Supporting Information

New one-pot method for the synthesis of pyrrolidinofullerenes

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General considerations. Commercially available [60]fullerene and methyl or ethyl isocyanoacetates (99.5% pure, Sigma-Aldrich) was used. The reaction products were analyzied on a HPLC chromatograph HEWLETT PACKARD (model HP 1090 Liquid Chromatograph) at 340 nm. The mixtures were separated on a metal preparative column Cosmosil Buckyprep Waters (250×10 mm) at ~20 ^{o}C. Toluene was used as eluent, the flow rate was 3.0 mL•min^{-1}. The ^{1}H and ^{13}C NMR spectra were run on a Bruker Avance-500 spectrometer at 500.17 and 125.78 MHz, respectively. The mixture of $CDCl_3$ and CS_2 (1:5) was used as a solvent. The mass spectra were obtained on a MALDI TOF/TOF Autoflex-III Bruker and UltraFlex III TOF/TOF (Bruker Daltonik GmbH, Germany) operating in a linear mode. S_8 and DCTB (trans-2-[3-(4-tret-butylphenyl)-2-methyl-2-propenyliden]malononitrile) are used as a matrix. For the application on a metal target, the toluene solutions of the samples were used.

General synthetic procedure. A 50 mL glass reactor was charged with C_{60} (20 mg, 0.0278 mmol) in dry chlorobenzene (3 mL), Ti(O*i*-Pr)$_4$ (0.04 mL, 0.14 mmol) and methyl or ethyl isocyanoacetate (0.2 mmol) under a dried argon atmosphere at 0 ^{o}C. The resulting solution was heated to 80 ^{o}C, and RMgBr (1 M solution in diethyl ether, 0.334 mmol) was added dropwise during 2—3 min. The reaction mixture stirred for 15 min and was quenched with an 8-10% (aq) solution of HCl. The layers were separated and the organic layer passed through a column with small amount of silica gel. The reaction products **1-6**, and the starting fullerene C_{60} were separated by the semi-preparative HPLC, eluent was toluene.

Electrochemistry. All experiments were carried out under a dry argon atmosphere. Bu_4NBF_4 was purchased from Aldrich and was used without further purification. Anhydrous solvent dichlorobenzene (Acros Organics) was obtained by distillation over phosphoric anhydride. Cyclic voltammograms were recorded with a BASi Epsilon potentiostate (USA) at room temperature in o- dichlorobenzene (10^{-3} M substrate concentration). 0.1 M Bu_4NBF_4 was used as the supporting electrolyte and a glassy carbon electrode was used as a working electrode. The auxiliary electrode was a platinum rod. All potentials are referenced against the $Ag/AgNO_3$ redox couple and recalculated from Fc^{+}/Fc. The scan rate was 100 mV·s^{-1}.

Electrophysics

Measure the resistance of the films was carried out using a digital meter resistance AKIP-8602 AKIP company. Accuracy of resistance measurement: ± 5%. On resistance is determined by the value of conductivity (G = 1/R). The temperature was measured with a digital multimeter APPA-107N with an accuracy of about ± 1%.

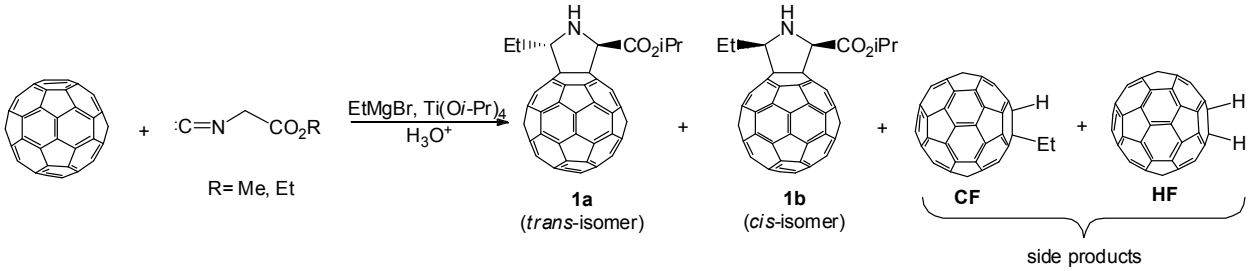

Table 1. Effect of the ratio of the reactant and catalyst components on the formation of the products reaction.

C_{60} : isocyanoacetate : [Mg] : [Ti]	**Conversion C_{60}, %**	**Ratio 1a : 1b : CF : HF**
1 : 1 : 8 : 3	100	Adducts of poly carbomagnesiation and hydrogenation of C_{60}
1 : 2 : 8 : 3	100	Adducts of poly carbomagnesiation and hydrogenation of C_{60}
1 : 4 : 8 : 3	**65**	**1 : 2 : < 0.1 : <0.1**
1 : 6 : 8 : 3	66	1 : 2 : < 0.1 : <0.1
1 : 4 : 1 : 1	2-3	not calculated
1 : 4 : 2 : 1	8	not calculated
1 : 4 : 4 : 2	15	1 : 2 : < 0.1 : <0.1
1 : 4 : 6 : 1	40	1 : 2 : 10 : 0
1 : 4 : 6 : 2	42	1 : 2 : 3 : 1
1 : 4 : 6 : 3	35	1 : 2 : <1 : < 1
1 : 4 : 10 : 4	75	1 : 2 : 2 : 6

Conditions: 80 ^{o}C, 15 min, solvent- dry chlorobenzene.

Table 2. Effect of temperature on the reaction pathway.

Temperature, ^{o}C	**Total yield 1a and 1b, %**
20	0
40	8
60	15
80	65
100	63

Conditions: C_{60} : isocyanoacetate : [Mg] : [Ti] ratio of 1 : 4 : 8 : 3, 15 min, solvent- dry chlorobenzene.

Mixture of isomers of *iso*-propyl (2-ethyl-3,4-fullero[60]pyrrolidine-5-yl)-1*H*-carboxylates 1a+1b (cis/trans = 2:1). IR: 527, 753, 1103, 1143, 1180, 1202, 1374, 1384, 1427, 1460, 1753, 3448 cm^{-1}. UV ($CHCl_3$), λ_{max}, nm: 256, 319, 429. ^{15}N NMR (50.69 MHz, $CDCl_3$): δ 55.17 (*trans*-isomer), 60.96 (*cis*-isomer). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.21 (d, 3H, CH_3, i-Pr (*trans*-isomer), J = 7.0), 1.22 (d, 3H, CH_3, i-Pr (*cis*-isomer), J = 7.0), 1.38 (d, 3H, CH_3, i-Pr (*cis*-isomer), J = 7.0), 1.41 (d, 3H, CH_3, i-Pr (*trans*-isomer), J = 7.0), 1.53 (t, 3H, CH_3, Et (*trans*-isomer), J = 7.0), 1.58 (t, 3H, CH_3, Et (*cis*-isomer), J = 7.0), 2.20 and 2.82 (both m, 2H, CH_2, Et (*cis*-isomer)), 2.32 and 2.58 (both m, 2H, CH_2, Et (*trans*-isomer)), 4.68 (dd, 1H, CH, pyrrolidine (*cis*-isomer), J = 2.0, J = 7.0), 5.17 (dd, 1H, CH, pyrrolidine (*trans*-isomer), J = 2.0, J = 7.0), 5.24 (m, 1H, CH, *i*-Pr (*cis*-isomer)), 5.26 (m, 1H, CH, *i*-Pr (*trans*-isomer)), 5.43 (s, 1H, CH, pyrrolidine (*cis*-isomer)), 5.56 (s, 1H, CH, pyrrolidine (*trans*-isomer)). ^{13}C NMR (125 MHz, $CDCl_3$): δ 13.01, 13.40, 21.89, 22.05, 22.08, 22.13, 26.90, 28.58, 69.77, 70.09, 72.32, 74.03, 74.20, 75.24, 76.77, 77.58, 78.34, 79.38, 135.07, 135.45, 135.84, 136.31, 136.39, 136.54, 136.74, 139.38, 139.49, 139.81, 140.02, 140.29, 140.32, 141.78, 141.85, 141.88, 141.89, 141.97, 142.02, 142.16, 142.20, 142.25, 142.30, 142.35, 142.53, 142.71, 142.75, 142.77, 142.81, 143.12, 143.16, 143.22, 143.28, 144.29, 144.34, 144.45, 144.49, 144.54, 144.57, 145.21, 145.24, 145.30, 145.33, 145.38, 145.45, 145.52, 145.58, 145.62, 145.71, 145.74, 145.88, 145.92, 146.02, 146.05, 146.10, 146.13, 146.26, 146.31, 146.39, 146.77, 147.03, 147.12, 147.23, 150.94, 151.46, 151.77, 152.38, 153.00, 153.33, 153.70, 154.96, 155.47, 168.76, 169.99. MALDI TOF: *m/z* found 878.044 $[M+H]^+$, calc. 877.110 ($C_{68}H_{15}NO_2$).

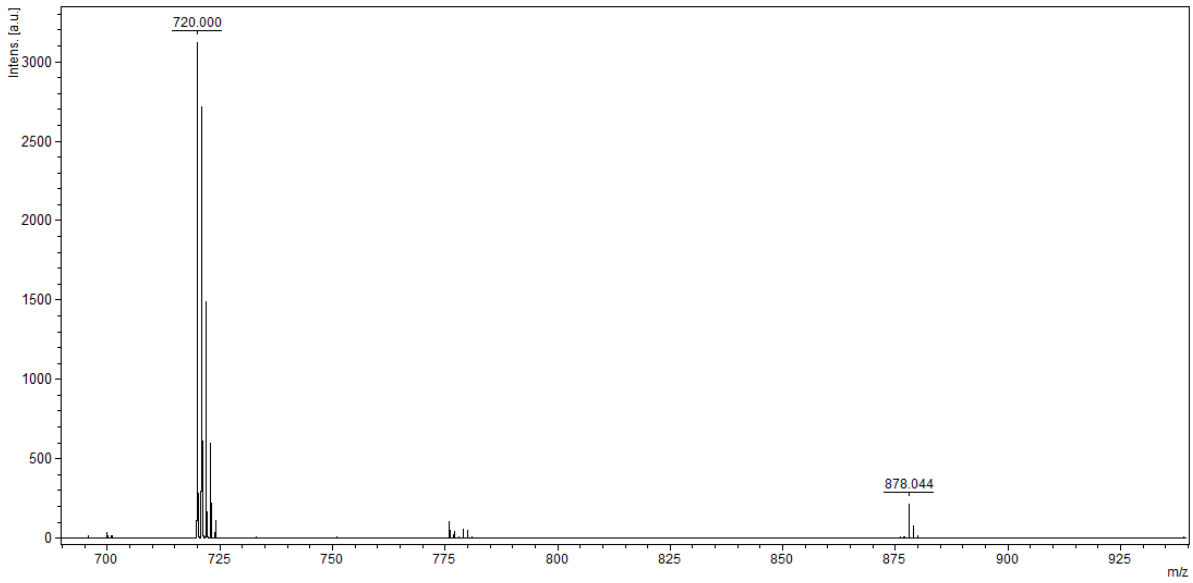

Figure 1. Mass spectra MALDI TOF of compound **1** (matrix – S_8).

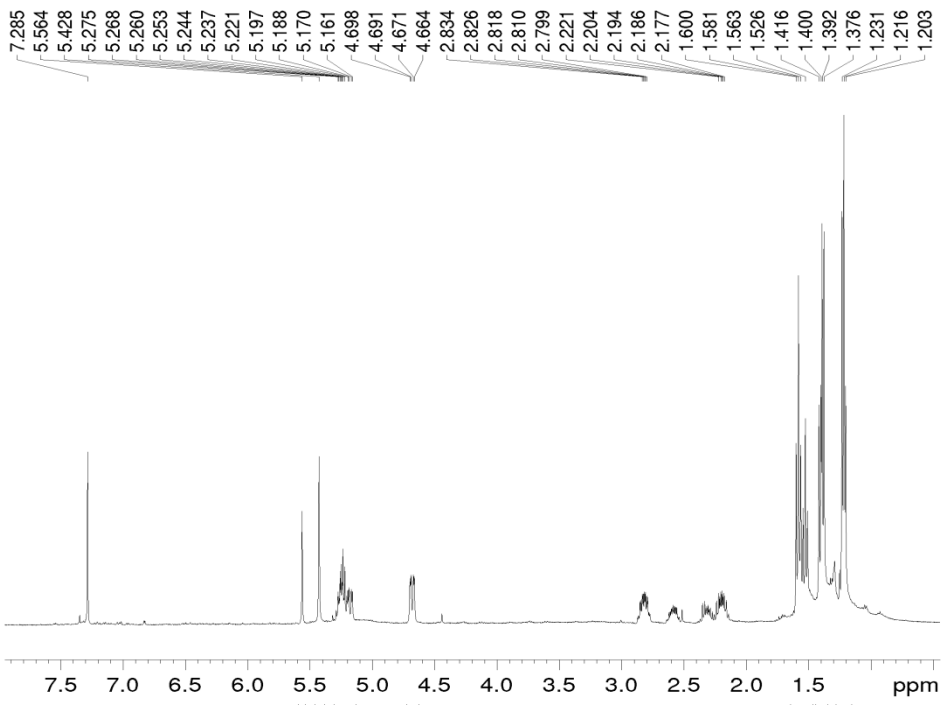

Figure 2. The ^{1}H NMR spectrum of compound **1** (500.17 MHz , solvent CS_2 : $CDCl_3$ = 5:1)

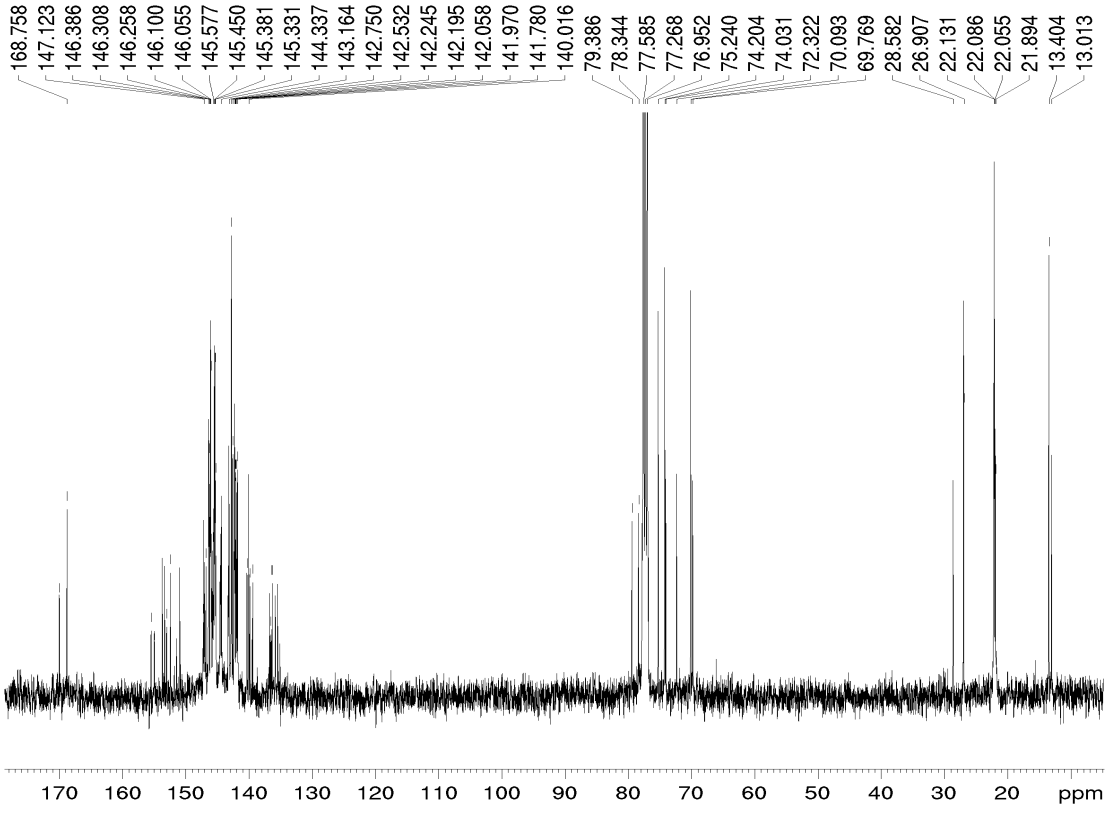

Figure 3. The ^{13}C NMR spectrum of compound **1** (125.78 MHz, solvent CS_2 : $CDCl_3$ = 5:1)

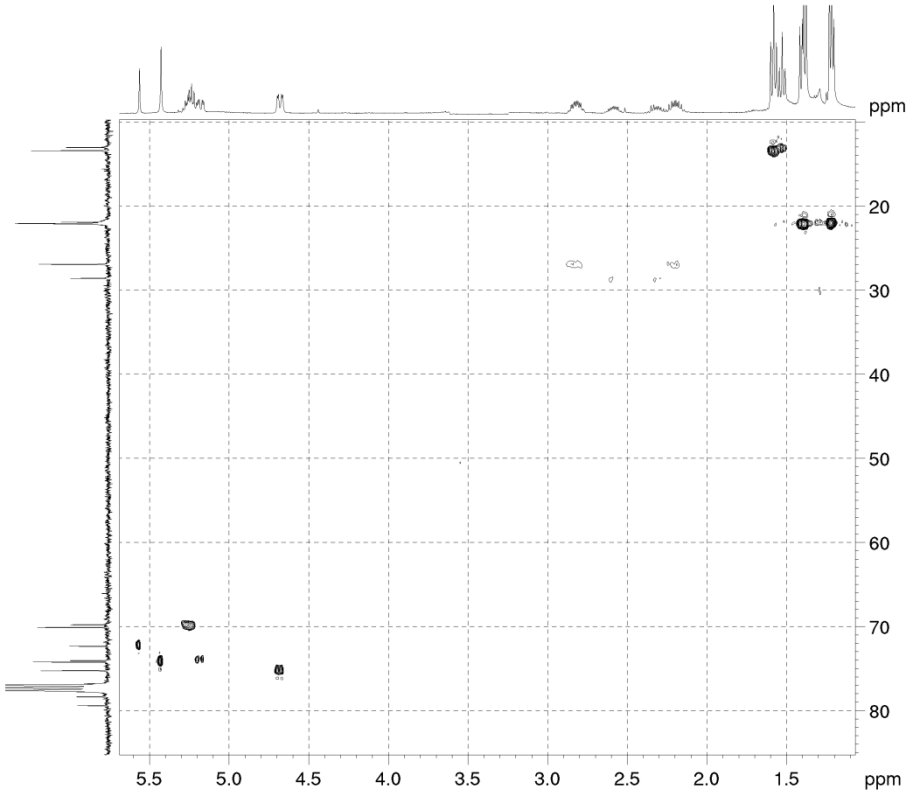

Figure 4. The HSQC spectrum of compound **1** (500.17 MHz for ^{1}H, 125.78 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

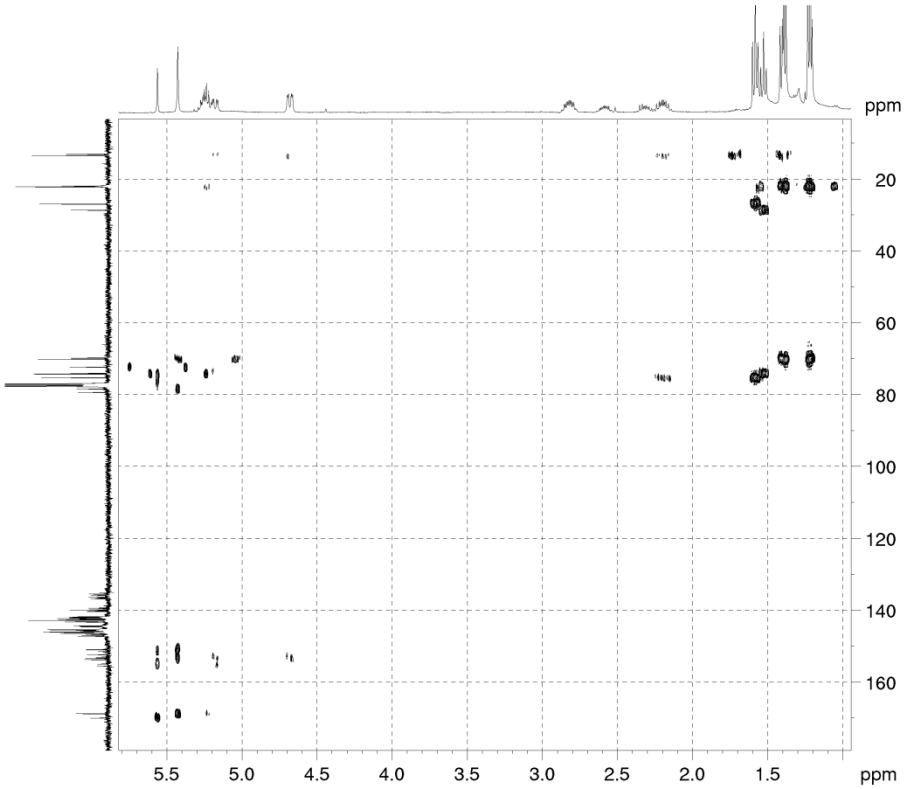

Figure 5. The HMBC spectrum of compound **1** (500.17 MHz for ^{1}H, 125.78 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

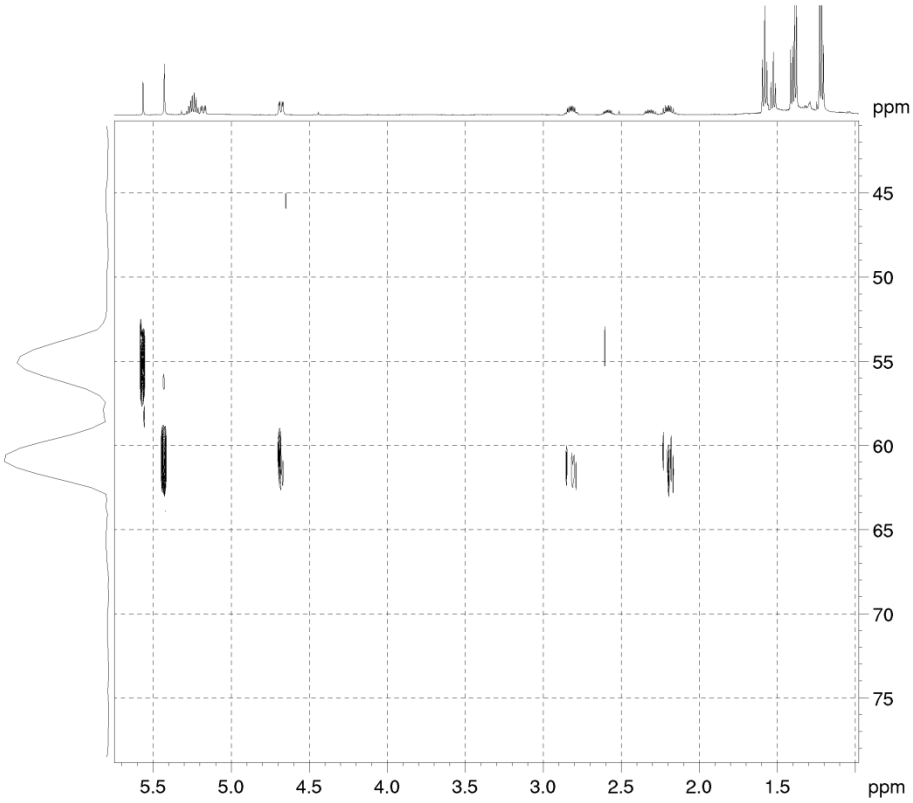

Figure 6. The ^{1}H-^{15}N HMBC spectrum of compound **1** (500.17 MHz for ^{1}H, 50.69 MHZ for ^{15}N, solvent CS_2 : $CDCl_3$ = 5:1)

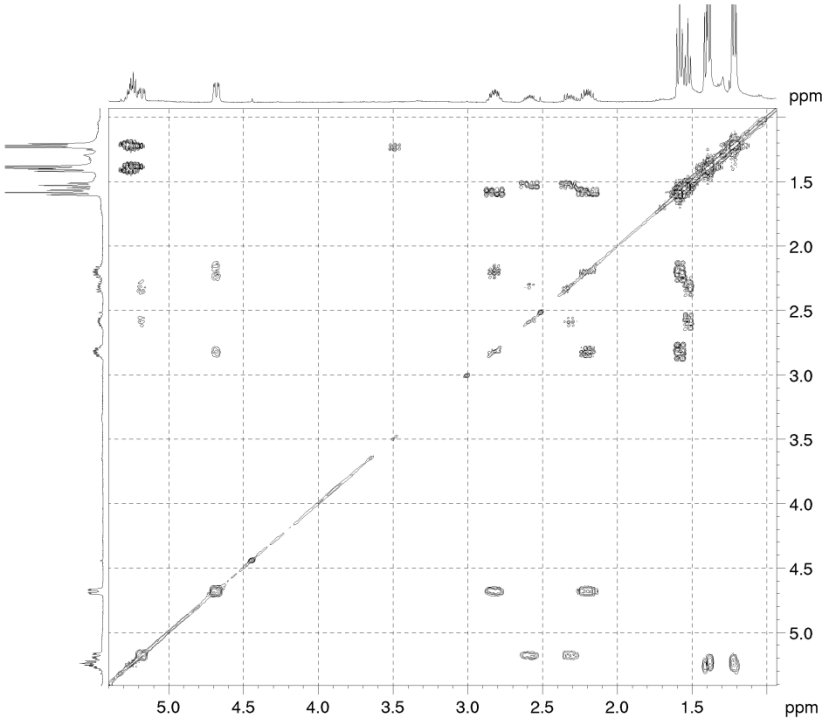

Figure 7. The H-H COSY spectrum of compound **1** (500.17 MHz for ^{1}H, solvent CS_2 : $CDCl_3$ = 5:1)

Mixture of isomers of *iso*-propyl (2-butyl-3,4-fullero[60]pyrrolidine-5-yl)-1*H*-carboxylates 2a+2b (cis/trans = 3:2). IR: 527, 1104, 1195, 1373, 1384, 1462, 1737, 3435 cm^{-1}. UV ($CHCl_3$), λ_{max}, nm: 258, 329, 429. ^1H NMR (500 MHz, $CDCl_3$): δ 1.09 (t, 3H, CH_3, Bu (*cis*-isomer), *J* = 7.0), 1.13 (t, 3H, CH_3, Bu (*trans*-isomer), *J* = 7.0), 1.20 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.21 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.37 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.40 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.64 (m, 2H, CH_2, Bu (*cis*-isomer)), 1.89 (m, 2H, CH_2, Bu (*cis*-isomer)), 2.91 (m, 2H, CH_2, Bu (*trans*-isomer)), 2.09 (m, 2H, CH_2, Bu (*trans*-isomer)), 2.18 and 2.73 (both m, 2H, CH_2, Bu (*cis*-isomer)), 2.28 and 2.53 (both m, 2H, CH_2, Bu (*trans*-isomer)), 4.78 (dd, 1H, CH, pyrrolidine (*cis*-isomer), *J* = 2.0, *J* = 7.0), 5.22 (dd, 1H, CH, pyrrolidine (*trans*-isomer), *J* = 2.0, *J* = 7.0), 5.24 (m, 1H, CH, *i*-Pr (*cis*-isomer)), 5.28 (m, 1H, CH, *i*-Pr (*trans*-isomer)), 5.43 (s, 1H, CH, pyrrolidine (*cis*-isomer)), 5.58 (s, 1H, CH, pyrrolidine (*trans*-isomer)). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.44, 14.53, 21.86, 22.03, 22.07, 22.12, 23.19, 23.37, 30.49, 30.86, 33.30, 34.88, 69.76, 70.11, 72.34, 72.41, 73.60, 74.22, 76.68, 77.73, 78.23, 79.45, 135.45, 135.84, 136.31, 136.71, 139.36, 139.80, 140.02, 140.29, 141.78, 141.87, 141.90, 141.96, 142.06, 142.15, 142.20, 142.25, 142.30, 142.35, 142.53, 142.74, 142.77, 142.80, 143.11, 143.16, 143.27, 144.29, 144.33, 144.45, 144.49, 144.54, 145.20, 145.24, 145.32, 145.38, 145.44, 145.50, 145.57, 145.71, 145.77, 145.80, 145.91, 145.98, 146.02, 146.05, 146.13, 146.25, 146.31, 146.34, 146.38, 146.77, 147.03, 147.12, 147.24, 150.96, 151.53, 152.40, 153.00, 153.32, 153.68, 155.07, 155.36, 168.81, 170.10. MALDI TOF: *m/z* found 903.085 $[M-2H]^-$, calc. 905.905 ($C_{70}H_{19}NO_2$).

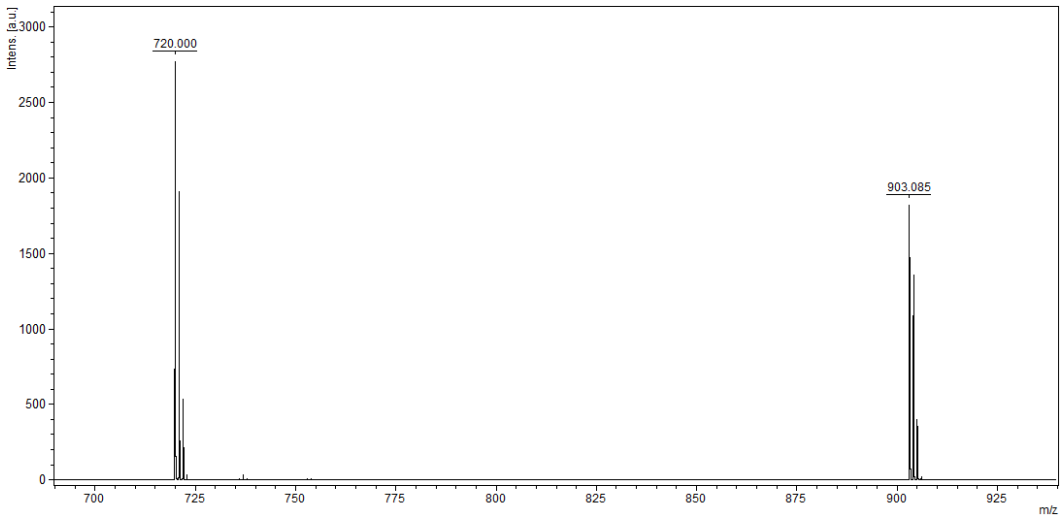

Figure 8. Mass spectra MALDI TOF of compound **2** (matrix – S_8).

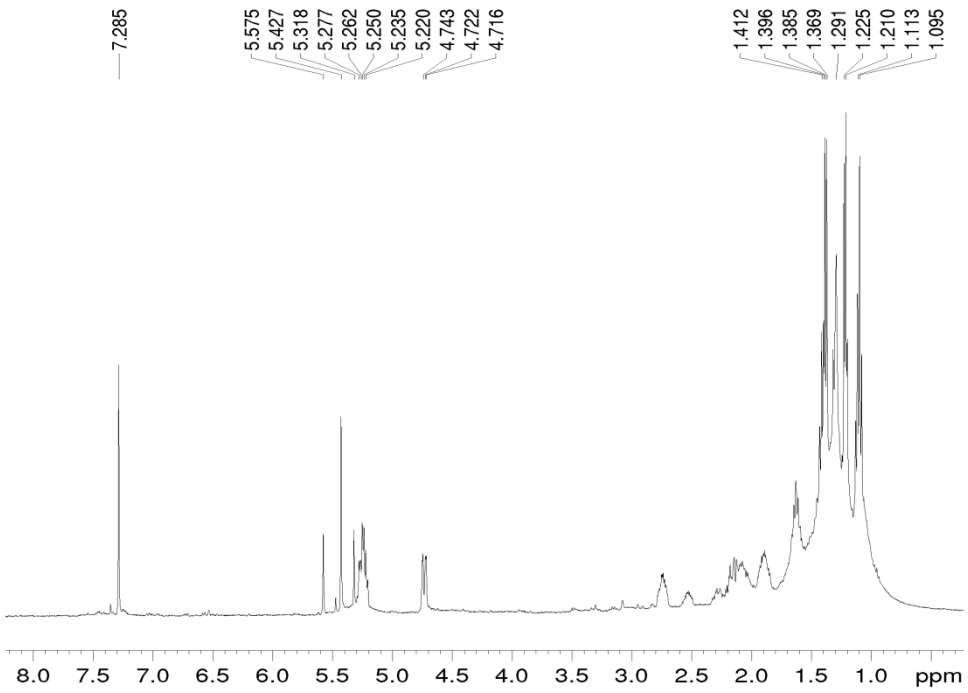

Figure 9. The ^{1}H NMR spectrum of compound **2** (500.17 MHz , solvent CS_2 : $CDCl_3$ = 5:1)

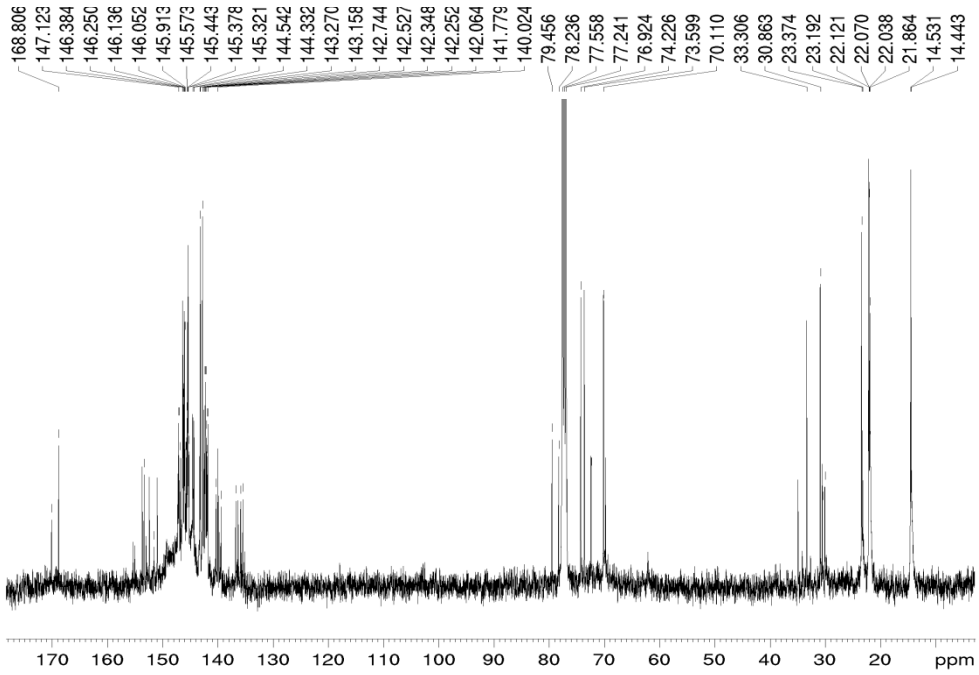

Figure 10. The ^{13}C NMR spectrum of compound **2** (125.78 MHz, solvent CS_2 : $CDCl_3$ = 5:1)

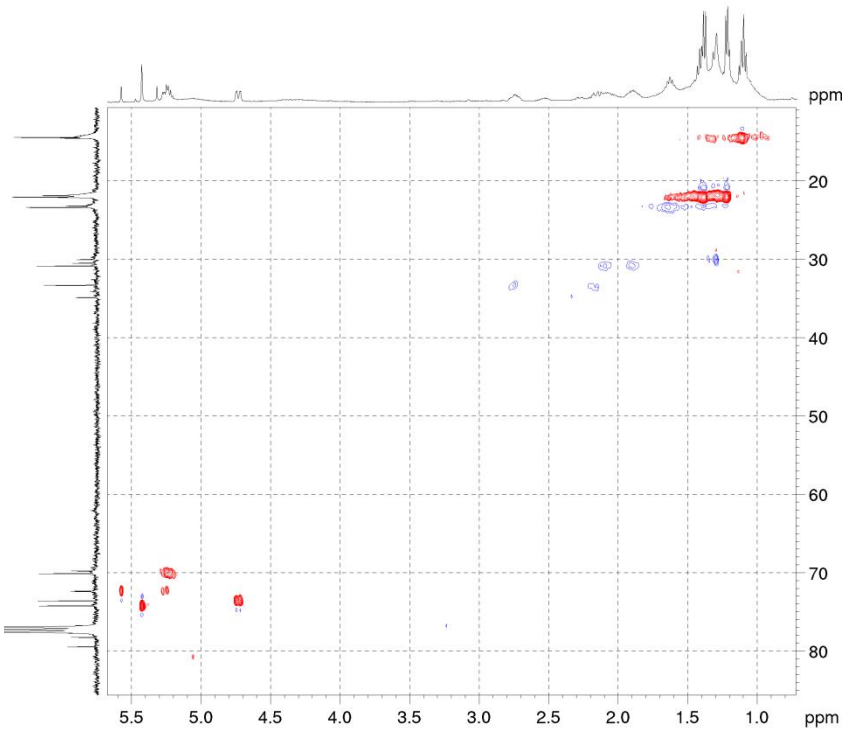

Figure 11. The HSQC spectrum of compound **2** (500.17 MHz for ^{1}H, 125.78 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

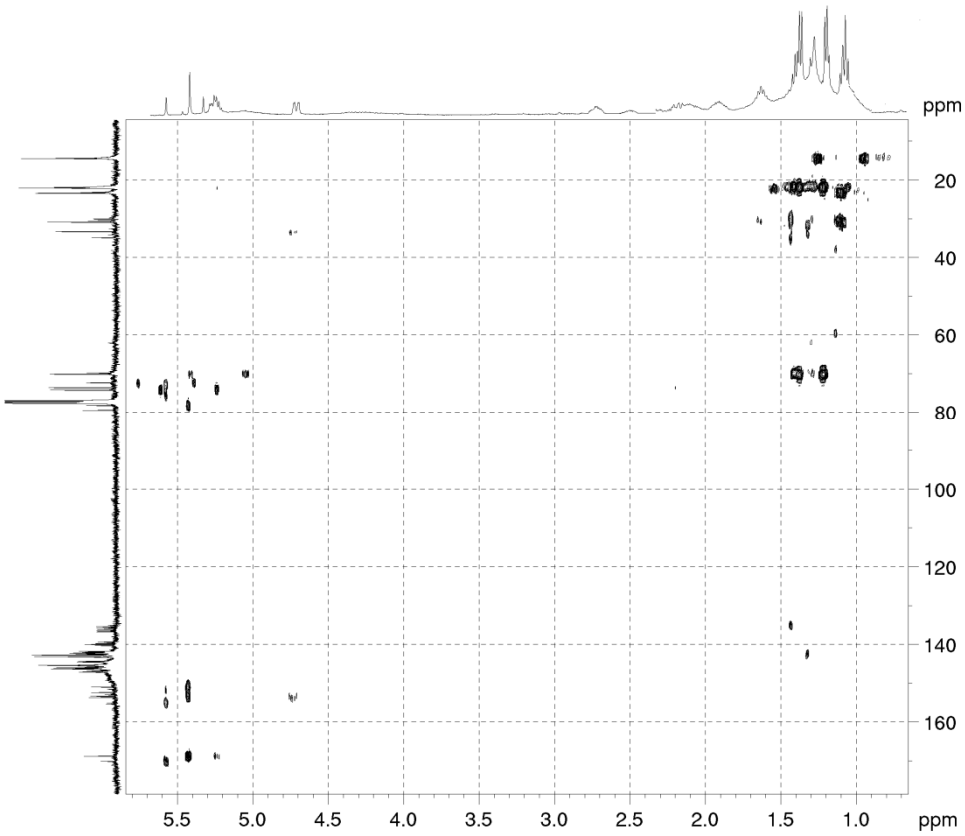

Figure 12. The HMBC spectrum of compound **2** (500.17 MHz for ^{1}H, 125.78 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

Mixture of isomers of *iso*-propyl (2-*iso*-propyl-3,4-fullero[60]pyrrolidine-5-yl)-1*H*-carboxylates 3a+3b (cis/trans = 1:1). IR: 527, 731, 1103, 1187, 1204, 1372, 1384, 1428, 1462, 1736, 3441 cm^{-1}. UV ($CHCl_3$), λ_{max}, nm: 257, 308, 429. ^1H NMR (500 MHz, $CDCl_3$): δ 1.19 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.22 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.38 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.41 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.49 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.53 (d, 3H, CH_3, i-Pr (*ciss*-isomer), *J* = 7.0), 1.60 (d, 6H, $2CH_3$, i-Pr (*trans* and *cis*-isomers), *J* = 7.0), 2.89 (m, H, CH, i-Pr (*cis*-isomer)), 2.99 (m, H, CH, i-Pr (*trans*-isomer)), 4.62 (d, 1H, CH, pyrrolidine (*cis*-isomer), *J* = 3.6), 5.06 (d, 1H, CH, pyrrolidine (*trans*-isomer), *J* = 5.2), 5.21 (m, 2H, 2CH, *i*-Pr (*cis*+*trans*-isomer)), 5.35 (s, 1H, CH, pyrrolidine (*cis*-isomer)), 5.52 (s, 1H, CH, pyrrolidine (*trans*-isomer)). ^{13}C NMR (125 MHz, $CDCl_3$): δ 19.46, 19.49, 21.94, 22.09, 22.15, 22.18, 23.74, 24.06, 29.58, 32.32, 69.71, 69.98, 73.00, 73.96, 78.34, 79.40, 77.12, 77.69, 78.68, 134.64, 135.41, 135.61, 135.83, 136.35, 136.44, 136.74, 139.38, 139.43, 139.81, 139.93, 140.06, 140.13, 141.74, 141.78, 141.84, 141.88, 141.91, 142.00, 142.21, 142.24, 142.27, 142.30, 142.38, 142.51, 142.55, 142.71, 142.76, 142.81, 143.15, 143.18, 143.26, 143.34, 144.31, 144.33, 144.41, 144.51, 144.55, 144.60, 145.22, 145.33, 145.36, 145.40, 145.44, 145.50, 145.52, 145.57, 145.68, 145.75, 145.86, 145.93, 146.03, 146.27, 146.31, 146.35, 146.40, 146.65, 146.98, 147.06, 147.08, 147.18, 151.06, 151.67, 152.42, 153.09, 153.77, 154.10, 155.01, 156.20, 168.61, 169.74. MALDI TOF, *m/z* 891.1 $[M]^-$.

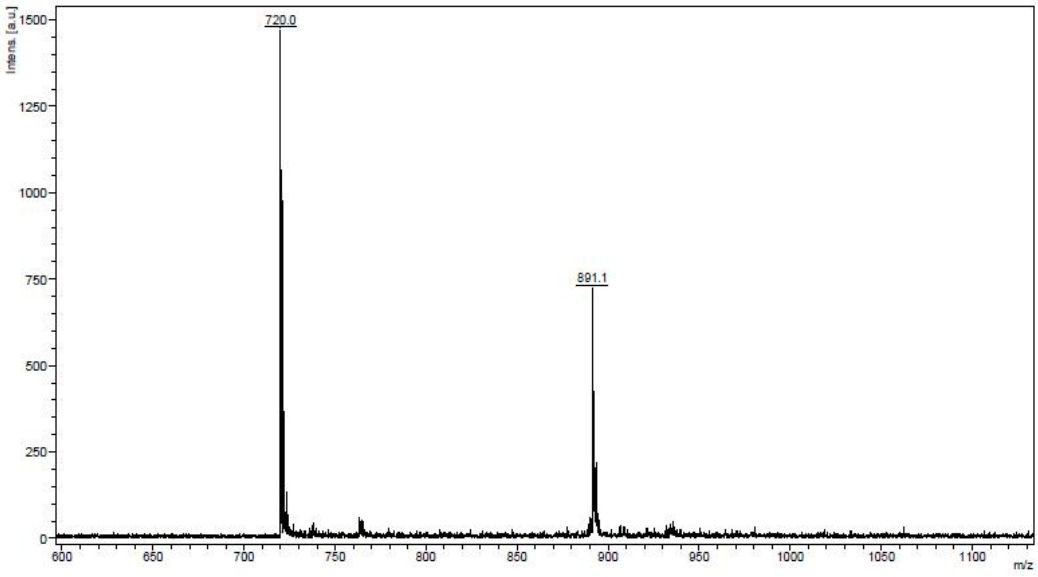

Figure 13. Mass spectra MALDI TOF/TOF of compound **3** (matrix – DCTB).

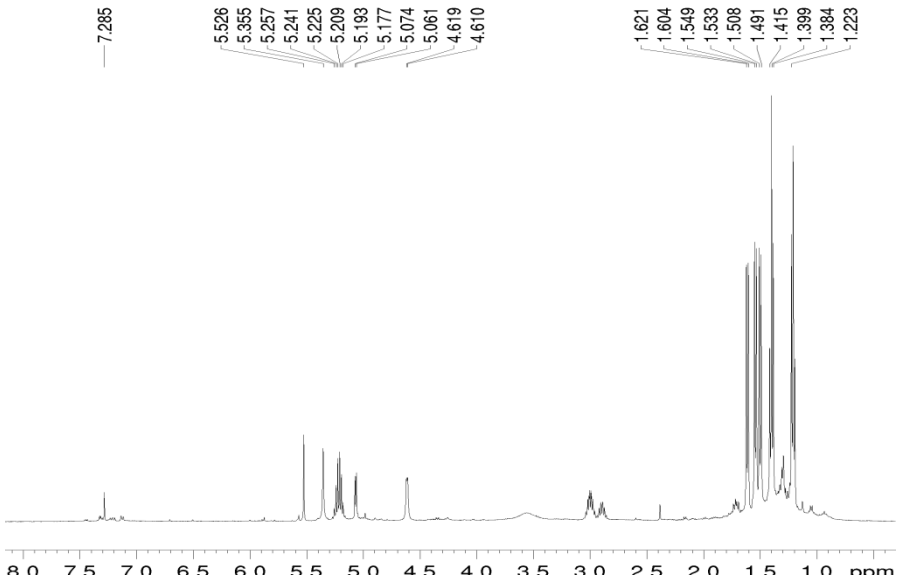

Figure 14. The ^{1}H NMR spectrum of compound **3** (400.13 MHz , solvent CS_2 : $CDCl_3$ = 5:1)

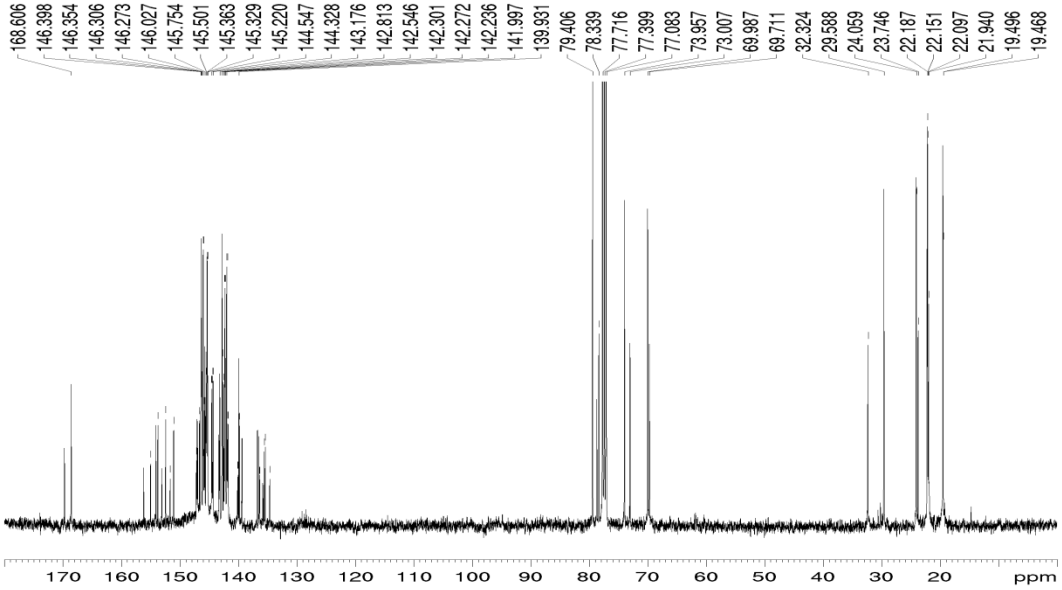

Figure 15. The ^{13}C NMR spectrum of compound **3** (100.62 MHz, solvent CS_2 : $CDCl_3$ = 5:1)

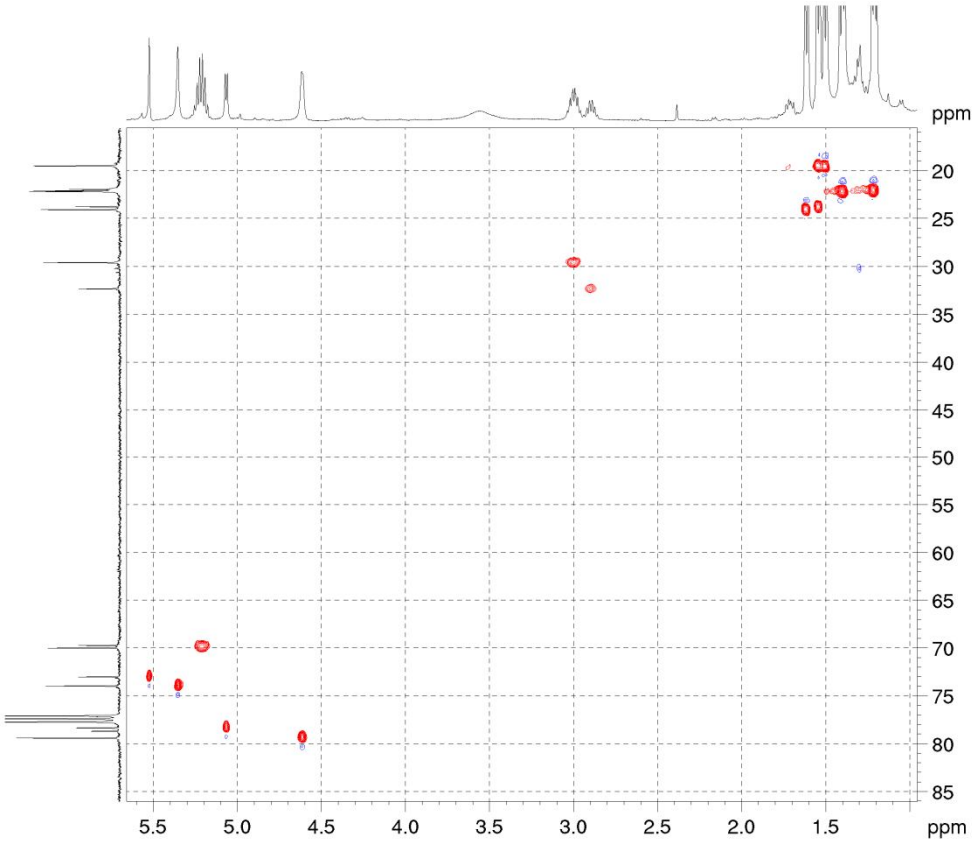

Figure 16. The HSQC spectrum of compound **3** (400.13 MHz for ^{1}H, 100.62 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

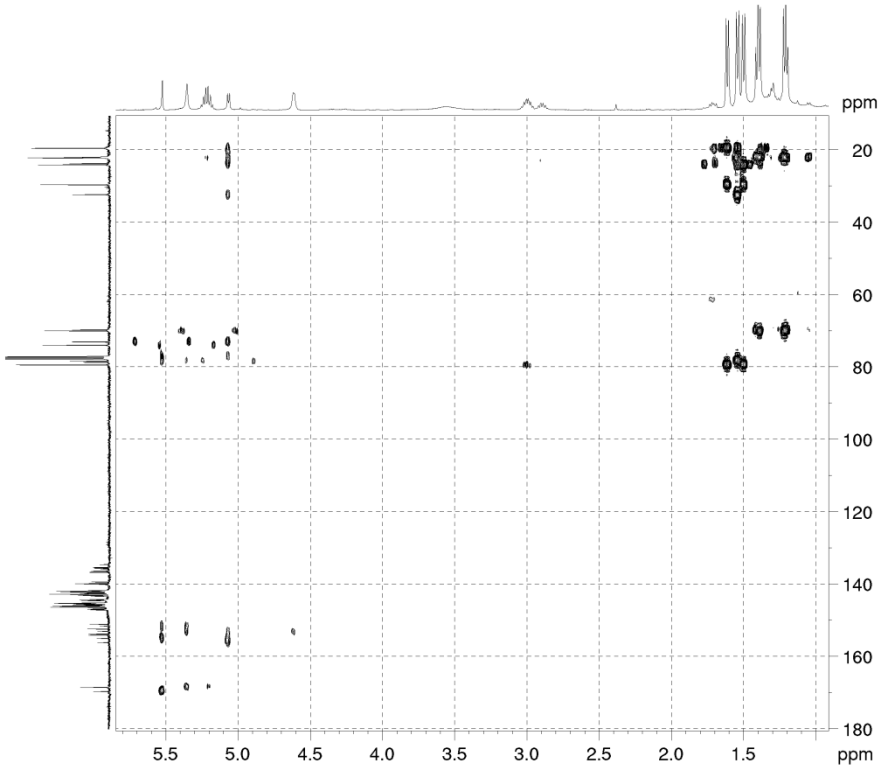

Figure 17. The HMBC spectrum of compound **3** (400.13 MHz for ^{1}H, 100.62 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

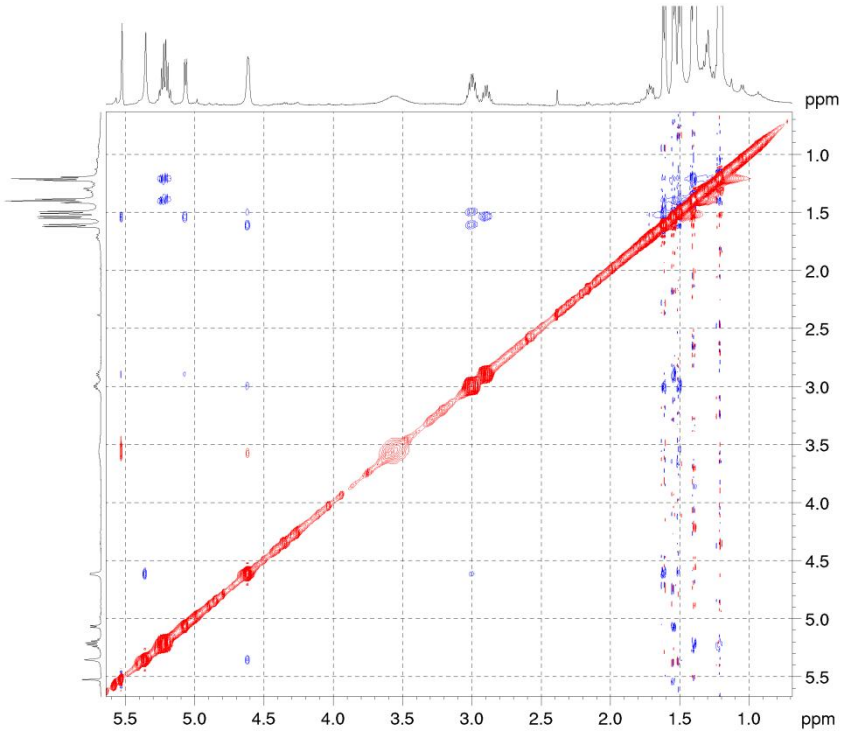

Figure 18. The NOESY spectrum of compound **3** (400.13 MHz for ^{1}H, solvent CS_2 : $CDCl_3$ = 5:1)

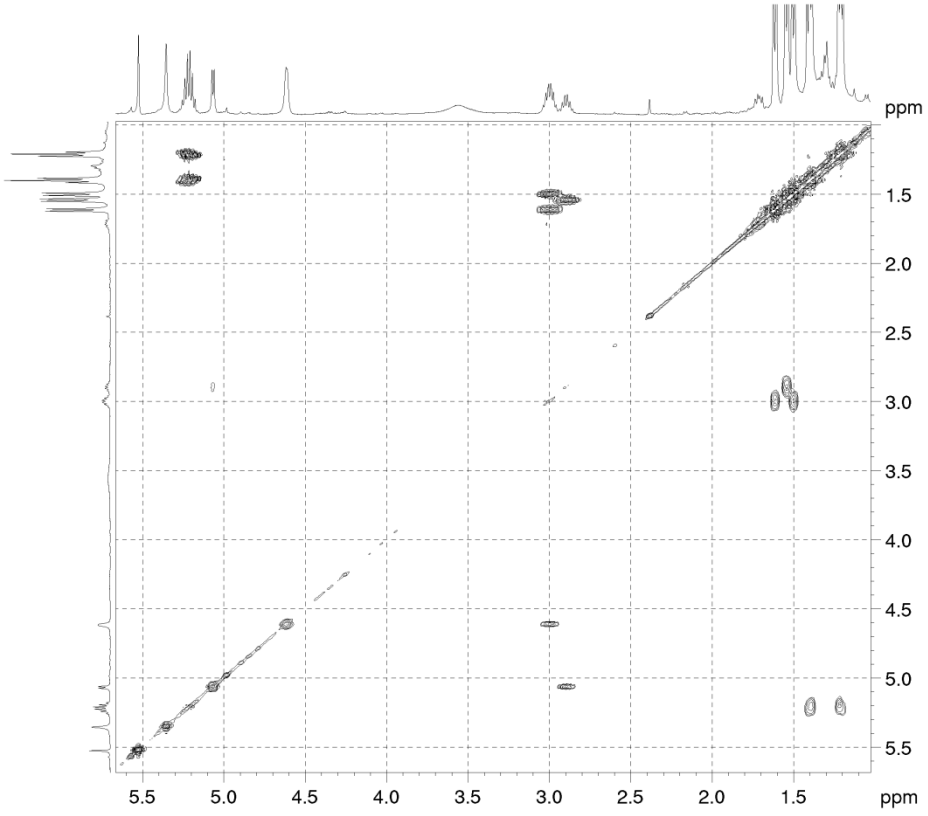

Figure 19. The H-H COSY spectrum of compound **3** (400.13 MHz for ^{1}H, solvent CS_2 : $CDCl_3$ = 5:1)

Mixture of isomers of *iso*-propyl (2-*cyclo*-hexyl-3,4-fullero[60]pyrrolidine-5-yl)-1*H*-carboxylates 4a+4b (cis/trans = 10:8). IR: 527, 732, 1102, 1280, 1480, 1729, 3435 cm^{-1}. UV ($CHCl_3$), λ_{max}, nm: 258, 330, 429. ^1H NMR (500 MHz, $CDCl_3$): δ 1.20 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.24 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.37 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.40 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.5-2.4 (m, 22H, 10CH_2, 2CH, 2Cy), 4.59 (d, 1H, CH, pyrrolidine (*cis*-isomer), *J* = 3.6), 5.05 (d, 1H, CH, pyrrolidine (*trans*-isomer), *J* = 3.6), 5.21-5.27 (m, 2H, 2CH, *i*-Pr (*cis*+*trans*-isomer)), 5.37 (s, 1H, CH, pyrrolidine (*cis*-isomer)), 5.54 (s, 1H, CH, pyrrolidine (*trans*-isomer)). ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.14, 21.75, 21.78, 21.87, 21.99, 22.04, 22.06, 22.13, 26.17, 26.53, 26.67, 26.74, 26.83, 26.87, 29.61, 30.02, 69.85, 70.11, 73.12, 73.43, 73.98, 75.66, 77.80, 78.51, 78.79, 86.07, 140.36, 140.69, 141.06, 141.75, 141.80, 141.83, 141.87, 141.97, 141.99, 142.05, 142.11, 142.18, 142.25, 142.29, 142.40, 142.44, 142.51, 142.68, 142.78, 142.80, 142.88, 143.16, 143.22, 143.27, 143.33, 144.31, 144.33, 144.39, 144.50, 144.53, 144.59, 145.21, 145.26, 145.43, 145.45, 145.51, 145.74, 14.02, 146.25, 146.30, 146.33, 146.40, 147.01, 147.08, 147.16, 150.59, 151.06, 152.32, 153.71, 154.02, 154.34, 154.93, 155.49, 168.92, 169.06.

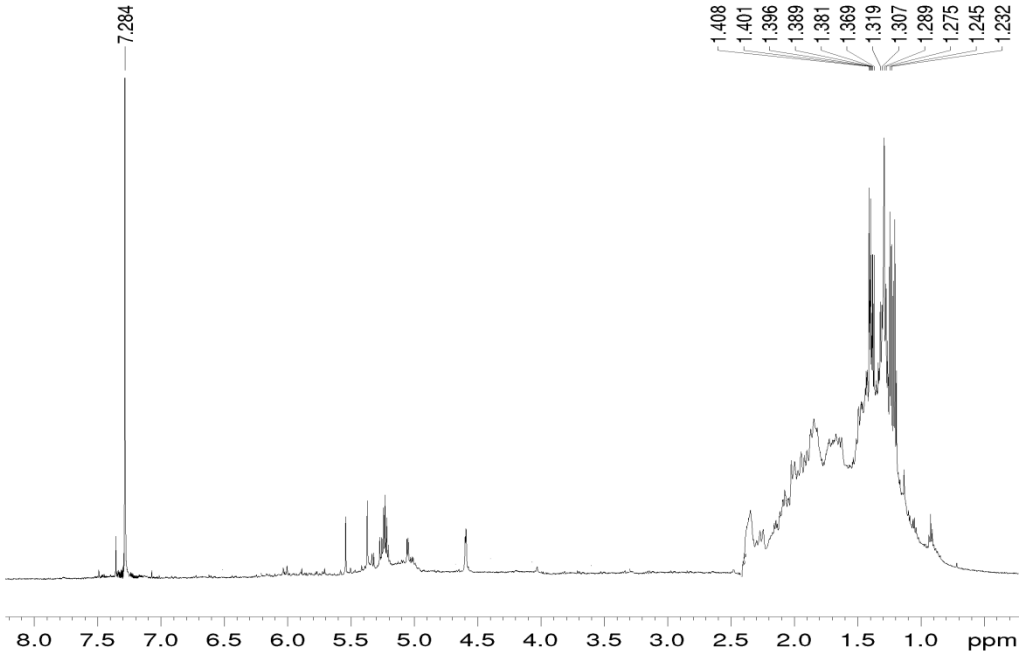

Figure 20. The ^1H NMR spectrum of compound **4** (500.17 MHz , solvent CS_2 : $CDCl_3$ = 5:1)

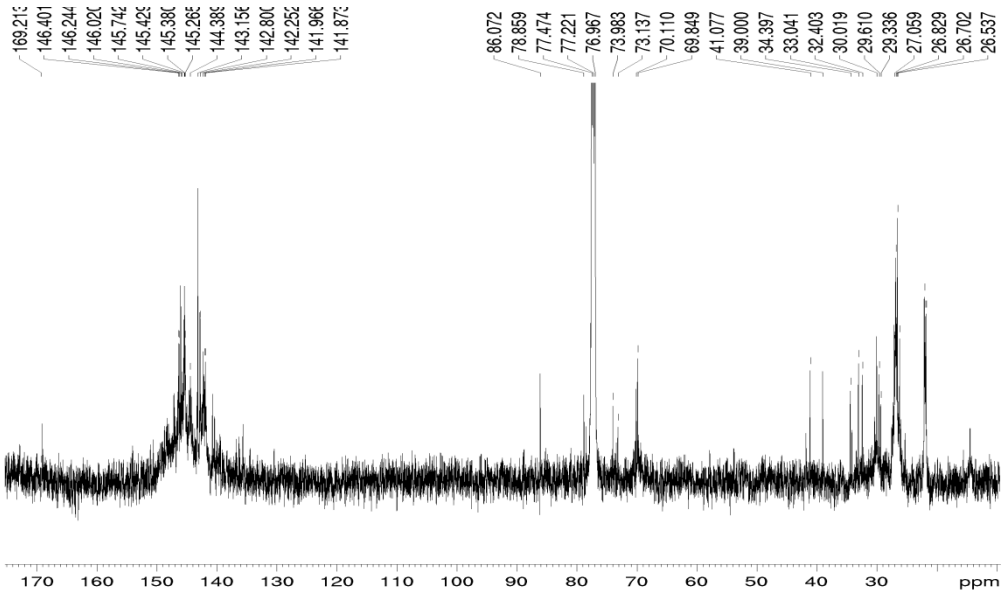

Figure 21. The ^{13}C NMR spectrum of compound **4** (125.78 MHz, solvent CS_2 : $CDCl_3$ = 5:1)

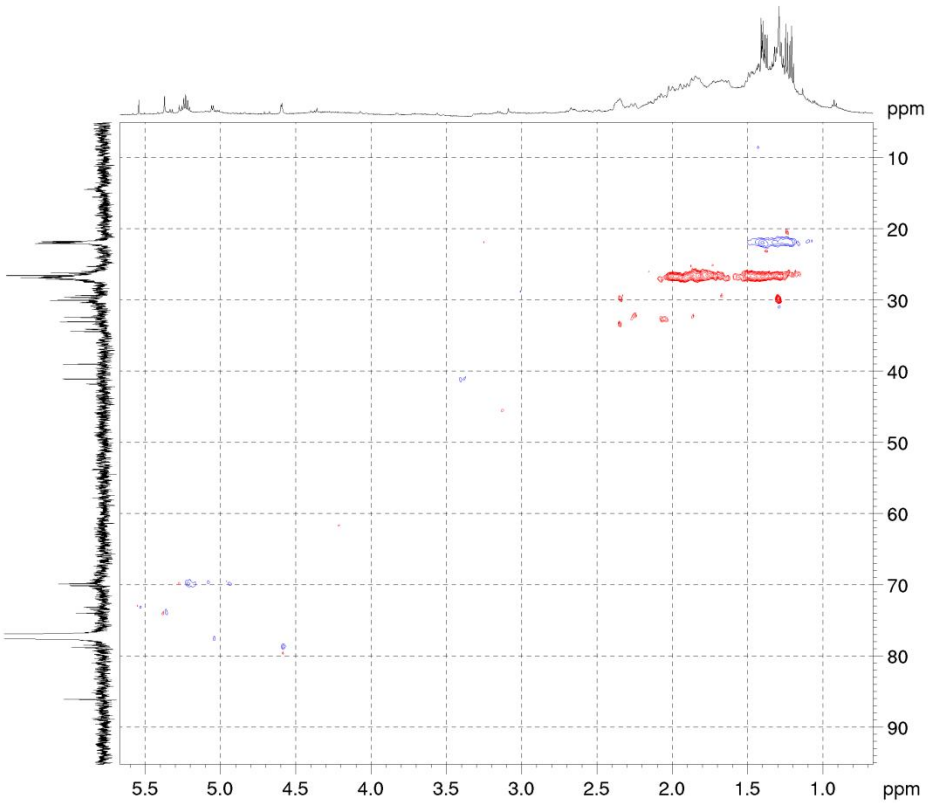

Figure 22. The HSQC spectrum of compound **4** (500.17 MHz for ^{1}H, 125.78 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

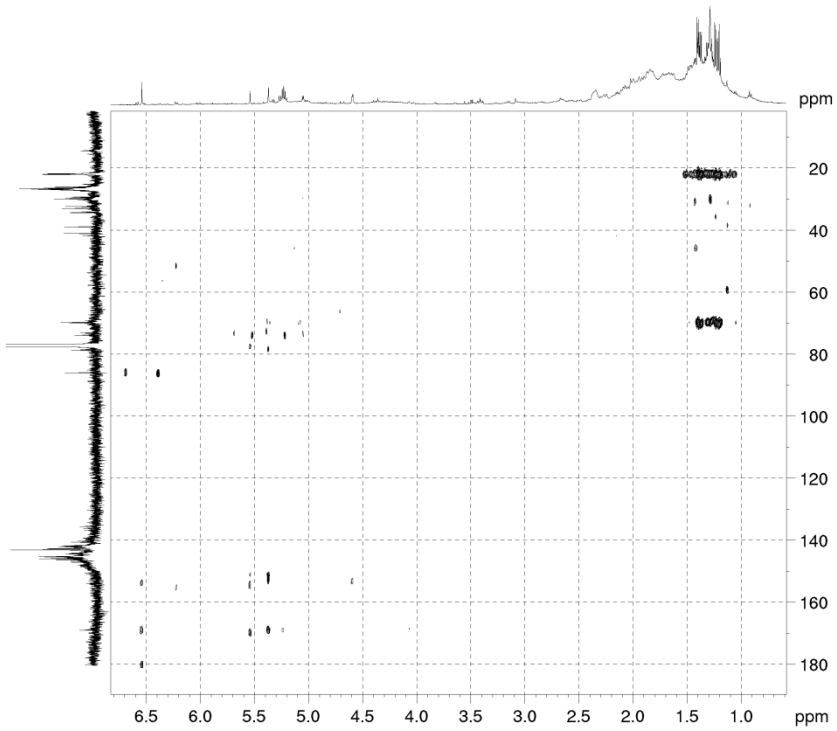

Figure 23. The HMBC spectrum of compound **4** (500.17 MHz for ^{1}H, 125.78 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

trans *iso*-Propyl (2-phenyl-3,4-fullero[60]pyrrolidine-5-yl)-1*H*-carboxylates 5. IR: 527, 732, 1102, 1182, 1261, 1429, 1461, 1731, 3445 cm^{-1}. UV ($CHCl_3$), λ_{max}, nm: 254, 329, 429. ^1H NMR (500 MHz, $CDCl_3$): δ 1.23 (d, 3H, CH_3, i-Pr, J = 7.0), 1.45 (d, 3H, CH_3, i-Pr, J = 7.0), 1.71 (broad s, 1H, NH), 5.34 (m, 1H, CH, *i*-Pr), 5.74 (s, 1H, CH, pyrrolidine), 6.54 (s, 1H, CH, pyrrolidine), 7.35 (d, 1H, CH, Ph, J = 7.0), 7.43 (t, 2H, 2CH, Ph, J = 7.0), 7.87 (d, 2H, 2CH, Ph, J = 7.0). ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.75, 22.23, 69.68, 70.72, 73.26, 74.24, 75.87, 128.54, 128.62, 128.73, 135.69, 135.83, 136.37, 136.49, 138.15, 139.58, 139.70, 140.08, 140.14, 141.64, 141.79, 141.92, 142.06, 142.13, 142.15, 142.22, 142.30, 142.36, 142.68, 142.73, 142.80, 143.14, 143.18, 144.33, 144.57, 144.67, 144.92, 145.25, 145.34, 145.40, 145.51, 145.55, 145.59, 145.72, 146.01, 146.04, 146.12, 146.15, 146.26, 146.29, 146.42, 146.45, 146.55, 146.78, 147.25, 147.44, 151.77, 153.16, 154.01, 156.14, 171.83. MALDI TOF, *m/z* 925.1 $[M]^-$.

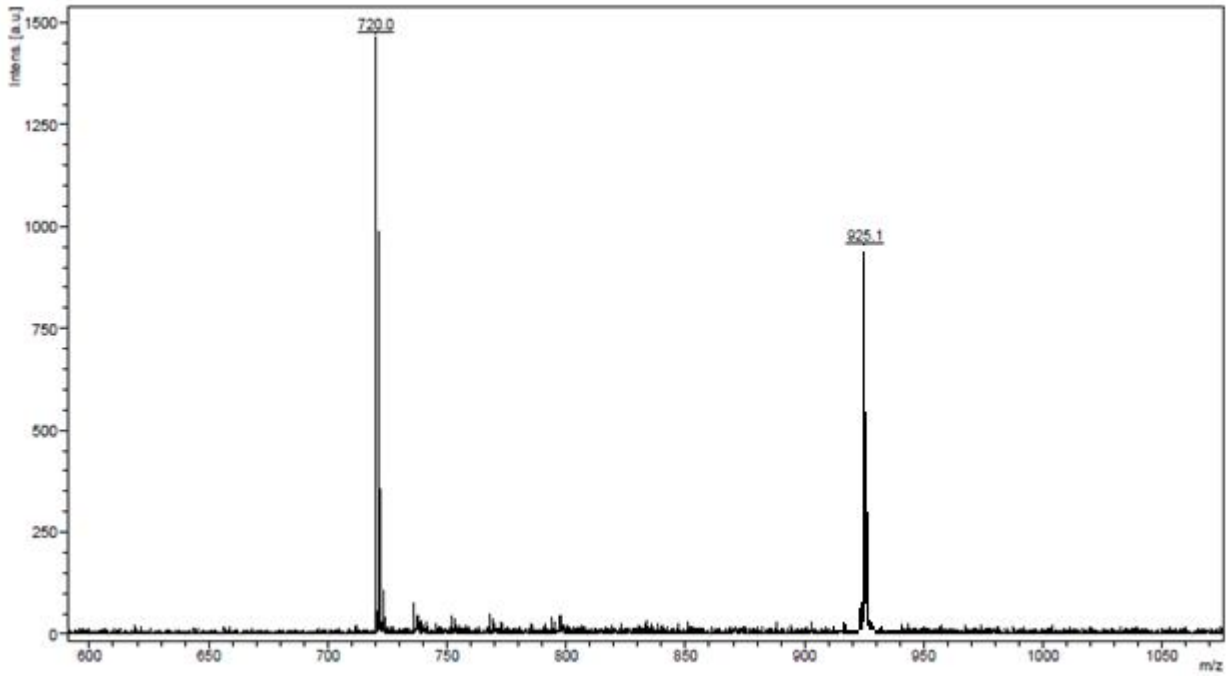

Figure 24. Mass spectra MALDI TOF/TOF of compound **5** (matrix – DCTB).

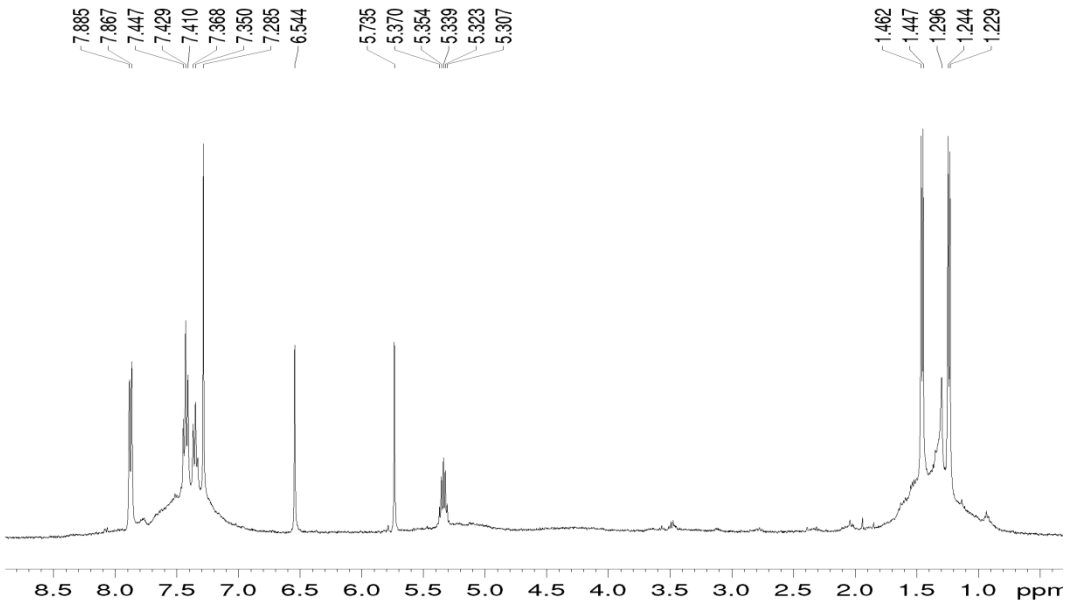

Figure 25. The ^{1}H NMR spectrum of compound **5** (400.13 MHz , solvent CS_2 : $CDCl_3$ = 5:1)

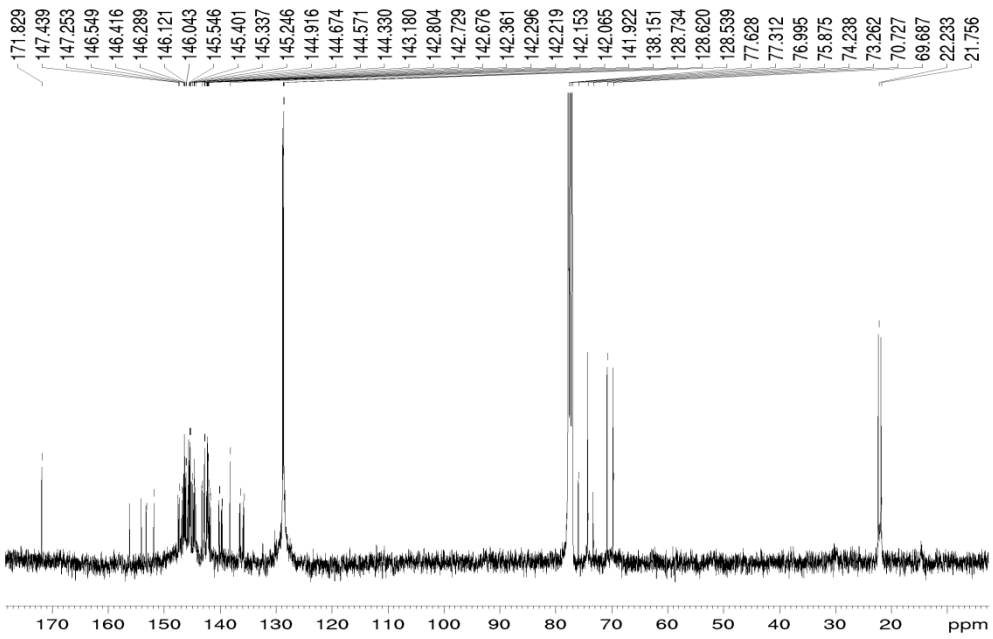

Figure 26. The ^{13}C NMR spectrum of compound **5** (100.62 MHz, solvent CS_2 : $CDCl_3$ = 5:1)

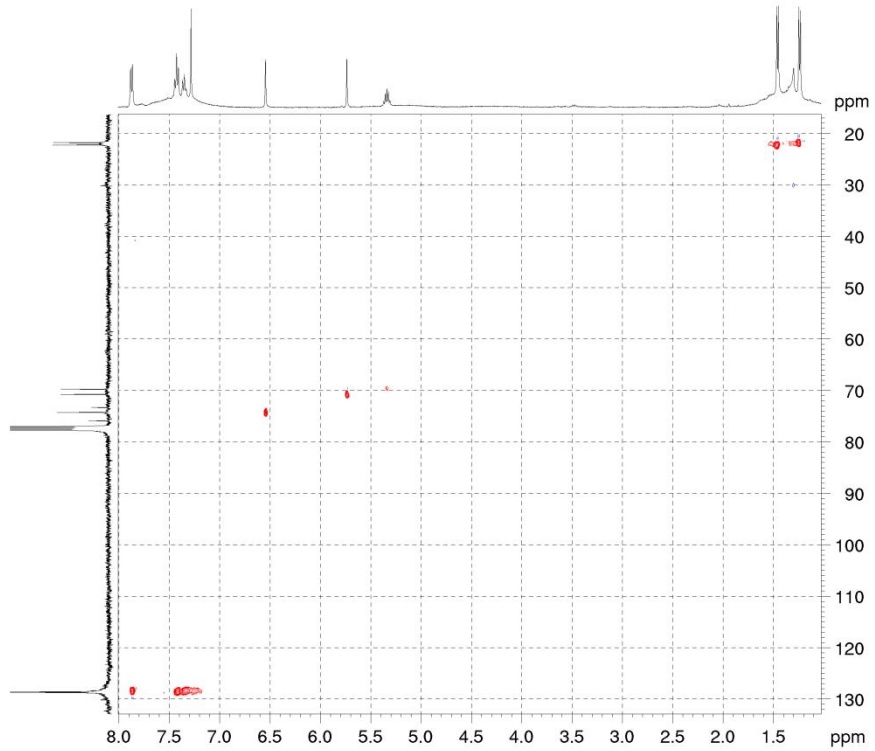

Figure 27. The HSQC spectrum of compound **5** (400.13 MHz for ^{1}H, 100.62 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

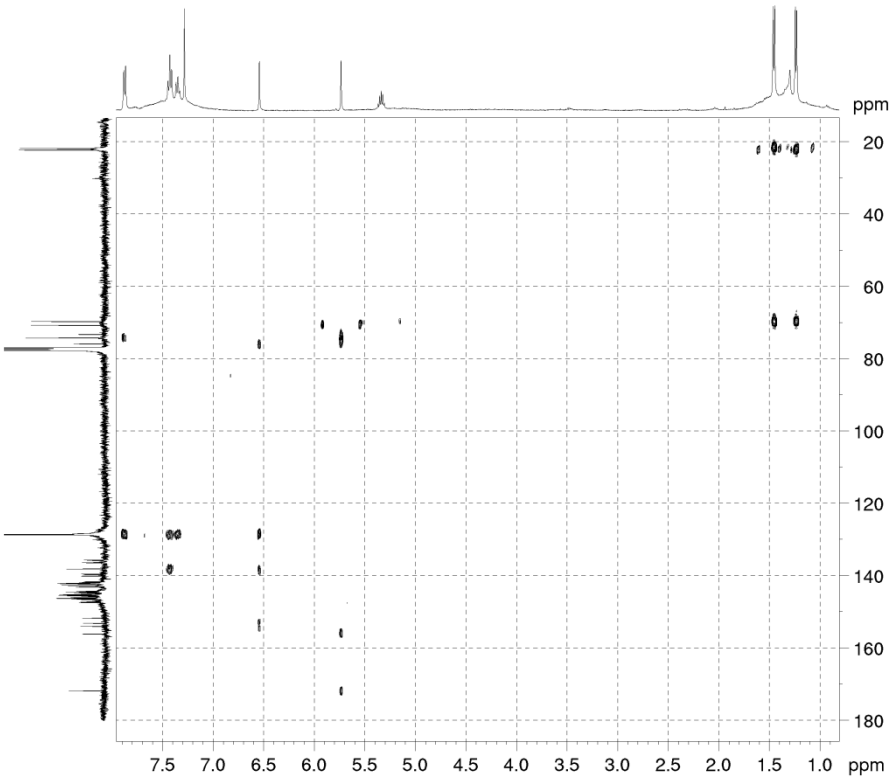

Figure 28. The HMBC spectrum of compound **5** (400.13 MHz for ^{1}H, 100.62 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

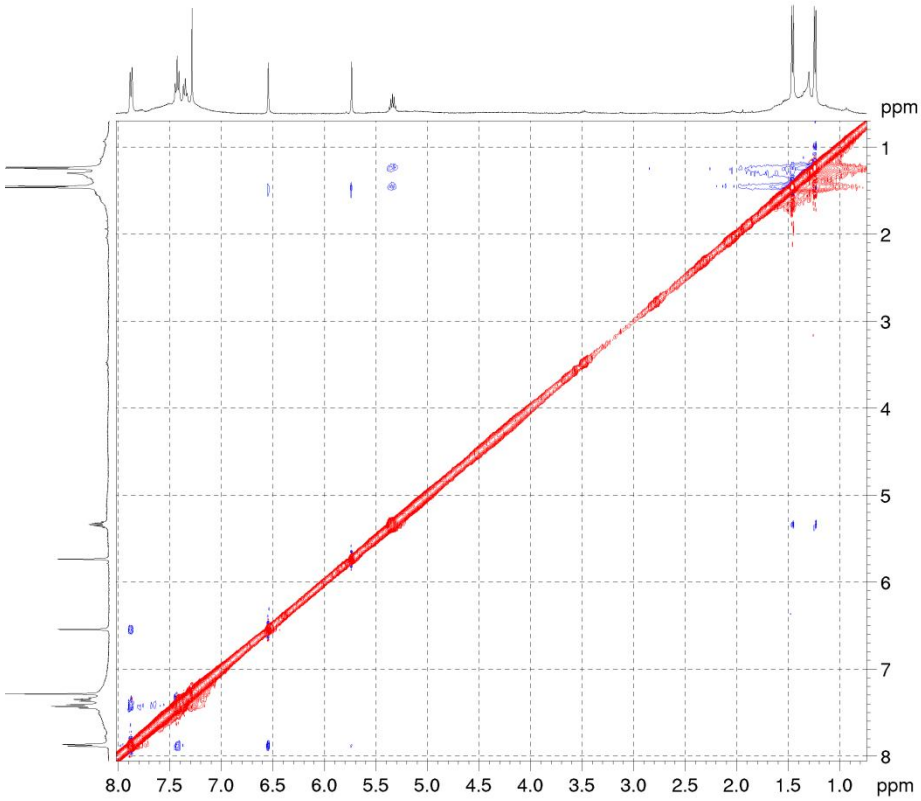

Figure 29. The NOESY spectrum of compound **5** (400.13 MHz for ^{1}H, solvent CS_2 : $CDCl_3$ = 5:1)

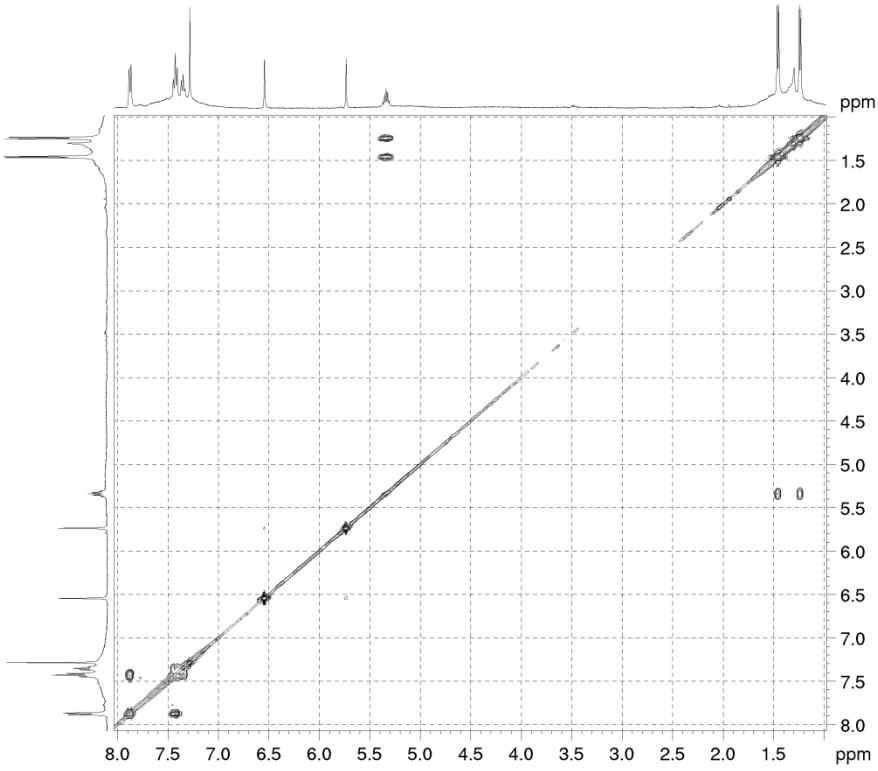

Figure 30. The H-H COSY spectrum of compound **5** (400.13 MHz for ^{1}H, solvent CS_2 : $CDCl_3$ = 5:1)

Mixture of isomers of *iso*-propyl (2-(*o*-tolyl)-3,4-fullero[60]pyrrolidine-5-yl)-1*H*-carboxylates 6a+6b (cis/trans = 1:2). IR: 527, 731, 758, 1103, 1183, 1202, 1373, 1429, 1461, 1488, 1735, 3438 cm^{-1}. UV ($CHCl_3$), λ_{max}, nm: 257, 331, 429. ^1H NMR (500 MHz, $CDCl_3$): δ 1.24 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 1.25 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.42 (d, 3H, CH_3, i-Pr (*cis*-isomer), *J* = 7.0), 1.46 (d, 3H, CH_3, i-Pr (*trans*-isomer), *J* = 7.0), 2.64 (s, 3H, CH_3, *trans*-isomer), 2.65 (s, 3H, CH_3, *cis*-isomer), 5.04 (dd, 1H, CH, pyrrolidine (*cis*-isomer), *J* = 2.0, *J* = 7.0), 5.11 (dd, 1H, CH, pyrrolidine (*trans*-isomer), *J* = 2.0, *J* = 7.0), 5.27 (m, 1H, 1CH, *i*-Pr (*cis*-isomer)), 5.32 (m, 1H, 1CH, *i*-Pr (*cis*-isomer)), 5.59 (s, 1H, CH, pyrrolidine (*cis*-isomer)), 5.72 (s, 1H, CH, pyrrolidine (*trans*-isomer)), 7.00-8.25 (m, 8H, 8CH, 2Ph). ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.78, 21.91, 21.97, 22.07, 22.13, 22.25, 69.61, 69.64, 70.13, 70.49, 71.79, 73.25, 73.66, 75.88, 76.42, 79.62, 126.58, 126.83, 127.69, 127.85, 128.26, 128.45, 128.81, 130.46, 130.60, 130.71, 131.01, 131.36, 136.29, 136.44, 136.58, 136.99, 139.68, 139.78, 139.84, 140.00, 140.07, 140.16, 141.66, 141.78, 141.98, 142.11, 142.14, 142.20, 142.32, 142.68, 142.73, 143.06, 143.17, 144.16, 144.28, 144.32, 144.40, 144.43, 144.53, 144.57, 144.63, 144.90, 145.21, 145.24, 145.26, 145.32, 145.41, 145.44, 145.49, 145.60, 145.65, 145.71, 145.74, 145.98, 146.03, 146.10, 146.15, 146.21, 146.23, 146.28, 146.31, 146.40, 146.92, 147.03, 147.06, 147.17, 147.24, 147.42, 151.18, 151.76, 152.87, 153.27, 153.65, 154.21, 156.44, 168.64, 171.94.

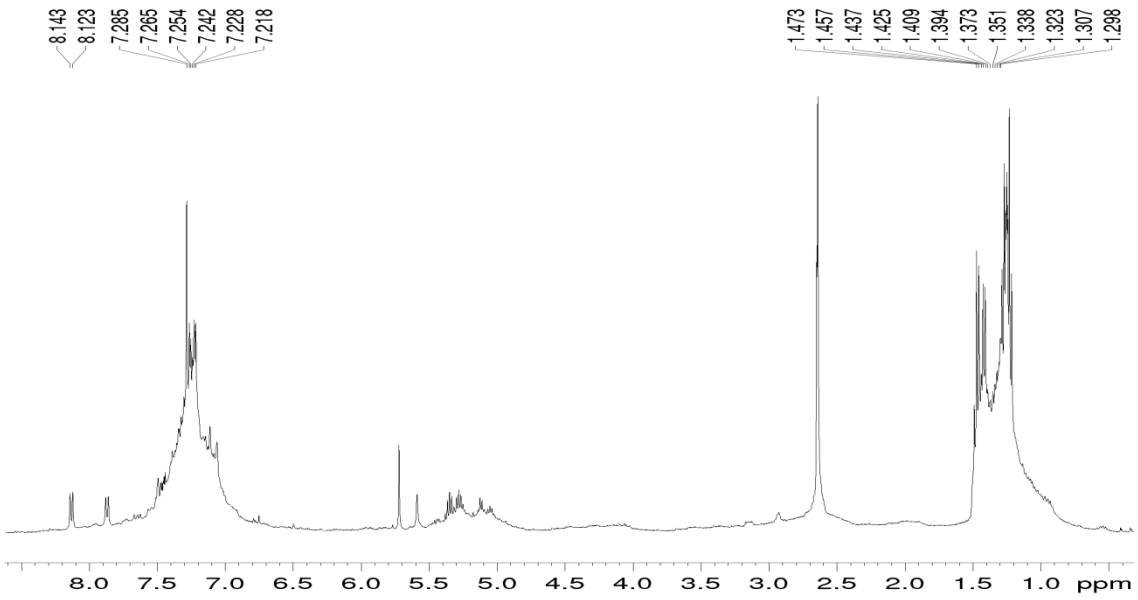

Figure 31. The ^1H NMR spectrum of compound **6** (400.13 MHz , solvent CS_2 : $CDCl_3$ = 5:1)

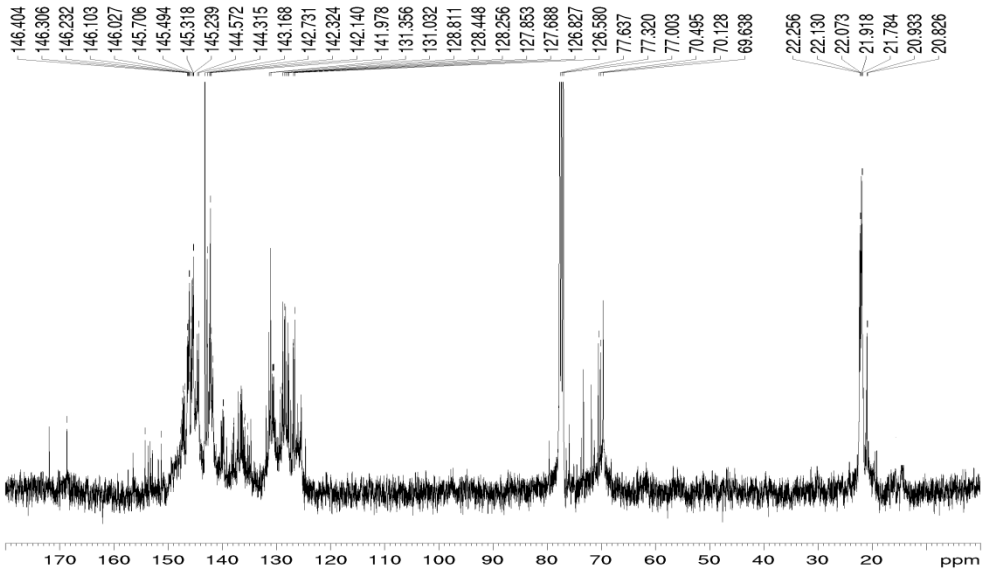

Figure 32. The ^{13}C NMR spectrum of compound **6** (100.62 MHz, solvent CS_2 : $CDCl_3$ = 5:1)

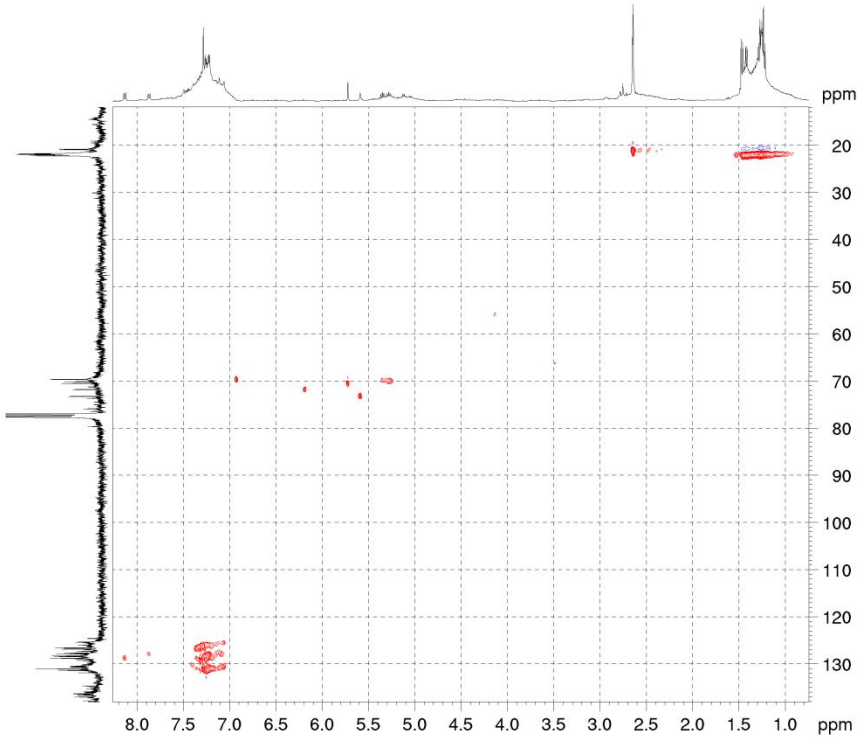

Figure 33. The HSQC spectrum of compound **6** (400.13 MHz for ^{1}H, 100.62 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)

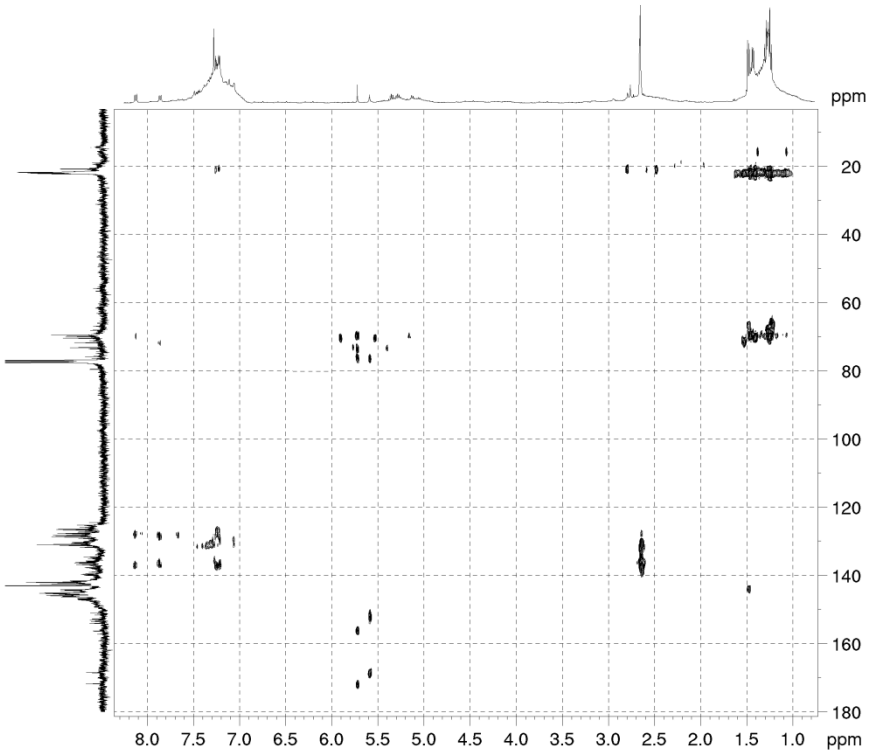

Figure 34. The HMBC spectrum of compound **6** (500.17 MHz for ^{1}H, 125.78 MHZ for ^{13}C, solvent CS_2 : $CDCl_3$ = 5:1)