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Lead(IV) acetate: an efficient reagent for the synthesis of pyrrolidinofullerenes *via* oxidative coupling of C_{60} with amino acids esters

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Compound	Eluent (toluene : MeOH, v/v)	Yield (%)*
2a	**100:0**	**30**
2b	**99.7:0.3**	**40**
2c	**99.5:0.5**	**24**
3b	**99.9:0.1**	**8**
3c	**99.8:0.2**	**9**

* Yields are given based on the amount of the fullerene introduced in the reaction. The amount of unreacted fullerene recovered after chromatographic separation was *ca*. 40-50%.

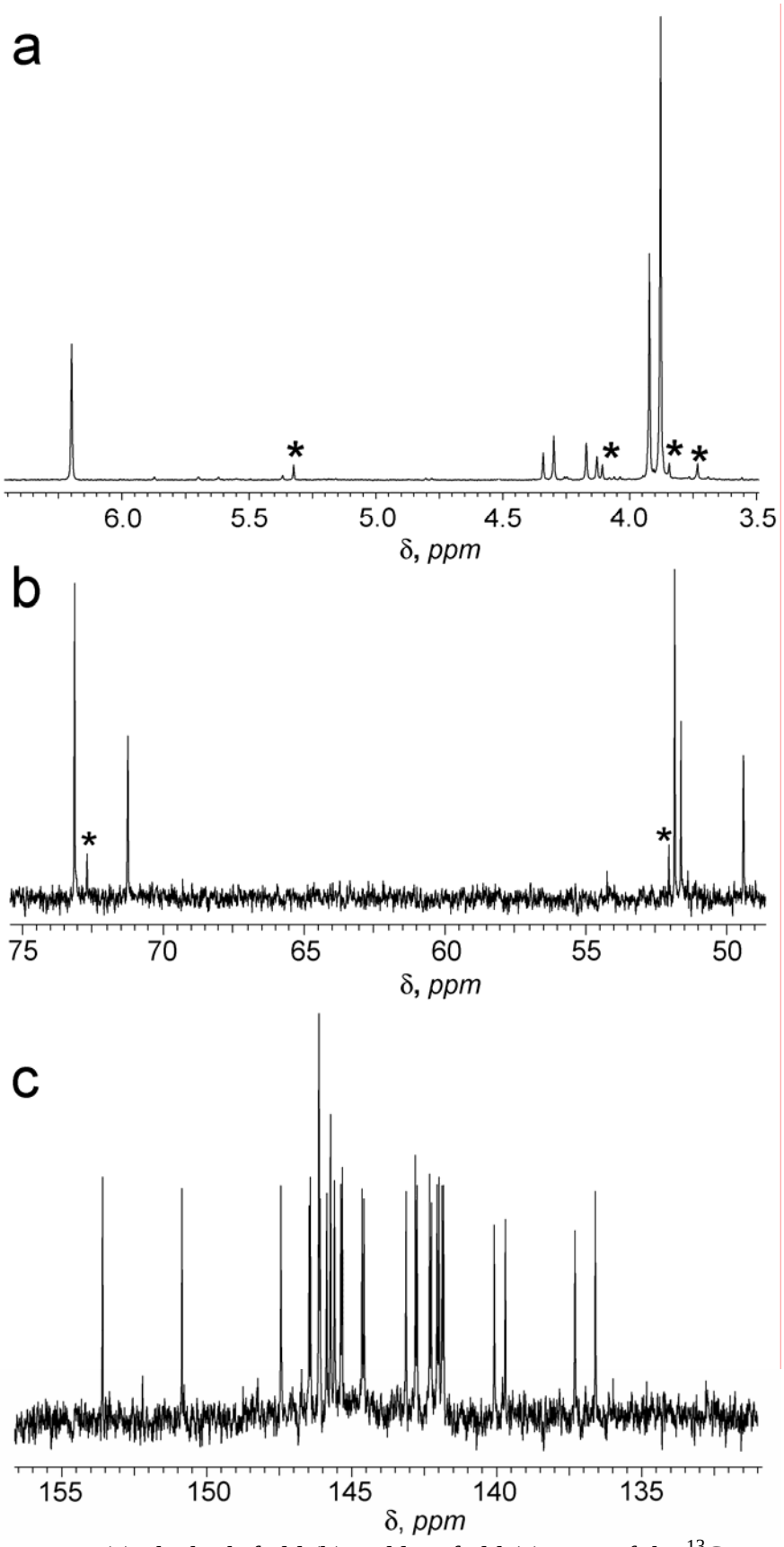
NMR data for compounds. 2a ^{1}H NMR (400 MHz, $CDCl_3$): δ= 3.88 (6H, s), 3.92 (3H, s), 4.15 (1H, d), 4.32 (1H, d), 6.20 (2H, s) ppm. ^{13}C NMR (100 MHz, CS_2-acetone-d^6 10:1): δ= 49.42, 51.64, 51.85, 71.26, 73.16, 136.6, 137.3, 139.71, 140.09, 141.84, 141.88, 141.99, 142.06, 142.26, 142.32, 142.75, 142.81, 143.13, 144.58, 144.64, 145.33, 145.38, 145.6, 145.73, 145.86, 146.09, 146.13, 146.43, 146.47, 147.44, 150.86, 153.59, 169.1, 169.44 ppm.

2b ^{1}H NMR (400 MHz, CS_2-acetone-d^6 10:1): δ= 3.19 (1H, m), 3.27 (2H, t), 3.48 (1H, m), 3.68 (6H, s), 3.74 (2H, d), 3.81 (6H, s), 3.89(2H, d), 6.09 (2H, s) ppm. ^{13}C NMR (150 MHz, CS_2-acetone-d^6 10:1):45.37, 50.15, 50.7, 50.86, 53.63, 70.7, 72.85, 124.58, 135.58, 136.19, 138.84, 139.2, 140.95, 141.05, 141.11, 141.27, 141.38, 141.44, 141.86, 141.92, 142.27, 143.75, 143.78, 144.45, 144.5, 144.68, 144.79, 144.86, 145.08, 145.25, 145.4, 145.54, 145.58, 146.57, 150.56, 153.25, 169.3, 169.94

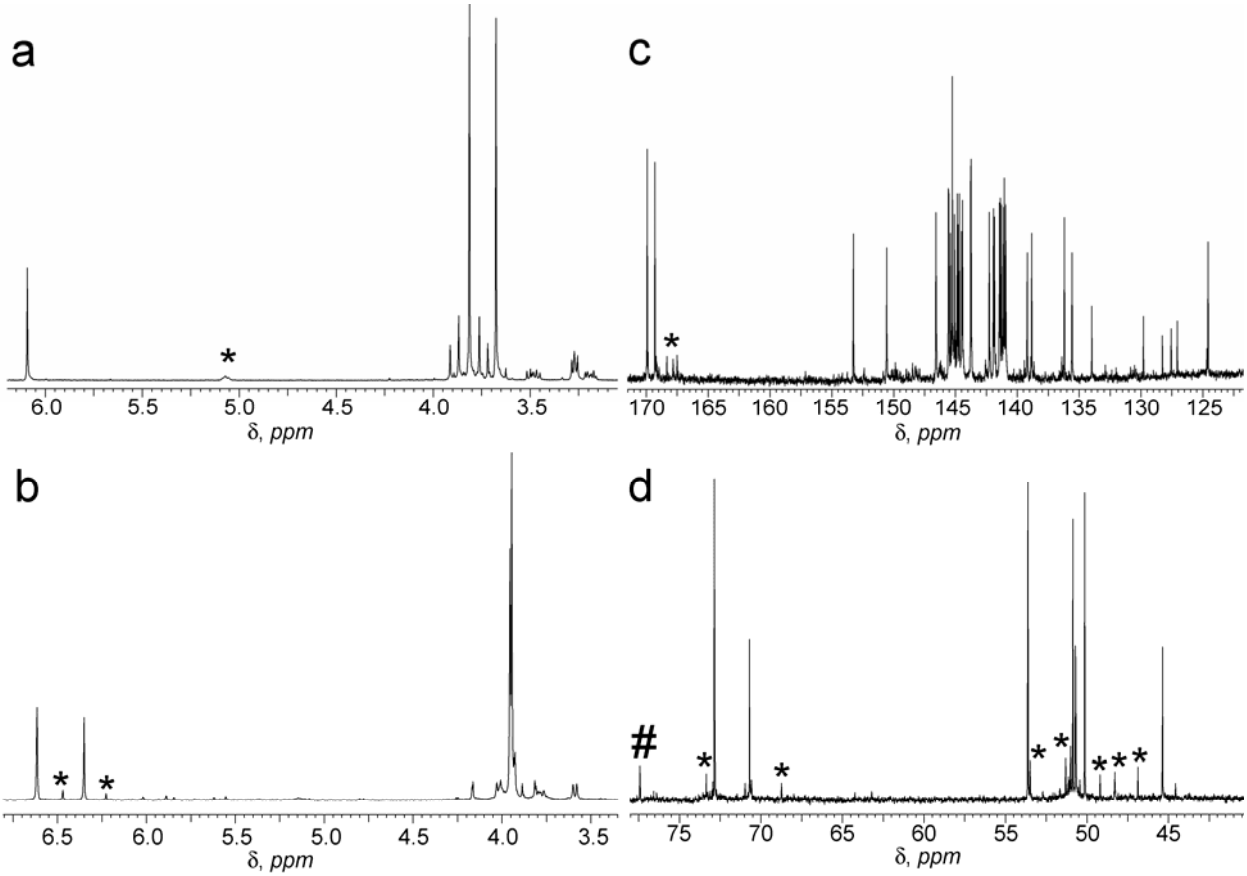
2c ^{1}H NMR (600 MHz, $CDCl_3$): δ= 1.52 (2H, m), 1.62 (2H, m), 2.06 (4H, m), 2.79 (2H, t) 3.08 (1H, m), 3.38 (1H, m), 3.61 (4H, s), 3.75 (6H, s), 3.88 (6H, s), 5.93 (2H, s) ppm. ^{13}C NMR (100 MHz, CS_2-acetone-d^6 10:1): δ= 28.43, 28.5, 30.3, 32.44, 48.38, 50.85, 51.72, 54.14, 54.23, 71.43, 73.87, 134.83, 136.62, 137.29, 139.72, 140.1, 141.82, 141.87, 141.97, 142.1, 142.22, 142.3, 142.72, 142.8, 143.12, 144.61, 144.62, 145.32, 145.37, 145.55, 145.7, 145.72, 145.84, 146.11, 146.16, 146.41, 146.44, 147.43, 151.26, 153.88, 169.73, 170.41 ppm.

3b ^{1}H NMR (400 MHz, $CDCl_3$): δ= 3.79 (2H, m), 3.94 (6H, s), 3.96 (6H, s), 4.01 (2H, m), 6.35 (2H, s), 6.62 (2H, s) ppm.

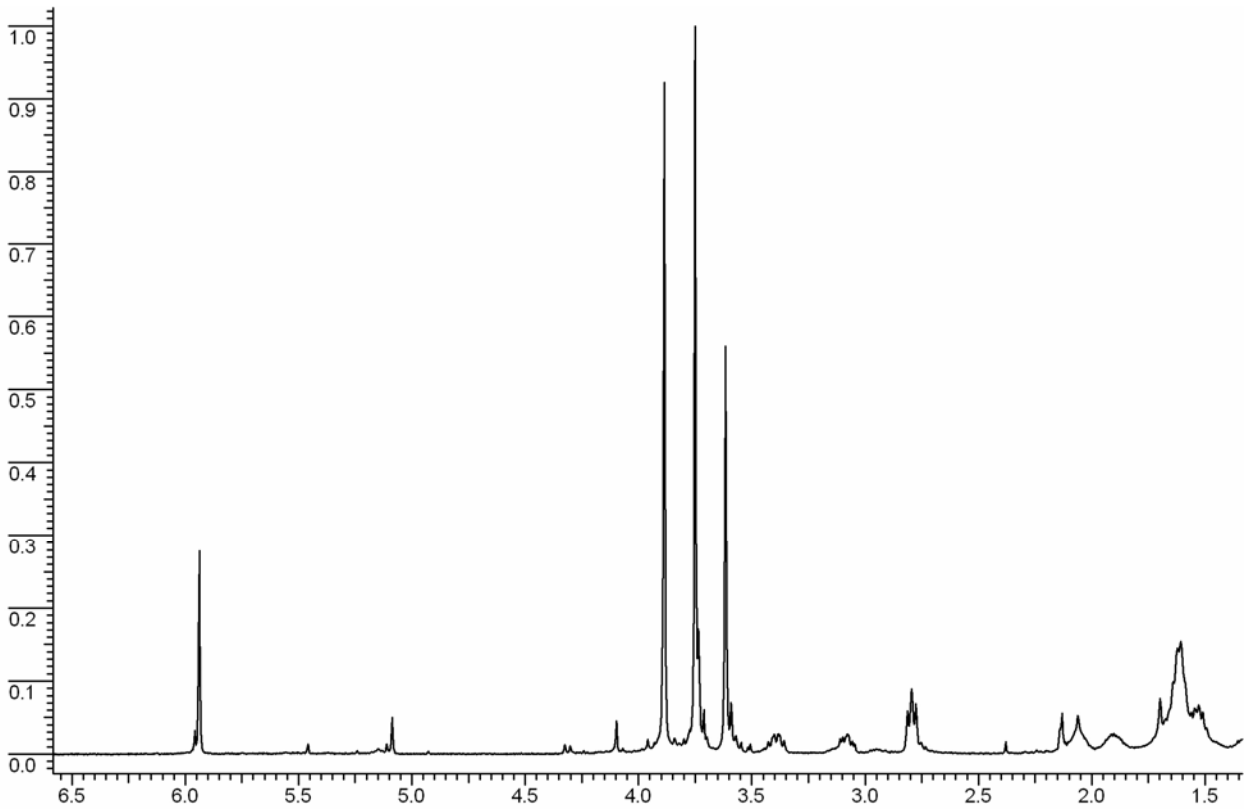
3c ^{1}H NMR (600 MHz, $CDCl_3$): δ= 2.10 (8H, m), 3.18 (2H, m), 3.47 (2H, m), 3.91 (12H, s), 5.99 (4H, s) ppm. ^{13}C NMR (100 MHz, CS_2-acetone-d^6 10:1): δ= 32.5, 33.26, 48.41, 51.76, 71.46, 73.89, 136.63, 136.65, 137.31, 137.33, 139.73, 140.12, 141.83, 141.90, 141.96, 142.12, 142.23, 142.3, 142.74, 142.81, 143.14, 144.62, 145.32, 145.37, 145.57, 145.71, 145.83, 146.13, 146.41, 146.44, 147.43, 151.22, 153.84, 169.75 ppm.



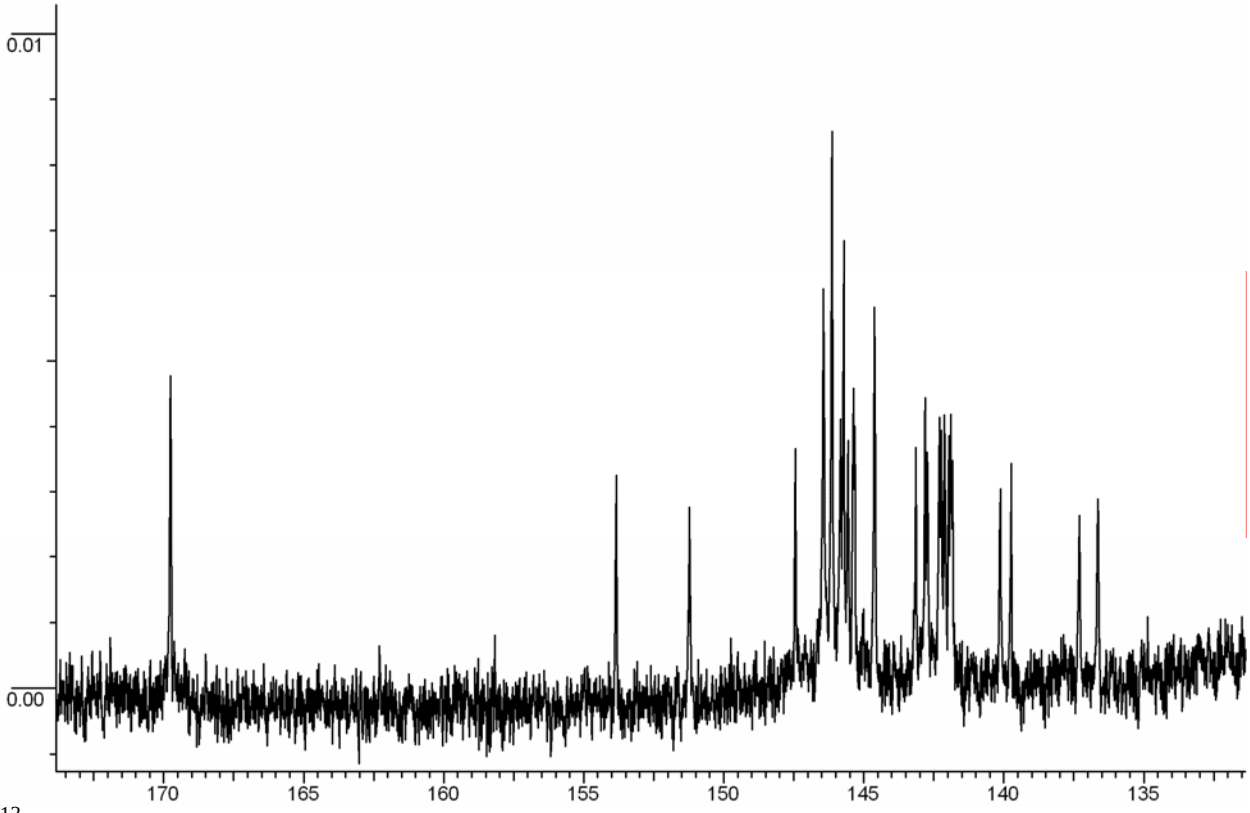
The ^{1}H NMR spectrum (a), the high-field (b) and low field (c) parts of the ^{13}C NMR spectrum of **2a.** Symbol "*" denotes signals assigned to the minor *cis*-isomer.



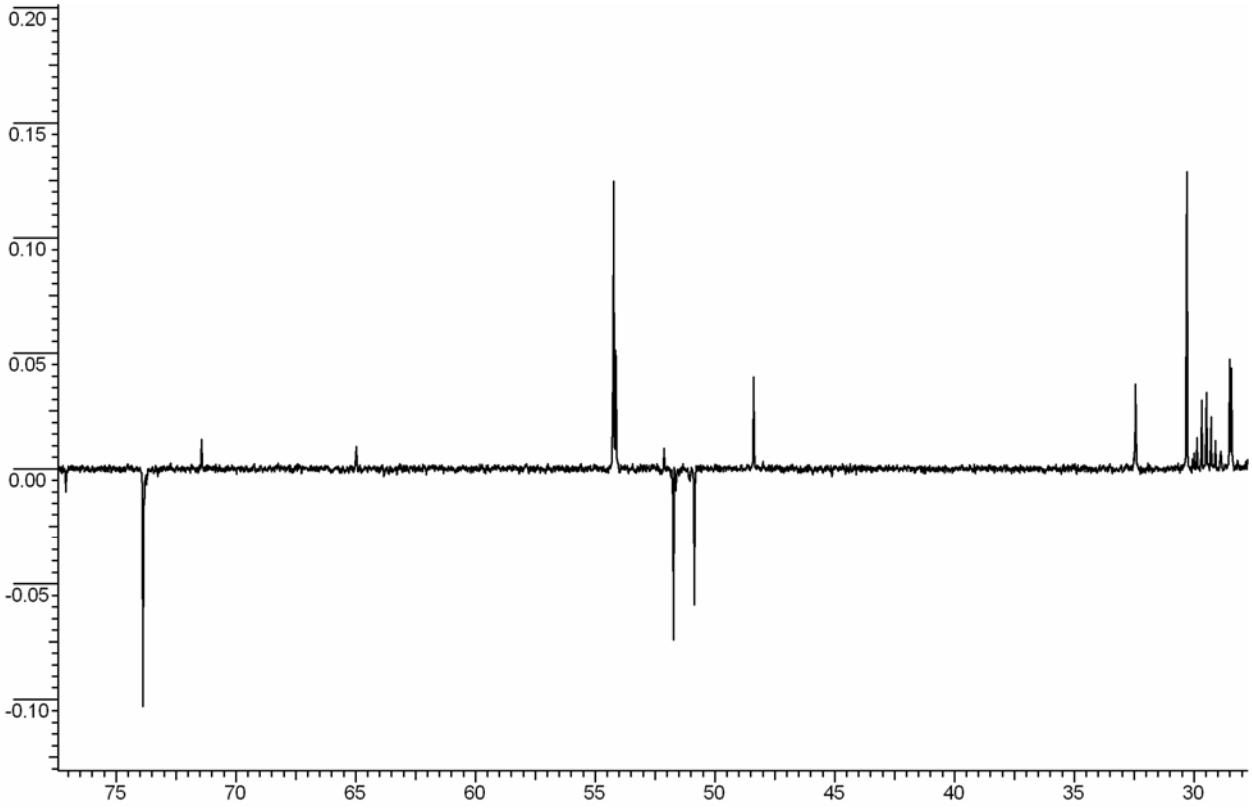
The ^{1}H NMR spectra of **2b** (a) and **3b** (b); the high-field (c) and low field (d) parts of the ^{13}C NMR spectrum of **2b**. Symbol "*" denotes signals assigned to the minor *cis*-products, symbol "#" denotes signal of $CHCl_3$ impurity.



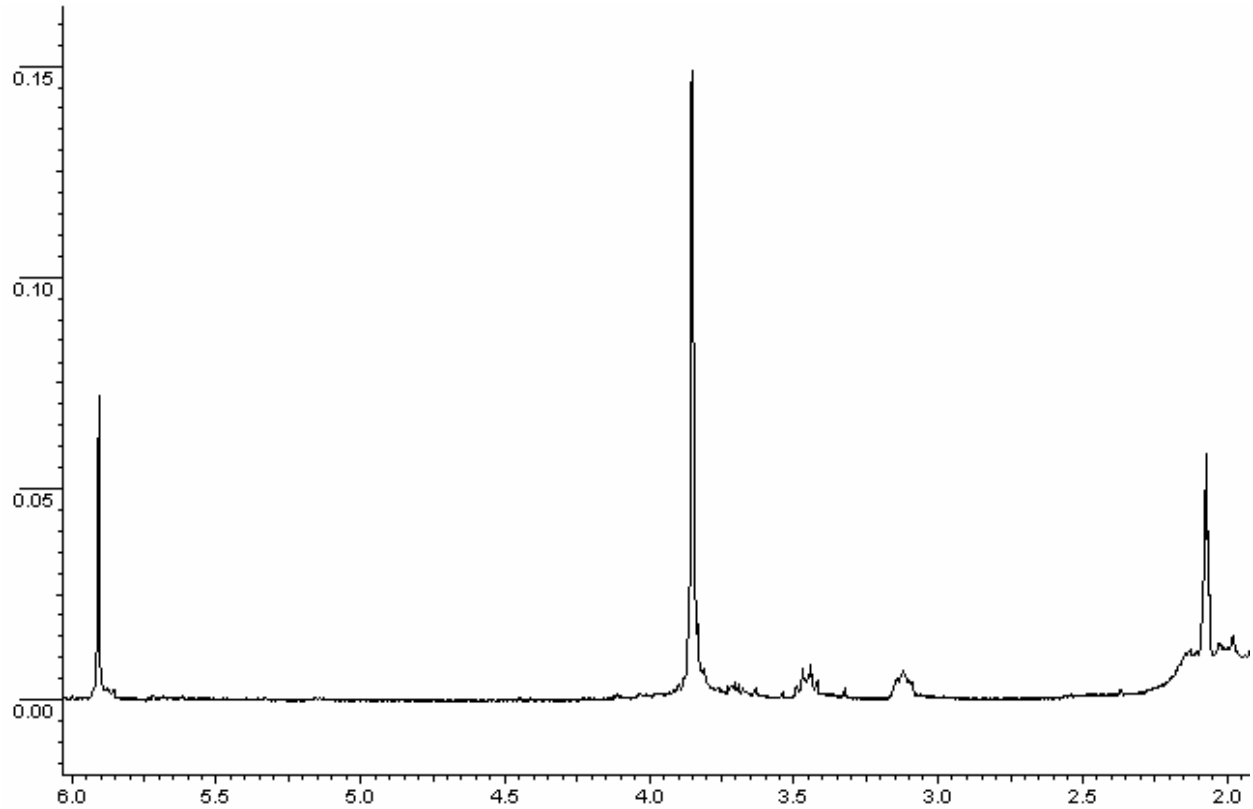
^{1}H NMR spectrum of **2c**



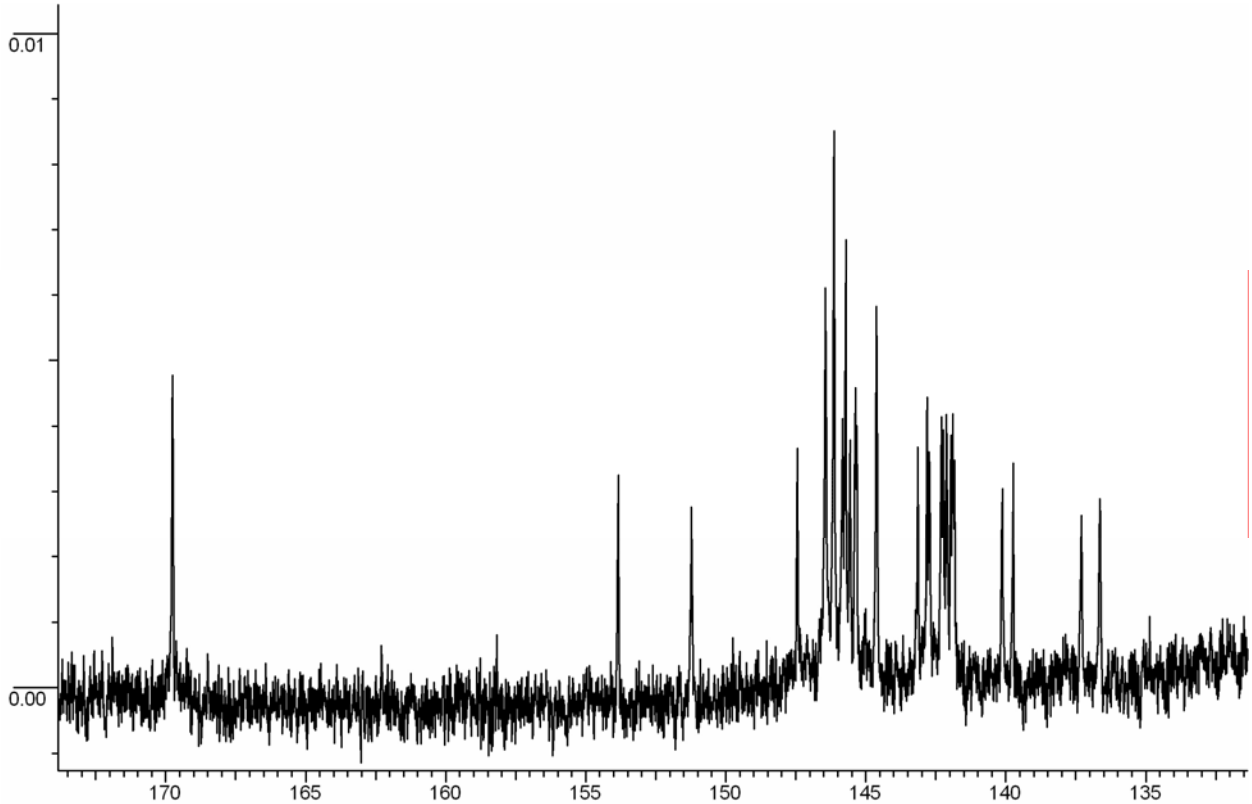
^{13}C NMR spectrum of **2c** (low field)



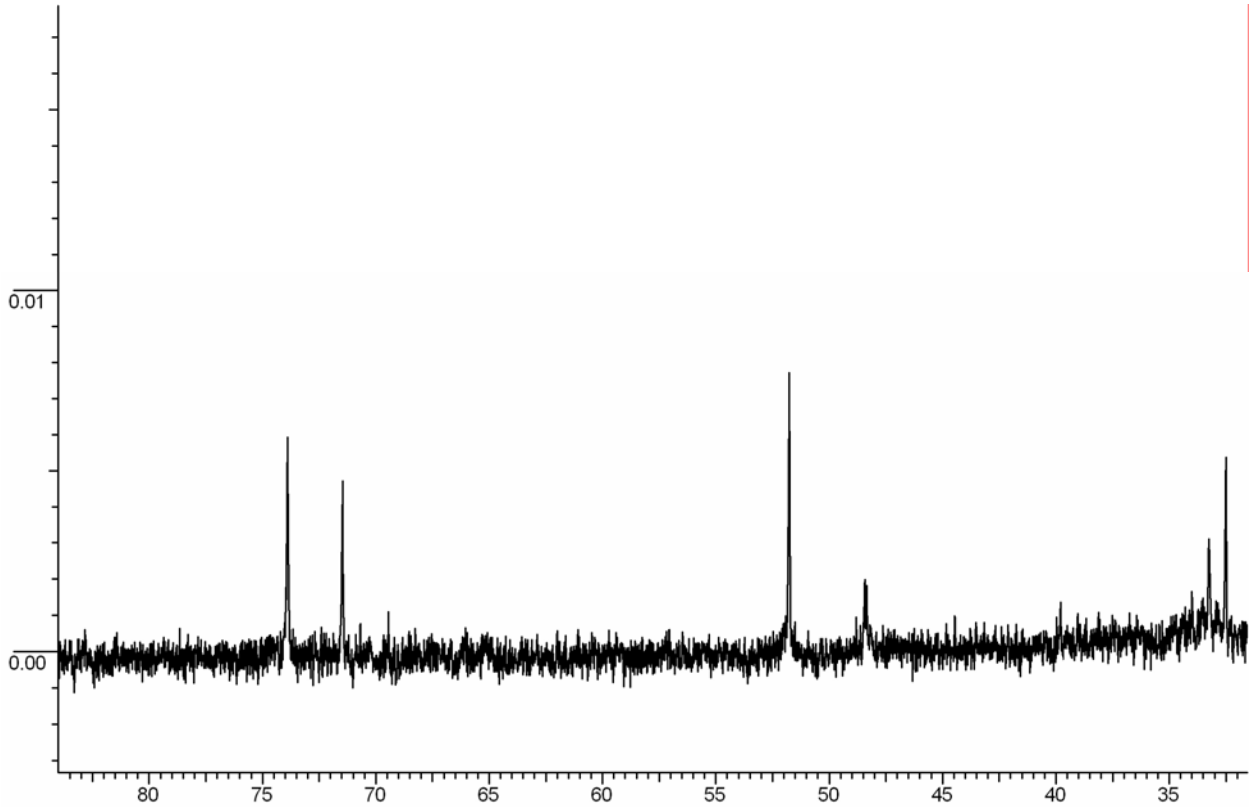
^{13}C NMR spectrum of **2c** (high field)



^{1}H NMR spectrum of **3c**



^{13}C NMR spectrum of **3c** (low field)



^{13}C NMR spectrum of **3c** (high field)