

*Advanced*  
**Synthesis &  
Catalysis**

Supporting Information

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## Experimental Section

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### List of MS and $^{13}\text{C}$ DEPT spectra for new compounds **1f**.

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### List of XPS and AES spectra

Page 42	(a) XPS survey spectrum for the sample prepared from the reaction residue of entry 10 in Table 1. (b) XPS spectrum of Cu <sub>2</sub> P.
Page 43	(c) XPS spectrum of I3d. (d) AES spectrum for Cu <sup>1+</sup> .

## Experimental Section

All melting points were determined on a Yanaco melting point apparatus and are uncorrected. IR spectra were recorded on a Nicolet FT-IR 5DX spectrometer. The  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on a JEOL JNM-ECA 300 spectrometer in  $\text{CDCl}_3$  with TMS as internal reference. The  $J$  values are given in Hz. MS were recorded on a VG-ZAB-MS spectrometer with 70 eV. Elementary analysis data were obtained on a Perkin-Elmer-241C apparatus.

**A Typical Procedure for Preparation of 1-Phenylpyrazole (1a):** A stirred suspension of PhI (**2a**, 810 mg, 4 mmol), pyrazole (**3a**, 41 mg, 6 mmol), Cu(0) powder (51 mg, 0.8 mmol) and  $\text{Cs}_2\text{CO}_3$  (2.60 g, 8 mmol) in MeCN was refluxed for 12 h (monitored by TLC) under the nitrogen. After it was cooled to room temperature, the solid was filtered off and MeCN was evaporated. The residue was purified by chromatography (silica gel, 40%  $\text{CH}_2\text{Cl}_2$  in PE) to give desired product **1a** (547 mg) in 95% yield.

The arylated products **1a-p** were prepared efficiently by using the similar procedure (See Table 3 and Table 4).

**1-Phenylpyrazole (1a).**<sup>1</sup> This compound was obtained in 95% yield as colorless oil after chromatography (silica gel, 50%  $\text{CH}_2\text{Cl}_2$  in PE).  $^1\text{H}$  NMR:  $\delta$  7.93-7.92 (d,  $J$  = 2.4 Hz, 1H), 7.73-7.68 (m, 3H), 7.50-7.43 (m, 2H), 7.31-7.24 (m, 1H), 6.47-6.45 (m, 1H);  $^{13}\text{C}$  NMR:  $\delta$  140.6, 139.8, 129.0, 126.3, 125.9, 118.7, 107.2.

**1-Phenylpyrrole (1b).** This compound was obtained in 96% yield as white solid after chromatography (silica gel, PE). Mp: 61-62 °C (PE) (lit.<sup>2</sup> 59-60 °C);  $^1\text{H}$  NMR:  $\delta$  7.43-7.40 (m, 4H), 7.26-7.23 (m, 1H), 7.10-7.08 (m, 2H), 6.36-6.34 (m, 2H);  $^{13}\text{C}$  NMR:  $\delta$  140.7, 129.5, 125.5, 120.4, 119.2, 110.4.

**1-Phenylimidazole (1c).**<sup>3</sup> This compound was obtained in 95% yield as colorless oil after chromatography (silica gel, 50% CH<sub>2</sub>Cl<sub>2</sub> in PE). <sup>1</sup>H NMR:  $\delta$  7.85 (s, 1H), 7.51-7.47 (m, 2H), 7.45-7.34 (m, 3H), 7.28 (s, 1H), 7.21 (s, 1H); <sup>13</sup>C NMR:  $\delta$  136.9, 135.1, 130.0, 129.4, 127.0, 120.9, 117.7.

**1-Phenyl-1,2,3-triazole (1d).** This compound was obtained in 45% yield as a white solid after chromatography (silica gel, PE: EtOAc: CH<sub>2</sub>Cl<sub>2</sub> = 7:2:1). mp: 55-56 °C (PE-EtOAc) (lit.<sup>4</sup> 60-61 °C); <sup>1</sup>H NMR:  $\delta$  8.03-8.02 (d, *J* = 1.0 Hz, 1H), 7.83 (s, 1H), 7.75-7.72 (m, 2H), 7.53-7.40 (m, 3H); <sup>13</sup>C NMR:  $\delta$  136.7, 134.2, 129.5, 128.5, 121.6, 120.3.

**1-Phenyl-1,2,4-triazole (1e).** This compound was obtained in 54% yield as white solid after chromatography (silica gel, PE: EtOAc: CH<sub>2</sub>Cl<sub>2</sub> = 3:1:1). mp: 45-47 °C (PE-EtOAc) (lit.<sup>1</sup> 47 °C); <sup>1</sup>H NMR:  $\delta$  8.56 (s, 1H), 8.10-8.08 (m, 1H), 7.69-7.66 (m, 2H), 7.52-7.47 (m, 2H), 7.41-7.38 (m, 1H); <sup>13</sup>C NMR:  $\delta$  152.5, 140.8, 136.9, 129.6, 128.1, 119.9.

**1,4-Diphenyl-1H-pyrrole-3-carboxylic acid ethyl ester (1f).** This compound was obtained in 91% yield as white solid after chromatography (silica gel, PE: EtOAc: CH<sub>2</sub>Cl<sub>2</sub> = 10:1:1). mp: 68-70 °C (PE-EtOAc); IR:  $\nu$  3141, 3049, 1672, 1601 cm<sup>-1</sup>; <sup>1</sup>H NMR:  $\delta$  7.77-7.76 (d, *J* = 2.7 Hz, 1H), 7.58-7.28 (m, 10H), 7.07-7.06 (d, *J* = 2.7 Hz, 1H), 4.25-4.20 (q, *J* = 7.2 Hz, 2H), 1.28-1.23 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR:  $\delta$  164.0, 139.0, 134.0, 129.3, 129.0, 128.0, 127.3, 126.4, 126.3, 125.5, 120.2, 119.2, 115.1, 59.3, 13.9; MS *m/z* (%): 293 (2.8), 292 (22.1), 291 (M<sup>+</sup>, 100), 246 (90.7); Anal Calcd for C<sub>19</sub>H<sub>17</sub>NO<sub>2</sub> (291.34): C, 78.33; H, 5.88; N, 4.81; Found: C, 78.03; H, 5.78; N, 4.85.

**1-Phenyl-indole (1g).**<sup>3</sup> This compound was obtained in 95% yield as light yellow oil after chromatography (silica gel, PE: EtOAc: CH<sub>2</sub>Cl<sub>2</sub> = 10:1:1). <sup>1</sup>H NMR:  $\delta$  7.68-7.66 (m, 1H), 7.59-7.55 (m, 1H), 7.49-7.47 (m, 4H), 7.33-7.31 (m, 2H),

7.24-7.11 (m, 2H), 6.68-6.67 (m, 1H);  $^{13}\text{C}$  NMR:  $\delta$  139.5, 135.6, 129.3, 127.6, 126.0, 123.9, 122.2, 121.0, 120.3, 110.4, 103.5.

**1-Phenyl-1H-benzoimidazole (1h).** This compound was obtained in 85% yield as white solid after chromatography (silica gel, 50%  $\text{CH}_2\text{Cl}_2$  in PE). Mp: 96-97 °C (PE) (lit.<sup>5</sup> 95-97 °C);  $^1\text{H}$  NMR:  $\delta$  8.11 (s, 1H), 7.92-7.88 (m, 1H), 7.57-7.45 (m, 6H), 7.34-7.31 (m, 2H);  $^{13}\text{C}$  NMR:  $\delta$  143.9, 142.1, 136.1, 133.5, 129.8, 127.8, 123.8, 123.5, 122.6, 120.4, 110.3.

**9-Phenylcarbazole (1i).** This compound was obtained in 80% yield as white solid after chromatography (silica gel, PE: EtOAc:  $\text{CH}_2\text{Cl}_2$  = 7:2:1). mp: 88-90 °C (PE-EtOAc) (lit.<sup>6</sup> 89-90 °C);  $^1\text{H}$  NMR:  $\delta$  8.15-8.12 (m, 2H), 7.60-7.53 (m, 4H), 7.50-7.39 (m, 5H), 7.30-7.22 (m, 2H);  $^{13}\text{C}$  NMR:  $\delta$  140.8, 137.6, 129.7, 127.3, 127.0, 125.9, 123.3, 120.2, 119.9, 109.7.

**9-Phenyl-2,3,4,9-tetrahydrocarbazole (1j).** This compound was obtained in 94% yield as white solid after chromatography (silica gel, PE: EtOAc:  $\text{CH}_2\text{Cl}_2$  = 7:2:1). mp: 84-86 °C (PE-EtOAc) (lit.<sup>7</sup> 86 °C);  $^1\text{H}$  NMR:  $\delta$  7.60-7.41 (m, 3H), 7.38-7.30 (m, 3H), 7.25-7.16 (m, 1H), 7.12-7.05 (m, 2H), 2.79-2.76 (m, 2H), 2.57-2.55 (m, 2H), 1.90-1.83 (m, 4H);  $^{13}\text{C}$  NMR:  $\delta$  138.0, 137.1, 135.7, 129.2, 127.7, 127.1, 126.7, 121.2, 119.5, 117.7, 110.9, 109.8, 23.4, 23.2, 23.1, 21.1.

**1-(4-Nitrophenyl)-pyrrole (1k).** This compound was obtained in 95% yield as light yellow solid after chromatography (silica gel, 50%  $\text{CH}_2\text{Cl}_2$  in PE). mp: 181-183 °C (EtOH) (lit.<sup>2</sup> 178-180 °C);  $^1\text{H}$  NMR:  $\delta$  8.31-8.28 (m, 2H), 7.52-7.49 (m, 2H), 7.18-7.16 (m, 2H), 6.43-6.41 (m, 2H);  $^{13}\text{C}$  NMR:  $\delta$  145.2, 144.7, 125.5, 119.4, 119.1, 112.5.

**Ethyl 4-(pyrrol-1-yl)-benzoate (1l).** This compound was obtained in 94% yield as white solid after chromatography (silica gel, 25%  $\text{CH}_2\text{Cl}_2$  in PE). mp: 74-76 °C

(PE) (lit.<sup>8</sup> 74.5-75 °C); <sup>1</sup>H NMR:  $\delta$  8.09-8.06 (d,  $J$  = 8.2 Hz, 2H), 7.42-7.39 (d,  $J$  = 8.6 Hz, 2H), 7.14-7.12 (m, 2H), 6.37-6.35 (m, 2H), 4.41-4.36 (q,  $J$  = 7.2 Hz, 2H), 1.41-1.36 (t,  $J$  = 7.2 Hz, 3H); <sup>13</sup>C NMR:  $\delta$  165.8, 143.8, 131.1, 127.2, 119.1, 118.9, 111.4, 60.9, 14.3.

**1-(4-Methoxyphenyl)-pyrrole (1m).** This compound was obtained in 95% yield as white solid after chromatography (silica gel, 50% CH<sub>2</sub>Cl<sub>2</sub> in PE). mp: 110-112 °C (PE) (lit.<sup>2</sup> 104-108 °C); <sup>1</sup>H NMR:  $\delta$  7.26-7.23 (m, 2H), 6.96-6.86 (m, 4H), 6.30-6.29 (m, 2H), 3.74 (s, 3H); <sup>13</sup>C NMR:  $\delta$  157.5, 134.3, 121.9, 119.5, 114.5, 109.8, 55.3.

**4-(Pyrrol-1-yl)-aniline (1n).** This compound was obtained in 95% yield as white solid after chromatography (silica gel, PE: EtOAc: CH<sub>2</sub>Cl<sub>2</sub> = 10:1:1). mp: 72-74 °C (PE-EtOAc) (lit.<sup>9</sup> 74-76 °C); <sup>1</sup>H NMR:  $\delta$  7.19-7.16 (d,  $J$  = 8.6 Hz, 2H), 6.97-6.96 (m, 2H), 6.73-6.69 (d,  $J$  = 8.6 Hz, 2H), 6.30-6.28 (m, 2H), 3.66 (br, 2H); <sup>13</sup>C NMR:  $\delta$  144.4, 132.5, 122.0, 119.5, 115.4, 109.3.

**1-(2-Bromophenyl)-pyrrole (1o).**<sup>10</sup> This compound was obtained in 85% yield as light yellow oil after chromatography (silica gel, PE). <sup>1</sup>H NMR:  $\delta$  7.64-7.61 (m, 1H), 7.33-7.20 (m, 2H), 7.18-7.14 (m, 1H), 6.84-6.82 (m, 2H), 6.31-6.29 (m, 2H); <sup>13</sup>C NMR:  $\delta$  140.2, 133.6, 128.6, 128.1, 128.0, 122.0, 119.6, 109.1.

**1-(4-Chlorophenyl)-pyrrole (1p).** This compound was obtained in 83% yield as white solid after chromatography (silica gel, PE). mp: 81-83 °C (PE) (lit.<sup>2</sup> 81-82 °C); <sup>1</sup>H NMR:  $\delta$  7.40-7.29 (m, 4H), 7.04-7.02 (m, 2H), 6.35-6.33 (m, 2H); <sup>13</sup>C NMR:  $\delta$  139.2, 130.9, 129.5, 121.4, 119.1, 110.8.

## References:

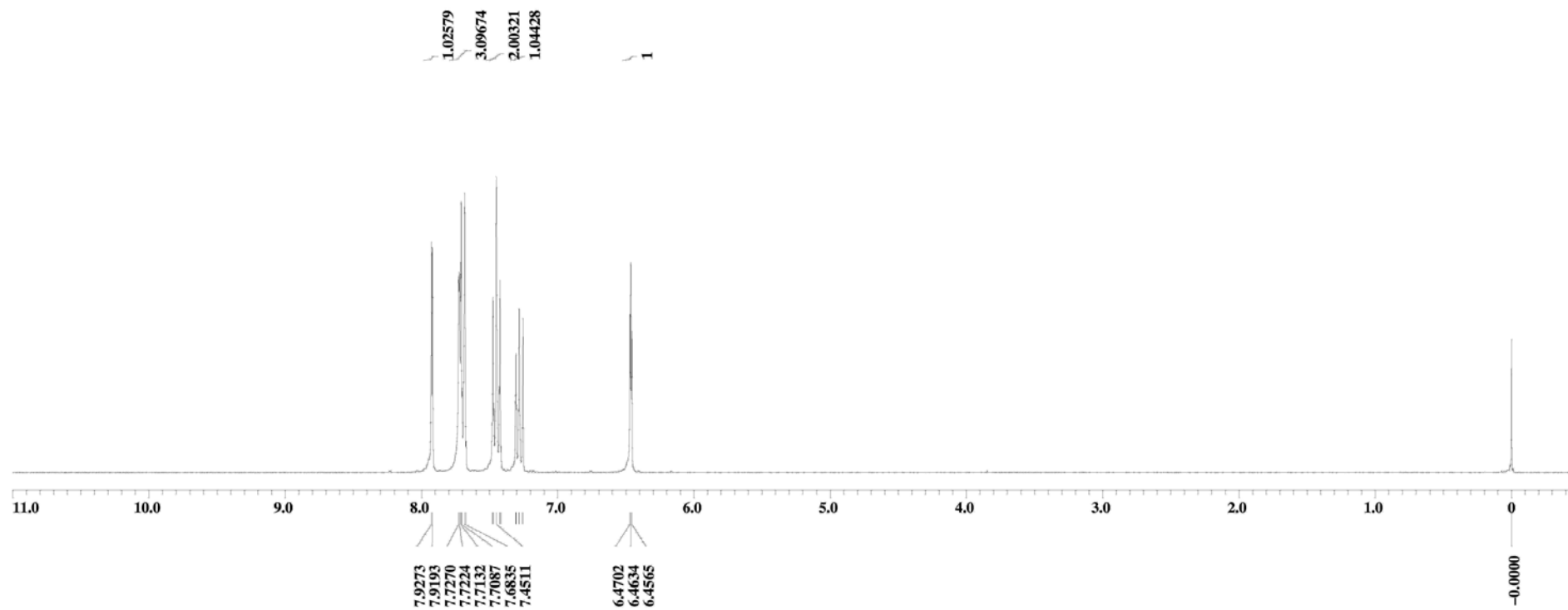
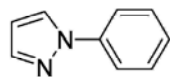
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**Preparation of the Cu<sup>1+</sup> sample for XPS and AES spectra:** A stirred suspension of PhI (**2a**, 810 mg, 4 mmol), pyrazole (**3a**, 41 mg, 6 mmol), Cu(0) powder (51 mg, 0.8 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (2.60 g, 8 mmol) in MeCN was refluxed for 12 h under the nitrogen. After it was cooled to room temperature, the solid was filtered off and MeCN was evaporated. The residue was diluted by petroleum ether (20 mL) and the resultant mixture was stirred for 10 min. Then the mixture was filtered and the solid was collected and washed with petroleum ether (50 mL). After the solid was dried in vacuum, it was used as the sample for the XPS and AES spectra.

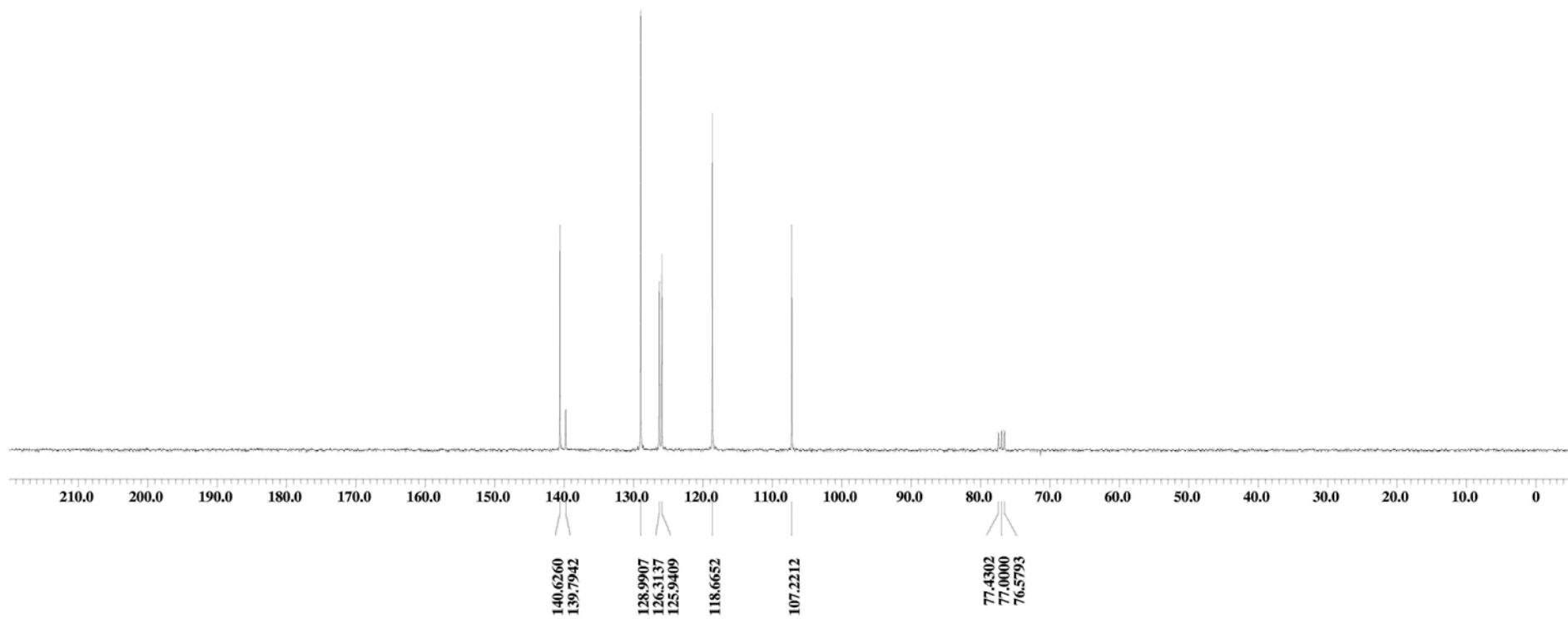
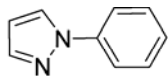
**General Conditions for XPS and AES spectra:** X-ray photoelectron spectroscopy data were obtained with an ESCALab220i-XL electron spectrometer from VG Scientific using 300W MgK $\alpha$  radiation. The base pressure was about  $3 \times 10^{-9}$  mbar. The binding energies were referenced to the C1s line at 284.8 eV from adventitious carbon.

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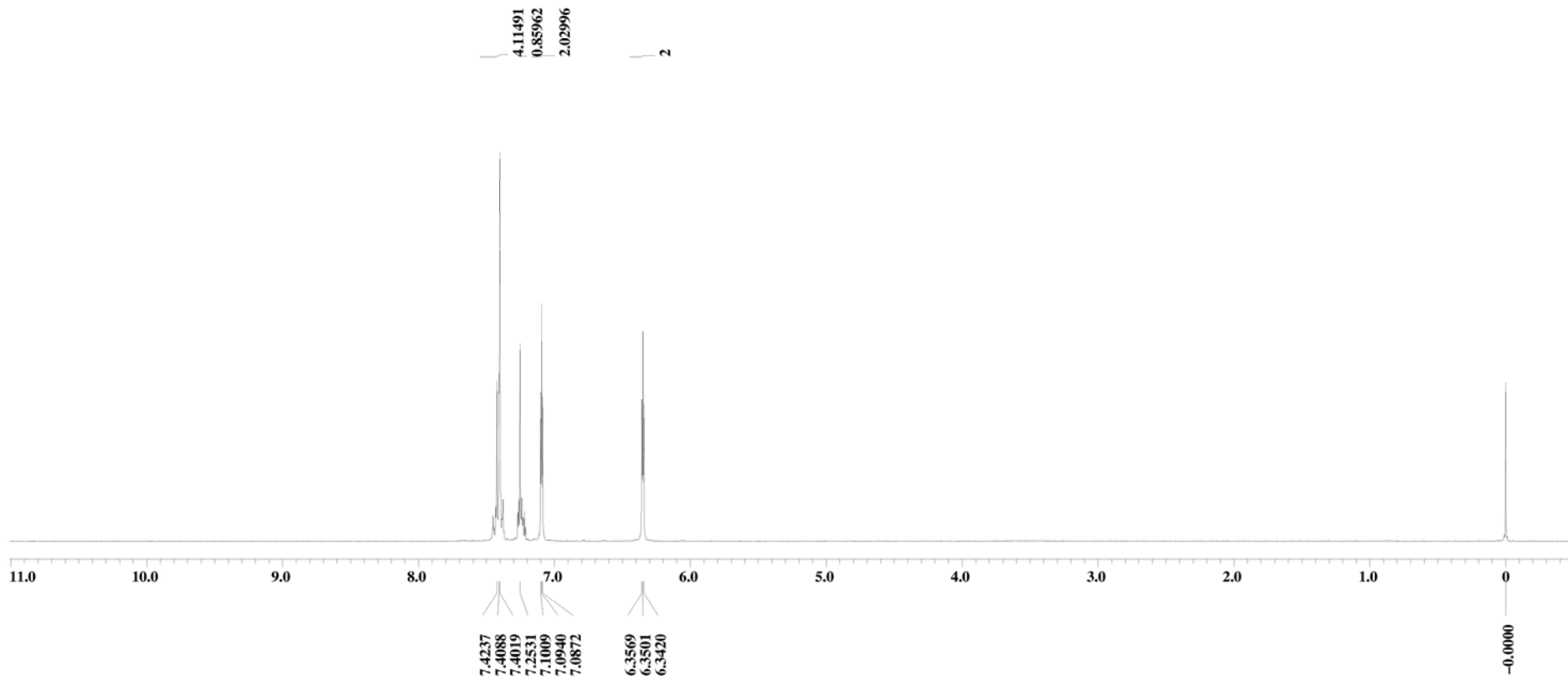
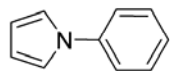
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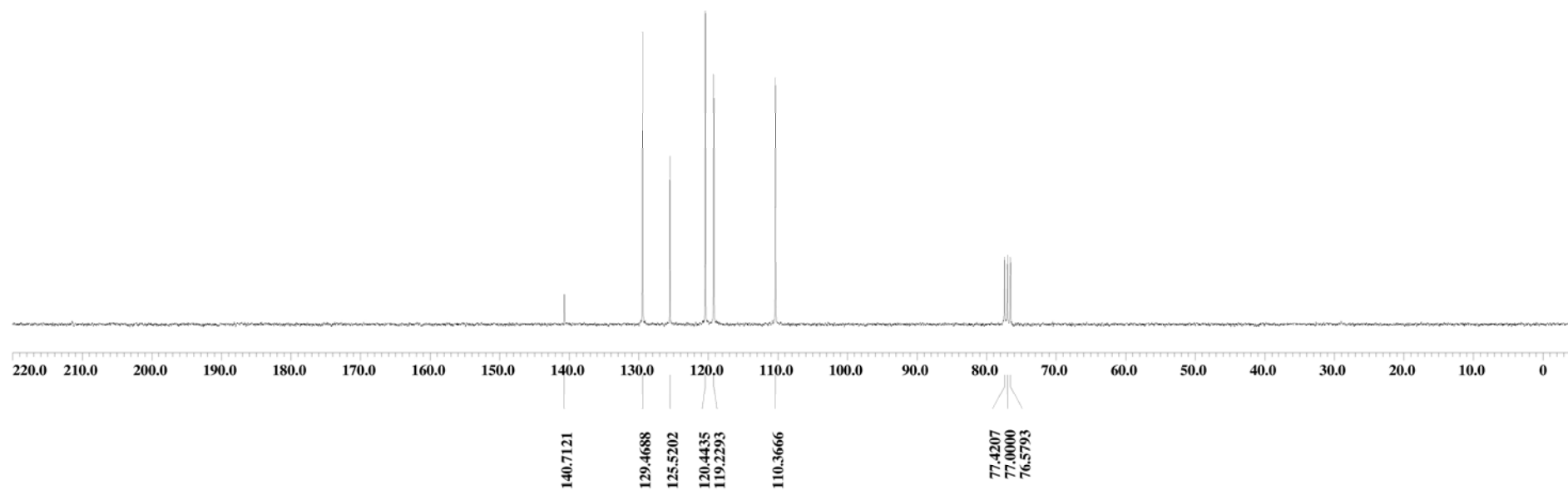
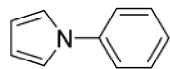
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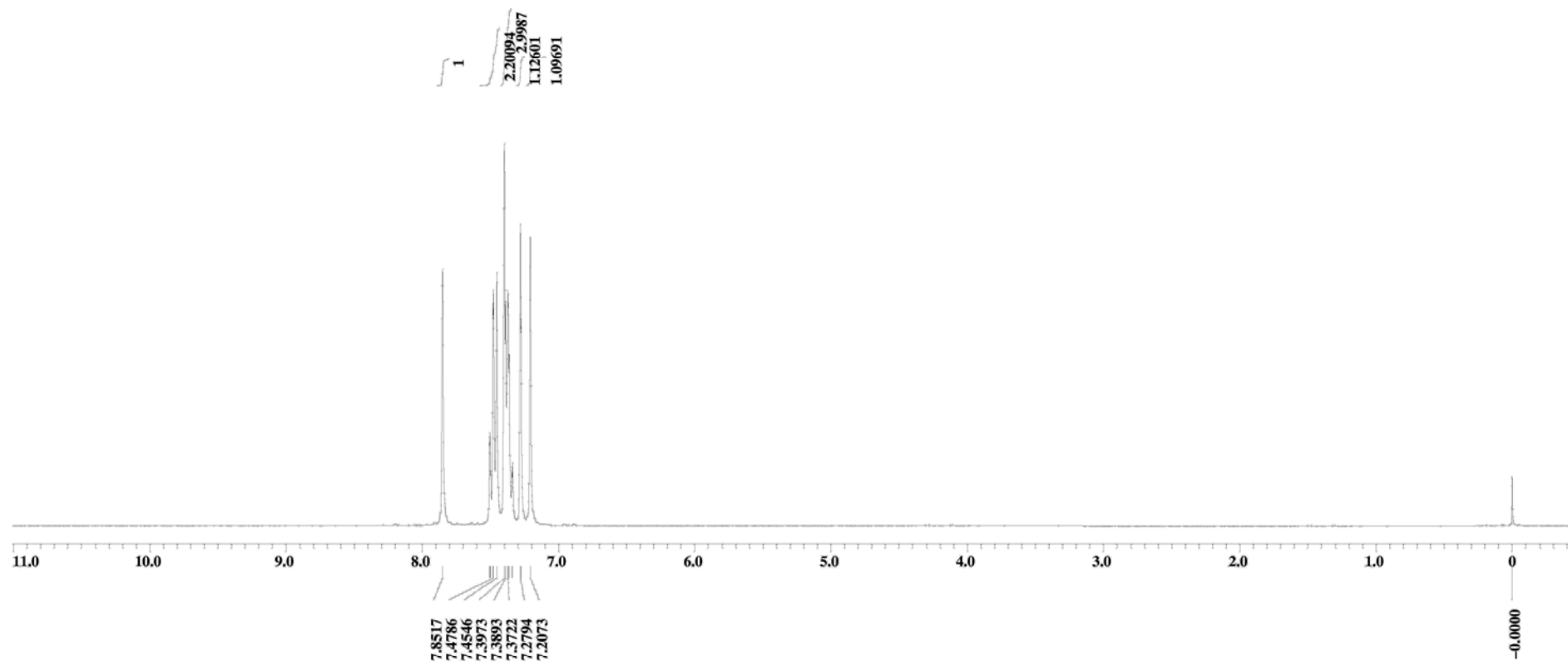
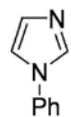
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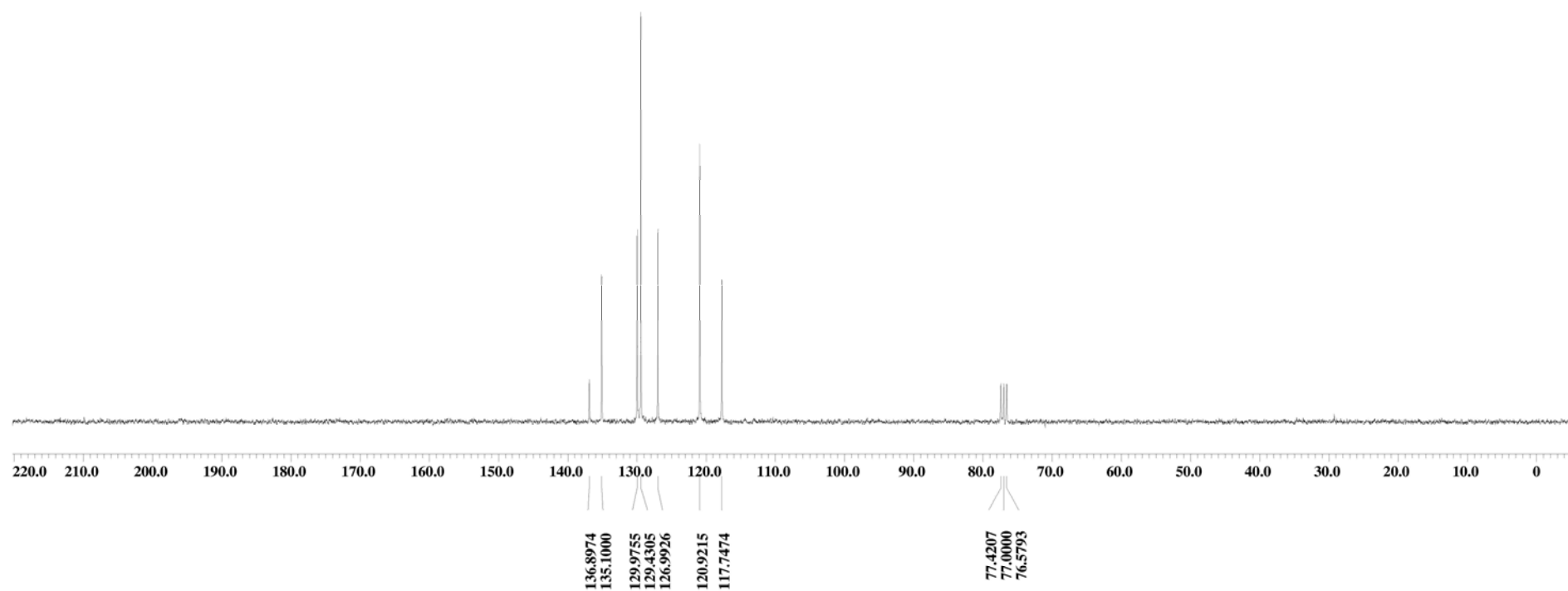
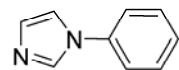
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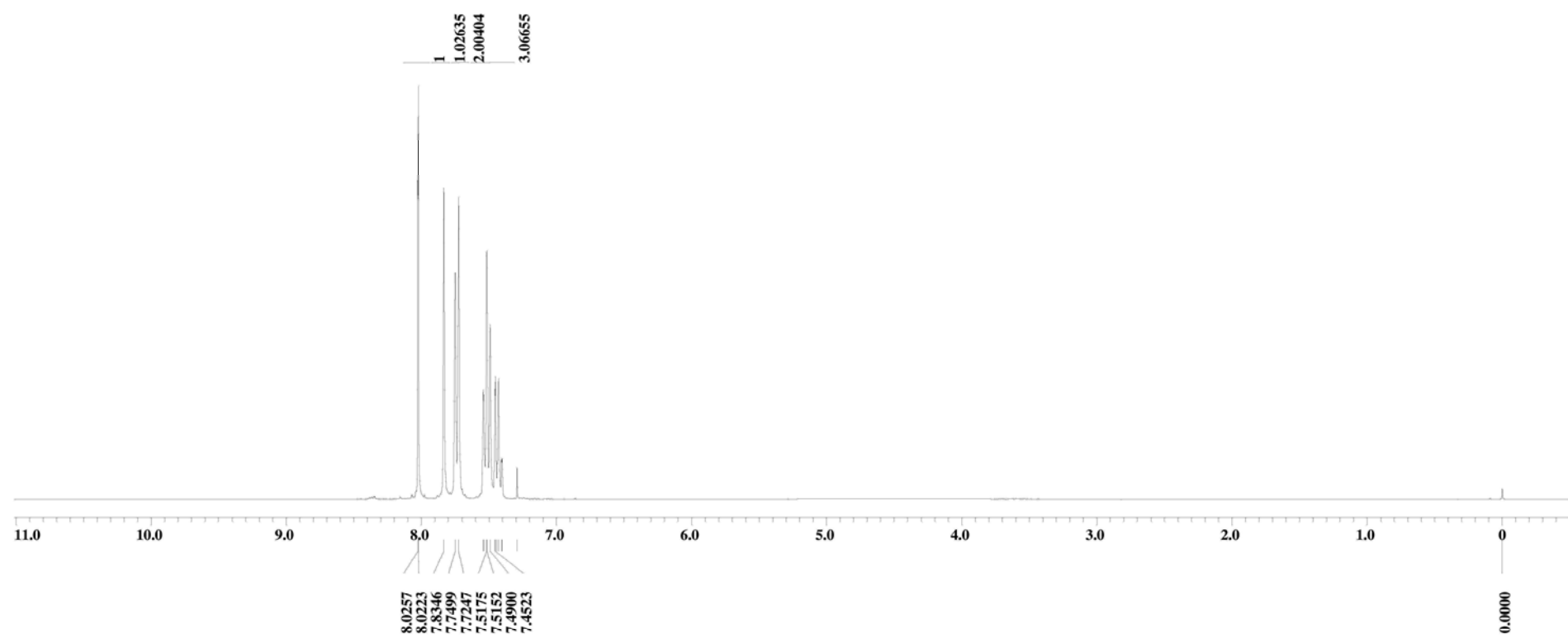
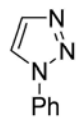
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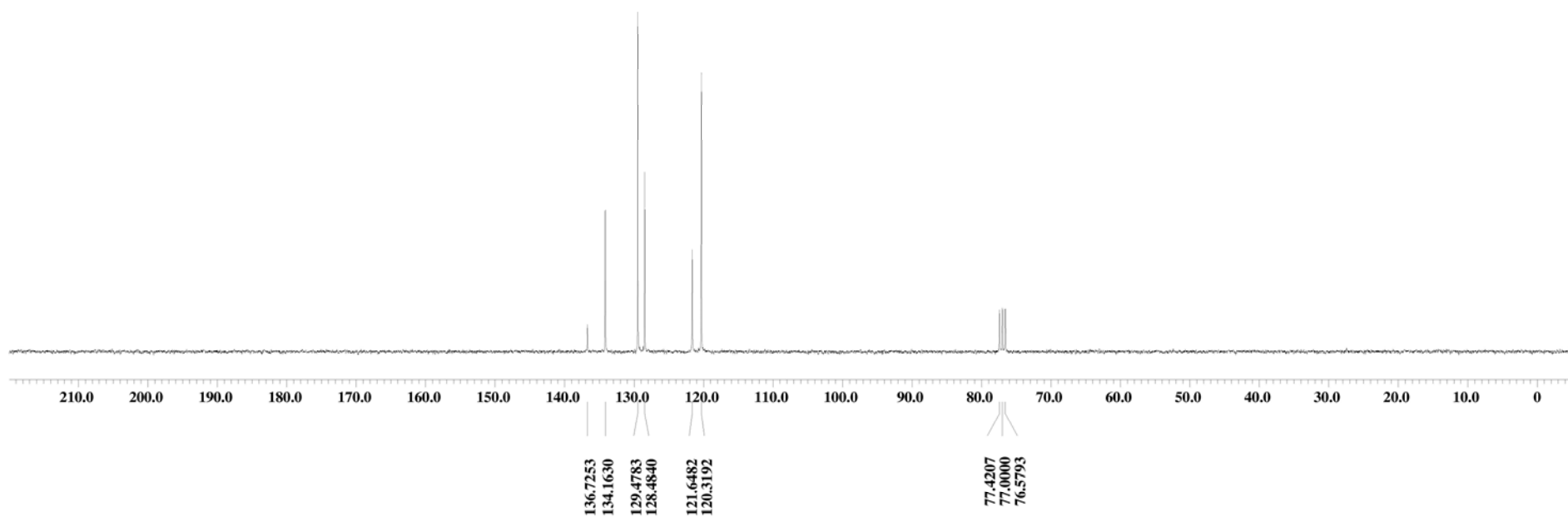
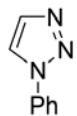
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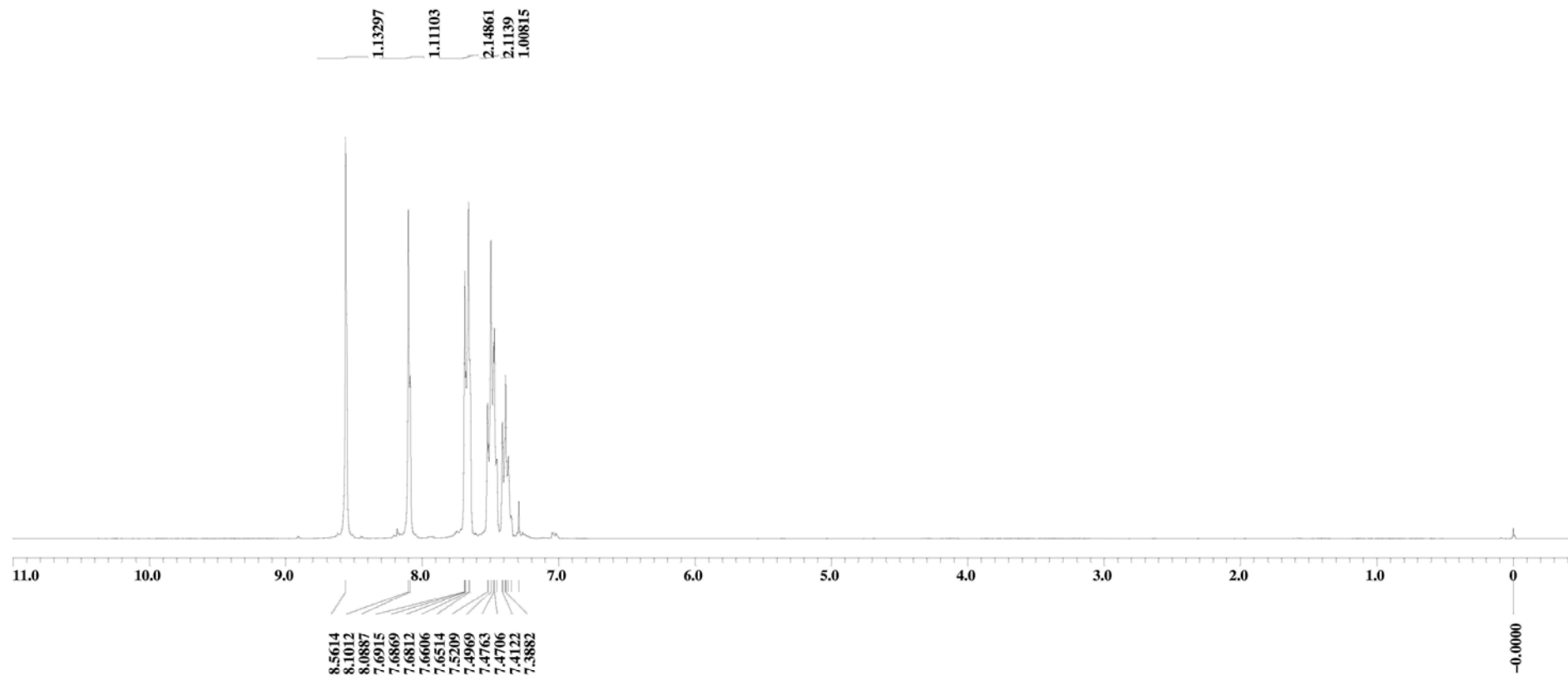
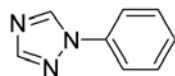
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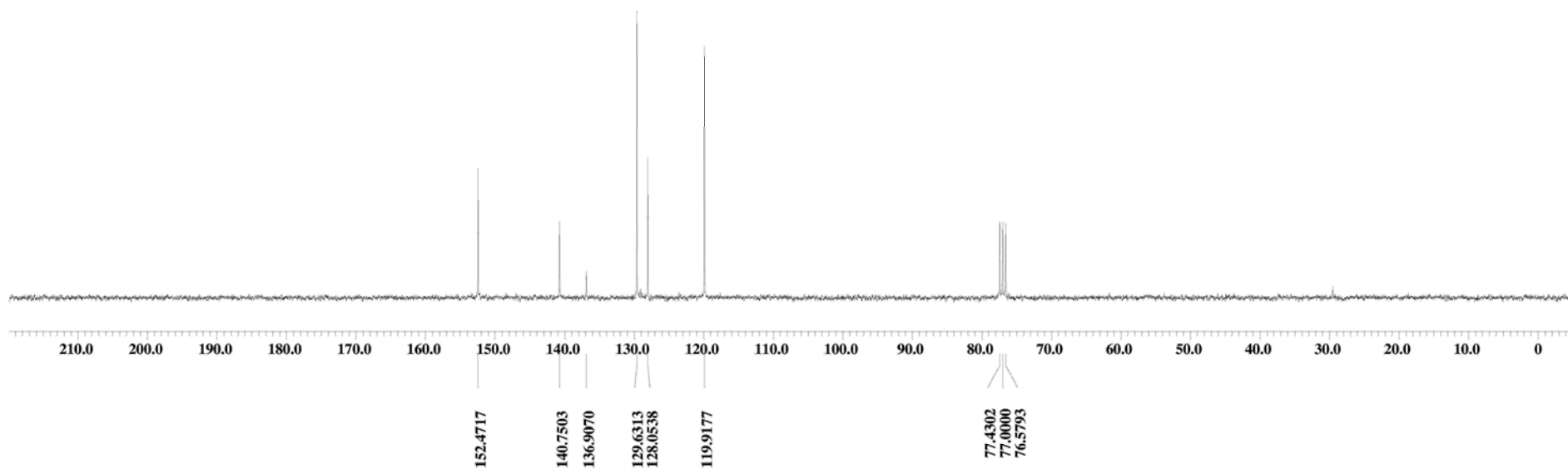
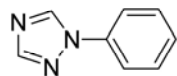


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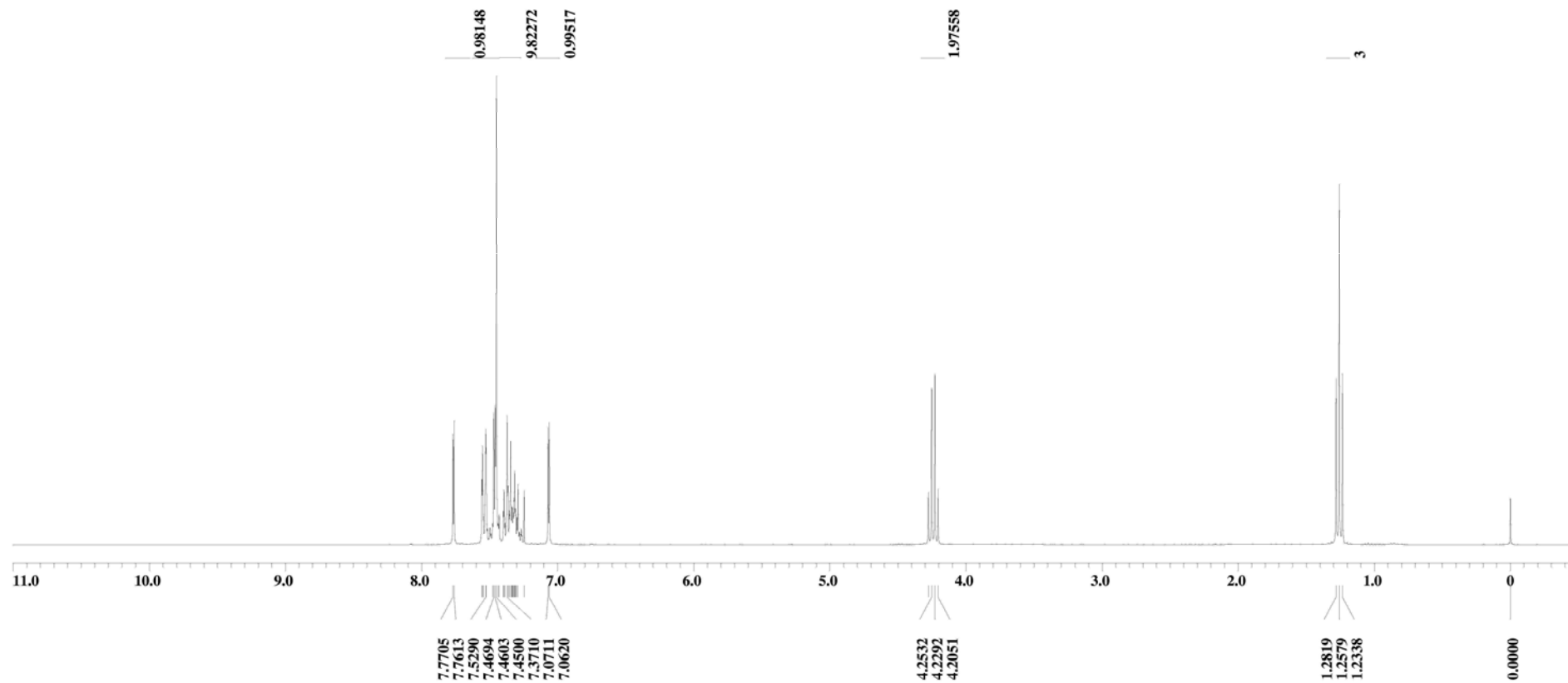
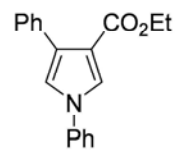


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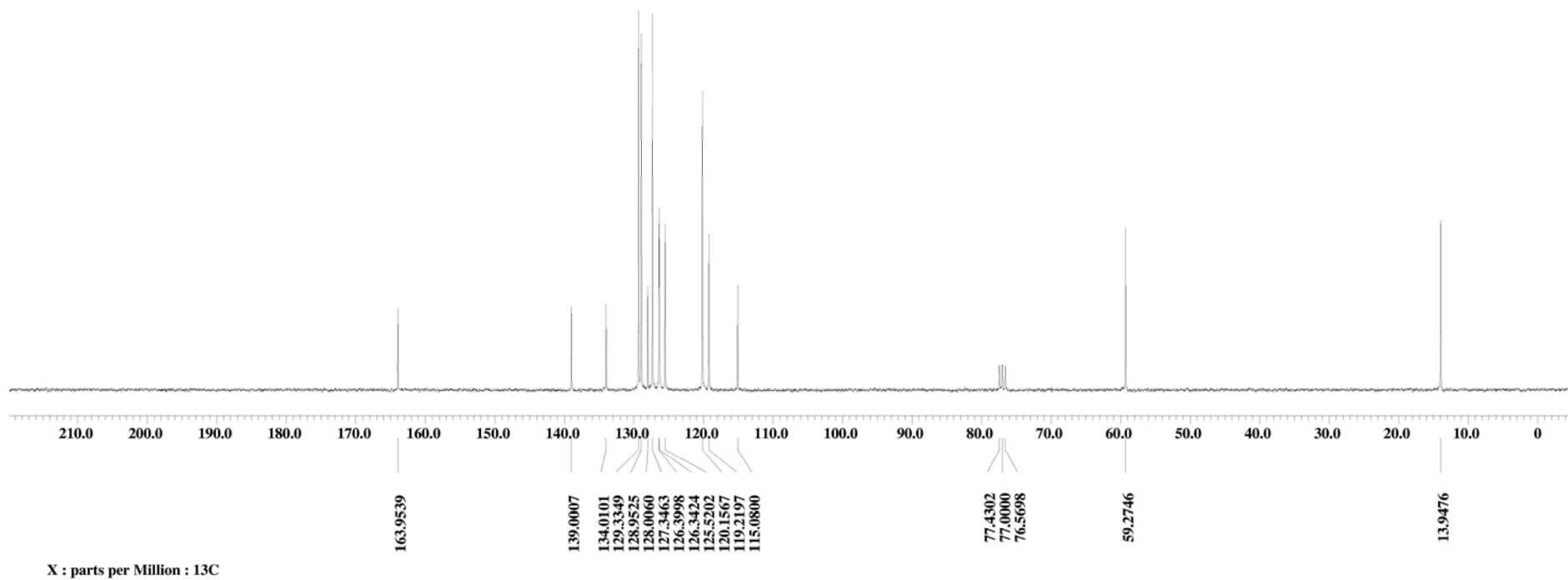
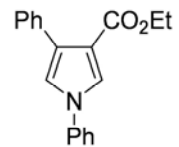
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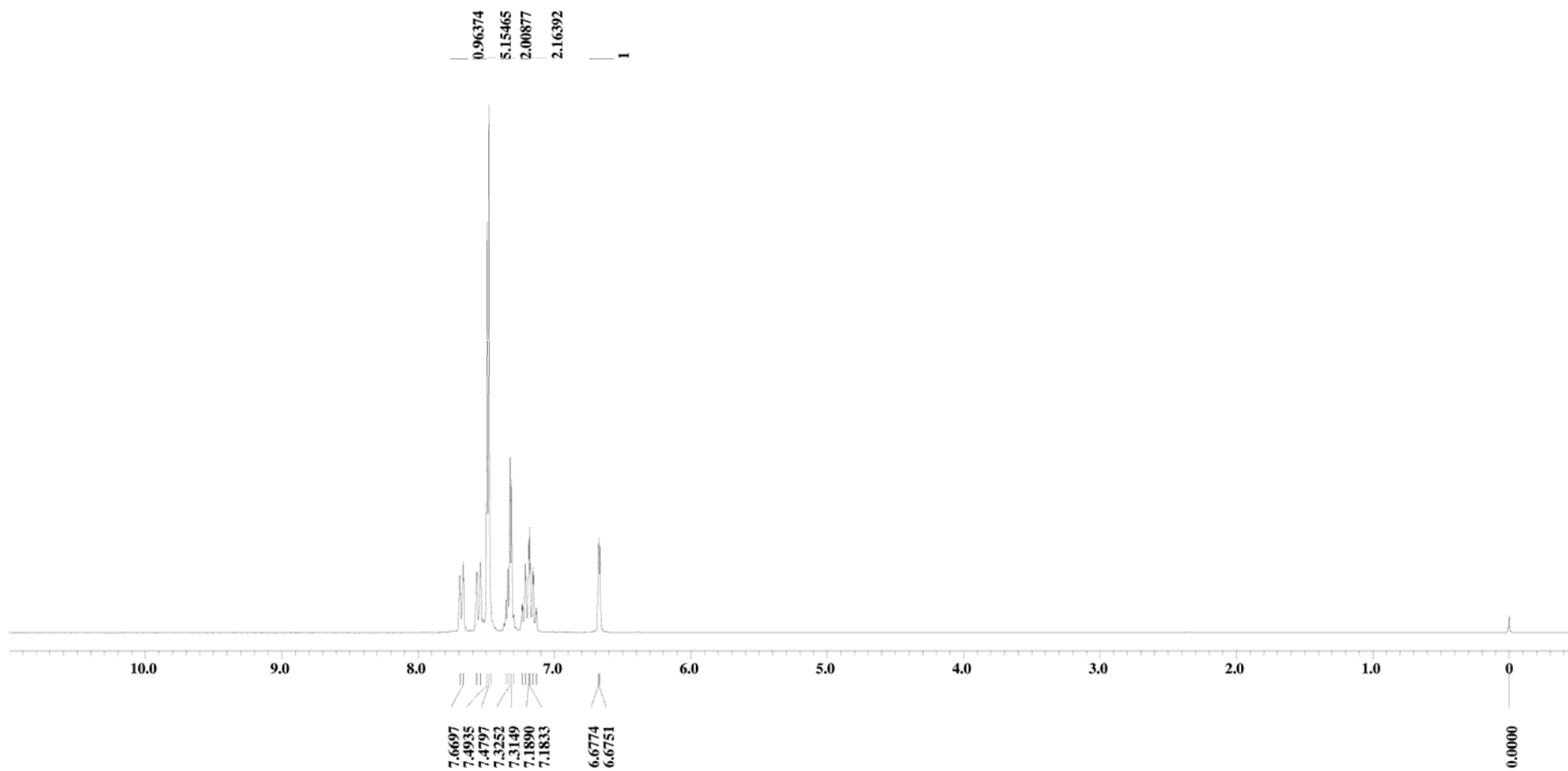
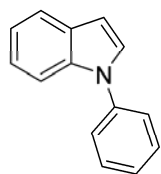


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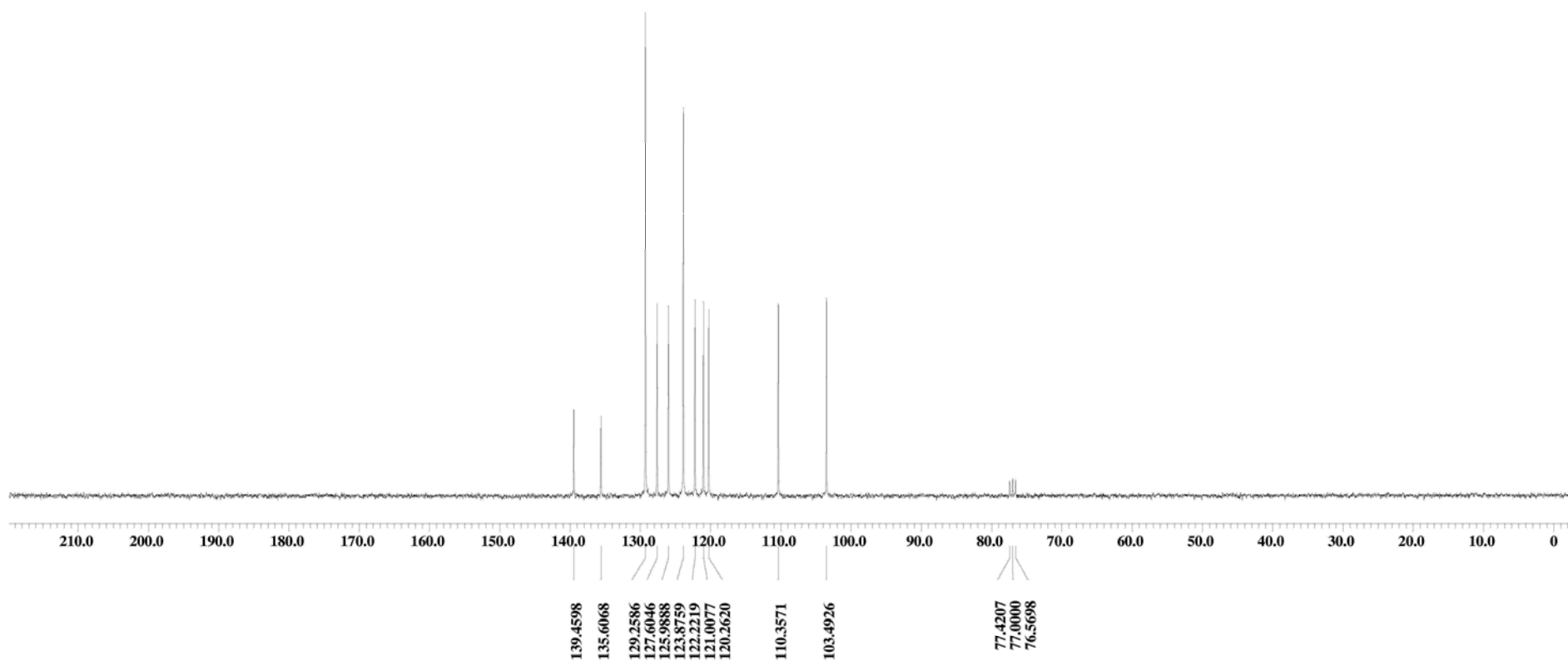
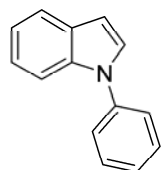


zr-070521-2-H-single\_pulse-2.jdf  
single\_pulse



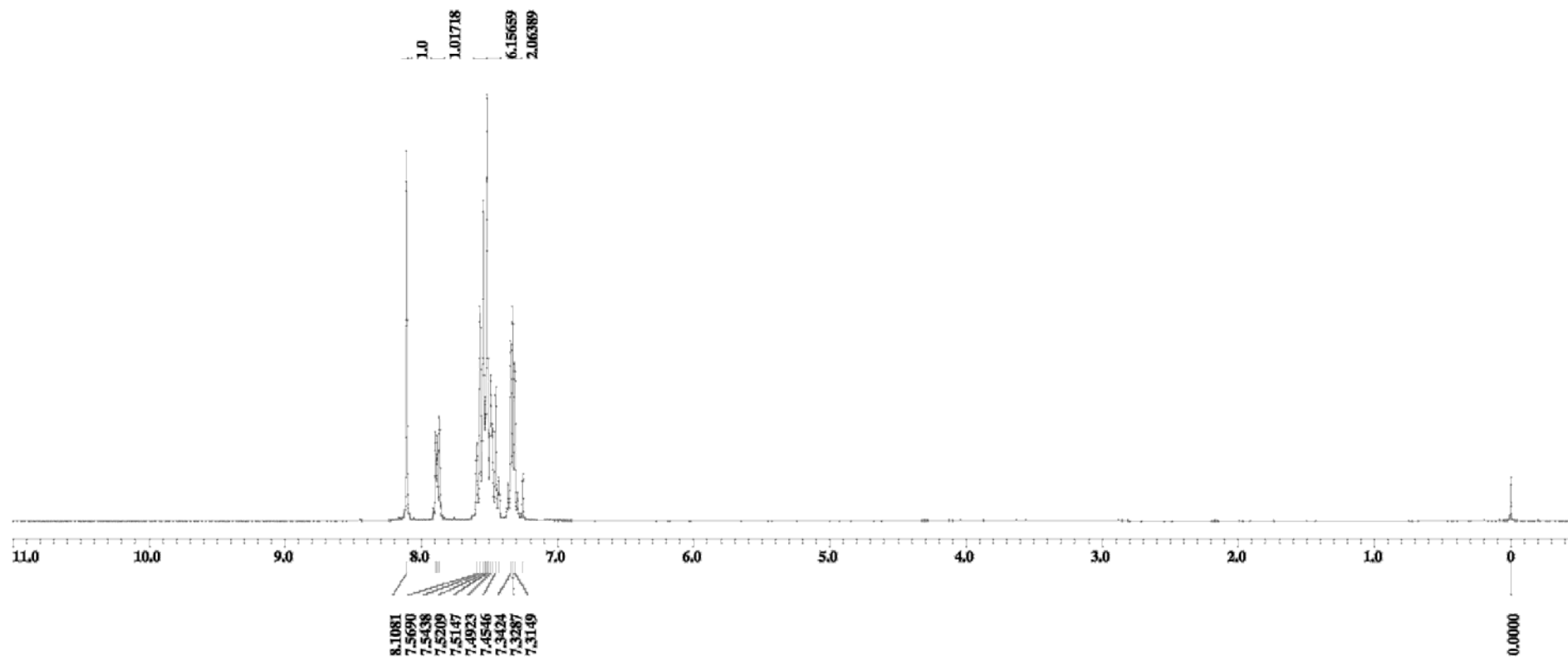
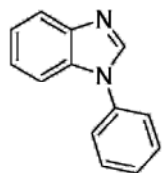
X : parts per Million : 1H

zr-070518-1-C-single\_pulse\_dec-2.jdf  
single pulse decoupled gated NOE



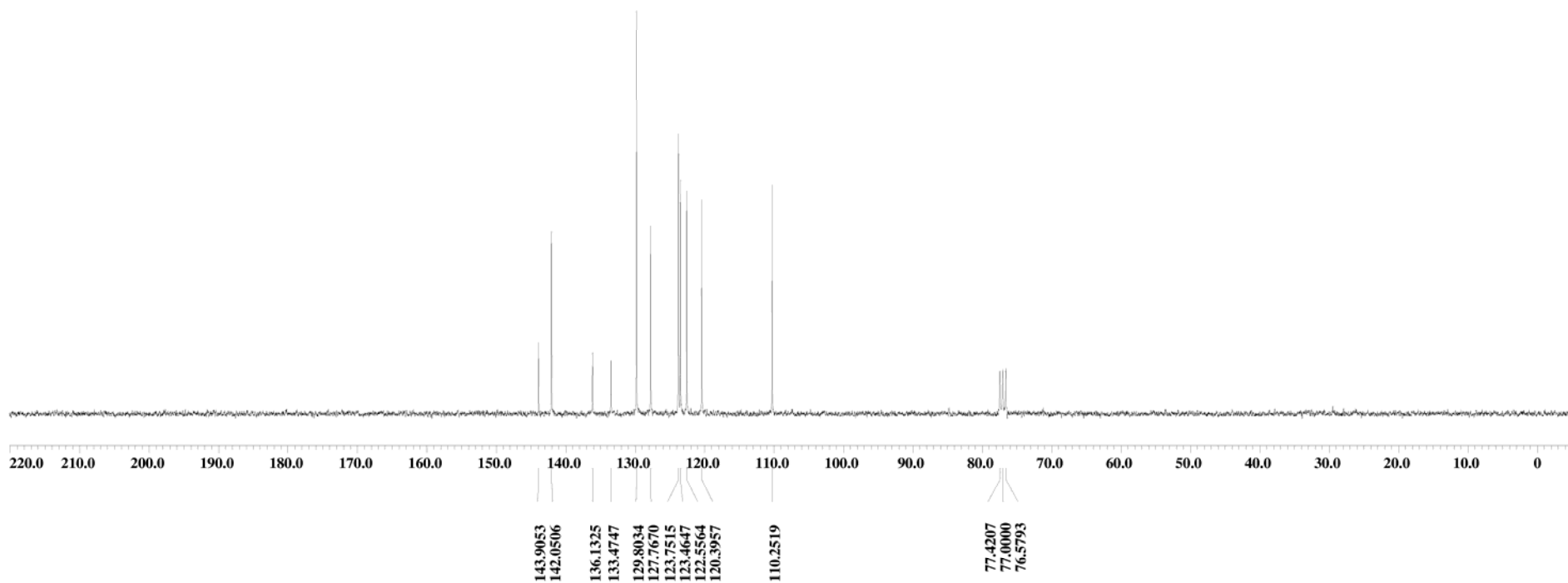
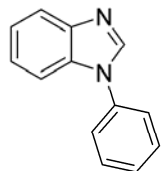
X : parts per Million : 13C

zr-070610-2-H-single\_pulse-2.jdf  
single\_pulse



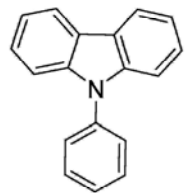
X : parts per Million : 1H

zr-070609-3-C-single\_pulse\_dec-2.jdf  
single pulse decoupled gated NOE

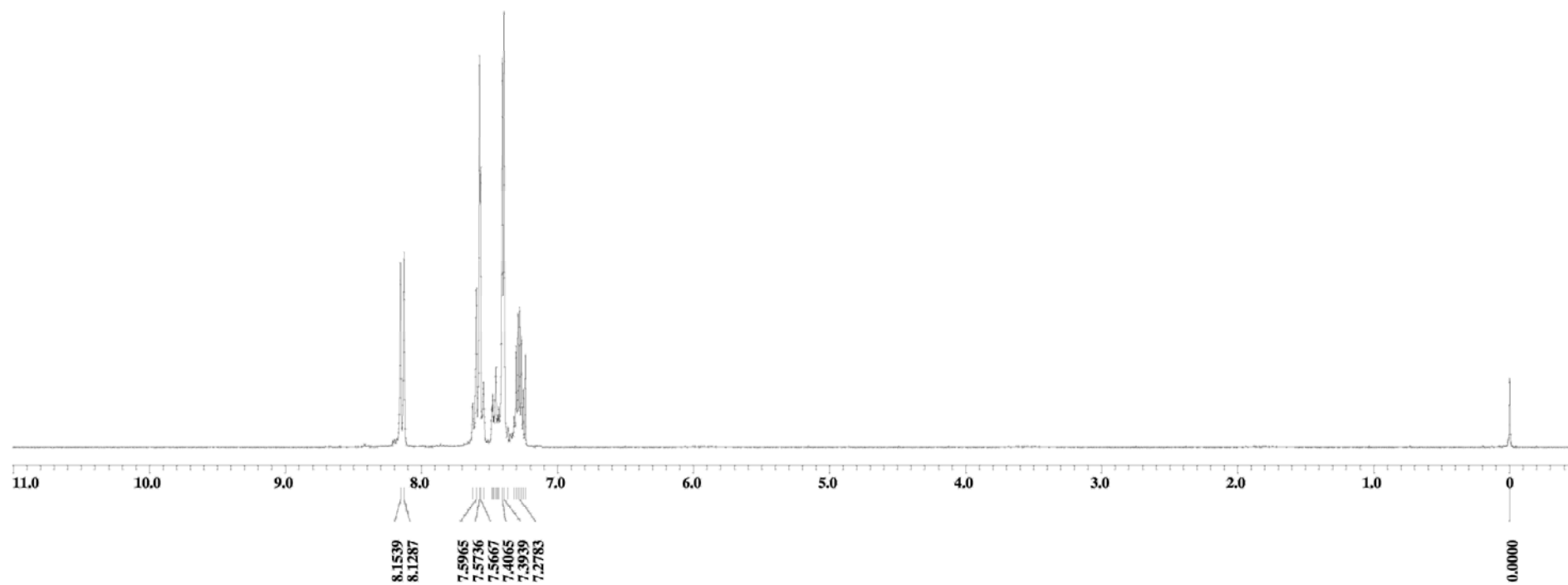


X : parts per Million : 13C

zr-070610-1-H-single\_pulse-2.jdf  
single\_pulse

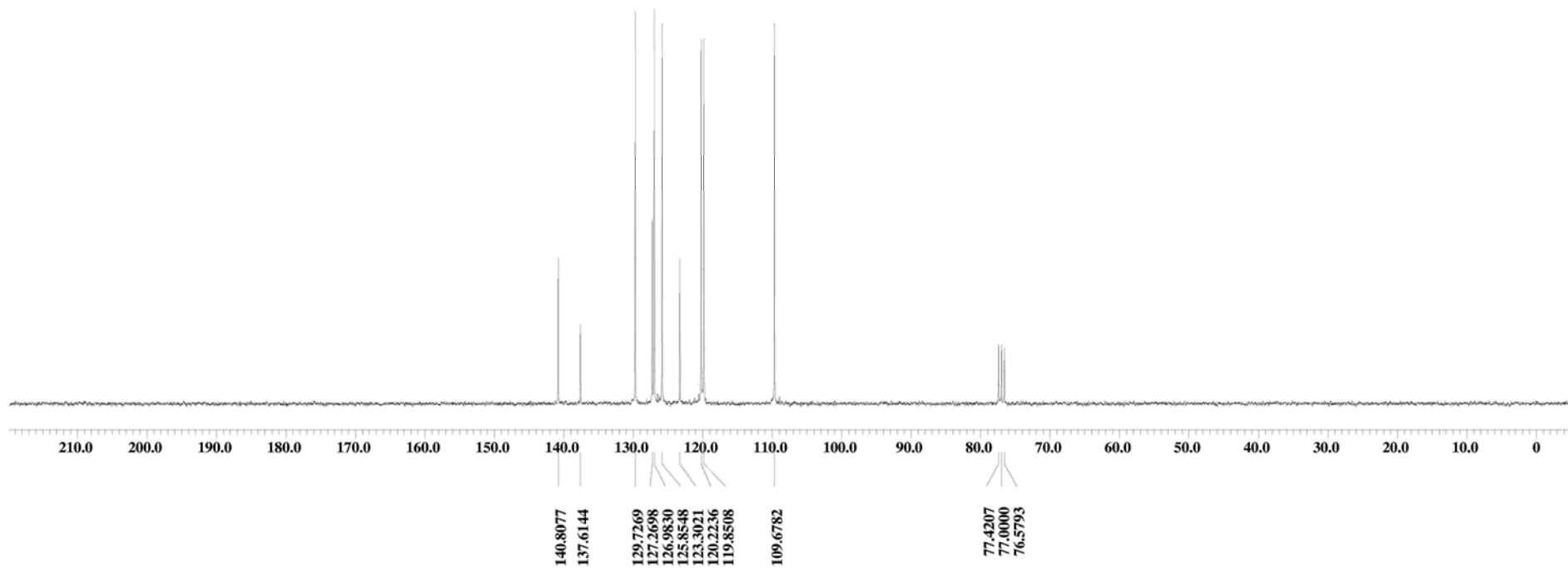
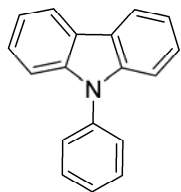


2  
4.02984  
4.98869  
2.11617



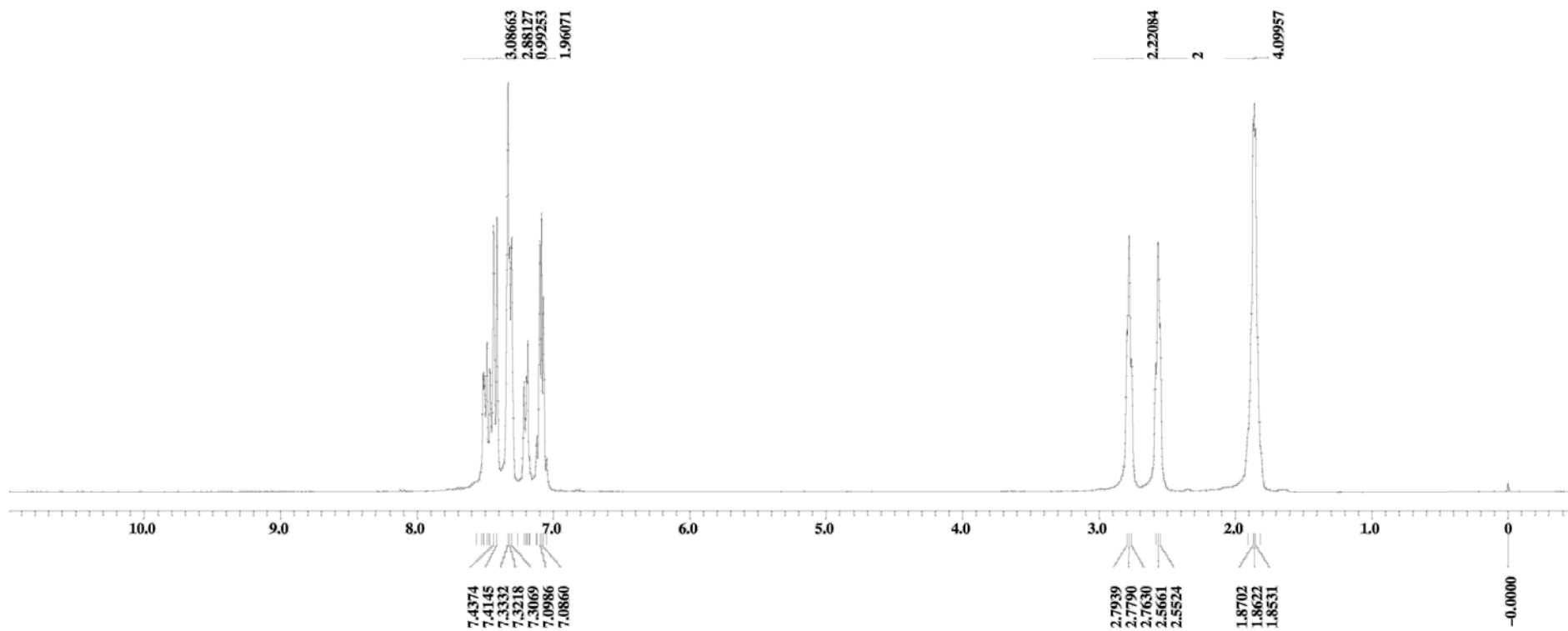
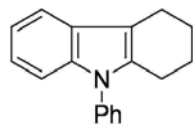
X : parts per Million : 1H

zr-070610-1-C-single\_pulse\_dec-2.jdf  
single pulse decoupled gated NOE



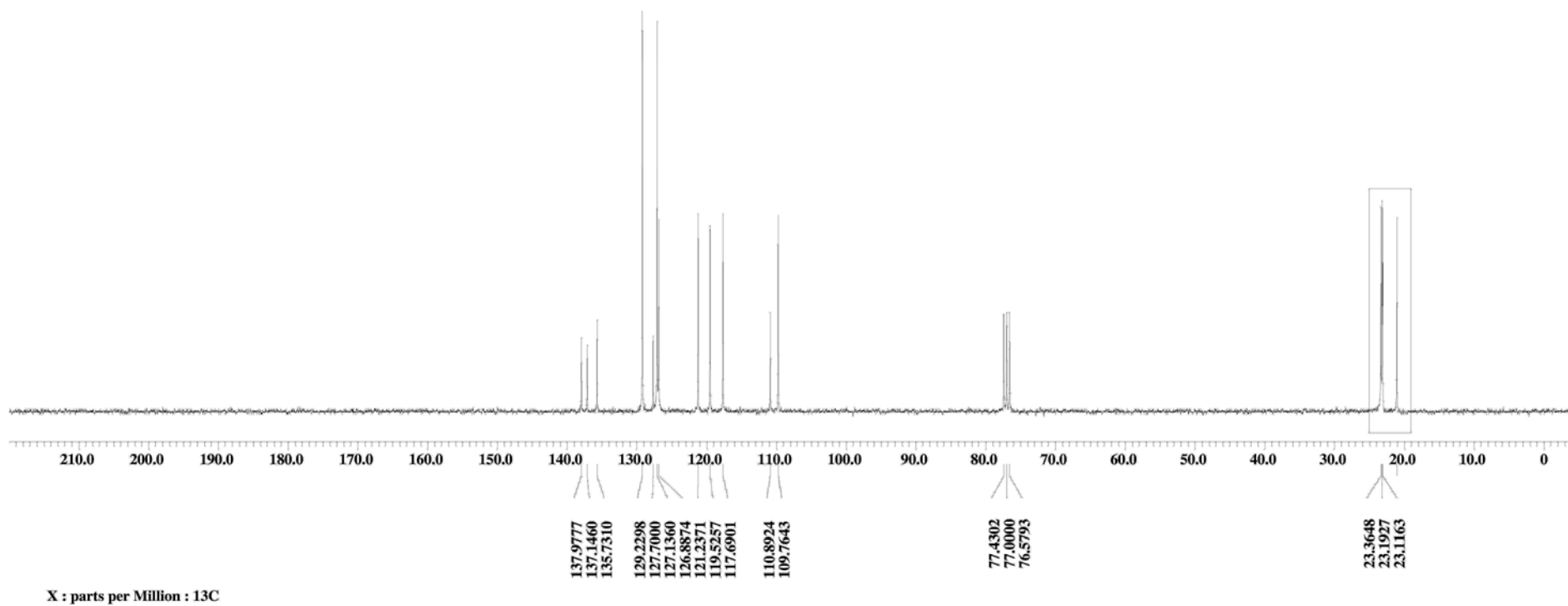
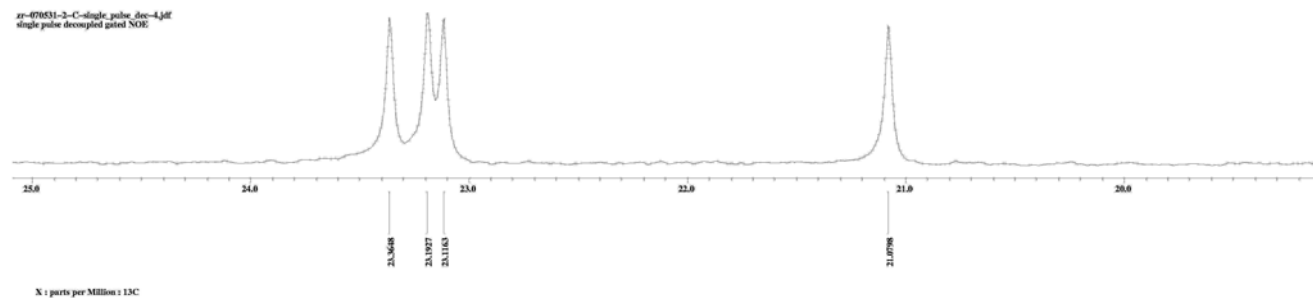
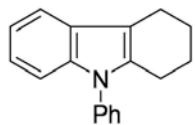
X : parts per Million : 13C

zr-070531-2-H-single\_pulse-3.jdf  
single\_pulse

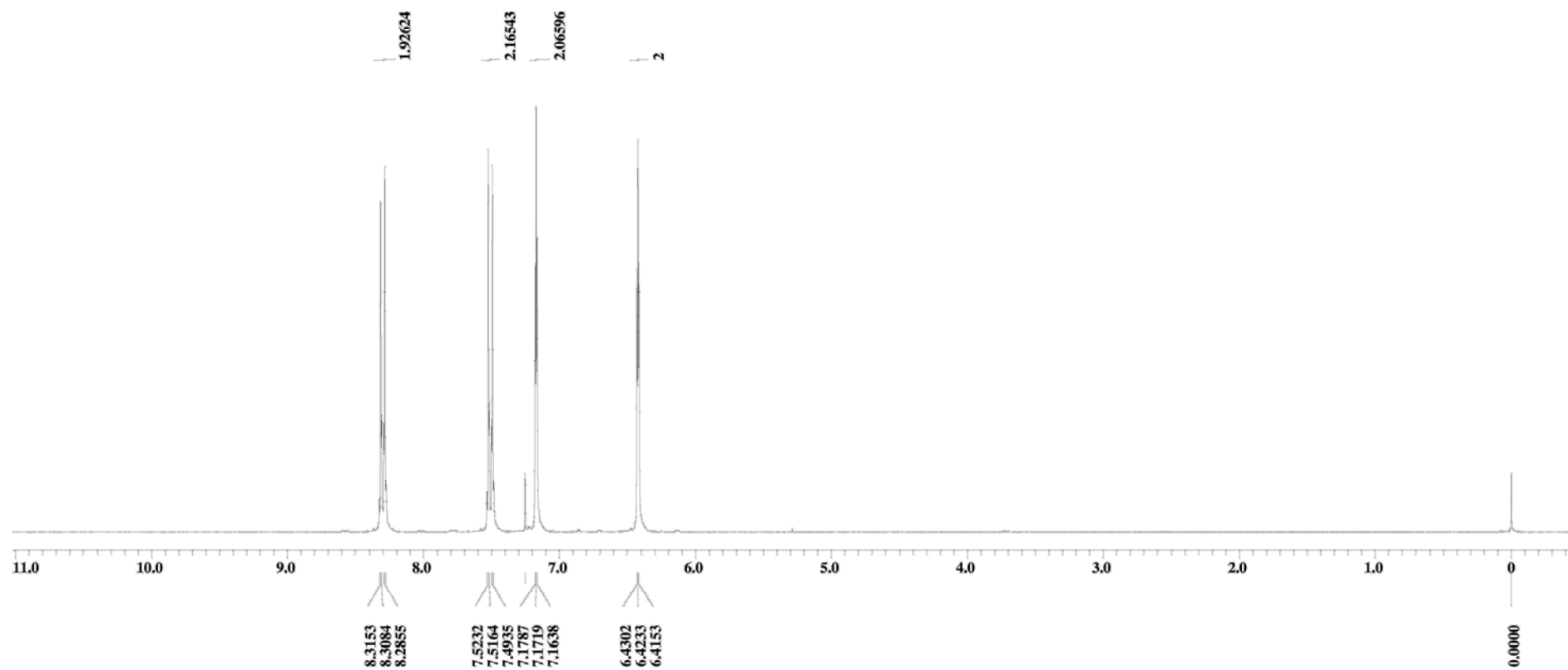
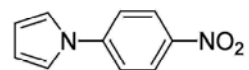


X : parts per Million : 1H

zr-070531-2-C-single\_pulse\_dec-4.jdf  
single pulse decoupled gated NOE

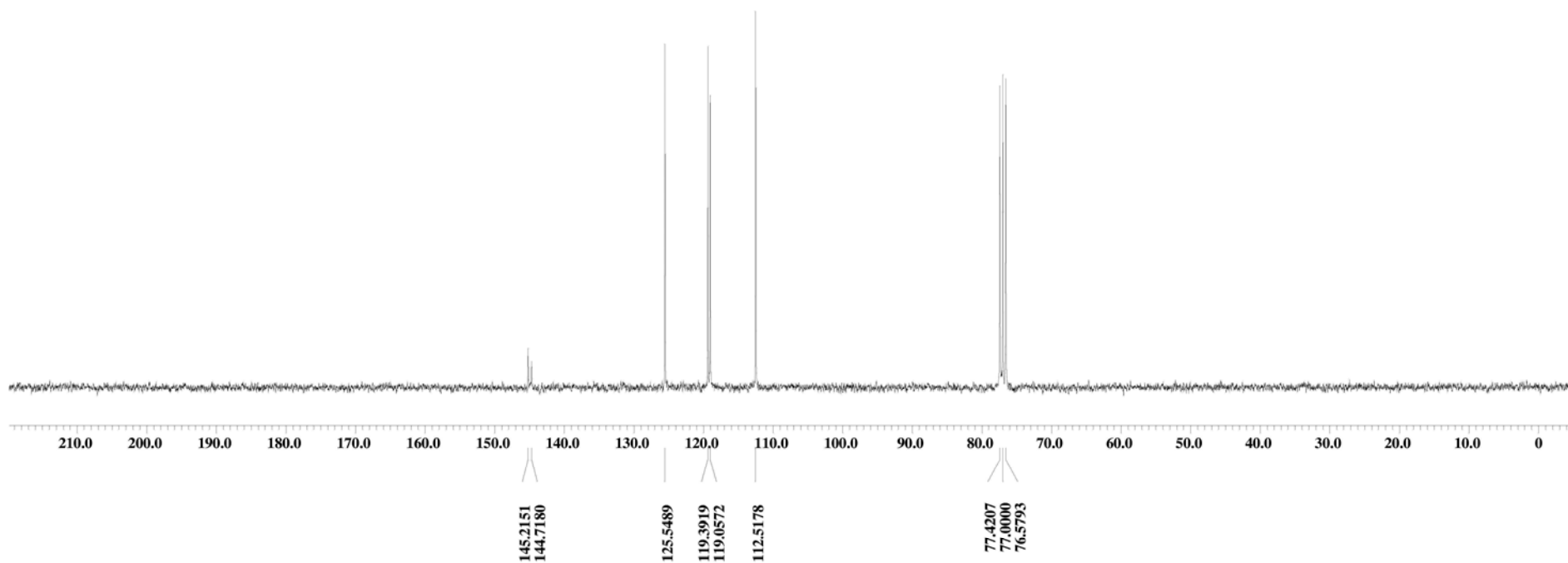
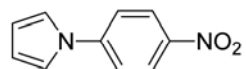


zr-070611-H-single\_pulse-2.jdf  
single\_pulse



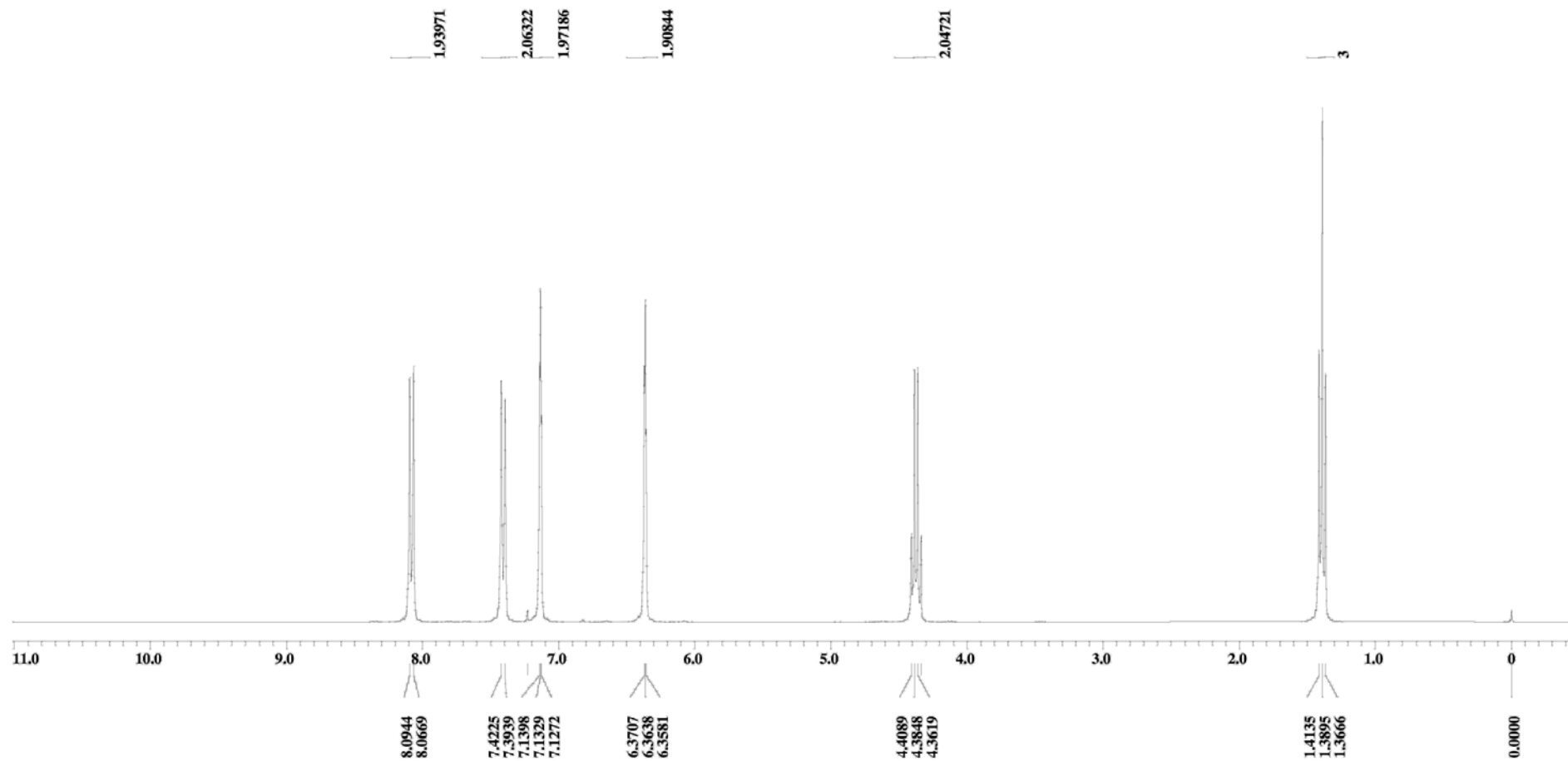
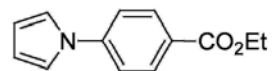
X : parts per Million : 1H

zr-070611-1-C-single\_pulse\_dec-3.jdf  
single pulse decoupled gated NOE



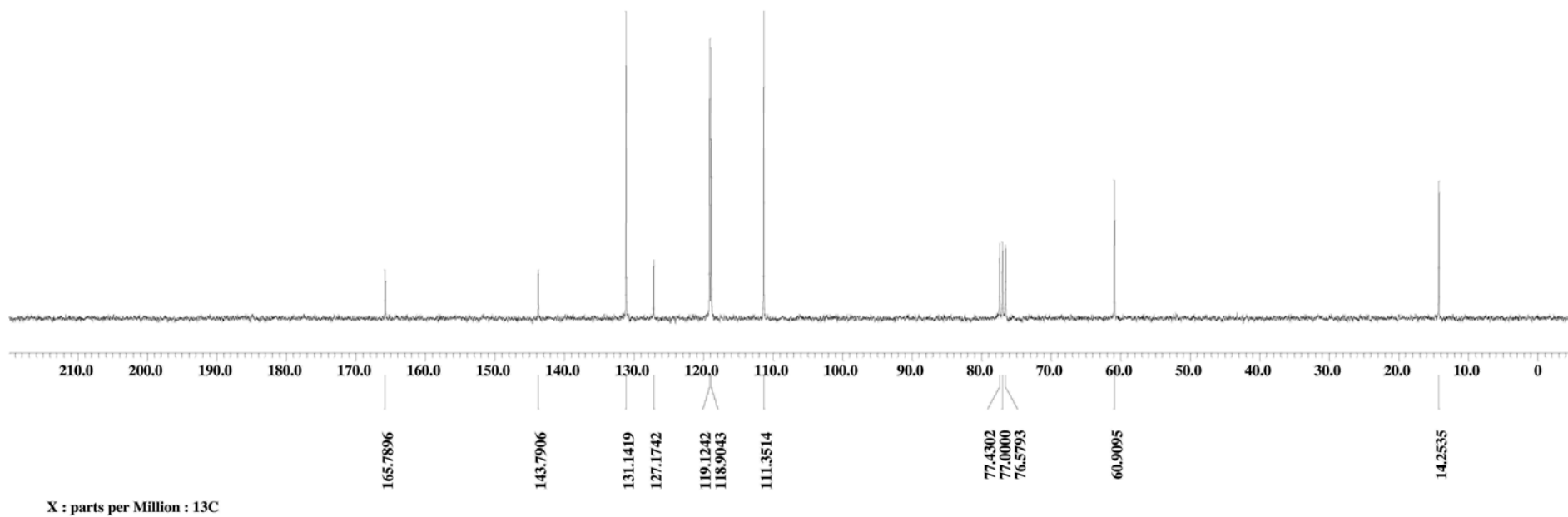
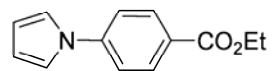
X : parts per Million : 13C

zr-070523-1-H-single\_pulse-2.jdf  
single\_pulse

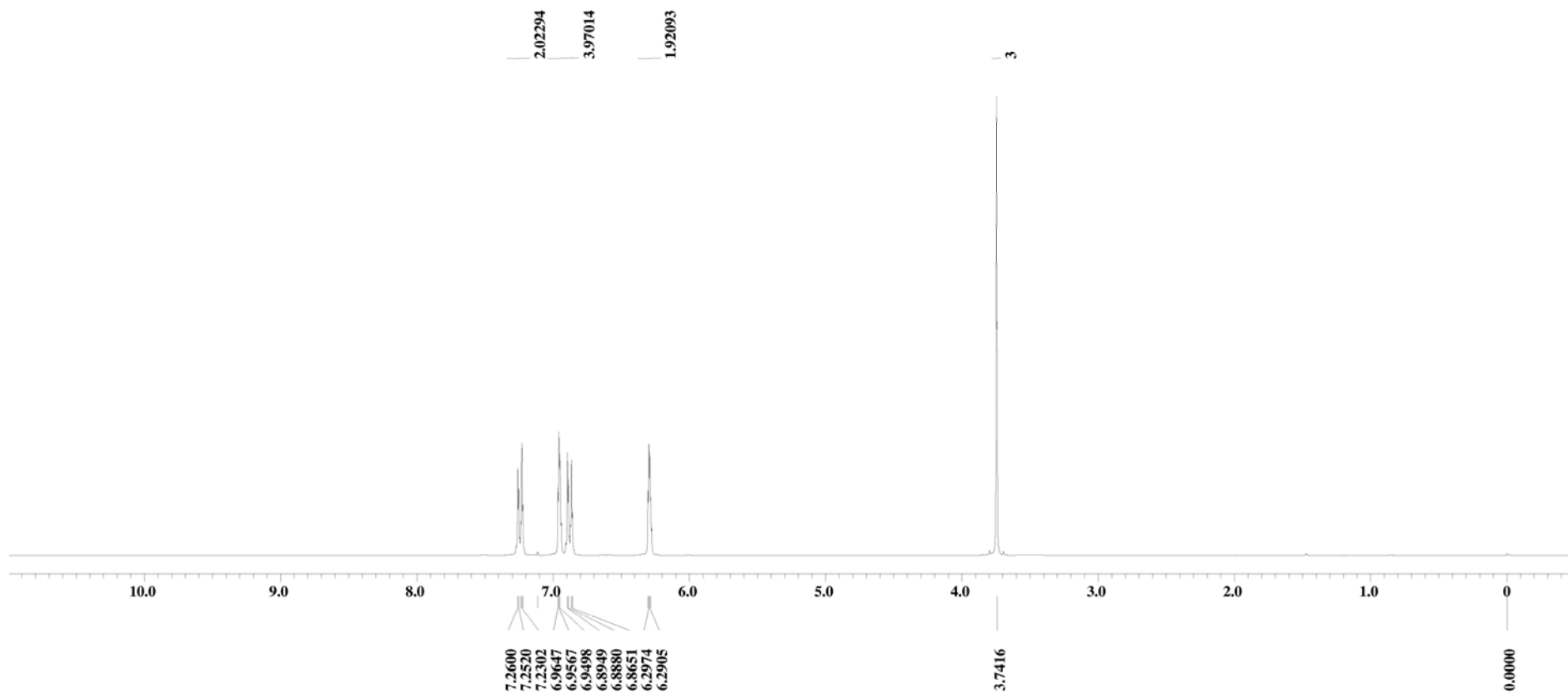
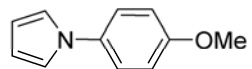


X : parts per Million : 1H

zr-070523-1-C-single\_pulse\_dec-2.jdf  
single pulse decoupled gated NOE

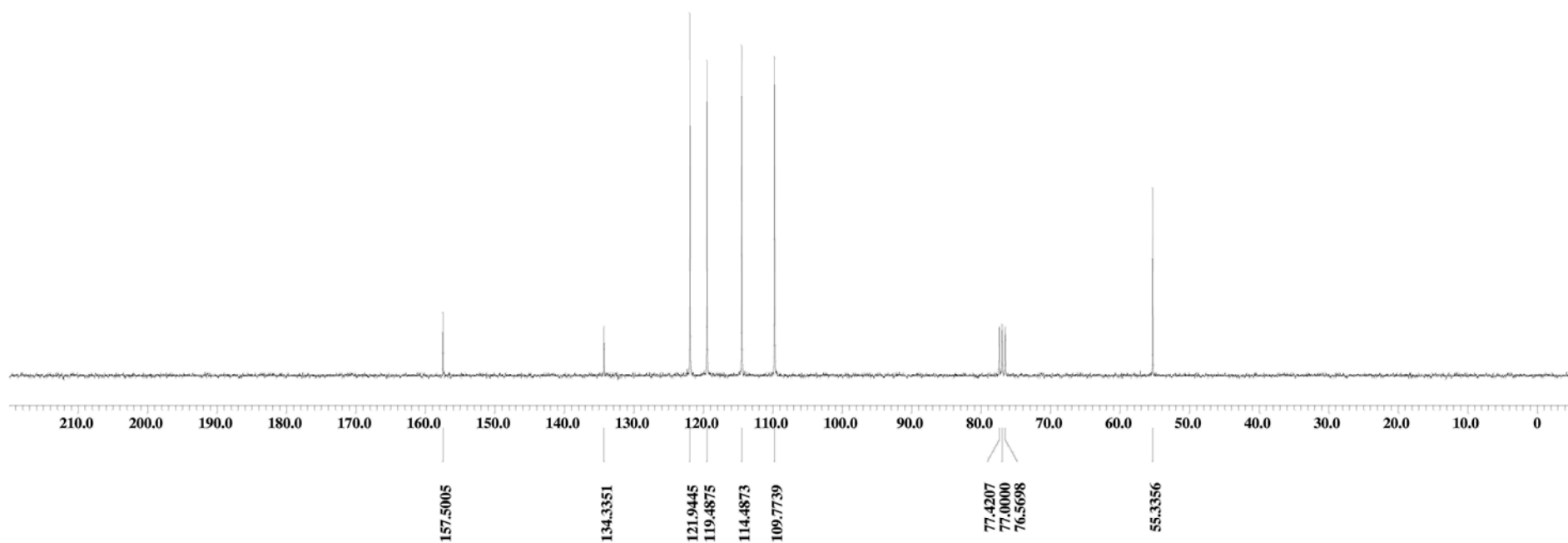
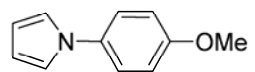


zr-070522-1-H-single\_pulse-2.jdf  
single\_pulse



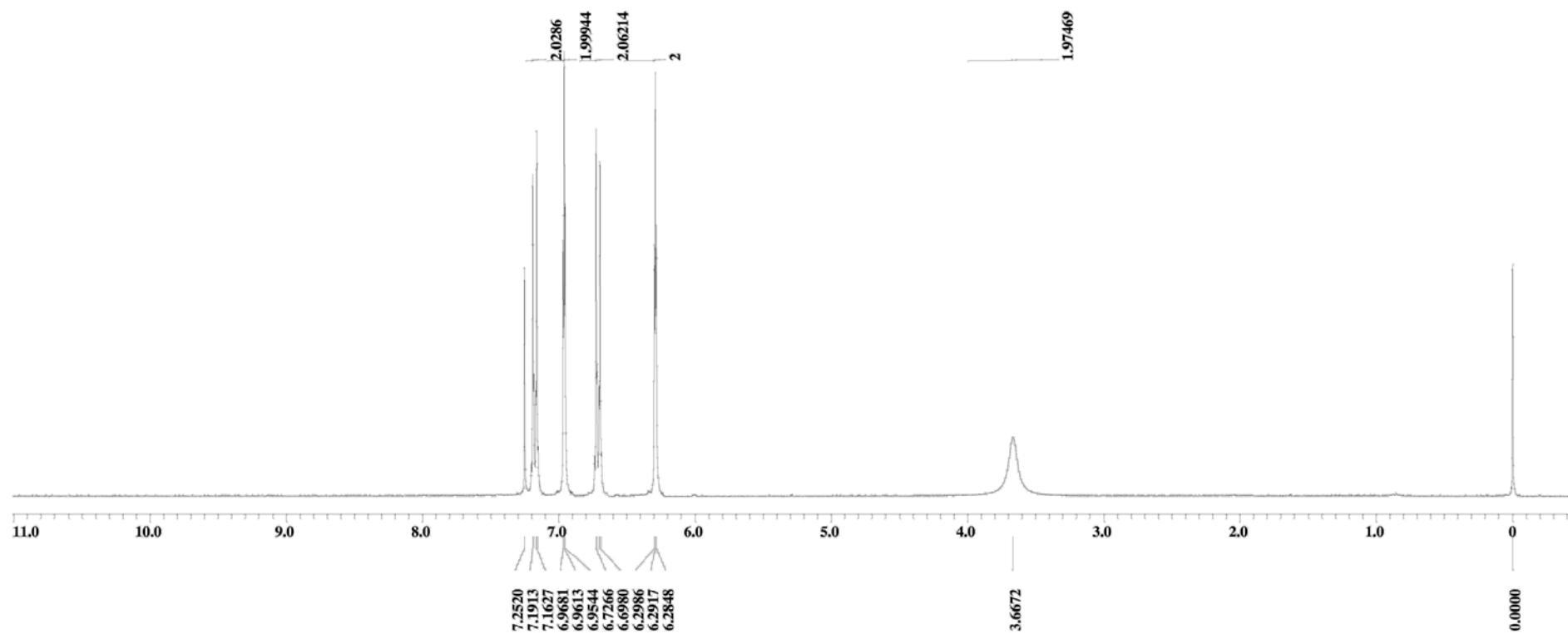
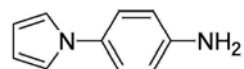
X : parts per Million : 1H

zr-070522-1-C-single\_pulse\_dec-2.jdf  
single pulse decoupled gated NOE



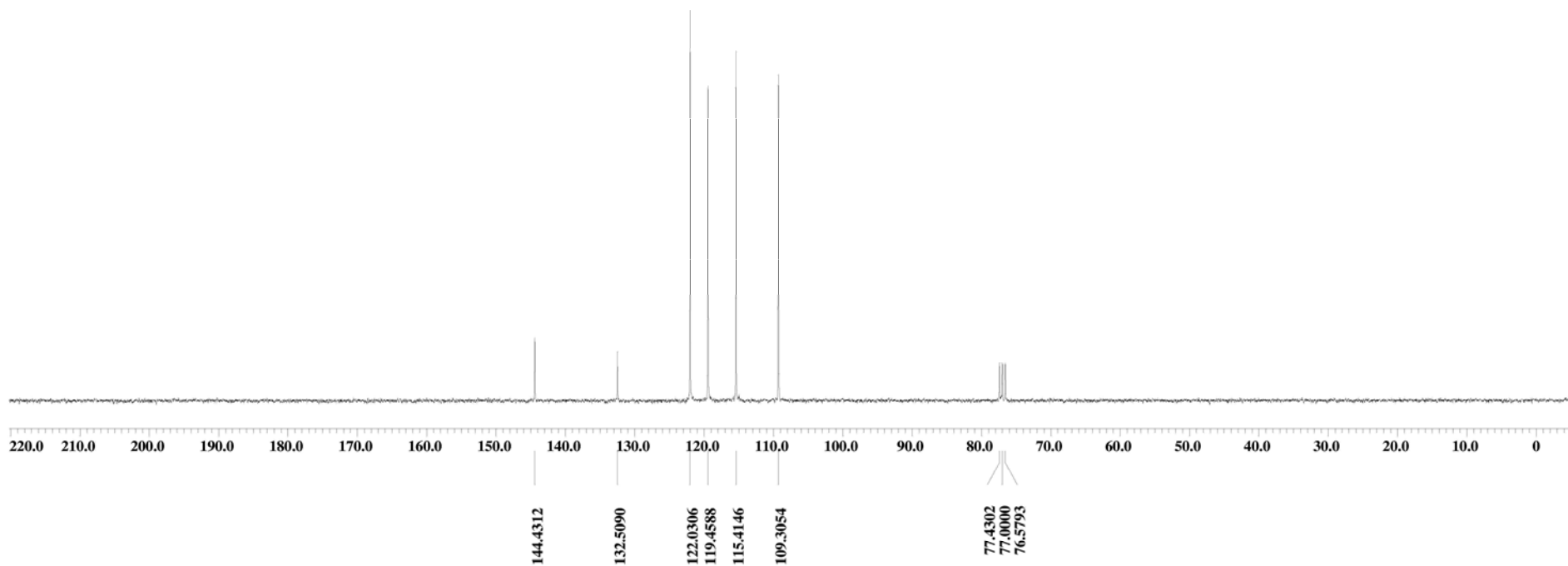
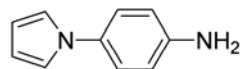
X : parts per Million : 13C

zr-070627-1-H-single\_pulse-2.jdf  
single\_pulse



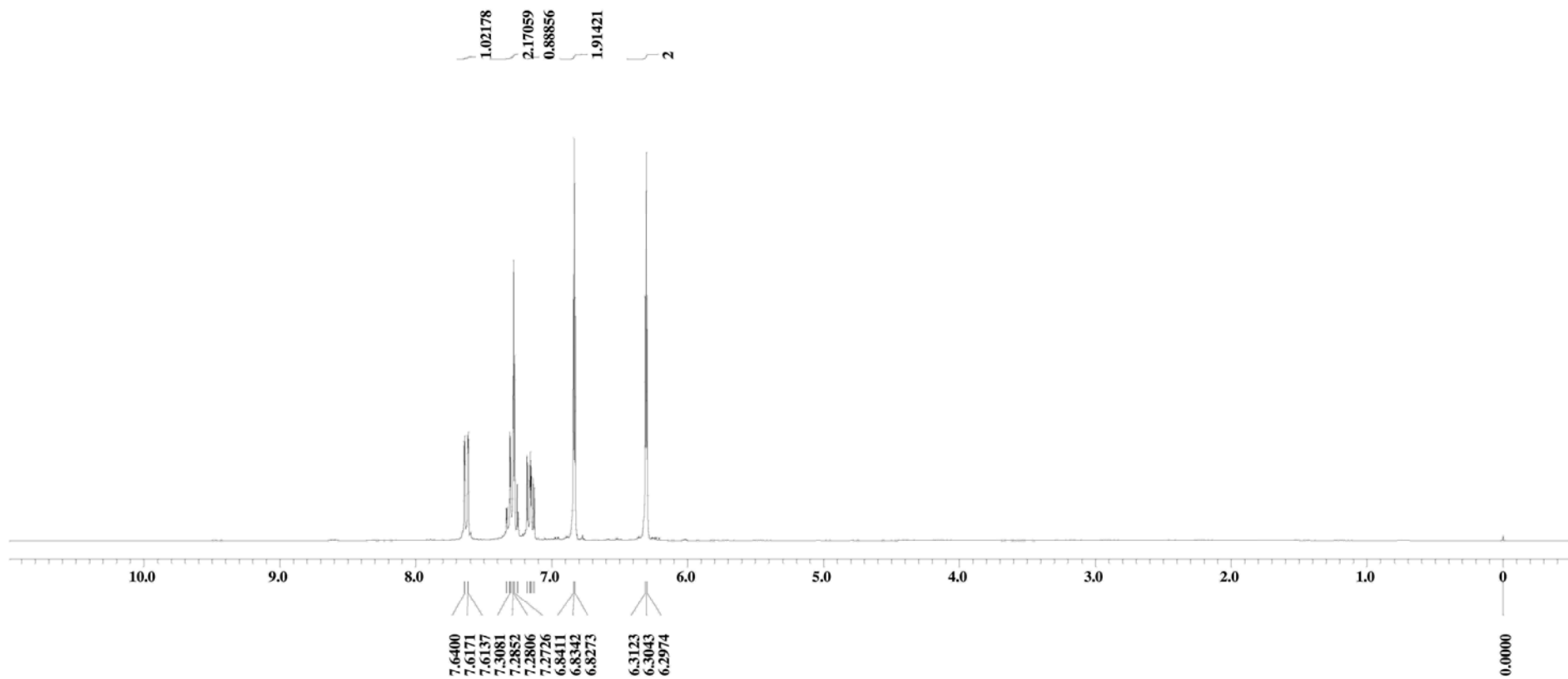
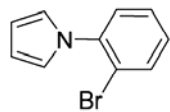
X : parts per Million : 1H

zr-070625-2-C-single\_pulse\_dec-2.jdf  
single pulse decoupled gated NOE



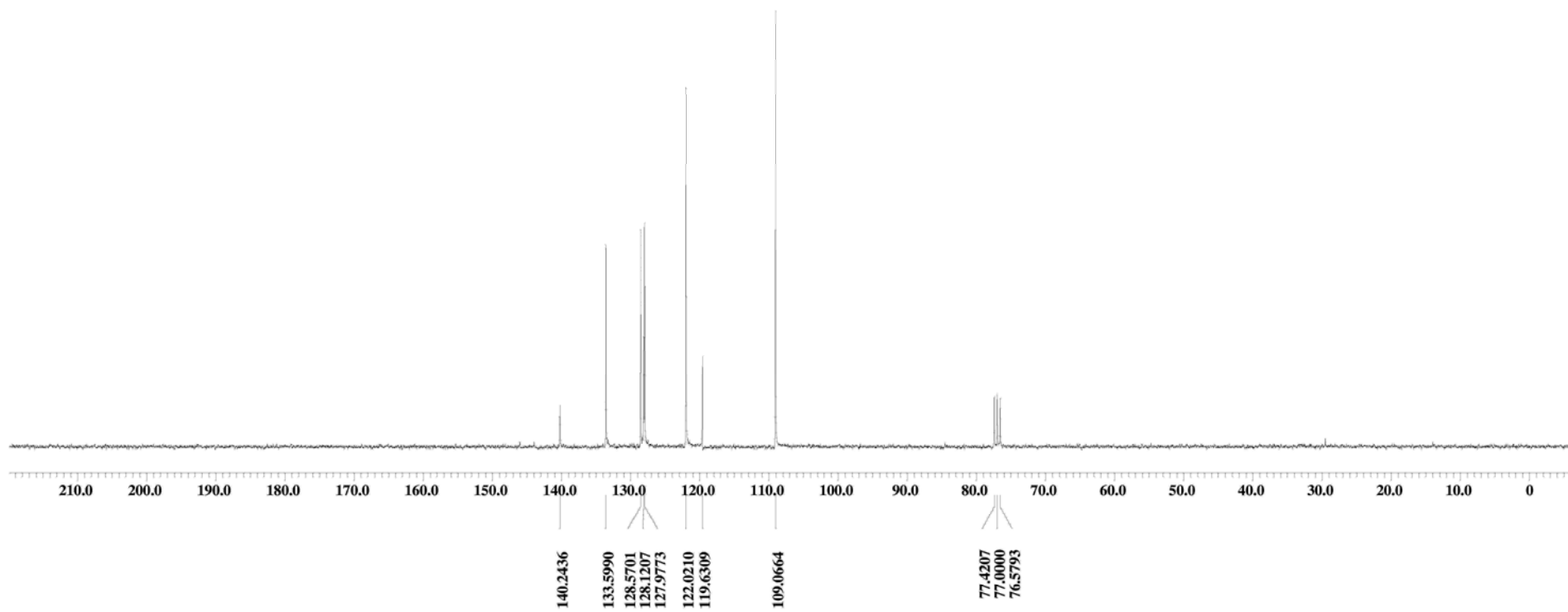
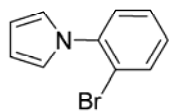
X : parts per Million : 13C

zr-070608-01-H-single\_pulse-3.jdf  
single\_pulse



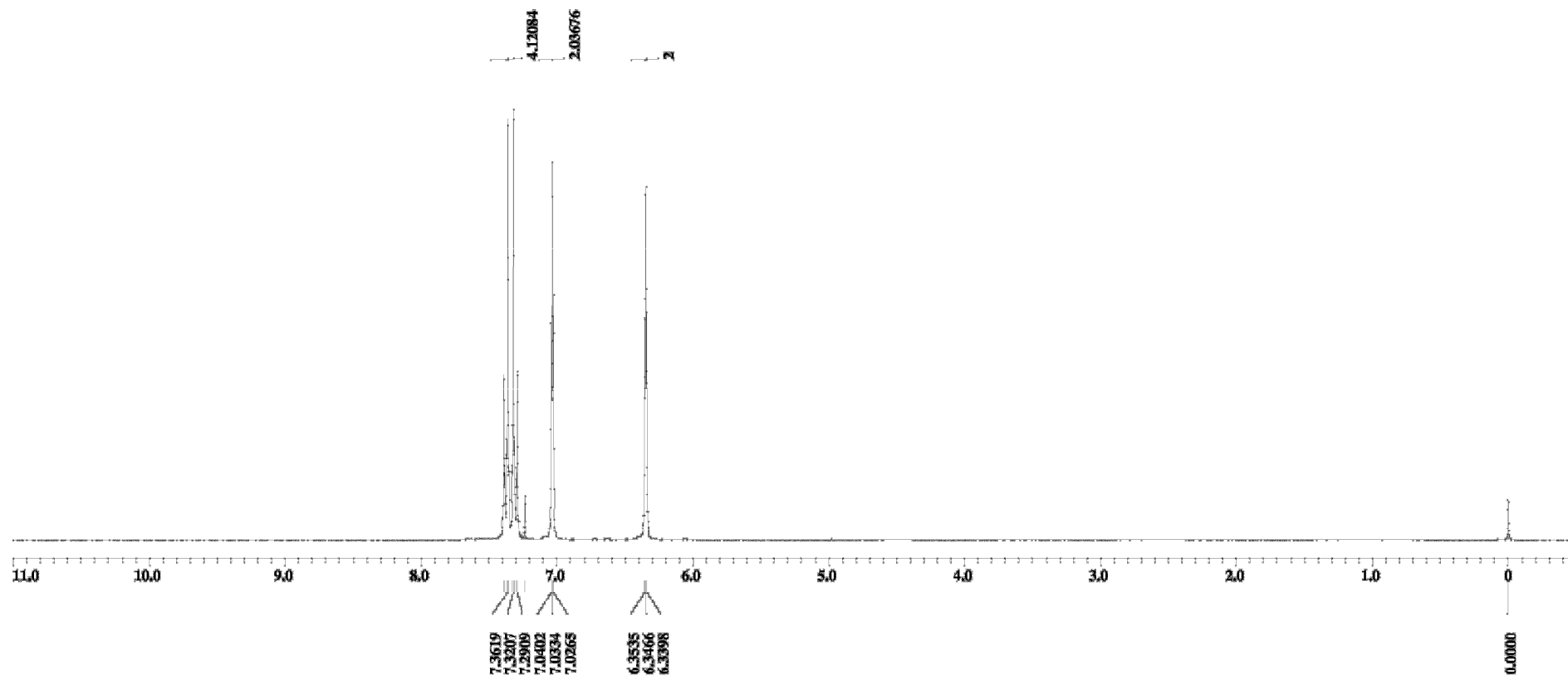
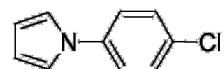
X : parts per Million : 1H

zr-070608-01-single\_pulse\_dec-5.jdf  
single pulse decoupled gated NOE



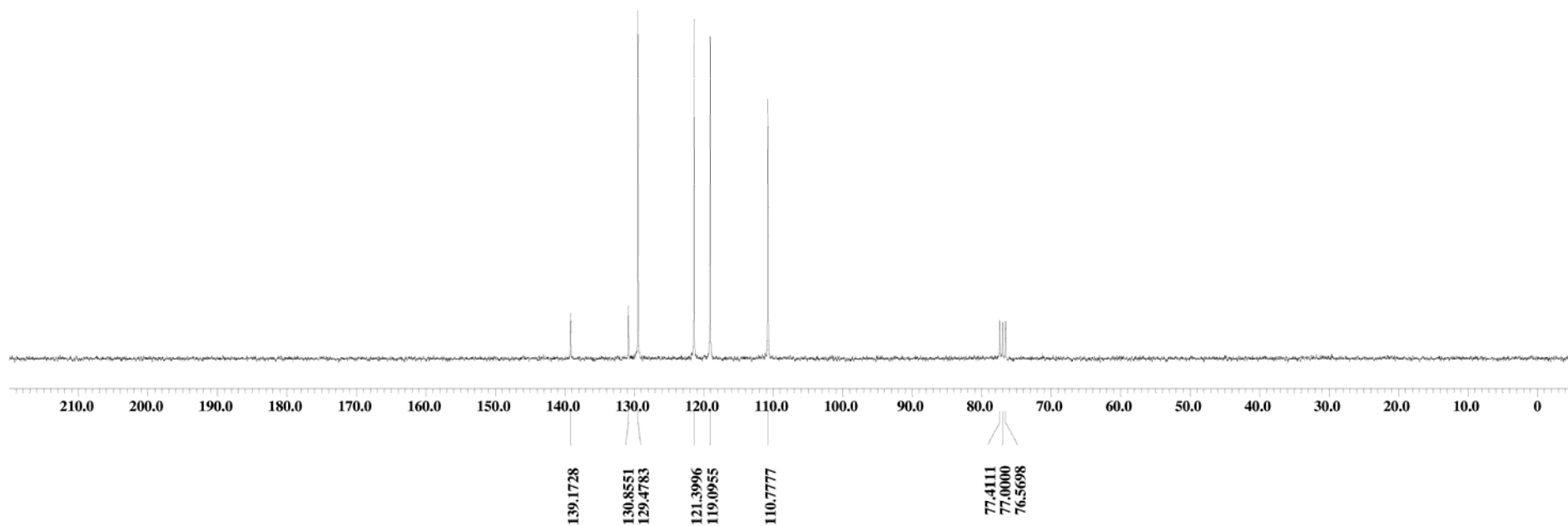
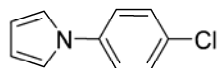
X : parts per Million : 13C

nr-070610-3-H-single\_pulse-2.jdf  
single\_pulse



X : parts per Million : 1H

zr-070609-4-C-single\_pulse\_dec-2.jdf  
single pulse decoupled gated NOE



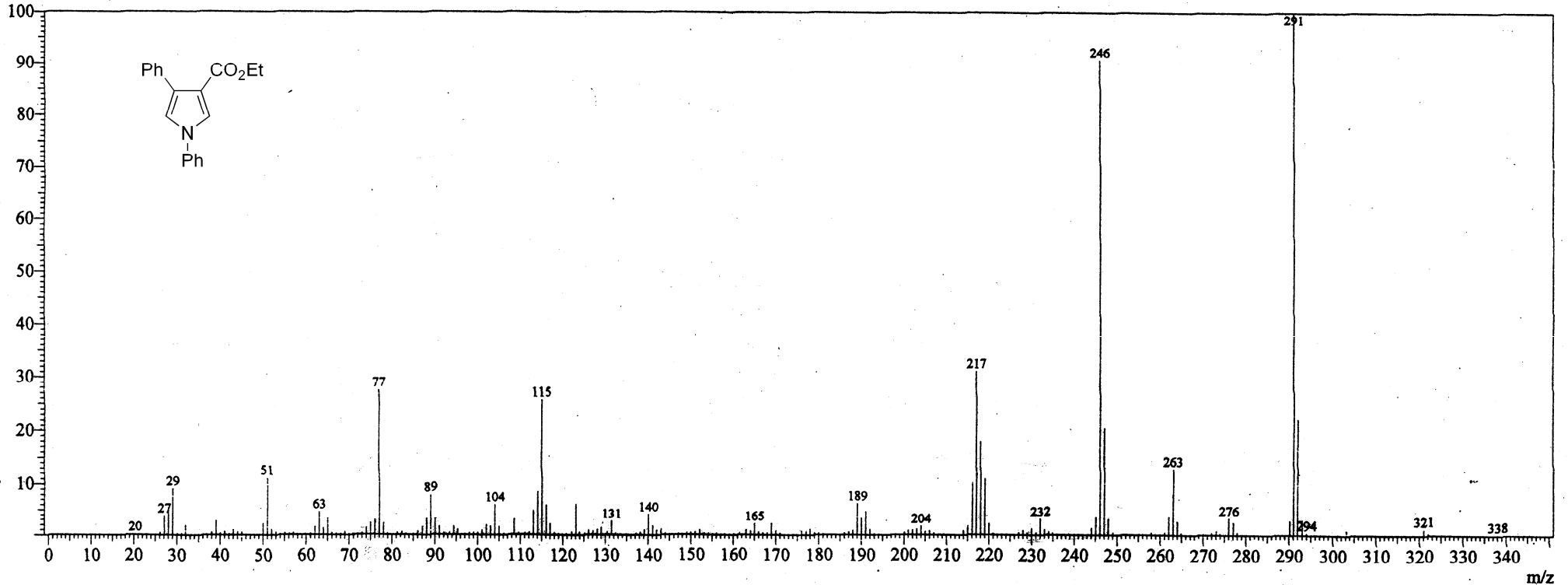
X : parts per Million : 13C

Sample Information

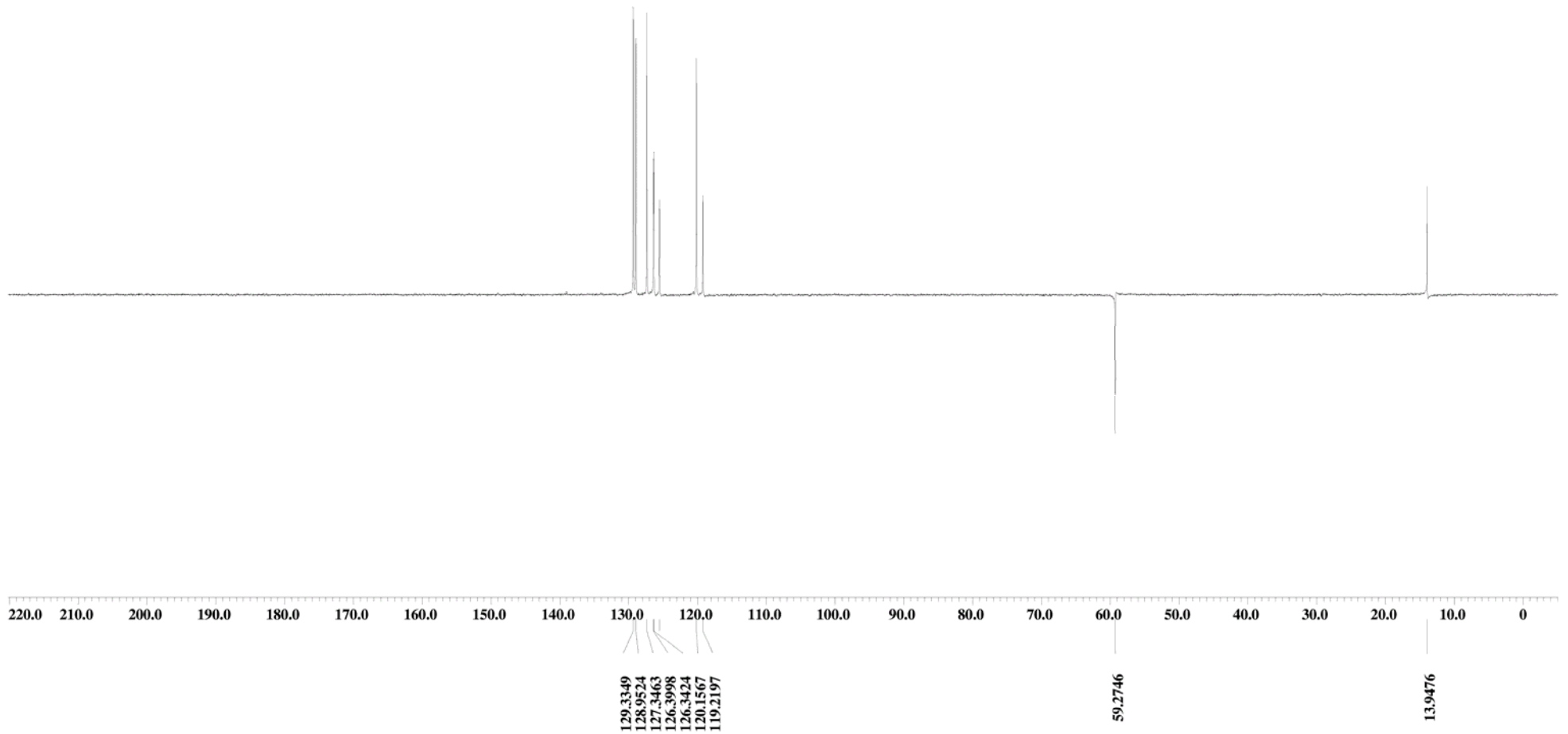
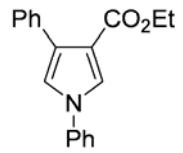
Analyzed by : Admin  
Analyzed : 2006-10-19 11:04:18  
Sample Type : Unknown  
Level # : 1  
Sample Name : 清华大学化学系 胡跃飞 朱锐 9#  
Sample ID :  
IS Amount : [1]=1.000  
Sample Amount : 1.000  
Dilution Factor : 1.000  
Vial # : 7  
Injection Volume : 1.000  
Data File : C:\GCMSsolution\Data\2006File F\061019-10.QGD  
Org Data File : C:\GCMSsolution\Data\2006File F\061019-10.QGD  
Method File : C:\GCMSsolution\Data\2006file F\061019-2.qgm  
Org Method File : C:\GCMSsolution\Data\2006file F\061019-2.qgm  
Report File :  
Tuning File : C:\GCMSsolution\System\Tune1\EI\_20060713.qgt  
Modified by : Admin  
Modified : 2006-10-19 11:08:21

Spectrum

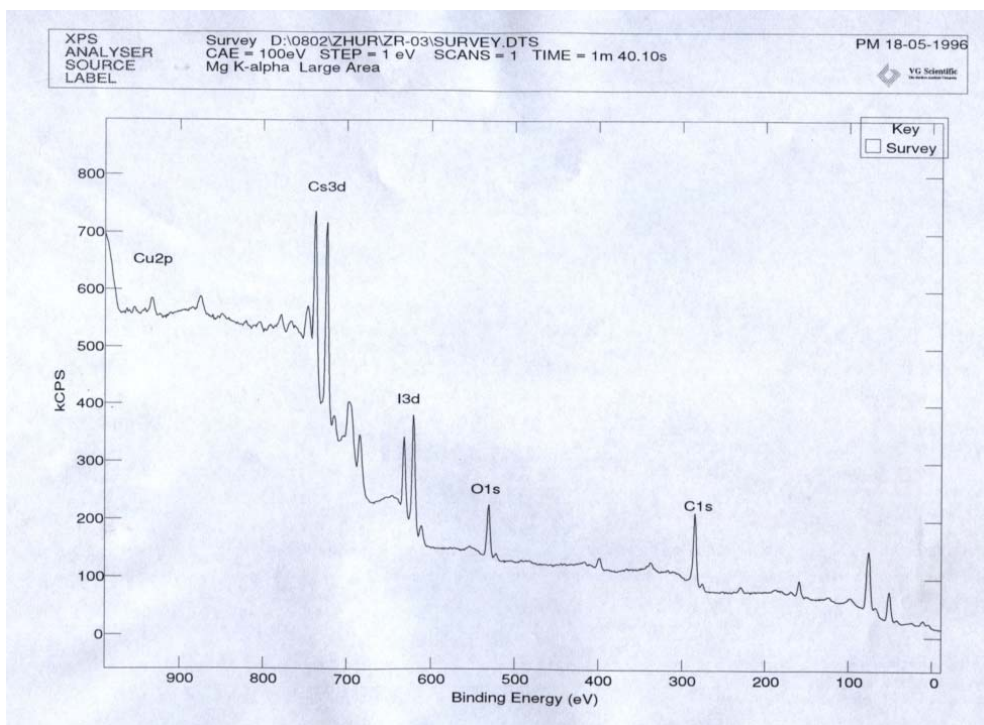
Line#:1 R.Time:0.2(Scan#:1)  
MassPeaks:211 BasePeak:291(153972)  
RawMode:Single 0.2(1)  
BG Mode:0.5(21)



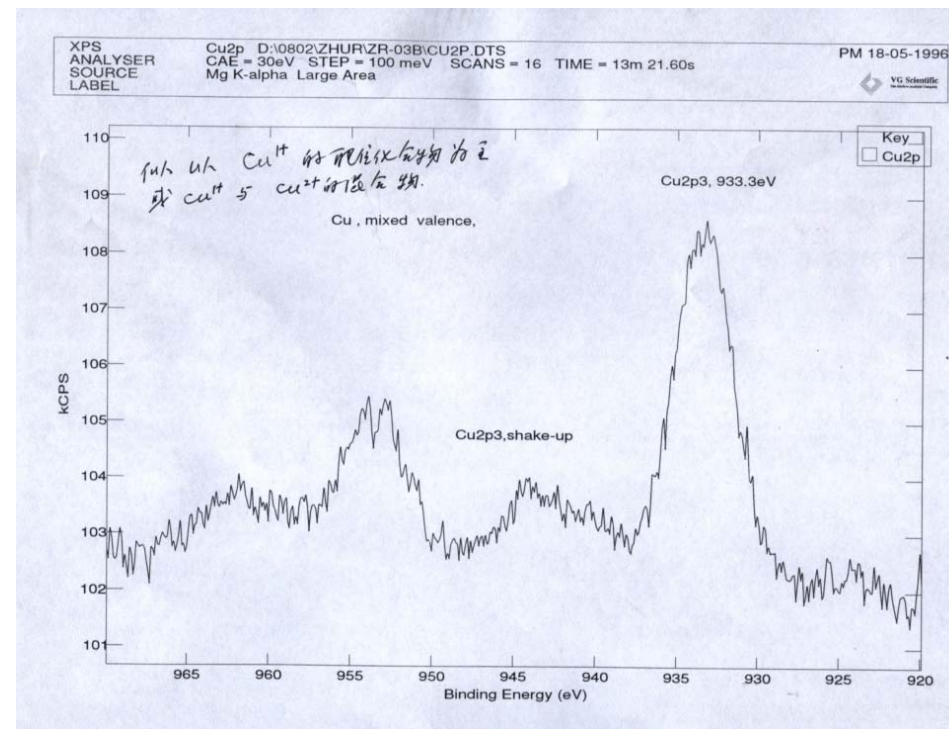
zr-070713-1-dept-3.jdf  
DEPT with decoupling



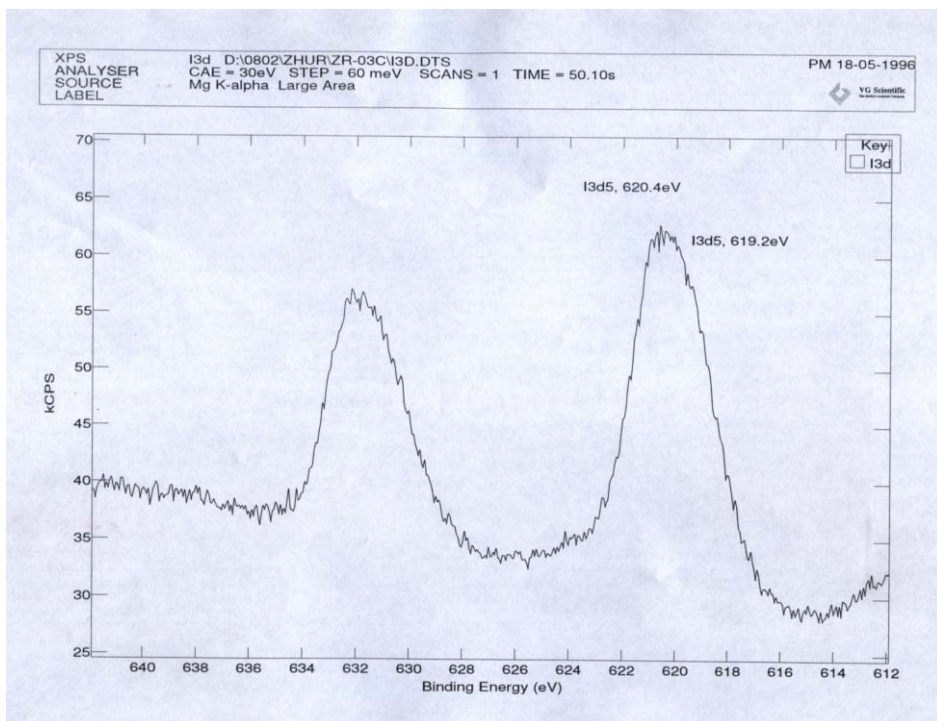
X : parts per Million : 13C



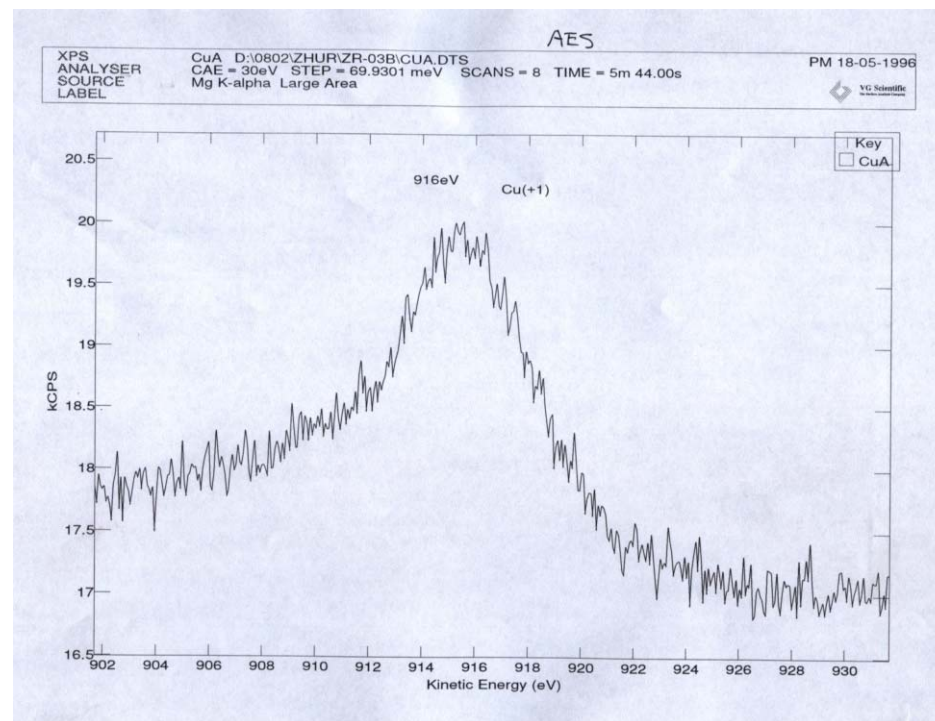
(a) XPS survey spectrum for the sample prepared from the reaction residue of entry 10 in Table 1.



(b) XPS spectrum of Cu2P.



(c) XPS spectrum zoomed on I3d.



(d) AES spectrum for Cu<sup>1+</sup>.