Supporting Information

Intramolecular Anodic Olefin Coupling Reactions: Using Competition Studies to Probe the Mechanism of Oxidative Cyclization Reactions

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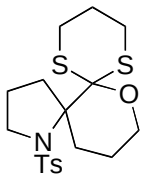
General procedure for electrolysis reactions:

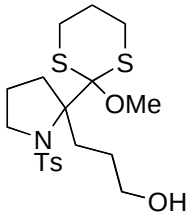
LiOMe (1.0 *M* in MeOH, 0.5 equiv) was added to a methanol solution of the substrate (0.03 *M*, 1 equiv) and the electrolyte Et_4NOTs (0.1 *M*) in a three-neck round bottom flask at rt under argon atmosphere. Two of the three septa were replaced by a reticulated vitreous carbon anode (100 PPI) and platinum wire cathode. The solution was sonicated for 10 min. The electrolysis reaction was carried out at constant current of 6.0 mA until complete consumption of the starting material (the progress of the reaction was monitored by ^{1}H-NMR). When complete, the reaction was concentrated under reduced pressure if Et_4NOTs was used as electrolyte. And then the residue was chromatographed through a silica gel column (slurry packed using 1% triethylamine in hexane solution) to give the desired product. In the case in which $LiClO_4$ was used as electrolyte, water and ether was added. The ether layer was separated and aqueous layer extracted with ether. The combined organic solution was dried and concentrated. The residue was chromatogaphed in a way described above to afford the desired product.

Spectra Data:



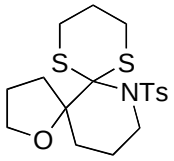
8a

IR (neat, cm^{-1}) 1355, 1100, 836, 754; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 4.11 (td, *J* = 11.4, 3.6 Hz, 1H), 3.76 (dd, *J* = 5.1 Hz, 1H), 3.56-3.26 (m, 4H), 2.96-2.83 (m, 2H), 2.70-2.59 (m, 2H), 2.38 (s, 3H), 2.10-1.68 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 138.9, 129.6, 128.1, 98.9, 74.9, 62.8, 52.4, 37.4, 31.0, 27.5, 26.5, 25.1, 24.6, 23.5, 21.7; ESI HRMS *m/z* $(M+Na)^+$ calcd 422.0899, obsd 422.0887.



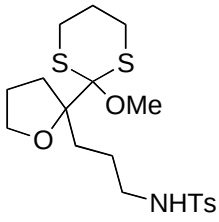
8b

IR (neat, cm^{-1}) 3647, 1154, 670, 589; ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 4.11-4.03 (m, 1H), 3.74 (t, *J* = 5.7 Hz, 2H), 3.55-3.46 (m, 1H), 3.35 (s, 3H), 2.88-2.64 (m, 6H), 2.12-1.71 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.2, 140.2, 128.8, 127.7, 102.2, 83.1, 63.3, 54.2, 52.7, 38.2, 31.1, 28.7, 28.1, 24.2, 22.3, 21.8; ESI HRMS *m/z* $(M+Na)^+$ calcd 454.1151, obsd 454.1144.



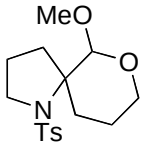
8c

IR (neat, cm^{-1}) 1330, 1050, 657, 572; ^1H NMR (300 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 4.22-4.17 (m, 1H), 4.04-3.98 (m, 1H), 3.76-3.62 (m, 2H), 3.08-3.04 (m, 1H), 3.00-2.91 (m, 1H), 2.79-2.62 (m, 2H), 2.58-2.51 (m, 1H), 2.39 (s, 3H), 2.01-1.90 (m, 2H), 1.88-1.71 (m, 5H), 1.69-1.60 (m, 1H), 1.57-1.50 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.4, 141.3, 128.8, 127.9, 89.5, 69.8, 45.2, 35.7, 33.7, 27.9, 26.9, 26.98, 23.6, 22.8, 21.8; ESI HRMS *m/z* $(M+Na)^+$ calcd 422.0899, obsd 422.0882.



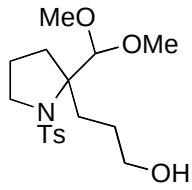
8d

IR (neat, cm^{-1}) 3283, 1193, 1159, 1058; ^1H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 4.54 (t, *J* = 6.3 Hz, 1H), 3.94-3.90 (m, 2H), 3.50 (s, 3H), 3.03-2.87 (m, 4H), 2.83-2.70 (m, 2H), 2.42 (s, 3H), 2.03-1.46 (m, 10 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.5, 137.3, 129.7, 127.4, 102.6, 92.8, 71.2, 53.6, 41.1, 33.7, 33.0, 27.6, 27.2, 27.1, 24.6, 24.0, 21.8; ESI HRMS *m/z* $(M+Na)^+$ calcd 454.1151, obsd 454.1173.



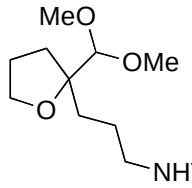
10a

IR (neat, cm^{-1}) 1102, 670, 587, 545; ^1H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 4.82 (s, 1H), 3.96-3.90 (m, 1H), 3.50 (td, *J* = 11.4, 3.6 Hz, 1H), 3.41-3.30 (m, 5H), 2.48-2.43 (m, 1H), 2.41 (s, 3H), 1.95-1.87 (m, 1H), 1.71-1.53 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.0, 139.0, 129.7, 127.3, 105.2, 69.7, 66.4, 56.7, 50.4, 34.7, 32.3, 25.2, 23.3, 21.7; ESI HRMS *m/z* $(M+Na)^+$ calcd 348.1240, obsd 348.1251.



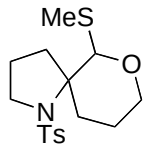
10b

IR (neat, cm^{-1}) 3495, 1328, 669, 592; ^1H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 4.66 (s, 1H), 3.61-3.56 (m, 2H), 3.52 (s, 3H), 3.41-3.28 (m, 5H), 2.40 (s, 3H), 2.35-2.28 (m, 1H), 2.12-2.03 (m, 1H), 1.81-1.41 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.0, 138.9, 129.6, 127.2, 110.5, 73.9, 63.3, 59.3, 57.5, 50.9, 32.2, 31.7, 27.4, 23.7, 21.7; ESI HRMS *m/z* $(M+Na)^+$ calcd 380.1502, obsd 380.1498.



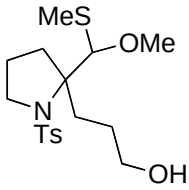
10d

IR (neat, cm^{-1}) 3276, 1080, 815, 662, 550; ^1H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.4 Hz, 7.29 (d, *J* = 8.4 Hz, 2H), 4.90 (t, *J* = 6.0 Hz, 1H), 4.00 (s, 1H), 3.80-3.75 (m, 2H), 3.48 (s, 3H), 3.41 (s, 3H), 2.95-2.87 (m, 2H), 2.41 (s, 3H), 2.06-1.73 (m, 3H), 1.56-1.45 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 137.3, 129.8, 127.3, 110.0, 86.6, 69.2, 58.6, 57.1, 44.0, 33.4, 30.9, 26.9, 23.8, 21.7; ESI HRMS *m/z* $(M+Na)^+$ calcd 380.1502, obsd 380.1494.



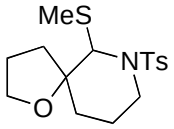
12a

Two isomers were obtained. Isomer 1: IR (neat, cm^{-1}) 1329, 1005, 662, 591, 574; ^1H NMR (300 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 5.18 (s, 1H), 4.03-3.98 (m, 1H), 3.53 (td, *J* = 11.1, 3.0 Hz, 1H), 3.37-3.27 (m, 2H), 2.65-2.58 (m, 1H), 2.43-2.35 (m, 4H), 2.24 (s, 3H), 1.96-1.62 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 138.5, 129.6, 127.5, 92.4, 69.7, 69.3, 49.9, 35.4, 33.9, 25.3, 23.5, 21.7, 14.6; ESI HRMS *m/z* $(M+Na)^+$ calcd 364.1012, obsd 364.1033. Isomer 2: IR (neat, cm^{-1}) 1338, 1068, 660, 592, 546; ^1H NMR (300 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 4.79 (s, 1H), 4.15 (td, *J* = 11.7, 3.9 Hz, 1H), 3.54-3.48 (m, 1H), 3.38-3.28 (m, 2H), 3.18 (td, *J* = 12.9, 5.4 Hz, 1H), 2.68 (ddd, *J* = 12.6, 6.0, 2.1 Hz), 2.40 (s, 3H), 2.04 (s, 3H), 1.90-1.57 (m, 5H), 1.48 (td, *J* = 12.0, 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 138.3, 129.6, 128.0, 88.8, 70.2, 58.5, 50.2, 38.2, 29.0, 24.6, 21.7, 21.5, 13.7; ESI HRMS *m/z* $(M+Na)^+$ calcd 364.1012, obsd 364.1022.



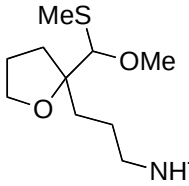
12b

IR (neat, cm^{-1}) 3512, 1322, 667, 590; ^1H NMR (300 MHz, $CDCl_3$, 1:4 mixture of isomers) δ 7.90 (d, *J* = 8.1 Hz, 0.4H), 7.74 (d, *J* = 8.1 Hz, 1.6H), 7.27 (d, *J* = 8.1 Hz, 2H), 4.98 (s, 0.2 H), 4.69 (s, 0.8H), 3.64-3.63 (m, 1.6H), 3.49-3.43 (m, 2.2H), 3.43-3.27 (m, 0.8H), 3.17 (s, 2.4H), 2.40 (s, 3H), 2.33 (s, 0.6H), 2.21-2.12 (m, 5H), 1.88-1.54 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.9, 138.9, 129.6, 127.2, 97.6, 76.0, 63.2, 56.0, 51.2, 33.7, 33.4, 27.7, 23.8, 21.7, 16.9; ESI HRMS *m/z* $(M+Na)^+$ calcd 396.1274, obsd 396.1266.



12c

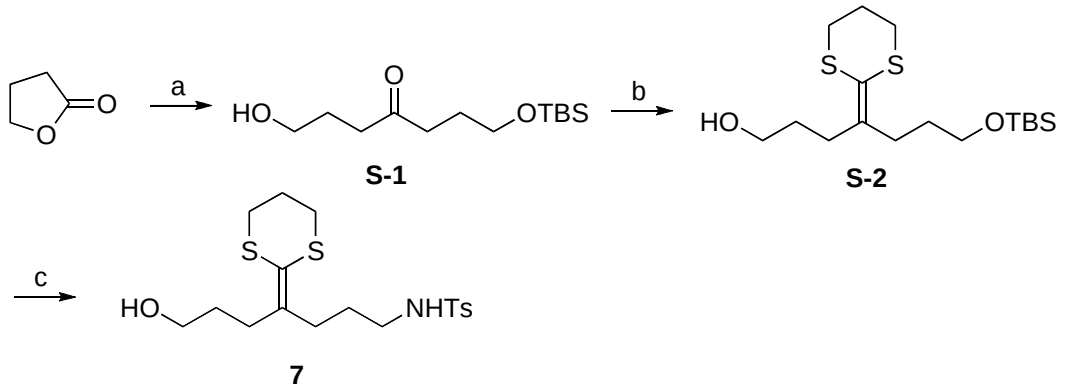
IR (neat, cm^{-1}) 1336, 1161, 1091, 655, 574; ^1H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 5.00 (s, 1H), 3.89 (t, *J* = 6.6 Hz, 2H), 3.56-3.50 (m, 1H), 3.01 (td, *J* = 12.6, 3.3 Hz, 1H), 2.42 (s, 3H), 2.14-1.91 (m, 7H), 1.74-1.62 (m, 2H), 1.51-1.45 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.8, 137.4, 129.8, 127.7, 84.0, 71.2, 68.1, 40.6, 35.1, 31.5, 25.9, 23.8, 21.8, 16.0; ESI HRMS *m/z* $(M+Na)^+$ calcd 364.1012, obsd 364.1026.



12d

IR (neat, cm^{-1}) 3267, 1326, 1093, 662, 550; ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.1 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 5.07-5.02 (m, 1H), 4.14 (s, 1H), 3.89-3.79 (m, 2H), 3.45, 3.44 (2s, 3H), 2.95-2.90 (m, 2H), 2.42 (s, 3H), 2.17, 2.12 (2s, 3H), 2.07-1.49 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 137.3, 129.9, 127.3, 96.5, 95.2, 88.2, 87.9, 69.5, 69.3, 58.0, 57.6, 43.9 (d), 34.5, 34.4, 33.2, 32.4, 26.9 (d), 24.1, 21.7, 15.7, 14.5; ESI HRMS *m/z* $(M+Na)^+$ calcd 396.1274, obsd 396.1269.

Synthesis of the Substrates:



Reaction conditions: a. $ICH_2CH_2CH_2OTBS$, *t*-BuLi, Et_2O, -78 °C, 48%; b. 2-trimethylsilyl-1,3-dithane, *n*-BuLi, 52%; c. 1[1]. TsNHBoc, Ph_3P, DEAD, THF, rt, 2. LiMe, -20 °C, Et_2O, 3. TBAF, THF, rt, 81%.

Synthesis of S-1: To a solution of *tert*-butyl(3-iodopropoxy)dimethylsilane (6.0 g, 20 mmol) was added *t*-BuLi solution (1.7 M in pentane, 23.5 mL, 40 mmol) at -78 °C under argon atmosphere. The resulting mixture was stirred at -78 °C for 0.5 h and then rt for 1 h. In another flask, a solution of butyrolactone (1.7 g, 20 mmol) in Et_2O was cooled to -78 °C and treated with the above lithium reagent. Water was added after 15 min and the cold bath was removed. Ether layer was separated and the aqueous phase extracted with ether. The combined organic solution was dried over $MgSO_4$, filtered, and concentrated. The residue was chromatographed through silica gel (eluted with acetone/hexane, 1:4) to give **S-1** (2.5 g, 48%) and. IR (neat, cm^{-1}) 3411, 1713, 1255, 1100, 836; ^1H NMR (300 MHz, $CDCl_3$) δ 3.63-3.56 (m, 4H), 2.57-2.47 (m, 4H), 1.83-1.74 (m, 4H), 0.86 (s, 9H), 0.03 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 211.8, 62.4, 62.3, 39.8, 39.4, 27.0, 26.7, 26.1, 18.5, -5.2; ESI HRMS *m/z* $(M+Na)^+$ calcd 283.1700, obsd 283.1689.

Synthesis of S-2: *n*-BuLi (1.6 M in hexanes, 13.2 mL, 21.2 mmol) was added drop-wise to a solution of 2-trimethylsilyl-1,3-dithiane (3.93 mL, 21.2 mmol) in THF (40 mL). The resulting mixture was stirred at -78 °C for 0.5 h and then 0 °C for an additional 0.5 h. The reaction was then cooled to -78 °C and treated with **S-1** (1.84 g, 7.07 mmol). Upon complete addition, the reaction was allowed to warm to rt slowly and stirred overnight. The reaction mixture was poured into water (50 mL) and extracted with ether. The combined extractions were dried over $MgSO_4$ and evaporated. Chromatography through silica gel afforded **S-1** (0.50 g, 27%) and **S-2** (1.34g, 52%). IR (neat, cm^{-1}) 3868, 1254, 1102, 835, 775; ^1H NMR (300 MHz, $CDCl_3$) δ 3.59-3.55 (m, 4H), 2.85-2.79 (m, 4H), 2.40-2.29 (m, 4H), 2.14-2.04 (m, 3H), 1.65-1.54 (m, 4H), 0.85 (s, 9H), 0.02 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.3, 120.9, 63.1, 62.1, 31.7, 30.9, 30.6, 30.5, 30.4, 29.9, 26.2, 25.2, 18.5; ESI HRMS *m/z* $(M+Na)^+$ calcd 385.1662, obsd 385.1651.

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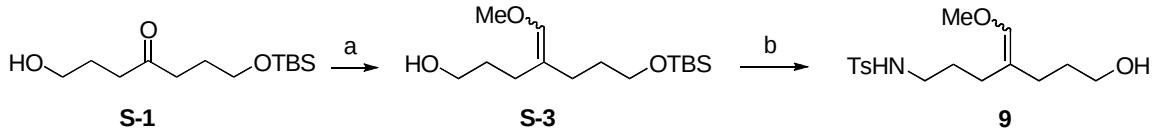
[1] Henry, J. R.; Marcin, L. R.; McIntosh, M. C.; Scola, P. M.; Harris, G. D.; Weinreb, S. M. *Tetrahedron Lett.* **1989**, *30*, 5709–5712.

Synthesis of 7: Diethyl azodicarboxylate solution (40 wt% soluion in toluene, 3.40 mL, 7.50 mmol) was added drop-wise to a solution of **S-2** (1.06 g, 2.92 mmol), *N*-(*tert*-butoxycarbonyl)-*p*-toluenesulfonamide (1.22 g, 4.50 mmol) and triphenylphosphine (2.36 g, 9.00 mmol) in THF (40 mL) at rt. After stirred overnight, the solvent was removed and the residue filtered through a pad of silica gel to give a mixture of the desired product and Ph_3P. The mixture was then dissolved in Et_2O (24 mL) and treated with LiMe (9.10 mL, 14.5 mmol) at -20 °C. Water was added slowly after 15 min and the cold bath was then removed. The organic layer was separated and aqueous phase extracted with ether. The combined organic solution was dried with $MgSO_4$, filtered and concentrated. The residue was dissolved in THF (15 mL) and treated with TBAF (4.80 mL, 4.80 mmol) at rt. The reaction was kept at rt for 2 h and then poured into water (40 mL). Ether was added and organic layer separated. The water layer was extracted with ether. The organic solutions were combined, dried with $MgSO_4$ and concentrated under reduced pressure. The residue was chromatographed (silica gel, ether) to give **7** (0.95 g, 81%). IR (neat, cm^{-1}) 3499, 3278, 1323, 814, 662, 550; ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 4.81 (t, *J* = 6.0 Hz, 1H), 3.61-3.55 (m, 2H), 2.97-2.91 (m, 2H), 2.86-2.81 (m, 4H), 2.42 (s, 3H), 2.34-2.26 (m, 4H), 2.14-2.06 (m, 2H), 1.79 (t, *J* = 5.7 Hz, 1H), 1.66-1.50 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.6, 142.8, 137.3, 130.0, 127.4, 122.0, 62.2, 43.0, 31.0, 30.6, 30.5, 29.8, 28.0, 25.0, 21.8; ESI HRMS *m/z* $(M+Na)^+$ calcd 424.1045, obsd 424.1043.



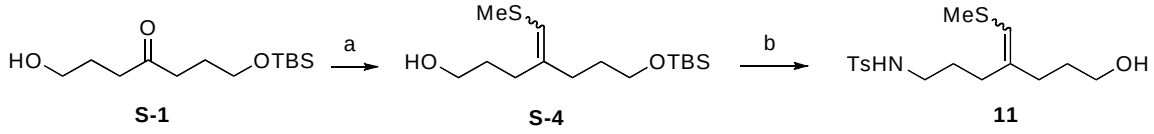
S-1 **S-3** **9**

Reaction conditions: a. Ph_3PCH_2OMeCl, NaHMDS, THF, 59%; b. 1. TsNHBoc, Ph_3P, DEAD, THF, rt, 2. LiMe, -20 °C, Et_2O, 3. TBAF, THF, rt, 70%.

Synthesis of S-3: To a suspension of methoxymethyltriphenylphosphonium chloride (27.4 g, 80.0 mmol) in THF (160 mL) was added NaHMDS solution (1.0 M in THF, 80.0 mL, 80.0 mmol) at 0 °C. The resulting reaction mixture was stirred at 0 °C for 0.5 h and then treated with **S-1** (5.2 g, 20.0 mmol). The reaction was stirred overnight and warmed to rt. Water was added, followed by ether. The organic layer was separated and aqueous layer extracted twice with ether. The combined organic layers were dried with $MgSO_4$ and concentrated *in vacuo*. Chromatography through silica gel gave **S-3** as a 2:3 mixture of isomers (3.40 g, 59%). IR (neat, cm^{-1}) 3363, 1255, 1100, 836; ^1H NMR (300 MHz, $CDCl_3$) δ 5.78 (s, 0.4H), 5.73 (s, 0.6H), 3.56-3.52 (m, 4H), 3.49 (s, 1.2H), 3.46 (s, 1.8H), 2.10 (t, *J* = 7.2 Hz, 1.2H), 2.02, (t, *J* = 7.8 Hz, 1.8H), 1.92-1.83 (m, 2H), 1.59-1.50 (m, 4H), 0.84 (s, 9H), -0.01 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.8, 142.6, 117.4, 116.9, 63.4, 62.8, 62.5, 61.5, 59.5, 59.3, 31.4, 31.3, 31.2, 29.9, 28.0, 27.7, 26.1, 23.3, 22.6, 18.5, -5.1; ESI HRMS *m/z* $(M+Na)^+$ calcd 311.2013, obsd 311.2006.

Synthesis of 9: Compound **9** was synthesized from **S-3** by following the same procedure described for the synthesis of **7**. IR (neat, cm^{-1}) 3499, 3280, 1323, 1128, 662, 550; ^1H NMR (300 MHz, $CDCl_3$) δ 7.70, 7.68 (2d, *J* = 8.1, 8.4 Hz, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 5.72 (s, 1H), 5.44-5.36 (m, 1H), 3.54-3.43 (m, 5H), 2.82 (q, *J* = 8.1 Hz, 2H), 2.68 (t, *J* =

6.3 Hz, 0.6H), 2.37 (s, 3.4H), 2.03-1.96 (m, 2H), 1.79 (t, 7.5 Hz, 2H), 1.55-1.41 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.5, 143.4, 143.2, 137.5, 137.2, 129.9, 129.8, 127.3, 127.2, 116.1, 115.8, 62.4, 61.6, 59.6, 42.8, 42.4, 31.1, 29.9, 28.4, 28.0, 27.5, 26.9, 23.4, 22.4, 21.9; ESI HRMS *m/z* $(M+Na)^+$ calcd 350.1397, obsd 350.1406.



S-1 **S-4** **11**

Reaction conditions: a. Ph_3PCH_2SMeCl, *n*-BuLi, THF, 34%; b. 1. TsNHBoc, Ph_3P, DEAD, THF, rt, 2. LiMe, -20 °C, Et_2O, 3. TBAF, THF, rt, 75%.

Synthesis of S-4: To a suspension of (methylthiomethyl)triphenylphosphonium chloride (21.5 g, 60.0 mmol) in THF (150 mL) was added drop-wise *n*-BuLi solution (1.6 M in hexanes, 37.5 mL, 60.0 mmol) at 0 °C under argon atmosphere. After complete addition, solution was stirred at 0 °C for 0.5 h and then treated with **S-1** (5.2 g, 20 mmol). The reaction was stirred at rt for 4 days. Brine and ether was added at 0 °C. The organic phase was separated and aqueous lay extracted with ether. The organic solutions were combined, dried over $MgSO_4$, and concentrated. Chromatography on silica gel afforded **S-1** (1.00 g, 19%) and **S-4** (2.10 g, 34%). IR (neat, cm^{-1}) 3350, 1255, 1102, 836; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.58 (s, 1H), 3.56 (t, *J* = 6.6 Hz, 4H), 2.25 (br, 1H), 2.18 (s, 3H), 2.15-2.06 (m, 4H), 1.67-1.54 (m, 4H), 0.85 (s, 9H), 0.01 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.6, 121.2, 63.3, 62.5, 33.0, 31.0, 30.9, 8.6, 26.1, 18.5, 17.4, -5.0; ESI HRMS *m/z* $(M+Na)^+$ calcd 327.1784, obsd 327.1790.

Synthesis of 11: Compound **11** was synthesized from **S-4** by following the same procedure described for the synthesis of **7**. IR (neat, cm^{-1}) 3494, 3280, 1321, 1157, 661, 550; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 5.54 (s, 1H), 5.40 (t, *J* = 6.0 Hz, 1H), 3.56-3.50 (q, *J* = 5.7 Hz, 2H), 2.89-2.82 (q, *J* = 6.6 Hz, 2H), 2.38-2.34 (m, 4H), 2.17 (s, 3H), 2.09 (t, *J* = 6.9 Hz, 2H), 2.00 (t, *J* = 8.1 Hz, 2H), 1.61-1.50 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.6, 138.3, 137.2, 129.9, 127.3, 122.1, 62.2, 42.9, 33.3, 30.3, 28.0, 21.7, 17.4; ESI HRMS *m/z* $(M+Na)^+$ calcd 366.1168, obsd 366.1162.

8a
S
O
S
N
Ts
ppm
1
2
3
4
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1.01
1.01
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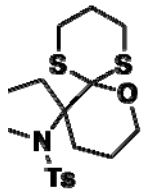

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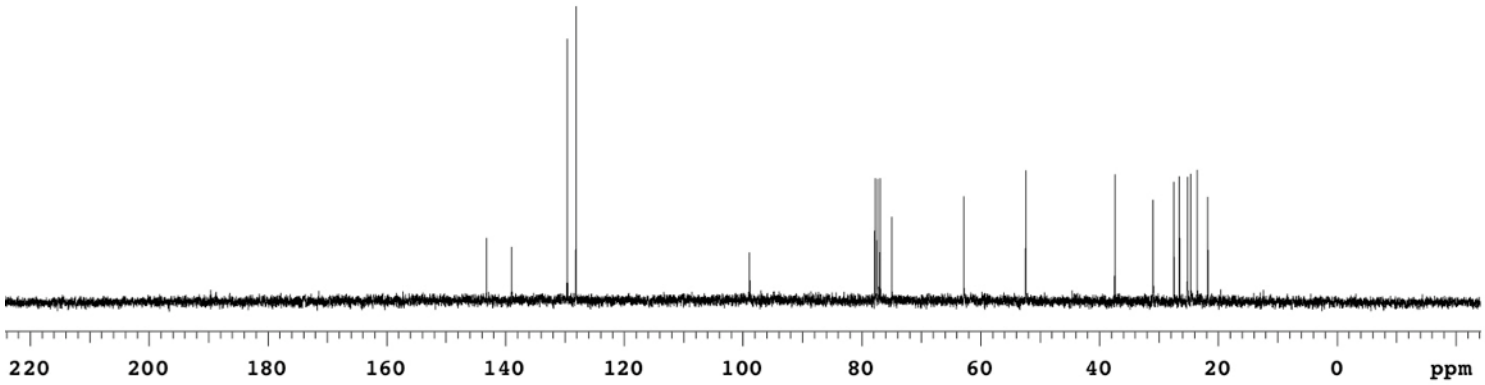

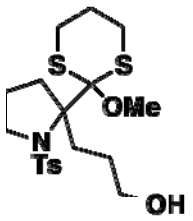

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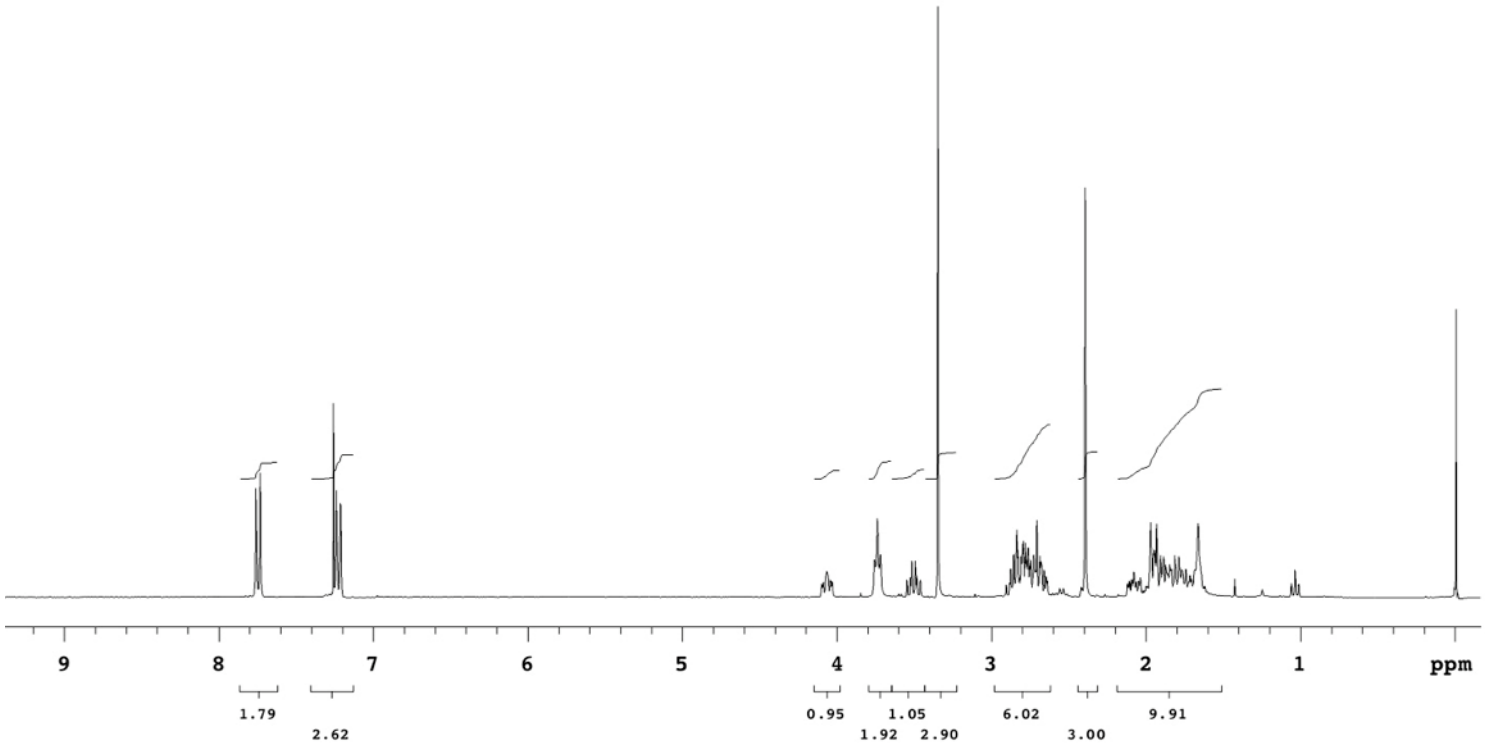

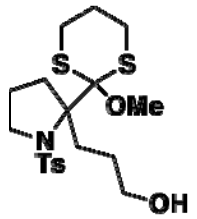

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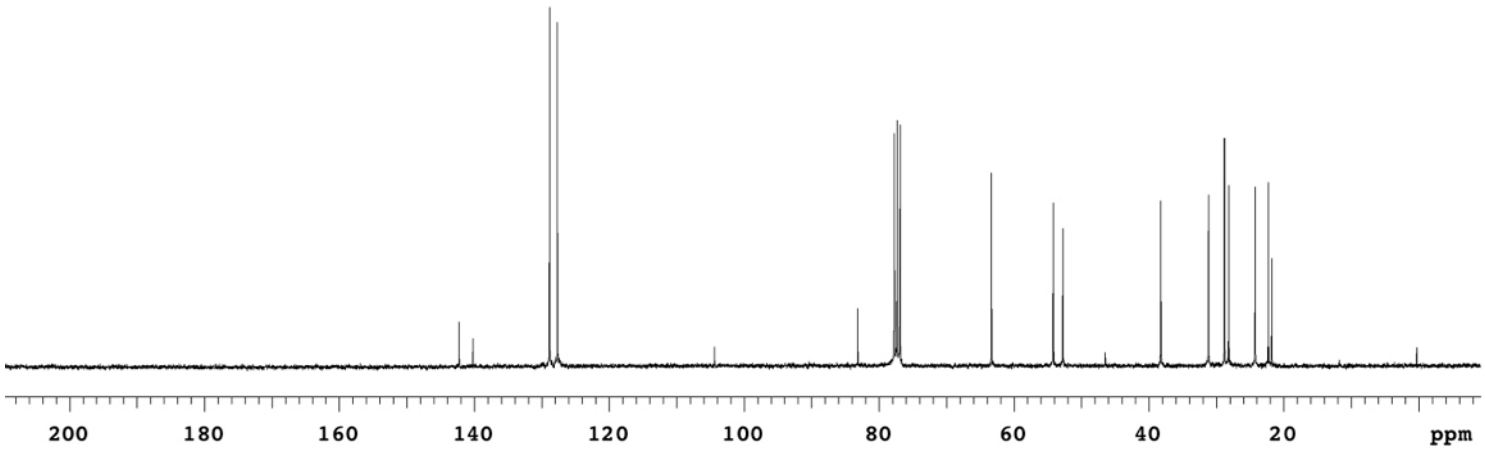

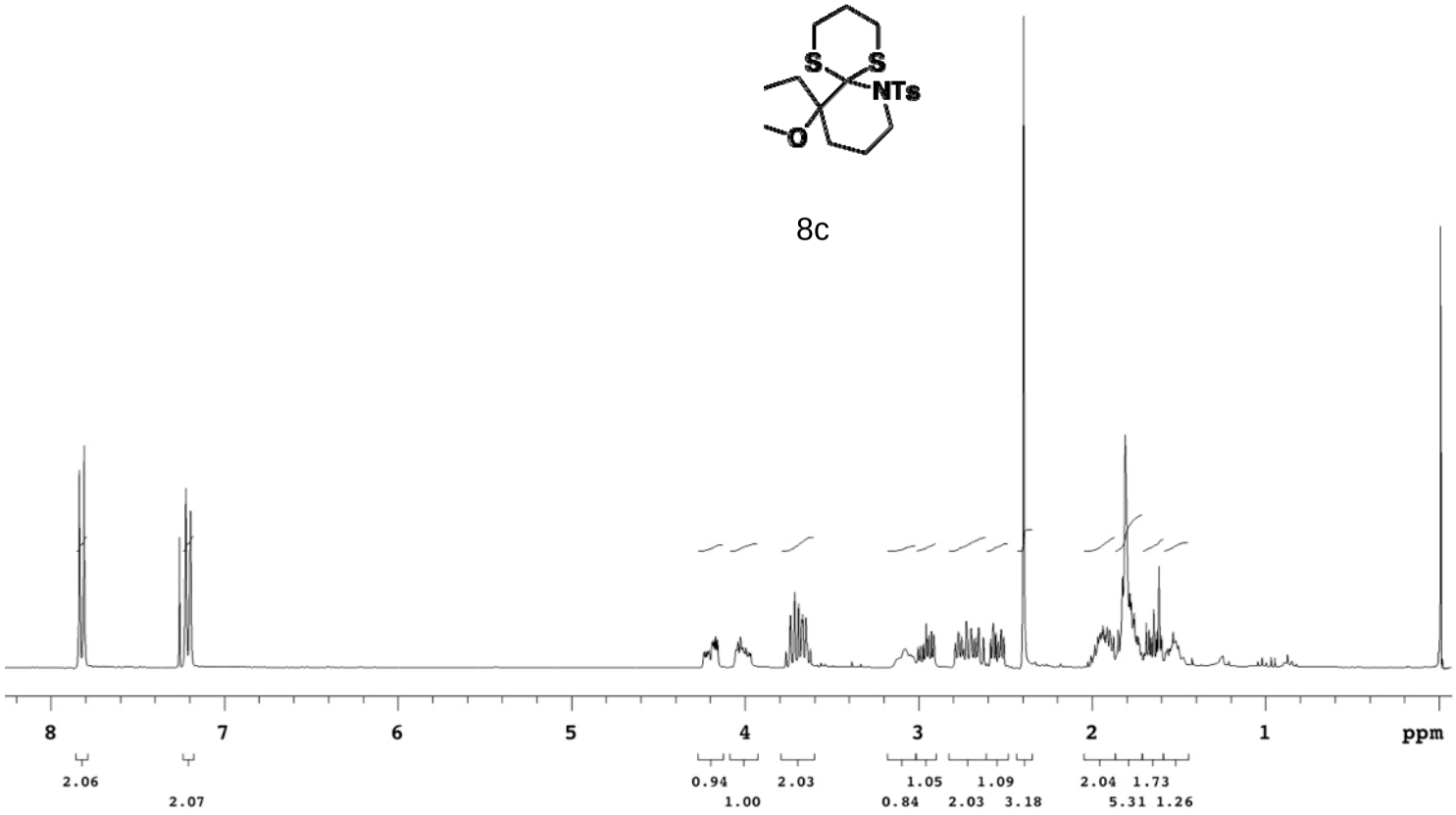
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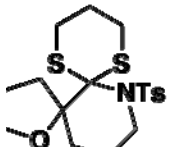

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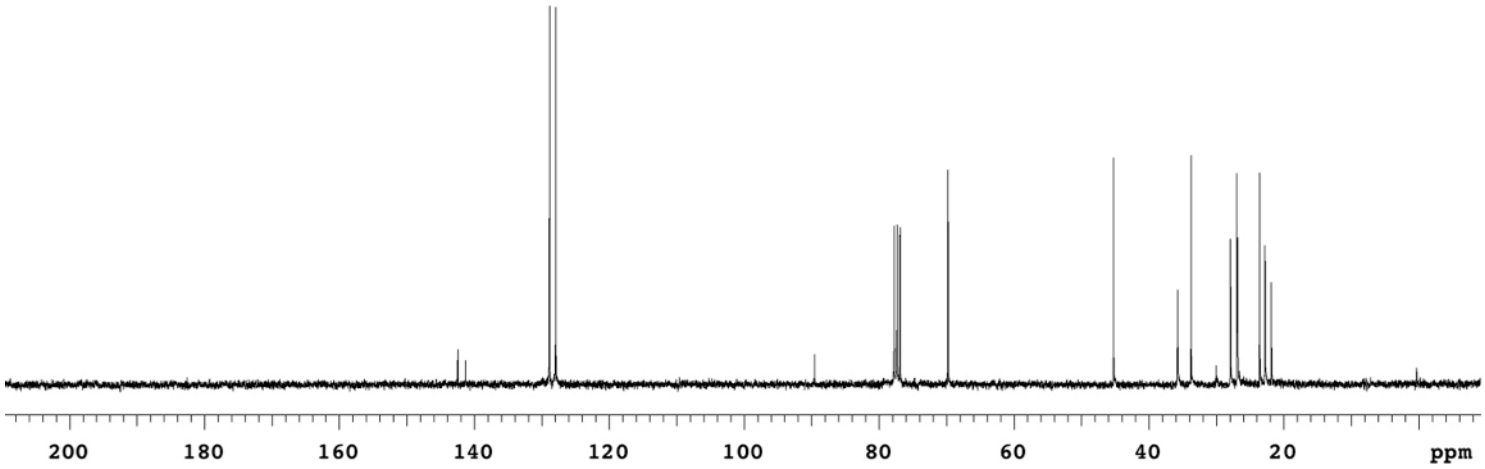

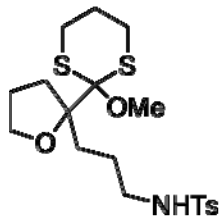
S
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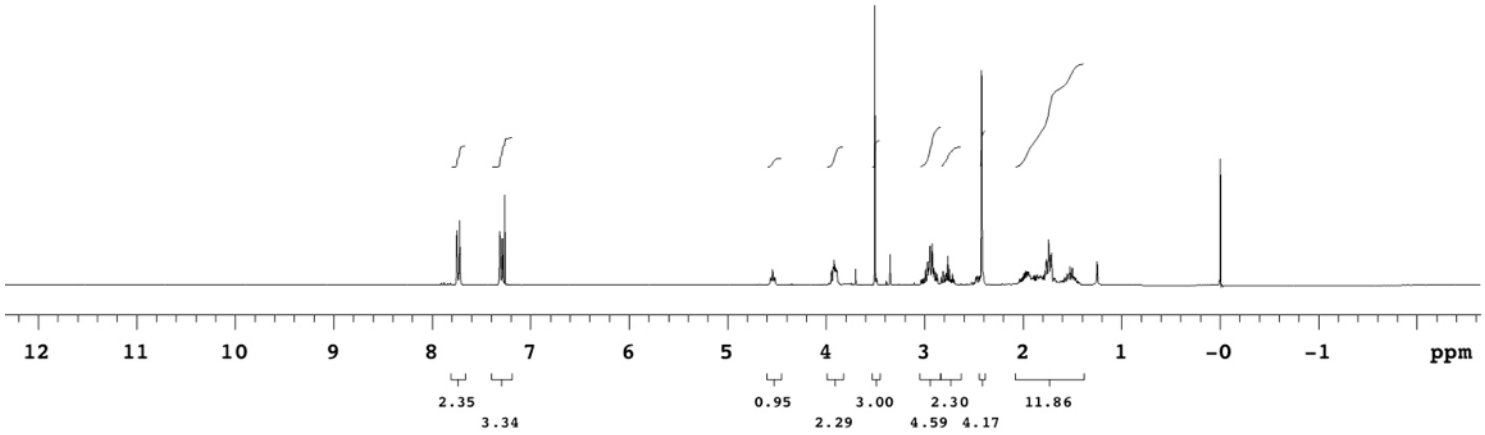
OMe
O
NHTs

8d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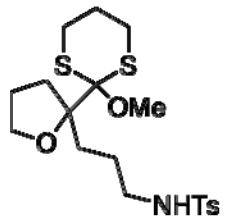

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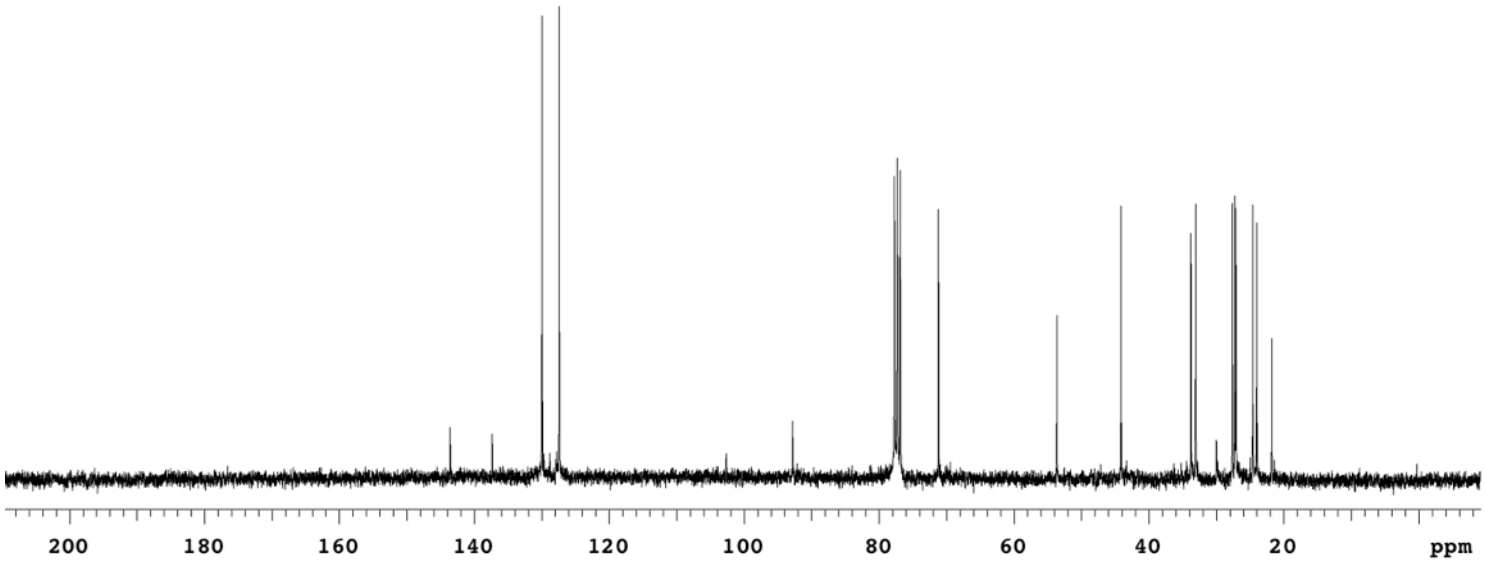

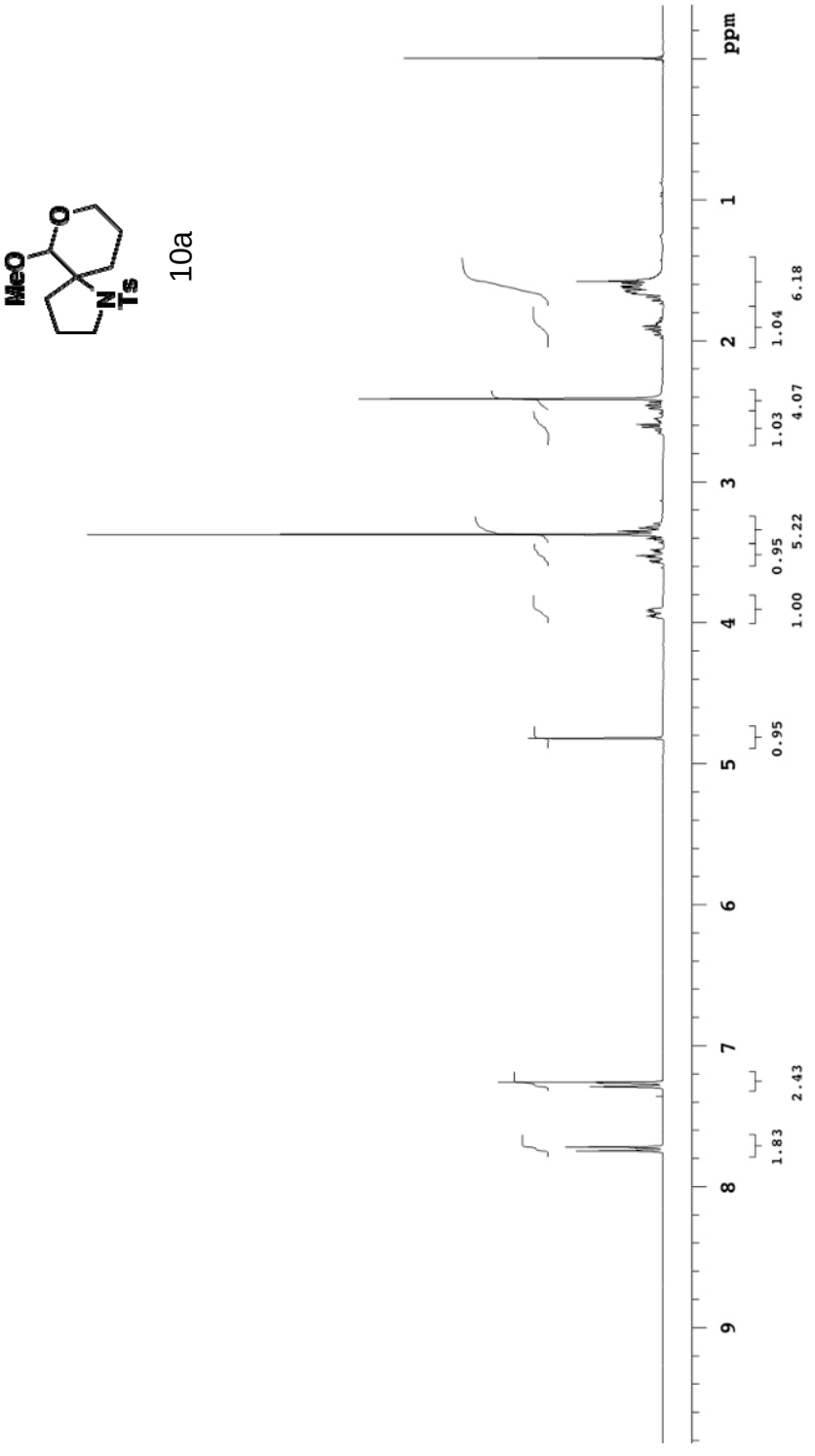
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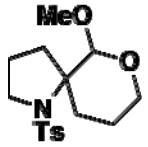

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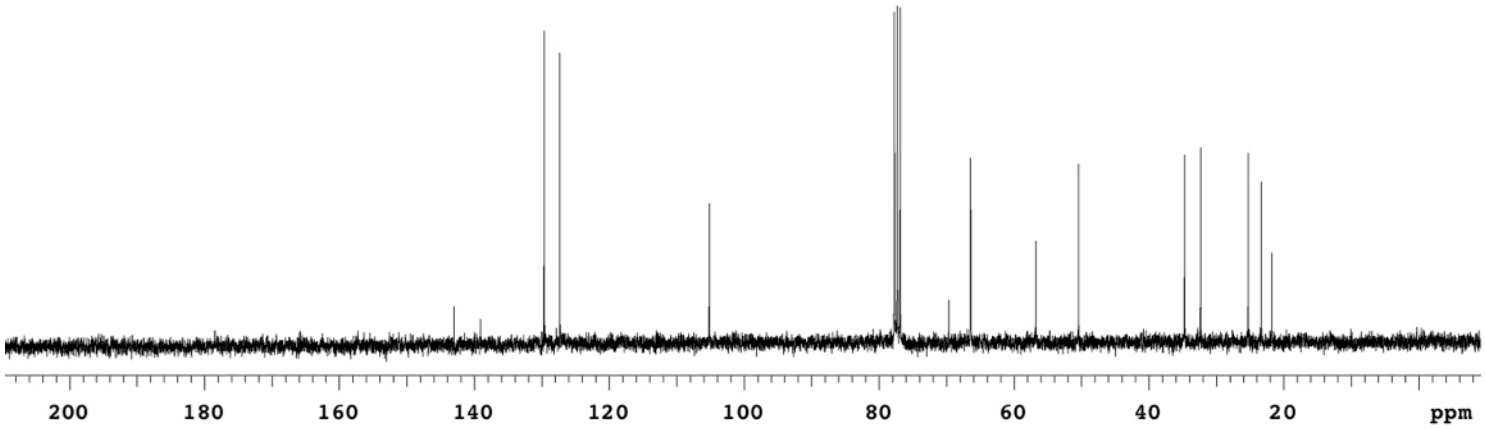

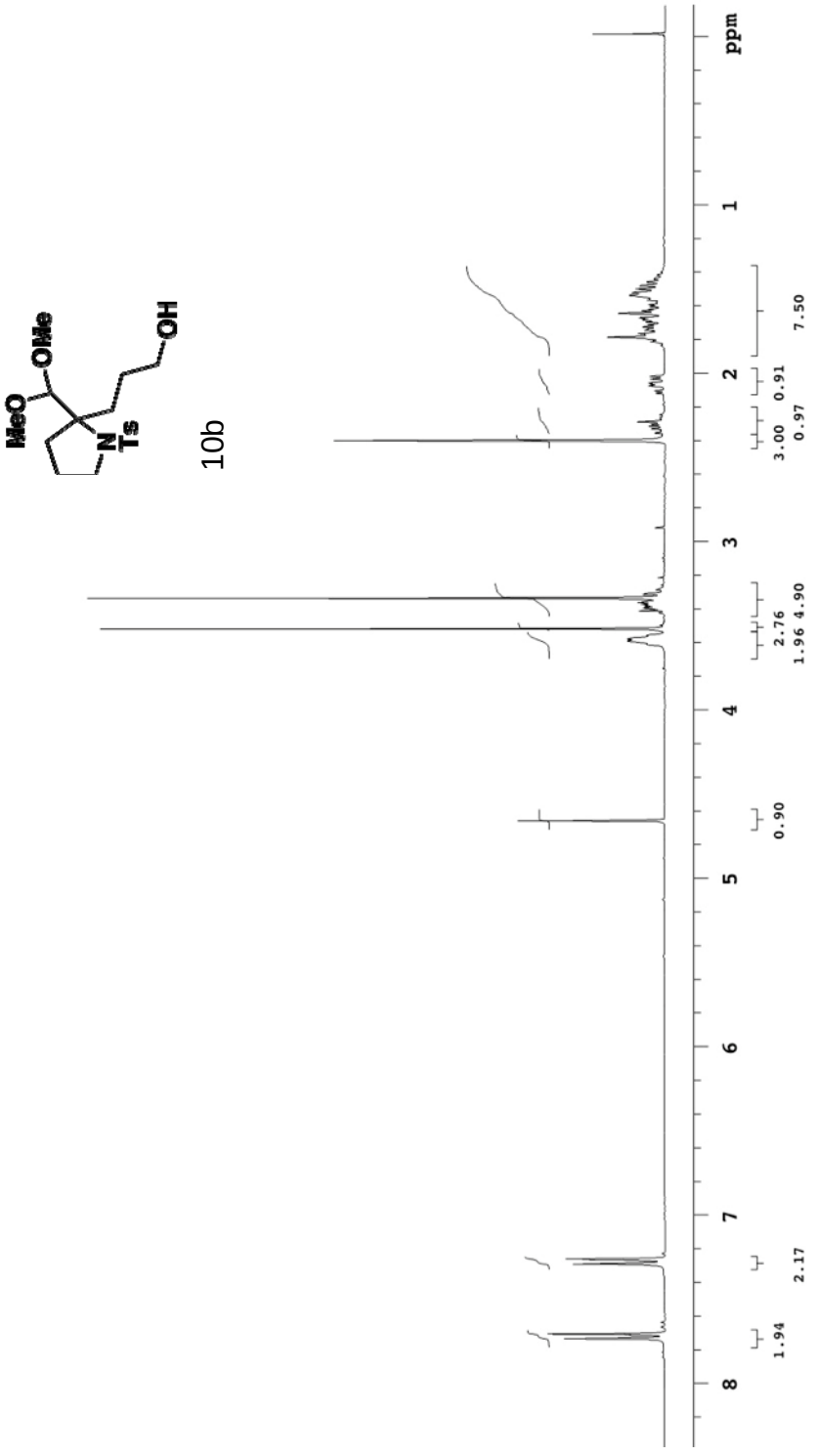
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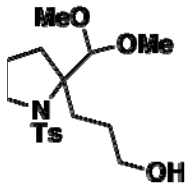

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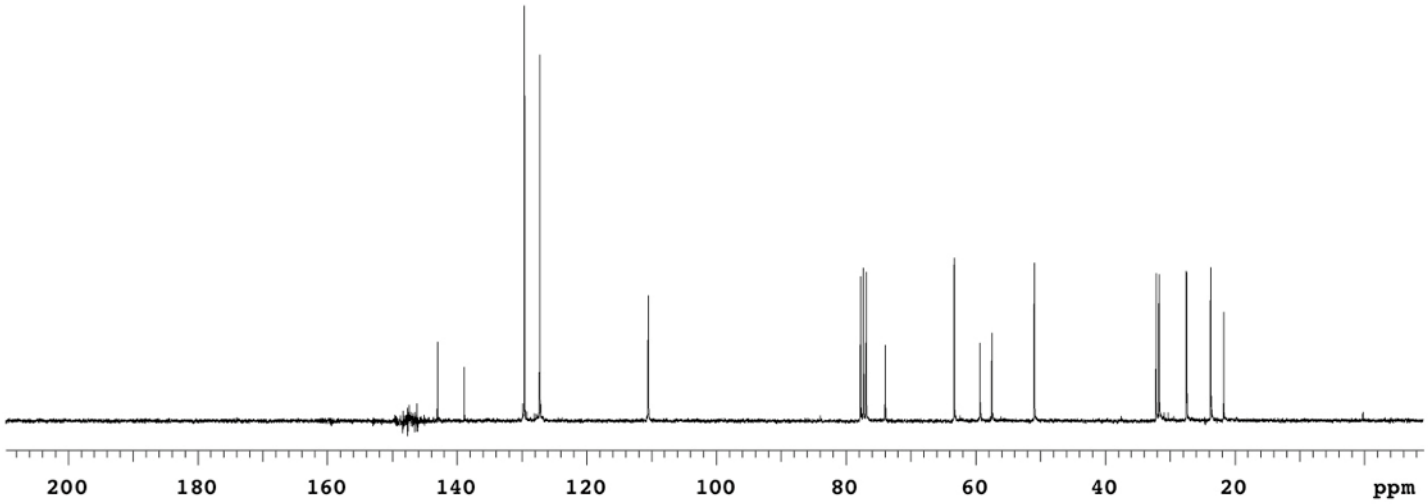

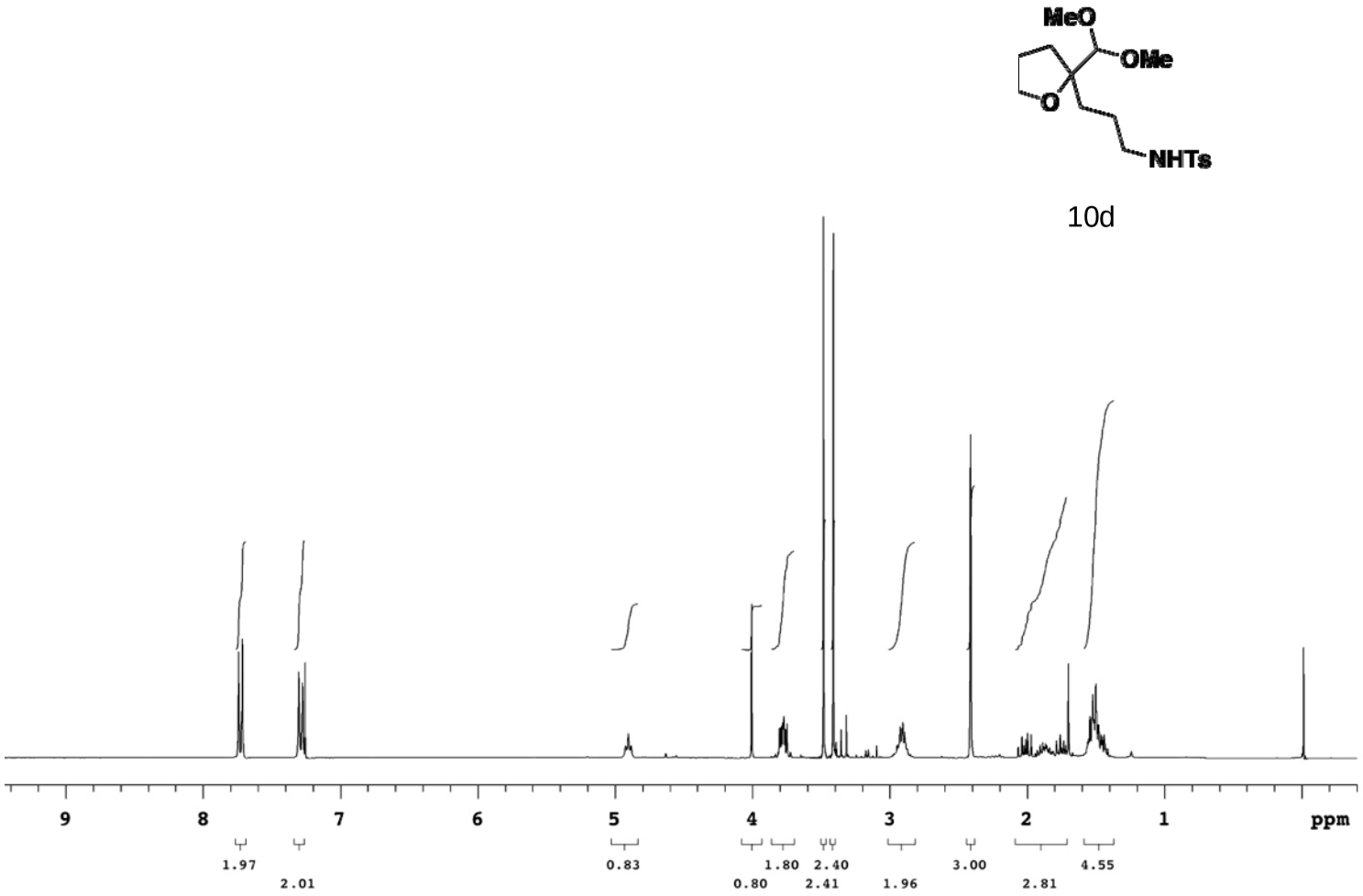
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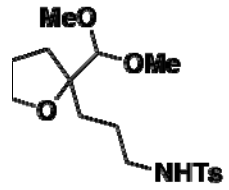

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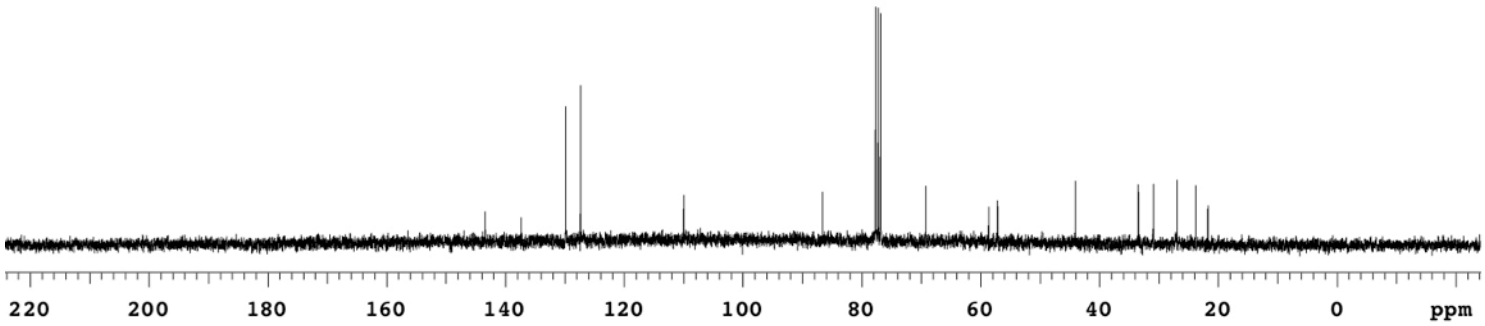

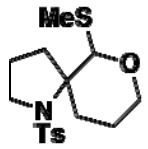

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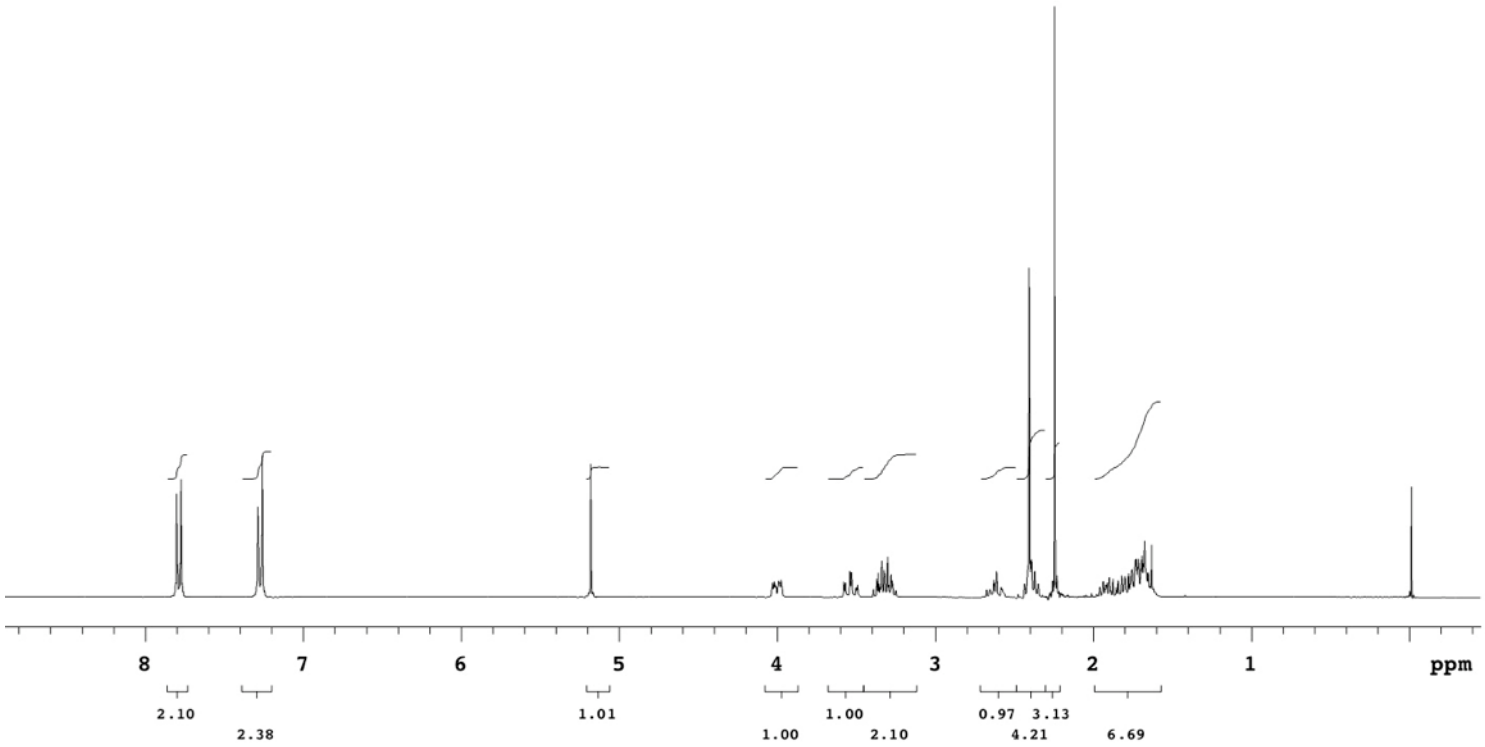

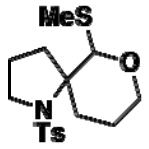

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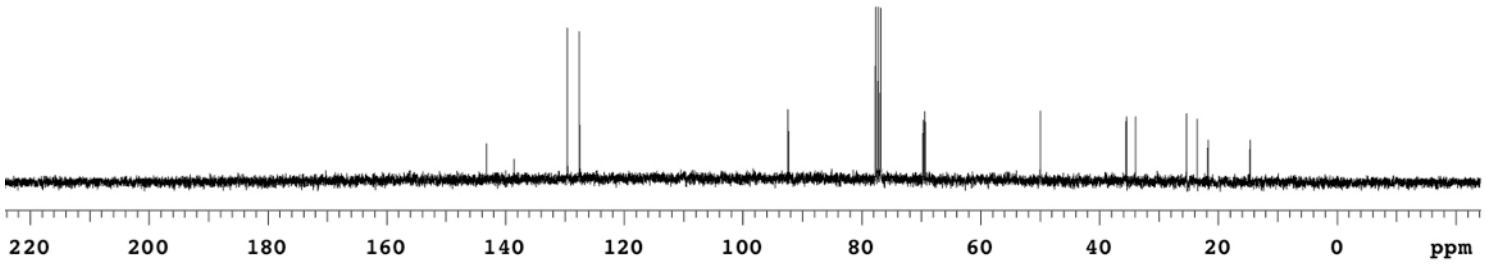

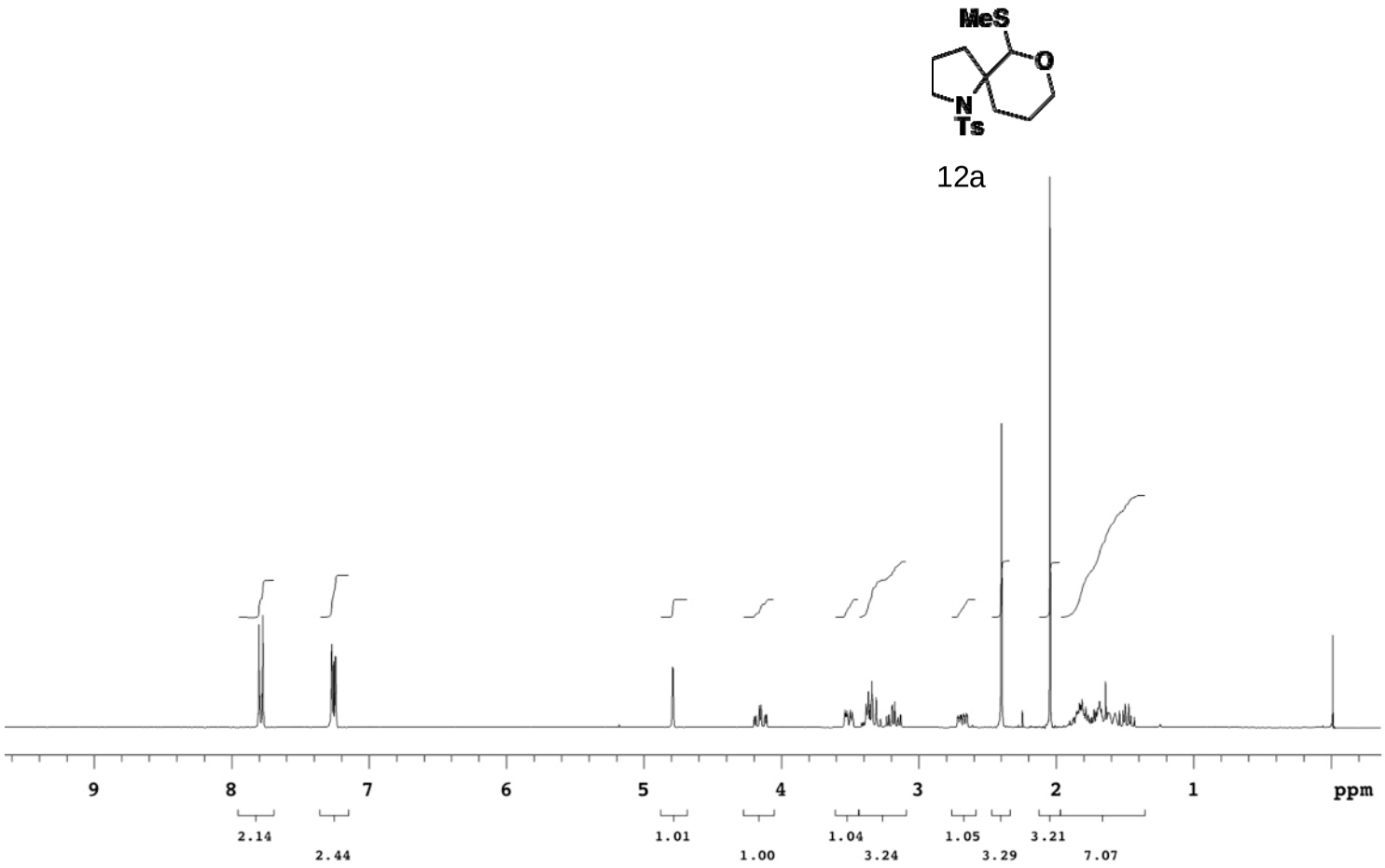
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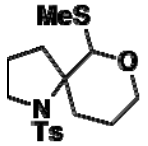

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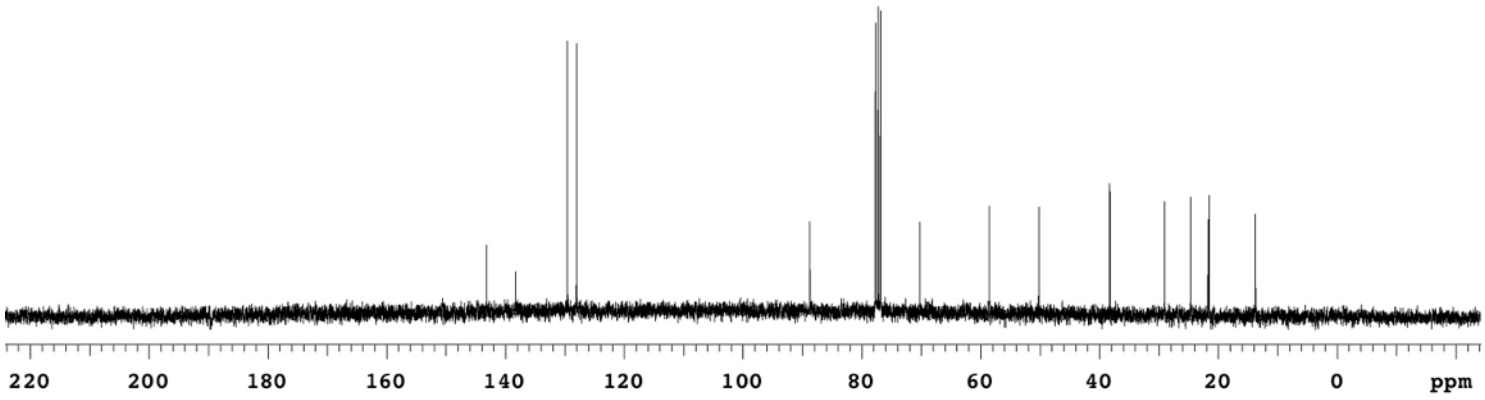
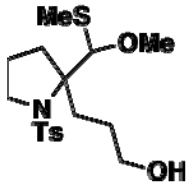
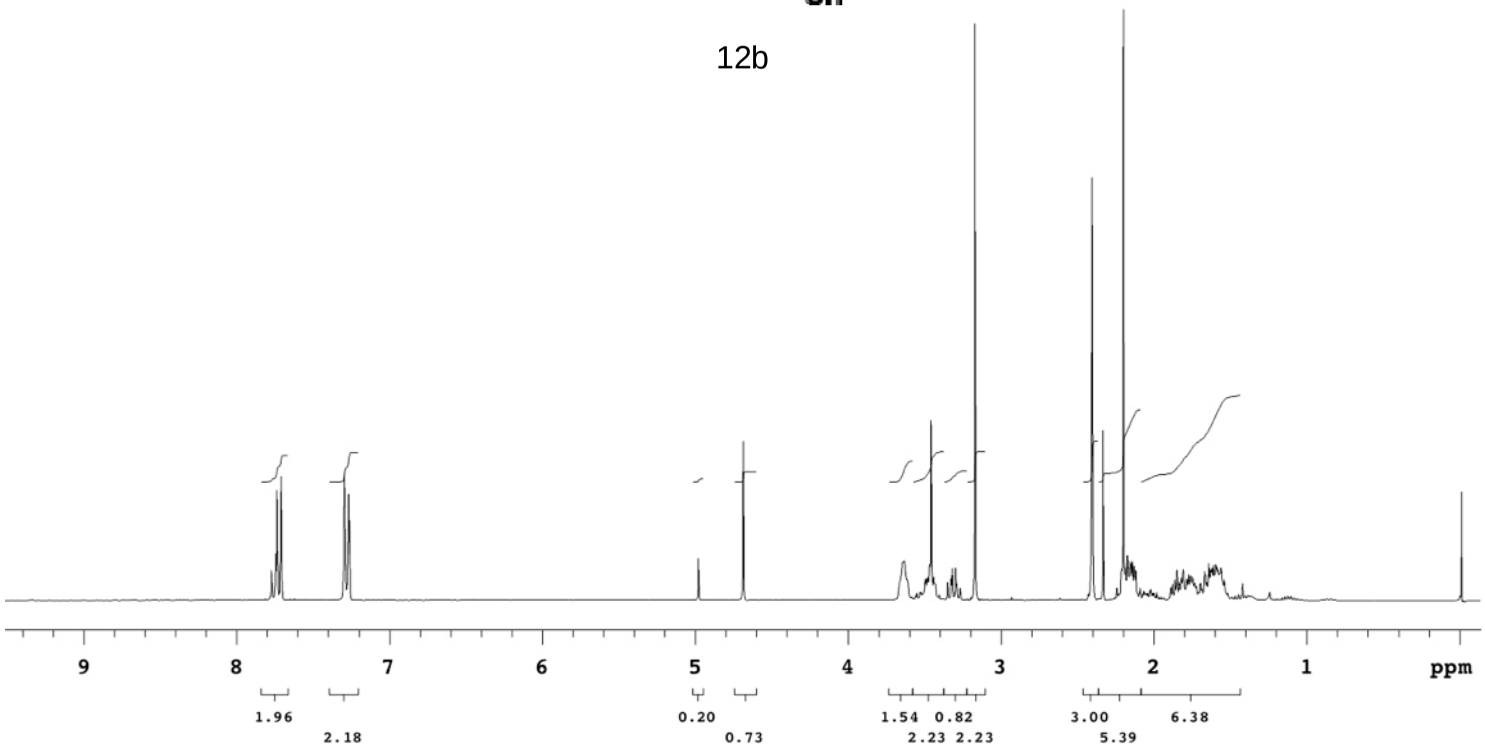



MeS
OMe
N
Ts
OH

12b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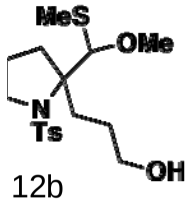

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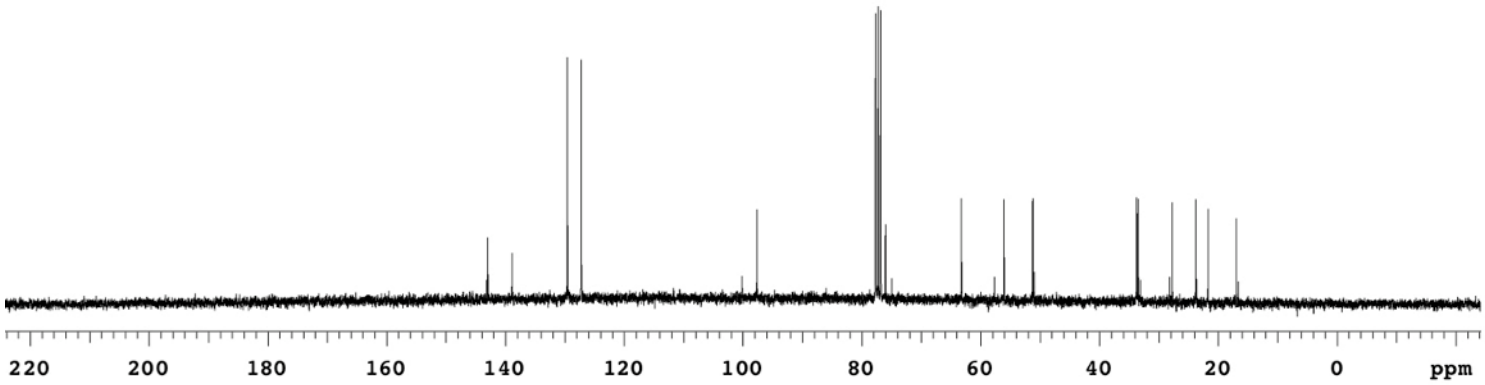

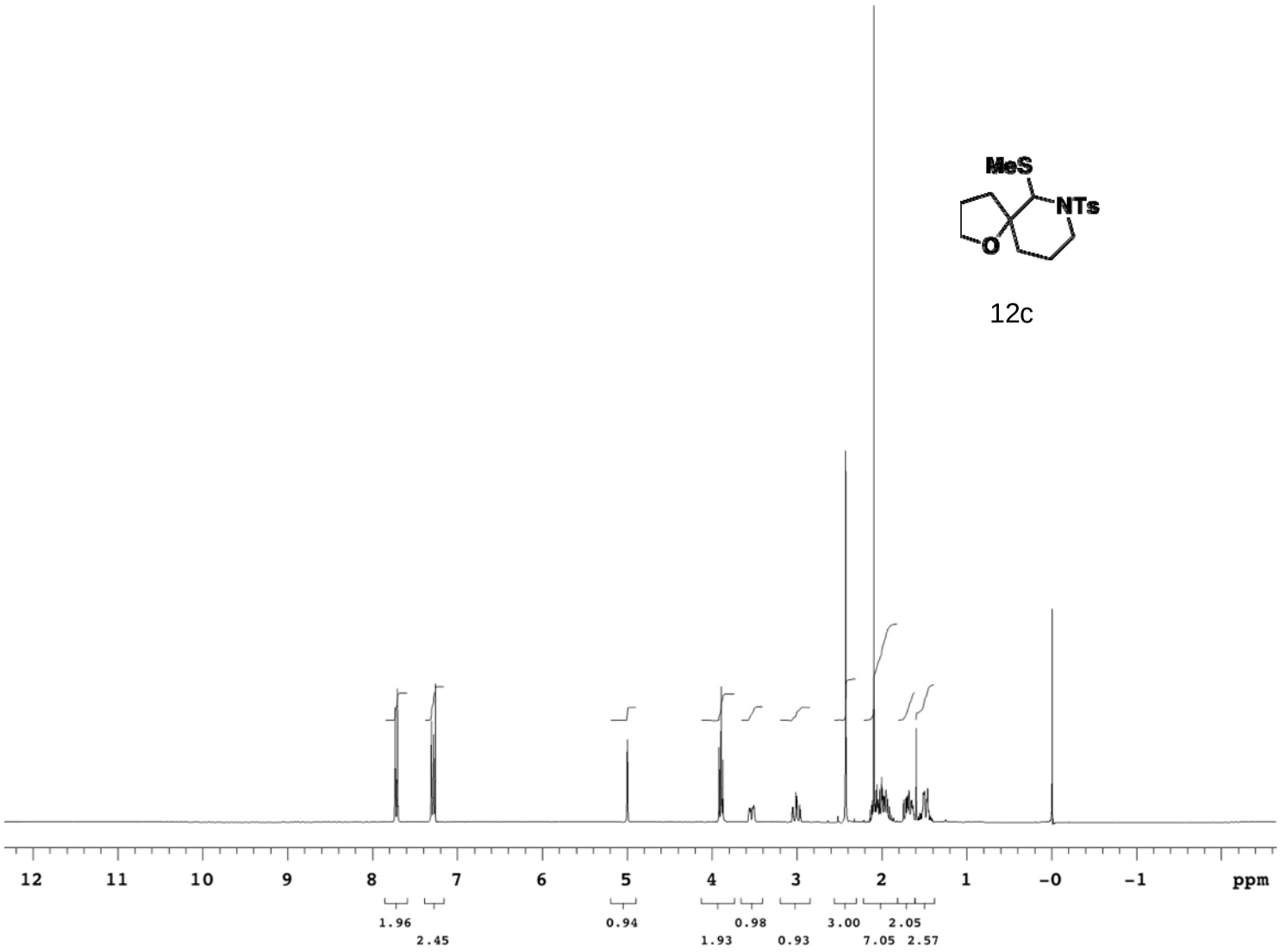
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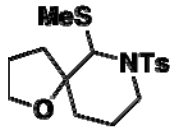

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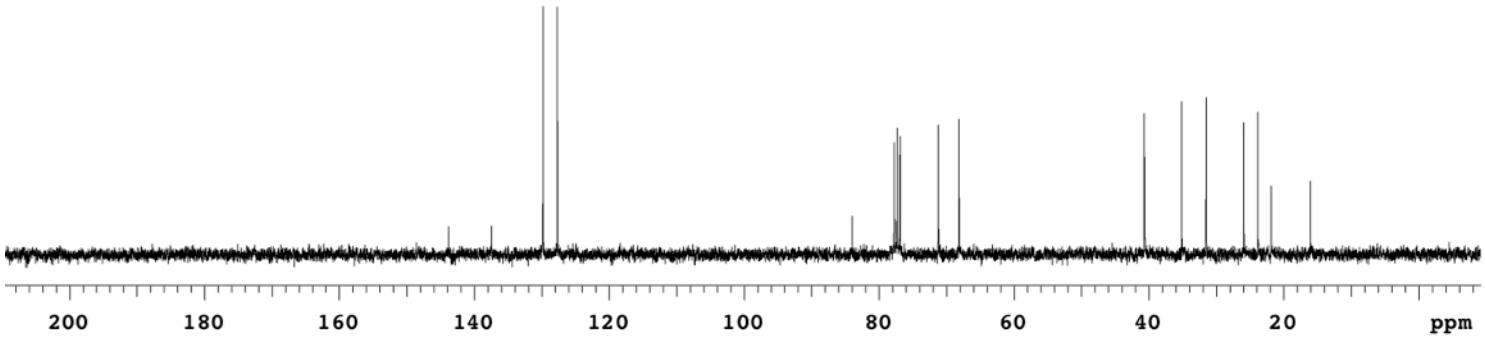
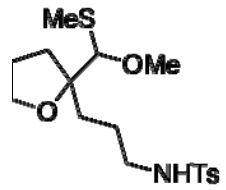
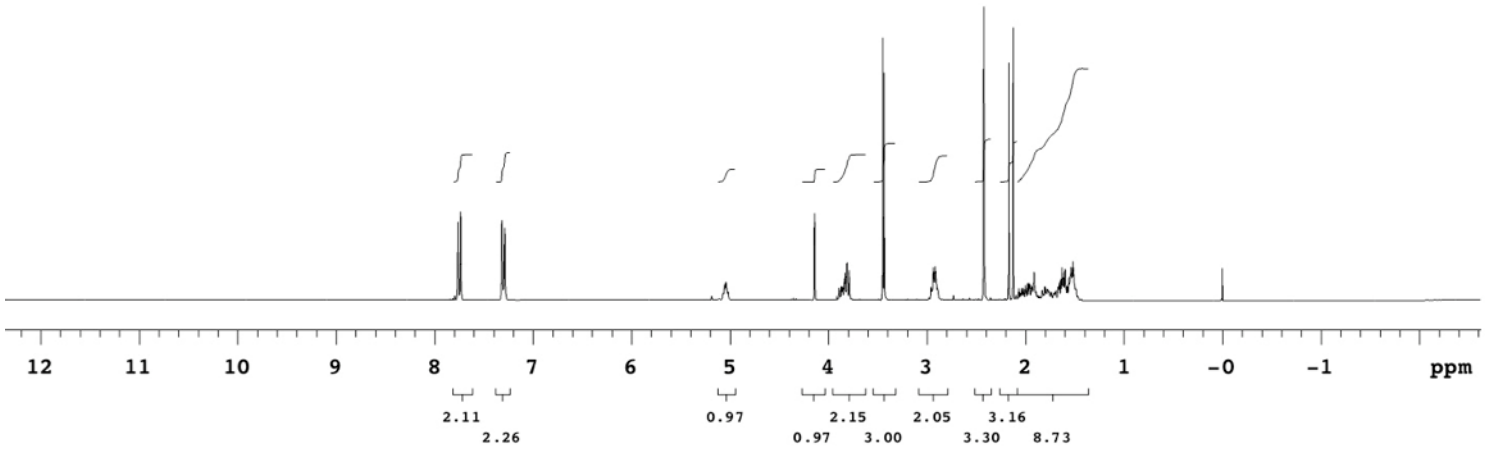



MeS
OMe
O
NHTs

12d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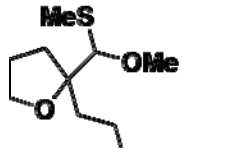
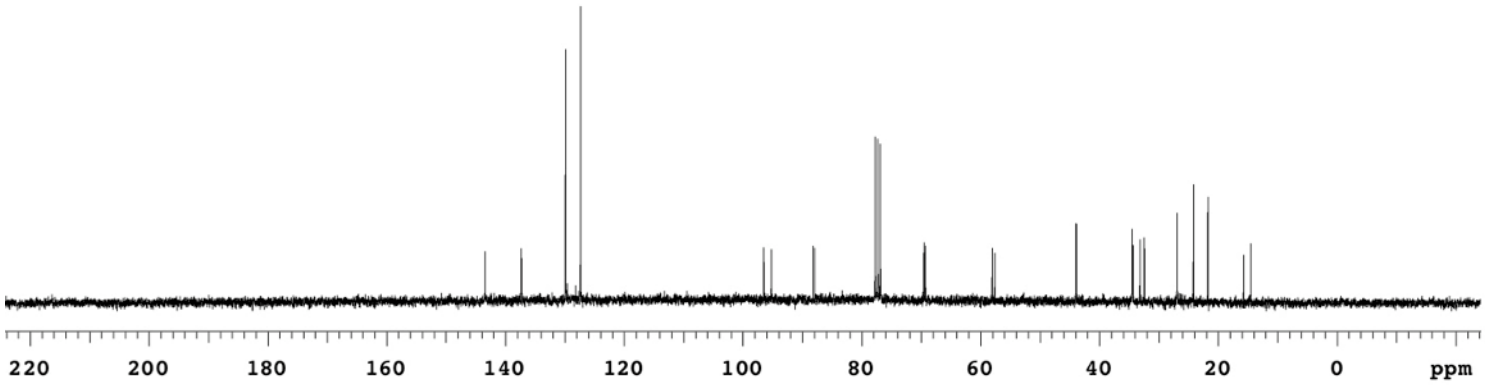
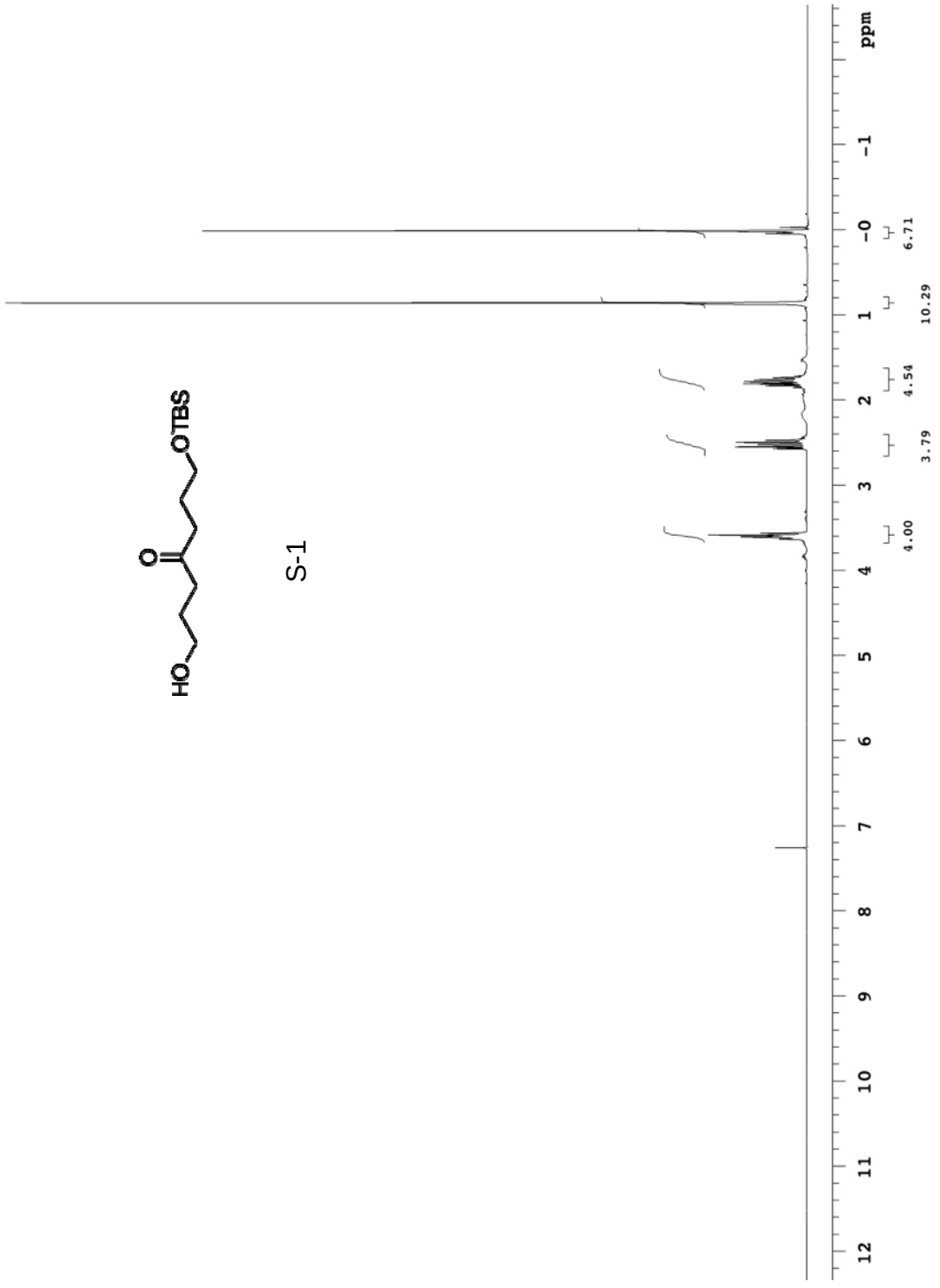
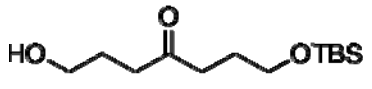


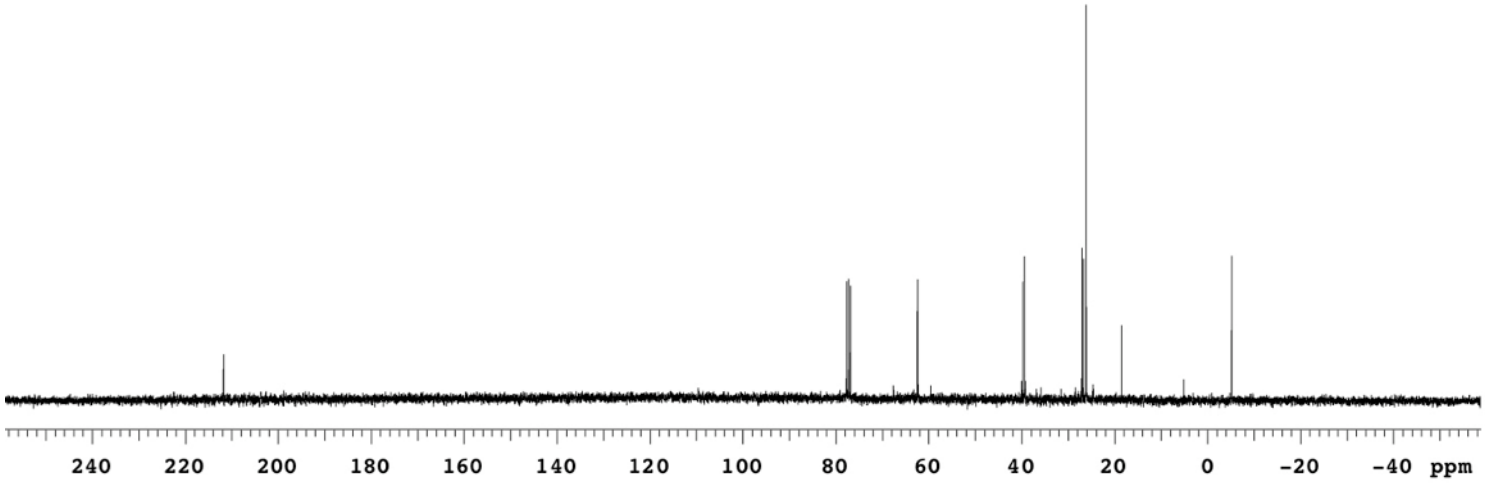
12d

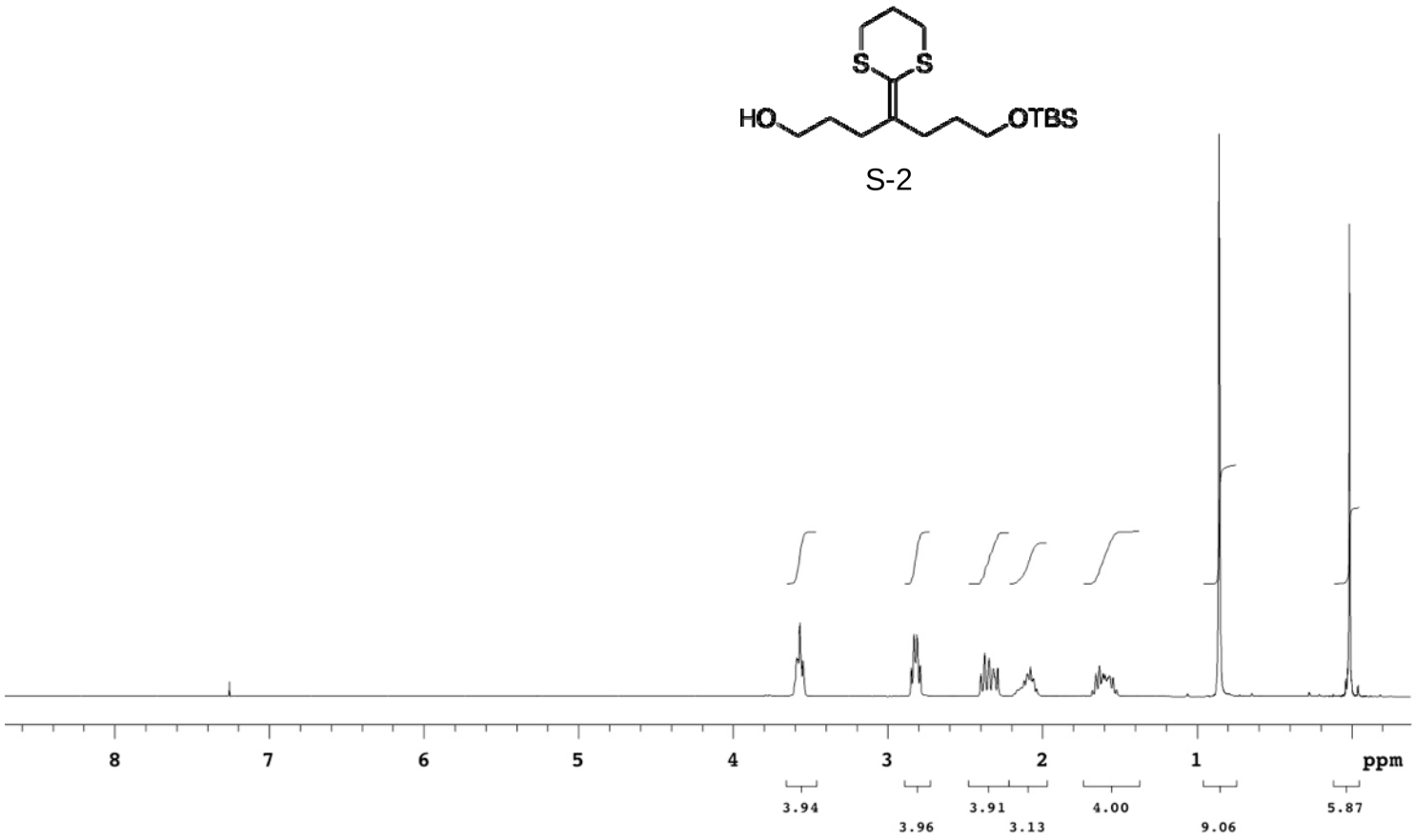


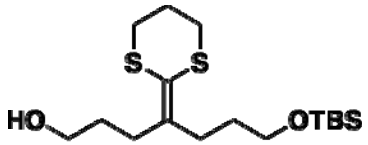

HO
O
OTBS
S-1
ppm
4.00
3.79
4.54
10.29
6.71



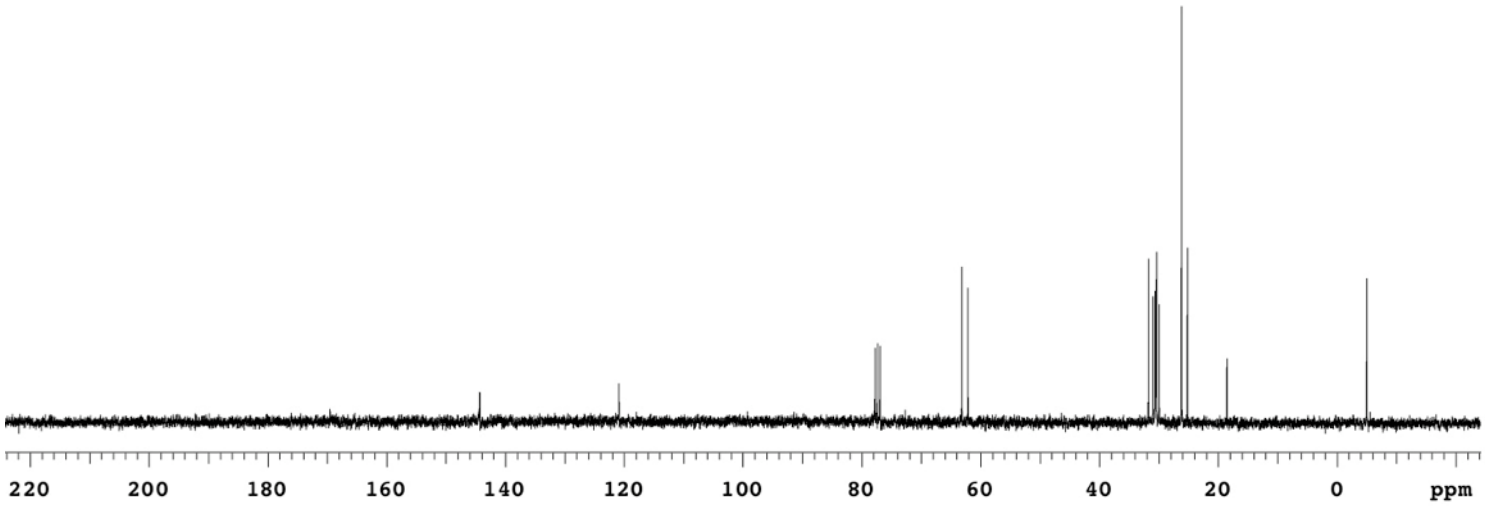
S-1

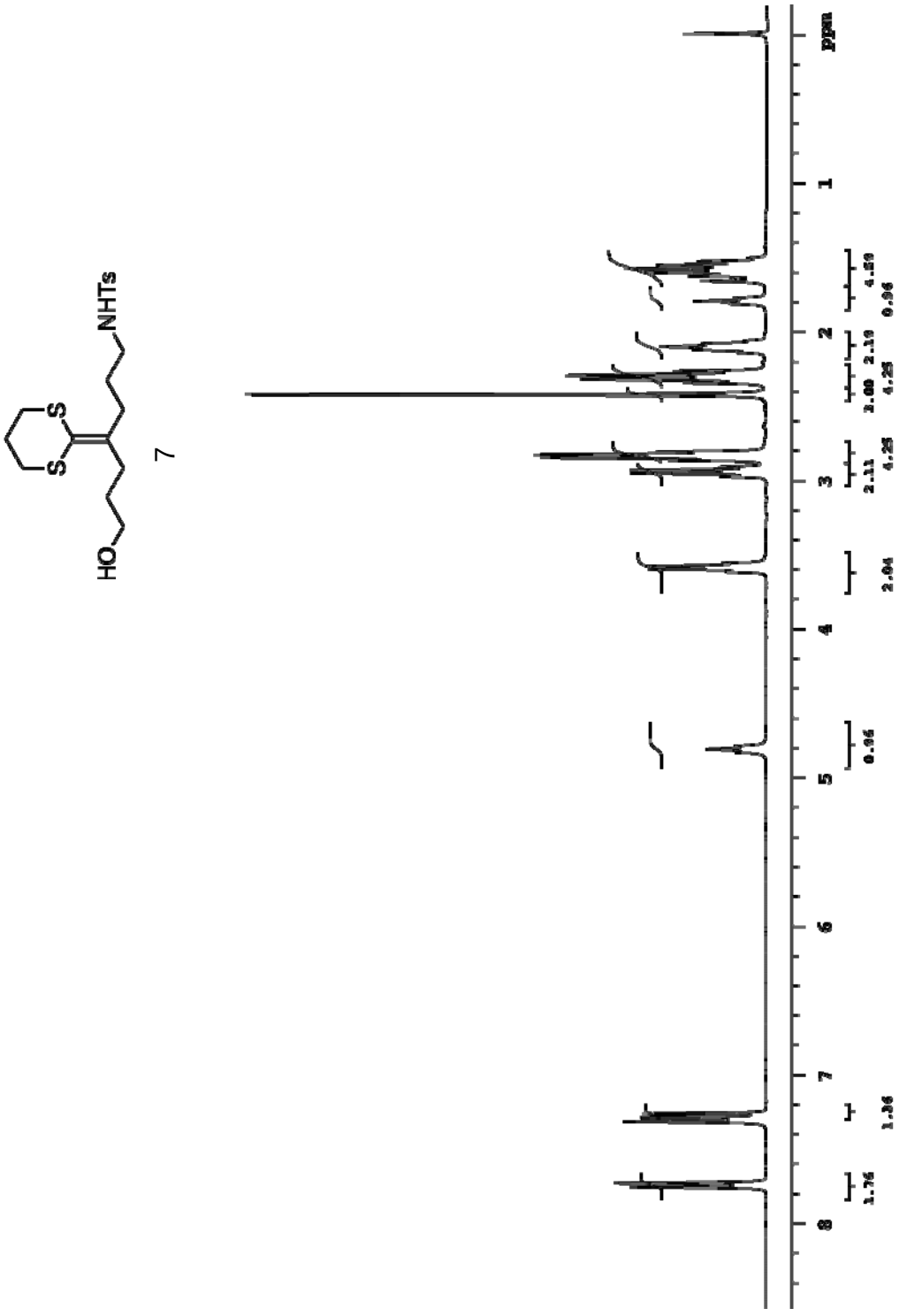



S
S
HO
OTBS
S-2
8
7
6
5
4
3
2
1
ppm
3.94
3.96
3.91
3.13
4.00
9.06
5.87

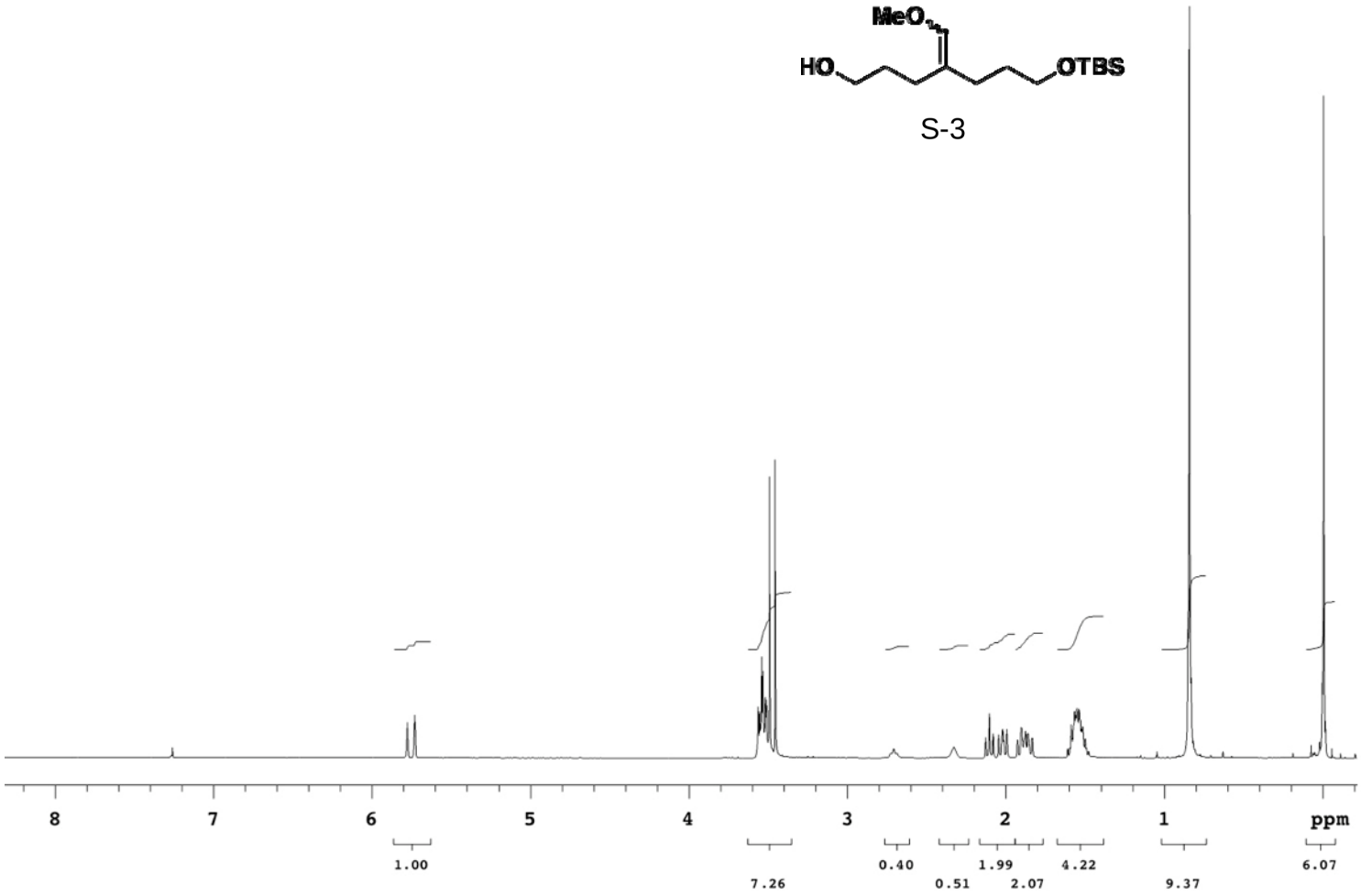


S-1

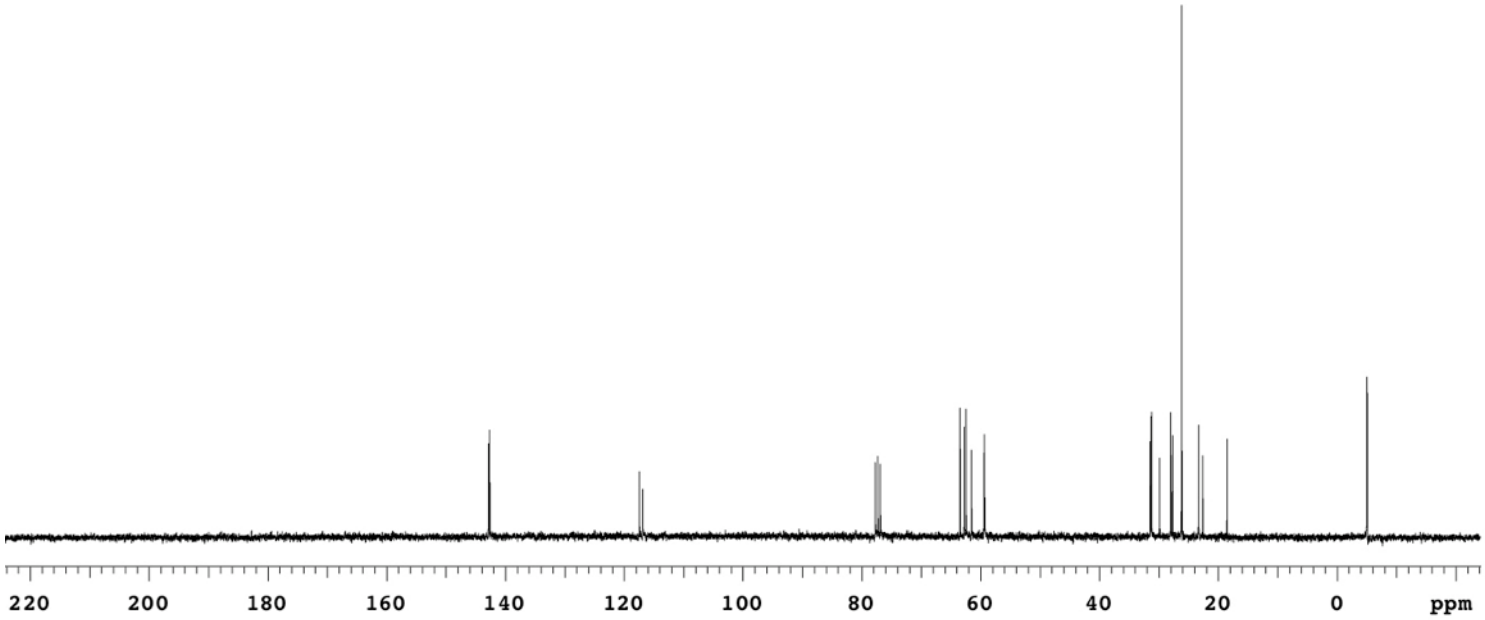


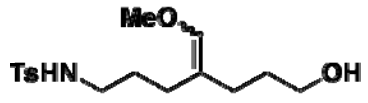

NHTs
S
S
HO
7
ppm
1
2
3
4
5
6
7
8
1.76
1.36
2.04
0.86
2.11
4.25
3.00
2.10
4.25
4.25
0.86

NHTs
S
S
HO
7
ppm
20
40
60
80
100
120
140
160
180
200

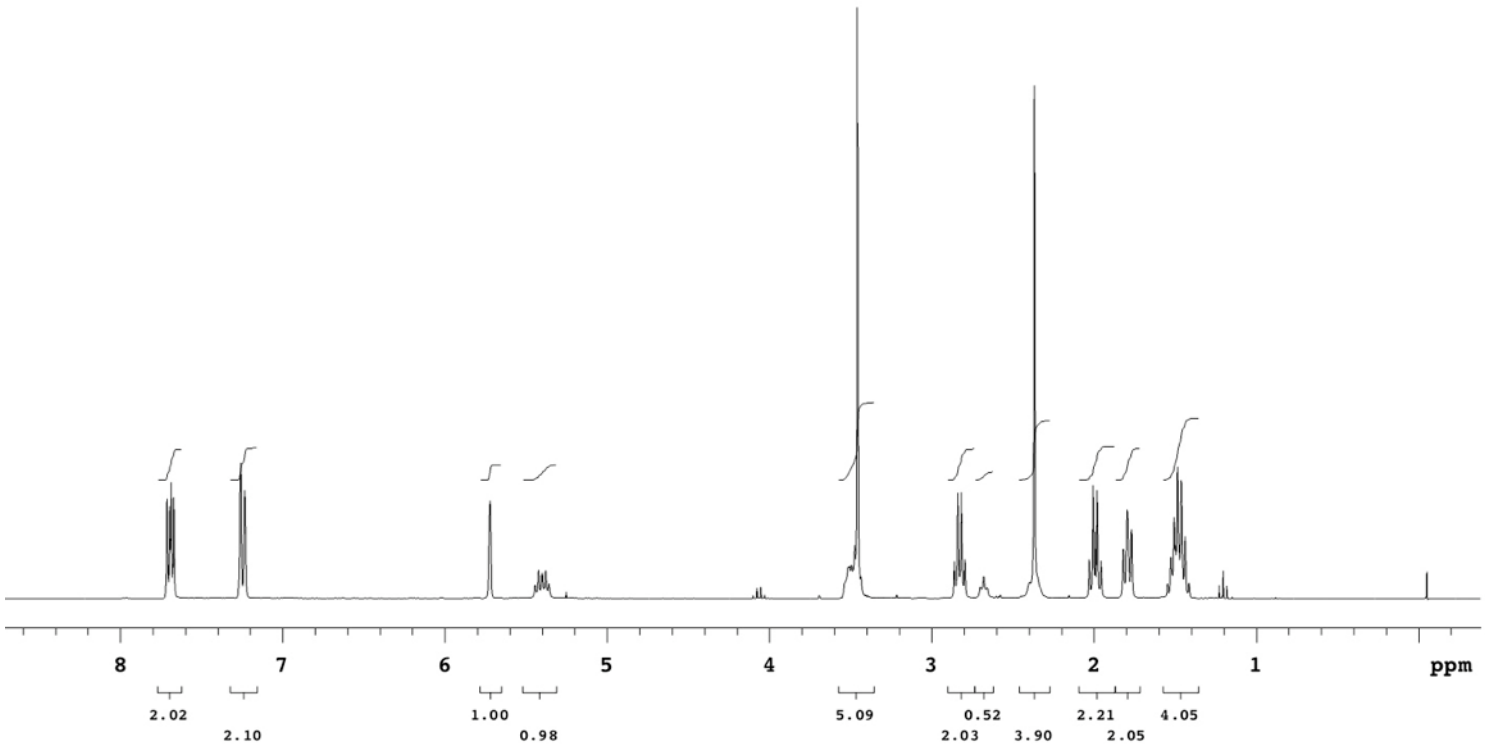

MeO
HO
OTBS
S-3
8
7
6
5
4
3
2
1
ppm
1.00
7.26
0.40
0.51
1.99
2.07
4.22
9.37
6.07

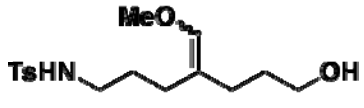
S-3



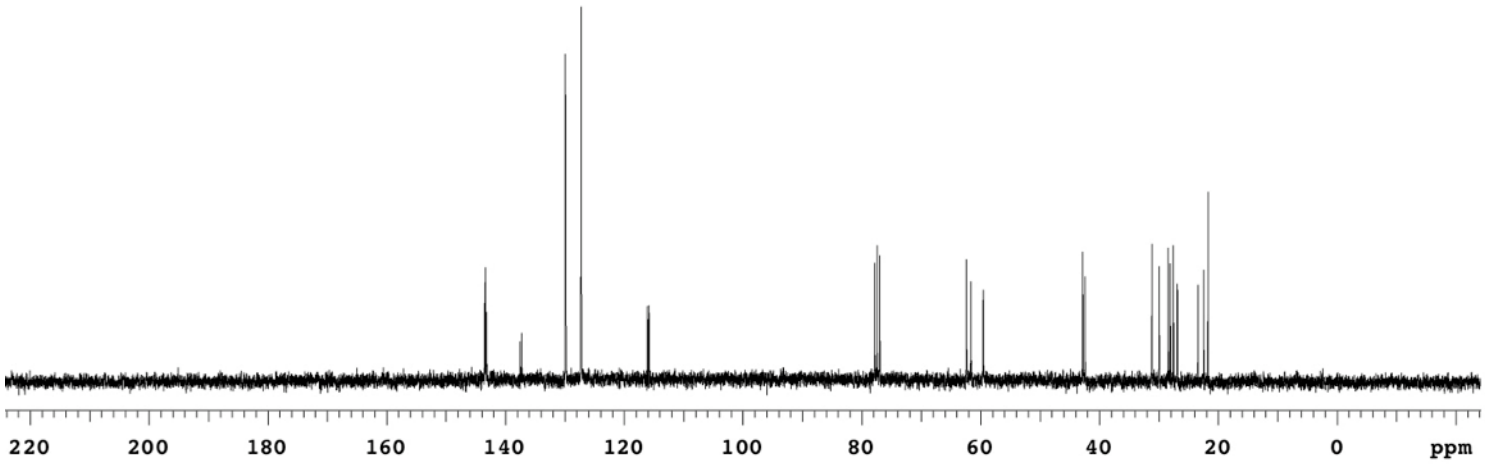

MeO
TsHN
OH

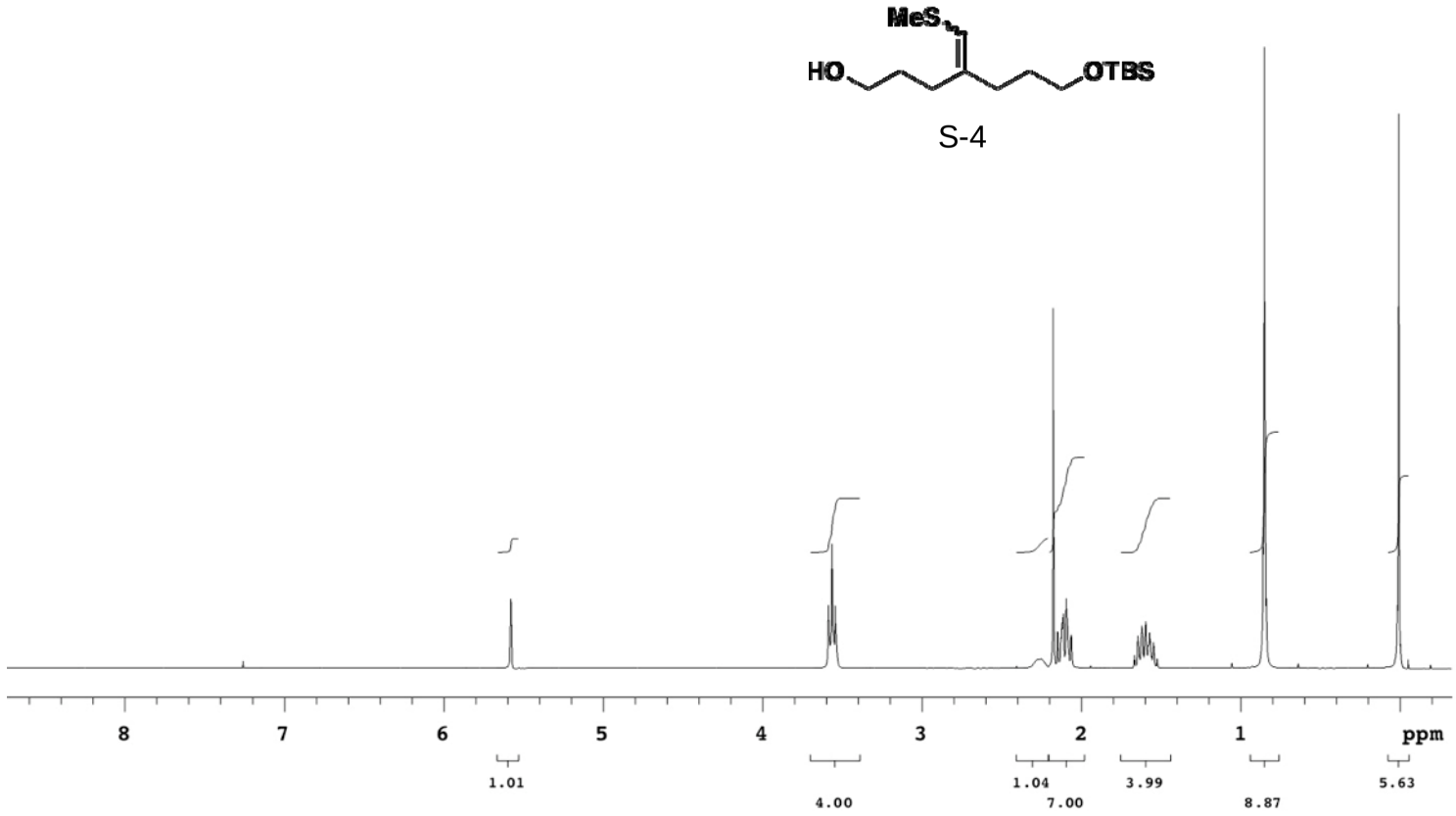
9

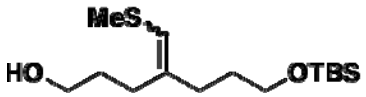

8
7
6
5
4
3
2
1
ppm
2.02
2.10
1.00
0.98
5.09
2.03
0.52
3.90
2.21
2.05
4.05



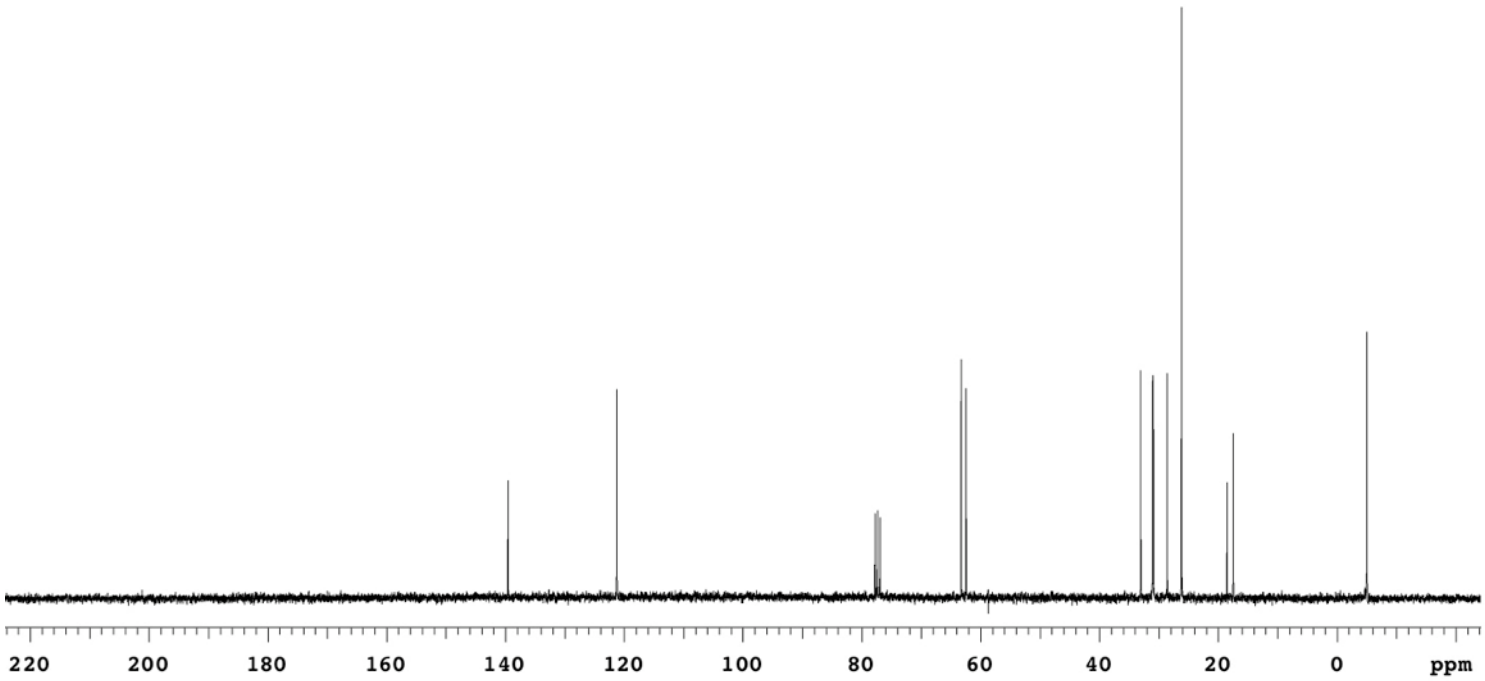
9

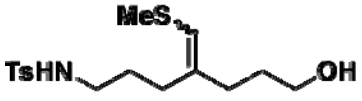



MeS
HO
OTBS
S-4
8
7
6
5
4
3
2
1
ppm
1.01
4.00
1.04
7.00
3.99
8.87
5.63

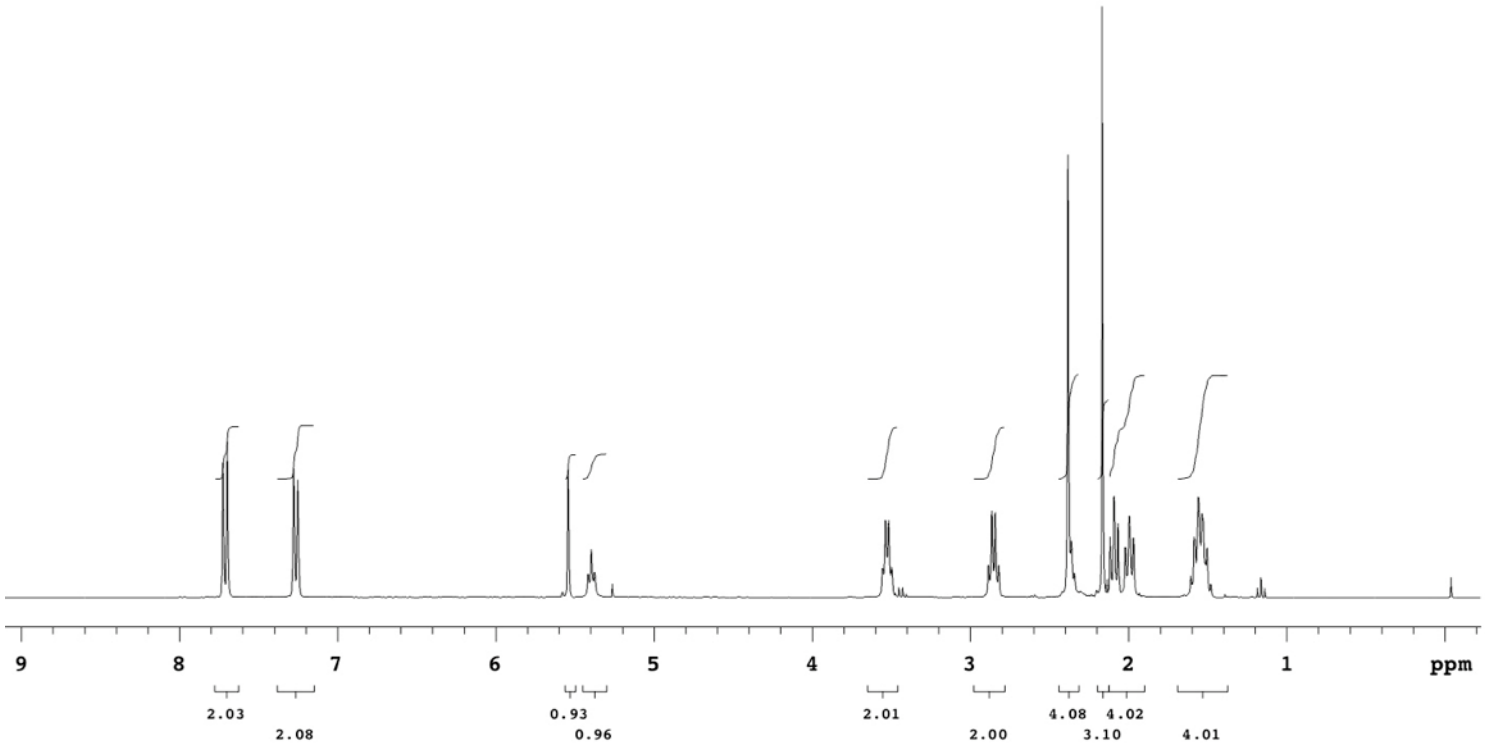


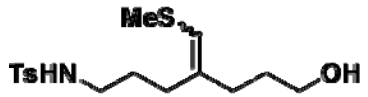
S-4




MeS
TsHN
OH

11


9
8
7
6
5
4
3
2
1
ppm
2.03
2.08
0.93
0.96
2.01
2.00
4.08
4.02
3.10
4.01



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