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S U P P L E M E N T A R Y M A T E R I A L

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B E L O N G I N G TO T H E P A P E R

Regioselective Transition Metal- and Halogen-Free Direct Dithiolation at C(sp^3)-H of Nitrotoluenes with Diaryl Disulfides

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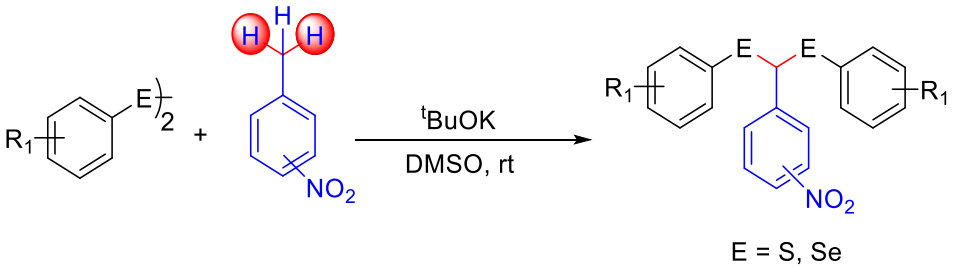
Contents

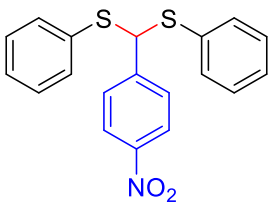
General Experimental Details

All reactions were carried out in oven-dried glassware with magnetic stirring. Nitrotoluenes used in this study were purchased from commercial suppliers and used without further purification. Various diphenyl disulfides used were prepared from corresponding thiols. Solvents screened in this report were used as purchased from suppliers. Silica gel (230-400 mesh size) was used for column chromatography. TLC analysis of reaction mixtures was performed using silica gel plates. All NMR experiments were carried out on Bruker 400/500 MHz spectrometers in DMSO-d_6 or $CDCl_3$ and NMR chemical shifts are reported in ppm referenced to the solvent peaks of $CDCl_3$ (7.26 ppm for ^1H and 77.16 (± 0.06) ppm for ^{13}C, respectively) or DMSO-d_6 (3.31 ppm for H_2O, 2.47 ppm for ^1H and 39.50 ppm for ^{13}C, respectively). The ^{77}Se NMR spectra were obtained at 76.31 MHz in $CDCl_3$ or DMSO-d_6. Chemical shifts are reported relative to dimethyl selenide (^{77}Se) (0 ppm). High resolution mass spectra (HRMS), electrospray mass spectra (ESMS), and low resolution mass spectra (LRMS) are reported for ions of ^{80}Se. High Resolution mass analysis is performed on quadruple-time of flight (Q-TOF) Bruker MicroTOF-Q II mass spectrometer equipped with an ESI source (-ve/+ve) and APCI (-ve/+ve). Single crystal X-ray data for compounds **6** and **14** were collected on Bruker D8 VENTURE diffractometer equipped with CMOS Photon 100 detector and Mo-Kα radiation (λ = 0.71073 Å) was used.

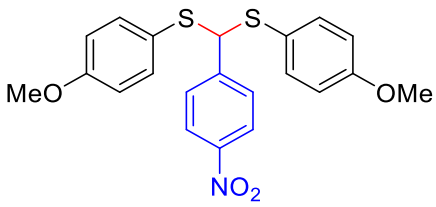
Scheme S1 General Procedure for Dithiolation of nitrotoluenes



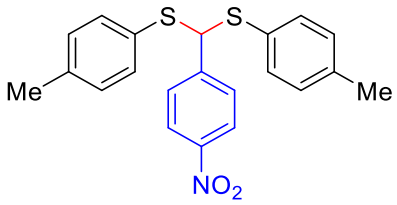
To a single neck flask, KO^{t}Bu (112 mg, 1 mmol) was added in one portion to the solution of the the diphenyl disulfide (109 mg, 0.5 mmol) and 4-nitrotoluene (137 mg, 1 mmol) in DMSO (2 mL) at room temperature. The resulted reaction mixture was stirred at room temperature for 6 hours. Next, saturated aqueous NaCl solution (15 mL) was added and the resulting mixture was extracted with ethyl acetate (3×20 mL). The organic layers were combined, dried over anhydrous Na_2SO_4, filtered and concentrated in *vacuo*. The crude product was purified by column chromatography by using dichloromethane/hexane (10/90). The desired compound **1** was obtained in yield (106 mg, 60%).



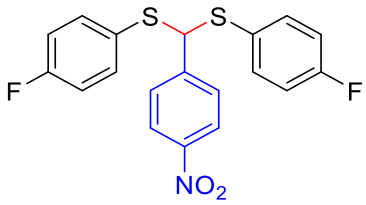
((4-nitrophenyl)methylene)bis(phenylsulfane) (1). Light yellow liquid, Yield 106 mg (60%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.73 Hz, 2H), 7.43 (d, *J* = 8.73 Hz, 2H), 7.34-7.32 (m, 4H), 7.26-7.24 (m, 6H), 5.42 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 147.1, 133.2, 133.1, 129.1, 128.8, 128.6, 123.7, 59.8. HRMS-ES$^-$ *m/z*: 352.0460 (calculated for $C_{19}H_{15}NO_2S_2$ - H^+: 352.0483).



((4-nitrophenyl)methylene)bis((4-methoxyphenyl)sulfane) (2). Colourless liquid, Yield 126 mg (61%); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10-8.08 (m, 2H), 7.38-7.36 (m, 2H), 7.30-7.27 (m, 4H), 6.82-6.79 (m, 4H), 5.21 (s, 1H), 3.80 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.4, 147.7, 147.2, 136.3, 128.8, 123.5, 123.4, 114.6, 61.8, 55.3. HRMS-APCI$^-$ *m/z*: 412.0672 (calculated for $C_{21}H_{19}NO_4S_2$ - H^+: 412.0676).

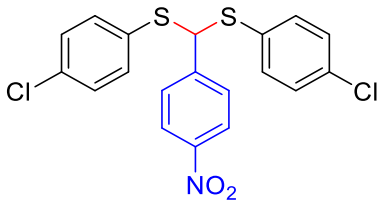


((4-nitrophenyl)methylene)bis(p-tolylsulfane) (3). Light yellow solid, Yield 138 mg (72%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.76 Hz, 2H), 7.41 (d, *J* = 8.76 Hz, 2H), 7.23 (d, *J* = 8.03 Hz, 4H), 7.06 (d, *J* = 8.03 Hz, 4H), 5.34 (s, 1H), 2.31 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 147.2, 138.9, 133.7, 129.9, 129.5, 128.8, 123.6, 60.5, 21.2. HRMS-ES$^+$ *m/z*: 404.0747 (calculated for $C_{21}H_{19}NO_2S_2$ + Na^+: 404.0749).

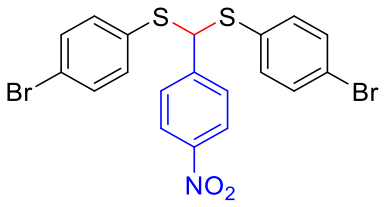


((4-nitrophenyl)methylene)bis((4-fluorophenyl)sulfane) (4). Light yellow liquid, Yield 133 mg (68%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.75 Hz, 2H), 7.37 (d, *J* = 8.75 Hz, 2H),

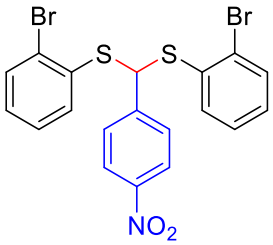
7.32-7.29 (m, 4H), 6.97-6.92 (m, 4H), 5.26 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2 (d, $^{1}J_{C-F}$ = 250.2 Hz), 147.4, 146.7, 136.3 (d, $^{3}J_{C-F}$ = 8.50 Hz), 128.7, 127.9 (d, $^{4}J_{C-F}$ = 3.45 Hz), 123.7, 116.3 (d, $^{2}J_{C-F}$ = 22.0 Hz), 61.2. HRMS-ES$^-$ *m/z*: 388.0301 (calculated for $C_{19}H_{13}F_2NO_2S_2$ - H^+: 388.0272).



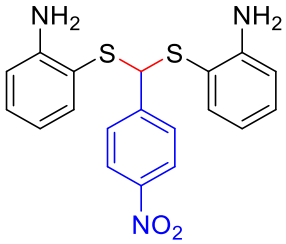
((4-nitrophenyl)methylene)bis((4-chlorophenyl)sulfane) (5). Light yellow solid, Yield 112 mg (53%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.71 Hz, 2H), 7.42 (d, *J* = 8.71 Hz, 2H), 7.26-7.21 (m, 8H), 5.33 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 146.3, 135.2, 134.8, 131.2, 129.4, 128.7, 123.8, 60.2. HRMS-APCI$^-$ *m/z*: 419.9657 (calculated for $C_{19}H_{13}Cl_2NO_2S_2$ - H^+: 419.9681).



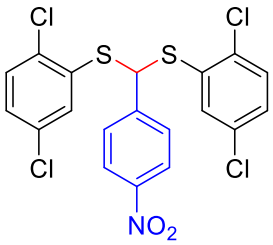
((4-nitrophenyl)methylene)bis((4-bromophenyl)sulfane) (6). Light yellow needles, mp 105 ^{o}C, Yield 115 mg (45%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.74 Hz, 2H), 7.43 (d, *J* = 8.74 Hz, 2H), 7.38 (d, *J* = 8.51 Hz, 4H), 7.17 (d, *J* = 8.51 Hz, 4H), 5.34 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 146.2, 134.8, 132.3, 131.8, 128.7, 123.9, 123.3, 59.9. HRMS-APCI$^-$ *m/z*: 507.8671 (calculated for $C_{19}H_{13}Br_2NO_2S_2$ - H^+: 507.8687).



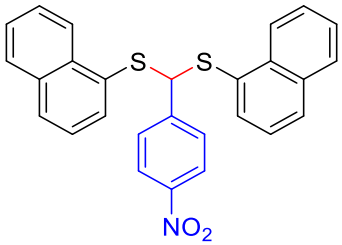
((4-nitrophenyl)methylene)bis((2-bromophenyl)sulfane) (7). Yellow semi-solid, Yield 153 mg (60%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.74 Hz, 2H), 7.60 (d, *J* = 8.74 Hz, 2H), 7.55 (dd, *J* = 7.87 Hz, *J* = 1.24 Hz, 2H), 7.44 (dd, *J* = 7.70 Hz, *J* = 1.54 Hz, 2H), 7.18 (dd, *J* = 7.50 Hz, *J* = 1.24 Hz, 2H), 7.11 (dd, *J* = 7.60 Hz, *J* = 1.54 Hz, 2H), 5.78 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 145.8, 134.8, 134.1, 133.4, 129.9, 129.0, 128.0, 127.8, 123.9, 56.9. HRMS-ES^{+} *m/z*: 531.8643 (calculated for $C_{19}H_{13}Br_2S_2NO_2$ + Na^{+}: 531.8647).



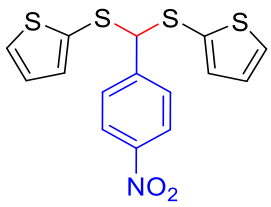
2,2'-(((4-nitrophenyl)methylene)bis(sulfanediyl))dianiline (8). Dark brown viscous liquid, Yield 88 mg (46%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.73 Hz, 2H), 7.41 (d, *J* = 8.73 Hz, 2H), 7.16-7.10 (m, 4H), 6.67 (d, *J* = 8.03 Hz, 2H), 6.57 (d, *J* = 7.50 Hz, 2H), 5.08 (s, 1H), 4.21 (bs, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.0, 148.0, 147.2, 137.4, 131.4, 128.5, 123.6, 118.4, 115.4, 115.2, 57.8. HRMS-APCI^{-} *m/z*: 382.0658 (calculated for $C_{19}H_{17}N_3O_2S_2$ - H^{+}: 382.0678)



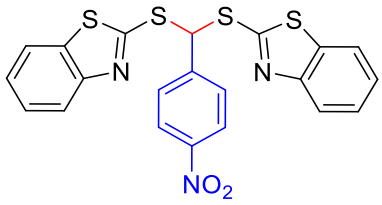
((4-nitrophenyl)methylene)bis((2,5-dichlorophenyl)sulfane) (9). Light grey semi-solid, Yield 125 mg (51%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.68 Hz, 2H), 7.60 (d, *J* = 8.68 Hz, 2H), 7.42 (d, *J* = 2.39 Hz, 2H), 7.30 (d, *J* = 8.60 Hz, 2H), 7.17 (dd, *J* = 8.60 Hz, *J* = 2.39 Hz, 2H), 5.77 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.9, 144.8, 135.1, 133.8, 133.6, 133.0, 130.9, 129.8, 128.8, 124.1, 56.2. HRMS-ES$^+$ *m/z*: 511.8875 (calculated for $C_{19}H_{11}Cl_4S_2NO_2$ + Na^+: 511.8878).



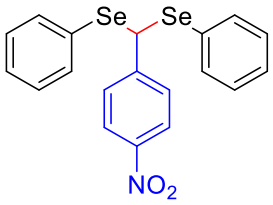
((4-nitrophenyl)methylene)bis(naphthalen-1-ylsulfane) (10). Yellow semi-solid, Yield 145 mg (64%) ; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.50 Hz, 2H), 8.01 (d, *J* = 8.70 Hz, 2H), 7.83 (d, *J* = 8.20 Hz, 4H), 7.61 (d, *J* = 7.10 Hz, 2H), 7.47 (d, *J* = 7.50 Hz, 2H), 7.37-7.30 (m, 6H), 5.35 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 147.3, 134.7, 134.2, 130.3, 130.1, 128.8, 128.7, 127.1, 126.4, 125.5, 125.2, 123.6, 60.2. HRMS-ES$^+$ *m/z*: 476.0748 (calculated for $C_{27}H_{19}S_2NO_2$ + Na^+: 476.0749).



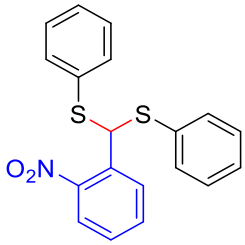
2,2'-(((4-nitrophenyl)methylene)bis(sulfanediyl))dithiophene (11). Cream viscous liquid, Yield 62 mg (34%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.77 Hz, 2H), 7.39 (dd, *J* = 5.34 Hz, *J* = 1.07 Hz, 2H), 7.35 (d, *J* = 8.77 Hz, 2H), 7.05 (dd, *J* = 3.58 Hz, *J* = 1.08 Hz, 2H), 6.95 (dd, *J* = 5.30 Hz, *J* = 3.67 Hz, 2H), 5.18 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 146.1, 136.6, 131.8, 130.2, 128.8, 127.7, 123.6, 63.9. HRMS-APCI^{+} *m/z* 363.9567 (calculated for $C_{15}H_{11}NO_2S_4$– H^{+} : 363.9589).



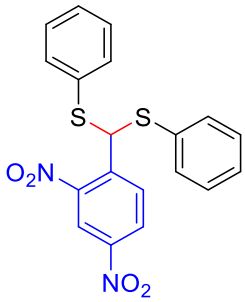
2,2'-(((4-nitrophenyl)methylene)bis(sulfanediyl))bis(benzo[*d*]thiazole) (12). Cream semi-solid, Yield 80 mg (34%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (d, *J* = 8.76 Hz, 2H), 7.90 (d, *J* = 8.76 Hz, 2H), 7.87 (d, *J* = 8.23 Hz, 2H), 7.74 (d, *J* = 7.76 Hz, 2H), 7.42 (t, *J* = 7.15 Hz, 2H), 7.32 (t, *J* = 7.15 Hz, 2H), 7.19 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1, 152.8, 145.1, 139.3, 135.7, 129.2, 126.4, 125.1, 124.0, 122.4, 121.2, 55.4. HRMS-ES^{+} *m/z* : 489.9784 (calculated for $C_{21}H_{13}N_3O_2S_4$ + Na^{+} : 489.9783).



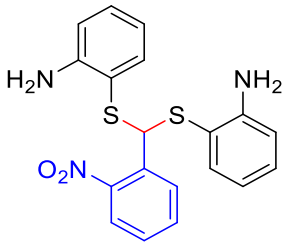
((4-nitrophenyl)methylene)bis(phenylselane) (13). Light yellow liquid, Yield 152 mg (68%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.75 Hz, 2H), 7.42-7.40 (m, 4H), 7.33 (d, *J* = 8.75 Hz, 2H), 7.27 (d, *J* = 7.30 Hz, 2H), 7.23-7.19 (m, 4H), 5.49 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.0, 146.8, 135.0, 129.8, 129.2, 128.9, 128.8 123.5, 41.8. ^{77}Se NMR (76 MHz, $CDCl_3$) δ 481.5. HRMS-APCI^{+} *m/z* : 447.9361 (calculated for $C_{19}H_{15}NO_2Se_2$ – H^{+} : 447.9353).



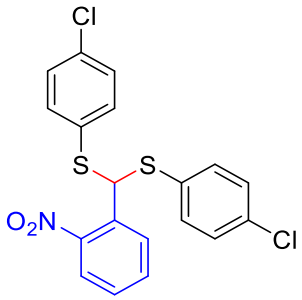
((2-nitrophenyl)methylene)bis(phenylsulfane) (14).[1] Brown crystals, mp 84 ºC, Yield 80 mg (45%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.10 Hz, 1H), 7.79 (d, *J* = 8.10 Hz, 1H), 7.53 (d, *J* = 7.80 Hz, 1H), 7.36-7.33 (m, 5H), 7.24-7.21 (m, 6H), 6.42 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 134.6, 133.3, 133.2, 132.9, 130.7, 129.1, 128.6, 128.4, 124.5, 54.5. HRMS-ES^{+} *m/z* : 354.0617 (calculated for $C_{19}H_{15}NO_2S_2$ + H^{+} : 354.0613).



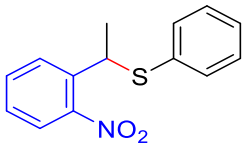
((2,4-dinitrophenyl)methylene)bis(phenylsulfane) (15). Dark brown viscous liquid, Yield 54 mg (27%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 2.26 Hz, 1H), 8.30 (dd, *J* = 8.71 Hz, *J* = 2.26 Hz, 1H), 8.06 (d, *J* = 8.71 Hz, 1H), 7.34-7.32 (m, 4H), 7.29-7.24 (m, 6H), 6.39 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 146.8, 141.6, 133.4, 132.3, 132.1, 129.4, 129.1, 127.0, 120.0, 54.1. HRMS-ES$^-$ *m/z* : 397.0311 (calculated for $C_{19}H_{14}N_2O_4S_2$ - H^+ : 397.0339).



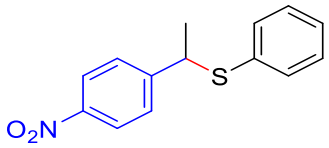
2,2'-(((2-nitrophenyl)methylene)bis(sulfanediyl))dianiline (16): Dark brown viscous liquid, Yield 71 mg (37%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.90 Hz, 1H), 7.68 (d, *J* = 8.17 Hz, 1H), 7.61 (t, *J* = 7.20 Hz, 1H), 7.36 (t, *J* = 7.20 Hz, 1H), 7.16 (dd, *J* = 7.70 Hz, *J* = 1.33 Hz, 2H), 7.10 (t, *J* = 7.70 Hz, 2H), 6.64 (d, *J* = 8.08 Hz, 2H), 6.55 (td, *J* = 7.51 Hz, *J* = 1.12 Hz, 2H), 5.87 (s, 1H), 4.20 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.1, 148.0, 137.3, 135.4, 133.0, 131.4, 131.2, 128.5, 123.9, 118.2, 115.1, 52.9. HRMS-ES$^+$ *m/z* : 384.0863 (calculated for $C_{19}H_{17}N_3O_2S_2$ + H^+ : 384.0835).



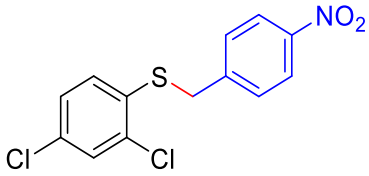
((2-nitrophenyl)methylene)bis((4-chlorophenyl)sulfane) (17): Brown viscous liquid, Yield 90 mg (43%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (td, *J* = 8.05 Hz, *J* = 0.95 Hz, 2H), 7.55 (td, *J* = 7.65 Hz, *J* = 0.95 Hz, 1H), 7.39 (td, *J* = 7.65 Hz, *J* = 1.18 Hz, 1H), 7.27-7.19 (m, 8H), 6.34 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 135.0, 134.4, 133.9, 133.3, 131.5, 130.5, 129.3, 129.0, 124.8, 55.0. HRMS-ES$^-$ *m/z* : 419.9707 (calculated for $C_{19}H_{13}Cl_2NO_2S_2 - H^+$: 419.9681).



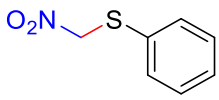
(1-(2-nitrophenyl)ethyl)(phenyl)sulfane (18): Colorless liquid, Yield 39 mg (30%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 7.95 Hz, *J* = 0.84 Hz, 1H), 7.55 (dd, *J* = 8.13 Hz, *J* = 0.97 Hz, 1H), 7.53 (t, *J* = 7.71 Hz, 1H), 7.31 (t, *J* = 7.71 Hz, 1H), 7.24-7.17 (m, 5H), 5.05 (q, *J* = 6.91 Hz, 1H), 1.67 (d, *J* = 6.91 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.1, 138.2, 133.8, 132.9, 132.6, 129.3, 128.9, 127.73, 127.69, 124.0, 42.1, 22.2. HRMS-ES$^+$ *m/z* : 282.0557 (calculated for $C_{14}H_{13}NO_2S + Na^+$: 282.0559).



(1-(4-nitrophenyl)ethyl)(phenyl)sulfane (19):[2] Colorless oil, Yield 55 mg (43%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.79 Hz, 2H), 7.37 (d, *J* = 8.79 Hz, 2H), 7.22-7.17 (m, 5H), 4.35 (q, *J* = 7.06 Hz, 1H), 1.64 (d, *J* = 7.06 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 146.9, 133.6, 133.2, 128.9, 128.1, 127.9, 123.6, 47.7, 21.7.

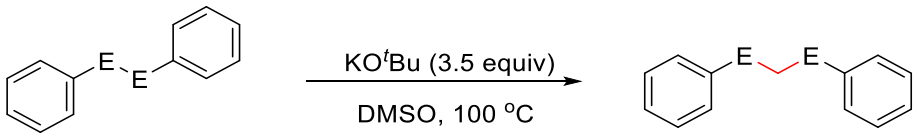


(2,4-dichlorophenyl)(4-nitrobenzyl)sulfane (20): Yellow liquid, Yield 75 mg (48%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.65 Hz, 2H), 7.42-7.40 (m, 3H), 7.11 (s, 2H), 4.15 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 144.2, 136.4, 133.8, 132.5, 132.1, 129.9, 129.7, 127.5, 123.8, 37.5.

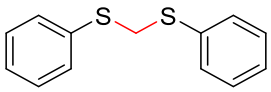


(nitromethyl)(phenyl)sulfane (21):[3] Light yellow oil, Yield 27 mg (32%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.48 (m, 2H), 7.37-7.35 (m, 3H), 5.44 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.2, 131.7, 129.6, 129.2, 79.5.

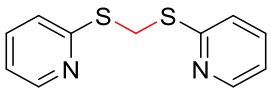
General Procedure for Synthesis of thio- and selenoacetals 22-25.



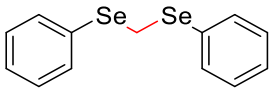
Diphenyl disulfide (110 mg, 0.5 mmol) was taken in DMSO in a 10 ml round bottom flask followed by addition of KO^{t}Bu (197 mg, 1.75 mmol). The reaction mixture was placed in a pre-heated oil bath at 100 °C and heated for 24 h. Then reaction mixture was poured in brine and extracted with dichloromethane (3 x 5 mL), dried over sodium sulfate and evaporated over rotary evaporator. Purification by chromatography (dichloromethane/hexane: 3/97) resulted in the pure product **22**.



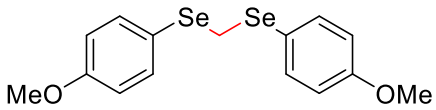
bis(phenylthio)methane (22):[4] Yield 29 mg (25%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.40 (m, 4H), 7.32-7.28 (m, 4H), 7.25-7.23 (m, 2H), 4.33 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.0, 130.7, 129.0, 127.2, 40.6. LRMS-GC *m/z* : 232.0 (calculated for $C_{13}H_{12}S_2$: 232.0).



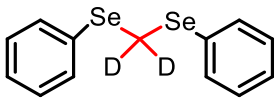
bis(pyridin-2-ylthio)methane (23):[4] Yield 27 mg (23%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.2 Hz, 2H), 7.47 (td, *J* = 7.9 Hz, *J* = 1.8 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 7.01 – 6.98 (m, 2H), 5.05 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3, 149.5, 136.0, 122.5, 119.7, 30.7. LRMS-GC *m/z* : 234.0 (calculated for $C_{11}H_{10}N_2S_2$: 234.0).



bis(phenylselanyl)methane (24):[4] Light Yellow liquid, Yield 106 mg (65%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.51 (m, 4H), 7.72-7.26 (m, 6H), 4.22 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.0, 130.8, 129.2, 127.6, 21.0. ^{77}Se NMR (76 MHz, $CDCl_3$) δ 345.5. LRMS-GC m/z : 327.9 (calculated for $C_{13}H_{12}Se_2$: 327.9).

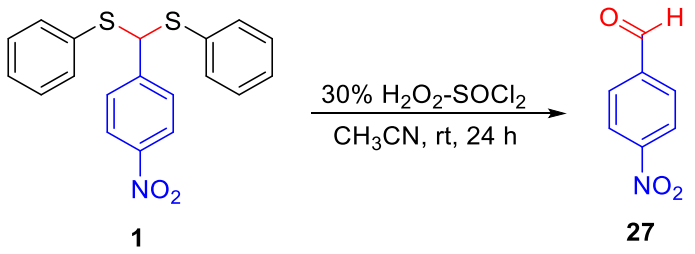


bis((4-methoxyphenyl)selanyl)methane (25). Orange semi-solid, Yield 116 mg (60%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.77 Hz, 4H), 6.80 (d, *J* = 8.77 Hz, 4H), 4.06 (s, 2H), 3.79 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 135.9, 120.8, 114.8, 55.3, 23.7. ^{77}Se NMR (76 MHz, $CDCl_3$) δ 336.5 HRMS-ES^{+} *m/z* : 426.9110 (calculated for $C_{15}H_{16}O_2Se_2$ + K^{+} : 426.9115).



bis(phenylselanyl)methane-*d*$_2$ (26): Yield 50 mg (30%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.46 (m, 4H), 7.33-7.18 (m, 6H), 3.43 (bs, H_2O), 2.47 (DMSO). HRMS-GC^{+} *m/z* : 329.9387 (calculated for $C_{13}H_{10}D_2Se_2$: 329.9395).

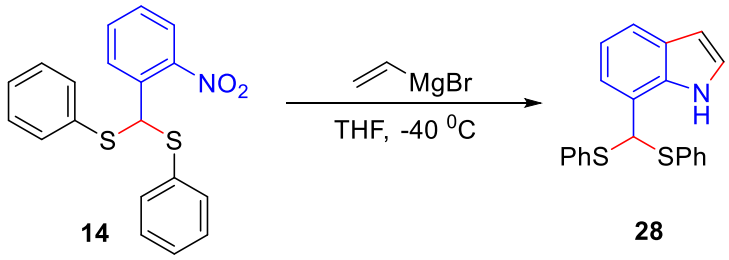
General Procedure for Synthesis of 4-Nitrobenzaldehyde[5]



A mixture of dithioacetal **1** (90 mg, 0.25 mmol), H_2O_2 (30%, 80 µl, 1 mmol) and $SOCl_2$ (36 µl, 0.5 mmol) was stirred in CH_3CN at 25 ^oC for 24 h. The progress of the reaction was monitored by TLC. After completion, the reaction was quenched by the addition of H_2O (10 mL) and the resulting mixture was extracted with EtOAc (4 x 3 mL). The combined organic phase was dried over anhydrous sodium sulfate, and evaporated. Chromatography on silica gel (DCM/Hexane: 50/50) gave a pure product **27** in 60% yield.

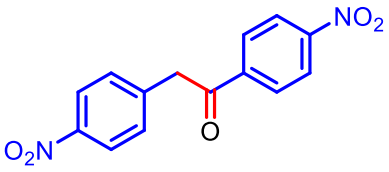
4-Nitrobenzaldehyde (27): Light yellow solid, Yield 38 mg (60%); ^1H NMR (400 MHz, $CDCl_3$) δ 10.15 (s, 1H), 8.39 (d, *J* = 8.63 Hz, 2H), 8.06 (d, *J* = 8.63 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.2, 151.2, 140.0, 130.5, 124.3.

General Procedure for Synthesis of 7-(bis(phenylthio)methyl)-1*H*-indole[6]



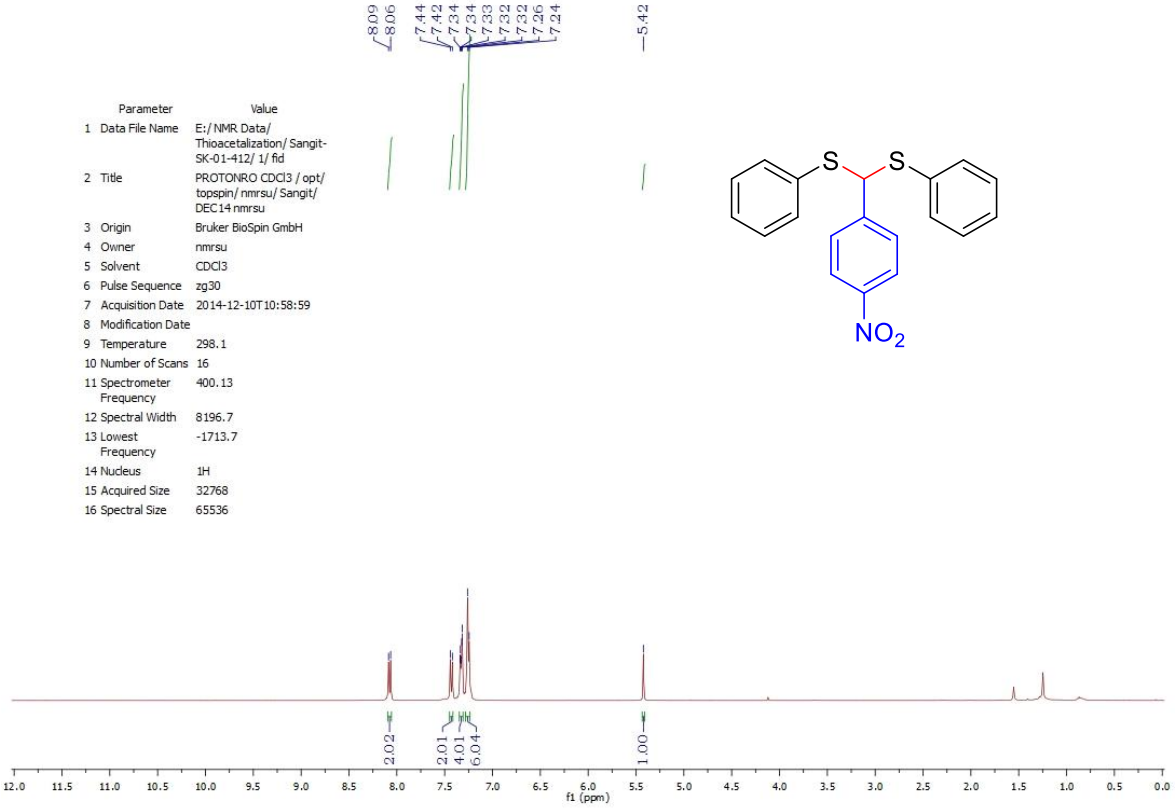
((2-nitrophenyl)methylene)bis(phenylsulfane) **14** (88 mg, 0.25 mmol) was dissolved in THF (3 mL) and the solution cooled to –40 °C. Then vinylmagnesium bromide (0.75 mL, 1.0 M in THF, 3.0 equiv) was added dropwise over 15 min. After completion of the addition, the reaction mixture was stirred at –40 °C. The progress of reaction was monitored by TLC. When all the starting material was consumed, the reaction mixture was quenched with saturated aqueous NH_4Cl solution (5 mL), extracted with ethyl acetate (3×10 mL), the combined organic layers were washed with brine (10 mL) and dried over Na_2SO_4. The crude product was purified by column chromatography (hexane: ethyl acetate. 95:5). Yield 60 mg (70%).

7-(bis(phenylthio)methyl)-1*H*-indole (28): Dark red semi-solid, Yield 60 mg (70%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.08 (brs, 1H), 7.55 (d, *J* = 7.64 Hz, 1H), 7.30-7.27 (m, 5H), 7.22-7.18 (m, 6H), 6.91-6.83 (m, 2H), 6.56 (t, *J* = 2.50 Hz, 1H), 5.67 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.0, 133.3, 132.8, 129.2, 128.9, 128.1, 124.3, 122.1, 121.3, 121.2, 119.2, 102.7, 59.9. HRMS-ES$^+$ *m/z* : 386.0434 (calculated for $C_{21}H_{17}NS_2 + K^+$: 386.0436).



1,2-bis(4-nitrophenyl)ethan-1-one (29): Light brown needles, mp 107 °C, Yield 57 mg (30%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.82 Hz, 2H), 8.19 (d, *J* = 8.71 Hz, 2H), 8.15 (d, *J* = 8.85 Hz, 2H), 7.42 (d, *J* = 8.71 Hz, 2H), 4.46 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.5, 150.6, 147.3, 140.7, 140.5, 130.7, 129.4, 124.1, 123.9, 45.4.

S1 ^{1}H & ^{13}C NMR of **1**



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9	Temperature	298.1
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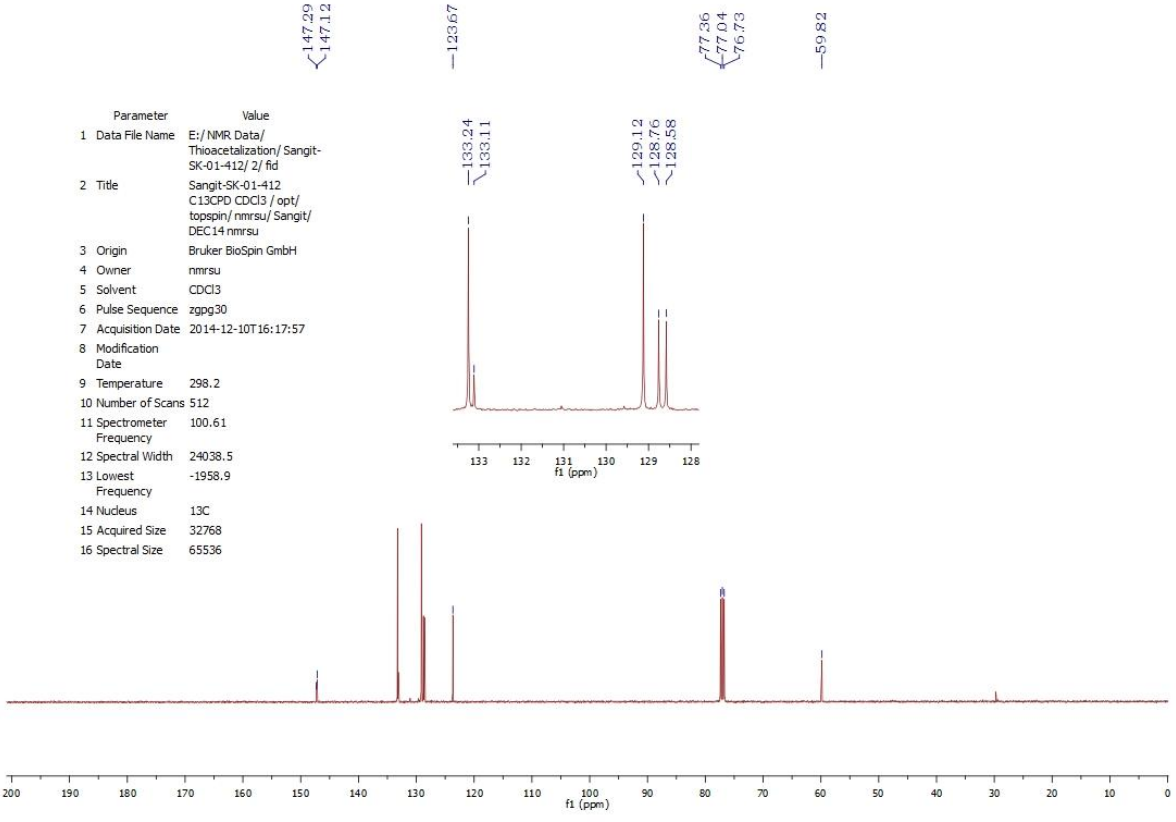
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



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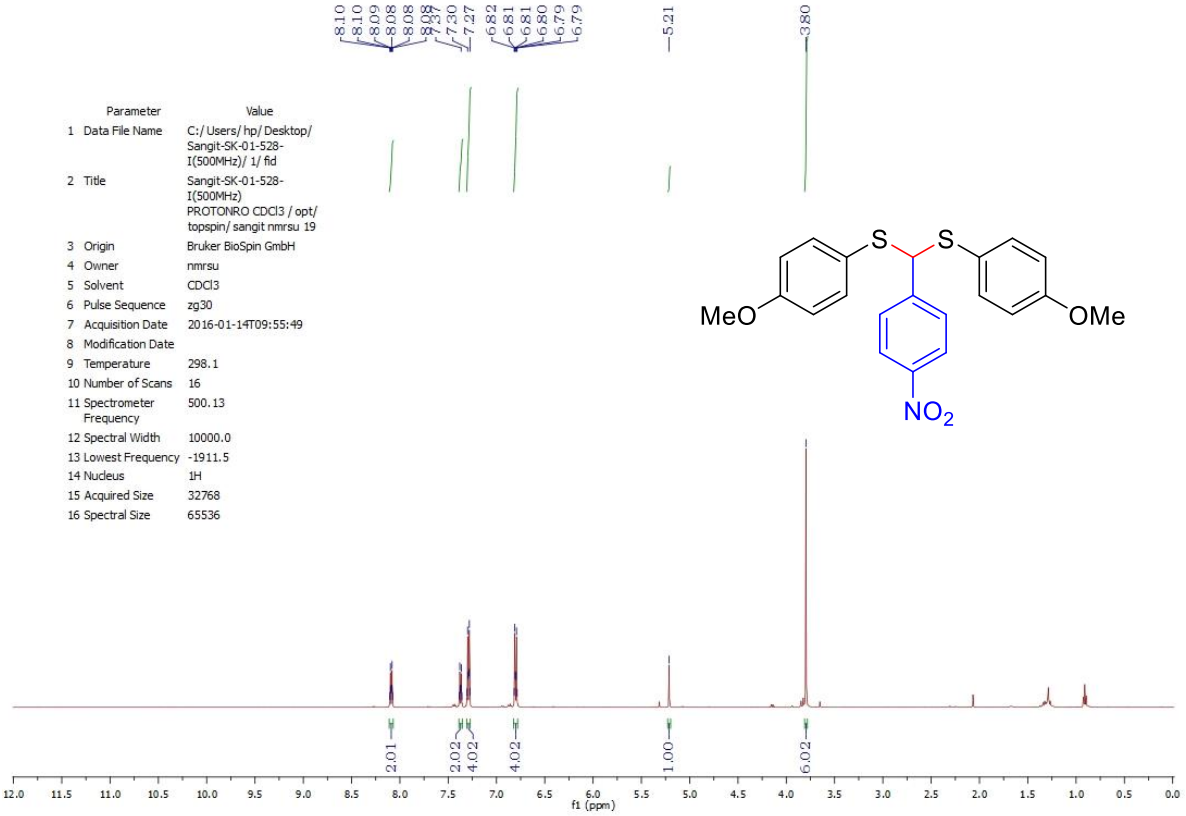
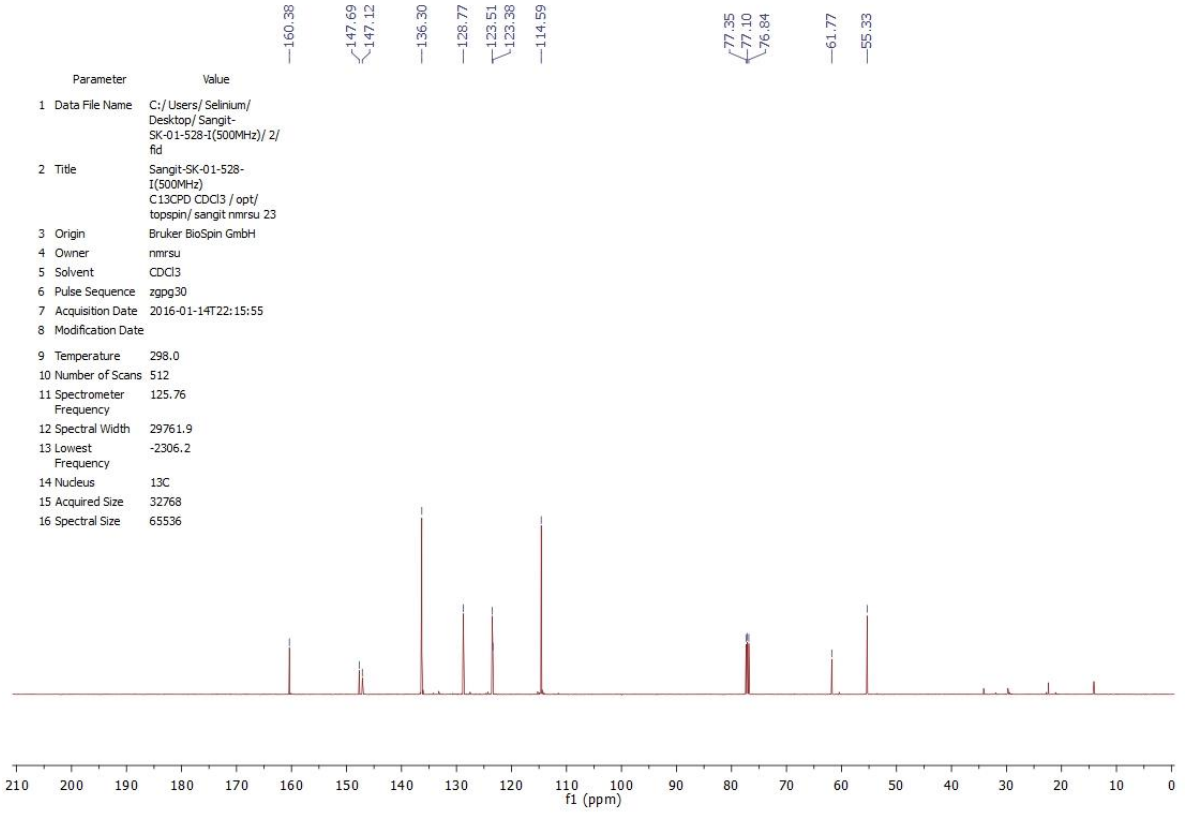
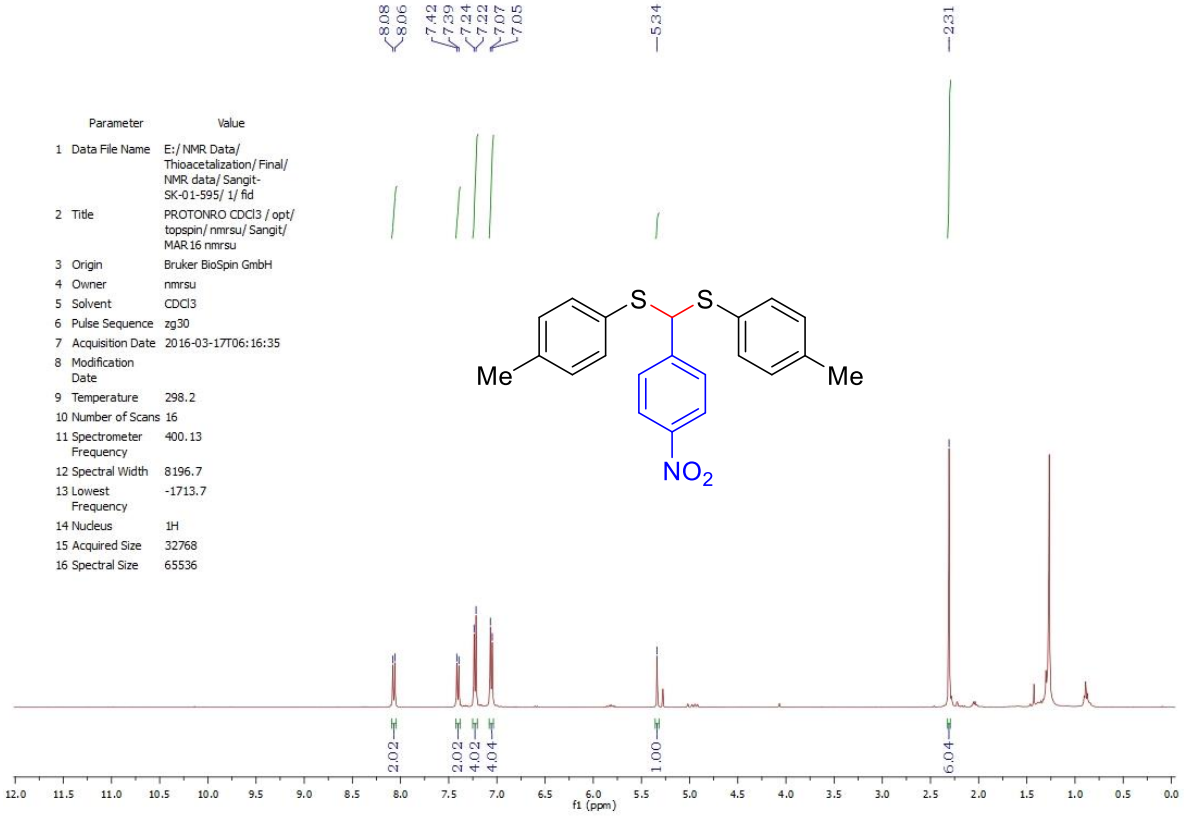
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S2 ^{1}H & ^{13}C NMR of **2**





S3 ^{1}H & ^{13}C NMR of **3**



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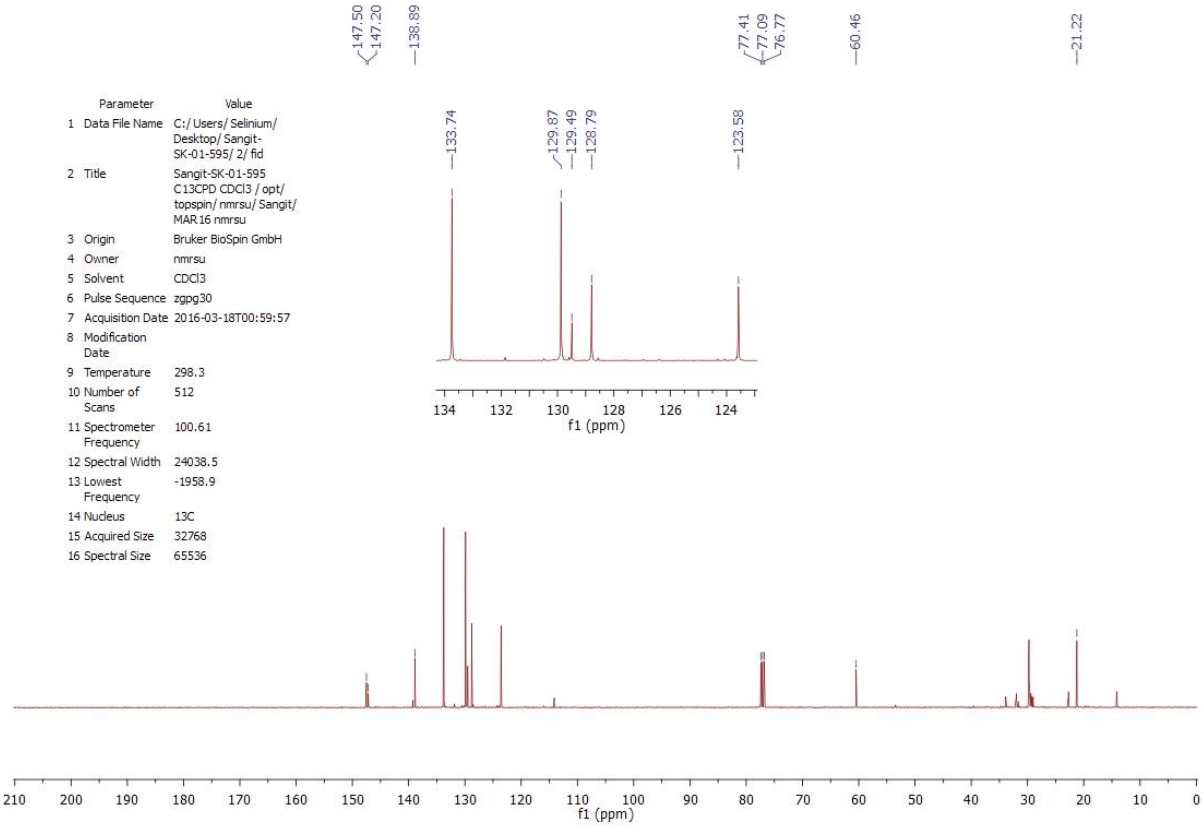

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12	Spectral Width	24038.5
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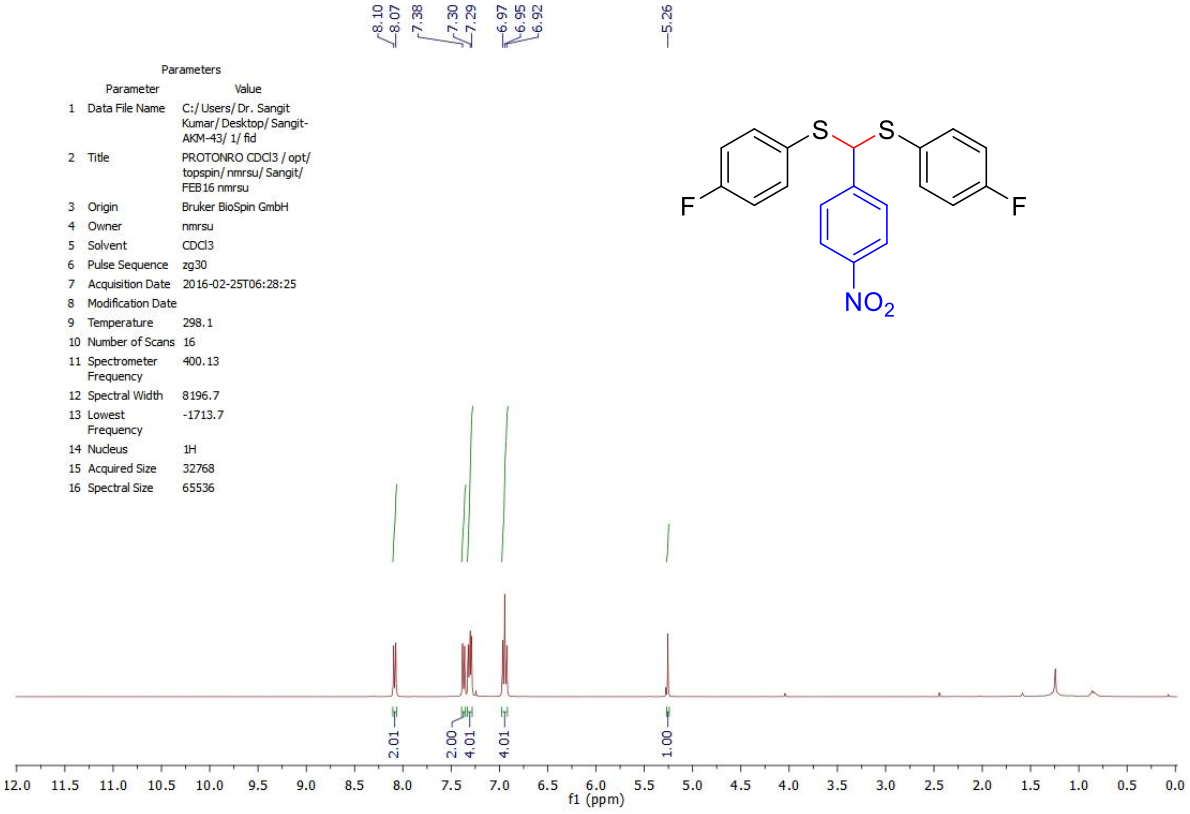
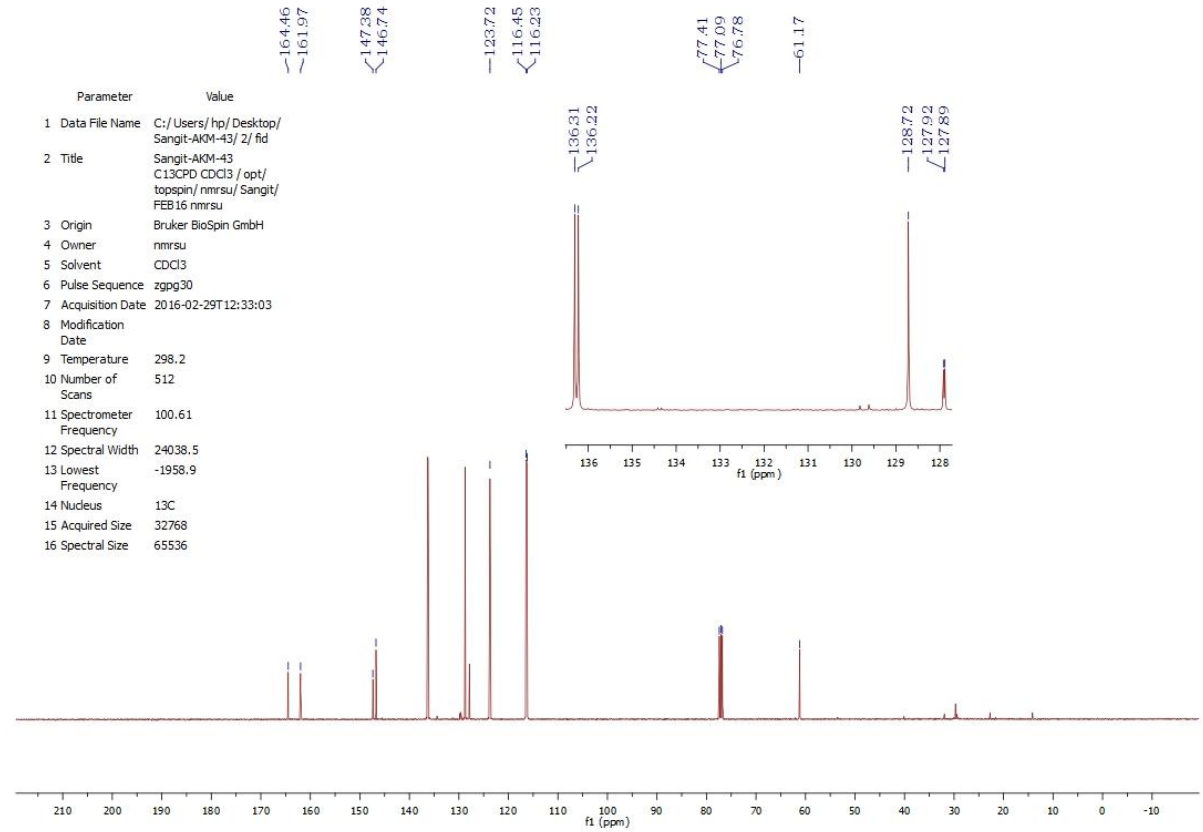
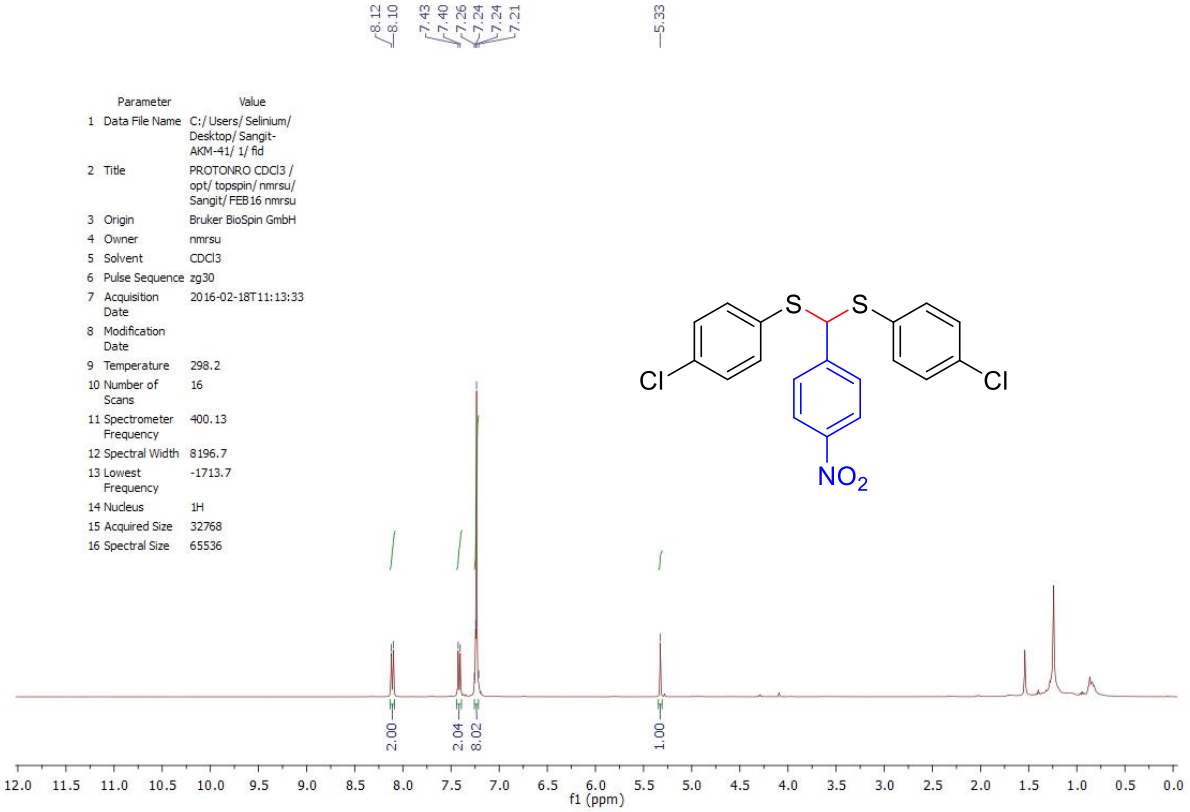
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

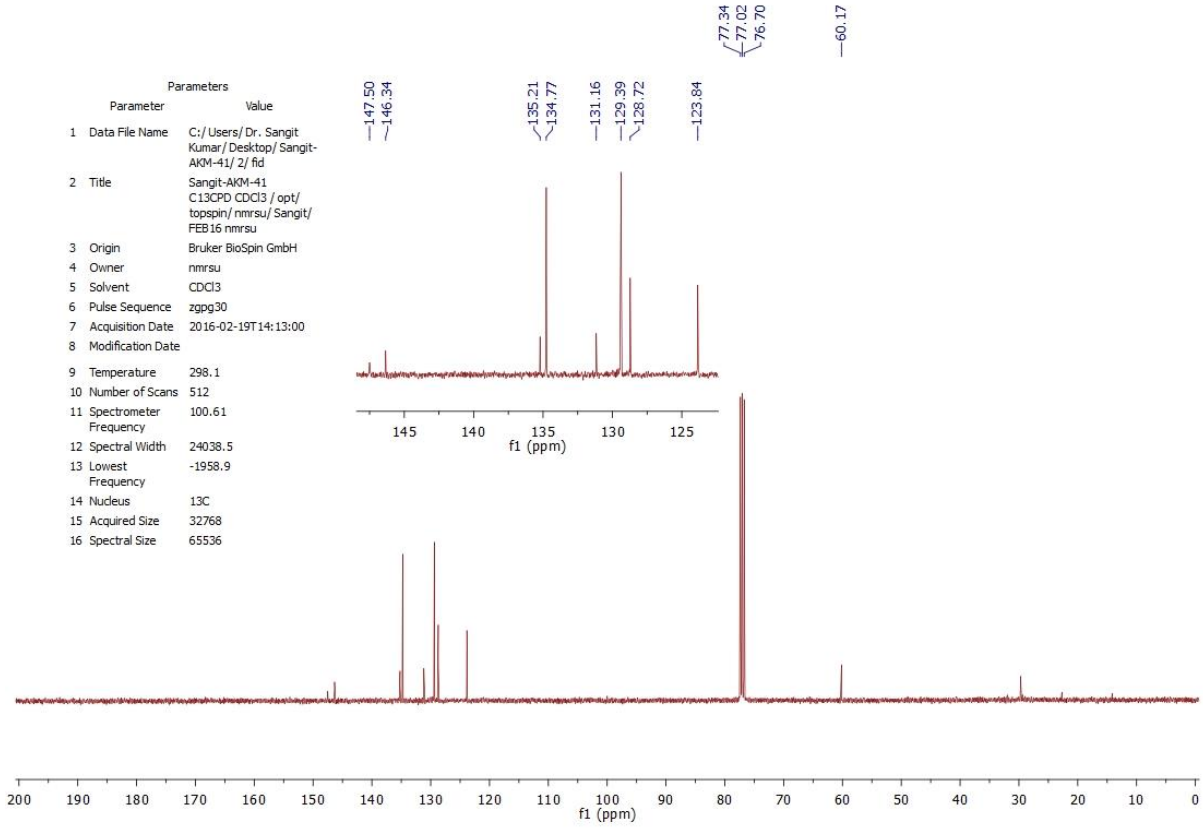
S4 ^{1}H & ^{13}C NMR of **4**



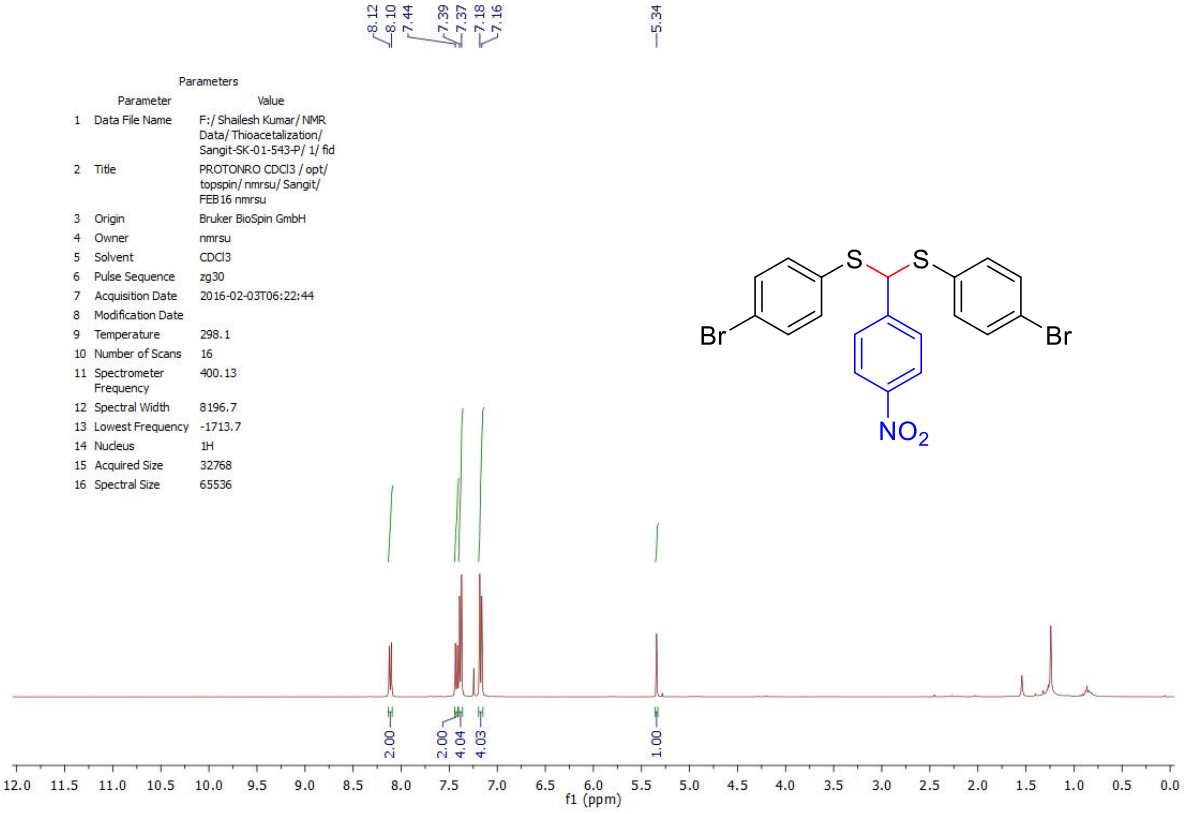


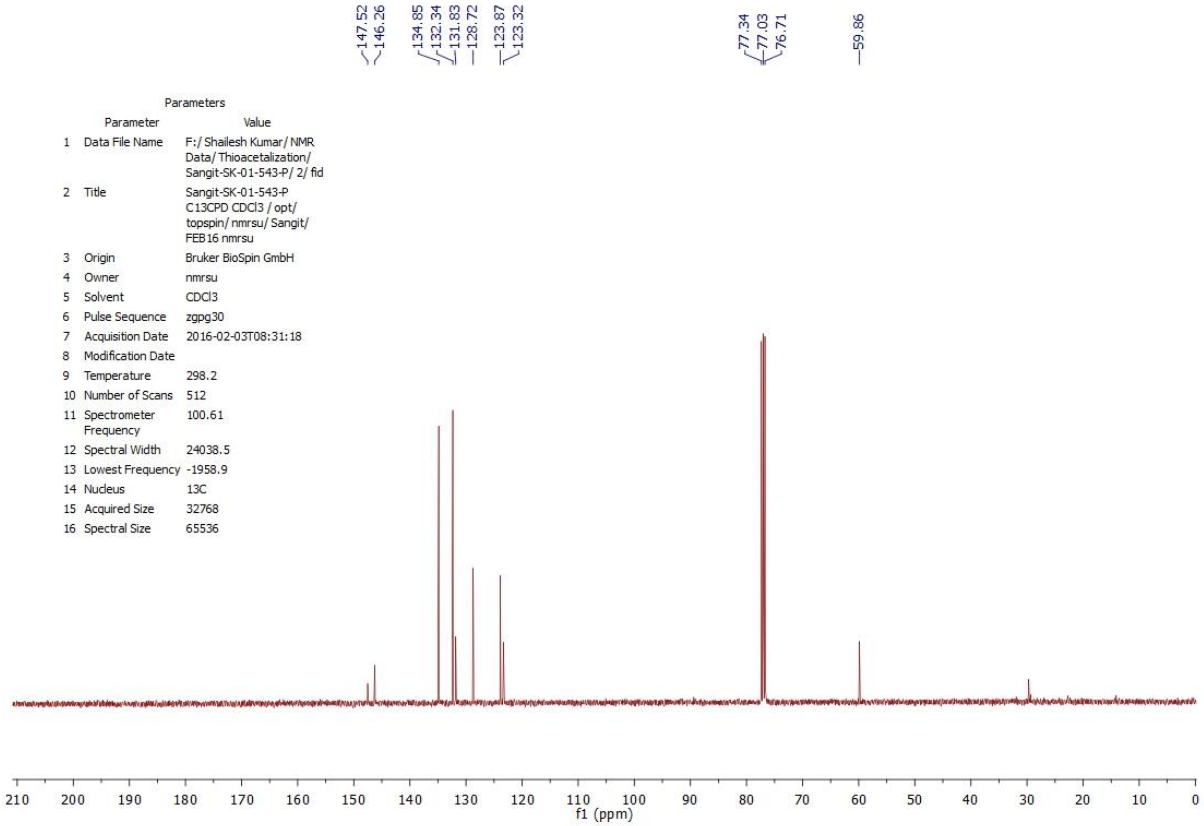
S5 ^{1}H & ^{13}C NMR of **5**



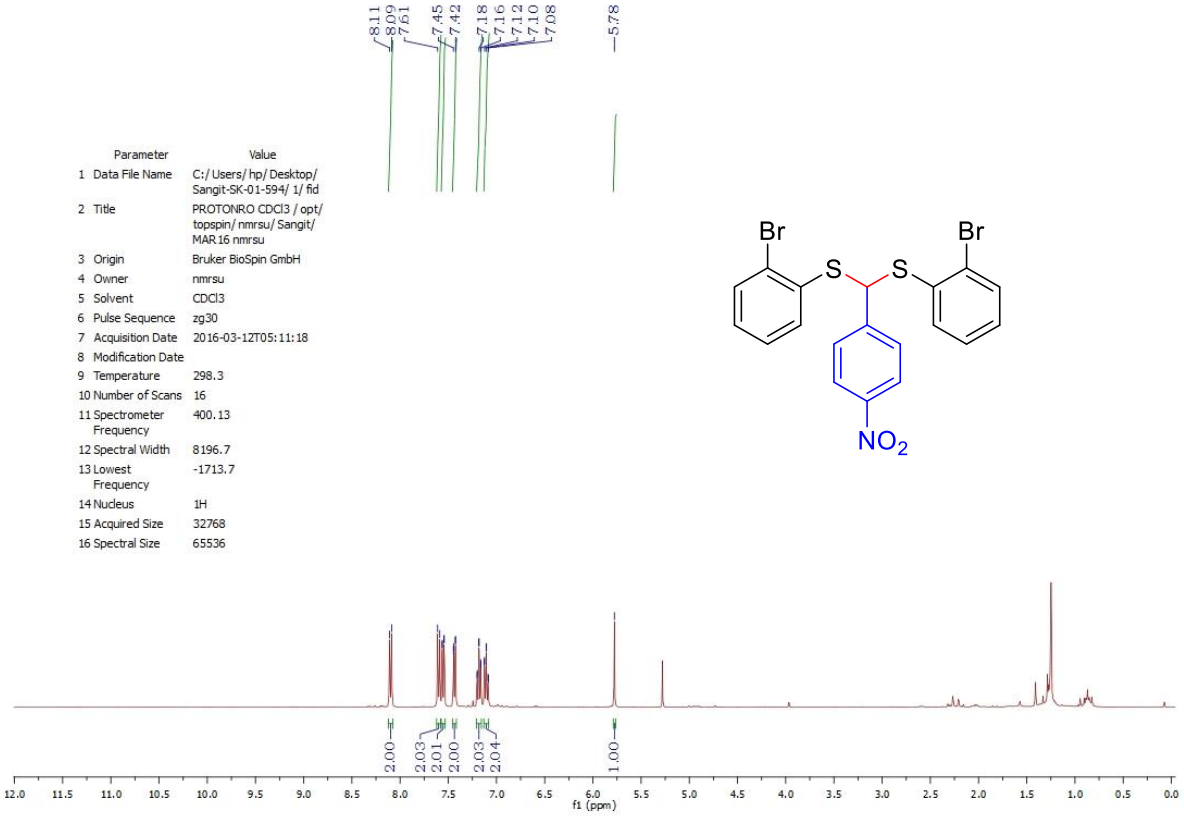


S6 ^{1}H & ^{13}C NMR of **6**





S7 ^{1}H & ^{13}C NMR of **7**



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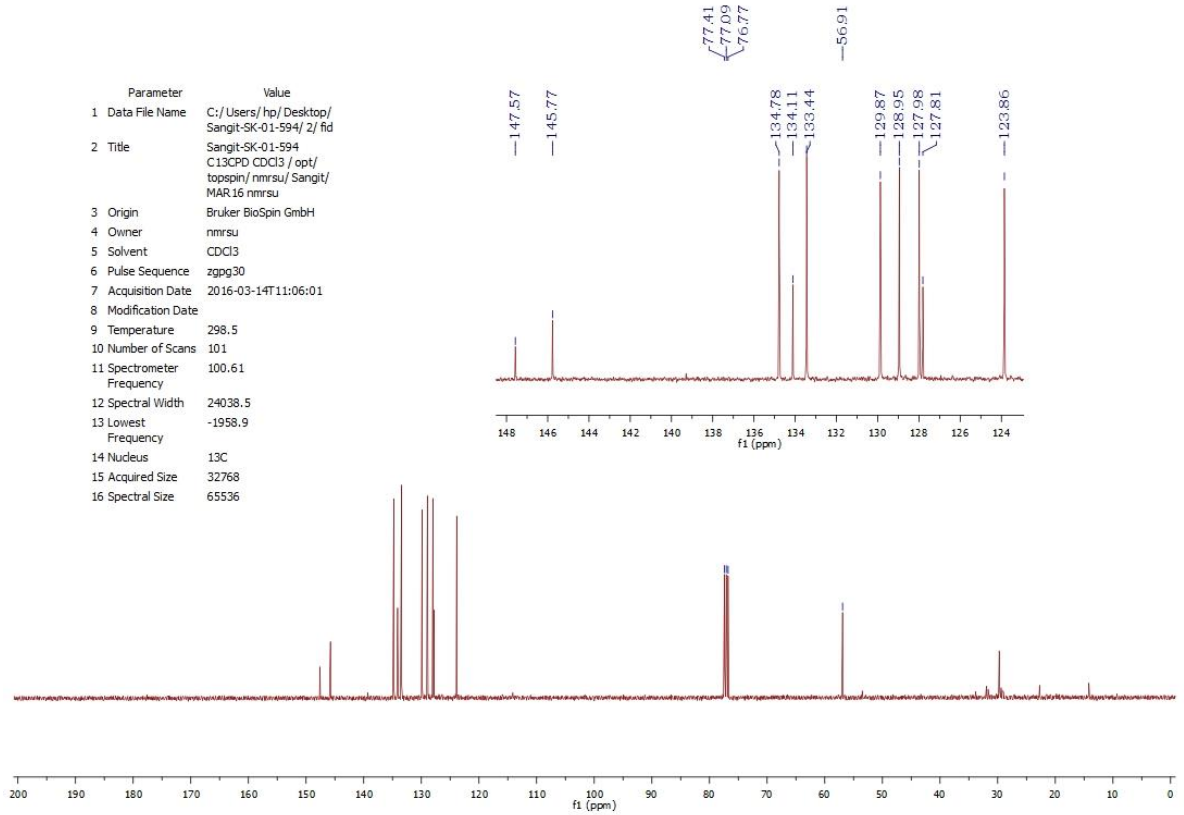

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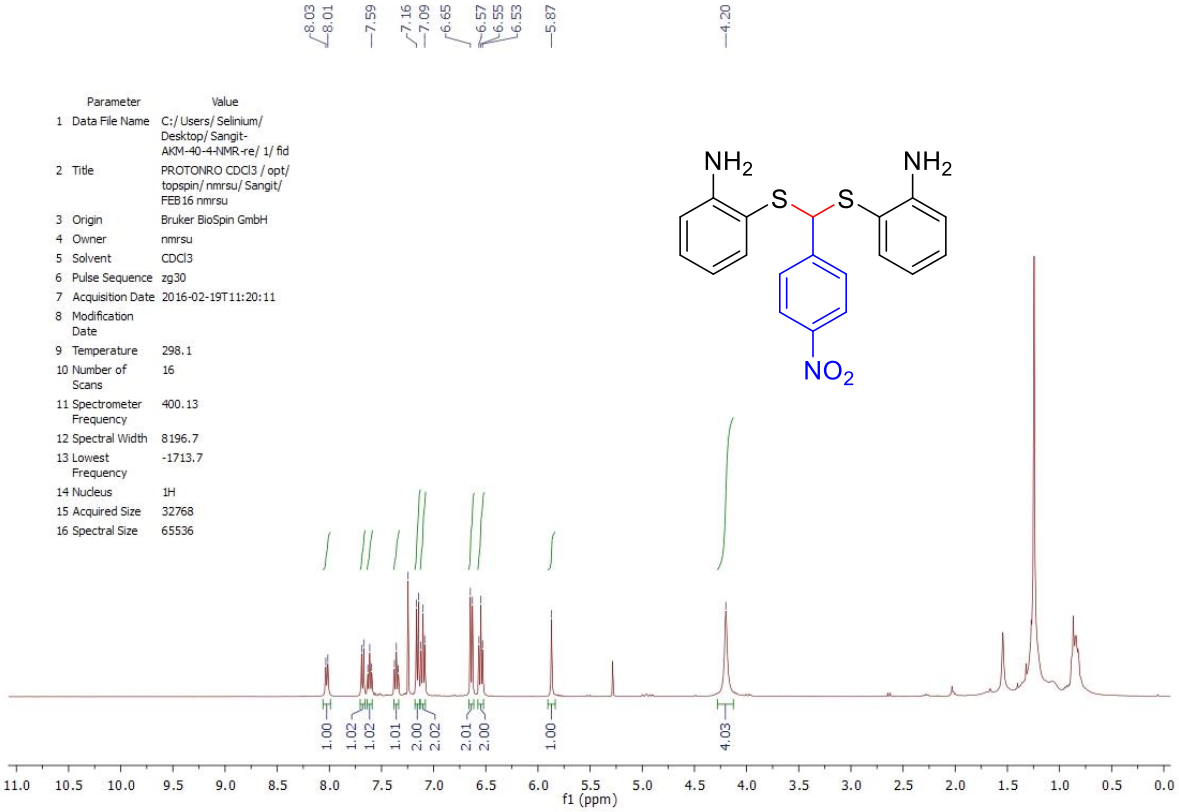
15 Acquired Size	32768
16 Spectral Size	65536

S8 ^{1}H & ^{13}C NMR of **8**



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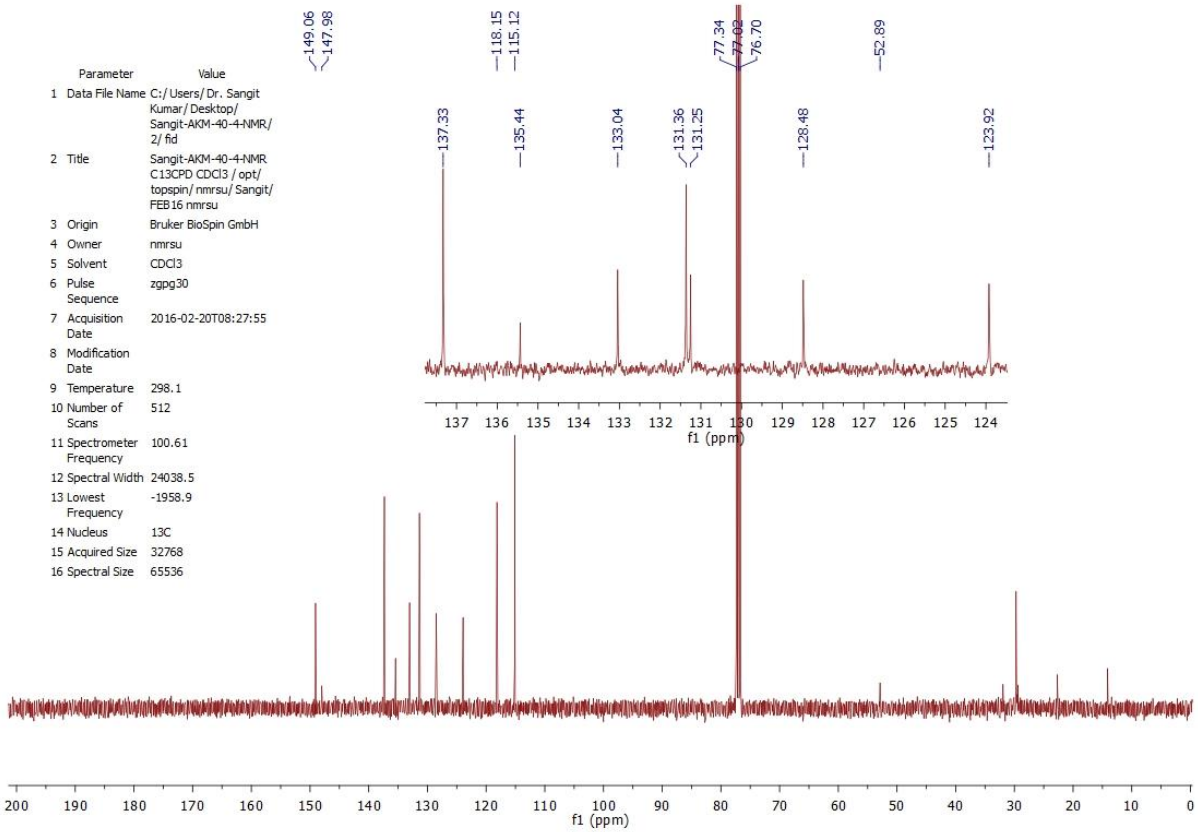
15	Acquired Size	32768
16	Spectral Size	65536



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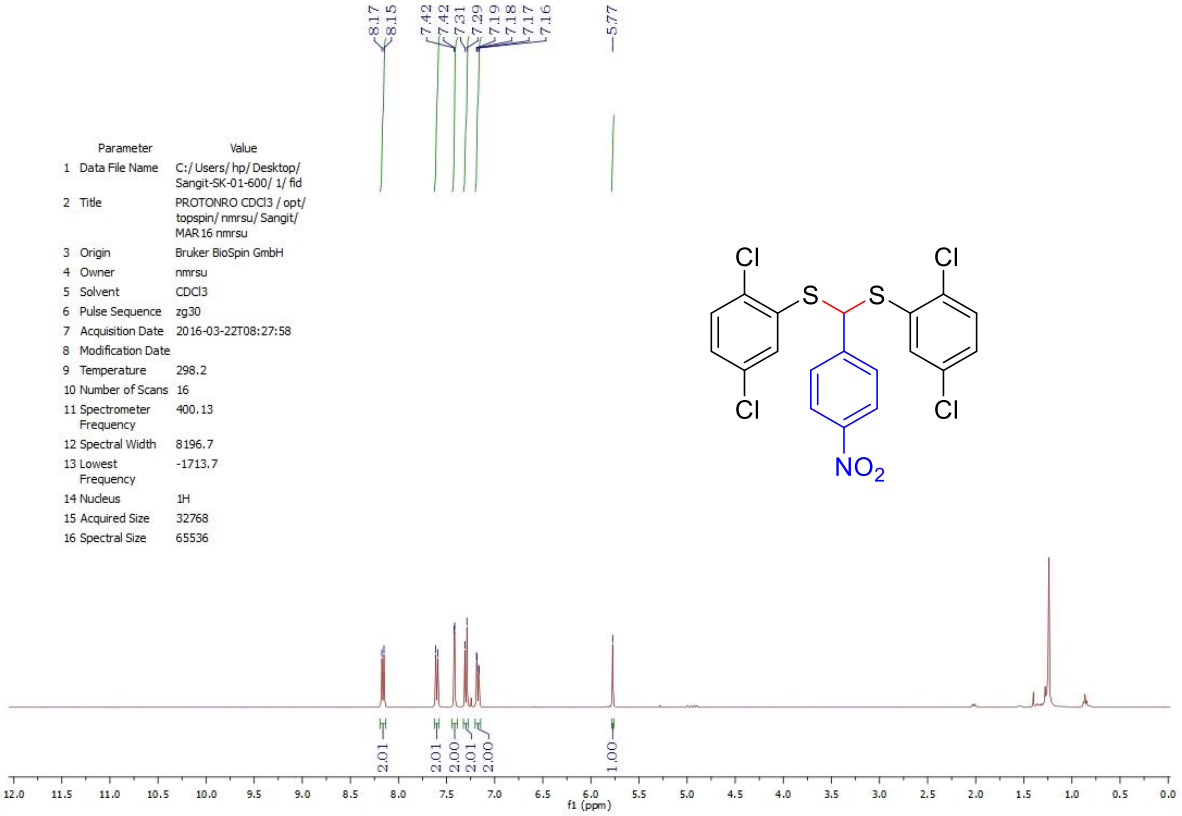
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2016-02-20T08:27:55
8	Modification Date	
9	Temperature	298.1
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S9 ^{1}H & ^{13}C NMR of **9**



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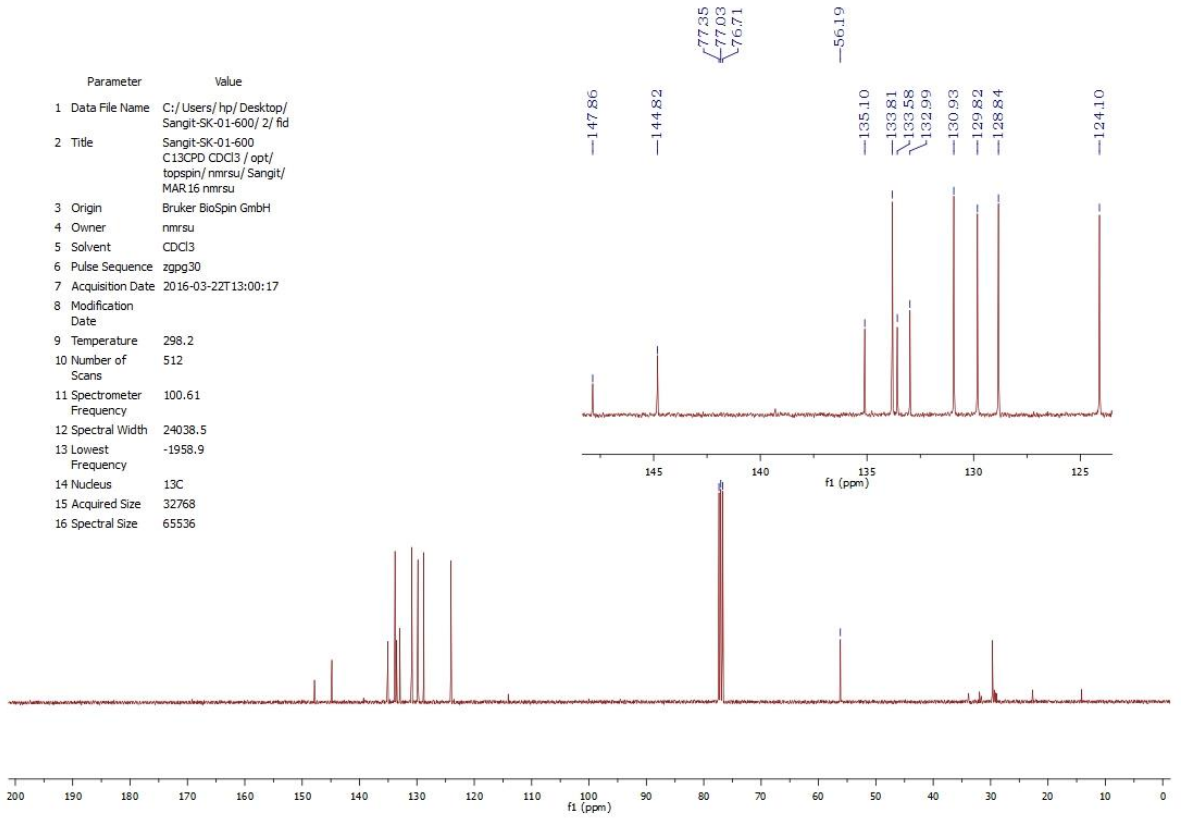
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
7	Acquisition Date	2016-03-22T08:27:58
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



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4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
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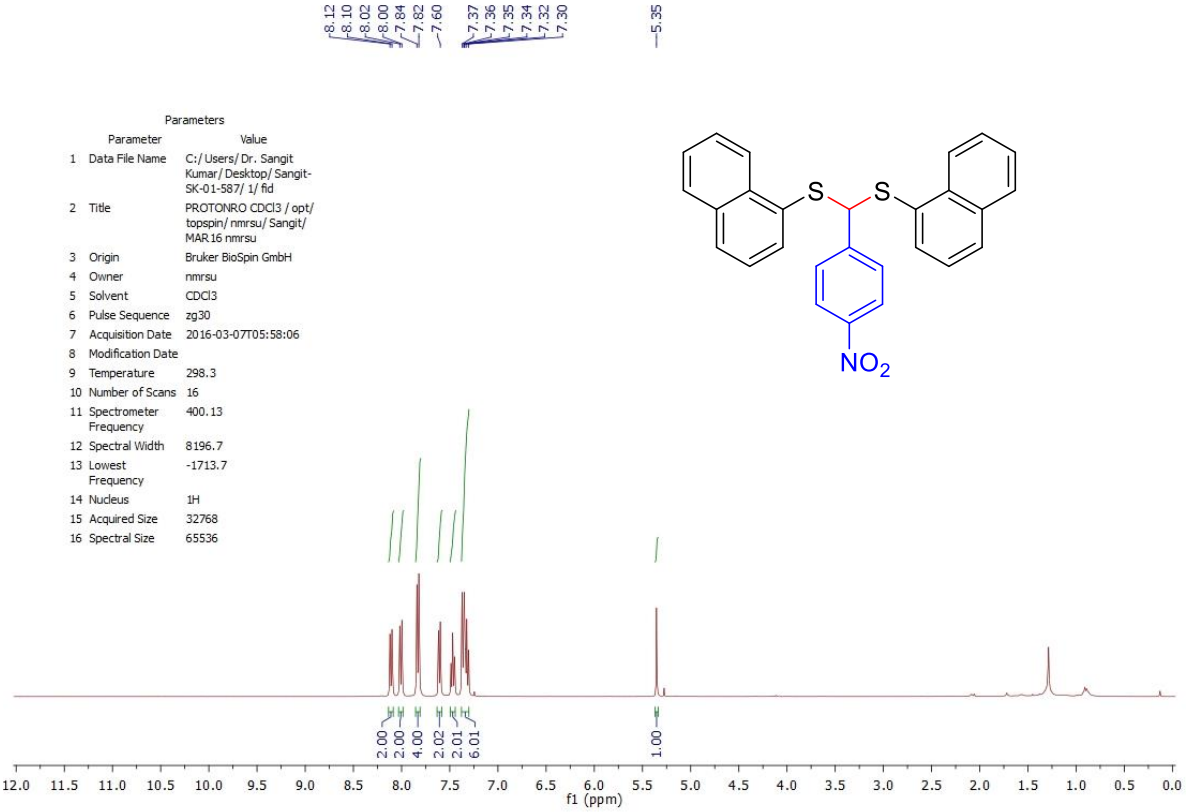
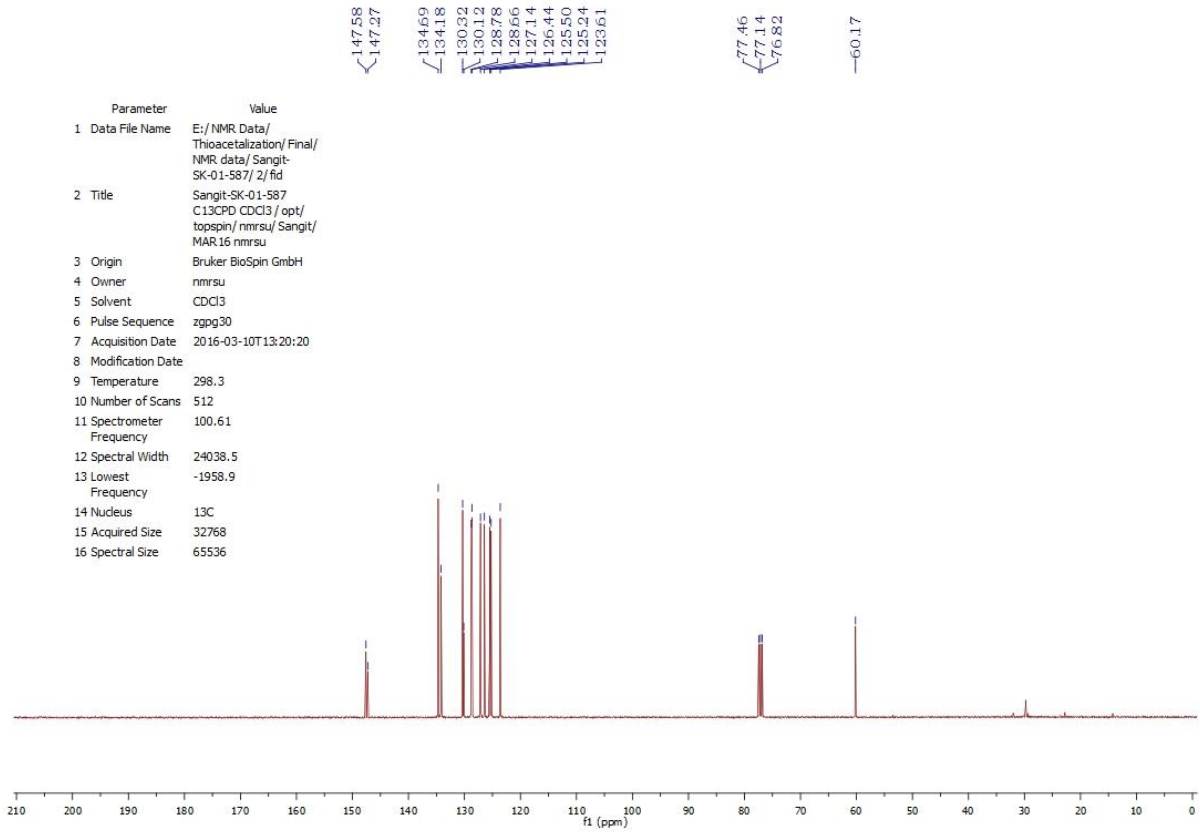
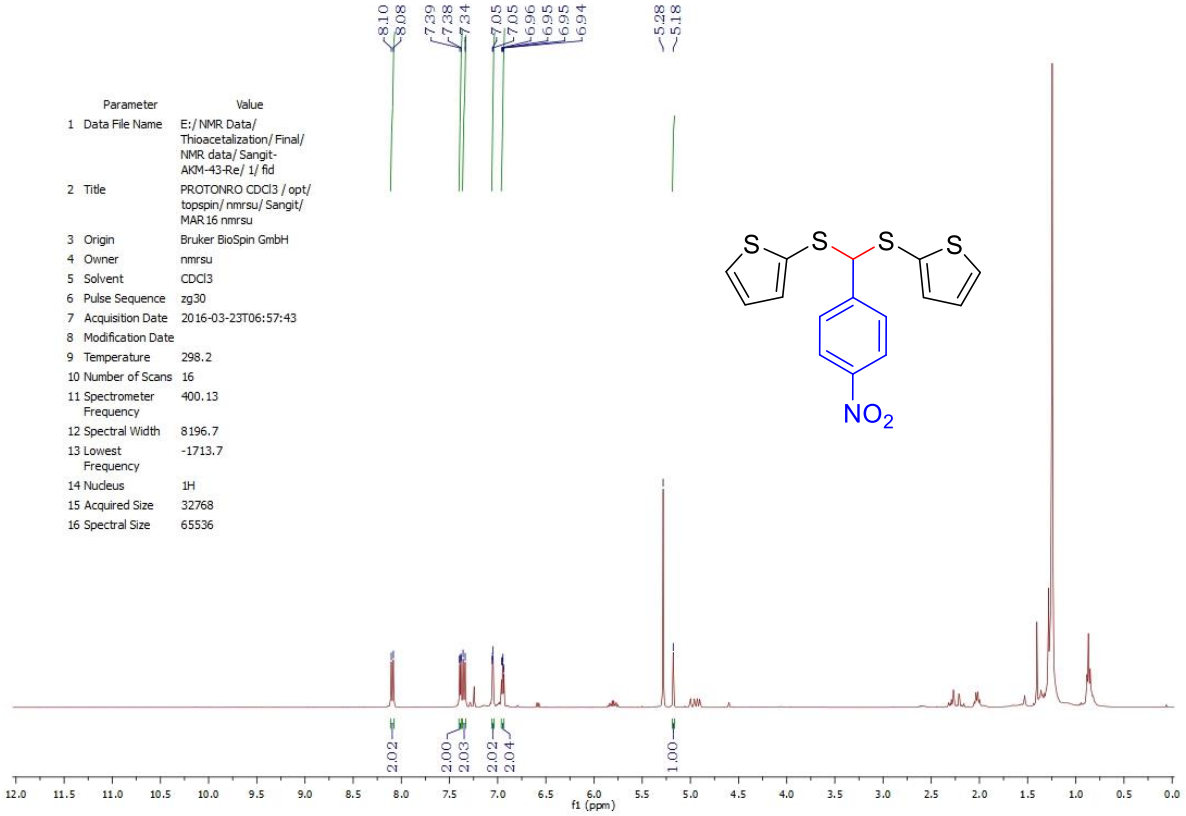
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S10 ^{1}H & ^{13}C NMR of **10**





S11 ^{1}H & ^{13}C NMR of **11**



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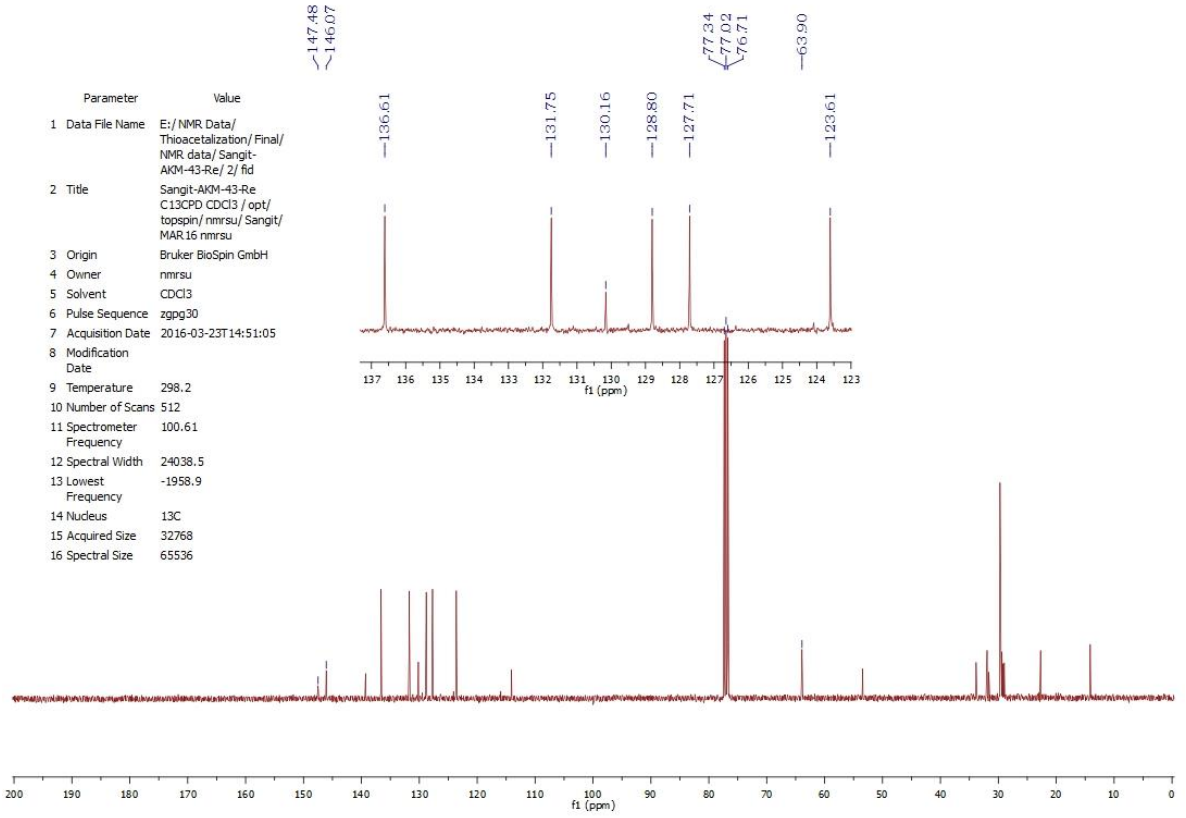
2	Title	PROTONRO CDCl3 / opt/ topspin/ nmrsu/ Sangit/ MAR16 nmrsu
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
7	Acquisition Date	2016-03-23T06:57:43
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



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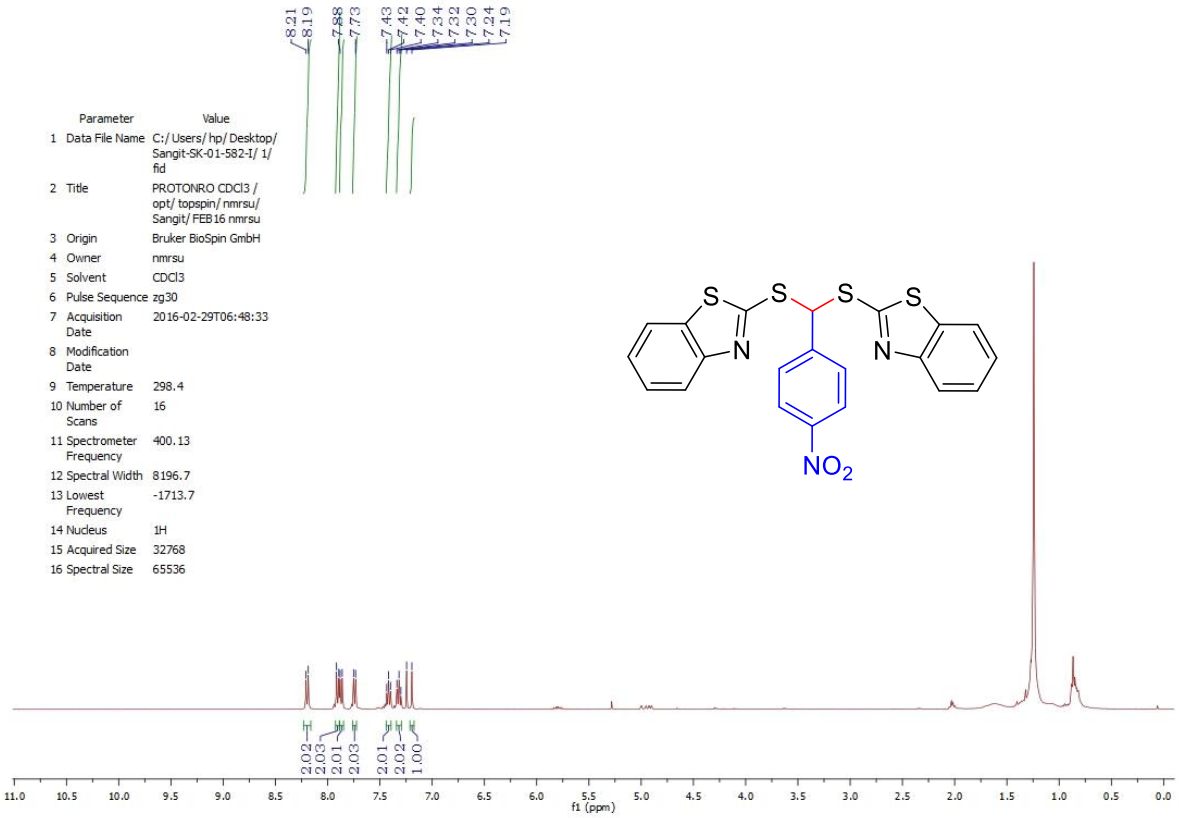
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2016-03-23T14:51:05
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S12 ^{1}H & ^{13}C NMR of **12**



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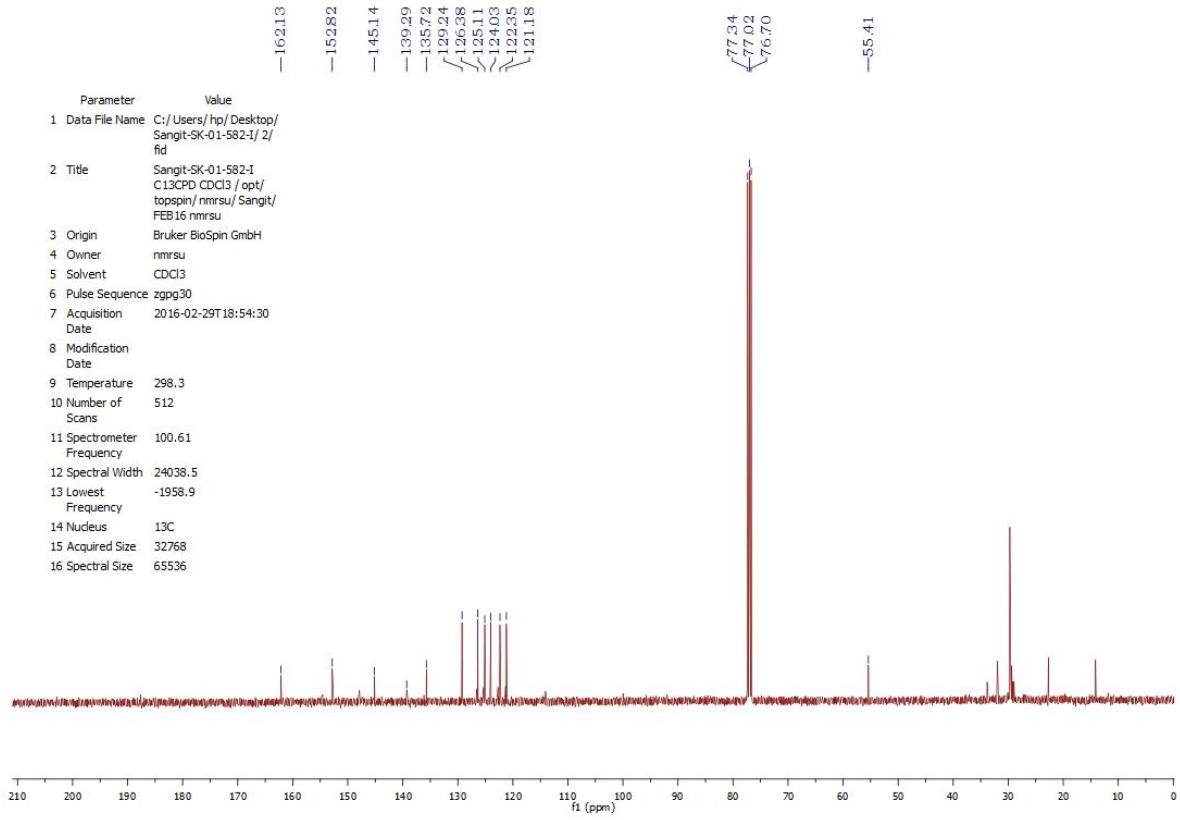
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
7	Acquisition Date	2016-02-29T06:48:33
8	Modification Date	
9	Temperature	298.4
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



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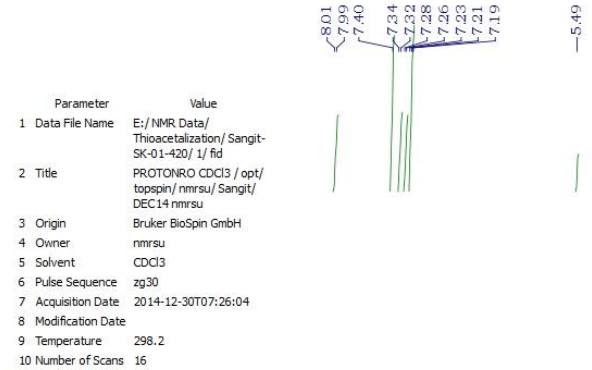
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2016-02-29T18:54:30
8	Modification Date	
9	Temperature	298.3
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S13 ^{1}H & ^{13}C NMR of **13**



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3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
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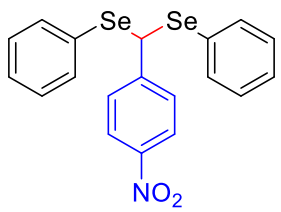
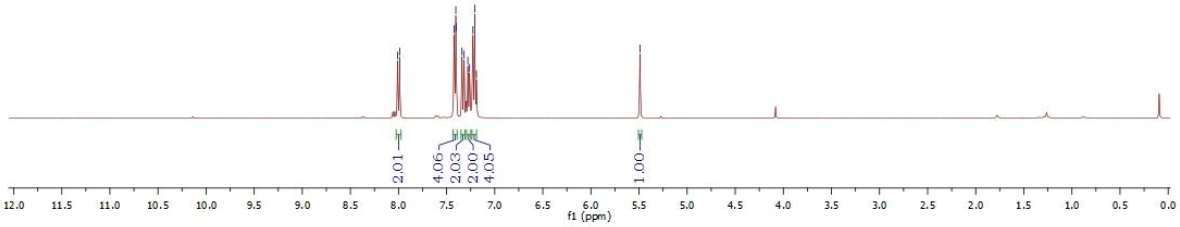
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536





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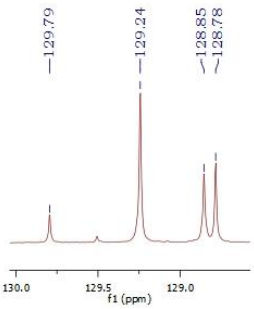
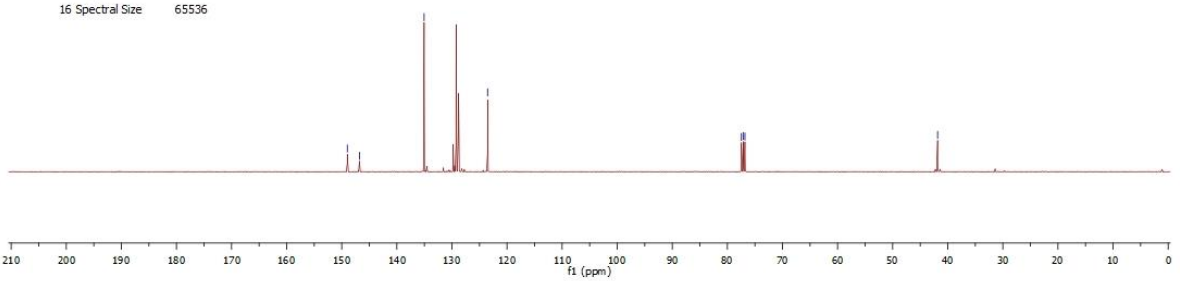
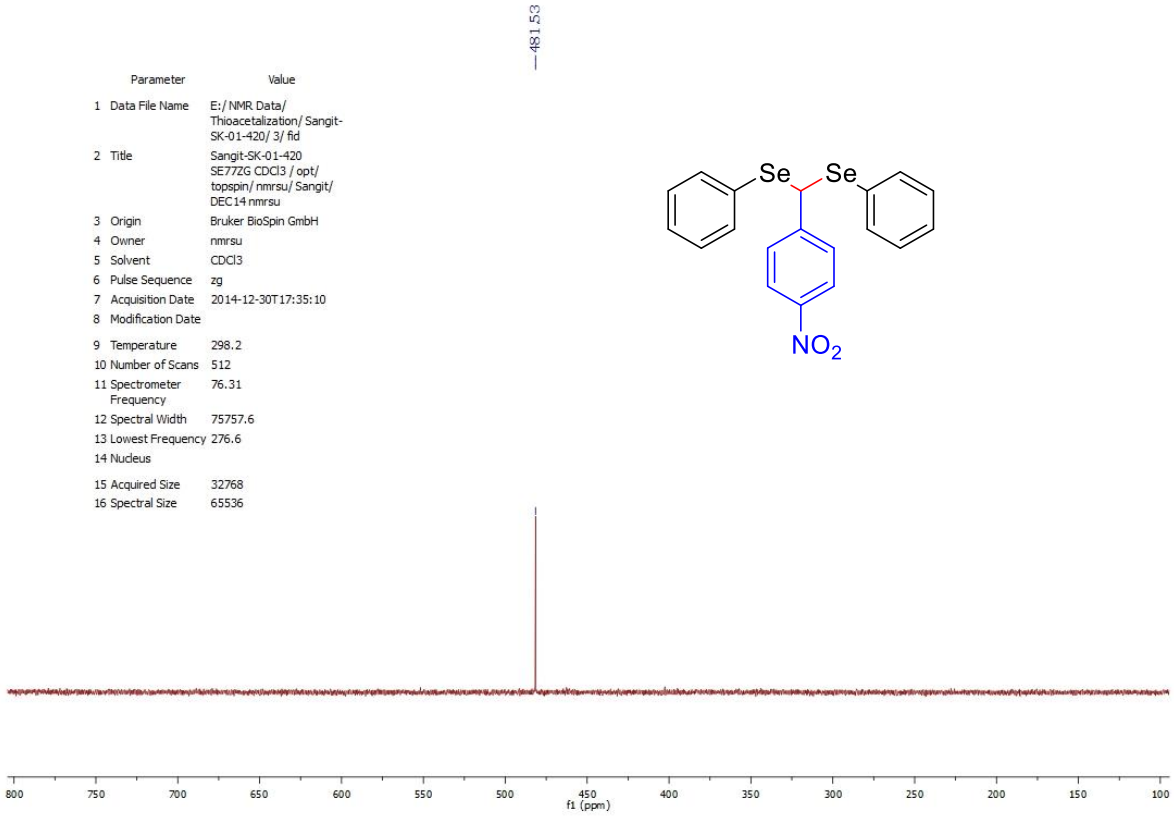
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2014-12-30T17:11:55
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	300
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

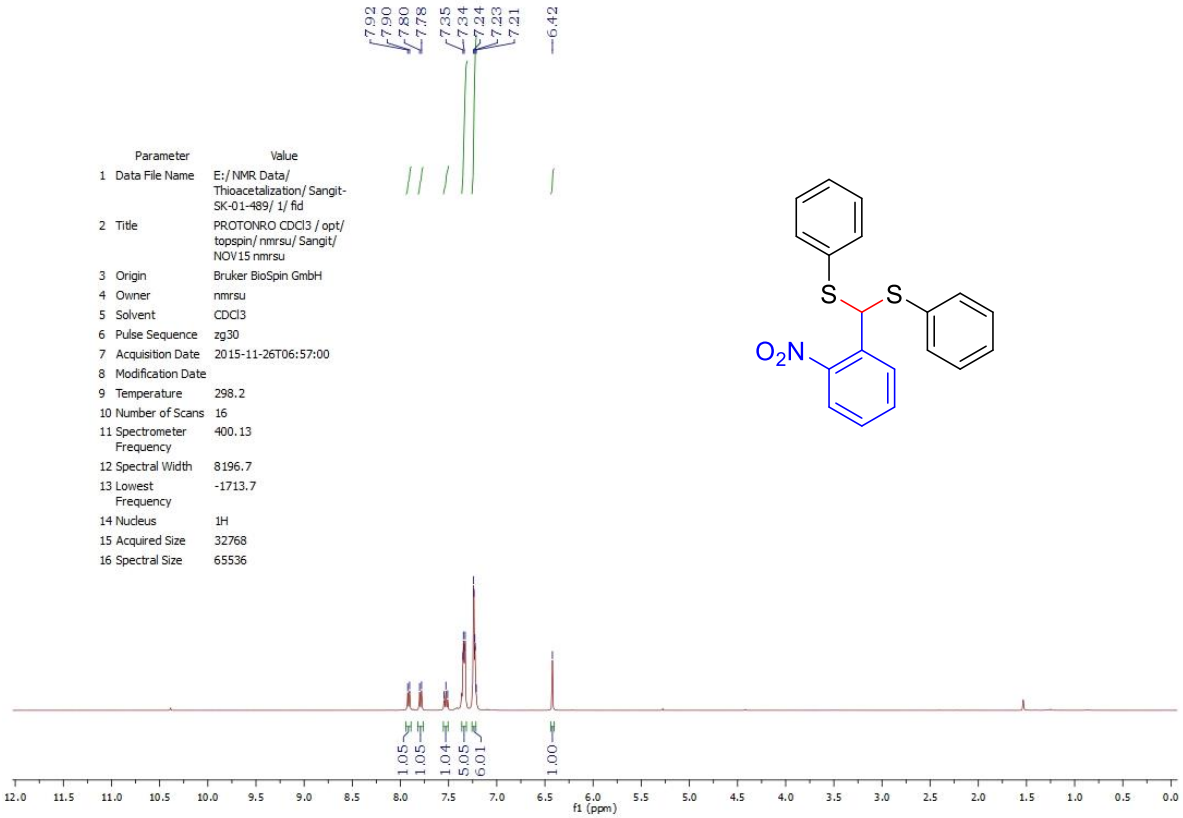


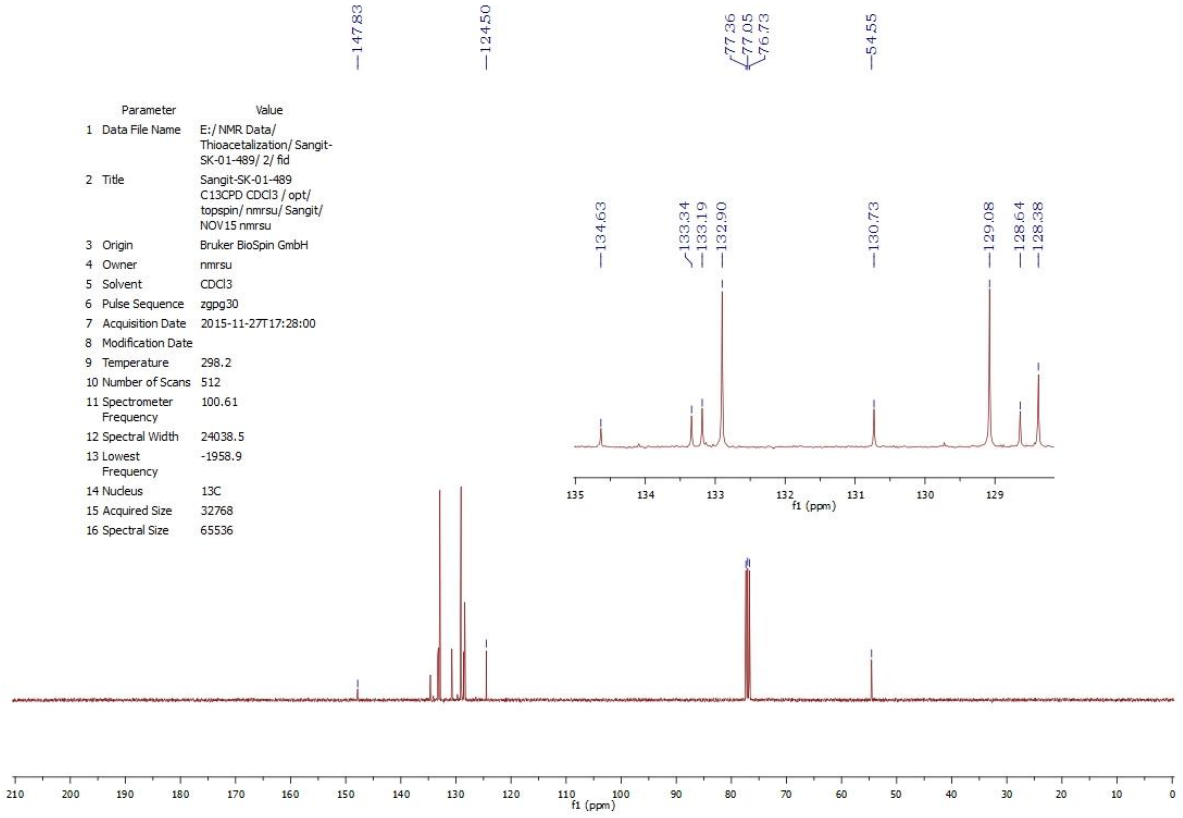


S14 ^{77}Se NMR of **13**

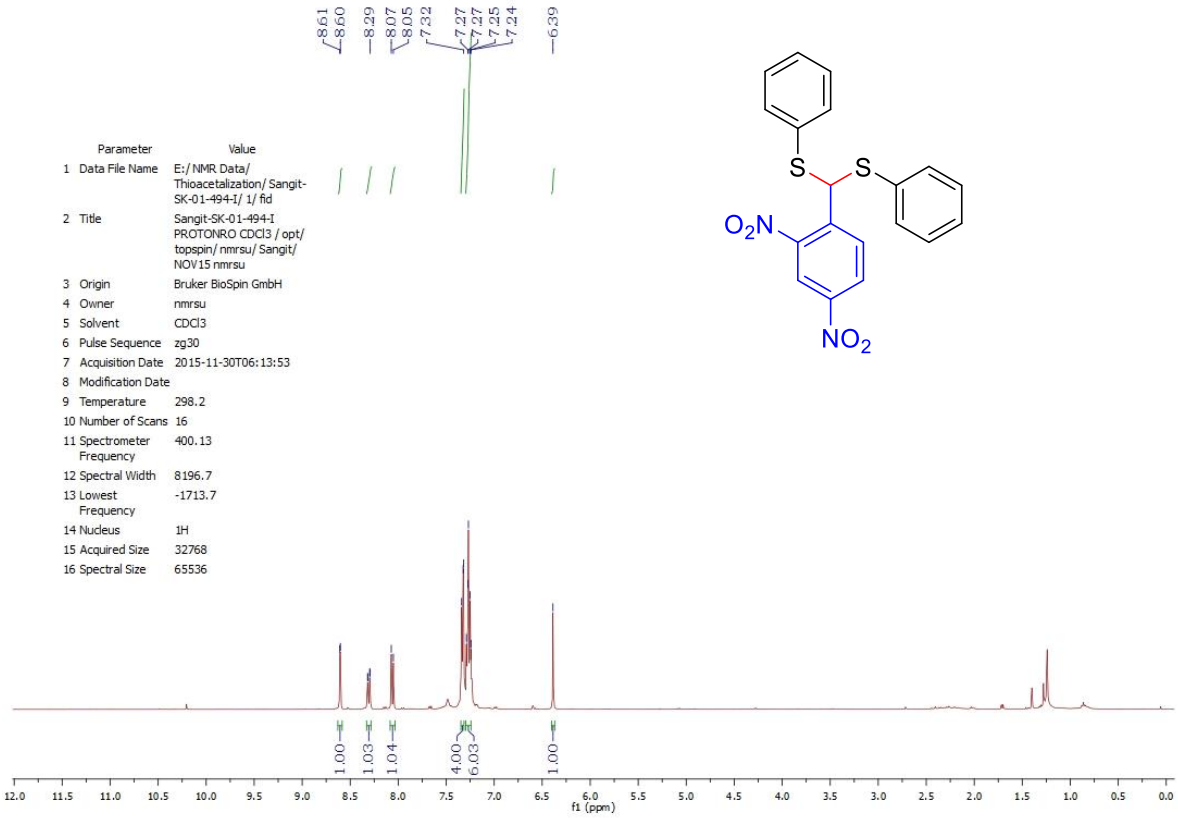


S15 ^{1}H & ^{13}C NMR of **14**





S16 ^{1}H & ^{13}C NMR of **15**



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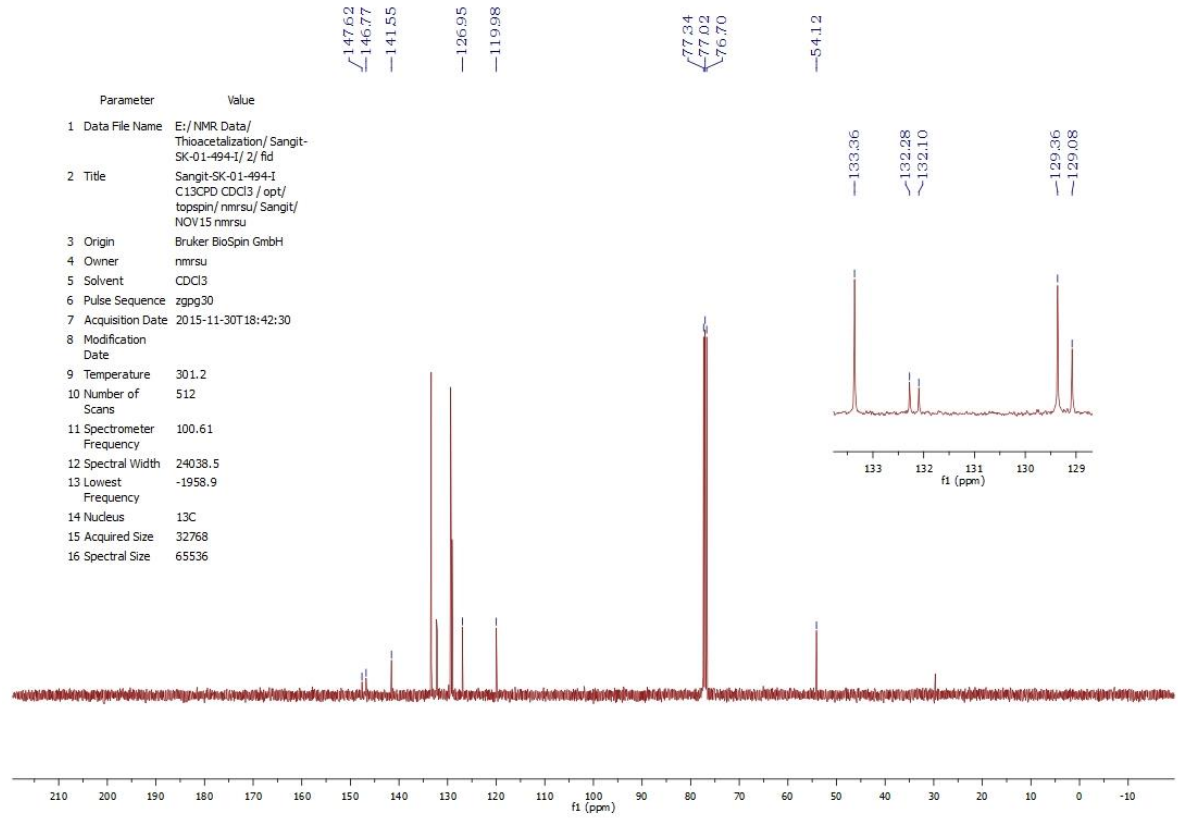
3 Origin	Bruker BioSpin GmbH
4 Owner	nmrsu
5 Solvent	CDCl3
6 Pulse Sequence	zg30
7 Acquisition Date	2015-11-30T06:13:53
8 Modification Date	
9 Temperature	298.2
10 Number of Scans	16
11 Spectrometer Frequency	400.13
12 Spectral Width	8196.7
13 Lowest Frequency	-1713.7
14 Nucleus	1H
15 Acquired Size	32768
16 Spectral Size	65536



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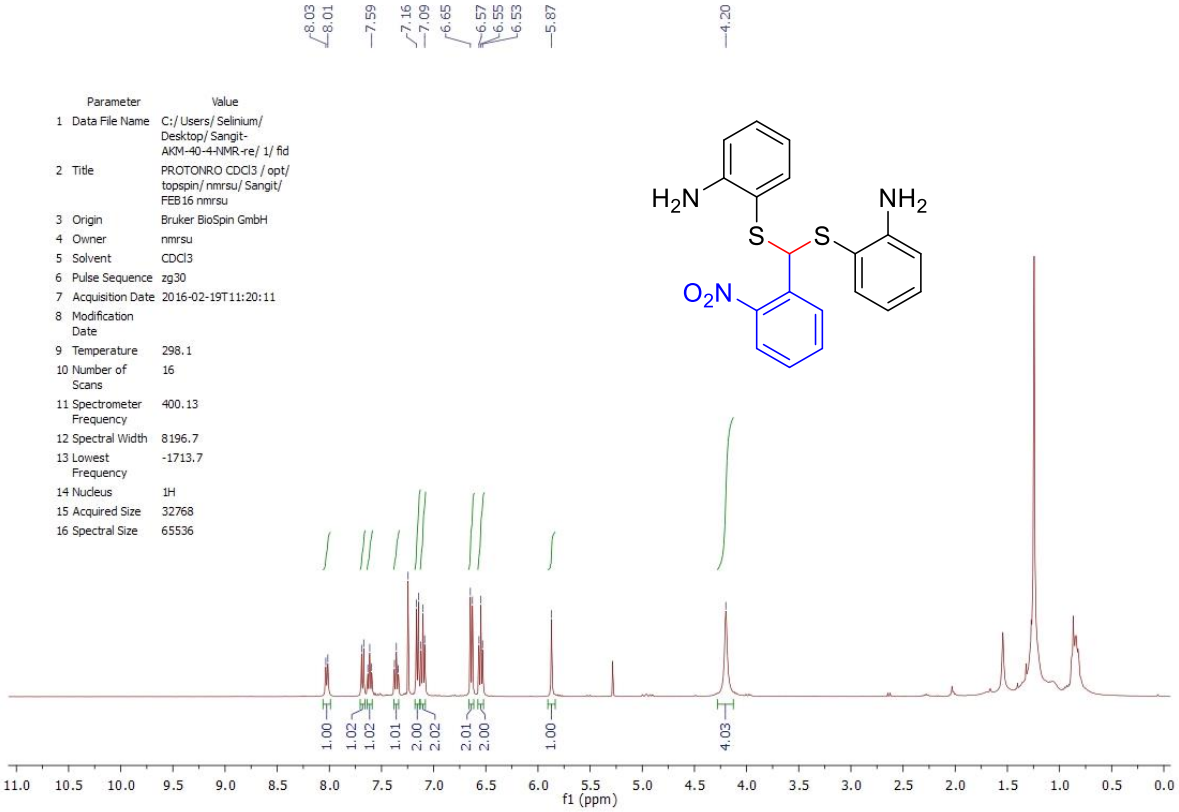
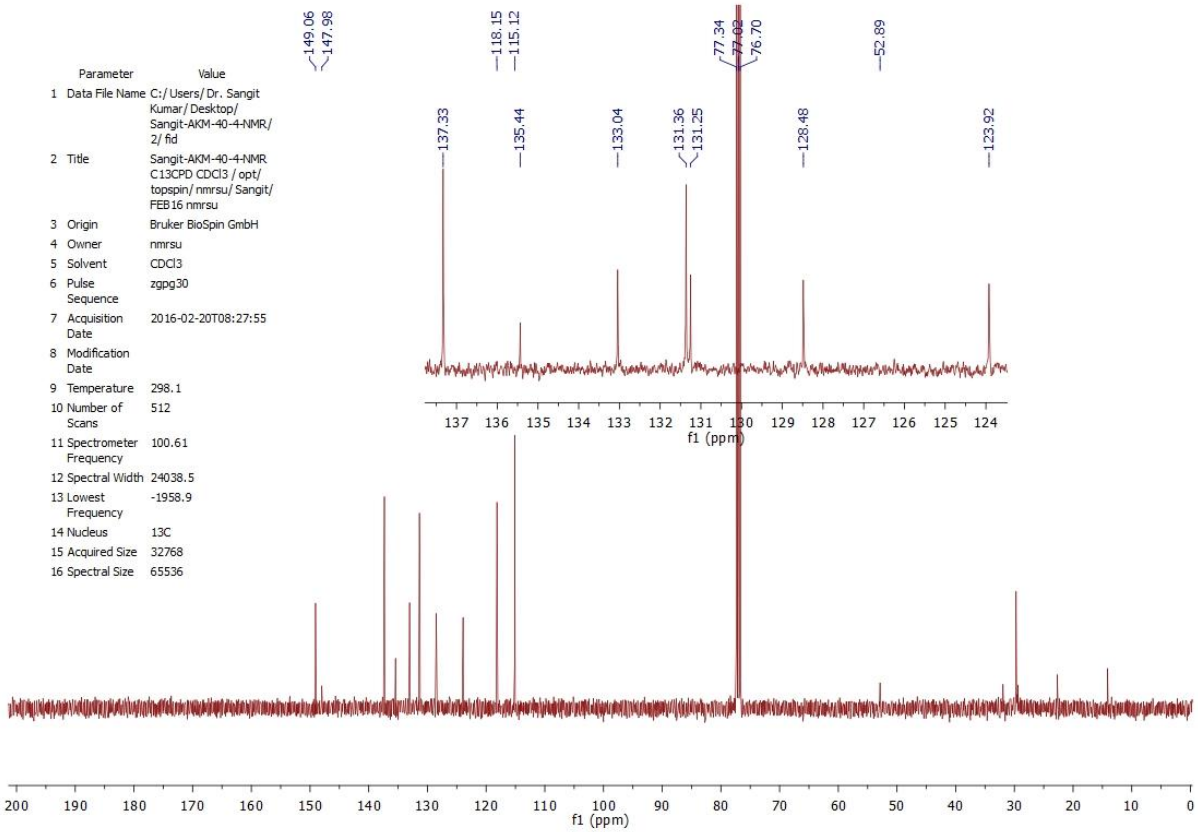
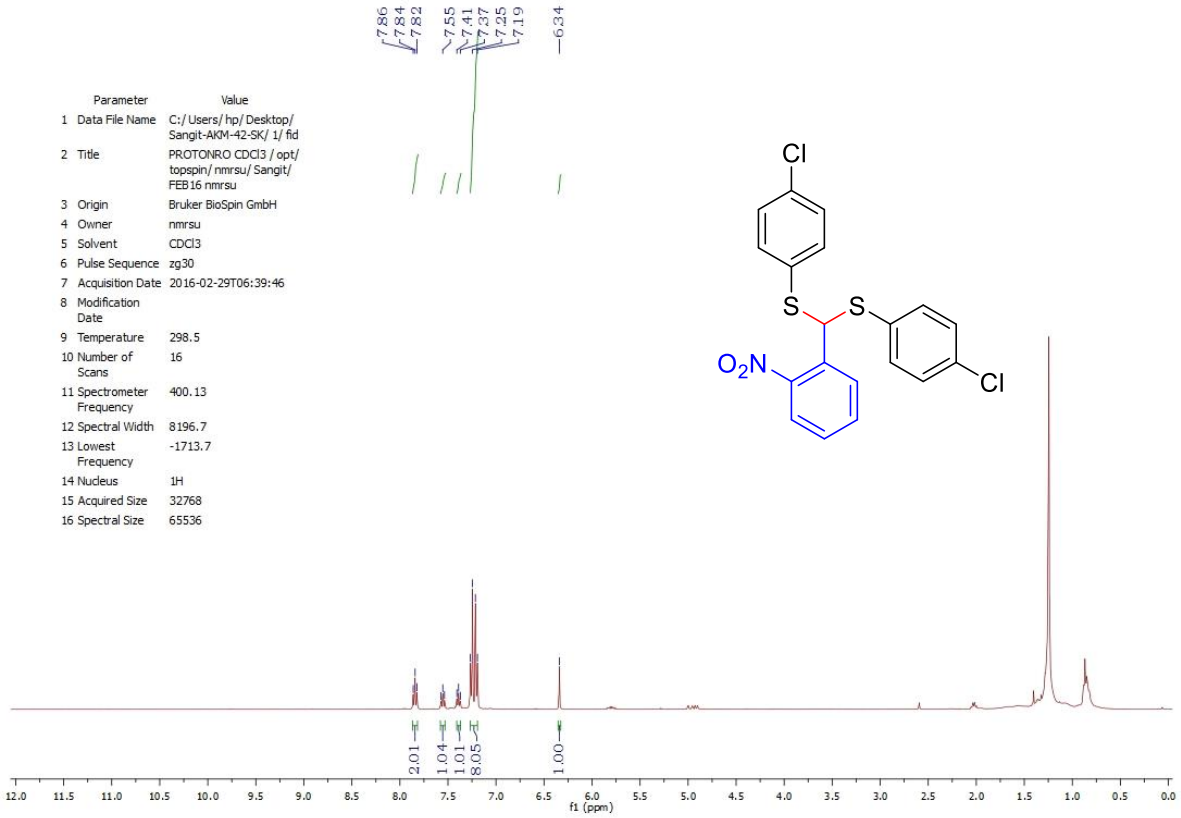
3 Origin	Bruker BioSpin GmbH
4 Owner	nmrsu
5 Solvent	CDCl3
6 Pulse Sequence	zgpg30
7 Acquisition Date	2015-11-30T18:42:30
8 Modification Date	
9 Temperature	301.2
10 Number of Scans	512
11 Spectrometer Frequency	100.61
12 Spectral Width	24038.5
13 Lowest Frequency	-1958.9
14 Nucleus	13C
15 Acquired Size	32768
16 Spectral Size	65536

S17 ^{1}H & ^{13}C NMR of **16**





S18 ^{1}H & ^{13}C NMR of **17**



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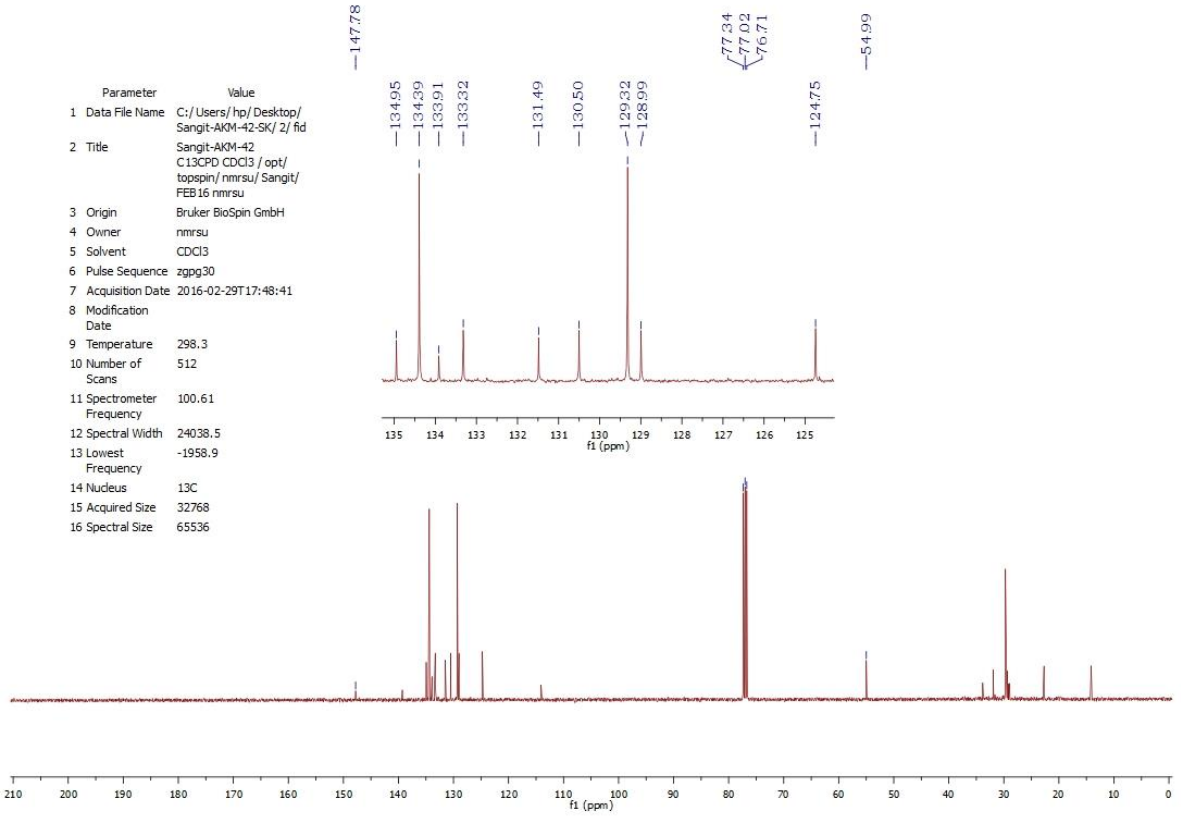
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
7	Acquisition Date	2016-02-29T06:39:46
8	Modification Date	
9	Temperature	298.5
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



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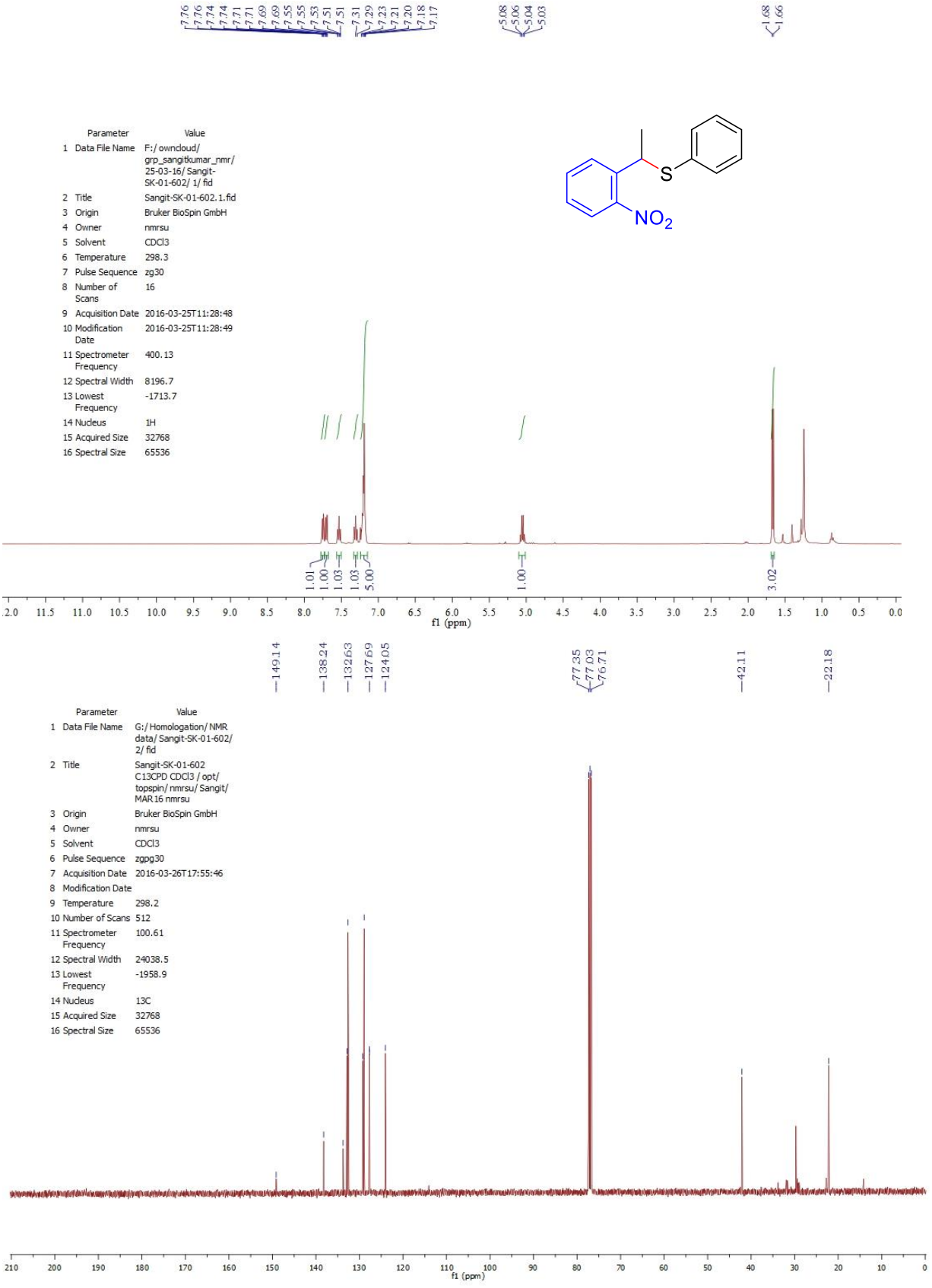
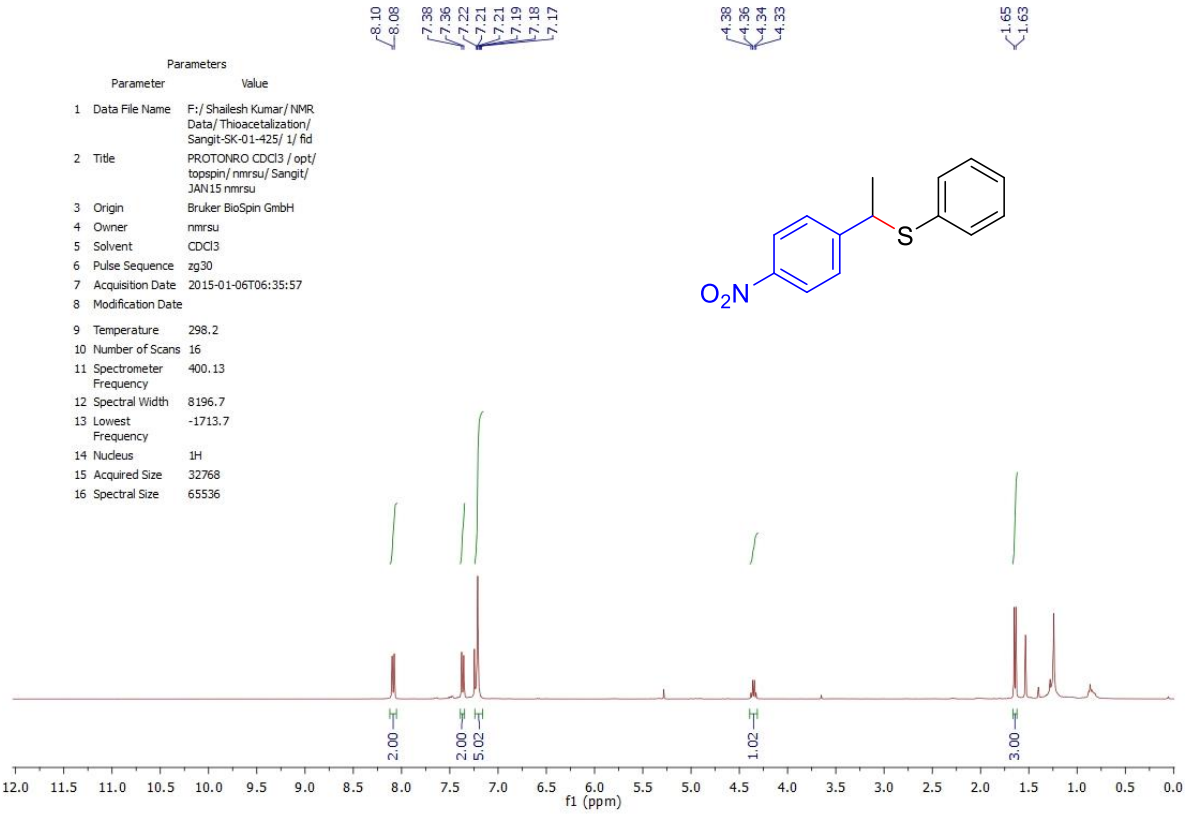
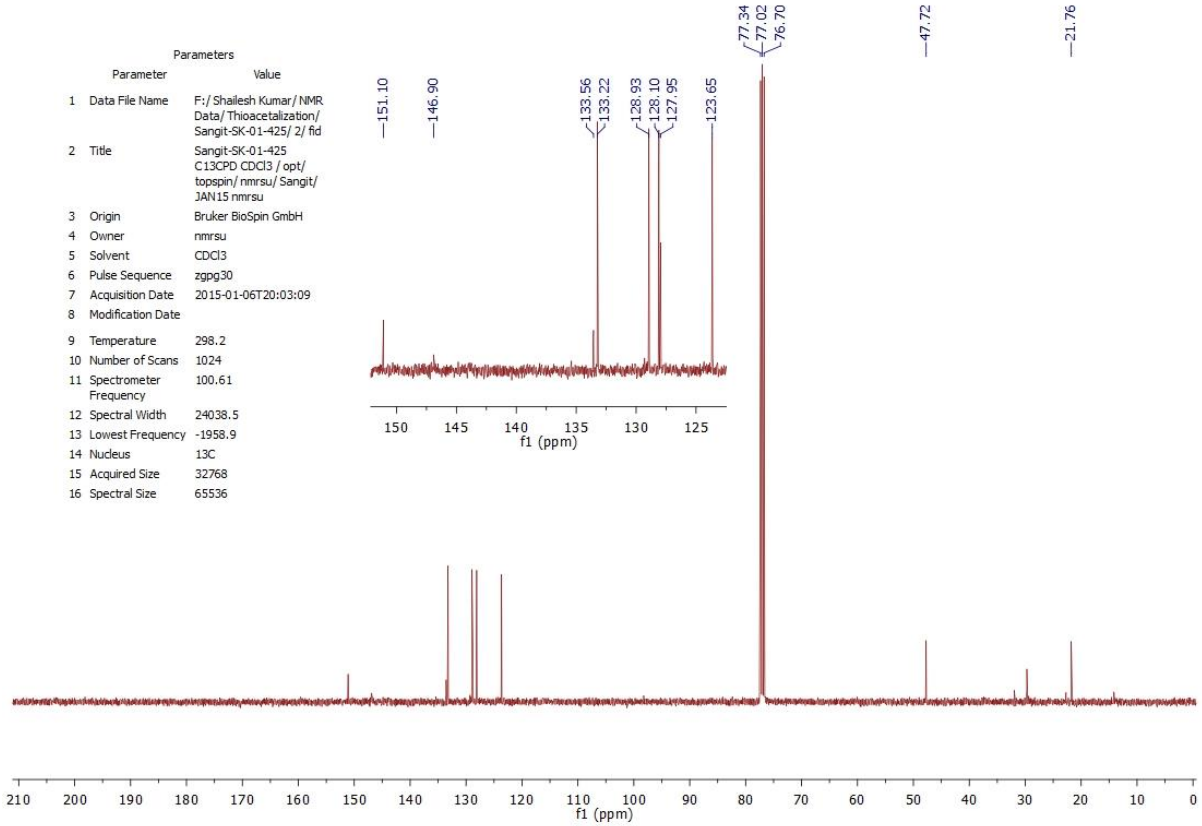
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2016-02-29T17:48:41
8	Modification Date	
9	Temperature	298.3
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S19 ^{1}H & ^{13}C NMR of **18**

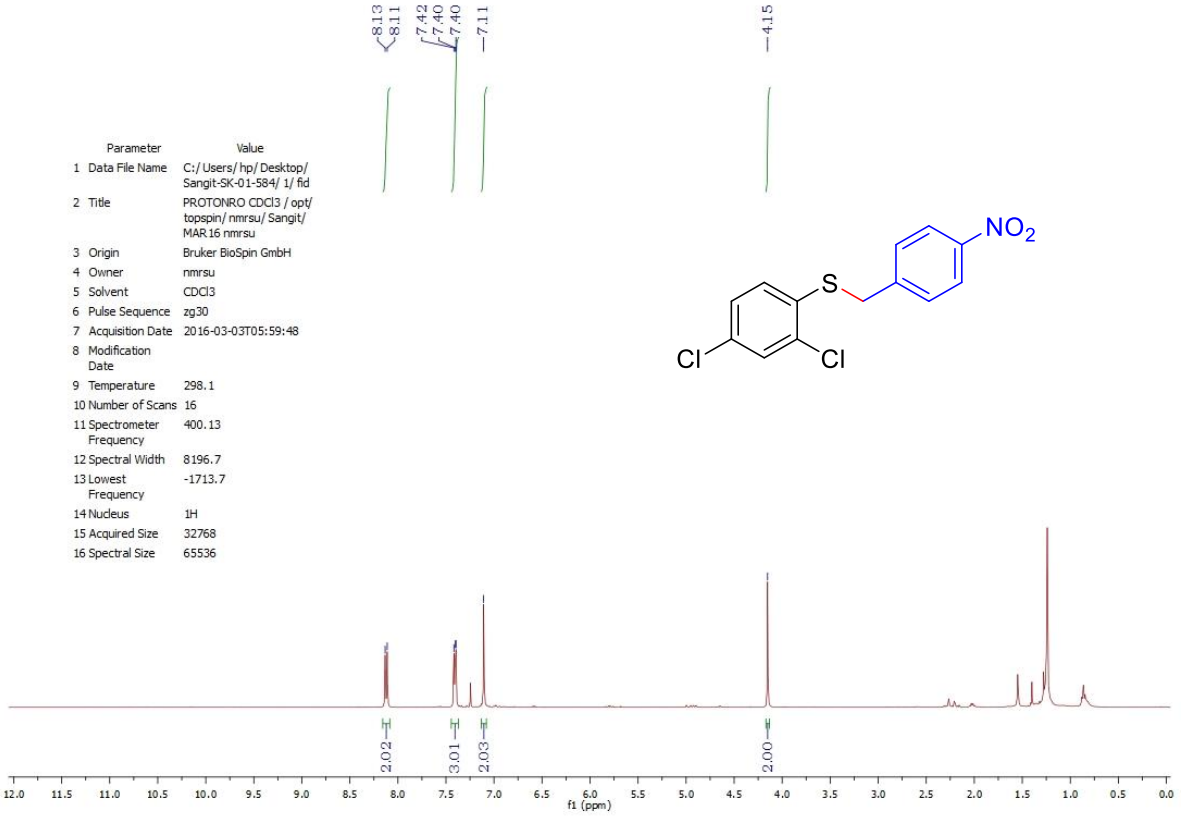


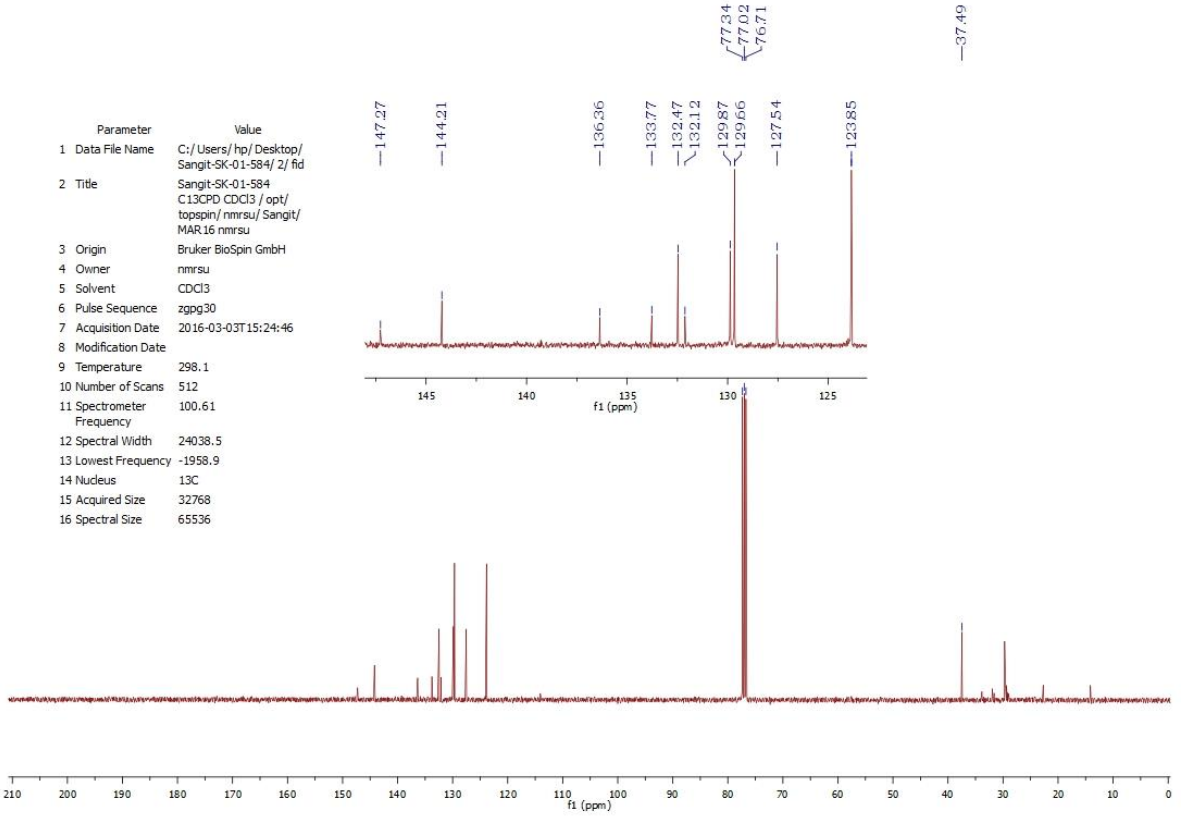
S20 ^{1}H & ^{13}C NMR of **19**



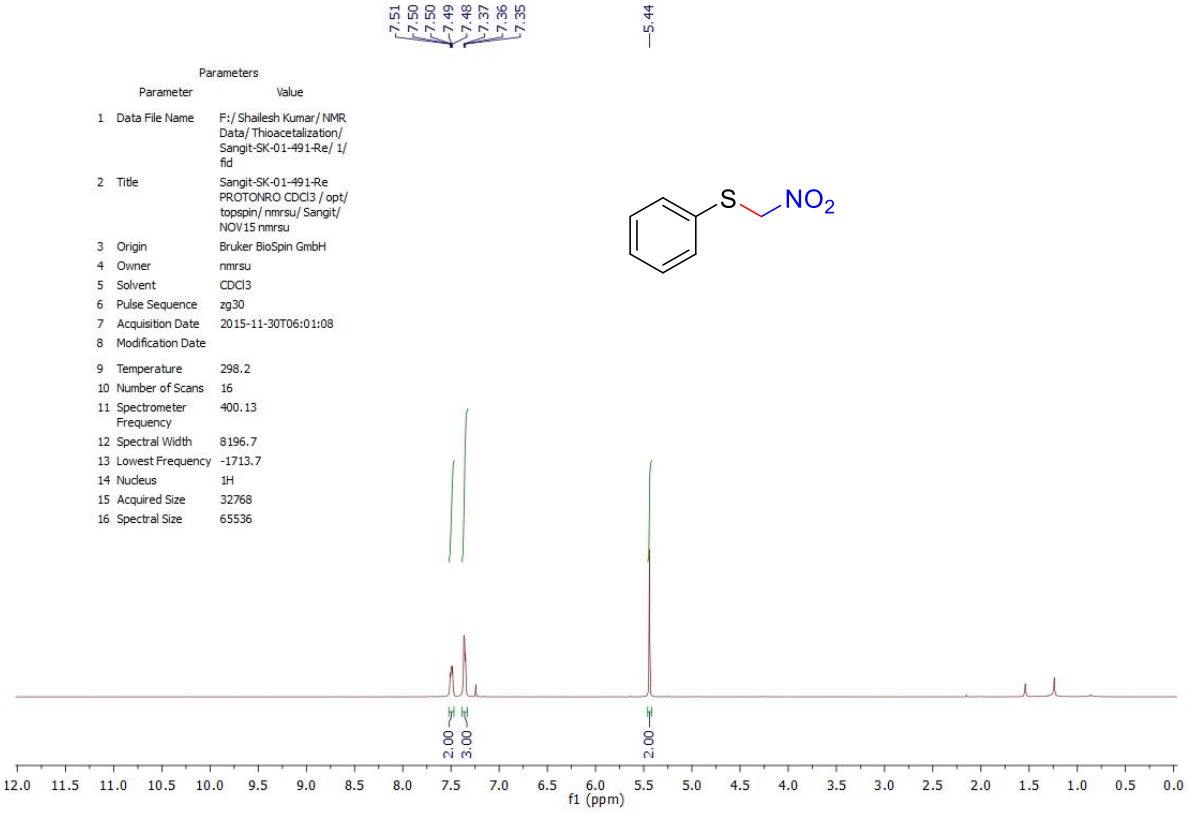


S21 ^{1}H & ^{13}C NMR of **20**





S22 ^{1}H & ^{13}C NMR of **21**



Parameters

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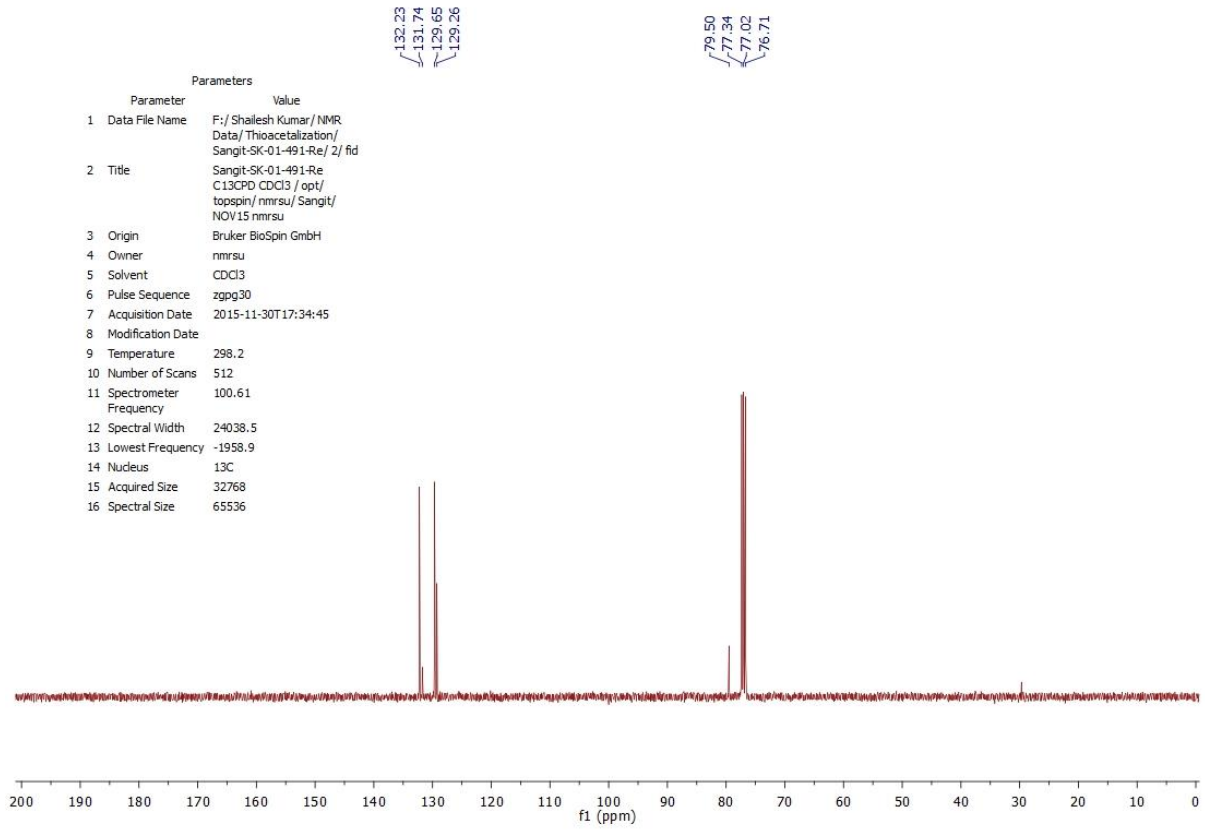
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
7	Acquisition Date	2015-11-30T06:01:08
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



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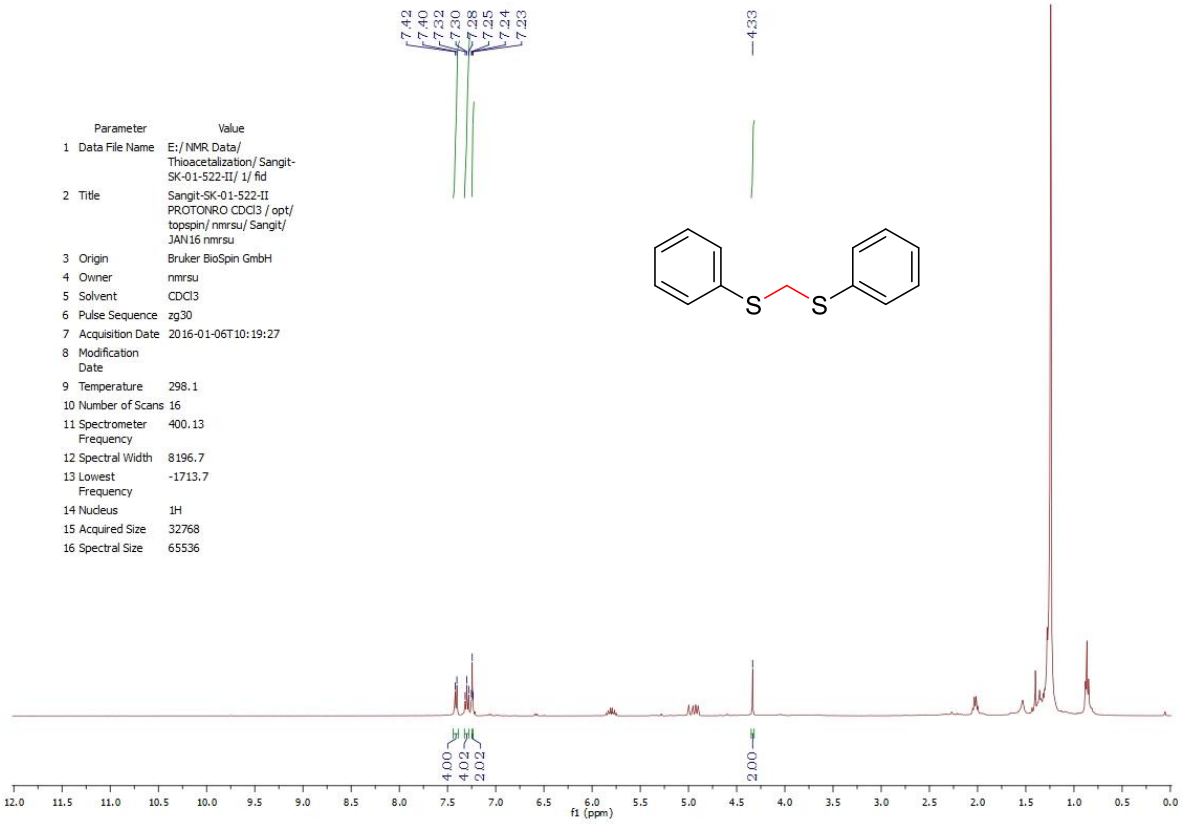
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2015-11-30T17:34:45
8	Modification Date	
9	Temperature	298.2
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S23 ^{1}H & ^{13}C NMR of **22**



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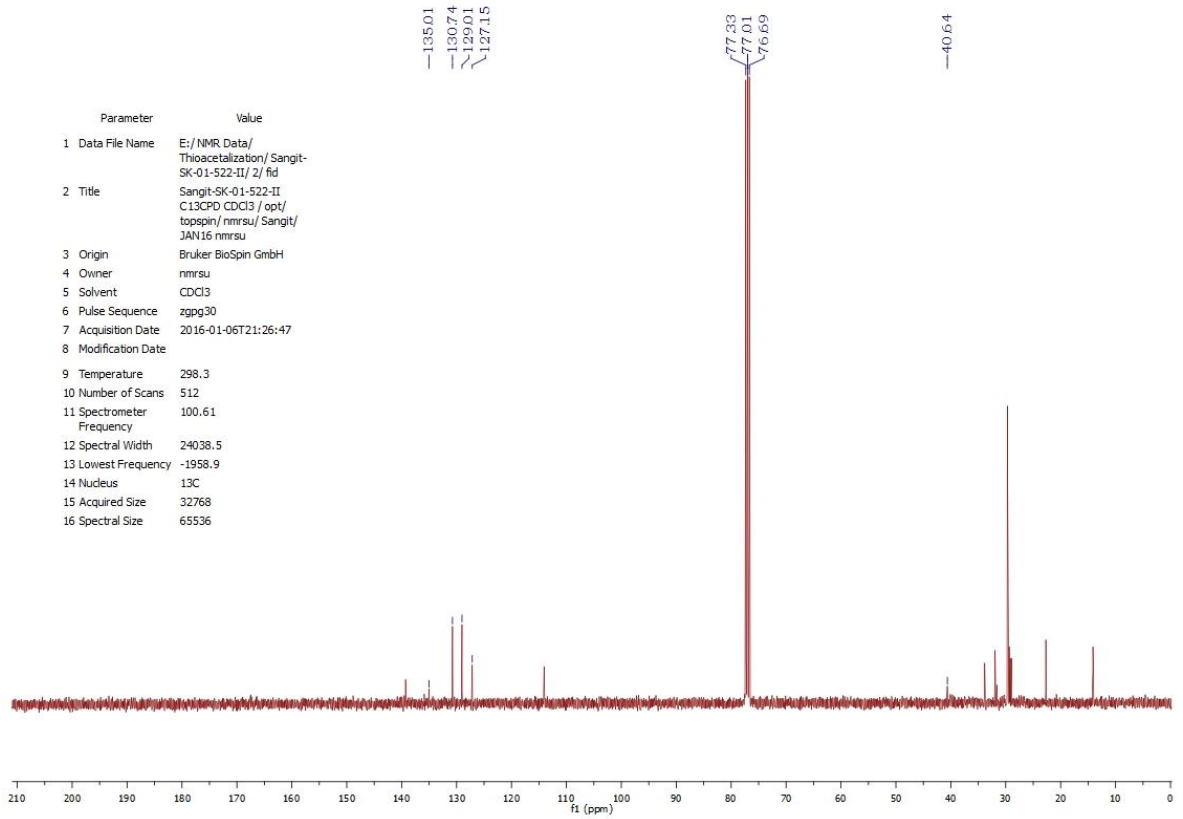
2 Title	Sangit-SK-01-522-II PROTONRO CDCl3 / opt/ topspin/ nmrsu/ Sangit/ JAN16 nmrsu
3 Origin	Bruker BioSpin GmbH
4 Owner	nmrsu
5 Solvent	CDCl3
6 Pulse Sequence	zg30
7 Acquisition Date	2016-01-06T10:19:27
8 Modification Date	
9 Temperature	298.1
10 Number of Scans	16
11 Spectrometer Frequency	400.13
12 Spectral Width	8196.7
13 Lowest Frequency	-1713.7
14 Nucleus	1H
15 Acquired Size	32768
16 Spectral Size	65536



Parameter	Value
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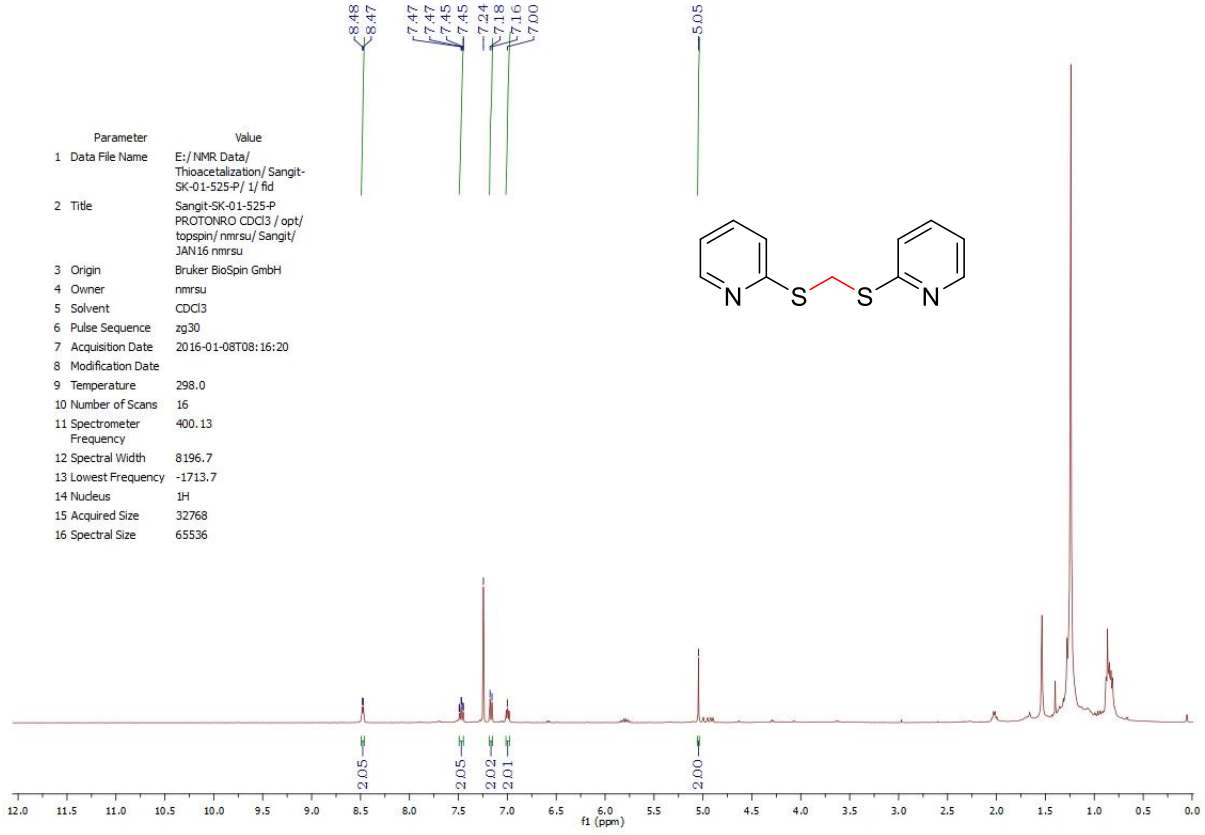
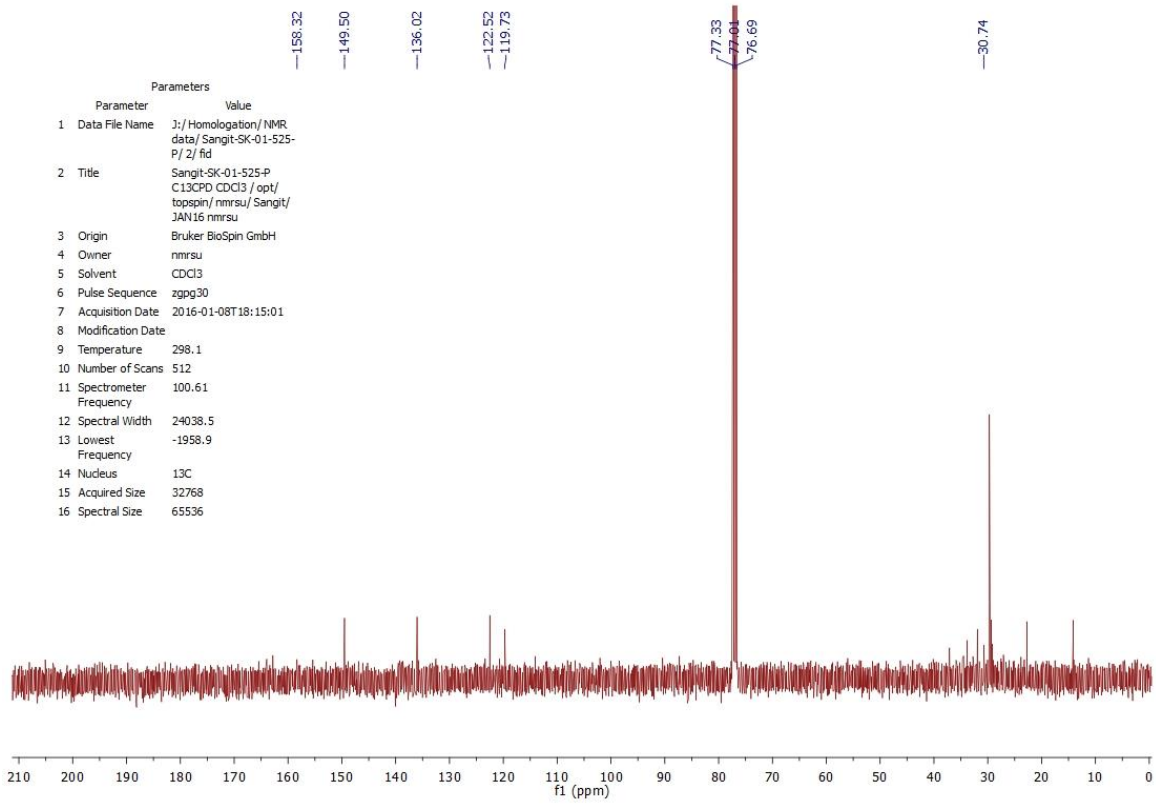
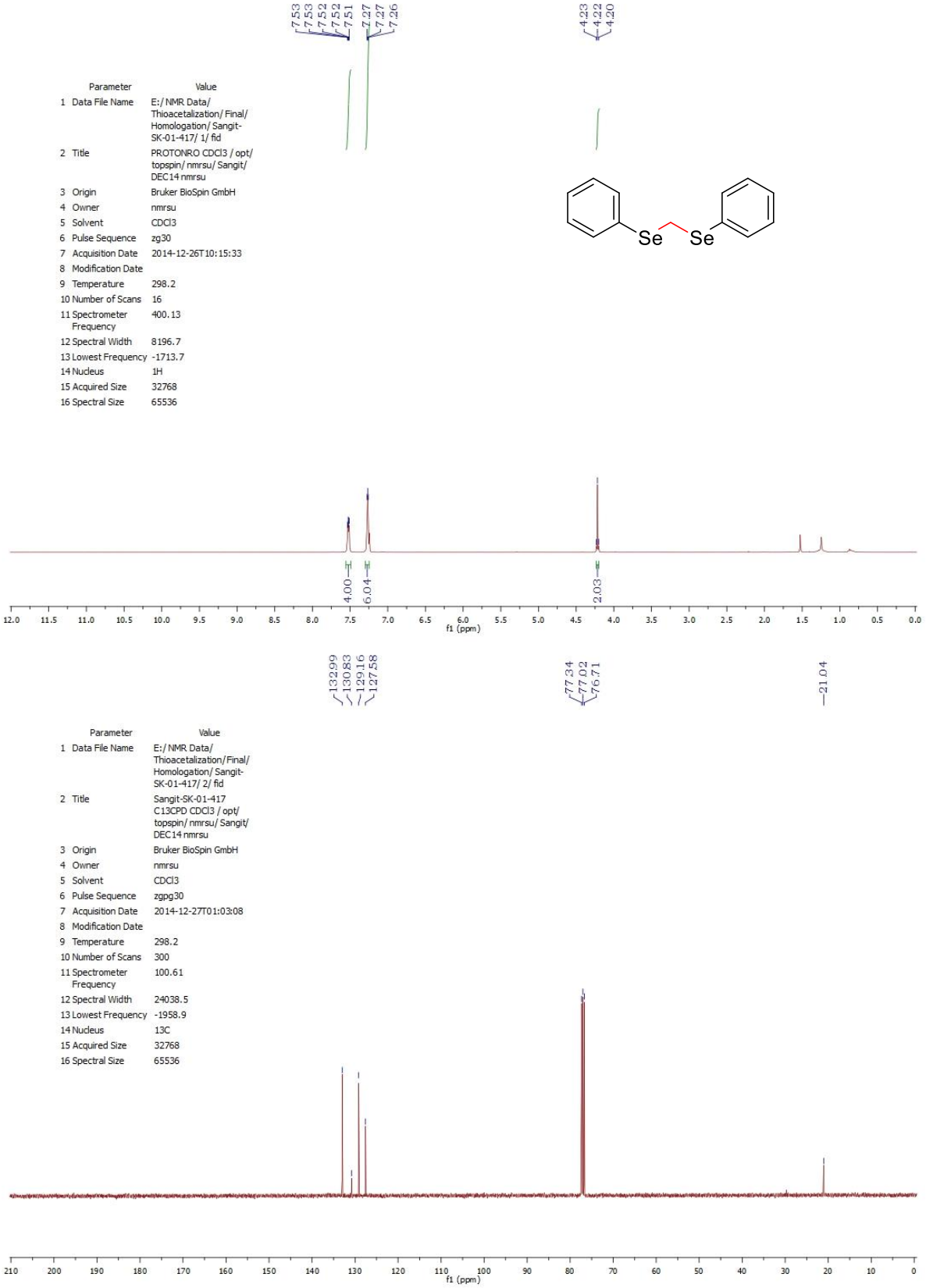
2 Title	Sangit-SK-01-522-II C13CPD CDCl3 / opt/ topspin/ nmrsu/ Sangit/ JAN16 nmrsu
3 Origin	Bruker BioSpin GmbH
4 Owner	nmrsu
5 Solvent	CDCl3
6 Pulse Sequence	zgpg30
7 Acquisition Date	2016-01-06T21:26:47
8 Modification Date	
9 Temperature	298.3
10 Number of Scans	512
11 Spectrometer Frequency	100.61
12 Spectral Width	24038.5
13 Lowest Frequency	-1958.9
14 Nucleus	13C
15 Acquired Size	32768
16 Spectral Size	65536

S24 ^{1}H & ^{13}C NMR of **23**



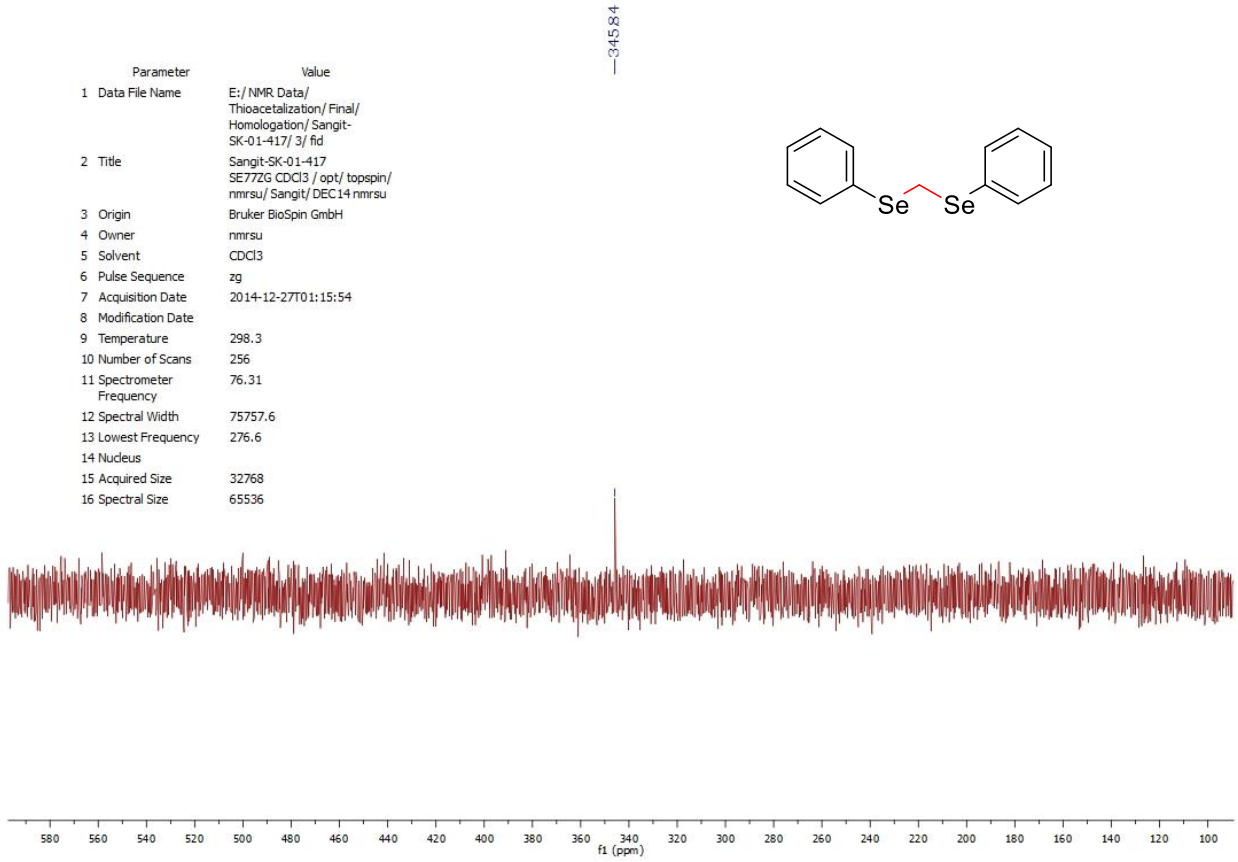


S25 ^{1}H & ^{13}C NMR of **24**

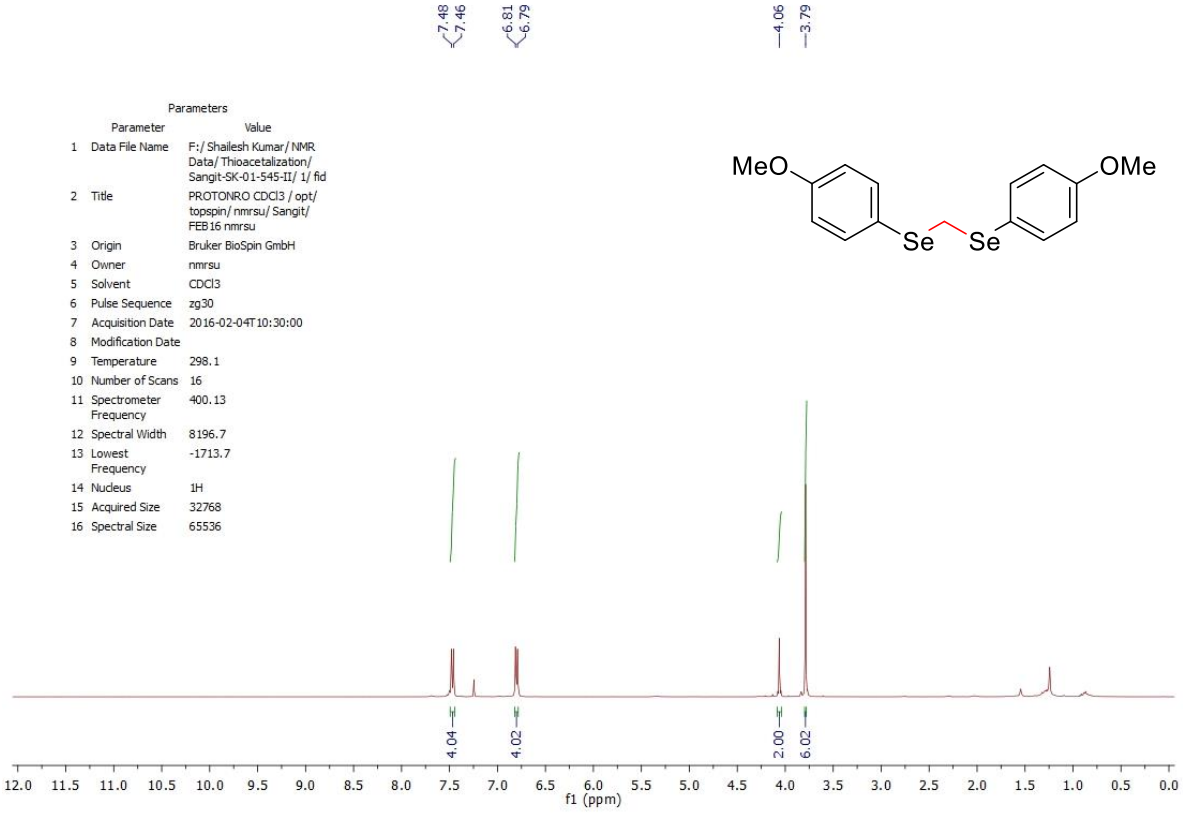


S26 ^{77}Se NMR of **24**

	Parameter	Value
1	Data File Name	E:/ NMR Data/ Thioacetalization/ Final/ Homologation/ Sangit-SK-01-417/ 3/ fid
2	Title	Sangit-SK-01-417 SE77ZG CDCl3 / opt/ topspin/ nmrsu/ Sangit/ DEC14 nmrsu
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg
7	Acquisition Date	2014-12-27T01:15:54
8	Modification Date	
9	Temperature	298.3
10	Number of Scans	256
11	Spectrometer Frequency	76.31
12	Spectral Width	75757.6
13	Lowest Frequency	276.6
14	Nucleus	
15	Acquired Size	32768
16	Spectral Size	65536



S27 ^{1}H & ^{13}C NMR of **25**



Parameters

	Parameter	Value
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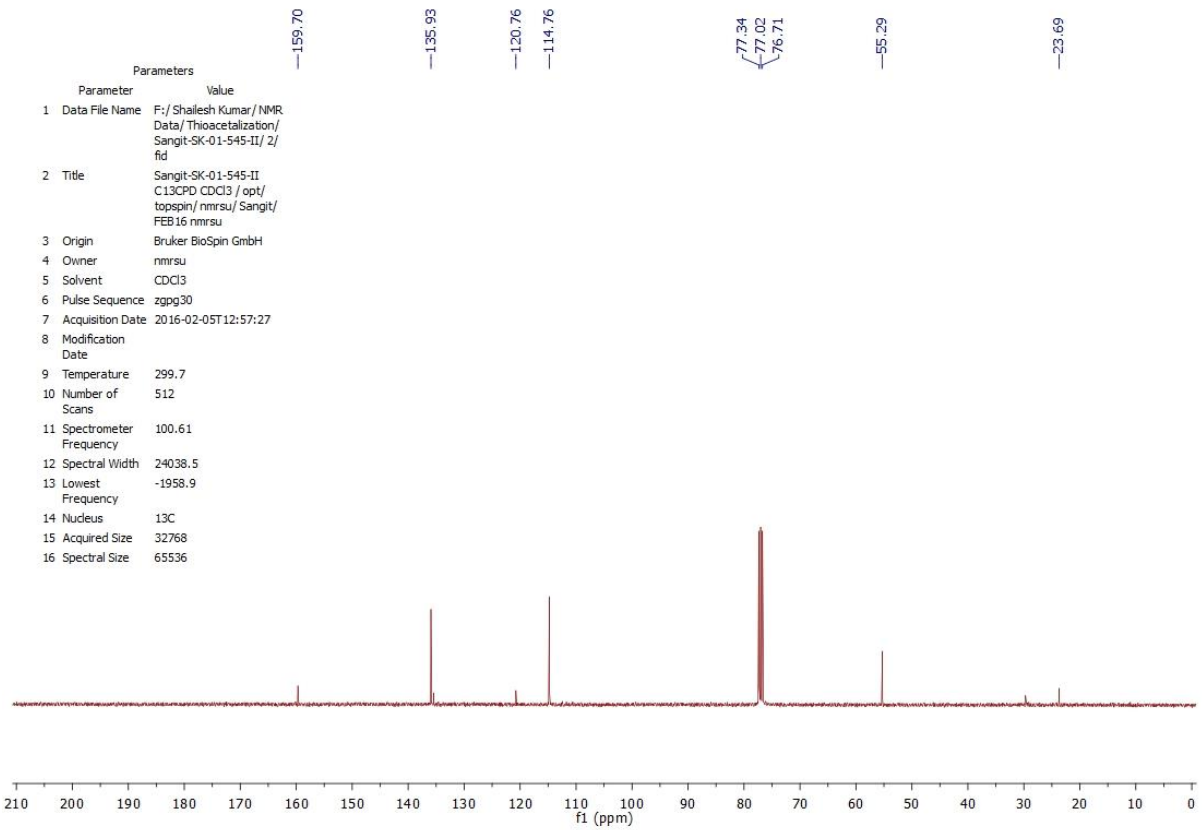
2	Title	PROTONRO CDCl3 / opt/ topspin/ nmrsu/ Sangit/ FEB16 nmrsu
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
7	Acquisition Date	2016-02-04T10:30:00
8	Modification Date	
9	Temperature	298.1
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



Parameters

	Parameter	Value
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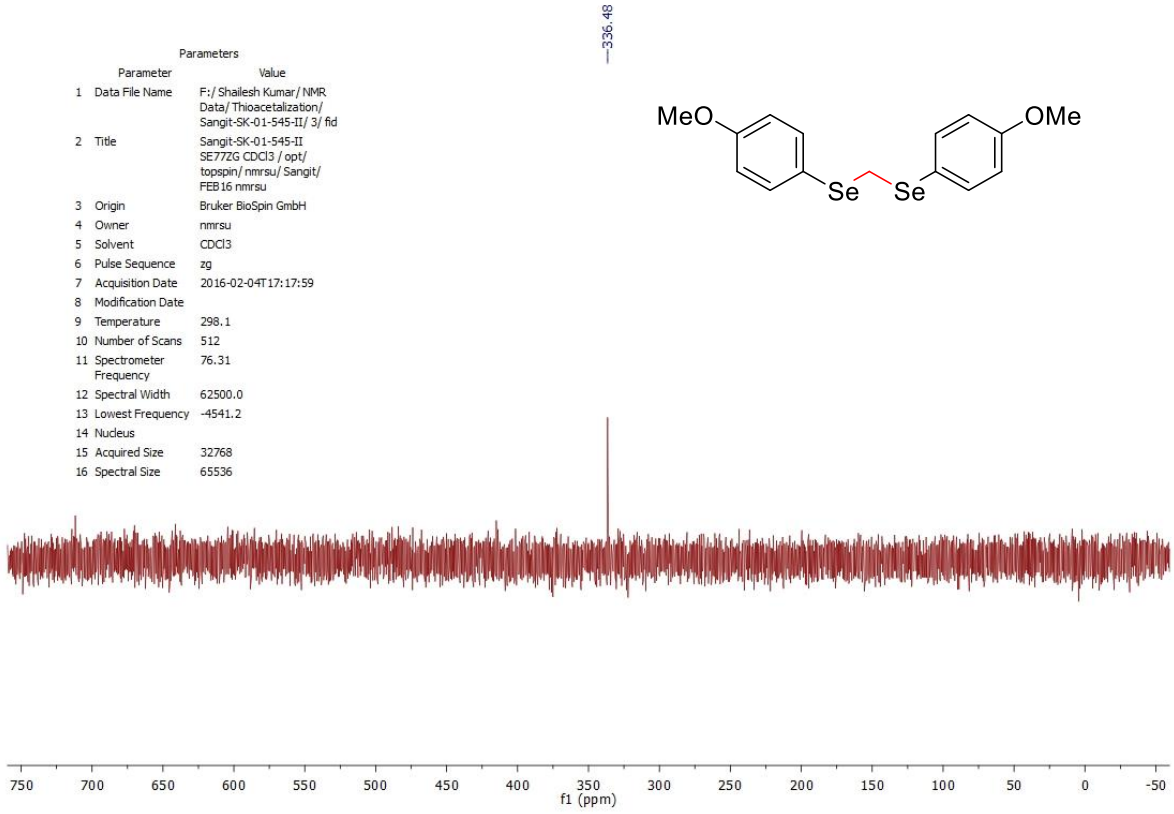
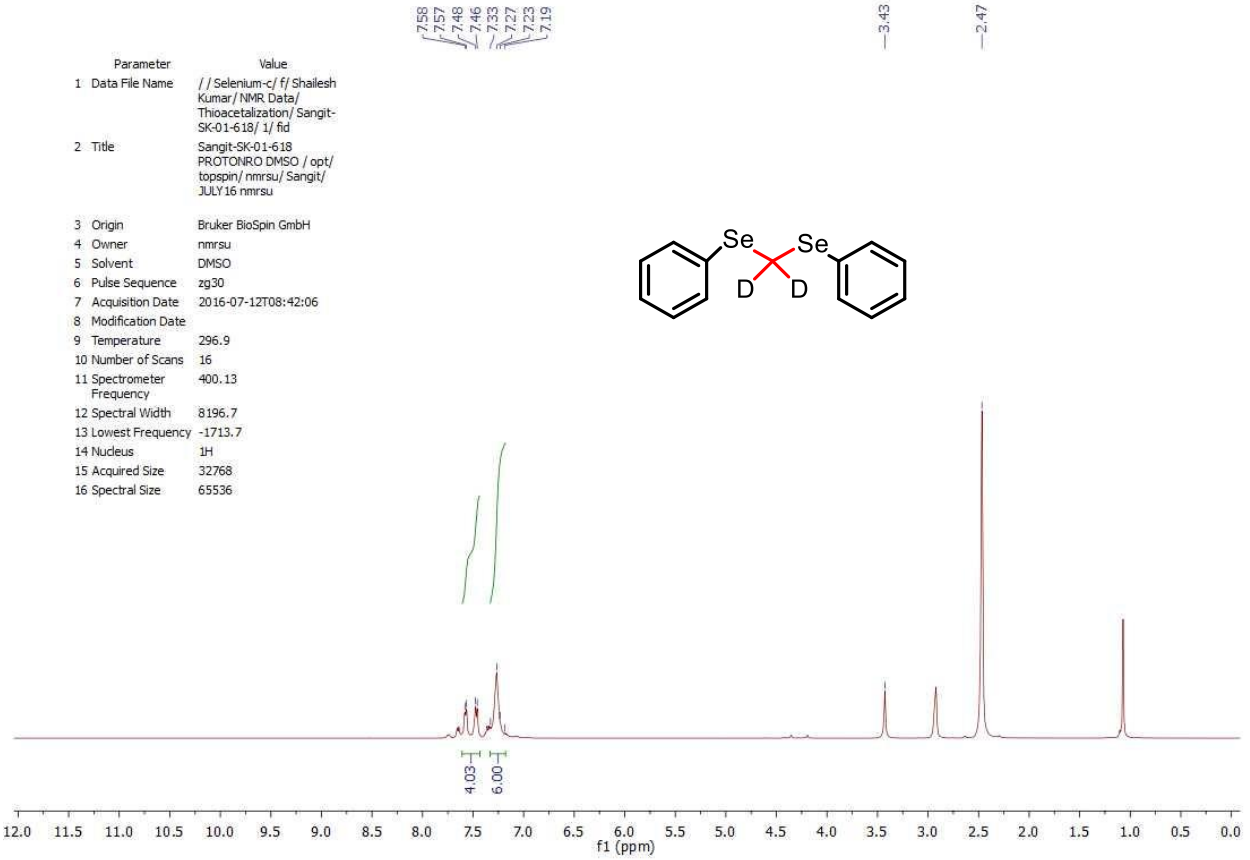
2	Title	Sangit-SK-01-545-II C13CPD CDCl3 / opt/ topspin/ nmrsu/ Sangit/ FEB16 nmrsu
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2016-02-05T12:57:27
8	Modification Date	
9	Temperature	299.7
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S28 ^{77}Se NMR of **25**

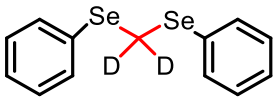


S29 ^{1}H NMR of **26**



Qualitative Compound Report

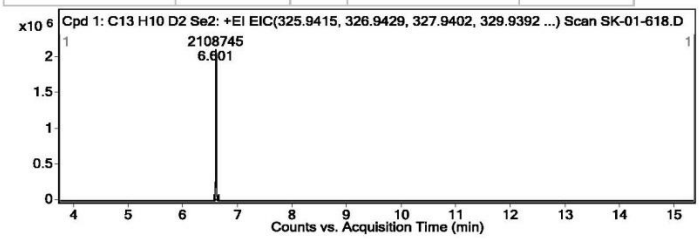
Data File	SK-01-618.D	**Sample Name**	SK-01-618
Sample Type		**Position**	4
Instrument Name	GCQTOF	**User Name**	Agilent-PC\admin
Acq Method	IISER_GENERAL_HP5_80.M	**Acquired Time**	13-Jul-16 11:20:53 AM
IRM Calibration Status	Success	**DA Method**	iiSER13MAY 2013.m
Comment			
Expected Barcode		**Sample Amount**	
Dual Inj Vol	0.1	**TuneName**	Install_tune.ei.tune_1_4_13.tune.xml
TunePath	D:\MassHunter\GCMS\1\7200\	**TuneDateStamp**	41954.1706
MSFirmwareVersion	G.7200.01.09	**OperatorName**	Agilent-PC\admin
RunCompletedFlag	TRUE		



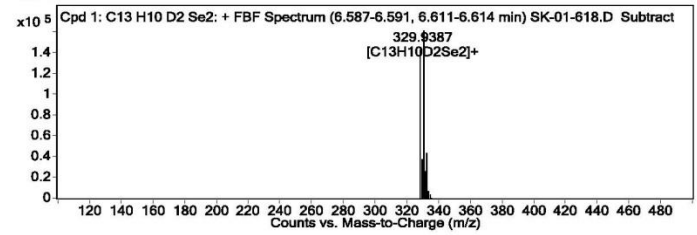
Compound Table

Compound Label	RT	Mass	Abund	Formula	Tgt Mass	Diff (ppm)	MFG Formula	DB Formula
Cpd 1: C13 H10 D2 Se2	6.601	317.9504	161276	C13 H10 D2 Se2	317.9514	-3.15	C13 H10 D2 Se2	C13 H10 D2 Se2

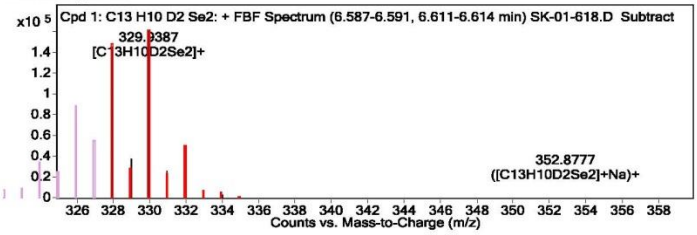
Compound Label	*m/z*	RT	Algorithm	Mass
Cpd 1: C13 H10 D2 Se2	329.9387	6.601	Find By Formula	317.9504



MS Spectrum



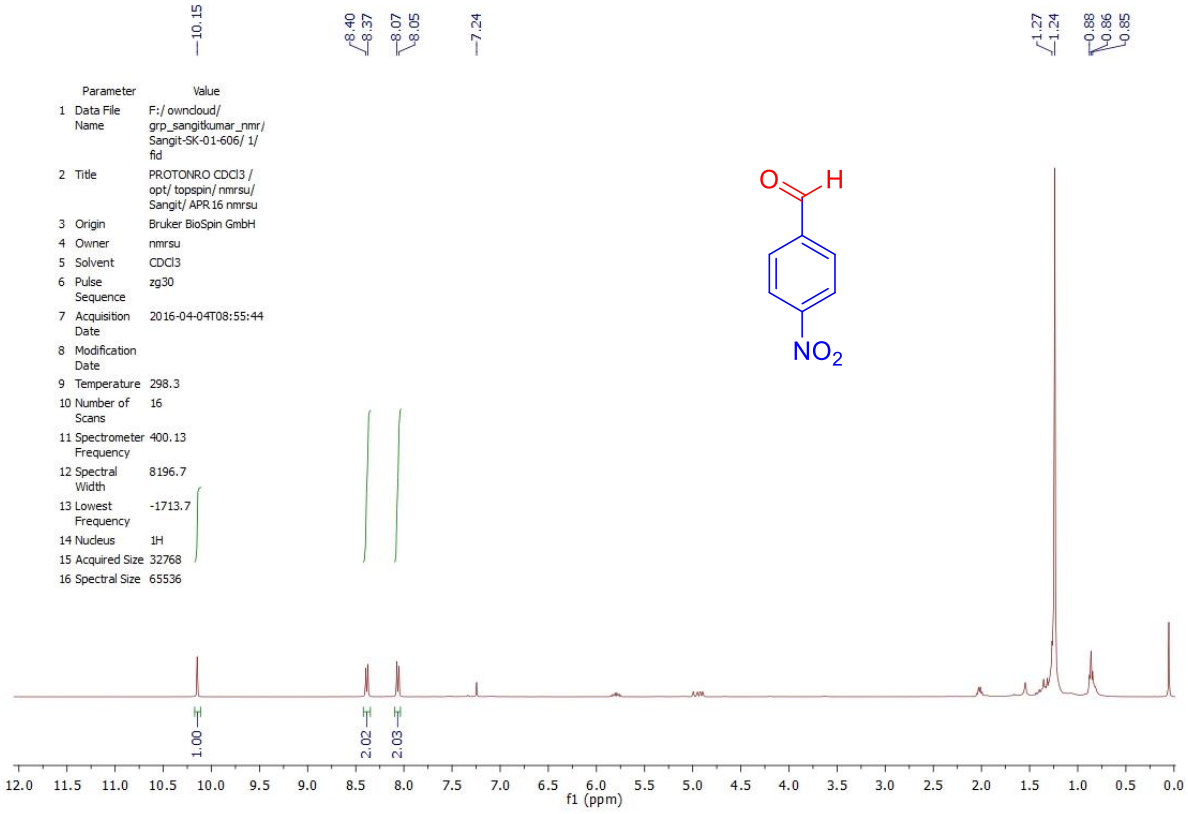
MS Zoomed Spectrum



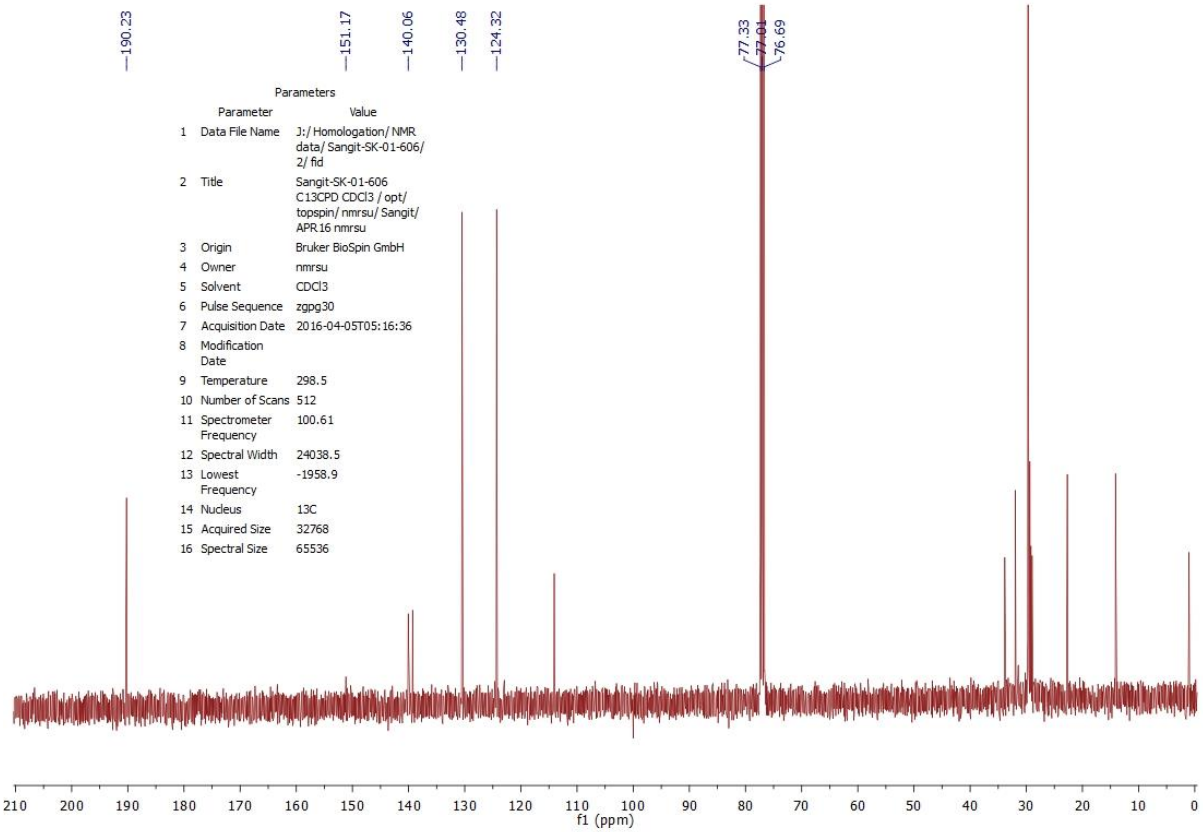
MS Spectrum Peak List

m/z	z	Abund	Formula	Ion
327.9395	1	142599.55	C13H10D2Se2	M+
328.9392	1	38474.62	C13H10D2Se2	M+

S31 ^{1}H & ^{13}C NMR of **27**



Parameter		Value
1	Data File Name	F:/ owncloud/ grp_sangitkumar_nmr/ Sangit-SK-01-606/ 1/ fid
2	Title	PROTONRO CDCl3 / opt/ topspin/ nmrsu/ Sangit/ APR16 nmrsu
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zg30
7	Acquisition Date	2016-04-04T08:55:44
8	Modification Date	
9	Temperature	298.3
10	Number of Scans	16
11	Spectrometer Frequency	400.13
12	Spectral Width	8196.7
13	Lowest Frequency	-1713.7
14	Nucleus	1H
15	Acquired Size	32768
16	Spectral Size	65536



Parameters

Parameter		Value
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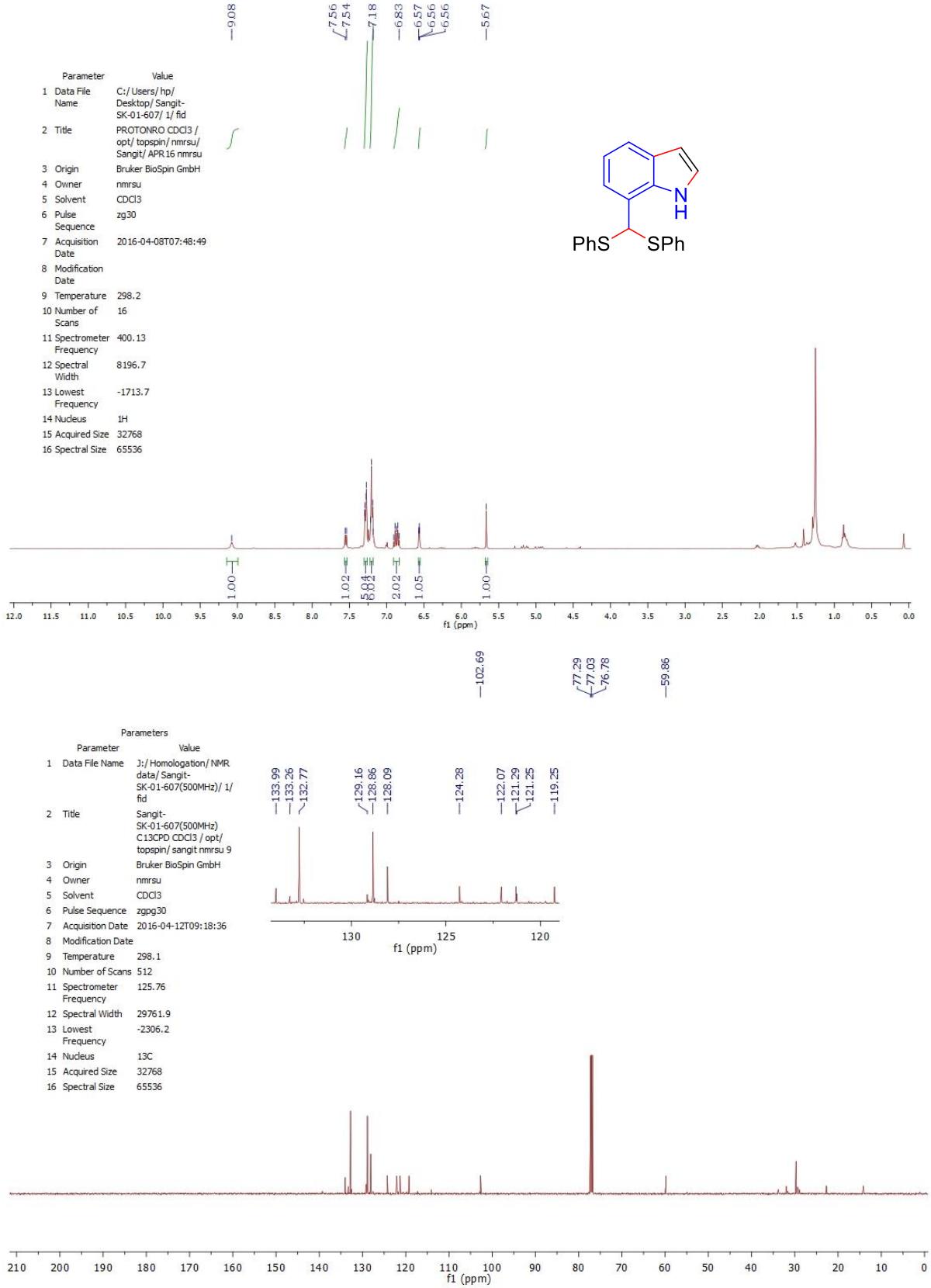
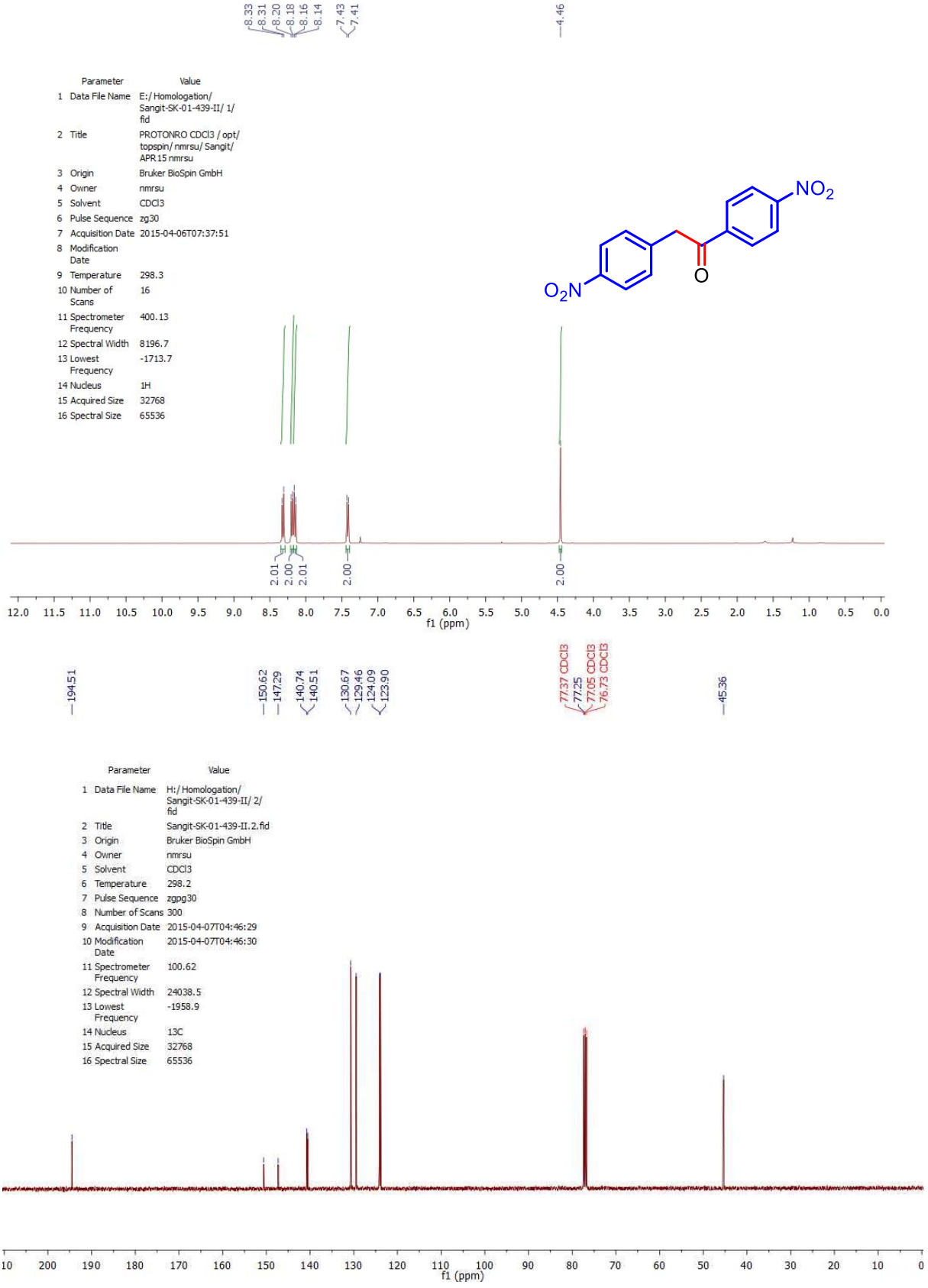
2	Title	Sangit-SK-01-606 C13CPD CDCl3 / opt/ topspin/ nmrsu/ Sangit/ APR16 nmrsu
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Pulse Sequence	zgpg30
7	Acquisition Date	2016-04-05T05:16:36
8	Modification Date	
9	Temperature	298.5
10	Number of Scans	512
11	Spectrometer Frequency	100.61
12	Spectral Width	24038.5
13	Lowest Frequency	-1958.9
14	Nucleus	13C
15	Acquired Size	32768
16	Spectral Size	65536

S32 ^{1}H & ^{13}C NMR of **28**



S33 ^{1}H & ^{13}C NMR of **29**



Ortep view of 6 with 40% ellipsoidal probability and hydrogens are omitted for clarity (CCDC No. 1487922)

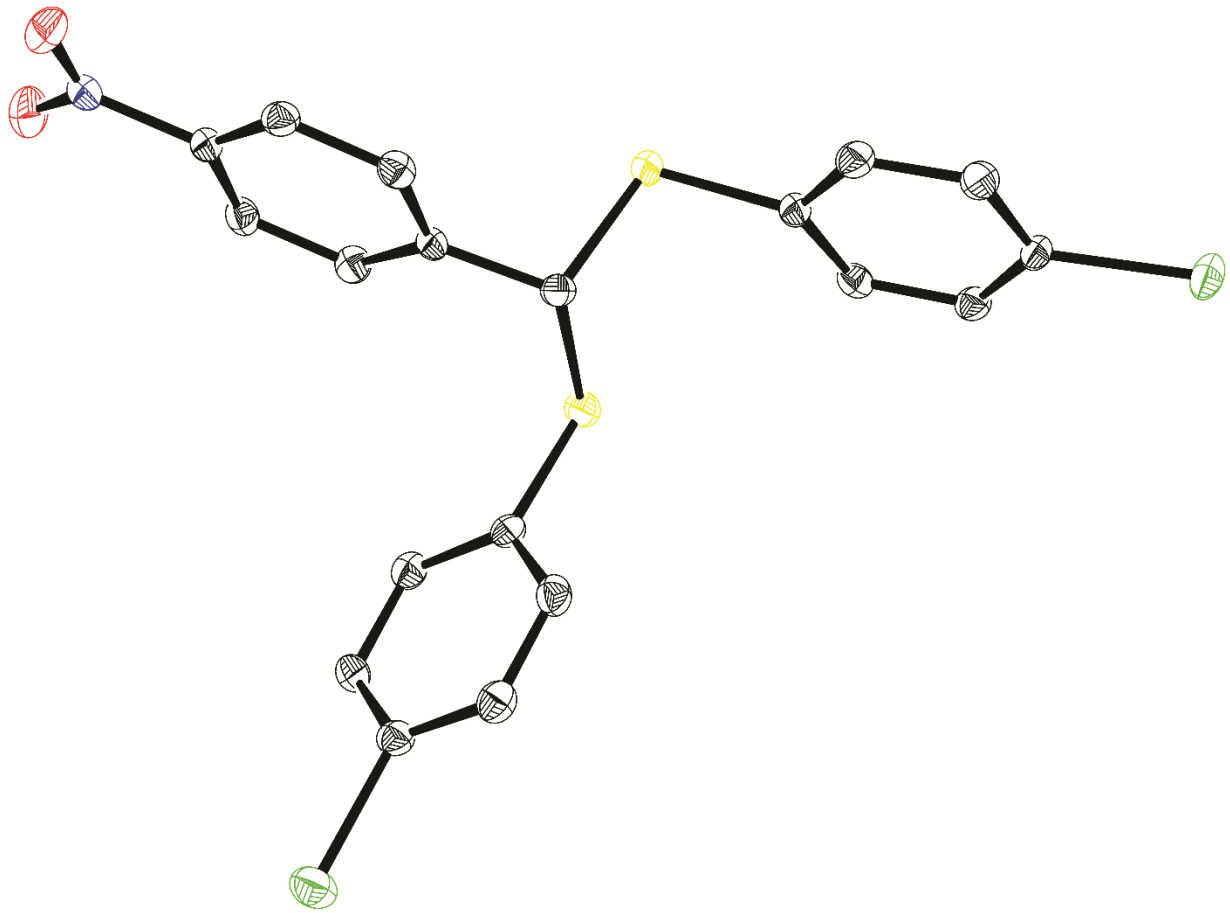


Table 1 Crystal data and structure refinement for 6

Identification code	SK_543_P (Final)
Empirical formula	$C_{19}H_{13}Br_2NO_2S_2$
Formula weight	511.22
Temperature/K	187.23
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	7.6546(8)
b/Å	29.982(3)
c/Å	8.7107(10)
α/°	90
β/°	108.845(4)
γ/°	90
Volume/Å^3	1892.0(4)
Z	4
ρ_{calc}g/cm^3	1.7948
μ/mm^{-1}	4.519
F(000)	1008.0
Crystal size/mm^3	0.3 × 0.25 × 0.2
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	5.12 to 54.96
Index ranges	$-9 \le h \le 9$, $0 \le k \le 38$, $0 \le l \le 11$
Reflections collected	4339
Independent reflections	4339 [R_{int} = 0.0000, R_{sigma} = 0.0268]
Data/restraints/parameters	4339/0/235
Goodness-of-fit on F^2	1.069
Final R indexes [I>=2σ (I)]	R_1 = 0.0318, wR_2 = 0.0571
Final R indexes [all data]	R_1 = 0.0434, wR_2 = 0.0603
Largest diff. peak/hole / e Å^{-3}	0.63/-0.53

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 6. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br2	7449.3(4)	3185.74(9)	3196.7(3)	29.83(8)
Br1	8890.1(4)	5095.29(10)	-8363.3(4)	33.27(8)
S1	2001.6(9)	3384.8(2)	-4134.4(8)	26.13(15)
S2	3546.1(9)	4227.3(2)	-4770.3(8)	24.80(14)
O1	-2165(3)	3487.1(7)	-12705(2)	41.2(5)
C17	74(3)	3325.8(8)	-10250(3)	22.0(5)
C5	7323(3)	4821.9(8)	-7316(3)	23.0(5)
N1	-1034(3)	3209.4(7)	-11930(3)	27.0(5)
C2	5028(3)	4436.2(8)	-5820(3)	21.9(5)
C8	3672(3)	3352.9(8)	-2162(3)	21.8(5)

C16	-115(3)	3747.2(8)	-9672(3)	23.9(5)
O2	-799(3)	2847.9(7)	-12457(2)	39.6(5)
C13	3952(3)	3709.8(8)	-1084(3)	22.7(5)
C15	944(3)	3856.0(8)	-8098(3)	23.1(5)
C18	1272(3)	3011.5(8)	-9312(3)	22.6(5)
C12	5125(3)	3666.0(8)	493(3)	22.7(5)
C1	3276(3)	3642.5(8)	-5362(3)	21.0(5)
C7	4318(3)	4734.5(8)	-7090(3)	24.0(5)
C9	4580(3)	2950.7(8)	-1649(3)	24.7(5)
C14	2159(3)	3548.1(8)	-7117(3)	19.8(5)
C6	5472(3)	4932.4(8)	-7842(3)	23.8(5)
C11	5977(3)	3256.5(8)	993(3)	21.3(5)
C19	2303(3)	3126.0(8)	-7742(3)	22.3(5)
C4	8064(4)	4527.6(8)	-6066(3)	25.6(5)
C3	6902(4)	4336.4(8)	-5307(3)	25.6(5)
C10	5731(3)	2900.0(8)	-61(3)	24.2(5)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 6. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br2	26.34(14)	35.18(15)	21.23(14)	1.24(11)	-1.63(10)	-0.60(11)
Br1	26.63(14)	37.34(16)	37.30(17)	-0.34(11)	12.32(12)	9.99(12)
S1	22.6(3)	34.5(4)	18.7(3)	-7.9(3)	3.1(2)	3.0(3)
S2	31.9(3)	22.7(3)	22.0(3)	-4.0(3)	11.7(3)	-3.2(2)
O1	43.8(12)	45.8(12)	25.2(11)	0.2(10)	-0.9(9)	7.2(9)
C17	22.4(12)	26.2(13)	17.4(12)	-5.3(10)	6.7(10)	0.2(10)
C5	25.7(13)	21.1(12)	23.4(13)	-3.7(10)	9.4(10)	-3.6(10)
N1	28.3(11)	32.2(13)	21.1(11)	-8.4(10)	8.6(9)	2.9(10)
C2	28.3(13)	16.9(12)	20.1(13)	-5.4(10)	7.4(10)	-5.9(9)
C8	19.7(12)	27.7(13)	17.5(12)	-4.2(10)	5.2(10)	2(1)
C16	23.4(12)	27.0(13)	20.8(13)	1.8(10)	6.3(10)	4.9(10)
O2	51.6(13)	34.9(11)	27.5(11)	-10.0(9)	6.3(9)	-8.7(9)
C13	23.9(12)	19.9(12)	24.9(13)	1.5(10)	8.9(10)	4.3(10)
C15	25.9(13)	21.3(12)	23.2(13)	1.7(10)	9.5(11)	-1.8(10)
C18	25.7(13)	20.0(12)	23.9(13)	-3.1(10)	10.3(11)	-2.8(10)
C12	26.0(12)	21.8(12)	20.9(13)	-4.3(10)	8.5(10)	-4.1(10)
C1	22.1(12)	20.3(12)	21.4(13)	-3.2(10)	8.1(10)	-1.2(10)
C7	22.7(12)	24.1(13)	23.0(13)	0.6(10)	4.2(10)	-0.9(10)
C9	27.5(13)	23.2(13)	23.3(13)	-3.9(10)	7.9(11)	-3.7(10)
C14	20.1(12)	23.5(12)	17.1(12)	-2.8(9)	7.9(10)	1.3(9)

C6	26.7(13)	22.3(12)	21.8(13)	0.7(10)	6.7(11)	4.7(10)
C11	17.4(11)	26.9(13)	17.1(12)	-3.3(10)	2.2(9)	-0(1)
C19	20.9(12)	22.0(12)	24.1(13)	2.4(10)	7.2(10)	3.3(10)
C4	23.6(12)	25.2(13)	25.8(14)	2.8(10)	4.8(11)	-0.8(11)
C3	31.8(14)	21.3(12)	20.8(13)	0.9(10)	4.5(11)	1.6(10)
C10	24.0(12)	21.4(12)	25.4(14)	0.8(10)	5.3(11)	1.1(10)

Table 4 Bond Lengths for 6

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br2	C11	1.898(2)	C2	C3	1.390(4)
Br1	C5	1.911(2)	C8	C13	1.393(3)
S1	C8	1.783(2)	C8	C9	1.391(4)
S1	C1	1.835(2)	C16	C15	1.387(3)
S2	C2	1.784(3)	C13	C12	1.383(3)
S2	C1	1.820(2)	C15	C14	1.391(3)
O1	N1	1.233(3)	C18	C19	1.383(3)
C17	N1	1.477(3)	C12	C11	1.392(3)
C17	C16	1.385(3)	C1	C14	1.517(3)
C17	C18	1.383(3)	C7	C6	1.391(4)
C5	C6	1.381(3)	C9	C10	1.387(4)
C5	C4	1.374(4)	C14	C19	1.396(3)
N1	O2	1.213(3)	C11	C10	1.382(3)
C2	C7	1.390(3)	C4	C3	1.391(4)

Table 5. Bond Angles for 6

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	S1	C8	103.51(11)	C14	C15	C16	120.8(2)
C1	S2	C2	103.03(11)	C19	C18	C17	118.2(2)
C16	C17	N1	118.8(2)	C11	C12	C13	118.9(2)
C18	C17	N1	118.9(2)	S2	C1	S1	105.87(12)
C18	C17	C16	122.3(2)	C14	C1	S1	106.00(16)
C6	C5	Br1	118.28(19)	C14	C1	S2	116.14(17)
C4	C5	Br1	119.45(19)	C6	C7	C2	120.4(2)
C4	C5	C6	122.3(2)	C10	C9	C8	120.4(2)
C17	N1	O1	117.4(2)	C1	C14	C15	122.9(2)
O2	N1	O1	124.1(2)	C19	C14	C15	118.9(2)
O2	N1	C17	118.6(2)	C19	C14	C1	118.2(2)
C7	C2	S2	118.94(19)	C7	C6	C5	118.7(2)

C3	C2	S2	121.37(19)	C12	C11	Br2	118.70(18)
C3	C2	C7	119.5(2)	C10	C11	Br2	119.65(18)
C13	C8	S1	120.97(19)	C10	C11	C12	121.6(2)
C9	C8	S1	118.97(19)	C14	C19	C18	121.3(2)
C9	C8	C13	119.8(2)	C3	C4	C5	118.5(2)
C15	C16	C17	118.5(2)	C4	C3	C2	120.7(2)
C12	C13	C8	120.3(2)	C11	C10	C9	118.9(2)

Table 6. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 6

Atom	***x***	***y***	***z***	**U(eq)**
H16	-950(3)	3957.2(8)	-10339(3)	28.7(6)
H13	3334(3)	3984.6(8)	-1434(3)	27.2(6)
H15	837(3)	4144.5(8)	-7686(3)	27.7(6)
H18	1383(3)	2724.6(8)	-9734(3)	27.2(6)
H12	5346(3)	3911.1(8)	1222(3)	27.2(6)
H1	4520(3)	3500.2(8)	-5102(3)	25.2(6)
H7	3038(3)	4803.6(8)	-7446(3)	28.8(6)
H9	4410(3)	2709.5(8)	-2389(3)	29.7(6)
H6	4995(3)	5139.6(8)	-8702(3)	28.6(6)
H19	3125(3)	2913.4(8)	-7077(3)	26.8(6)
H4	9341(4)	4456.3(8)	-5727(3)	30.8(7)
H3	7395(4)	4135.6(8)	-4430(3)	30.7(7)
H10	6341(3)	2624.6(8)	295(3)	29.1(6)

Ortep view of 14 with 40% ellipsoidal probability and hydrogens are omitted for clarity (CCDC No. 1487921)

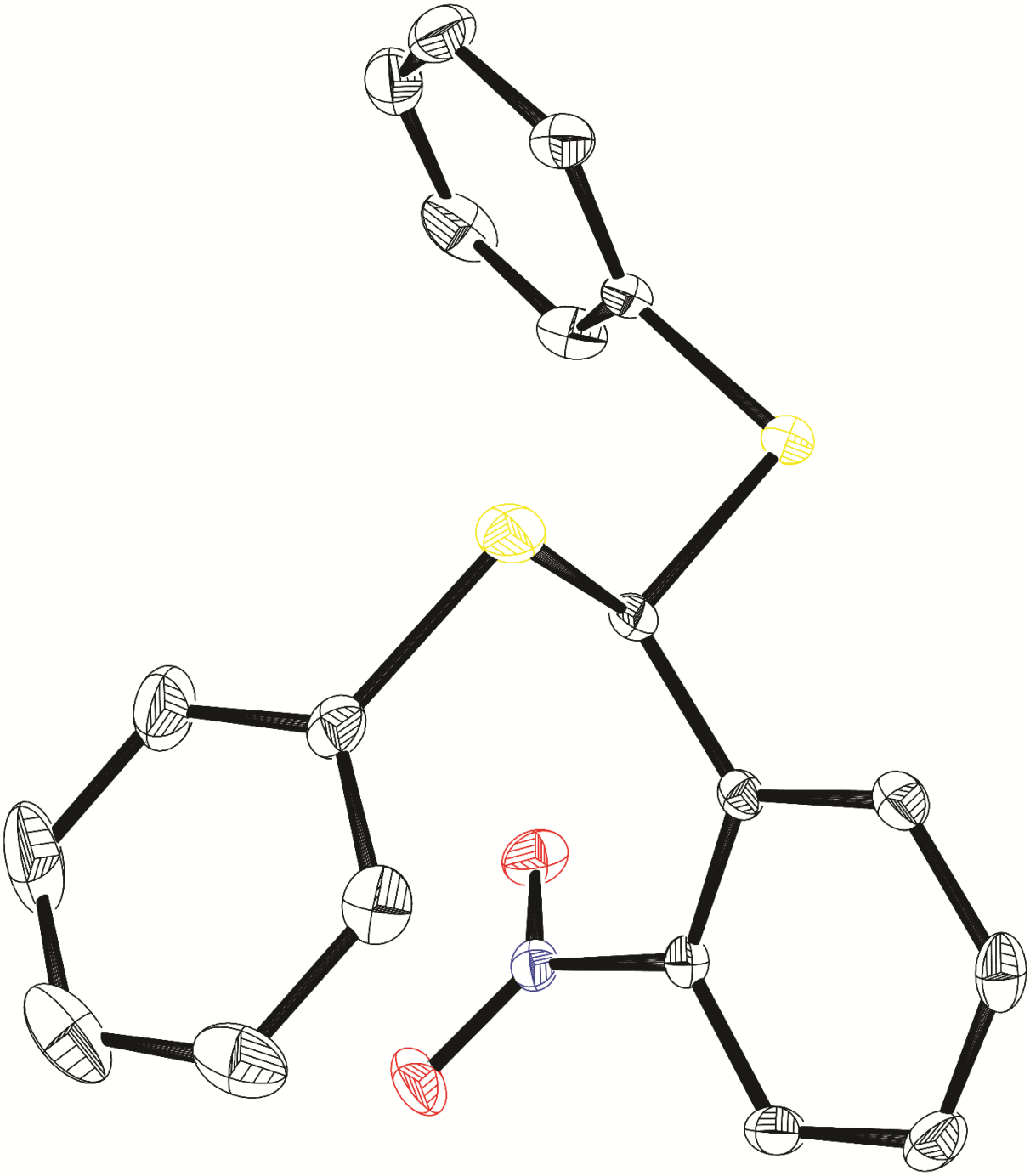


Table 1 Crystal data and structure refinement for 14

Identification code	SK_01_489 (Final)
Empirical formula	$C_{19}H_{15}NO_2S_2$
Formula weight	353.44
Temperature/K	140.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	16.385(2)
b/Å	13.037(2)
c/Å	8.2869(14)
α/°	90
β/°	103.740(6)
γ/°	90
Volume/Å^3	1719.6(5)
Z	4
ρ_{calc}g/cm^3	1.3652
μ/mm^{-1}	0.320
F(000)	736.0
Crystal size/mm^3	0.25 × 0.2 × 0.15
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	5.12 to 47.06
Index ranges	$-18 \leq h \leq 17$, $-14 \leq k \leq 14$, $-7 \leq l \leq 9$
Reflections collected	8283
Independent reflections	2559 [R_{int} = 0.0607, R_{sigma} = 0.0552]
Data/restraints/parameters	2559/0/216
Goodness-of-fit on F^2	1.051
Final R indexes [I>=2σ (I)]	R_1 = 0.0381, wR_2 = 0.0760
Final R indexes [all data]	R_1 = 0.0623, wR_2 = 0.0844
Largest diff. peak/hole / e Å^{-3}	0.38/-0.38

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 14. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	8612.1(4)	9076.8(5)	1423.9(9)	25.4(2)
S2	8325.7(4)	9973.7(6)	4568.9(9)	28.8(2)
O1	5456.9(10)	10437.8(14)	1615(2)	31.4(5)
O2	6317.6(10)	10303.3(14)	-10(2)	28.2(5)

C15	6299.0(13)	8992.6(18)	1869(3)	15.9(6)
C19	7366.3(15)	7830.7(19)	3122(3)	21.3(6)
C14	7153.2(13)	8794.2(18)	2428(3)	15.6(6)
N1	6008.6(12)	9984.9(16)	1112(3)	19.7(5)
C1	7822.2(13)	9588.6(19)	2431(3)	18.5(6)
C2	9085.0(14)	10244.3(19)	995(3)	20.7(6)
C8	7450.7(14)	10615(2)	5043(3)	23.2(6)
C3	9807.5(14)	10598(2)	2081(3)	26.3(7)
C7	8746.6(15)	10791(2)	-434(4)	32.1(7)
C11	6067(2)	11601(3)	5701(4)	50.4(9)
C10	6180.1(17)	10573(3)	6002(4)	39.2(8)
C13	7331.7(19)	11652(2)	4738(4)	42.1(8)
C18	6762.5(15)	7113(2)	3222(3)	25.1(6)
C6	9124.4(17)	11693(2)	-775(4)	39.5(8)
C5	9838.5(17)	12048(2)	331(4)	36.3(8)
C16	5679.7(15)	8292(2)	1995(3)	23.0(6)
C17	5913.3(15)	7338(2)	2660(3)	26.7(7)
C12	6639(2)	12137(3)	5068(5)	56.3(10)
C9	6867.8(16)	10078(2)	5679(3)	28.9(7)
C4	10177.2(16)	11500(2)	1742(4)	34.3(8)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 14. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S1	19.4(3)	22.0(4)	37.8(5)	-1.3(3)	12.6(3)	-4.8(3)
S2	18.6(3)	37.0(4)	27.5(4)	-0.4(3)	-0.9(3)	-9.4(3)
O1	23.3(10)	30.8(12)	37.3(13)	12.5(9)	1.4(9)	-7.0(9)
O2	25.9(10)	26.5(11)	31.0(12)	-2.1(8)	4.2(9)	11.4(9)
C15	19.4(13)	14.2(14)	14.0(14)	1.1(11)	4.0(11)	0.2(11)
C19	20.1(13)	22.6(16)	20.4(16)	3.6(11)	3.4(11)	-1.1(12)
C14	16.7(13)	16.8(15)	13.5(14)	1.4(10)	3.9(11)	-2.4(11)
N1	15.3(11)	18.4(12)	21.8(13)	0.8(9)	-2.7(10)	-4.3(10)
C1	13.8(12)	18.0(14)	22.6(16)	2.4(10)	2.6(11)	-1.4(12)
C2	14.8(13)	22.3(16)	25.8(16)	0.5(11)	6.4(12)	-3.1(12)
C8	24.5(14)	24.5(17)	18.5(16)	-3.9(12)	1.2(12)	-5.6(12)
C3	21.9(14)	31.2(17)	26.3(17)	-2.1(12)	6.5(12)	-0.9(13)
C7	21.8(14)	42(2)	31.8(19)	2.0(13)	5.7(13)	2.2(15)
C11	55(2)	64(3)	37(2)	24.9(18)	19.8(17)	-2.6(18)
C10	36.5(17)	60(2)	25.4(18)	2.1(16)	16.5(14)	0.4(16)
C13	57(2)	22.4(18)	54(2)	-6.4(15)	27.2(18)	-4.0(15)

C18	41.3(16)	15.2(15)	20.0(16)	4.3(12)	9.7(13)	4.2(12)
C6	38.3(17)	44(2)	41(2)	15.1(15)	19.0(16)	19.9(16)
C5	39.9(17)	26.3(18)	52(2)	-2.8(14)	28.5(17)	-1.6(16)
C16	17.1(13)	28.1(17)	24.7(16)	-2.7(12)	6.5(11)	-3.5(13)
C17	31.7(15)	22.5(17)	28.6(18)	-8.8(12)	12.3(13)	0.9(13)
C12	87(2)	25(2)	65(3)	18.9(18)	34(2)	-0.2(17)
C9	38.3(16)	28.7(17)	20.0(16)	-0.2(13)	7.5(13)	1.7(13)
C4	28.4(15)	37.4(19)	40(2)	-11.8(14)	13.3(15)	-10.7(16)

Table 4 Bond Lengths for 14

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	C1	1.825(2)	C2	C7	1.380(4)
S1	C2	1.782(3)	C8	C13	1.381(4)
S2	C1	1.838(3)	C8	C9	1.386(3)
S2	C8	1.782(3)	C3	C4	1.381(4)
O1	N1	1.232(2)	C7	C6	1.389(4)
O2	N1	1.232(3)	C11	C10	1.368(4)
C15	C14	1.390(3)	C11	C12	1.369(5)
C15	N1	1.467(3)	C10	C9	1.379(4)
C15	C16	1.387(3)	C13	C12	1.382(4)
C19	C14	1.391(3)	C18	C17	1.390(3)
C19	C18	1.379(3)	C6	C5	1.383(4)
C14	C1	1.508(3)	C5	C4	1.370(4)
C2	C3	1.385(3)	C16	C17	1.377(4)

Table 5 Bond Angles for 14

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	S1	C1	99.67(11)	C7	C2	C3	119.5(2)
C8	S2	C1	98.06(11)	C13	C8	S2	120.3(2)
N1	C15	C14	120.3(2)	C9	C8	S2	120.7(2)
C16	C15	C14	123.3(2)	C9	C8	C13	119.0(3)
C16	C15	N1	116.4(2)	C4	C3	C2	120.0(3)
C18	C19	C14	121.6(2)	C6	C7	C2	120.3(3)
C19	C14	C15	116.1(2)	C12	C11	C10	119.7(3)
C1	C14	C15	123.2(2)	C9	C10	C11	120.3(3)
C1	C14	C19	120.6(2)	C12	C13	C8	120.0(3)
O2	N1	O1	124.0(2)	C17	C18	C19	120.8(2)

C15	N1	O1	117.9(2)	C5	C6	C7	119.7(3)
C15	N1	O2	118.08(19)	C4	C5	C6	119.9(3)
S2	C1	S1	109.63(11)	C17	C16	C15	119.1(2)
C14	C1	S1	110.03(16)	C16	C17	C18	119.1(2)
C14	C1	S2	110.41(17)	C13	C12	C11	120.7(3)
C3	C2	S1	120.0(2)	C10	C9	C8	120.4(3)
C7	C2	S1	120.5(2)	C5	C4	C3	120.6(3)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 14

Atom	*x*	*y*	*z*	U(eq)
H19	7942.2(15)	7663.1(19)	3537(3)	25.5(8)
H1	7555.9(13)	10204.8(19)	1801(3)	22.1(7)
H3	10048.9(14)	10220(2)	3058(3)	31.6(8)
H7	8253.4(15)	10549(2)	-1187(4)	38.5(9)
H11	5594(2)	11941(3)	5930(4)	60.4(11)
H10	5782.8(17)	10198(3)	6435(4)	47(1)
H13	7725.6(19)	12031(2)	4303(4)	50.6(10)
H18	6928.8(15)	6456(2)	3682(3)	30.1(8)
H6	8893.3(17)	12065(2)	-1763(4)	47.4(10)
H5	10093.7(17)	12670(2)	113(4)	43.6(9)
H16	5102.7(15)	8468(2)	1628(3)	27.7(8)
H17	5499.7(15)	6841(2)	2734(3)	32.1(8)
H12	6559(2)	12851(3)	4853(5)	67.6(12)
H9	6942.5(16)	9364(2)	5895(3)	34.7(8)
H4	10670.7(16)	11742(2)	2495(4)	41.2(9)

Ortep view of 29 with 40% ellipsoidal probability (CCDC No. 1500695) and hydrogens are omitted for clarity

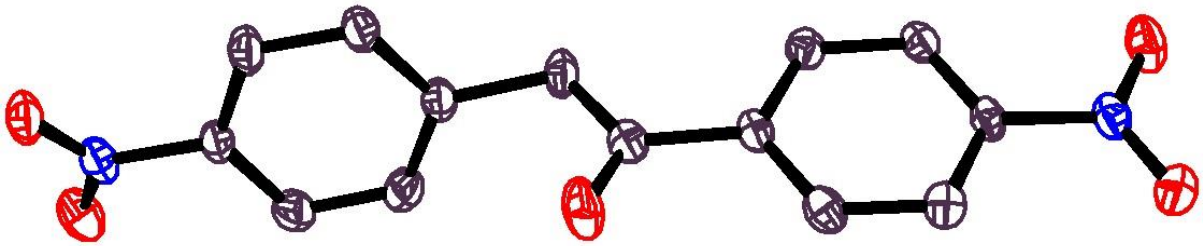


Table 1 Crystal data and structure refinement for 29

Identification code	SK-01-439 (Final)
Empirical formula	$C_{14}H_{10}N_2O_5$
Formula weight	286.24
Temperature/K	140.02
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	13.122(6)
b/Å	12.993(6)
c/Å	7.922(4)
α/°	90
β/°	103.660(12)
γ/°	90
Volume/Å^3	1312.5(10)
Z	4
ρ_{calc}g/cm^3	1.449
μ/mm^{-1}	0.112
F(000)	592.0
Crystal size/mm^3	0.2 × 0.15 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.476 to 51.602
Index ranges	$-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $-9 \leq l \leq 9$
Reflections collected	14834
Independent reflections	2517 [R_{int} = 0.0967, R_{sigma} = 0.0729]
Data/restraints/parameters	2517/0/190
Goodness-of-fit on F^2	1.062
Final R indexes [I>=2σ (I)]	R_1 = 0.0590, wR_2 = 0.1061
Final R indexes [all data]	R_1 = 0.1293, wR_2 = 0.1284
Largest diff. peak/hole / e Å^{-3}	0.20/-0.23

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for SK-01-439 (Final). U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O5	7803.6(14)	6045.7(19)	424(3)	43.4(6)
O1	13194.0(14)	6649.6(19)	-88(3)	45.2(6)
N2	8269.3(17)	6173(2)	-732(3)	31.0(6)
C12	9420.0(19)	6264(2)	-244(4)	26.5(7)
C14	11016(2)	6230(2)	1908(4)	28.7(7)
O2	18170.3(15)	4947(2)	4069(3)	45.3(7)

C10	11025(2)	6447(2)	-1093(4)	31.0(8)
O4	7825.8(14)	6210.6(19)	-2272(3)	44.4(7)
O3	18331.8(14)	6597.8(19)	4306(3)	41.0(6)
C1	12748(2)	6402(2)	1040(4)	29.7(8)
C11	9943(2)	6399(2)	-1551(4)	30.3(8)
N1	17804.1(18)	5815(2)	4024(3)	34.7(7)
C9	11569.2(19)	6361(2)	629(4)	25.7(7)
C6	16650(2)	5914(3)	3641(3)	28.9(8)
C7	16225(2)	6792(3)	4142(4)	35.9(8)
C13	9927.0(19)	6184(2)	1476(4)	28.8(7)
C8	15143(2)	6860(3)	3854(4)	34.9(8)
C3	14509(2)	6059(3)	3079(4)	30.8(8)
C5	16045(2)	5110(3)	2851(4)	38.2(9)
C4	14962(2)	5196(3)	2560(4)	39.7(9)
C2	13332.2(19)	6122(3)	2840(4)	35.6(8)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for SK-01-439 (Final). The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O5	25(1)	57.4(19)	50.4(13)	11.8(13)	14(1)	1.9(10)
O1	27.4(11)	58.0(18)	53.4(14)	13.7(13)	16.3(10)	-5.9(11)
N2	23.9(13)	25.5(18)	43.5(16)	-0.9(13)	7.8(12)	2.7(11)
C12	19.1(13)	19(2)	41.3(17)	-0.7(15)	7.0(12)	1.0(11)
C14	26.3(15)	24(2)	34.3(16)	-0.3(15)	4.9(12)	2.3(12)
O2	36.2(13)	44.7(19)	57.2(15)	9.2(13)	15.6(11)	15.6(12)
C10	29.1(16)	29(2)	38.0(18)	4.2(16)	14.2(13)	-0.4(13)
O4	29.8(11)	60(2)	40.2(13)	-6.3(12)	2.1(10)	-1.7(11)
O3	24.7(11)	45.0(18)	54.9(14)	-0.7(13)	12.7(10)	-7.6(11)
C1	27.8(15)	19(2)	43.6(18)	-4.0(15)	11.6(14)	-3.4(13)
C11	29.3(16)	27(2)	34.0(17)	3.9(15)	5.9(13)	-0.1(13)
N1	25.2(14)	45(2)	35.5(15)	5.7(15)	10.9(11)	4.6(14)
C9	21.8(14)	18(2)	39.0(17)	-1.0(14)	9.4(12)	-0.9(11)
C6	20.5(14)	34(2)	32.4(16)	0.9(16)	7.6(12)	0.2(13)
C7	22.4(15)	35(2)	51.0(19)	-3.8(18)	10.2(13)	-4.9(14)
C13	25.1(15)	25(2)	39.1(18)	-2.4(15)	12.7(13)	-0.7(12)
C8	24.7(15)	31(2)	50.8(19)	-6.5(17)	11.9(14)	-0.2(14)
C3	22.0(14)	36(2)	34.0(17)	0.5(16)	6.2(12)	-1.2(14)
C5	35.1(18)	34(2)	48(2)	-9.7(17)	14.4(15)	0.7(15)
C4	31.9(17)	37(2)	49(2)	-12.9(18)	6.3(14)	-7.5(15)
C2	20.3(14)	45(3)	40.5(18)	-0.9(17)	6.0(13)	-5.3(14)

Table 4 Bond Lengths for 29

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O5	N2	1.226(3)	O3	N1	1.221(3)
O1	C1	1.222(3)	C1	C9	1.504(4)
N2	C12	1.472(3)	C1	C2	1.496(4)
N2	O4	1.222(3)	N1	C6	1.478(3)
C12	C11	1.381(4)	C6	C7	1.370(4)
C12	C13	1.372(4)	C6	C5	1.371(4)
C14	C9	1.389(4)	C7	C8	1.387(4)
C14	C13	1.390(4)	C8	C3	1.382(4)
O2	N1	1.223(3)	C3	C4	1.377(4)
C10	C11	1.381(4)	C3	C2	1.513(4)
C10	C9	1.387(4)	C5	C4	1.390(4)

Table 5 Bond Angles for 29

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O5	N2	C12	118.4(2)	C14	C9	C1	122.3(3)
O4	N2	O5	123.2(2)	C10	C9	C14	119.4(2)
O4	N2	C12	118.4(2)	C10	C9	C1	118.3(2)
C11	C12	N2	118.3(2)	C7	C6	N1	118.7(3)
C13	C12	N2	118.8(2)	C7	C6	C5	122.4(3)
C13	C12	C11	122.9(2)	C5	C6	N1	118.8(3)
C9	C14	C13	120.6(3)	C6	C7	C8	118.4(3)
C11	C10	C9	120.9(3)	C12	C13	C14	118.1(2)
O1	C1	C9	119.8(3)	C3	C8	C7	120.7(3)
O1	C1	C2	122.4(2)	C8	C3	C2	120.3(3)
C2	C1	C9	117.9(2)	C4	C3	C8	119.3(3)
C10	C11	C12	118.1(3)	C4	C3	C2	120.4(3)
O2	N1	C6	117.5(3)	C6	C5	C4	118.3(3)
O3	N1	O2	124.1(3)	C3	C4	C5	120.9(3)
O3	N1	C6	118.3(3)	C1	C2	C3	114.3(2)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 29

Atom	*x*	*y*	*z*	U(eq)
H14	11386	6171	3090	34
H10	11401	6541	-1970	37
H11	9569	6457	-2730	36
H7	16662	7343	4675	43
H13	9545	6099	2346	35
H8	14834	7463	4193	42
H5	16358	4509	2510	46
H4	14528	4652	1996	48
H2A	13074	5447	3145	43
H2B	13174	6639	3660	43

References

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