

Supporting Information

A Metal- and Solvent-free Ultrasonic Multicomponent Synthesis of (Z)- β -Iodo vinylthiocyanates

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1. General Information

Unless otherwise specified, all reagents and solvents were obtained from commercial suppliers and used without further purification. All reagents were weighed and handled in air at room temperature. Chromatographic purifications were carried out on a Biotage Isolera Four instrument. ^1H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz by using a Bruker Avance 400 spectrometer. Chemical shifts were reported in parts per million (δ) relative to tetramethylsilane (TMS). The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad singlet. HRMS spectra were performed on a spectrometer operating on EI.

Ultrasound set-up

Ultrasound for sonochemical synthesis is generated with the help of ultrasonic instrument set-up (horn type). The specification and details of the set-up, processing parameters used during the experiments are:

Make: Yinyu Gaoke, China.

Operating frequency: 28-68 kHz.

Rated output power: 25-50 W.

Diameter of stainless steel tip of horn: 1.3×10^2 m.

Surface area of ultrasound irradiating face: 1.32×10^4 m².

2. Experiment Section

General Procedure for the Synthesis of *Z*-vinyl thiocyanates **2**: In a vial was placed alkyne (0.2 mmol), KSCN (39 mg, 0.4 mmol), I₂ (25 mg, 0.1 mmol), (NH₄)₂S₂O₈ (45 mg, 0.2 mmol), HOAc (0.4 mmol), then the contents were reacted under ultrasound irradiation. The progress of the reaction was monitored by TLC. The reaction typically took 5 min. Upon completion, the reaction mixture was purified by column chromatography on silica gel (eluent: hexanes/ethyl acetate) to afford **2**.

3. Characterization Data

(Z)-1-(2-iodo-1-thiocyanatovinyl)-4-methylbenzene (2a): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.33 (d, $J = 8.0$ Hz, 2H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.18 (s, 1H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.5, 132.8, 130.9, 129.6, 128.9, 109.4, 80.4, 21.5. HRMS Calcd (EI) m/z for $\text{C}_{10}\text{H}_8\text{INS}$: $[\text{M}]^+$ 300.9422, found: 300.9420.

(Z)-(2-iodo-1-thiocyanatovinyl)benzene (2b)^[1]: Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.48–7.44 (m, 5H), 7.26 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 135.9, 130.8, 130.2, 129.0, 128.9, 109.2, 81.3.

(Z)-1-(2-iodo-1-thiocyanatovinyl)-4-methoxybenzene (2c): Light yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.41 (d, $J = 8.8$ Hz, 2H), 7.16 (s, 1H), 6.97 (d, $J = 8.8$ Hz, 2H), 3.86 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 160.8, 130.8, 130.7, 127.8, 114.2, 109.5, 79.8, 55.3. HRMS Calcd (EI) m/z for $\text{C}_{10}\text{H}_8\text{INOS}$: $[\text{M}]^+$ 316.9371, found: 316.9366.

(Z)-1-ethyl-4-(2-iodo-1-thiocyanatovinyl)benzene (2d): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.41 (d, $J = 8.4$ Hz, 2H), 7.33 (d, $J = 8.4$ Hz, 2H), 7.25 (s, 1H), 2.75 (q, $J = 7.6$ Hz, 2H), 1.32 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 146.6, 133.0, 130.9, 129.0, 128.4, 109.4, 80.3, 28.7, 15.0. HRMS Calcd (EI) m/z for $\text{C}_{11}\text{H}_{10}\text{INS}$: $[\text{M}]^+$ 314.9579, found: 314.9573.

(Z)-4-(2-iodo-1-thiocyanatovinyl)-1,1'-biphenyl (2e): Light yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.69 (d, $J = 8.4$ Hz, 2H), 7.62–7.63 (m, 2H), 7.54 (d, $J = 8.4$ Hz, 2H), 7.50–7.46 (m, 2H), 7.42–7.38 (m, 1H), 7.30 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.0, 139.9, 134.6, 130.7, 129.6, 128.9, 128.0, 127.6, 127.2, 109.3, 81.3. HRMS Calcd (EI) m/z for $\text{C}_{15}\text{H}_{10}\text{INS}$: $[\text{M}]^+$ 362.9579, found: 362.9576.

(Z)-1-fluoro-4-(2-iodo-1-thiocyanatovinyl)benzene (2f): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.47–7.43 (m, 2H), 7.28 (s, 1H), 7.18–7.14 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 164.6, 162.1, 131.3, 131.2, 130.1, 130.0, 116.3, 116.1, 109.0, 82.3. ^{19}F

NMR (376 MHz, CDCl₃) δ -108.9. HRMS Calcd (EI) m/z for C₉H₅FINS: [M]⁺ 304.9171, found: 304.9173.

(Z)-1-chloro-4-(2-iodo-1-thiocyanatovinyl)benzene (2g): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.45 (d, *J* = 8.8 Hz, 2H), 7.39 (t, *J* = 8.8 Hz, 2H), 7.31 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 136.3, 134.3, 130.4, 129.9, 129.3, 108.9, 82.9. HRMS Calcd (EI) m/z for C₉H₅ClINS: [M]⁺ 320.8876, found: 320.8871.

(Z)-1-bromo-4-(2-iodo-1-thiocyanatovinyl)benzene (2h): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.32 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 134.8, 132.2, 130.6, 129.5, 124.6, 108.9, 83.0. HRMS Calcd (EI) m/z for C₉H₅BrINS: [M]⁺ 364.8371, found: 364.8368.

(Z)-1-(2-iodo-1-thiocyanatovinyl)-3-methylbenzene (2i): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.37–7.33 (m, 1H), 7.28–7.24 (m, 3H), 7.22 (s, 1H), 2.42 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 138.7, 135.7, 131.0, 129.4, 128.8, 126.1, 109.3, 80.7, 21.3. HRMS Calcd (EI) m/z for C₁₀H₈INS: [M]⁺ 300.9422, found: 300.9420.

(Z)-1-(2-iodo-1-thiocyanatovinyl)-2-methylbenzene (2j): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.40–7.36 (m, 1H), 7.32–7.28 (m, 2H), 7.25 (s, 1H), 7.18–7.15 (m, 1H), 2.34 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 136.1, 135.6, 130.7, 130.7, 130.2, 128.9, 126.5, 108.8, 82.5, 19.0. HRMS Calcd (EI) m/z for C₁₀H₈INS: [M]⁺ 300.9422, found: 300.9420.

(Z)-1-ethynyl-4-(2-iodo-1-thiocyanatovinyl)benzene (2k): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.58 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.32 (s, 1H), 3.20 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 136.1, 132.6, 129.1, 128.1, 124.1, 108.9, 82.7, 82.0, 79.3. HRMS Calcd (EI) m/z for C₁₁H₆INOS: [M]⁺ 310.9266, found: 310.9260.

(Z)-3-(2-iodo-1-thiocyanatovinyl)thiophene (2l): Light yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.72–7.71 (m, 1H), 7.43–7.41 (m, 1H), 7.38–7.36 (m, 1H), 7.25 (s, 1H). ¹³C

NMR (100 MHz, CDCl₃) δ 135.6, 128.2, 127.4, 126.6, 125.9, 109.3, 80.9. HRMS Calcd (EI) m/z for C₇H₄INS₂: [M]⁺ 292.8830, found: 292.8825.

(Z)-3-(2-iodo-1-thiocyanatovinyl)pyridine (2m): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 8.62–8.60 (m, 1H), 8.57–8.56 (m, 1H), 7.68–7.65 (m, 1H), 7.38–7.35 (m, 1H), 7.04 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 150.8, 148.2, 135.9, 135.2, 123.6, 120.8, 108.4, 94.8. HRMS Calcd (EI) m/z for C₈H₅IN₂S: [M]⁺ 287.9218, found: 287.9211.

(Z)-(2-iodo-1-thiocyanatovinyl)ferrocene (2n): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 6.55 (s, 1H), 4.57 (s, 2H), 4.52 (s, 2H), 4.28 (s, 5H). ¹³C NMR (100 MHz, CDCl₃) δ 132.2, 110.2, 109.2, 76.5, 71.1, 70.3, 69.9. HRMS Calcd (EI) m/z for C₁₃H₁₀FeINS: [M]⁺ 394.8928, found: 394.8926.

(Z)-1-iodo-2-thiocyanatooct-1-ene (2o): Light yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 6.89 (s, 1H), 2.58 (t, *J* = 7.6 Hz, 2H), 1.64–1.57 (m, 2H), 1.39–1.30 (m, 6H), 0.90 (t, *J* = 6.8 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 132.2, 109.3, 82.3, 37.3, 31.3, 28.3, 26.9, 22.4, 14.0. HRMS Calcd (EI) m/z for C₉H₁₄INS: [M]⁺ 294.9892, found: 294.9895.

(Z)-(2-iodo-1-thiocyanatovinyl)cyclopropane (2p): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.11 (s, 1H), 2.00–1.93 (m, 1H), 1.07–1.02 (m, 2H), 0.93–0.88 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 132.2, 109.9, 85.6, 18.9, 7.8. HRMS Calcd (EI) m/z for C₆H₆INS: [M]⁺ 250.9266, found: 250.9262.

(Z)-(2-iodo-1-thiocyanatovinyl)cyclohexane (2q): Colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 6.94 (s, 1H), 2.98–2.91 (m, 1H), 1.85–1.81 (m, 2H), 1.75–1.72 (m, 2H), 1.46–1.31 (m, 5H), 1.25–1.18 (m, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 135.9, 110.2, 81.9, 46.6, 30.0, 25.7, 25.3. HRMS Calcd (EI) m/z for C₉H₁₂INS: [M]⁺ 292.9735, found: 292.9733.

(Z)-5-chloro-1-iodo-2-thiocyanatopent-1-ene (2r): Light yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.02 (s, 1H), 3.60 (t, *J* = 6.0 Hz, 2H), 2.79–2.75 (m, 2H), 2.13–2.06 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 130.6, 108.9, 84.5, 43.4, 34.9, 29.7. HRMS Calcd (EI) m/z for $\text{C}_6\text{H}_7\text{ClINS}$: $[\text{M}]^+$ 286.9032, found: 286.9033.

(Z)-4-bromo-1-iodo-2-thiocyanatobut-1-ene (2s): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.25 (s, 1H), 3.61 (t, $J = 7.2$ Hz, 2H), 3.18 (t, $J = 7.2$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 129.0, 108.7, 88.8, 39.6, 27.3. HRMS Calcd (EI) m/z for $\text{C}_5\text{H}_5\text{BrINS}$: $[\text{M}]^+$ 316.8371, found: 316.8366.

(Z)-3-iodo-2-thiocyanatoprop-2-en-1-ol (2t): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.04 (s, 1H), 4.29 (s, 2H), 1.97 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 104.0, 79.8, 70.8. HRMS Calcd (EI) m/z for $\text{C}_4\text{H}_4\text{INOS}$: $[\text{M}]^+$ 240.9058, found: 240.9054.

(Z)-7-iodo-6-thiocyanatohept-6-enoic acid (2u): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 6.95 (s, 1H), 2.63 (t, $J = 7.2$ Hz, 2H), 2.44 (t, $J = 7.2$ Hz, 2H), 1.75–1.68 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 179.1, 131.5, 109.2, 83.6, 37.0, 33.5, 26.3, 23.5. HRMS Calcd (EI) m/z for $\text{C}_8\text{H}_{10}\text{INO}_2\text{S}$: $[\text{M}]^+$ 310.9477, found: 310.9480.

(Z)-(2-iodo-1-thiocyanatovinyl)trimethylsilane (2v): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.49 (s, 1H), 0.35 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 128.5, 113.1, 109.0, 0.4. HRMS Calcd (EI) m/z for $\text{C}_6\text{H}_{10}\text{INSSi}$: $[\text{M}]^+$ 282.9348, found: 282.9350.

(Z)-ethyl 3-iodo-2-thiocyanatoacrylate (2w): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.61 (s, 1H), 4.30 (q, $J = 6.4$ Hz, 2H), 1.38 (t, $J = 6.4$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 163.8, 140.9, 111.7, 79.1, 64.0, 13.9. HRMS Calcd (EI) m/z for $\text{C}_6\text{H}_6\text{INO}_2\text{S}$: $[\text{M}]^+$ 282.9164, found: 282.9161.

(Z)-5-iodo-6-thiocyanatodec-5-ene (2x): White solid, m.p. 107–109 $^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 2.79–2.72 (m, 4H), 1.64–1.45 (m, 4H), 1.41–1.36 (m, 2H), 1.33–1.28 (m, 2H), 0.97 (t, $J = 7.2$ Hz, 3H), 0.95 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 124.9, 112.5, 109.7, 43.7, 42.5, 31.1, 29.4, 22.0, 21.5, 13.8. HRMS Calcd (EI) m/z for $\text{C}_{11}\text{H}_{18}\text{INS}$: $[\text{M}]^+$ 323.0205, found: 323.0201.

(Z)-(2-iodo-1-thiocyanatoprop-1-en-1-yl)benzene (2y): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.46–7.41 (m, 3H), 7.34–7.31 (m, 2H), 2.90 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.3, 129.3, 129.0, 128.7, 125.3, 109.0, 104.9, 32.9. HRMS Calcd (EI) m/z for $\text{C}_{10}\text{H}_8\text{INS}$: $[\text{M}]^+$ 300.9422, found: 300.9420.

(Z)-(1-iodo-2-thiocyanatoethene-1,2-diyl)dibenzene (2z): Colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.54–7.40 (m, 10H). ^{13}C NMR (100 MHz, CDCl_3) δ 142.0, 140.3, 129.8, 129.6, 129.2, 129.0, 128.9, 128.8, 128.1, 109.2, 100.9. HRMS Calcd (EI) m/z for $\text{C}_{15}\text{H}_{10}\text{INS}$: $[\text{M}]^+$ 362.9579, found: 362.9577.

***p*-tolyl (Z)-3-iodo-3-phenyl-2-thiocyanatoacrylate (2aa):** White solid, m.p. 94–95 $^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.65–7.57 (m, 3H), 7.36–7.35 (m, 2H), 7.32–7.29 (m, 2H), 7.15–7.13 (m, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 163.5, 150.9, 148.3, 141.4, 136.6, 130.4, 130.2, 129.3, 127.6, 120.6, 109.8, 82.0, 20.9. HRMS Calcd (EI) m/z for $\text{C}_{17}\text{H}_{12}\text{INO}_2\text{S}$: $[\text{M}]^+$ 420.9633, found: 420.9630.

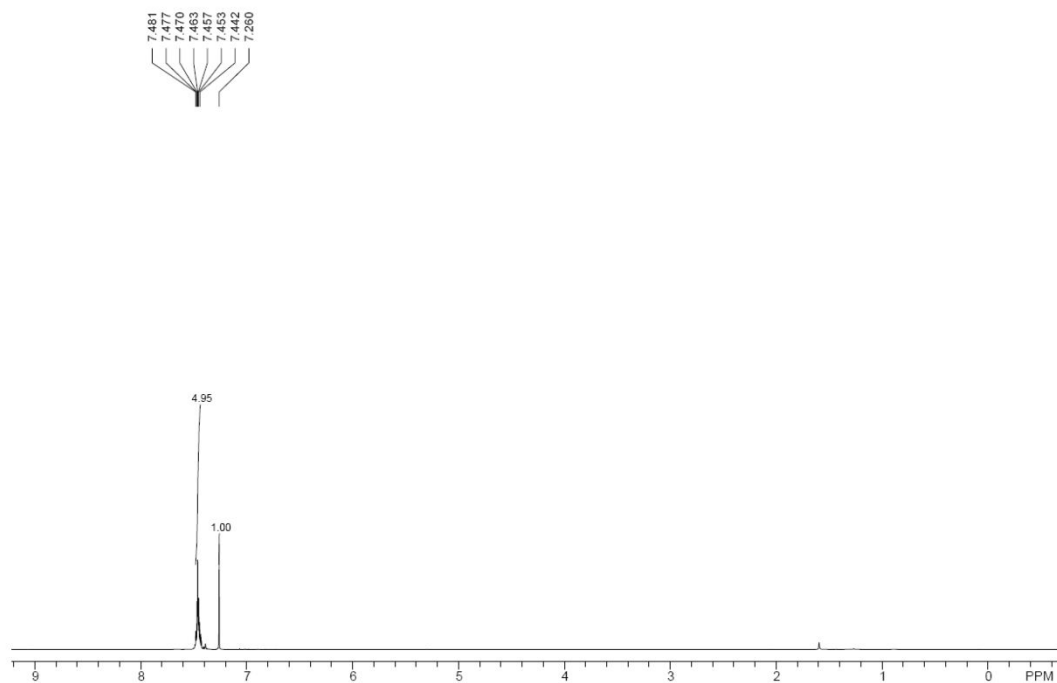
(E)-1-(1,2-diiodovinyl)-4-methylbenzene (3a)^[2]: Light yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.19 (d, $J = 8.0$ Hz, 2H), 7.14 (s, 1H), 7.09 (d, $J = 8.0$ Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.1, 139.0, 129.1, 128.4, 96.6, 80.2, 21.4.

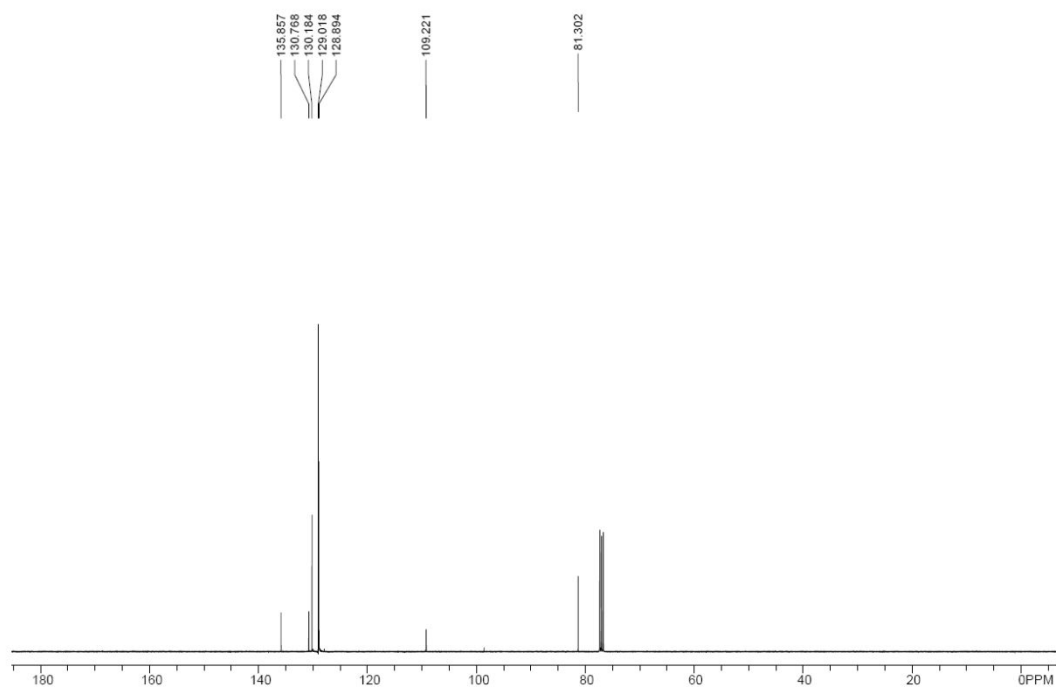
4. References

1. Jiang, G.; Zhu, C.; Li, J.; Wu, W.; Jiang, H. Silver-Catalyzed Regio- and Stereoselective Thiocyanation of Haloalkynes: Access to (Z)-Vinyl Thiocyanates. *Adv. Synth. Catal.* **2017**, 359, 1208–1212, DOI: 10.1002/adsc.201601142.
2. Liu, Y.; Huang, D.; Huang, J.; Maruoka, K. Hypervalent Iodine Mediated Chemoselective Iodination of Alkynes. *J. Org. Chem.* **2017**, 82, 11865–11871, DOI: 10.1021/acs.joc.7b01555

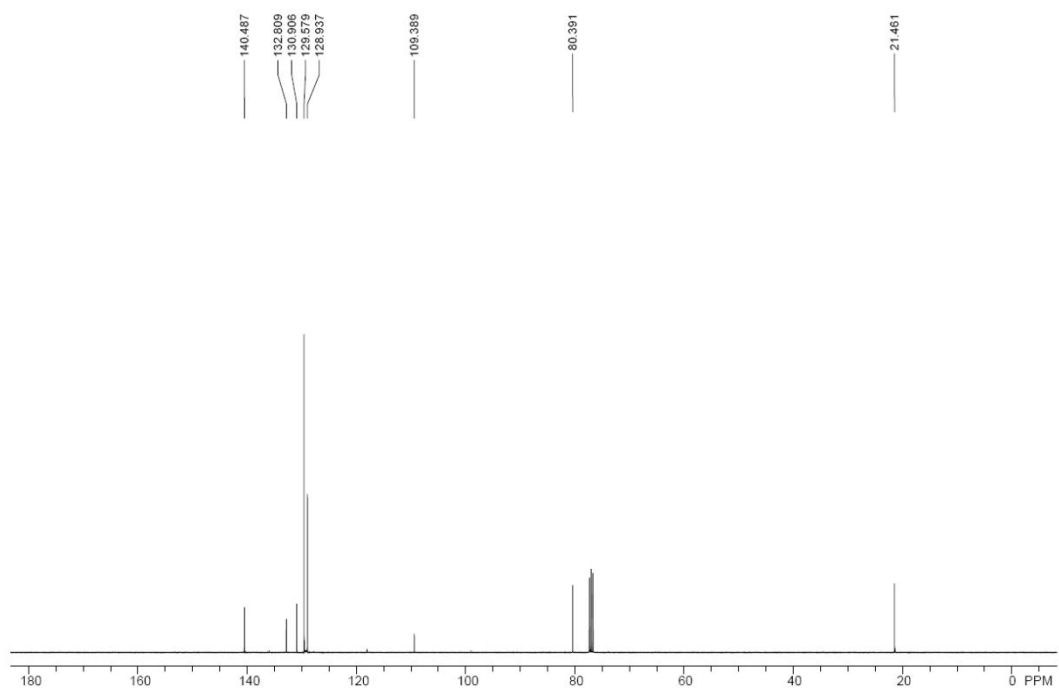
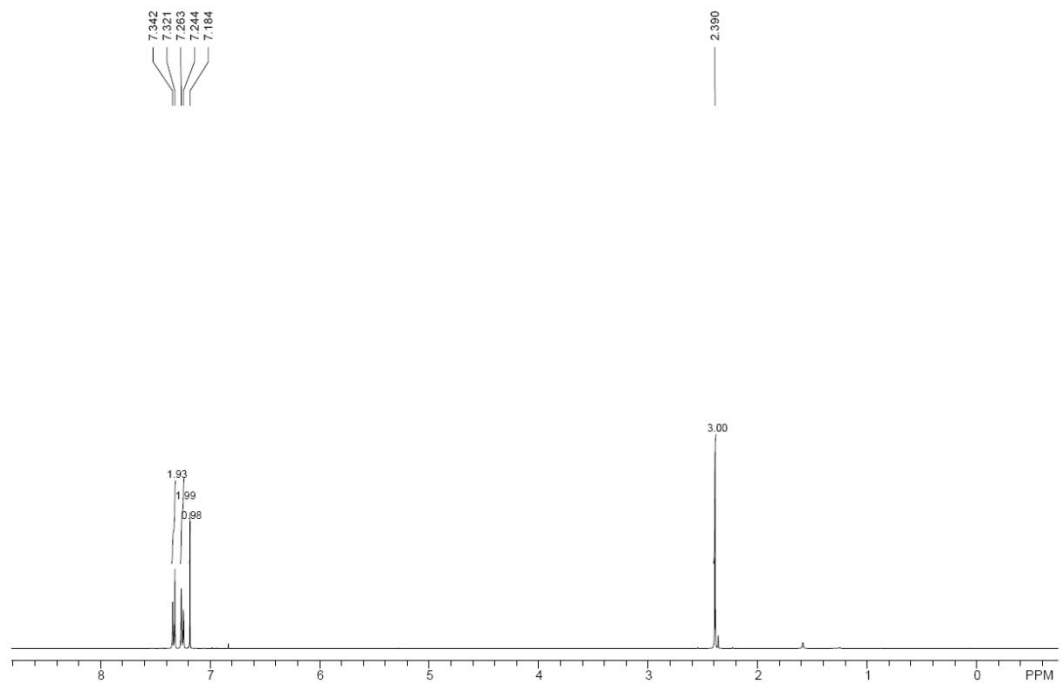
5. ^1H and ^{13}C NMR spectra

(Z)-(2-iodo-1-thiocyanatovinyl)benzene (**2b**)

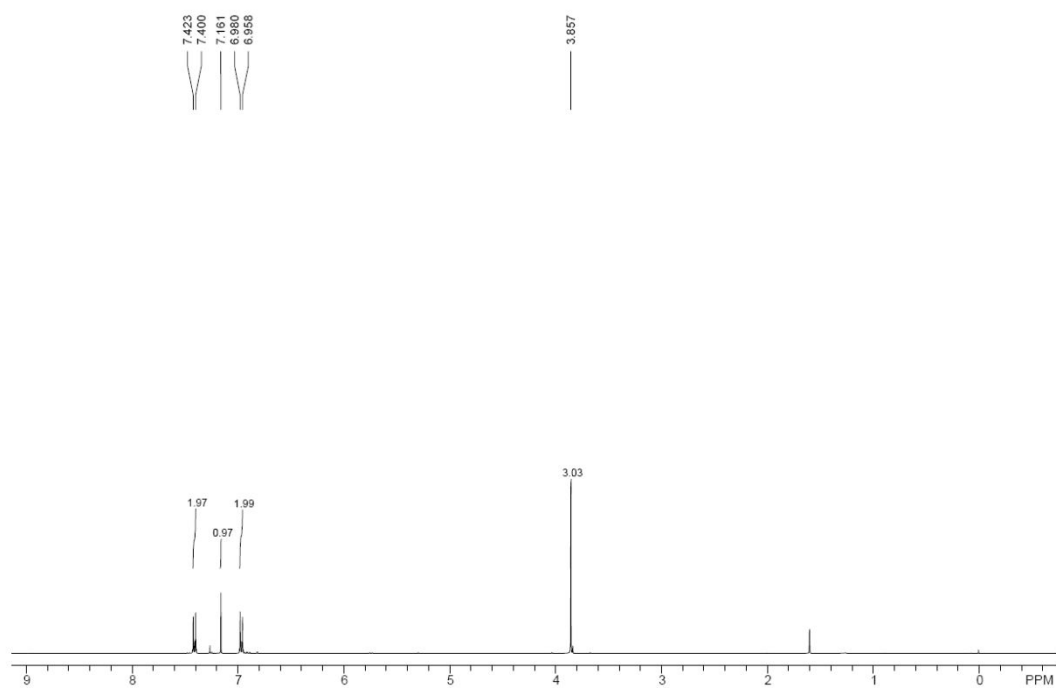


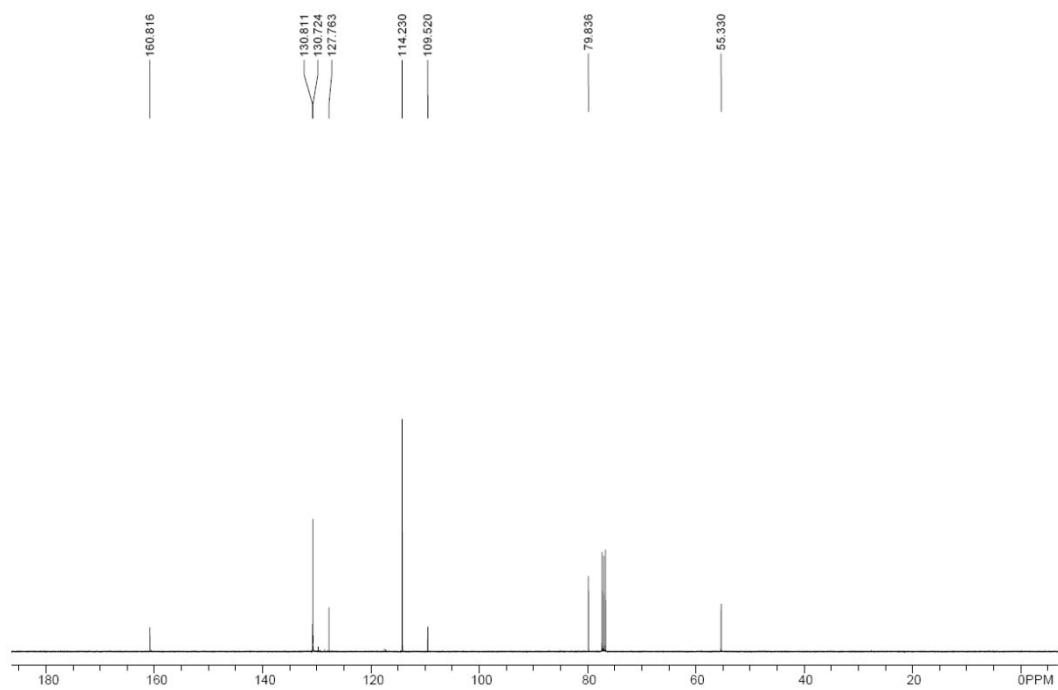


***(Z)*-1-(2-iodo-1-thiocyanatovinyl)-4-methylbenzene (2a)**

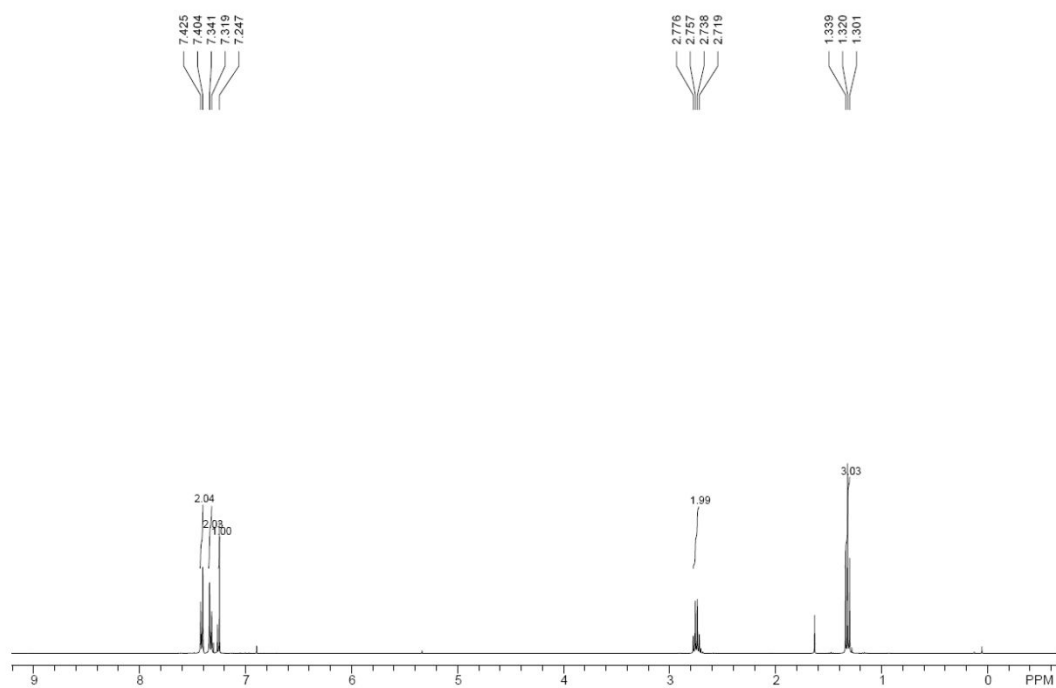


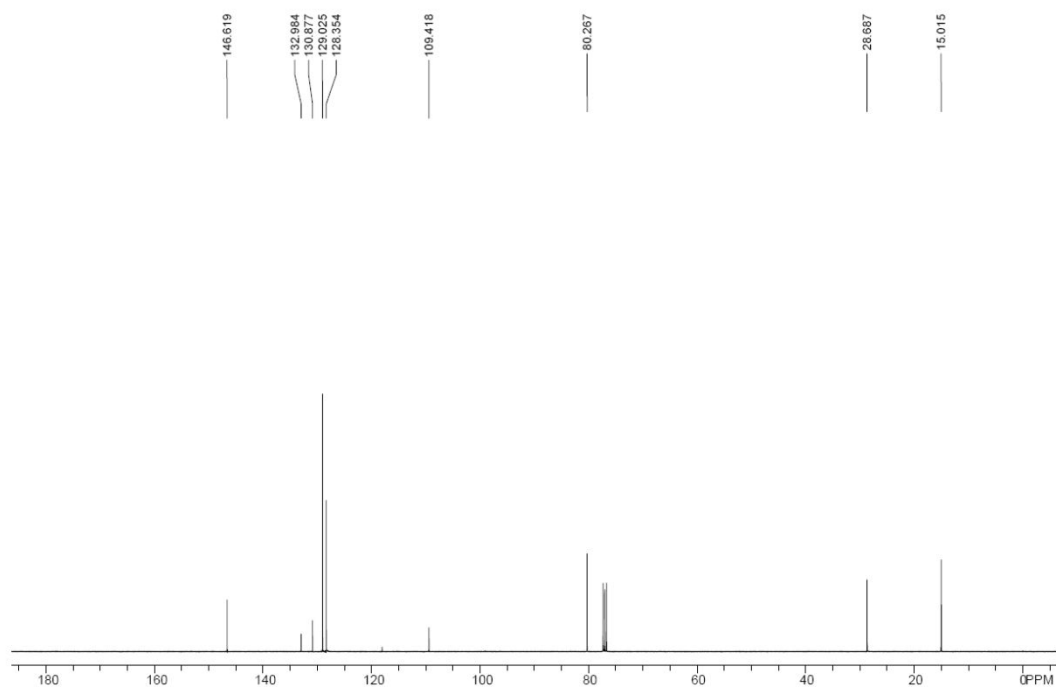
***(Z)*-1-(2-iodo-1-thiocyanatovinyl)-4-methoxybenzene (2c)**



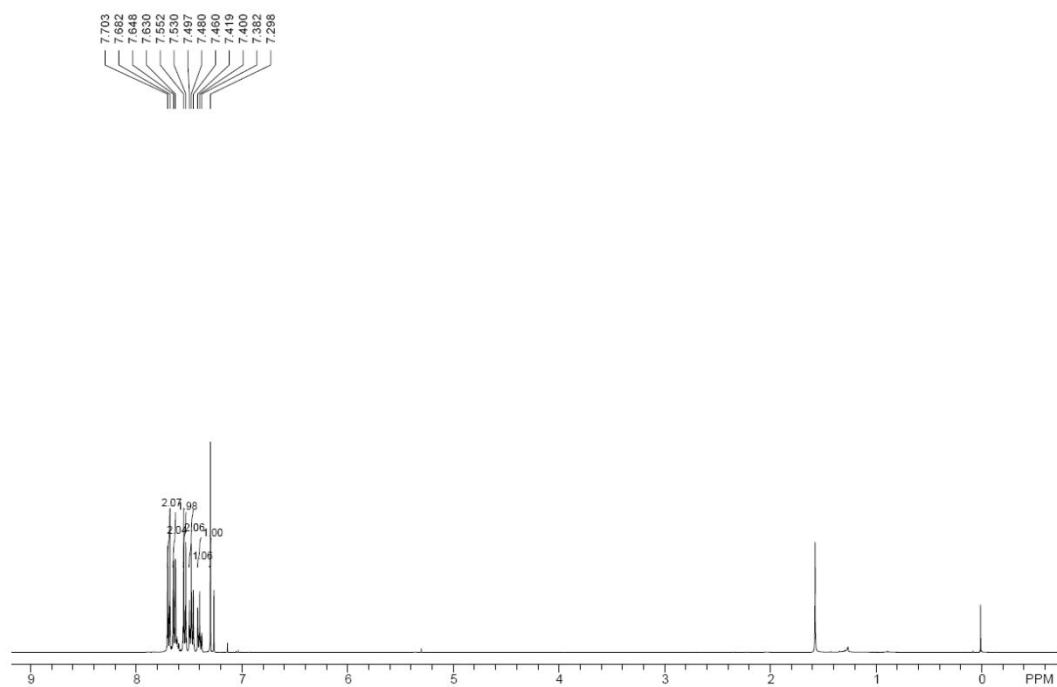


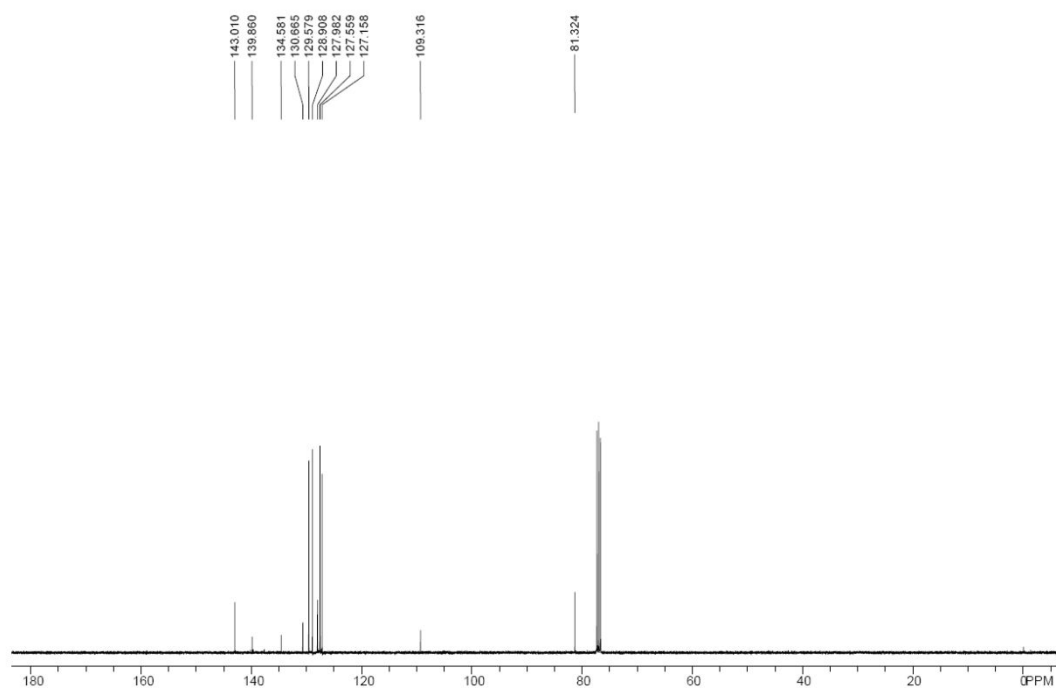
***(Z)*-1-ethyl-4-(2-iodo-1-thiocyanatovinyl)benzene (2d)**



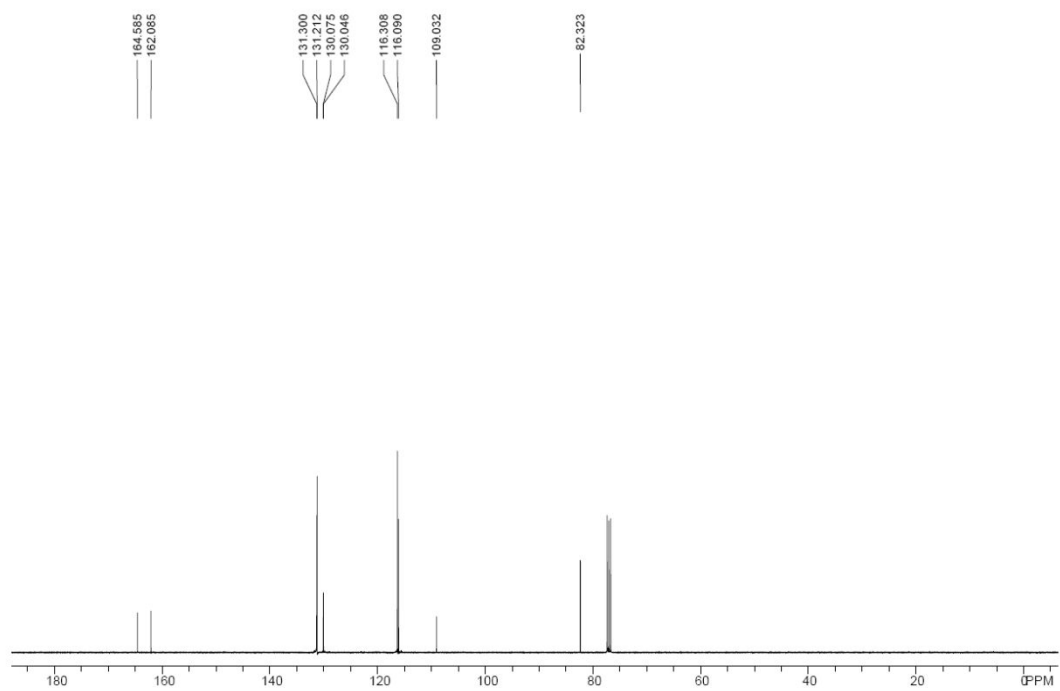
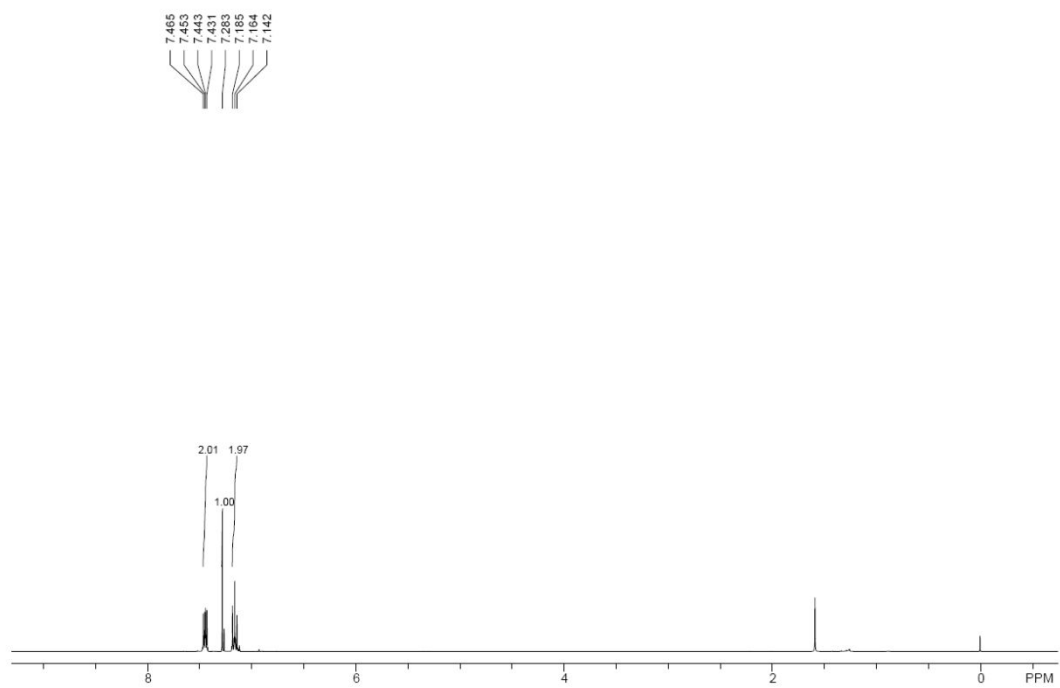


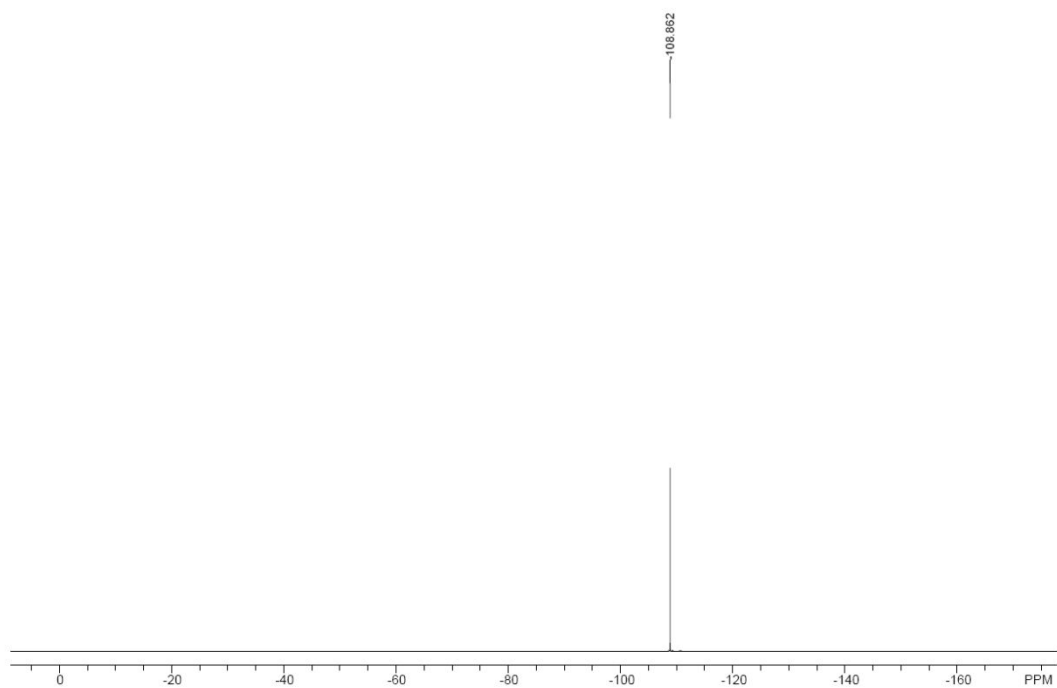
(Z)-4-(2-iodo-1-thiocyanatovinyl)-1,1'-biphenyl (2e)



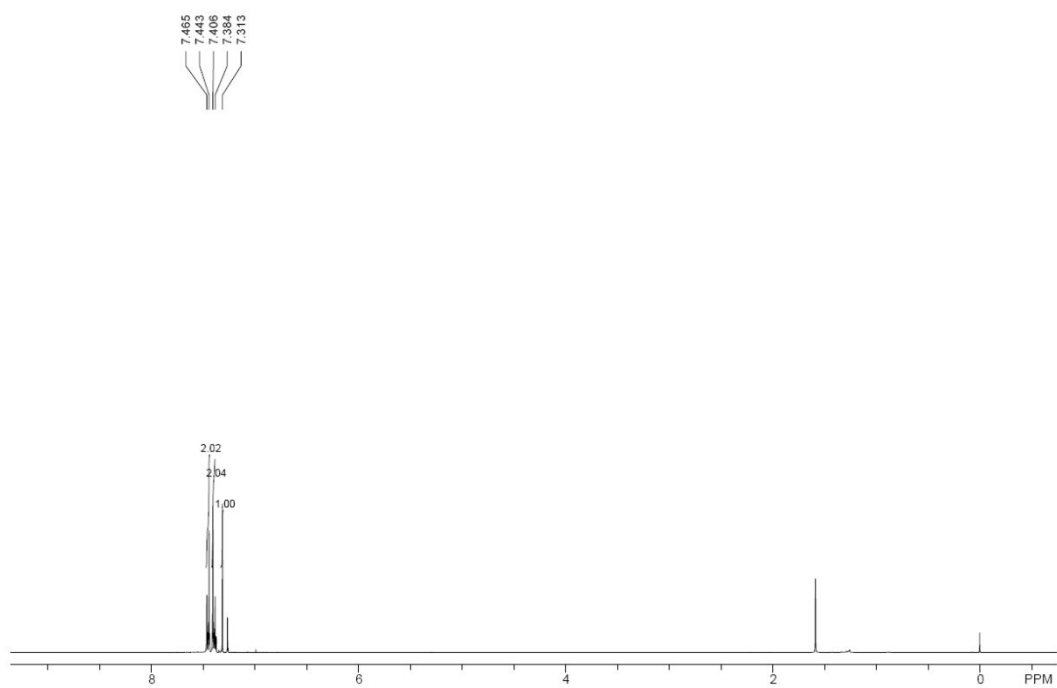


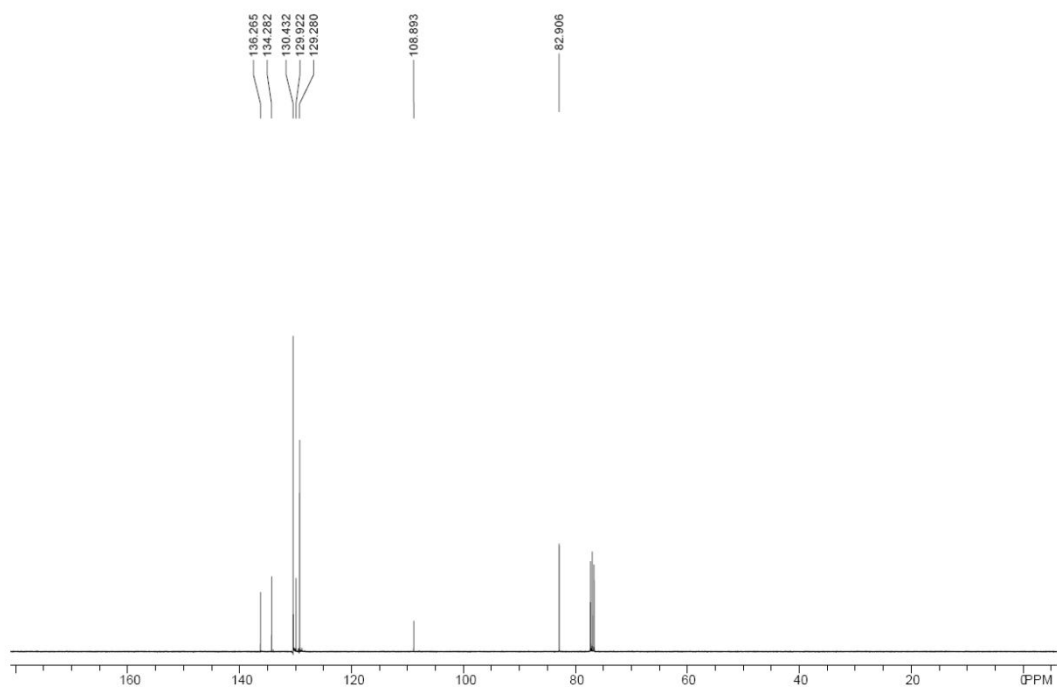
(Z)-1-fluoro-4-(2-iodo-1-thiocyanatovinyl)benzene (2f)



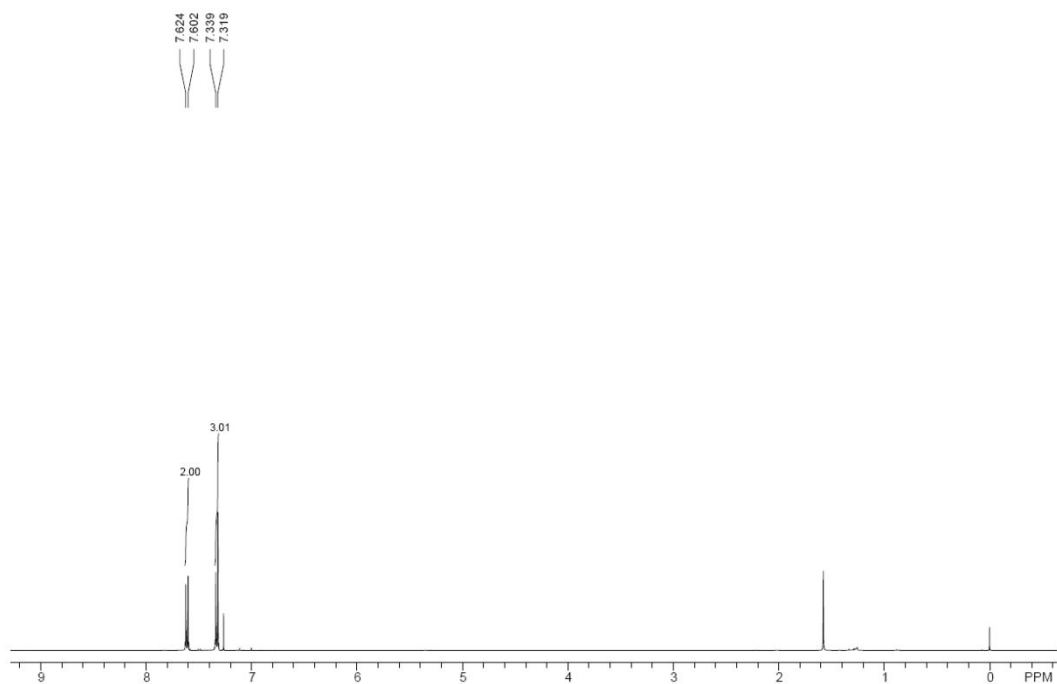


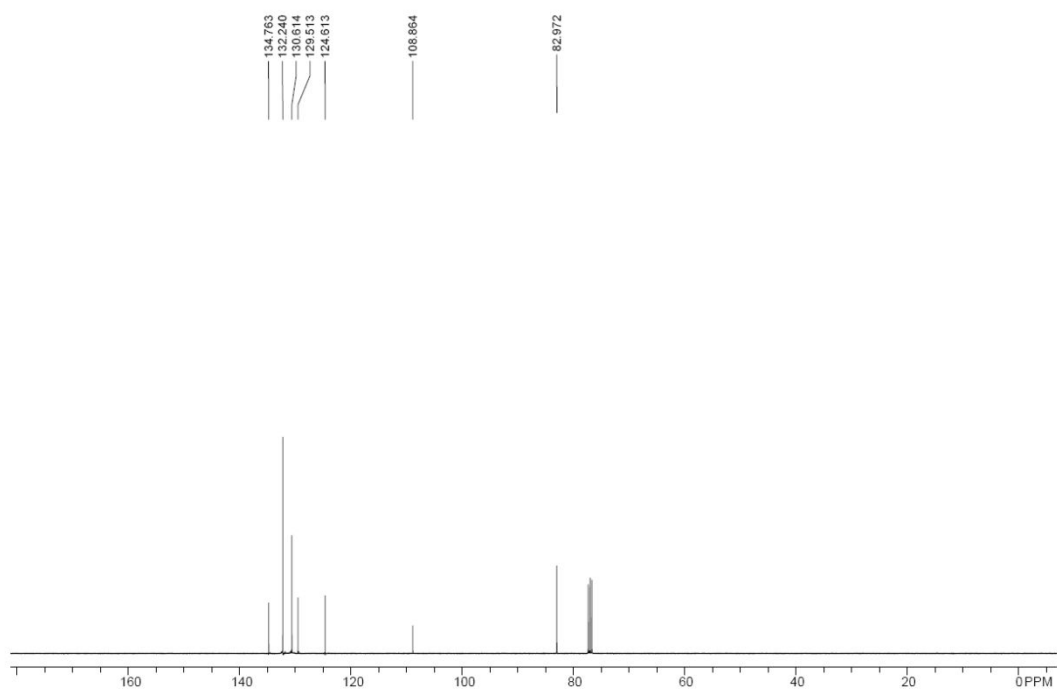
***(Z)*-1-chloro-4-(2-iodo-1-thiocyanatovinyl)benzene (2g)**



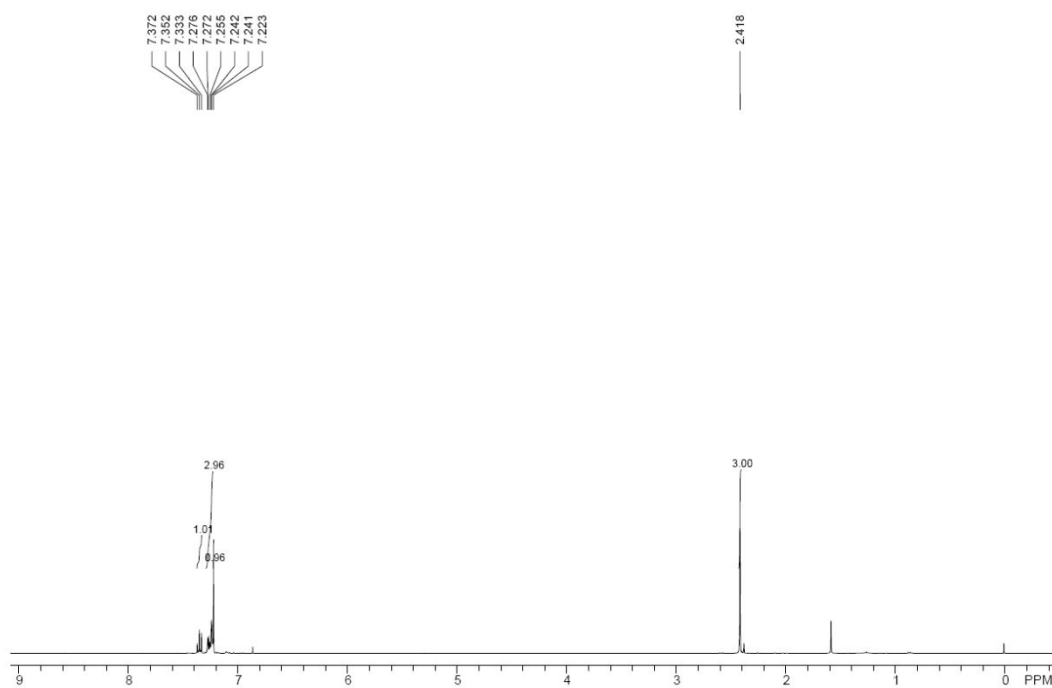


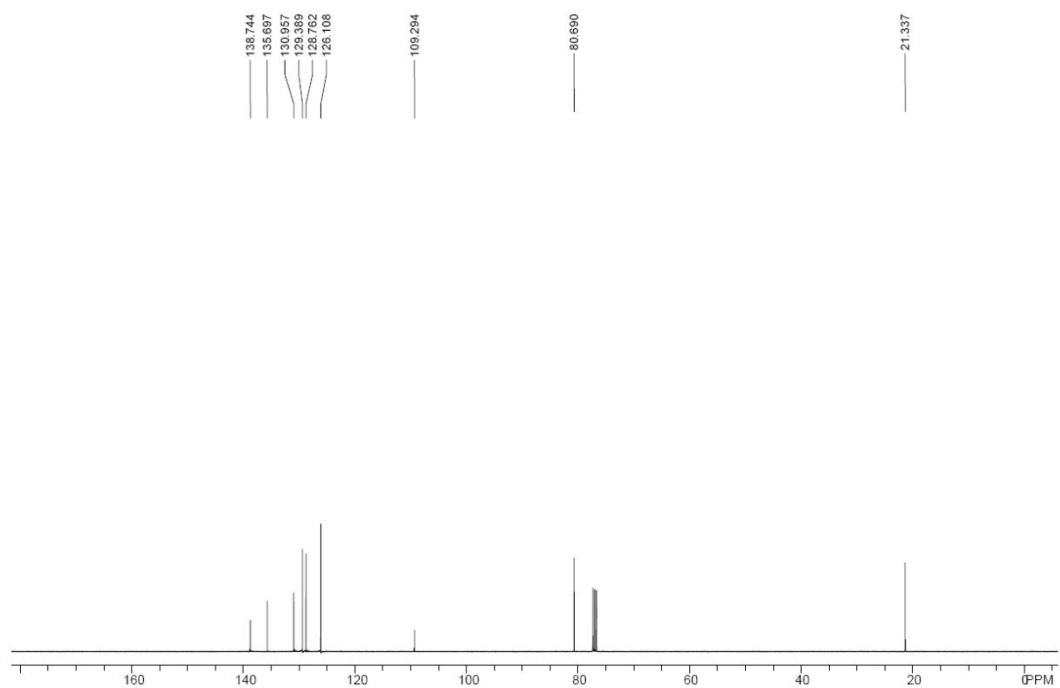
***(Z)*-1-bromo-4-(2-iodo-1-thiocyanatovinyl)benzene (2h)**



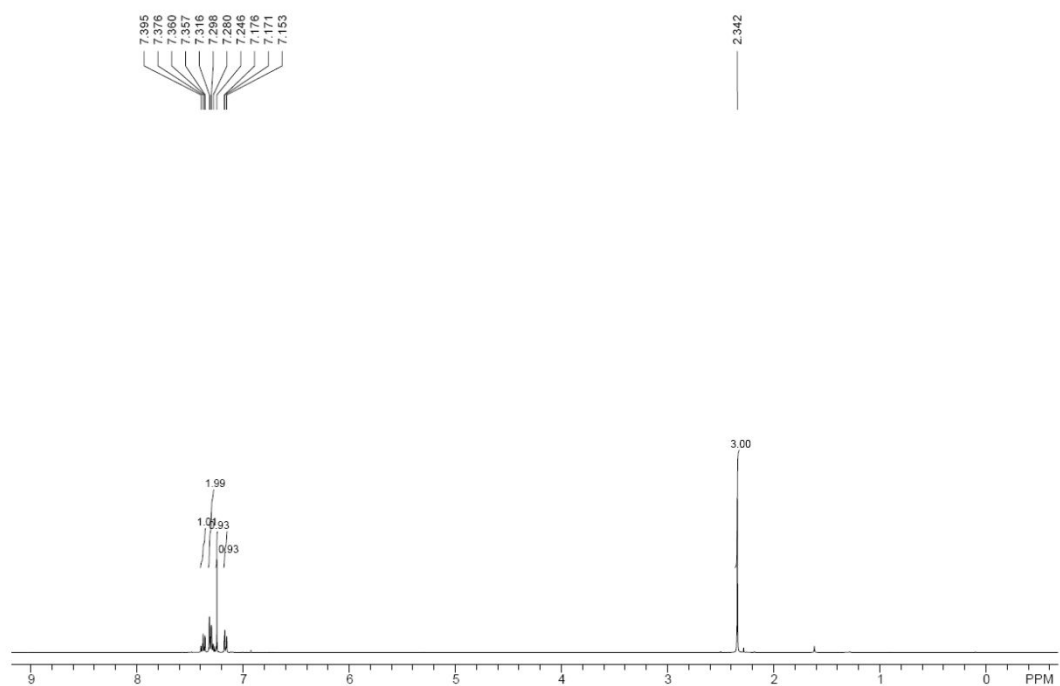


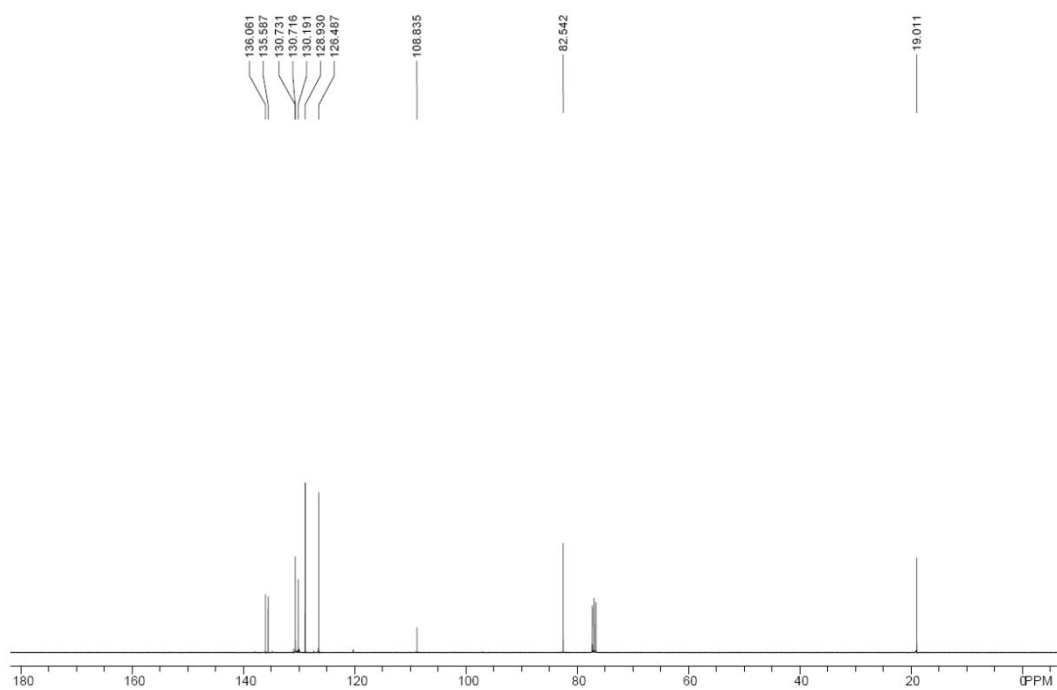
***(Z)*-1-(2-iodo-1-thiocyanatovinyl)-3-methylbenzene (2i)**



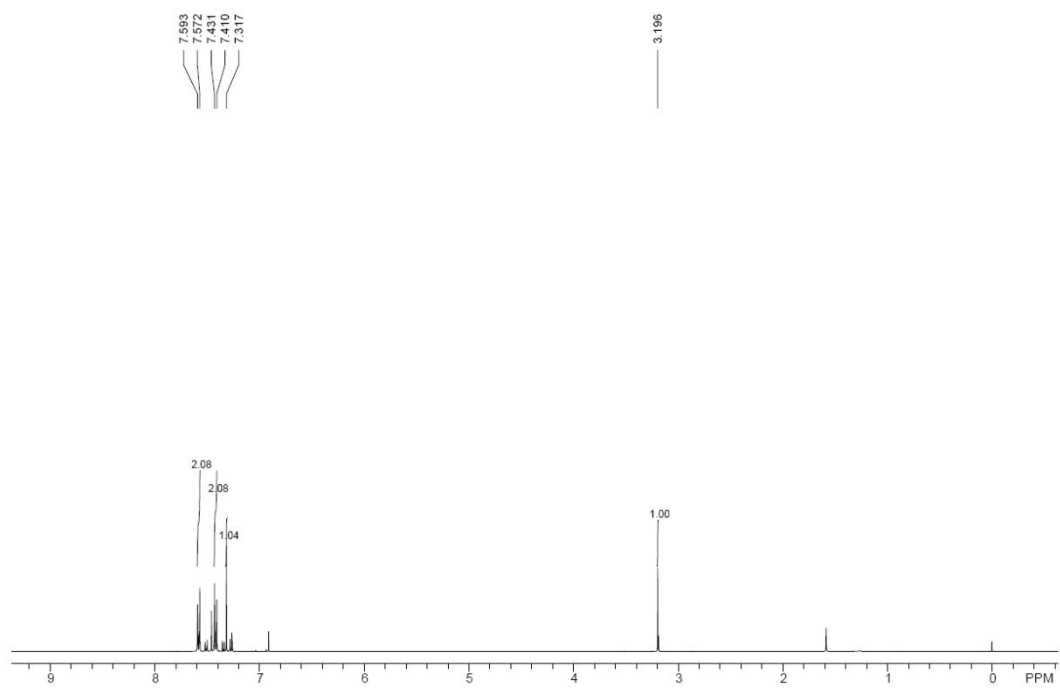


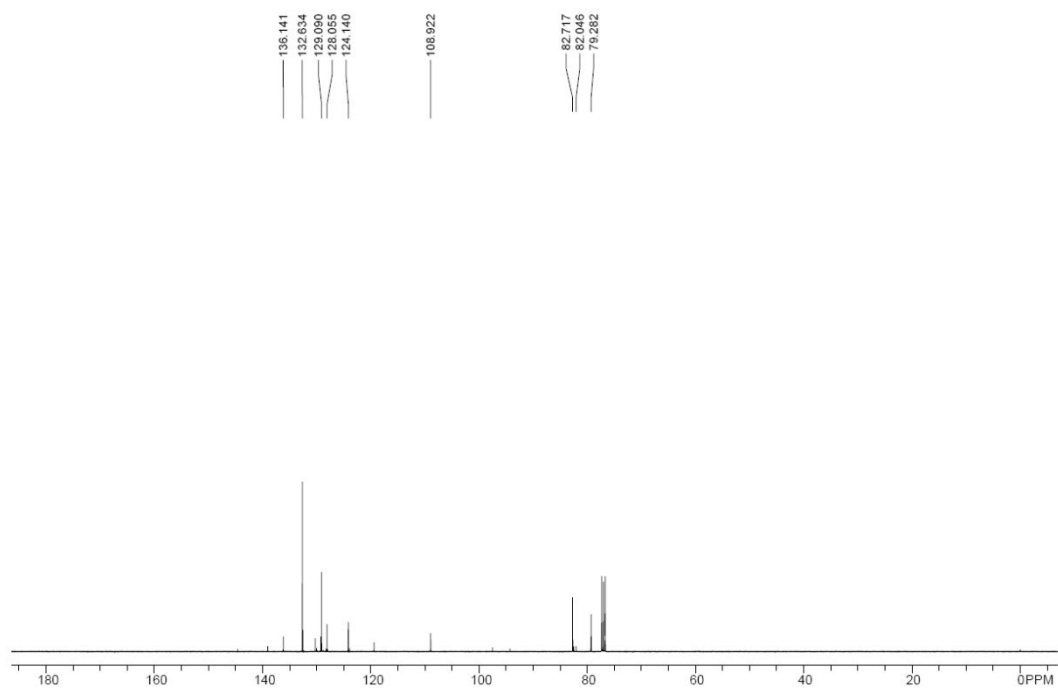
***(Z)*-1-(2-iodo-1-thiocyanatovinyl)-2-methylbenzene (2j)**



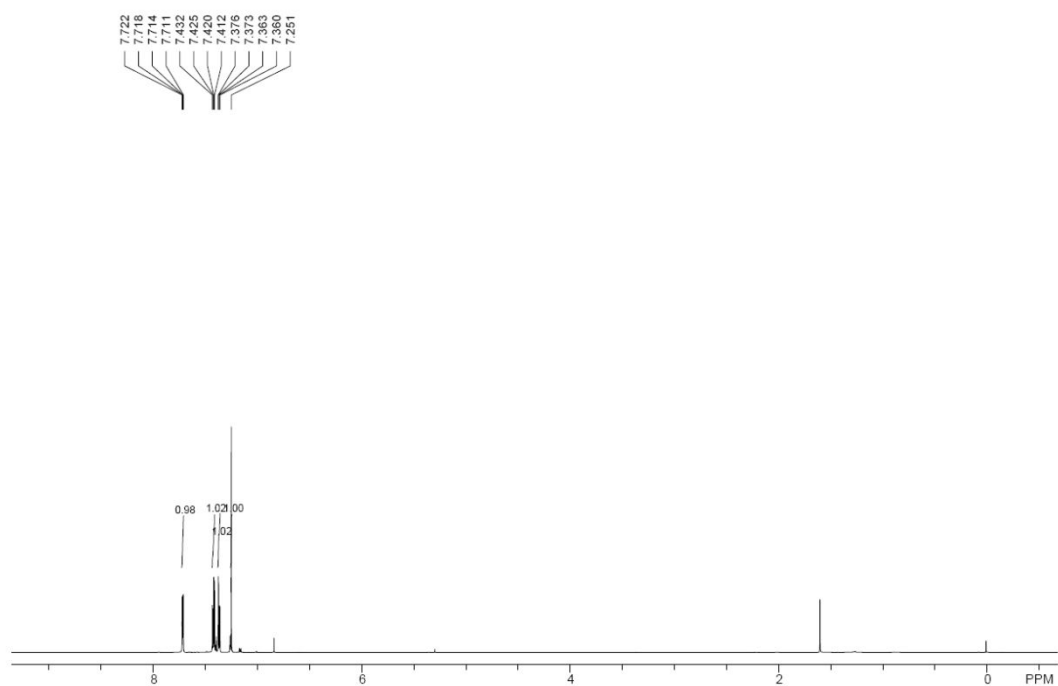


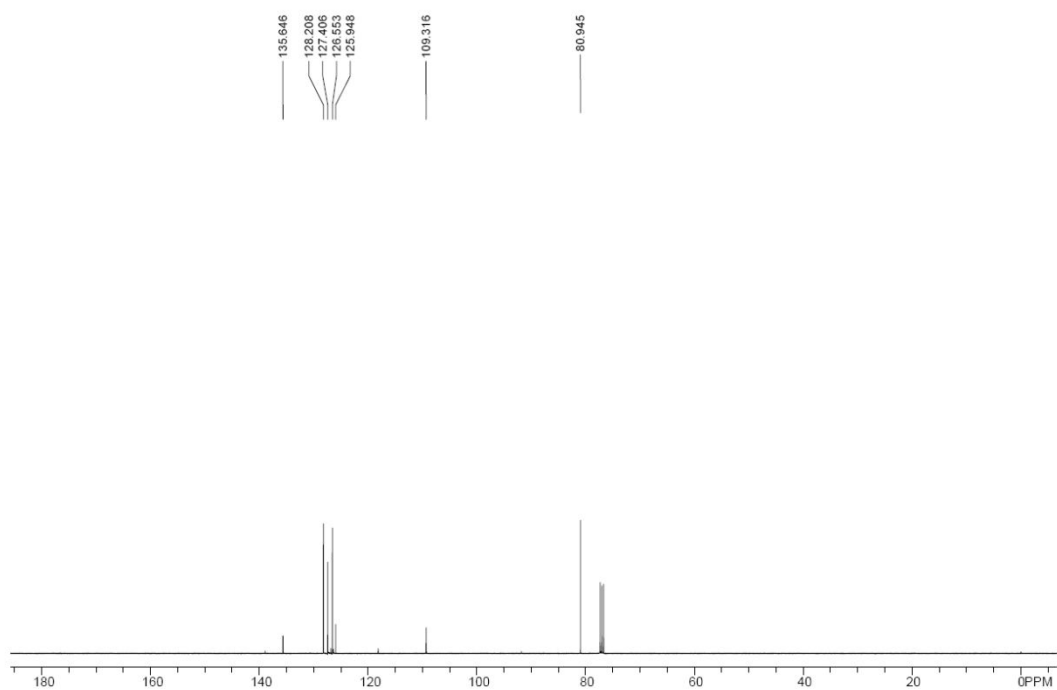
***(Z)*-1-ethynyl-4-(2-iodo-1-thiocyanatovinyl)benzene (2k)**



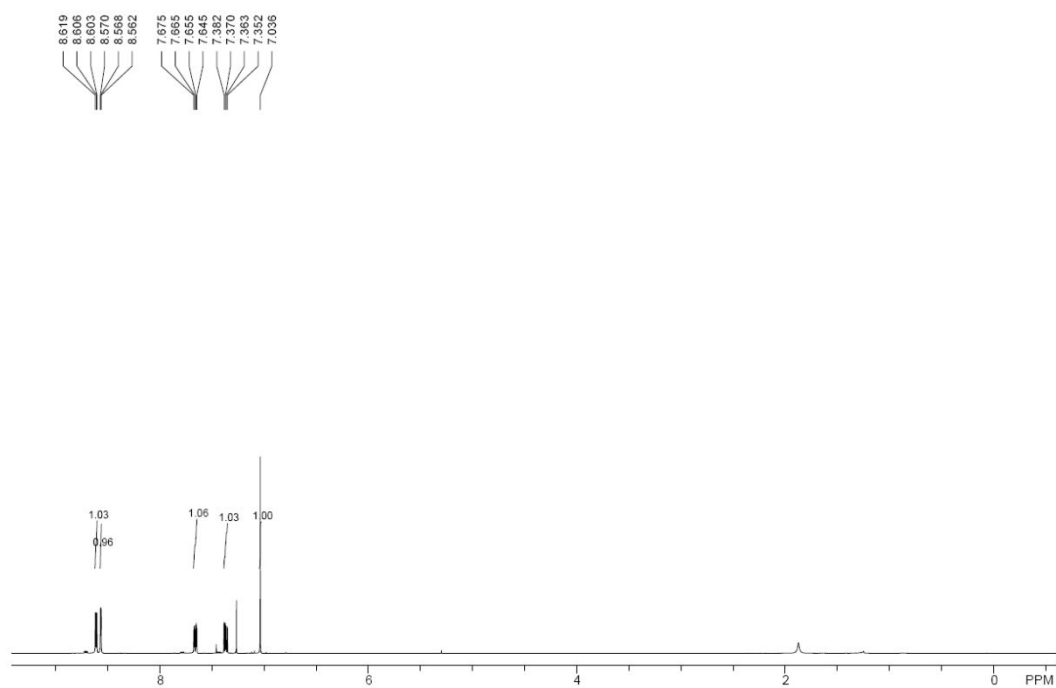


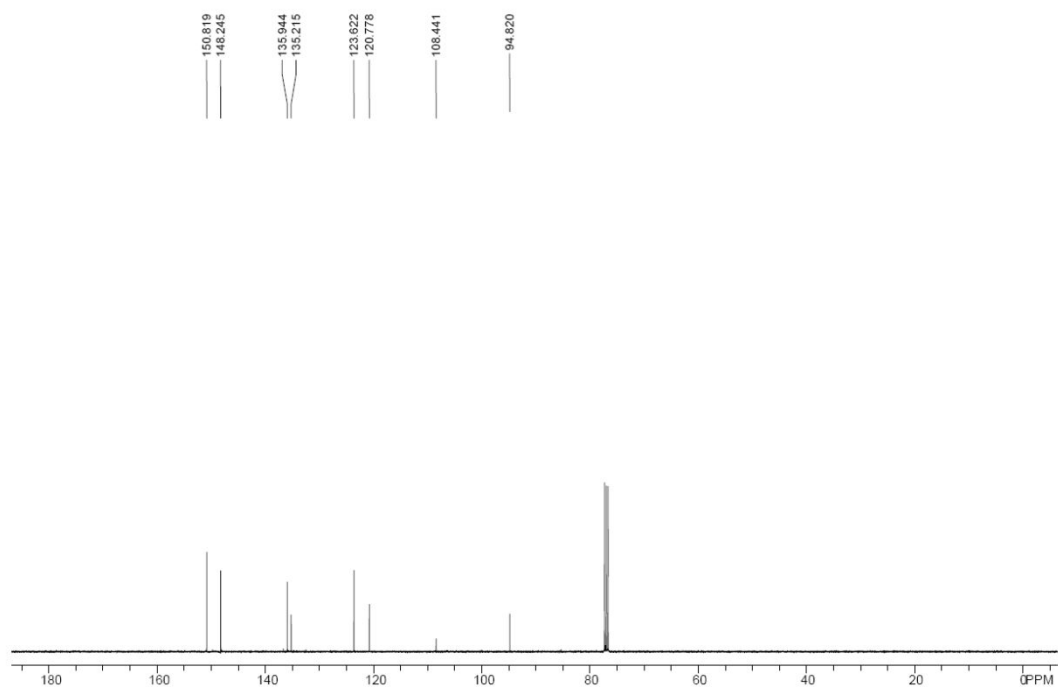
***(Z)*-3-(2-iodo-1-thiocyanatovinyl)thiophene (2l)**



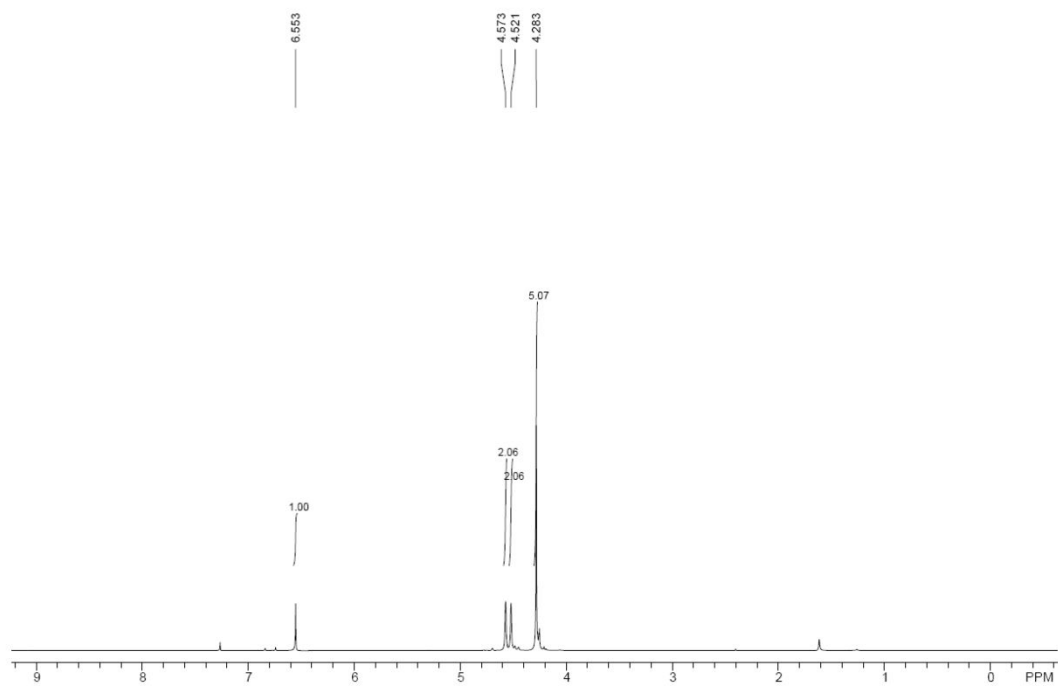


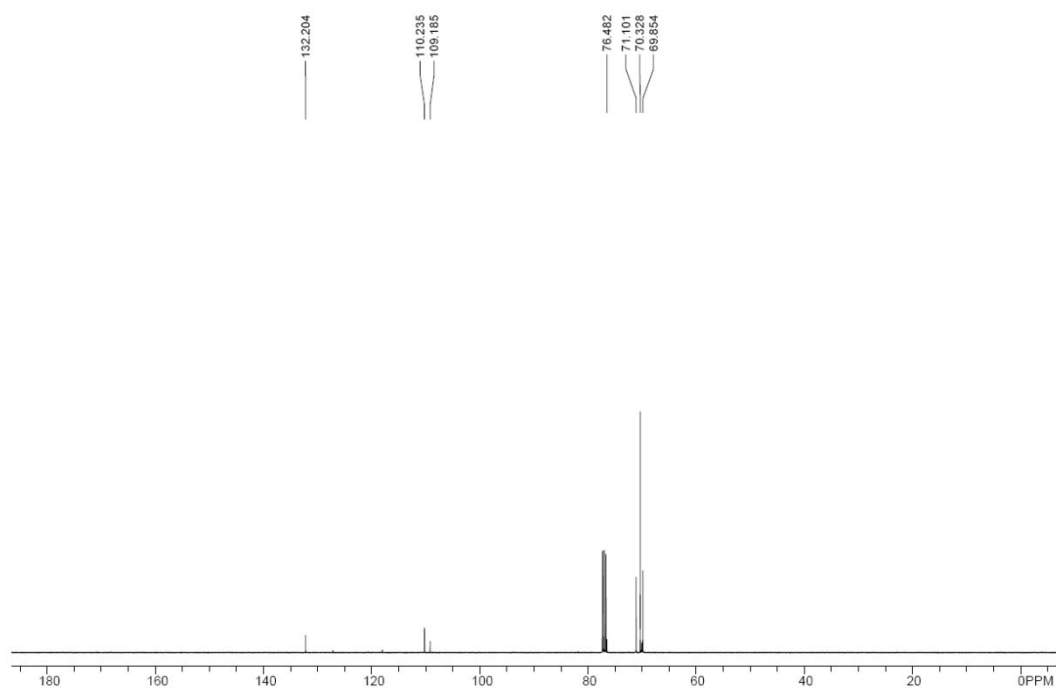
***(Z)*-3-(2-iodo-1-thiocyanatovinyl)pyridine (2m)**



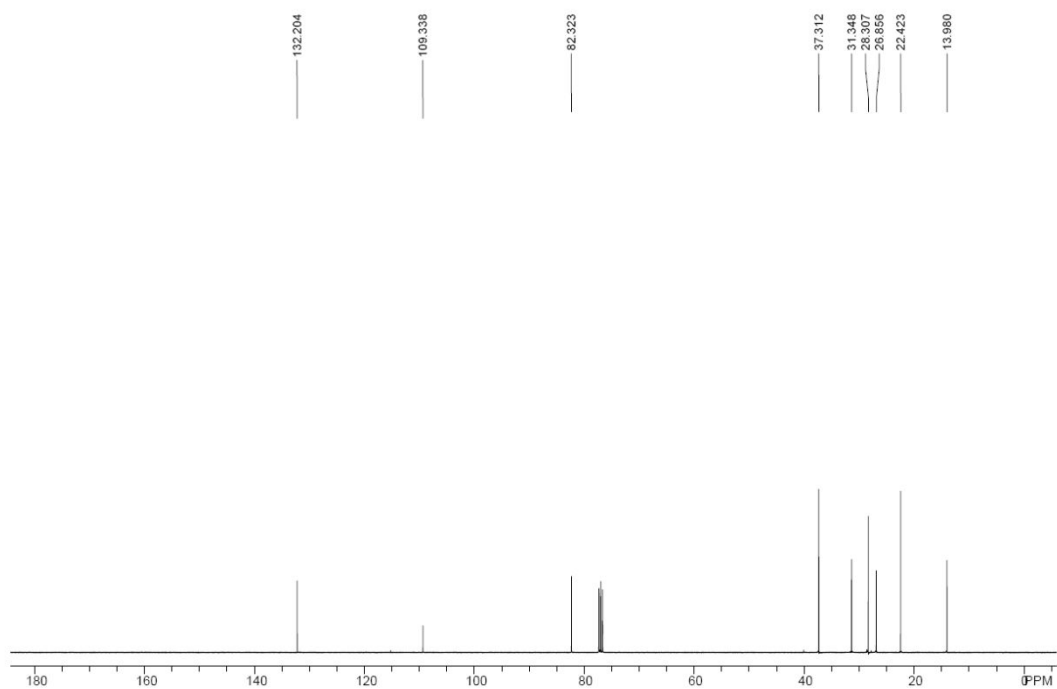
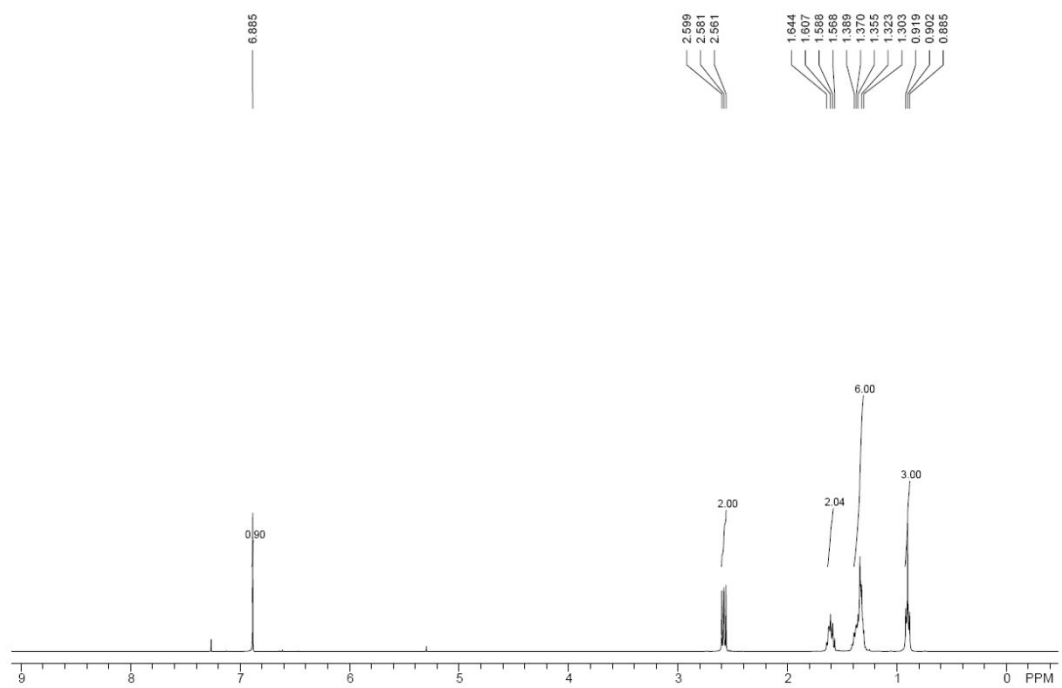


(Z)-(2-iodo-1-thiocyanatovinyl)ferrocene (2n)

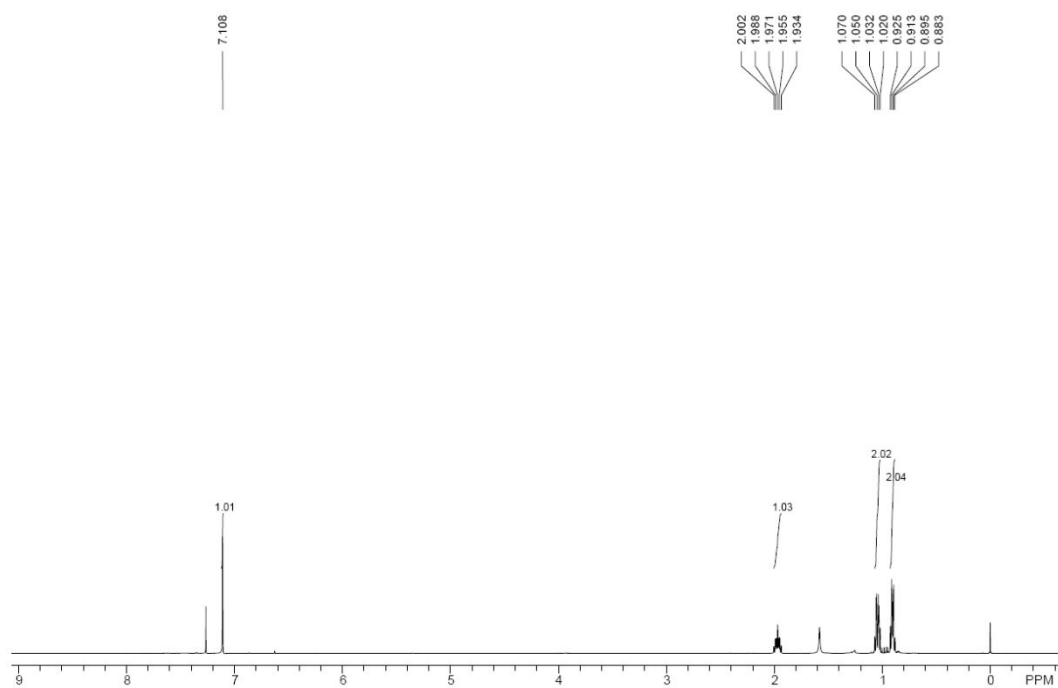


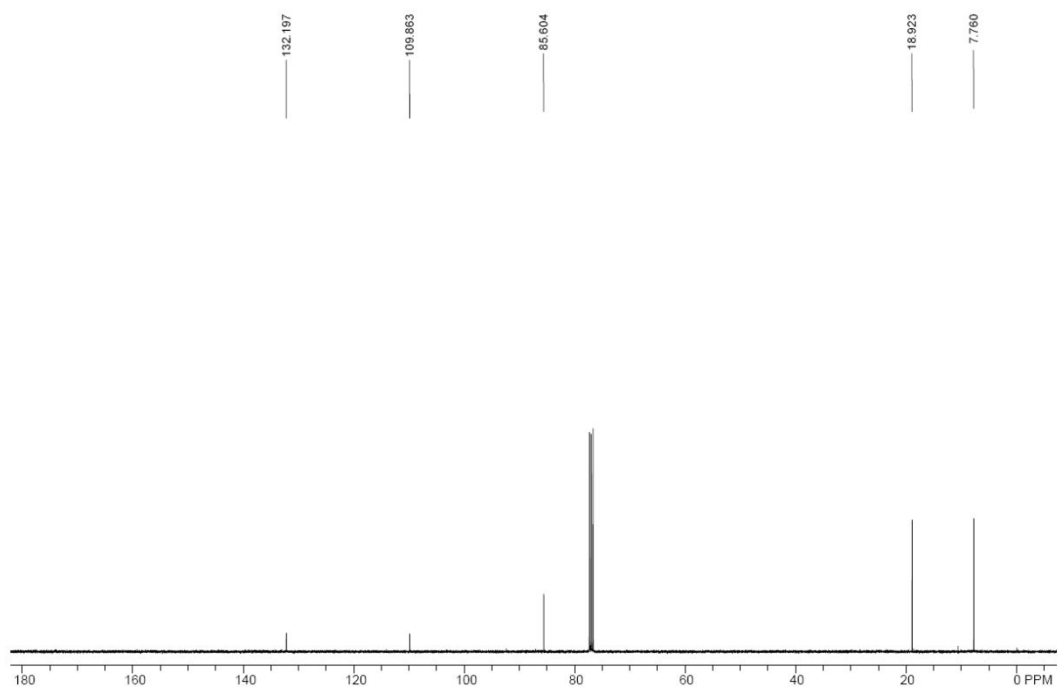


(Z)-1-iodo-2-thiocyanatooct-1-ene (2o)

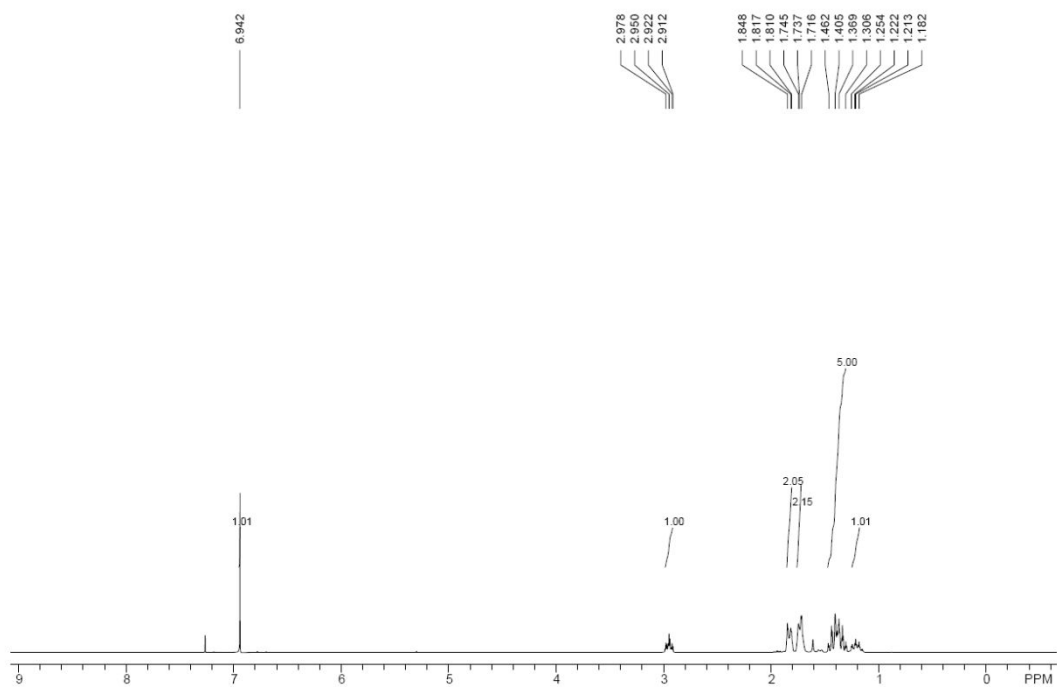


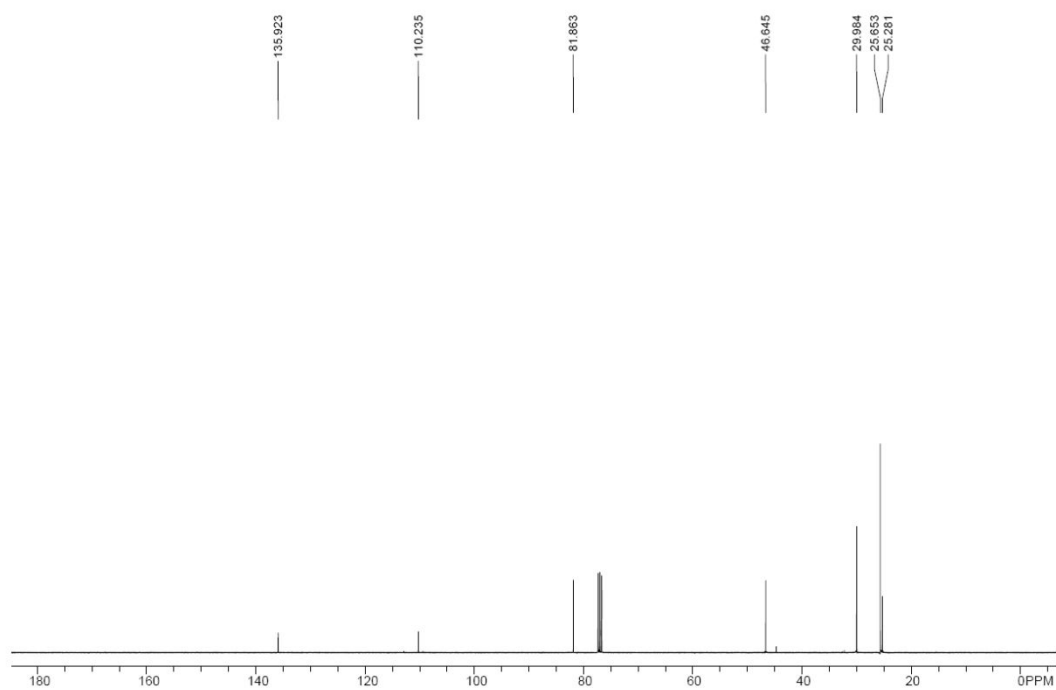
***(Z)*-(2-iodo-1-thiocyanatovinyl)cyclopropane (2p)**



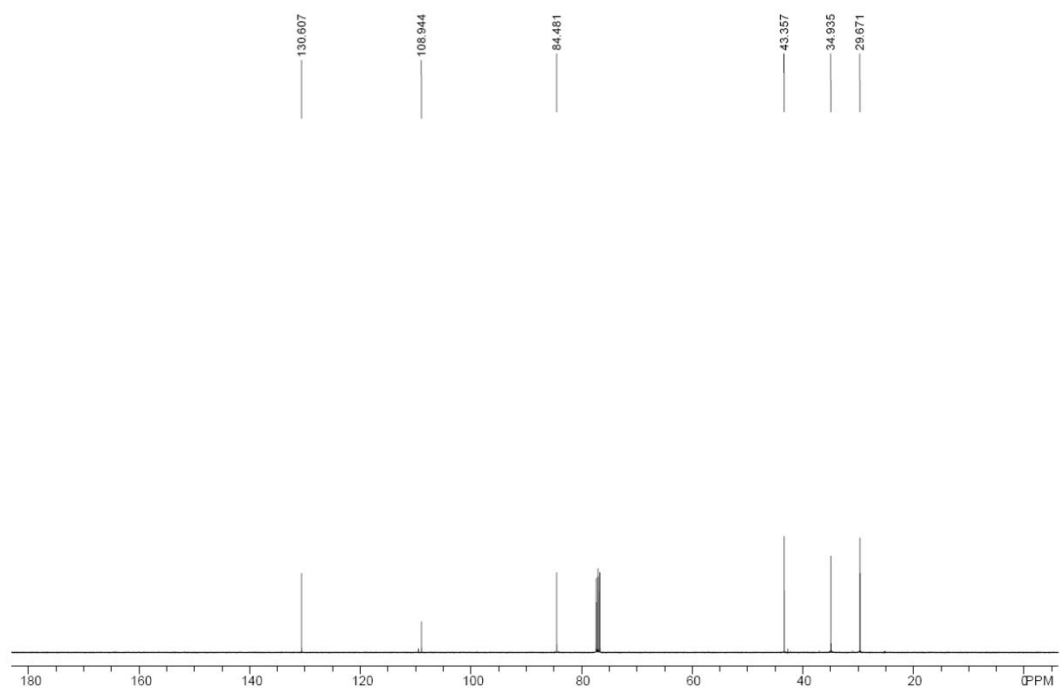
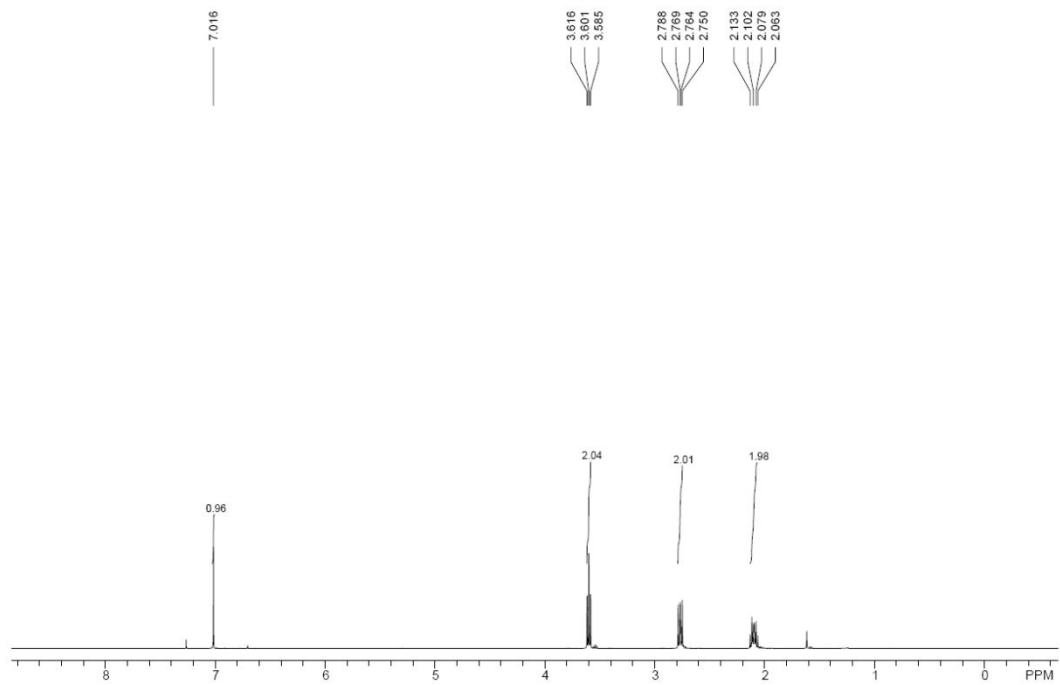


(Z)-(2-iodo-1-thiocyanatovinyl)cyclohexane (2q)

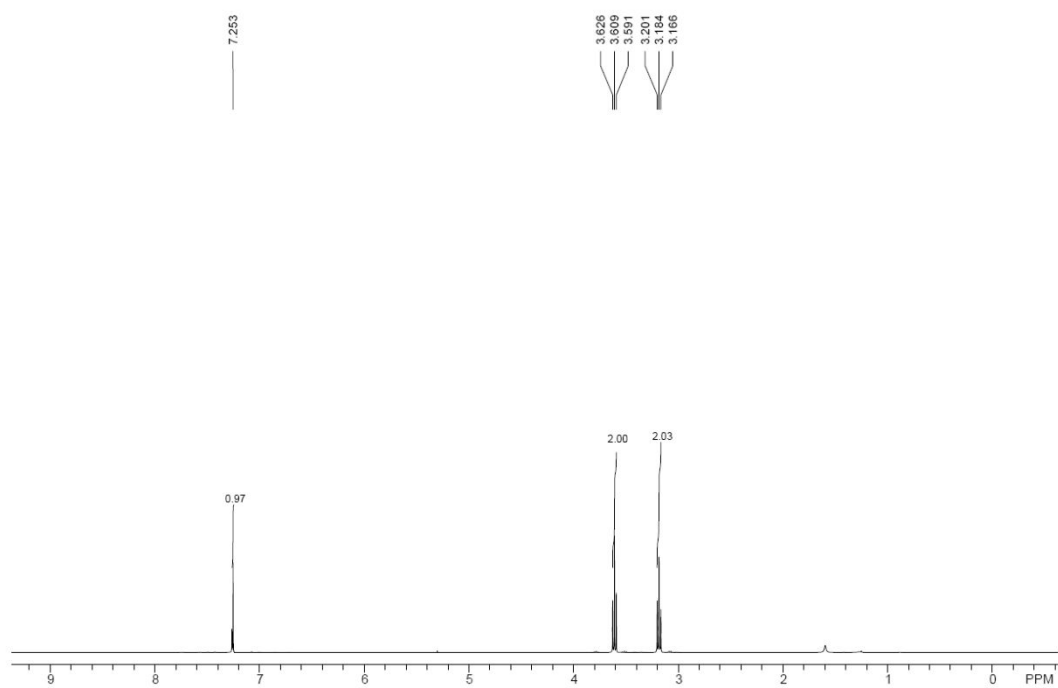


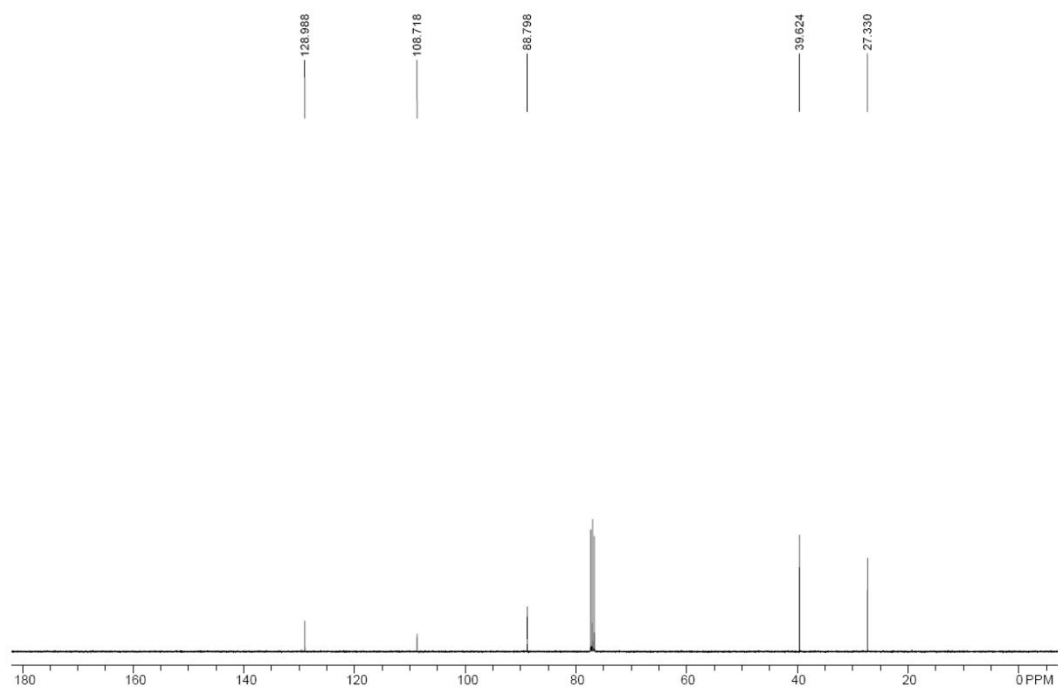


(Z)-5-chloro-1-iodo-2-thiocyanatopent-1-ene (2r)

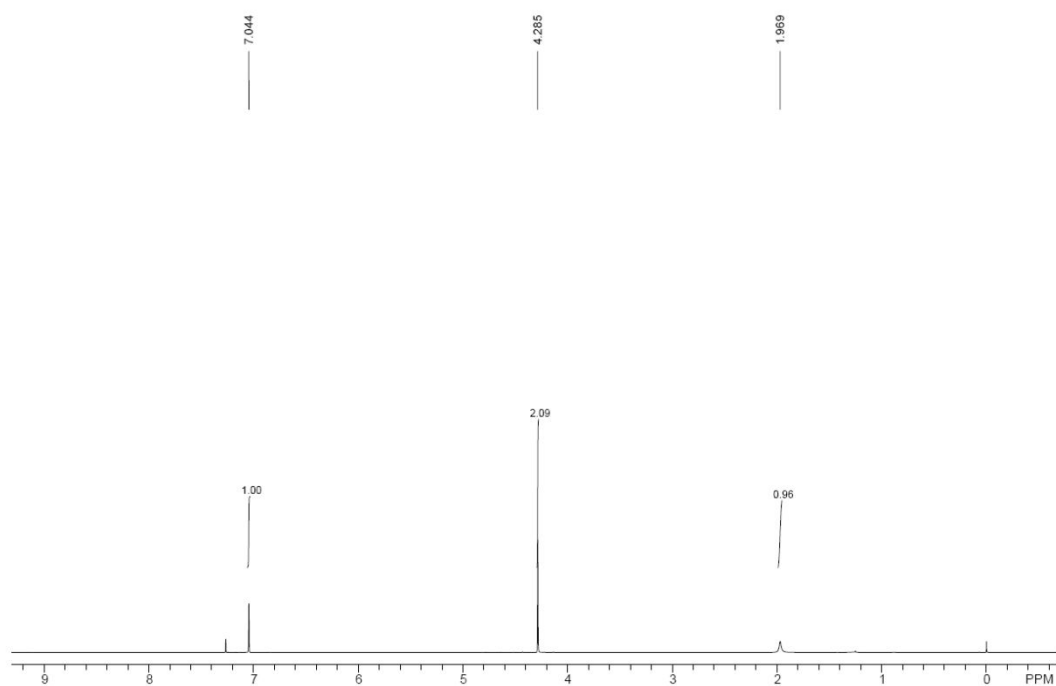


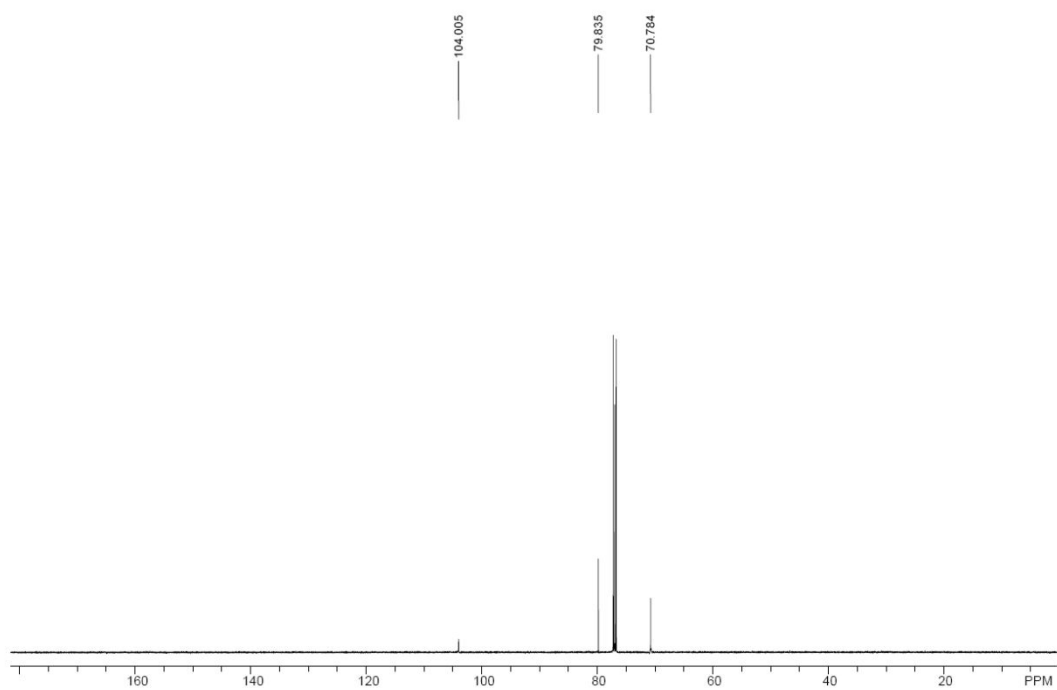
***(Z)*-4-bromo-1-iodo-2-thiocyanatobut-1-ene (2s)**



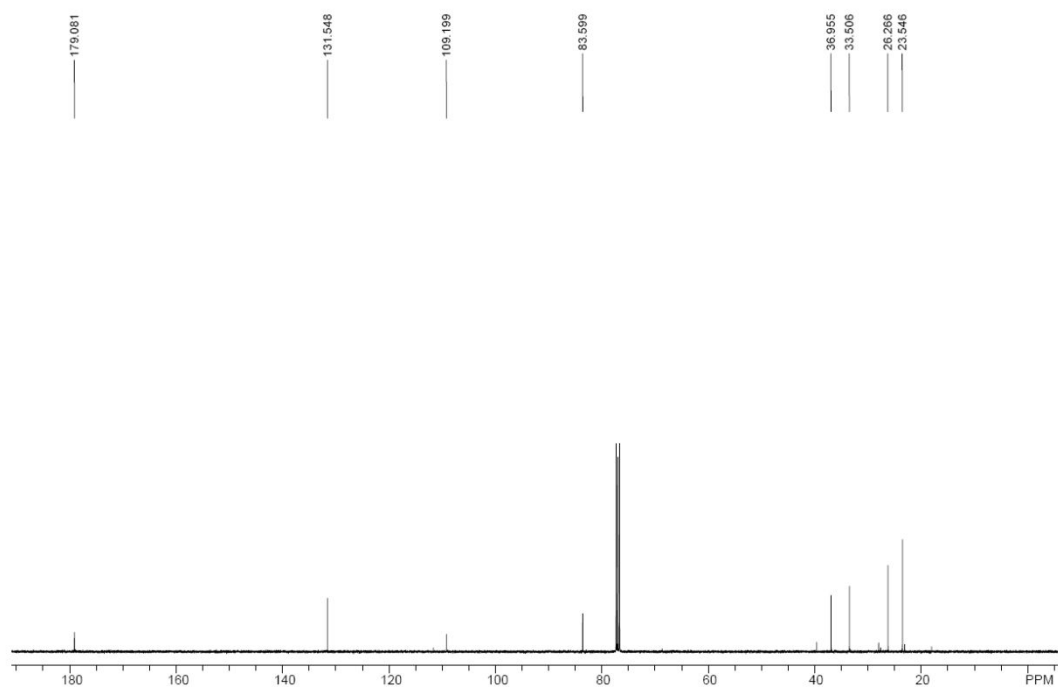
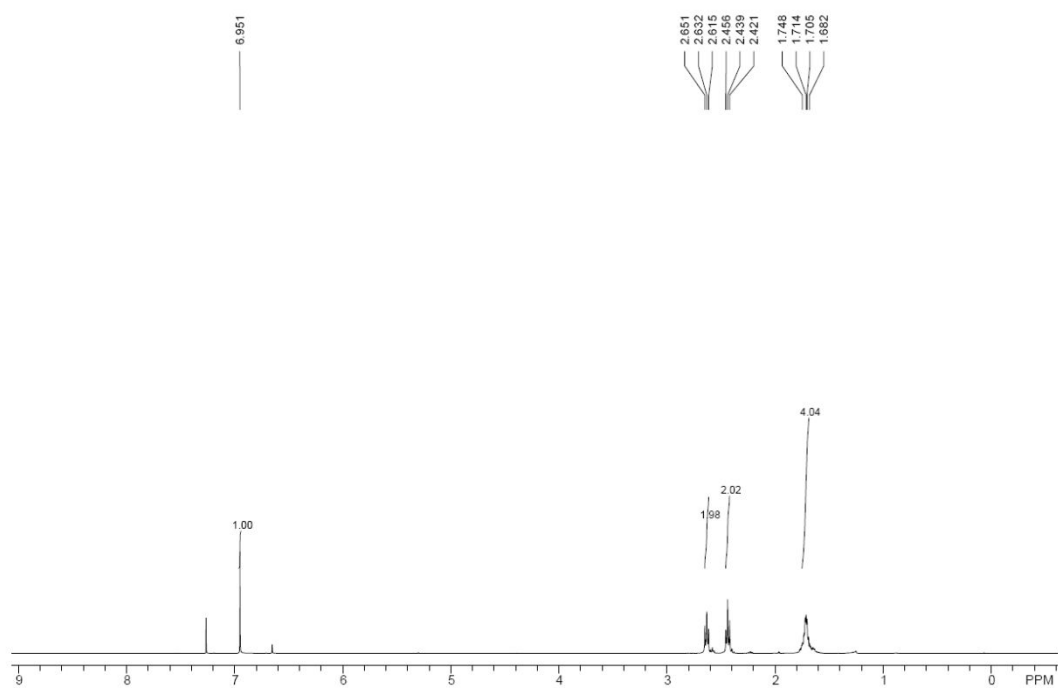


(Z)-3-iodo-2-thiocyanatoprop-2-en-1-ol (2t)

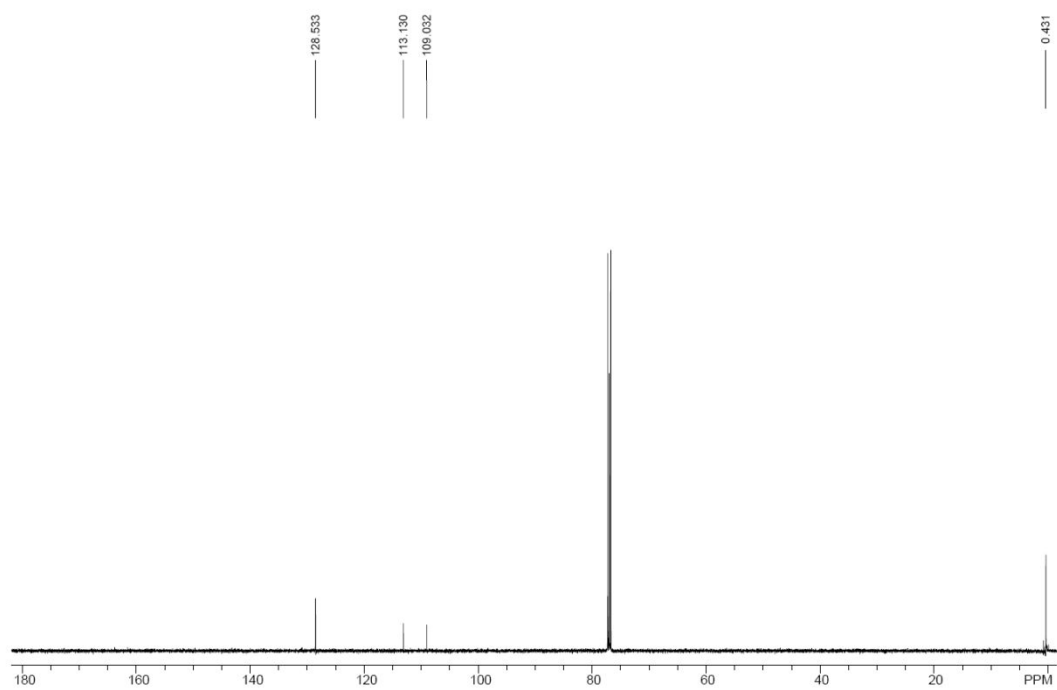
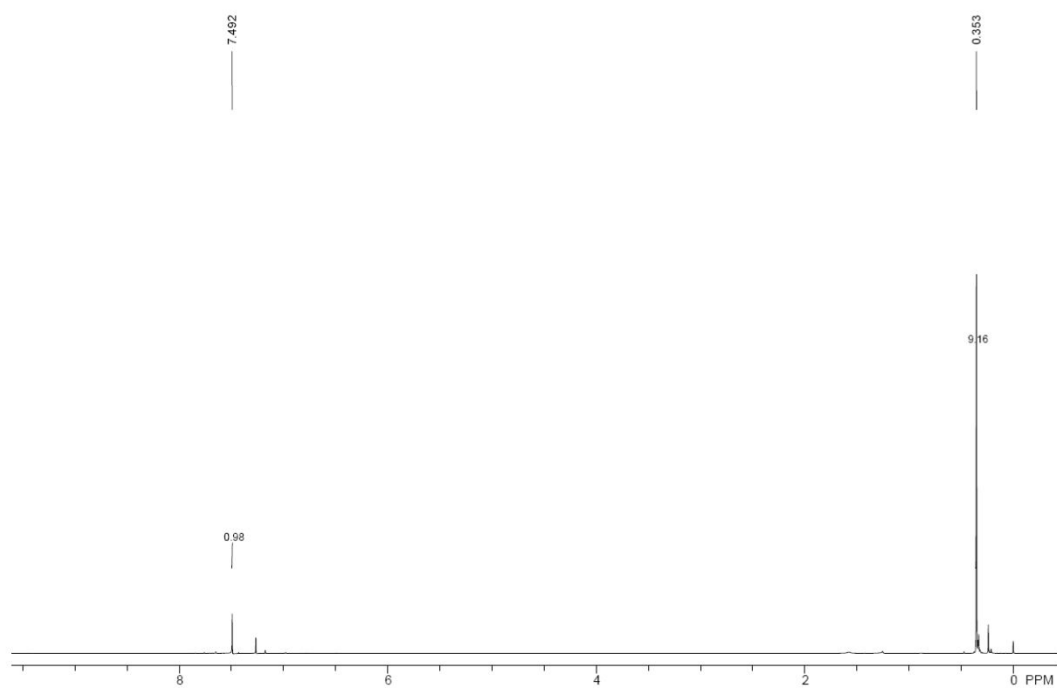




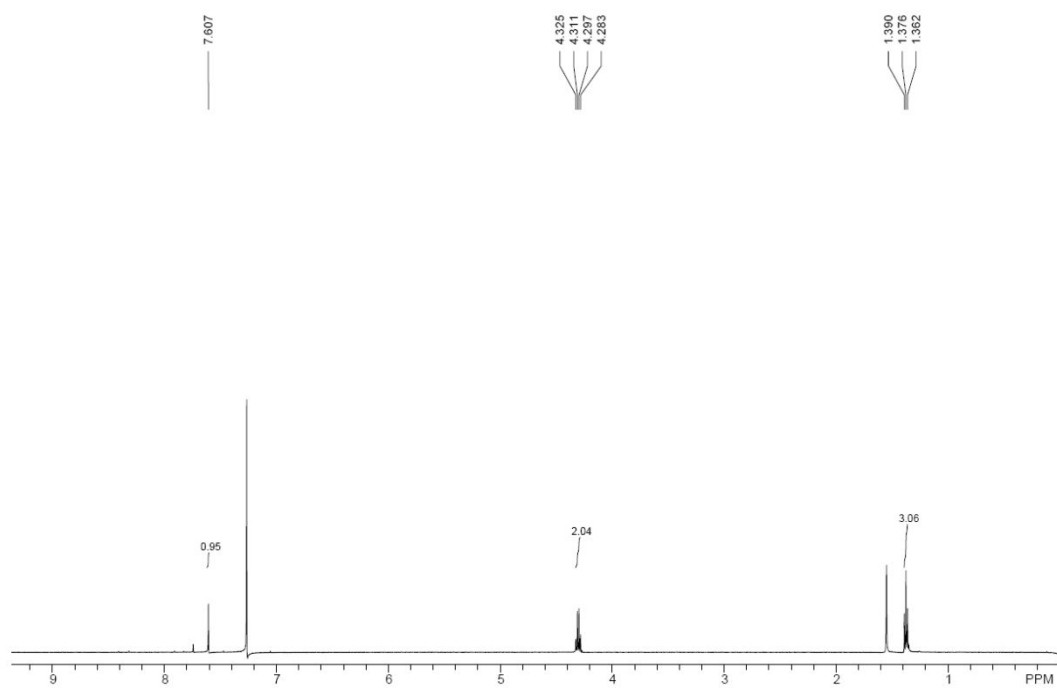
(Z)-7-iodo-6-thiocyanatohept-6-enoic acid (2u)

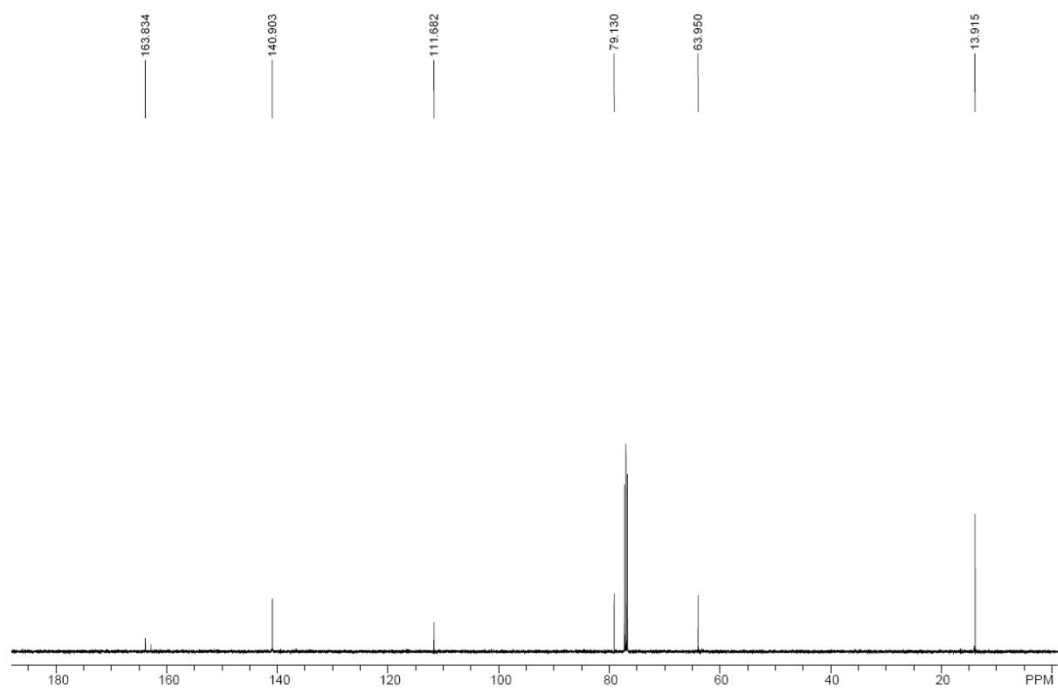


***(Z)*-(2-iodo-1-thiocyanatovinyl)trimethylsilane (2v)**

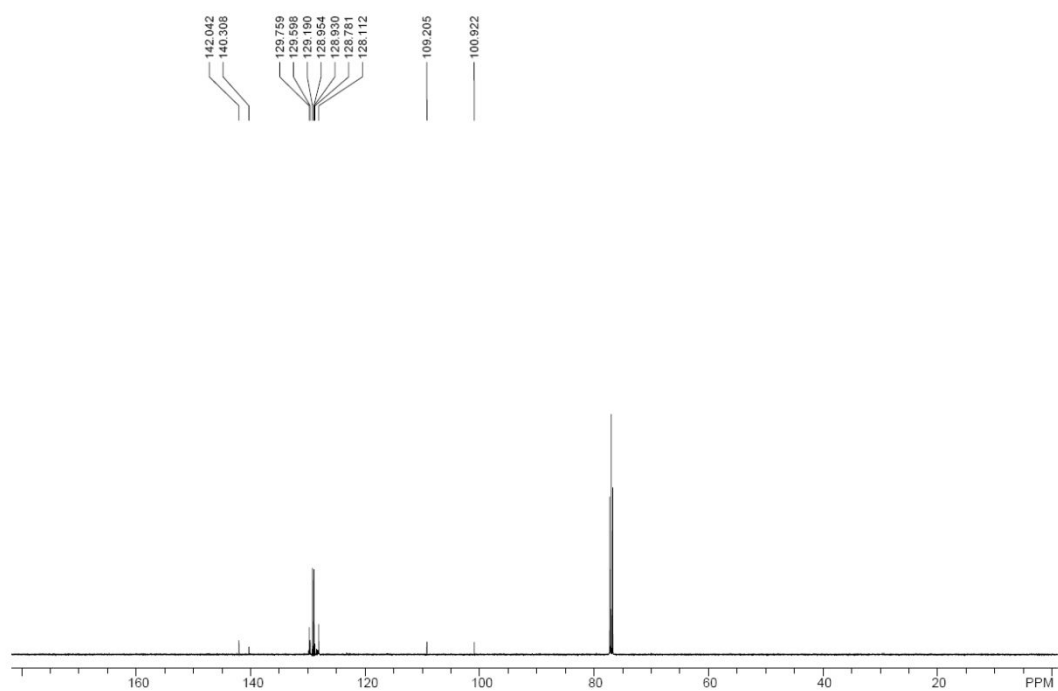
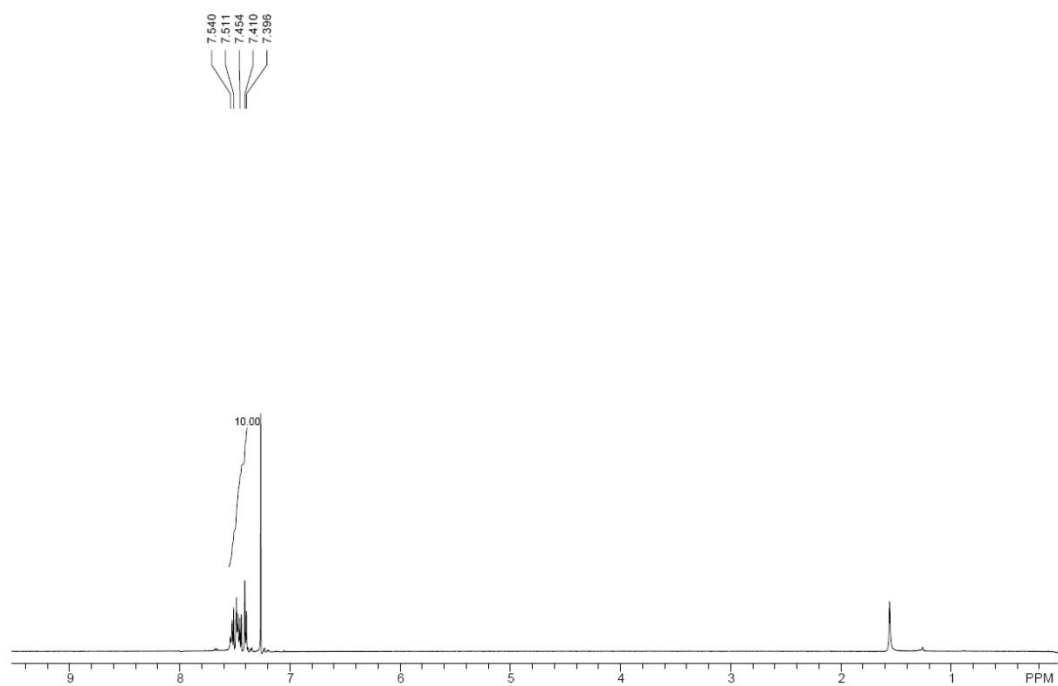


***(Z)*-ethyl 3-iodo-2-thiocyanatoacrylate (2w)**

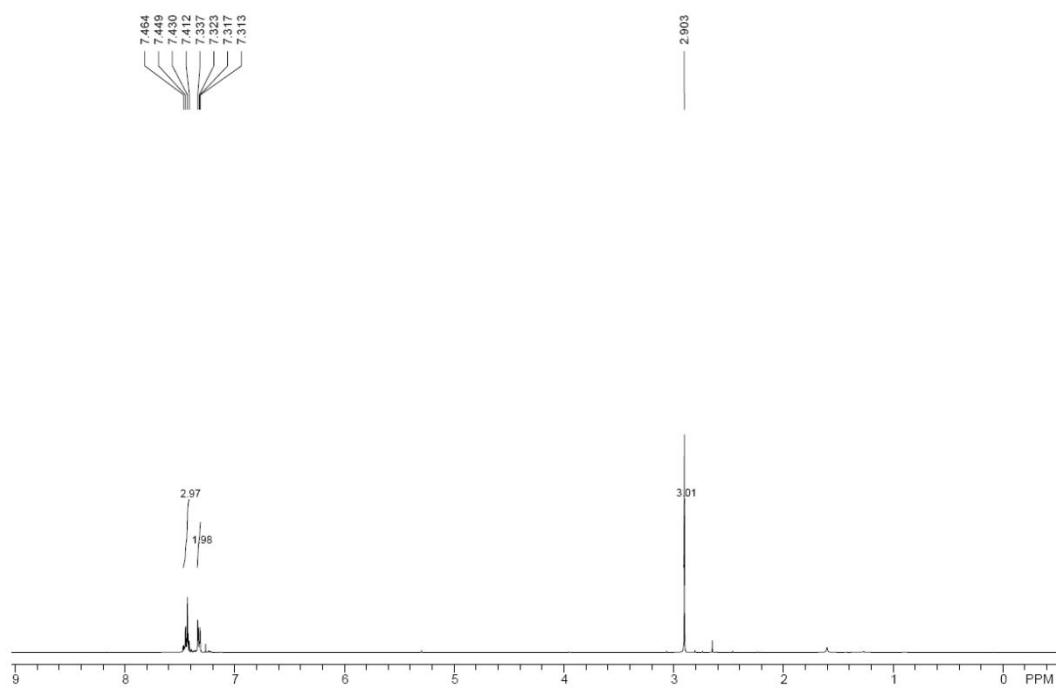


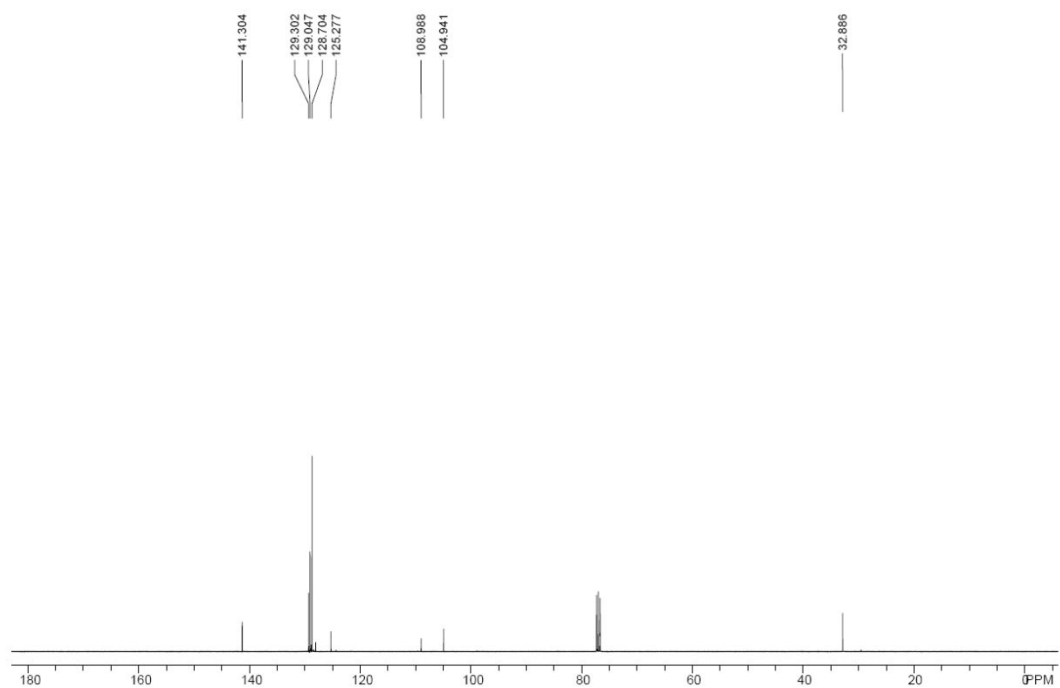


(Z)-5-iodo-6-thiocyanatodec-5-ene (2x)

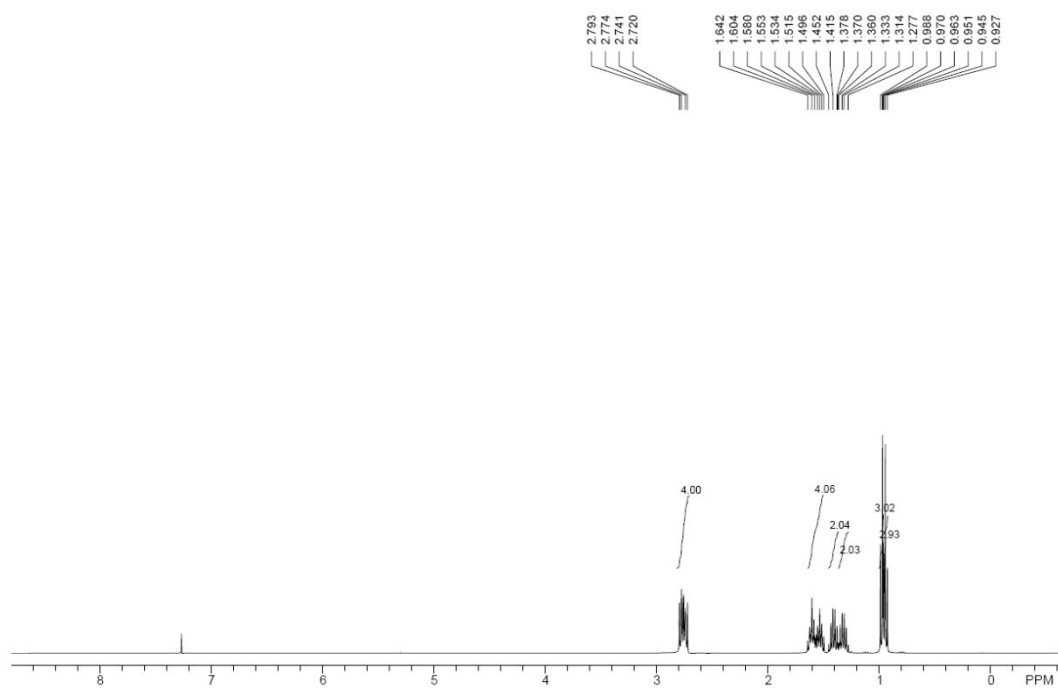


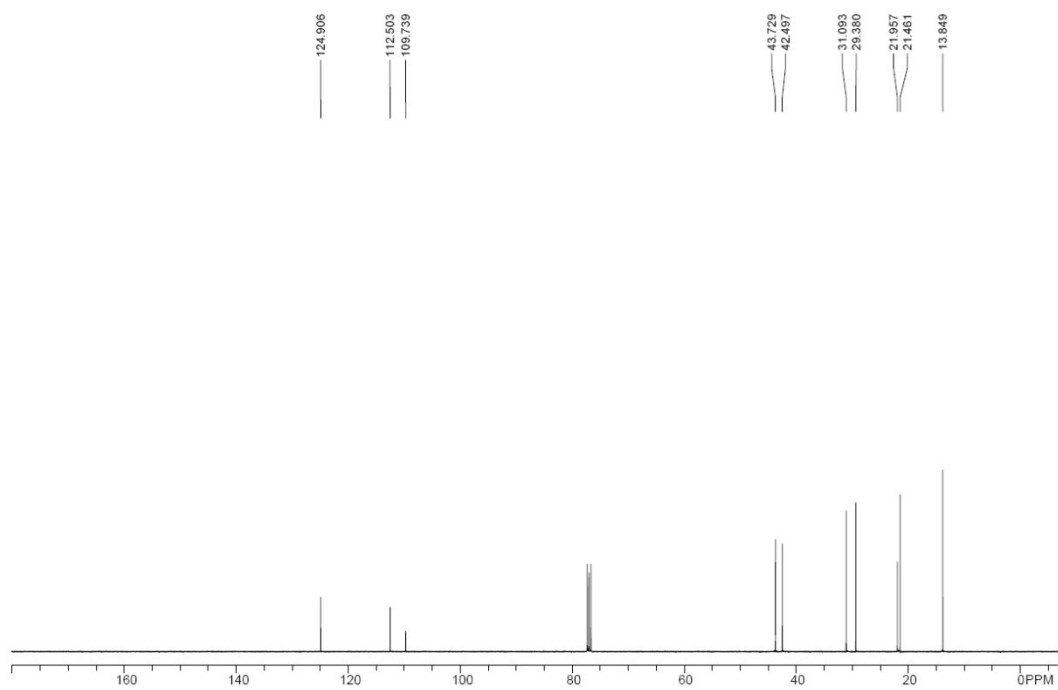
***(Z)*-(2-iodo-1-thiocyanatoprop-1-en-1-yl)benzene (2y)**



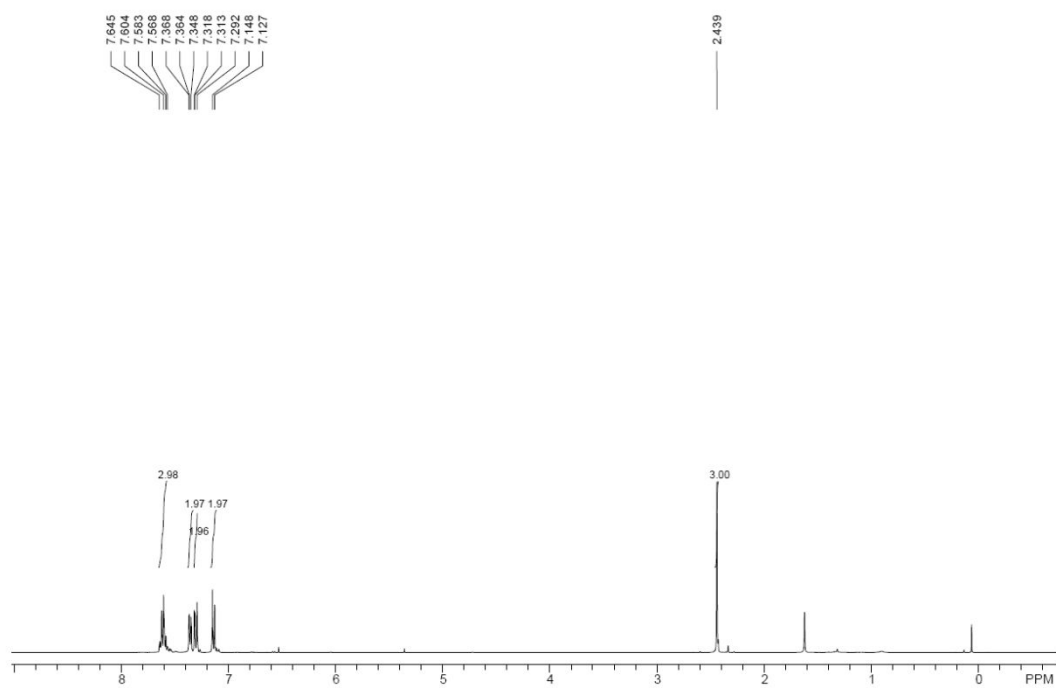


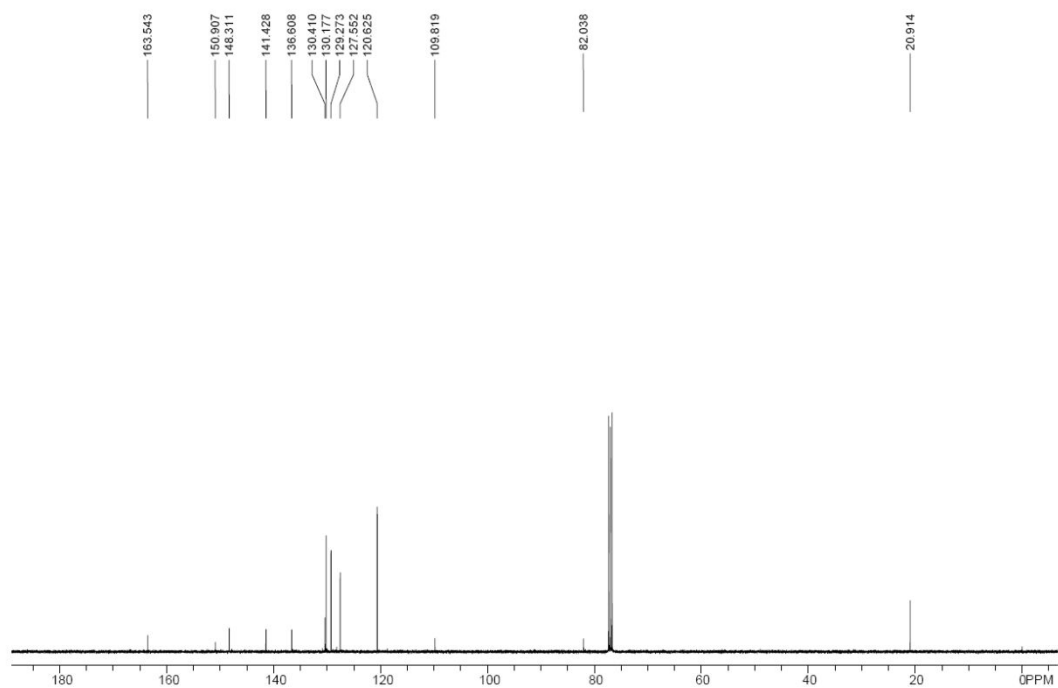
***(Z)*-(1-iodo-2-thiocyanatoethene-1,2-diyl)dibenzene (2z)**





***p*-tolyl (Z)-3-iodo-3-phenyl-2-thiocyanatoacrylate (2aa)**





***(E)*-1-(1,2-diiodovinyl)-4-methylbenzene (3a)**

