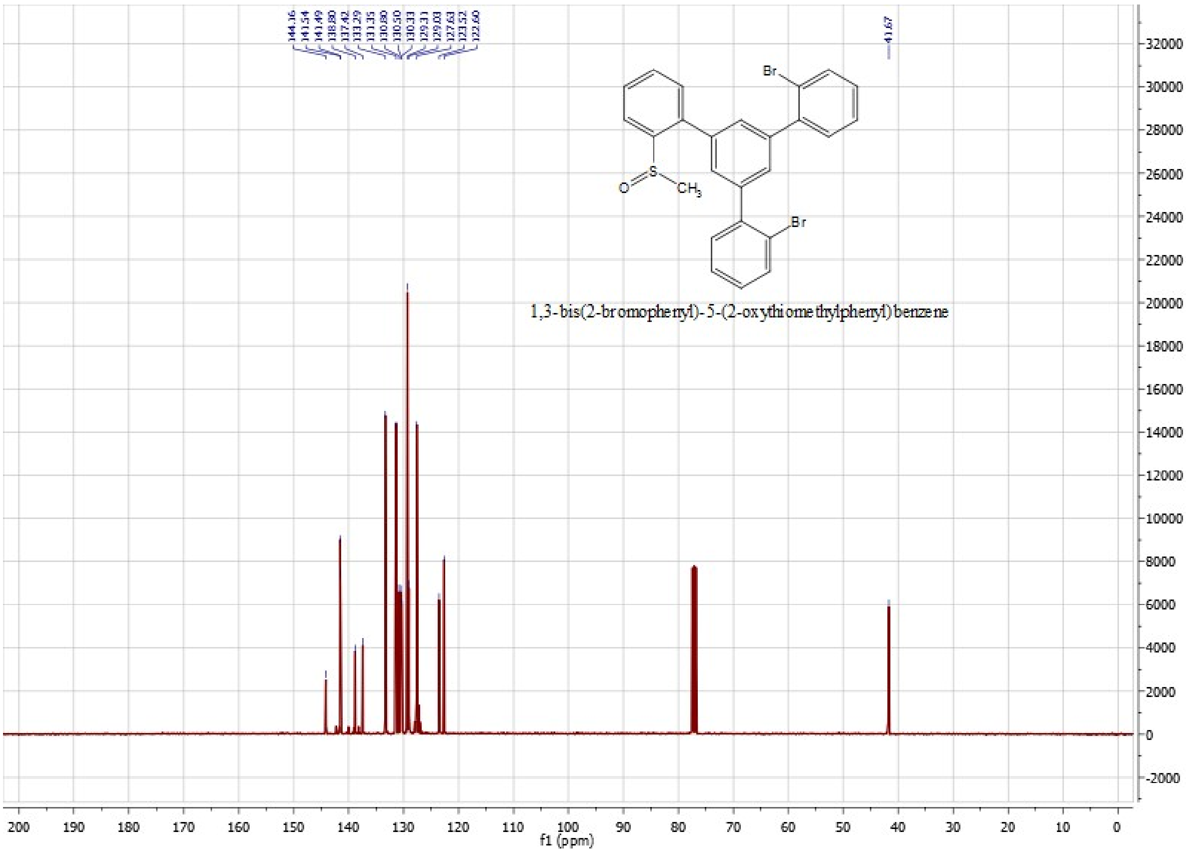
7.44
7.42
7.40
7.38
7.36
7.26
7.24
7.22
7.20
1.02
3.35
1.09
8.10
2.10
2.49
2.47
3.00
Br
Br
O
S
CH3
1,3-bis(2-bromophenyl)-5-(2-oxythiomethylphenyl)benzene
f1 (ppm)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130.80
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130.33
129.31
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127.63
123.52
122.60
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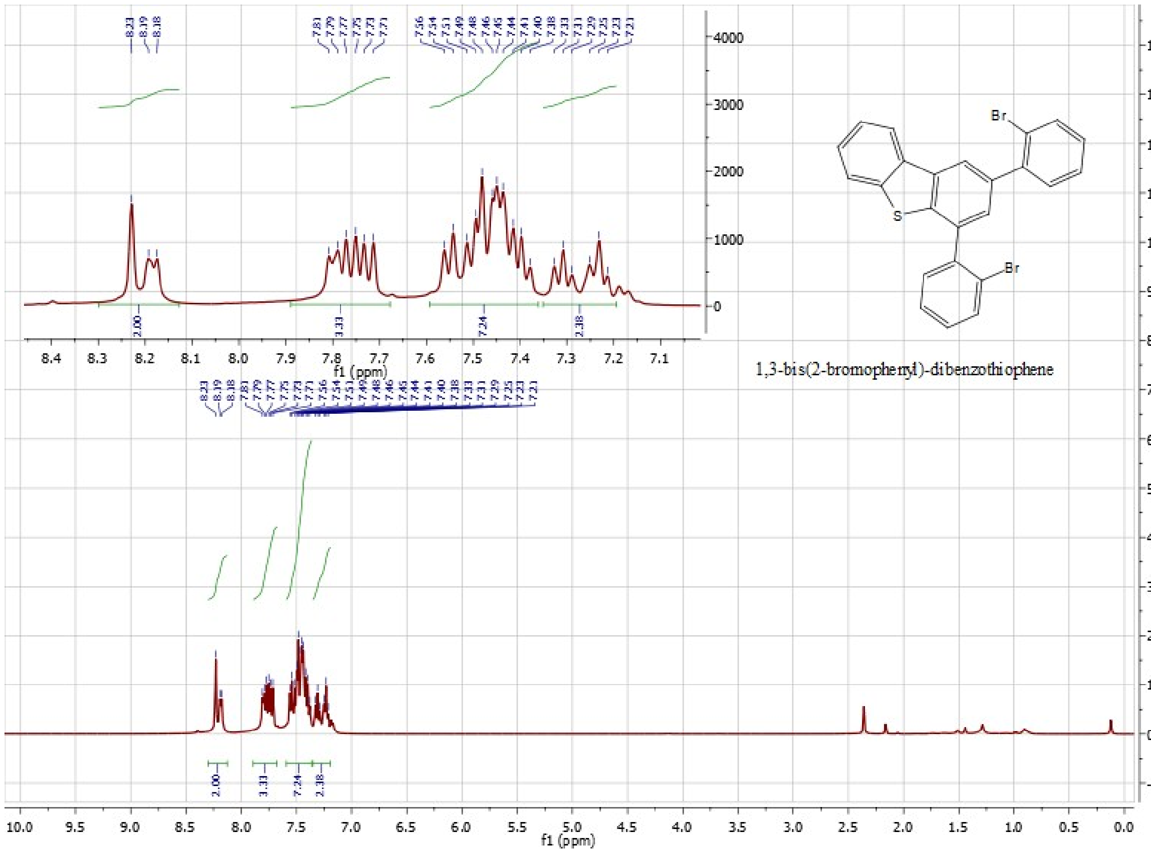
Br
Br
O
S
CH3
1,3-bis(2-bromophenyl)-5-(2-oxythiomethylphenyl)benzene
f1 (ppm)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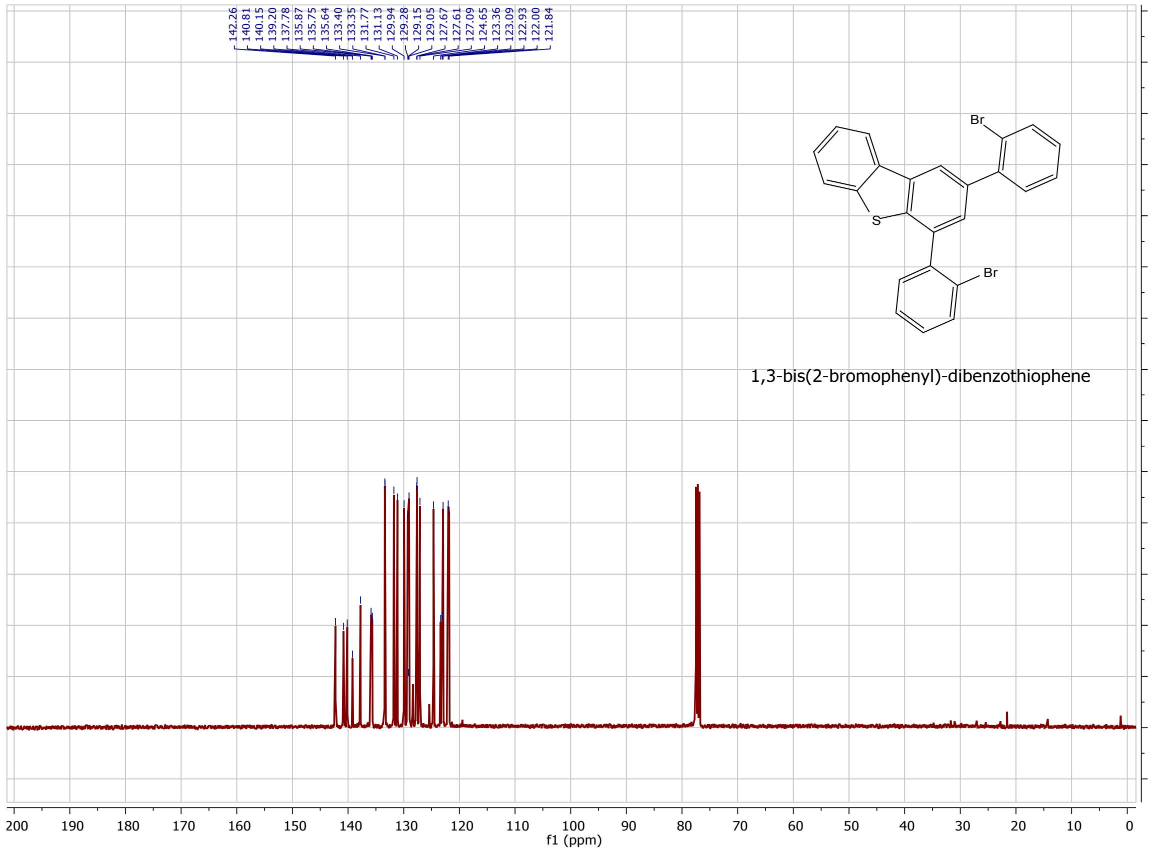
7.48
7.46
7.45
7.44
7.41
7.40
7.38
7.33
7.31
7.29
7.25
7.23
7.21
2.00
3.33
7.24
2.38
f1 (ppm)
Br
S
Br
1,3-bis(2-bromophenyl)-dibenzothiophene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133.35
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131.13
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129.28
129.15
129.05
127.67
127.61
127.09
124.65
123.36
123.09
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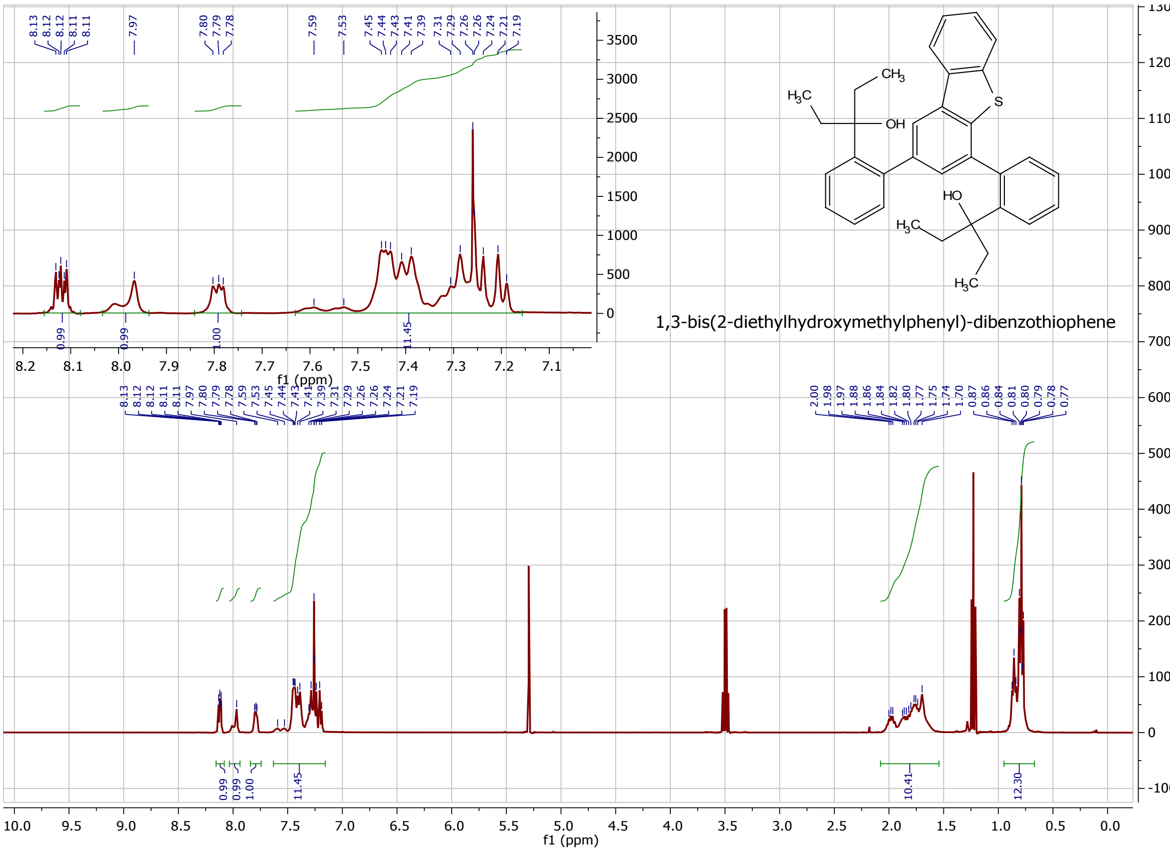
122.00
121.84
f1 (ppm)
Br
S
Br
1,3-bis(2-bromophenyl)-dibenzothiophene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2.00 1.98 1.97 1.88 1.86 1.84 1.82 1.80 1.77 1.75 1.74 1.70 0.87 0.86 0.84 0.81 0.80 0.79 0.78 0.77
0.99 0.99 1.00 11.45 10.41 12.30
3500 3000 2500 2000 1500 1000 500 0
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1
f1 (ppm)
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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
f1 (ppm)
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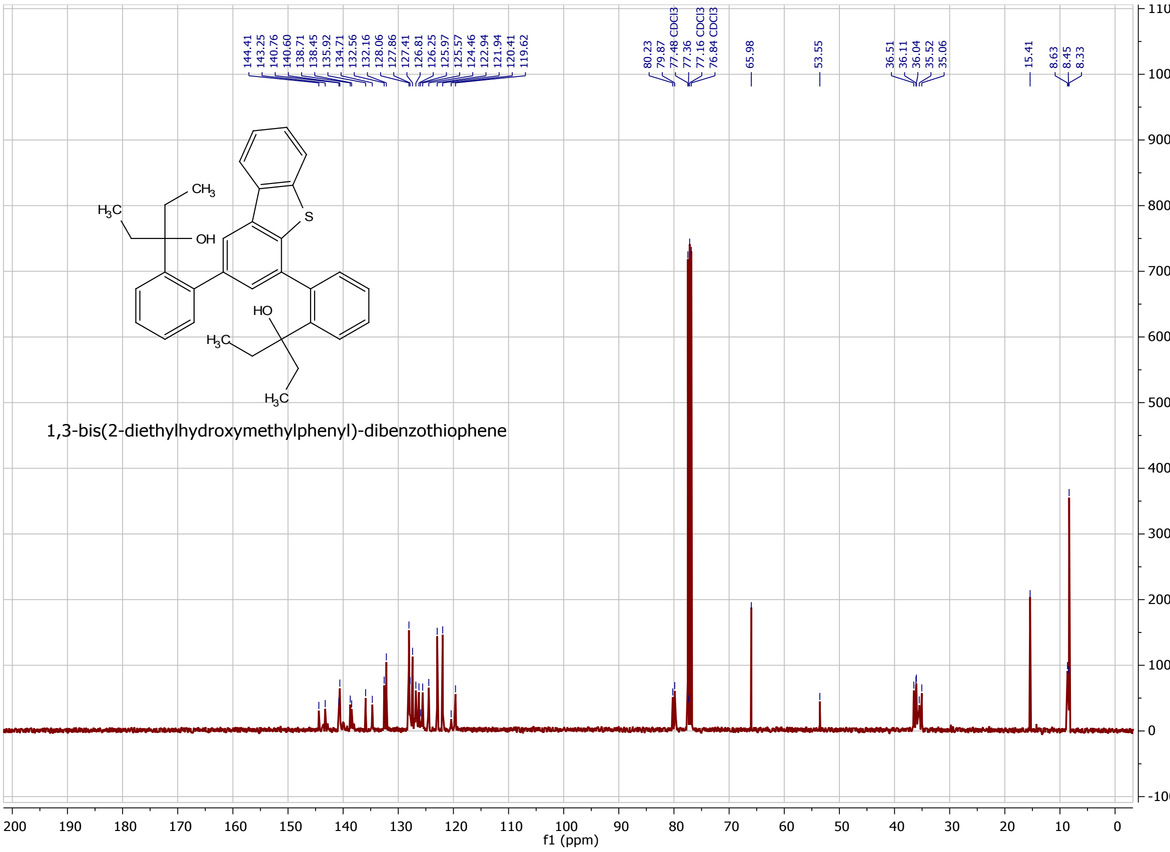
H3C
OH
S
HO
H3C
H3C
1,3-bis(2-diethylhydroxymethylphenyl)-dibenzothiophene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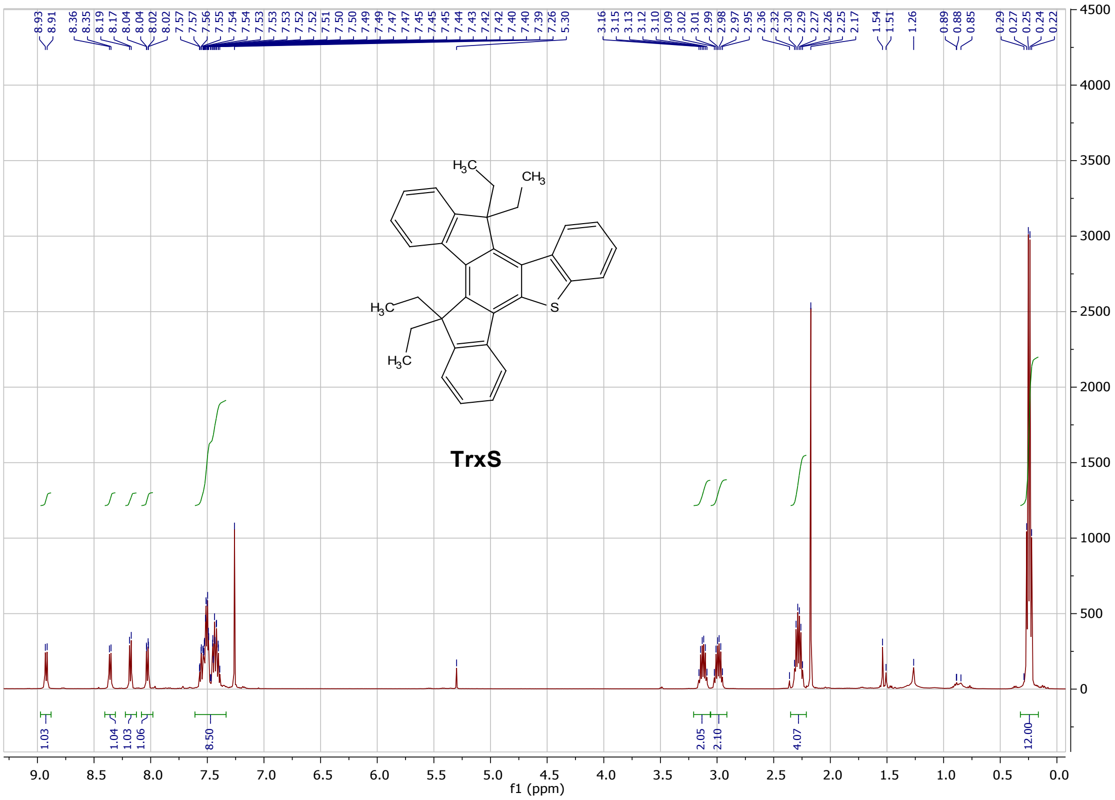
80.23 79.87 77.48 CDCl3 77.36 77.16 CDCl3 76.84 CDCl3
65.98
53.55
36.51 36.11 36.04 35.52 35.06
15.41
8.63 8.45 8.33
11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
CH3
H3C
OH
S
HO
H3C
H3C
1,3-bis(2-diethylhydroxymethylphenyl)-dibenzothiophene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2.30
2.29
2.27
2.26
2.25
2.17
1.54
1.51
1.26
0.89
0.88
0.85
0.29
0.27
0.25
0.24
0.22
1.03
1.04
1.03
1.06
8.50
2.05
2.10
4.07
12.00
H3C
CH3
H3C
H3C
S
TrxS
f1 (ppm)
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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
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1500
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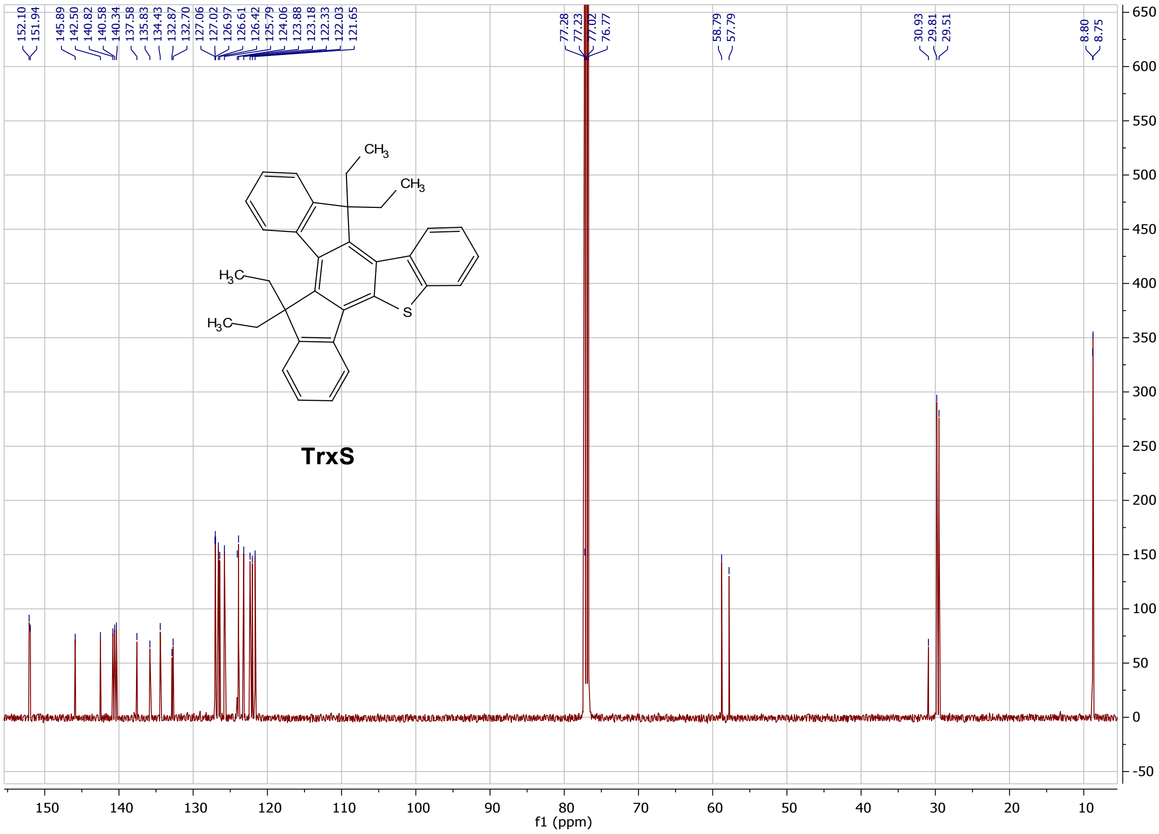
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127.06
127.02
126.97
126.61
126.42
125.79
124.06
123.88
123.18
122.33
122.03
121.65
77.28
77.23
77.02
76.77
58.79
57.79
30.93
29.81
29.51
8.80
8.75
CH3
CH3
H3C
H3C
S
TrxS
f1 (ppm)
150
140
130
120
110
100
90
80
70
60
50
40
30
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10
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600
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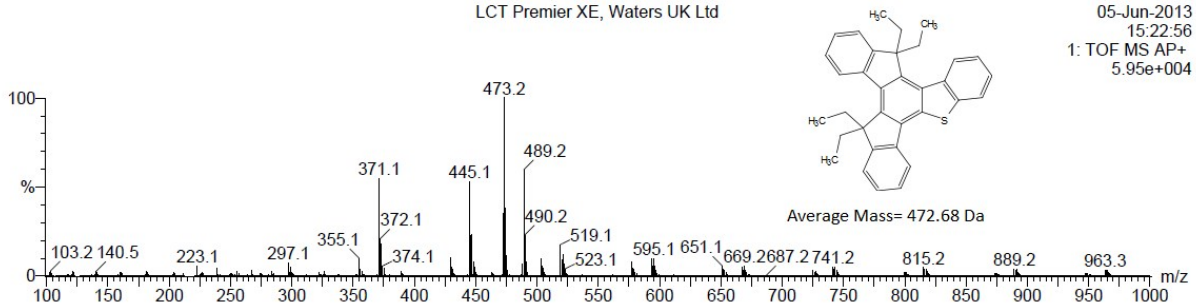
LCT Premier XE, Waters UK Ltd
05-Jun-2013
15:22:56
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5.95e+004
H₃C
CH₃
H₃C
H₃C
S
Average Mass= 472.68 Da
100
%
0
473.2
489.2
371.1
445.1
372.1
490.2
355.1
519.1
103.2
140.5
223.1
297.1
374.1
523.1
595.1
651.1
669.2
687.2
741.2
815.2
889.2
963.3
m/z
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850
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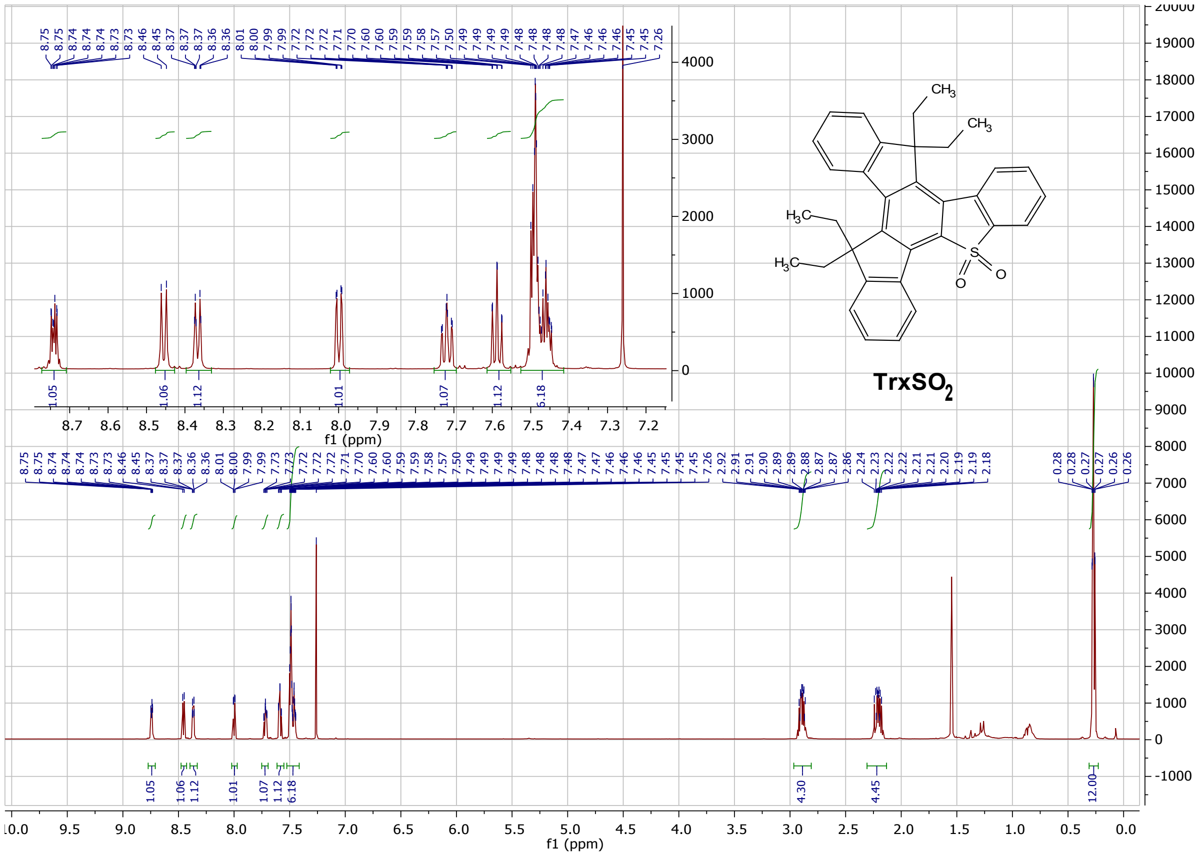
950
1000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2.92 2.91 2.90 2.89 2.89 2.88 2.87 2.87 2.86 2.24 2.23 2.22 2.22 2.21 2.21 2.20 2.19 2.19 2.18
0.28 0.28 0.27 0.27 0.26 0.26
1.05
1.06
1.12
1.01
1.07
1.12
6.18
4.30
4.45
12.00
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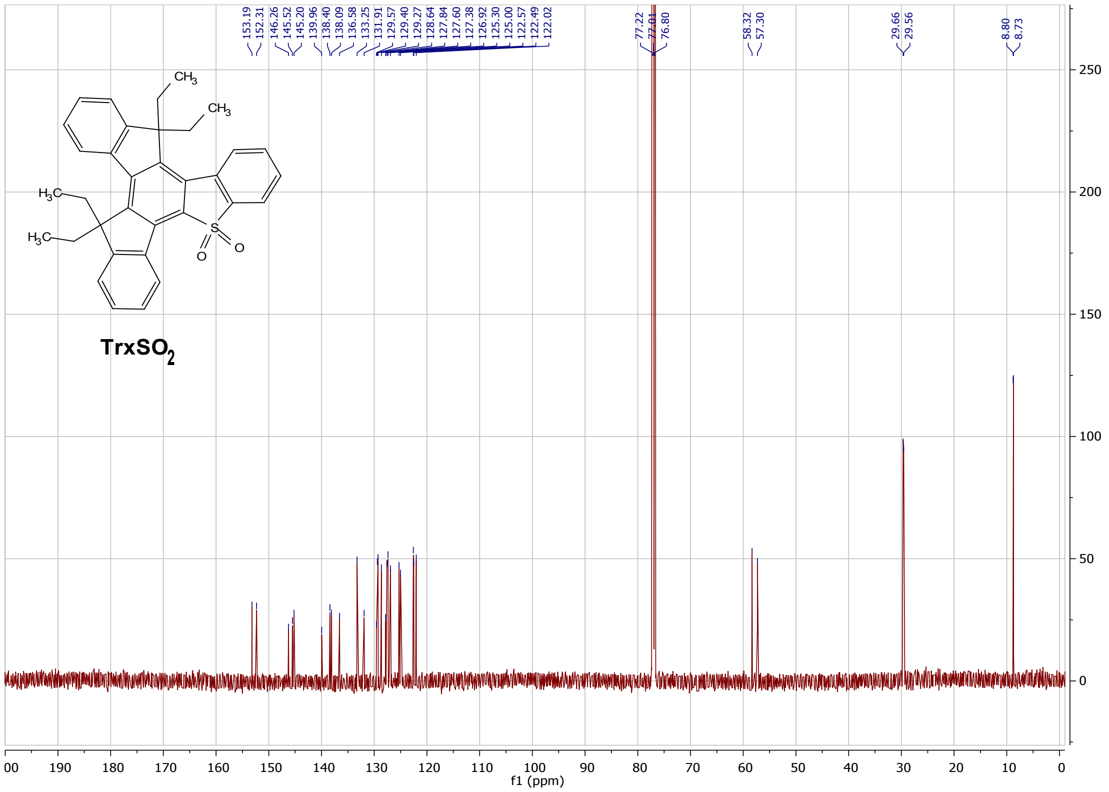
CH3
H3C
H3C
S
O
O
TrxSO2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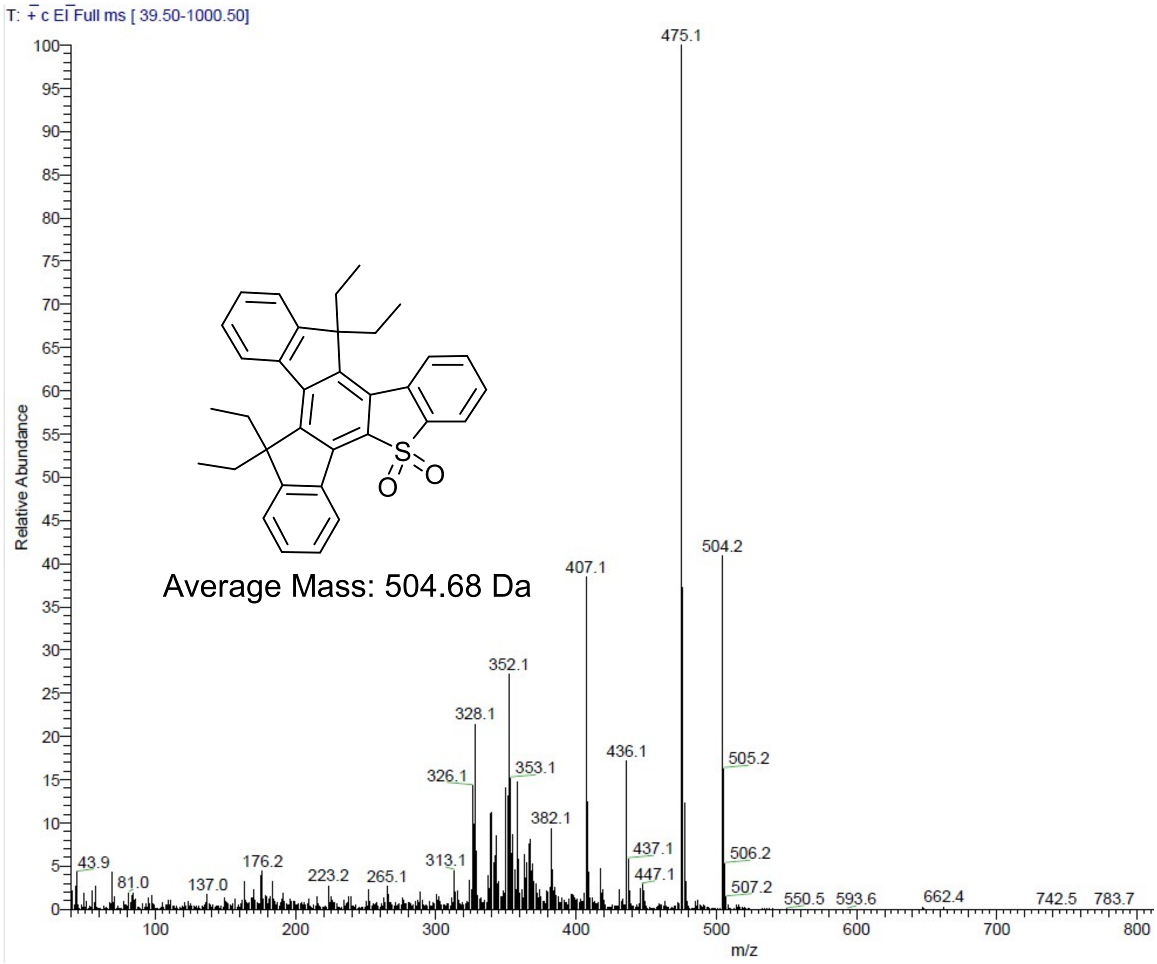
58.32 57.30
29.66 29.56
8.80 8.73
f1 (ppm)
CH3
CH3
H3C
H3C
S
O
O
TrxSO2


T: + c EI Full ms [39.50-1000.50]
Relative Abundance
m/z
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15
20
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30
35
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55
60
65
70
75
80
85
90
95
100
100
200
300
400
500
600
700
800
475.1
504.2
407.1
352.1
328.1
436.1
505.2
326.1
353.1
382.1
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43.9
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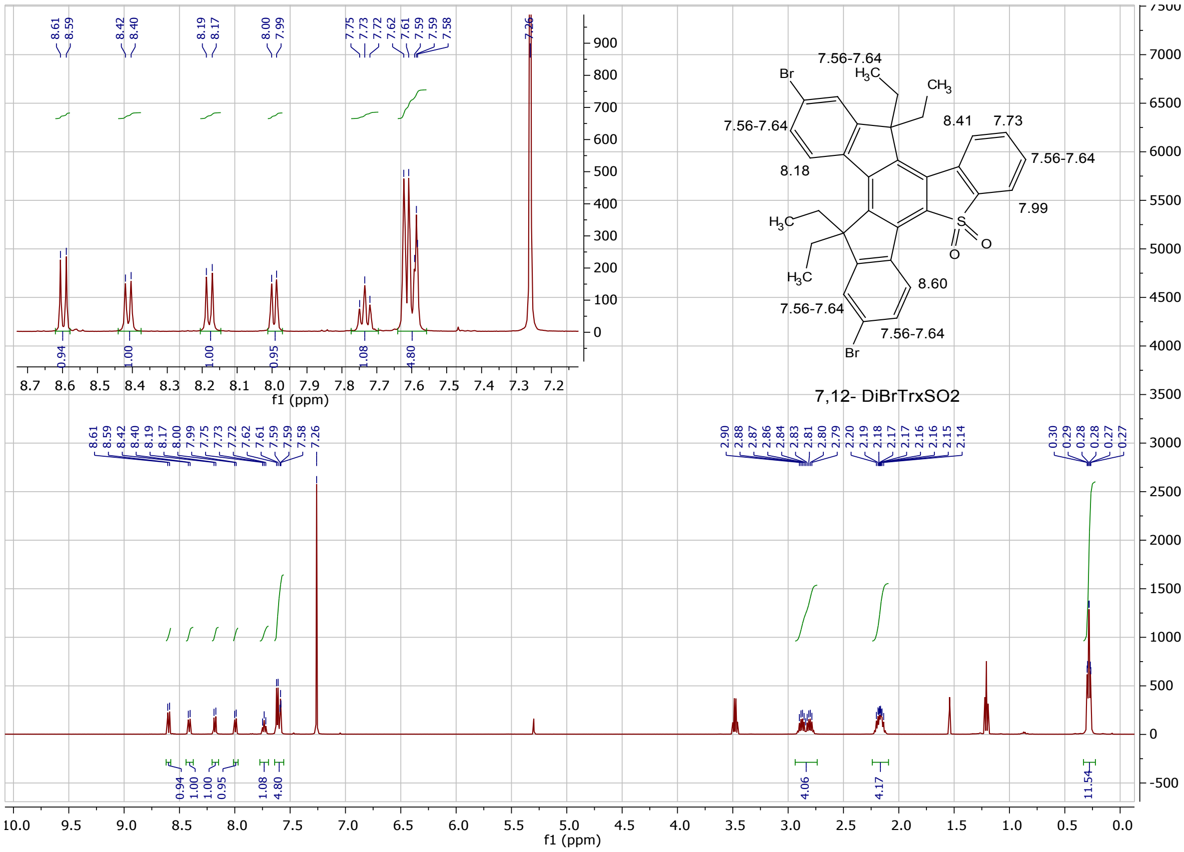
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0.94 1.00 1.00 0.95 1.08 4.80
8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2
f1 (ppm)
900 800 700 600 500 400 300 200 100 0
7.56-7.64
Br
H_3C
CH_3
8.41
7.73
7.56-7.64
7.56-7.64
8.18
7.99
H_3C
S
O
O
H_3C
8.60
7.56-7.64
7.56-7.64
Br
7,12- DiBrTrxSO2
8.61 8.59 8.42 8.40 8.19 8.17 8.00 7.99 7.75 7.73 7.72 7.62 7.61 7.59 7.59 7.58 7.26
2.90 2.88 2.87 2.86 2.84 2.83 2.81 2.80 2.79 2.20 2.19 2.18 2.17 2.17 2.16 2.16 2.15 2.14
0.30 0.29 0.28 0.28 0.27 0.27
0.94 1.00 1.00 0.95 1.08 4.80 4.06 4.17 11.54
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
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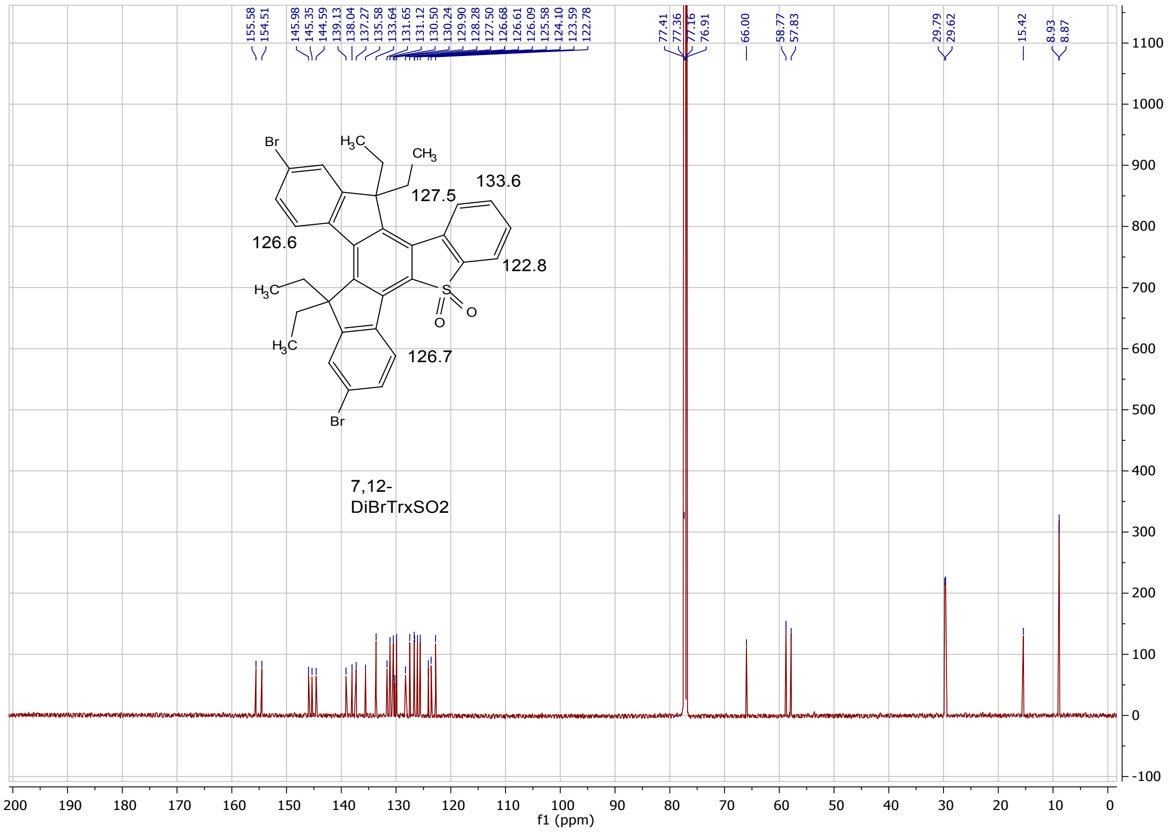
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77.41 77.36 77.16 76.91 66.00 58.77 57.83 29.79 29.62 15.42 8.93 8.87
Br
H_3C
CH_3
127.5
133.6
126.6
122.8
H_3C
S
O
O
H_3C
126.7
Br
7,12-
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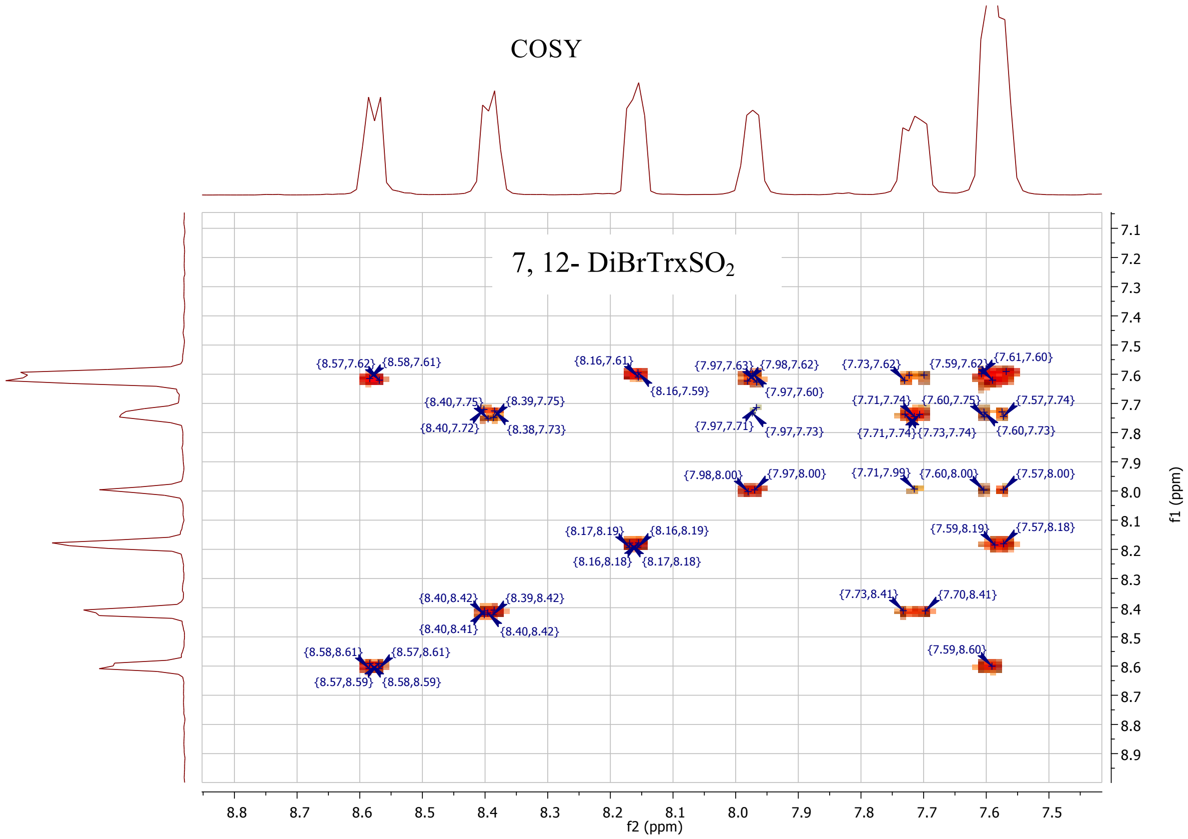
f1 (ppm)

COSY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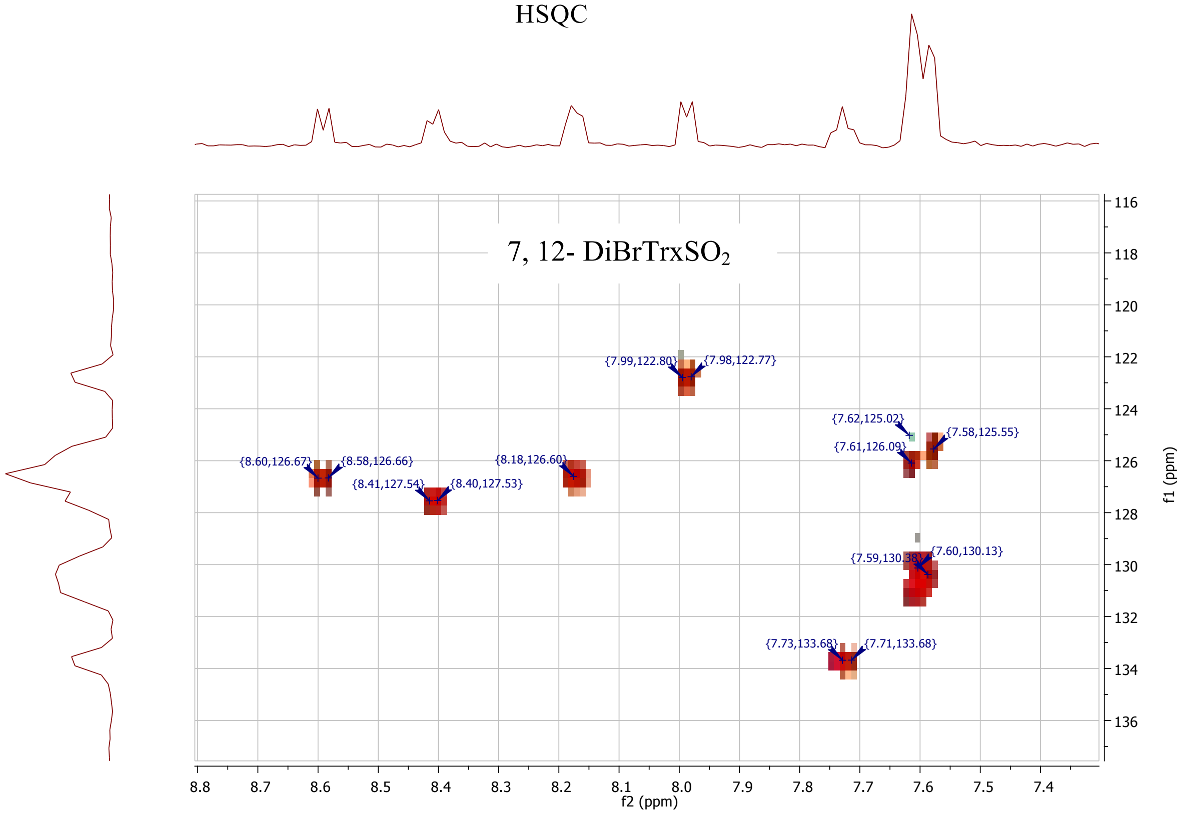
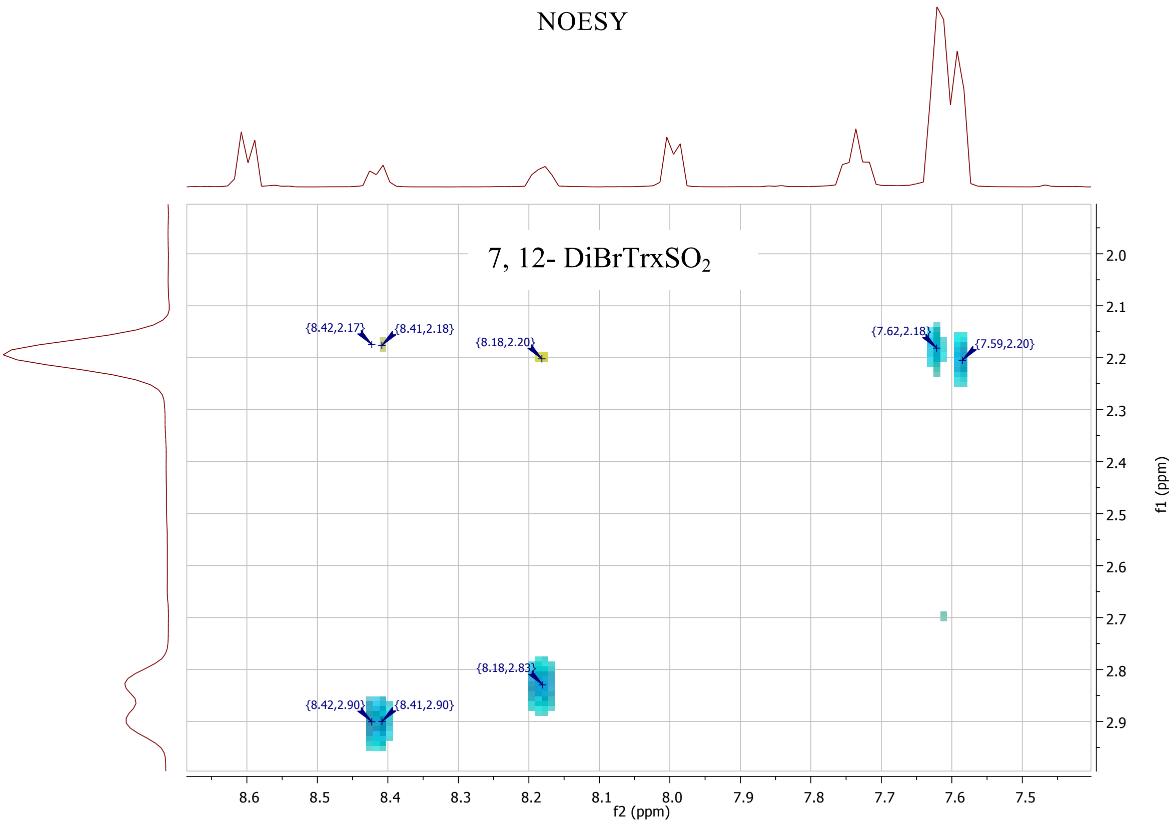
{7.59,8.60}
{8.57,8.59}
{8.58,8.59}
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.1
8.2
8.3
8.4
8.5
8.6
8.7
8.8
8.9
f1 (ppm)
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
f2 (ppm)

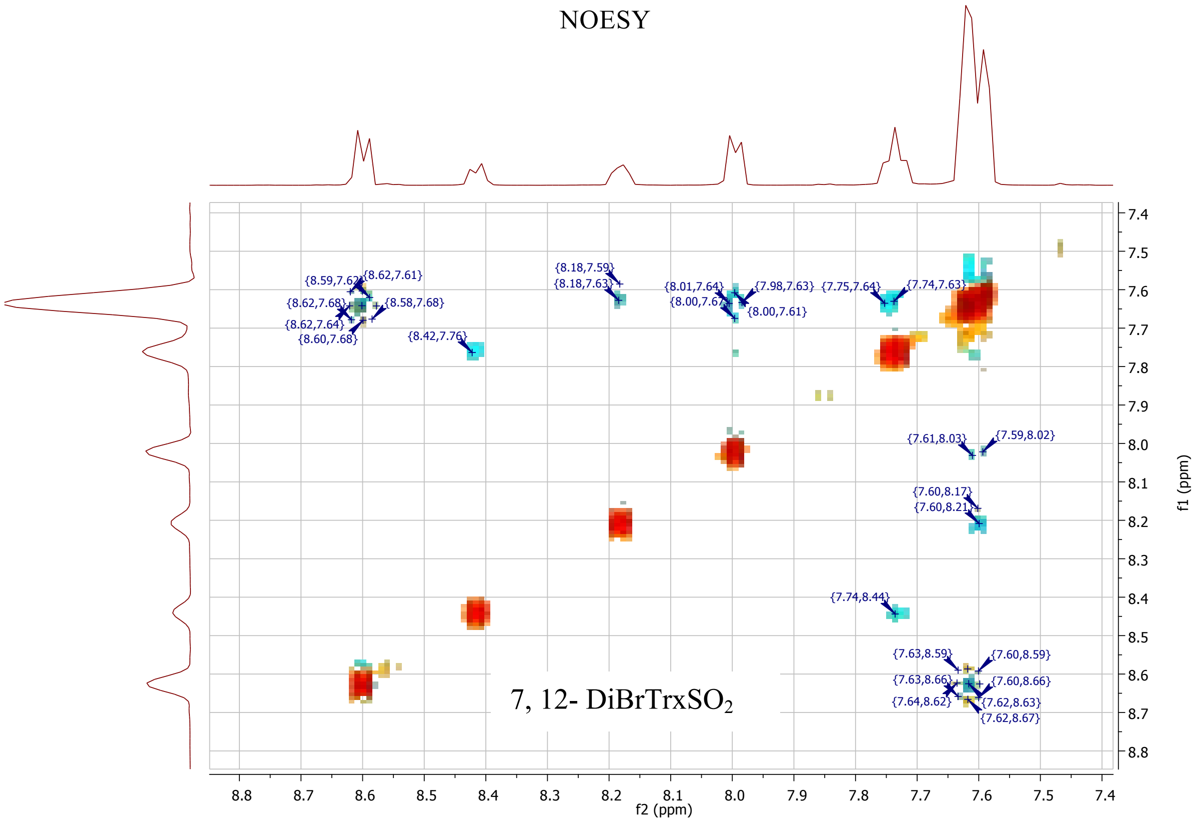
HSQC

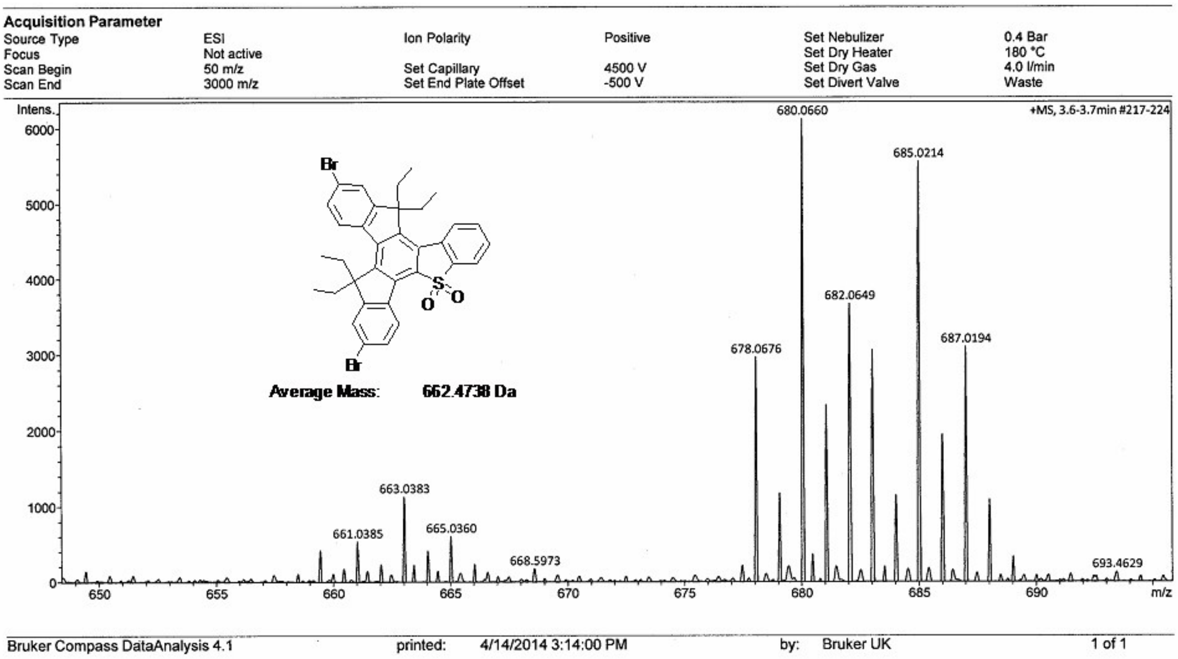

7, 12- DiBrTrxSO$_2$
{7.99,122.80}
{7.98,122.77}
{7.62,125.02}
{7.58,125.55}
{7.61,126.09}
{8.60,126.67}
{8.58,126.66}
{8.18,126.60}
{8.41,127.54}
{8.40,127.53}
{7.59,130.38}
{7.60,130.13}
{7.73,133.68}
{7.71,133.68}
116
118
120
122
124
126
128
130
132
134
136
f1 (ppm)
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
f2 (ppm)

NOESY


7, 12- DiBrTrxSO$_2$
{8.42,2.17}
{8.41,2.18}
{8.18,2.20}
{7.62,2.18}
{7.59,2.20}
{8.18,2.83}
{8.42,2.90}
{8.41,2.90}
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
f2 (ppm)
2.0
2.1
2.2
2.3
2.4
2.5
2.6
2.7
2.8
2.9
f1 (ppm)

NOESY


{8.59,7.62}
{8.62,7.61}
{8.62,7.68}
{8.58,7.68}
{8.62,7.64}
{8.60,7.68}
{8.42,7.76}
{8.18,7.59}
{8.18,7.63}
{8.01,7.64}
{8.00,7.67}
{7.98,7.63}
{8.00,7.61}
{7.75,7.64}
{7.74,7.63}
{7.61,8.03}
{7.59,8.02}
{7.60,8.17}
{7.60,8.21}
{7.74,8.44}
{7.63,8.59}
{7.60,8.59}
{7.63,8.66}
{7.60,8.66}
{7.64,8.62}
{7.62,8.63}
{7.62,8.67}
7, 12- DiBrTrxSO$_2$
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
f2 (ppm)
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.1
8.2
8.3
8.4
8.5
8.6
8.7
8.8
f1 (ppm)



Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

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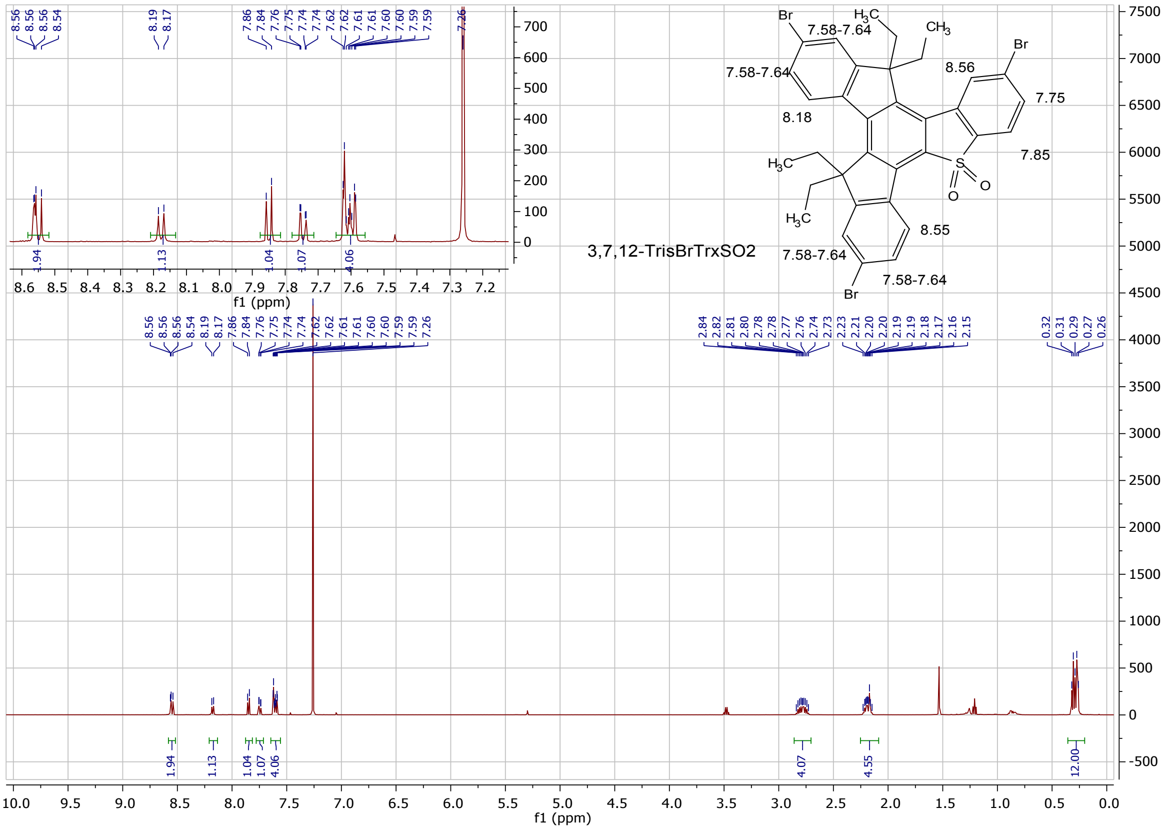
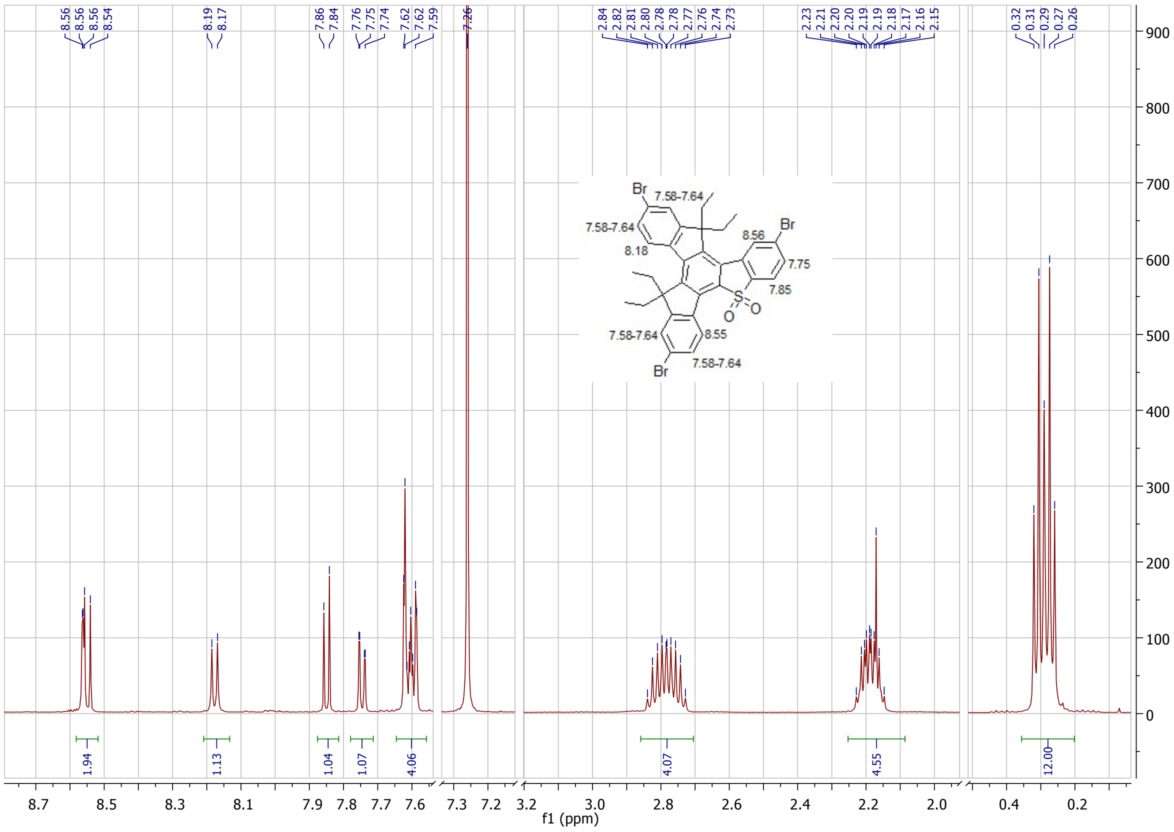
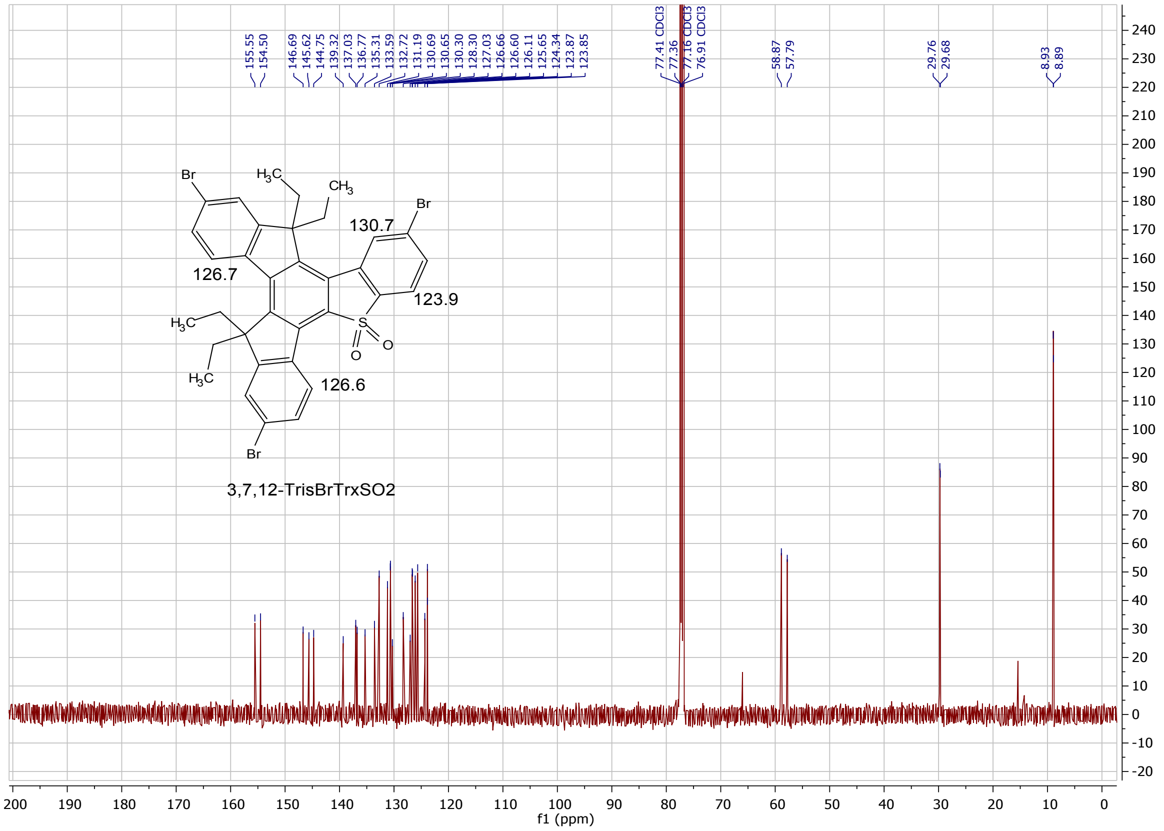
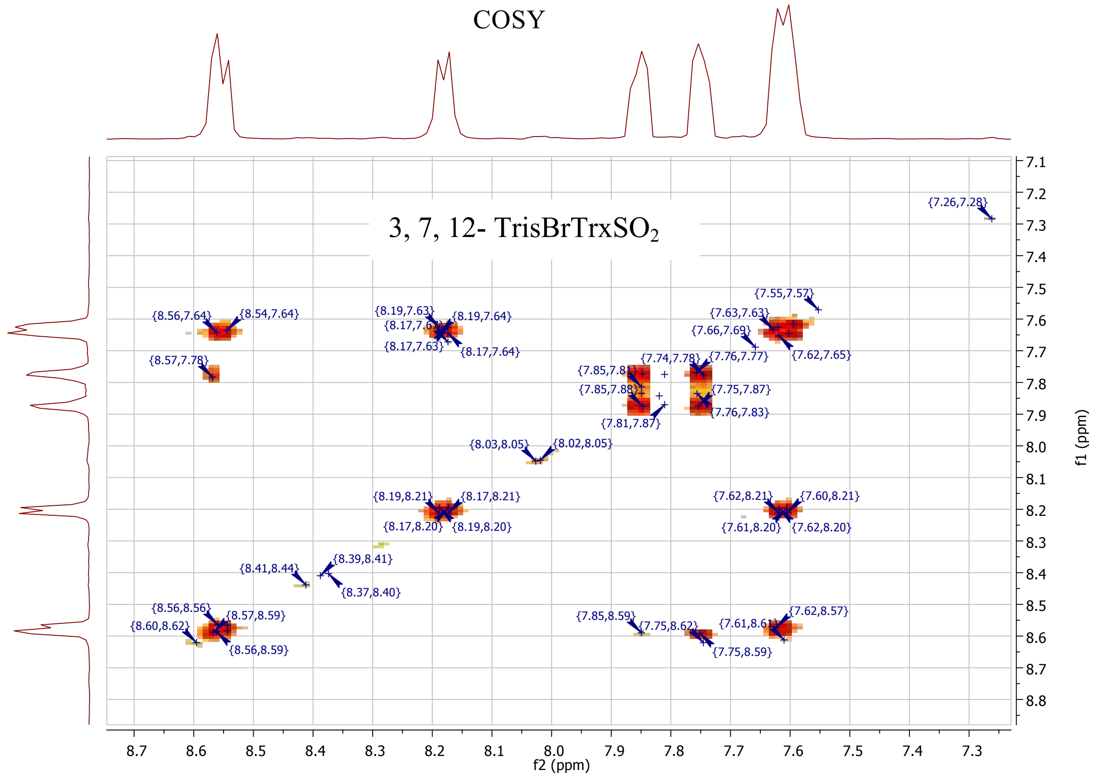

3,7,12-TrisBrTrxSO2
Br
H$_3$C
CH$_3$
7.58-7.64
7.58-7.64
8.56
7.75
8.18
7.85
S
O
8.55
7.58-7.64
7.58-7.64
1.94
1.13
1.04
1.07
4.06
4.07
4.55
12.00
f1 (ppm)

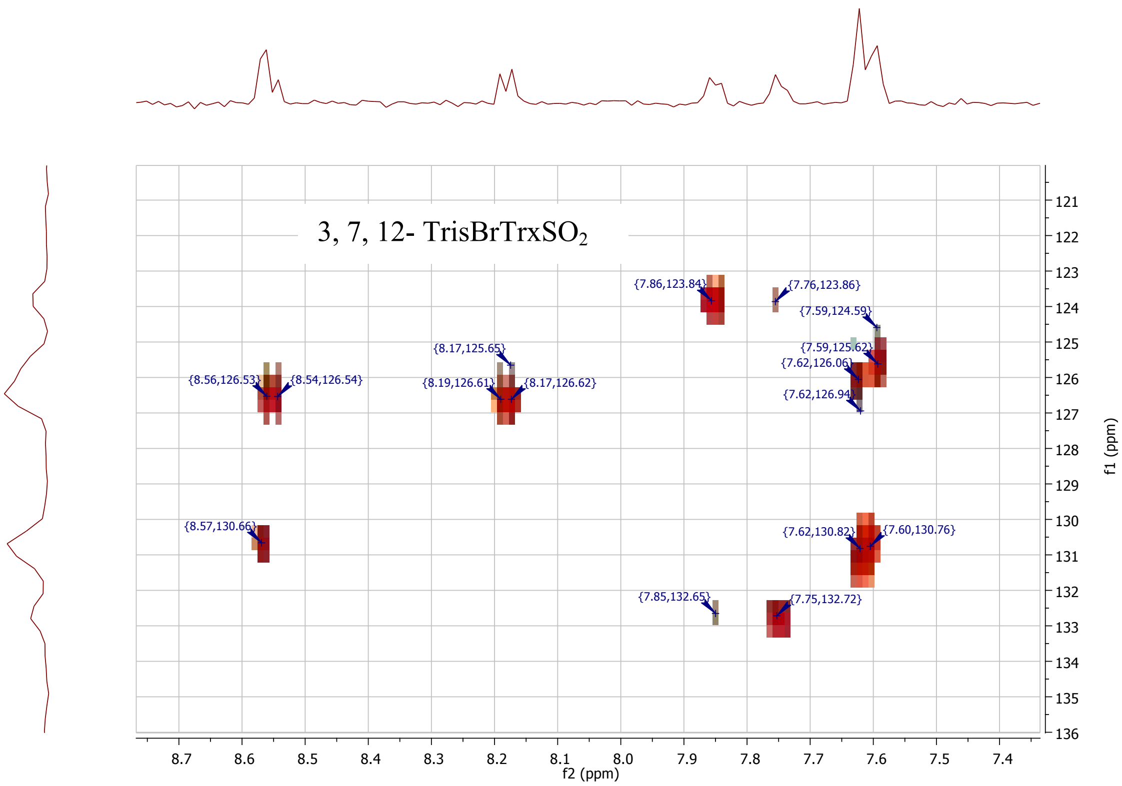

Br
7.58-7.64
7.58-7.64
8.56
8.18
7.75
7.85
S
O
7.58-7.64
8.55
7.58-7.64
1.94
1.13
1.04
1.07
4.06
4.07
4.55
12.00
f1 (ppm)


155.55
154.50
146.69
145.62
144.75
139.32
137.03
136.77
135.31
133.59
132.72
131.19
130.69
130.65
130.30
128.30
127.03
126.66
126.60
126.11
125.65
124.34
123.87
123.85
77.41 CDCl3
77.36
77.16 CDCl3
76.91 CDCl3
58.87
57.79
29.76
29.68
8.93
8.89
Br
H$_3$C
CH$_3$
Br
130.7
126.7
123.9
H$_3$C
S
O
O
H$_3$C
126.6
Br
3,7,12-TrisBrTrxSO2
f1 (ppm)

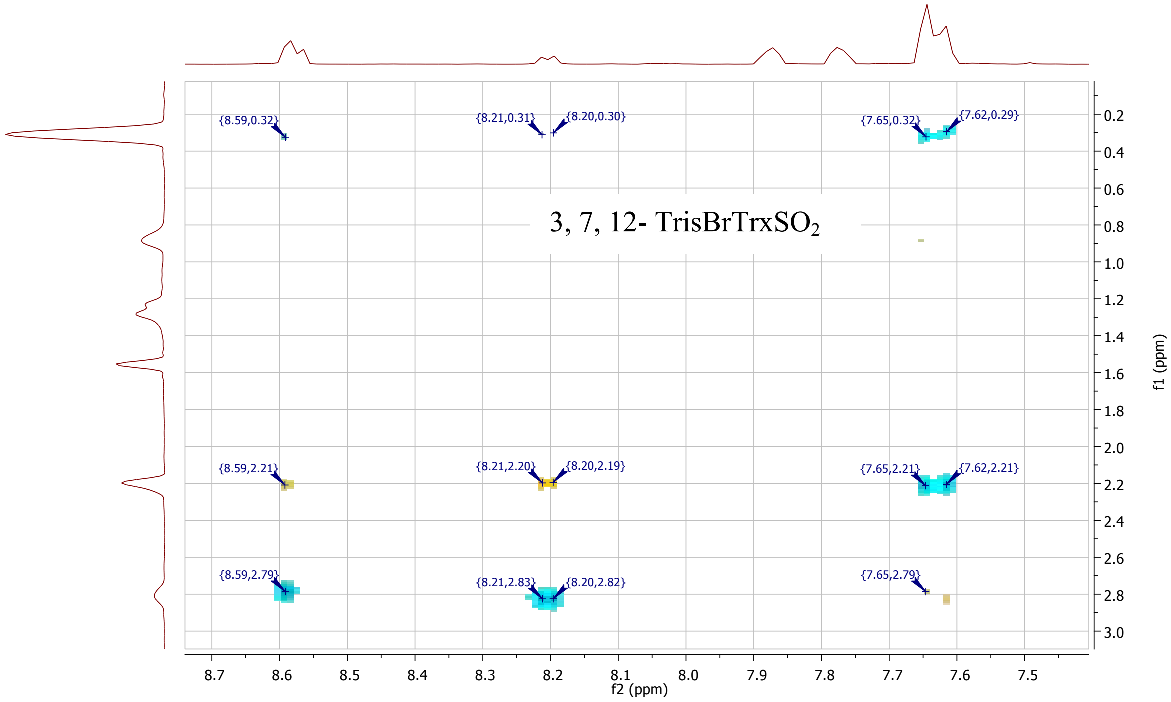
COSY


3, 7, 12- TrisBrTrxSO$_2$
{7.26,7.28}
{8.56,7.64}
{8.54,7.64}
{8.57,7.78}
{8.19,7.63}
{8.17,7.6}
{8.19,7.64}
{8.17,7.63}
{8.17,7.64}
{7.55,7.57}
{7.63,7.63}
{7.66,7.69}
{7.62,7.65}
{7.74,7.78}
{7.76,7.77}
{7.85,7.8}
{7.85,7.88}
{7.75,7.87}
{7.81,7.87}
{7.76,7.83}
{8.03,8.05}
{8.02,8.05}
{8.19,8.21}
{8.17,8.21}
{8.17,8.20}
{8.19,8.20}
{7.62,8.21}
{7.60,8.21}
{7.61,8.20}
{7.62,8.20}
{8.39,8.41}
{8.41,8.44}
{8.37,8.40}
{8.56,8.56}
{8.57,8.59}
{8.60,8.62}
{8.56,8.59}
{7.85,8.59}
{7.75,8.62}
{7.61,8.61}
{7.62,8.57}
{7.75,8.59}
f2 (ppm)
f1 (ppm)

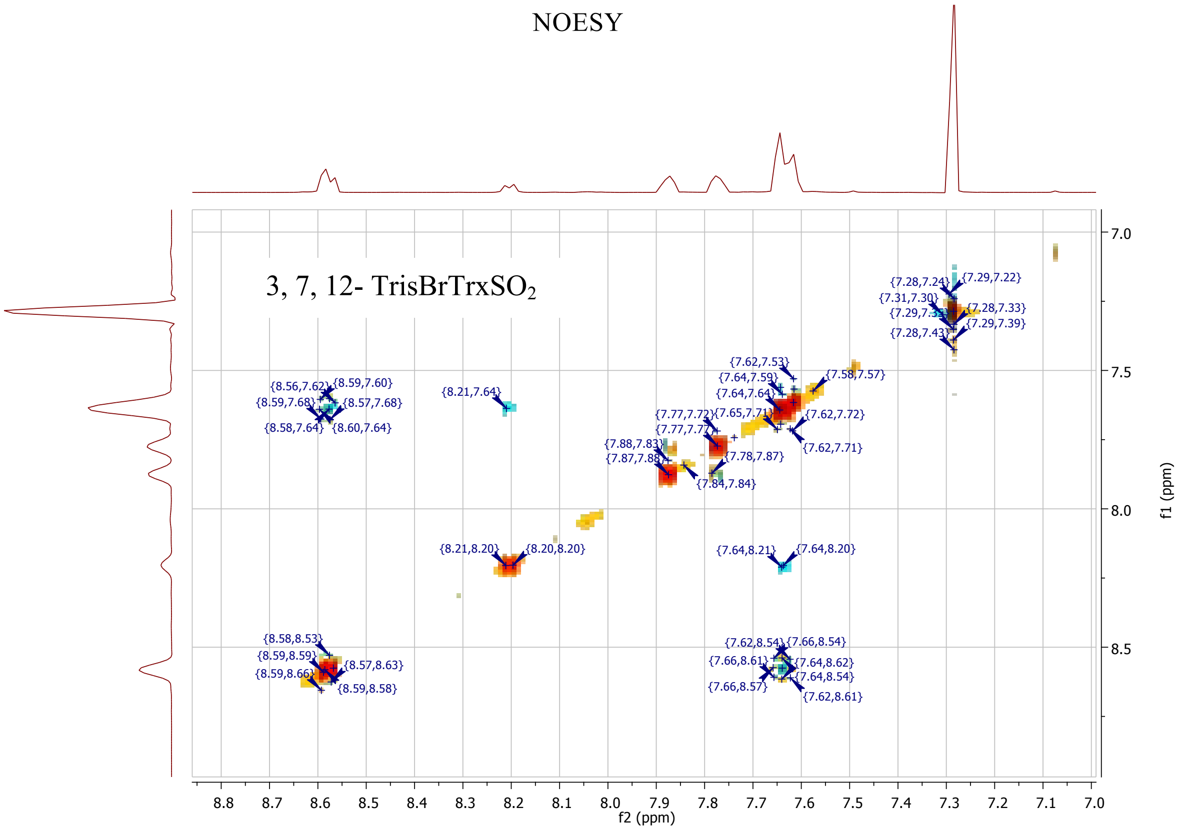
HSQC

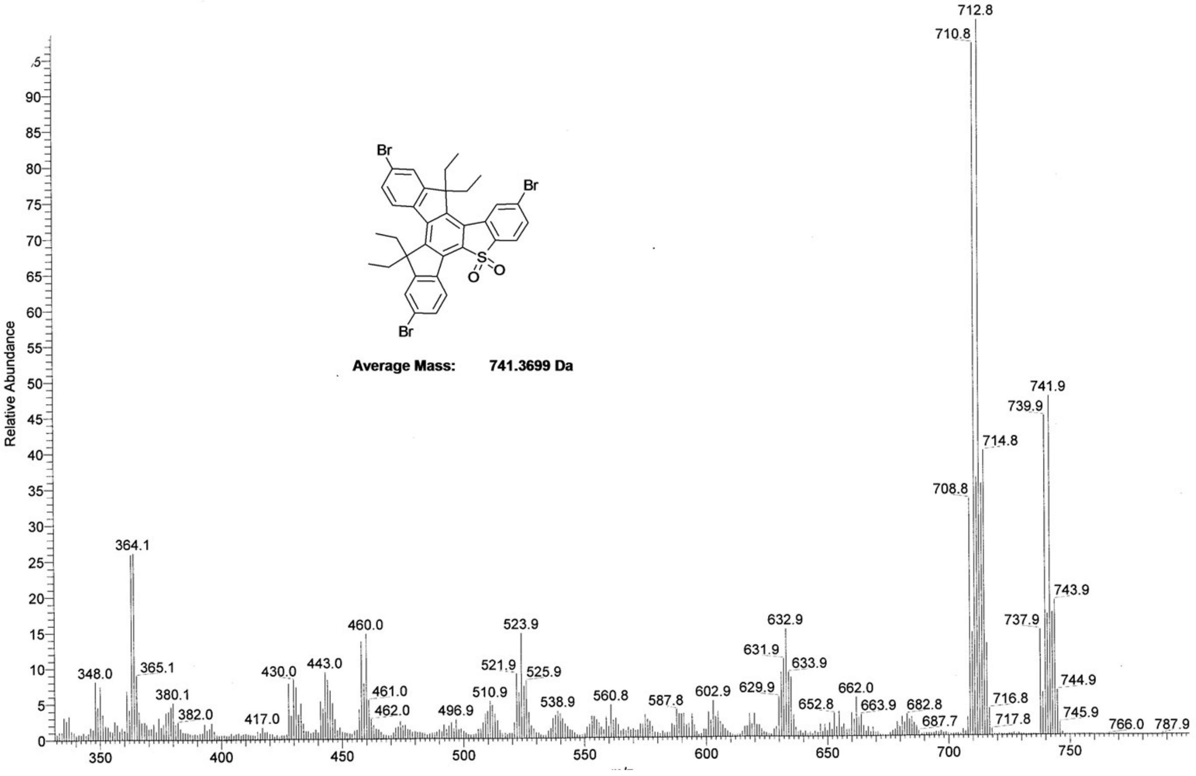

3, 7, 12- TrisBrTrxSO$_2$
{7.86,123.84}
{7.76,123.86}
{7.59,124.59}
{8.17,125.65}
{7.59,125.62}
{7.62,126.06}
{8.56,126.53}
{8.54,126.54}
{8.19,126.61}
{8.17,126.62}
{7.62,126.94}
{8.57,130.66}
{7.62,130.82}
{7.60,130.76}
{7.85,132.65}
{7.75,132.72}
f1 (ppm)
f2 (ppm)
121
122
123
124
125
126
127
128
129
130
131
132
133
134
135
136
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4

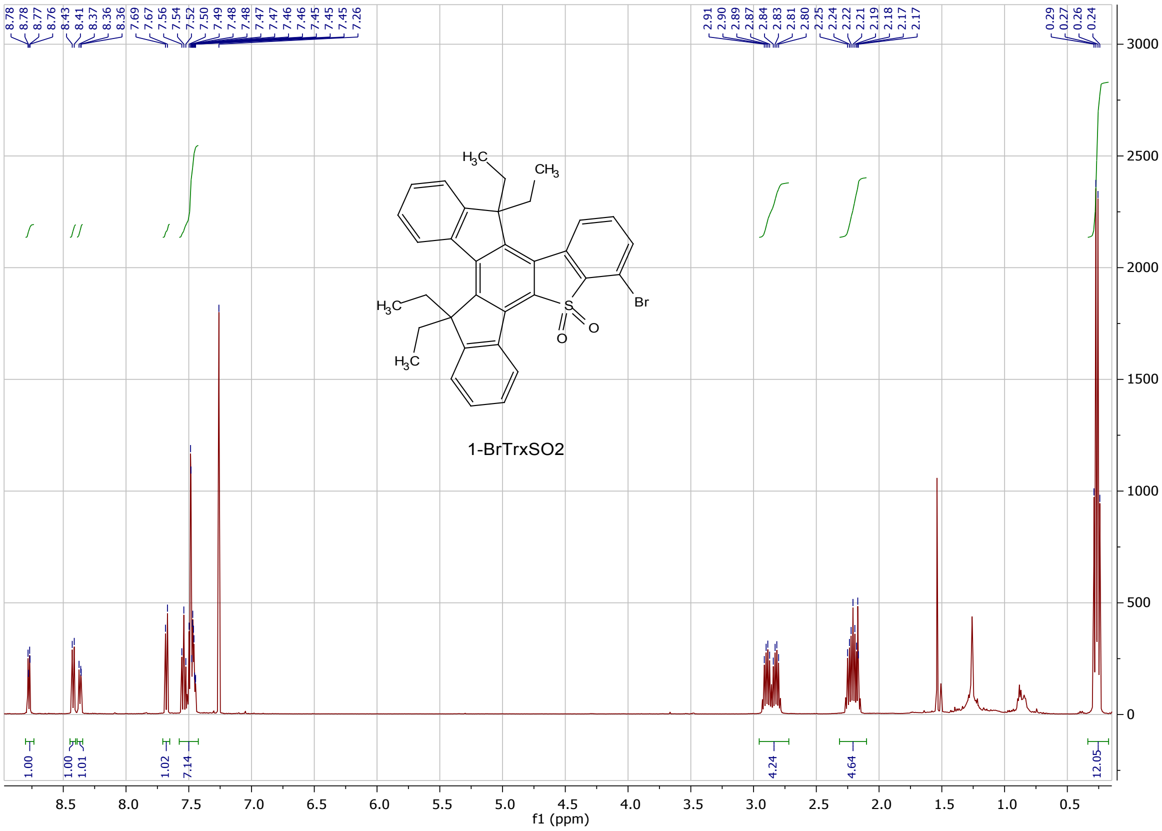
NOESY

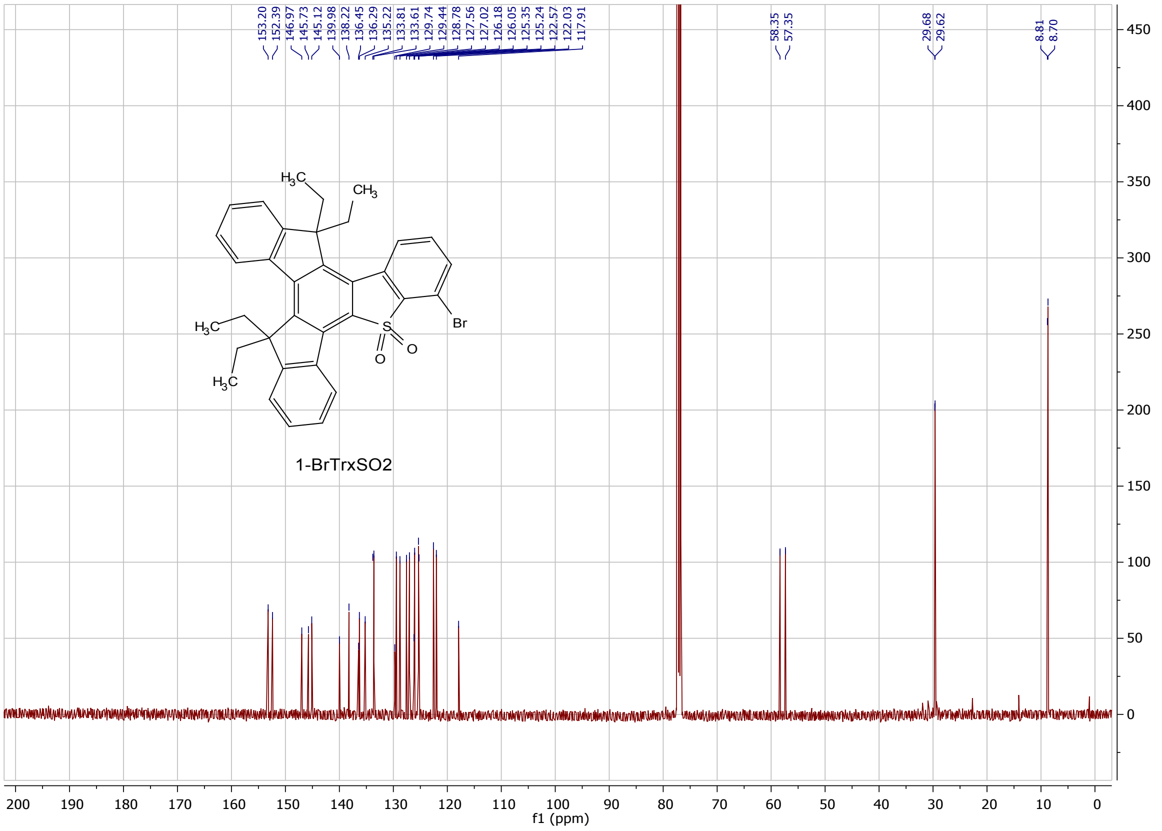

3, 7, 12- TrisBrTrxSO$_2$
{8.59,0.32}
{8.21,0.31}
{8.20,0.30}
{7.65,0.32}
{7.62,0.29}
{8.59,2.21}
{8.21,2.20}
{8.20,2.19}
{7.65,2.21}
{7.62,2.21}
{8.59,2.79}
{8.21,2.83}
{8.20,2.82}
{7.65,2.79}
f1 (ppm)
f2 (ppm)
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5

NOESY


3, 7, 12- TrisBrTrxSO$_2$
{7.28,7.24}
{7.29,7.22}
{7.31,7.30}
{7.29,7.33}
{7.28,7.33}
{7.29,7.39}
{7.28,7.43}
{7.62,7.53}
{7.64,7.59}
{7.58,7.57}
{7.64,7.64}
{8.56,7.62}
{8.59,7.60}
{8.59,7.68}
{8.57,7.68}
{8.58,7.64}
{8.60,7.64}
{8.21,7.64}
{7.77,7.72}
{7.65,7.71}
{7.62,7.72}
{7.77,7.77}
{7.88,7.83}
{7.87,7.88}
{7.78,7.87}
{7.62,7.71}
{7.84,7.84}
{8.21,8.20}
{8.20,8.20}
{7.64,8.21}
{7.64,8.20}
{8.58,8.53}
{8.59,8.59}
{8.57,8.63}
{8.59,8.66}
{8.59,8.58}
{7.62,8.54}
{7.66,8.54}
{7.66,8.61}
{7.64,8.62}
{7.64,8.54}
{7.66,8.57}
{7.62,8.61}
8.8
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
f2 (ppm)
7.0
7.5
8.0
8.5
f1 (ppm)

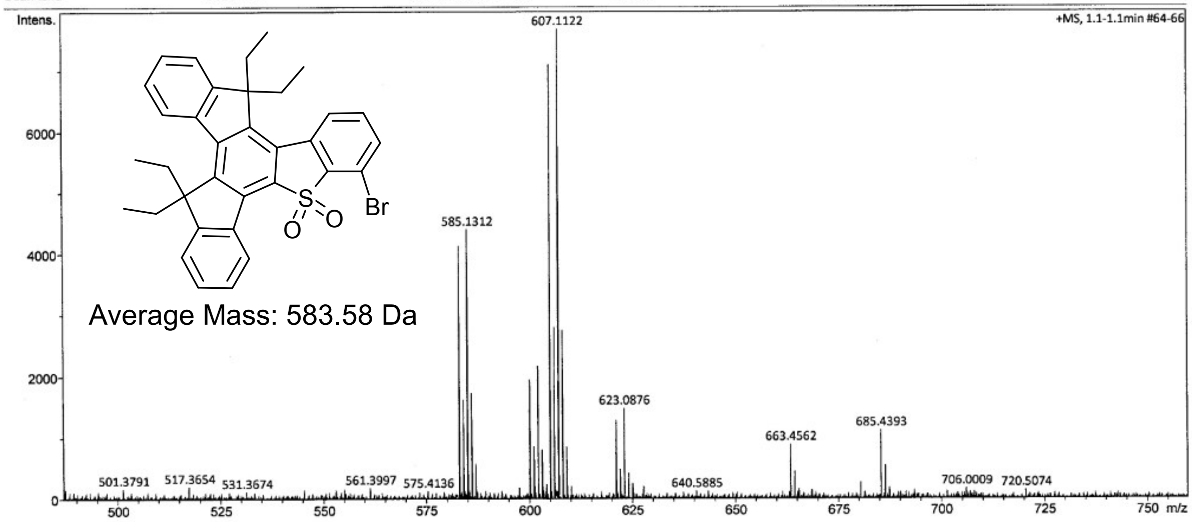

Br
Br
Br
S
O
O
Average Mass: 741.3699 Da
Relative Abundance
0
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
5
350
400
450
500
550
600
650
700
750
712.8
710.8
741.9
739.9
714.8
708.8
743.9
737.9
744.9
716.8
717.8
745.9
766.0
787.9
364.1
348.0
365.1
380.1
382.0
417.0
430.0
443.0
460.0
461.0
462.0
496.9
510.9
521.9
523.9
525.9
538.9
560.8
587.8
602.9
629.9
631.9
632.9
633.9
652.8
662.0
663.9
682.8
687.7


8.78
8.78
8.77
8.76
8.43
8.41
8.37
8.36
8.36
7.69
7.67
7.56
7.54
7.52
7.50
7.49
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.45
7.45
7.26
2.91
2.90
2.89
2.87
2.84
2.83
2.81
2.80
2.25
2.24
2.22
2.21
2.19
2.18
2.17
2.17
0.29
0.27
0.26
0.24
H3C
CH3
H3C
H3C
S
O
O
Br
1-BrTrxSO2
1.00
1.00
1.01
1.02
7.14
4.24
4.64
12.05
f1 (ppm)


153.20
152.39
146.97
145.73
145.12
139.98
138.22
136.45
136.29
135.22
133.81
133.61
129.74
129.44
128.78
127.56
127.02
126.18
126.05
125.35
125.24
122.57
122.03
117.91
58.35
57.35
29.68
29.62
8.81
8.70
H3C
CH3
H3C
H3C
S
O
O
Br
1-BrTrxSO2
f1 (ppm)

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste