Electronic Supplementary Material (ESI) for ChemComm.

Gold(I) Operational in Synergistic Catalysis for the Intermolecular α-Addition Reaction of Aldehydes across Allenamides

Alberto Ballesteros,[a] Pablo Morán-Poladura,[a] and José M. González*[a]

[a] *Departamento de Química Orgánica e Inorgánica and Instituto Universitario de Química Organometálica "Enrique Moles"-Unidad Asociada al C.S.I.C., Universidad de Oviedo (Spain).*

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General Considerations:

All reactions were carried out using oven dried glassware under an atmosphere of nitrogen (99.99 %) or argon (99.999 %). The solvents used in column chromatography: hexane, ethyl acetate and dichloromethane were obtained from commercial suppliers and used without further distillation.

Acetonitrile and toluene used in the addition reactios were purified through a Innovative Technology System, provided with two one metre length columns, filled with activated alumina. Addition reactions were performed in a RR9803012 place Carousel Reaction StationTM from Radleys Discovery Technologies, equipped with gastight threaded caps with a valve, cooling reflux head system, and digital temperature controller.

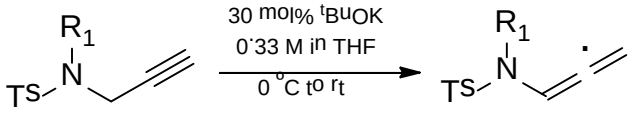
TLC was performed on aluminium-backed plates coated with silica gel 60 with F254 indicator (Merck), using UV light as a visualizing agent and phosphomolybdic acid in ethanol, and heat as developing agent. Flash chromatography was performed on silica gel 60 (230-400 mesh).

^{1}H NMR (300, 400, 600 MHz) and ^{13}C NMR (75.5, 100 MHz) spectra were measured in $CDCl_3$ at room temperature on a Bruker DPX-300, Bruker AV-300 MHz, Bruker AV-400 and Bruker AV-600 instruments, with $CDCl_3$ (δ = 7.26, ^{1}H NMR; δ = 77.16, ^{13}C NMR) as internal standard. Data are reported as follows: chemical shift, multiplicity (s: singlet, d: doublet, t: triplet, q: quartet, hex: hexet; br: broad, m: multiplet), coupling constants (J in Hz) and integration. Carbon multiplicities were assigned by DEPT techniques. 2D NMR experiments were recorded on a Bruker AV-400 MHz. Enantiomer ratios were determined by chiral HPLC analyses with a Vis-UV photodiode Array 2996 or 996 as detector, and compared with the authentic racemic products.

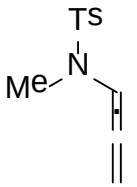
All common reagents and solvents were obtained from commercial suppliers and used without any further purification unless otherwise noted. The different aldehydes used were purchased from available commercial sources and were distilled under argon atmosphere before used. High-resolution mass spectra (HRMS) were determined by Universidad de Burgos and Universidad de Vigo (CACTI) with a VG AutoSpec M Mass Spectrometers and a microTOF focus (Bruker Daltonics, Bremen Germany) respectively.

Synthesis of N-Allenamides and Characterization Data

N-tosylallenamides (1a-1g) were prepared through base promoted isomerization reaction of *N*-tosyl propargylamines[1] according to previously reported procedures.[2]



To a solution of *N*-tosyl propargylamine (5.0 mmol) in 15 ml of anhydrous THF under N_2 atmosphere at 0°C was added in portions 169 mg of KO^{t}Bu (1,5 mmol). The reaction was allowed to stir at room temperature. After 12 h the mixture was diluted with 10 ml of Et_2O, and then filtrated over celite. The residue was washed with diethyl ether. The collected filtrate was concentrated in vacuo and the residue purified by flash column chromatography on silica gel (Hexane: Et_2O = 5:1), affording the *N*-tosylallenamides as white solids.



***N*,4-dimethyl-*N*-(propa-1,2-dien-1-yl)benzenesulfonamid (1a).** White solid (88%). **^{1}H-NMR** (400MHz, $CDCl_3$): δ (ppm) = 7.68 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 6.90 (t, *J* = 6.2 Hz, 1H), 5.30 (d, *J* = 6.2 Hz, 2H), 2.72 (s, 3H), 2.44 (s, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 201.5 (C), 143.8 (C), 133.4 (C), 129.7 (2 CH), 129.6 (2 CH), 101.7 (CH), 87.7 (CH_2), 33.2 (CH_3), 21.6 (CH_3). **HRMS** (EI): calcd for $C_{11}H_{13}NO_2S$: 223.0667. Found: 223.0650.

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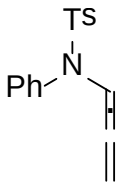
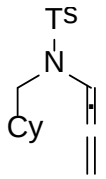
[1] *N*-tosylpropargylamines were prepared according to previous procedure described in the literature. See for instance: (a) J. Barluenga, M. Trincado, M. Marco-Arias, A. Ballesteros, E. Rubio, J. M. González. *Chem. Commun.* **2005**, 2008; (b) S. Suárez-Pantiga, D. Palomas, E. Rubio, J. M. González, *Angew. Chem. Int. Ed.* **2009**.

[2] a) A. González-Gómez, L. Añorbe, A. Poblador, G. Domínguez, J. Pérez-Castells. *Eur. J. Org. Chem*. **2008**, 1370; b) X.-X. Li, L.-L. Zhu, W. Zhou, Z. Chen, *Org. Lett.* **2012**, *14*, 436-439; c) S. Suárez-Pantiga, C. Hernández-Díaz, M. Piedrafita, E. Rubio, J. M. González, *Adv. Synth. Catal.* **2012**, *354*, 1651-1657; d) H. Xiong, M. R. Tracey, T. P. Grebe, J. A. Mulder, R. P. Hsung, P. Wipf, J. Smotryski, *Org. Synth.* **2005**, 81, 147; e) S. Suárez-Pantiga, C. Hernández-Díaz, E. Rubio, J. M. González, *Angew. Chem. Int. Ed.* **2012**, *51,* 11552-11555.

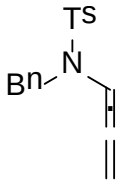


4-methyl-*N*-phenyl-*N*-(propa-1,2-dien-1-yl)benzenesulfonamide (1b). White solid, (94%). **^{1}H-NMR** (400MHz, $CDCl_3$): δ (ppm) = 7.56 (d, *J* = 8.1 Hz, 2H), 7.55 - 7.27 (m, 5H), 7.12 (t, *J* = 6.2 Hz, 1H), 7.01 (m, 2H), 5.03 (d, *J* = 6.2 Hz, 2H), 2.45 (s, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 201.1 (C), 143.9 (C), 137.2 (C), 135.3 (C), 129.6 (2 CH), 129.5 (2 CH), 128.7 (2 CH), 128.6 (2 CH), 127.7 (CH), 102.4 (CH), 87.5 (CH_2), 21.6 (CH_3). **HRMS** (EI): calcd for $C_{16}H_{15}NO_2S$: 285.0824. Found: 285.0819.



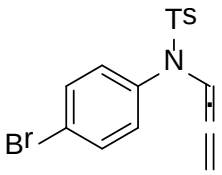
***N*-(cyclohexylmethyl)-4-methyl-*N*-(propa-1,2-dien-1-yl)benzenesulfonamide (1c).** White solid (59%). **^{1}H-NMR** (300MHz, $CDCl_3$): δ (ppm) = 7.67 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 6.82 (t, *J* = 6.2 Hz, 1H), 5.26 (d, *J* = 6.2 Hz, 2H), 2.87 (d, *J* = 7.1 Hz, 2H), 2.43 (s, 3H), 1.81 – 1.55 (m, 6H), 1.31 – 1.09 (m, 3H), 0.98 – 0.81 (m, 2H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 202.0 (C), 143.6 (C), 135.4 (C), 129.8 (2 CH), 127.3 (2 CH), 100.9 (CH), 87.6 (CH_2), 52.7 (CH_2), 36.2 (CH), 30.7 (2 CH_2), 26.6 (CH_2), 26.0 (2 CH_2), 21.7 (CH_3).

HRMS (EI): calcd for $C_{17}H_{23}NO_2S$: 305.1450. Found: 305.1453.

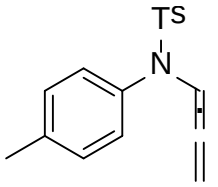


***N*-benzyl-4-methyl-*N*-(propa-1,2-dien-1-yl)benzenesulfonamide (1d).** White solid (88%). **^{1}H-NMR** (300MHz, $CDCl_3$): δ (ppm) = 7.75 (d, *J* = 8.3 Hz, 2H), 7.40 – 7.22 (m, 7H), 6.86 (t, *J* = 6.2 Hz, 1H), 5.17 (d, *J* = 6.2 Hz, 2H), 4.33 (s, 2H), 2.47 (s, 3H).

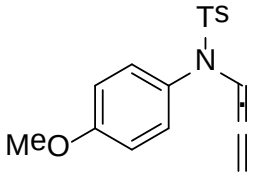
^{13}C-NMR (75 MHz, $CDCl_3$): δ (ppm) = 202.2 (C), 143.8 (C), 136.2 (C), 135.3 (C), 129.8 (2 CH), 128.3 (2 CH), 127.9 (2 CH), 127.4 (CH), 127.2 (2 CH), 100.1 (CH), 88.1 (CH_2), 50.0 (CH_2), 21.6 (CH_3). **HRMS** (EI): calcd for $C_{17}H_{17}NO_2S$: 299.0980. Found: 299.0980.



***N*-(4-bromophenyl)-4-methyl-*N*-(propa-1,2-dien-1-yl)benzenesulfonamide (1e).** Light yellow solid (91%). **^{1}H-NMR** (400MHz, $CDCl_3$): δ (ppm) = 7.56 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.7 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.10 (t, *J* = 6.3 Hz, 1H), 6.89 (d, *J* = 8.7 Hz, 2H), 5.07 (d, *J* = 6.3 Hz, 2H), 2.47 (s, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 202.3 (C), 143.8 (C), 137.9 (C), 135.1 (C), 132.6 (C), 129.5 (2 CH), 129.2 (2 CH), 124.6 (2 CH), 120.1 (2 CH), 102.4 (CH), 87.3 (CH_2), 21.3 (CH_3). **HRMS** (EI): calcd for $C_{16}H_{14}BrNO_2S$: 362.9929. Found: 362.9931.



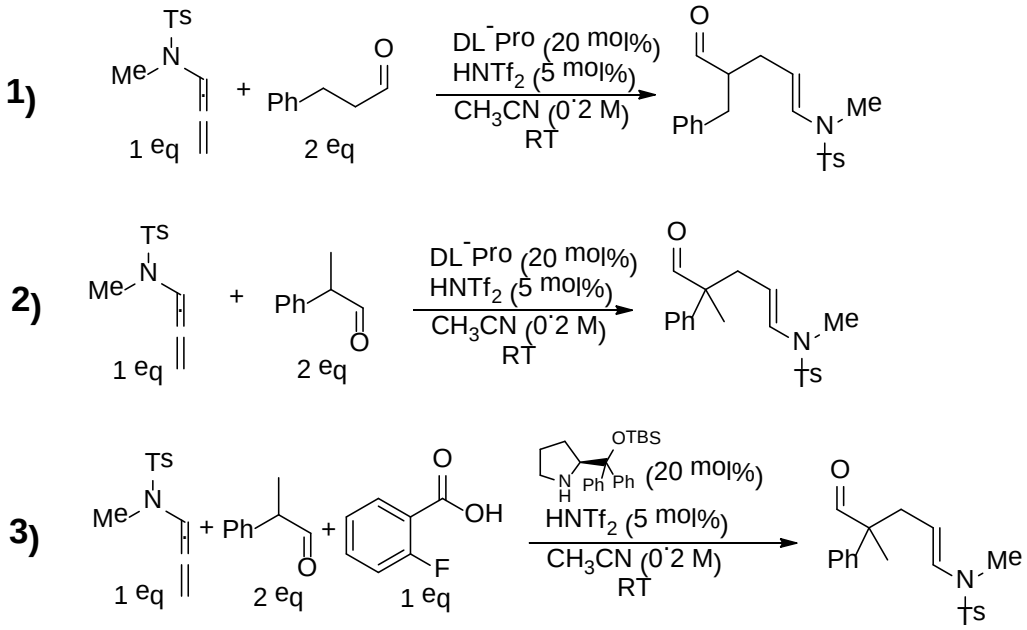
4-methyl-*N*-(propa-1,2-dien-1-yl)-*N*-(p-tolyl)benzenesulfonamide (1f). White solid (92%). **^{1}H-NMR** (400MHz, $CDCl_3$): δ (ppm) = 7.57 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.3 Hz, 2H), 7.13 (t, *J* = 6.2 Hz, 1H), 6.88 (d, *J* = 8.3 Hz, 2H), 5.06 (d, *J* = 6.2 Hz, 2H), 2.47 (s, 3H), 2.36 (s, 3H).**^{13}C-NMR** (100 MHz, $CDCl_3$): δ (ppm) = 201.1 (C), 144.2 (C), 138.9 (C), 135.3 (C), 134.6 (C), 129.6 (2 CH), 129.4 (2 CH), 129.2 (2 CH), 127.6 (2 CH), 102.4 (CH), 87.1 (CH_2), 21.3 (CH_3), 20.9 (CH_3). **HRMS** (EI): Calcd for $C_{17}H_{17}NO_2S$: 299.0980. Found: 299.0969.



***N*-(4-methoxyphenyl)-4-methyl-*N*-(propa-1,2-dien-1-yl)benzenesulfonamide (1g).** Yellow solid (74%). **^{1}H-NMR** (300MHz, $CDCl_3$): δ (ppm) = 7.56 (d, *J* = 6.6 Hz, 2H), 7.28 (d, *J* = 7.7 Hz, 2H), 7.13 (t, *J* = 6.0 Hz, 1H), 6.88 – 66.93 (m, 2H), 6.76 – 6.81 (m, 2H), 5.04 (d, *J* = 6.0 Hz, 2H), 3.80 (s, 3H), 2.45 (s, 3H).**^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 200.9 (C), 159.5 (C), 143.8 (C), 135.1 (C), 130.7 (C), 129.5 (2 CH), 129.5 (2 CH), 127.7 (2 CH), 113.8 (2 CH), 102.7 (CH), 87.5 (CH_2), 55.3 (CH_3), 21.6 (CH_3). **HRMS** (EI): Calcd for $C_{17}H_{17}NO_3S$: 315.0929. Found: 315.0927.

Control experiments with $HNTf_2$

The following control experiments with $HNTf_2$ were performed:



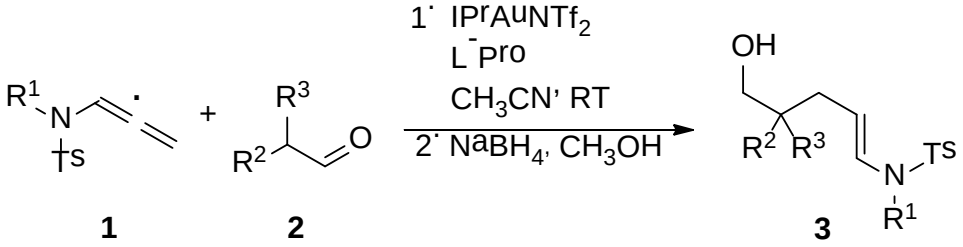
Experiment 1 yielded 11 % of the reaction product. Only 60 % of conversion was achieved, determined by internal standard.

Experiment 2 yielded 51 % of the isolated product.

Experiment 3 yielded 18% of the reaction product, determined by internal standard.

General Procedure for the addition reaction

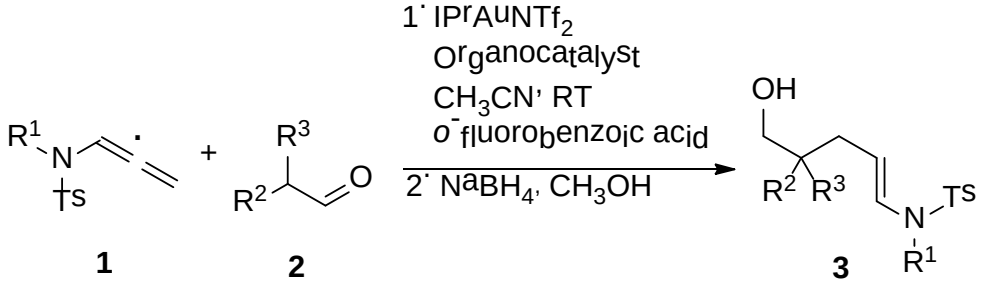
- *Method A:*



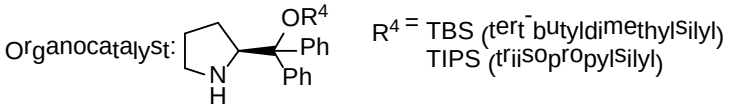
1 **2** **3**

First, L-Pro (4.6 mg, 0.04 mmol) was suspended in 1 mL of acetonitrile. Then aldehyde 2 was added (0.4 mmol, unless otherwise noted).The mixture was stirred for 10 minutes. After that, $IPrAuNTf_2$ (8.7 mg, 0.01 mmol) and allenamide 1 (0.2 mmol) were added. The reaction was monitored until all the allenamide is consumed (TLC or GCMS). The reaction is stopped by the addition of PPh_3 (5.2 mg, 0.02 mmol). Then 0.5 mL of MeOH and $NaBH_4$ (15.2 mg, 0.4 mmol) were added and the reaction was stirred for 20 minutes. The mixture was concentrated in vacuo and purified by flash chromatography on silica gel (4 Hex: 2 DCM: 1 AcOEt). All the reported yields are isolated yields.

- *Method B*



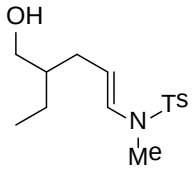
1 **2** **3**



First, the organocatalyst (0.04 mmol) was dissolved in 1 mL of acetonitrile. Then 2-fluorobenzoic acid (28 mg, 0.2 mmol), $IPrAuNTf_2$ (8.7 mg, 0.01 mmol), allenamide 1 (0.2 mmol) and aldehyde 2 (0.4 mmol) were added. The reaction was monitored until all the allenamide is consumed (TLC or GCMS). The reaction is stopped by the addition of PPh_3 (5.2 mg, 0.02 mmol). Then 0.5 mL of MeOH and $NaBH_4$ (15.2 mg, 0.4 mmol) were added and the reaction was stirred

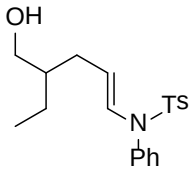
for 20 minutes. The mixture was concentrated in vacuo and purified by flash chromatography on silica gel (4 Hex: 2 DCM: 1 AcOEt). All the reported yields are isolated yields.

Characterization data:



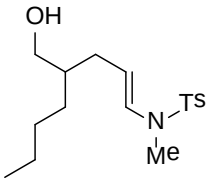
(*E*)-*N*-(4-(hydroxymethyl)hex-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3a) was prepared following *method A*; but 1 mmol (5 eq) of aldehyde was used. The reaction was stirred for 2h 45 min. Colorless oil (63 %).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.63 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.73 (d, *J* = 14.0 Hz, 1H), 4.68 (dt, *J* = 14.4, 7.5 Hz, 1H), 3.50 (s, 2H), 2.82 (s, 3H), 2.42 (s, 3H), 2.08 (ddd, *J* = 7.3, 5.4, 2.2 Hz, 2H), 1.47 – 1.38 (m, 1H), 1.36 – 1.13 (m, 3H), 0.89 (t, *J* = 7.4 Hz, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.7 (C), 134.4 (C), 129.7 (2 CH), 128.8 (CH), 127.1 (2 CH), 109.4 (CH), 64.8 (CH_2), 42.6 (CH), 32.3 (CH_3), 31.2 (CH_2), 22.9 (CH_2), 21.5 (CH_3), 11.2 (CH_3). **HRMS** (ESI): calcd for $C_{15}H_{24}NO_3S$: 298.1471. Found: 298.1474



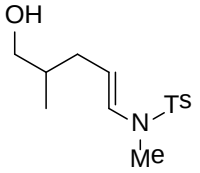
(*E*)-*N*-(4-(hydroxymethyl)hex-1-en-1-yl)-4-methyl-*N*-phenylbenzenesulfonamide (3b) was prepared following *method A*. The reaction was stirred for 2h 45 min. Colorless oil (32 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.58 (d, *J* = 8.2 Hz, 2H), 7.43 – 7.24 (m, 5H), 7.04 – 6.93 (m, 3H), 4.40 (dt, *J* = 14.5, 7.6 Hz, 1H), 3.46 (d, *J* = 4.9 Hz, 2H), 2.46 (s, 3H), 2.04 (t, *J* = 6.9 Hz, 2H), 1.63 (bs, 1H), 1.47 – 1.14 (m, 3H), 0.87 (t, *J* = 7.2 Hz, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.8 (C), 137.0 (C), 135.9 (C), 130.0 (2 CH), 129.8 (CH), 129.5 (2 CH), 129.4 (2 CH), 128.8 (CH), 127.5 (2 CH), 110.8 (CH), 64.9 (CH_2), 42.5 (CH), 31.0 (CH_2), 23.1 (CH_2), 21.6 (CH_3), 11.2 (CH_3). **HRMS** (ESI): Calcd for $C_{20}H_{26}NO_3S$: 360.1628. Found: 360.1634.



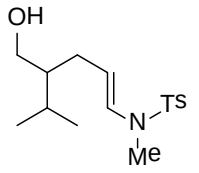
(*E*)-*N*-(4-(hydroxymethyl)oct-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3c) was prepared following *method A*; but 1 mmol (5 eq) of aldehyde was used. The reaction was stirred for 4h 35 min. Colorless oil (70 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.64 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 6.75 (dd, *J* = 14.1, 1.3 Hz, 1H), 4.70 (dt, *J* = 14.1, 7.5 Hz, 1H), 3.51 (t, *J* = 5.7 Hz, 2H), 2.85 (s, 3H), 2.43 (s, 3H), 2.12 – 2.10 (m, 2H), 1.60 – 1.42 (m, 1H), 1.55 – 1.04 (m, 7H), 1.00 – 0.79 (m, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.7 (C), 134.4 (C), 129.7 (2 CH), 128.8 (CH), 127.0 (2 CH), 109.5 (CH), 65.2 (CH_2), 41.0 (CH), 32.3 (CH_3), 31.6 (CH_2), 30.1 (CH_2), 29.1 (CH_2), 23.0 (CH_2), 21.5 (CH_3), 14.1 (CH_3). **HRMS** (ESI): calcd for $C_{17}H_{28}NO_3S$: 326.1784. Found: 326.1780



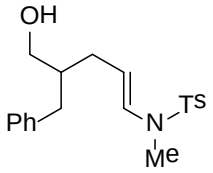
(*E*)-*N*-(5-hydroxy-4-methylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3d) was prepared following *method A*. The reaction was stirred for 4h. Colorless oil (52 %).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.62 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 6.72 (d, *J* = 14.0 Hz, 1H), 4.67 (dt, *J* = 14.5, 7.5 Hz, 1H), 3.49 – 3.38 (m, 2H), 2.82 (s, 3H), 2.41 (s, 3H), 2.15 (dt, *J* = 14.1, 6.8 Hz, 1H), 1.90 (dt, *J* = 14.4, 7.6 Hz, 1H), 1.70 – 1.57 (m, 2H), 0.86 (d, *J* = 6.8 Hz, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.8 (C), 134.6 (C), 129.8 (2 CH), 129.0 (CH), 127.2 (2 CH), 109.5 (CH), 67.7 (CH_2), 36.5 (CH), 33.9 (CH_2), 32.5 (CH_3), 21.7 (CH_3), 16.3 (CH_3). **HRMS** (ESI): calcd for $C_{14}H_{22}NO_3S$: 284.1315. Found: 284.1320.



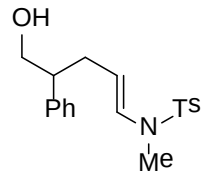
(*E*)-*N*-(4-(hydroxymethyl)-5-methylhex-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3e) was prepared following *method A*; but 1 mmol (5 eq) of aldehyde was used. The reaction was stirred for 4h 5 min. Colorless oil (59 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.62 (dt, *J* = 8.3, 1.8 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.73 (dt, *J* = 14.0, 1.2 Hz, 1H), 4.70 (dt, *J* = 14.3, 7.5 Hz, 1H), 3.55 (qd, *J* = 10.6, 5.8 Hz, 2H), 2.82 (s, 3H), 2.42 (s, 3H), 2.20 – 1.97 (m, 2H), 1.80 – 1.67 (m, 1H), 1.39 – 1.24 (m, 1H), 1.04 (bs, 1H), 0.89 (d, *J* = 6.9 Hz, 6H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.7 (C), 134.3 (C), 129.7 (2 CH), 128.6 (CH), 127.0 (2 CH), 110.5 (CH), 63.3 (CH_2), 47.2 (CH), 32.3 (CH_3), 28.9 (CH_2), 27.7 (CH), 21.6 (CH_3), 19.7 (CH_3), 19.6 (CH_3). **HRMS** (ESI): calcd for $C_{16}H_{26}NO_3S$: 312.1628. Found: 312.1619



(*E*)-*N*-(4-benzyl-5-hydroxypent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3f) was prepared following *method A*. The reaction was stirred for 1h 10 min. Colorless oil (74 %).

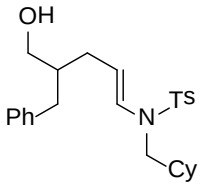
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.63 (d, *J* = 8.3 Hz, 2H), 7.27 (t, *J* = 7.7 Hz, 4H), 7.23 – 7.17 (m, 1H), 7.16 – 7.10 (m, 2H), 6.74 (d, *J* = 14.1 Hz, 1H), 4.67 (dt, *J* = 14.4, 7.5 Hz, 1H), 3.49 (t, *J* = 5.0 Hz, 2H), 2.81 (s, 3H), 2.67 – 2.47 (m, 2H), 2.39 (s, 3H), 2.20 – 2.00 (m, 2H), 1.90 – 1.76 (m, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.8 (C), 140.4 (C), 134.4 (C), 129.7 (2 CH), 129.1 (CH), 129.1 (2 CH), 128.4 (2 CH), 127.0 (2 CH), 126.0 (CH), 109.3 (CH), 64.5 (CH_2), 43.2 (CH), 37.2 (CH_2), 32.3 (CH_3), 31.5 (CH_2), 21.5 (CH_3). **HRMS** (ESI): calcd for $C_{20}H_{26}NO_3S$: 360.1628. Found: 360.1628



(*E*)-*N*-(5-hydroxy-4-phenylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3g) was prepared following *method A*, but in this case the organocatalyst and the aldehyde were not stirred for 10 minutes before the other components were added. The reaction was stirred for 3h. Colorless oil (78 %).

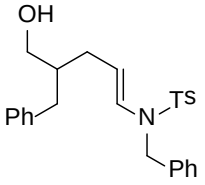
^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.49 (d, *J* = 8.3 Hz, 2H), 7.38 – 7.20 (m, 5H), 7.20 – 7.12 (m, 2H), 6.72 (dt, *J* = 14.1, 1.2 Hz, 1H), 4.57 (dt, *J* = 14.3, 7.3 Hz, 1H), 3.74 (d, *J* = 6.7 Hz, 2H), 2.86 – 2.75 (m, 1H), 2.72 (s, 3H), 2.54 – 2.43 (m, 1H), 2.41 (s, 3H), 2.39 – 2.29 (m, 1H), 1.30 (bs, 1H) **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.6 (C), 141.5 (C), 134.4 (C), 129.7 (2 CH), 129.1

(CH), 128.7 (2 CH), 128.0 (2 CH), 126.9 (2 CH), 126.8 (CH), 108.6 (CH), 66.8 (CH_2), 49.1 (CH), 32.7 (CH_2), 32.2 (CH_3), 21.5 (CH_3). **HRMS** (ESI): calcd for $C_{19}H_{24}NO_3S$: 346.1471. Found: 346.1474



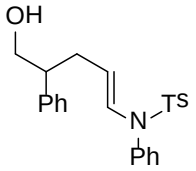
(*E*)-*N*-(4-benzyl-5-hydroxypent-1-en-1-yl)-*N*-(cyclohexylmethyl)-4-methylbenzenesulfonamide (3h) was prepared following *method A*. The reaction was stirred for 5h 20min. Colorless oil (55 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.62 (d, *J* = 8.3 Hz, 2H), 7.34 – 7.16 (m, 5H), 7.16 – 7.09 (m, 2H), 6.48 (d, *J* = 14.1 Hz, 1H), 4.81 (dt, *J* = 14.5, 7.5 Hz, 1H), 3.48 (t, *J* = 4.7 Hz, 2H), 3.03 (d, *J* = 7.0 Hz, 2H), 2.57 (h, *J* = 7.2 Hz, 2H), 2.38 (s, 3H), 2.20 – 2.01 (m, 2H), 1.89 – 1.78 (m 1H), 1.78 – 1.62 (m, 6H), 1.29 – 1.10 (m, 4H), 1.01 – 0.84 (m, 2H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.4 (C), 140.4 (C), 135.9 (C), 129.6 (2 CH), 129.1 (2 CH), 128.4 (2 CH), 127.9 (CH), 126.9 (2 CH), 126.0 (CH), 111.7 (CH), 64.5 (CH_2), 51.9 (CH_2), 43.2 (CH), 37.2 (CH_2), 35.1 (CH), 31.8 (CH_2), 30.8 (2 CH_2), 26.4 (CH_2), 25.9 (2 CH_2), 21.5 (CH_3). **HRMS** (ESI): calcd for $C_{26}H_{36}NO_3S$: 442.2410. Found: 442.2413.



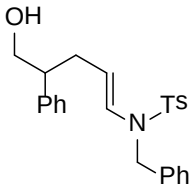
(*E*)-*N*-benzyl-*N*-(4-benzyl-5-hydroxypent-1-en-1-yl)-4-methylbenzenesulfonamide (3i) was prepared following *method A*. The reaction was stirred for 5h 20 min. Colorless oil (71 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.71 (d, *J* = 8.3 Hz, 2H), 7.41 – 7.14 (m, 10H), 7.05 – 6.96 (m, 2H), 6.63 (d, *J* = 14.1 Hz, 1H), 4.66 (dt, *J* = 14.4, 7.5 Hz, 1H), 4.50 (s, 2H), 3.26 (p, *J* = 5.3 Hz, 2H), 2.44 (s, 3H), 2.36 (ddd, *J* = 13.6, 6.8, 6.3 Hz, 2H), 1.99 (t, *J* = 7.0 Hz, 2H), 1.75 – 1.53 (m, 2H), 1.08 (bs, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.8 (C), 140.3 (C), 135.9 (C), 135.6 (C), 129.8 (2 CH), 129.0 (2 CH), 128.6 (2 CH), 128.3 (2 CH), 127.5 (CH), 127.0 (2 CH), 127.0 (2 CH), 126.8 (CH), 125.9 (CH), 111.8 (CH), 64.3 (CH_2), 49.5 (CH_2), 43.0 (CH), 36.7 (CH_2), 31.6 (CH_2), 21.6 (CH_3). **HRMS** (ESI): calcd for $C_{26}H_{30}NO_3S$: 436.1941. Found: 436.1944.



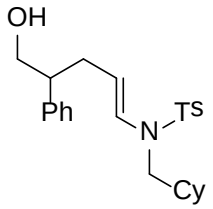
(*E*)-*N*-(5-hydroxy-4-phenylpent-1-en-1-yl)-4-methyl-*N*-phenylbenzenesulfonamide (3j) was prepared following *method A*, but in this case the organocatalyst and the aldehyde were not stirred for 10 minutes before the other components were added. The reaction was stirred for 6h. Colorless oil (61 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.45 – 7.37 (m, 2H), 7.35 – 7.18 (m, 8H), 7.15 – 7.05 (m, 2H), 6.92 – 6.78 (m, 3H), 4.27 (dt, *J* = 13.9, 7.5 Hz, 1H), 3.69 (m, 2H), 2.71 (p, *J* = 6.9 Hz, 1H), 2.44 (s, 3H), 2.41 – 2.33 (m, 1H), 2.31 – 2.20 (m, 1H), 1.27 (bs, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.8 (C), 141.8 (C), 136.9 (C), 136.0 (C), 130.4 (CH), 130.2 (2 CH), 129.7 (2 CH), 129.4 (2 CH), 128.9 (CH), 128.8 (2 CH), 128.1 (2 CH), 127.5 (2 CH), 126.9 (CH), 110.1 (CH), 66.7 (CH_2), 49.0 (CH), 32.9 (CH_2), 21.7 (CH_3). **HRMS** (ESI): calcd for $C_{24}H_{26}NO_3S$: 408.1628. Found: 436.1636.



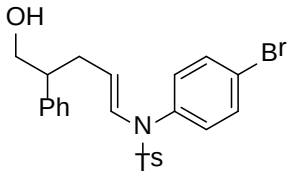
(*E*)-*N*-benzyl-*N*-(5-hydroxy-4-phenylpent-1-en-1-yl)-4-methylbenzenesulfonamide (3k) was prepared following *method A*, but in this case the organocatalyst and the aldehyde were not stirred for 10 minutes before the other components were added. The reaction was stirred for 4h. Colorless oil (94 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.58 (d, *J* = 8.3 Hz, 2H), 7.36 – 7.15 (m, 10H), 7.03 – 6.96 (m, 2H), 6.62 (d, *J* = 14.1 Hz, 1H), 4.58 (dt, *J* = 14.6, 7.4 Hz, 1H), 4.40 (d, *J* = 3.0 Hz, 2H), 3.62 (t, *J* = 5.7 Hz, 2H), 2.73 – 2.60 (m, 1H), 2.46 (s, 3H), 2.42 – 2.20 (m, 2H), 1.19 (bs, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.6 (C), 141.3 (C), 135.9 (C), 135.5 (C), 129.8 (2 CH), 128.6 (2 CH), 128.5 (2 CH), 127.9 (2 CH), 127.3 (CH), 127.0 (CH), 126.9 (2 CH), 126.9 (2 CH), 126.7 (CH), 110.5 (CH), 66.4 (CH_2), 49.3 (CH_2), 48.8 (CH), 33.2 (CH_2), 21.6 (CH_3). **HRMS** (ESI): calcd for $C_{25}H_{28}NO_3S$: 422.1784. Found: 422.1787.



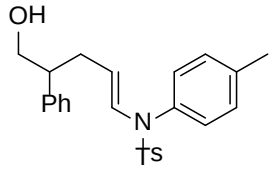
(*E*)-*N*-(cyclohexylmethyl)-*N*-(5-hydroxy-4-phenylpent-1-en-1-yl)-4-methylbenzenesulfonamide (3l) was prepared following *method A*, but in this case the organocatalyst and the aldehyde were not stirred for 10 minutes before the other components were added. In this case 1 mmol (5 eq) of aldehyde was used. The reaction was stirred for 8h. Colorless oil (88 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.48 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.13 (m, 7H), 6.46 (d, *J* = 14.2 Hz, 1H), 4.64 (dt, *J* = 14.4, 7.3 Hz, 1H), 3.74 (d, *J* = 5.0 Hz, 2H), 2.97 – 2.76 (m, 3H), 2.48 (dt, *J* = 13.2, 6.7 Hz, 1H), 2.40 (s, 3H), 2.33 (dt, *J* = 14.3, 7.7 Hz, 1H), 1.80 – 1.57 (m, 5H), 1.24 (m, 2H), 1.22 – 1.04 (m, 3H), 0.94 – 0.74 (m, 2H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.2 (C), 141.4 (C), 135.9 (C), 129.6 (2 CH), 128.7 (2 CH), 128.1 (2 CH), 127.7 (CH), 126.9 (CH), 126.8 (2 CH), 111.0 (CH), 66.9 (CH_2), 51.7 (CH_2), 49.2 (CH), 34.8 (CH), 33.0 (CH_2), 30.8 (2 CH_2), 26.4 (CH_2), 25.8 (2 CH_2), 21.5 (CH_3). **HRMS** (ESI): calcd for $C_{25}H_{34}NO_3S$: 428.2254. Found: 428.2255.



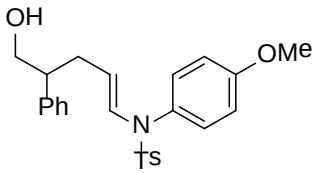
(*E*)-*N*-(4-bromophenyl)-*N*-(5-hydroxy-4-phenylpent-1-en-1-yl)-4-methylbenzenesulfonamide (3m) was prepared following *method A*, but in this case the organocatalyst and the aldehyde were not stirred for 10 minutes before the other components were added. The reaction was stirred for 6h. Colorless oil (27 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.42 (dd, *J* = 8.4, 6.3 Hz, 4H), 7.36 – 7.21 (m, 5H), 7.15 – 7.08 (m, 2H), 6.84 (d, *J* = 14.0 Hz, 1H), 6.69 (d, *J* = 8.7 Hz, 2H), 4.30 (dt, *J* = 14.3, 7.4 Hz, 1H), 3.72 (d, *J* = 6.4 Hz, 2H), 2.74 (dt, *J* = 13.3, 6.6 Hz, 1H), 2.46 (s, 3H), 2.46 – 2.34 (m, 1H), 2.26 (dt, *J* = 15.4, 8.0 Hz, 1H), 1.27 (bs, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.9 (C), 141.5 (C), 135.9 (C), 135.4 (C), 132.6 (2 CH), 131.6 (2 CH), 130.0 (CH), 129.6 (2 CH), 128.6 (2 CH), 128.0 (2 CH), 127.3 (2 CH), 126.8 (CH), 122.9 (C), 110.5 (CH), 66.6 (CH_2), 48.8 (CH), 32.6 (CH_2), 21.6 (CH_3). **HRMS** (ESI): calcd for $C_{24}H_{25}BrNO_3S$: 486.0733. Found: 486.0732.



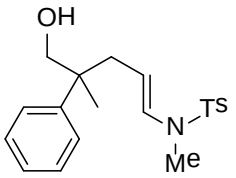
(*E*)-*N*-(5-hydroxy-4-phenylpent-1-en-1-yl)-4-methyl-*N*-(p-tolyl)benzenesulfonamide (3n) was prepared following *method A*, but in this case the organocatalyst and the aldehyde were not stirred for 10 minutes before the other components were added. The reaction was stirred for 7h 30m. Colorless oil (42 %).

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.41 (d, *J* = 8.3 Hz, 2H), 7.34 – 7.18 (m, 5H), 7.15 – 7.04 (m, 4H), 6.86 (d, *J* = 13.9 Hz, 1H), 6.70 (d, *J* = 8.2 Hz, 2H), 4.27 (dt, *J* = 14.2, 7.5 Hz, 1H), 3.70 (d, *J* = 7.2 Hz, 2H), 2.71 (p, *J* = 7.1 Hz, 1H), 2.43 (s, 3H), 2.41 – 2.34 (m, 1H), 2.33 (s, 3H), 2.31 – 2.18 (m, 1H), 1.26 (bs, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.7 (C), 141.9 (C), 139.0 (C), 136.1 (C), 134.1 (C), 130.5 (CH), 130.1 (2 CH), 129.9 (2 CH), 129.6 (2 CH), 128.8 (2 CH), 128.1 (2 CH), 127.5 (2 CH), 126.9 (CH), 109.7 (CH), 66.7 (CH_2), 49.0 (CH), 33.0 (CH_2), 21.7 (CH_3), 21.3 (CH_3). **HRMS** (ESI): calcd for $C_{25}H_{28}NO_3S$: 422.1784. Found: 422.1786.



(*E*)-*N*-(5-hydroxy-4-phenylpent-1-en-1-yl)-*N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide (3o) was prepared following *method A*, but in this case the organocatalyst and the aldehyde were not stirred for 10 minutes before the other components were added. The reaction was stirred for 7h 30m. Colorless oil (47 %).

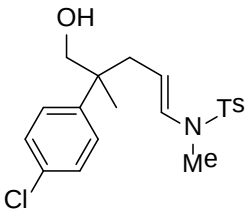
^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.40 (d, *J* = 8.3 Hz, 2H), 7.33 – 7.16 (m, 5H), 7.10 (d, *J* = 6.8 Hz, 2H), 6.87 (d, *J* = 14.0 Hz, 1H), 6.82 – 6.66 (m, 4H), 4.26 (dt, *J* = 14.2, 7.5 Hz, 1H), 3.79 (s, 3H), 3.75 – 3.65 (m, 2H), 2.71 (p, *J* = 7.1 Hz, 1H), 2.43 (s, 3H), 2.41 – 2.32 (m, 1H), 2.31 – 2.18 (m, 1H), 1.24 (bs, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 159.7 (C), 143.7 (C), 141.8 (C), 136.0 (C), 131.3 (2 CH), 130.6 (CH), 129.6 (2 CH), 129.1 (C), 128.8 (2 CH), 128.1 (2 CH), 127.5 (2 CH), 126.9 (CH), 114.7 (2 CH), 109.4 (CH), 66.7 (CH_2), 55.6 (CH_3), 49.1 (CH), 32.9 (CH_2), 21.8 (CH_3). **HRMS** (ESI): calcd for $C_{25}H_{28}NO_4S$: 438.1734. Found: 438.1735.



(*E*)-*N*-(5-hydroxy-4-methyl-4-phenylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3p) was prepared following:

- *Method A*. The reaction was stirred for 23h. Colorless oil (47 %).
- *Method B, R^4 = TBS.* The reaction was stirred for 4h. Colorless oil (72 %, 76 %ee)
- *Method B, R^4 = TIPS.* The reaction was stirred for 3h. Colorless oil (25 %, 86 %ee)

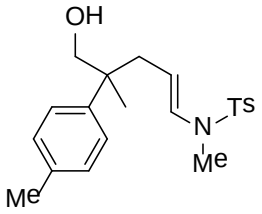
^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.45 (d, *J* = 8.3 Hz, 2H), 7.31 – 7.11 (m, 7H), 6.64 (d, *J* = 14.0 Hz, 1H), 4.36 (dt, *J* = 14.3, 7.6 Hz, 1H), 3.63 (d, *J* = 10.9 Hz, 1H), 3.49 (d, *J* = 10.9 Hz, 1H), 2.62 (s, 3H), 2.45 (dd, *J* = 13.9, 6.8 Hz, 1H), 2.34 (s, 3H), 2.24 (dd, *J* = 14.3, 7.8 Hz, 1H), 1.53 (bs, 1H), 1.20 (s, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 144.3 (C), 143.6 (C), 134.4 (C), 129.8 (CH), 129.7 (2 CH), 128.5 (2 CH), 127.0 (2 CH), 126.6 (2 CH), 126.3 (CH), 106.6 (CH), 71.6 (CH_2), 43.6 (C), 39.1 (CH_2), 32.2 (CH_3), 21.7 (CH_3), 21.5 (CH_3). **HRMS** (ESI): calcd for $C_{20}H_{26}NO_3S$: 360.1628. Found: 360.1635.



(*E*)-*N*-(4-(4-chlorophenyl)-5-hydroxy-4-methylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3q) was prepared following:

- *Method A*. The reaction was stirred for 8h. Colorless oil (28 %).
- *Method B, R^4 = TBS.* The reaction was stirred for 3h. Colorless oil (27 %, 24 %ee)
- *Method B, R^4 = TIPS.* The reaction was stirred for 8h. Colorless oil (23 %, 60 %ee)

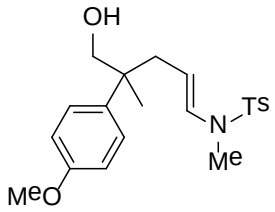
^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.52 (d, *J* = 8.3 Hz, 2H), 7.34 – 7.18 (m, 6H), 6.71 (dt, *J* = 14.0, 0.9 Hz, 1H), 4.40 (dt, *J* = 14.1, 7.6 Hz, 1H), 3.68 (dd, *J* = 10.7, 4.9 Hz, 1H), 3.55 (dd, *J* = 10.8, 6.4 Hz, 1H), 2.70 (s, 3H), 2.49 (ddd, *J* = 13.9, 7.3, 1.0 Hz, 1H), 2.42 (s, 3H), 2.30 (ddd, *J* = 14.0, 7.9, 1.0 Hz, 1H), 1.26 (s, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.9 (C), 143.1 (C), 134.6 (C), 132.3 (C), 130.2 (CH), 129.8 (2 CH), 128.7 (2 CH), 128.3 (2 CH), 127.1 (2 CH), 106.1 (CH), 71.6 (CH_2), 43.6 (C), 39.2 (CH_2), 32.3 (CH_3), 21.8 (CH_3), 21.7 (CH_3). **HRMS** (ESI): calcd for $C_{20}H_{25}ClNO_3S$: 394.1238. Found: 394.1240.



(*E*)-*N*-(5-hydroxy-4-methyl-4-(p-tolyl)pent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3r) was prepared following:

- *Method A*. The reaction was stirred for 23h. Colorless oil (46 %).
- *Method B, R^4 = TBS.* The reaction was stirred for 2h 15m. Colorless oil (80 %, 60 %ee)
- *Method B, R^4 = TIPS.* The reaction was stirred for 7h. Colorless oil (35 %, 82 %ee)

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.54 (d, *J* = 8.0 Hz, 2H), 7.29 – 7.24 (m, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.72 (d, *J* = 14.1 Hz, 1H), 4.45 (dt, *J* = 14.5, 7.5 Hz, 1H), 3.68 (d, *J* = 10.7 Hz, 1H), 3.54 (d, *J* = 10.7 Hz, 1H), 2.70 (s, 3H), 2.50 (dd, *J* = 13.9, 6.9 Hz, 1H), 2.42 (s, 3H), 2.33 (s, 3H), 2.32 – 2.26 (m, 1H), 1.25 (s, 3H), 1.21 (bs, 1H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 143.6 (C), 141.2 (C), 135.8 (C), 134.4 (C), 129.8 (CH), 129.6 (2 CH), 129.2 (2 CH), 127.0 (2 CH), 126.5 (2 CH), 106.8 (CH), 71.7 (CH_2), 43.3 (C), 39.1 (CH_2), 32.2 (CH_3), 21.7 (CH_3), 21.6 (CH_3), 20.9 (CH_3). **HRMS** (ESI): Calcd for $C_{21}H_{28}NO_3S$: 374.1784. Found: 374.1791.



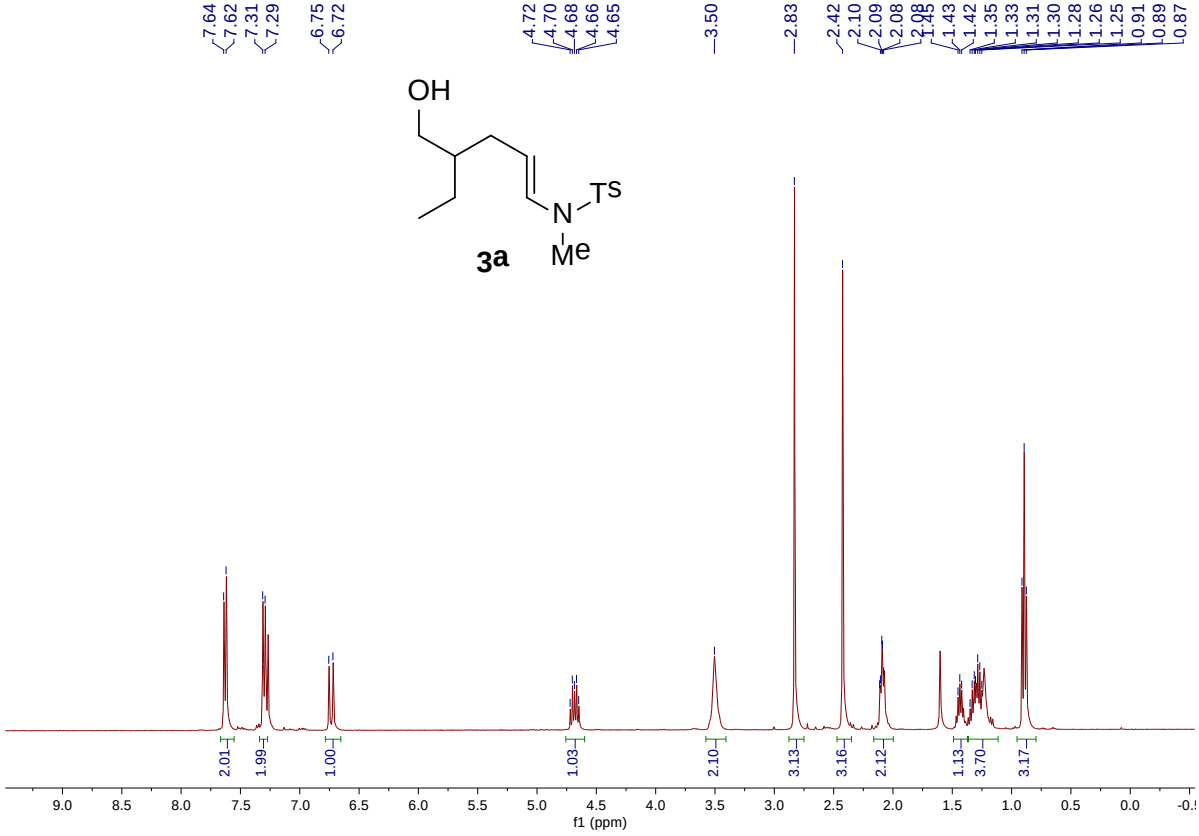
(*E*)-*N*-(5-hydroxy-4-(4-methoxyphenyl)-4-methylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3s) was prepared following:

- *Method A*. The reaction was stirred for 30h. Colorless oil (25 %).
- *Method B, R^4 = TBS.* The reaction was stirred for 4h. Colorless oil (73 %, 68 %ee)
- *Method B, R^4 = TIPS.* The reaction was stirred for 7h. Colorless oil (32 %, 80 %ee)

^{1}H-NMR (300 MHz, $CDCl_3$): δ (ppm) = 7.53 (d, *J* = 7.8 Hz, 2H), 7.34 – 7.13 (m, 4H), 6.86 (d, *J* = 8.4 Hz, 2H), 6.71 (d, *J* = 14.0 Hz, 1H), 4.43 (dt, *J* = 14.3, 7.4 Hz, 1H), 3.80 (s, 3H), 3.66 (d, *J* = 10.3 Hz, 1H), 3.54 (d, J = 9.6, 1H), 2.69 (s, 3H), 2.48 (dd, *J* = 14.1, 7.2 Hz, 1H), 2.41 (s, 3H), 2.28 (dd, *J* = 13.7, 8.1 Hz, 1H), 1.24 (s, 3H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 157.9 (C), 143.6 (C),

136.1 (C), 134.4 (C), 129.8 (CH), 129.6 (2 CH), 127.7 (2 CH), 127.0 (2 CH), 113.8 (2 CH), 106.7 (CH), 71.8 (CH_2), 55.2 (CH_3), 43.0 (C), 39.2 (CH_2), 32.2 (CH_3), 21.8 (CH_3), 21.5 (CH_3). **HRMS** (ESI): calcd for $C_{21}H_{28}NO_4S$: 390.1734. Found: 390.1735.

NMR Spectra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4.70
4.68
4.66
4.65
3.50
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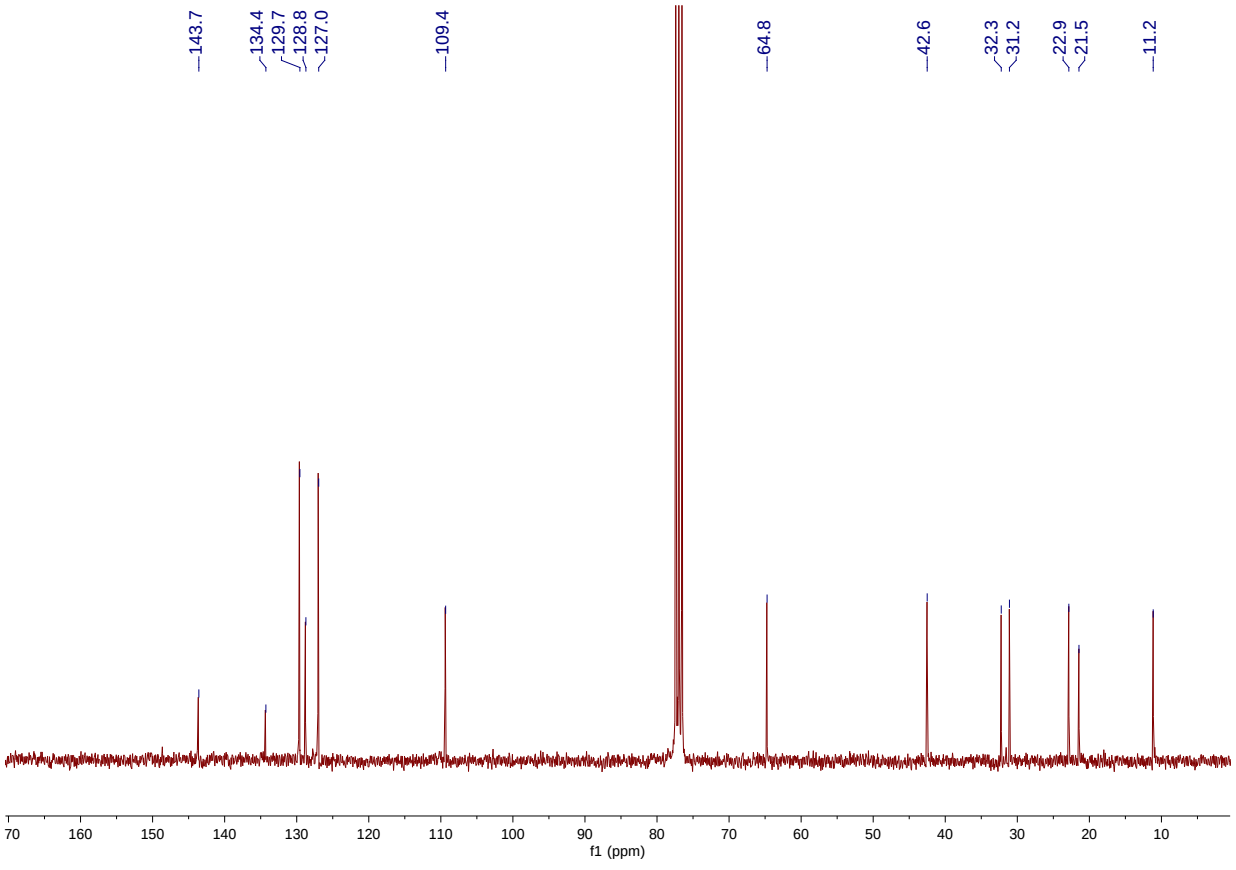
2.42
2.10
2.09
2.08
1.43
1.42
1.35
1.33
1.31
1.30
1.28
1.26
1.25
0.91
0.89
0.87
OH
N
Ts
Me
3a
2.01
1.99
1.00
1.03
2.10
3.13
3.16
2.12
1.13
3.70
3.17
f1 (ppm)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109.4
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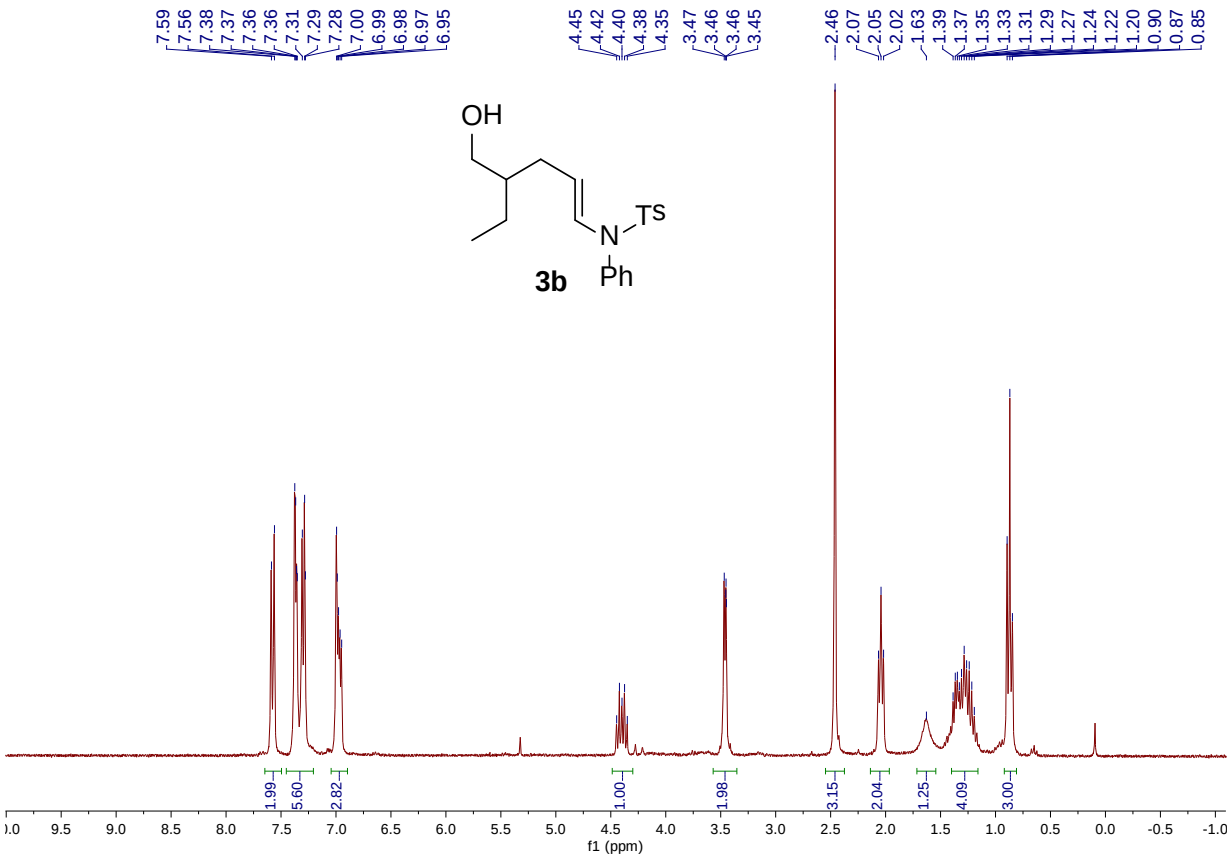
42.6
32.3
31.2
22.9
21.5
11.2
f1 (ppm)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2.46
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2.05
2.02
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1.39
1.37
1.35
1.33
1.31
1.29
1.27
1.24
1.22
1.20
0.90
0.87
0.85
OH
TS
N
Ph
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1.98
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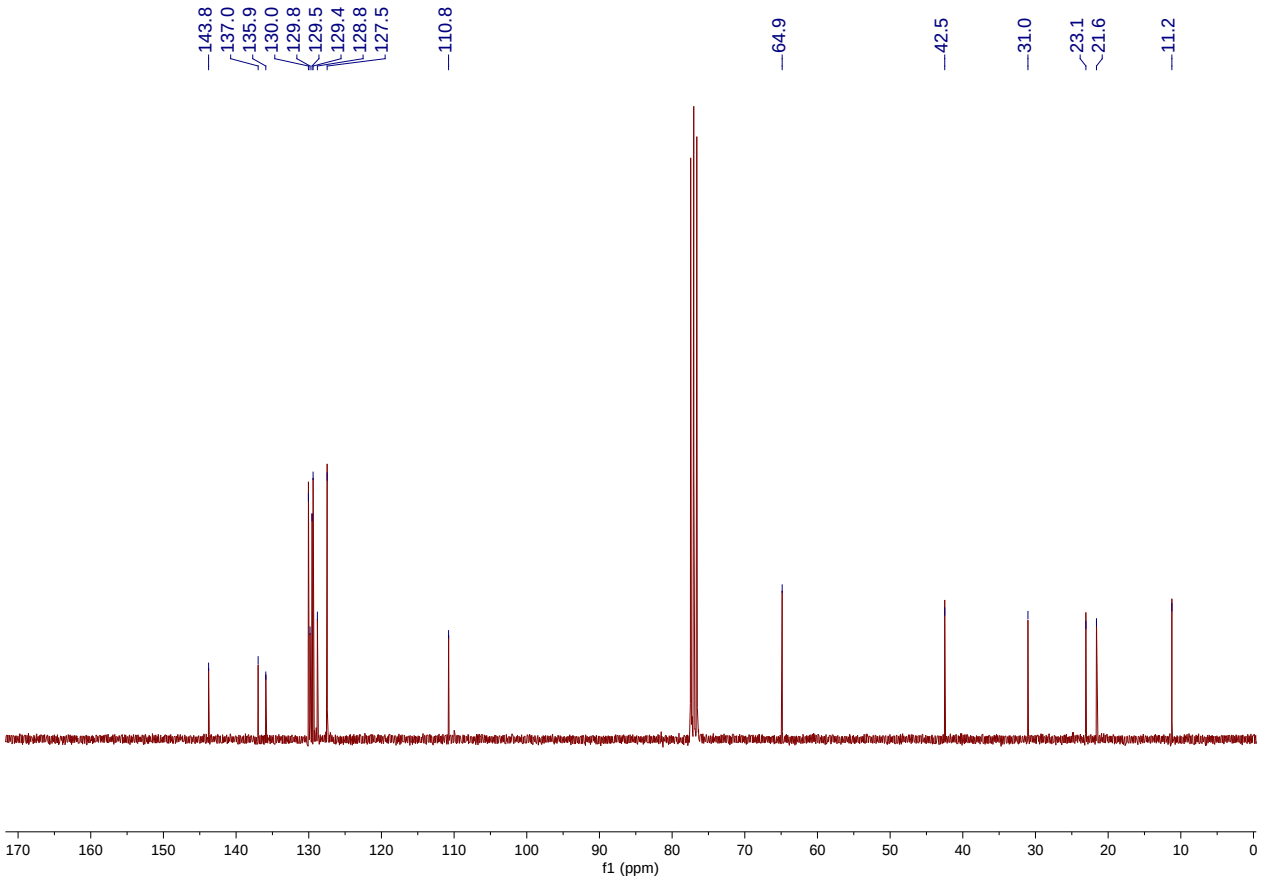
1.25
4.09
3.00
f1 (ppm)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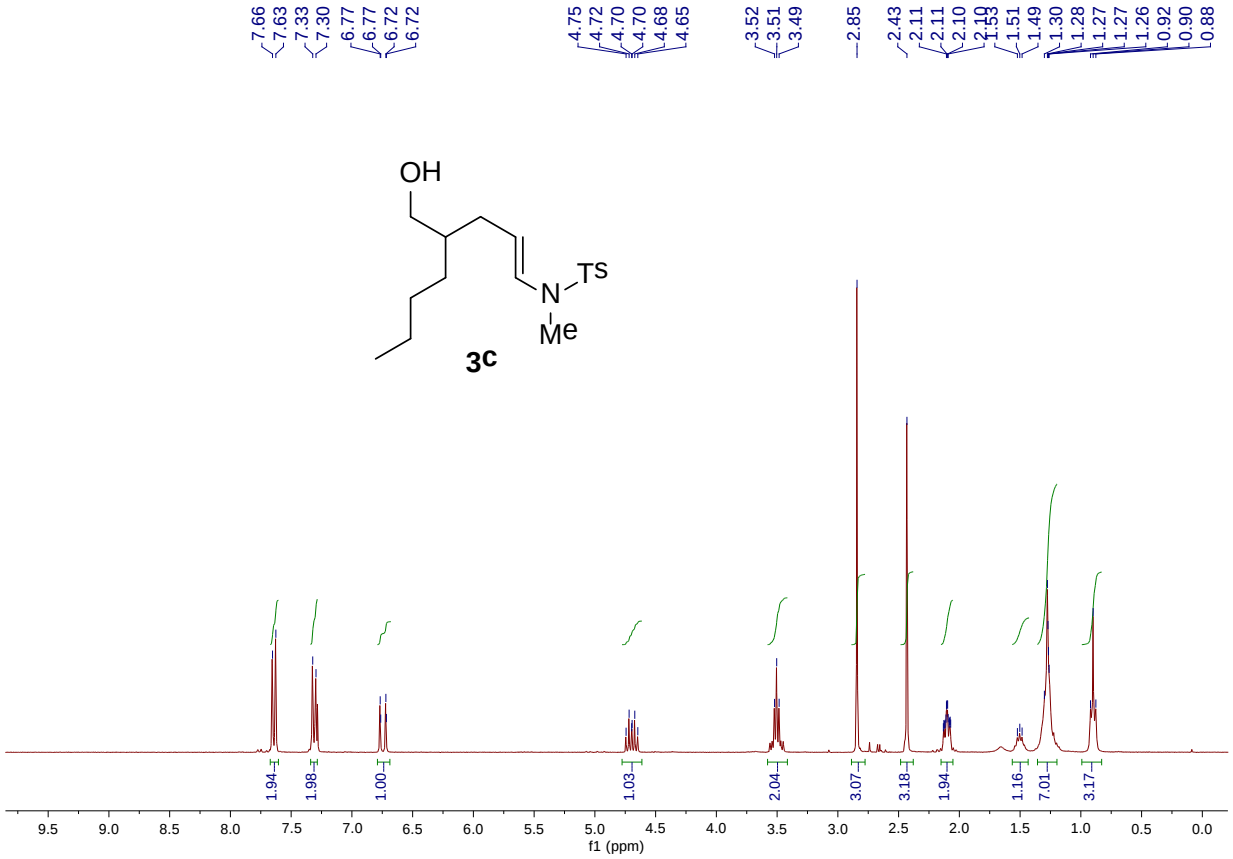
110.8
64.9
42.5
31.0
23.1
21.6
11.2
f1 (ppm)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2.10
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1.27
1.27
1.26
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0.88
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N
Ts
Me
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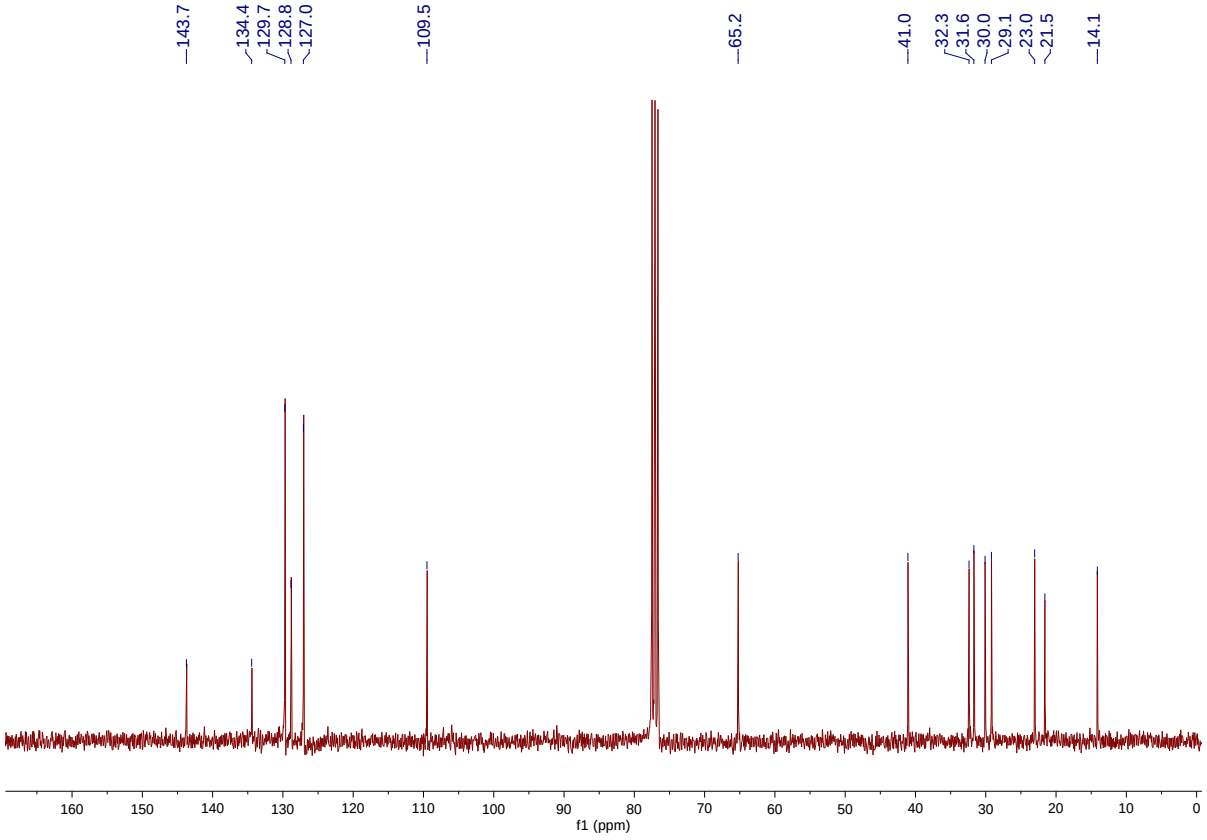
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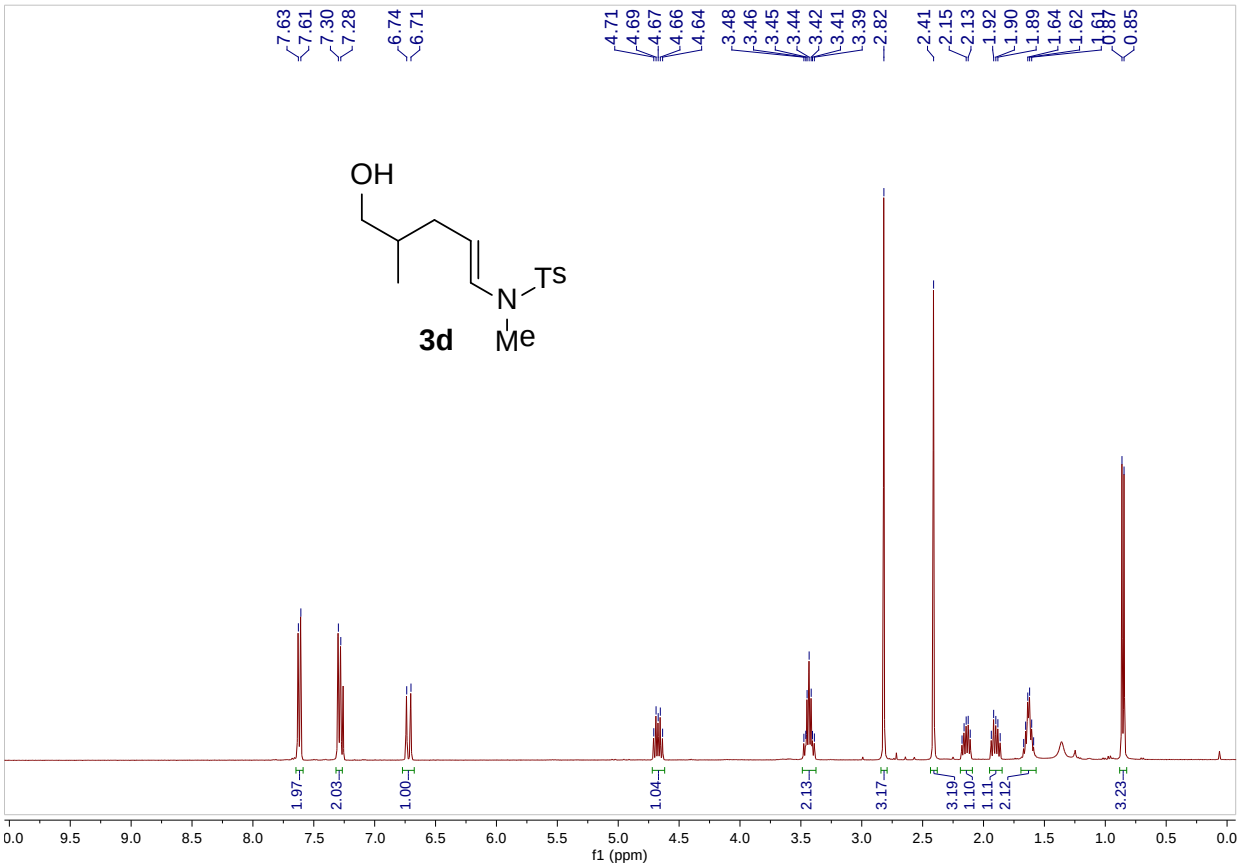
30.0
29.1
23.0
21.5
14.1
f1 (ppm)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3.39
2.82
2.41
2.15
2.13
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1.89
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1.61
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0.85
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TS
N
Me
3d
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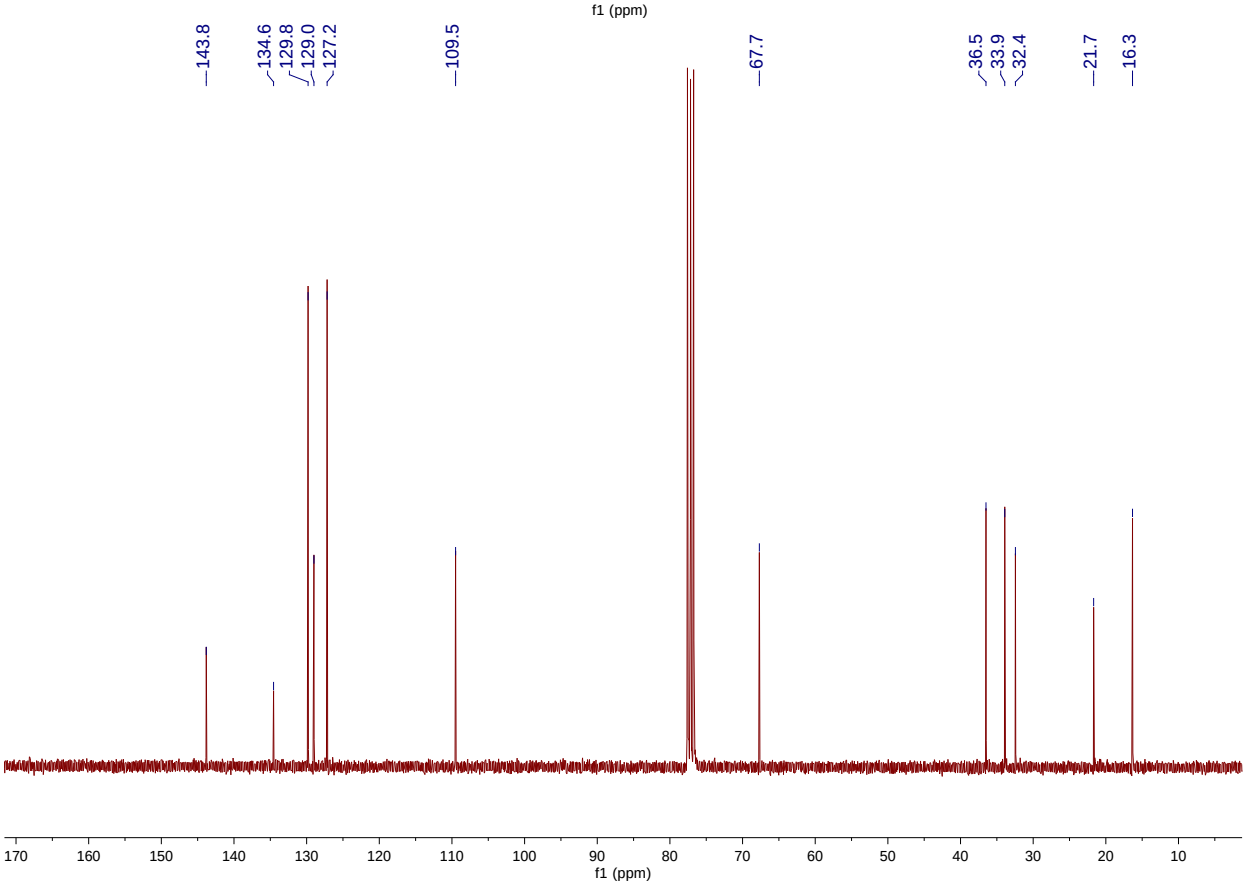
3.19
1.10
1.11
2.12
3.23
f1 (ppm)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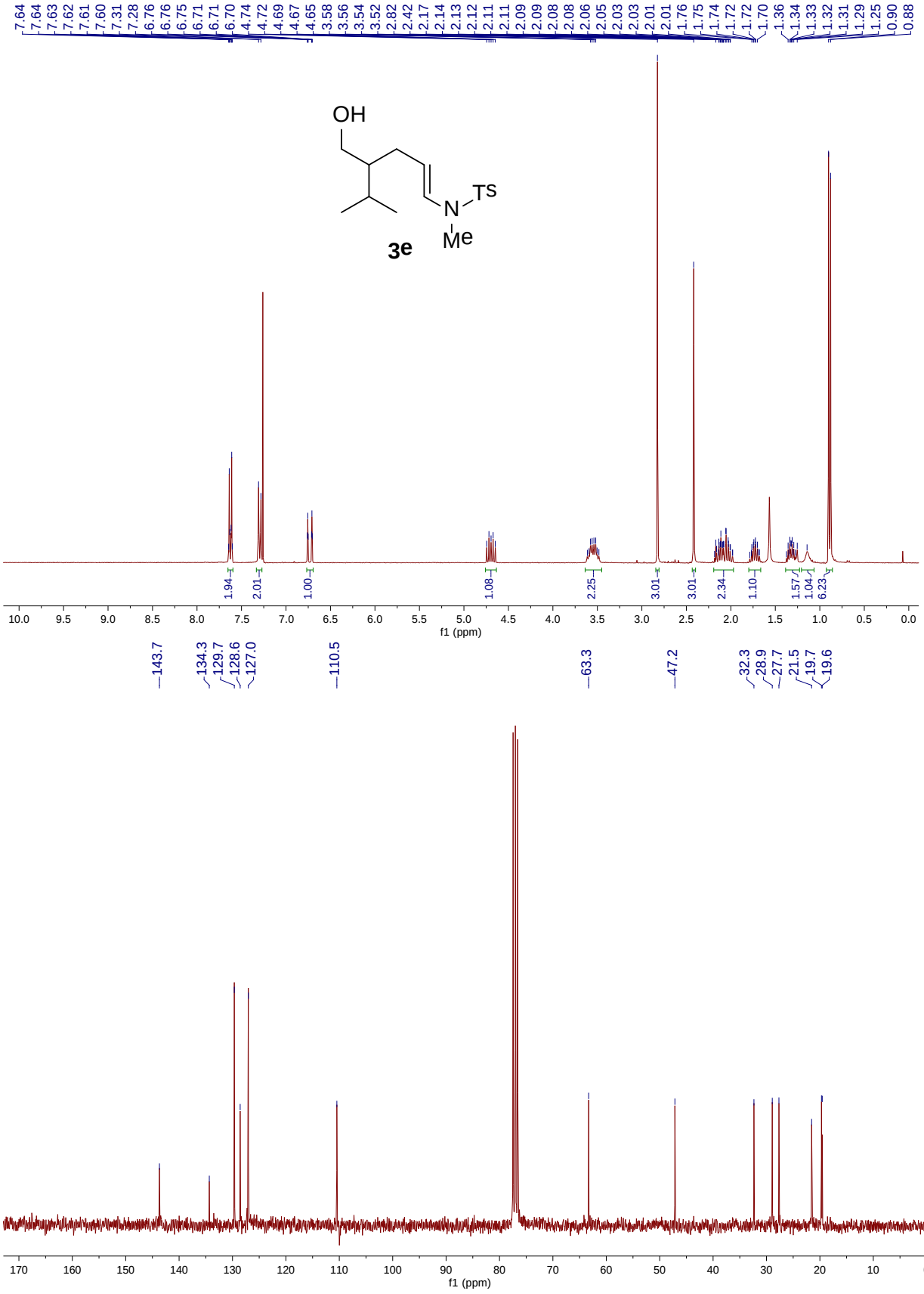
32.4
21.7
16.3
f1 (ppm)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OH
TS
N
Me
3e
1.94 2.01 1.00 1.08 2.25 3.01 3.01 2.34 1.10 1.57 1.04 6.23
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
143.7 134.3 129.7 128.6 127.0 110.5 63.3 47.2 32.3 28.9 27.7 21.5 19.7 19.6
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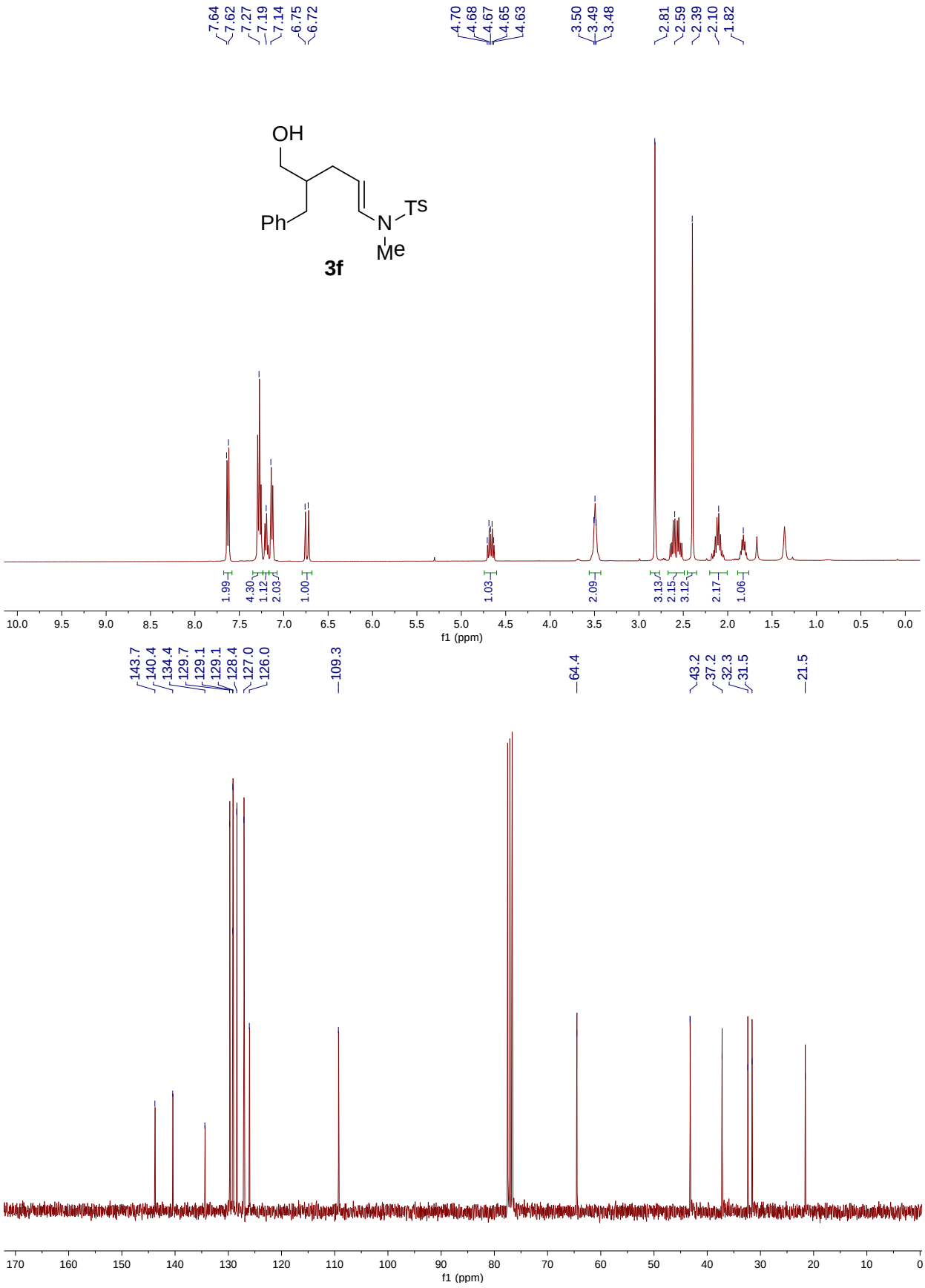
f1 (ppm)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2.10
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OH
Ph
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Ts
Me
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4.30
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2.03
1.00
1.03
2.09
3.13
2.15
3.12
2.17
1.06
f1 (ppm)
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129.7
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64.4
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32.3
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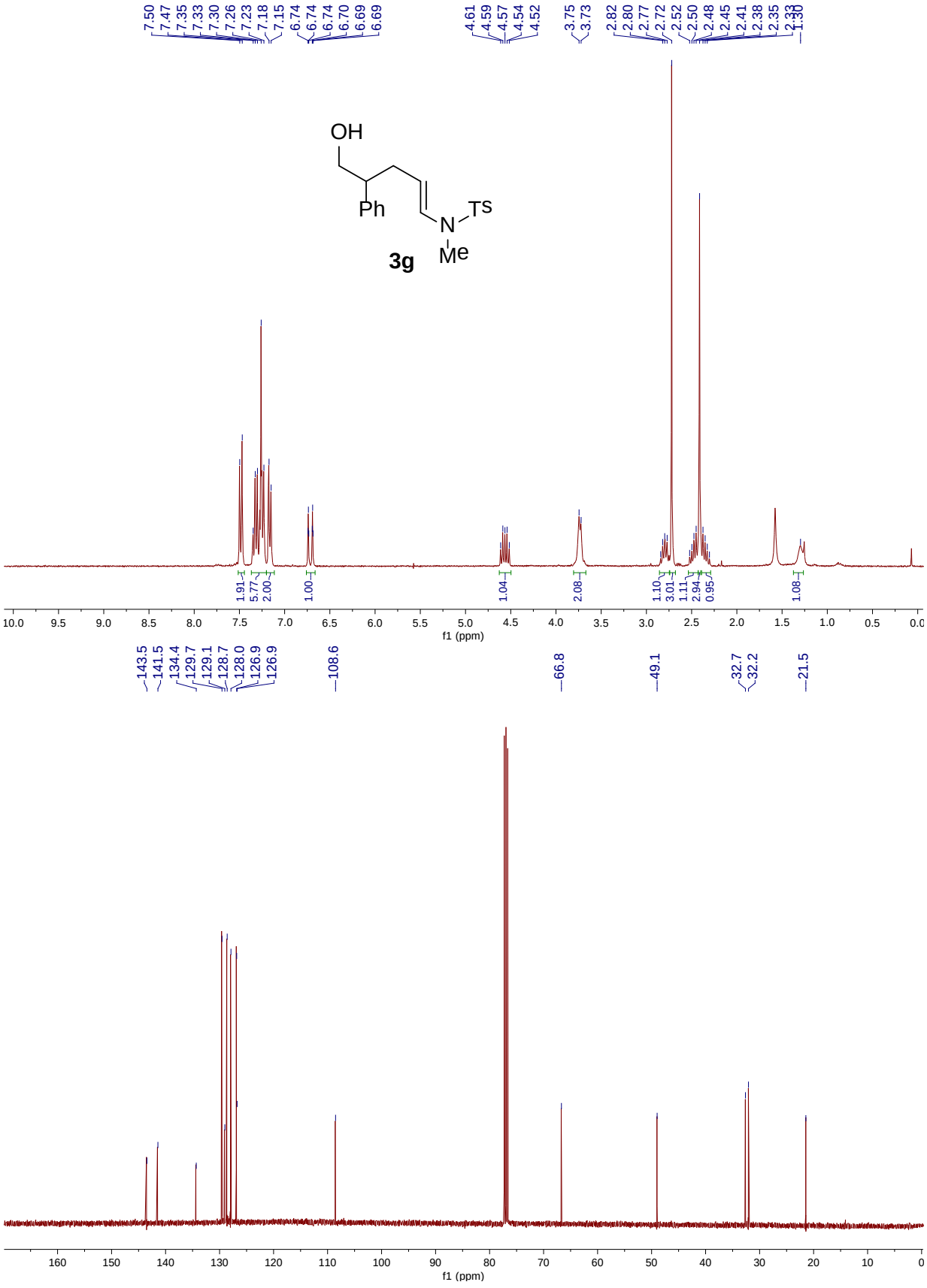
21.5
f1 (ppm)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2.33
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f1 (ppm)
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32.7
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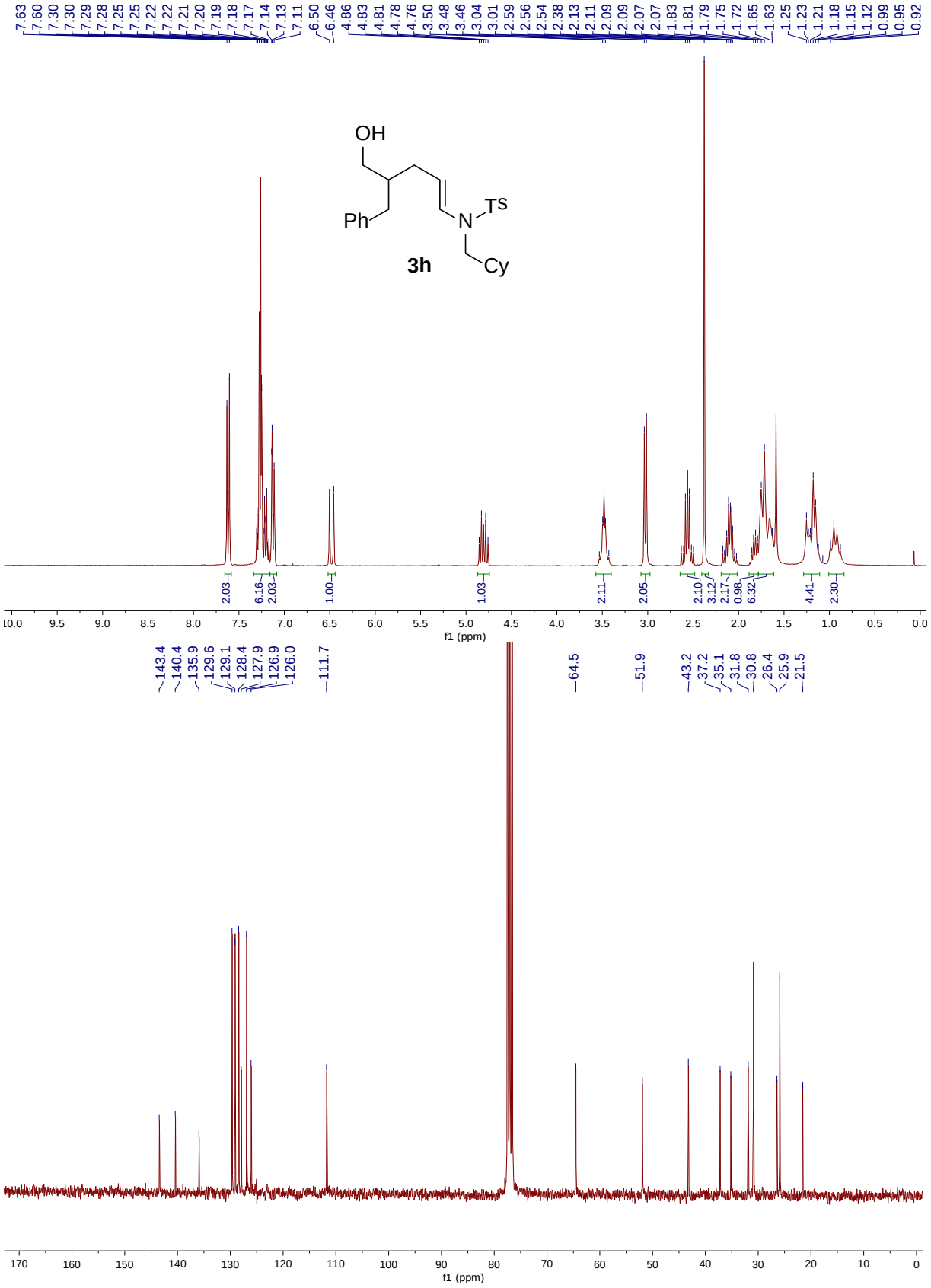
21.5
f1 (ppm)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TS
Cy
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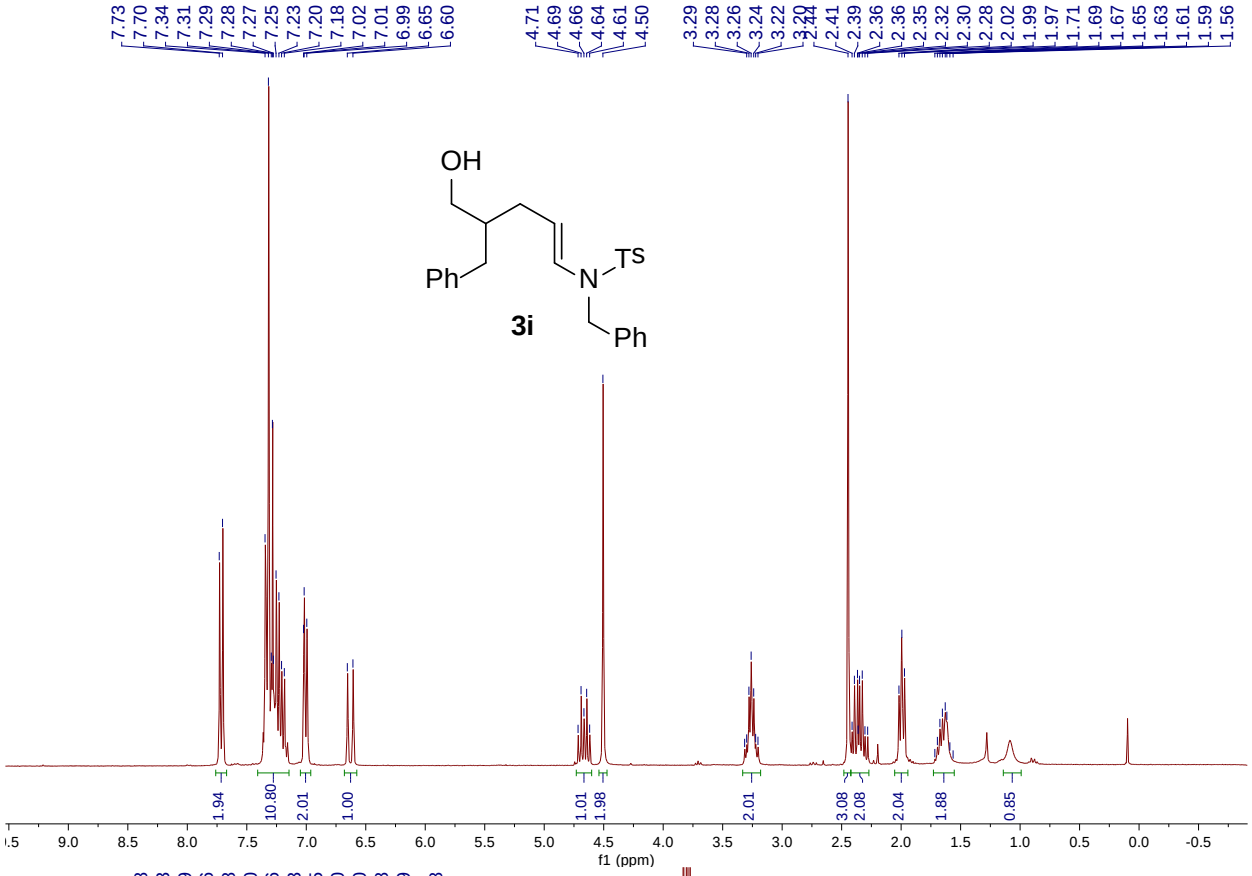
f1 (ppm)
f1 (ppm)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4.61
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3.28
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3.24
3.22
2.41
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2.32
2.30
2.28
2.02
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1.56
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Ph
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TS
Ph
3i
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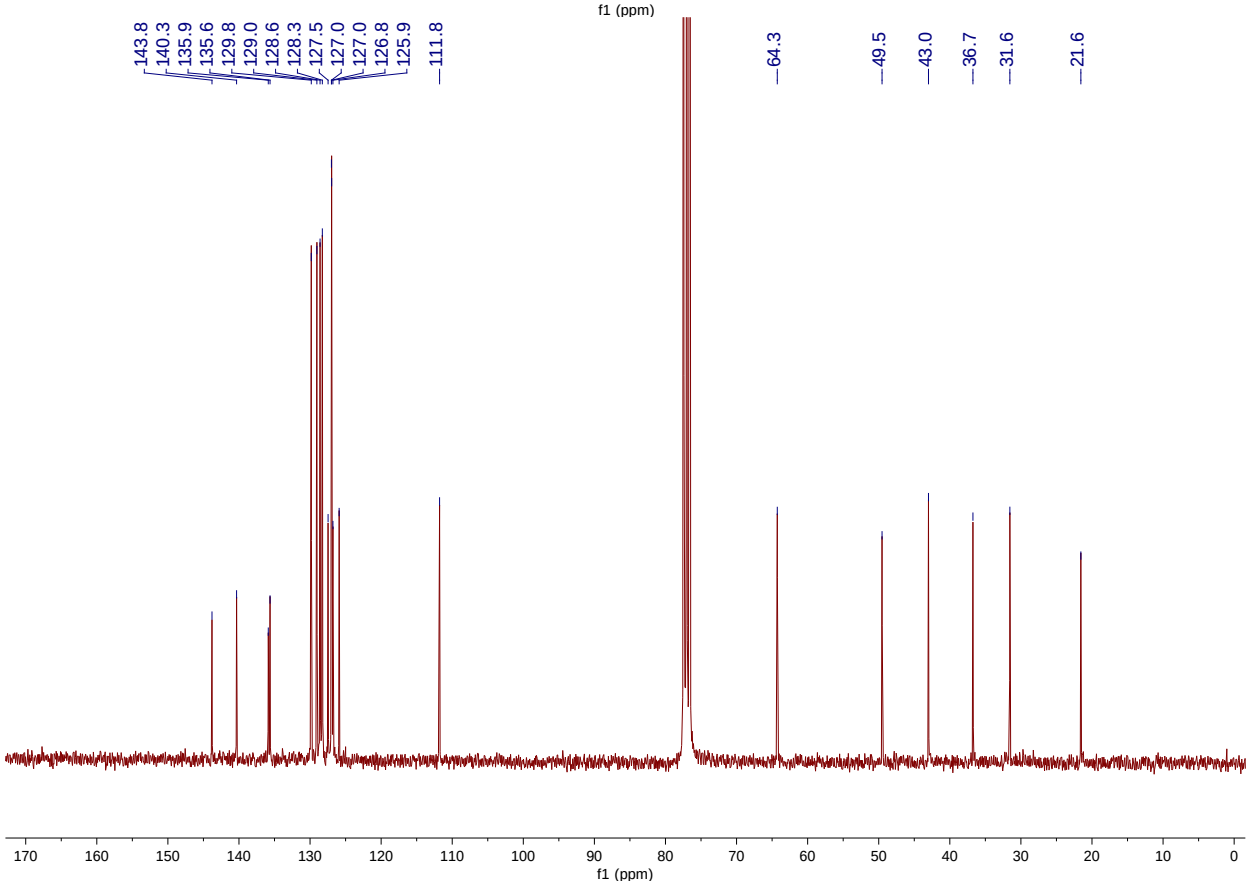
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64.3
49.5
43.0
36.7
31.6
21.6
f1 (ppm)


7.42
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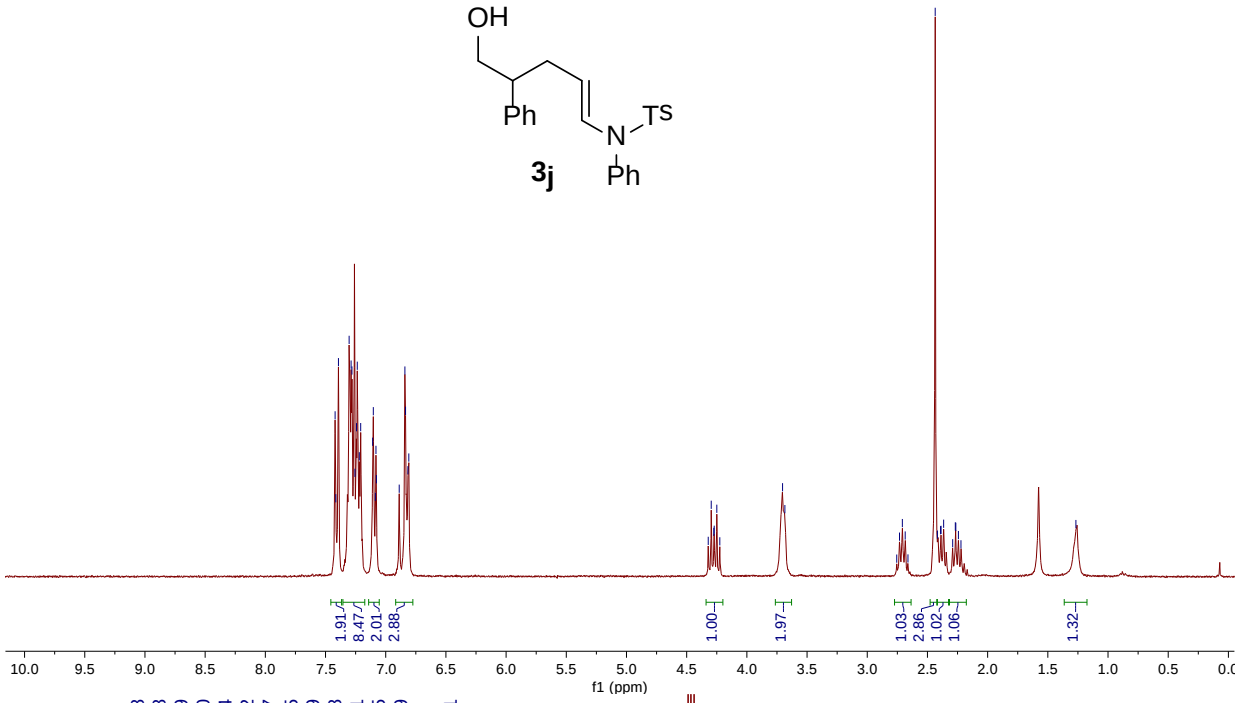
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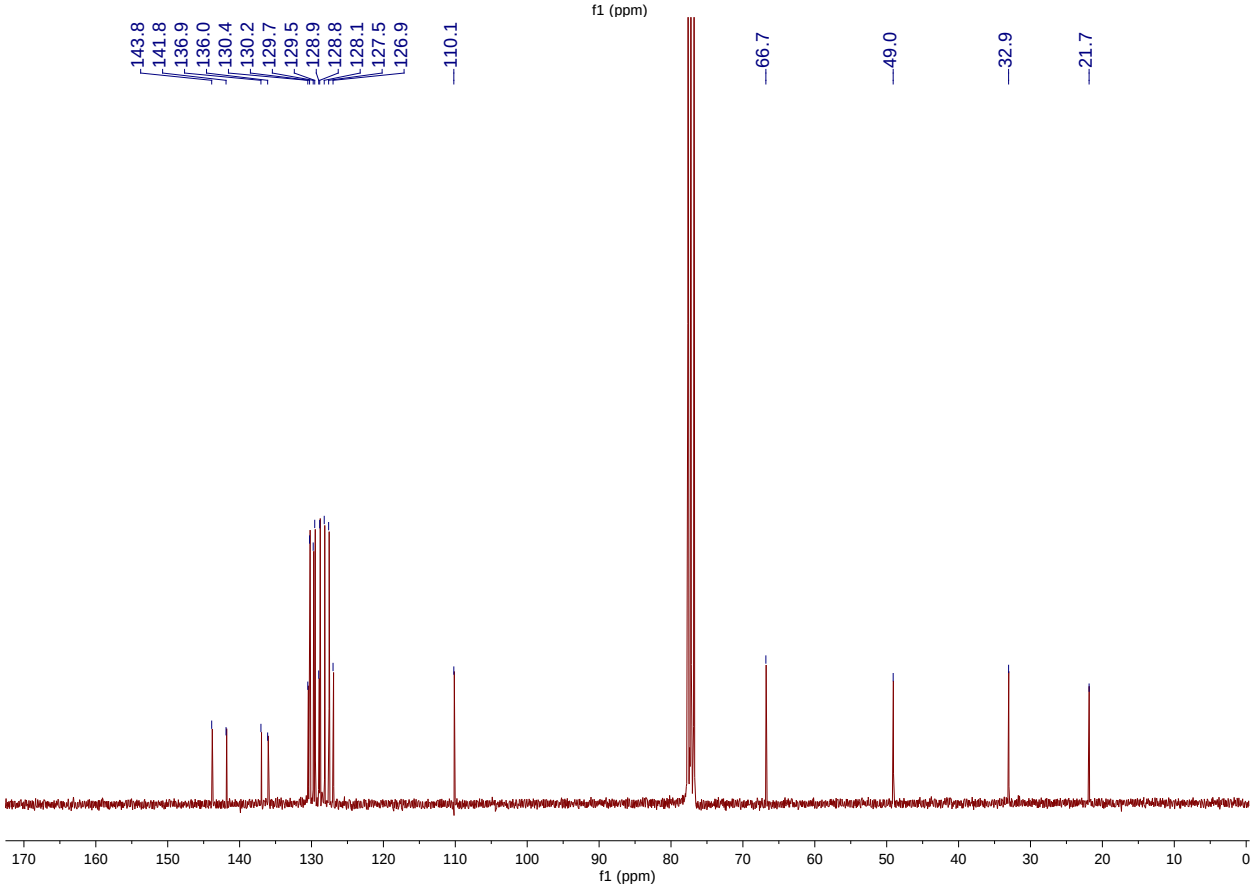
f1 (ppm)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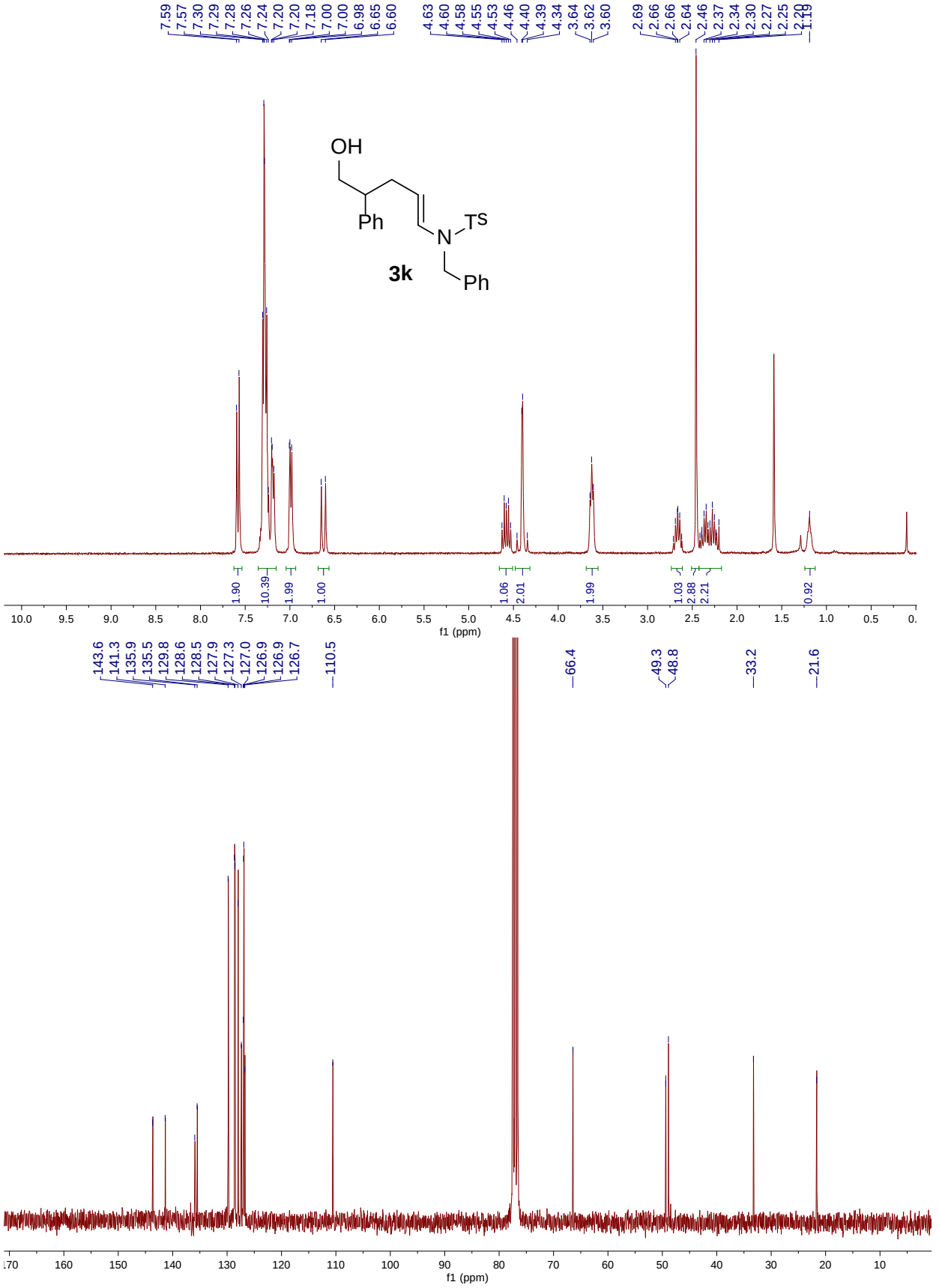
21.7
f1 (ppm)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Ph
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TS
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1.99
1.03
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2.21
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f1 (ppm)
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141.3
135.9
135.5
129.8
128.6
128.5
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110.5
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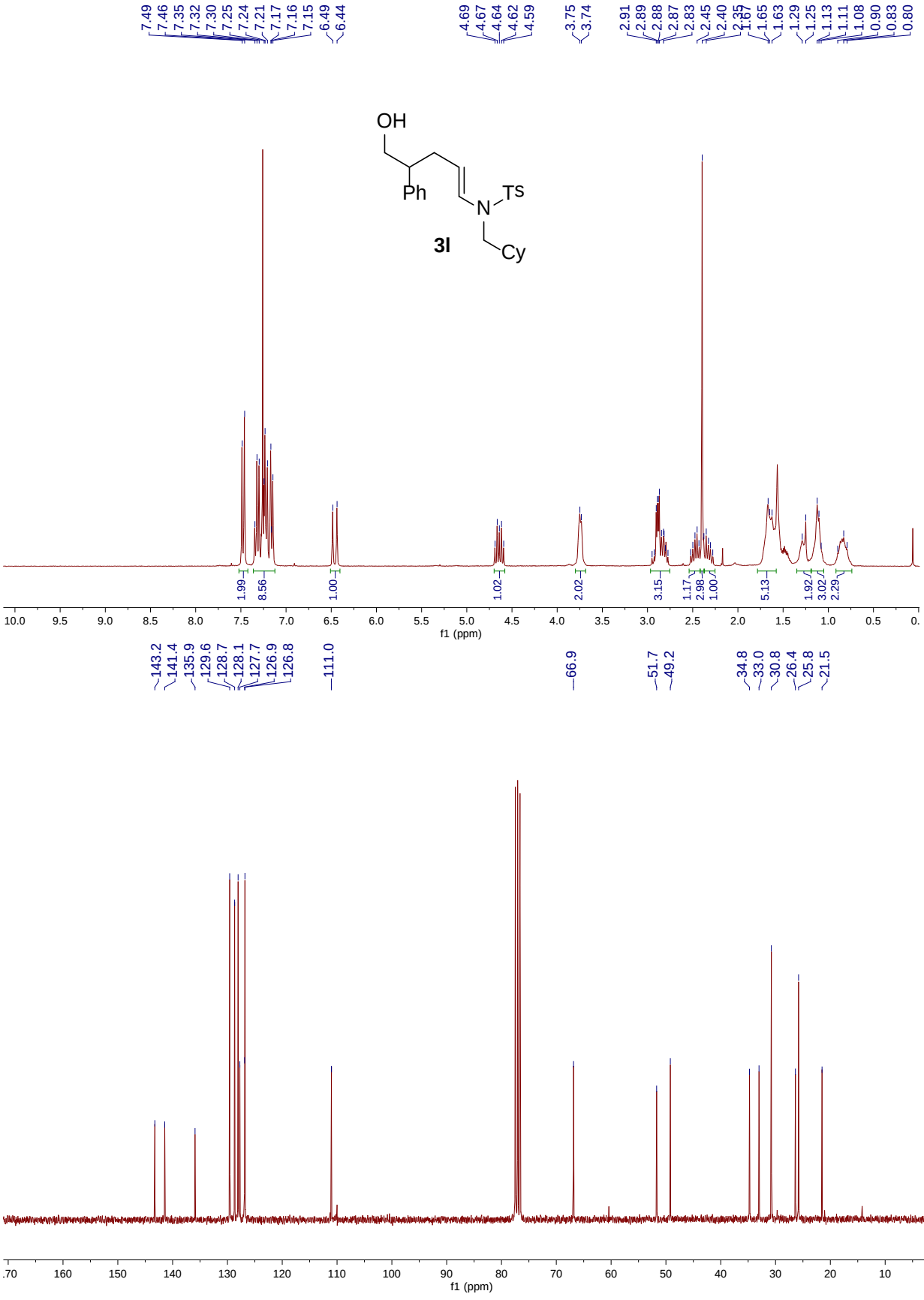
48.8
33.2
21.6
f1 (ppm)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6.44
4.69
4.67
4.64
4.62
4.59
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3.74
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2.89
2.88
2.87
2.83
2.45
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1.11
1.08
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0.80
OH
Ph
N
Ts
Cy
3l
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2.02
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1.17
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1.00
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2.29
f1 (ppm)
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135.9
129.6
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126.8
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34.8
33.0
30.8
26.4
25.8
21.5
f1 (ppm)


7.44
7.42
7.41
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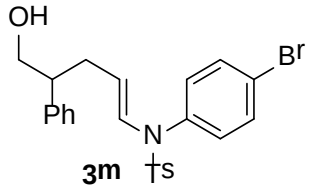
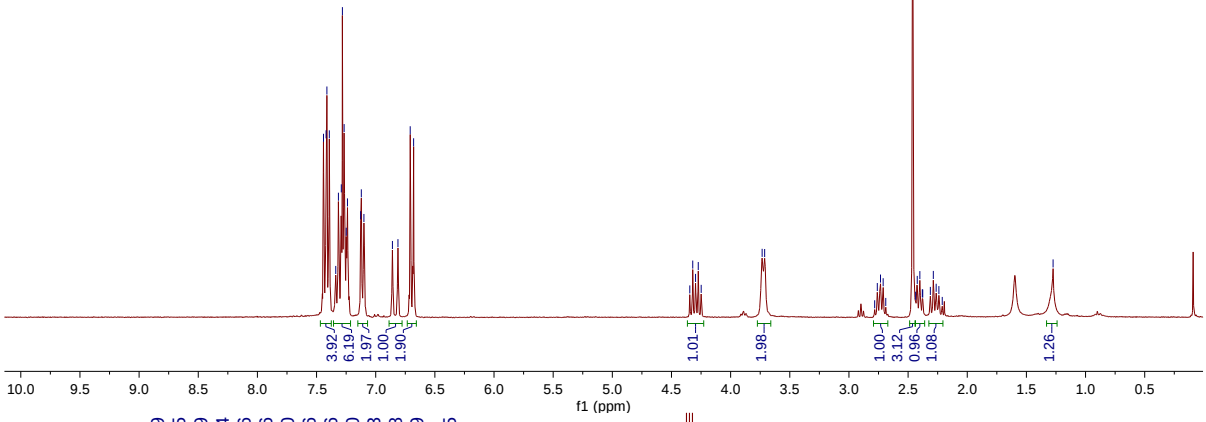
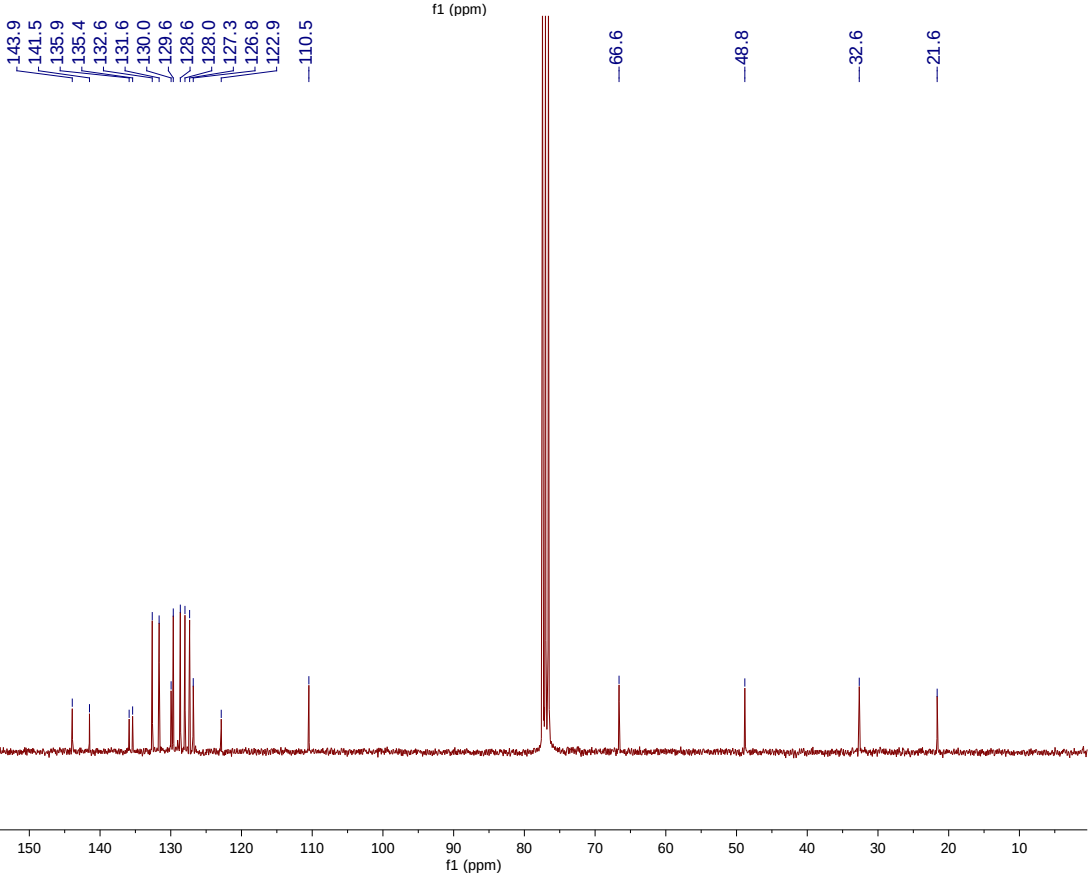
2.38
2.31
2.29
2.26
2.24
1.27


OH
Br
Ph
N
Ts

3m


3.92
6.19
1.97
1.00
1.90
1.01
1.98
1.00
3.12
0.96
1.08
1.26
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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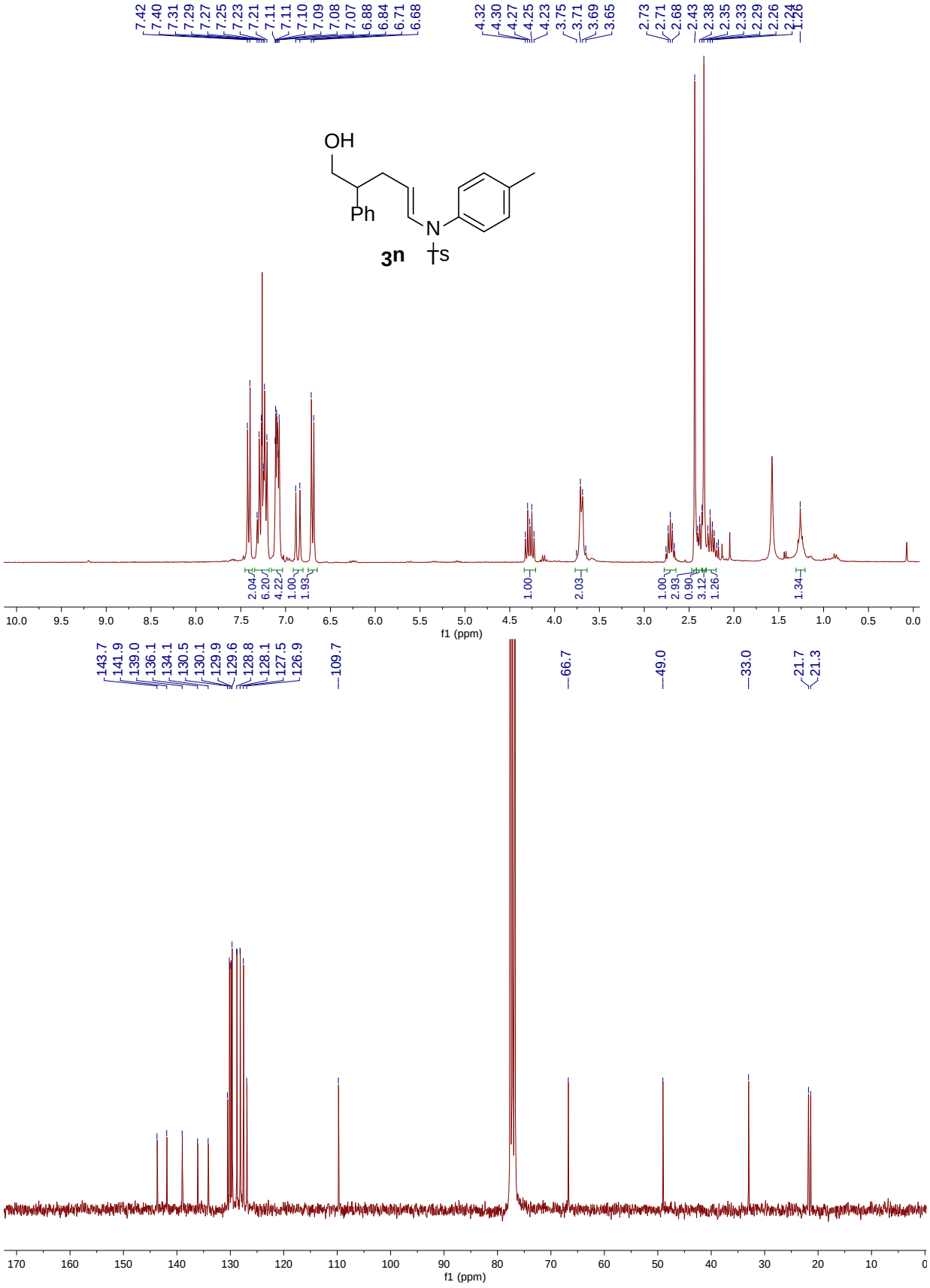
131.6
130.0
129.6
128.6
128.0
127.3
126.8
122.9
110.5
66.6
48.8
32.6
21.6
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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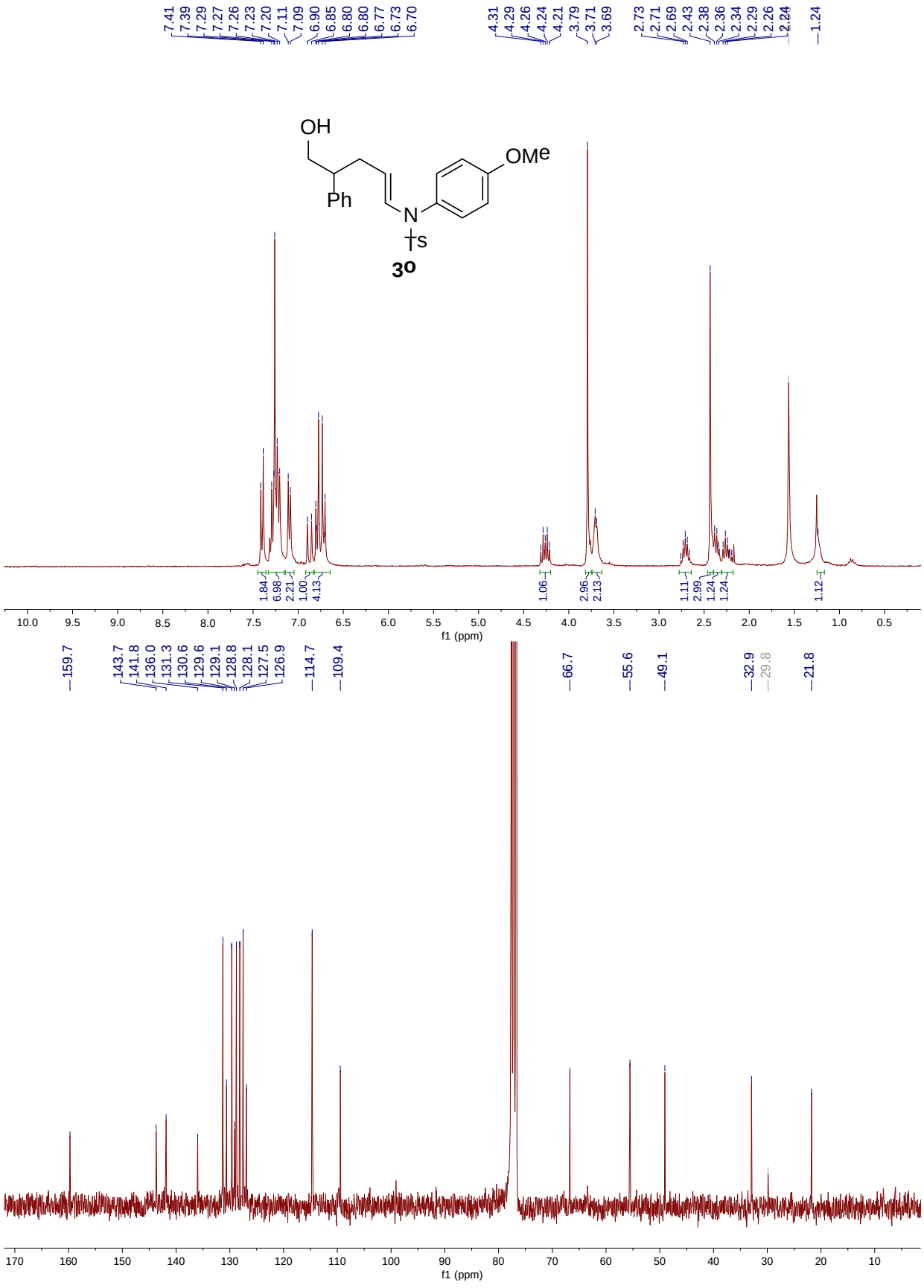
7.23
7.21
7.11
7.11
7.10
7.09
7.08
7.07
6.88
6.84
6.71
6.68
4.32
4.30
4.27
4.25
4.23
3.75
3.71
3.69
3.65
2.73
2.71
2.68
2.43
2.38
2.35
2.33
2.29
2.26
2.24
1.26
OH
Ph
N
Ts
3n
2.04
6.20
4.22
1.00
1.93
1.00
2.03
1.00
2.93
0.90
3.12
1.26
1.34
f1 (ppm)
143.7
141.9
139.0
136.1
134.1
130.5
130.1
129.9
129.6
128.8
128.1
127.5
126.9
109.7
66.7
49.0
33.0
21.7
21.3
f1 (ppm)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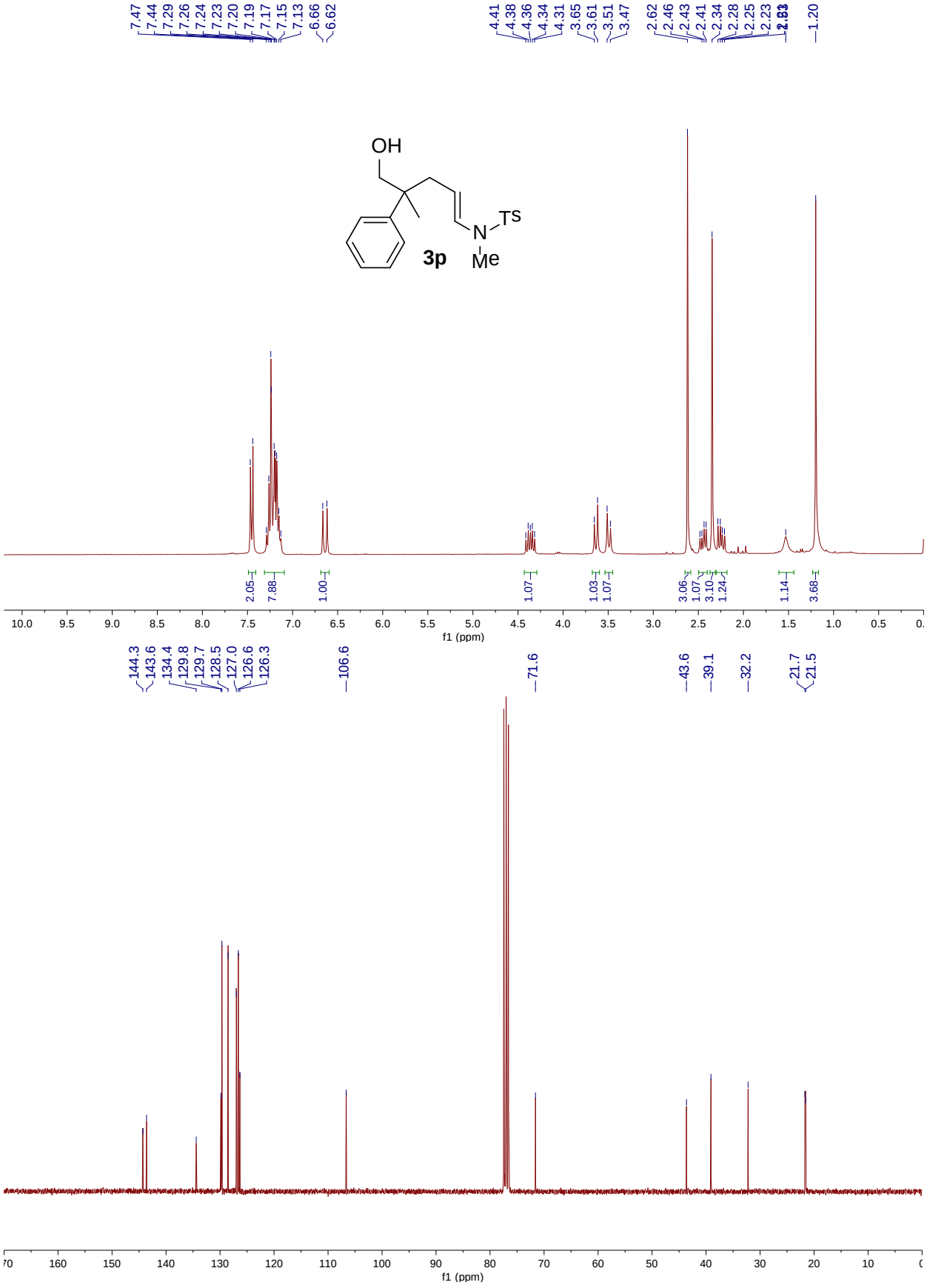
7.39
7.29
7.27
7.26
7.23
7.20
7.11
7.09
6.90
6.85
6.80
6.80
6.77
6.73
6.70
4.31
4.29
4.26
4.24
4.21
3.79
3.71
3.69
2.73
2.71
2.69
2.43
2.38
2.36
2.34
2.29
2.26
2.24
1.24
OH
Ph
N
Ts
OMe
3o
1.84
6.98
2.21
1.00
4.13
1.06
2.96
2.13
1.11
2.99
1.24
1.24
1.12
f1 (ppm)
159.7
143.7
141.8
136.0
131.3
130.6
129.6
129.1
128.8
128.1
127.5
126.9
114.7
109.4
66.7
55.6
49.1
32.9
29.8
21.8
f1 (ppm)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7.24
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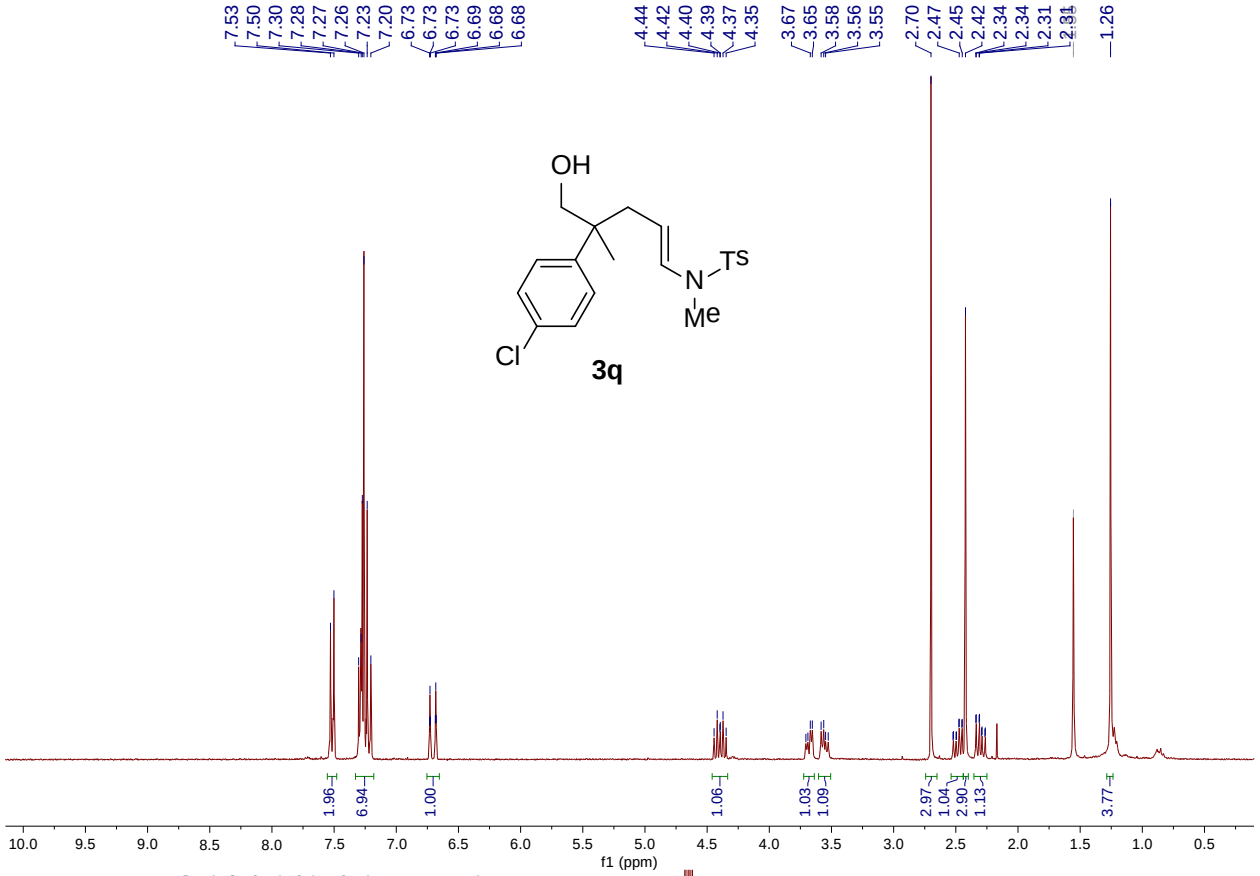
7.20
7.19
7.17
7.15
7.13
6.66
6.62
4.41
4.38
4.36
4.34
4.31
3.65
3.61
3.51
3.47
2.62
2.46
2.43
2.41
2.34
2.28
2.25
2.23
1.20
OH
TS
N
Me
3p
2.05
7.88
1.00
1.07
1.03
1.07
3.06
1.07
3.10
1.24
1.14
3.68
f1 (ppm)
144.3
143.6
134.4
129.8
129.7
128.5
127.0
126.6
126.3
106.6
71.6
43.6
39.1
32.2
21.7
21.5
f1 (ppm)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7.30
7.28
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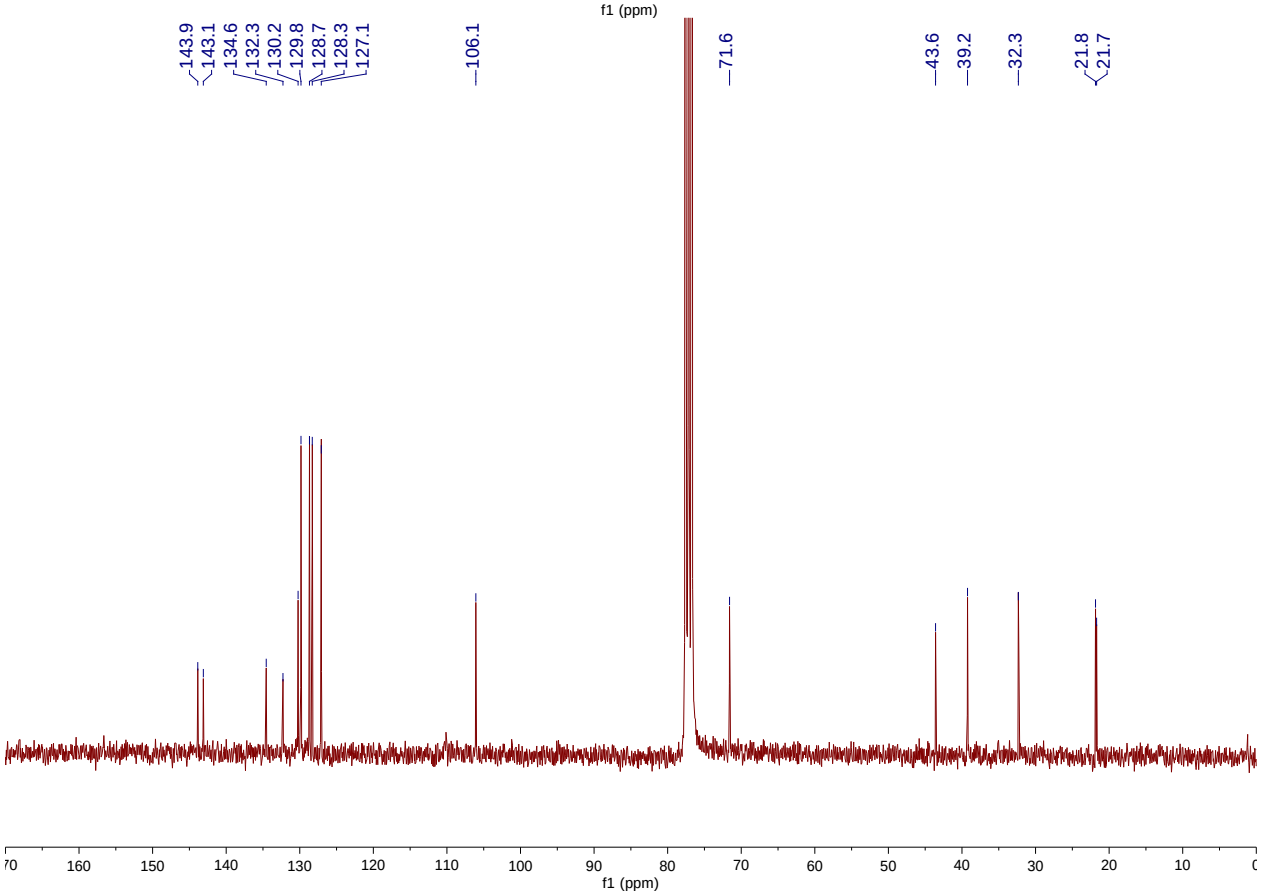
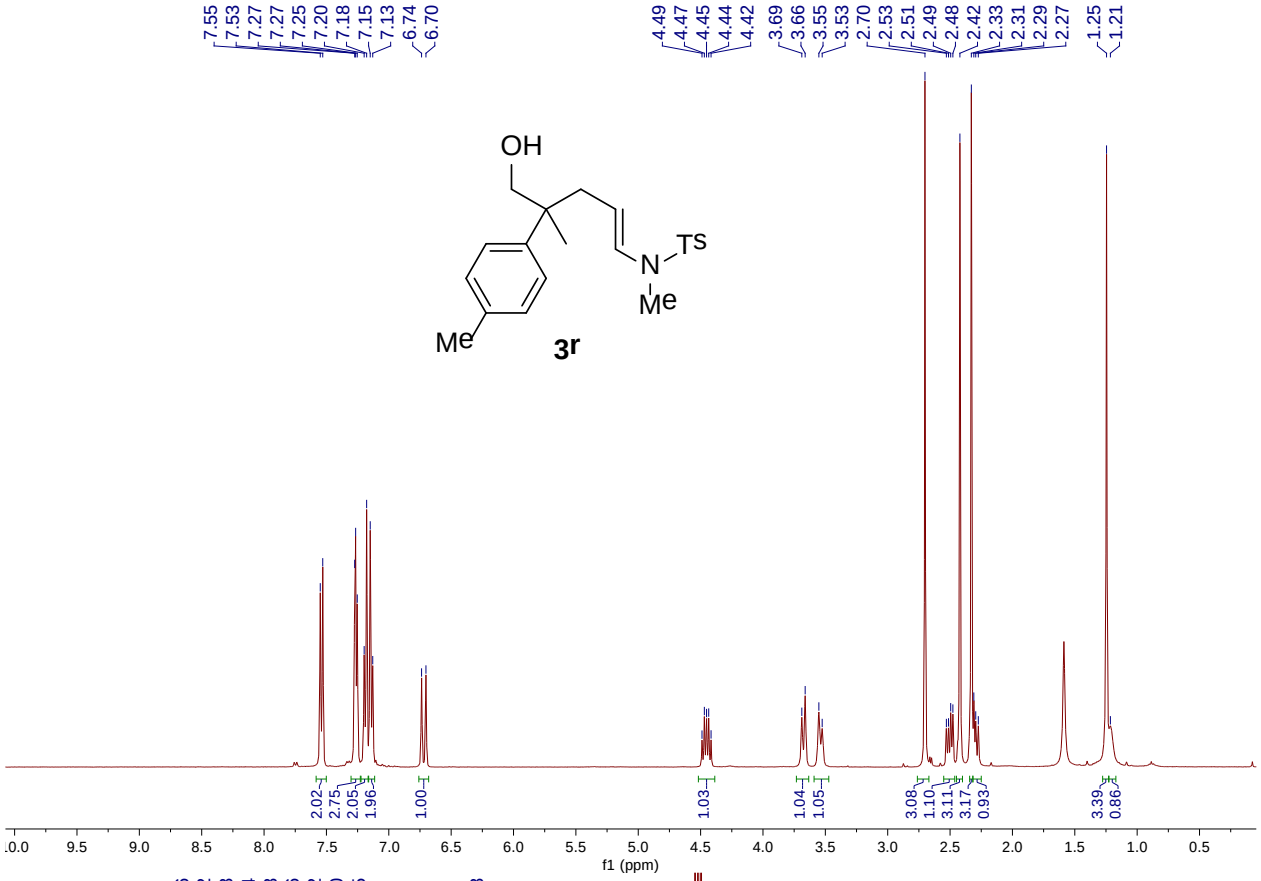
7.26
7.23
7.20
6.73
6.73
6.73
6.69
6.68
6.68
4.44
4.42
4.40
4.39
4.37
4.35
3.67
3.65
3.58
3.56
3.55
2.70
2.47
2.45
2.42
2.34
2.34
2.31
1.26
OH
N
Ts
Me
Cl
3q
1.96
6.94
1.00
1.06
1.03
1.09
2.97
1.04
2.90
1.13
3.77
f1 (ppm)


143.9
143.1
134.6
132.3
130.2
129.8
128.7
128.3
127.1
106.1
71.6
43.6
39.2
32.3
21.8
21.7
f1 (ppm)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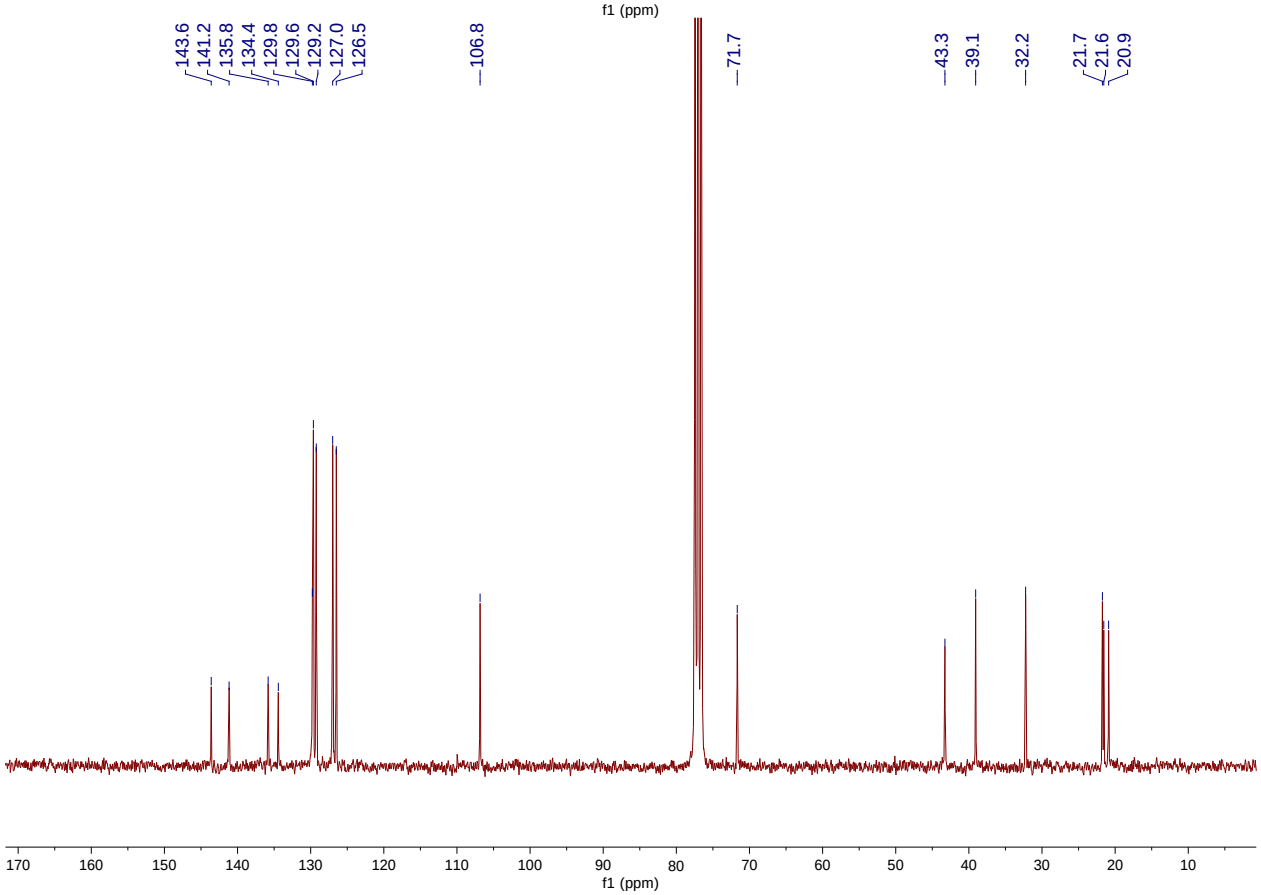
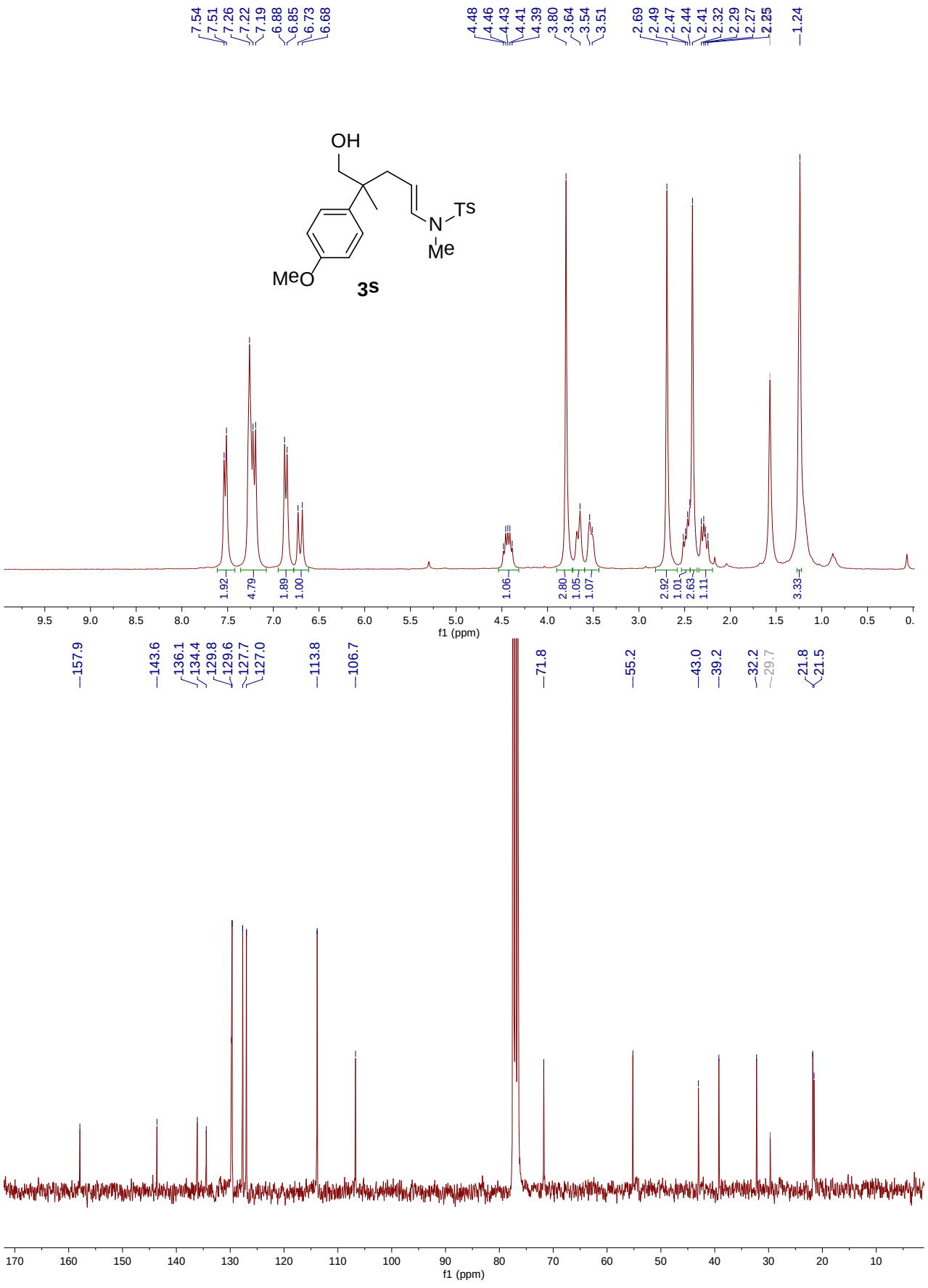
7.27
7.27
7.25
7.20
7.18
7.15
7.13
6.74
6.70
4.49
4.47
4.45
4.44
4.42
3.69
3.66
3.55
3.53
2.70
2.53
2.51
2.49
2.48
2.42
2.33
2.31
2.29
2.27
1.25
1.21
OH
Ts
N
Me
Me
3r
2.02
2.75
2.05
1.96
1.00
1.03
1.04
1.05
3.08
1.10
3.11
3.17
0.93
3.39
0.86
f1 (ppm)


143.6
141.2
135.8
134.4
129.8
129.6
129.2
127.0
126.5
106.8
71.7
43.3
39.1
32.2
21.7
21.6
20.9
f1 (ppm)


7.54
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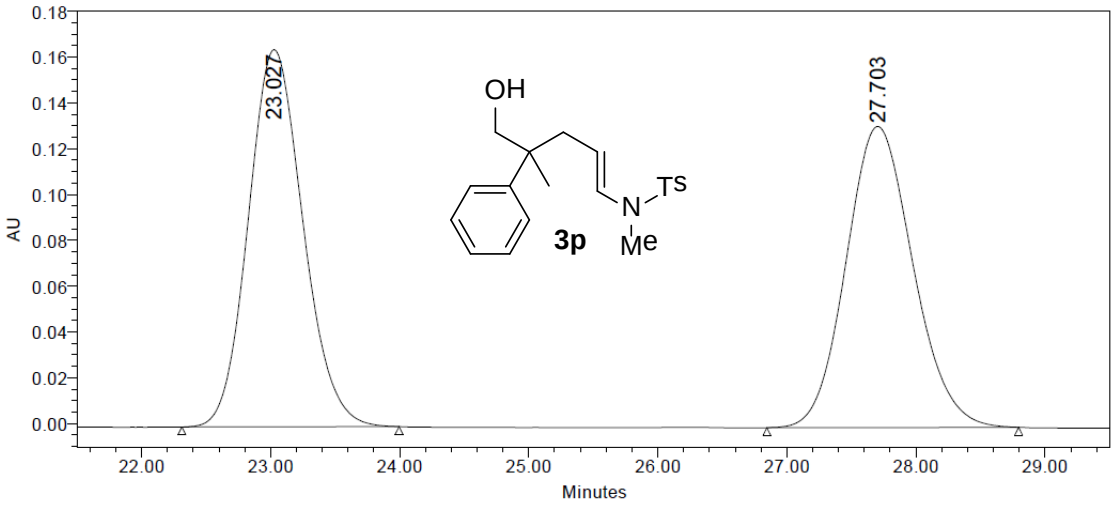
7.26
7.22
7.19
6.88
6.85
6.73
6.68
4.48
4.46
4.43
4.41
4.39
3.80
3.64
3.54
3.51
2.69
2.49
2.47
2.44
2.41
2.32
2.29
2.27
2.25
1.24
OH
TS
N
Me
MeO
3s
1.92
4.79
1.89
1.00
1.06
2.80
1.05
1.07
2.92
1.01
2.63
1.11
3.33
f1 (ppm)
157.9
143.6
136.1
134.4
129.8
129.6
127.7
127.0
113.8
106.7
71.8
55.2
43.0
39.2
32.2
29.7
21.8
21.5
f1 (ppm)

HPLC-Chromatogram

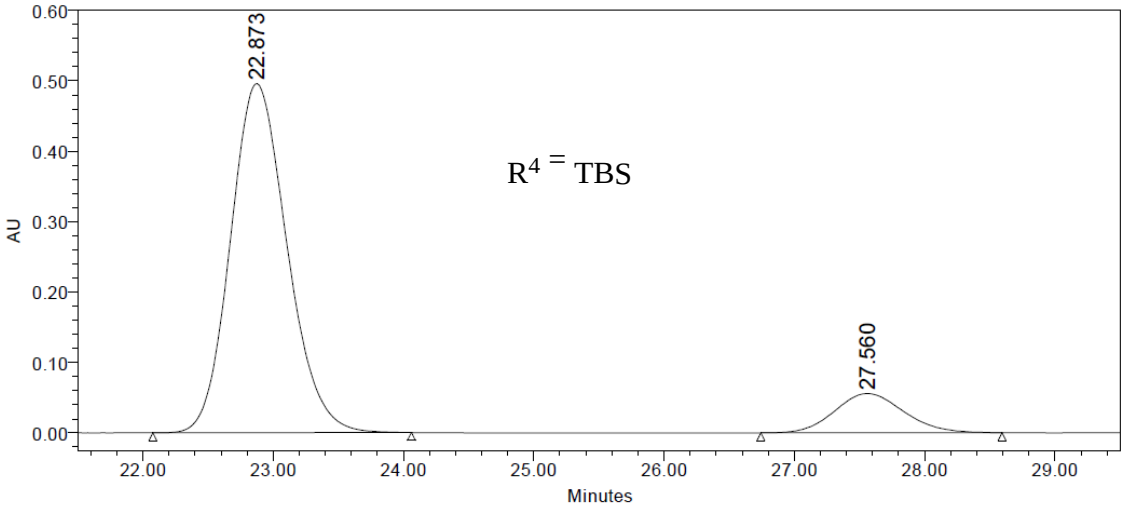
(*E*)-*N*-(5-hydroxy-4-methyl-4-phenylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3p)

CHIRALPAK ADH n-Hexane : IPrOH 80:20, flow 0.6 ml/min (λ250.8 nm)



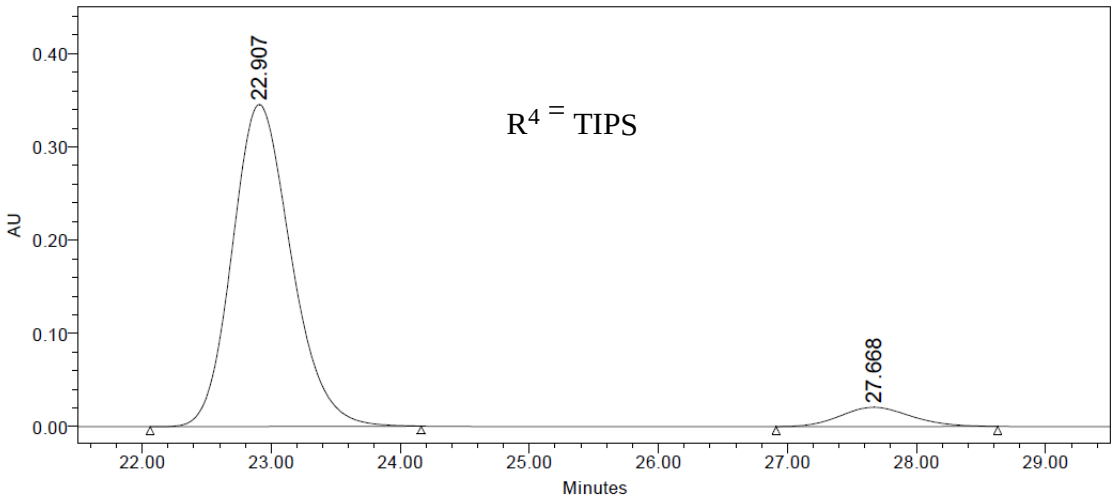
Peak Results

	RT	Area	Height	% Area
1	23.027	4853493	164781	50.65
2	27.703	4729833	131515	49.35



Peak Results

	RT	Area	Height	% Area
1	22.873	14958071	495830	87.93
2	27.560	2052768	55754	12.07

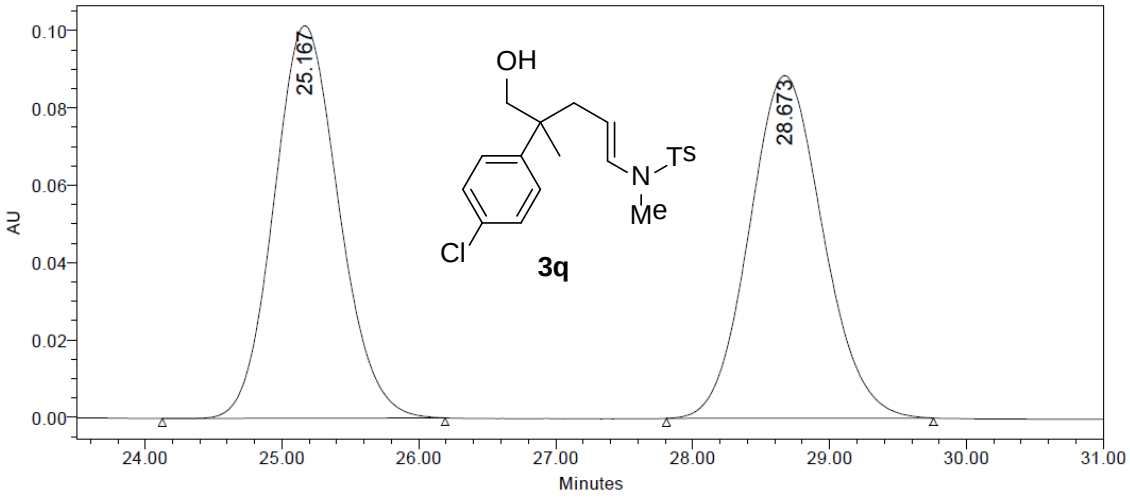


Peak Results

	RT	Area	Height	% Area
1	22.907	10964947	345520	93.30
2	27.668	787076	20608	6.70

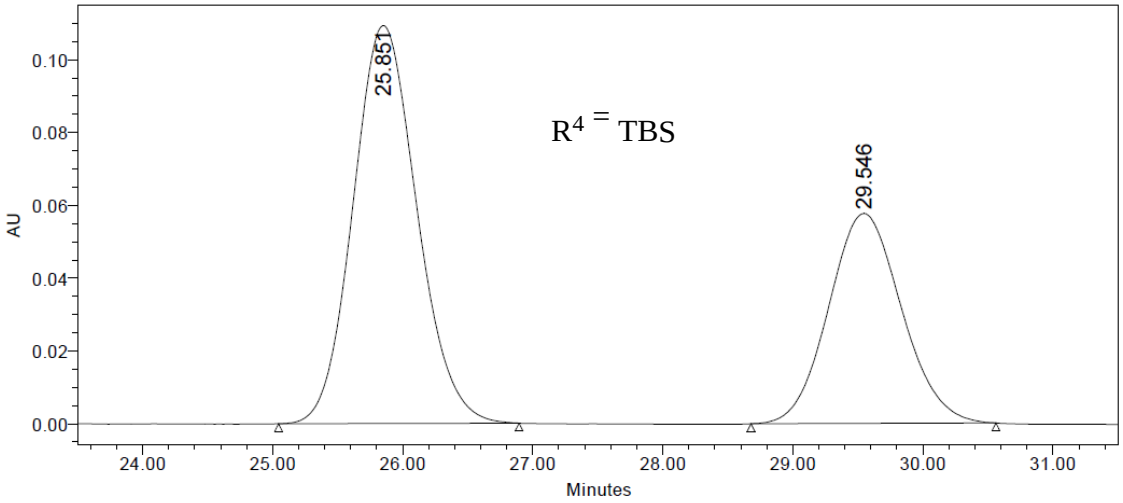
(*E*)-*N*-(4-(4-chlorophenyl)-5-hydroxy-4-methylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3q)

CHIRALPAK ADH n-Hexane : IPrOH 80:20, flow 0.6 ml/min (λ252.0 nm)



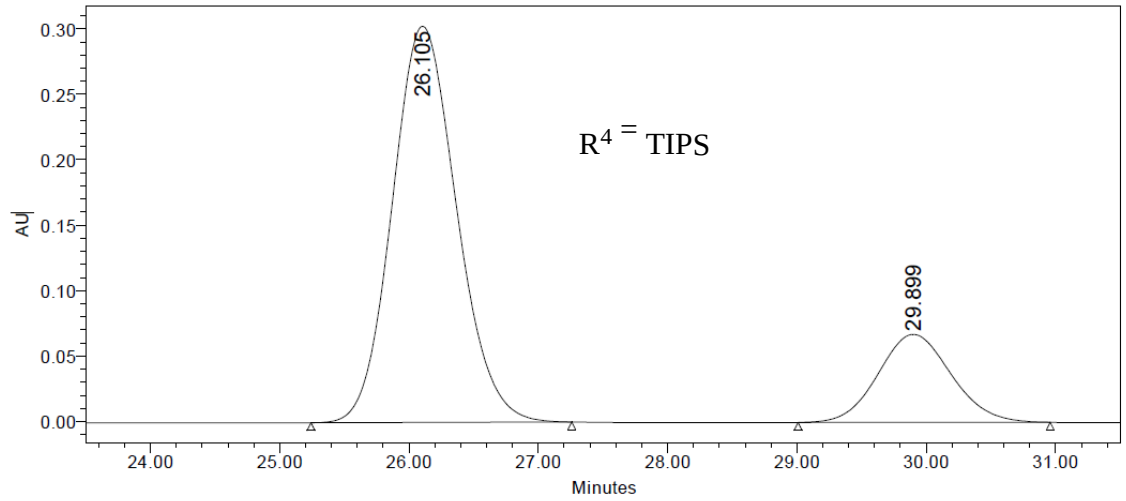
Peak Results

	RT	Area	Height	% Area
1	25.167	3348849	101552	50.17
2	28.673	3325823	88616	49.83



Peak Results

	RT	Area	Height	% Area
1	25.851	3728469	109405	62.38
2	29.546	2248549	57698	37.62

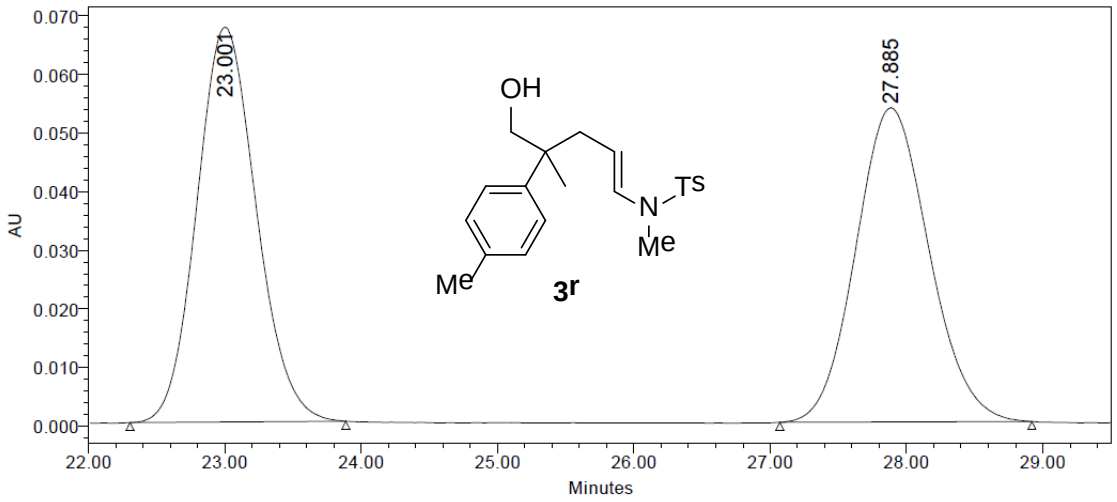


Peak Results

	RT	Area	Height	% Area
1	26.105	10466781	302747	79.90
2	29.899	2632929	67190	20.10

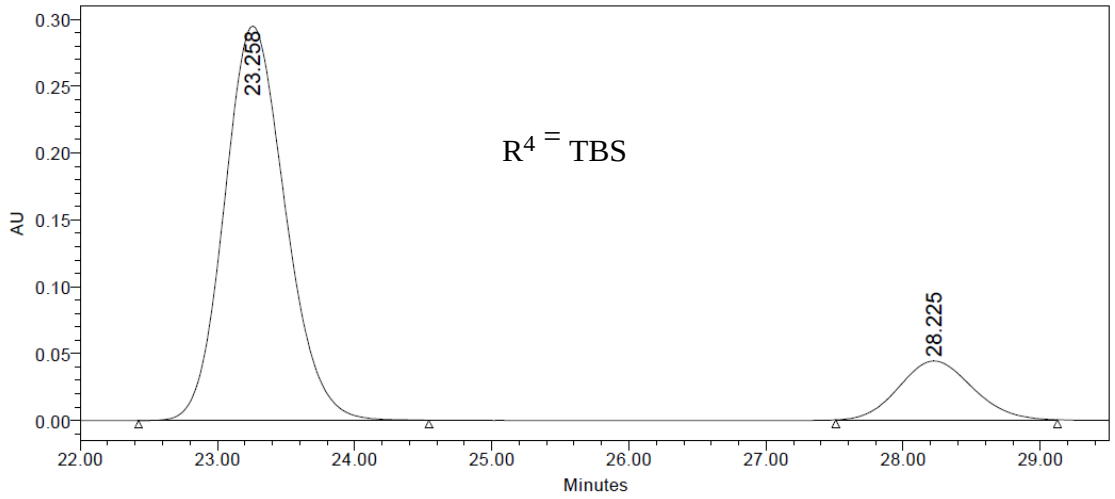
(*E*)-*N*-(5-hydroxy-4-methyl-4-(p-tolyl)pent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3r)

CHIRALPAK ADH n-Hexane : IPrOH 80:20, flow 0.6 ml/min (λ252.0 nm)



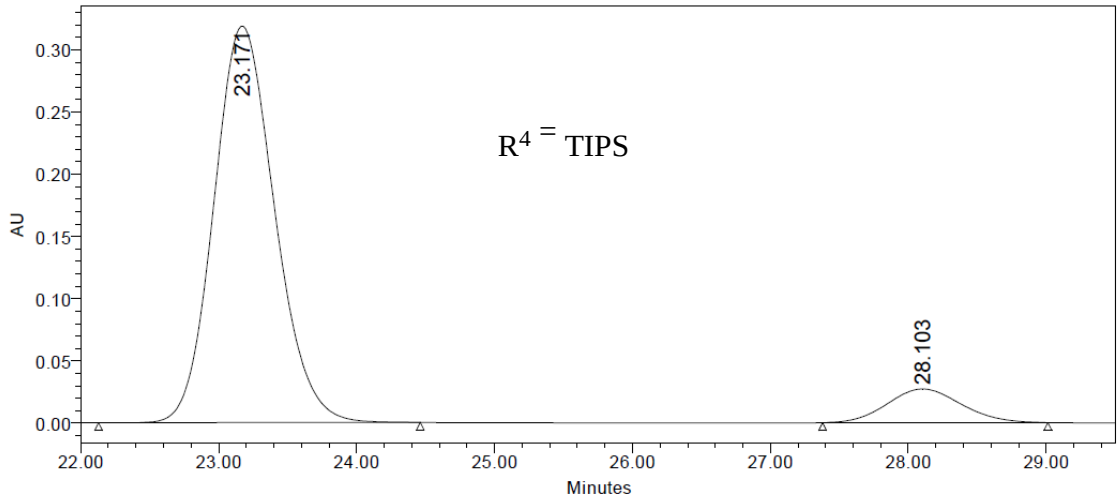
Peak Results

	RT	Area	Height	% Area
1	23.001	2040503	67388	50.44
2	27.885	2004541	53583	49.56



Peak Results

	RT	Area	Height	% Area
1	23.258	9082866	294750	84.76
2	28.225	1632896	43965	15.24

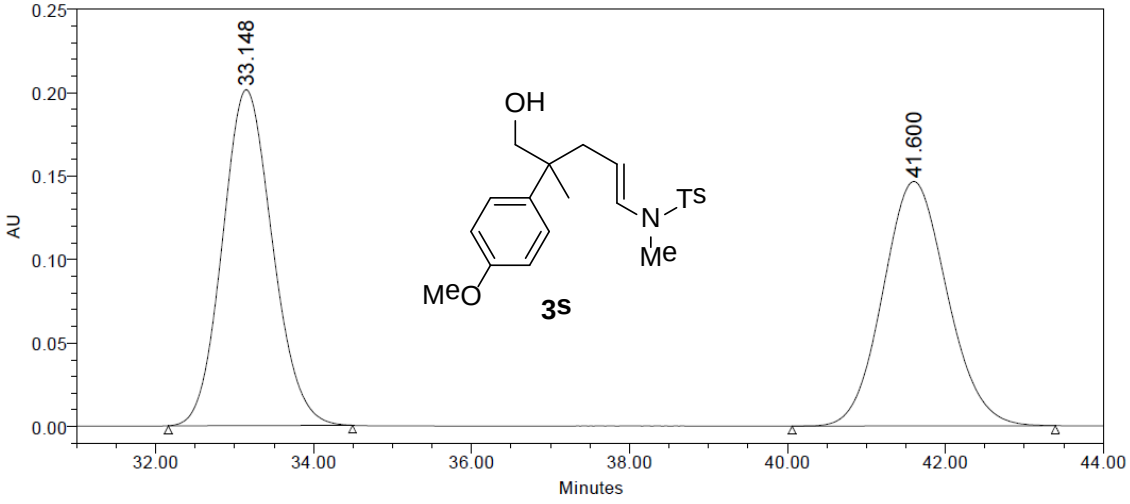


Peak Results

	RT	Area	Height	% Area
1	23.171	9750357	318652	90.66
2	28.103	1004526	27054	9.34

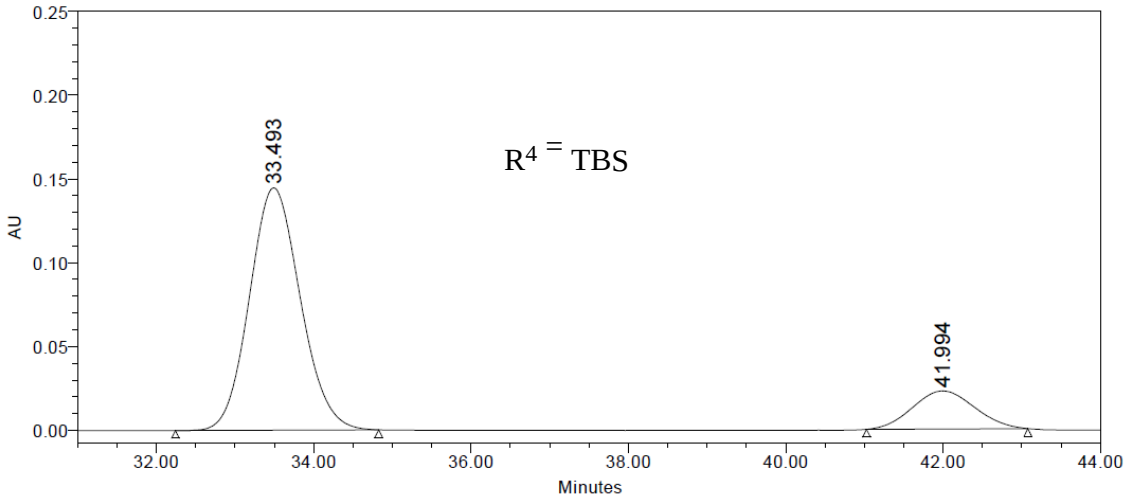
(*E*)-*N*-(5-hydroxy-4-(4-methoxyphenyl)-4-methylpent-1-en-1-yl)-*N*,4-dimethylbenzenesulfonamide (3s)

CHIRALPAK ADH n-Hexane : IPrOH 80:20, flow 0.6 ml/min (λ254.4 nm)



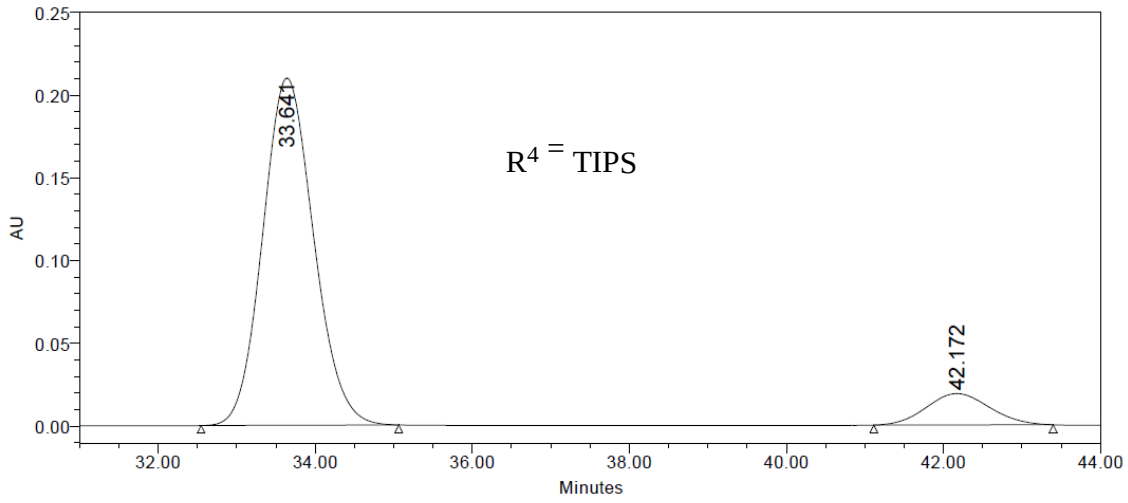
Peak Results

	RT	Area	Height	% Area
1	33.148	8887738	201301	51.77
2	41.600	8278581	146495	48.23



Peak Results

	RT	Area	Height	% Area
1	33.493	6495179	144691	83.93
2	41.994	1243578	22693	16.07



Peak Results

	RT	Area	Height	% Area
1	33.641	9466719	209910	89.90
2	42.172	1063149	18986	10.10