# Supplementary Information

## Elemental sulfur as the "S" source: visible-light-mediated four-component reactions leading to thiocyanates

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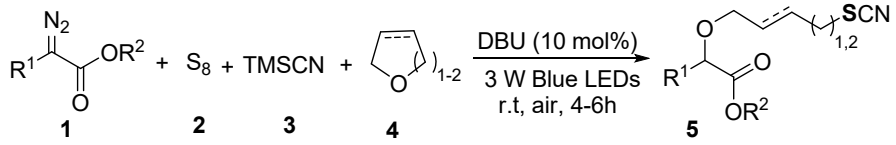
## 1.General information

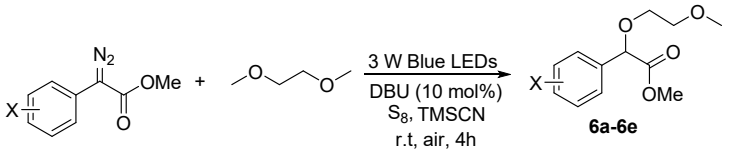
All commercially available reagent grade chemicals were purchased from Aldrich, Acros, Bidepharm and Energy Chemical Company and used as received without further purification unless otherwise stated. $^{1}H$ NMR and $^{13}C$ NMR were recorded in $CDCl_3$ on a Bruker Avance III spectrometer with TMS as internal standard (500 MHz $^{1}H$, 125 MHz $^{13}C$ ) at room temperature, the chemical shifts ($\delta$) were expressed in ppm and *J* values were given in Hz. The following abbreviations are used to indicate the multiplicity: singlet (s), doublet (d), triplet (t), quartet (q), doublet of doublets (dd), doublet of triplets (dt), and multiplet (m). All first order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted were designated as multiplet (m). Mass analyses and HRMS were obtained on a Finnigan-LCQDECA mass spectrometer and a Bruker Daltonics Bio-TOF-Q mass spectrometer by the ESI method, respectively. Column chromatography was performed on silica gel (200-300 mesh). There is 3.0 cm distance between the reactor and LEDs.

## 2. The procedure for visible-light-initiated four-component reaction of α-diazoesters, elemental sulfur, cyclic ethers and TMSCN leading to thiocyanates.




To a mixture of α-diazoesters **1** (0.2 mmol), $S_8$ **2**(0.2 mmol), TMSCN **3** (0.1 mmol) and DBU (0.01mmol) was added cyclic ethers **4** (2 mL). The reaction mixture was stirred in air under the irradiation of 3W blue LED at room temperature for 4-6h. After completion of the reaction, the solution was concentrated in vacuum. The residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **5.**

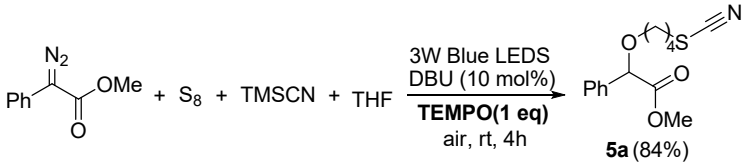
## 3. The procedure for two-component reaction of α-diazoesters with dimethoxyethane leading to α-alkoxyl esters.




To a mixture of α-diazoesters **1** (0.2 mmol), $S_8$ (0.2 mmol), TMSCN (0.1 mmol) and DBU (0.01mmol) was added dimethoxyethane (2 mL). The reaction mixture was stirred in air under the irradiation of 3W blue LED at room temperature for 4h. After completion of the reaction, the solution was concentrated in vacuum. The residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **6.**

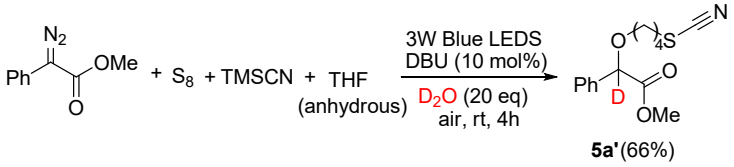
## 4.Preliminary mechanistic studies.

### 4.1 The addition of TEMPO in the model reaction system.



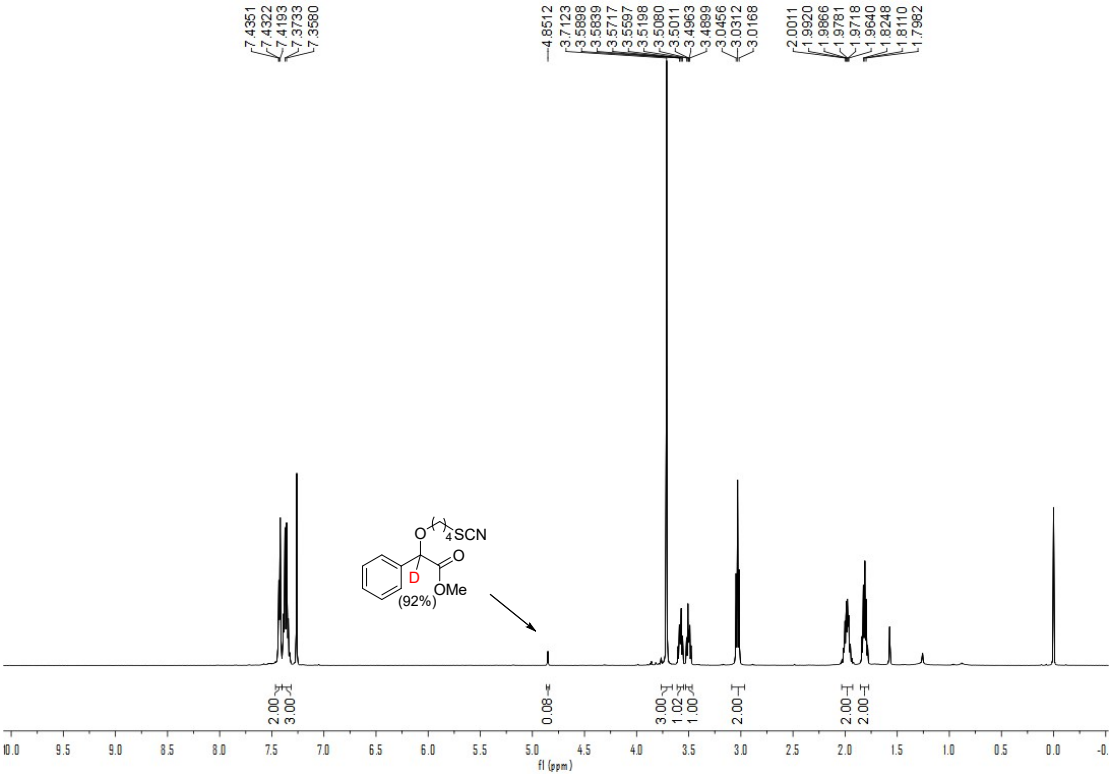

To a mixture of α-diazoester **1a** (0.2 mmol), $S_8$ **2** (0.2 mmol), TMSCN **3** (0.1 mmol) and DBU (0.01mmol), TEMPO (0.1 mmol) was added THF (AR, 2 mL). The reaction mixture was stirred in air under the irradiation of 3W blue LED at room temperature for 4h. After completion of the reaction, the solution was concentrated in vacuum, the desired product **5a** was obtained in 84% yield. This result indicated that a radical process might not be involved in the present transformations.

### 4.3 Deuterium experiment.

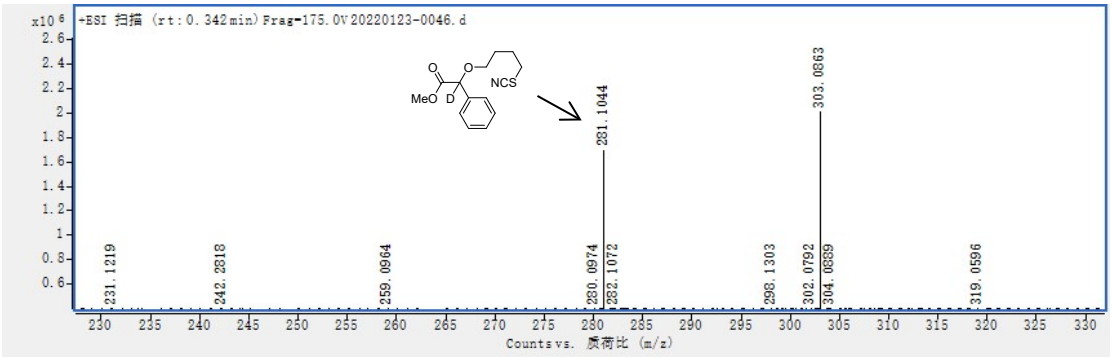



To a mixture of α-diazoester **1a** (0.2 mmol), $S_8$ **2** (0.2 mmol), TMSCN **3** (0.1 mmol), DBU (0.01mmol), and $D_2O$ (2 mmol) was added anhydrous THF (2 mL). The

reaction mixture was stirred in air under the irradiation of 3W blue LED at room temperature for 4h. After completion of the reaction, the solution was concentrated in vacuum, the desired product D-**5a** was obtained in 66% yield. The deuterated product was determined by $^{1}H$ NMR and HRMS. This result indicated which indicated that proton at α-position of ester came from water in solvent.




NMR




HRMS

**4.2 The procedures for Light On/off experiments.**

To a mixture of α-diazoester **1a** (0.4 mmol), $S_8$ **2** (0.2 mmol), diethyl H-phosphonate **3a** (0.1 mmol), and DBU (0.1 mmol) was added THF (2 mL). The reaction mixture was separately stirred and irradiated by 3 W blue LEDs at room temperature for 1h, 2h, and 3h. The desired product **5a** was isolated in 25%, 50%, and 71%, respectively. Additionally, the reaction mixture was stirred and irradiated by 3 W Blue LEDs at room temperature for 1h, then the reaction mixture was continuously stirred in the dark for 1h, the corresponding product was also obtained in 25.1% yield. Additionally, when the reaction mixture was stirred and irradiated by 3 W blue LEDs at room temperature for 2h, then the reaction mixture was continuously stirred in the dark for 1h, the corresponding product was obtained in 50% yield. The above results suggested that the continuous visible-light irradiation is necessary for promoting this transformation.

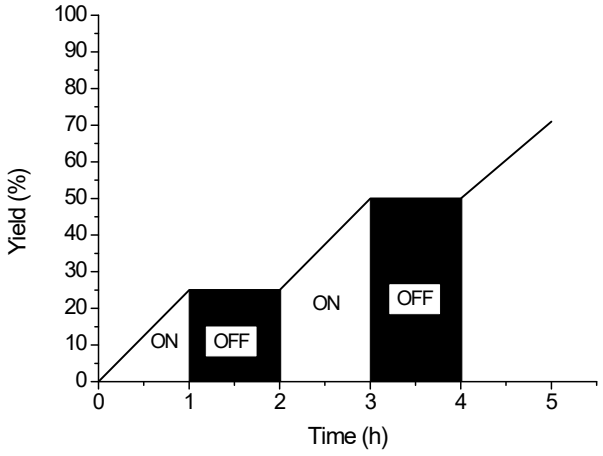
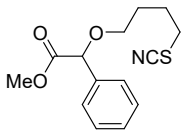


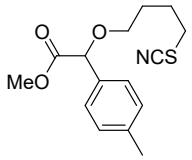

**Fig S1.** On/off experiments.

## 5. Characterization data of products 5a- 5x



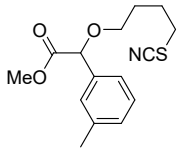

**methyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5a** was obtained in 84% yield (23.4 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.42 (m, 2H), 7.39 – 7.32 (m, 3H), 4.85 (s, 1H), 3.71 (s, 3H), 3.60 – 3.56 (m, 1H), 3.52 – 3.48 (m, 1H), 3.03 (t, *J* = 7.2 Hz, 2H), 1.99 – 1.96 (m, 2H), 1.83 – 1.78 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.2, 136.4, 128.8, 128.7, 127.2, 112.3, 81.1, 68.8, 52.3, 33.8, 27.7, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{18}NO_3S$ 280.1007; found 280.1008.



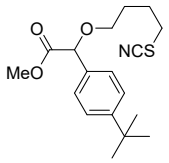

**methyl 2-(4-thiocyanatobutoxy)-2-(p-tolyl)acetate**

Compound **5b** was obtained in 89% yield (26.2 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 4.81 (s, 1H), 3.70 (s, 3H), 3.56 – 3.53 (m, 1H), 3.50 – 3.46 (m, 1H), 3.02 (t, *J* = 7.2 Hz, 2H), 2.35 (s, 3H), 1.99 – 1.94 (m, 2H), 1.80 (t, *J* = 6.7 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.4, 138.7, 133.4, 129.4, 129.3, 127.1, 112.3, 81.0, 68.6, 52.2, 33.9, 27.7, 27.0, 21.2; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{20}NO_3S$ 294.1164; found 294.1166.



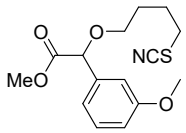

**methyl 2-(4-thiocyanatobutoxy)-2-(m-tolyl)acetate**

Compound **5c** was obtained in 84% yield (24.5 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.27 – 7.20 (m, 3H), 7.15 (d, *J* = 7.4 Hz, 1H), 4.81 (s, 1H), 3.71 (s, 3H), 3.57 – 3.54 (m, 1H), 3.51 – 3.48 (m, 1H), 3.03 (t, *J* = 7.2 Hz, 2H), 2.36 (s, 3H), 1.99 – 1.94 (m, 2H), 1.83 – 1.77 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.3, 138.5, 136.2, 129.6, 128.6, 127.8, 124.3, 112.3, 81.2, 68.8, 52.3, 33.9, 27.7, 27.0, 21.4; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{20}NO_3S$ 294.1164; found 294.1168.



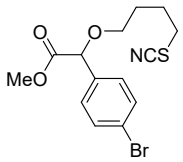

**methyl 2-(4-(tert-butyl)phenyl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5d** was obtained in 79% yield (26.3 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 4.83 (s, 1H), 3.71 (s, 3H), 3.57 – 3.54 (m, 1H), 3.52 – 3.49 (m, 1H), 3.03 (t, *J* = 7.2 Hz, 2H), 1.99 – 1.96 (m, 2H), 1.83 – 1.79 (m, 2H), 1.31 (s, 9H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.4, 151.8, 133.3, 126.9 , 125.7, 112.3, 81.0, 68.7, 52.2, 34.6, 33.9, 31.3, 27.8, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{26}NO_3S$ 336.1633; found 336.1636.



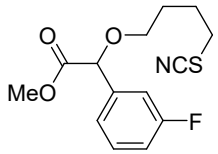

**methyl 2-(3-methoxyphenyl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5e** was obtained in 70% yield (21.6 mg) according to the general procedure (6h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 1H), 7.00 – 6.97 (m, 2H), 6.89 – 6.97 (m, 1H), 4.83 (d, *J* = 7.5 Hz, 1H), 3.81 (d, *J* = 3.1 Hz, 3H), 3.71 (s, 3H), 3.59 – 3.55 (m, 1H), 3.52 – 3.48 (m, 1H), 3.01 (t, *J* = 7.2 Hz, 2H), 2.00 – 1.96 (m, 2H), 1.83 – 1.79 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.1, 159.9, 137.8, 129.7, 119.5, 114.5, 112.5, 112.3, 81.0, 68.8, 55.3, 52.3, 33.8, 27.7, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{20}NO_4S$ 310.1113; found 310.1115.




**methyl 2-(4-bromophenyl)-2-(4-thiocyanatobutoxy)acetate**

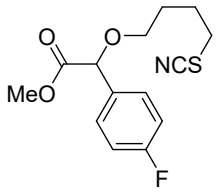
Compound **5f** was obtained in 80% yield (28.7 mg) according to the general procedure (6h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 4.81 (s, 1H), 3.71 (s, 3H), 3.61 – 3.56 (m, 1H), 3.51 – 3.48 (m, 1H), 3.03 (t, *J* = 7.2 Hz, 2H), 2.10 – 1.95 (m, 2H), 1.84 – 1.79 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.8 , 135.4, 131.9, 128.8, 122.9, 112.2, 80.5, 69.0, 52.4, 33.8, 27.7, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}BrNO_3S$ 358.0113; found 358.0117.




**methyl 2-(3-fluorophenyl)-2-(4-thiocyanatobutoxy)acetate**

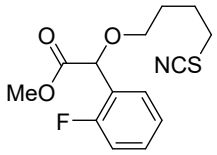
Compound **5g** was obtained in 84% yield (25.3 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.32 (m, 1H), 7.21 (d, *J* = 7.7 Hz, 1H), 7.16 (d, *J* = 9.4 Hz, 1H), 7.06 – 7.15 (m, 1H), 4.85 (s, 1H), 3.73 (s, 3H), 3.61 – 3.58 (m, 1H), 3.53 – 3.50 (m, 1H), 3.04 (t, *J* = 7.2 Hz, 2H), 2.01 – 1.97 (m, 2H), 1.85 – 1.81 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.8, 162.8 (d, *J* = 245.3 Hz), 138.7 (d, *J* = 7.3 Hz), 130.2 (d, *J* = 8.1 Hz), 122.7 (d, *J* = 3.0 Hz), 115.7(d, *J* =

21.0 Hz), 114.1(d, *J* = 22.5 Hz), 112.3, 80.5 (d, *J* = 1.7 Hz), 69.0, 52.4, 33.8, 27.7, 26.9; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}FNO_3S$ 298.0913; found 298.0910.



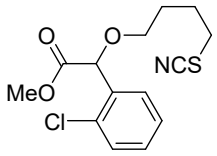

**methyl 2-(4-fluorophenyl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5h** was obtained in 86% yield (25.6 mg) according to the general procedure (4h). Yellow oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.40 (m, 2H), 7.07 – 7.04 (m, 2H), 4.83 (s, 1H), 3.72 (s, 3H), 3.60 – 3.56 (m, 1H), 3.51 – 3.47 (m, 1H), 3.03 (t, *J* = 7.2 Hz, 2H), 2.00 – 1.96 (m, 2H), 1.84 – 1.78 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.1, 163.9 (d, *J* = 246.0 Hz), 132.2 (d, *J* = 3.2 Hz), 128.9 (d, *J* = 8.3 Hz), 115.7(d, *J* = 22.5 Hz), 112.3, 80.4, 68.9, 52.4, 33.8, 27.7, 26.9; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}FNO_3S$ 298.0913; found 298.0909.



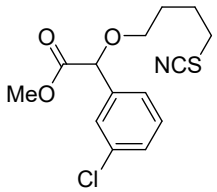

**methyl 2-(2-fluorophenyl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5i** was obtained in 83% yield (24.6 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.42 (m, 1H), 7.35 – 7.32 (m, 1H), 7.18 (t, *J* = 7.3 Hz, 1H), 7.08 (t, *J* = 9 Hz, 1H), 5.20 (s, 1H), 3.73 (d, *J* = 2.6 Hz, 3H), 3.65 – 3.62 (m, 1H), 3.53 – 3.50 (m, 1H), 3.02 (t, *J* = 7.2 Hz, 2H), 1.99 – 1.93 (m, 2H), 1.83 – 1.77 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.7, 160.4 (d, *J* = 246.8 Hz), 130.5 (d, *J* = 8.2 Hz), 128.7 (d, *J* = 3.3 Hz), 124.6 (d, *J* = 3.6 Hz), 123.9 (d, *J* = 14.2 Hz), 115.6 (d, *J* = 21.6 Hz), 112.3, 74.1 (d, *J* = 3.0 Hz), 69.6, 69.1, 52.4, 33.8, 27.7, 26.9; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}FNO_3S$ $[M+H]^+$ 298.0913, found 298.0898.



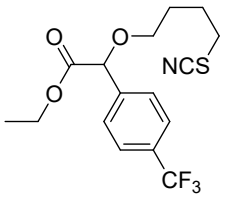

**methyl 2-(2-chlorophenyl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5j** was obtained in 85% yield (26.4 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.49 – 7.46 (m, 1H), 7.41 – 7.39 (m, 1H), 7.32 – 7.28 (m, 2H), 5.34 (s, 1H), 3.73 (s, 3H), 3.67 – 3.63 (m, 1H), 3.54 – 3.50 (m, 1H), 3.02 (t, *J* = 7.2 Hz, 2H), 1.98 – 1.94 (m, 2H), 1.82 – 1.79 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.7, 134.4, 133.9, 129.9, 129.7, 128.7, 127.3, 112.3, 69.2, 52.4, 33.8, 27.7, 26.9; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}ClNO_3S$ 314.0618; found 314.0614.



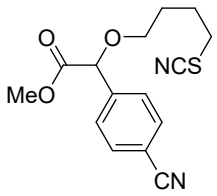

**methyl 2-(3-chlorophenyl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5k** was obtained in 79% yield (34.1 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.34 – 7.30 (m, 3H), 4.82 (s, 1H), 3.73 (s, 3H), 3.60 – 3.58 (m, 1H), 3.52 – 3.48 (m, 1H), 3.04 (t, *J* = 7.2 Hz, 2H), 2.01 – 1.95 (m, 2H), 1.85 – 1.80 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.7, 138.3, 134.6, 129.9, 128.9, 127.2, 125.2, 112.3, 80.4, 69.1, 52.5, 33.8, 27.7, 26.9; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{14}H_{17}ClNO_3S$ 314.0618; found 314.0616.



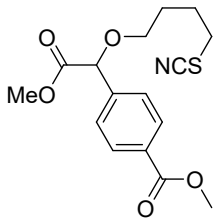

**ethyl 2-(4-thiocyanatobutoxy)-2-(4-(trifluoromethyl)phenyl)acetate**

Compound **5l** was obtained in 77% yield (27.7 mg) according to the general procedure (6h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.64 – 7.57 (m, 4H), 4.89 (s, 1H), 4.23 – 4.13 (m, 2H), 3.65 – 3.63 (m, 1H), 3.55 – 3.51 (m, 1H), 3.05 (t, *J* = 7.2 Hz, 2H), 2.02 – 1.97 (m, 2H), 1.86 – 1.81 (m, 2H), 1.23 (t, *J* = 7.1 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.1, 140.3, 130.8 (d, *J* = 32.3 Hz), 127.2, 125.6 (q, *J* = 3.7 Hz), 123.9 (d, *J* = 272.1 Hz), 80.6, 69.2, 61.6, 33.8, 27.8, 26.9, 14.1; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{16}H_{19}F_3NO_3S$ 362.1038; found 362.1042.



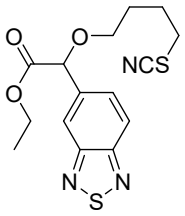

**methyl 2-(4-cyanophenyl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5m** was obtained in 86% yield (25.1 mg) according to the general procedure (6h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.1 Hz, 2H), 7.57 (d, *J* = 8.0 Hz, 2H), 4.91 (s, 1H), 3.73 (s, 3H), 3.67 – 3.63 (m, 1H), 3.55 – 3.50 (m, 1H), 3.04 (t, *J* = 7.2 Hz, 2H), 2.02 – 1.99 (m, 2H), 1.87 – 1.83 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.2, 141.4, 132.5, 127.7, 118.4, 112.7, 112.2, 80.4, 69.4, 52.6, 33.7, 27.8, 26.9; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{17}N_2O_3S$ $[M+H]^+$ 305.0960, found 305.0955.



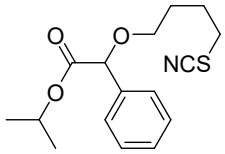
**methyl 4-(2-methoxy-2-oxo-1-(4-thiocyanatobutoxy)ethyl)benzoate**

Compound **5n** was obtained in 93% yield (31.6 mg) according to the general procedure (6h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.44 (d, *J* = 8.3 Hz, 2H), 4.84 (s, 1H), 3.85 (s, 3H), 3.65 (s, 3H), 3.57 – 3.53 (m, 1H), 3.46 – 13.42 (m, 1H), 2.97 (t, *J* = 7.2 Hz, 2H), 1.94 – 1.90 (m, 2H), 1.77 – 1.75 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.7 , 166.6, 141.2, 130.5, 129.9, 127.0, 112.2, 80.7, 69.1, 52.5, 52.2, 33.8, 27.7, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{16}H_{20}NO_5S$ $[M+H]^+$ 338.1062, found 338.1067.



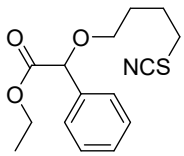
**ethyl 2-(benzo[c][1,2,5]thiadiazol-5-yl)-2-(4-thiocyanatobutoxy)acetate**

Compound **5o** was obtained in 52% yield (18.3 mg) according to the general procedure (6h). Yellow oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 8.08 (s, 1H), 8.01 (d, *J* = 9.1 Hz, 1H), 7.72 – 7.70 (m, 1H), 5.01 (s, 1H), 4.24 – 4.18 (m, 2H), 3.70 – 3.67 (m, 1H), 3.61 – 3.58 (m, 1H), 3.07 (t, *J* = 7.2 Hz, 2H), 2.04 – 2.00 (m, 2H), 1.88 – 1.85 (m, 2H), 1.24 (t, *J* = 5.8 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 169.9, 154.8, 154.6, 138.2, 128.2, 121.8, 120.0, 112.2, 80.8, 69.2, 61.7, 33.8, 27.8, 27.0, 14.1; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{18}N_3O_3S_2$ $[M+H]^+$ 352.0790, found 352.0777.



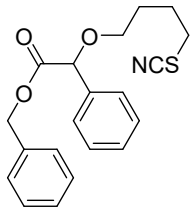
**isopropyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5p** was obtained in 79% yield (19.4 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.41(m, 2H), 7.38 – 7.32 (m, 3H), 5.06 – 5.02 (m, 1H), 4.79 (s, 1H), 3.61 – 3.57 (m, 1H), 3.53 – 3.49 (m, 1H), 3.05 (t, *J* = 7.2 Hz, 2H), 2.01 – 1.98 (m, 2H), 1.84 – 1.79 (m, 2H), 1.25 (d, *J* = 6.3 Hz, 3H), 1.12 (d, *J* = 6.2 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.4, 136.6, 128.6,128.6, 127.0, 112.4, 81.3, 68.8, 68.7, 33.9, 27.8, 27.1, 21.8, 21.5; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{16}H_{22}NO_3S$ $[M + H]^+$ 308.1320; found 308.1322.



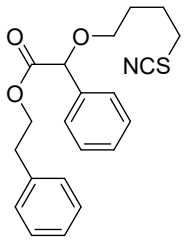

**ethyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5q** was obtained in 92% yield (27.0 mg) according to the general procedure (4h). Colorless oil. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.42 (m, 2H), 7.38 – 7.33 (m, 3H), 4.83 (s, 1H), 4.21 – 4.11 (m, 2H), 3.61 – 3.57 (m, 1H), 3.52 – 3.48 (m, 1H), 3.04 (t, *J* = 7.2 Hz, 2H), 2.02 – 1.95 (m, 2H), 1.84 – 1.79 (m, 2H), 1.22 (t, *J* = 7.1 Hz, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.8, 136.5, 128.7, 128.6, 128.6, 127.1, 112.3, 81.2, 68.8, 61.3, 33.9, 27.7, 27.0, 14.1; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{20}NO_3S$ $[M+Na]^+$ 294.1164, found 294.1166.



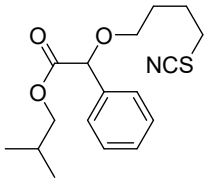

**benzyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5r** was obtained in 87% yield (30.8 mg) according to the general procedure (4h). Colorless oil. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.41 (m, 2H), 7.37 – 7.33 (m, 3H), 7.31 – 7.30 (m, 3H), 7.25 – 7.21 (m, 2H), 5.17 – 5.10 (m, 2H), 4.89 (s, 1H), 3.58 – 3.54 (m, 1H), 3.52 – 3.49 (m, 1H), 2.99 (t, *J* = 7.2 Hz, 2H), 1.98 – 1.93 (m, 2H), 1.81 – 1.76 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.7, 136.3, 135.4, 128.8, 128.7, 128.6, 128.3, 128.0, 127.2, 112.3, 81.2, 68.8, 66.8, 33.8, 27.7, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_3S$ $[M+Na]^+$ 356.1320, found 356.1315.



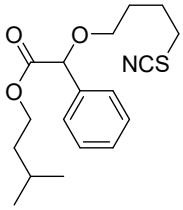

**phenethyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5s** was obtained in 81% yield (29.8 mg) according to the general procedure (4h). Yellow solid. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 5H), 7.25 – 7.20 (m, 3H), 7.08 – 7.07 (m, 2H), 4.80 (s, 1H), 4.37 – 4.32 (m, 2H), 3.52 – 3.44 (m, 2H), 3.01 (t, *J* = 7.2 Hz, 2H), 2.89 – 2.85 (m, 2H), 2.00 – 1.93 (m, 2H), 1.80 – 1.70 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.7, 137.4, 136.4, 128.9, 128.5, 127.1, 126.6, 112.4, 81.2, 68.8, 65.6, 34.9, 33.9, 27.7, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{21}H_{24}NO_3S$ 370.1477; found 370.1480.



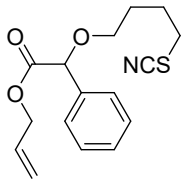

**isobutyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5t** was obtained in 79% yield (30.6 mg) according to the general procedure (the reaction was complete for 4h). Colorless oil. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.42 (m, 2H), 7.38 – 7.32 (m, 3H), 4.84 (s, 1H), 3.93 – 3.86 (m, 2H), 3.62 – 3.58 (m, 1H), 3.53 – 3.49 (m, 1H), 3.04 (t, *J* = 7.2 Hz, 2H), 2.01 – 1.97 (m, 2H), 1.88 – 1.84 (m, 1H), 1.84 – 1.80 (m, 2H), 0.83 – 0.81 (m, 6H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.9, 136.7, 128.7, 128.6, 127.1, 112.3, 81.2, 71.1, 68.8, 33.9, 27.8, 28.7, 27.0, 18.9; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{24}NO_3S$ $[M+H]^+$ 322.1477, found 322.1481.



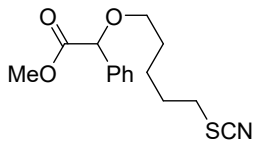

**isopentyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5u** was obtained in 82% yield (30.5 mg) according to the general procedure (the reaction was complete for 6h). Yellow oil. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.42 (d, *J* = 6.6 Hz, 2H), 7.38 – 7.31 (m, 3H), 4.82 (s, 1H), 4.15 – 4.12 (m, 2H), 3.59 – 3.57 (m, 1H), 3.52 – 3.49 (m, 1H), 3.04 (t, *J* = 7.2 Hz, 2H), 2.00 – 1.97 (m, 2H), 1.84 – 1.80 (m, 2H), 1.59 – 1.51 (m, 1H), 1.48 – 1.44 (m, 2H), 0.84 (dd, *J* = 9.8, 6.6 Hz, 6H);$^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.9, 136.5, 128.7, 128.6, 127.1, 112.4, 81.2, 68.8, 63.9, 37.1, 33.9, 27.7, 27.0, 24.9, 22.4, 22.3; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{26}NO_3S$ $[M+H]^+$ 336.1633, found 336.1637.



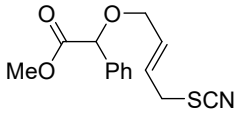

**allyl 2-phenyl-2-(4-thiocyanatobutoxy)acetate**

Compound **5v** was obtained in 88% yield (37.0 mg) according to the general procedure (the reaction was complete for 6h). Yellow oil. $^{1}$H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.3 (m, 2H), 7.39 – 7.33 (m, 3H), 5.89 – 5.80 (m, 1H), 5.21 – 5.17 (m, 2H), 4.87 (s, 1H), 4.82 – 4.60 (m, 1H), 3.62 – 3.57 (m, 1H), 3.53 – 3.49 (m, 1H), 3.03 (t, *J* = 7.2 Hz, 2H), 2.00 – 1.95 (m, 2H), 1.84 – 1.80 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.5, 136.4, 131.6, 128.8, 128.7, 127.2, 127.1, 118.5, 112.3, 81.2, 68.8, 65.6, 33.9, 27.7, 27.0; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{16}H_{20}NO_3S$ $[M+H]^+$ 306.1164, found 306.1167.



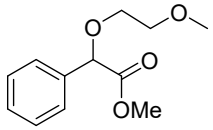

**methyl 2-phenyl-2-((5-thiocyanatopentyl)oxy)acetate**

Compound **5w** was obtained in 64% yield (18.8 mg) according to the general procedure (the reaction was complete for 6h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.43 (m, 2H), 7.39 – 7.33 (m, 3H), 4.86 (s, 1H), 3.71(s, 3H), 3.56 – 3.53 (m, 1H), 3.48 – 3.45(m, 1H), 2.95 (t, *J* = 7.3 Hz, 2H), 1.88 – 1.81 (m, 2H), 1.72 – 1.67 (m, 2H), 1.60 – 1.53 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.3, 136.5, 128.7, 128.6, 127.2, 112.3, 81.1, 69.3, 52.3, 33.9, 29.6, 28.9, 24.7; HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{25}H_{25}N_2O_3S$ [M+H]$^+$ 294.1164, found 294.1166.



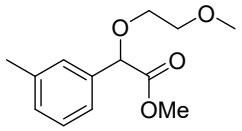

**methyl (E)-2-phenyl-2-((4-thiocyanatobut-2-en-1-yl)oxy)acetate**

Compound **5x** was obtained in 47% yield (11.8 mg) according to the general procedure (the reaction was complete for 4h). Yellow oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.43 (m, 2H), 7.40 – 7.35 (m, 3H), 5.97 – 5.93 (m, 1H), 5.82 – 5.77 (m, 1H), 4.92 (s, 1H), 4.24 – 4.20 (m, 1H), 4.16 – 4.12 (m, 1H), 3.76 – 3.74 (m, 1H), 3.72 (s, 3H), 3.67 – 3.63 (m, 1H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.9, 135.9, 131.8, 129.0, 128.8, 127.4, 125.9, 111.9, 80.5, 64.7, 52.4, 30.8; HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{14}H_{16}NO_3S$ 278.0851; found 278.0852.




**methyl 2-(2-methoxyethoxy)-2-phenylacetate**

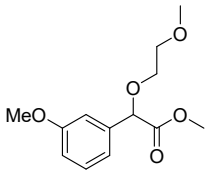
Compound **6a** was obtained in 57% yield (12.8 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.47 – 7.45 (m, 2H), 7.38 – 7.30 (m, 3H), 4.99 (s, 1H), 3.72 – 3.69 (m, 1H), 3.71 (s, 3H), 3.66 – 3.63 (m, 1H), 3.62 – 3.57 (m, 2H), 3.37 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.3, 136.4, 128.7, 128.6, 127.3, 81.4, 72.0, 69.0, 59.0, 52.2; HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{12}H_{17}O_4$ 225.2217; found 225.2219.




**methyl 2-(2-methoxyethoxy)-2-(m-tolyl)acetate**

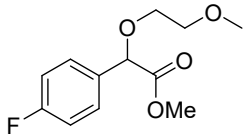
Compound **6b** was obtained in 56% yield (13.3 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.27 (s, 1H), 7.24 – 7.23 (m, 2H), 7.14 – 7.13 (m, 1H), 4.95 (s, 1H), 3.71 (s, 3H), 3.70 – 3.67 (m, 1H), 3.65 – 3.63 (m, 3H), 3.36 (s, 3H), 2.35 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.4, 138.4,

136.3, 129.5, 128.5, 127.9, 124.5, 81.4, 72.0, 68.9, 59.0, 52.2, 21.4; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{19}O_4$ 239.1283; found 239.1287.



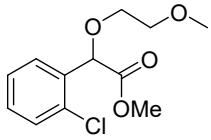

**methyl 2-(2-methoxyethoxy)-2-(3-methoxyphenyl)acetate**

Compound **6c** was obtained in 66% yield (16.8 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.26 (t, *J* = 7.8 Hz, 1H), 7.02 (t, *J* = 4.9 Hz, 2H), 6.88 – 6.86 (m, 1H), 4.96 (s, 1H), 3.81 (s, 3H), 3.71 (s, 3H), 3.69-3.67 (m, 1H), 3.66-3.58 (m, 3H), 3.37 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.2, 159.8, 137.9, 129.6, 119.7, 114.6, 112.4, 81.3, 72.0, 69.0, 59.0, 55.3, 52.3; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{19}O_5$ 255.1232; found 255.1236.




**methyl 2-(4-fluorophenyl)-2-(2-methoxyethoxy)acetate**

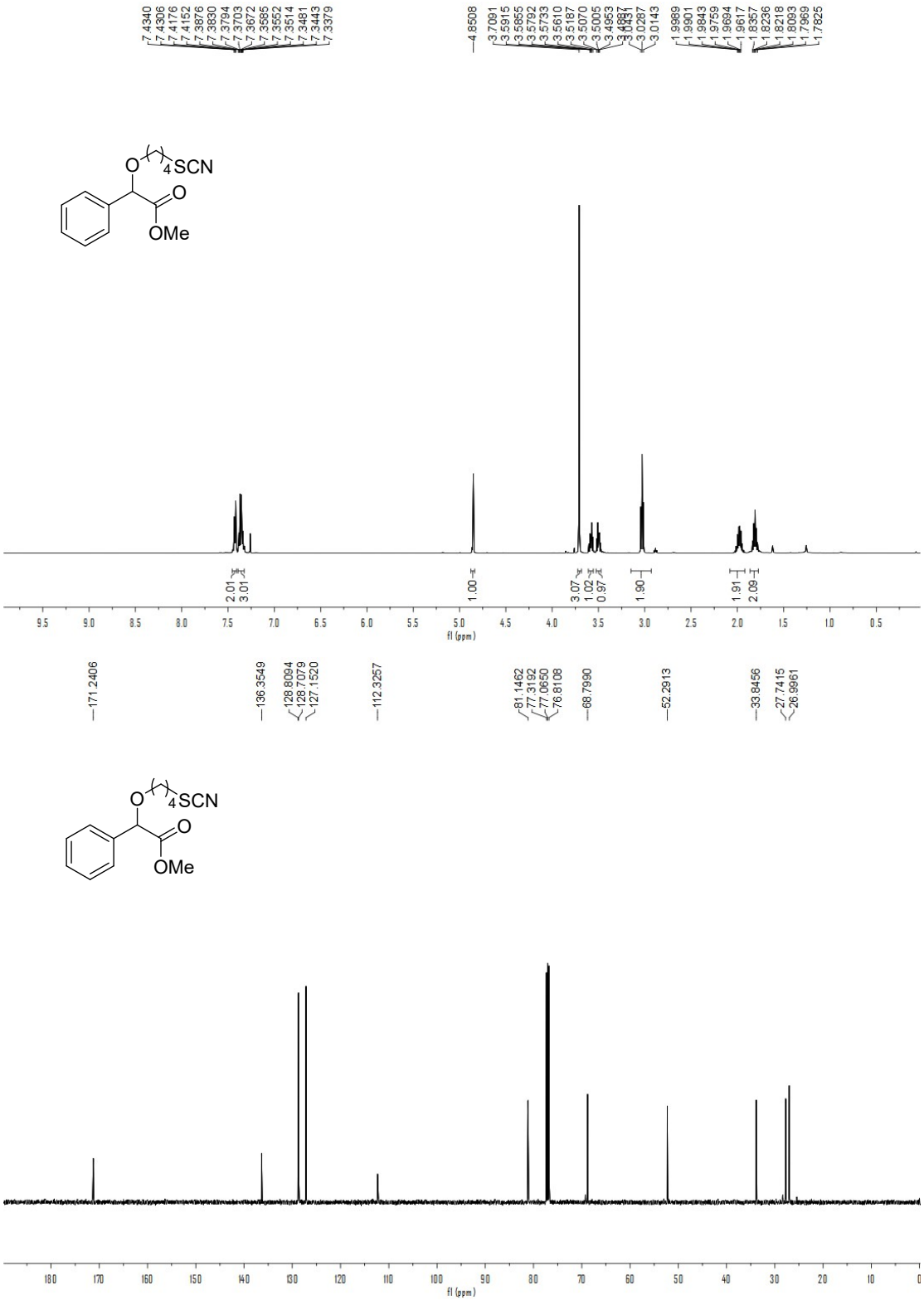
Compound **6d** was obtained in 53% yield (12.8 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.45-7.42 (m, 2H), 7.06-7.03 (m, 2H), 4.97 (s, 1H), 3.71 (s, 3H), 3.69 – 3.67 (m, 1H), 3.65 – 3.58 (m, 1H), 3.37 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 171.2, 162.9 (d, *J* = 245.8 Hz), 132.3 (d, *J* = 3.2 Hz), 129.0 (d, *J* = 8.3 Hz), 115.5 (d, *J* = 21.5 Hz), 80.7, 72.0, 69.1, 59.0, 52.3; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{16}FO_4$ 243.1033; found 243.1037.




**methyl 2-(2-chlorophenyl)-2-(2-methoxyethoxy)acetate**

Compound **6e** was obtained in 76% yield (19.6 mg) according to the general procedure (4h). Colorless oil. $^1$H NMR (500 MHz, $CDCl_3$) δ 7.55 – 7.53 (m, 1H), 7.39 – 7.37 (m, 1H), 7.30 – 7.26 (m, 2H), 5.47 (s, 1H), 3.79 – 3.74 (m, 1H), 3.72 (s, 3H), 3.69 – 3.64 (m, 1H), 3.60 – 3.58 (m, 2H), 3.36 (s, 3H); $^{13}$C NMR (125 MHz, $CDCl_3$) δ 170.6, 134.5, 133.8, 129.8, 129.6, 128.9, 127.2, 77.6, 71.8, 69.4, 59.0, 52.3; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{16}ClO_4$ 259.0737; found 259.0741.

## 6.Copies of NMR spectra for 5a−5x, 6a-6e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1.7969
1.7825
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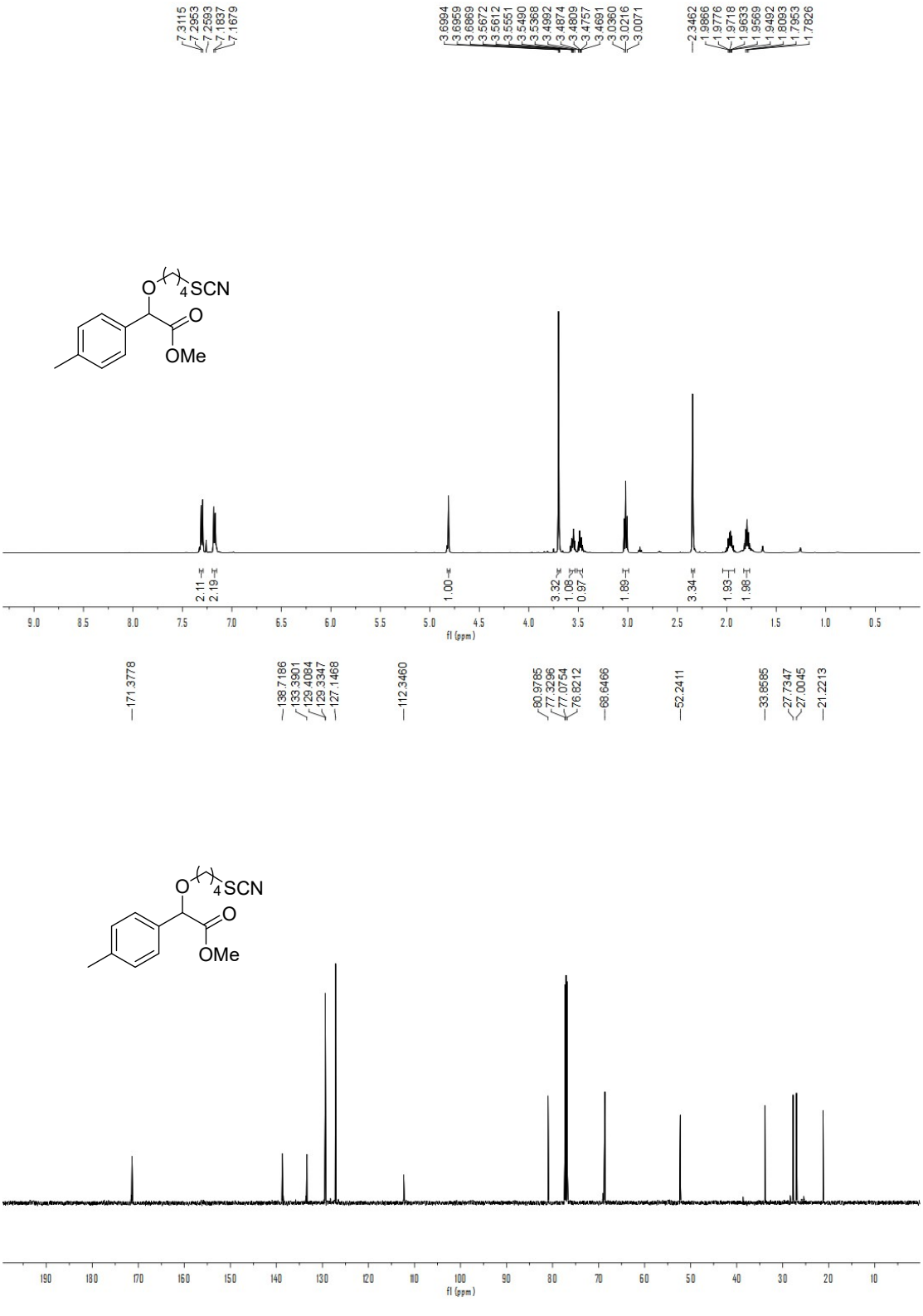
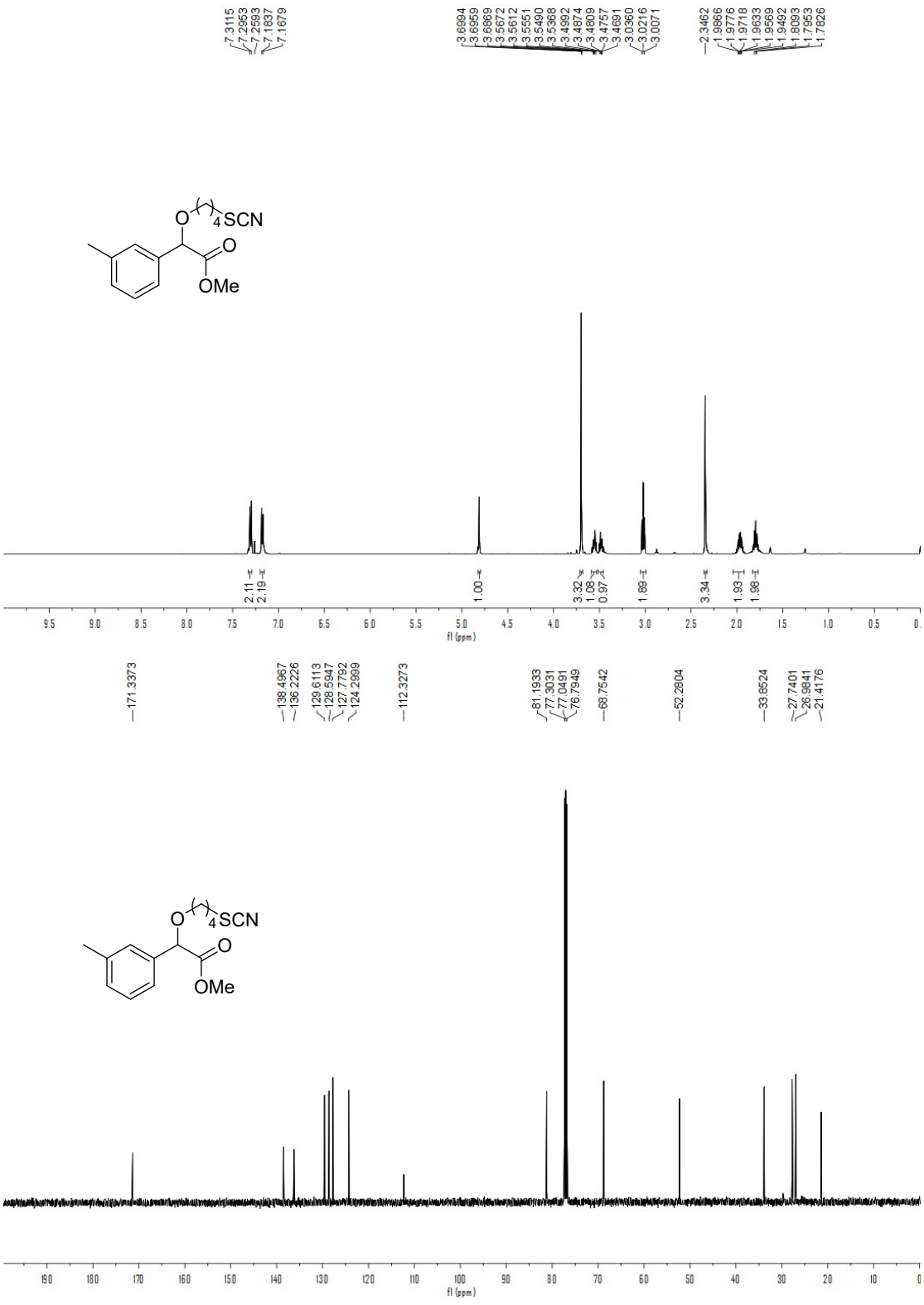
4SCN
O
OMe
2.01
3.01
1.00
3.07
1.02
0.97
1.90
1.91
2.09
f1 (ppm)
171.2406
136.3549
128.8094
128.7079
127.1520
112.3257
81.1462
77.3192
77.0650
76.8108
68.7990
52.2913
33.8456
27.7415
26.9961
O
4SCN
O
OMe
f1 (ppm)

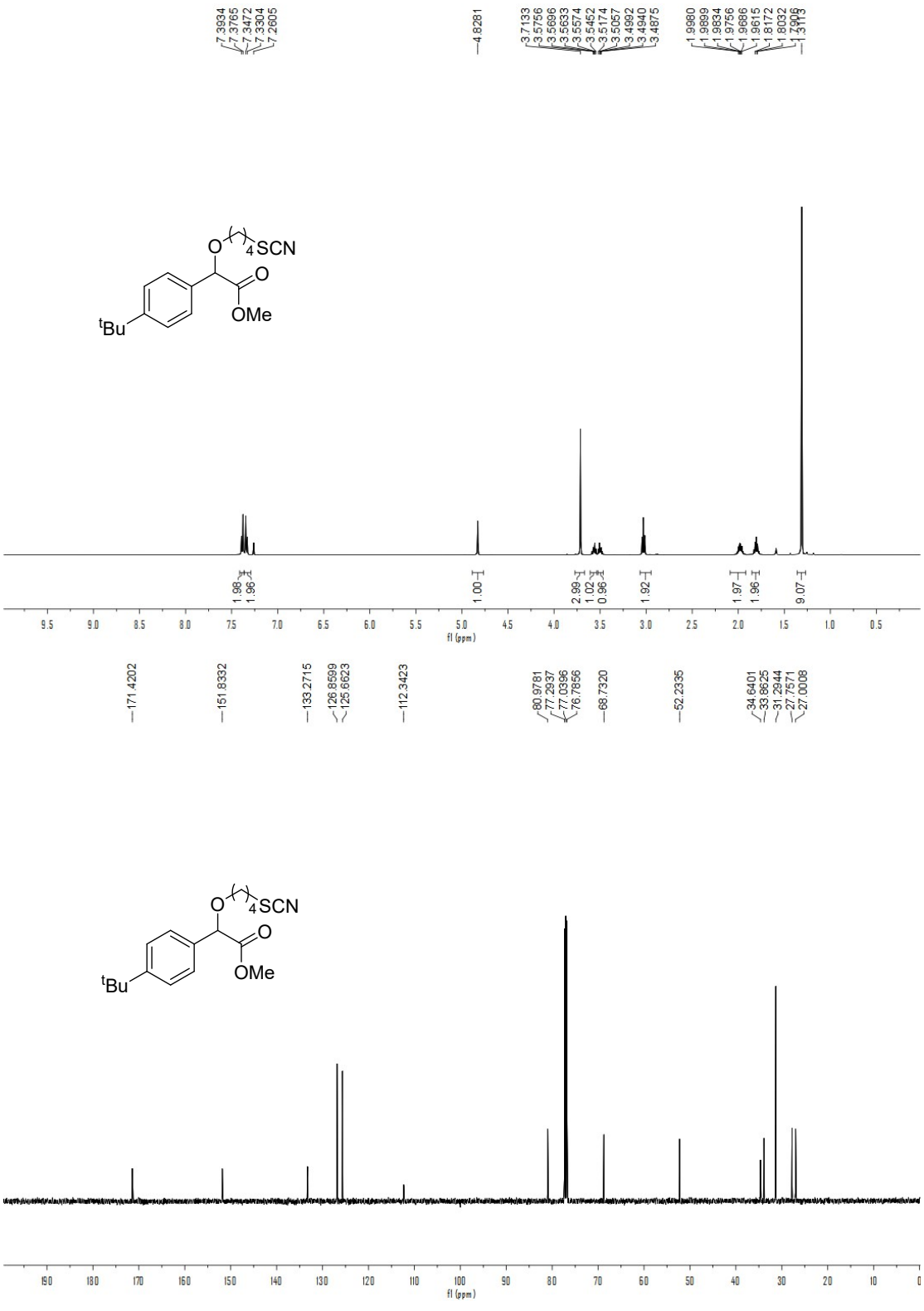
**5a**


7.3115
7.2953
7.2593
7.1837
7.1679
3.6994
3.6959
3.6869
3.5672
3.5612
3.5551
3.5490
3.5368
3.4992
3.4874
3.4809
3.4757
3.4691
3.0360
3.0216
3.0071
2.3462
1.9866
1.9776
1.9718
1.9633
1.9569
1.9492
1.8093
1.7953
1.7826
O
4
SCN
O
OMe
2.11
2.19
1.00
3.32
1.08
0.97
1.89
3.34
1.93
1.98
f1 (ppm)
171.3778
138.7186
133.3901
129.4084
129.3347
127.1468
112.3460
80.9785
77.3296
77.0754
76.8212
68.6466
52.2411
33.8585
27.7347
27.0045
21.2213
f1 (ppm)

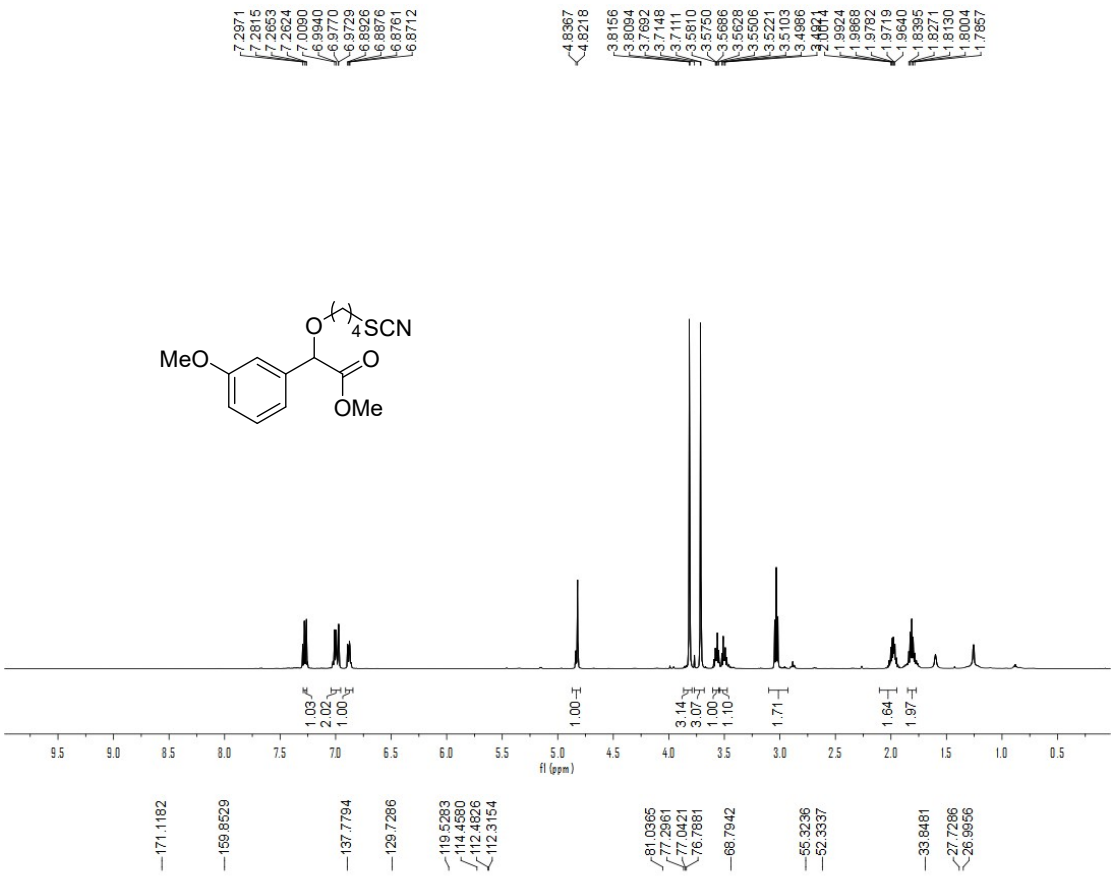
**5b**


7.3115
7.2953
7.2593
7.1837
7.1679
3.6994
3.6959
3.6869
3.5672
3.5612
3.5551
3.5490
3.5368
3.4992
3.4874
3.4809
3.4757
3.4691
3.0360
3.0216
3.0071
2.3462
1.9866
1.9776
1.9718
1.9633
1.9569
1.9492
1.8093
1.7953
1.7826
O
4
SCN
O
OMe
2.11
2.19
1.00
3.32
1.08
0.97
1.89
3.34
1.93
1.98
f1 (ppm)
171.3373
138.4967
136.2226
129.6113
128.5947
127.7792
124.2999
112.3273
81.1933
77.3031
77.0491
76.7949
68.7542
52.2804
33.8524
27.7401
26.9841
21.4176
O
4
SCN
O
OMe
f1 (ppm)

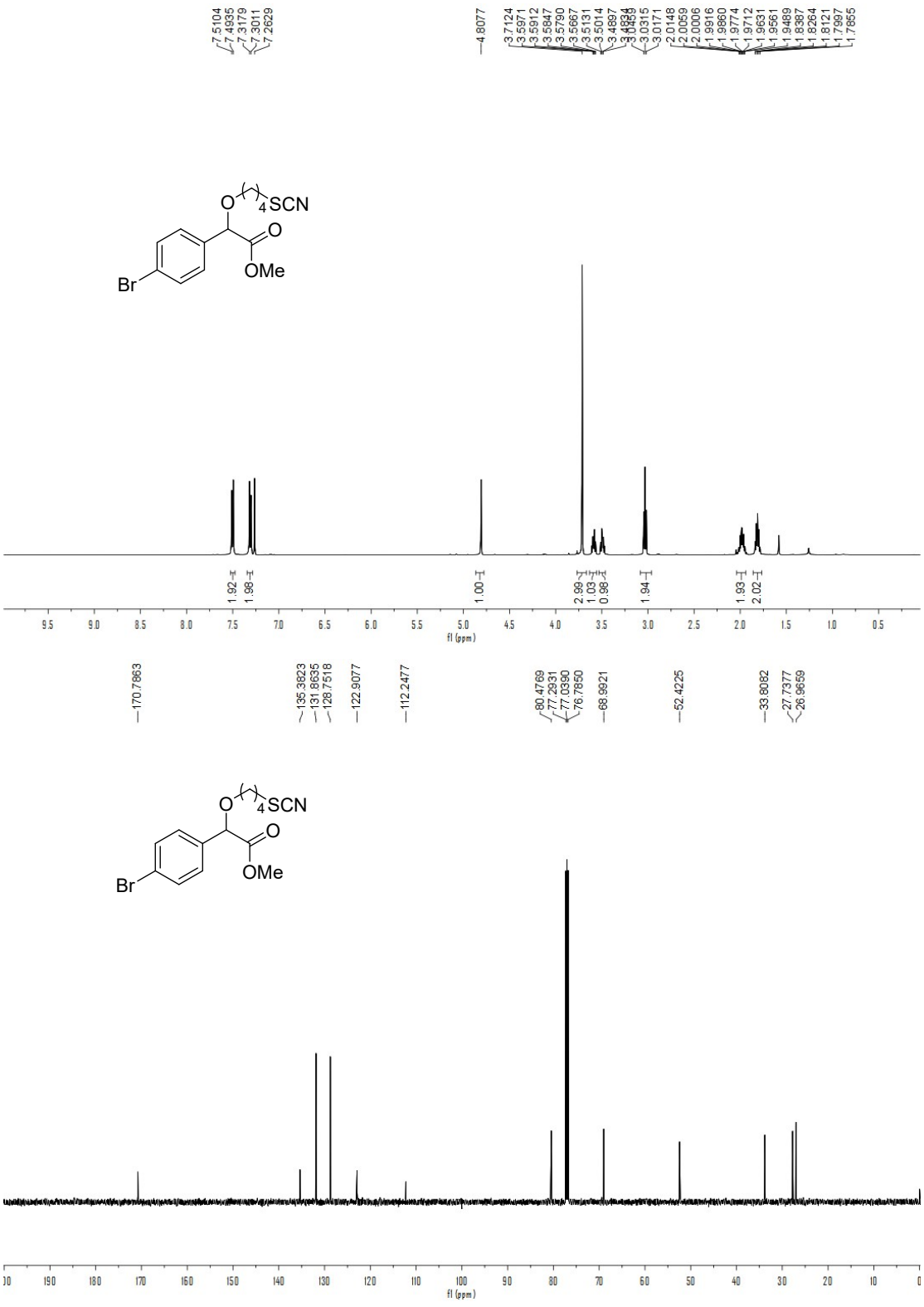
**5c**


7.3934
7.3765
7.3472
7.3304
7.2605
4.8281
3.7133
3.5756
3.5696
3.5633
3.5574
3.5452
3.5174
3.5057
3.4992
3.4940
3.4875
1.9980
1.9899
1.9834
1.9756
1.9686
1.9615
1.8172
1.8032
1.3115
O
4SCN
O
OMe
tBu
1.98
1.96
1.00
2.99
1.02
0.96
1.92
1.97
1.96
9.07
f1 (ppm)
171.4202
151.8332
133.2715
126.8599
125.6623
112.3423
80.9781
77.2937
77.0396
76.7856
68.7320
52.2335
34.6401
33.8625
31.2944
27.7571
27.0008
f1 (ppm)

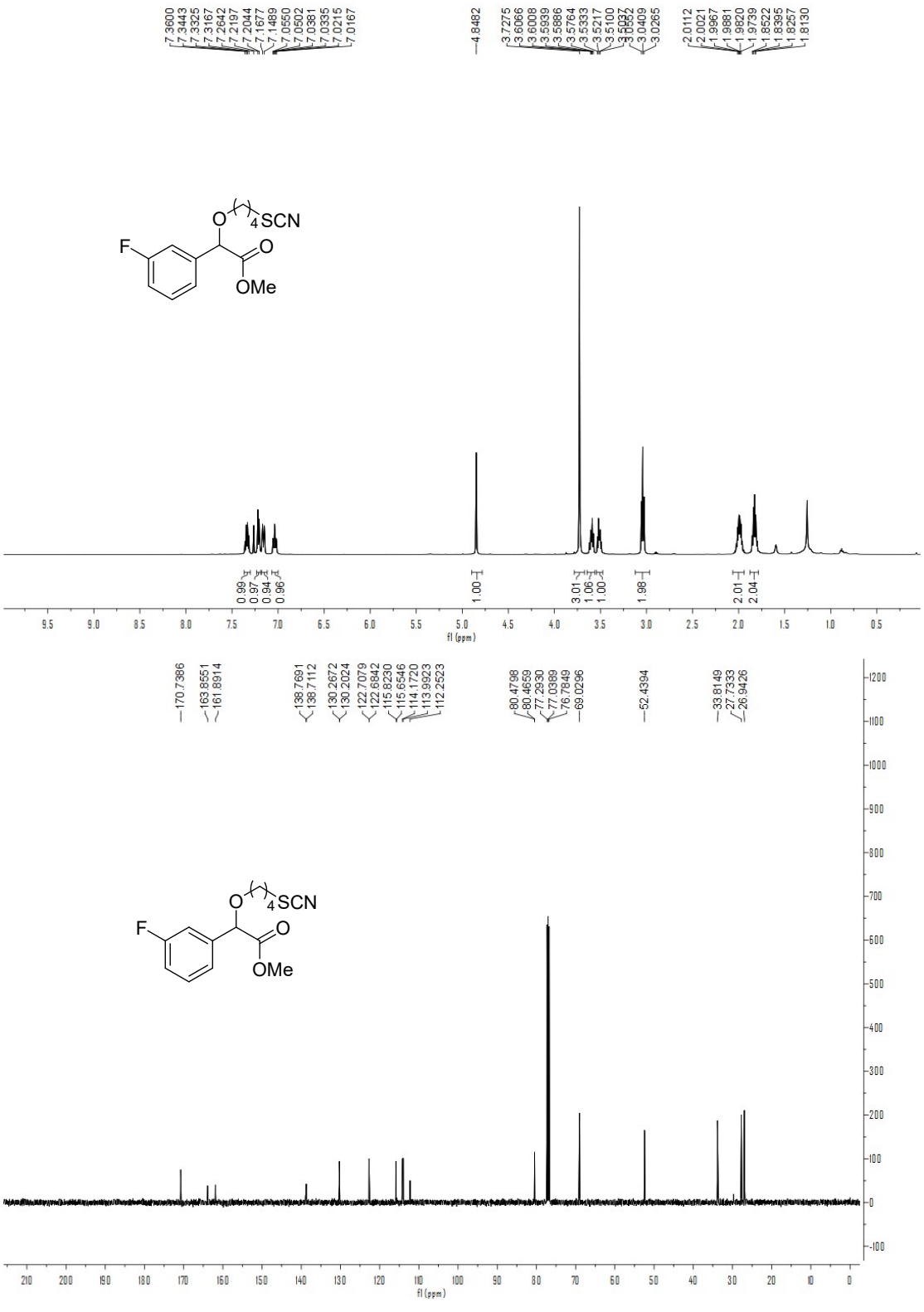
**5d**


7.2971
7.2815
7.2653
7.2624
7.0090
6.9940
6.9770
6.9729
6.8926
6.8876
6.8761
6.8712
4.8367
4.8218
3.8156
3.8094
3.7692
3.7148
3.7111
3.5810
3.5750
3.5686
3.5628
3.5506
3.5221
3.5103
3.4986
1.9924
1.9868
1.9782
1.9719
1.9640
1.8395
1.8271
1.8130
1.8004
1.7857
O
4SCN
MeO
O
OMe
1.03
2.02
1.00
1.00
3.14
3.07
1.00
1.10
1.71
1.64
1.97
f1 (ppm)
171.1182
159.8529
137.7794
129.7286
119.5283
114.4580
112.4826
112.3154
81.0365
77.2961
77.0421
76.7881
68.7942
55.3236
52.3337
33.8481
27.7286
26.9956
f1 (ppm)

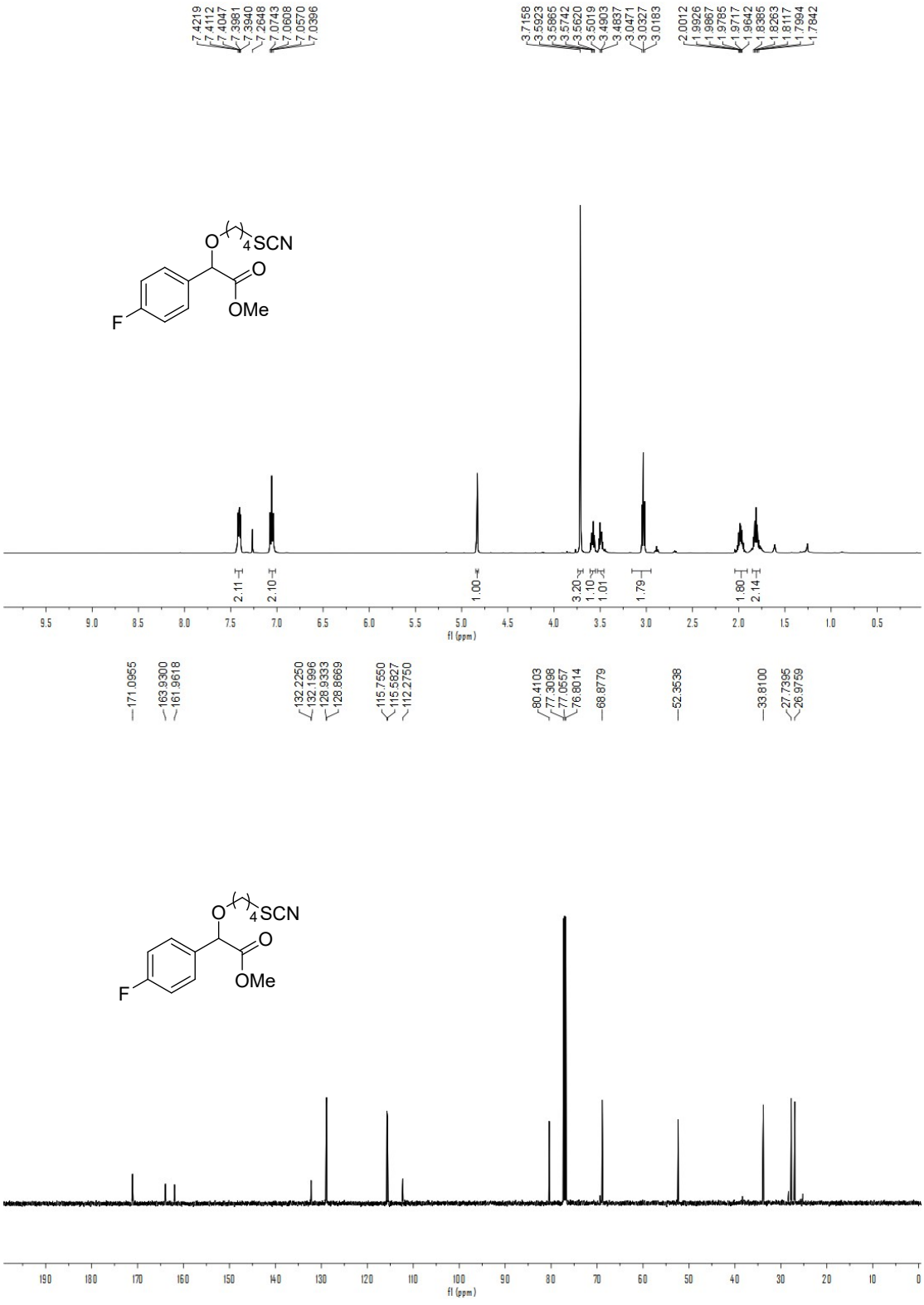
**5e**


7.5104
7.4935
7.3179
7.3011
7.2629
4.8077
3.7124
3.5971
3.5912
3.5847
3.5790
3.5667
3.5131
3.5014
3.4897
3.4834
3.4459
3.0315
3.0171
2.0148
2.0059
2.0006
1.9916
1.9860
1.9774
1.9712
1.9631
1.9561
1.9489
1.8387
1.8264
1.8121
1.7997
1.7855
O
4
SCN
O
OMe
Br
1.92
1.98
1.00
2.99
1.03
0.98
1.94
1.93
2.02
f1 (ppm)
170.7863
135.3823
131.8635
128.7518
122.9077
112.2477
80.4769
77.2931
77.0390
76.7850
68.9921
52.4225
33.8082
27.7377
26.9659
f1 (ppm)

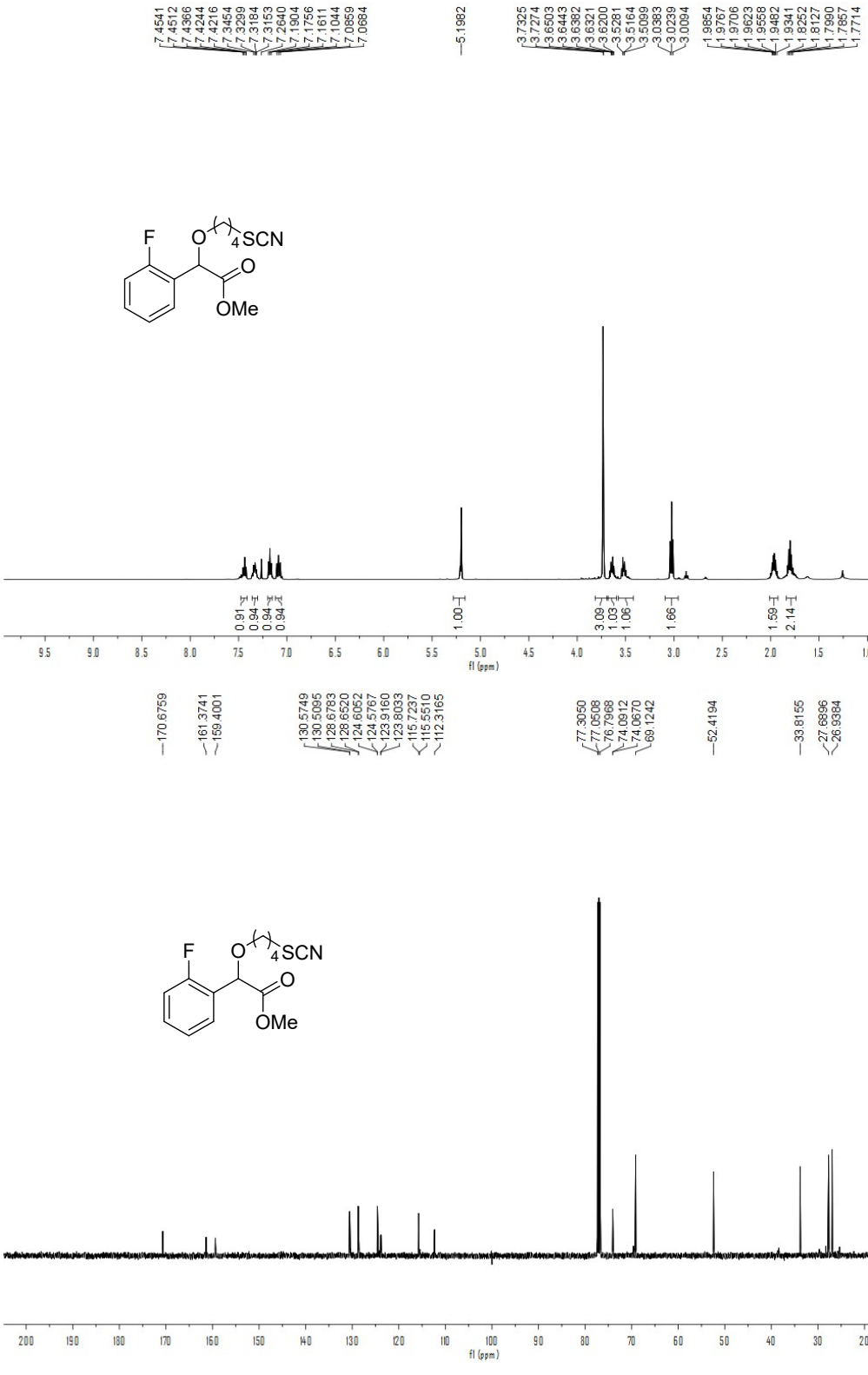
**5f**


7.3600
7.3443
7.3325
7.3167
7.2642
7.2197
7.2044
7.1677
7.1489
7.0550
7.0502
7.0381
7.0335
7.0215
7.0167
4.8482
3.7275
3.6066
3.6008
3.5939
3.5886
3.5764
3.5533
3.5217
3.5100
3.0552
3.0409
3.0265
2.0112
2.0021
1.9967
1.9881
1.9820
1.9739
1.8522
1.8395
1.8257
1.8130
O
4SCN
F
O
OMe
0.99
0.97
0.94
0.96
1.00
3.01
1.06
1.00
1.98
2.01
2.04
f1 (ppm)
170.7366
163.8551
161.8914
138.7691
138.7112
130.2672
130.2024
122.7079
122.6842
115.8230
115.6546
114.1720
113.9923
112.2523
80.4798
80.4659
77.2930
77.0389
76.7849
69.0296
52.4394
33.8149
27.7333
26.9426
f1 (ppm)

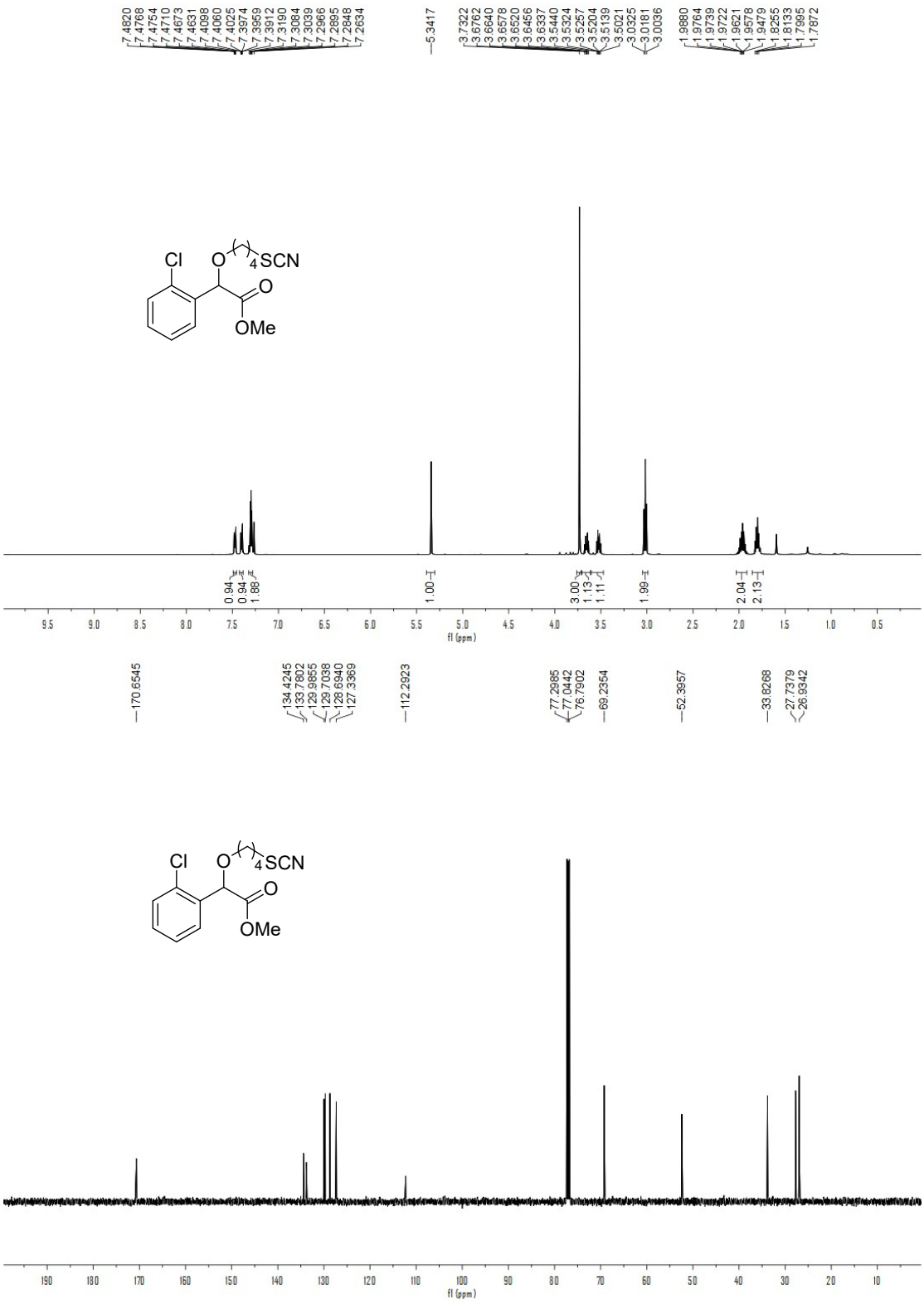
**5g**


7.4219
7.4112
7.4047
7.3981
7.3940
7.2648
7.0743
7.0608
7.0570
7.0396
3.7158
3.5923
3.5865
3.5742
3.5620
3.5019
3.4903
3.4837
3.0471
3.0327
3.0183
2.0012
1.9926
1.9867
1.9785
1.9717
1.9642
1.8385
1.8263
1.8117
1.7994
1.7842
O
4
SCN
O
OMe
F
2.11
2.10
1.00
3.20
1.10
1.01
1.79
1.80
2.14
f1 (ppm)
171.0955
163.9300
161.9618
132.2250
132.1996
128.9333
128.8669
115.7550
115.5827
112.2750
80.4103
77.3098
77.0557
76.8014
68.8779
52.3538
33.8100
27.7395
26.9759
f1 (ppm)

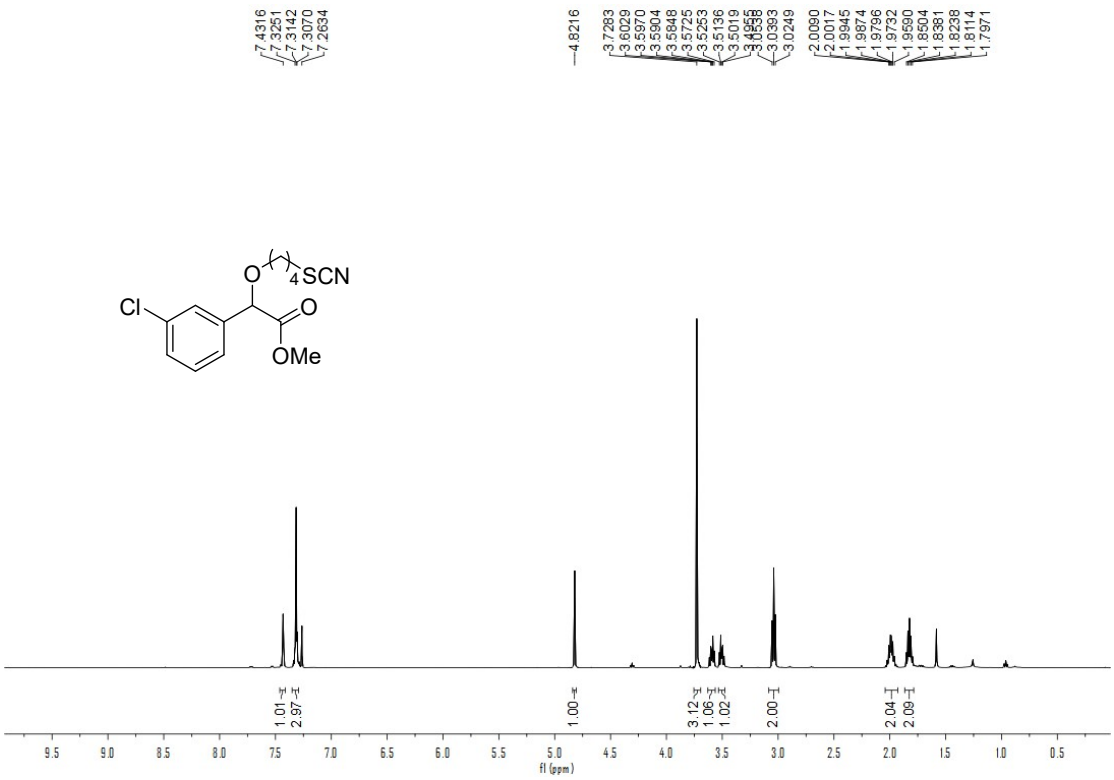
**5h**

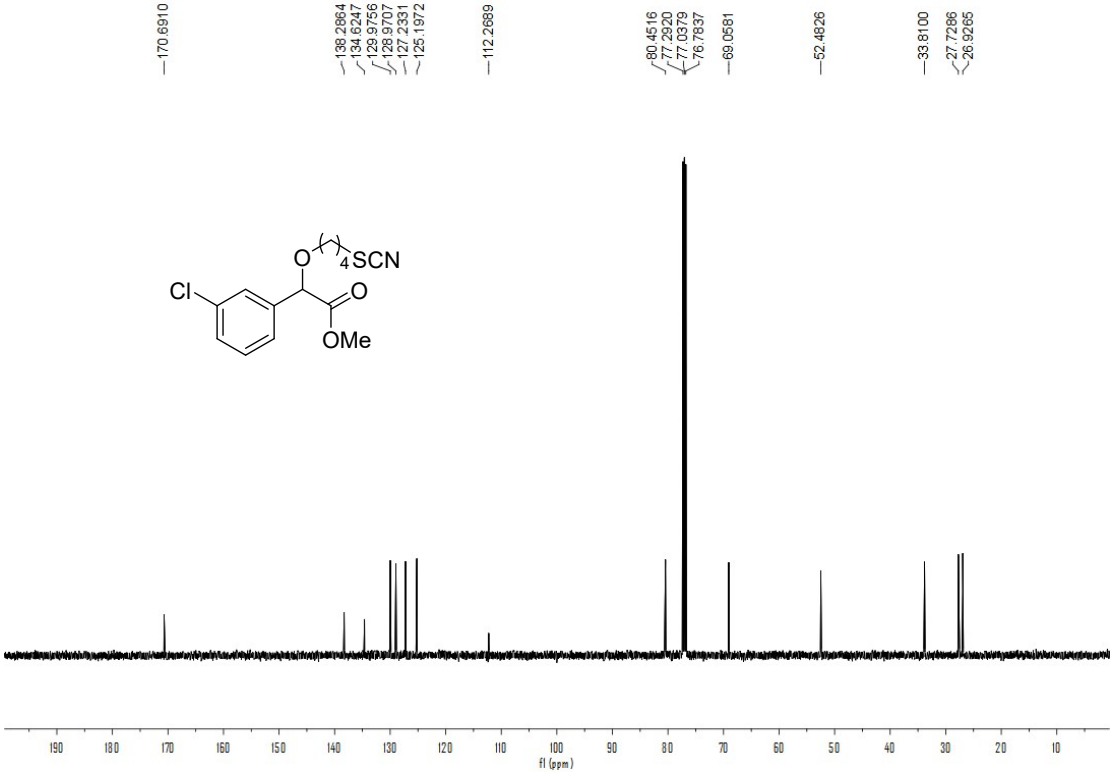

7.4541
7.4512
7.4366
7.4244
7.4216
7.3454
7.3299
7.3184
7.3153
7.2640
7.1904
7.1756
7.1611
7.1044
7.0859
7.0684
5.1982
3.7325
3.7274
3.6503
3.6443
3.6382
3.6321
3.6200
3.5281
3.5164
3.5099
3.0383
3.0239
3.0094
1.9854
1.9767
1.9706
1.9623
1.9558
1.9482
1.9341
1.8252
1.8127
1.7990
1.7857
1.7714
F
O
4SCN
O
OMe
0.91
0.94
0.94
0.94
1.00
3.09
1.03
1.06
1.66
1.59
2.14
f1 (ppm)
170.6759
161.3741
159.4001
130.5749
130.5095
128.6783
128.6520
124.6052
124.5767
123.9160
123.8033
115.7237
115.5510
112.3165
77.3050
77.0508
76.7968
74.0912
74.0670
69.1242
52.4194
33.8155
27.6896
26.9384
F
O
4SCN
O
OMe
f1 (ppm)

**5i**

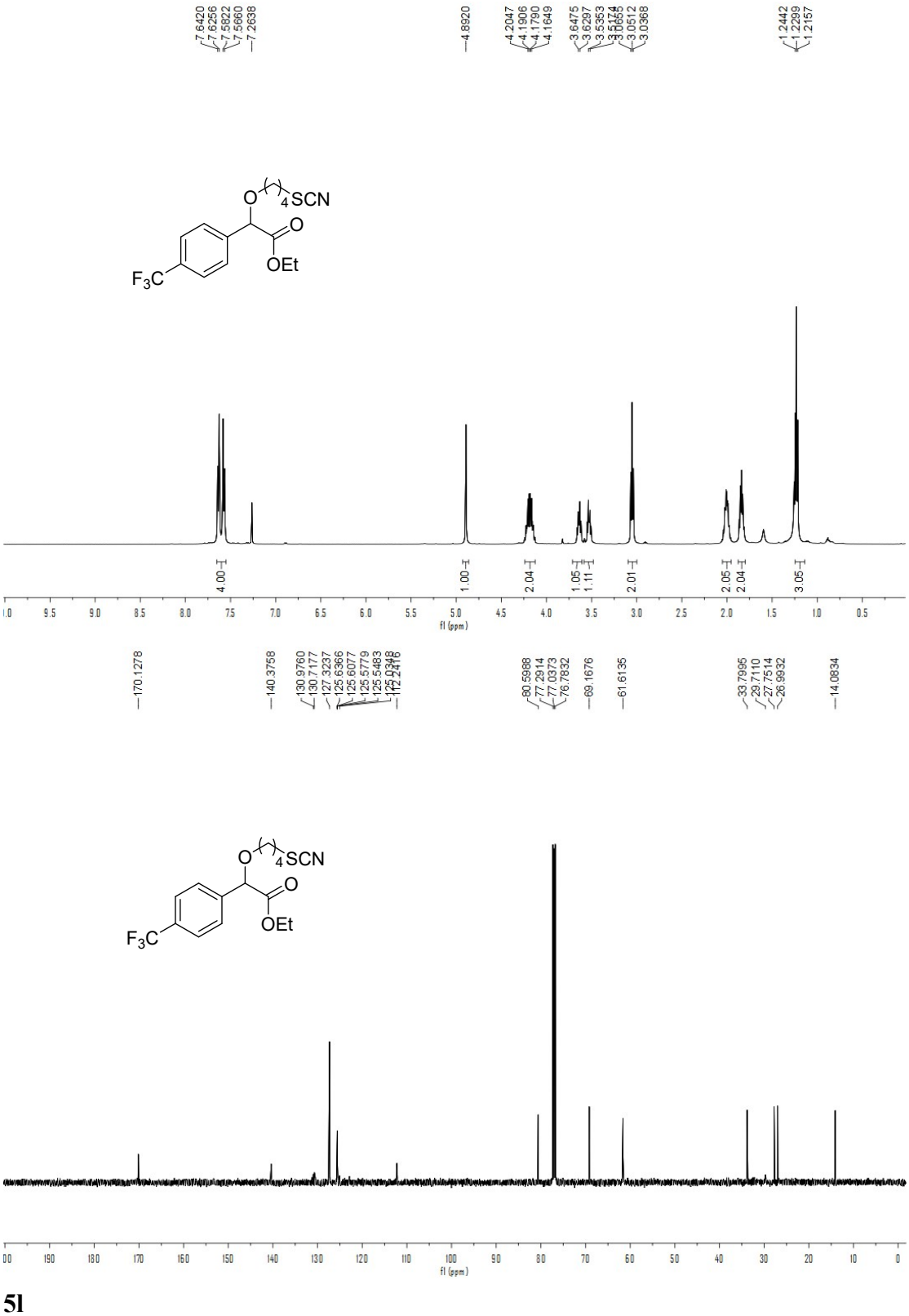

7.4820
7.4768
7.4754
7.4710
7.4673
7.4631
7.4098
7.4060
7.4025
7.3974
7.3959
7.3912
7.3190
7.3084
7.3039
7.2966
7.2895
7.2848
7.2634
5.3417
3.7322
3.6762
3.6640
3.6578
3.6520
3.6456
3.6337
3.5440
3.5324
3.5257
3.5204
3.5139
3.5021
3.0325
3.0181
3.0036
1.9880
1.9764
1.9739
1.9722
1.9621
1.9578
1.9479
1.8255
1.8133
1.7995
1.7872
Cl
O
4SCN
O
OMe
0.94
0.94
1.88
1.00
3.00
1.13
1.11
1.99
2.04
2.13
f1 (ppm)
170.6545
134.4245
133.7802
129.9855
129.7038
128.6940
127.3369
112.2923
77.2985
77.0442
76.7902
69.2354
52.3957
33.8268
27.7379
26.9342
f1 (ppm)

**5j**

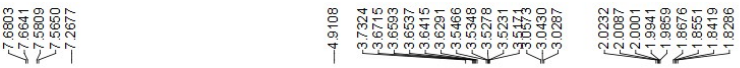

7.4316
7.3251
7.3142
7.3070
7.2634
4.8216
3.7283
3.6029
3.5970
3.5904
3.5848
3.5725
3.5253
3.5136
3.5019
3.0393
3.0249
2.0090
2.0017
1.9945
1.9874
1.9796
1.9732
1.9590
1.8504
1.8381
1.8238
1.8114
1.7971
O
4SCN
Cl
O
OMe
1.01
2.97
1.00
3.12
1.06
1.02
2.00
2.04
2.09
f1 (ppm)

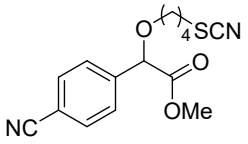

170.6910
138.2864
134.6247
129.9756
128.9707
127.2331
125.1972
112.2689
80.4516
77.2920
77.0379
76.7837
69.0581
52.4826
33.8100
27.7286
26.9265
O
4SCN
Cl
O
OMe
f1 (ppm)

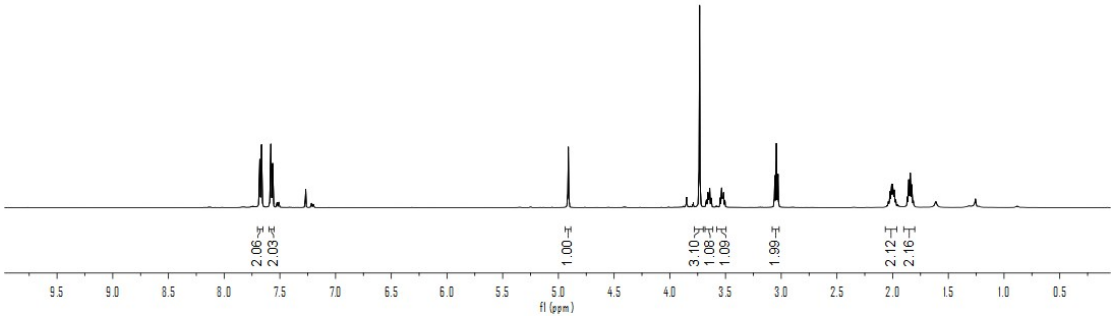
**5k**


7.6420
7.6256
7.5822
7.5660
7.2638
4.8920
4.2047
4.1906
4.1790
4.1649
3.6475
3.6297
3.5353
3.5174
3.0655
3.0512
3.0368
1.2442
1.2299
1.2157
O
4SCN
O
OEt
F3C
4.00
1.00
2.04
1.05
1.11
2.01
2.05
2.04
3.05
f1 (ppm)
170.1278
140.3758
130.9760
130.7177
127.3237
125.6366
125.6077
125.5779
125.5483
125.0348
122.2416
80.5988
77.2914
77.0373
76.7832
69.1676
61.6135
33.7995
29.7110
27.7514
26.9932
14.0834

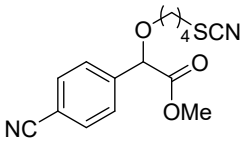
**5l**

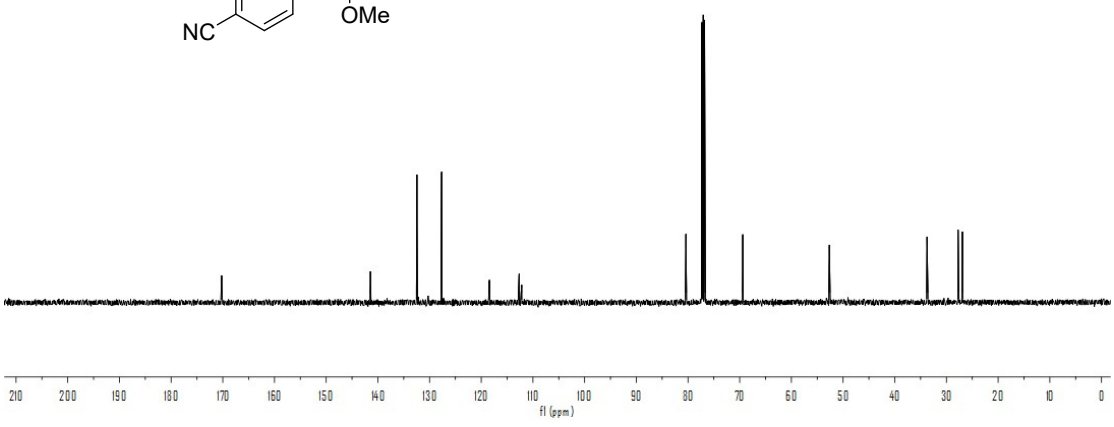

7.6803
7.6641
7.5809
7.5650
7.2677
4.9108
3.7324
3.6715
3.6593
3.6537
3.6415
3.6291
3.5466
3.5348
3.5278
3.5231
3.5171
3.0573
3.0430
3.0287
2.0232
2.0087
2.0001
1.9941
1.9859
1.8676
1.8551
1.8419
1.8286


O
4
SCN
O
OMe
NC

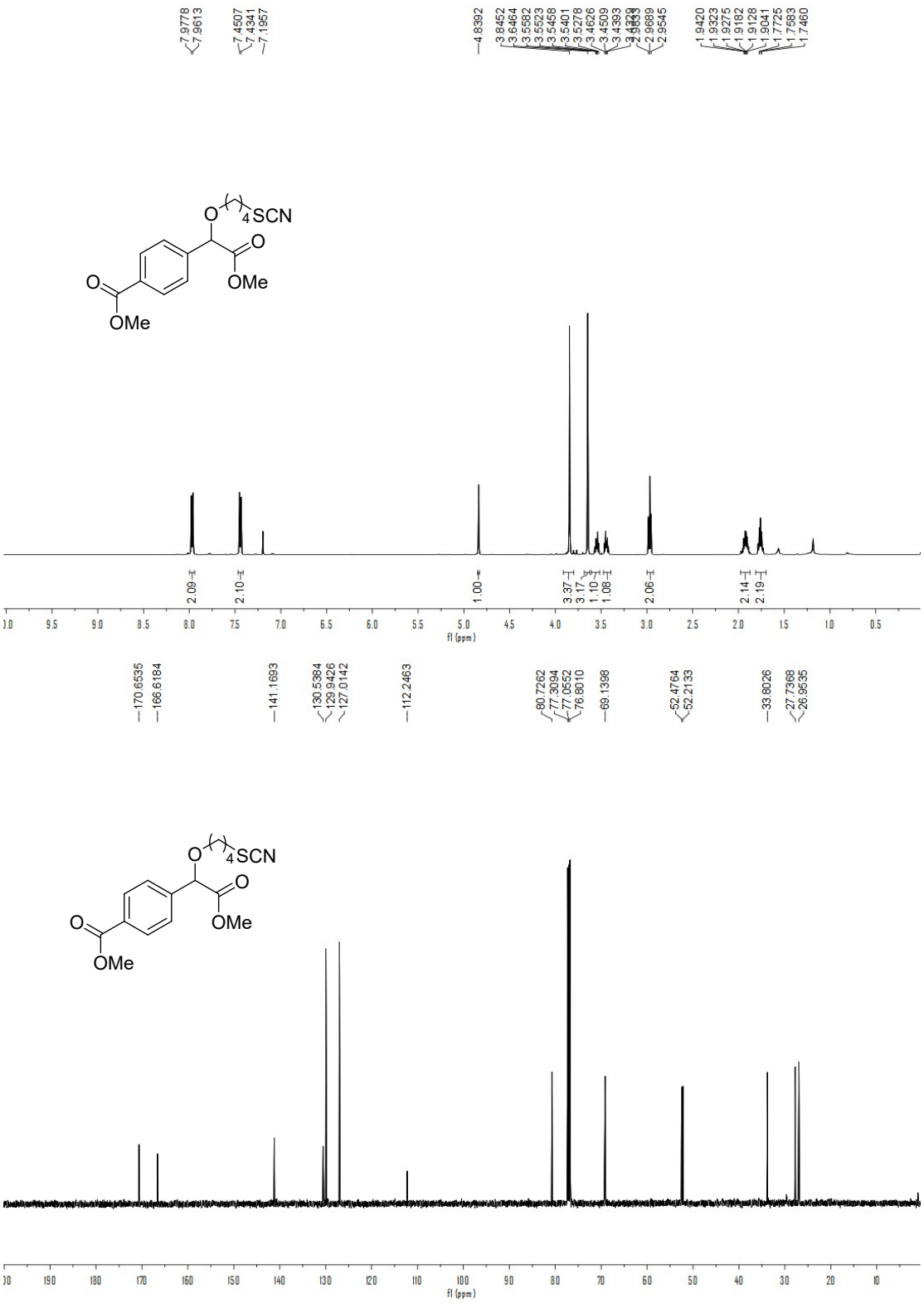

2.06
2.03
1.00
3.10
1.08
1.09
1.99
2.12
2.16
f1 (ppm)


170.2351
141.4280
132.4568
127.6520
118.4239
112.6642
112.1748
80.3974
77.3002
77.0461
76.7921
69.3910
52.6457
33.7458
27.7502
26.9250

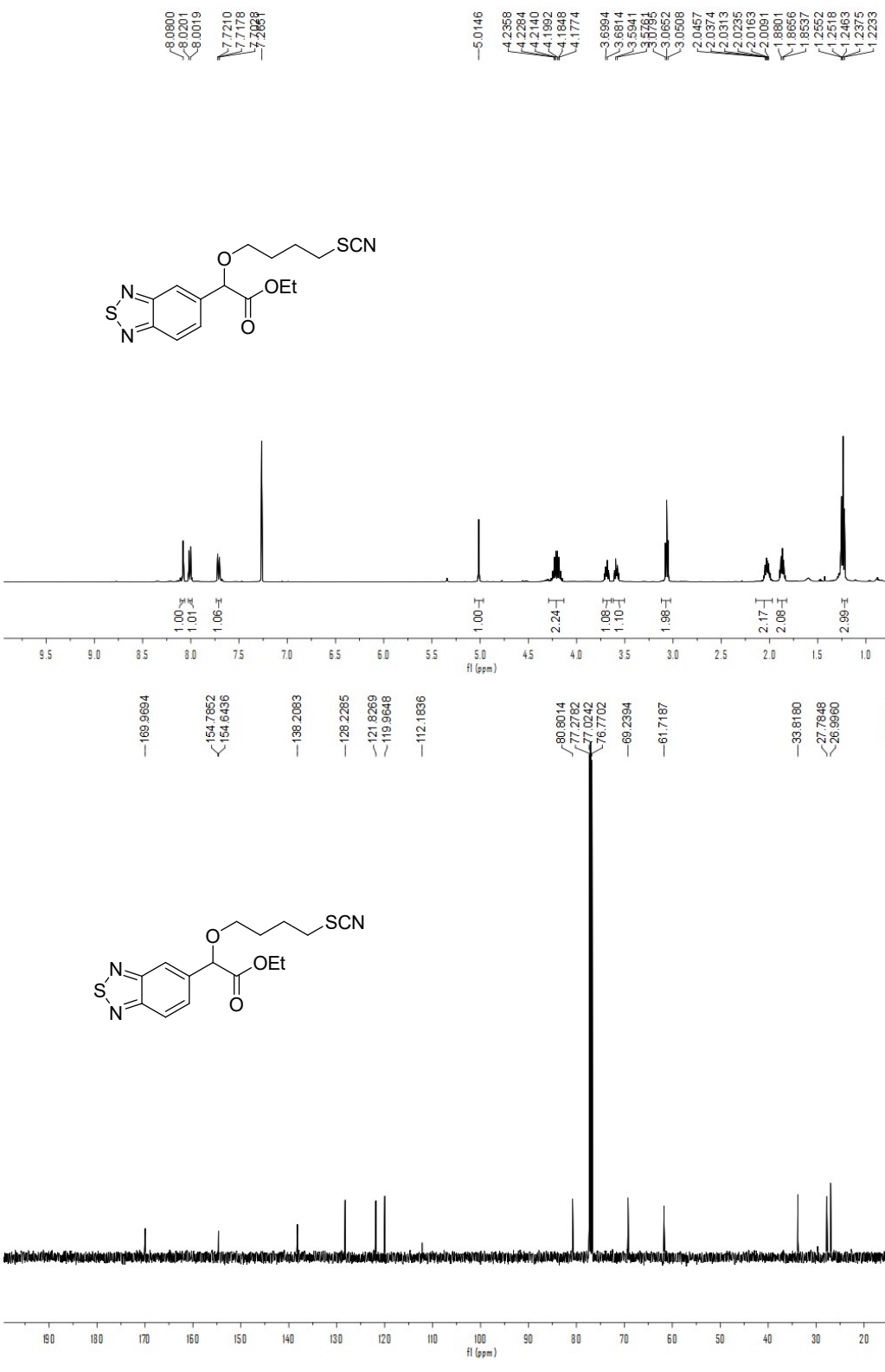

O
4
SCN
O
OMe
NC


f1 (ppm)

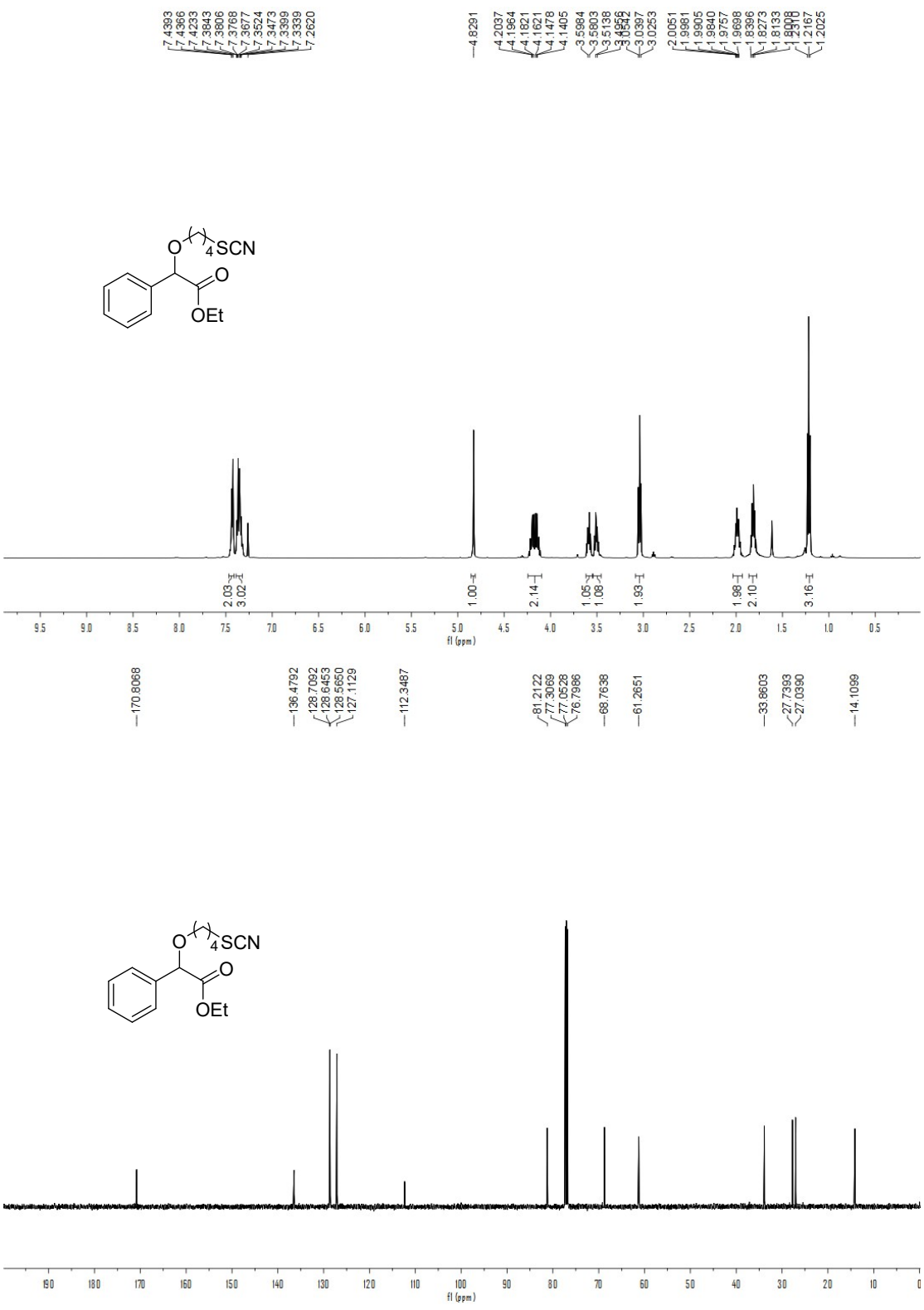
**5m**


7.9778
7.9613
7.4507
7.4341
7.1957
4.8392
3.8452
3.6464
3.5582
3.5523
3.5458
3.5401
3.5278
3.4626
3.4509
3.4393
2.9689
2.9545
1.9420
1.9323
1.9275
1.9182
1.9128
1.9041
1.7725
1.7583
1.7460
O
4SCN
O
O
OMe
OMe
2.09
2.10
1.00
3.37
3.17
1.10
1.08
2.06
2.14
2.19
f1 (ppm)
170.6535
166.6184
141.1693
130.5384
129.9426
127.0142
112.2463
80.7262
77.3094
77.0552
76.8010
69.1398
52.4764
52.2133
33.8026
27.7368
26.9535
f1 (ppm)

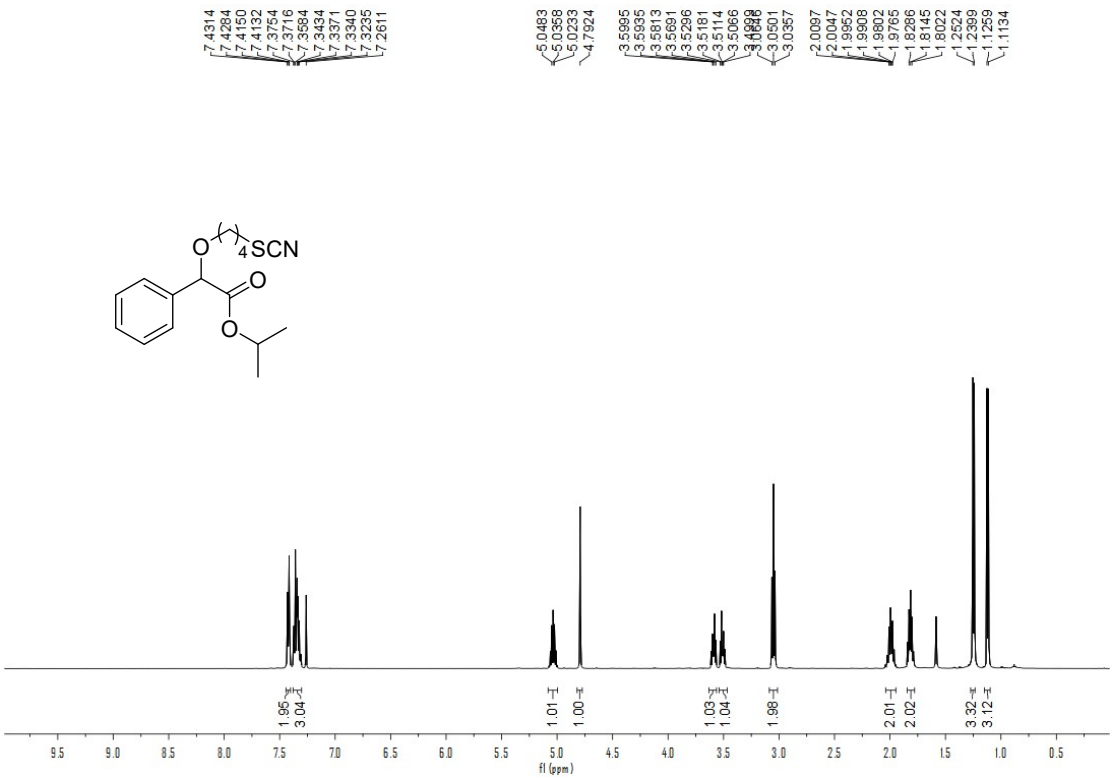
**5n**

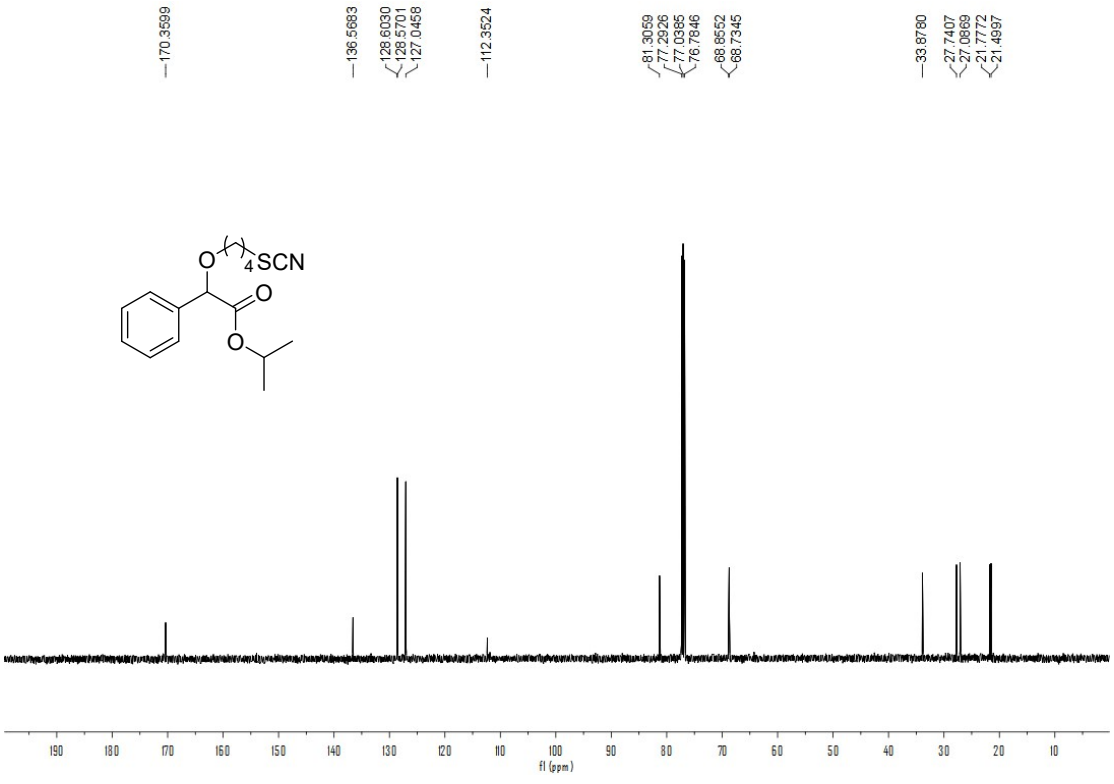

8.0800
8.0201
8.0019
7.7210
7.7178
7.7028
7.2651
5.0146
4.2358
4.2284
4.2140
4.1992
4.1848
4.1774
3.6994
3.6814
3.5941
3.5783
3.5630
3.0652
3.0508
2.0457
2.0374
2.0313
2.0235
2.0163
2.0091
1.8801
1.8656
1.8537
1.2552
1.2518
1.2463
1.2375
1.2233
SCN
O
OEt
O
N
S
N
1.00
1.01
1.06
1.00
2.24
1.08
1.10
1.98
2.17
2.08
2.99
f1 (ppm)
169.9694
154.7852
154.6436
138.2083
128.2285
121.8269
119.9648
112.1836
80.8014
77.2782
77.0242
76.7702
69.2394
61.7187
33.8180
27.7848
26.9960
14.1105
SCN
O
OEt
O
N
S
N
f1 (ppm)

**5o**

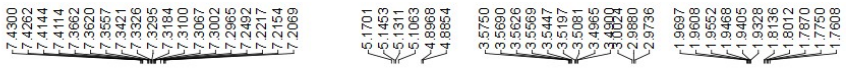

7.4393
7.4366
7.4233
7.3843
7.3806
7.3768
7.3677
7.3524
7.3473
7.3399
7.3339
7.2620
4.8291
4.2037
4.1964
4.1821
4.1621
4.1478
4.1405
3.5984
3.5803
3.5138
3.0397
3.0253
2.0051
1.9981
1.9905
1.9840
1.9757
1.9698
1.8396
1.8273
1.8133
1.2310
1.2167
1.2025
O
4SCN
O
OEt
2.03
3.02
1.00
2.14
1.05
1.08
1.93
1.98
2.10
3.16
f1 (ppm)
170.8068
136.4792
128.7092
128.6453
128.5650
127.1129
112.3487
81.2122
77.3069
77.0528
76.7986
68.7638
61.2651
33.8603
27.7393
27.0390
14.1099
f1 (ppm)

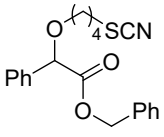
**5p**

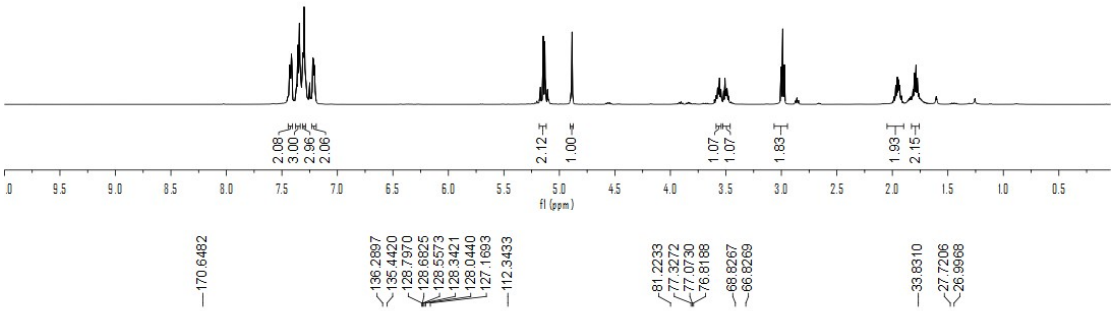

7.4314
7.4284
7.4150
7.4132
7.3754
7.3716
7.3584
7.3434
7.3371
7.3340
7.3235
7.2611
5.0483
5.0358
5.0233
4.7924
3.5995
3.5935
3.5813
3.5691
3.5296
3.5181
3.5114
3.5066
3.0501
3.0357
2.0097
2.0047
1.9952
1.9908
1.9802
1.9765
1.8286
1.8145
1.8022
1.2524
1.2399
1.1259
1.1134
O
4SCN
O
O
1.95
3.04
1.01
1.00
1.03
1.04
1.98
2.01
2.02
3.32
3.12
f1 (ppm)


170.3599
136.5683
128.6030
128.5701
127.0458
112.3524
81.3059
77.2926
77.0385
76.7846
68.8552
68.7345
33.8780
27.7407
27.0869
21.7772
21.4997
O
4SCN
O
O
f1 (ppm)

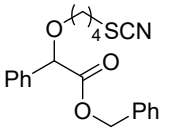
**5q**

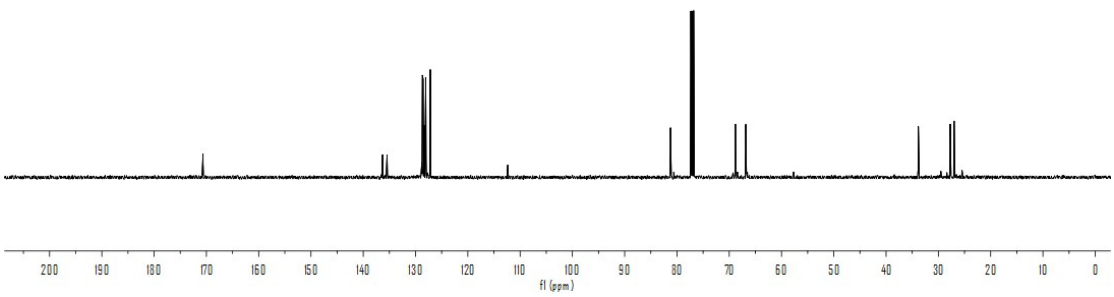



O
4SCN
Ph
O
O
Ph

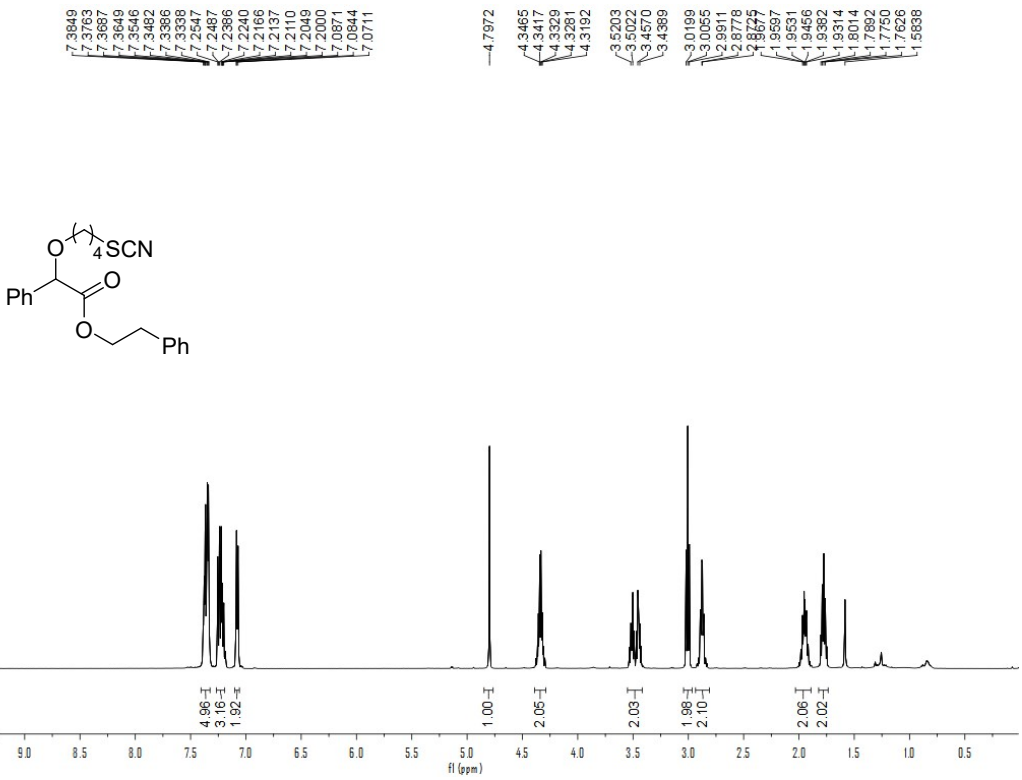

2.08
3.00
2.96
2.06
2.12
1.00
1.07
1.07
1.83
1.93
2.15
f1 (ppm)

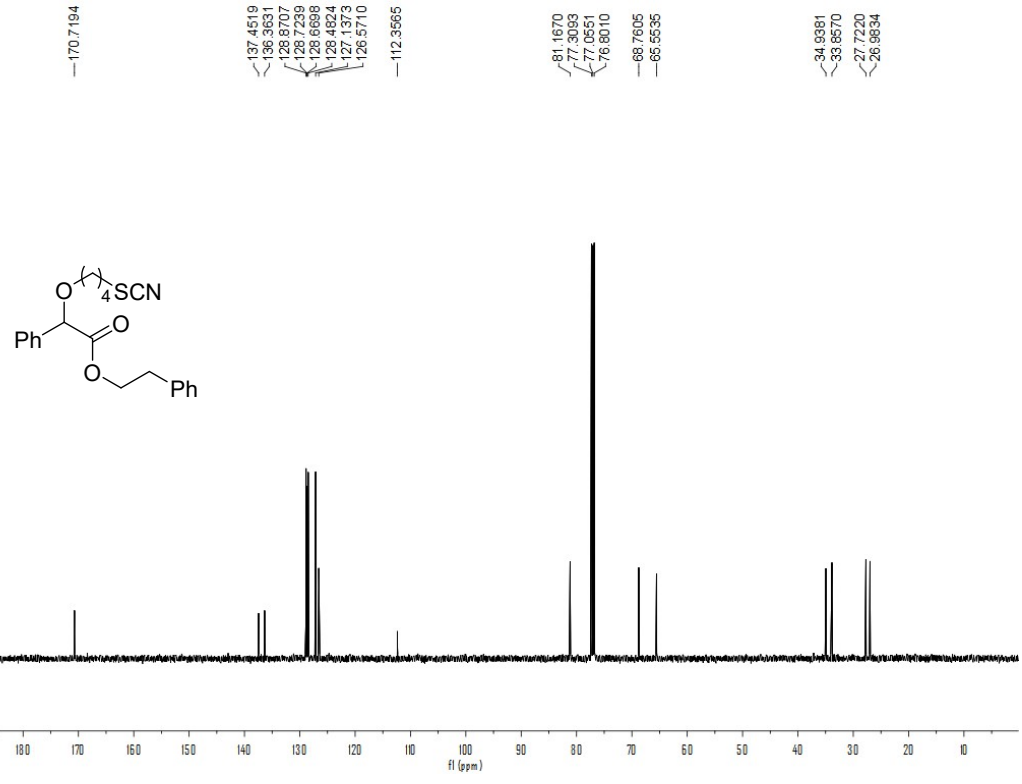

170.6482
136.2897
135.4420
128.7970
128.6825
128.5573
128.3421
128.0440
127.1693
112.3433
81.2233
77.3272
77.0730
76.8188
68.8267
66.8269
33.8310
27.7206
26.9968


O
4SCN
Ph
O
O
Ph

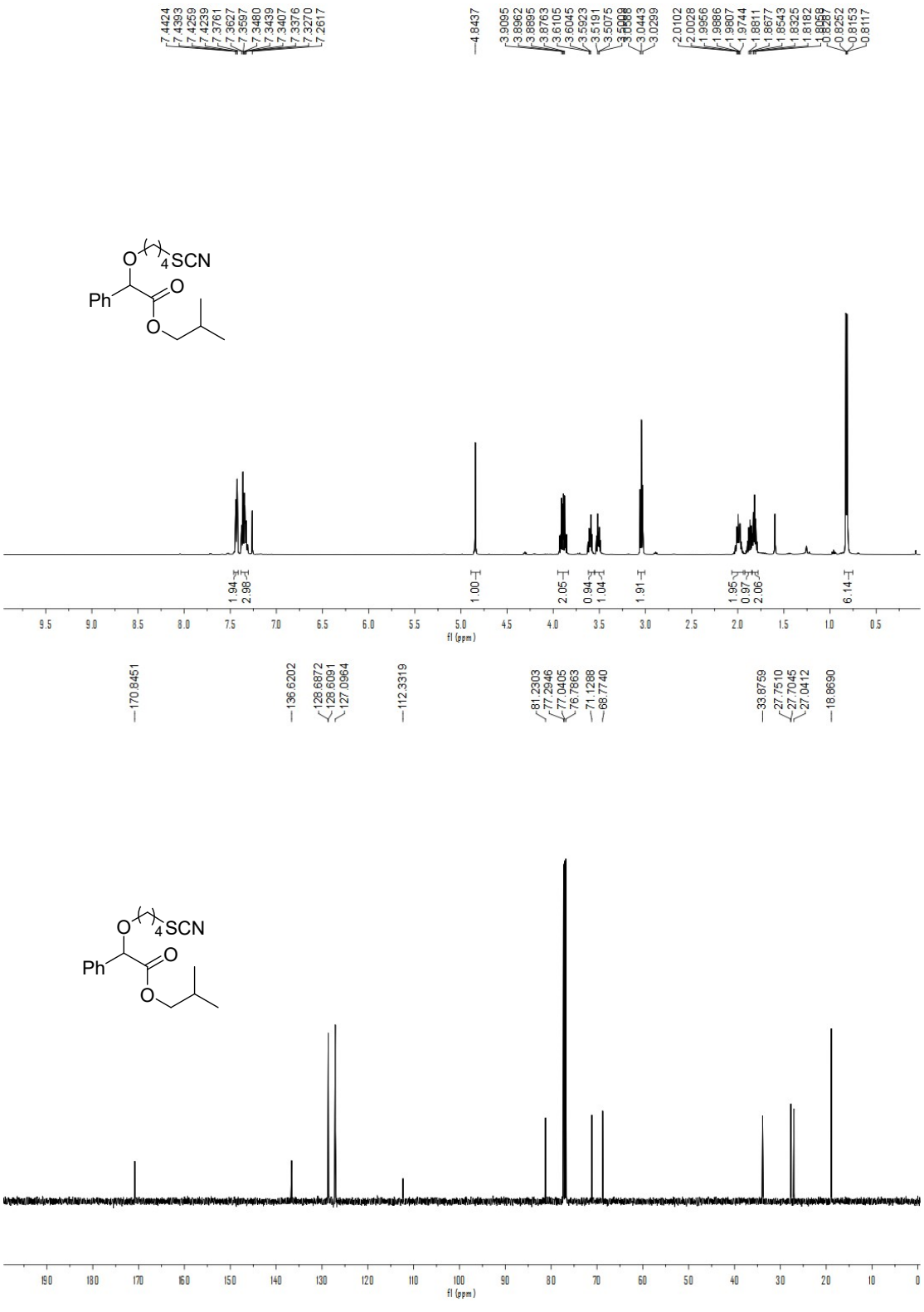

f1 (ppm)

**5r**

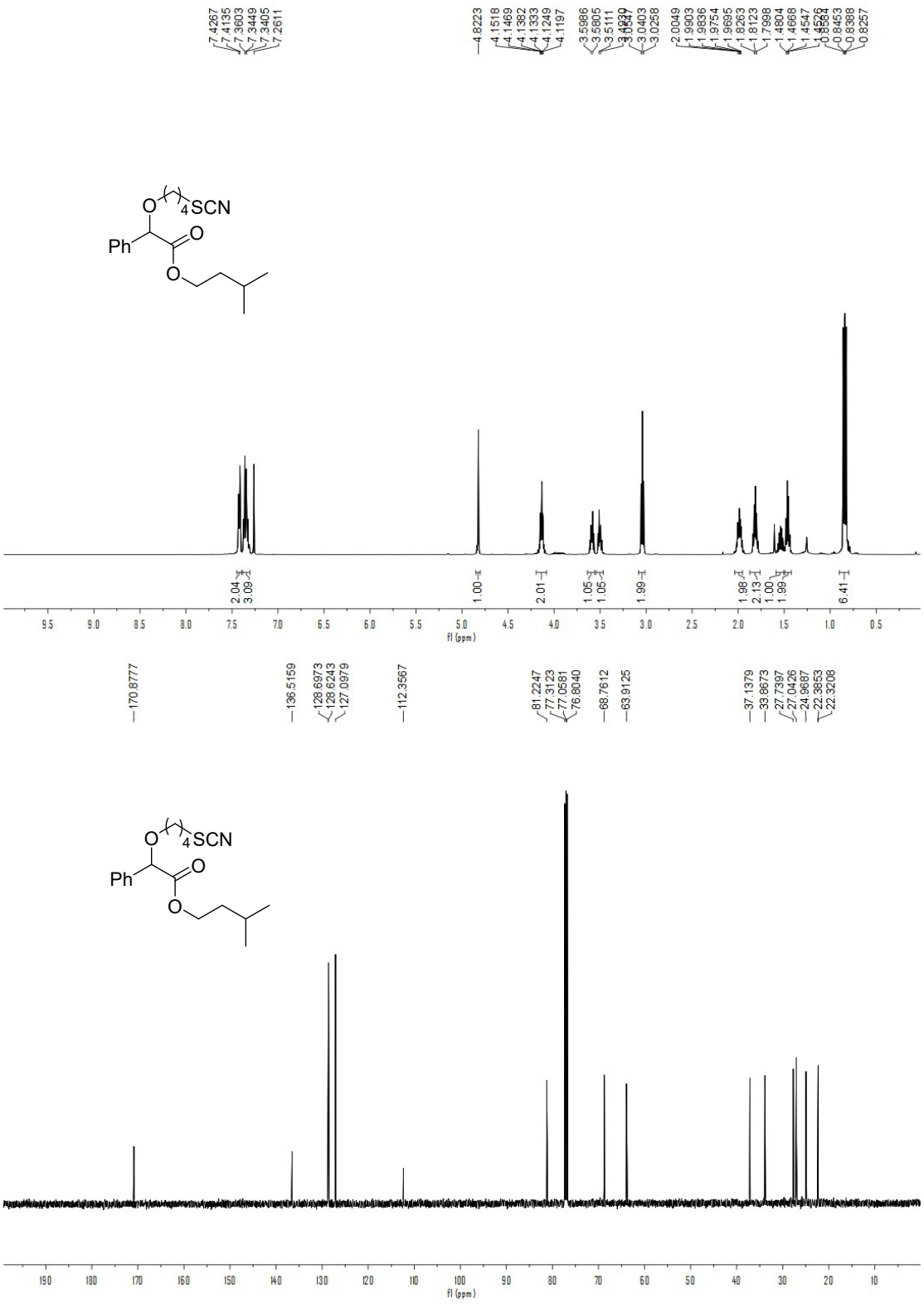

7.3849
7.3763
7.3687
7.3649
7.3546
7.3482
7.3386
7.3338
7.2547
7.2487
7.2386
7.2240
7.2166
7.2137
7.2110
7.2049
7.2000
7.0871
7.0844
7.0711
4.7972
4.3465
4.3417
4.3329
4.3281
4.3192
3.5203
3.5022
3.4570
3.4389
3.0199
3.0055
2.9911
2.8778
1.9597
1.9531
1.9456
1.9382
1.9314
1.8014
1.7892
1.7750
1.7626
1.5838
O
4
SCN
O
Ph
O
Ph
4.96
3.16
1.92
1.00
2.05
2.03
1.98
2.10
2.06
2.02
f1 (ppm)


170.7194
137.4519
136.3631
128.8707
128.7239
128.6698
128.4824
127.1373
126.5710
112.3565
81.1670
77.3093
77.0551
76.8010
68.7605
65.5535
34.9381
33.8570
27.7220
26.9834
O
4
SCN
O
Ph
O
Ph
f1 (ppm)

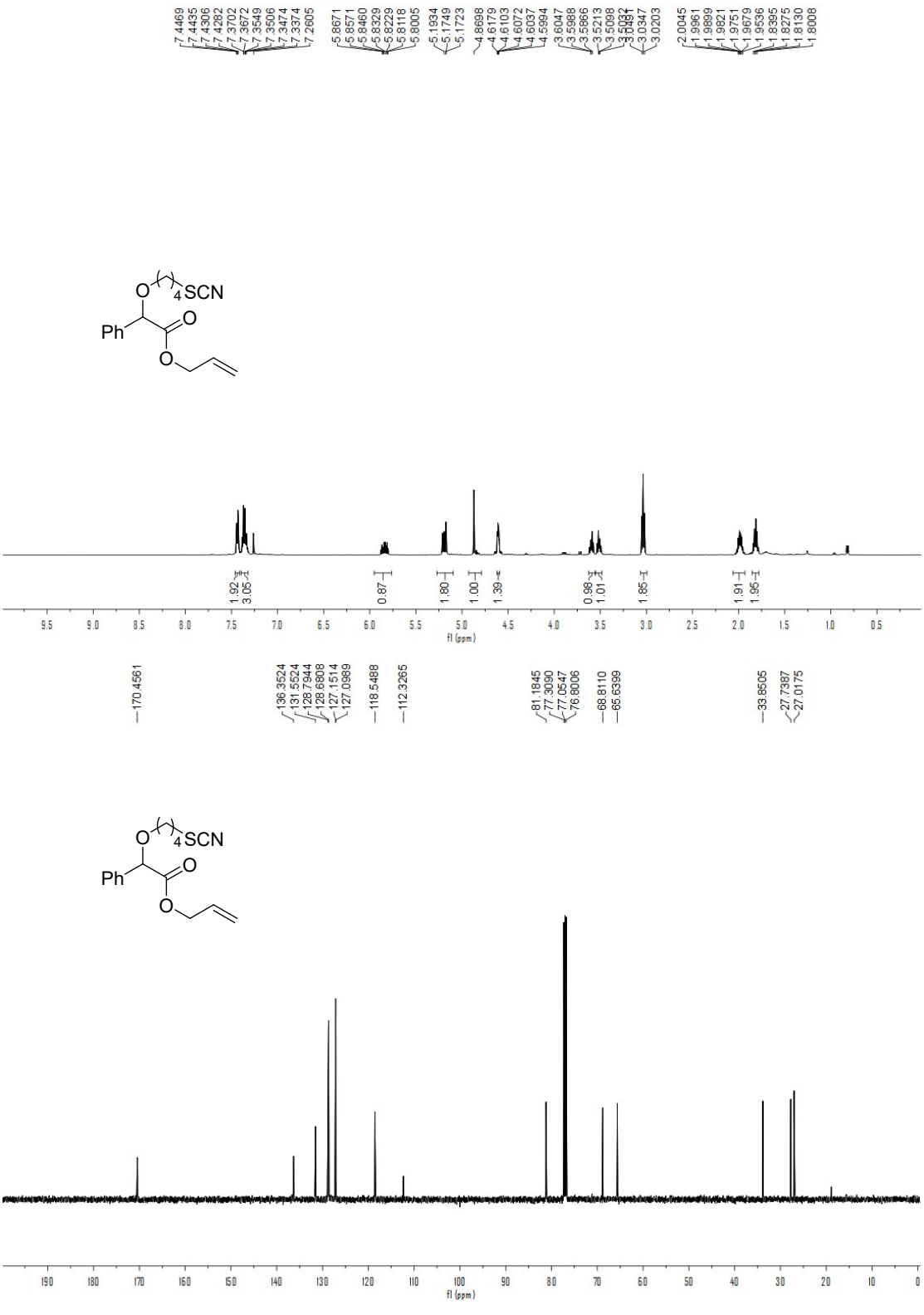
**5s**


7.4424
7.4393
7.4259
7.4239
7.3761
7.3627
7.3597
7.3480
7.3439
7.3407
7.3376
7.3270
7.2617
4.8437
3.9095
3.8962
3.8895
3.8763
3.6105
3.6045
3.5923
3.5191
3.5075
3.0443
3.0239
2.0102
2.0028
1.9956
1.9886
1.9807
1.9744
1.8811
1.8677
1.8543
1.8325
1.8182
0.8252
0.8153
0.8117
O
4SCN
Ph
O
O
1.94
2.98
1.00
2.05
0.94
1.04
1.91
1.95
0.97
2.06
6.14
f1 (ppm)
170.8451
136.6202
128.6872
128.6091
127.0964
112.3319
81.2303
77.2946
77.0405
76.7863
71.1288
68.7740
33.8759
27.7510
27.7045
27.0412
18.8690
f1 (ppm)

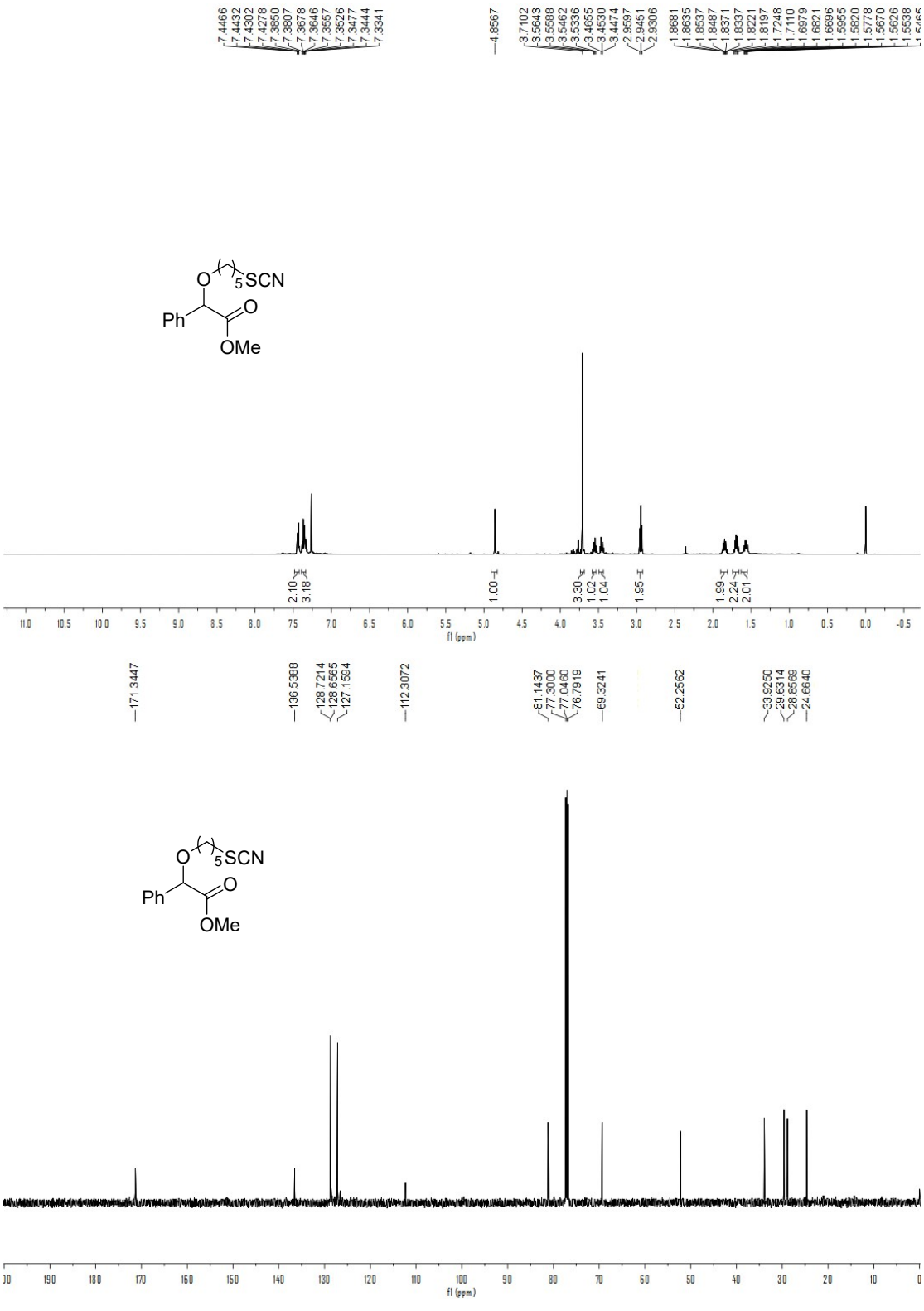
**5t**


7.4267
7.4135
7.3603
7.3449
7.3405
7.2611
4.8223
4.1518
4.1469
4.1382
4.1333
4.1249
4.1197
3.5986
3.5805
3.5111
3.0547
3.0403
3.0258
2.0049
1.9903
1.9836
1.9754
1.9695
1.8263
1.8123
1.7998
1.4804
1.4668
1.4547
0.9526
0.9453
0.8388
0.8257
O
4SCN
Ph
O
O
2.04
3.09
1.00
2.01
1.05
1.05
1.99
1.98
2.13
1.00
1.99
6.41
f1 (ppm)
170.8777
136.5159
128.6973
128.6243
127.0979
112.3567
81.2247
77.3123
77.0581
76.8040
68.7612
63.9125
37.1379
33.8673
27.7397
27.0426
24.9687
22.3853
22.3208
f1 (ppm)

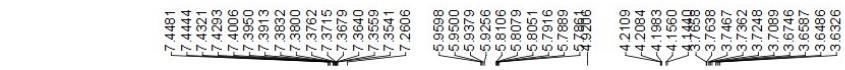
**5u**

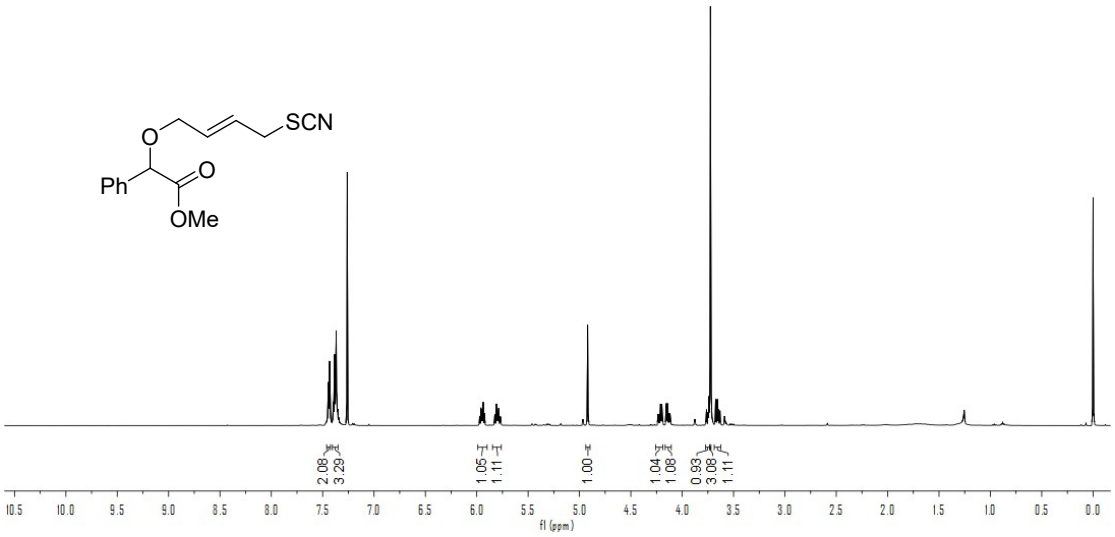

7.4469
7.4435
7.4306
7.4282
7.3702
7.3672
7.3549
7.3506
7.3474
7.3374
7.2605
5.8671
5.8571
5.8460
5.8329
5.8229
5.8118
5.8005
5.1934
5.1749
5.1723
4.8698
4.6179
4.6103
4.6072
4.6037
4.5994
3.6047
3.5988
3.5866
3.5213
3.5098
3.5032
3.0491
3.0347
3.0203
2.0045
1.9961
1.9899
1.9821
1.9751
1.9679
1.9536
1.8395
1.8275
1.8130
1.8008
O
4
SCN
Ph
O
O
1.92
3.05
0.87
1.80
1.00
1.39
0.98
1.01
1.85
1.91
1.95
f1 (ppm)
170.4561
136.3524
131.5524
128.7944
128.6808
127.1514
127.0989
118.5488
112.3265
81.1845
77.3090
77.0547
76.8006
68.8110
65.6399
33.8505
27.7387
27.0175
f1 (ppm)

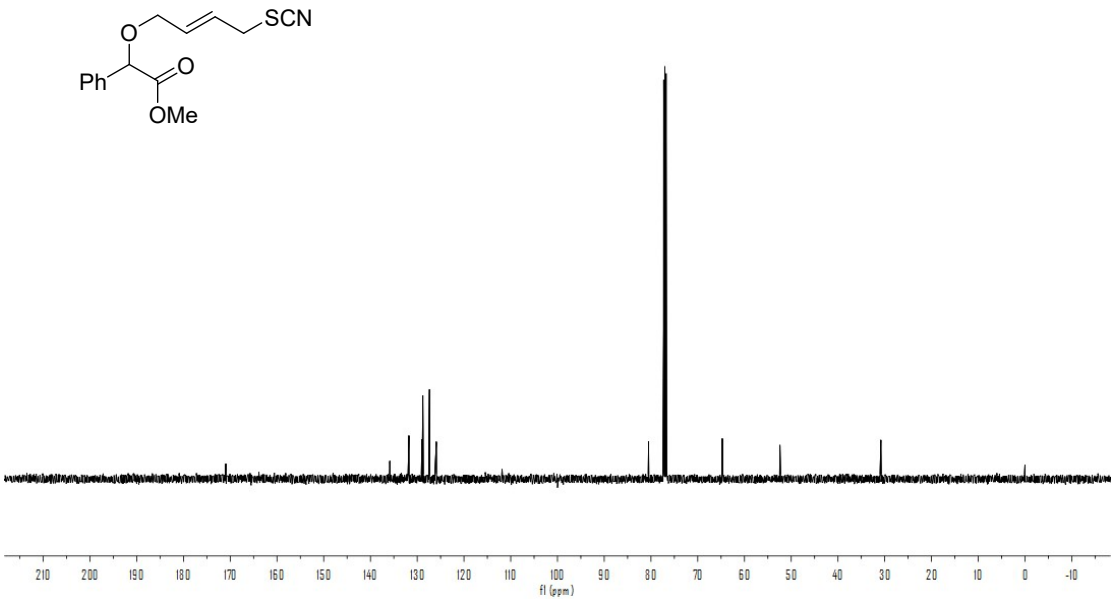
**5v**


7.4466
7.4432
7.4302
7.4278
7.3850
7.3807
7.3678
7.3646
7.3557
7.3526
7.3477
7.3444
7.3341
4.8567
3.7102
3.5643
3.5588
3.5462
3.5336
3.4655
3.4530
3.4474
2.9597
2.9451
2.9306
1.8681
1.8635
1.8537
1.8487
1.8371
1.8337
1.8221
1.8197
1.7248
1.7110
1.6979
1.6821
1.6696
1.5955
1.5820
1.5778
1.5670
1.5626
1.5538
O
5
SCN
Ph
O
OMe
2.10
3.18
1.00
3.30
1.02
1.04
1.95
1.99
2.24
2.01
f1 (ppm)
171.3447
136.5388
128.7214
128.6565
127.1594
112.3072
81.1437
77.3000
77.0460
76.7919
69.3241
52.2562
33.9250
29.6314
28.8569
24.6640
O
5
SCN
Ph
O
OMe
f1 (ppm)

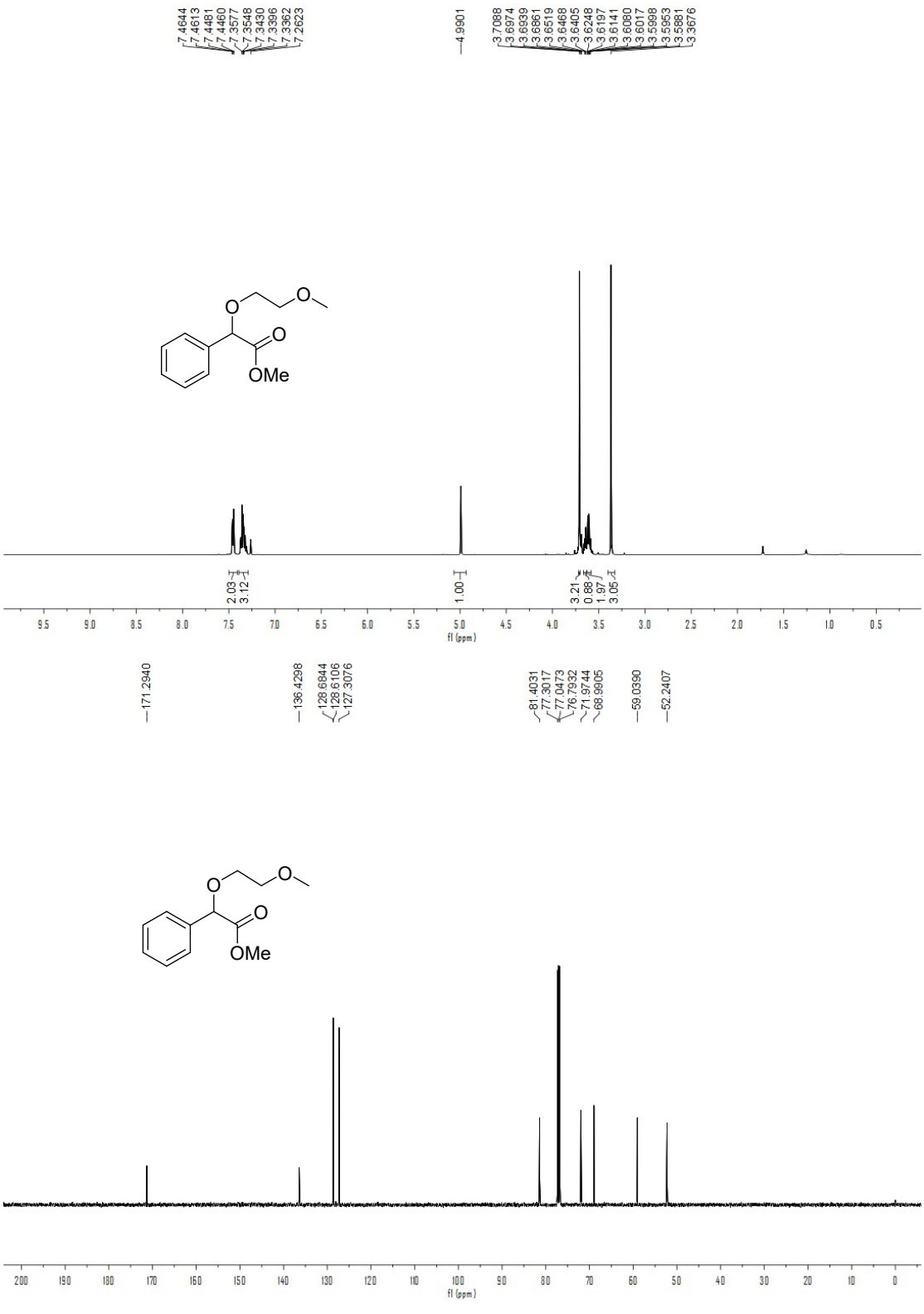
**5w**



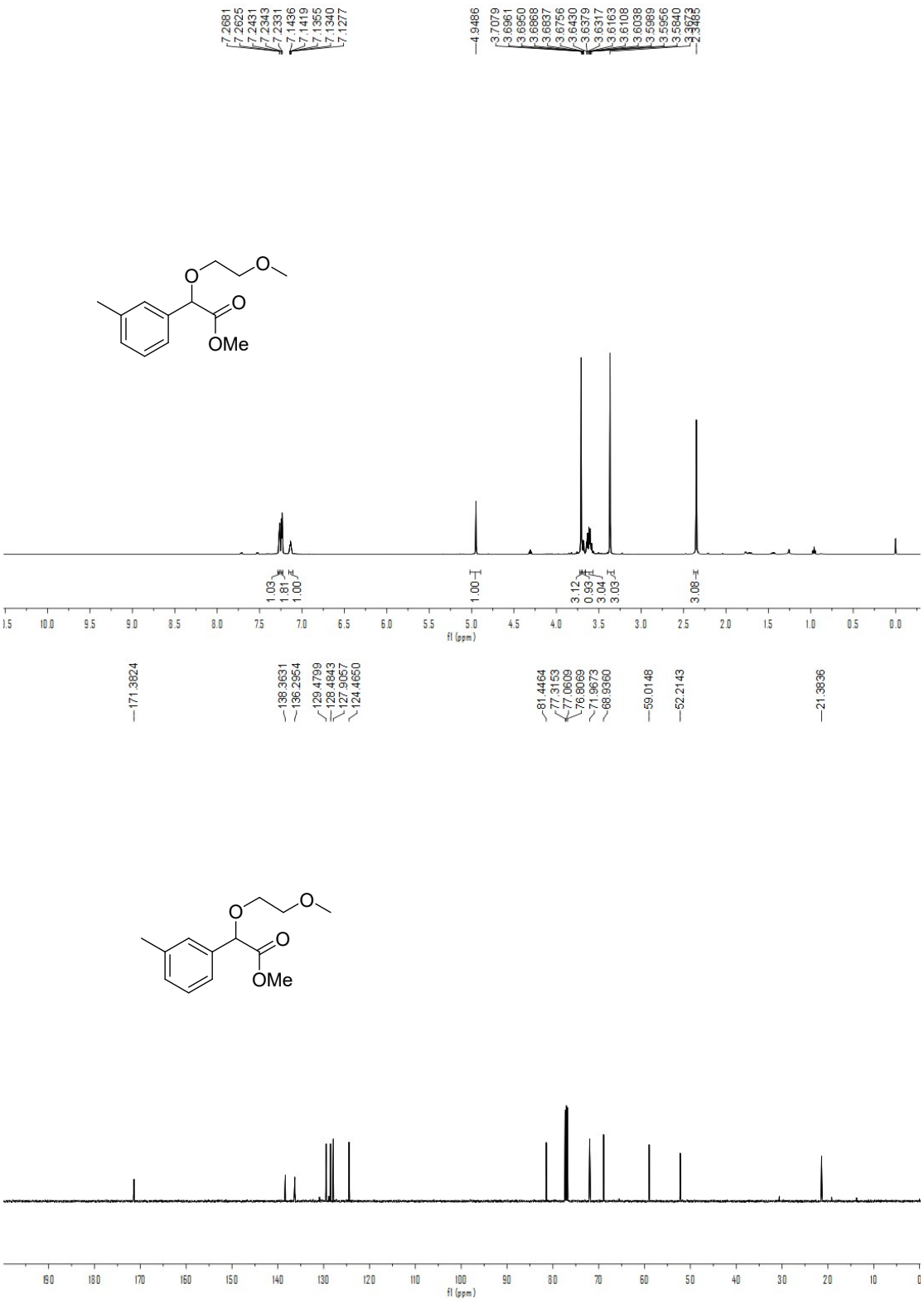

SCN
O
Ph
O
OMe
2.08
3.29
1.05
1.11
1.00
1.04
1.08
0.93
3.08
1.11
f1 (ppm)


SCN
O
Ph
O
OMe
f1 (ppm)

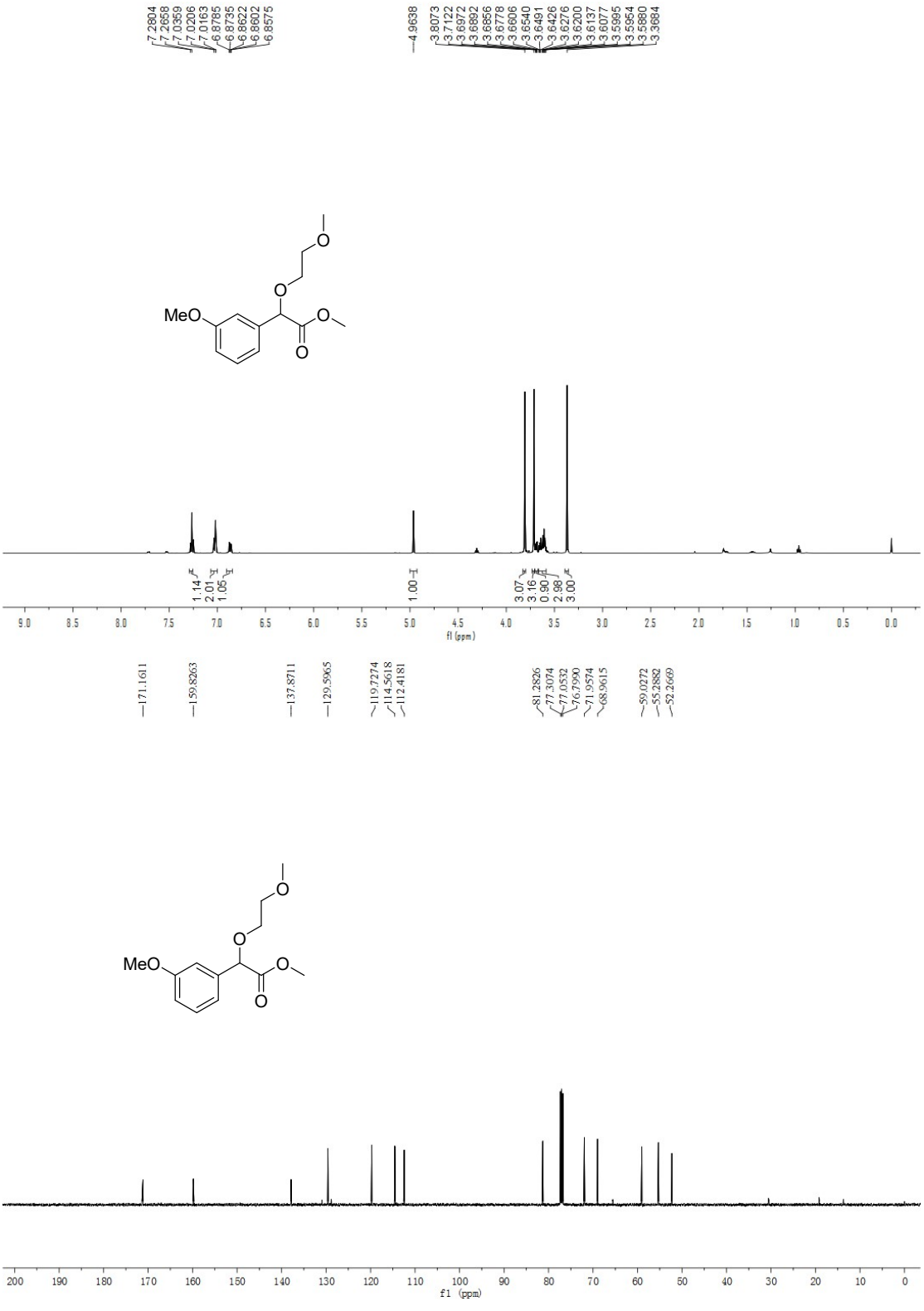
**5x**


7.4644
7.4613
7.4481
7.4460
7.3577
7.3548
7.3430
7.3396
7.3362
7.2623
4.9901
3.7088
3.6974
3.6939
3.6861
3.6519
3.6468
3.6405
3.6248
3.6197
3.6141
3.6080
3.6017
3.5998
3.5953
3.5881
3.3676
O
O
O
OMe
2.03
3.12
1.00
3.21
0.88
1.97
3.05
f1 (ppm)
171.2940
136.4298
128.6844
128.6106
127.3076
81.4031
77.3017
77.0473
76.7932
71.9744
68.9905
59.0390
52.2407
O
O
O
OMe
f1 (ppm)

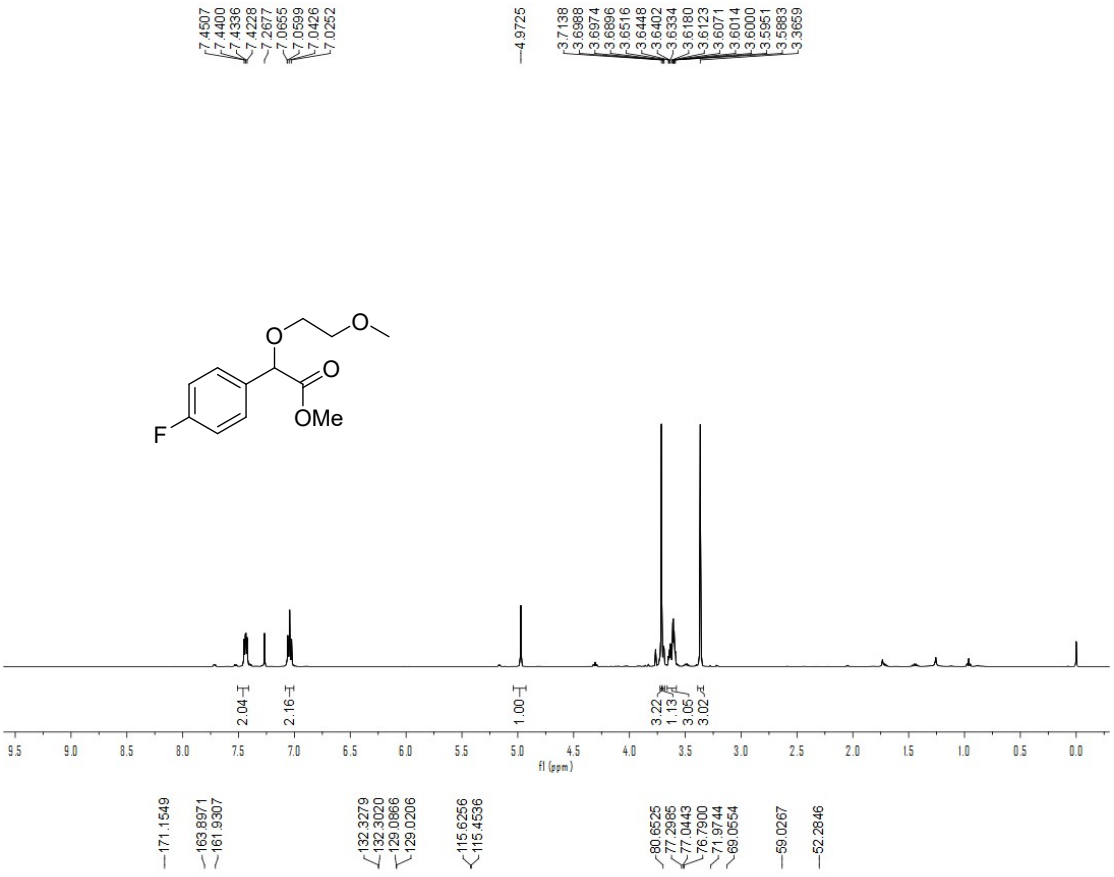
**6a**


7.2681
7.2625
7.2431
7.2343
7.2331
7.1436
7.1419
7.1355
7.1340
7.1277
4.9486
3.7079
3.6961
3.6950
3.6868
3.6837
3.6756
3.6430
3.6379
3.6317
3.6163
3.6108
3.6038
3.5989
3.5956
3.5840
2.3863
O
O
O
OMe
1.03
1.81
1.00
1.00
3.12
0.93
3.04
3.03
3.08
f1 (ppm)
171.3824
138.3631
136.2954
129.4799
128.4843
127.9057
124.4650
81.4464
77.3153
77.0609
76.8069
71.9673
68.9360
59.0148
52.2143
21.3836
f1 (ppm)

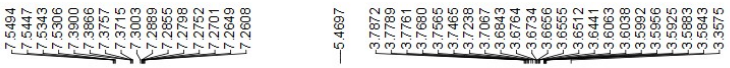
**6b**

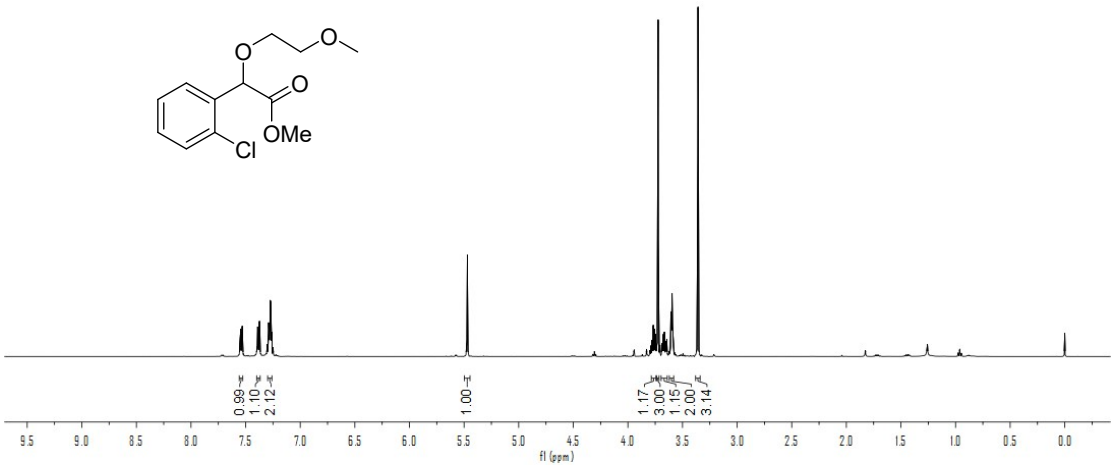

7.2804
7.2658
7.0359
7.0206
7.0163
6.8785
6.8735
6.8622
6.8602
6.8575
4.9638
3.8073
3.7122
3.6972
3.6892
3.6856
3.6778
3.6606
3.6540
3.6491
3.6426
3.6276
3.6200
3.6137
3.6077
3.5995
3.5954
3.5880
3.3684
MeO
O
O
O
O
1.14
2.01
1.05
1.00
3.07
3.16
0.90
2.98
3.00
f1 (ppm)
171.1611
159.8263
137.8711
129.5965
119.7274
114.5618
112.4181
81.2826
77.3074
77.0532
76.7990
71.9574
68.9615
59.0272
55.2882
52.2669
f1 (ppm)

**6c**


7.4507
7.4400
7.4336
7.4228
7.2677
7.0655
7.0599
7.0426
7.0252
4.9725
3.7138
3.6988
3.6974
3.6896
3.6516
3.6448
3.6402
3.6334
3.6180
3.6123
3.6071
3.6014
3.6000
3.5951
3.5883
3.3659
O
O
O
OMe
F
2.04
2.16
1.00
3.22
1.13
3.05
3.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
171.1549
163.8971
161.9307
132.3279
132.3020
129.0886
129.0206
115.6256
115.4536
80.6525
77.2985
77.0443
76.7900
71.9744
69.0554
59.0267
52.2846
O
O
O
OMe
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

**6d**


7.5494
7.5447
7.5343
7.5306
7.3900
7.3866
7.3757
7.3715
7.3003
7.2889
7.2855
7.2798
7.2752
7.2701
7.2649
7.2608
5.4697
3.7872
3.7789
3.7761
3.7680
3.7565
3.7465
3.7238
3.7067
3.6843
3.6764
3.6734
3.6656
3.6555
3.6512
3.6441
3.6063
3.6038
3.5992
3.5956
3.5925
3.5883
3.5843
3.3575


O
O
O
OMe
Cl
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1.10
2.12
1.00
1.17
3.00
1.15
2.00
3.14
f1 (ppm)


170.6406
134.5378
133.7730
129.8210
129.5864
128.8528
127.2293
77.5718
77.3249
77.0707
76.8163
71.8071
69.4449
58.9905
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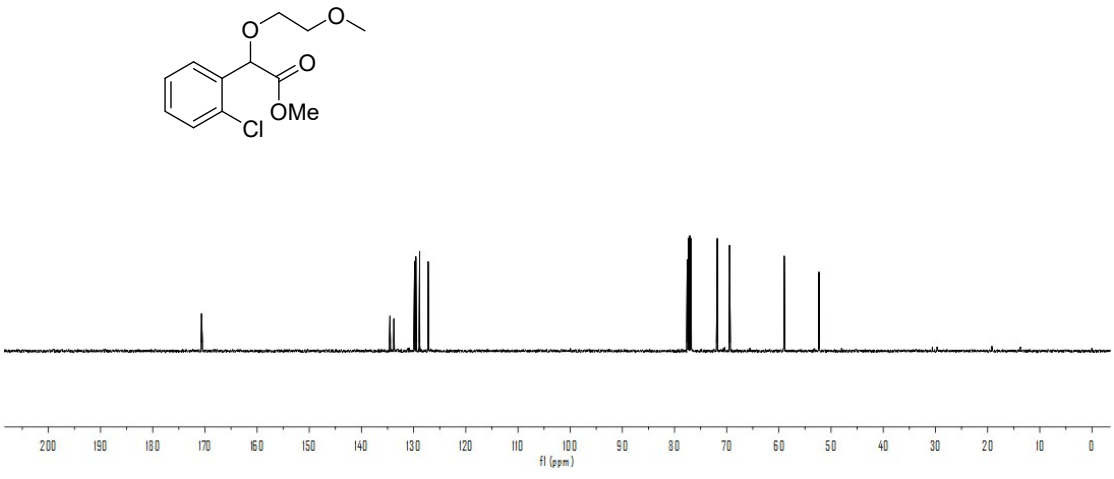
O
O
O
OMe
Cl
f1 (ppm)

**6e**