

## Supporting Information

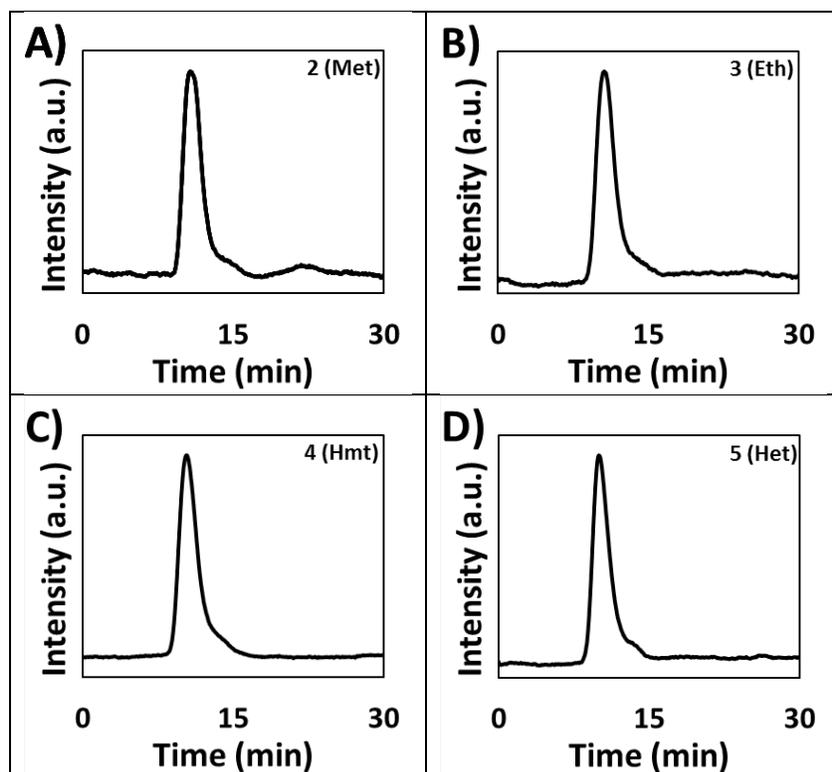
### Influence of sulfoxide group placement on polypeptide conformational stability

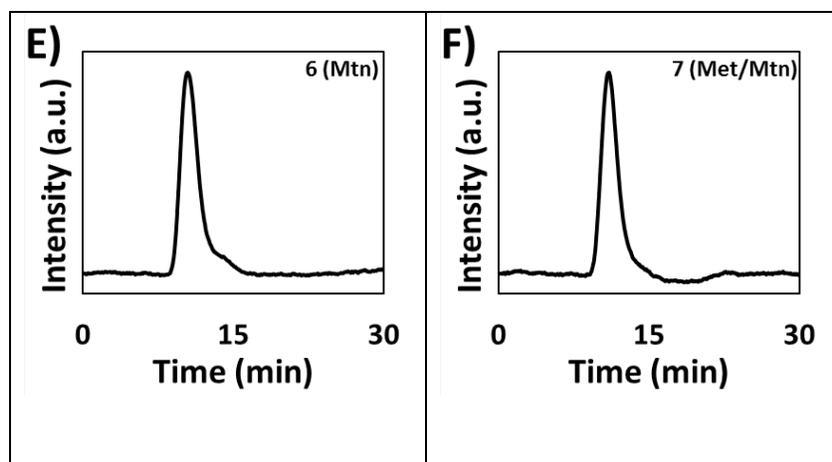
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**Table S1.** Solubility of polypeptides 1-6 in common solvents.

Polymer	HFiP	TFA	CH <sub>3</sub> CN	DMF	CHCl <sub>3</sub>	THF	toluene	alkanes	alcohols
1	-	+	-	-	-	-	-	-	-
2	+	+	-	-	+	+	-	-	-
3	+	+	-	-	+	+	-	-	-
4	+	+	-	-	+	+	-	-	-
5	+	+	-	-	+	+	-	-	-
6	+	+	-	-	+	+	-	-	-

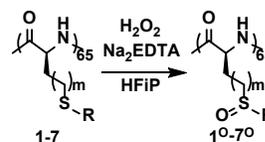
+ = soluble; - = insoluble



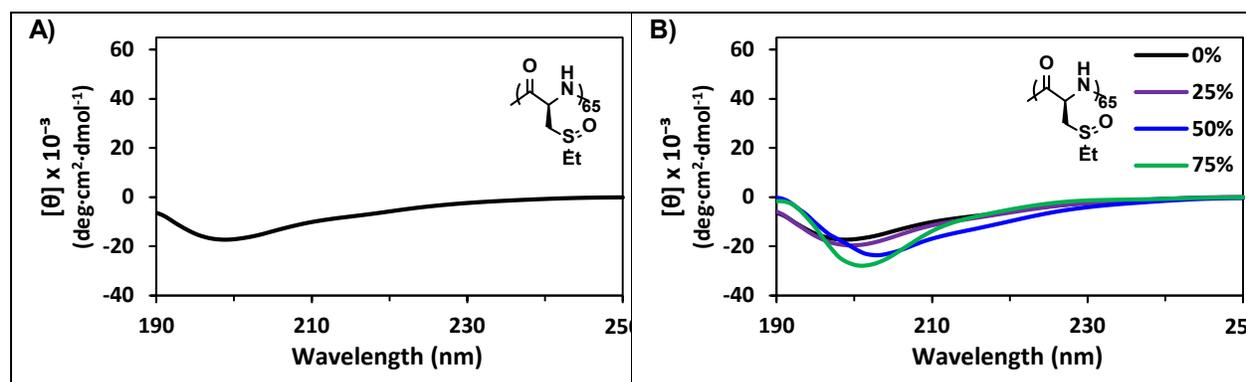


**Figure S1.** GPC Chromatograms of polypeptides **2-7** in 0.5 wt% KTFA in HFiP at 10 mg/mL.

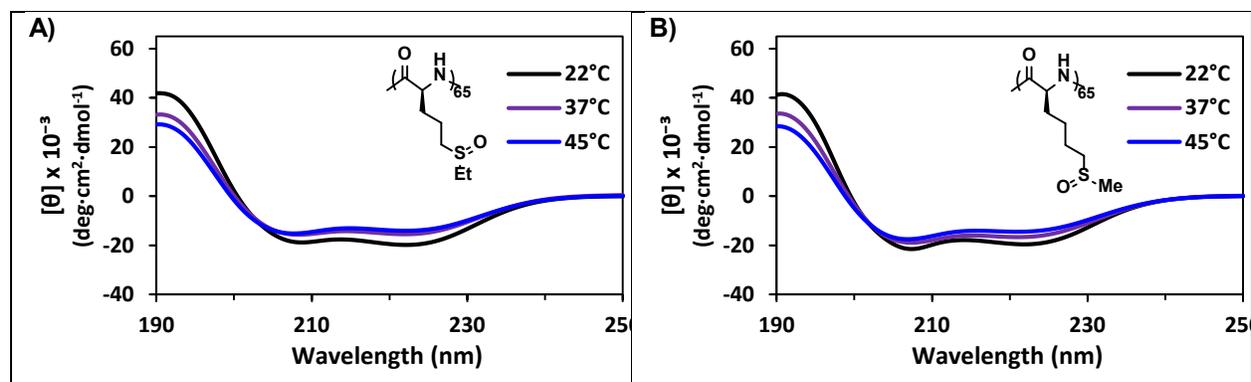
Product	R	m	Yield (%)
<b>1<sup>0</sup></b>	Et	0	90
<b>2<sup>0</sup></b>	Me	1	100
<b>3<sup>0</sup></b>	Et	1	100
<b>4<sup>0</sup></b>	Me	2	100
<b>5<sup>0</sup></b>	Et	2	96
<b>6<sup>0</sup></b>	Me	3	97
<b>7<sup>0</sup></b>	Me	1,3 <sup>a</sup>	98



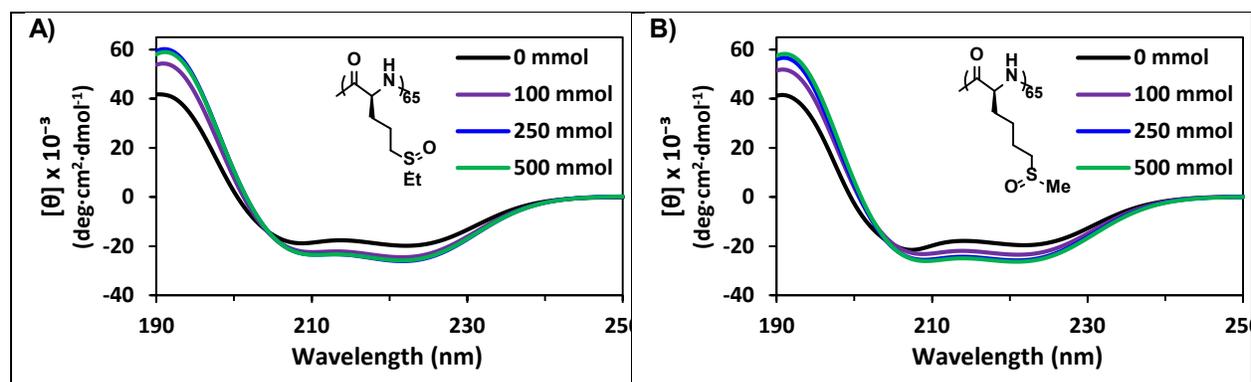
**Figure S2.** Oxidation of thioether containing polypeptides to sulfoxide derivatives. a = 1:1 statistical copolymer.



**Figure S3.** CD Spectra of polypeptide **1<sup>0</sup>**. (A) CD spectrum in H<sub>2</sub>O at 22 °C; (B) CD spectra in aqueous methanol mixtures (volume percent methanol indicated). Note that **1<sup>0</sup>** is not soluble in 92% MeOH. All spectra for polymer solutions (0.4-0.5 mg/mL) were recorded in H<sub>2</sub>O or MeOH/H<sub>2</sub>O mixtures at 22 °C.



**Figure S4.** CD spectra of aqueous polypeptide solutions at different temperatures. (A) CD spectra of  $5^0$  (0.4-0.5 mg/mL) in  $H_2O$ . (B) CD spectra of  $6^0$  (0.4-0.5 mg/mL) in  $H_2O$ . Sample temperatures indicated in plots.



**Figure S5.** CD spectra of aqueous polypeptide solutions at different  $NaClO_4$  concentrations. (A) CD spectra of  $5^0$  (0.4-0.5 mg/mL). (B) CD spectra of  $6^0$  (0.4-0.5 mg/mL).  $NaClO_4$  concentrations indicated in plots.

Polymer	R	m	Helicity (%) at MeOH Concentration (% v/v)				
			0	25	50	75	92
$2^0$	Me	1	2	17	64	87	101
$3^0$	Et	1	3	31	72	90	102
$4^0$	Me	2	28	63	79	98	102
$5^0$	Et	2	58	75	88	100	99
$6^0$	Me	3	58	82	91	99	100
$7^0$	Me	1,3 <sup>a</sup>	11	26	69	93	100

**Figure S6.** Conformations of sulfoxide containing polypeptides in aqueous methanol mixtures.  $\alpha$ -Helix content (helicity) was determined from intensity of minima at 222 nm in CD spectra of polypeptides (0.4-0.5 mg/mL) in MeOH/ $H_2O$  mixtures at 22 °C. a = 1:1 statistical copolymer. **R** and **m** correlate with structures shown in Figure 2.

## 1) Materials and Methods

NCA purifications and polymerizations were performed in a glovebox under N<sub>2</sub> atmosphere. Polypeptide oxidations were performed under ambient atmosphere. Small molecule reactions were performed under N<sub>2</sub> using oven dried glassware. Reactions at elevated temperature were controlled using a Corning PC 420D thermostated hotplate equipped with a thermocouple probe. Dialysis was performed using DI water (18.2 MΩ-cm) prepared by passing in-house reverse osmosis purified water through a Millipore Milli-Q Biocel A10 unit. In all other cases, in-house reverse osmosis purified water was used. Thin-layer chromatography was performed with EMD gel 60 F254 plates (0.25 mm thickness) and visualized using a UV lamp or permanganate stain. Column chromatography was performed using Silicycle Siliaflash G60 silica (60-200 μm). Chromatography eluents are reported as volume percents (v/v). Dialysis was performed using regenerated cellulose dialysis tubing obtained from Spectrum Labs. CD spectra were recorded on either an Olis DSM 10 spectrophotometer or a JASCO J-715 spectrophotometer, using a 0.1 cm path length quartz cell. Percent α-helical content was calculated using % helicity =  $(-[\theta_{222}] + 3000)/39000 \times 100$  where  $[\theta_{222}]$  is the molar ellipticity at 222 nm.<sup>1</sup> Infrared spectra of solution samples were collected using a Perkin Elmer 1605 FTIR Spectrophotometer. ATR-FTIR spectroscopy of solids was collected using a JASCO FT/IR-4100 fitted with an ATR PRO450-S accessory. NMR spectra were recorded on a Bruker AV300, AV400 or DRX500 instruments with chemical shifts reported relative to residual solvent signal. Abbreviations of splitting pattern designations are listed in the abbreviation section. ESI-MS was performed using a Waters LCT Premier spectrometer. NCA samples were prepared in 1% CHCl<sub>3</sub>/THF (1 mg/mL) and injected at a rate of 20 μL/min. Tandem gel permeation chromatography/light scattering (GPC/LS) was performed on an SSI Accuflow Series III liquid chromatograph pump equipped with Wyatt DAWN EOS LS and Optilab rEX refractive index (RI) detectors. Separations were achieved using xStream H<sub>2</sub>O 500 Å Jordi Labs 5 μm mixed bed column with 0.5 wt% potassium trifluoroacetate in HFiP as the eluent at 30 °C. The following reagents were used as received: porcine kidney acylase I (Calzyme), allyl bromide (Alfa), 4-bromo-1-butene (Oakwood), diethyl acetamidomalonate (Oakwood, Sigma), anhydrous DMF (Acros), DMPA (Sigma), ethanethiol (Sigma), ethyl bromide (Alfa), HFiP (Oakwood), H<sub>2</sub>O<sub>2</sub>, 60% NaH (Sigma), phosgene solution (Sigma), *rac*-homocysteine thiolactone hydrochloride (Sigma), D-TFA (Cambridge Isotope), Dowex® 50WX8 (Sigma). THF and hexanes were degassed with N<sub>2</sub> and passed through an alumina column before use. *N*-acetyl-*rac*-homocysteine thiolactone was prepared by acylating *rac*-homocysteine thiolactone hydrochloride with excess Ac<sub>2</sub>O in the presence of stoichiometric TEA; acylations with AcCl unexpectedly formed complex mixtures under a variety of conditions. Methanethiol solutions in MeOH were prepared by reducing an excess of dimethyl disulfide in hexanes with BuLi (1 eq); after concentrating, the residue was dissolved in MeOH, acidified with con. H<sub>3</sub>PO<sub>4</sub> (1 eq) and distilled.

*Abbreviations:* monomer to initiator ratio (M:I), dispersity ( $M_w/M_n$  or  $D$ ), degree of polymerization (DP), circular dichroism (CD), ultraviolet light irradiation ( $h\nu$ ), molecular weight cut-off (MWCO), deionized (DI), room temperature (RT), equivalents (eq), thin layer chromatography (TLC), *N*-carboxyanhydride (NCA), L-methionine (Met), S-ethyl-

L-cysteine (Ecy), L-ethionine (Eth), L-homomethionine (Hmt), L-homoethionine (Het), 6-(methylthio)-L-norleucine (Mtn), L-2,2-dimethoxy-2-phenylacetophenone (DMPA),  $\alpha$ -methoxy- $\omega$ -isocyanoethyl-poly(ethylene glycol)<sub>45</sub> (PEG<sub>45</sub>NCO), liquid nitrogen (LN<sub>2</sub>), isopropyl alcohol (IPA), acetonitrile (MeCN), tetrahydrofuran (THF), chloroform (CHCl<sub>3</sub>), methanol (MeOH), methanethiol (MeSH), ethyl acetate (EtOAc), glacial acetic acid (AcOH), 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP), formic acid (HCOOH), diethyl ether (Et<sub>2</sub>O), dichloromethane (DCM), *N,N*-dimethylformamide (DMF), *N*-methyl-2-pyrrolidone (NMP), potassium hydroxide (KOH), potassium tert-butoxide (KOtBu), potassium trifluoroacetate (KTFA), disodium ethylenediaminetetraacetate dehydrate (Na<sub>2</sub>EDTA), 1x phosphate-buffered saline (PBS), concentrated hydrochloric acid (Con. HCl), broad (br), doublet (d), doublet of doublets (dd), doublet of doublet of quartets (ddq), doublet of quartets (dq), doublet of triplets (dt), multiplet (m), pentet (pent), septet (sep), sextet (sext), quartet (q), quartet of doublet (qd), singlet (s), triplet (t), electrospray ionization-mass spectrometry (ESI-MS).

## 2) General Synthetic Procedures

### Saponification and decarboxylation of diethyl 2-alkyl-2-acetamidomalonates<sup>2,3</sup>

The desired diethyl 2-alkyl-2-acetamidomalonate (1 eq) was suspended in aqueous 1 N KOH (2.6 eq) and the mixture was refluxed with vigorous stirring. The initially biphasic mixture became homogenous after *ca.* 30 min. Heating was maintained for 3 h in total. The mixture was adjusted to pH 3.5 (temperature compensated) with Con. HCl. The mixture was refluxed for 1 h. The pH was readjusted back to pH 3.5 using Con. HCl (*Note:* the pH rises as the decarboxylation progresses), and the mixture was refluxed for an additional 2 h. The solution was then concentrated to about 2/3 its original volume by rotary evaporation. The resultant amber mixture was treated with activated charcoal (0.25 mL/mmol substrate). After stirring for 10 min, the suspension was filtered. The colorless filtrate was adjusted to pH 2.0 with Con. HCl. This was extracted with EtOAc (4x using 1.6 mL per mmol substrate). The organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated by rotary evaporation to provide the *N*-acetyl *rac*-amino acid as a colorless solid.

### *N*-Acetyl *rac*-amino acid kinetic resolution

Performed by modification of a previously reported procedure.<sup>4</sup> An aqueous solution of 300 mM *N*-acetyl *rac*-amino acid and 5 mM KH<sub>2</sub>PO<sub>4</sub> was prepared. The solution was adjusted to pH 7.5-8.0 with aqueous 4 N KOH. The mixture was placed on a 40 °C oil bath and degassed by stirring under N<sub>2</sub> for 15 min. Porcine kidney acylase I (1100 U/mg protein; 4.2 U acylase per mmol of *N*-acetyl amino acid) was added and the reaction mixture was gently stirred. At selected time points 50  $\mu$ L aliquots were removed and analyzed using a ninhydrin colorimetric assay.<sup>5</sup> The pH of the solution was periodically measured and if necessary readjusted to pH 7.5-8.0 by addition of 4 N KOH. When the ninhydrin assay showed no further increase in amino acid concentration, the resolution was suspended by briefly heating the mixture to 60 °C and stirring with activated charcoal (0.25 mL per mmol of substrate). The resulting mixture was vacuum filtered to provide a colorless filtrate, which was then adjusted to pH 1.5 with Con. HCl. The mixture was washed with EtOAc (3x using 1.7 mL/mmol of substrate). The aqueous phase was applied onto a

Dowex® 50WX8 column (1.7 mL/mmol of substrate) and desalted by flushing the column with H<sub>2</sub>O until the eluent became neutral. Desorption of the amino acid was accomplished by eluting with aqueous 1.0 N NH<sub>3</sub>. The L-amino acid was recovered as a colorless solid by rotary evaporation followed by lyophilization.

### **Thiol-ene reactions**

The alkene containing L-amino acid (1 eq) in 50% (v/v) MeOH/H<sub>2</sub>O was treated with the desired thiol (1.5 eq) followed by DMPA (0.025 eq) ([alkene] in reaction mixture was *ca.* 0.11 M). The mixture was stirred under nitrogen for 30 min and subsequently irradiated with an Exo Terra Repti-Glo 5.0 compact fluorescent tropical terrarium lamp for 2.5 h. Substrate specific details and purification procedures are listed in the monomer synthesis section.

### **Amino acid phosgenation**

A suspension of amino acid (300 mM) in THF was prepared and vigorously stirred on a RT water bath under dinitrogen atmosphere. A 1.4 M phosgene solution in toluene (1.75 eq) was added in one portion. The water bath was heated to 45 °C, whereupon the amino acid gradually dissolved. The reaction mixture was stirred for 1 h after becoming limpid (generally 2 h in total). The reaction mixture was cooled to *ca.* -10 °C by briefly immersing into a LN<sub>2</sub> bath, and was subsequently concentrated under vacuum. The residue was purified by chromatography and/or precipitation from THF/Hexanes.<sup>6</sup> The NCA products were recovered as colorless crystalline solids.

### **NCA polymerization**

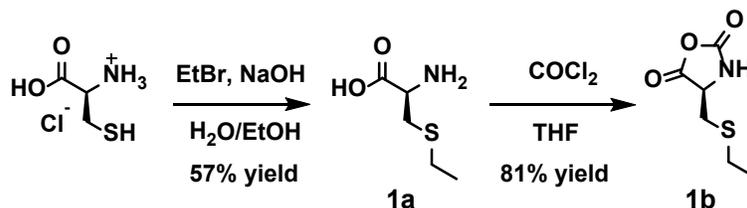
In a dinitrogen filled glove box, a solution of NCA (25-50 mg/mL) in THF was treated with 50 mM Co(PMe<sub>3</sub>)<sub>4</sub> initiator in THF at NCA to give monomer:initiator ratios ranging from 15:1 to 60:1.<sup>7</sup> A small sample of the polymerization mixture was withdrawn after 2 h for analysis by FTIR spectroscopy. In all cases FTIR analysis showed complete consumption of monomer (disappearance of NCA monomer stretch at ~1790 cm<sup>-1</sup>). A small aliquot of the polymerization mixture (150-350 µL) was then removed and then treated with excess PEG<sub>45</sub>NCO. The resulting chain-end modified polypeptide was isolated and analyzed using <sup>1</sup>H NMR to determine DP via end-group analysis.<sup>8</sup> The remainder of the polymerization mixture was removed from the glove box and treated with Con. HCl (40 µL per mL of solution). The bulk of the solvent was evaporated under a gentle stream of air. The precipitated polypeptide was subsequently triturated with H<sub>2</sub>O (1x), 100 mM aqueous Na<sub>2</sub>EDTA (2x) and subsequently H<sub>2</sub>O (2x). In each case centrifugation was used to assist separating the polymer from the supernatant. Finally, residual H<sub>2</sub>O was removed by lyophilization to provide the polypeptide as a colorless solid (> 95% typical yield).

### **Oxidation of polypeptide thioether groups**

A 50 mM solution (based on thioether residues) of polypeptide in HFiP was prepared. Na<sub>2</sub>EDTA (0.07 eq per thioether residue) was added. The solution was briefly stirred, a portion of the Na<sub>2</sub>EDTA remains undissolved. The polypeptide solution was treated with aqueous 9.8 M H<sub>2</sub>O<sub>2</sub> (2.75 eq per thioether residue). The solution was vigorously shaken, then allowed to stand

at room temperature for 18 h. The oxidant was then quenched with a few drops of aqueous 10% NaHSO<sub>3</sub>. Subsequently, the reaction mixture was transferred to a 2 kDa MWCO dialysis bag and dialyzed against H<sub>2</sub>O (36 h, 5 dialysate changes). The retentate was lyophilized to provide the fully oxidized polypeptide sulfoxide (> 95% typical yield).

### 3) Monomer Synthesis



#### S-Ethyl L-cysteine, Ecy (**1a**)

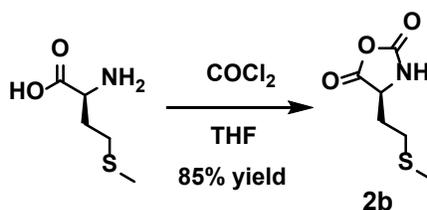
Prepared as previously reported.<sup>9</sup>

<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 3.94 (dd, *J* = 7.6, 4.3 Hz, 1 H), 3.17 (dd, *J* = 14.9, 4.3 Hz, 1 H), 3.06 (dd, *J* = 14.9, 7.6 Hz, 1 H), 2.65 (qd, *J* = 7.5, 1.5 Hz, 2 H), 1.27 (t, *J* = 7.3 Hz, 3 H). [α]<sub>D</sub><sup>24</sup> = -26.8 (c 1.00, H<sub>2</sub>O).

#### S-Ethyl L-cysteine-*N*-carboxyanhydride, Ecy NCA (**1b**)

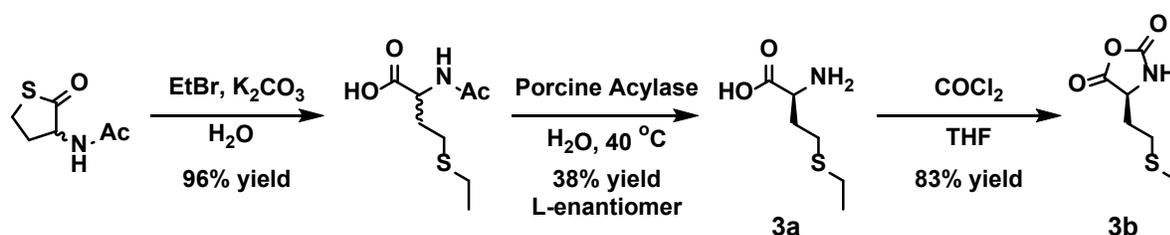
Prepared from *S*-ethyl-L-cysteine (2.0 g, 13 mmol) using the *Amino acid phosgenation* procedure. The crude product was diluted with THF and precipitated into hexanes. The precipitate was isolated by decantation and precipitated again in this manner to provide **2b** (1.9 g, 81 % yield) as a colorless solid. Spectra were in agreement with previously reported data.<sup>9</sup>

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, 25 °C): δ 6.91 (s, 1 H), 4.57 (dd, *J* = 6.4, 3.7 Hz, 1 H), 3.12 (dd, *J* = 14.4, 3.5 Hz, 1 H), 2.88 (dd, *J* = 14.3, 6.6 Hz, 1 H), 2.68-2.57 (m, 2 H), 1.26 (t, *J* = 7.4 Hz, 3 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, 25 °C): δ 168.5, 152.6, 58.0, 33.2, 27.1, 14.7. ESI-MS *m/z* = 174.0364 [M-H]<sup>-</sup> (calcd 174.0225 for C<sub>6</sub>H<sub>8</sub>NO<sub>3</sub>S).



#### L-Methionine-*N*-carboxyanhydride, Met NCA (**2b**)

Prepared from, L-methionine using the *Amino acid phosgenation* procedure. Purified by column chromatography, 25-30% THF/hexanes. *R<sub>F</sub>* = 0.55; 40% THF/hexanes. Spectra were in agreement with previously reported data.<sup>6</sup>



### *N*-Acetyl-*rac*-ethionine

A degassed aqueous 10%  $K_2CO_3$  solution (100 mL, 72 mmol, 2.3 eq) was transferred via cannula into a flask containing *N*-acetyl-*rac*-homocysteine thiolactone (5.0 g, 32 mmol, 1.0 eq). The mixture was stirred until homogenous (5-10 min) and subsequently, ethyl bromide (3.1 mL, 41 mmol, 1.3 eq) was added. After stirring for 18 h, the solution was concentrated to approximately 50 mL by rotary evaporation. The solution pH was adjusted to 1.5 with Con. HCl and the mixture was extracted with EtOAc (4 x 60 mL). The pooled extracts were dried over  $Na_2SO_4$  and concentrated. *N*-acetyl *rac*-ethionine (6.2 g, 96% yield) was recovered as a colorless solid.

$^1H$  NMR (500 MHz,  $CDCl_3$ , 25 °C):  $\delta$  11.61 (br s, 1 H), 6.61 (d,  $J = 7.6$  Hz, 1 H), 4.70 (qm,  $J = 7.6$  Hz, 1 H), 2.59 (t,  $J = 7.3$  Hz, 2 H), 2.55 (q,  $J = 7.7$  Hz, 2 H), 2.20 (m, 1 H), 2.07 (s, 3 H), 2.02 (m, 1 H), 1.24 (t,  $J = 7.3$  Hz, 3 H).

### L-Ethionine, Eth (3a)<sup>10</sup>

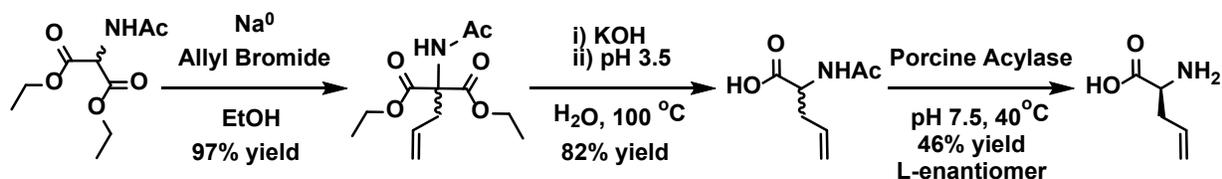
*N*-Acetyl *rac*-ethionine (6.0 g, 29 mmol) was treated using the *N*-Acetyl *rac*-amino acid kinetic resolution procedure to provide L-ethionine (1.8 g, 38% yield of L-enantiomer).  $[\alpha]_D^{24} = +24.1$  (c 2.00, 2.0 N HCl); Lit:  $[\alpha]_D^{25} = +23.7$  (c 2.00, 2.0 N HCl).

$^1H$  NMR (400 MHz,  $D_2O$ , 25 °C):  $\delta$  3.87 (dd,  $J = 7.0, 5.5$  Hz, 1 H), 2.71 (dt,  $J = 6.9, 1.2$  Hz, 2 H), 2.64 (q,  $J = 7.4$  Hz, 2 H), 2.16 (m, 2 H), 1.26 (t, 3 H).

### L-Ethionine-*N*-carboxyanhydride, Eth NCA (3b)

The NCA was prepared from L-ethionine (1.5 g, 9.2 mmol) using the *Amino acid phosgenation* procedure. The crude product was purified by column chromatography, 30% THF/hexanes, and after concentration the residue was diluted with THF and precipitated into hexanes to provide **3b** (1.4 g, 83% yield).  $R_F = 0.66$ ; 40% THF/hexanes.

$^1H$  NMR (400 MHz,  $CDCl_3$ , 25 °C):  $\delta$  7.04 (s, 1 H), 4.51 (dd,  $J = 6.7, 5.2$  Hz, 1 H), 2.69 (dt,  $J = 9.0, 6.7$  Hz, 2 H), 2.54 (q,  $J = 7.4$  Hz, 2 H), 2.24 (m, 1 H), 2.07 (dq,  $J = 14.5, 7.3$  Hz, 1 H), 1.24 (t,  $J = 7.4$  Hz, 3 H).  $^{13}C$  NMR (100 MHz,  $CDCl_3$ , 25 °C):  $\delta$  169.8, 152.8, 56.7, 30.8, 27.1, 25.6, 14.5. ESI-MS  $m/z = 188.0486$   $[M-H]^-$  (calcd 188.0381 for  $C_7H_{10}NO_3S$ ).



### Diethyl 2-allyl-2-acetamidomalonate

Prepared as previously reported<sup>11</sup> (97 % yield).  $R_F = 0.59$ ; 50% EtOAc/Hexanes.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C):  $\delta$  6.73 (s, 1 H), 5.56 (ddt,  $J = 14.9, 9.6, 7.5$  Hz, 1 H), 5.11 (m, 2 H), 4.25 (qd,  $J = 7.2, 0.9$  Hz, 4 H), 3.07 (dt,  $J = 7.5, 1.0$  Hz, 2 H), 2.03 (s, 3 H), 1.25 (t,  $J = 7.1$  Hz, 6 H).

### *N*-Acetyl-*rac*-allylglycine

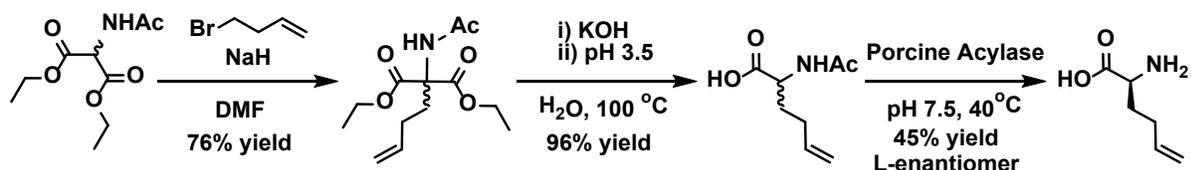
Prepared from diethyl 2-allyl-2-acetamidomalonate (10 g, 39 mmol), using the *Saponification and decarboxylation of diethyl 2-alkyl-2-acetamidomalonates* procedure to provide *N*-acetyl-*rac*-allylglycine (5.1 g, 82 % yield) as a colorless solid. Spectra were in agreement with previously reported data.<sup>2</sup>

$^1\text{H NMR}$  (300 MHz,  $\text{D}_2\text{O}$ , 25 °C):  $\delta$  5.78 (ddt,  $J = 17.2, 10.1, 7.0$  Hz, 1 H), 5.18 (m, 2 H), 4.43 (dd,  $J = 7.8, 5.2$  Hz, 1 H), 2.57 (m, 2 H), 2.02 (s, 3 H).

### L-Allylglycine

Prepared from *N*-acetyl-*rac*-allylglycine (46 g, 290 mmol) using the *N*-Acetyl *rac*-amino acid kinetic resolution procedure to provide L-allylglycine (15.3 g, 46% yield of L-enantiomer) as a colorless solid. Spectra were in agreement with previously reported data.<sup>12</sup>

$^1\text{H NMR}$  (400 MHz,  $\text{D}_2\text{O}$ , 25 °C):  $\delta$  5.80 (ddt,  $J = 17.2, 10.1, 7.3$  Hz, 1 H), 5.30 (m, 2 H), 3.82 (dd,  $J = 7.1, 4.9$  Hz, 1 H), 2.66 (m, 2 H).  $[\alpha]_{\text{D}}^{24} = -37.9$  (c 4.00,  $\text{H}_2\text{O}$ ); Lit:  $[\alpha]_{\text{D}}^{24} = -37.1$  (c 4.00,  $\text{H}_2\text{O}$ ).



### Diethyl 2-(3-butenyl)-2-acetamidomalonate

Synthesized by modification of a previously reported procedure.<sup>3,13</sup> A solution of diethyl 2-acetamidomalonate (45 g, 210 mmol, 1.0 eq) in DMF (300 mL) was made. Separately, a suspension of 60% NaH (9.1 g, 230 mmol, 1.1 eq) in DMF (100 mL) was prepared and stirred on a RT water bath. The malonate solution was cannula transferred into the hydride suspension (*caution*: exothermic). The resulting mixture was stirred 10 min. 4-bromobutene (23 mL, 230 mmol, 1.1 eq) was added in one portion. The mixture was stirred for 16 h on a 60 °C oil bath. The reaction was quenched with AcOH (2 mL) and concentrated by rotary evaporation, and then under high vacuum. The residue was dissolved in  $\text{Et}_2\text{O}$  (250 mL) and washed with  $\text{H}_2\text{O}$  (2x 200 mL). The organic phase was dried over  $\text{MgSO}_4$  and concentrated. This crude material was purified by flash chromatography (25-35%  $\text{EtOAc}$ /hexanes) to provide diethyl 2-(3-butenyl)-2-acetamidomalonate as a pale yellow solid (43 g, 76 % yield).  $R_F = 0.32$ ; 25%  $\text{EtOAc}$ /hexanes.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C):  $\delta$  6.79 (s, 1 H), 5.75 (ddq,  $J = 17.1, 10.3, 6.5$  Hz, 1 H), 5.03 (dq,  $J = 17.1, 1.7$  Hz, 1 H), 4.97 (dq,  $J = 10.2, 1.5$  Hz, 1 H), 4.25 (q,  $J = 7.1$  Hz, 4 H), 2.45 (m, 2 H), 2.04 (s, 3 H), 1.92 (m, 2 H), 1.25 (t,  $J = 7.0$ , 6 H).

### ***N*-Acetyl-*rac*-homoallylglycine**

Prepared from diethyl 2-(3-butenyl)-2-acetamidomalonate (13 g, 49 mmol), using the *Saponification and decarboxylation of diethyl 2-alkyl-2-acetamidomalonates* procedure to provide *N*-acetyl-*rac*-homoallylglycine (7.7 g, 96 % yield) as a colorless solid. Spectra were in agreement with previously reported data.<sup>13</sup>

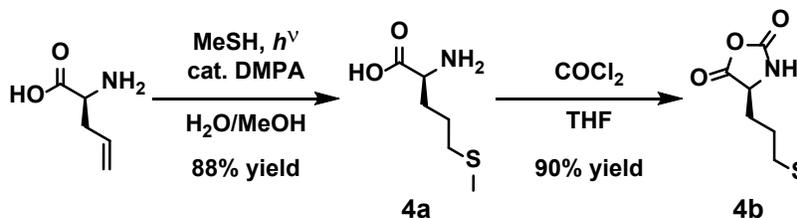
<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 5.89 (ddq, *J* = 17.2, 10.3, 6.8 Hz, 1 H), 5.12 (dq, *J* = 17.2, 1.7 Hz, 1 H), 5.06 (m, 1 H), 4.35 (dd, *J* = 9.5, 4.7 Hz, 1 H), 2.21 (m, 2 H), 2.06 (s, 3 H), 2.01 (m, 1 H), 1.86 (m, 1 H).

### **L-Homoallylglycine**

Prepared from *N*-acetyl-*rac*-homoallylglycine (21 g, 130 mmol) using the *N*-Acetyl *rac*-amino acid kinetic resolution procedure to provide L-homoallylglycine (7.2 g, 45% yield of L-enantiomer) as a colorless solid. Spectra were in agreement with previously reported data.<sup>3,13</sup>

<sup>1</sup>H NMR (400 MHz, 0.5 % D-TFA/D<sub>2</sub>O, 25 °C): δ 5.80 (m, 1 H), 5.09 (m, 1 H), 5.02 (m, 1 H), 4.02 (m, 1 H), 2.15 (m, 2 H), 2.0 (m, 2 H).

$[\alpha]_{\text{D}}^{24} = +27.4$  (c 1.00, 1 N HCl).



### **L-Homomethionine, Hmt (4a)**

Prepared from L-allylglycine (2.0 g, 17 mmol) using methanethiol (1.0 M solution in MeOH) by the *Thiol-ene Reactions* procedure ([alkene] in reaction mixture: 0.15 M). After irradiation, the reaction mixture was concentrated and recrystallized from 50% (v/v) EtOH/H<sub>2</sub>O to provide 4a (2.5 g, 88% yield).

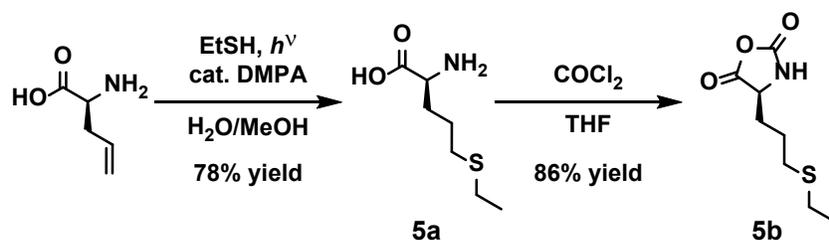
<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 4.07 (t, *J* = 6.3 Hz, 1 H), 2.54 (t, *J* = 7.1 Hz, 2 H), 2.04 (s, 3 H), 2.00 (m, 2 H), 1.71 (m, 2 H).

$[\alpha]_{\text{D}}^{24} = +23.0$  (c 0.30, 6.0 N HCl); Lit:  $[\alpha]_{\text{D}}^{25.5} = +21.0$  (c 0.30, 6.0 N HCl).<sup>14</sup>

### **L-Homomethionine-*N*-carboxyanhydride, Hmt NCA (4b)**

Prepared from L-homomethionine (2.4 g, 15 mmol) using the *Amino acid phosgenation* procedure. The crude product was purified by column chromatography, 30-35% THF/hexanes, and after concentration the residue was diluted with THF and precipitated into hexanes. The precipitate was collected by decantation and precipitated again in the same manner to provide 4b (2.5 g, 90% yield). *R<sub>F</sub>* = 0.52; 40% THF/hexanes.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, 25 °C): δ 7.06 (s, 1 H), 4.39 (t, *J* = 6.1 Hz, 1 H), 2.53 (t, *J* = 7.0 Hz, 2 H), 2.07 (s, 3 H), 2.06-1.86 (m, 2 H), 1.83-1.63 (m, 2 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, 25 °C): δ 169.6, 153.0, 57.4, 33.3, 30.6, 24.0, 15.4. ESI-MS *m/z* = 188.0487 [M-H]<sup>-</sup> (calcd 188.0381 for C<sub>7</sub>H<sub>10</sub>NO<sub>3</sub>S).



### L-Homoethionine, Het (5a)

Prepared from L-allylglycine (1.0 g, 8.7 mmol) using ethanethiol by the *Thiol-ene Reactions* procedure ([alkene] in reaction mixture: 0.17 M). After irradiation, the reaction mixture was concentrated to dryness and the residue was recrystallized from 50% (v/v) EtOH/H<sub>2</sub>O to provide **5a** (1.2 g, 78% yield).

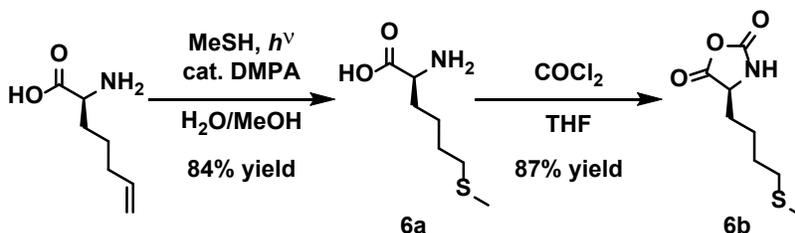
<sup>1</sup>H NMR (400 MHz, 1% D-TFA/D<sub>2</sub>O, 25 °C):  $\delta$  4.02 (t,  $J$  = 6.4 Hz, 1 H), 2.54 (t,  $J$  = 7.1 Hz, 2 H), 2.49 (q,  $J$  = 7.4 Hz, 2 H), 1.98 (m, 2 H), 1.66 (m, 2 H), 1.12 (t,  $J$  = 7.5 Hz, 3 H).

<sup>13</sup>C NMR (100 MHz, 1% D-TFA/D<sub>2</sub>O, 25 °C):  $\delta$  171.6, 52.4, 29.7, 28.7, 25.0, 24.1, 13.8. ESI-MS  $m/z$  = 178.0911 [M+H]<sup>+</sup> (calcd 178.0902 for C<sub>7</sub>H<sub>16</sub>NO<sub>2</sub>S). [ $\alpha$ ]<sub>D</sub><sup>24</sup> = +25.3 (c 1.00, 6 N HCl).

### L-Homoethionine-N-carboxyanhydride, Het NCA (5b)

Prepared from, L-homoethionine (1.2 g, 6.8 mmol) using the *Amino acid phosgenation* procedure. The crude product was purified by column chromatography, 25% THF/hexanes, and after concentration the residue was diluted with THF and precipitated into hexanes. The precipitate was collected by decantation and precipitated again in the same manner to provide **5b** (1.2 g, 87% yield).  $R_F$  = 0.57; 35% THF/hexanes.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, 25 °C):  $\delta$  6.86 (br s, 1 H), 4.36 (t,  $J$  = 6.3 Hz, 1H), 2.56 (t,  $J$  = 7.0 Hz, 2 H), 2.50 (q,  $J$  = 7.4 Hz, 2 H), 2.04 (m, 1 H), 1.92 (m, 1 H), 1.72 (m, 2 H), 1.22 (t,  $J$  = 7.4 Hz, 3 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, 25 °C):  $\delta$  169.6, 153.0, 57.5, 31.0, 30.9, 26.1, 24.7, 14.9. ESI-MS  $m/z$  = 188.0487 [M-H]<sup>-</sup> (calcd 188.0381 for C<sub>7</sub>H<sub>10</sub>NO<sub>3</sub>S).



### 6-(Methylthio)-L-norleucine, Mtn (6a)

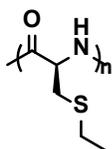
Prepared from L-homoallylglycine (2.8 g, 22 mmol) using methanethiol (1.4 M solution in MeOH) by the *Thiol-ene reactions* procedure. After irradiation the reaction mixture was concentrated and recrystallized from 50% (v/v) EtOH/H<sub>2</sub>O to provide **6a** (3.2 g, 84% yield). <sup>1</sup>H NMR (400 MHz, 0.5% D-TFA/D<sub>2</sub>O, 25 °C):  $\delta$  4.06 (t,  $J$  = 6.1 Hz, 1 H), 2.53 (t,  $J$  = 7.2 Hz, 2 H), 2.05 (s, 3 H), 1.95 (m, 2 H), 1.63 (p,  $J$  = 7.2 Hz, 2 H), 1.48 (m, 2 H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, 25 °C):  $\delta$  169.7, 153.0, 57.8, 33.8, 31.5, 28.4, 23.9, 15.7. ESI-MS  $m/z$  = 178.0919 [M+H]<sup>+</sup> (calcd 178.0902 for C<sub>7</sub>H<sub>16</sub>NO<sub>2</sub>S). [ $\alpha$ ]<sub>D</sub><sup>24</sup> = +26.5 (c 1.00, 6.0 N HCl).

### 6-(Methylthio)-L-norleucine N-carboxyanhydride, Mtn NCA (6b)

Prepared from 6-(methylthio)-L-norleucine (3.2 g, 18 mmol) using the *Amino acid phosgenation* procedure. The crude product was purified by column chromatography, 20-25% THF/hexanes, and after concentration the residue was diluted with THF and precipitated into hexanes. The precipitate was collected by decantation and precipitated again in the same manner to provide **6b** (3.2 g, 87% yield).  $R_F = 0.54$  35% THF/hexanes.

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C):  $\delta$  6.84 (s, 1H), 4.35 (dd,  $J = 7.1, 5.3$  Hz, 1 H), 2.51 (t,  $J = 7.1$  Hz, 2 H), 2.08 (s, 3 H), 2.00-1.77 (m, 2 H), 1.70-1.43 (m, 4 H).  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C):  $\delta$  169.5, 152.8, 57.6, 33.7, 31.3, 28.2, 23.7, 15.5. ESI-MS  $m/z = 202.0527$  [M-H] $^-$  (calcd 202.0538 for  $\text{C}_8\text{H}_{12}\text{NO}_3\text{S}$ ).

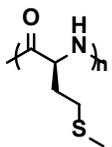
#### 4) Details of Specific Thioether Containing Polypeptides



##### **Poly(S-ethyl-L-cysteine), poly(Ecy), 1**

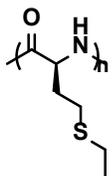
Prepared from **1b** using the *NCA polymerization* procedure. A slight modification to that procedure was made: the NCA stock solution was prepared in NMP at 15 mg/mL as this solvent better solubilized the polypeptide compared to THF. After completion of polymerization, the NMP was removed *in vacuo* before triturating the polymer with water, followed by lyophilization (95% yield).

$^1\text{H NMR}$  (400 MHz, D-TFA, 25 °C):  $\delta$  4.85 (t,  $J = 6.9$ , 1 H), 3.07 (br m, 2 H), 2.64 (q,  $J = 7.3$  Hz, 2 H), 1.25 (t,  $J = 7.4$  Hz, 3 H).



##### **Poly(L-methionine), poly(Met), 2**

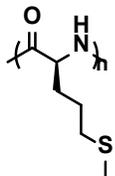
Prepared from **2b** using the *NCA polymerization* procedure (97% yield). Spectra were consistent with previously reported data.<sup>6</sup>



##### **Poly(L-ethionine), poly(Eth), 3**

Prepared from **3b** using the *NCA polymerization* procedure (99% yield).

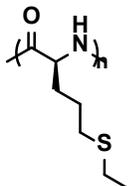
$^1\text{H NMR}$  (400 MHz, D-TFA, 25 °C):  $\delta$  4.82 (t,  $J = 8.1$  Hz, 1 H), 2.70 (m, 2 H), 2.62 (q,  $J = 7.2$  Hz, 2 H), 2.16 (m, 2 H), 1.25 (t,  $J = 7.4$  Hz, 3 H).



**Poly(L-homomethionine), poly(Hmt), 4**

Prepared from **4b** using the *NCA polymerization* procedure (98% yield).

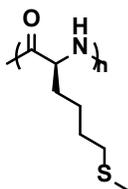
$^1\text{H NMR}$  (400 MHz, D-TFA, 25 °C):  $\delta$  4.65 (dd,  $J = 8.6, 5.4$  Hz, 1 H), 2.59 (p,  $J = 7.25$  Hz, 2 H), 2.12 (s, 3 H), 2.05-1.82 (br m, 2 H), 1.82-1.59 (br m, 2 H).



**Poly(L-homoethionine), poly(Het), 5**

Prepared from **5b** using the *NCA polymerization* procedure (99% yield).

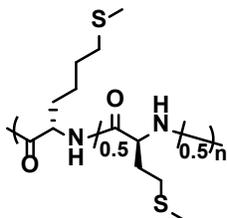
$^1\text{H NMR}$  (400 MHz, D-TFA, 25 °C):  $\delta$  4.70 (dd,  $J = 5.7, 8.4$  Hz, 1 H), 2.81-2.54 (br m, 4 H), 2.11-1.87 (br m, 2 H), 1.87-1.67 (br m, 2 H), 1.30 (t,  $J = 7.24$  Hz, 3 H).



**Poly(6-(methylthio)-L-norleucine), poly(Mtn), 6**

Prepared from **6b** using the *NCA polymerization* procedure (100% yield).

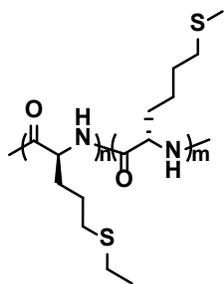
$^1\text{H NMR}$  (400 MHz, D-TFA, 25 °C):  $\delta$  4.61 (dd,  $J = 8.0, 6.2$  Hz, 1 H), 2.57 (p,  $J = 6.6$  Hz, 2 H), 2.12 (s, 3 H), 1.84 (br m, 2 H), 1.66 (p,  $J = 6.9$  Hz, 2 H), 1.49 (br m, 2 H).



**Poly[(6-(methylthio)-L-norleucine) $_{0.50}$ -stat-(L-methionine) $_{0.50}$ ], poly(Mtn $_{0.50}$ /Met $_{0.50}$ ), 7**

Prepared from **2b** and **6b** by slight modification of the *NCA polymerization* procedure. A **2b** stock solution (43 mg/mL, 0.25 M) was diluted with an equal volume of **6b** stock solution (50 mg/mL, 0.25 M). Initiator solution was then added at the desired M:I, and no further deviations were made from the general procedure (97% yield).

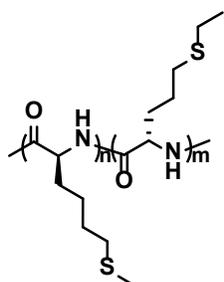
$^1\text{H NMR}$  (300 MHz, D-TFA, 25 °C):  $\delta$  4.83 (m, 0.5 H), 4.82 (m, 0.5 H), 2.57 (m, 2 H), 2.40-1.72 (br m, 5 H), 1.71-1.37 (br m, 2 H).



**Poly(L-homoethionine)<sub>n</sub>-block-poly(6-(methylthio)-L-norleucine)<sub>m</sub>, poly(Het)<sub>n</sub>-b-poly(Mtn)<sub>m</sub>**

Prepared from **5b** and **6b** by slight modification of the *NCA polymerization* procedure. First, **5b** was polymerized following the general procedure. After completion of the first segment (**poly(Het)**) was confirmed by FTIR spectroscopy, an aliquot was removed for chain length analysis. The polymerization mixture was then treated with a 3-fold molar excess (relative to **5b**) of **6b** in THF. The polymerization of the second block was allowed to reach completion (< 2 h) as confirmed by FTIR spectroscopy. The copolymer was subsequently isolated without further deviation from the general procedure (97% yield). The DP and  $M_n$  of each segment were determined by end-group analysis via  $^1\text{H NMR}$ , and  $D (M_w/M_n)$  of the final copolymer was determined by GPC.

$^1\text{H NMR}$  (400 MHz, D-TFA, 25 °C):  $\delta$  4.90-4.63 (br m, 1.33 H), 2.90-2.64 (br m, 3.33 H), 2.35 (s, 3 H), 2.20-1.46 (br m, 7.33 H), 1.42 (t,  $J = 7.7$  Hz, 1 H).

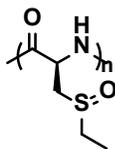


**Poly(6-(methylthio)-L-norleucine)<sub>n</sub>-block-poly(L-homoethionine)<sub>m</sub>, poly(Mtn)<sub>n</sub>-b-poly(Het)<sub>m</sub>**

Prepared as above, except **6b** was used as the monomer for the first block, which was then treated with a 3-fold excess of **5b** to furnish the diblock copolymer (97% yield). The DP and  $M_n$  of each segment were determined by end-group analysis via  $^1\text{H NMR}$ , and  $D (M_w/M_n)$  of the final copolymer was determined by GPC.

$^1\text{H NMR}$  (400 MHz, D-TFA, 25 °C):  $\delta$  4.86-4.60 (br m, 1.33 H), 3.02-2.47 (br m, 4.66 H), 2.46-2.14 (br s, 1 H), 2.11-1.49 (br m, 6 H), 1.33 (t,  $J = 7.4$  Hz, 3 H).

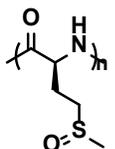
## 5) Details of Specific Sulfoxide Containing Polypeptides



### Poly(*S*-ethyl-L-cysteine sulfoxide), poly(Ecy<sup>O</sup>), 1<sup>O</sup>

Prepared from **1** using the *Oxidation of polypeptide thioether groups* procedure (90% yield).

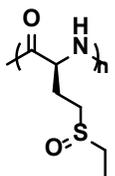
<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 5.04-4.82 (br m, 1 H), 3.64-3.15 (br m, 2 H), 3.15-2.80 (br m, 2 H), 1.36 (td, *J* = 7.3, 2.0 Hz, 3 H).



### Poly(L-methionine sulfoxide), poly(Met<sup>O</sup>), 2<sup>O</sup>

Prepared from **2** using the *Oxidation of polypeptide thioether groups* procedure (100% yield).

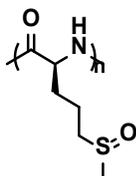
<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 4.61-4.40 (br m, 1 H), 3.11-2.88 (br m, 2 H), 2.71 (m, 3 H), 2.41-2.06 (br m, 2 H).



### Poly(L-ethionine sulfoxide), poly(Eth<sup>O</sup>), 3<sup>O</sup>

Prepared from **3** using the *Oxidation of polypeptide thioether groups* procedure (100% yield).

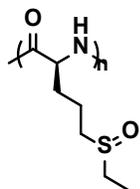
<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 4.56-4.40 (br m, 1 H), 3.22-2.71 (br m, 4 H), 2.50-2.01 (br m, 2 H), 1.33 (t, *J* = 7.4 Hz, 3 H). ATR-FTIR: 1640, 1546, 1032, 1011 cm<sup>-1</sup>.



### Poly(L-homomethionine sulfoxide), poly(Hmt<sup>O</sup>), 4<sup>O</sup>

Prepared from **4** using the *Oxidation of polypeptide thioether groups* procedure (100% yield).

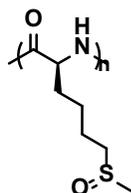
<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 4.50-4.73 (br m, 1 H), 3.10-2.84 (br m, 2 H), 2.71 (br s, 3 H), 2.32-1.64 (br m, 4 H). ATR-FTIR: 1640, 1546, 1035, 1011 cm<sup>-1</sup>.



**Poly(L-homoethionine sulfoxide), poly(Het<sup>O</sup>), 5<sup>O</sup>**

Prepared from **5** using the *Oxidation of polypeptide thioether groups* procedure (96% yield).

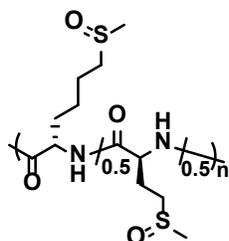
<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 4.40-4.00 (br m, 1 H), 3.13-2.74 (br m, 4 H), 2.37-1.66 (br m, 4 H), 1.33 (t, *J* = 7.4 Hz, 3 H). ATR-FTIR: 1648, 1541, 1013 cm<sup>-1</sup>.



**Poly(6-(methylthio)-L-norleucine), poly(Mtn<sup>O</sup>), 6<sup>O</sup>**

Prepared from **6** using the *Oxidation of polypeptide thioether groups* procedure (97% yield).

<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): δ 4.49-3.95 (br m, 1 H), 2.93 (br s, 1 H), 2.72 (s, 3 H), 2.30-1.32 (br m, 6 H). ATR-FTIR: 1649, 1540, 1012 cm<sup>-1</sup>.



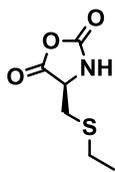
**Poly[(6-(methylthio)-L-norleucine)<sub>0.50</sub>-*stat*-(L-methionine sulfoxide)<sub>0.50</sub>], poly(Mtn<sup>O</sup><sub>0.50</sub>/Met<sup>O</sup><sub>0.50</sub>), 7<sup>O</sup>**

Prepared from **7** using the *Oxidation of polypeptide thioether groups* procedure (98% yield).

<sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O, 25 °C): 4.58-4.44 (br m, 1 H), 4.40-4.17 (br m, 1 H), 3.14-2.86 (br m, 4 H), 2.76 (s, 3 H), 2.72 (s, 3 H), 2.42-2.10 (br m, 2 H), 2.10-1.71 (br m, 4 H), 1.71-1.35 (br m, 2 H).

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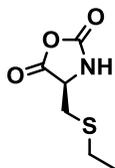
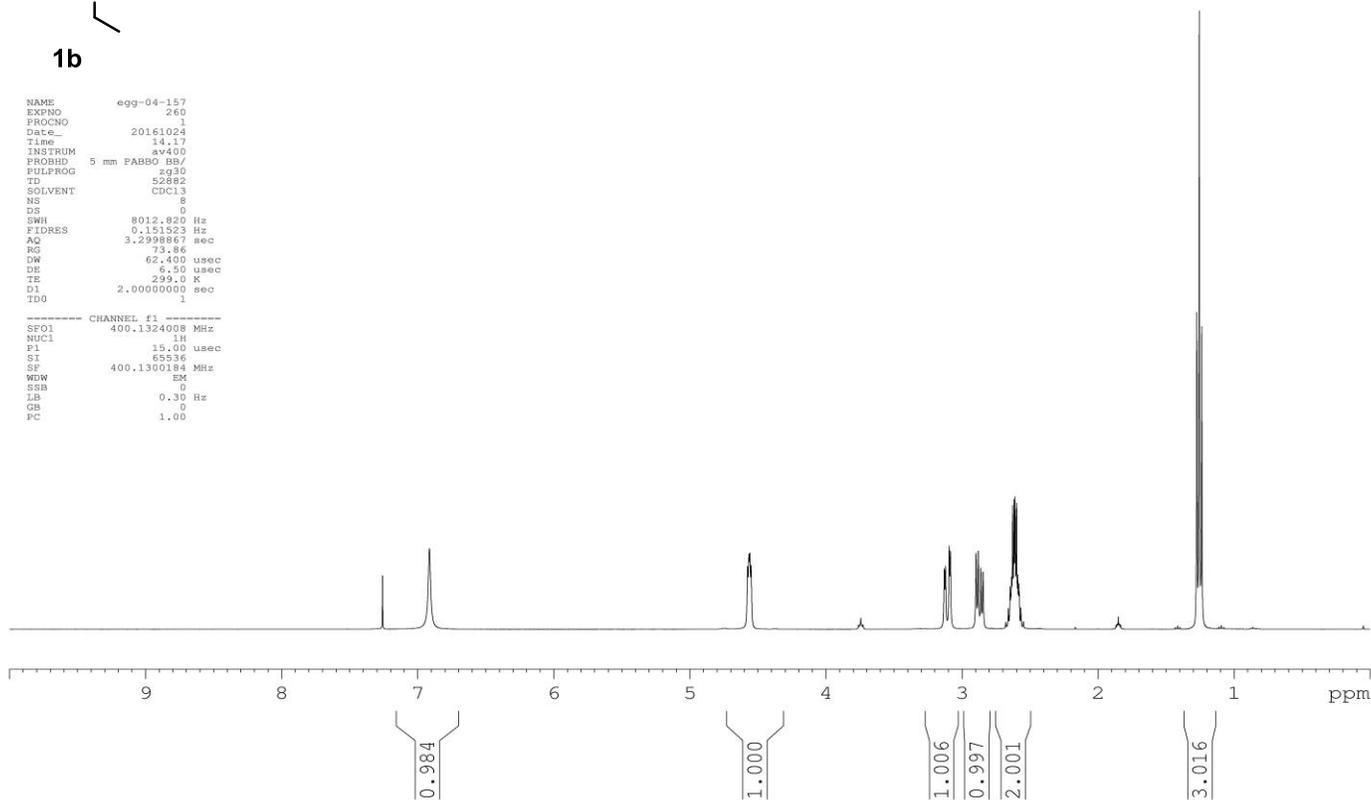
1b

```

NAME      egg-04-157
EXPNO     260
PROCNO    1
Date_     20161024
Time      14.17
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   CDCl3
NS         8
DS         0
SWH       8012.820 Hz
FIDRES    0.151323 Hz
AQ        3.2998867 sec
RG         73.86
DW        62.400 usec
DE         6.50 usec
TE        299.0 K
D1        2.0000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1      1H
P1        15.00 usec
SI        65536
SF        400.1300184 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



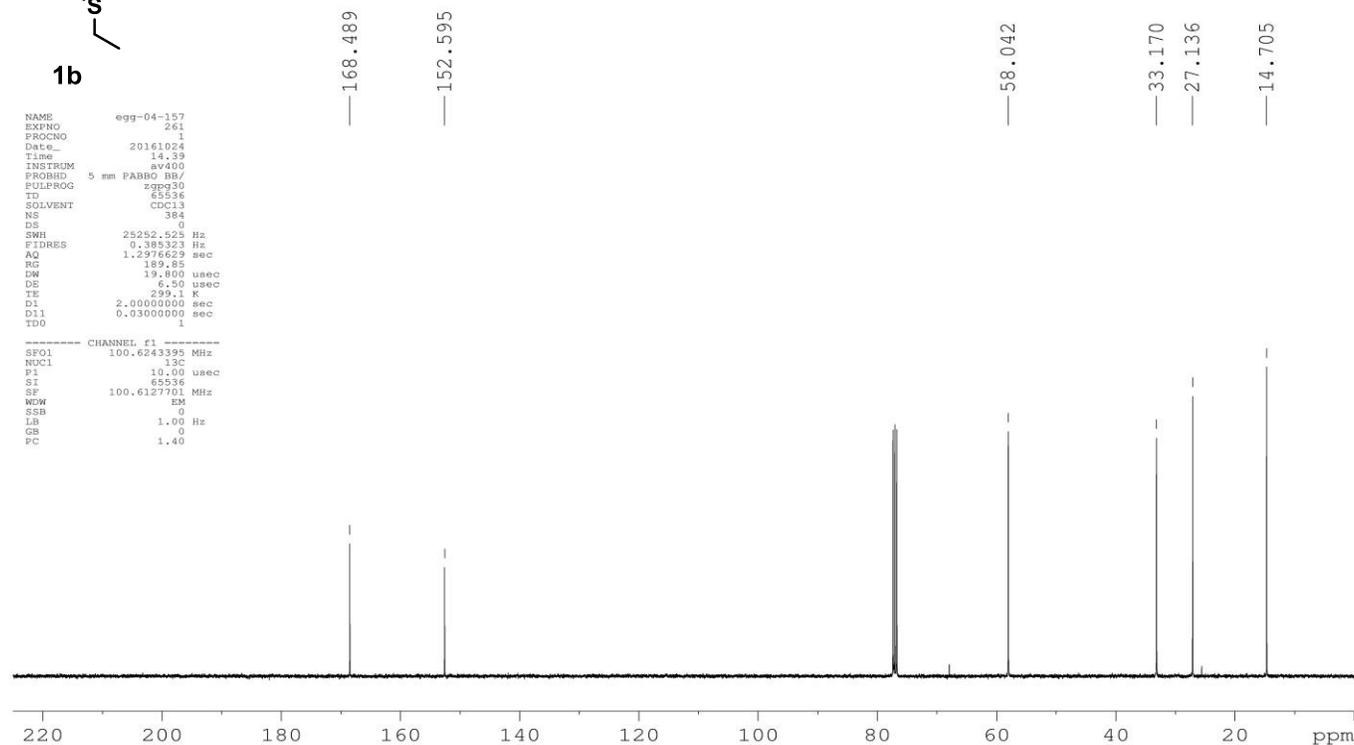
1b

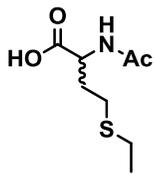
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NAME      egg-04-157
EXPNO     261
PROCNO    1
Date_     20161024
Time      14.39
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         384
DS         0
SWH       25252.525 Hz
FIDRES    0.388323 Hz
AQ        1.2976629 sec
RG         189.85
DW        19.800 usec
DE         6.50 usec
TE        299.1 K
D1        2.0000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SFO1      100.6243395 MHz
NUC1      13C
P1        10.00 usec
SI        65536
SF        100.6127701 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```





```

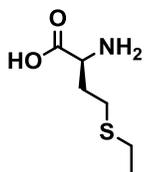
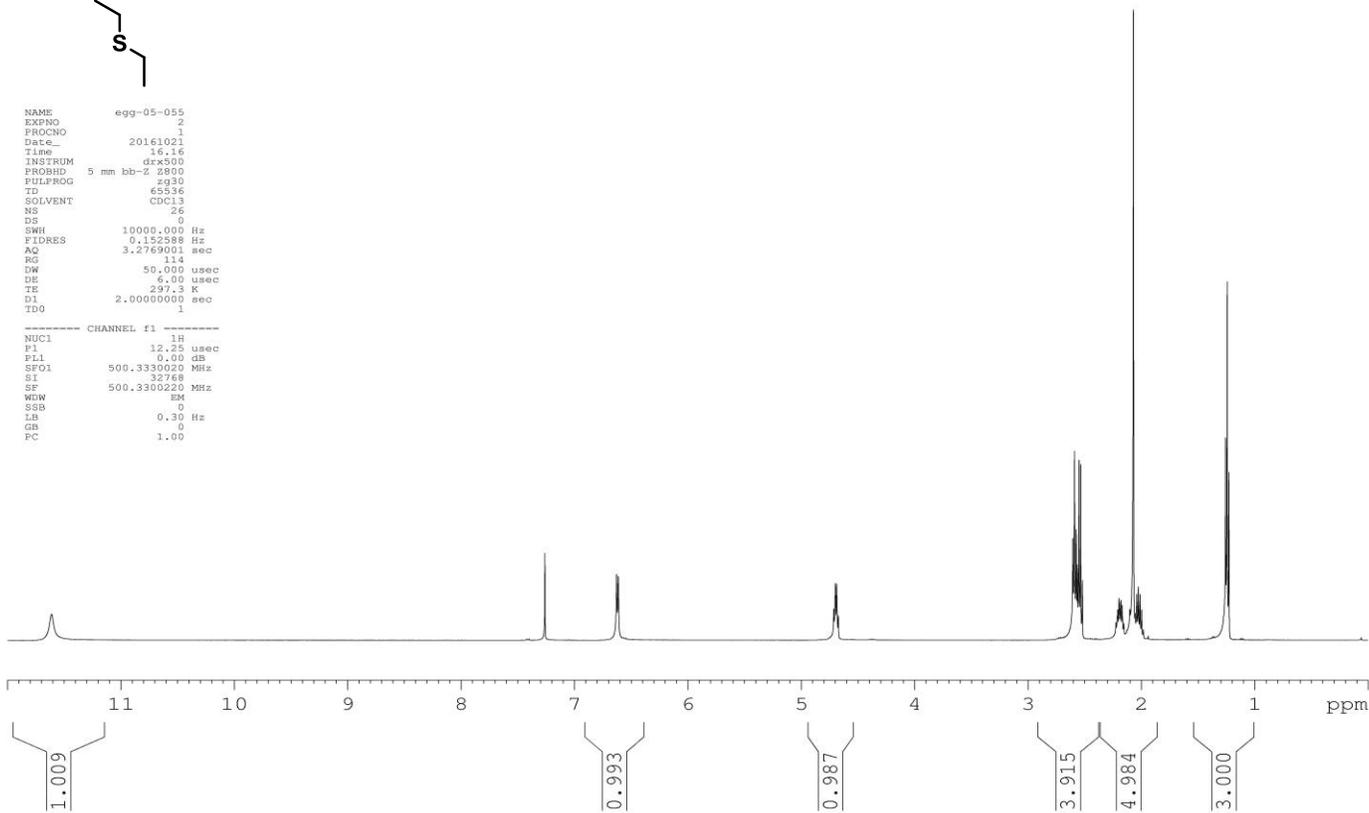
NAME      egg-05-055
EXPNO     2
PROCNO    1
Date_     20161021
Time      16.16
INSTRUM   grx300
PROBHD    5 mm bb-2 2800
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         26
DS         0
SWH        10000.000 Hz
FIDRES    0.152988 Hz
AQ         3.2769001 sec
RG         114
DW         50.000 usec
DE         6.00 usec
TE         297.3 K
DI         2.0000000 sec
TDO        1

```

```

----- CHANNEL f1 -----
NUC1      1H
P1        12.25 usec
PL1       0.00 dB
SFO1      500.330020 MHz
SI        32768
SF        500.3300220 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```



3a

```

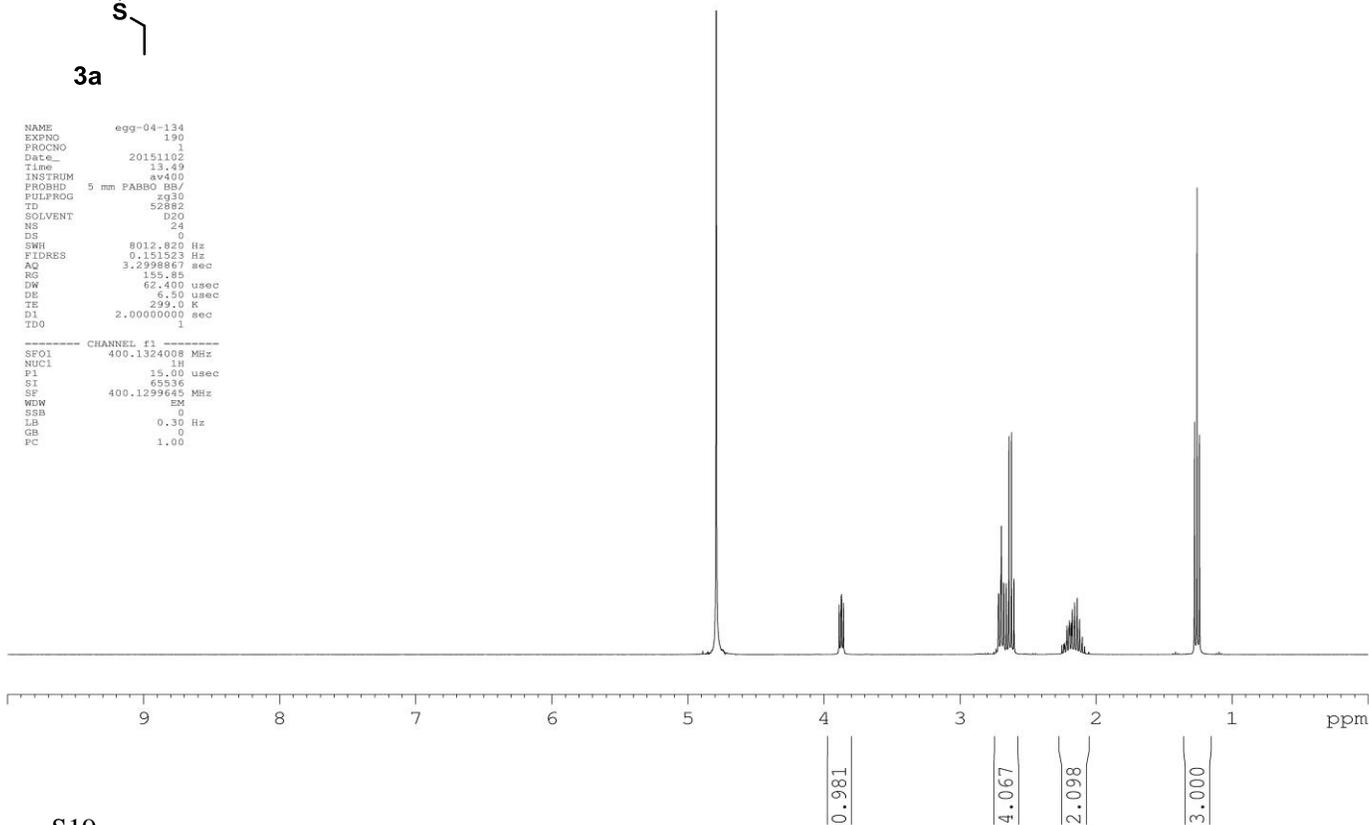
NAME      egg-04-134
EXPNO     190
PROCNO    1
Date_     20151102
Time      13.49
INSTRUM   av400
PROBHD    5 mm FAPBO BB/
PULPROG   zg30
TD         52882
SOLVENT   D2O
NS         24
DS         0
SWH        8012.820 Hz
FIDRES    0.151323 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.0 K
DI         2.0000000 sec
TDO        1

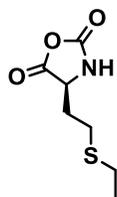
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1      1H
P1        15.00 usec
SI        65536
SF        400.1299645 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```





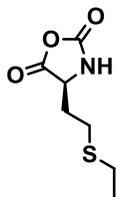
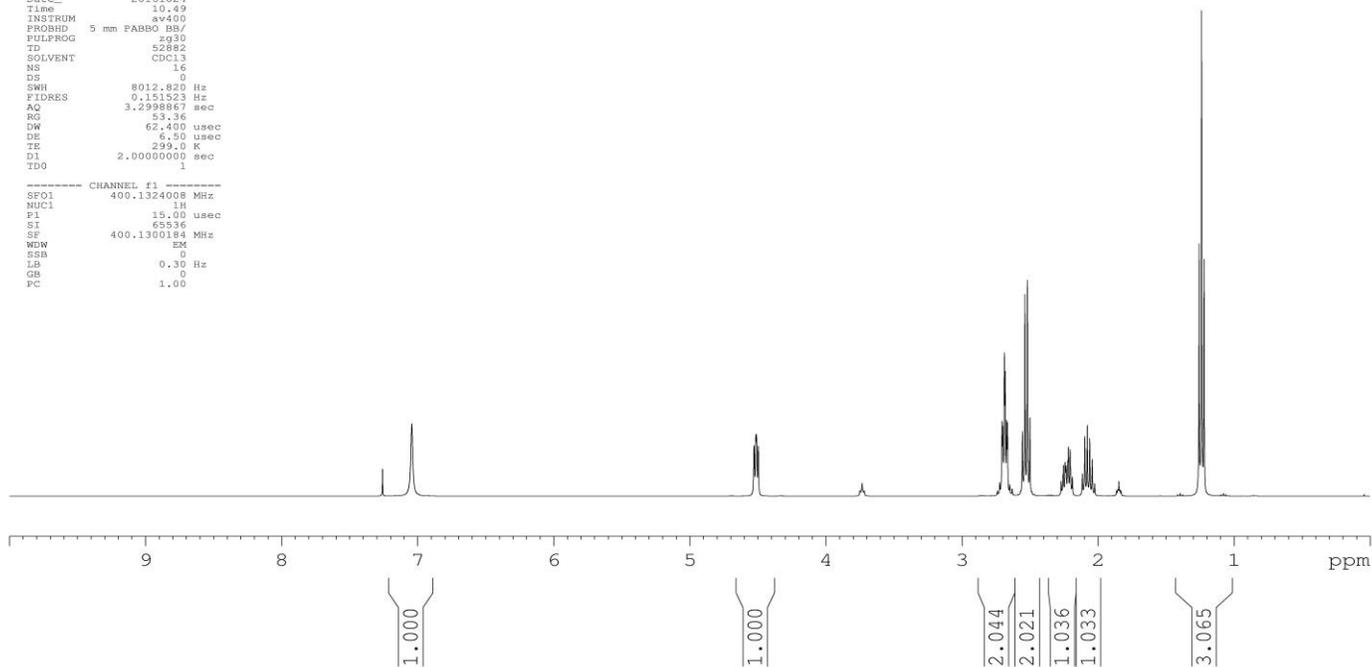
3b

```

NAME      egg-04-137
EXPNO    70
PROCNO   1
Date_    20161024
Time     10.49
INSTRUM  av400
PROBHD   5 mm FABBO BB/
PULPROG  zg30
TD        52882
SOLVENT  CDCl3
NS        16
DS        0
SWH       8012.820 Hz
FIDRES   0.151323 Hz
AQ        3.2998867 sec
RG        53.36
DW        62.400 usec
DE        6.50 usec
TE        299.0 K
D1        2.0000000 sec
TDO       1
  
```

```

----- CHANNEL f1 -----
SFO1     400.1324008 MHz
NUC1      1H
P1        15.00 usec
SI        65536
SF        400.1300184 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



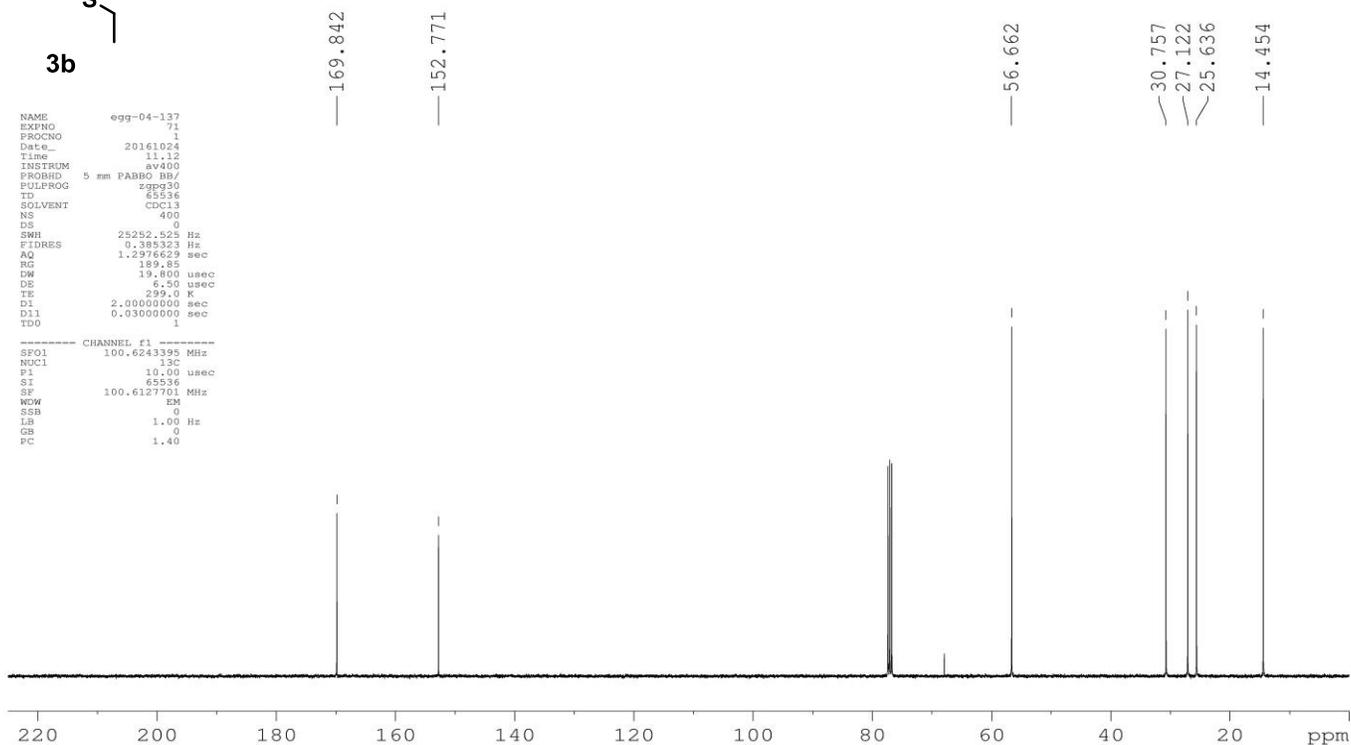
3b

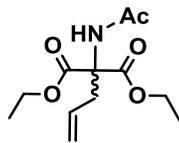
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NAME      egg-04-137
EXPNO    71
PROCNO   1
Date_    20161024
Time     11.12
INSTRUM  av400
PROBHD   5 mm FABBO BB/
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        400
DS        0
SWH       25252.525 Hz
FIDRES   0.388323 Hz
AQ        1.2976629 sec
RG        189.85
DW        19.800 usec
DE        6.50 usec
TE        299.0 K
D1        2.0000000 sec
D11       0.03000000 sec
TDO       1
  
```

```

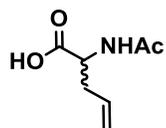
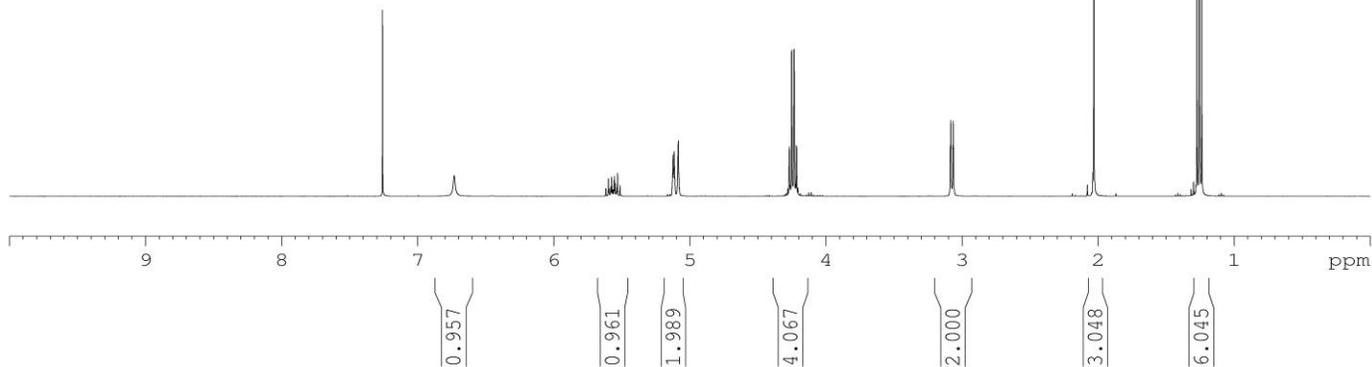
----- CHANNEL f1 -----
SFO1     100.6243395 MHz
NUC1      13C
P1        10.00 usec
SI        65536
SF        100.6127701 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```





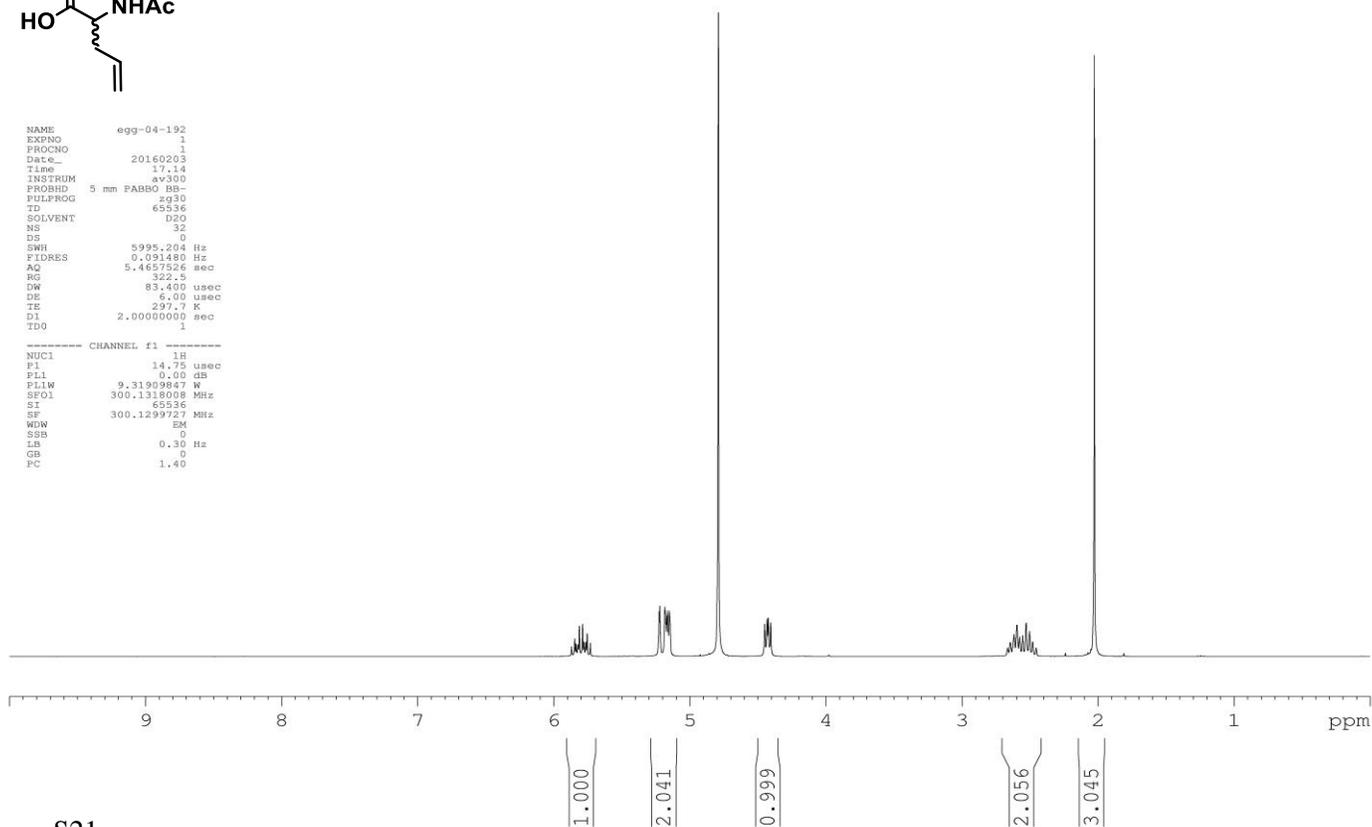
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 EXPNO 1  
 PROCNO 270  
 Date\_ 20160202  
 Time 13.39  
 INSTRUM av400  
 PROBHD 5 mm FABBO BB/  
 PULPROG zg30  
 TD 52882  
 SOLVENT CDCl3  
 NS 24  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.151323 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 DI 2.0000000 sec  
 TDO 1

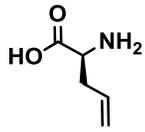
----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65536  
 SF 400.1300184 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



NAME egg-04-192  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20160203  
 Time 17.14  
 INSTRUM av300  
 PROBHD 5 mm FABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT D2O  
 NS 32  
 DS 0  
 SWH 5995.204 Hz  
 FIDRES 0.091480 Hz  
 AQ 5.4657526 sec  
 RG 322.5  
 DW 83.400 usec  
 DE 6.00 usec  
 TE 297.7 K  
 DI 2.0000000 sec  
 TDO 1

----- CHANNEL f1 -----  
 NUC1 1H  
 P1 14.75 usec  
 PLL 0.00 dB  
 PL1W 9.31999847 W  
 SFO1 300.1318008 MHz  
 SI 65536  
 SF 300.1299727 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.40





```

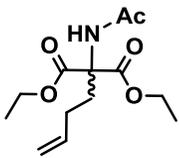
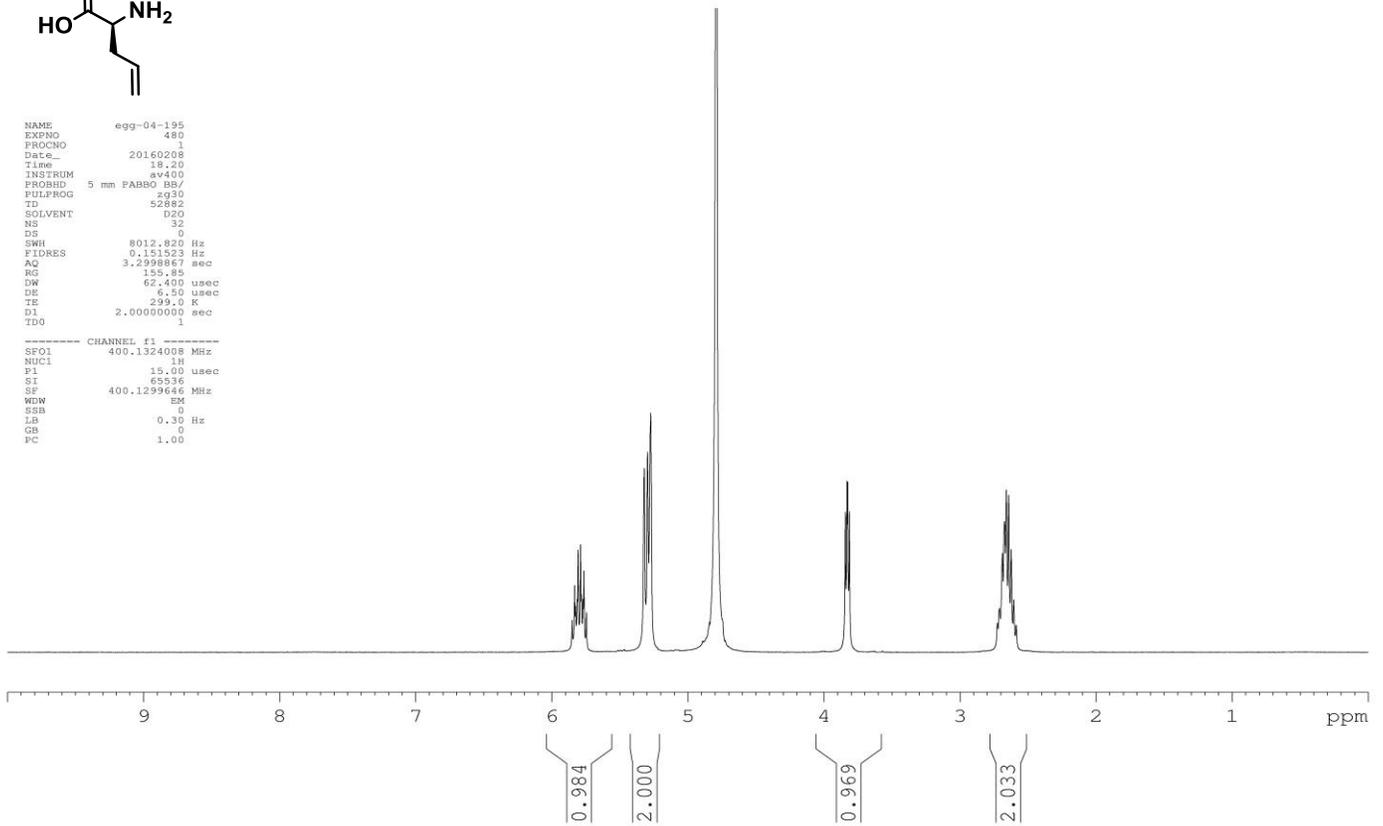
NAME      egg-04-195
EXPNO     480
PROCNO    1
Date_     20160208
Time      18.20
INSTRUM   av400
PROBHD    5 mm FABS0 BB/
PULPROG   zg30
TD         52882
SOLVENT   D2O
NS         32
DS         0
SWH        8012.820 Hz
FIDRES     0.151323 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TDO        1

```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1299646 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

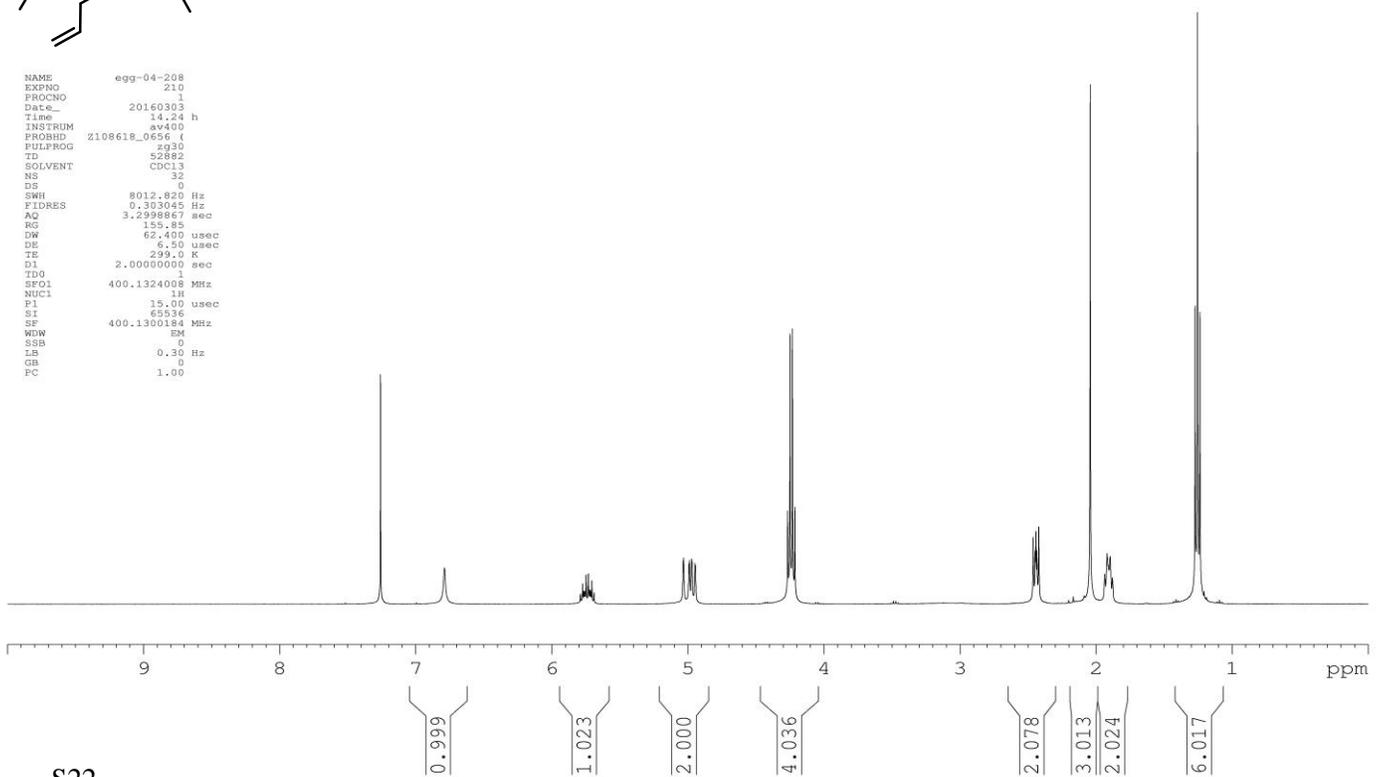
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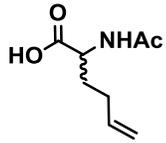


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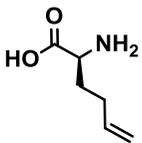
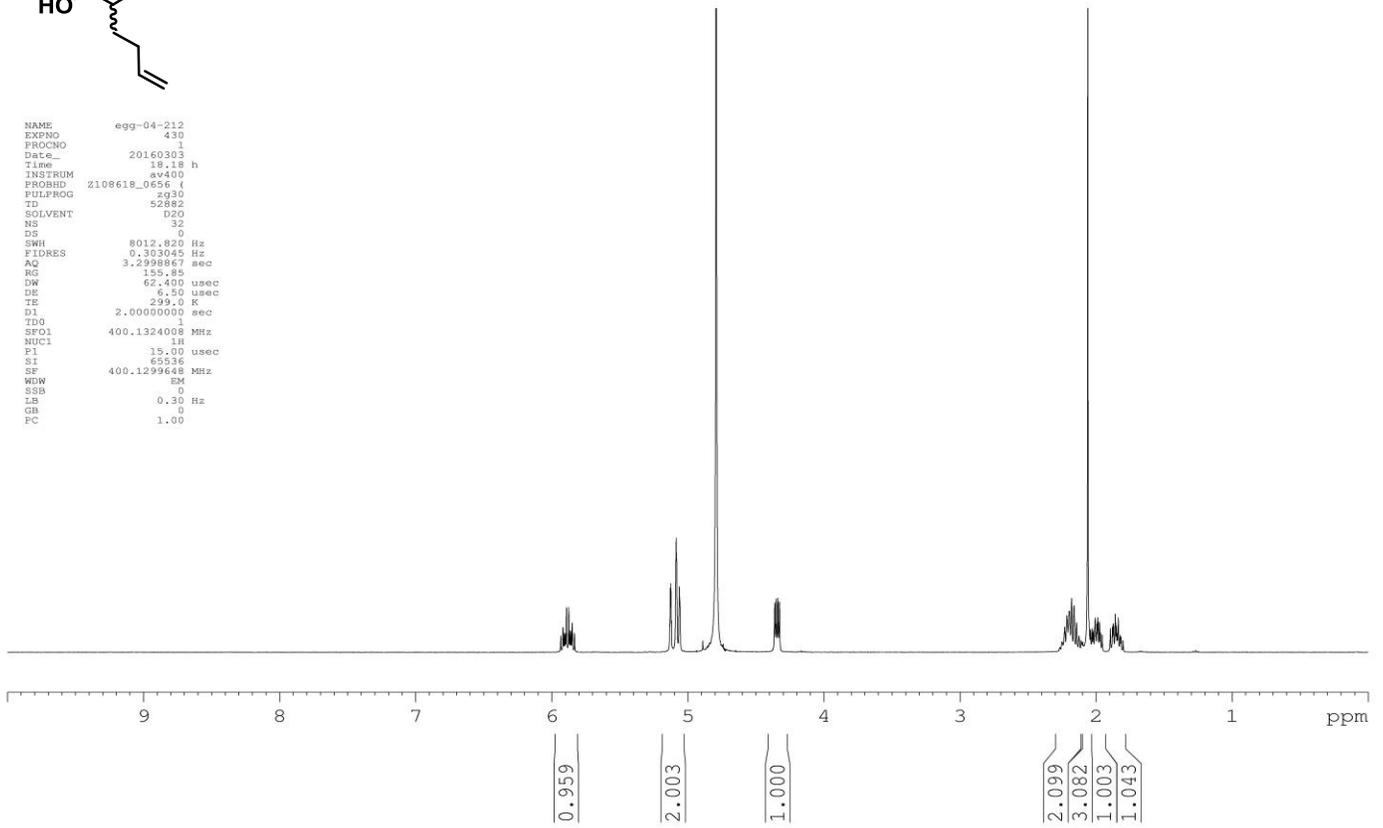
NAME      egg-04-208
EXPNO     210
PROCNO    1
Date_     20160303
Time      14.24 h
INSTRUM   av400
PROBHD    2108618_0656 (
PULPROG   zg30
TD         52882
SOLVENT   CDCl3
NS         32
DS         0
SWH        8012.820 Hz
FIDRES     0.303045 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TDO        1
SFO1      400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1300184 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```

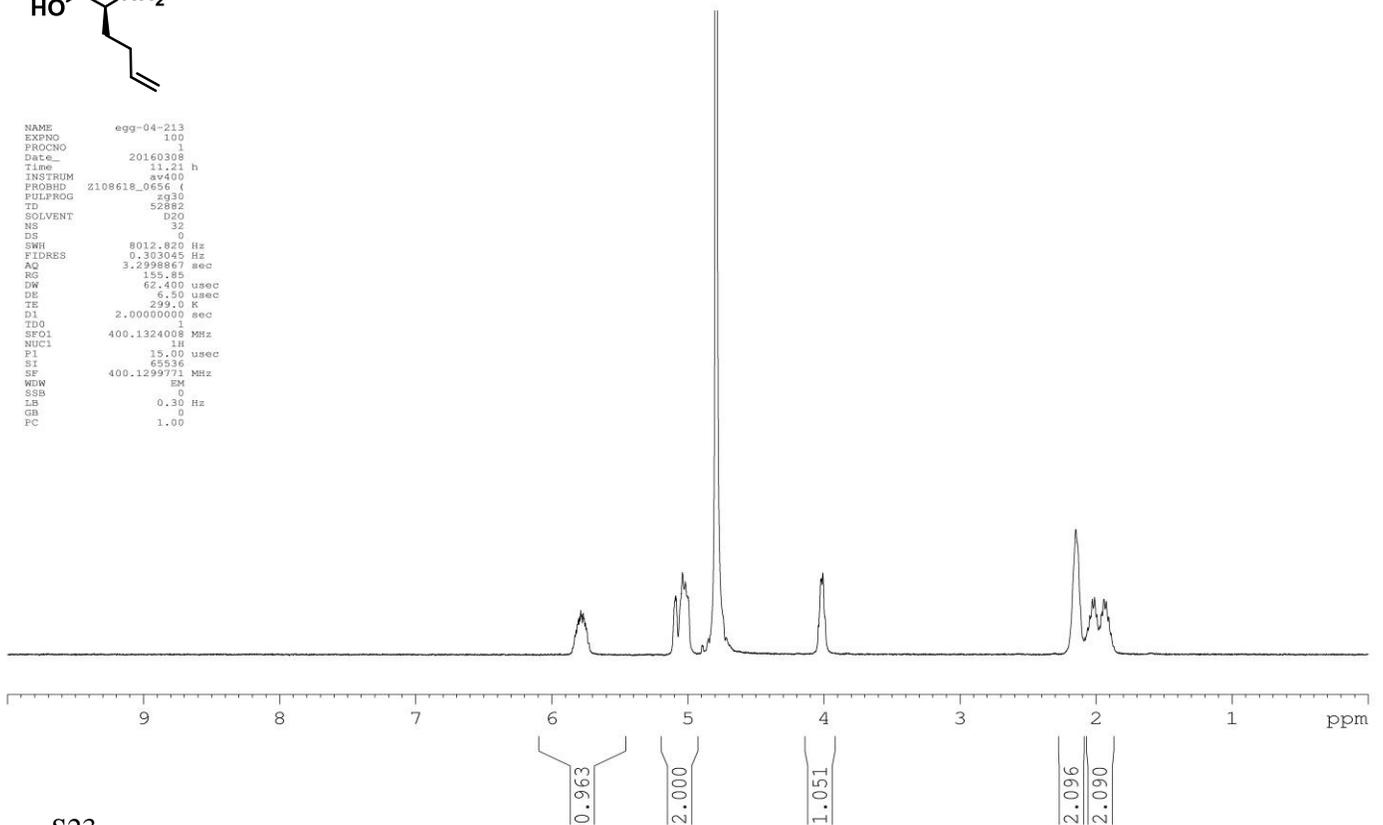


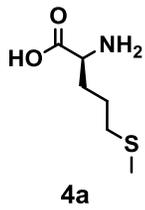


NAME egg-04-212  
 EXPNO 430  
 PROCNO 1  
 Date\_ 20160303  
 Time 18.18 h  
 INSTRUM av400  
 PROBHD 2108618\_0656 (   
 PULPROG zg30  
 TD 52882  
 SOLVENT D2O  
 NS 32  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.303045 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 DI 2.0000000 sec  
 TDO 1  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65536  
 SF 400.1299648 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



NAME egg-04-213  
 EXPNO 100  
 PROCNO 1  
 Date\_ 20160308  
 Time 11.21 h  
 INSTRUM av400  
 PROBHD 2108618\_0656 (   
 PULPROG zg30  
 TD 52882  
 SOLVENT D2O  
 NS 32  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.303045 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 DI 2.0000000 sec  
 TDO 1  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65536  
 SF 400.1299771 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00





```

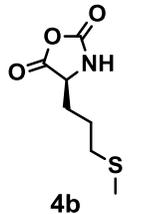
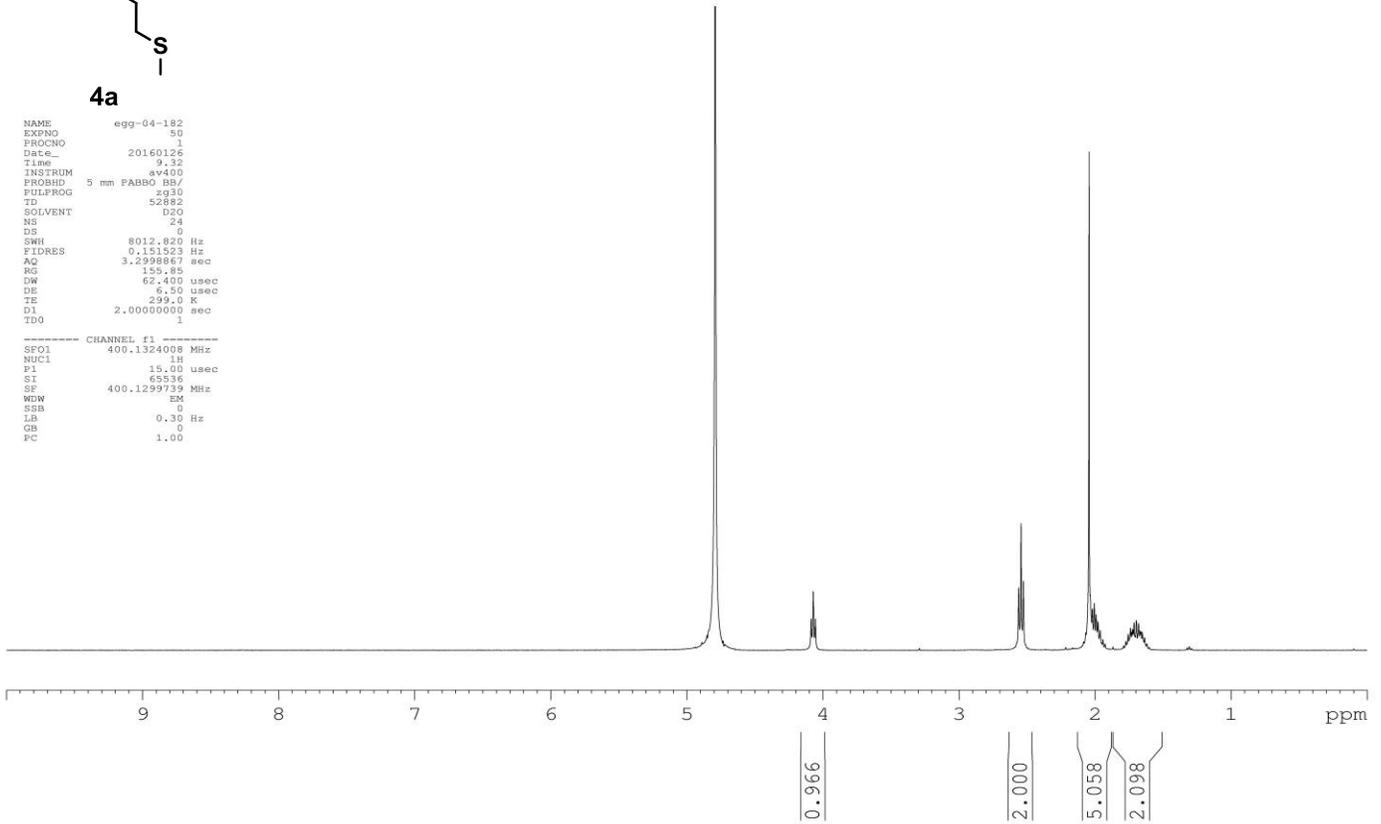
NAME      egg-04-182
EXPNO     50
PROCNO    1
Date_     20160126
Time      9.32
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD        52882
SOLVENT   D2O
NS        24
DS        0
SWH       8012.820 Hz
FIDRES    0.151323 Hz
AQ        3.2998867 sec
RG        155.85
DW        62.400 usec
DE        6.50 usec
TE        299.0 K
D1        2.0000000 sec
TDO       1

```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1      1H
P1        15.00 usec
SI        65536
SF        400.1299739 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```



```

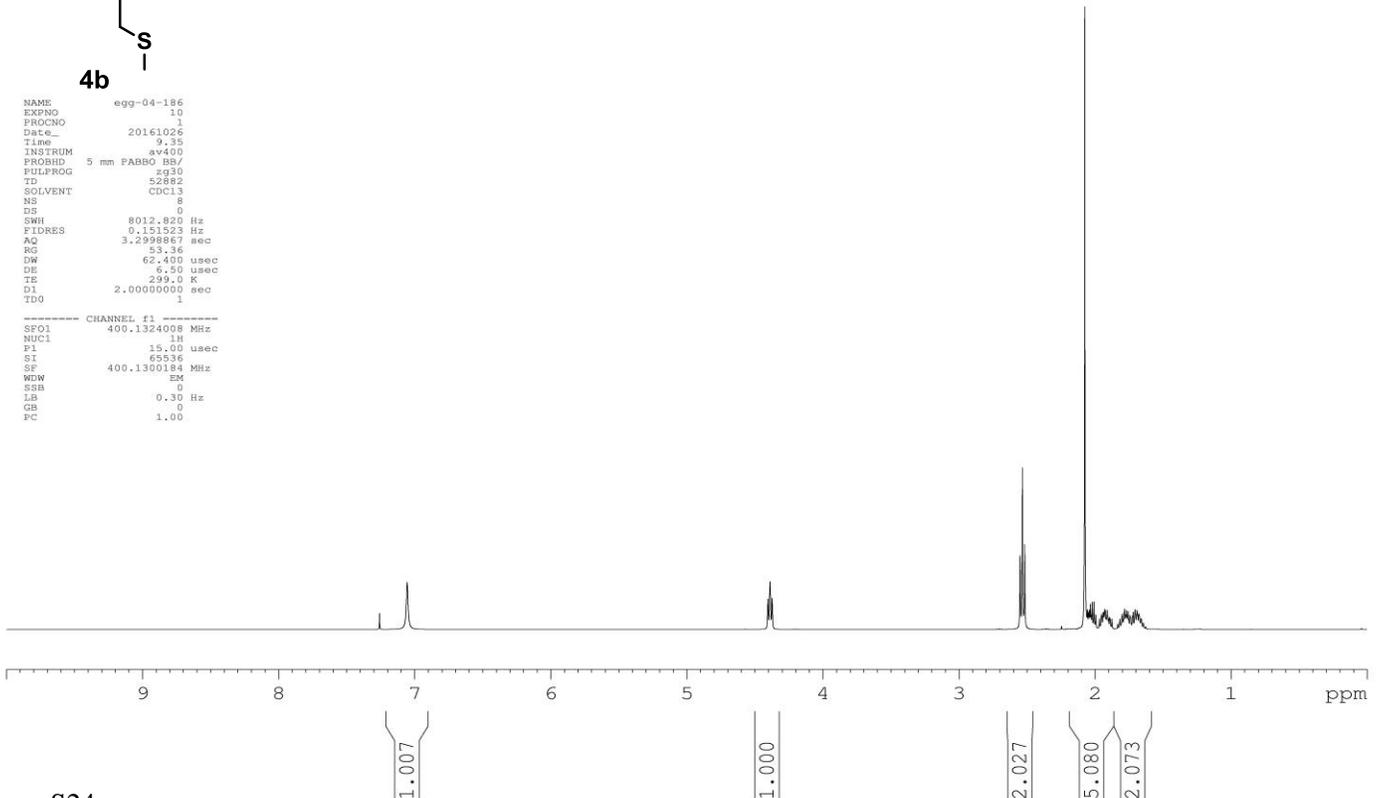
NAME      egg-04-186
EXPNO     10
PROCNO    1
Date_     20161026
Time      9.35
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD        52882
SOLVENT   CDCl3
NS        8
DS        0
SWH       8012.820 Hz
FIDRES    0.151323 Hz
AQ        3.2998867 sec
RG        53.36
DW        62.400 usec
DE        6.50 usec
TE        299.0 K
D1        2.0000000 sec
TDO       1

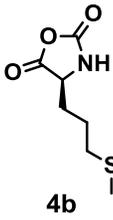
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1      1H
P1        15.00 usec
SI        65536
SF        400.1300184 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```





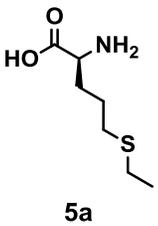
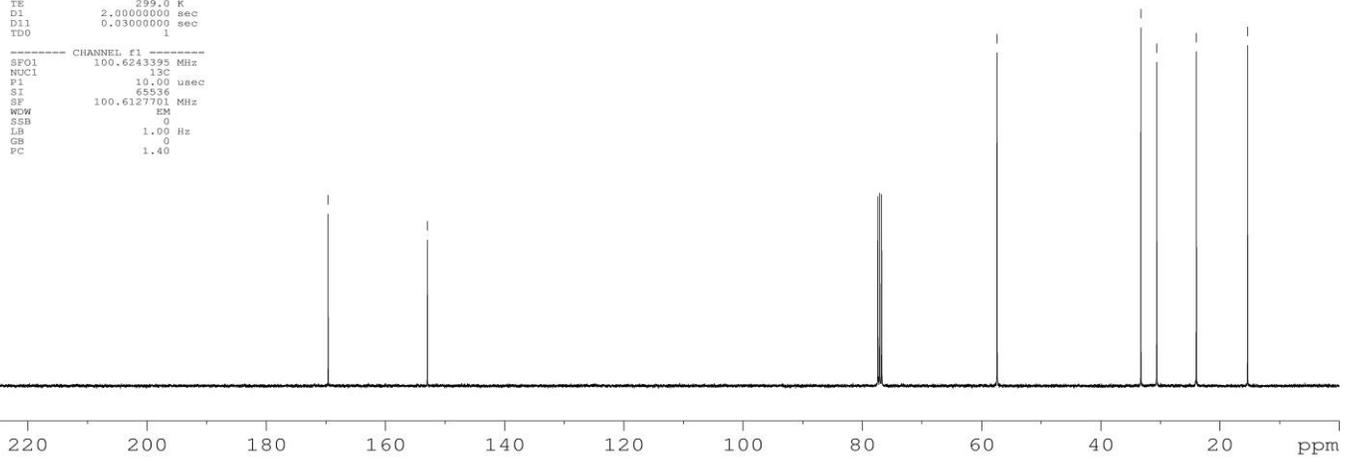
```

NAME      egg-04-186
EXPNO     11
PROCNO    1
Date_     20161026
Time      9.58
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         384
DS         0
SWH        25252.525 Hz
FIDRES     0.388323 Hz
AQ          1.2976629 sec
RG          189.85
DM          19.800 usec
DE          6.50 usec
TE          299.0 K
D1          2.0000000 sec
D11         0.03000000 sec
TD0         1
  
```

```

----- CHANNEL f1 -----
SFO1      100.6243395 MHz
NUC1       13C
P1         10.00 usec
SI         65536
SF         100.6127701 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

— 169.615 — 152.972 — 57.410 — 33.278 — 30.632 — 23.990 — 15.398

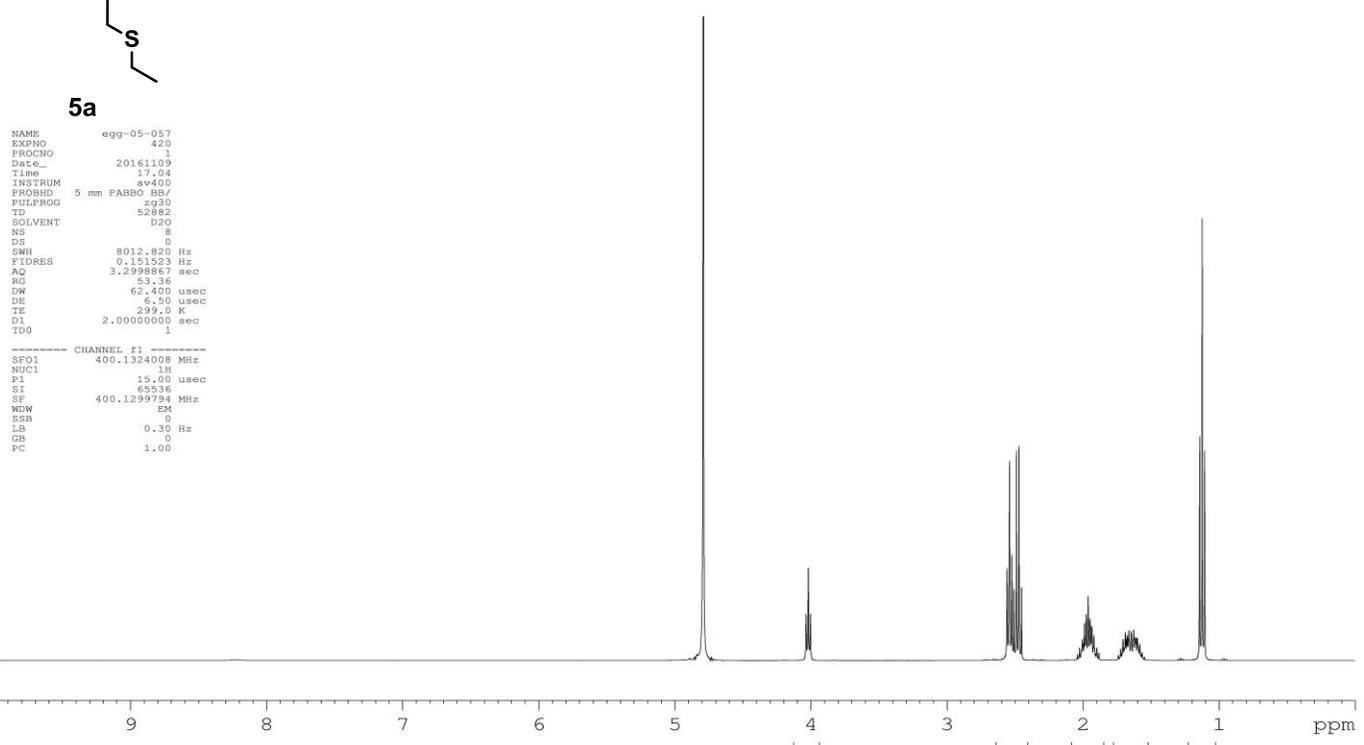


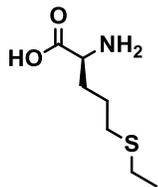
```

NAME      egg-05-057
EXPNO     420
PROCNO    1
Date_     20161109
Time      17.04
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   D2O
NS         8
DS         0
SWH        8012.820 Hz
FIDRES     0.151323 Hz
AQ          3.2998867 sec
RG          53.36
DM          62.400 usec
DE          6.50 usec
TE          299.0 K
D1          2.0000000 sec
TD0         1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1299794 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
  
```





5a

```

NAME      egg-05-057
EXPNO     421
PROCNO    1
Date_     20161109
Time      17.28
INSTRUM   av400
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   D2O
NS         400
DS         0
SWH        25252.525 Hz
FIDRES     0.385323 Hz
AQ         1.2975629 sec
RG         189.85
DM         19.800 usec
DE         6.50 usec
TE         299.2 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
SFO1      100.6243395 MHz
NUC1       13C
P1         10.00 usec
SI         65536
SF         100.6127701 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

171.690

52.399

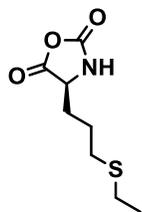
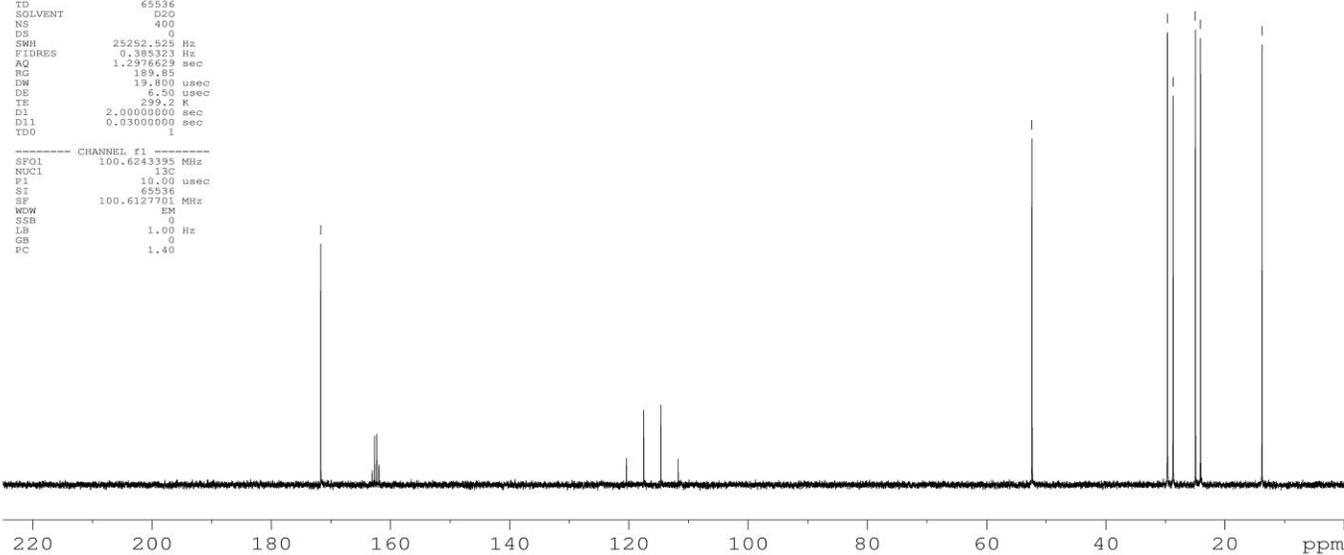
29.681

28.723

24.965

24.126

13.806



5b

```

NAME      egg-05-058
EXPNO     90
PROCNO    1
Date_     20161109
Time      11.12
INSTRUM   av400
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   CDCl3
NS         8
DS         0
SWH        8012.820 Hz
FIDRES     0.151323 Hz
AQ         3.2998867 sec
RG         83.63
DM         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1300255 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

0.997

0.990

1.948

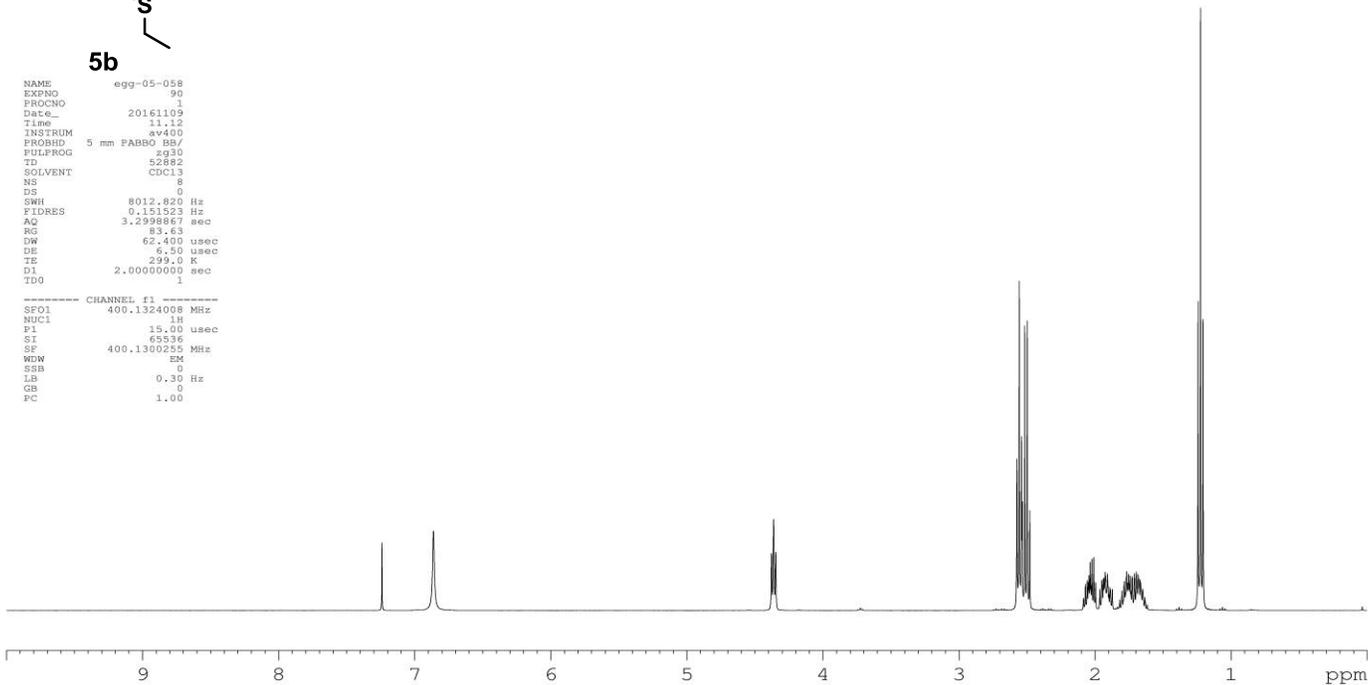
2.018

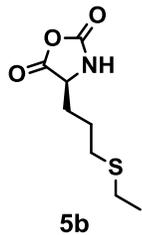
1.007

1.034

2.055

3.000



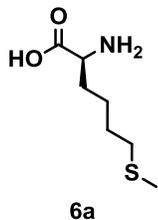
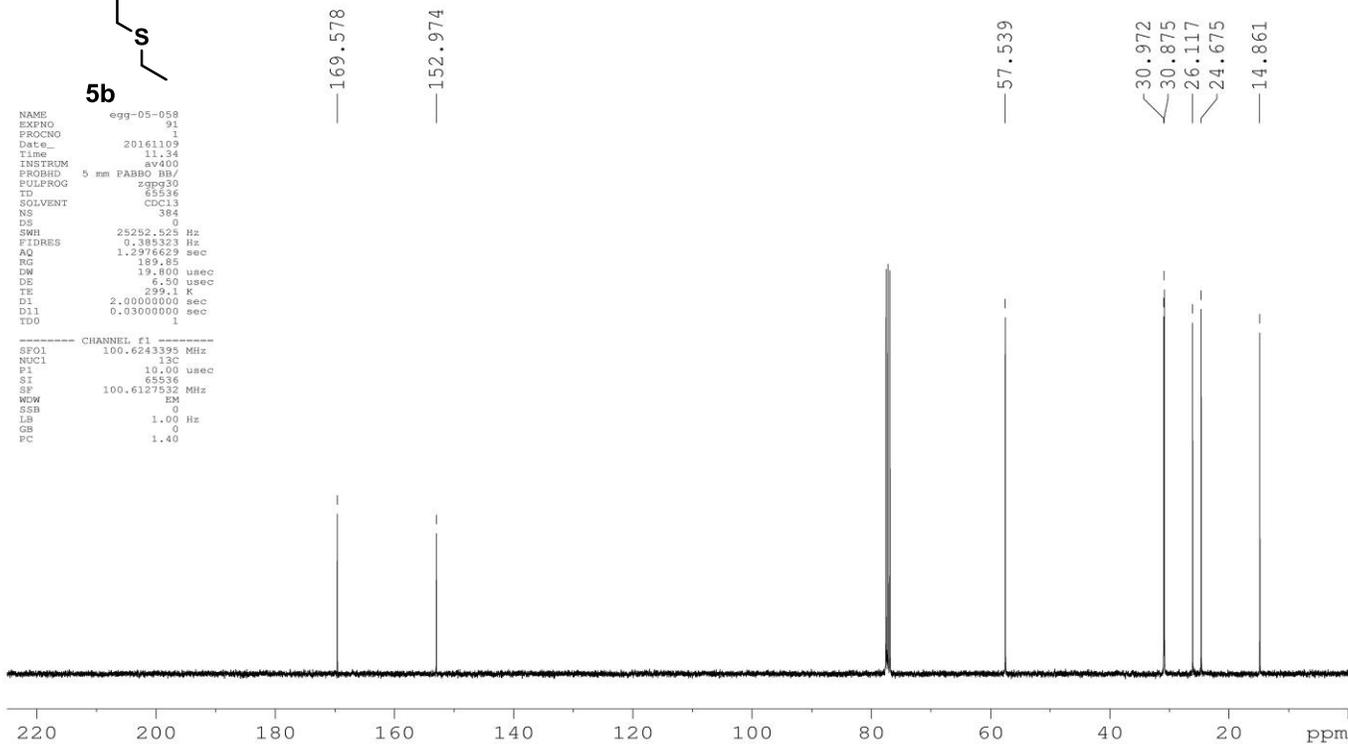


```

NAME      egg-05-058
EXPNO    91
PROCNO    1
Date_    20161109
Time     11.34
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        384
DS        0
SWH       25252.525 Hz
FIDRES    0.388323 Hz
AQ        1.2976629 sec
RG        189.85
DM        19.800 usec
DE        6.50 usec
TE        299.1 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SFO1     100.624395 MHz
NUC1     13C
P1       10.00 usec
SI       65536
SF       100.6127532 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

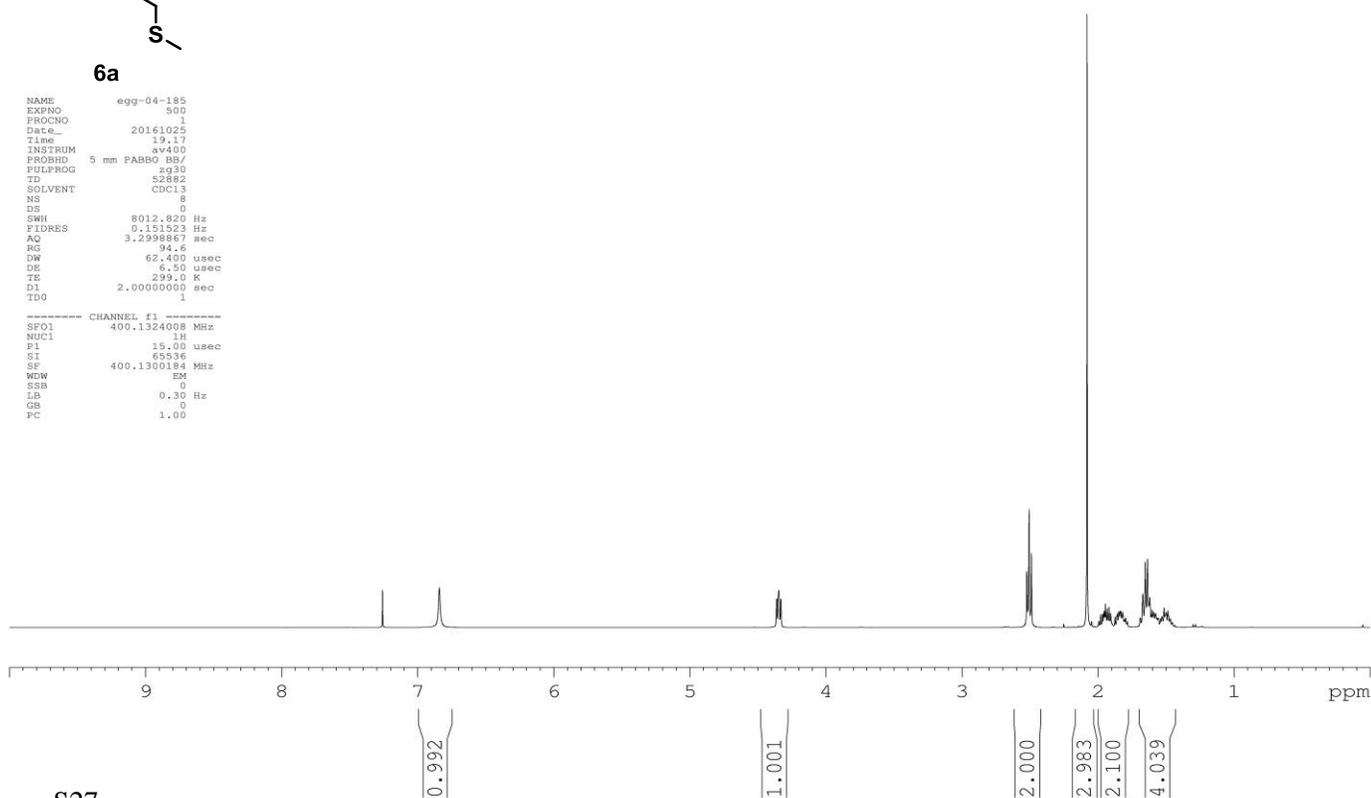


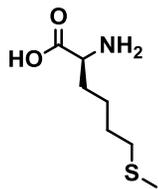
```

NAME      egg-04-185
EXPNO    500
PROCNO    1
Date_    20161025
Time     19.17
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD        52882
SOLVENT   CDCl3
NS        8
DS        0
SWH       8012.820 Hz
FIDRES    0.151323 Hz
AQ        3.2998867 sec
RG        94.6
DM        62.400 usec
DE        6.50 usec
TE        299.0 K
D1        2.0000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SFO1     400.1324008 MHz
NUC1     1H
P1       15.00 usec
SI       65536
SF       400.1300184 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```





6a

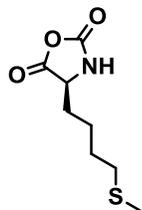
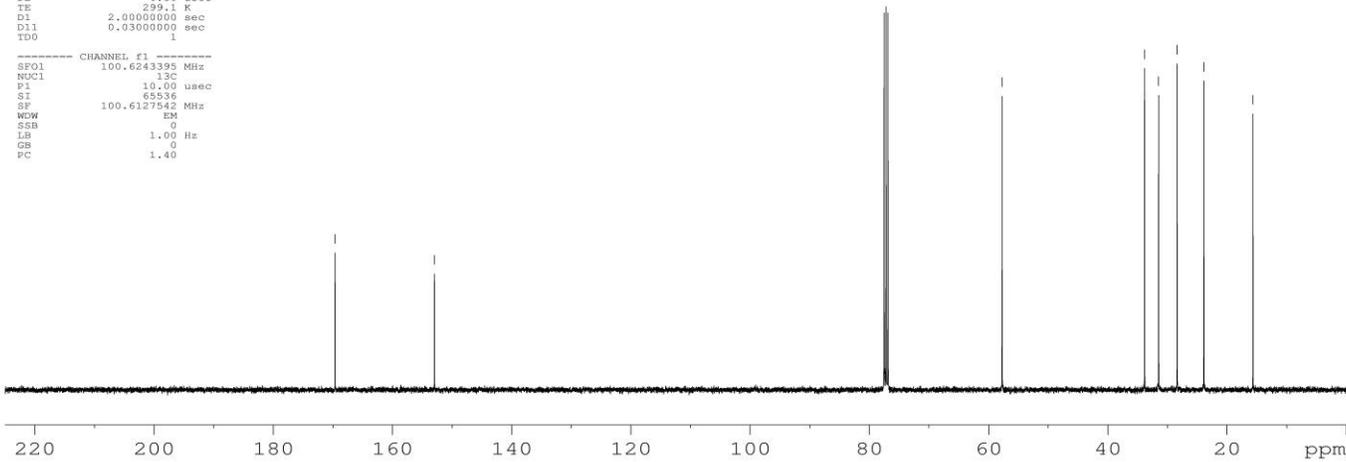
```

NAME      egg-04-185
EXPNO     501
PROCNO    1
Date_     20161025
Time      19.39
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         384
DS         0
SWH        25252.525 Hz
FIDRES    0.388323 Hz
AQ         1.2976629 sec
RG         189.85
DM         19.800 usec
DE         6.50 usec
TE         299.1 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
SFO1      100.6243395 MHz
NUC1       13C
P1         10.00 usec
SI         65536
SF         100.6127542 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

— 169.665 — 153.006 — 57.754 — 33.835 — 31.495 — 28.371 — 23.908 — 15.665



6b

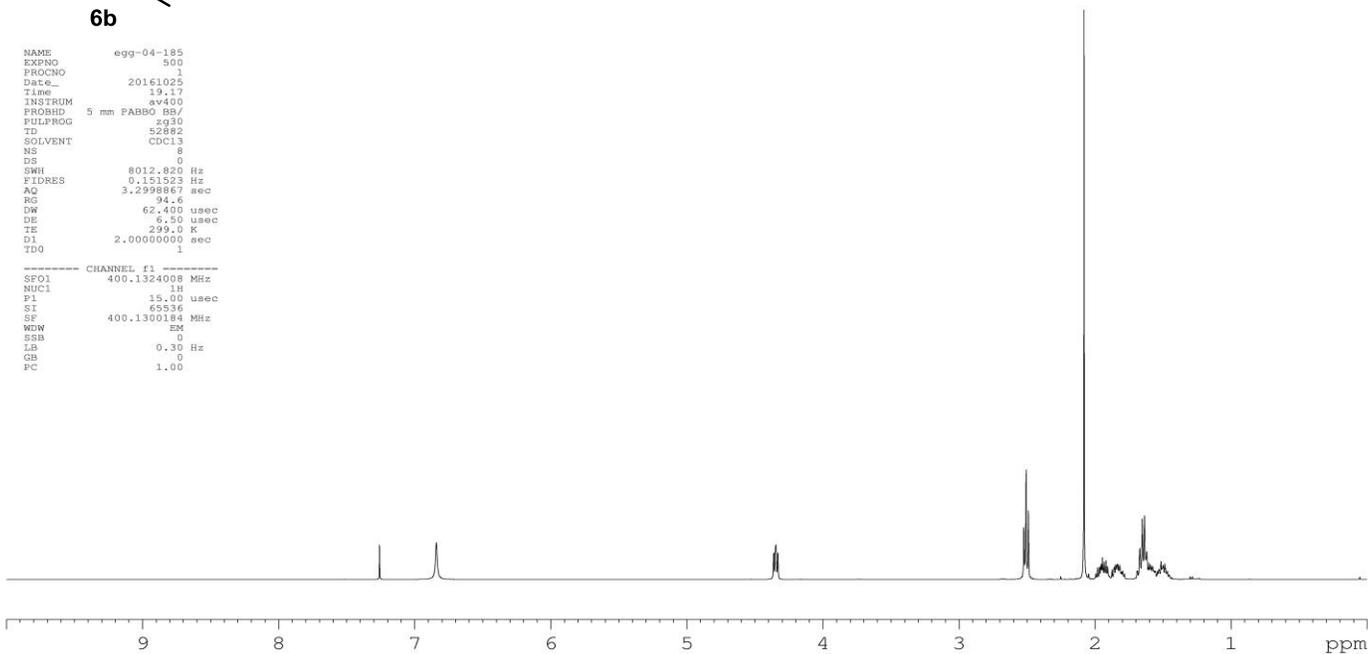
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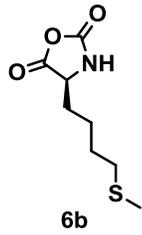
NAME      egg-04-185
EXPNO     500
PROCNO    1
Date_     20161025
Time      19.17
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   CDCl3
NS         8
DS         0
SWH        8012.820 Hz
FIDRES    0.151323 Hz
AQ         3.2998867 sec
RG         94.6
DM         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1300184 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```

0.992 1.001 2.000 2.983 2.100 4.039





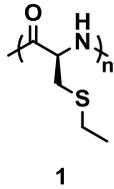
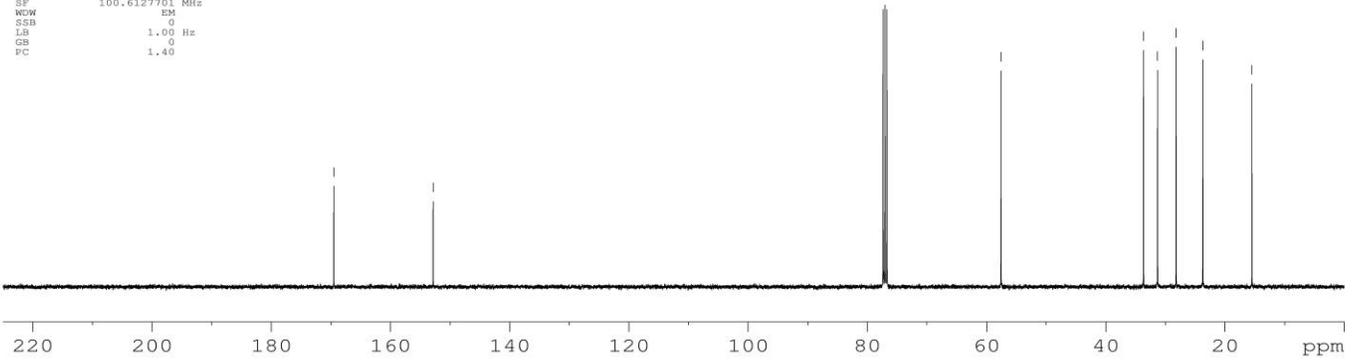
```

NAME      egg-04-185
EXPNO    501
PROCNO    1
Date_    20161025
Time     19.39
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        384
DS        0
SWH       25252.525 Hz
FIDRES    0.388323 Hz
AQ        1.2976629 sec
RG        189.85
DM        19.800 usec
DE        6.50 usec
TE        299.1 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SFO1     100.624395 MHz
NUC1     13C
P1       10.00 usec
SI       65536
SF       100.6127701 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

— 169.506 — 152.848 — 57.596 — 33.677 — 31.337 — 28.213 — 23.750 — 15.507

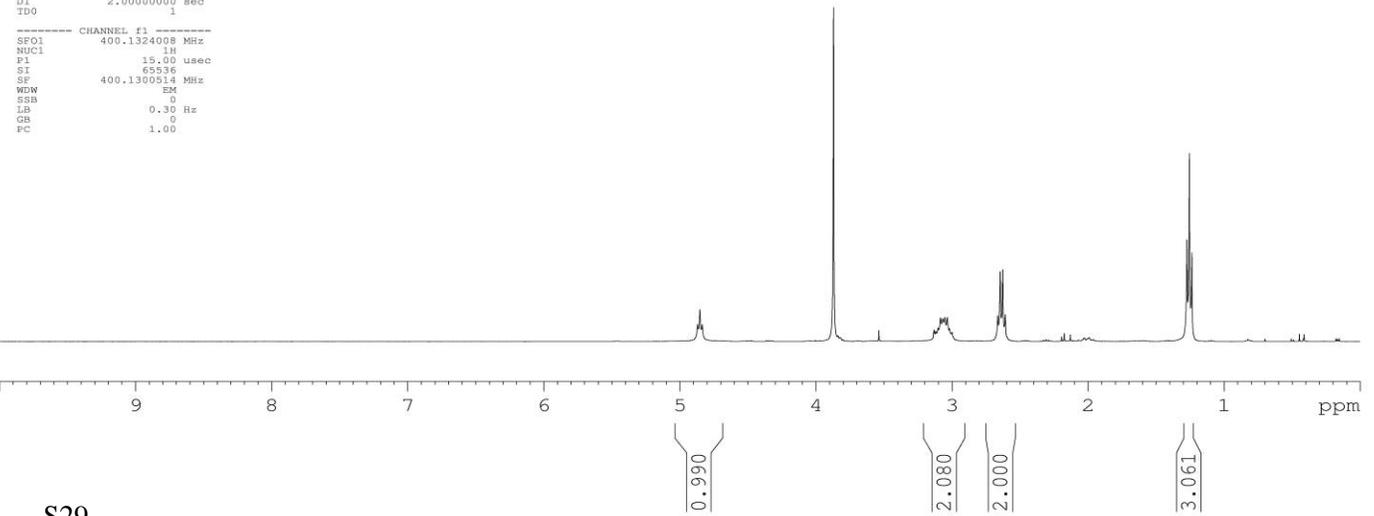


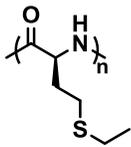
```

NAME      egg-04-163
EXPNO    210
PROCNO    1
Date_    20151207
Time     13.51
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD        52882
SOLVENT   TFA-d
NS        32
DS        0
SWH       8012.820 Hz
FIDRES    0.151323 Hz
AQ        3.2998867 sec
RG        155.85
DM        62.400 usec
DE        6.50 usec
TE        299.0 K
D1        2.0000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
SFO1     400.1324008 MHz
NUC1     1H
P1       15.00 usec
SI       65536
SF       400.1300514 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```





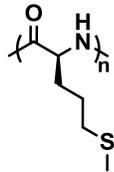
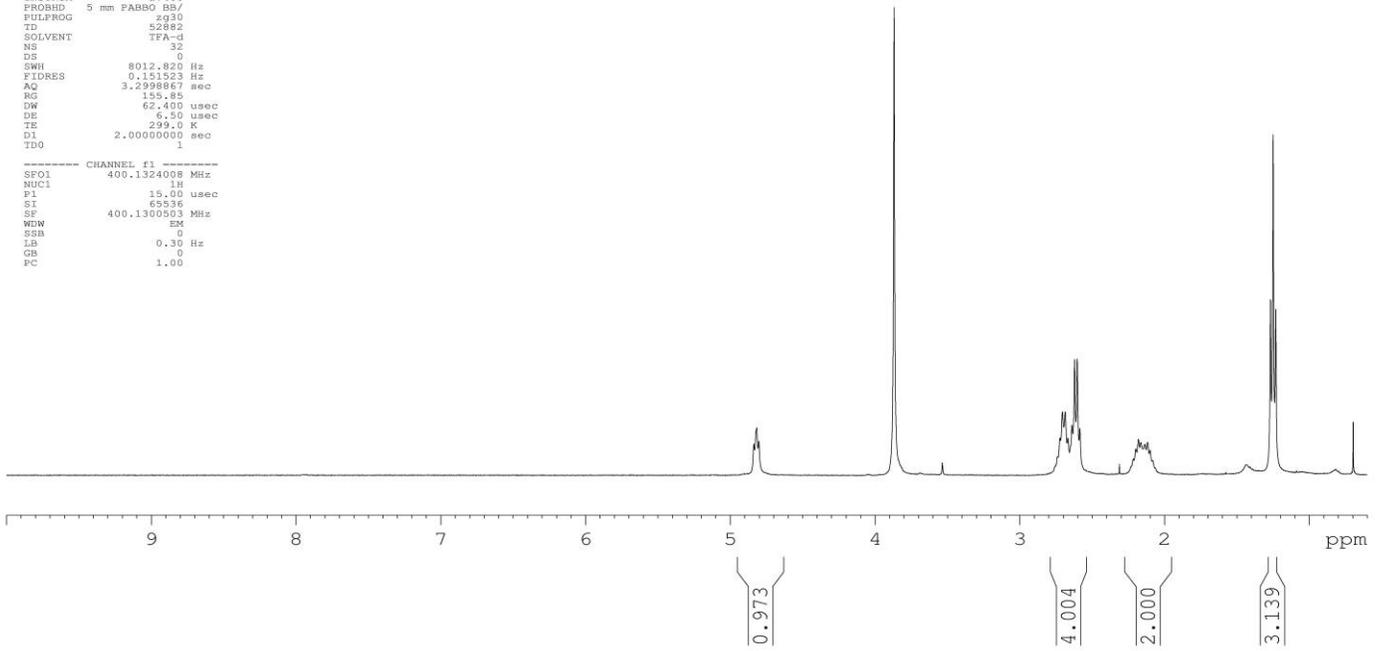
3

```

NAME      egg-04-145
EXPNO     250
PROCNO    1
Date_     20151111
Time      16.04
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   TFA-d
NS         32
DS         0
SWH        8012.820 Hz
FIDRES    0.151323 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1300503 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



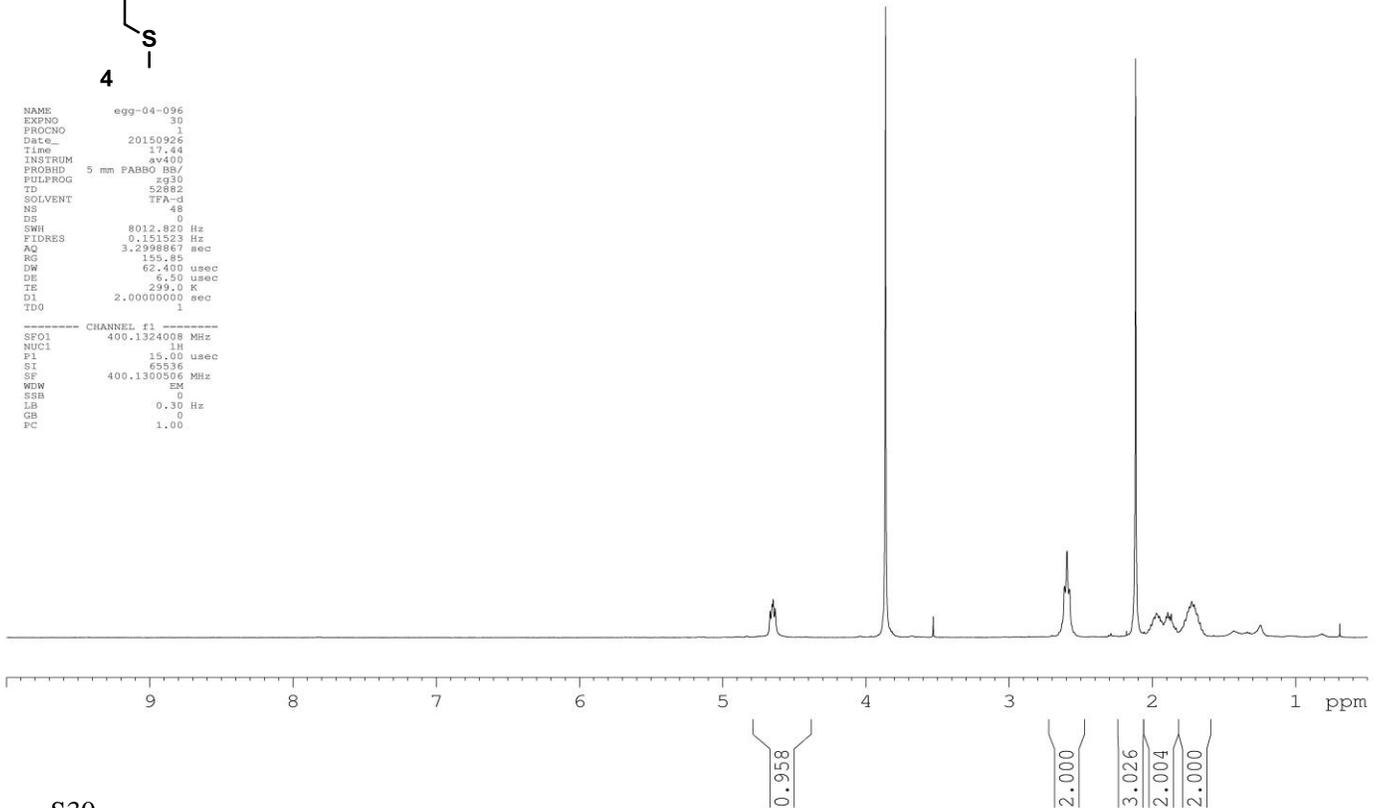
4

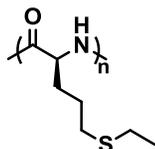
```

NAME      egg-04-096
EXPNO     30
PROCNO    1
Date_     20150926
Time      17.44
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   TFA-d
NS         48
DS         0
SWH        8012.820 Hz
FIDRES    0.151323 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1300506 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```





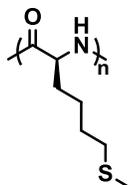
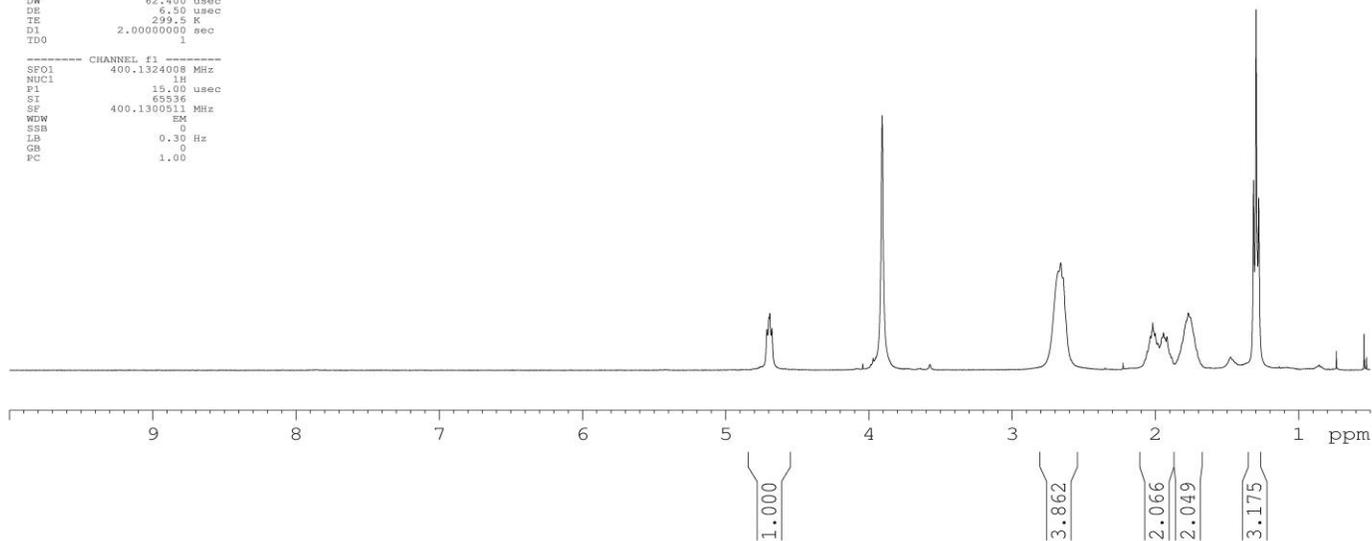
5

```

NAME      egg-04-178
EXPNO     170
PROCNO    1
Date_     20160123
Time      17.27
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   TFA-d
NS         32
DS         0
SWH        8012.820 Hz
FIDRES    0.151323 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.5 K
D1         2.0000000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1      1H
P1         15.00 usec
SI         65536
SF         400.1300511 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```



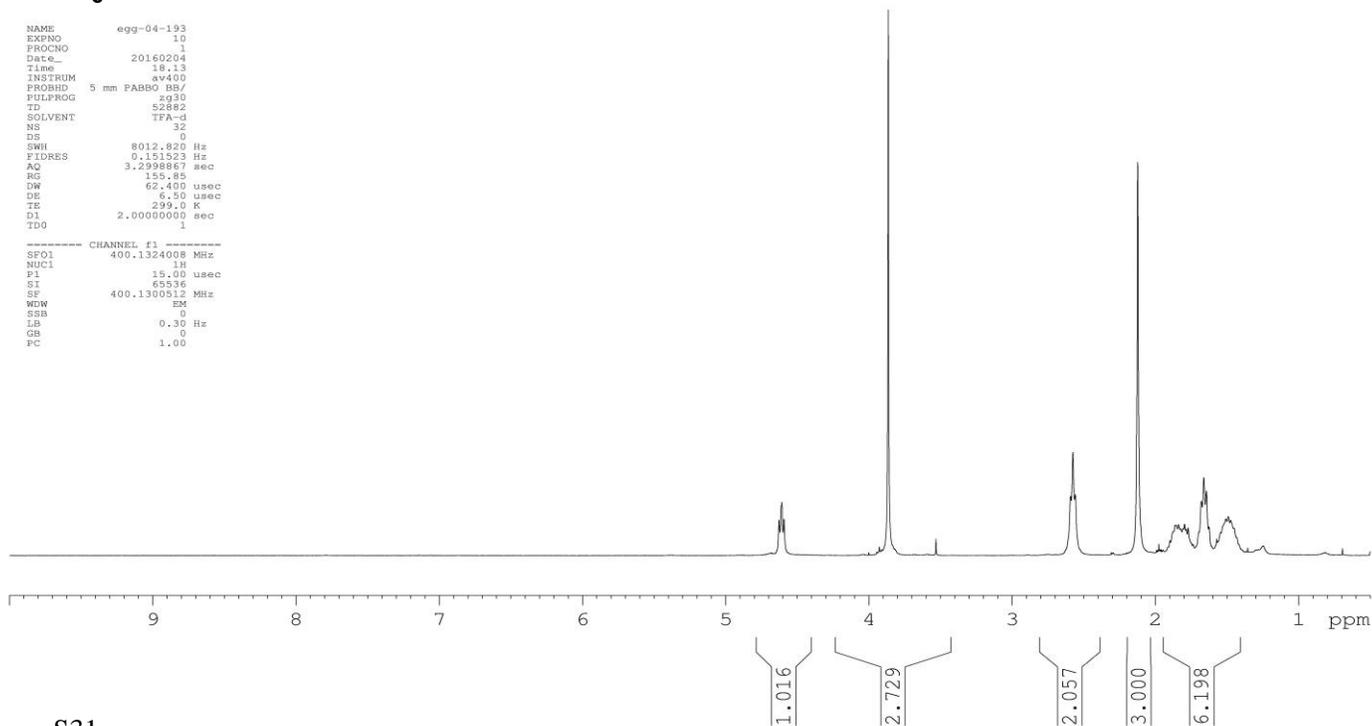
6

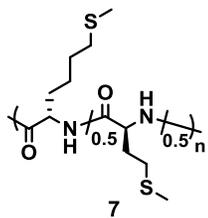
```

NAME      egg-04-193
EXPNO     10
PROCNO    1
Date_     20160204
Time      18.13
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   TFA-d
NS         32
DS         0
SWH        8012.820 Hz
FIDRES    0.151323 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
SFO1      400.1324008 MHz
NUC1      1H
P1         15.00 usec
SI         65536
SF         400.1300512 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```





```

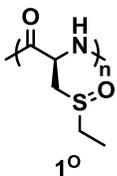
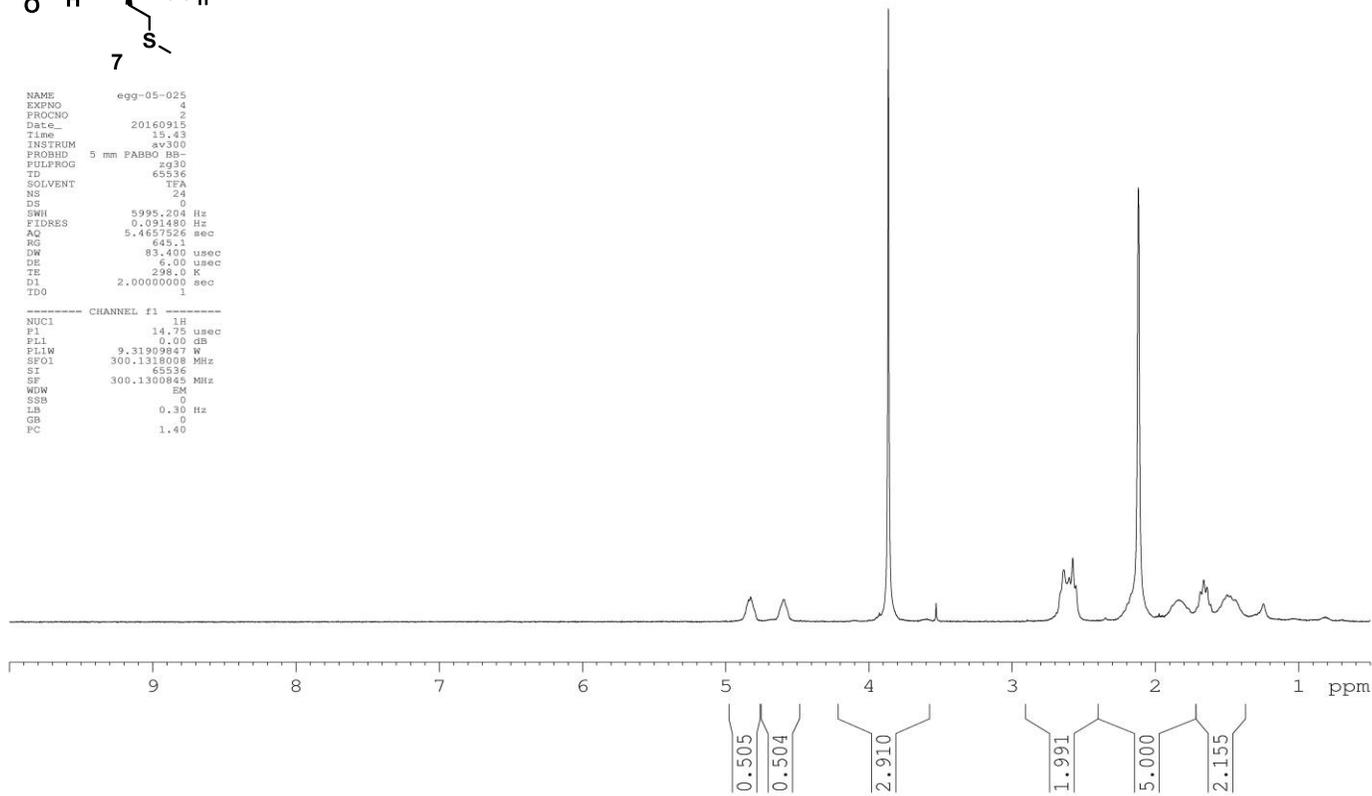
NAME      egg-05-025
EXPNO     4
PROCNO    2
Date_     20160915
Time      15.43
INSTRUM   av300
PROBHD    5 mm FABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   TFA
NS         24
DS         0
SWH        5995.204 Hz
FIDRES     0.091480 Hz
AQ         5.4657526 sec
RG         645.1
DW         83.400 usec
DE         6.00 usec
TE         298.0 K
D1         2.0000000 sec
TDO        1

```

```

----- CHANNEL f1 -----
NUC1       1H
P1         14.75 usec
PL1        0.00 dB
PL1W       9.3199987 W
SFO1       300.1318008 MHz
SI         65536
SF         300.1300845 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.40

```



```

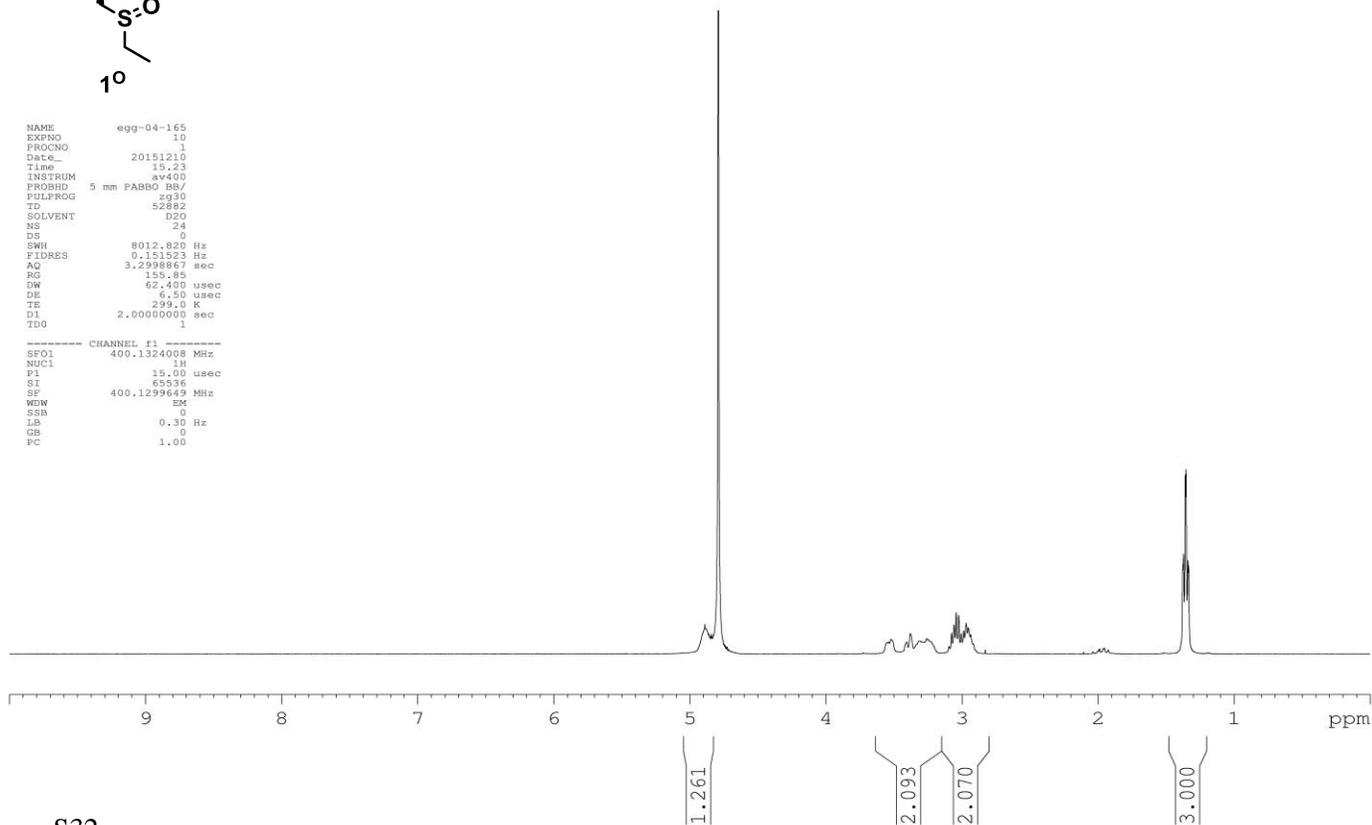
NAME      egg-04-165
EXPNO     10
PROCNO    1
Date_     20151210
Time      15.23
INSTRUM   av400
PROBHD    5 mm FABBO BB/
PULPROG   zg30
TD         52882
SOLVENT   D2O
NS         24
DS         0
SWH        8012.820 Hz
FIDRES     0.151323 Hz
AQ         3.2998867 sec
RG         155.85
DW         62.400 usec
DE         6.50 usec
TE         299.0 K
D1         2.0000000 sec
TDO        1

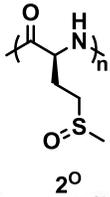
```

```

----- CHANNEL f1 -----
SFO1       400.1324008 MHz
NUC1       1H
P1         15.00 usec
SI         65536
SF         400.1299649 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

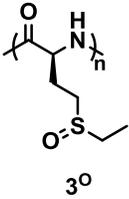
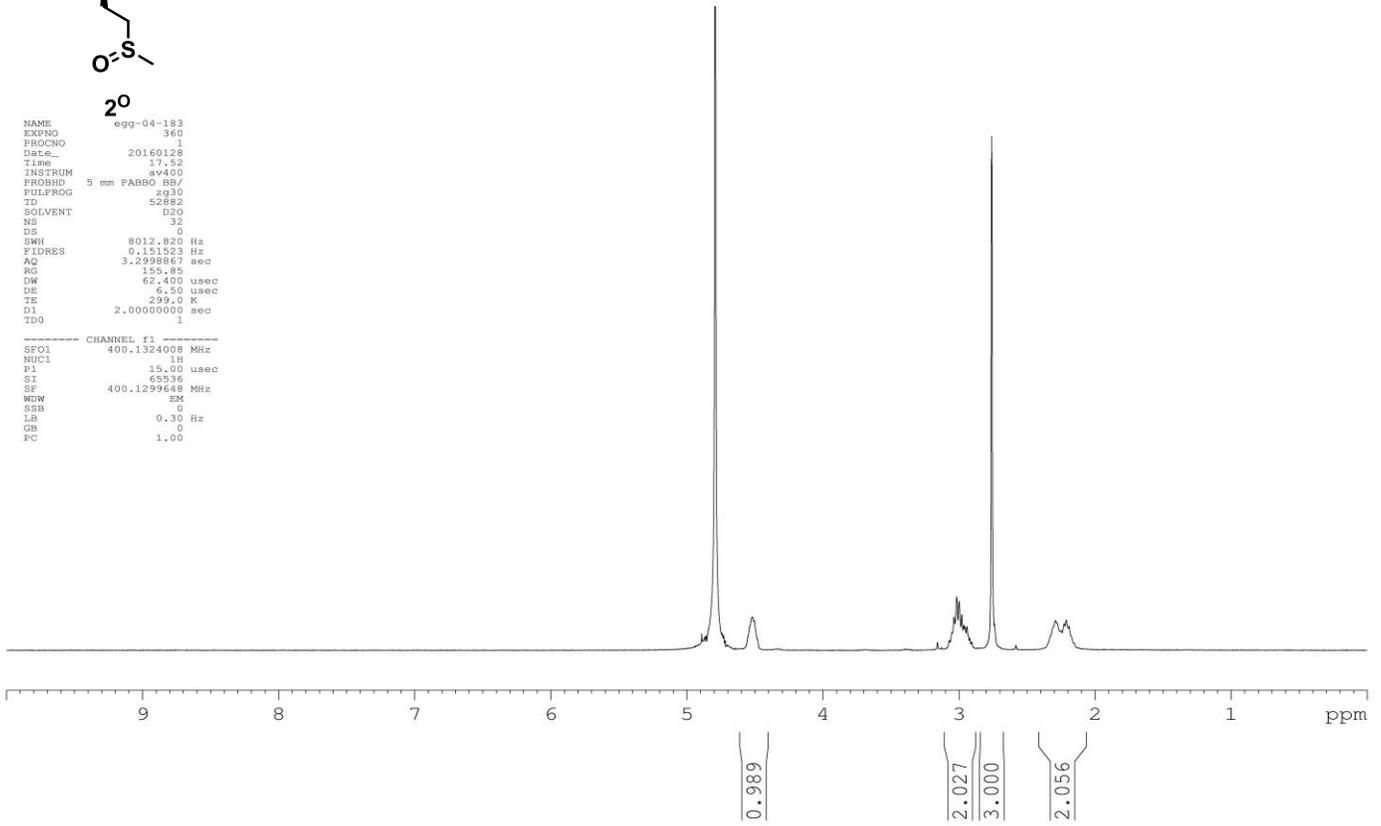
```





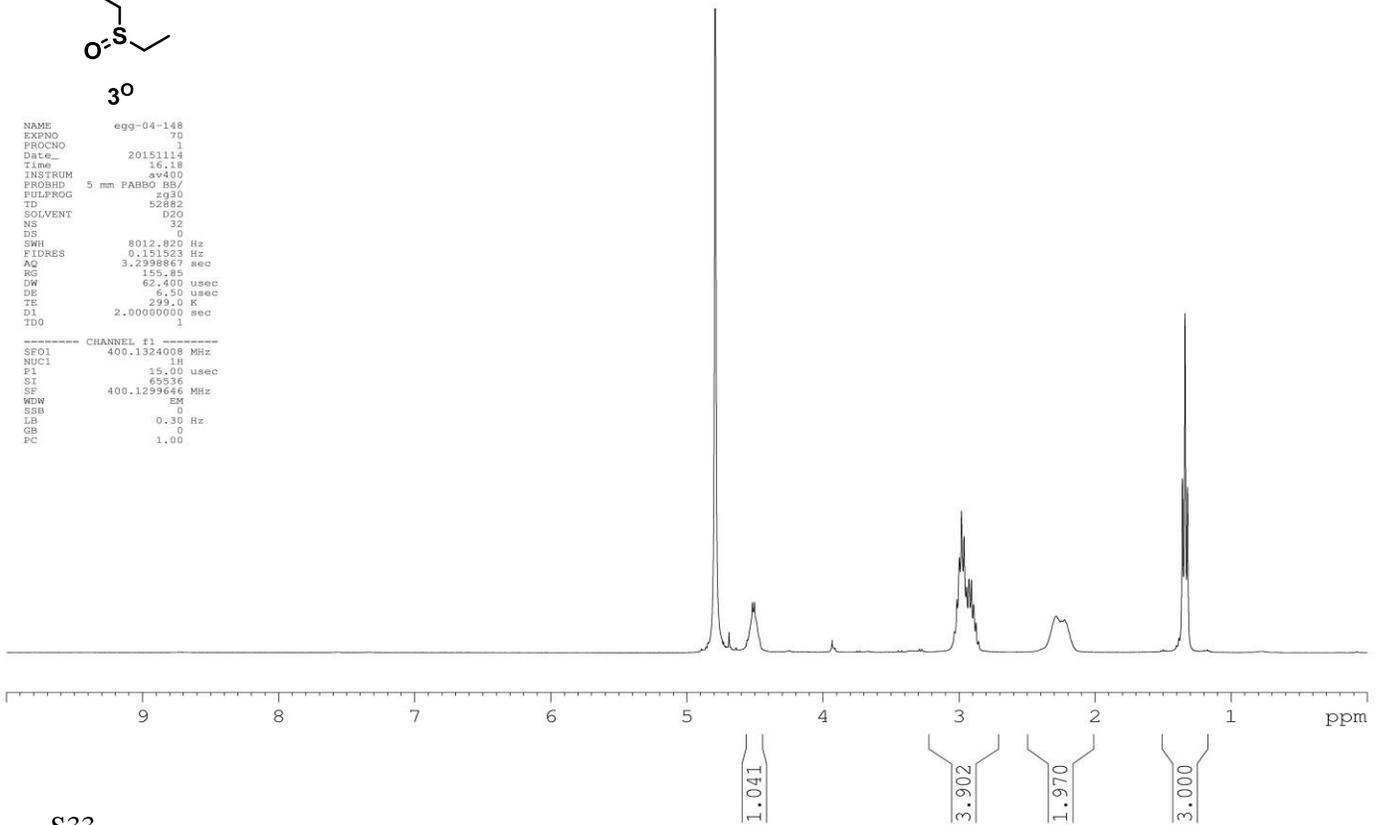
NAME egg-04-183  
 EXPNO 360  
 PROCNO 1  
 Date\_ 20160128  
 Time 17.52  
 INSTRUM av400  
 PROBHD 5 mm FABBO BB/  
 PULPROG zg30  
 TD 52882  
 SOLVENT D2O  
 NS 32  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.151323 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 DI 2.0000000 sec  
 TDO 1

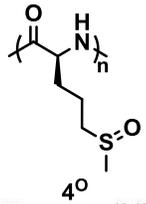
----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65336  
 SF 400.1299648 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 FC 1.00



NAME egg-04-148  
 EXPNO 70  
 PROCNO 1  
 Date\_ 20151114  
 Time 16.18  
 INSTRUM av400  
 PROBHD 5 mm FABBO BB/  
 PULPROG zg30  
 TD 52882  
 SOLVENT D2O  
 NS 32  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.151323 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 DI 2.0000000 sec  
 TDO 1

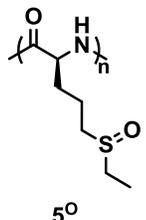
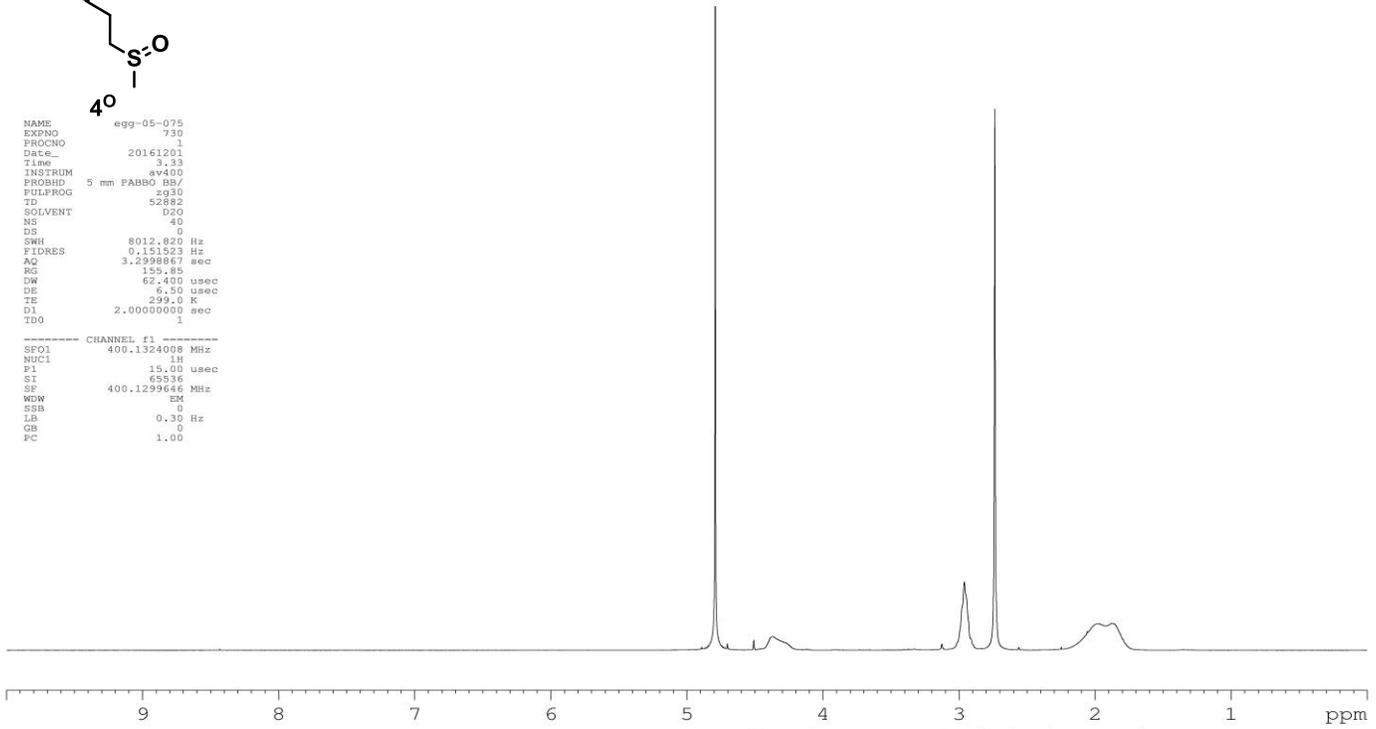
----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65336  
 SF 400.1299646 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 FC 1.00





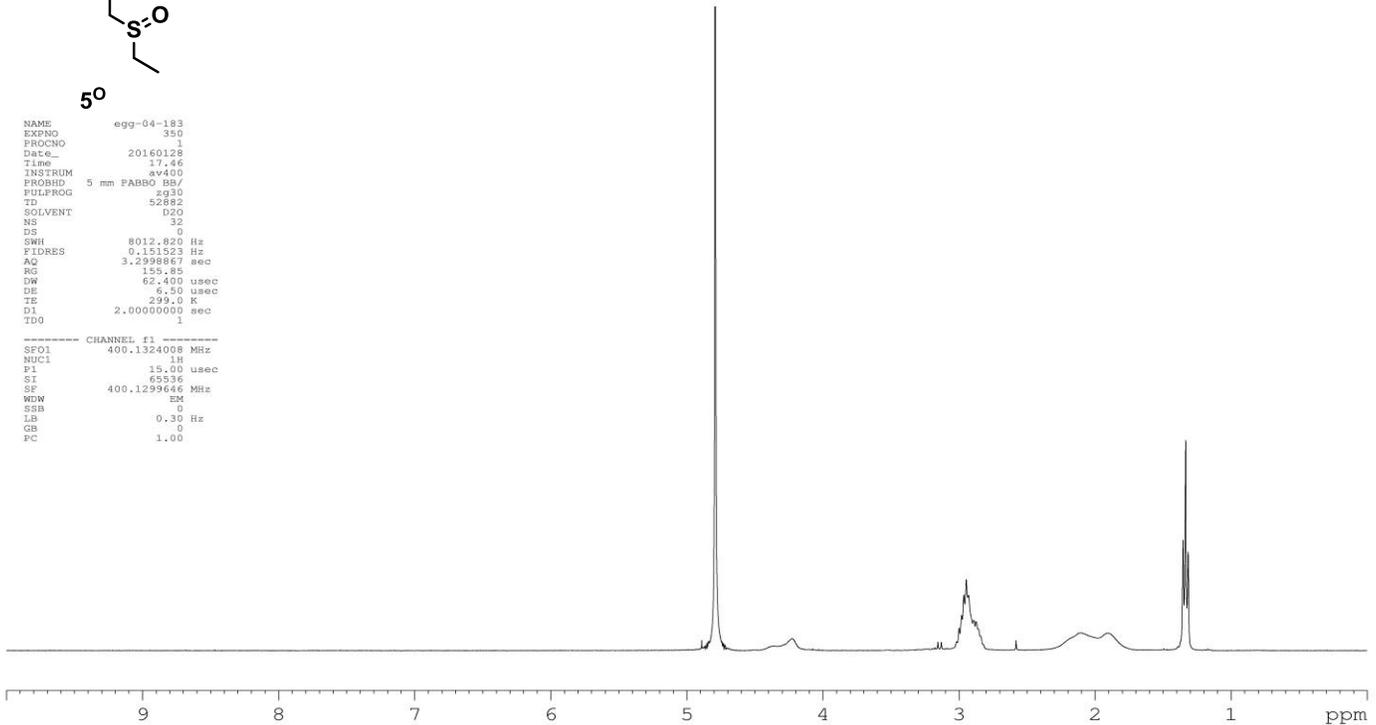
NAME egg-05-075  
 EXPNO 730  
 PROCNO 1  
 Date\_ 20161201  
 Time 3.33  
 INSTRUM av400  
 PROBHD 5 mm FABBO BB/  
 PULPROG zg30  
 TD 52882  
 SOLVENT D2O  
 NS 40  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.151323 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 D1 2.0000000 sec  
 TDO 1

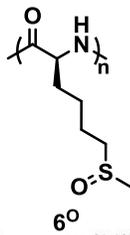
----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65336  
 SF 400.1299646 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 FC 1.00



NAME egg-04-183  
 EXPNO 350  
 PROCNO 1  
 Date\_ 20160128  
 Time 17.46  
 INSTRUM av400  
 PROBHD 5 mm FABBO BB/  
 PULPROG zg30  
 TD 52882  
 SOLVENT D2O  
 NS 32  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.151323 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 D1 2.0000000 sec  
 TDO 1

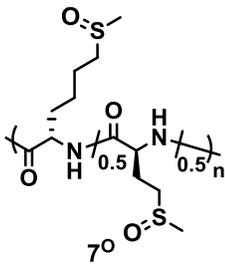
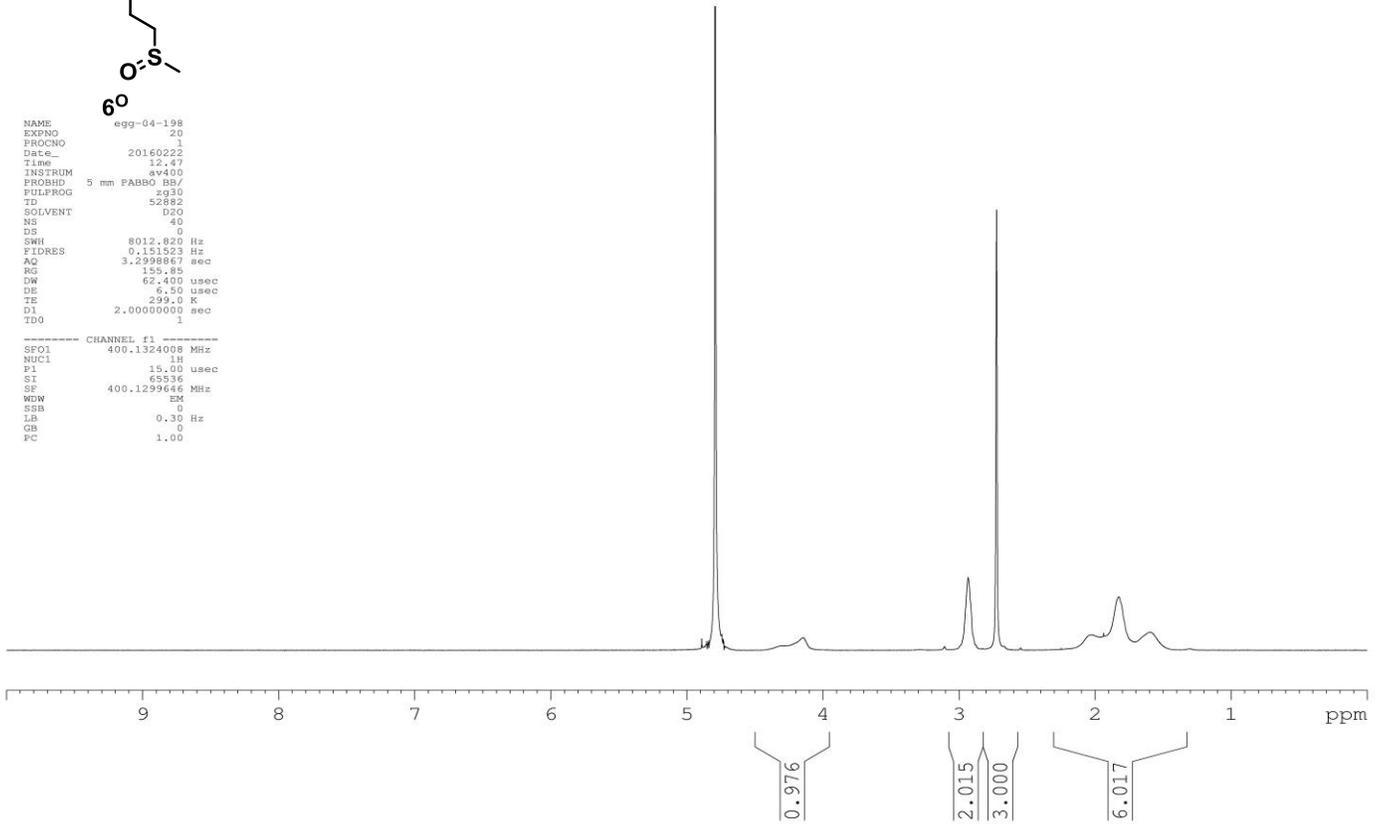
----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65336  
 SF 400.1299646 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 FC 1.00





NAME egg-04-198  
 EXPNO 20  
 PROCNO 1  
 Date\_ 20160222  
 Time 12.47  
 INSTRUM av400  
 PROBHD 5 mm FABBO BB/  
 PULPROG zg30  
 TD 52882  
 SOLVENT D2O  
 NS 40  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.151323 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 DI 2.0000000 sec  
 TDO 1

----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65536  
 SF 400.1299646 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 FC 1.00



NAME egg-05-029  
 EXPNO 310  
 PROCNO 1  
 Date\_ 20160922  
 Time 15.27  
 INSTRUM av400  
 PROBHD 5 mm FABBO BB/  
 PULPROG zg30  
 TD 52882  
 SOLVENT CDCl3  
 NS 32  
 DS 0  
 SWH 8012.820 Hz  
 FIDRES 0.151323 Hz  
 AQ 3.2998867 sec  
 RG 155.85  
 DW 62.400 usec  
 DE 6.50 usec  
 TE 299.0 K  
 DI 2.0000000 sec  
 TDO 1

----- CHANNEL f1 -----  
 SFO1 400.1324008 MHz  
 NUC1 1H  
 P1 15.00 usec  
 SI 65536  
 SF 400.1309888 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 FC 1.00

