

## **Staudinger/aza-Wittig reaction to access $N\beta$ -protected amino alkyl isothiocyanates**

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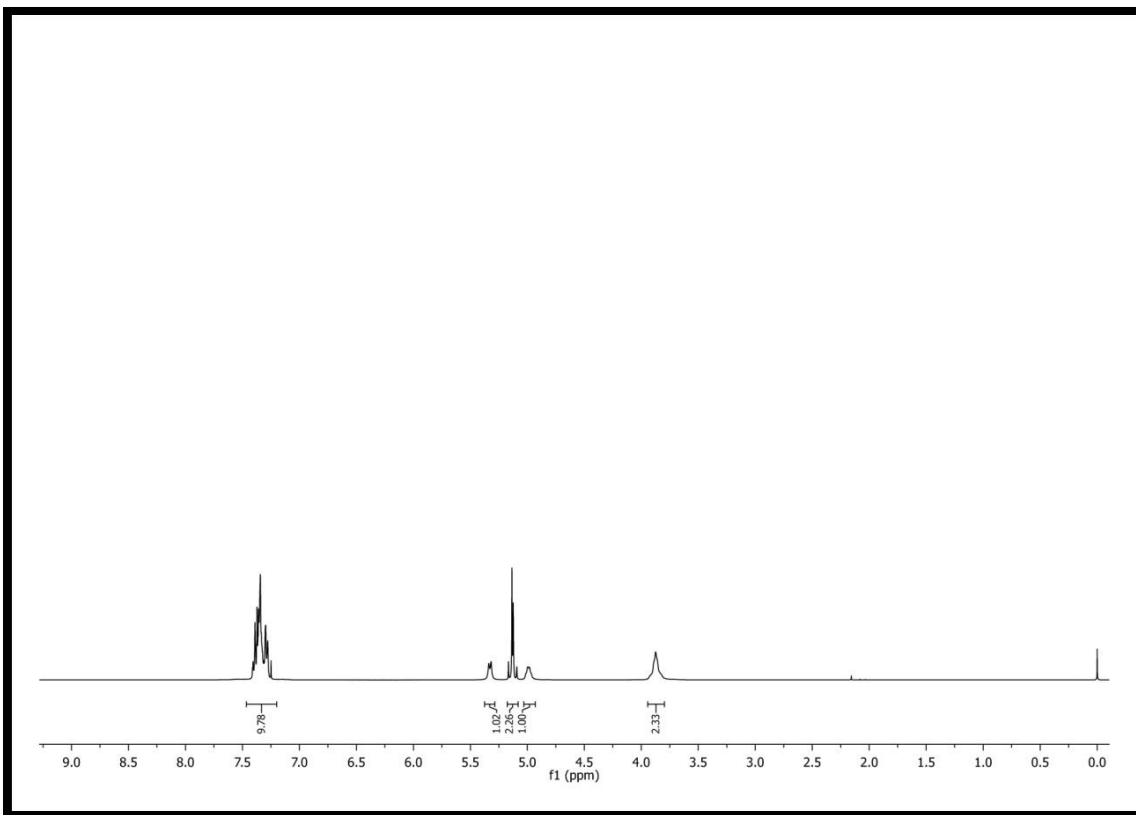
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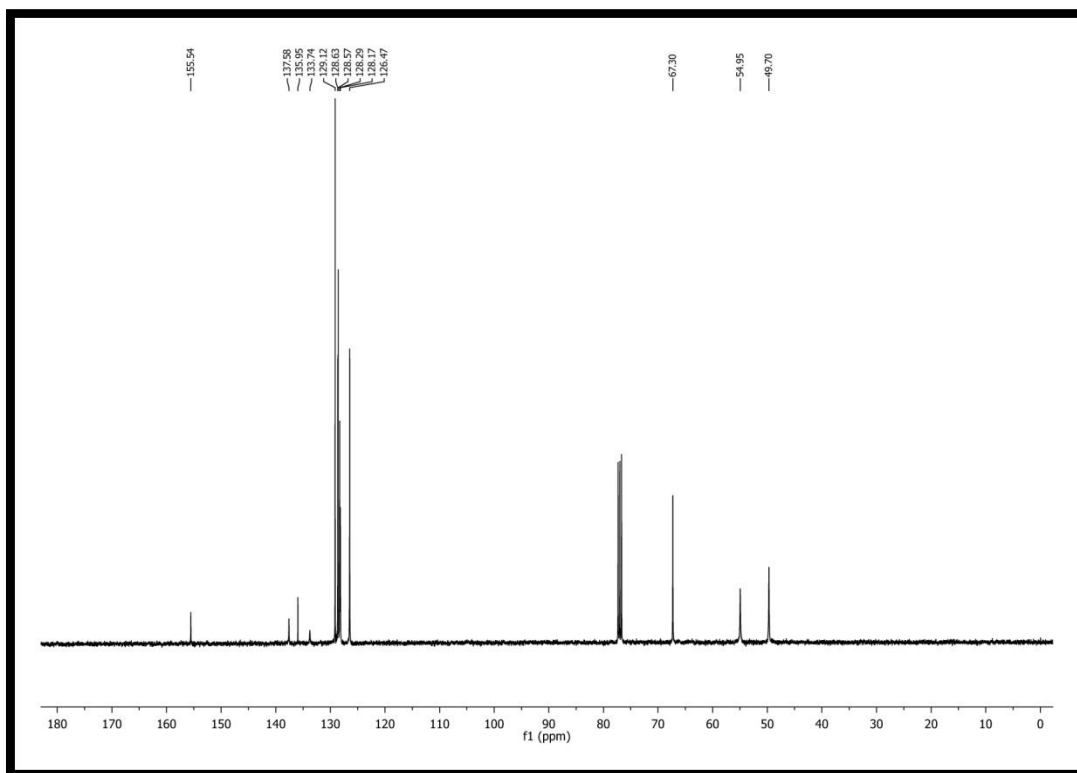
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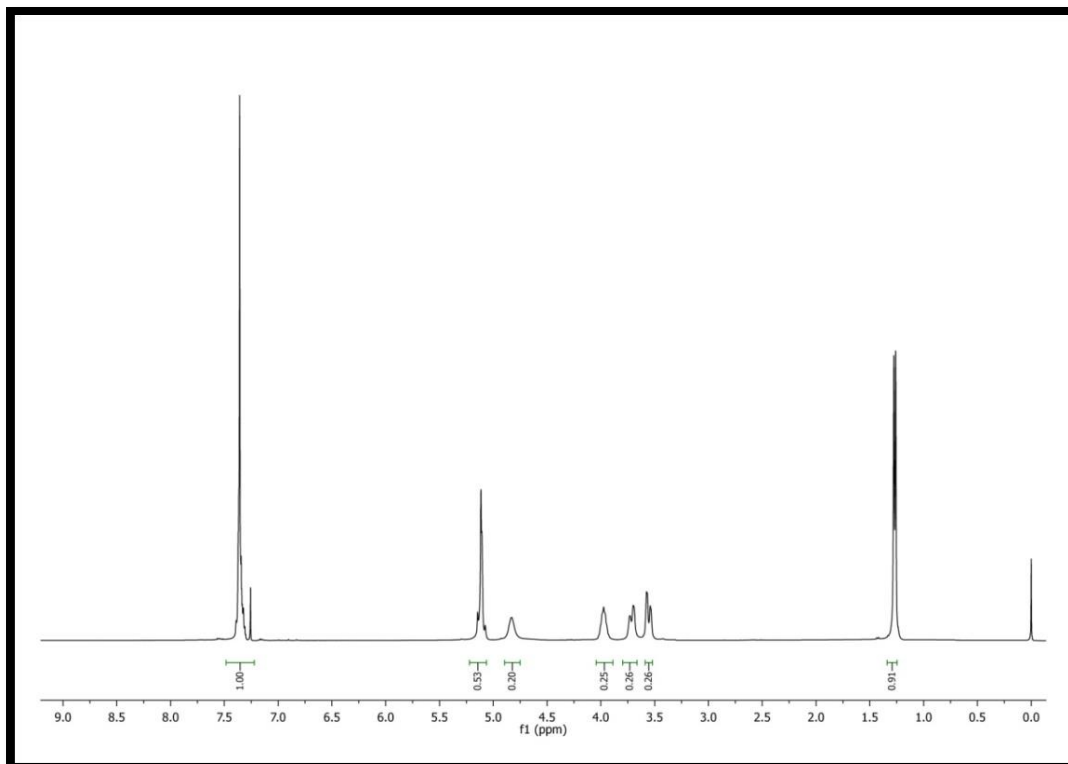
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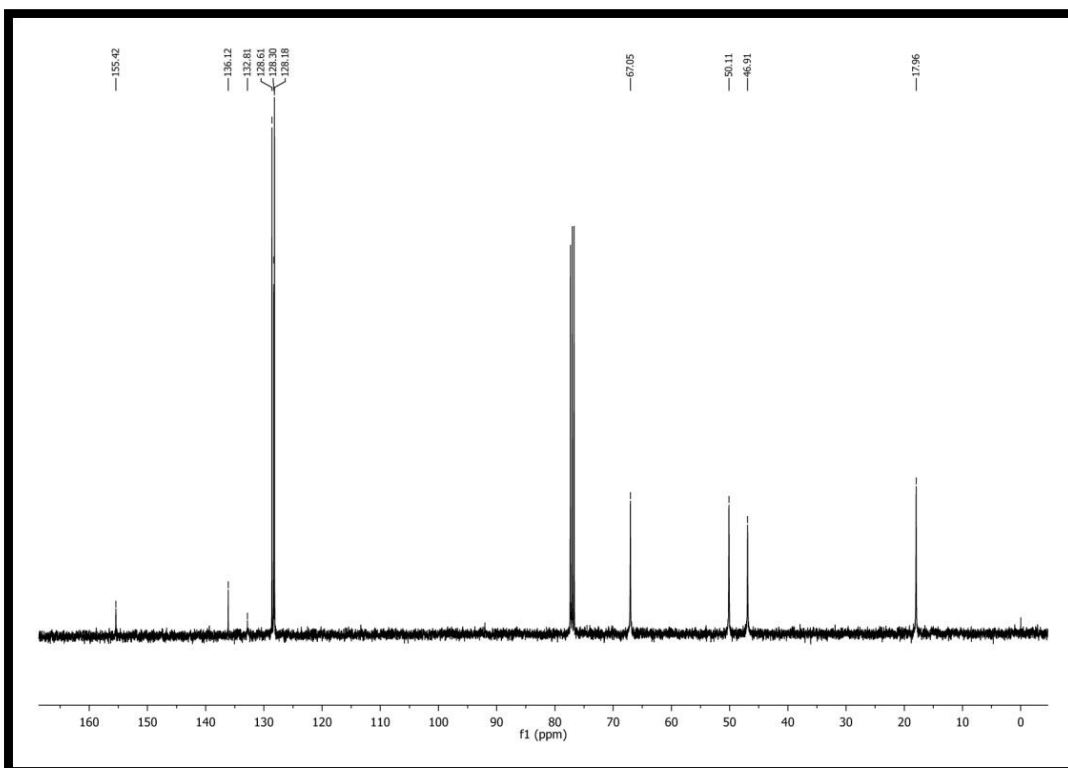
$^1\text{H}$  NMR Spectrum of Cbz-phg- $\psi$ [CH<sub>2</sub>NCS], 2a



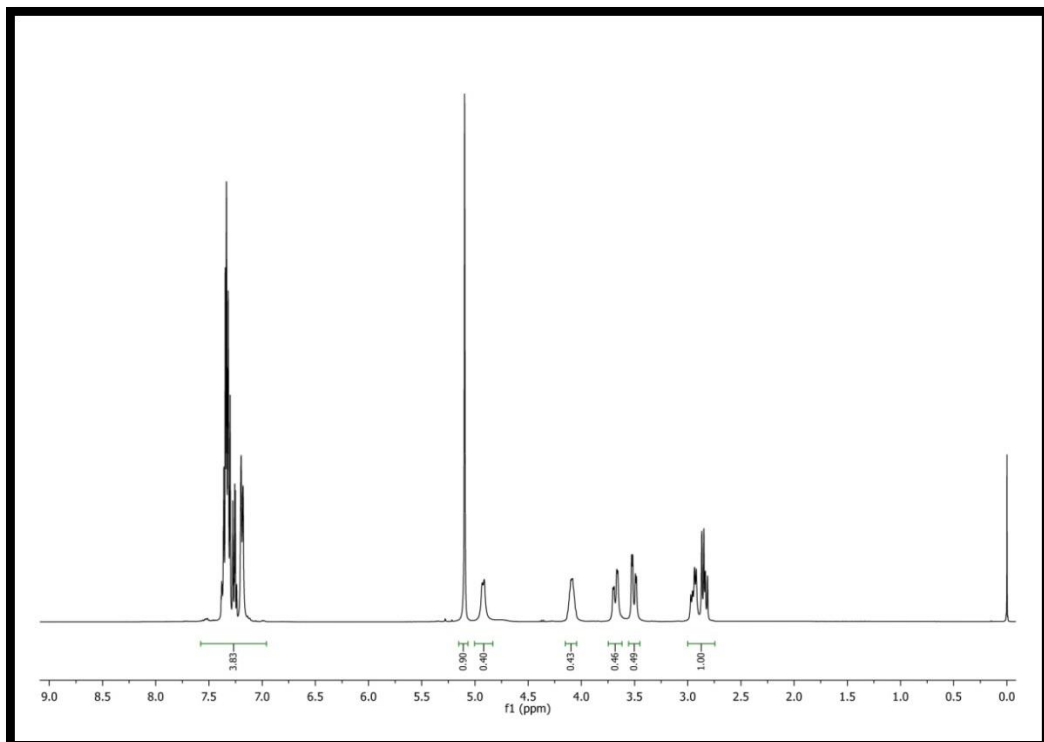
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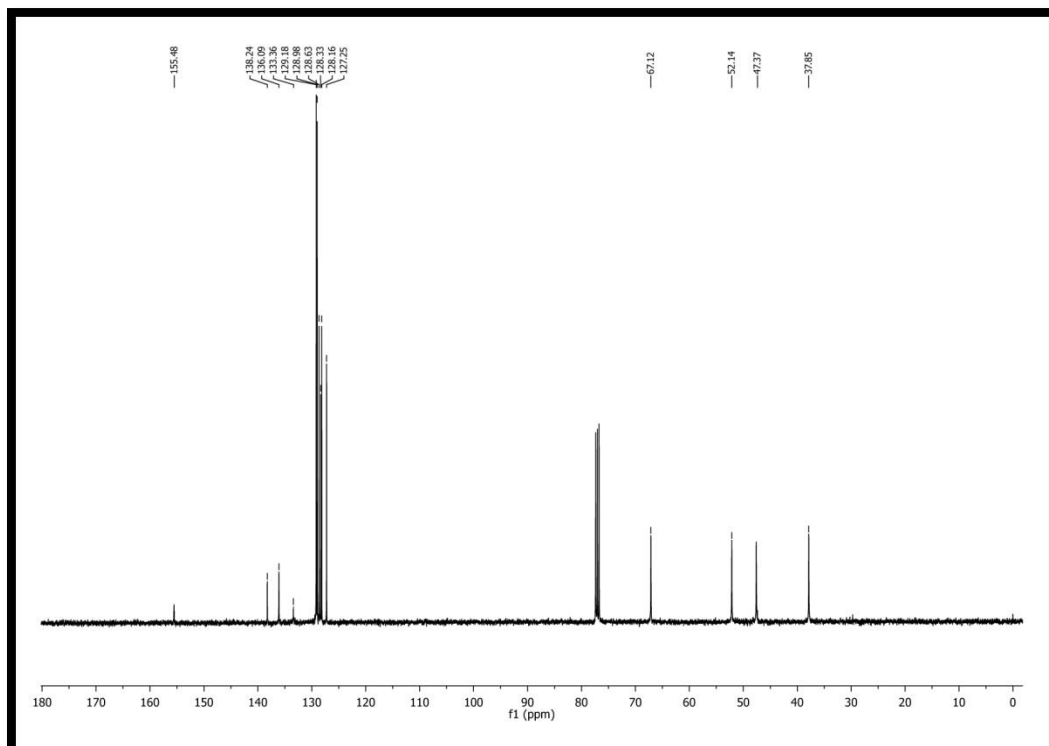
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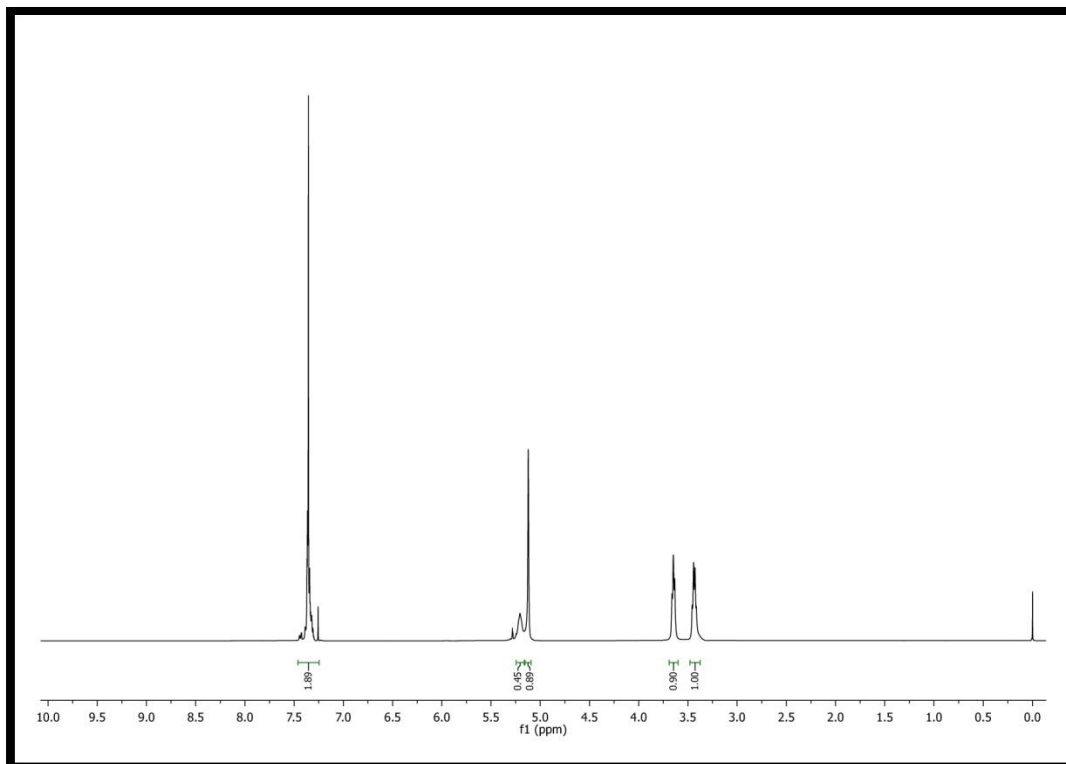
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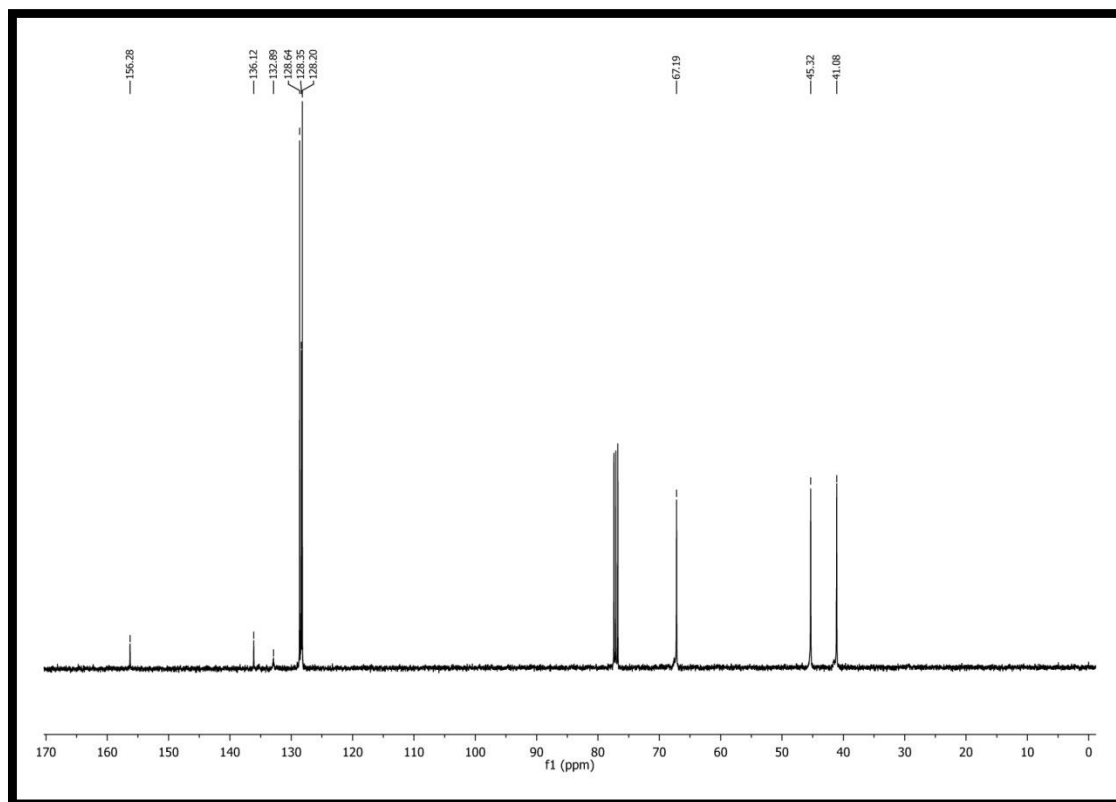
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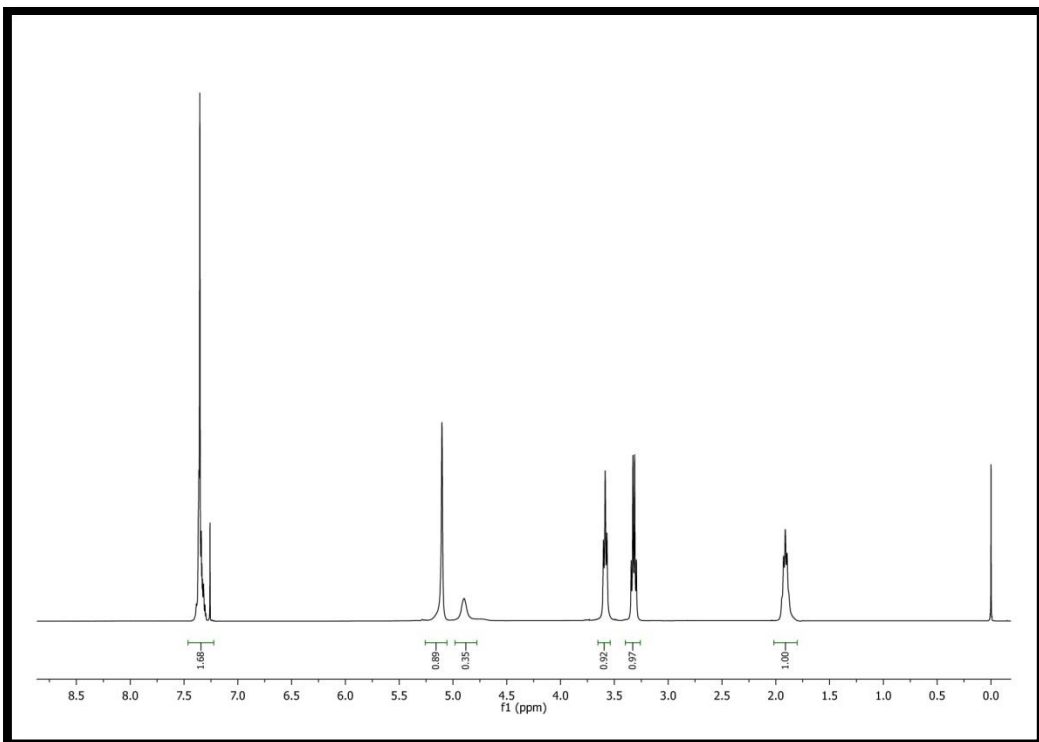
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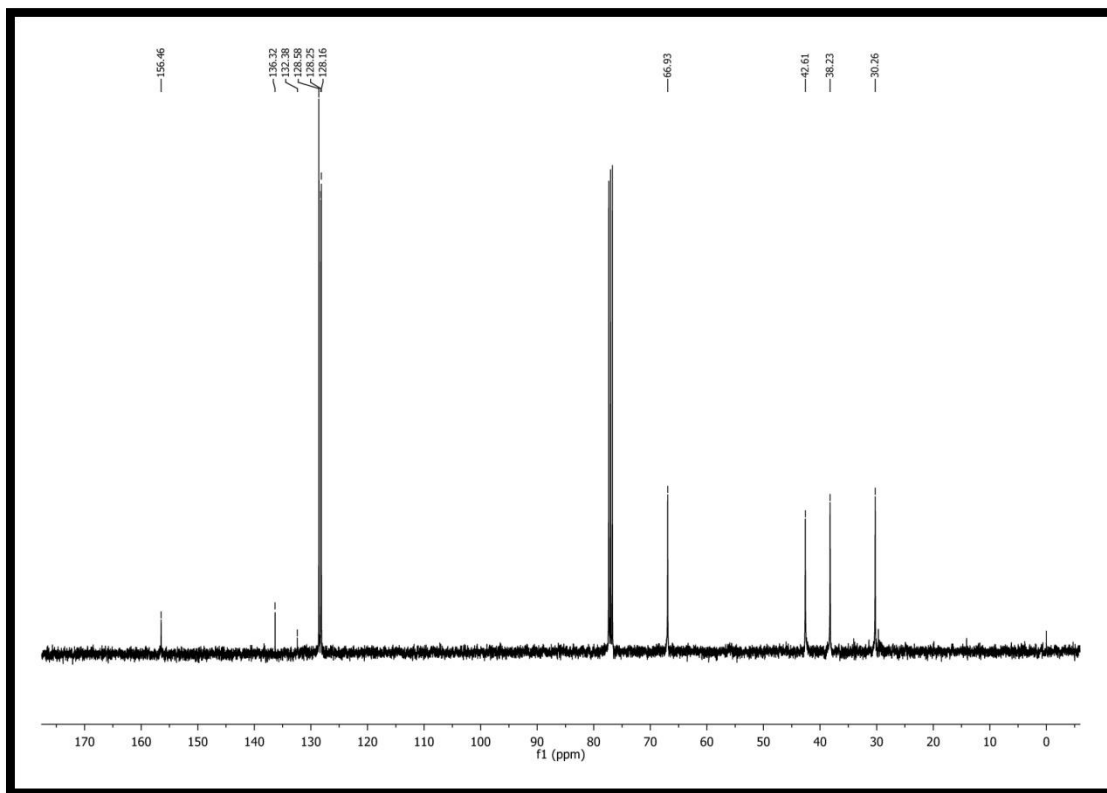
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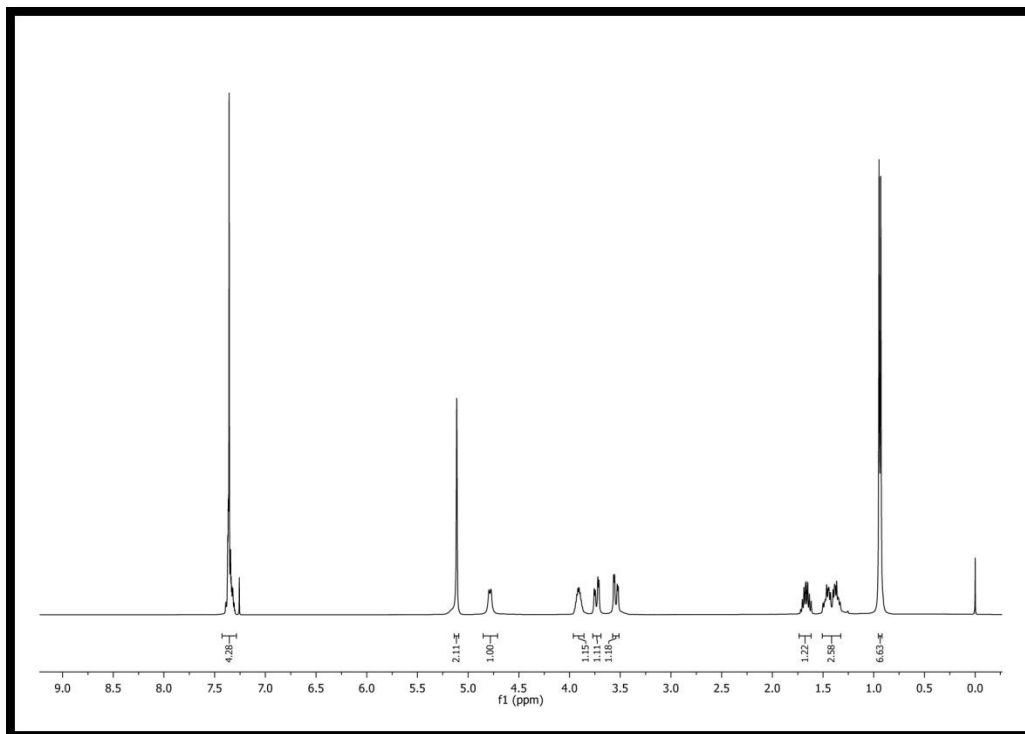
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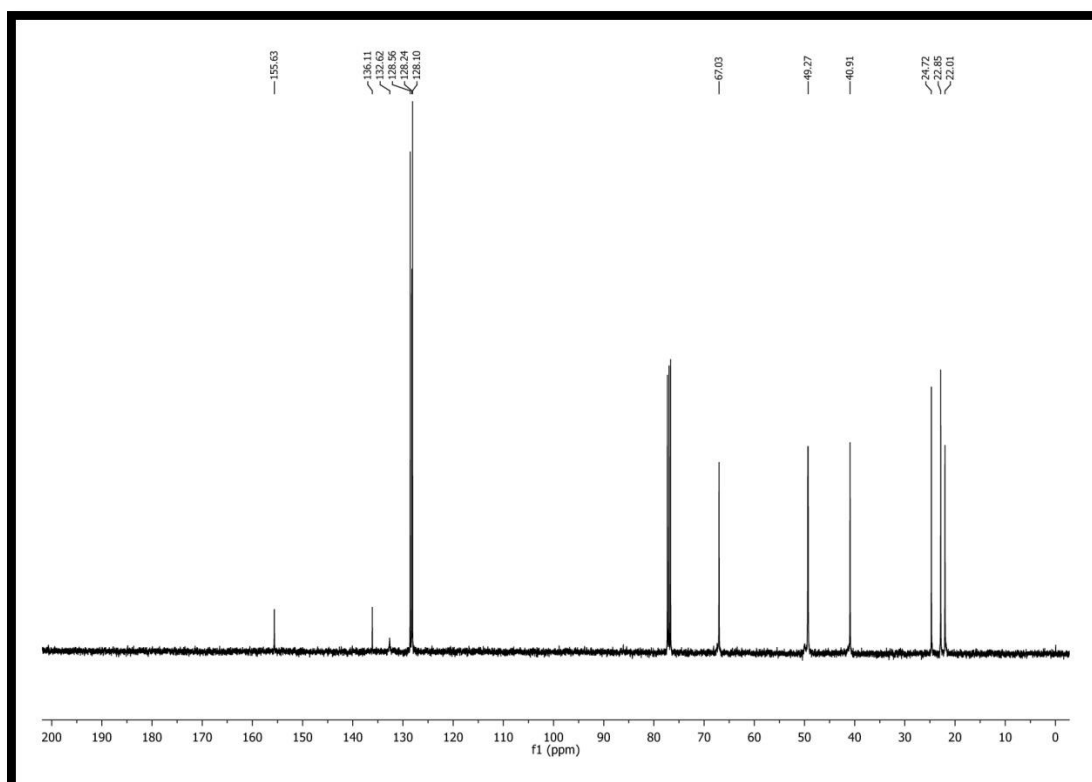
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$^{13}\text{C}$  NMR Spectrum of Cbz- $\beta$ -ala- $\psi$ [CH<sub>2</sub>NCS], 2e

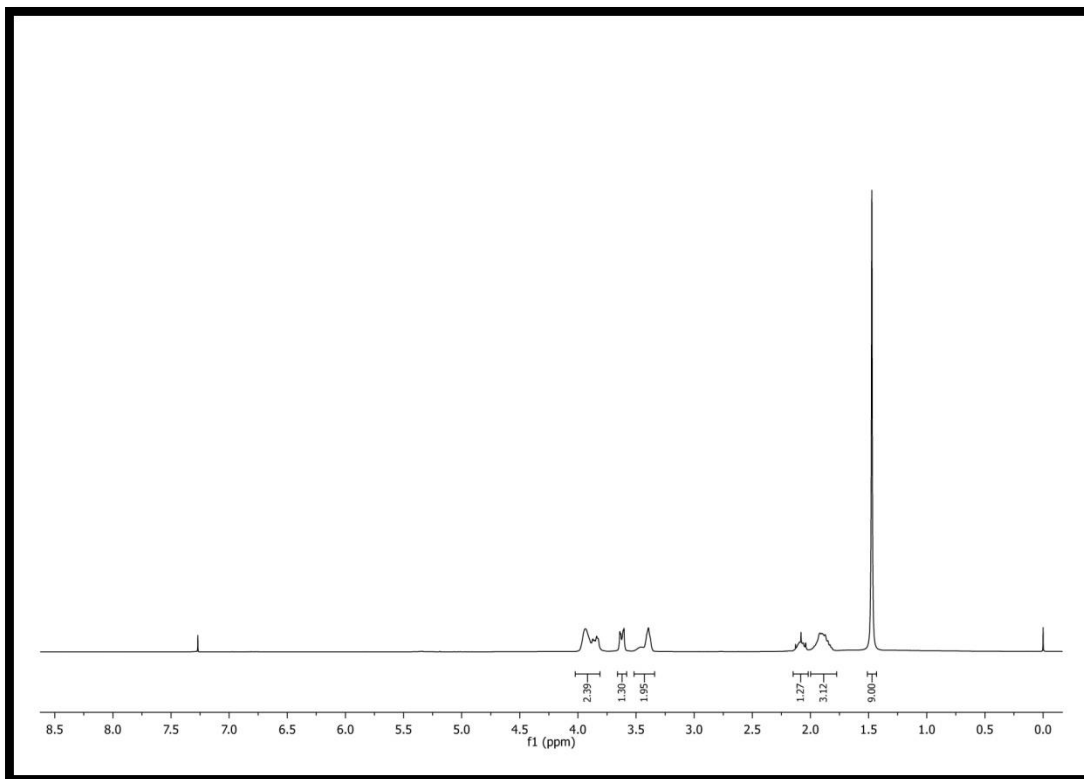


<sup>1</sup>H NMR Spectrum of Cbz-Leu-ψ[CH<sub>2</sub>NCS], 2f

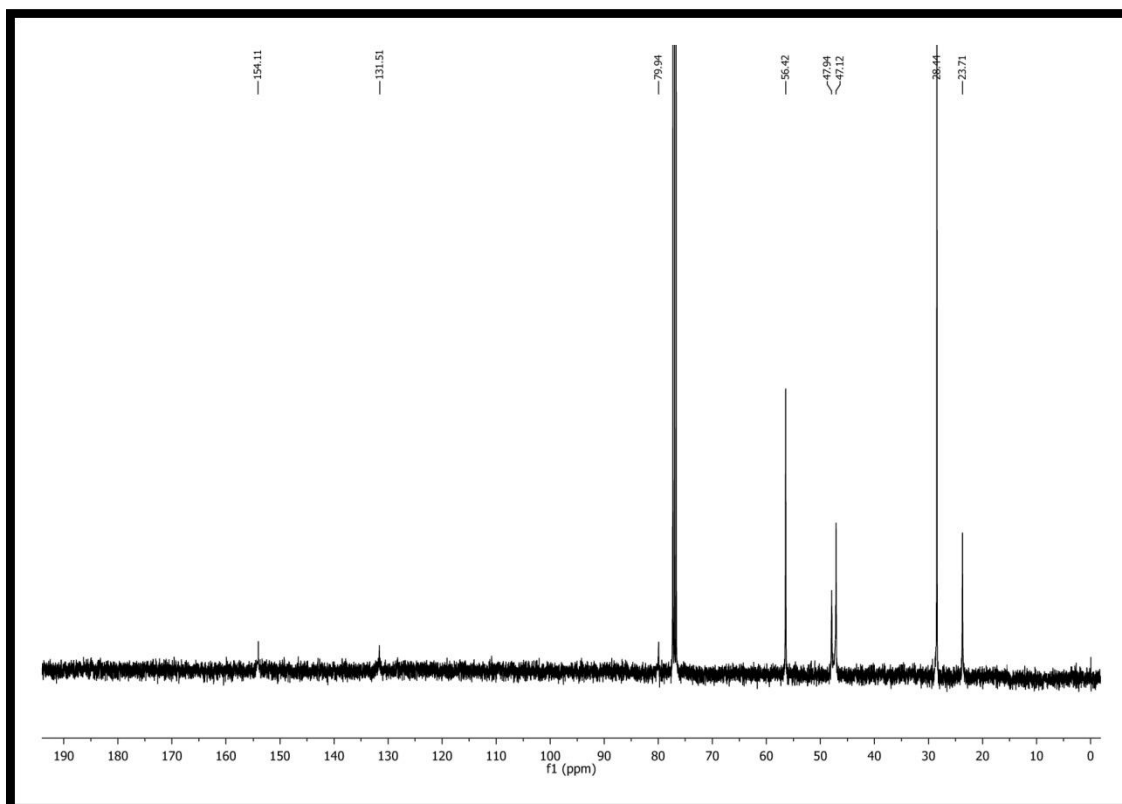


<sup>13</sup>C NMR Spectrum of Cbz-Leu-ψ[CH<sub>2</sub>NCS], 2f

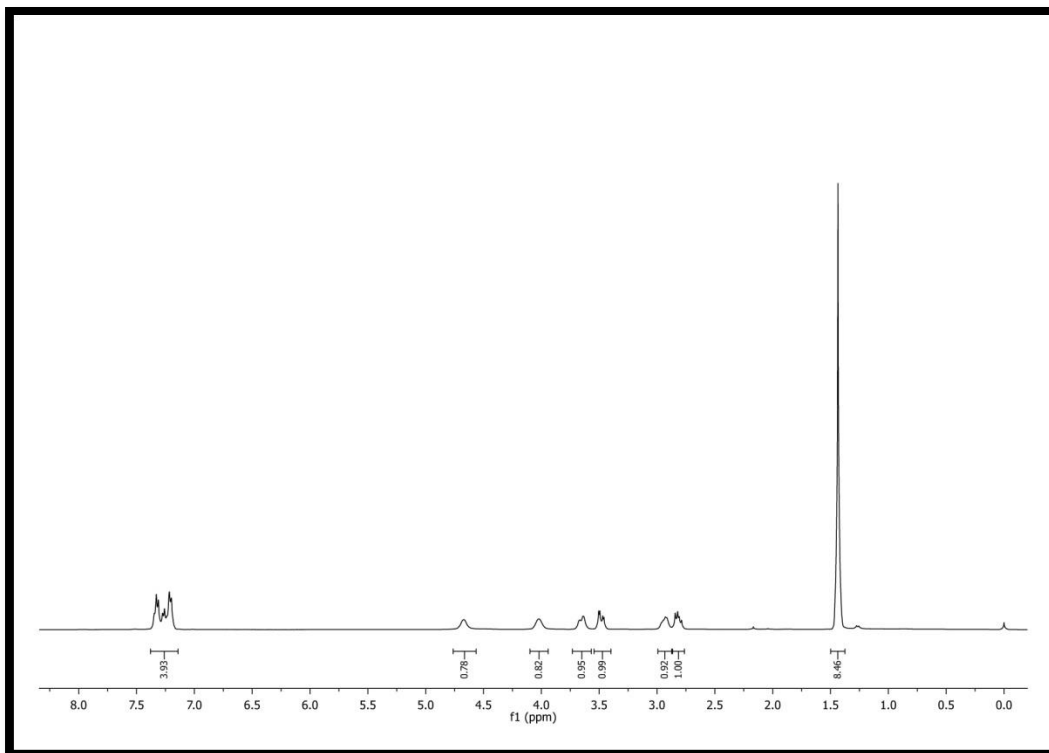




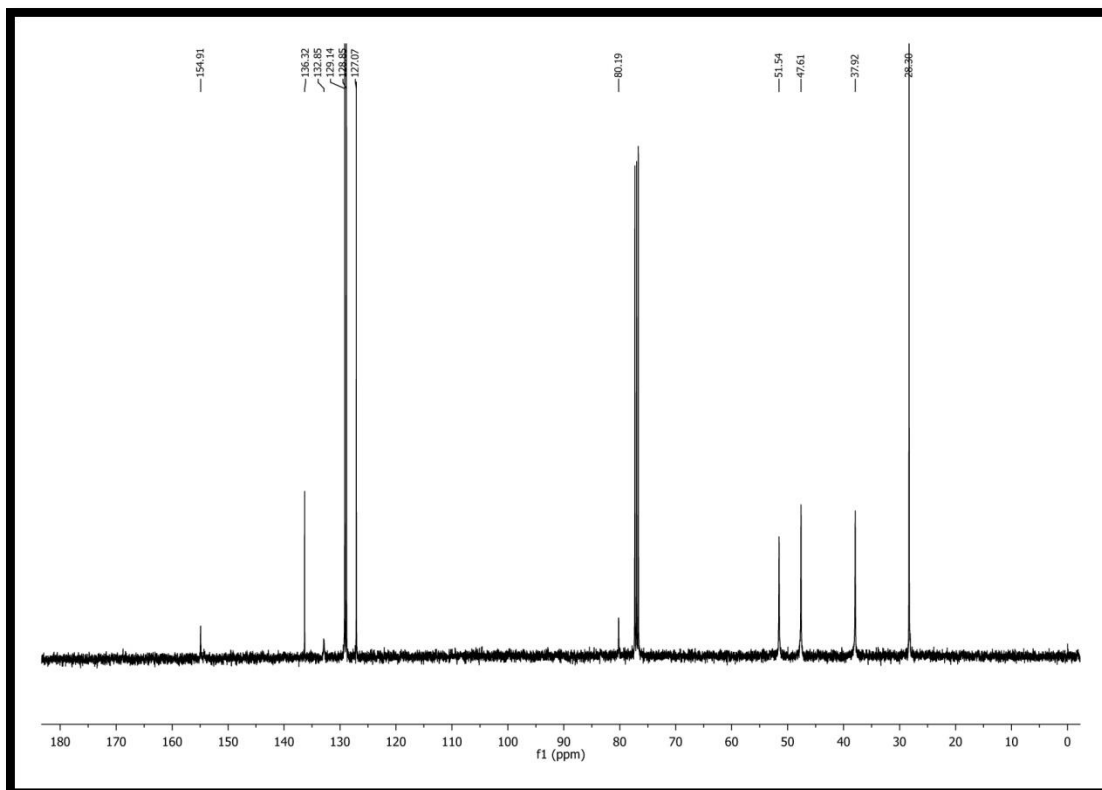
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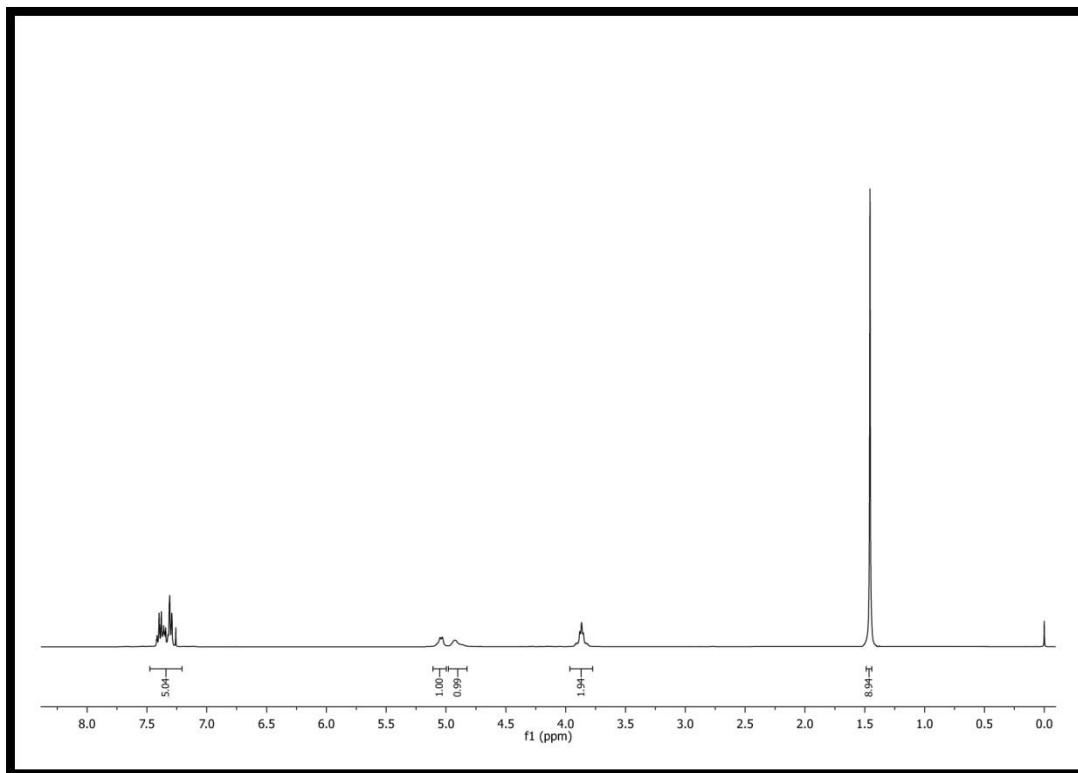
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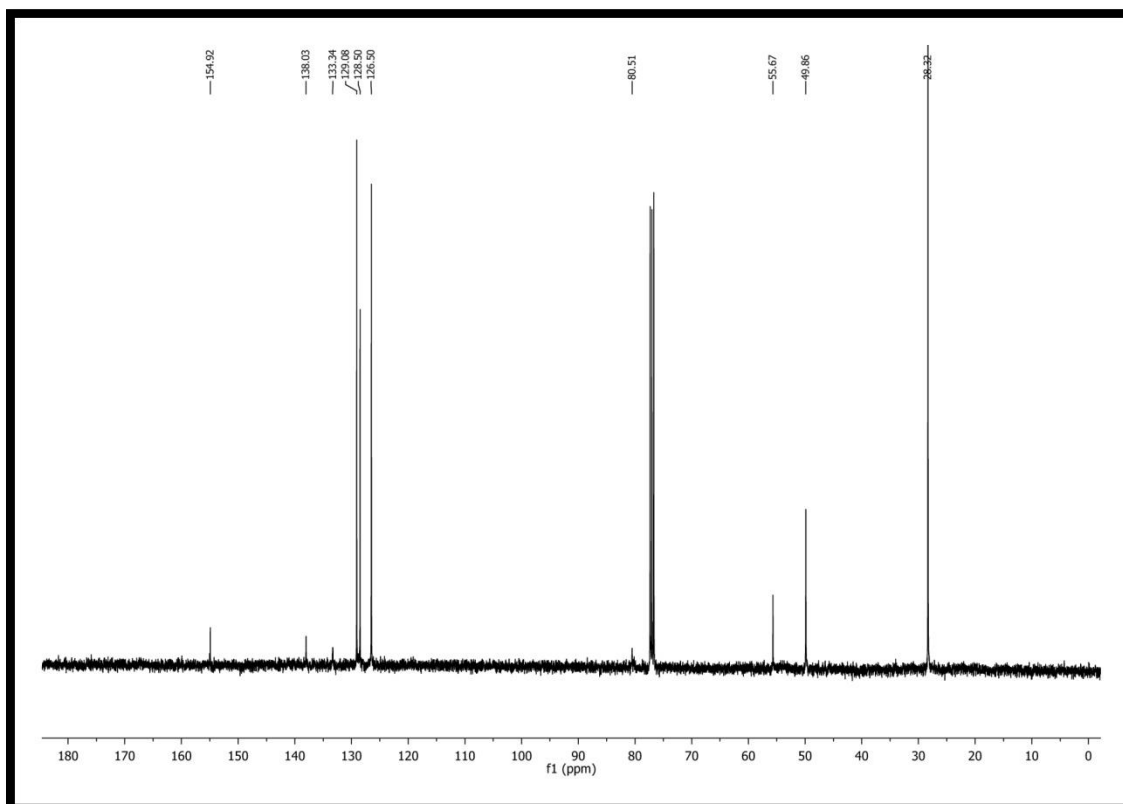
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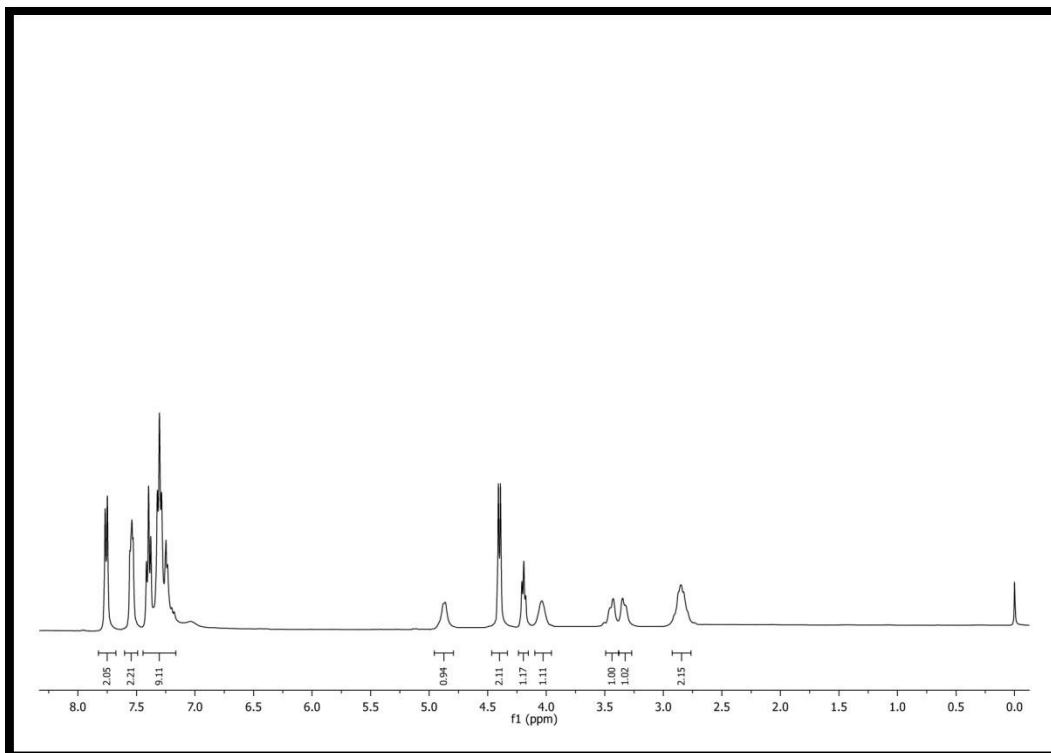
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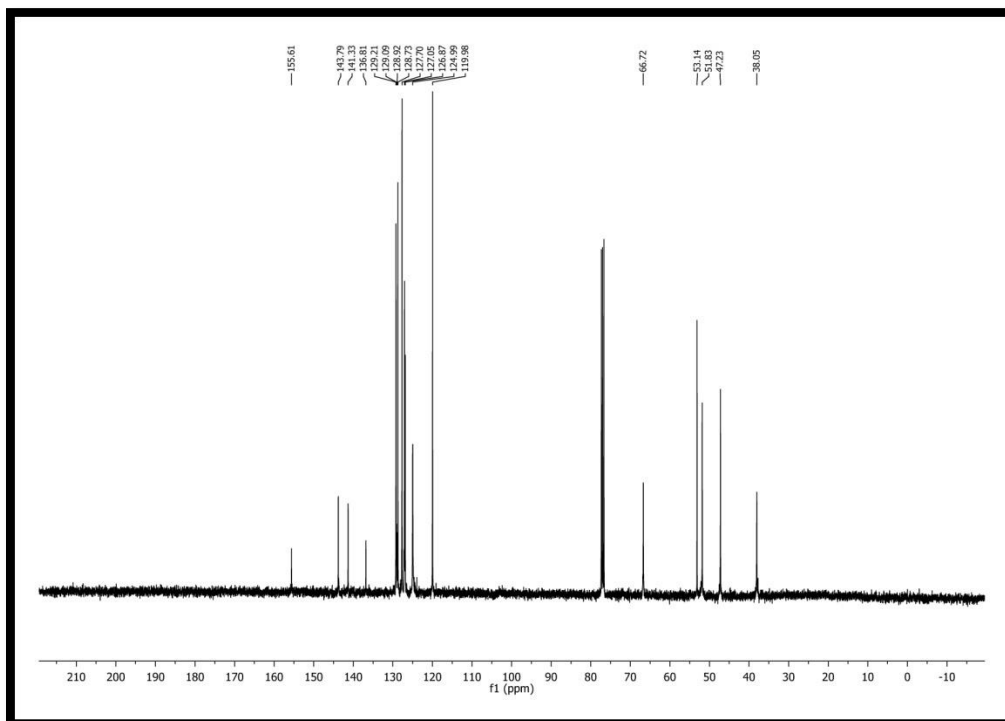
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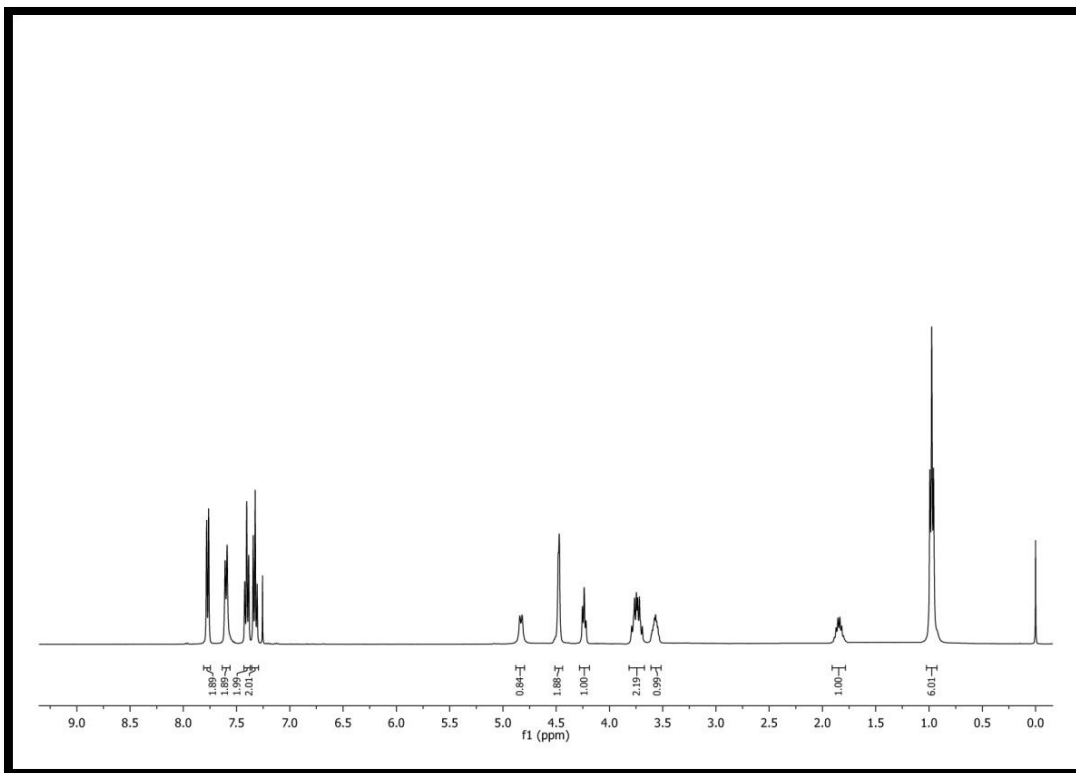
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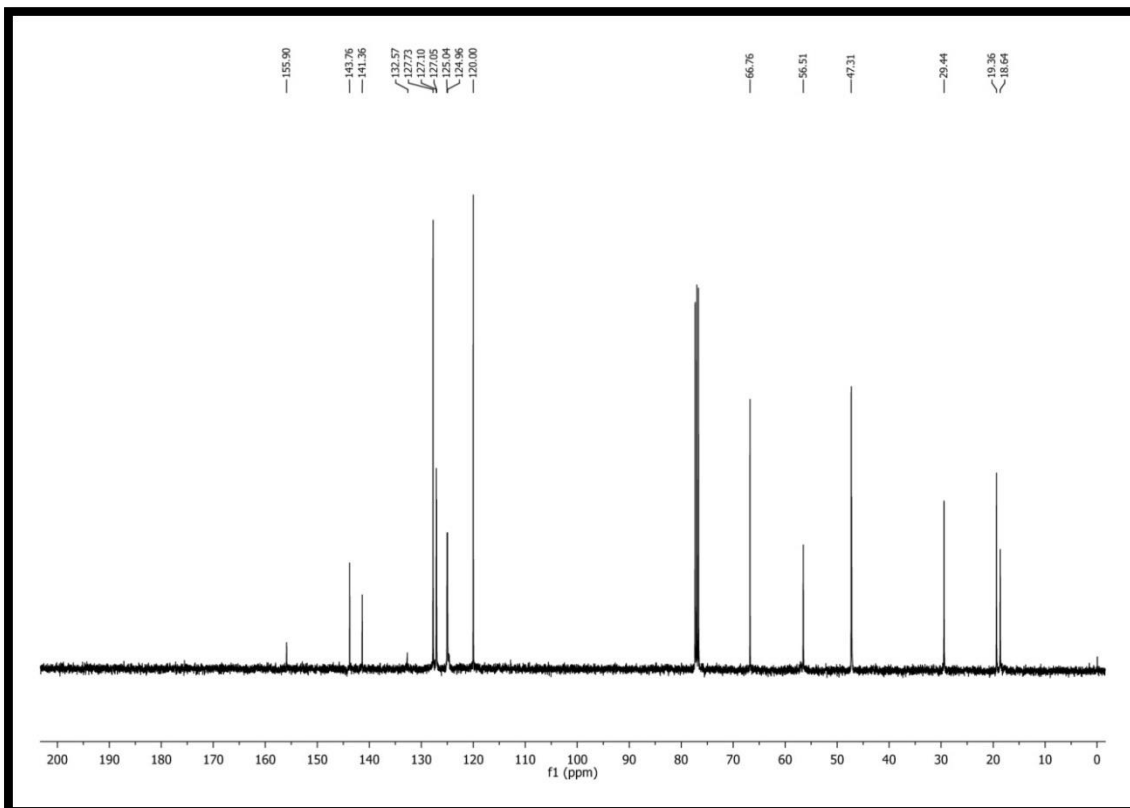
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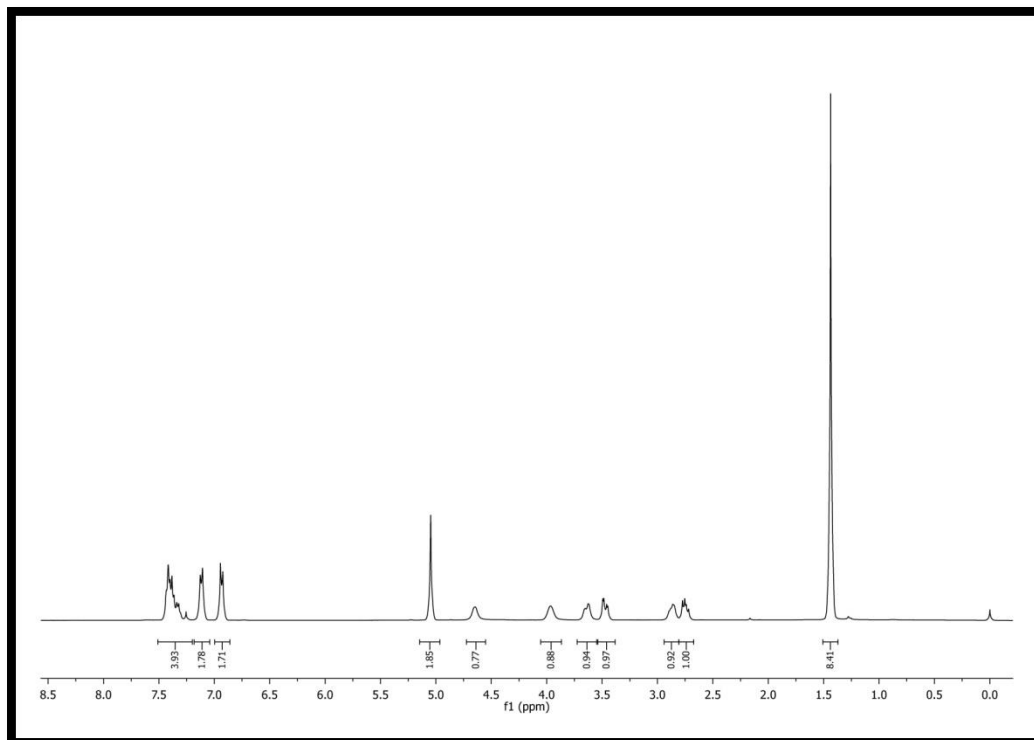
<sup>13</sup>C NMR Spectrum of Fmoc-Phe-ψ[CH<sub>2</sub>NCS], 2j



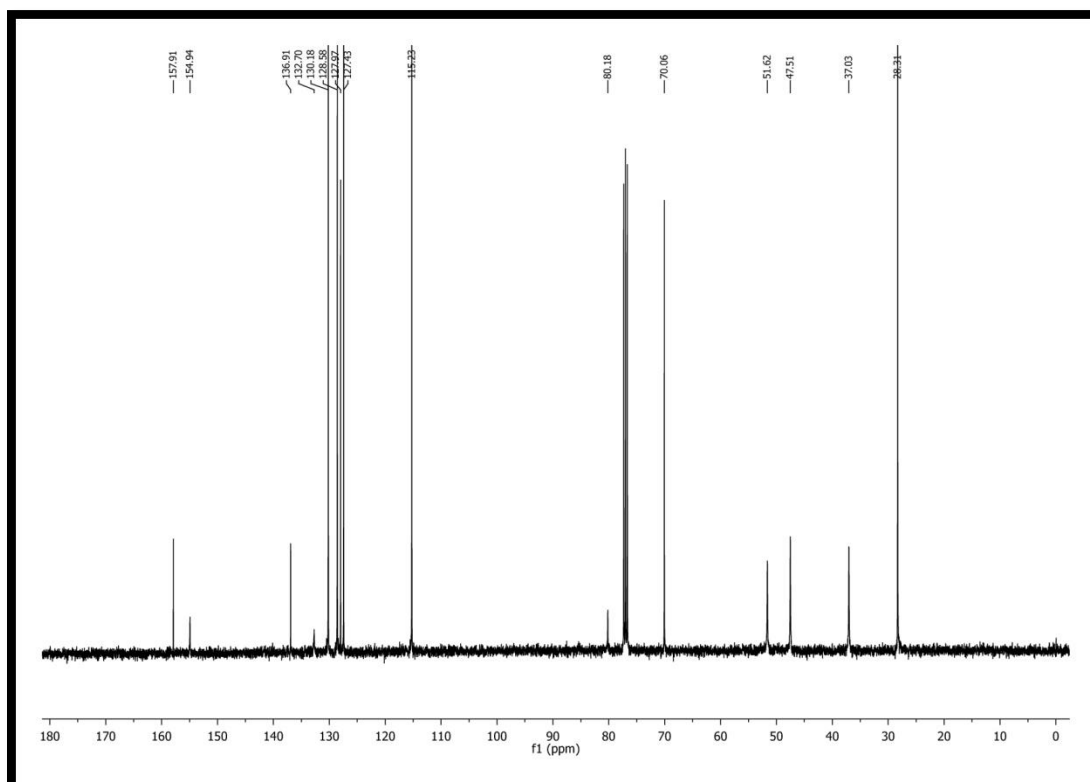
<sup>1</sup>H NMR Spectrum of Fmoc-Val-ψ[CH<sub>2</sub>NCS], 2k



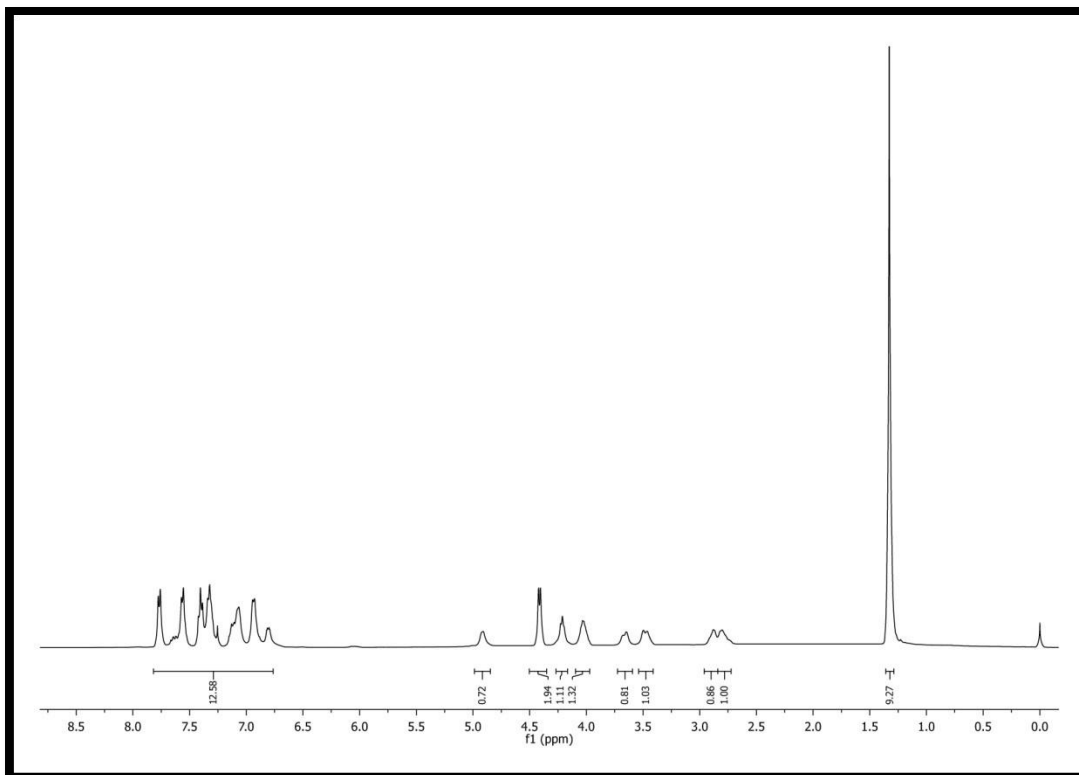
<sup>13</sup>C NMR Spectrum of Fmoc-Val-ψ[CH<sub>2</sub>NCS], 2k



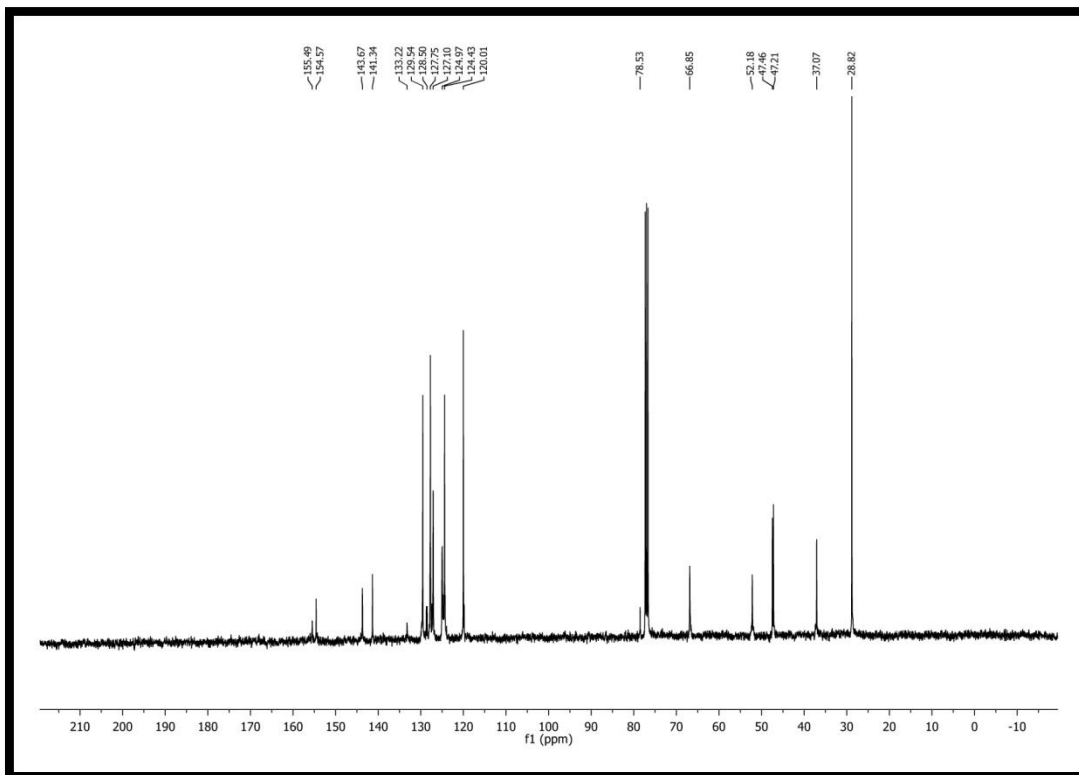
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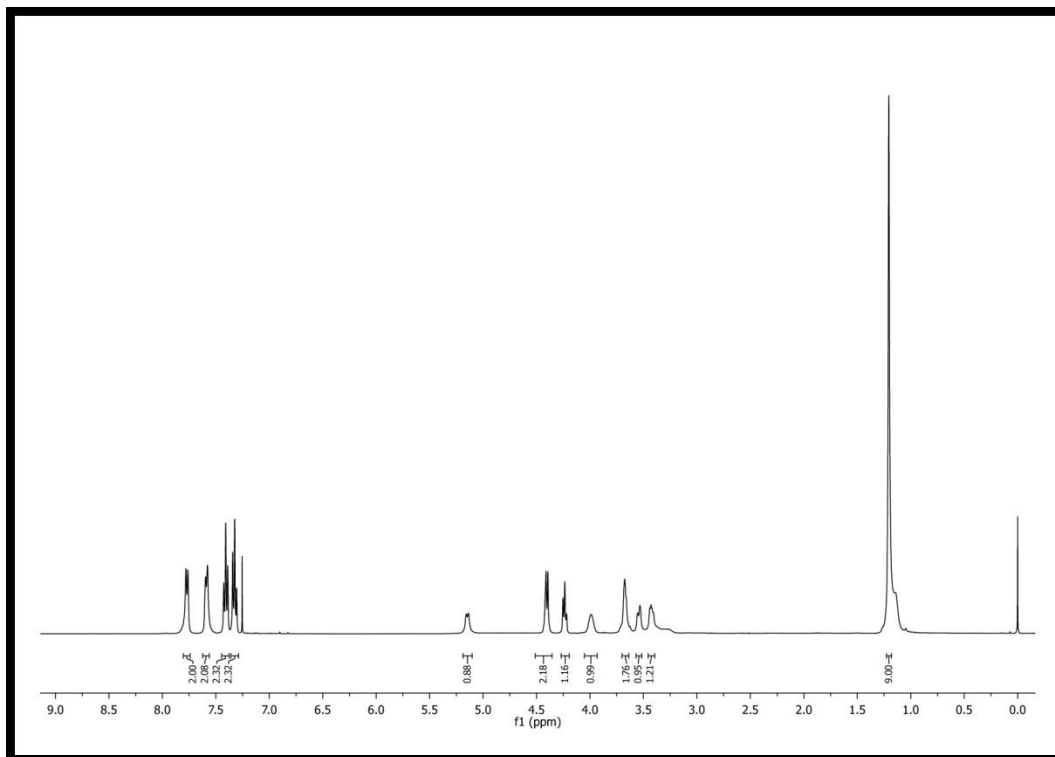
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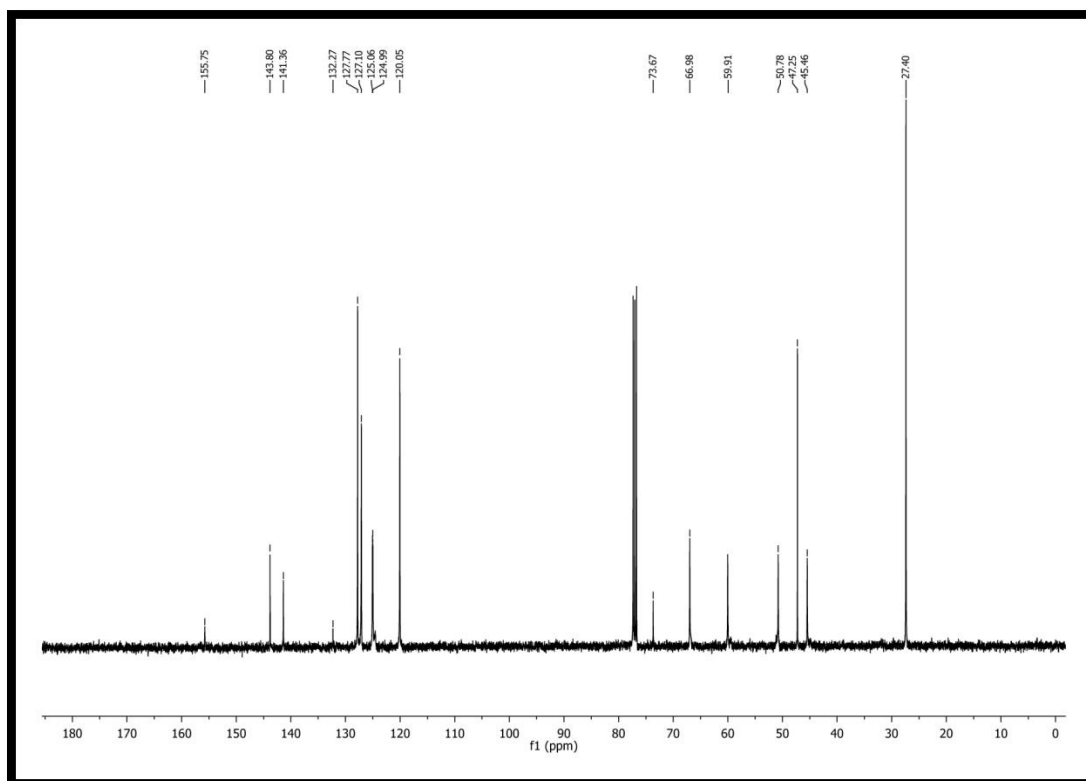
<sup>1</sup>H NMR Spectrum of Fmoc-Tyr(<sup>t</sup>Bu)-ψ[CH<sub>2</sub>NCS], 2m



<sup>13</sup>C NMR Spectrum of Fmoc-Tyr(<sup>t</sup>Bu)-ψ[CH<sub>2</sub>NCS], 2m

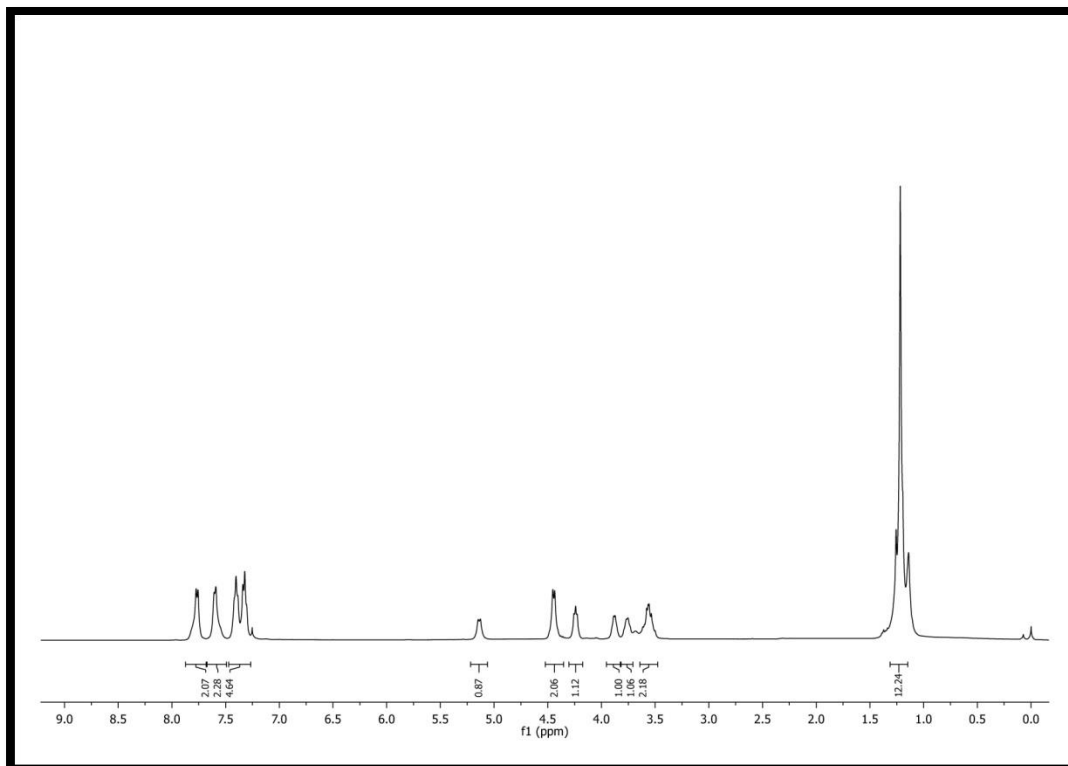


<sup>1</sup>H NMR Spectrum of Fmoc-Ser(<sup>t</sup>Bu)-ψ[CH<sub>2</sub>NCS], 2n

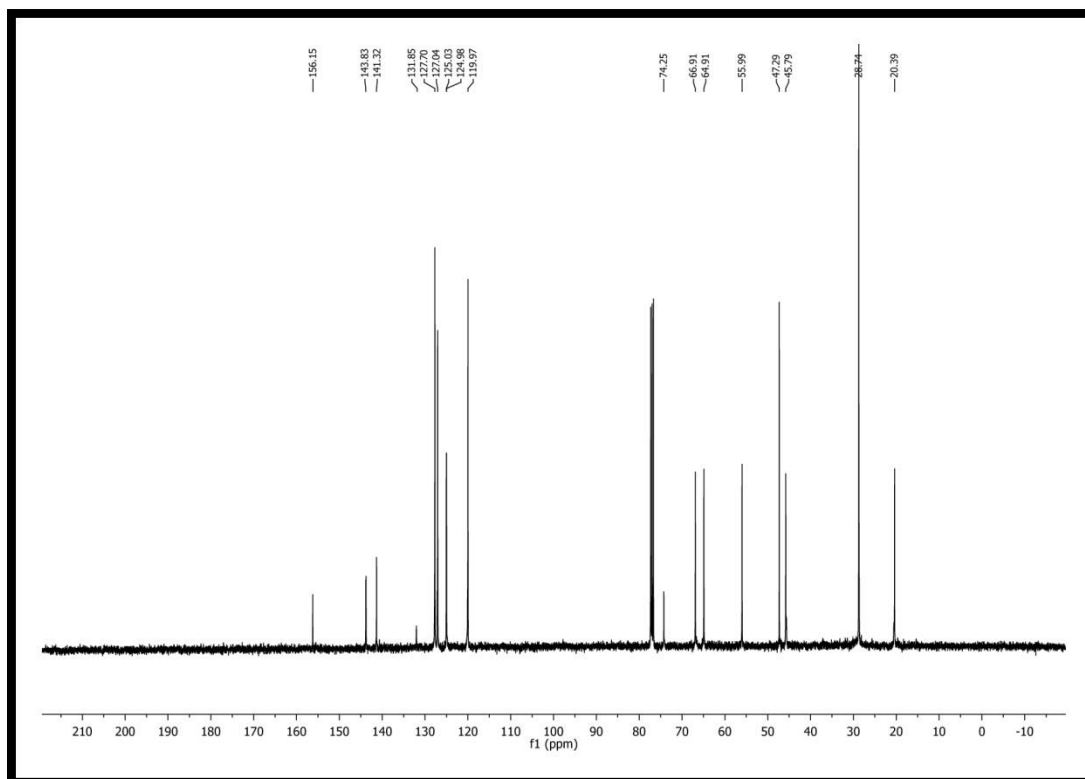


<sup>13</sup>C NMR Spectrum of Fmoc-Ser(<sup>t</sup>Bu)-ψ[CH<sub>2</sub>NCS], 2n

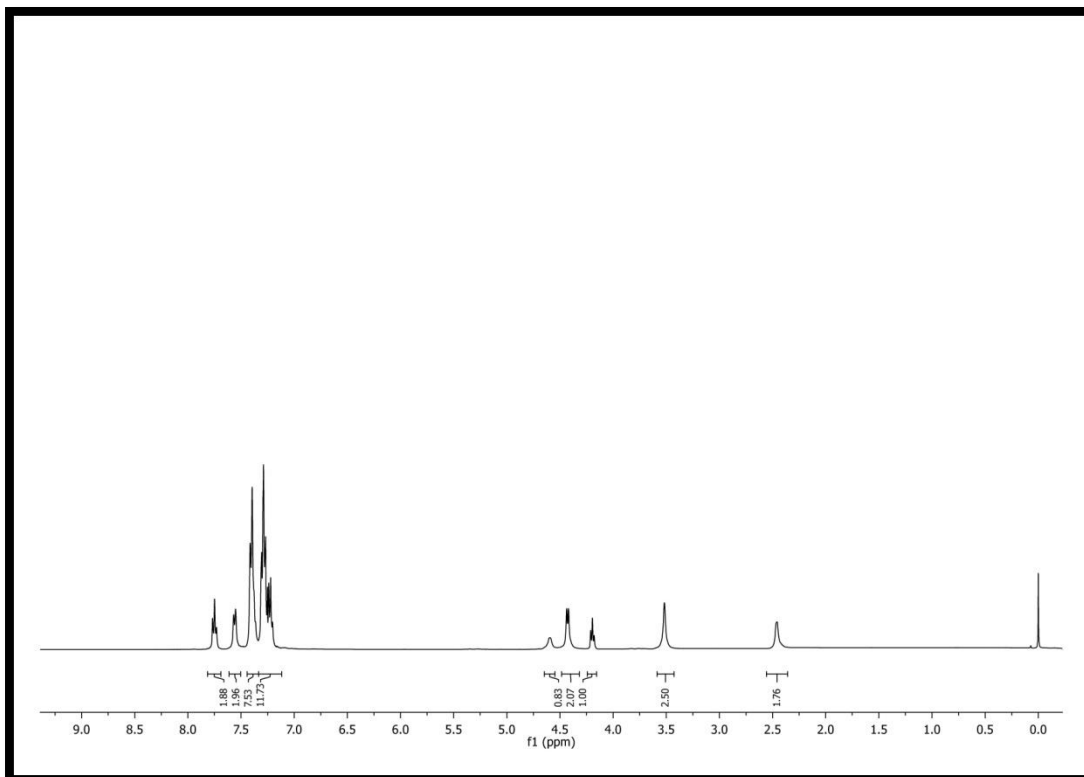




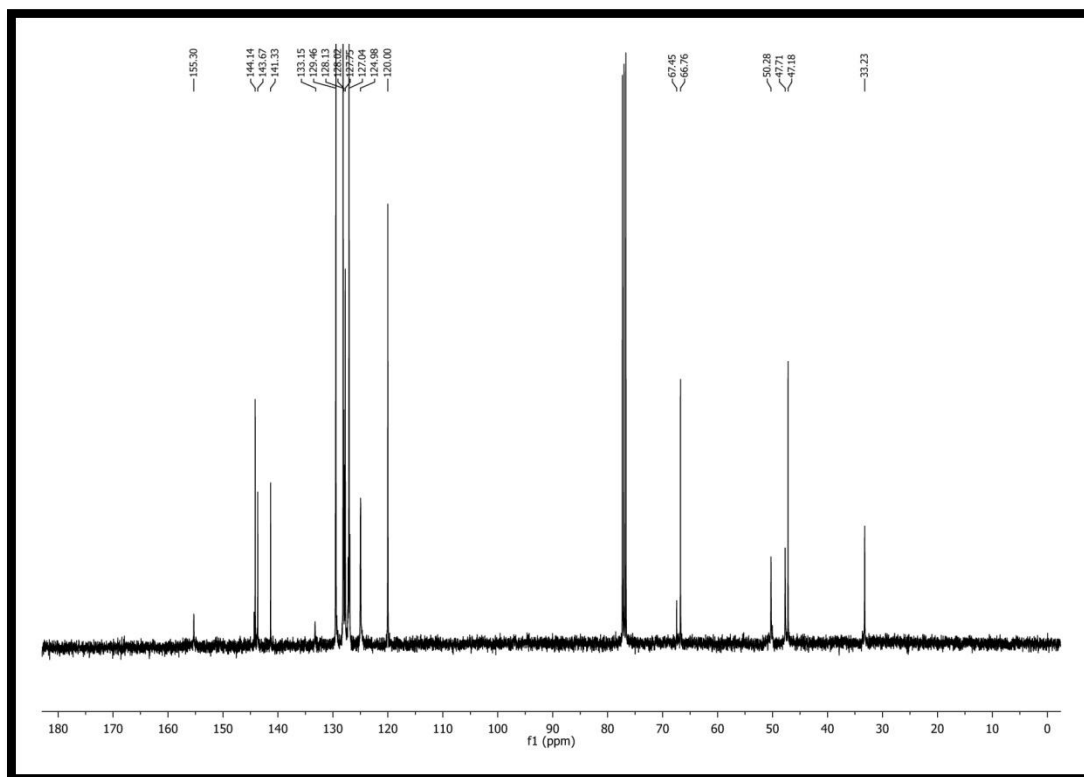
$^1\text{H}$  NMR Spectrum of Fmoc-Thr( $^t\text{Bu}$ )- $\psi[\text{CH}_2\text{NCS}]$ , 2o



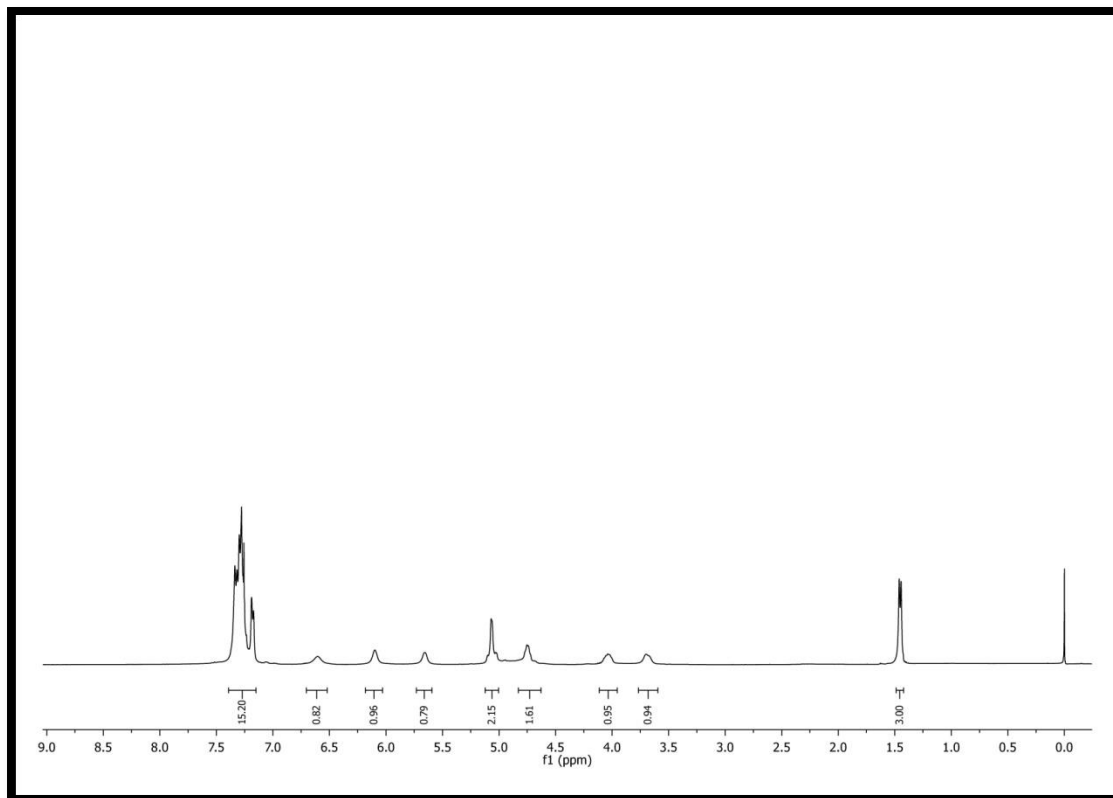
$^{13}\text{C}$  NMR Spectrum of Fmoc-Thr( $^t\text{Bu}$ )- $\psi[\text{CH}_2\text{NCS}]$ , 2o



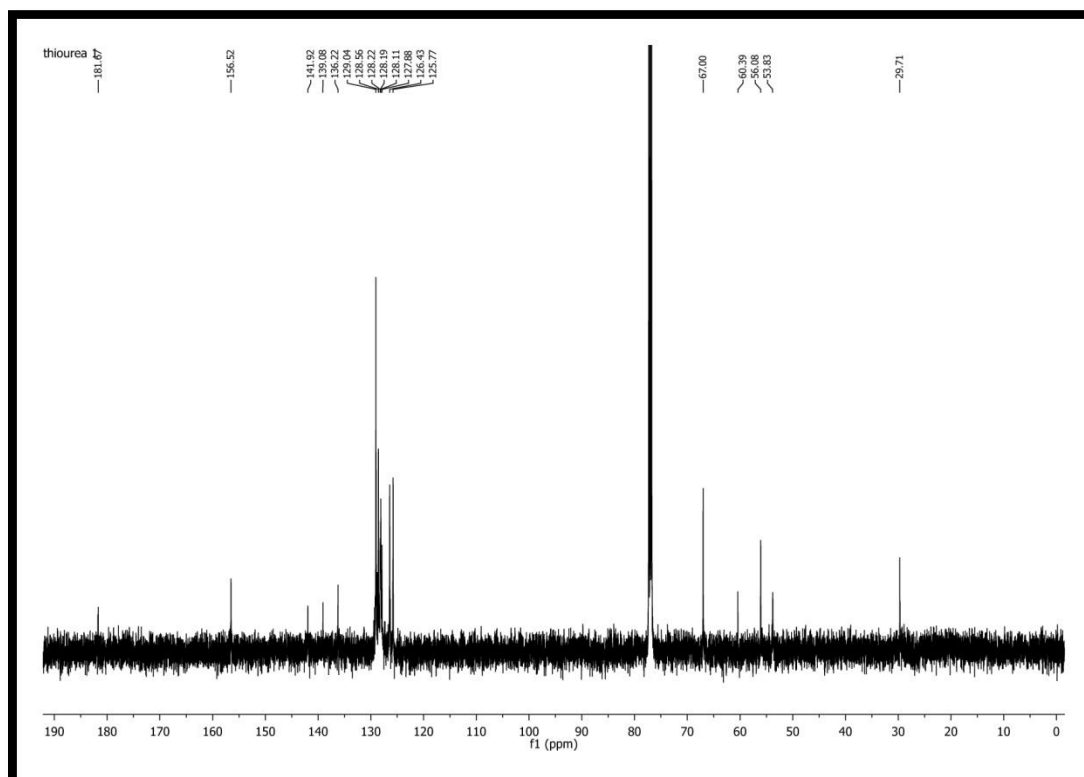
$^1\text{H}$  NMR Spectrum of Fmoc-Cys(Trt)- $\psi$ [CH<sub>2</sub>NCS], 2p



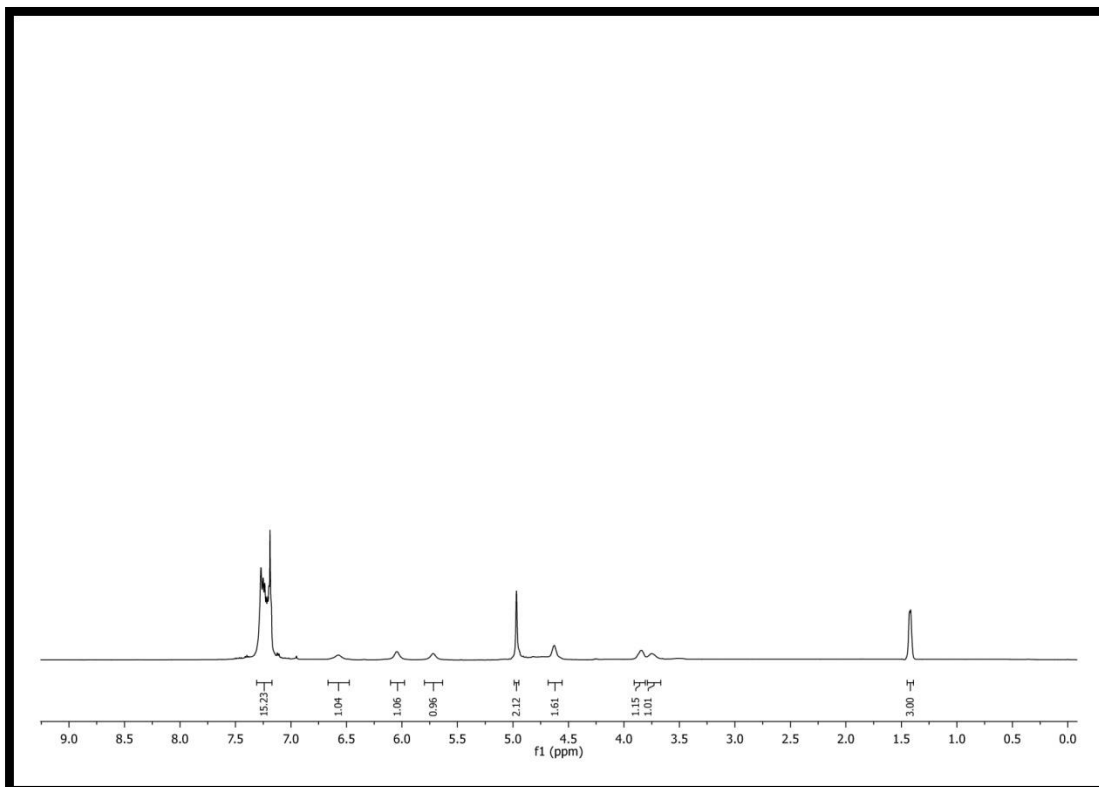
$^{13}\text{C}$  NMR Spectrum of Fmoc-Cys(Trt)- $\psi$ [CH<sub>2</sub>NCS], 2p



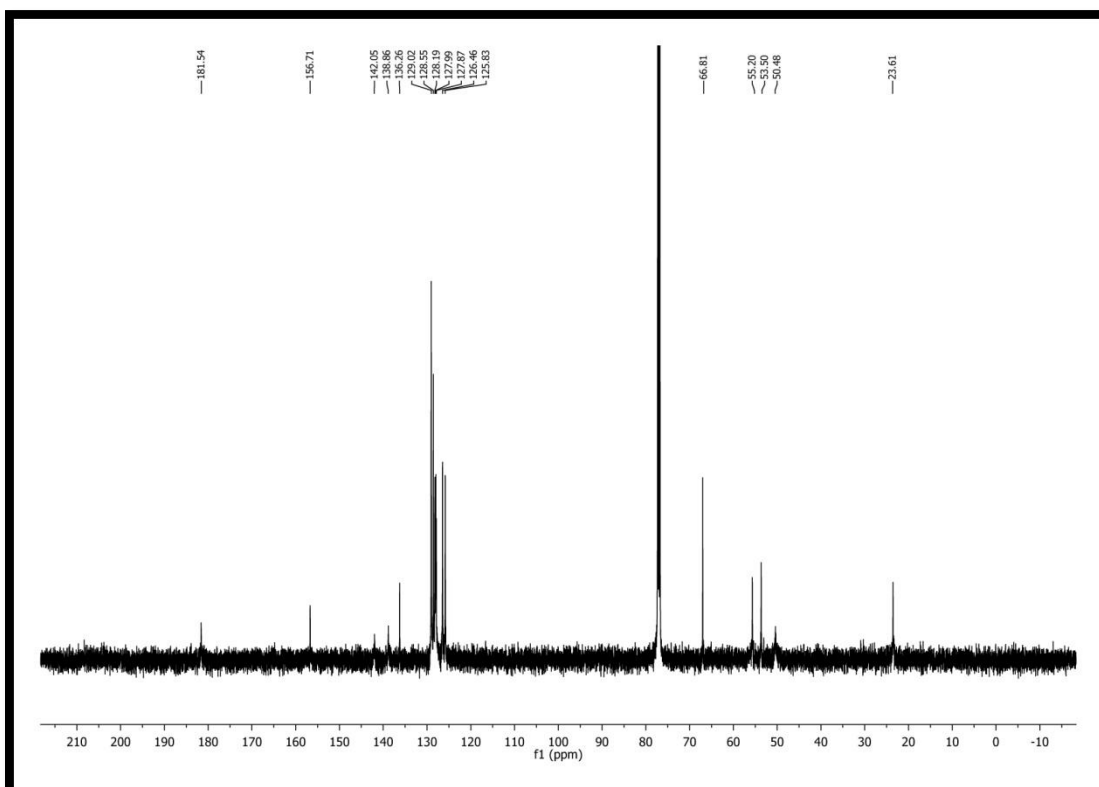
<sup>1</sup>H NMR Spectrum of Cbz-phg-ψ[CH<sub>2</sub>NHCSNH]-(*R*)-(+)-1-phenylethylamine, 3a



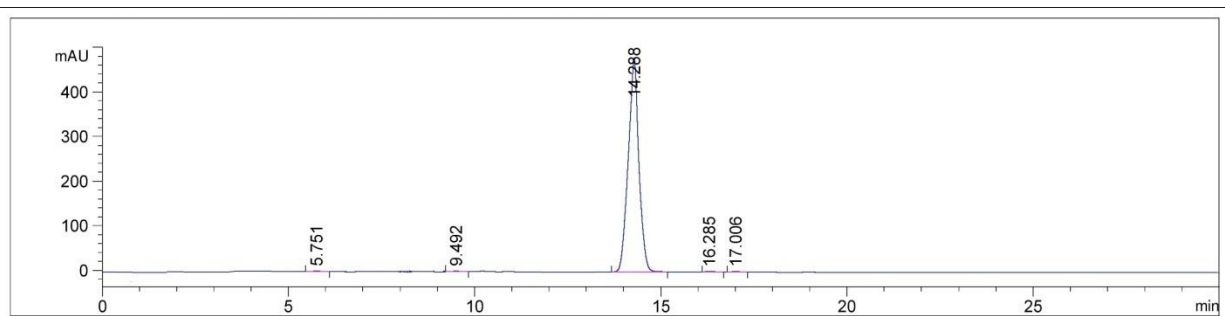
<sup>13</sup>C NMR Spectrum of Cbz-phg-ψ[CH<sub>2</sub>NHCSNH]-(*R*)-(+)-1-phenylethylamine, 3a



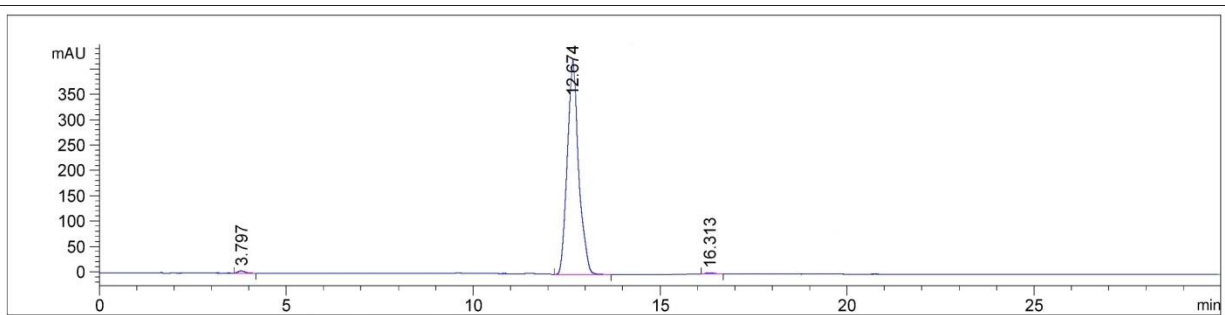
<sup>1</sup>H NMR Spectrum of Cbz-phg-ψ[CH<sub>2</sub>NHCSNH]-(S)-(-)-1-phenylethylamine, 3a\*



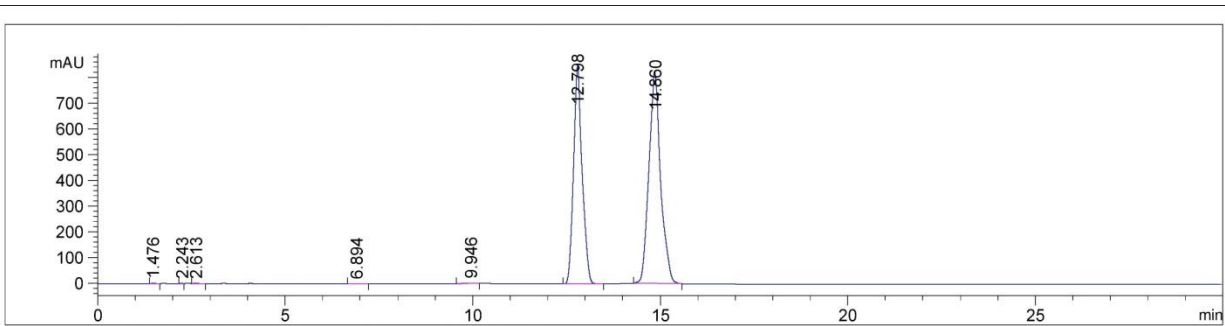
<sup>13</sup>C NMR Spectrum of Cbz-phg-ψ[CH<sub>2</sub>NHCSNH]-(S)-(-)-1-phenylethylamine, 3a\*



RP-HPLC Chromatogram 3a

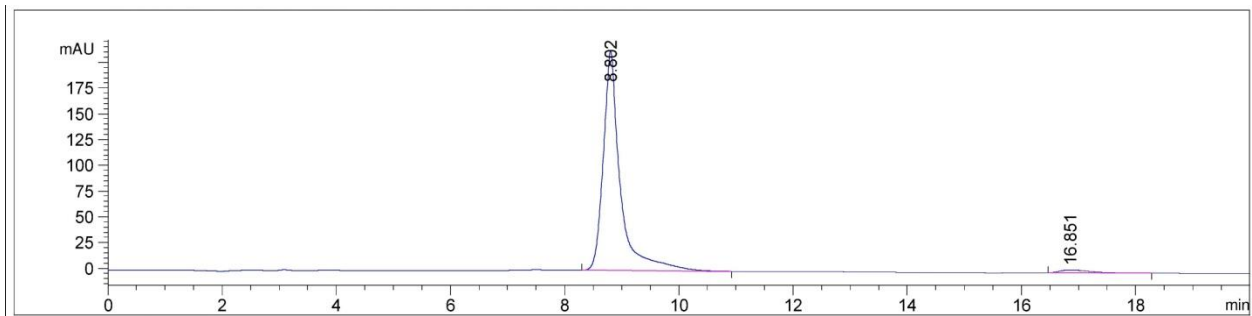


RP-HPLC Chromatogram 3a\*

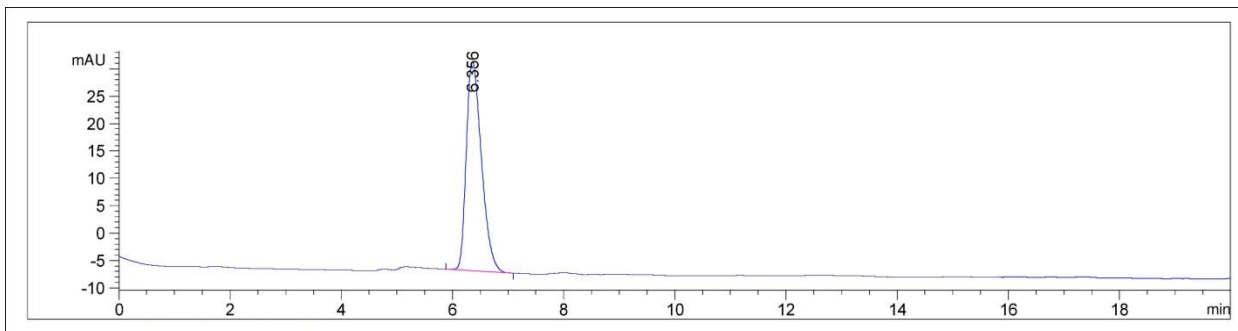


RP-HPLC Chromatogram of equimolar mixture of 3a and 3a\*

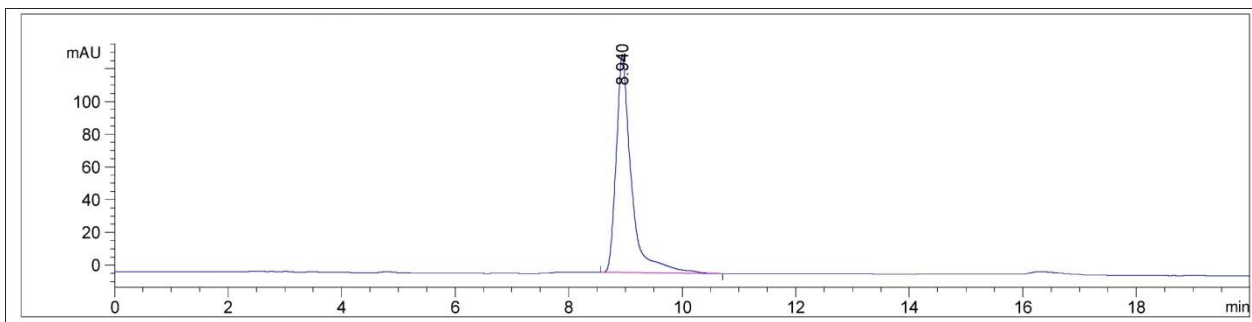
RP-HPLC profiles of (3a) and (3a\*) (method: gradient 0.1% TFA water-acetonitrile (0-100%) in 30 min; VWD at  $\lambda = 254$  nm; flow rate: 1.0 mL/min; column: Agilent Eclipse, XDB-C18, pore size-5  $\mu\text{m}$ , diameter x length = 4.6 x 150 nm).



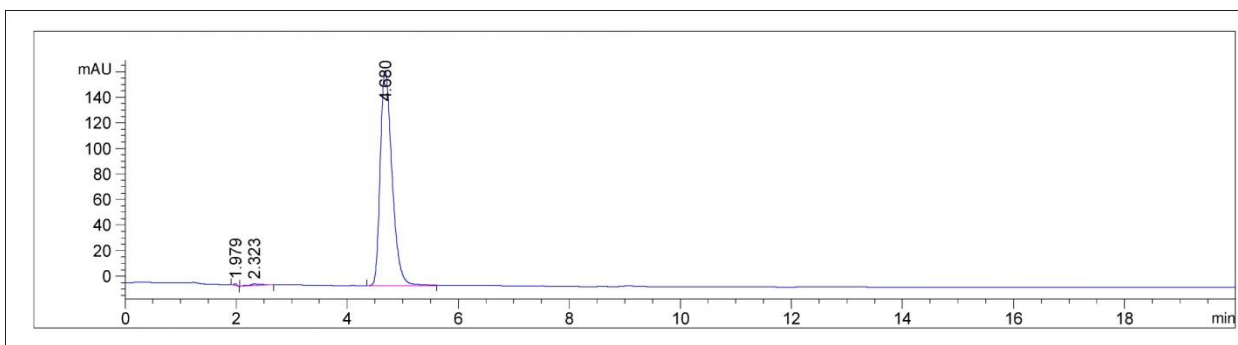
RP-HPLC Chromatogram of 2a



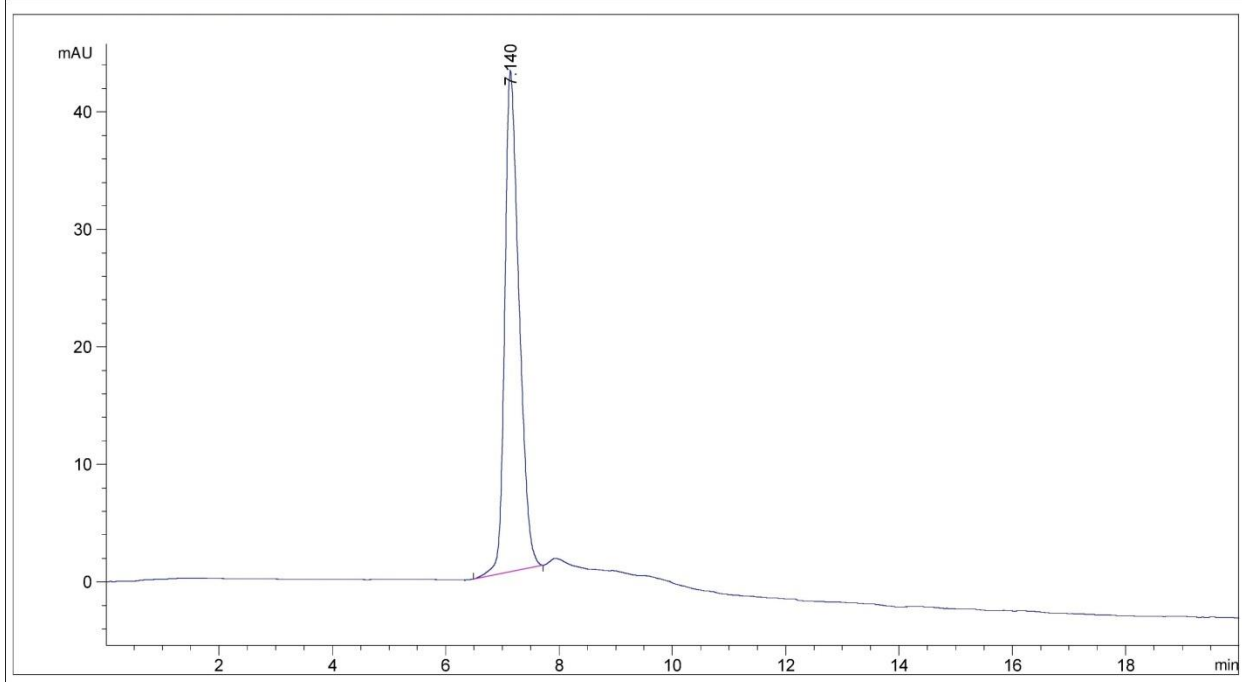
RP-HPLC Chromatogram of 2b



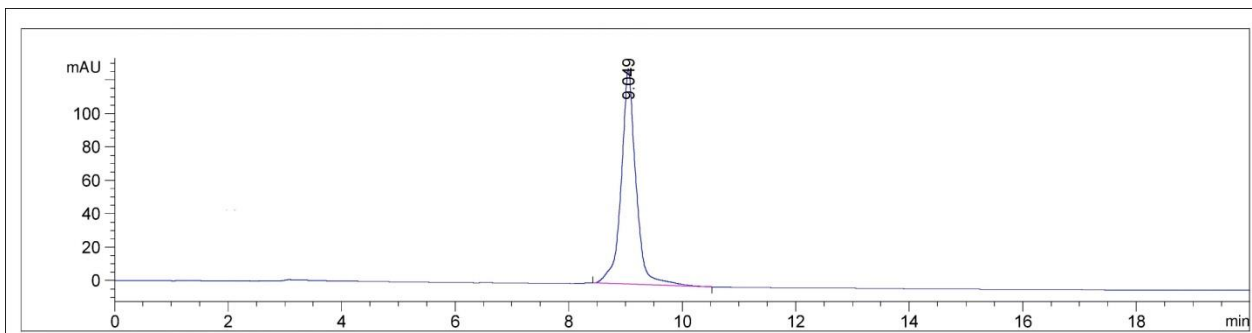
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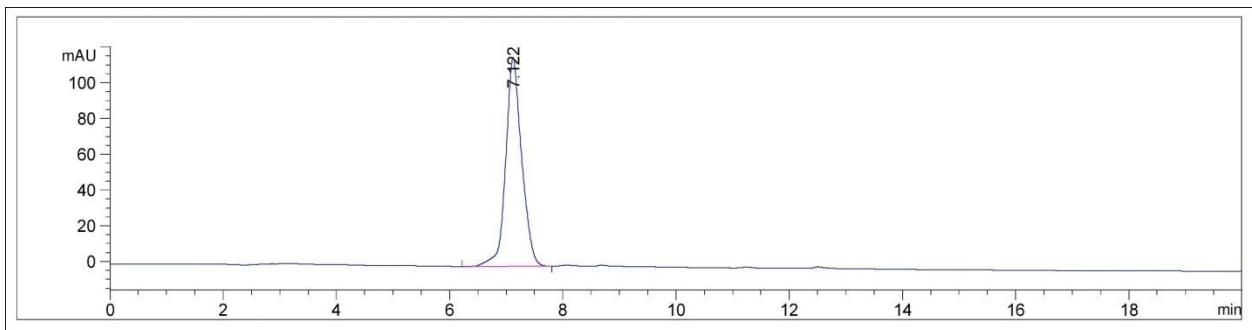
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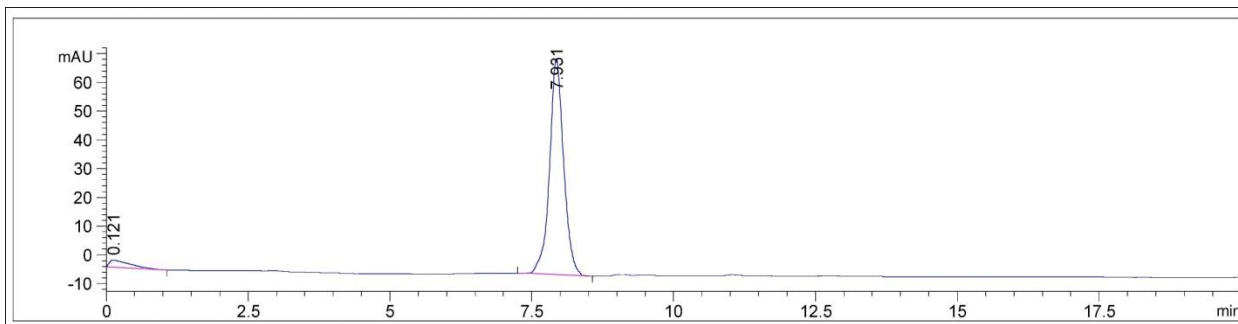
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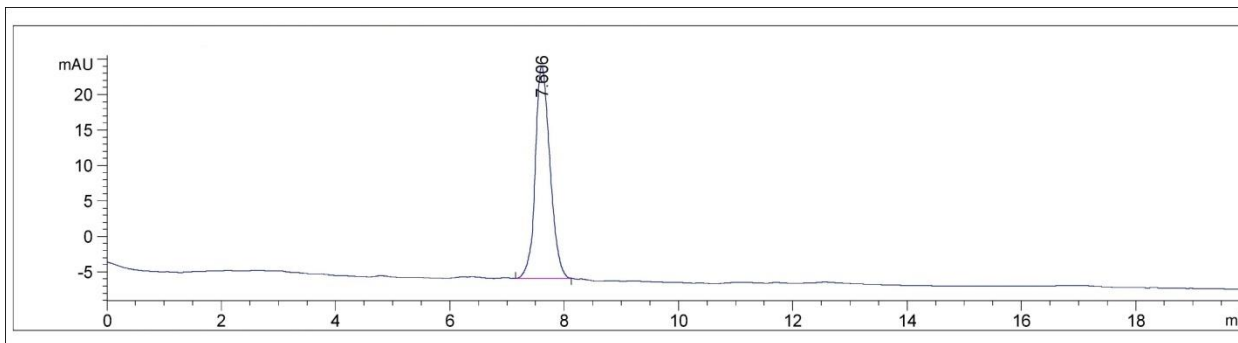
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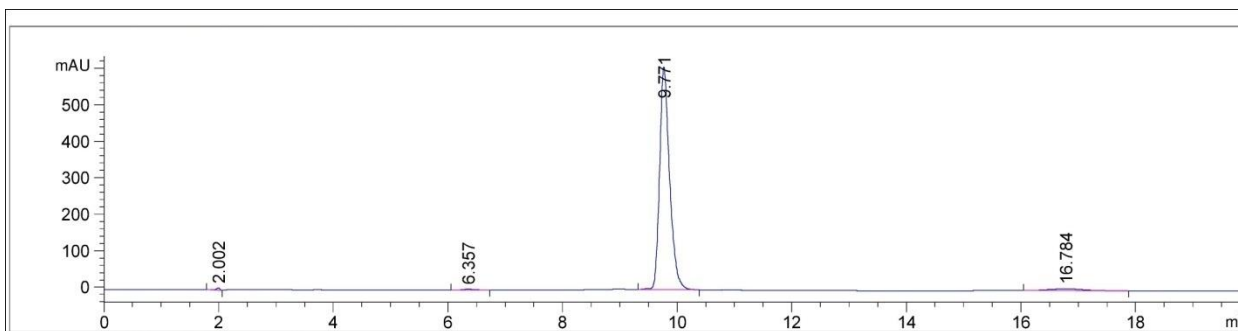
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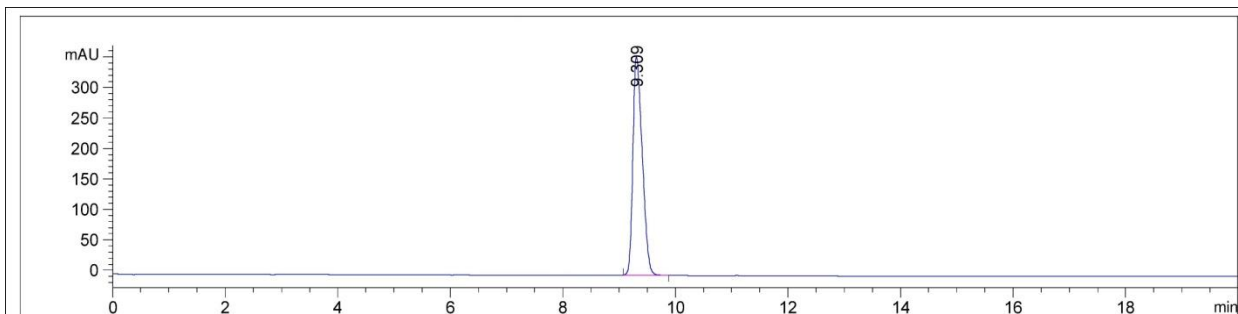
RP-HPLC Chromatogram of 2h



RP-HPLC Chromatogram of 2i

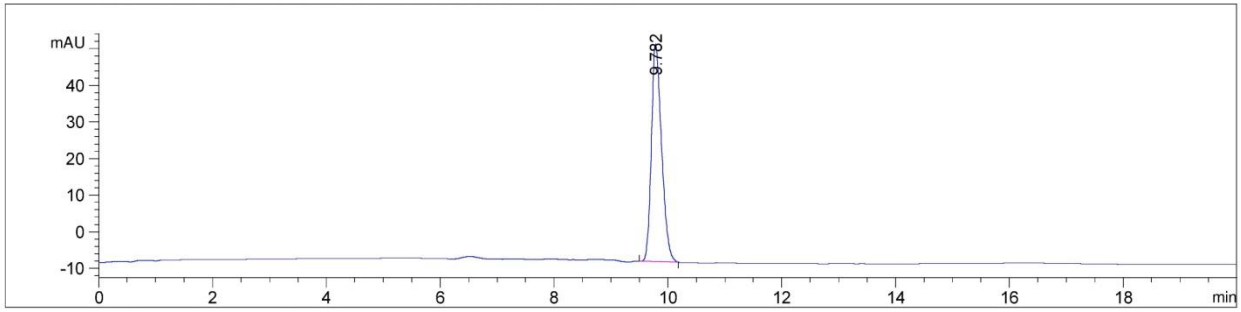


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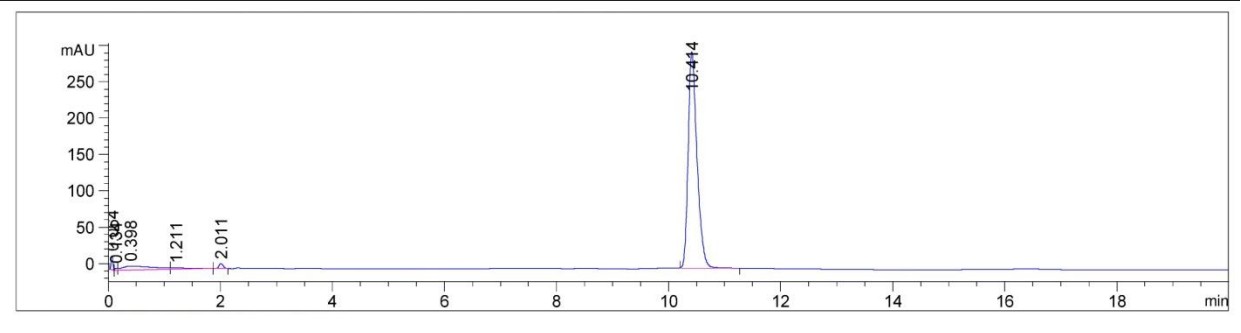


RP-HPLC Chromatogram of 2k

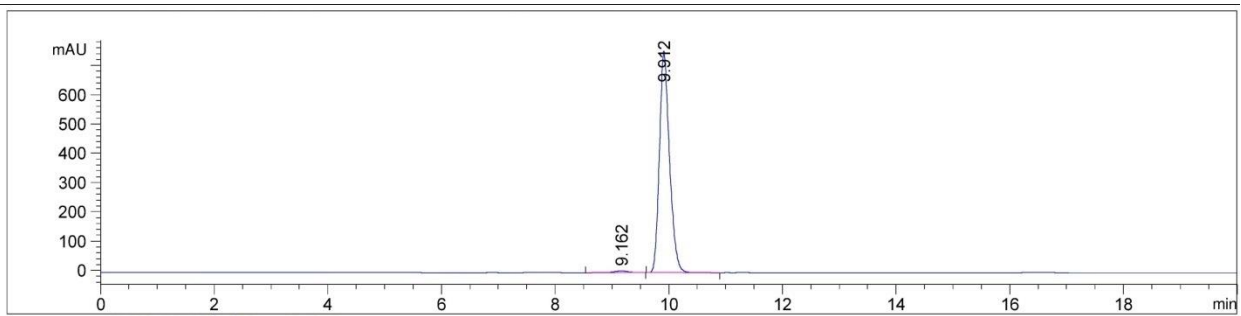




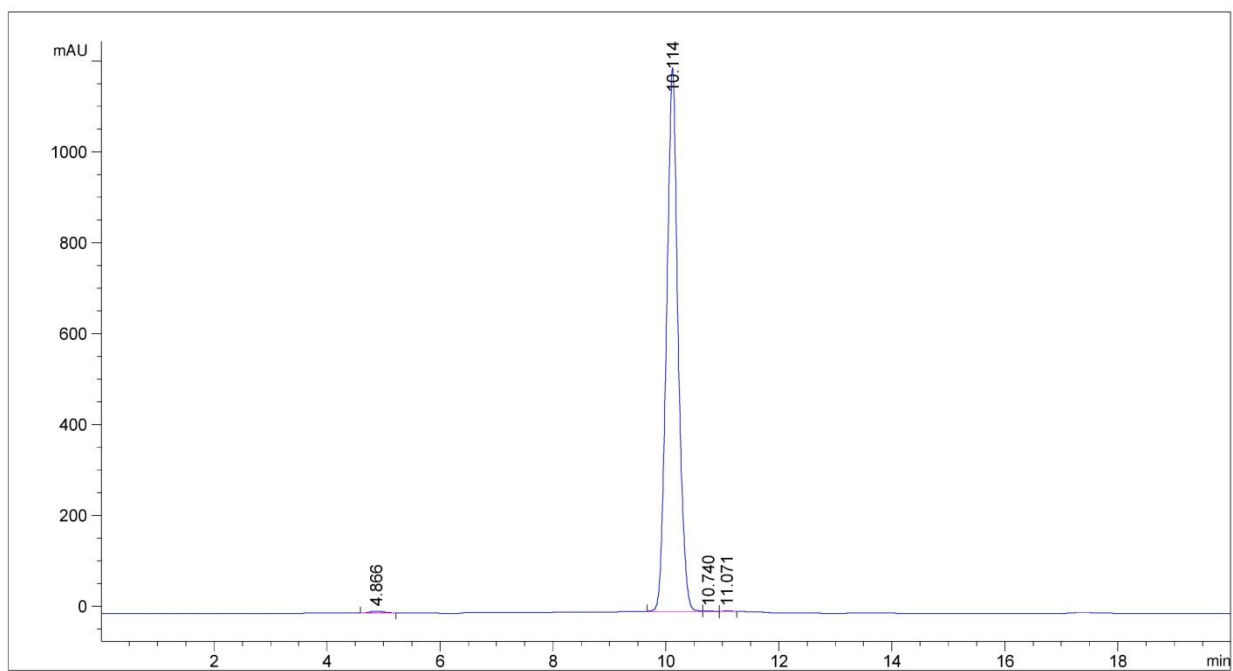
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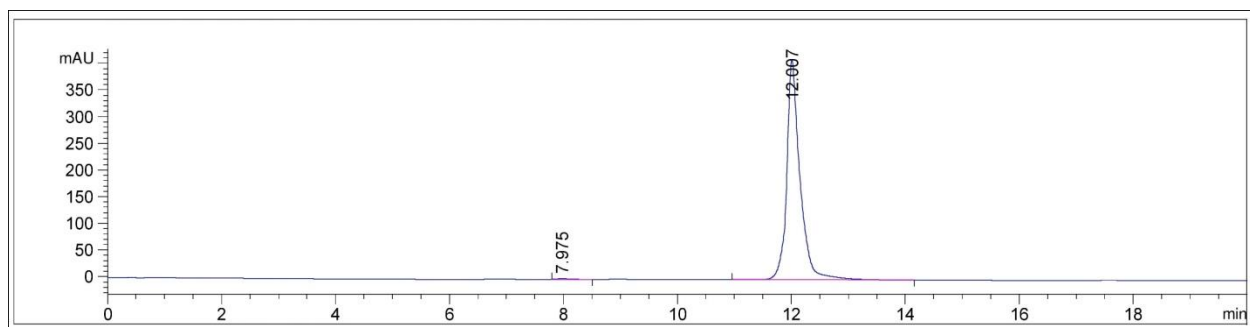
RP-HPLC Chromatogram of 2m



RP-HPLC Chromatogram of 2n



RP-HPLC Chromatogram of 2o



RP-HPLC Chromatogram of 2p

RP-HPLC profiles of 2a-p (method: gradient 0.1% TFA water-acetonitrile (0-100%) in 20 min; VWD at  $\lambda = 254$  nm; flow rate: 1.0 mL/min; column: Agilent Eclipse, XDB-C18, pore size-5  $\mu\text{m}$ , diameter x length = 4.6 x 150 mm).