Supplementary information

Catalyst Free Decarboxylative Trichloromethylation of Aldimines

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Summary

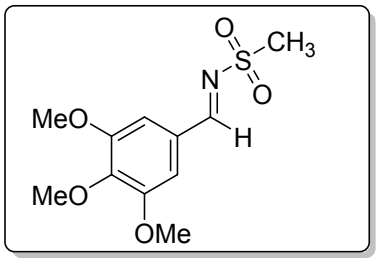
EXPERIMENTAL SECTION

1. General Procedures. Unless otherwise noted, all reagents were obtained commercially and used without further purification. Unless otherwise noted, all reaction mixtures were carried out in flame-dried glassware under nitrogen atmosphere. Analytical thin layer chromatography (TLC) was performed on Merck precoated glass-backed TLC plates (silica gel 60 F254) and visualized by UV lamp (254 nm). Yields refer to chromatographically purified and spectroscopically pure compounds, unless stated otherwise. ^{1}H and ^{13}C spectra were recorded on a Bruker DPX-500 and DRX 300 spectrometer. Chemical shifts are reported in ppm. ^{1}H NMR spectra were referenced to $CDCl_3$ (7.26 ppm) and ^{13}C NMR spectra were referenced to $CDCl_3$ (77.0 ppm). All ^{13}C spectra were measured with complete proton decoupling. Peak multiplicities are designated by the following abbreviations: s, singlet; d, doublet; dd, double doublet; t, triplet; m, multiplet; and J, coupling constant in Hz. High Resolution Mass Spectra (HRMS) were recorded on a Xevo G2-S Q-Tof spectrometer. Infrared experiments were acquired on a ReactIR 45m (Mettler Toledo). EasyMax 102 (Mettler Toledo). Microwave experimentals were performed on Anton Paar Monowave 300 and Microwave reactor CEM Discovery.

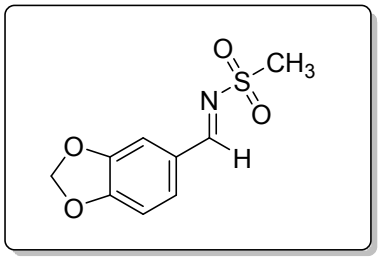
2. 1. General procedure for the preparation of *N*-sulfonyl aldimines Characterization data for the inedited sulfonylimines (1h and 1i)

A mixture of aromatic aldehyde (5.0 mmol), metanosulfonamide (5.0 mmol) and tetraethylortosilicate (5.25 mmol) was stirred and heated at 120 °C for 6-8 h. After completion of reaction (monitored by TLC) the crude reaction mixture was cooled, then solubilized with dichloromethane and washed with water (2:1). The organic phases were combined and concentrated under reduced pressure and the solid was recrystallized with

a mixture of hexanes/ethyl acetate. The resulting solid was collected by filtration and then dried in vacuum. The yields for the formation of *N*-sulfonyl imines ranging from 32% to 77%.

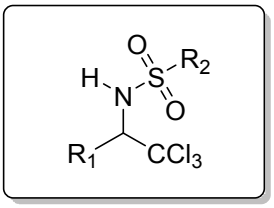


***(E)-N*-(3,4,5-trimethoxybenzylidene)methanesulfonamide:** The imine **1h** was obtained as a crystal colorless solid (887.3 mg, 65%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.91 (s, 1H), 7.21 (s, 2H), 3.97 (s, 3H), 3.93 (s, 6H), 3.14 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 171.2, 153.6, 144.4, 127.0, 108.5, 61.2, 56.4, 40.4. HRMS: calcd for $[C_{11}H_{15}NO_5S]$+ ([M+Na]+): *m/z* 296.0569, found 296.0572.

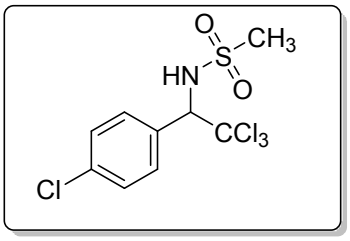


***(E)-N*-(benzo[d][1,3]dioxol-5-ylmethylene)methanesulfonamide:** The product **1i** was obtained as a crystal solid (887.3 mg, 68 %). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.87 (s, 1H), 7.48 (s, 1H), 7.44 (d, 1H, *J* = 8.0 Hz), 6.93 (d, 1H, *J* = 8.0 Hz), 6.11 (s, 2H), 3.11 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 170.4, 154.0, 148.9, 130.8, 126.9, 108.7, 107.9, 102.4, 40.4. HRMS: calcd for $[C_9H_9NO_4S]$+ ([M+Na]+): *m/z* 250.0150, found 250.0157.

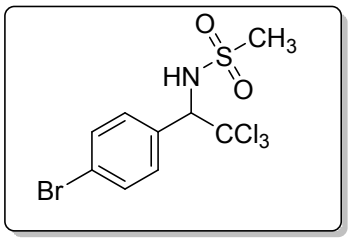
3. General procedure and characterization data for the trichloromethyl sulfonamides (3a-o)



To a solution of imine (0.5 mmol) in DMSO (0.7 mmol.mL^{-1}) was added potassium trichloroacetate salt (302.3 mg, 1.5 mmol) and stirred at room temperature for 40 minutes. After completion, it was added dichloromethane (5 mL) and the solution was extracted with water (4 x 10 mL). The aqueous phases was extracted with AcOEt (2 x 20 mL). The volatiles was dried with anhydrous sodium sulfate and evaporated under reduced pressure. The product was obtained after purification through chromatography column (elution: ethyl acetate/ hexanes, 3:1).

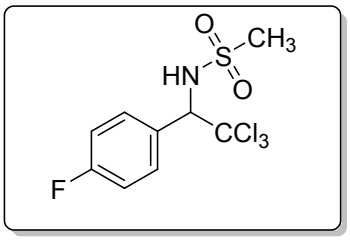


***N*-(2,2,2-trichloro-1-(4-chlorophenyl)ethyl)methanesulfonamide:** The product **3a** was obtained as a white solid (138.2 mg, 82%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.50 (d, 2H, *J* = 8.5 Hz), 7.42 (d, 2H) *J* = 8.5 Hz), 5.85 (d, 1H, *J* = 8.9 Hz), 5.26 (d, 1H, *J* = 8.9 Hz), 2.89 (s, 3H). ^{13}C NMR (75 MHz) δ: 136.0, 133.2, 130.5, 128.9, 100.3, 71.2, 42.5. IR (KBr, cm^{-1}): 3262, 2972, 1503, 1442, 780, 712. HRMS: calcd for $[C_9H_9Cl_4NO_2S]$+ ([M+Na]+): *m/z* 357.90060, found 357.89980.

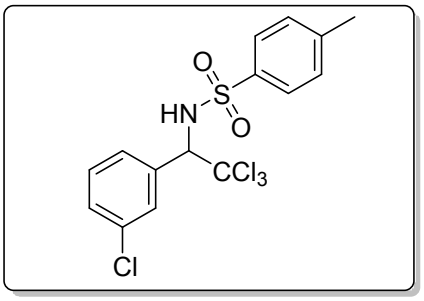


***N*-(2,2,2-trichloro-1-(4-bromophenyl)ethyl)methanesulfonamide:** The product **3b** was obtained as a white solid (135.4 mg, 71%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.59 (d, 2H, *J* = 8.5 Hz), 7.43 (d, 2H, *J* = 8.5 Hz), 5.74 (d, 1H, *J* = 9.2 Hz), 5.25 (d, 1H, *J* = 9.2 Hz), 2.89 (s, 3H). ^{13}C NMR (75 MHz) δ: 133.7, 131.8, 130.7, 124.2, 100.1, 71.2, 42.5. IR (KBr, cm^{-1}): 3271, 2969,

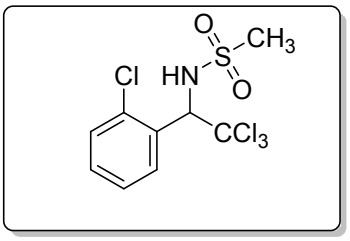
1581, 1494, 824, 787. HRMS: calcd for $[C_9H_9BrCl_3NO_2]$+ ([M+Na]+): *m/z* 401.8501, found 401.84930.



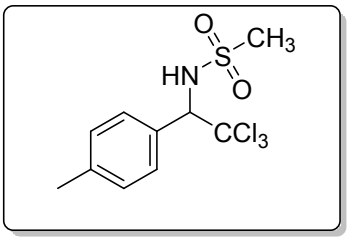
***N*-(2,2,2-trichloro-1-(4-fluorophenyl)ethyl)methanesulfonamide**: The product **3c** was obtained as an yellow oil (110.6 mg, 69%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.58 (dd, *J* = 8.60, 5.2 Hz, 2H), 7.10 (t, *J* = 8.60 Hz, 2H), 6.91 (d, 1H, *J* = 10.1 Hz), 5.25 (d, 1H, *J* = 10.1 Hz), 2.80 (s, 3H); ^{13}C NMR (75 MHz) δ: 163.3 (d, 1F, *J* = 248.5 Hz), 131.3 (d, 1F, *J* = 8.2 Hz), 130.6 (d, 1F, *J* = 3.2 Hz) 115.6 (d, 1F, *J* = 21,8 Hz), 100.8, 71.2, 42.2. IR (KBr, cm^{-1}): 3265, 2982, 1577, 1491, 832. HRMS: calcd for $[C_9H_9Cl_3FNO_2S]$+ ([M+Na]+): *m/z* 341.9301, found 341.9297.



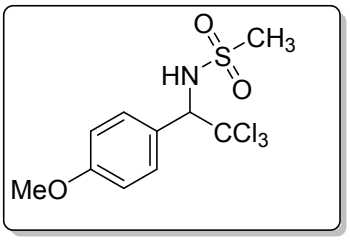
4-methyl-*N*-(2,2,2-trichloro-1-(3-chlorophenyl)ethyl)benzenesulfonamide: The product **3d** was obtained as a white solid (89.3 mg, 53%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.54 (d, 2H, *J* = 8.4 Hz), 7.26-7.23 (m, 2H), 7.19 (d, 1H, *J* = 7.8 Hz), 7.16 (t, 1H, *J* = 1.8 Hz), 7.12 (d, 2H, *J* = 8.4 Hz), 5.72 (d, 1H, *J* = 9.4 Hz), 5.08 (d, 1H, *J* = 9.4 Hz), 2.37 (s, 3H). ^{13}C NMR (125 MHz) δ: 144.0, 136.4, 135.7, 134.0, 129.43, 129.42, 129.3, 129.2, 127.5, 127.0, 99.8, 71.3, 53.4. IR (KBr, cm^{-1}): 3259, 2964, 1593, 1440, 755, 720. HRMS calcd for $[C_{15}H_{13}Cl_4NO_2S]$+ ([M+Na]+): *m/z* 433.9319, found 433.9321.



***N*-(2,2,2-trichloro-1-(2-chlorophenyl)ethyl)methanesulfonamide:** The product **3e** was obtained as yellow oil (87.6 mg, 52%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.73 (t, 1H, *J* = 4,5 Hz), 7.51-7.49 (m, 1H); 7.40-7.38 (m, 2H); 6.06 (d, 1H, *J* = 10.0 Hz), 5.98 (d, 1H, *J* = 10.0 Hz), 2.88 (s, 3H). ^{13}C NMR (125 Hz) δ: 135.4, 133.5, 130.8, 130.1, 128.4, 127.4, 100.5, 66.4, 42.1. IR (KBr, cm^{-1}): 3253, 2969, 1581, 1469, 1442, 776, 760, 725. HRMS: calcd for $[C_9H_9Cl_4NO_2S]$+ ([M+Na]+): *m/z* 357.90060, found 357.90052.

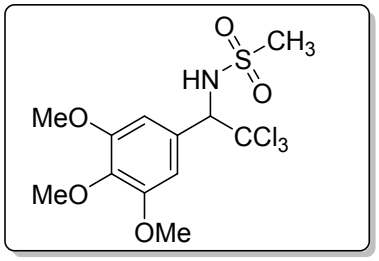


***N*-(2,2,2-trichloro-1-(p-tolyl)ethyl)methanesulfonamide:** The product **3f** was obtained as a white solid (113.9 mg, 72%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.44 (d, 2H, *J* = 8.0 Hz), 7.24 (d, 2H, *J* =8.0 Hz), 6.08 (d,1H, *J* = 9.8 Hz), 5.23 (d, 1H, *J* = 9.9 Hz), 2.79 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (75 MHz) δ: 139.9, 131.6, 129.3, 129.1, 100.9, 71.6, 42.2, 21.3. IR (KBr, cm^{-1}): 3279, 2961, 2909, 1529, 1451, 1327, 1164, 759, 721. HRMS: calcd for $[C_{10}H_{12}Cl_3NO_2S]$+ ([M+Na]+): *m/z* 337.95520, found 337.95405.

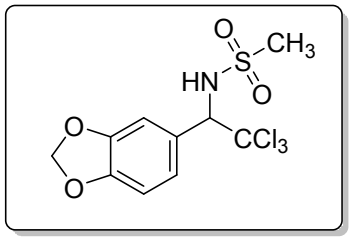


***N*-(2,2,2-trichloro-1-(4-methoxyphenyl)ethyl)methanesulfonamide:** The product **3g** was obtained as a white solid (128.0 mg, 77%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.49 (d, 2H, *J* = 9.0 Hz), 6,94 (d, 2H, *J* = 9.0 Hz), 6.30 (d, 1H, *J* = 10.2 Hz), 5.21 (d, *J* = 10.2 Hz, 1H), 3.84 (s, 3H), 2,79 (s,

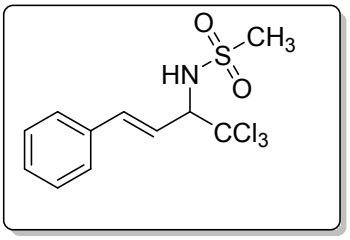
3H). ^{13}C NMR (75 MHz) δ: 160.5, 130.5, 126.4, 114.0, 101.2, 71.4, 55.3, 42.2. IR (KBr, cm^{-1}): 3248, 3012, 2951, 1607, 1567, 1294, 1135, 811, 753, 717. HRMS: calcd for $[C_{10}H_{12}Cl_3NO_3S]+$ ([M+Na]+): *m/z* 353.9485, found 353.9496.



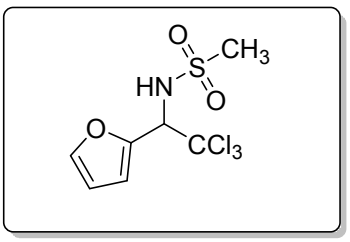
***N*-(2,2,2-trichloro-1-(3,4,5-trimethoxyphenyl)ethyl)methanesulfonamide:** The product **3h** was obtained as a white solid (166.8 mg, 85%). ^1H NMR (300 MHz, $CDCl_3$) δ: 6.85-6,82 (m, 2H), 5.15 (d, 1H, *J* = 10.2Hz), 3.89 (s, 3H), 8.88 (s, 3H), 2.79 (s, 3H), 2.67 (s, 3H) . ^{13}C NMR (75 MHz) δ: 153.1, 139.1, 129.9, 106.8, 101.0, 72.1, 60.9, 56.4, 42.1. IR (KBr, cm^{-1}): 3257, 1576, 1503, 1459, 1419, 816, 790. **HRMS:** calcd for $[C_{12}H_{17}Cl_3NO_5S]+$ ([M+H]+): *m/z* 391.9893, found 391.9884.



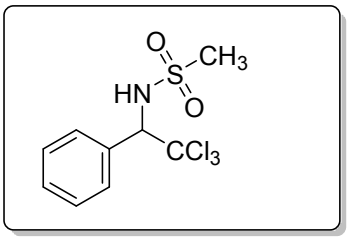
***N*-(1-(benzo[d][1,3]dioxol-5-yl)-2,2,2-trichloroethyl)methanesulfonamide:** The product **3i** was obtained as yellow oil (114.3 mg, 66%). ^1H NMR (300 MHz, $CDCl_3$) δ: 7.06-7.01 (m, 2H); 6.85 (d, 1H, *J* = 8.0 Hz), 6.10 (d, 1H, *J* = 9.8Hz), 6.04 (s, 2H), 5.18 (d, 1H, *J* = 9.8 Hz), 2.86 (s, 3H). ^{13}C NMR (75 MHz) δ: 148.8, 147.9, 128.2, 123.7, 109.0, 108.2, 101.6, 100.9, 71.6, 42.3. IR (KBr, cm^{-1}): 3253, 2934, 2891, 1503, 1452, 1253, 1149, 766, 730. HRMS: calcd for $[C_{10}H_{10}Cl_3NO_4S]+$ ([M+Na]+): *m/z* 367.92940, found 367.92819.



***(E)-N*-(1,1,1-trichloro-4-phenylbut-3-en-2-yl)methanesulfonamide:** The product **3j** was obtained as yellow oil (95.3 mg, 58%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.47-7.45 (m, 2H), 7.40-7.33 (m, 3H); 6.90 (d, 1H, J = 15.9 Hz), 6.38 (dd, 1H, J = 15.9, J = 7.1 Hz), 5.73 (d, J = 9.1 Hz, 1H), 4.89 (dd, 1H, J = 9.1, J = 7.1 Hz), 3.12 (s, 3H); ^{13}C NMR (75 MHz) δ: 137.5, 135.2, 129.0, 128.8, 127.0, 122.0, 101.2, 70.9, 42.8. IR (KBr, cm^{-1}): 3253, 2846, 1520, 1442, 758, 721. HRMS: calcd for $[C_{11}H_{12}Cl_3NO_2S]$+ ([M+Na]+): *m/z* 349.95570, found 349.95465.

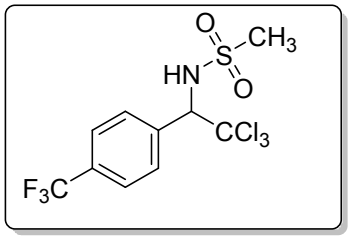


***N*-(2,2,2-trichloro-1-(furan-2-yl)ethyl)methanesulfonamide:** The product **3k** was obtained as a brown oil (83.3 mg, 57%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.51-7.50 (m, 1H), 6.64 (d, 1H, J = 3.1 Hz), 6.45 (dd, 1H, J = 3.1 Hz, J = 1.9 Hz), 5.71 (d, 1H J = 10.2 Hz), 5.36 (d, 1H, J = 10.2 Hz), 2.94 (s, 3H). ^{13}C NMR (75 MHz) δ: 147.1, 143.8, 112.2, 111.2, 99.7, 66.5, 42.3. IR (KBr, cm^{-1}): 3253, 2926, 2849, 1520, 1442, 1331, 1154, 750, 729. HRMS: calcd for $[C_7H_8Cl_3NO_3S]$+ ([M+Na]+): *m/z* 313.9188, found 313.9190.

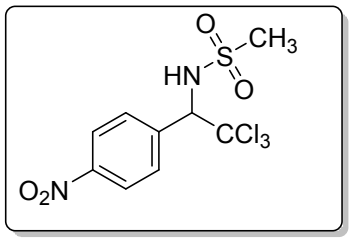


***N*-(2,2,2-trichloro-1-phenylethyl)methanesulfonamide:** The product **3l** was obtained as a white solid (131.6 mg, 87%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.59-7.56 (m, 2H), 7.45-7.44 (m, 3H), 6.26 (d, 1H, J = 10.2 Hz,), 5.27 (d, 1H, J = 10.2 Hz), 2.80 (s, 3H); ^{13}C NMR (75 MHz) δ: 134.7, 129.9, 129.4, 128.8, 100.9, 72.0, 42.4. IR (KBr, cm^{-1}): 3297,

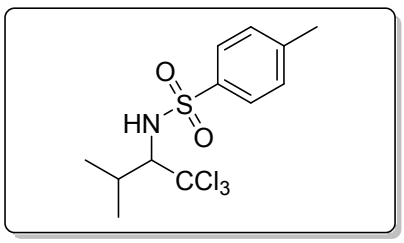
2969, 2944, 1503, 1460, 1339, 872, 787, 749. HRMS: calcd for $[C_9H_{10}Cl_3NO_2S]+$ ([M+Na]+): *m/z* 323.93960, found 323.93855.



***N*-(2,2,2-trichloro-1-(4-(trifluoromethyl)phenyl)ethyl)methanesulfonamide:** The product **3m** was obtained as a white solid yellow oil (144.4 mg, 78%). ^{1}H NMR (500 MHz, $CDCl_3$) δ : 7.71 (s, 4H); 5.91 (d, *J* = 9.3 Hz, 1H), 5.36 (d, *J* = 9.3 Hz, 1H), 2.93 (s, 3H).^{13}C NMR (125 MHz) δ: 138.6, 131.7 (q, *J* = 32.5 Hz), 129.7, 126.9, 125.6 (q, *J* = 270 Hz), 100.0, 71.3, 42.6. IR (KBr, cm^{-1}): 3279, 2969, 1641, 1460, 1321, 1174, 871, 817, 737, 619. HRMS: calcd for $[C_{10}H_9Cl_3F_3NO_2S]+$ ([M+Na]+): *m/z* 391.9269, found 391.9272.



***N*-(2,2,2-trichloro-1-(4-nitrophenyl)ethyl)methanesulfonamide:** The product **3n** was obtained as a yellow oil (147.7 mg, 85%); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.31 (d, 2H, *J* = 8.5 Hz), 7.78 (d, 2H, *J* = 8.5 Hz); 5.85 (d, 1H, *J* = 9.5 Hz), 5.42 (d, 1H, *J* = 9.5 Hz), 3.00 (s, 3H); ^{13}C NMR (125 MHz) δ: 148.6, 141.6, 130.4, 123.7, 99.5, 71.0, 42.6. IR (KBr, cm^{-1}): 3230, 3009, 2932, 1598, 1529, 1316, 992, 776, 739. HRMS: calcd for $[C_9H_9Cl_3N_2O_4S]+$ ([M+Na]+): *m/z* 368.9246, found 368.9249.

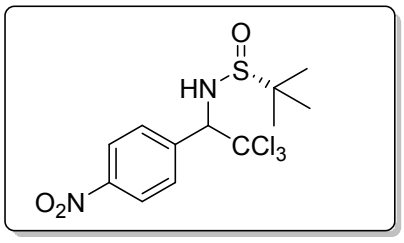


4-methyl-N-(1,1,1-trichloro-3-methylbutan-2-yl)benzenesulfonamide: The product **3o** was obtained

as a white solid (56.4 mg, 42%); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.81 (d, 2H, *J* = 8.2 Hz); 7.31 (d, 2H, *J* = 8.2 Hz), 4.96 (d, 1H *J* = 10.3 Hz), 4.11 (dd, 1H, *J* = 10.3 Hz, *J* = 2.0 Hz), 2.66-2.60 (m, 1H), 2.44 (s, 3H), 1.09 (d, 3H, *J* = 6.8 Hz), 1.03 (d, 3H, *J* = 6.8 Hz). ^{13}C NMR (125 MHz) δ: 143.6, 138.3, 129.5, 127.2, 102.4, 72.8, 29.9, 22.9, 21.5, 17.2. IR (KBr, cm^{-1}): 3199, 1452, 1312, 721. HRMS: calcd for $[C_{12}H_{16}Cl_3NO_2S]$+ ([M+Na]+): *m/z* 365.9865, found 365.9875.

4. General procedure and characterization data for the chiral trichloromethyl sulfinimide (5)

To a solution of imine (50.8 mg, 0.2 mmol) in DMSO *d6* (0.7 $mmol.mL^{-1}$) was added potassium trichloroacetate salt (161.2 mg, 0.8 mmol) and stirred at room temperature for 40 minutes. After completion, it was added dichloromethane (5 mL) and the solution was extracted with water (4 x 10 mL). The aqueous phases was extracted with AcOEt (2 x 20 mL). The volatiles was dried with anhydrous sodium sulfate and evaporated under reduced pressure. The product was obtained after purification through chromatography column (elution: ethyl acetate/ hexanes, 3:1).

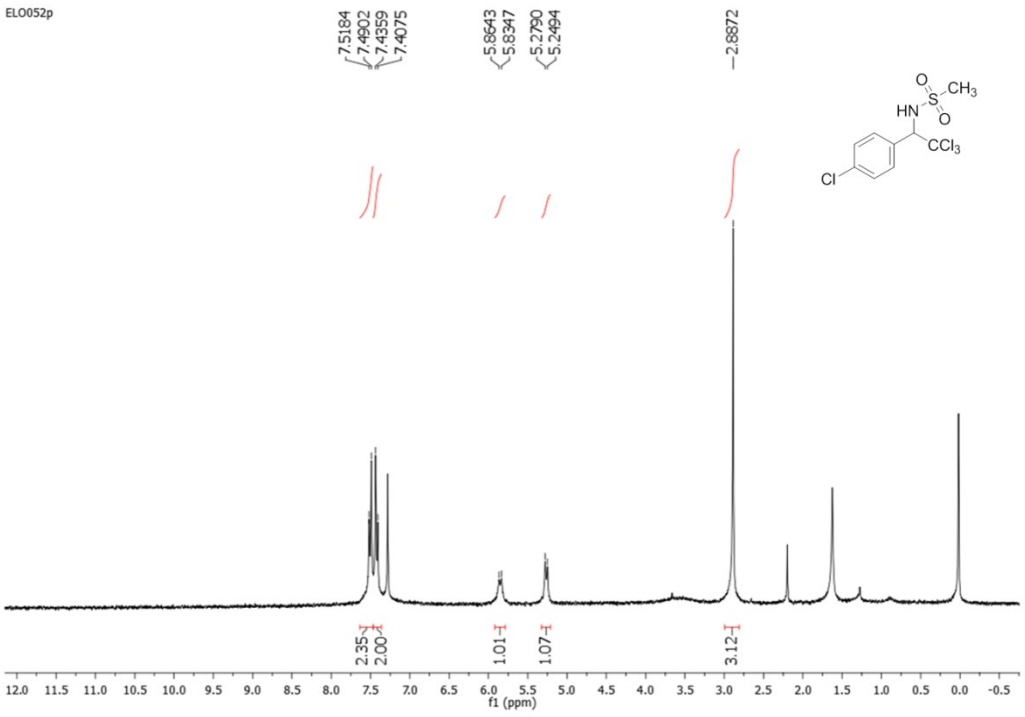


(R)-2-methyl-N-(2,2,2-trichloro-1-(4-nitrophenyl)ethyl)propane-2-sulfinamide

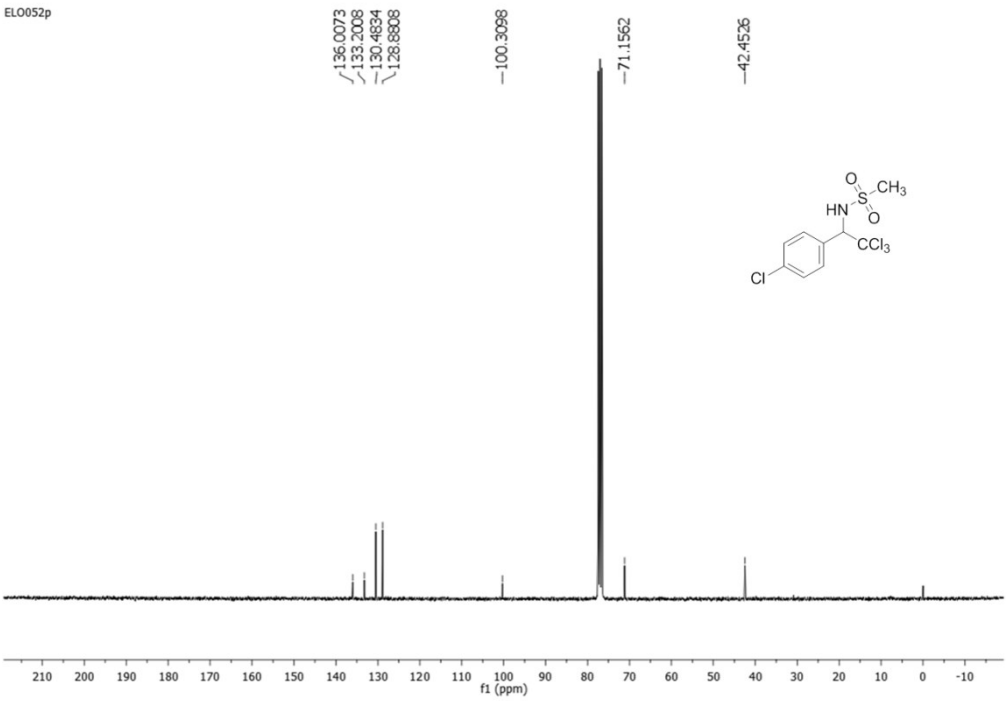
The product **5** was obtained as a white solid (56.4 mg, 75%). ^{1}H NMR (200 MHz, $CDCl_3$) δ: 8.28 (d, 2H, *J* = 8.0 Hz); 7.76 (d, 2H, *J* = 8.0 Hz), 5,16 (d, 1H, *J* = 8.9 Hz), 4.25 (d, 1H, *J* = 8.9 Hz), 1.31

(s, 9H). ^{13}C NMR (50 MHz) δ: 148.5, 143.0, 130.3, 123.8, 101.3, 74.1, 57.9, 22.7. HRMS: calcd for $[C_{12}H_{16}Cl_3N_2O_3S]$+ ([M+H]+): *m/z* 372.9947, found 372.9933.

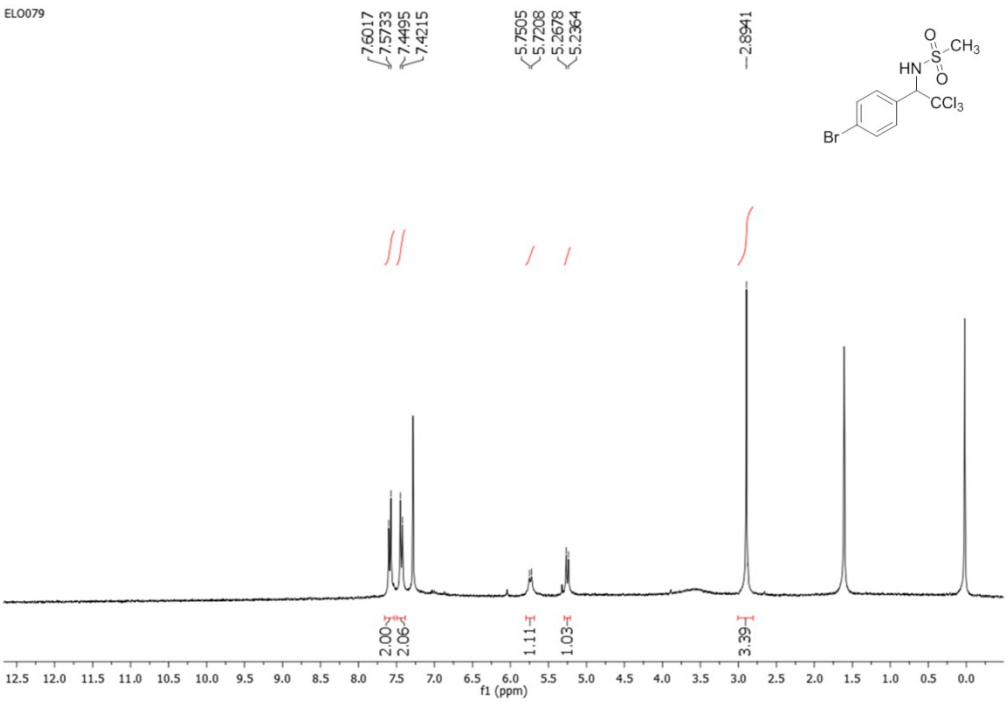
5. ^{1}H and ^{13}C NMR spectra of trichloromethyl sulfonamide and sulfinamide derivatives



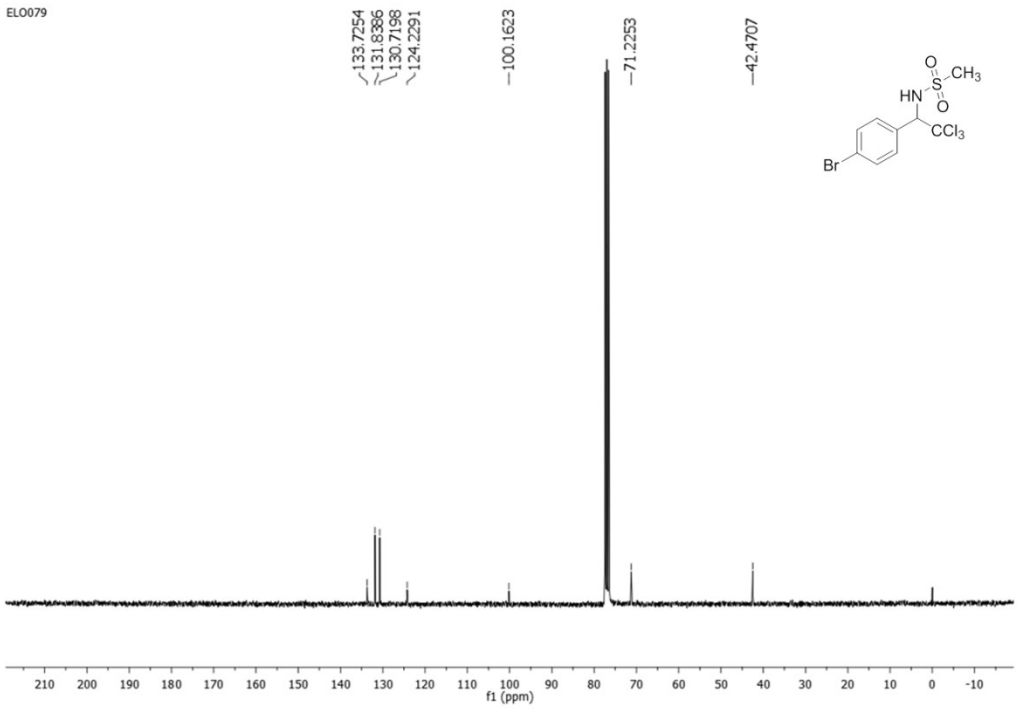
^{1}H NMR spectrum of **3a** (300 MHz, $CDCl_3$)



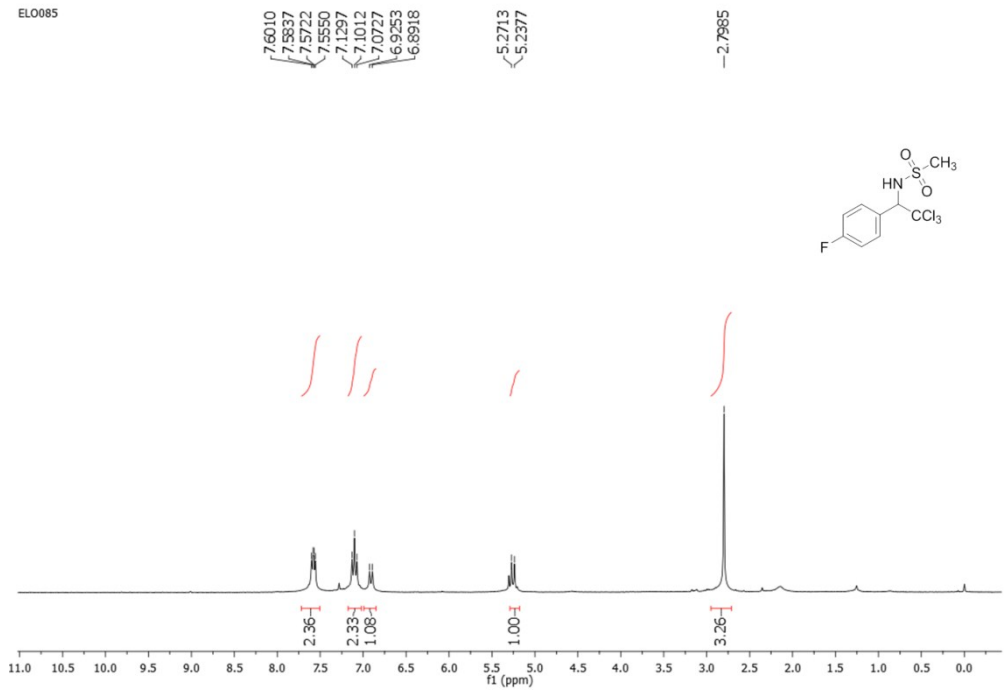
^{13}C NMR spectrum of **3a** (75 MHz, $CDCl_3$)



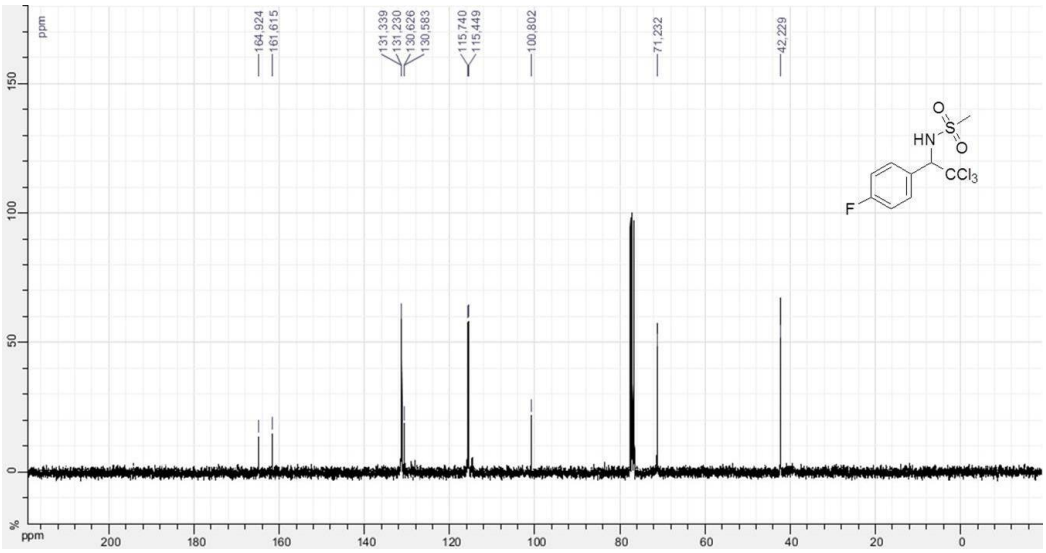
^{1}H NMR spectrum of **3b** (300 MHz, $CDCl_3$)



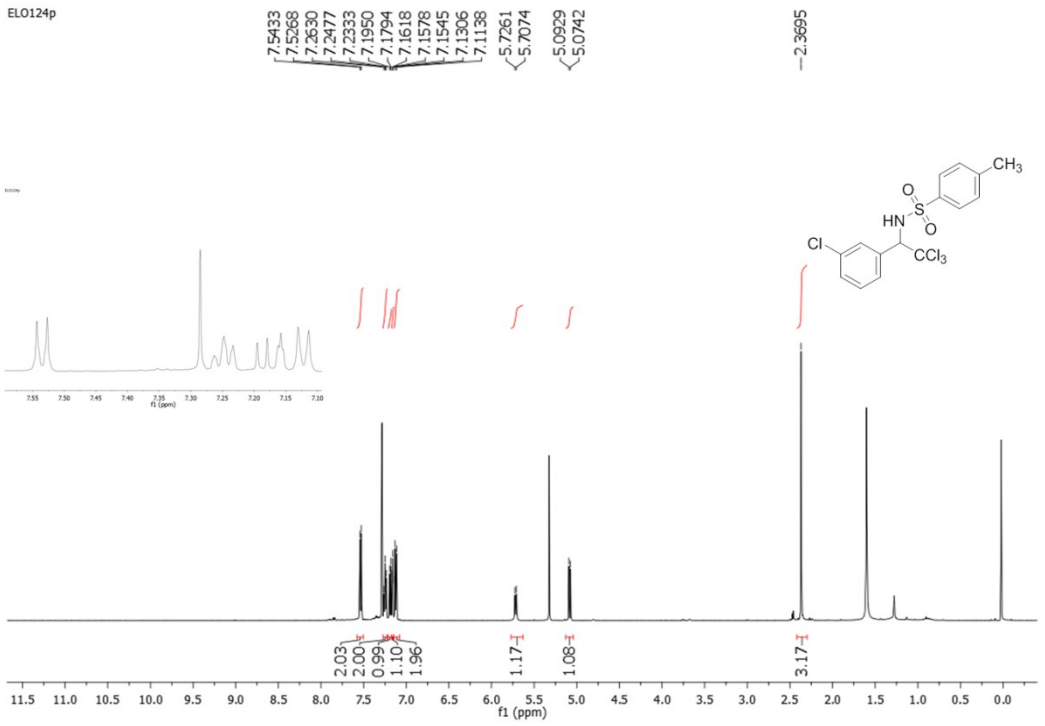
^{13}C NMR spectrum of **3b** (75 MHz, $CDCl_3$)



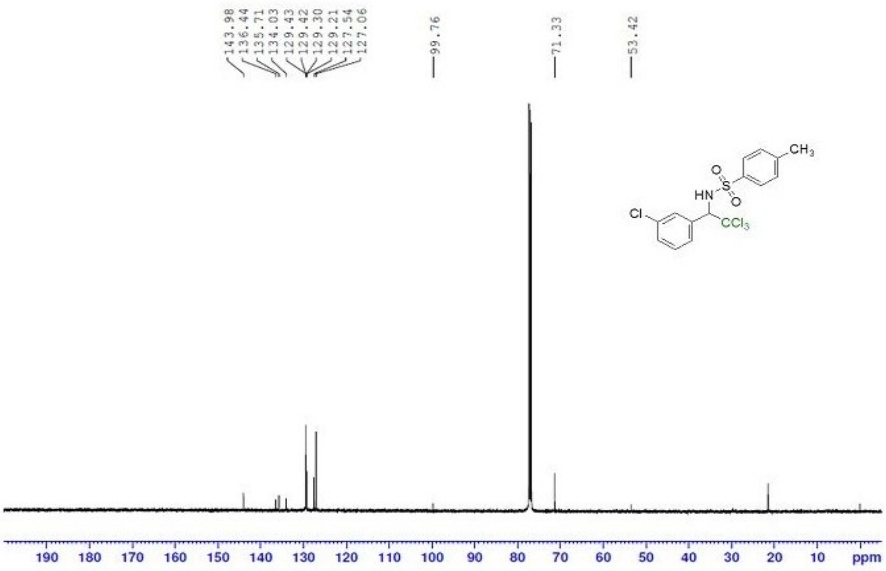
^{1}H NMR spectrum of **3c** (300 MHz, $CDCl_3$)



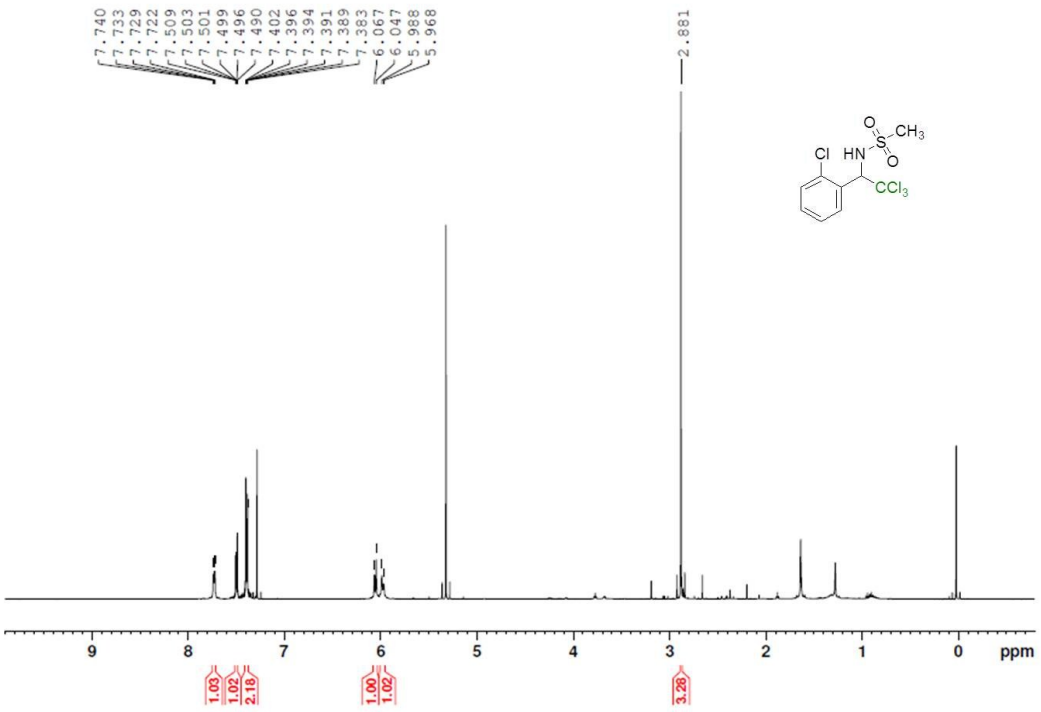
^{13}C NMR spectrum of **3c** (75 MHz, $CDCl_3$)



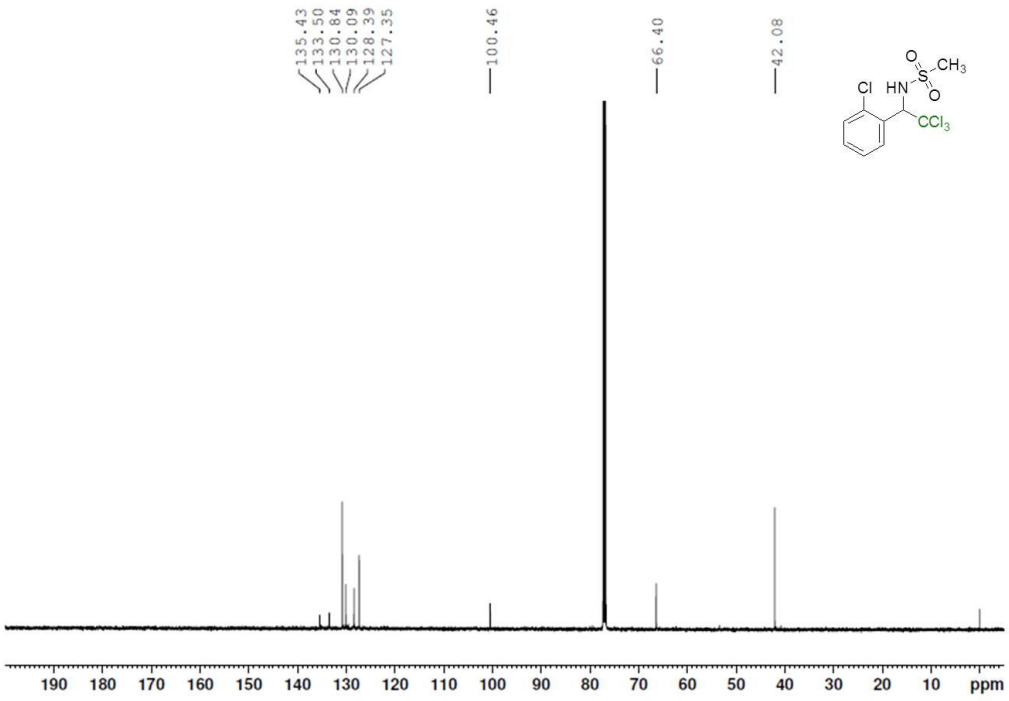
^{1}H NMR spectrum of **3d** (500 MHz, $CDCl_3$)



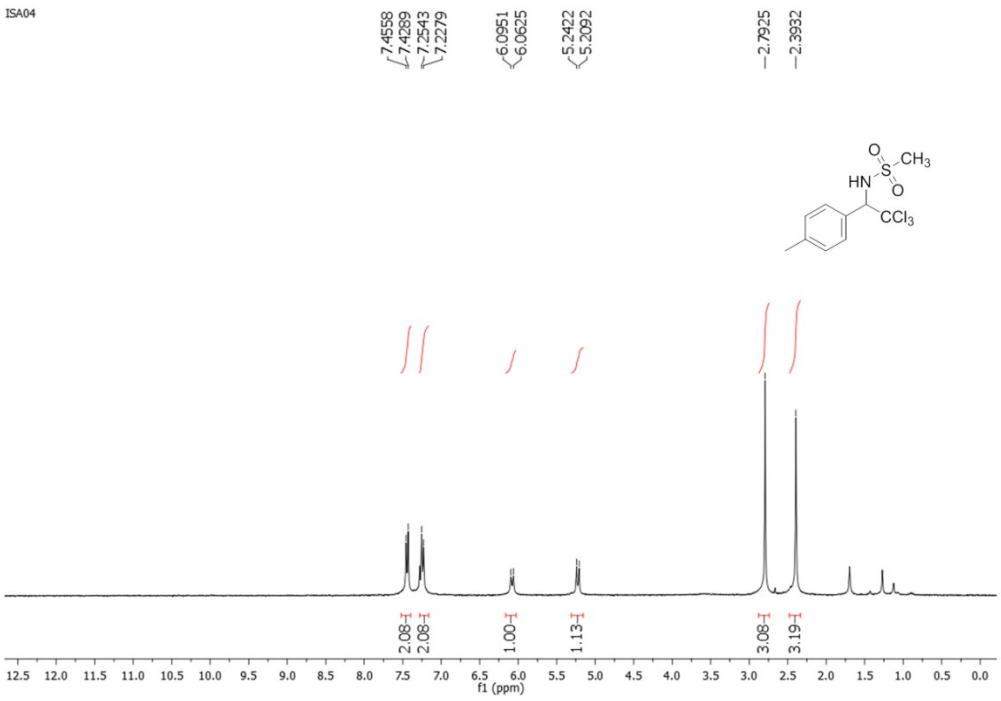
^{13}C NMR spectrum of **3d** (125 MHz, $CDCl_3$)



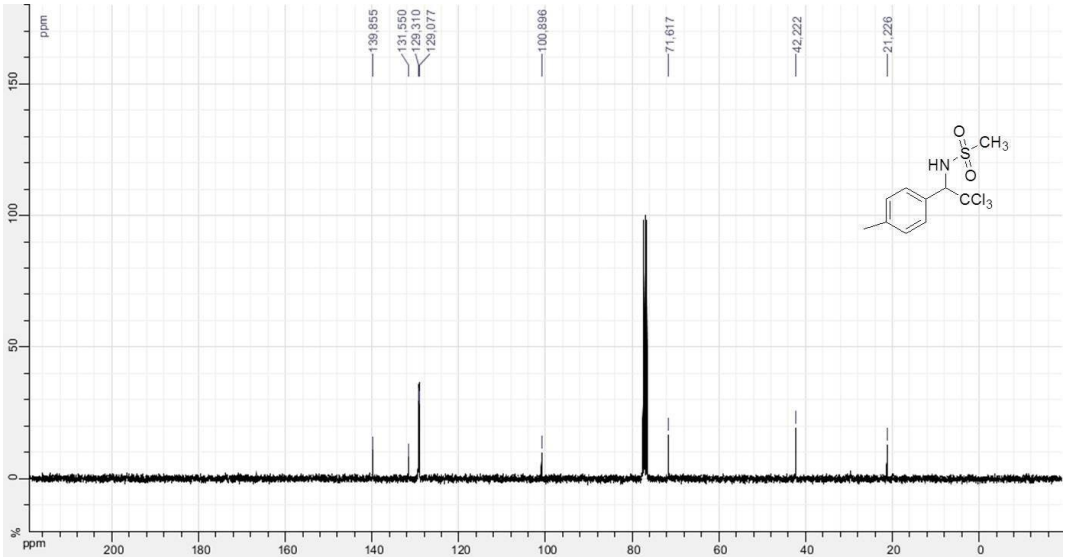
^{1}H NMR spectrum of **3e** (500 MHz, $CDCl_3$)



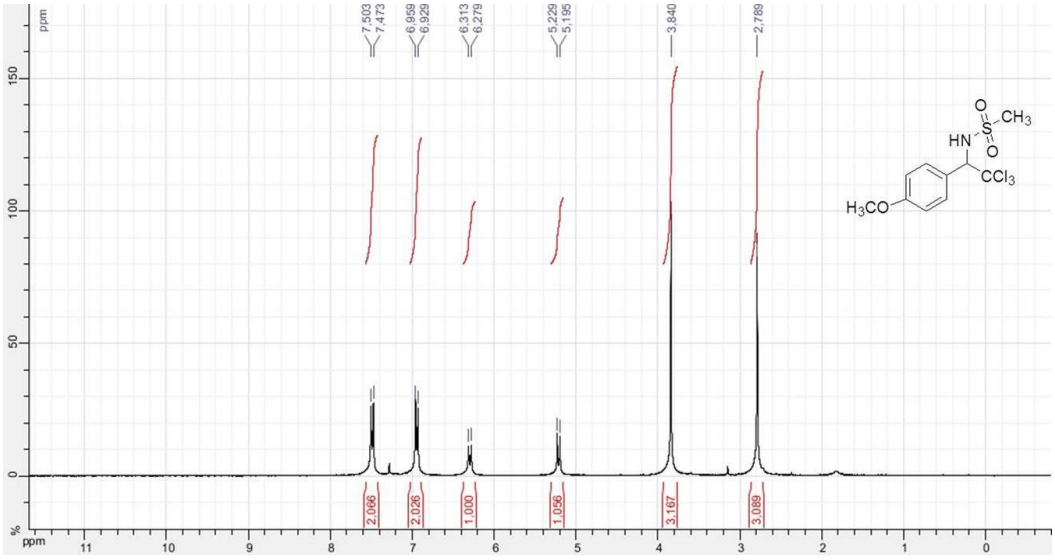
^{13}C NMR spectrum of **3e** (125 MHz, $CDCl_3$)



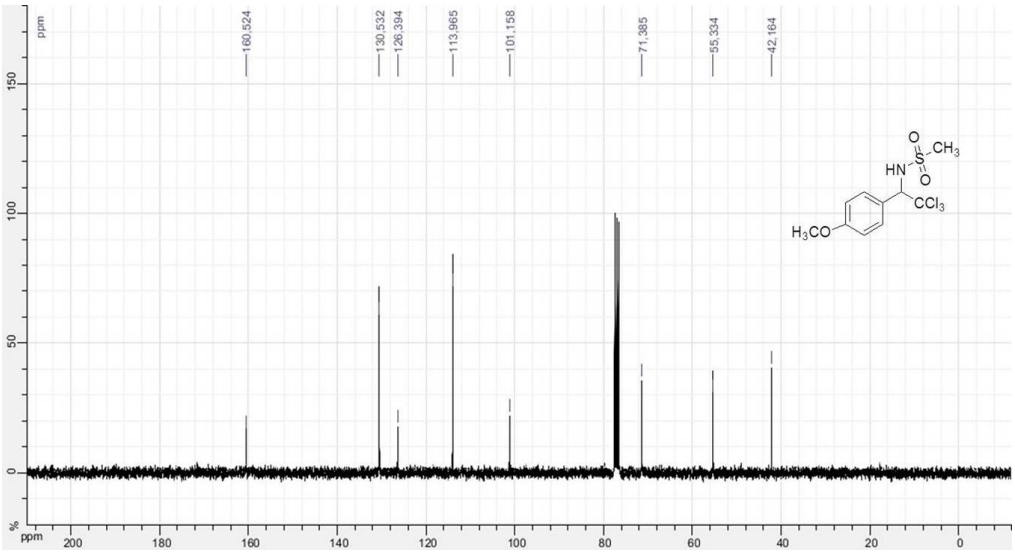
^{1}H NMR spectrum of **3f** (300 MHz, $CDCl_3$)



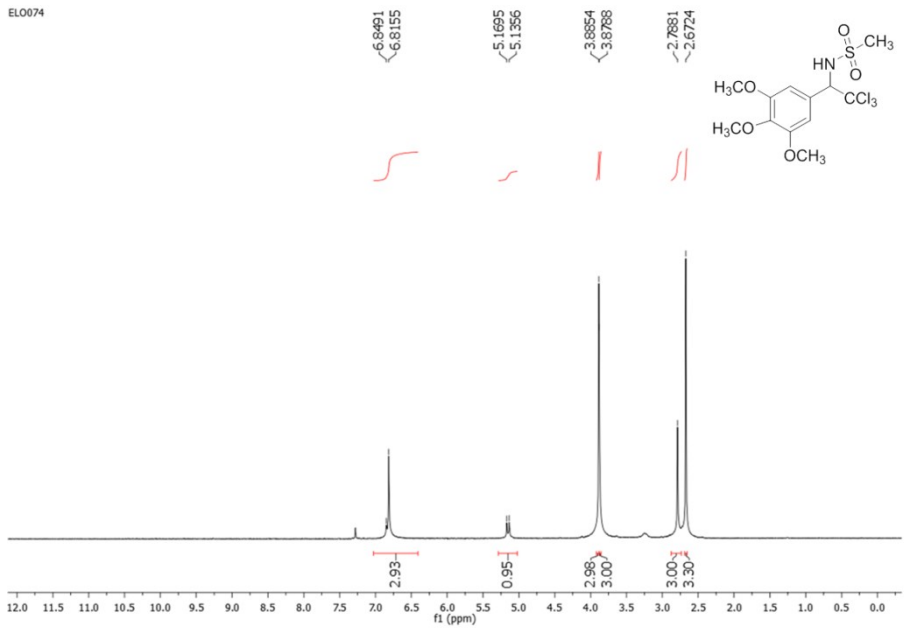
^{13}C NMR spectrum of **3f** (75 MHz, $CDCl_3$)



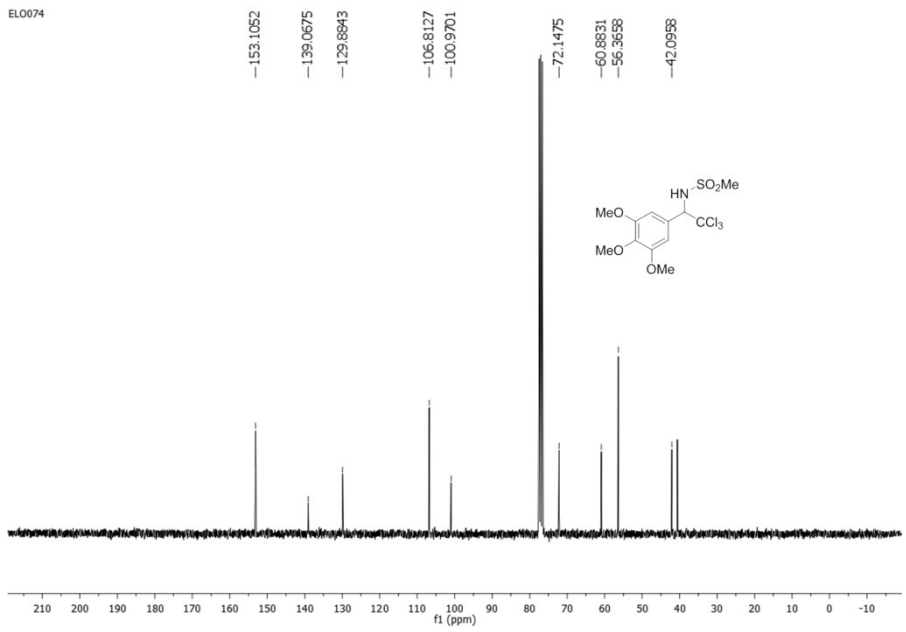
^{1}H NMR spectrum of **3g** (300 MHz, $CDCl_3$)



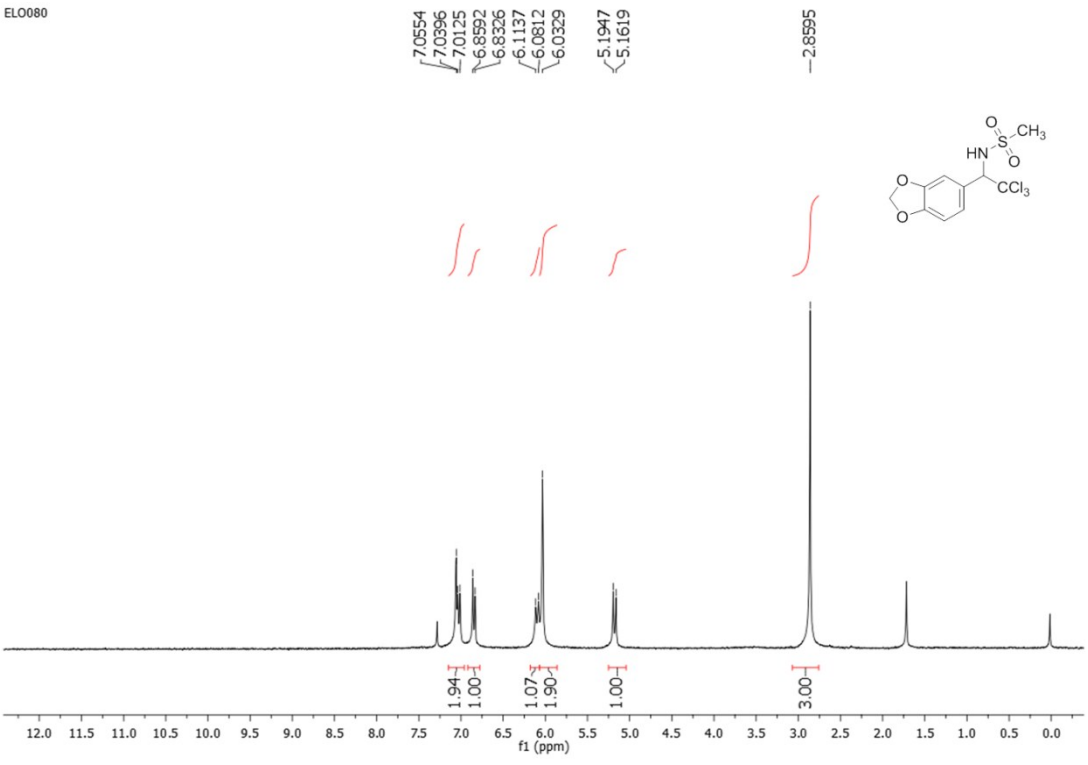
^{13}C NMR spectrum of **3g** (75 MHz, $CDCl_3$)



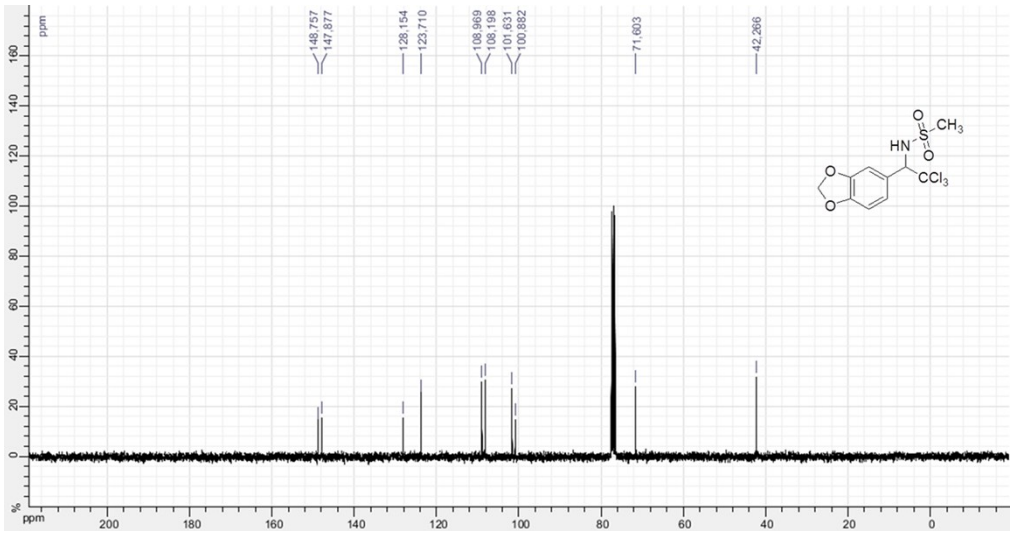
^{1}H NMR spectrum of **3h** (300 MHz, $CDCl_3$)



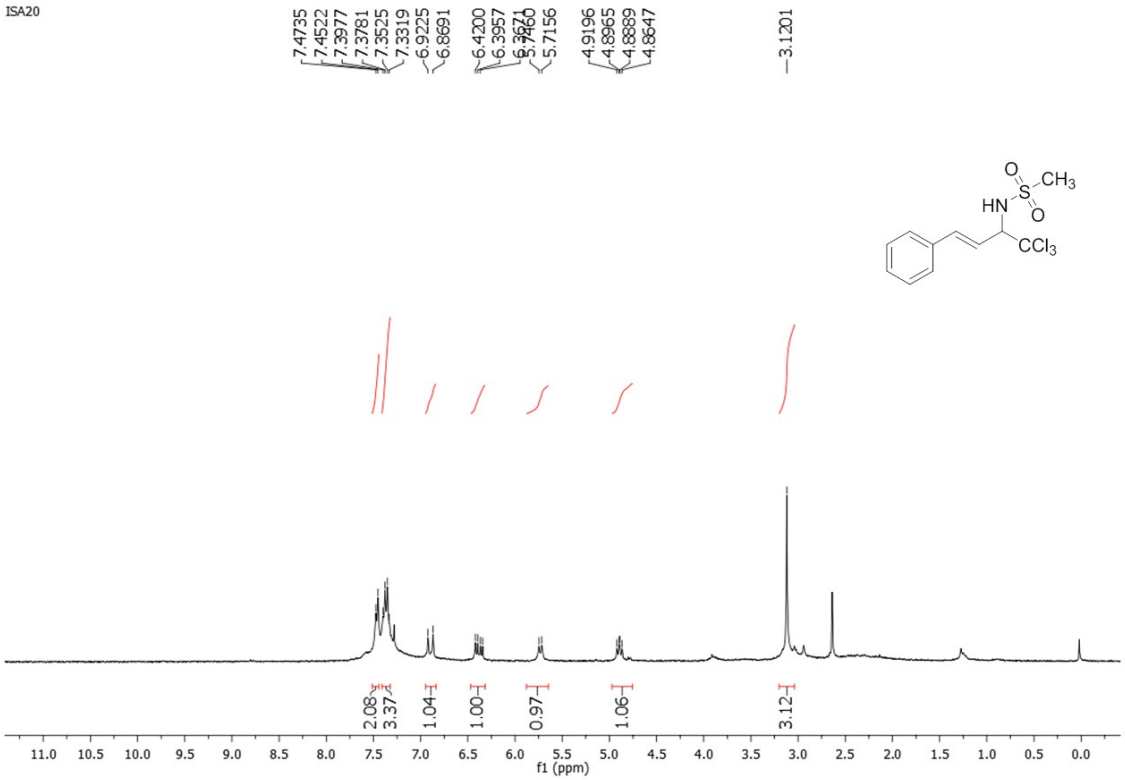
^{13}C NMR spectrum of **3h** (75 MHz, $CDCl_3$)



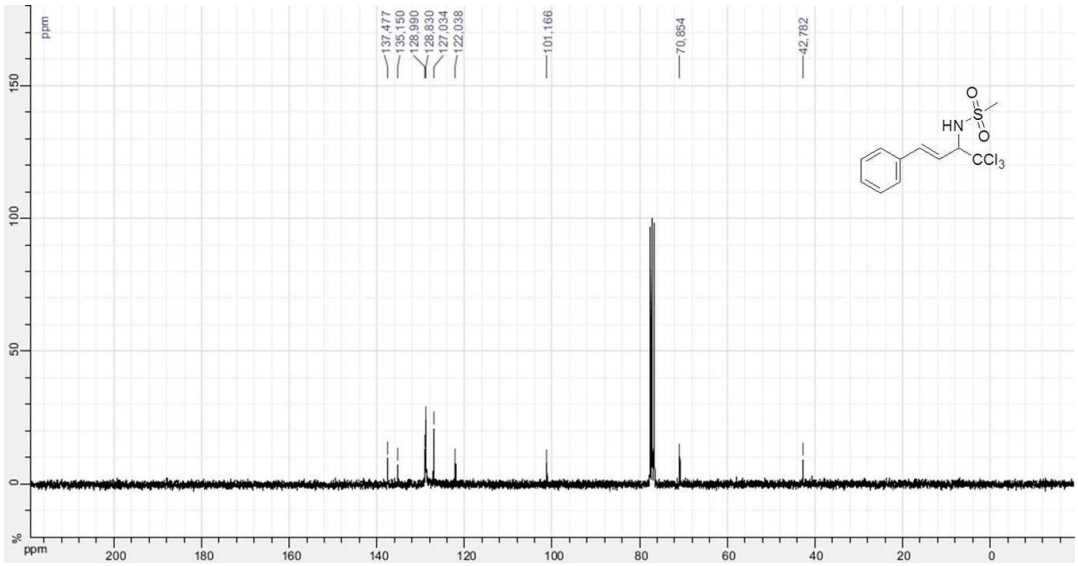
^{1}H NMR spectrum of **3i** (300 MHz, $CDCl_3$)



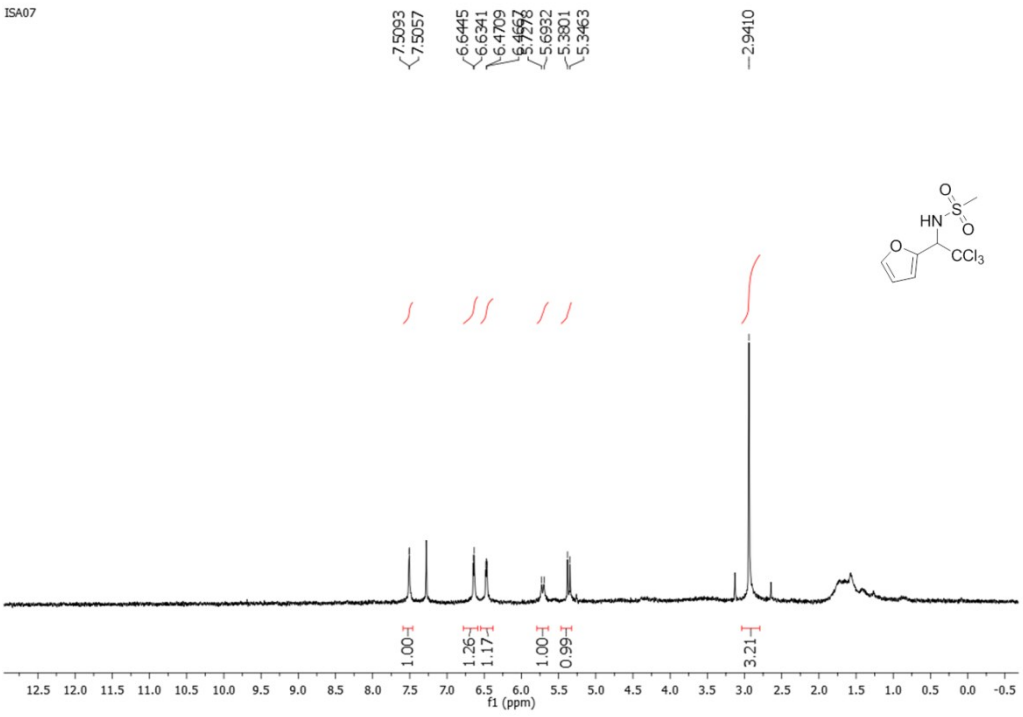
^{13}C NMR spectrum of **3i** (75 MHz, $CDCl_3$)



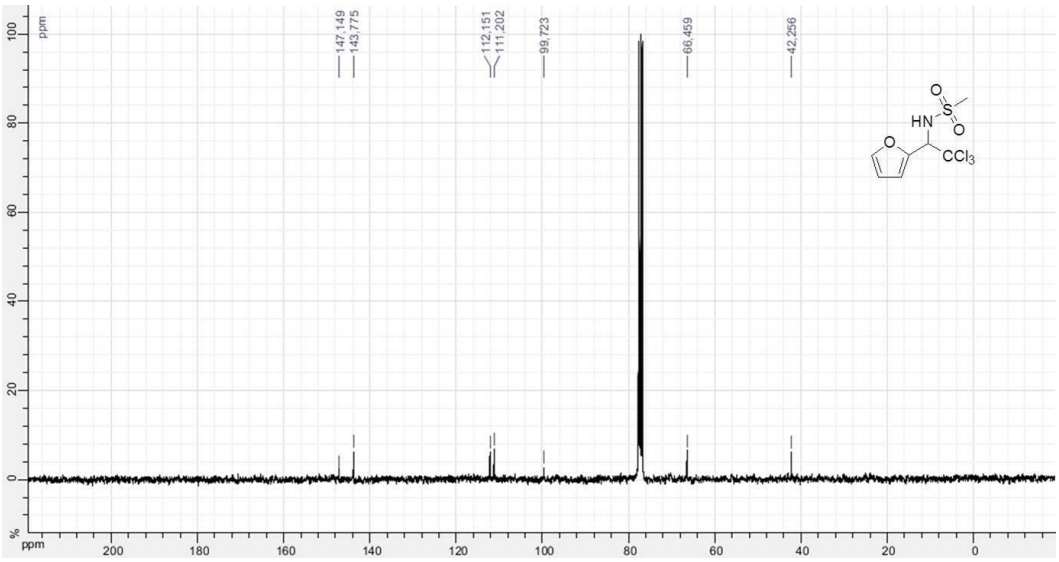
^{1}H NMR spectrum of **3j** (300 MHz, $CDCl_3$)



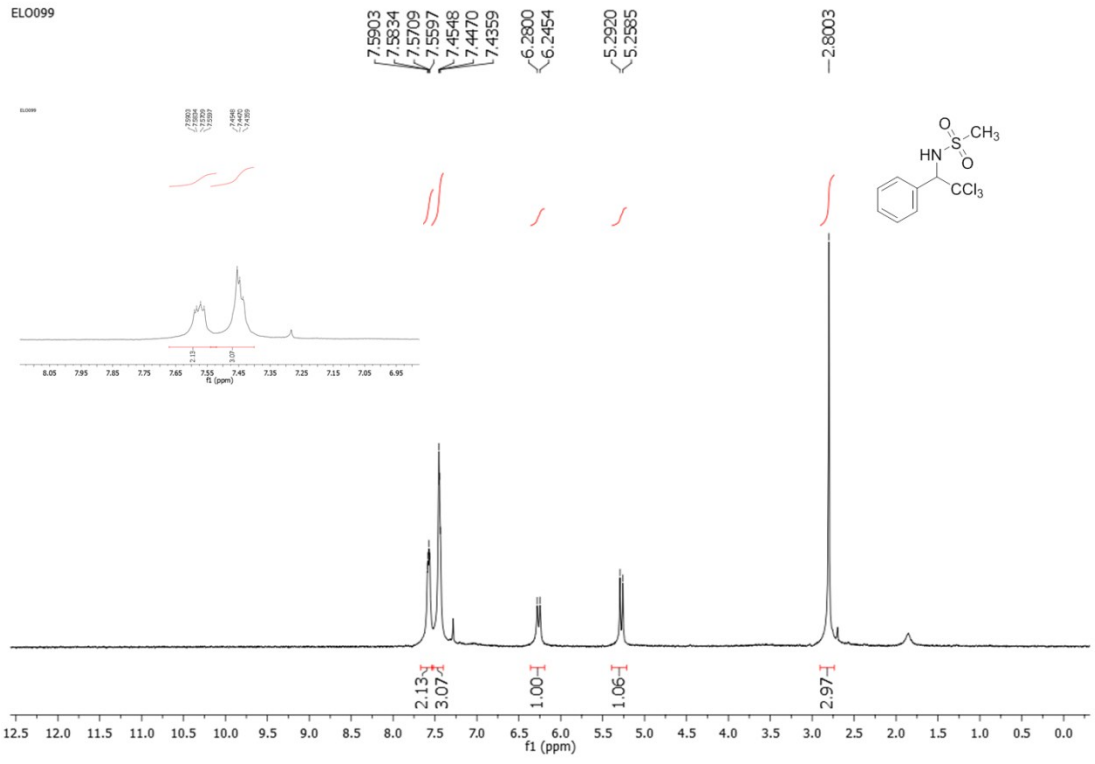
^{13}C NMR spectrum of **3j** (75 MHz, $CDCl_3$)



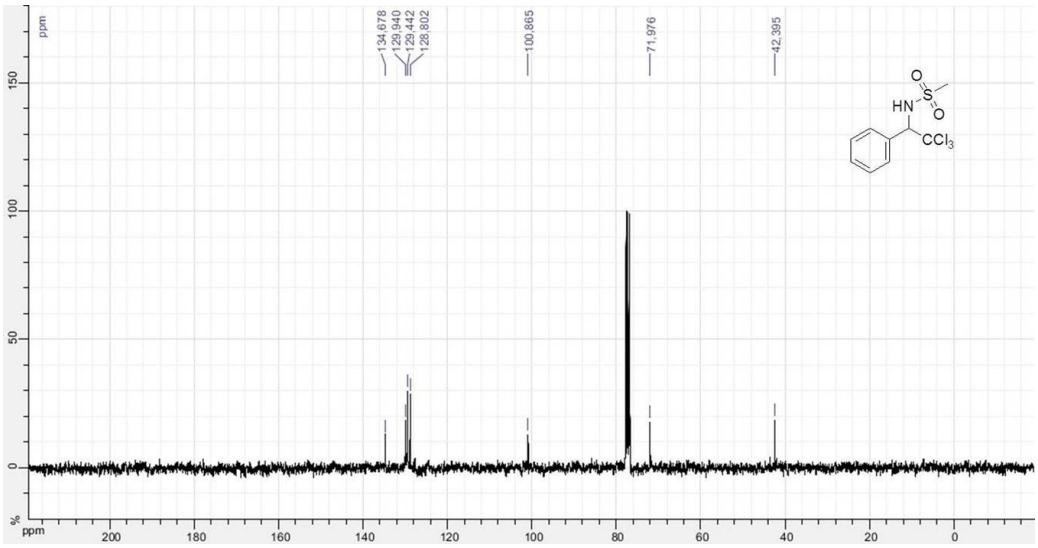
^{1}H NMR spectrum of 3**k** (300 MHz, $CDCl_3$)



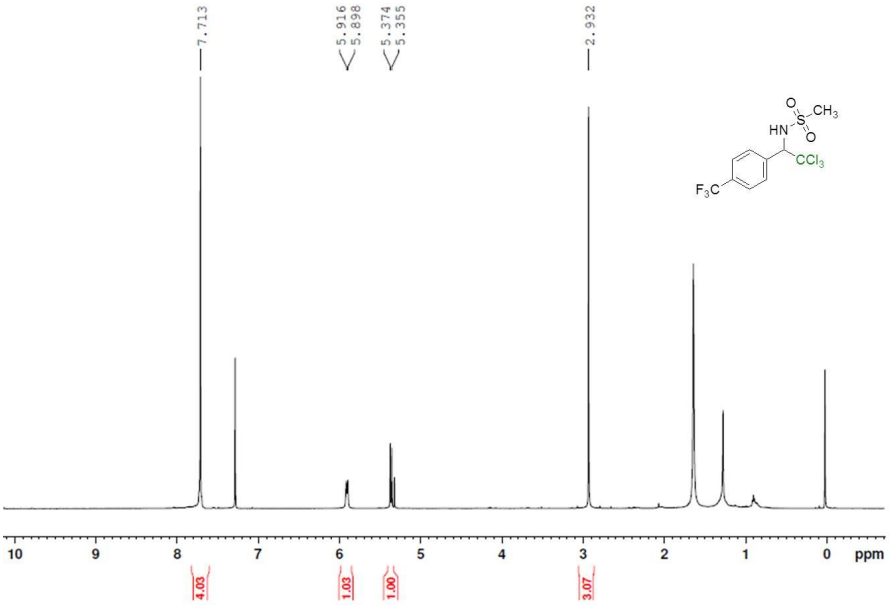
^{13}C NMR spectrum of **3k** (75 MHz, $CDCl_3$)



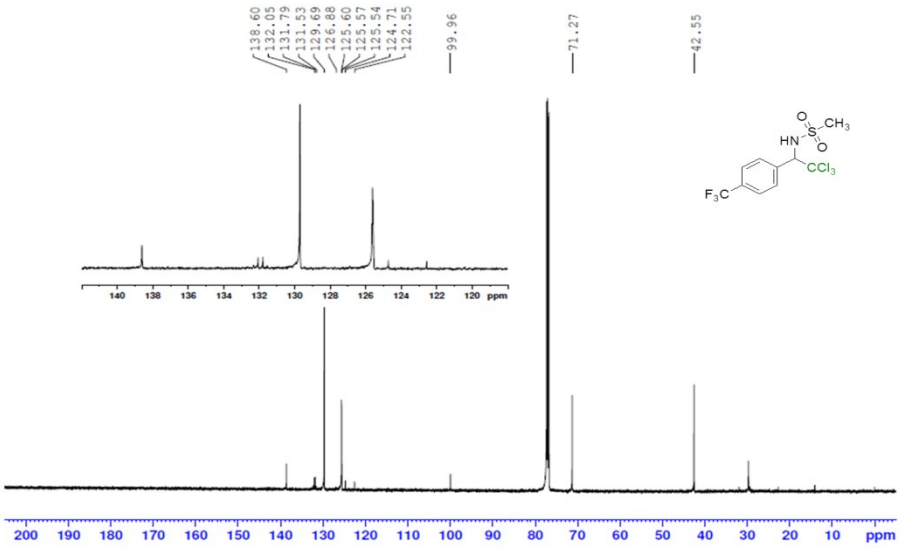
^{1}H NMR spectrum of **3l** (300 MHz, $CDCl_3$)



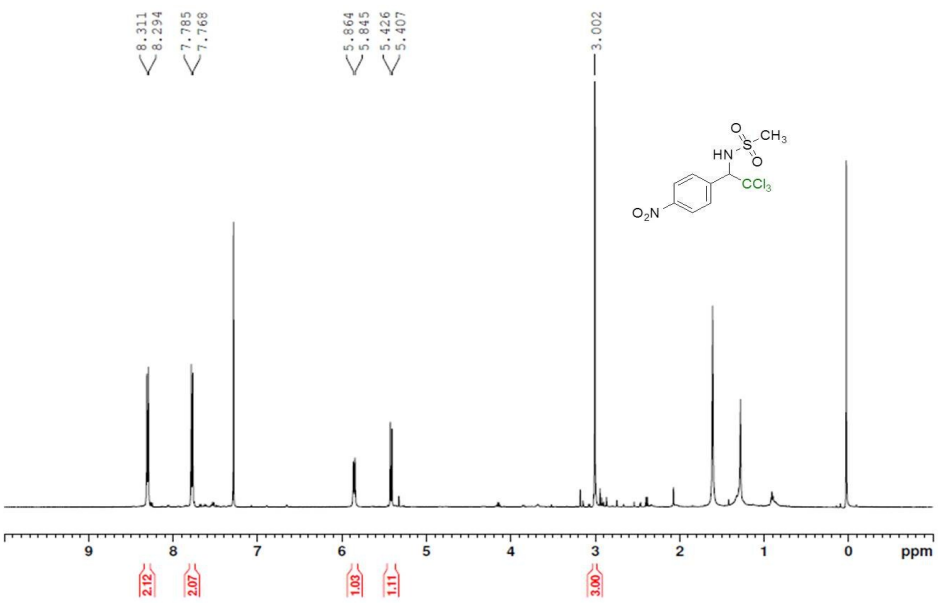
^{13}C NMR spectrum of **3l** (75 MHz, $CDCl_3$)



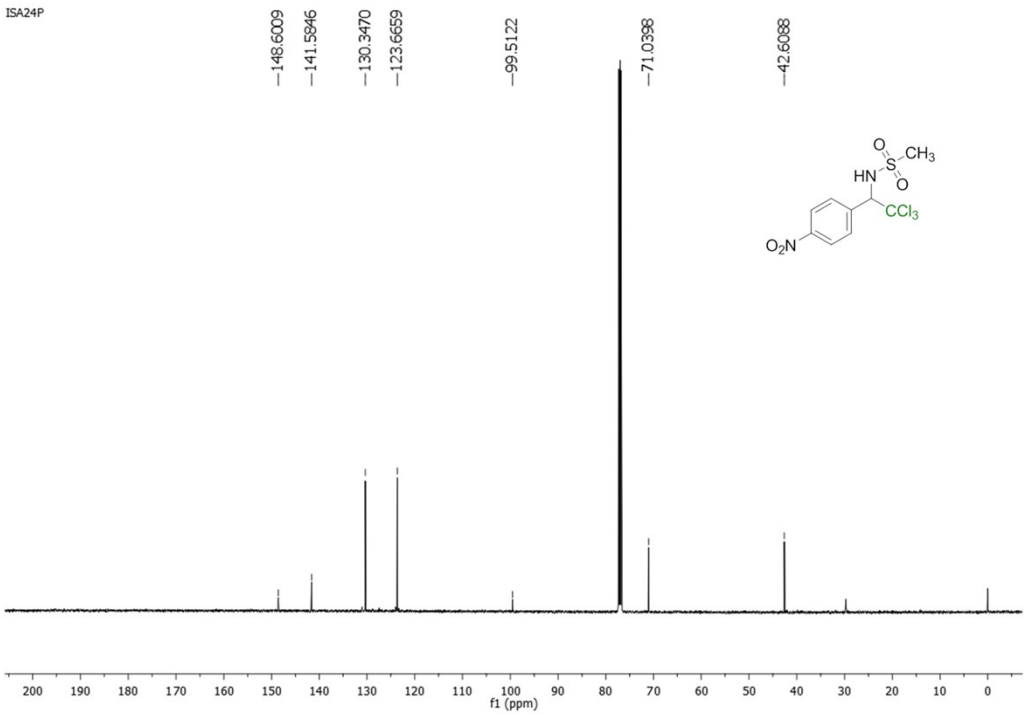
^{1}H NMR spectrum of **3m** (500 MHz, $CDCl_3$)



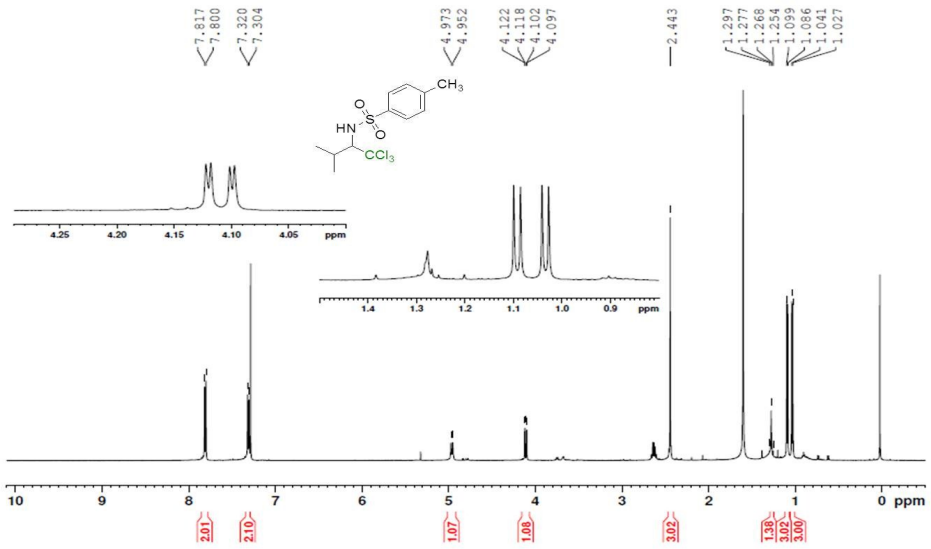
^{13}C NMR spectrum of **3m** (75 MHz, $CDCl_3$)



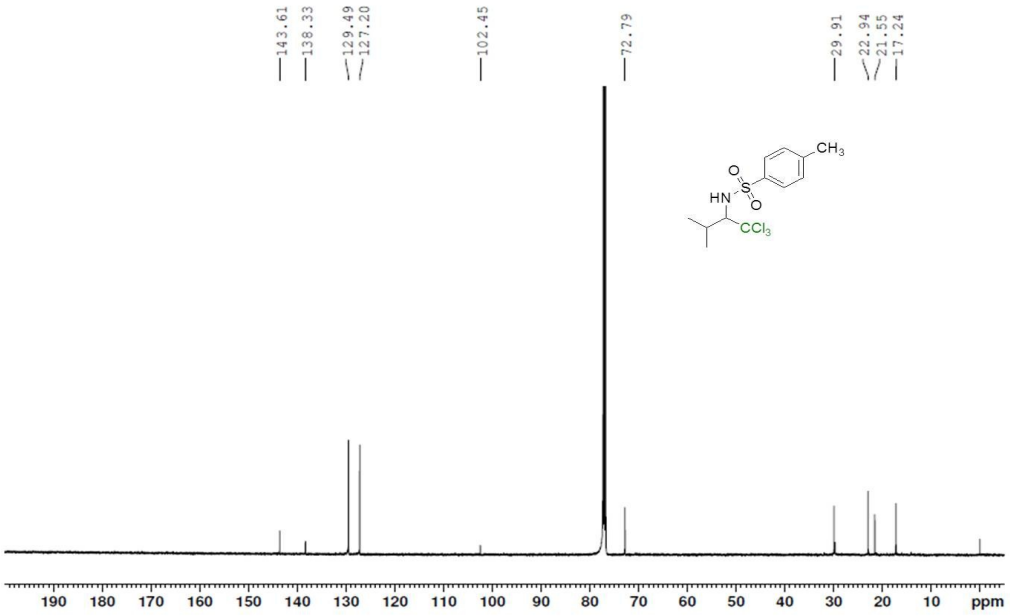
^{1}H NMR spectrum of **3n** (500 MHz, $CDCl_3$)



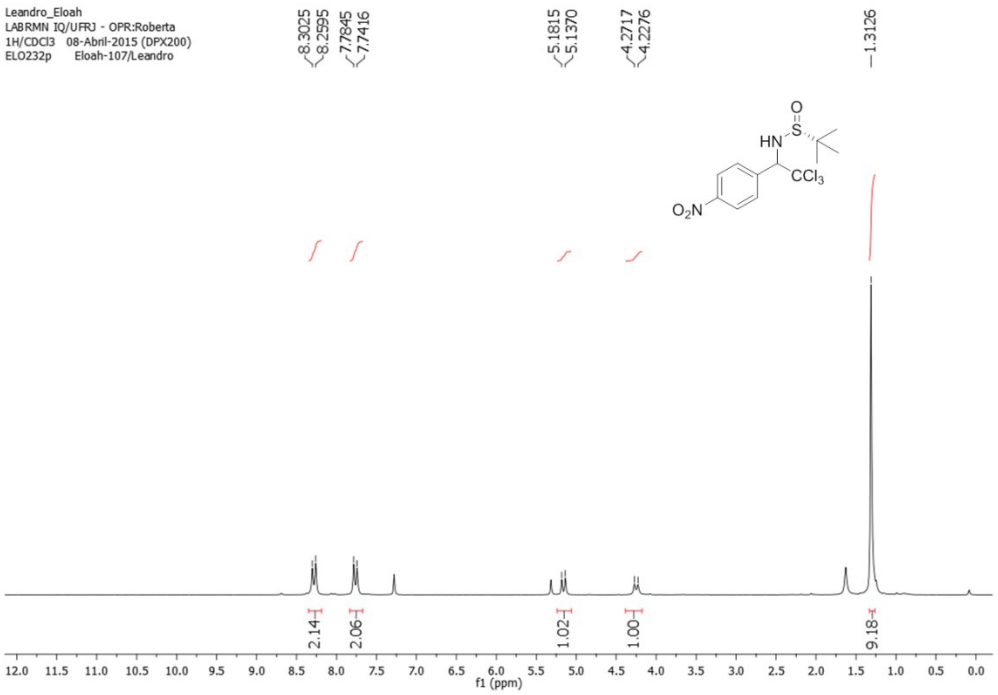
^{13}C NMR spectrum of **3n** (125 MHz, $CDCl_3$)



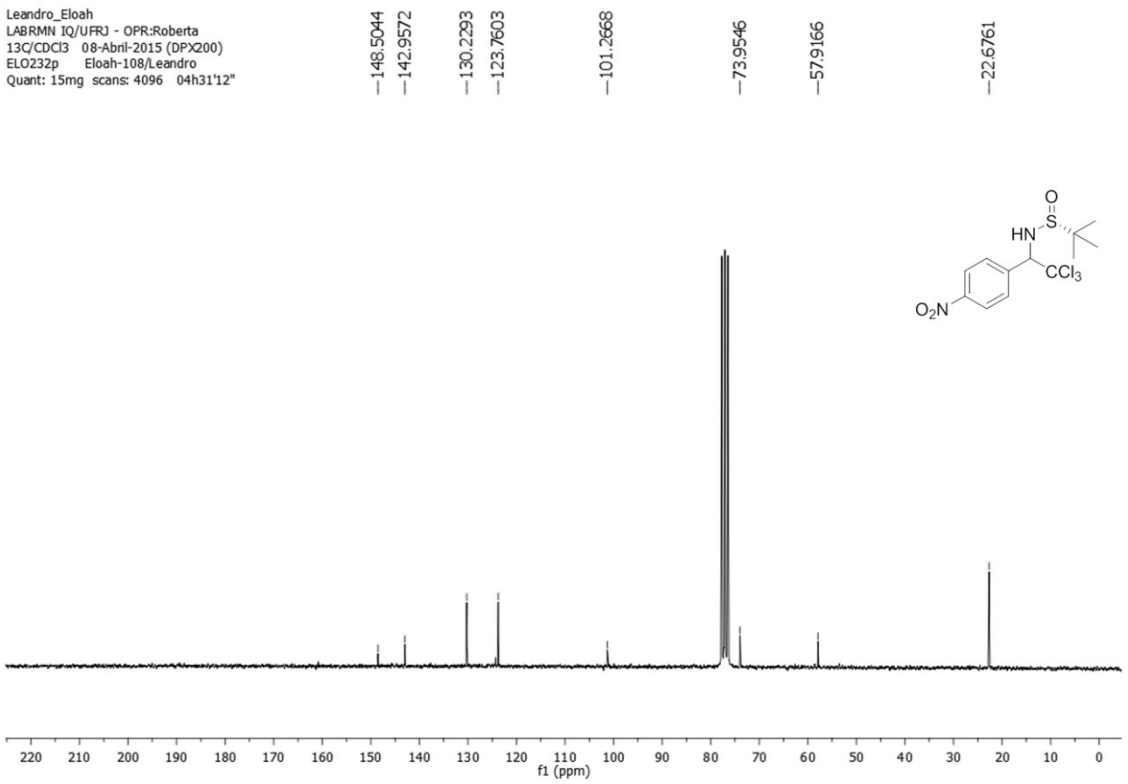
^{1}H NMR spectrum of **3o** (500 MHz, $CDCl_3$)



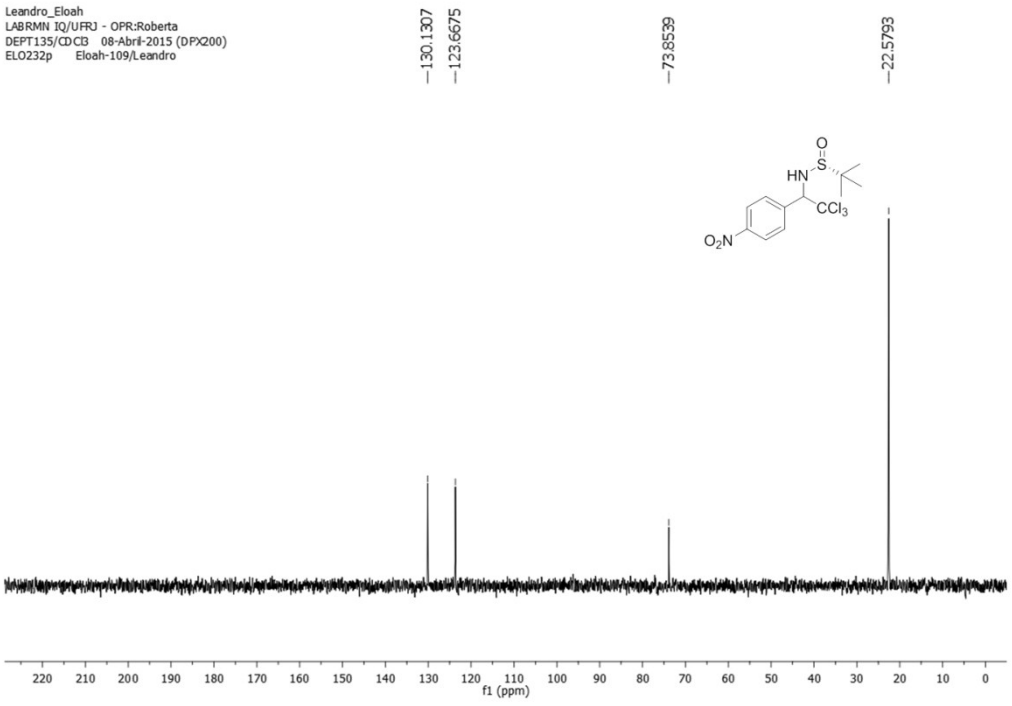
^{13}C NMR spectrum of **3o** (125 MHz, $CDCl_3$)



^{1}H NMR spectrum of **5** (200 MHz, $CDCl_3$)



^{13}C NMR spectrum of **5** (50 MHz, $CDCl_3$)



DEPT135 of **5**

6. Single crystal X-ray diffraction

Single crystal X-ray data were collected using an Oxford Gemini A. Ultra-diffractometer with Mo-Kα line (λ = 0.71073 Å) at temperature of 150 K. For data collection, reduction and refinement of the unit cells, the program CrysAlis RED, Oxford diffraction Ltd – version 1.171.32.38 [1] was used. The structure was solved and refined through the SHELX-2013 program [2]. The absorption correction *multiscan* was used [3]. Additionally, anisotropic displacement parameters were assigned to all non-hydrogen atoms. Hydrogen atoms were located from Fourier maps and the parameters of isotropic displacement were refined or fixed. The structures were designed using the following programs: ORTEP-3 for Windows [4] and Mercury 3.5.1 [5, 6]. The sample characteristics, data collection and refinement parameters for representative temperature measurements are listed in Tab. 1.

Table 1: Crystal data of compound.

Formula	$C_{10}H_{10}Cl_3NO_4S$
Formula weight/g mol^{-1}	346.60
Temperature/K	150
Crystal system	Triclinic
Space group	*P-1*
a/Å	6.9570 (6)
b/Å	9.0926 (10)
c/Å	12.0788 (12)
α / º	110.282 (9)
β / º	100.464 (7)
γ / º	96.922 (8)
V/Å^3	690.69 (13)
Z	2
Crystal size/mm	0.45 × 0.41 × 0.17
D_{calc}/g cm^{-3}	1.667
μ(Mo Kα)/cm^{-1}	0.82
Transmission factors $_{(min/max)}$	0.883 / 0.950
Reflections measured/unique	5866 / 2829
Observed reflections	2122
Nº. of parameters refined	177
***R*[F_o>2σ(F_o)]**	0.040
***wR*[F_o2>2σ (F_o)^2]**	0.085
S	1.03
RMS peak/	0.089

Table 2: Selection of the main geometric parameters, bond distance (Å), bond angles (º) torsion (º) and hydrogen bonds (Å and º).

Cl2—C9	1.775 (3)	O1—C1	1.425 (4)
S1—O4	1.426 (2)	N1—C8	1.457 (3)
S1—O6	1.442 (2)	C8—C5	1.512 (4)
S1—N1	1.615 (2)	C8—C9	1.554 (3)
S1—C10	1.752 (3)	C4—C2	1.371 (4)
Cl1—C9	1.765 (3)	C4—C5	1.411 (4)
Cl3—C9	1.787 (3)	C5—C6	1.382 (4)
O2—C3	1.393 (3)	C2—C3	1.374 (4)
O2—C1	1.408 (4)	C3—C7	1.363 (4)

O1—C2	1.381 (3)	C6—C7	1.401 (4)
Cl1···O4^{i}	3.172 (2)	O6···S1^{ii}	3.497 (2)
Cl1···N1	3.034 (2)	O6···N1^{ii}	2.891 (3)
Cl3···N1	3.026 (2)		
O4—S1—O6	119.40 (11)	C8—C9—Cl1	111.53 (18)
O4—S1—N1	108.01 (12)	C8—C9—Cl2	109.88 (19)
O6—S1—N1	105.33 (12)	Cl1—C9—Cl2	109.17 (13)
O4—S1—C10	108.99 (14)	C8—C9—Cl3	109.60 (16)
O6—S1—C10	106.70 (13)	Cl1—C9—Cl3	108.75 (15)
N1—S1—C10	107.89 (13)	Cl2—C9—Cl3	107.82 (14)
C3—O2—C1	104.8 (2)	C4—C2—C3	122.7 (3)
C2—O1—C1	105.0 (3)	C4—C2—O1	127.2 (3)
C8—N1—S1	123.2 (2)	C3—C2—O1	110.0 (2)
N1—C8—C5	114.0 (2)	C7—C3—C2	122.1 (3)
N1—C8—C9	107.0 (2)	C7—C3—O2	127.8 (3)
C5—C8—C9	113.47 (18)	C2—C3—O2	110.1 (3)
C2—C4—C5	116.4 (3)	C5—C6—C7	122.2 (3)
C6—C5—C4	120.2 (2)	O2—C1—O1	110.2 (3)
C6—C5—C8	118.9 (2)	C3—C7—C6	116.3 (3)
C4—C5—C8	120.9 (2)		
O4—S1—N1—C8	−31.1 (3)	N1—C8—C9—Cl1	59.4 (2)
O6—S1—N1—C8	−159.7 (2)	N1—C8—C9—Cl2	−179.42 (16)
S1—N1—C8—C5	−109.3 (2)	N1—C8—C9—Cl3	−61.1 (2)
S1—N1—C8—C9	124.5 (2)	C5—C8—C9—Cl1	−67.2 (2)
C4—C5—C8—N1	−41.7 (3)	C5—C8—C9—Cl2	54.0 (2)
C6—C5—C8—N1	139.9 (2)	C5—C8—C9—Cl3	172.31 (16)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···O6^{ii}	0.78 (3)	2.16 (2)	2.891 (3)	157 (2)
C1—H1*B*···O6^{iii}	0.97	2.46	3.296 (5)	145
C4—H4···O4^{i}	0.93	2.55	3.321 (4)	140
C8—H8···O4	0.98	2.44	2.939 (3)	111
C10—H10*C*···O2^{iv}	0.96	2.57	3.389 (4)	144

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y-1, -z+1$; (iii) $x+1, y+1, z$; (iv) $-x, -y, -z+1$.

References:

[1] O. Diffraction, CrysAlis PRO. Oxford Diffraction Ltd, (2010).

[2] T. Gruene, H.W. Hahn, A. V. Luebben, F. Meilleur, G.M. Sheldrick, Refinement of macromolecular structures against neutron data with SHELXL2013, in: J. Appl. Crystallogr. 747 (2014) 462–466.

[3] R.H. Blessing, Acta Crystallogr. A. 51 (1995) 33–38.

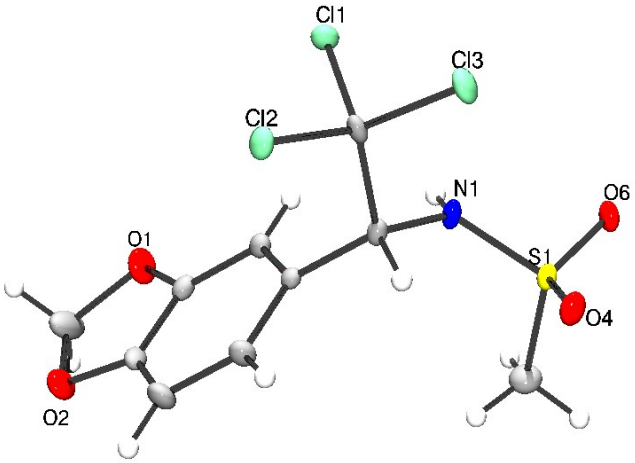
[4] L.J. Farrugia, J. Appl. Crystallogr. 30 (1997) 565.

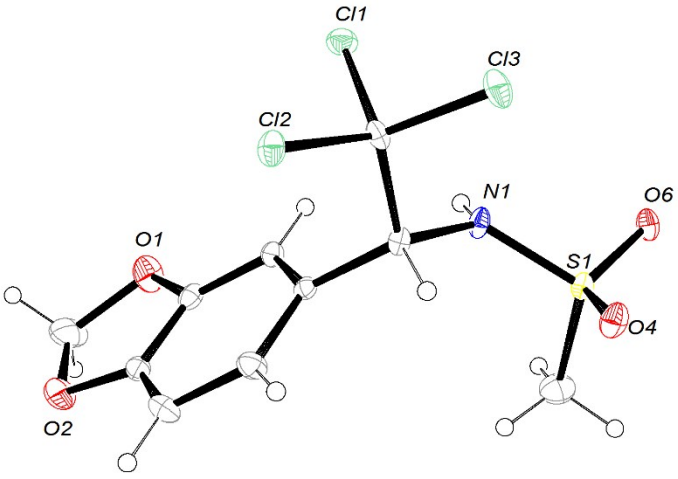
[5] I.R. Thomas, I.J. Bruno, J.C. Cole, C.F. MacRae, E. Pidcock, P.A. Wood, J. Appl. Crystallogr. 43 (2010) 362–366.

[6] C.F. Macrae, P.R. Edgington, P. McCabe, E. Pidcock, G.P. Shields, R. Taylor, M. Towler, J. Van De Streek, J. Appl. Crystallogr. 39 (2006) 453–457.

Appendix A. Supplementary data

All crystallographic data for structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center. Copies of this data (CCDC 1505456) may be obtained free of change upon application to CCDC, 12, Union Road, Cambridge CB2 1EZ, UK (e-mail: deposit@ccdc.cam.ac.uk).


Cl1
Cl3
Cl2
N1
O6
O1
S1
O4
O2


Cl1
Cl3
Cl2
N1
O6
O1
S1
O4
O2