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of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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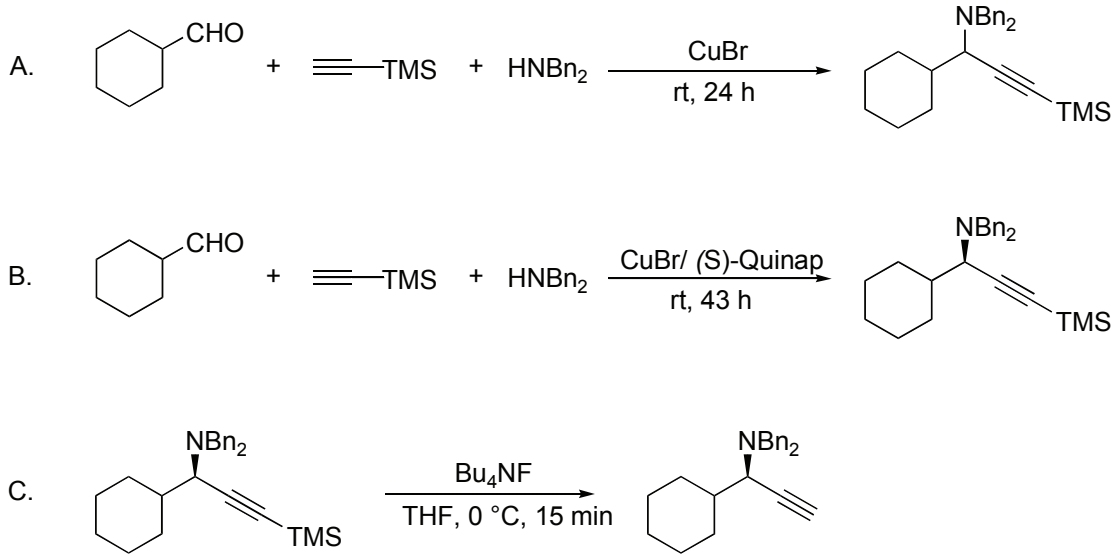
N,N-DIBENZYL-*N*-[1-CYCLOHEXYL-3-(TRIMETHYLSILYL)-2-PROPYNYL]-AMINE FROM CYCLOHEXANECARBALDEHYDE, TRIMETHYLSILYLACETYLENE AND DIBENZYLAMINE



Submitted by Nina Gommermann and Paul Knochel.[1]
Checked by Jason C. Rech and Jonathan A. Ellman.

1. Procedure

A. (±)-N,N-Dibenzyl-N-[1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-amine. A 100-mL Schlenk flask equipped with a magnetic stirring bar and a rubber septum is flame-dried under vacuum (5 x 10^{-2} mbar), cooled to rt, flushed with argon and charged with copper(I) bromide (143 mg, 1.0 mmol) (Note 1). The flask is evacuated (5 x 10^{-2} mbar) for 15 min, flushed with argon and charged with dry toluene (40 mL) (Note 2) giving a white suspension. Molecular sieves 4 Å (10 g) (Note 3) is added under a flow of argon. Trimethylsilylacetylene (1.96 g, 2.85 mL, 20.0 mmol) (Note 4) followed by cyclohexanecarbaldehyde (2.24 g, 2.42 mL, 20.0 mmol) (Note 5) are added via syringe (Note 6) in one portion. Dibenzylamine (3.95 g, 3.87 mL, 20.0 mmol) (Note 7) is added during 5 min via syringe. During the addition, the solution becomes clear and turns green at the end of addition. The reaction is slightly exothermic during the addition of dibenzylamine, and subsequently turns milky again. The reaction mixture is stirred for 24 h. After this time, GC-analysis (Note 8) shows more than 96% conversion to

the desired product. The solution is filtered (Note 9), and the molecular sieves 4 Å are washed with diethyl ether (150 mL). The solution is concentrated by rotary evaporation (55 °C, 760 mmHg, then 38 mmHg). The resulting light green oil is purified by column chromatography (Note 10) affording *N,N*-dibenzyl-*N*-[1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-amine (6.86 g, 17.6 mmol) as a clear, colorless oil which solidifies upon standing at rt (88%) (Note 11).

B. (R)-(+)-N,N-Dibenzyl-N-[1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-amine. A 25-mL Schlenk flask equipped with a magnetic stirring bar and a rubber septum is flame-dried under vacuum (5 x 10^{-2} mbar), cooled to rt, flushed with argon and charged with copper(I) bromide (21.5 mg, 0.150 mmol) (Note 1) and (*S*)-Quinap (72.5 mg, 0.165 mmol) (Note 12). The flask is evacuated (5 x 10^{-2} mbar) for 15 min, flushed with argon and charged with dry toluene (9 mL) (Note 2) giving a yellow solution with white particles. The mixture is stirred for 30 min at rt, during this time, the solution turns milky. Molecular sieves 4 Å (1.5 g) (Note 3) are added under a flow of argon. Trimethylsilylacetylene (295 mg, 0.43 mL, 3.0 mmol) (Note 4) followed by cyclohexanecarbaldehyde (337 mg, 0.36 mL, 3.0 mmol) (Note 5) are added via syringe (Note 6) in one portion. Dibenzylamine (592 mg, 0.58 mL, 3.0 mmol) (Note 7) is added during one minute via syringe. The suspension is stirred for 43 h at rt, after which time GC-analysis (Note 8) shows more than 96% conversion to the desired product. The solution is filtered (Note 9) and the molecular sieves 4 Å are washed with diethyl ether (50 mL). The solution is concentrated by rotary evaporation (55 °C, 760 mmHg, then 38 mmHg). The resulting yellow oil is dissolved in pentane:diethyl ether (99:1) and filtered from the precipitated catalyst (Note 13). The clear filtrate is subjected to column chromatographic purification (Note 14) affording (*R*)-(+)-*N*,*N*-dibenzyl-*N*-[1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-amine (1.07 g, 2.7 mmol) as a clear, colorless oil which solidifies upon standing at rt (91%, 95 % ee) (Note 15).

C. (R)-(+)-N,N-Dibenzyl-N-(1-cyclohexyl-2-propynyl)-amine. (*R*)-(+)-*N*,*N*-Dibenzyl-*N*-[1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-amine (390 mg, 1.00 mmol), placed in a 25-mL round-bottomed flask equipped with a magnetic stirring bar and a rubber septum, is dissolved in dry THF (5 mL) (Note 16). The solution is cooled to 0 °C for 10 min and Bu_4NF (0.30 mmol, 0.30 mL, 1 M solution in THF) (Note 17, 18) is added dropwise. During the addition, the solution turns slightly yellow. After 15 min, GC analysis (Note 8) shows full conversion. The reaction mixture is quenched with water (30

mL), and the aqueous phase is extracted with diethyl ether (3 x 40 mL). The combined organic fractions are dried over Na_2SO_4 (Note 19) and concentrated by rotary evaporation (55 °C, 760 mmHg, then 38 mmHg). The resulting light yellow oil is purified by column chromatography (Note 20) affording *N*,*N*-dibenzyl-N-(1-cyclohexyl-2-propynyl)-amine (307 mg, 0.97 mmol) as a clear, colorless oil. (97%) (Note 21).

2. Notes

1. CuBr was prepared as described by Taylor:[2] $CuBr_2$ (5.00 g, 22.4 mmol) was placed in a 100-mL round bottomed flask equipped with a magnetic stirring bar and dissolved in water (5 mL). A freshly prepared solution of Na_2SO_3 (3.80 g, 30.1 mmol) in water (25 mL) was added slowly via an addition funnel during 10 min. The initially dark brown mixture turned white and rapidly deposited a white precipitate of CuBr. After the addition was complete, the mixture was stirred for an additional 15 min. Afterwards, the suspension was poured into water (400 mL) which contained Na_2SO_3 (0.50 g) and concentrated hydrochloric acid (1 mL). The mixture was stirred vigorously for 10 min, after which the CuBr was allowed to settle. The supernatant liquid was decanted and the precipitate was transferred to a sintered-glass filter (pore size P4; 75 mL) with diluted sulfuric acid (100 mL, 0.1 M) to prevent oxidation. For the transfer, as well as for the following washing steps, it was ensured that liquid covered the precipitate at all times. With application of gentle suction (375 mmHg), the precipitate was washed with glacial acetic acid (4 x 15 mL), absolute ethanol (3 x 15 mL) and anhydrous diethyl ether (6 x 10 mL). The white crystalline CuBr was immediately transferred to a 50-mL Schlenk flask and dried under vacuum (4×10^{-2} mmHg). CuBr (3.08 g, 21.5 mmol, 96%) was obtained as a white solid and stored under argon, but the solid can be handled routinely in air.

2. Toluene was dried by alumina column (checkers) or dried by distillation under argon from sodium (submitters).

3. Molecular sieves 4 Å, 8 to 12 mesh, were obtained from Acros Organics and used as obtained.

4. Trimethylsilylacetylene was purchased from Lancaster (checkers) or obtained as a generous gift from Wacker Chemie GmbH, Burghausen (submitters), and used as obtained.

5. Cyclohexanecarbaldehyde (98%) was obtained from Aldrich and freshly distilled under reduced pressure (24 mmHg) before use. Reduced yields (65–80%), but not reduced enantioselectivities, were observed when aged starting materials were employed in this procedure.

6. Although the liquid starting materials were transferred via syringe, weighing of the compounds in the syringes instead of measuring the volumes was found to be advantageous. Especially for the smaller scale of the reaction in presence of the chiral ligand (Procedure B), yields were found to be more reliable when the amounts of starting materials were determined by weighing. Therefore, the use of the given mass-values instead of volumes is strongly recommended.

7. Dibenzylamine (98%) was obtained from Acros Organics and freshly distilled under reduced pressure (0.04 mmHg) before use.

8. GC-analysis was carried out using a Hewlett&Packard 5890 Series 2 machine equipped with a HP Ultra-2.5 %-phenylmethylpolysiloxane column (12m x 0.2 mm x 0.33 μm). Oven program for GC-Analysis: Starting temperature, 90 °C for 1 min; heating to 250 °C by a rate of 50 °C per min; 8 min at 250 °C. Retention time of *N*,*N*-Dibenzyl-*N*-[1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-amine: 7.34 min. The reaction was monitored by the disappearance of the enamine (retention time: 5.83 min).

9. A glass filter with pore size P3 and a volume of 75 mL was used. Filtration was carried out under reduced pressure (300 mmHg).

10. The oil is taken up in 20 mL of the solvent mixture (pentane/diethyl ether, 99:1) and applied to a 5-cm diameter column packed with 200 g Merck silica gel 60 mesh (0.063–0.200 mm), R_f= 0.35 (TLC, aluminum sheets, silica gel 60 F254, obtained from Merck). Approximately 1.5 L of the solvent mixture is used.

11. The submitters reported yields ranging from 6.97–7.01g (88–89%). Spectral data are as follows: checkers mp 68–70 °C, submitters mp 81–82 °C; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.28 (s, 9 H). 0.67–0.89 (m, 2 H), 1.01–1.28 (m, 3 H), 1.52–1.71 (m, 4 H), 1.96 (m, 1H), 2.27 (m, 1 H), 3.01 (d, *J* = 10.5 Hz, 1 H), 3.34 (d, *J* = 13.8 Hz, 2 H), 3.78 (d, *J* = 13.8 Hz, 2 H), 7.19–7.23 (m, 2 H), 7.26–7.34 (m, 4 H), 7.37–7.45 (m, 4 H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 0.5, 25.9, 26.1, 26.6, 30.2, 31.2, 39.4, 54.8, 58.5, 90.1, 103.5, 126.8, 128.2, 128.8, 139.8. MS (70 eV, EI): 307 (27), 306 (M^+–c-Hex, 100), 91 (34). HRMS (EI): Calcd. for $C_{26}H_{36}NSi$ [M+H]: 390.2617, found: 390.2628. IR (film): 2924, 2850, 2160, 1494, 1450, 1249, 1004, 841, 746,

698 cm^{-1}. Anal Calcd. for $C_{26}H_{35}NSi$: C: 80.14, H: 9.05, N: 3.59, Found: C: 79.98, H: 8.82, N: 3.79.

12. (S)-Quinap was obtained from Strem Chemicals, Inc. and used as obtained.

13. The oil was mixed with approximately 5 mL of the solvent mixture (pentane/diethyl ether, 99:1) and a light yellow precipitate was formed. The solid was collected by filtration and washed with the solvent mixture (10 mL).

14. Column chromatography was carried out on a 2.5-cm diameter column packed with 50 g Merck silica gel 60 mesh (0.063–0.200 mm) using pentane/diethyl ether, 99:1 as eluent, (R_f = 0.35, TLC aluminum sheets, Silica gel 60 F254, obtained from Merck). Approximately 0.5 L of the solvent mixture is used.

15. The submitters report yields in the range of 1.02–1.04g (88–89%, 95% ee). The enantiomeric excess was determined by HPLC analysis using a Chiracel OD-H column and n-heptane as the eluent after deprotection to form the desilylated derivative (see procedure C), retention times: 36.6 min (*S*), 41.9 min (*R*) (submitters). The enantiomeric excess can also be determined using a Chiracel OD column with hexane as the eluent, retention times: 7.45 min (*S*), 7.89 min (*R*) (checkers). Analytical data: $[\alpha]_D^{20}$ = +168 (c = 1.00, $CHCl_3$) (checkers). The purity of the product was established by ^{1}H-NMR analysis to be >98 %, showing the same data as reported in Note 10.

16. THF was dried by alumina column (checkers) or by distillation from sodium/benzophenone under nitrogen (submitters).

17. Bu_4NF was obtained as a 1M solution in THF from Alfa Aesar and used as obtained.

18. The use of only 0.4 equiv. of Bu_4NF for the cleavage of a TMS-group from acetylenes has been reported by Nishikawa.[3]

19. Na_2SO_4 was obtained from Acros Organics and used as obtained.

20. Column chromatography was carried out on a 2.5-cm diameter column packed with 20 g Merck silica gel 60 mesh (0.063–0.200 mm) using pentane/diethyl ether, 99:1 as eluent, (R_f= 0.30, TLC aluminum sheets, silica gel 60 F254, obtained from Merck).

21. The submitters reported a yield of 307 mg (99%). Spectral data as follows: $[\alpha]_D^{20}$ = +135 (c = 1.10, $CHCl_3$). ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 0.67–0.89 (m, 2 H), 1.01–1.28 (m, 3 H), 1.52–1.71 (m 4 H), 2.00 (m, 1 H),

2.29 (m, 1 H), 2.34 (d, J = 2.4 Hz, 1 H), 3.03 (dd, J = 10.5, 2.1 Hz, 1 H), 3.37 (d, J = 13.5 Hz, 2 H), 3.81 (d, J = 13.5 Hz, 2 H), 7.18-7.25 (m, 2 H), 7.26–7.35 (m, 4 H), 7.36–7.45 (m, 4 H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 25.9, 26.1, 26.5, 30.2, 31.2, 39.5, 54.8, 57.6, 73.4, 81.0, 126.8, 128.2, 128.8, 139.7. MS (70 eV, EI): 235 (21), 234 (M+–c-Hex, 100), 91 (88). HRMS (EI): Calcd. for $C_{23}H_{28}N$ [M+H]: 318.2222, found: 318.2222. IR (film): 3300, 2923, 2850, 1490, 1450, 745, 698 cm^{-1}. Anal Calcd. for $C_{23}H_{27}N$: C: 87.02, H: 8.57, N: 4.41, Found: C: 86.78, H: 8.63, N: 4.55. The checkers observed that the colorless oil solidifies upon standing: mp 75–76 °C.

Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

The catalytic, enantioselective formation of new carbon-carbon bonds is an important class of reactions. Especially attractive are multi-component reactions that allow the formation of several bonds including new carbon-carbon bonds in a one-pot procedure. The present procedure describes a convenient method for the preparation of enantiomerically enriched propargylamines in good selectivities and excellent yields.[4] The reaction is catalyzed by the chiral complex formed from Cu(I)Br and commercially available (*R*)- or (*S*)-Quinap[5] (5 mol%). The reaction tolerates a broad range of different aldehydes, including aromatic and heteroaromatic aldehydes, as well as branched and non-branched aliphatic aldehydes. Many types of terminal alkynes are tolerated, but the use of trimethylsilylacetylene provides the best enantiomeric excesses. Furthermore, desilylation to the terminal alkyne potentiates many different transformations (Scheme 1). As the third component, only aliphatic amines can be used; amides or anilines do not undergo the reaction. A major advantage of this reaction is the convenience of the procedure. The reaction is carried out at room temperature with equimolar amounts of starting materials and only 5 mol% of catalyst is necessary, which demonstrates a very atom economical reaction.[6] The propargylamines obtained by this procedure are valuable building blocks for organic synthesis. A related synthesis involving imines

that uses CuOTf/pybox as the catalytic system is also known, but the reaction is limited to aromatic aldehydes and anilines.[7]

Scheme 1. Transformations of terminal propargylamines.

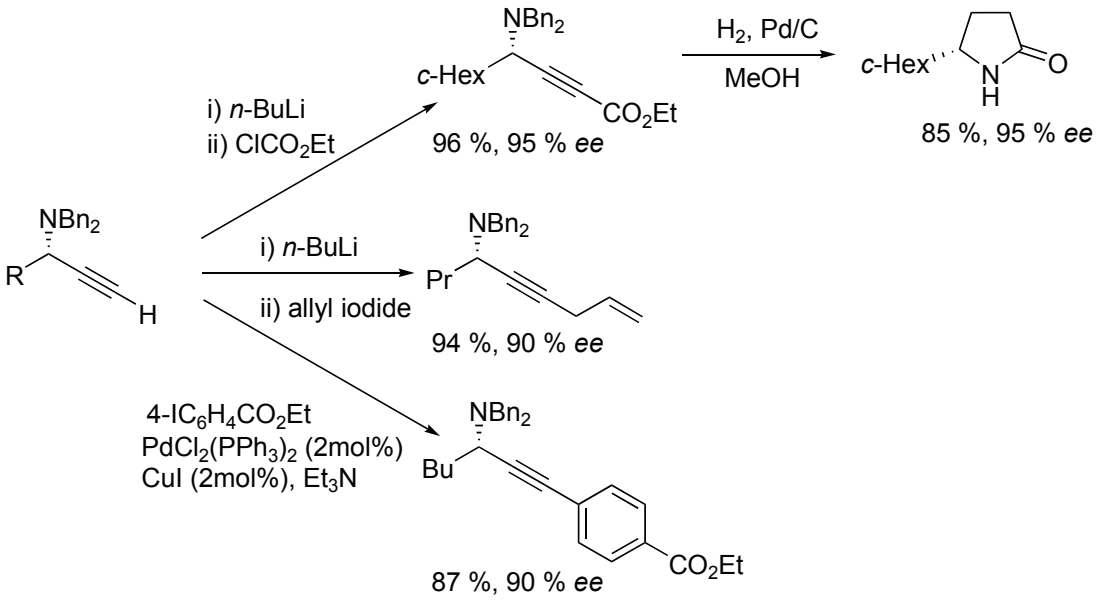


Table 1: Enantiomerically enriched propargylamines obtained by the Cu(I)-Quinap catalyzed three-component coupling of aldehydes, secondary amines and terminal alkynes.

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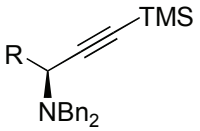
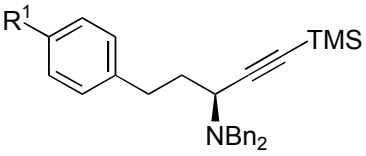
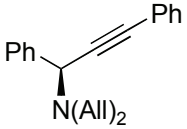
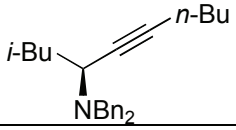
				
1	*n*-Pr	R= n-Pr	88	90
2	*n*-Bu	R= n-Bu	75	90
3	*i*-Bu	R= i-Bu	84	94
4	*i*-Pr	R= i-Pr	84	96
5	*s*-Pent	R= s-Pent	97	98
6	*c*-Pr	R= c-Pr	97	92
7	*c*-Pent	R= c-Pent	97	96
8	3-Benzothiophene	R=3-Benzothiophene	92	82
9	$(C_6H_5)CH=CH$	R= $(C_6H_5)CH=CH$	93	82
10	$(C_6H_5)_2C=CH$	R= $(C_6H_5)_2C=CH$	80	84
				
11	$C_6H_5(CH_2)_2$	R^1= H	76	88
12	4-Br-$C_6H_4(CH_2)_2$	R^1= Br	72	88
13	4-CO_2Et-$C_6H_4(CH_2)_2$	R^1= CO_2Et	83	87
14	Ph		91	70
15	*i*-Bu		85	82

[*a*] Isolated yield of analytically pure product; [*b*] Enantiomeric excess determined by HPLC using Chiracel OD-H column (*n*-heptane : *i*-PrOH).

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7. Wei, C.; Li, C.-J. *J. Am. Chem. Soc.* **2002**, *124*, 5638.

Appendix
Chemical Abstracts Nomenclature; (Registry Number)

Trimethylsilylacetylene: Silane, ethynyltrimethyl-; (1066-54-2)
Cyclohexanecarbaldehyde: Cyclohexanecarboxaldehyde; (2043-61-0)
Dibenzylamine: Benzenemethanamine, *N*-(phenylmethyl)-; (103-49-1)
Copper(I) bromide; (7787-70-4)
(*S*)-Quinap: Isoquinoline, 1-[2-(diphenylphosphino)-1-naphthalenyl]-, (1*S*)-; (149341-33-3)
(*R*)-(+)-*N*,*N*-Dibenzyl-*N*-[1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-amine: Benzenemethanamine, *N*-[(1*R*)-1-cyclohexyl-3-(trimethylsilyl)-2-propynyl]-*N*-(phenylmethyl)-; (872357-80-7)
Tetra-*n*-butylammonium fluoride: 1-Butanaminium, *N,N,N*-tributyl-, fluoride; (429-41-4)
N,*N*-Dibenzyl-N-(1-cyclohexyl-2-propynyl)-amine: Benzenemethanamine, *N*-[(1*R*)-1-cyclohexyl-2-propynyl]-*N*-(phenylmethyl)-; (872357-86-3)

Paul Knochel was born in 1955 in Strasbourg, France. He completed his undergraduate studies at the University of Strasbourg and his Ph.D. at the ETH Zurich with D. Seebach. He spent 4 years with Prof. J.-F. Normant (Paris) and 1 year with Prof. M. F. Semmelhack (Princeton) as a postdoctoral researcher. After professorships at the University of Michigan and the Philipps-Universität (Marburg), he moved to the Ludwig-Maximilians-Universität (Munich) in 1999. His research interests include the development of new synthetic methods with organometallic reagents, new asymmetric catalysts and natural product synthesis.

Nina Gommermann was born in 1978 in Kassel, Germany. After undergraduate studies at the Philipps-Universität in Marburg and the Ludwig-Maximilans-Universität (Munich), she joined the group of Prof. Knochel in 2002. She obtained her Ph.D. in 2005 on the enantioselective synthesis of propargylamines and their application in organic synthesis. Since November 2005, she has been a postdoctoral scholar (DAAD fellow) in the group of Prof. David MacMillan at the California Institute of Technology on natural product synthesis.

Jason Rech (born 1977) graduated from Allegheny College with a B.S. degree in chemistry in 1999 where he worked in the laboratory of Professor Martin J. Serra. He worked for the Bayer Corporation in polymer research until entering the Ph.D. program at the University of Pittsburgh in 2000. Under the guidance of Professor Paul E. Floreancig he earned his Ph.D. in 2005 and is currently pursuing postdoctoral research with Professor Jonathan A. Ellman at the University of California, Berkeley.

Jason Rech 05-05-06 2 , (AV 300) Org. Syn 1st step

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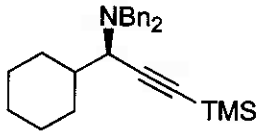

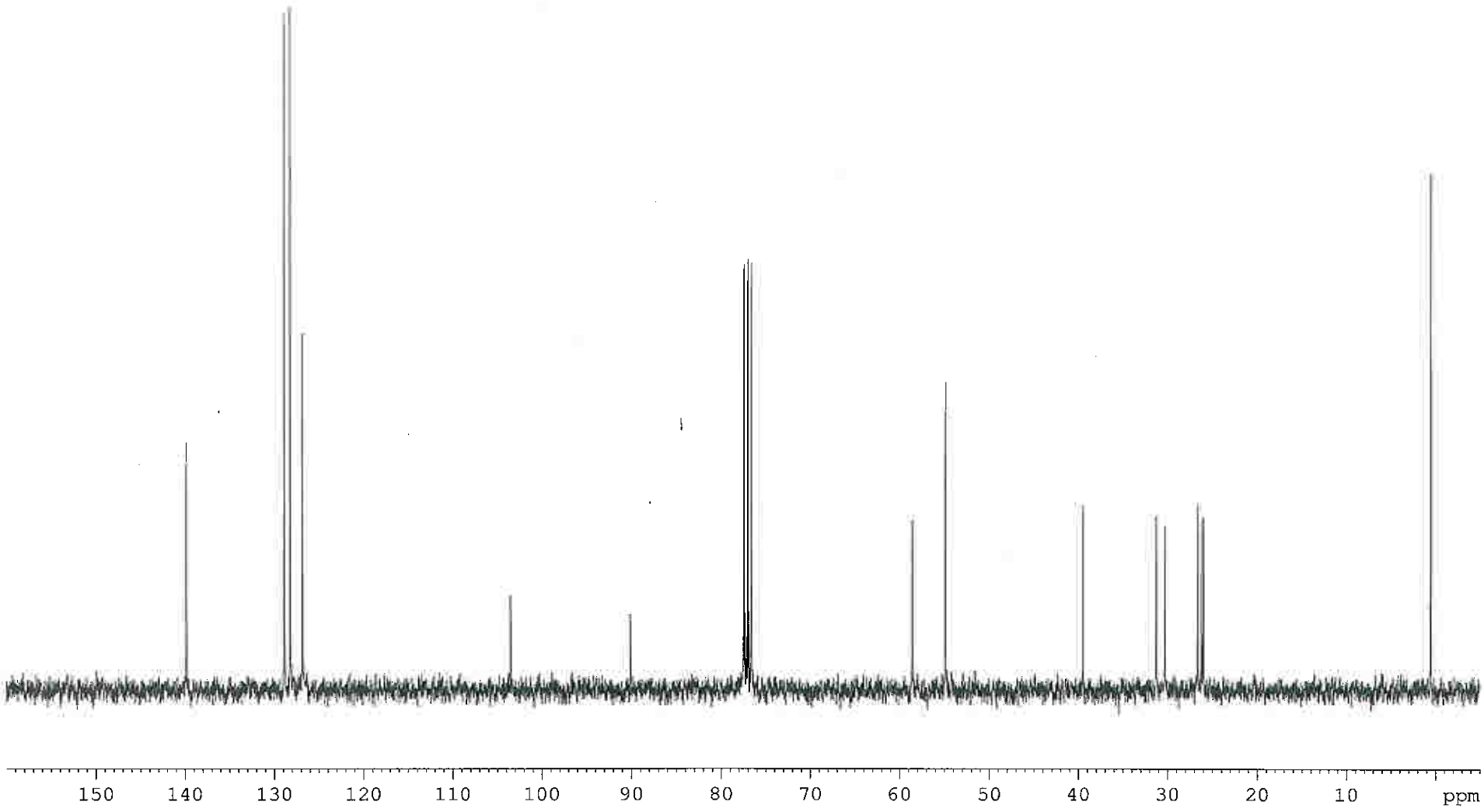

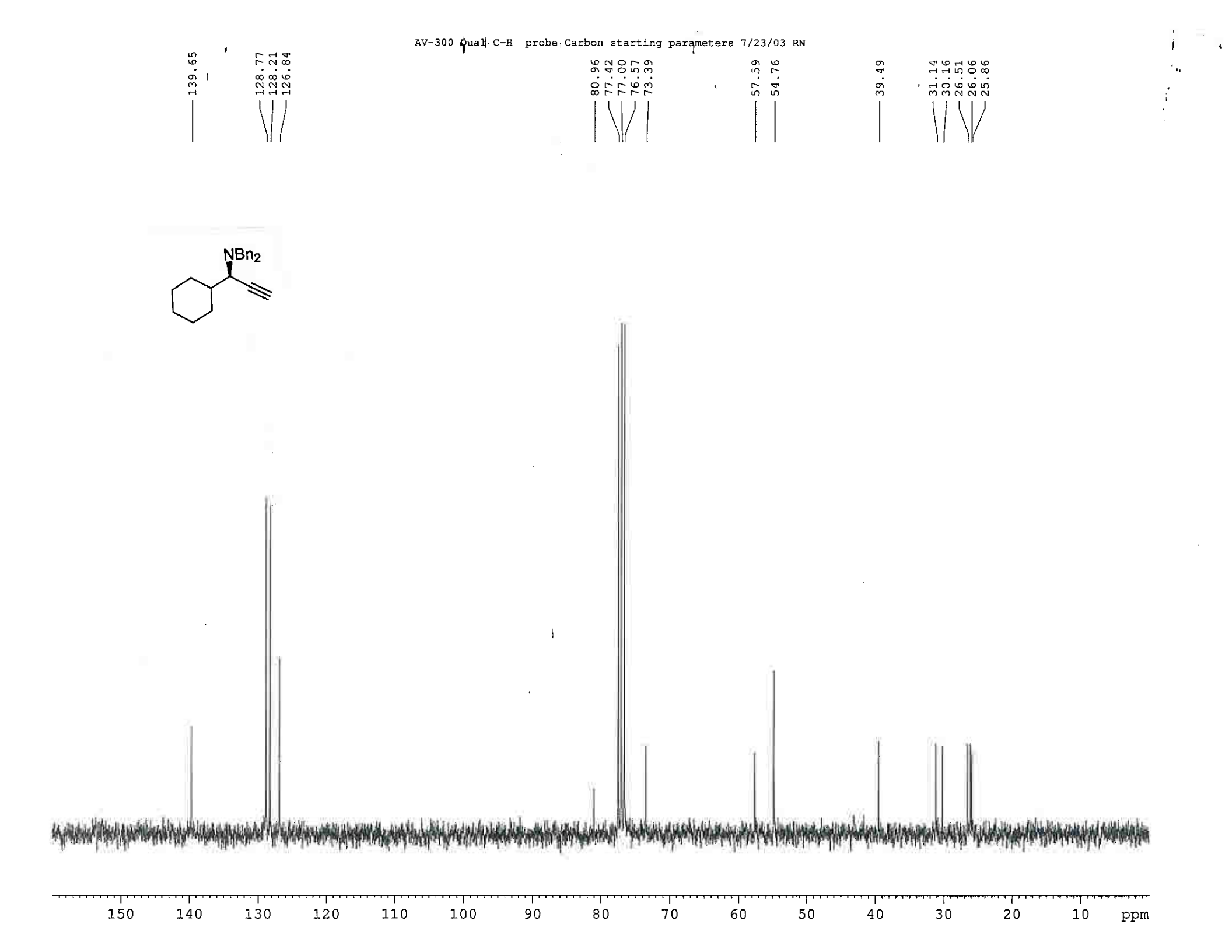
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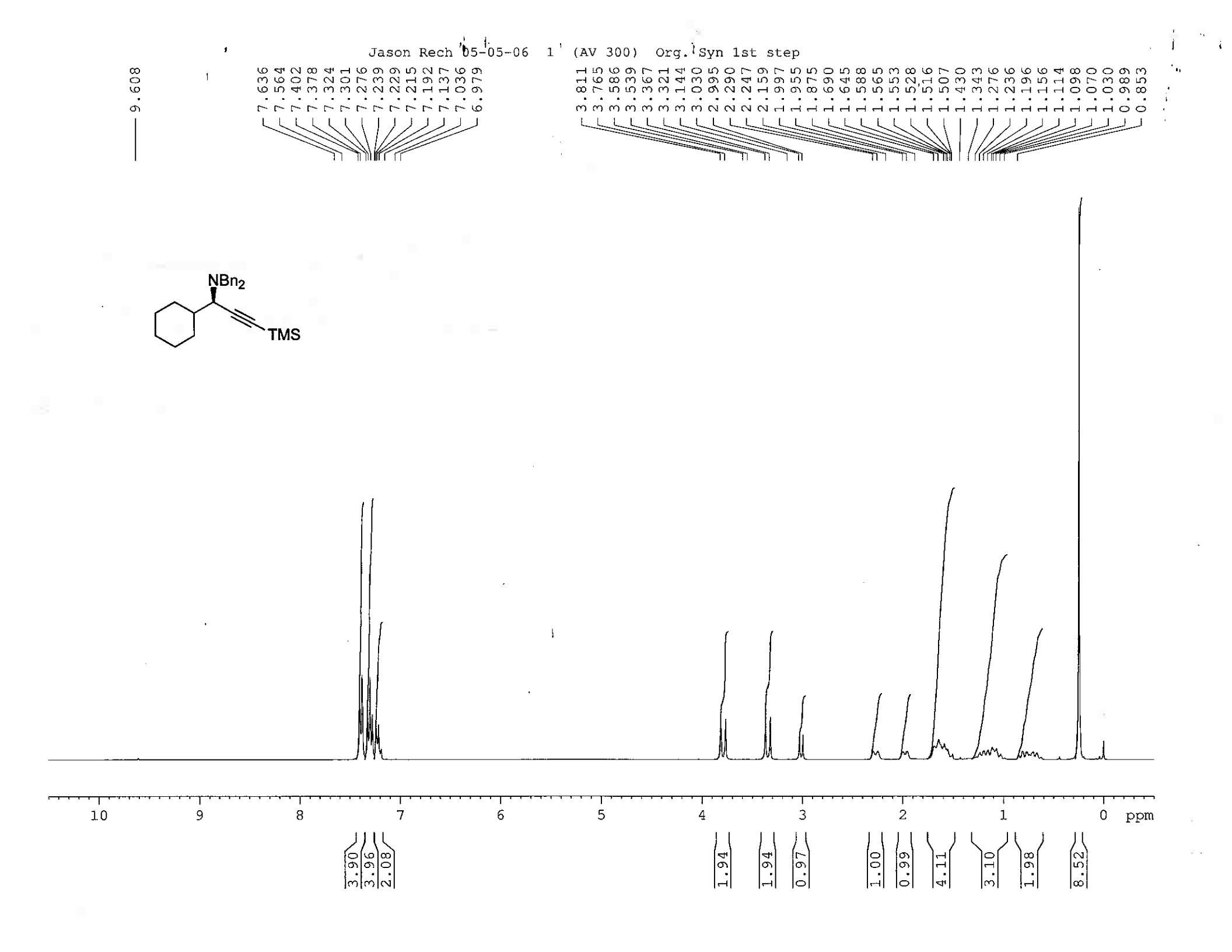
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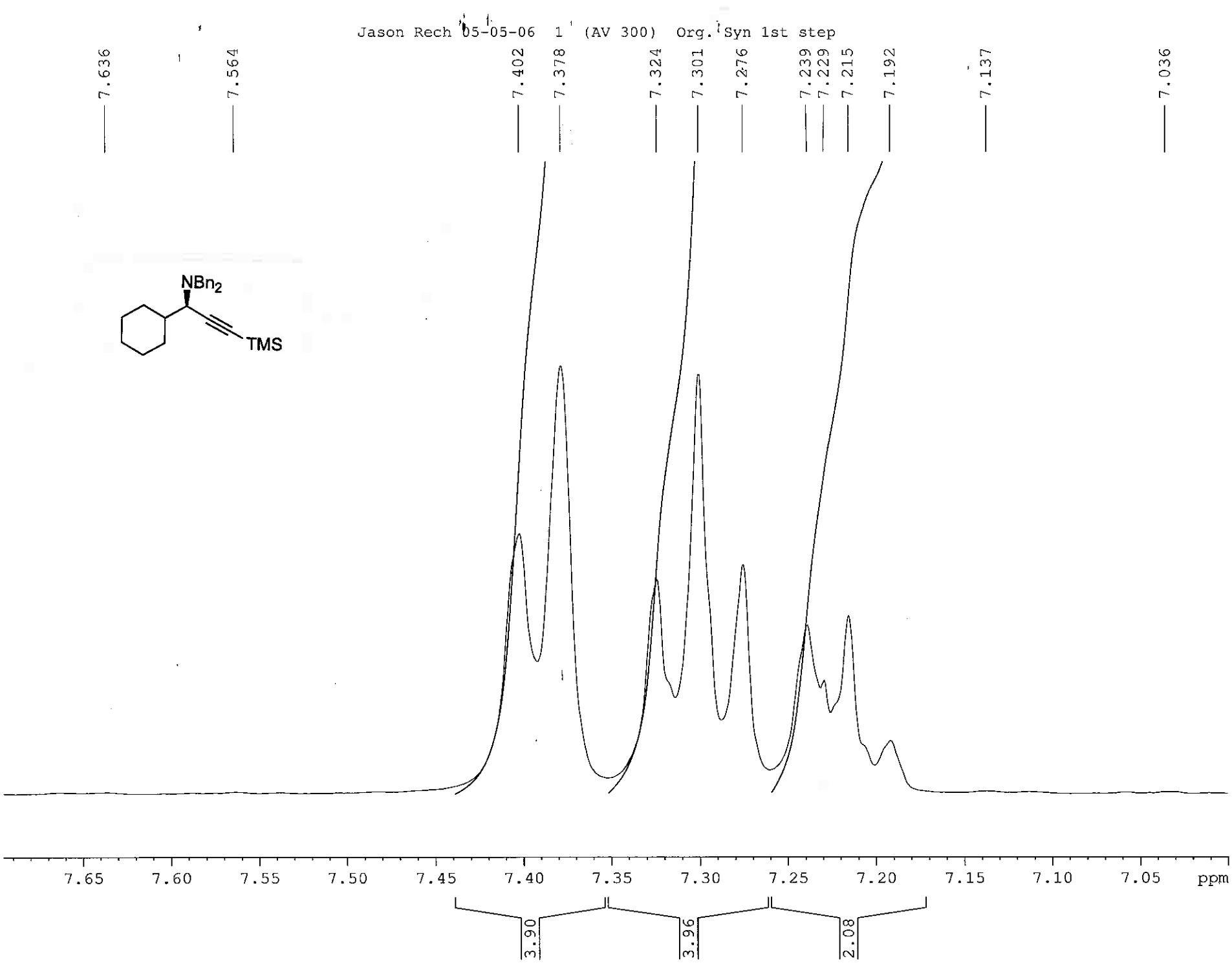
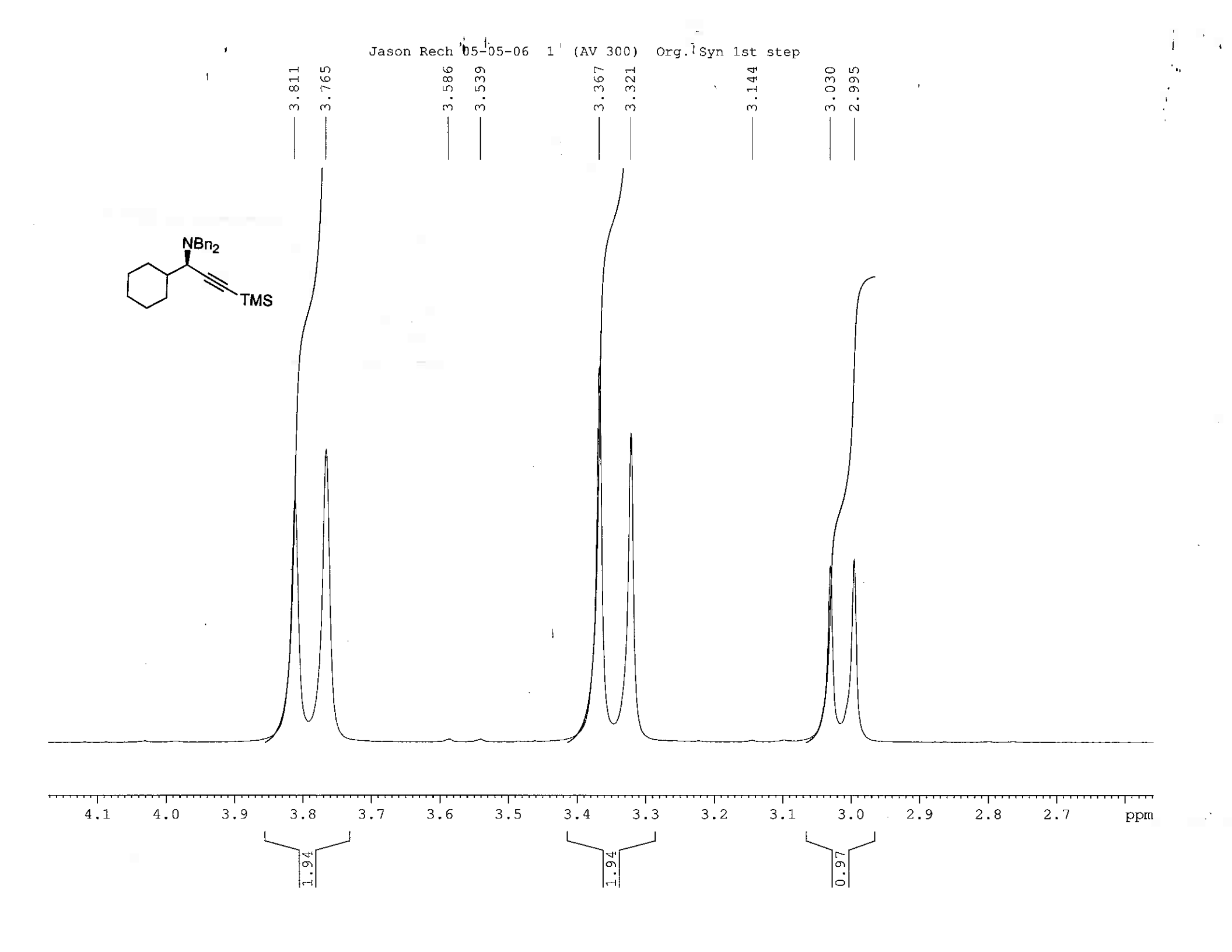
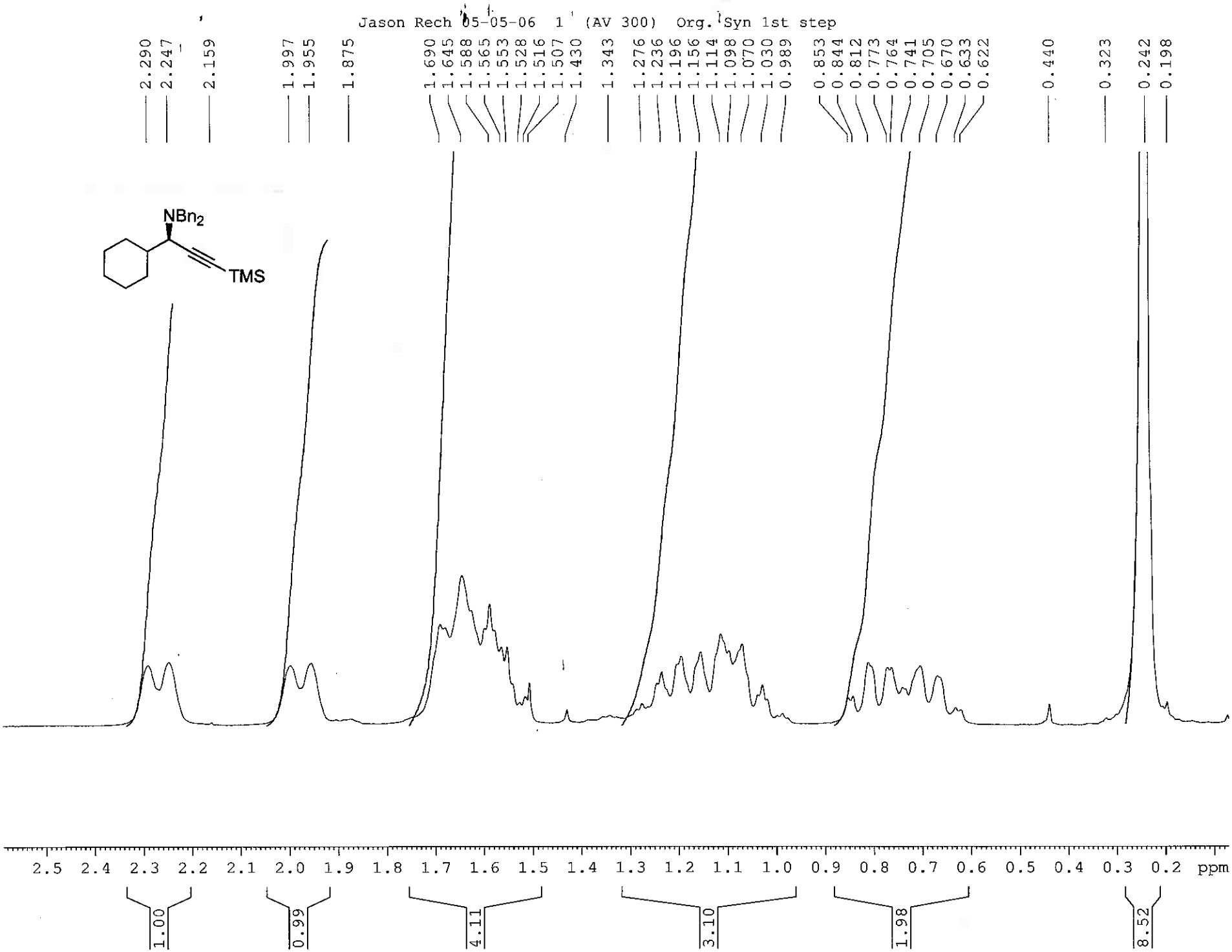
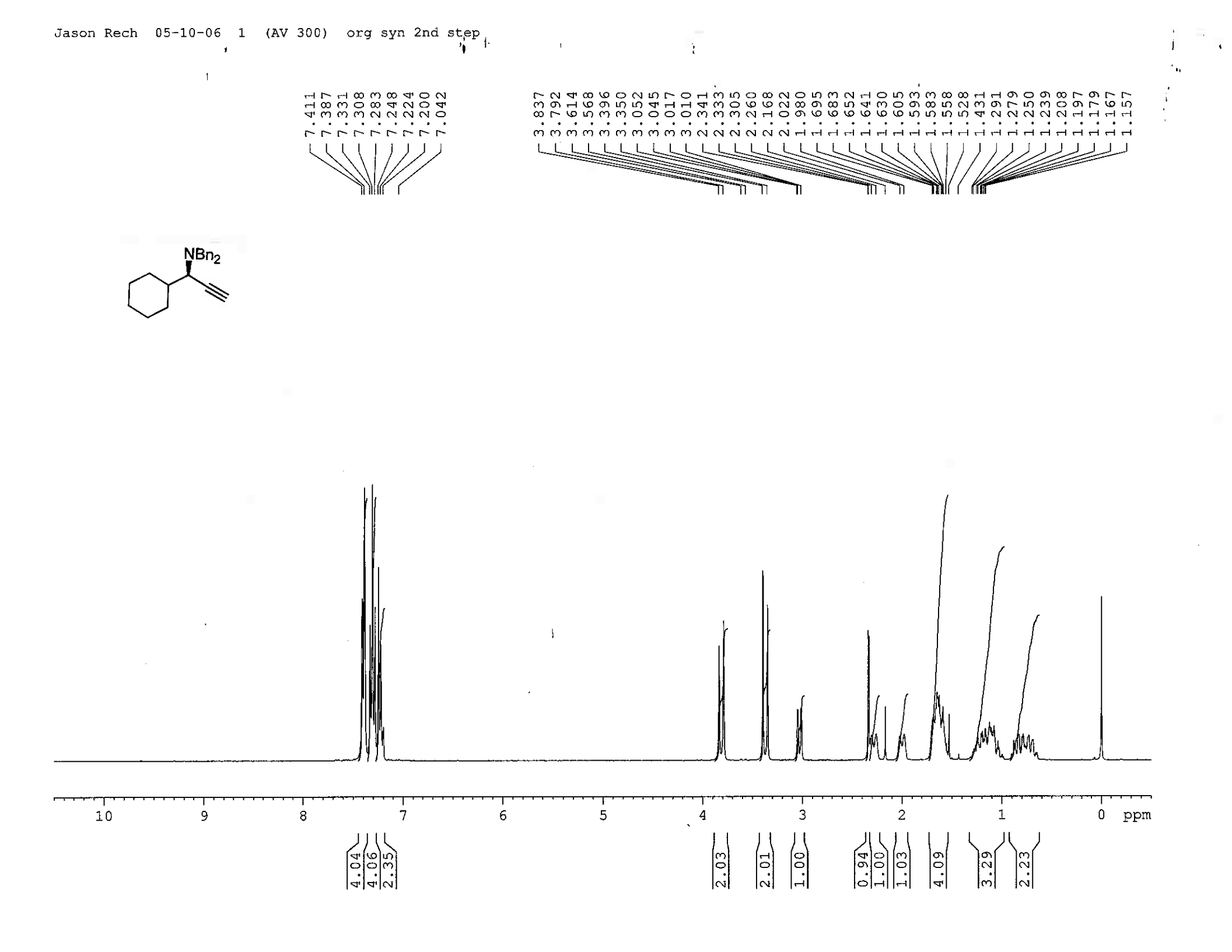
7.036
6.979
3.811
3.765
3.586
3.539
3.367
3.321
3.144
3.030
2.995
2.290
2.247
2.159
1.997
1.955
1.875
1.690
1.645
1.588
1.565
1.553
1.528
1.516
1.507
1.430
1.343
1.276
1.236
1.196
1.156
1.114
1.098
1.070
1.030
0.989
0.853
NBn2
TMS
10
9
8
7
6
5
4
3
2
1
0
ppm
3.90
3.96
2.08
1.94
1.94
0.97
1.00
0.99
4.11
3.10
1.98
8.52


Jason Rech 05-05-06 1 (AV 300) Org. Syn 1st step
7.636
7.564
7.402
7.378
7.324
7.301
7.276
7.239
7.229
7.215
7.192
7.137
7.036
NBn2
TMS
7.65
7.60
7.55
7.50
7.45
7.40
7.35
7.30
7.25
7.20
7.15
7.10
7.05
ppm
3.90
3.96
2.08

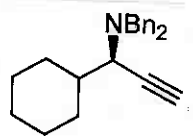

Jason Rech 05-05-06 1 (AV 300) Org. Syn 1st step
3.811
3.765
3.586
3.539
3.367
3.321
3.144
3.030
2.995
NBn2
TMS
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
ppm
1.94
1.94
0.97


Jason Rech 05-05-06 1 (AV 300) Org. Syn 1st step
2.290
2.247
2.159
1.997
1.955
1.875
1.690
1.645
1.588
1.565
1.553
1.528
1.516
1.507
1.430
1.343
1.276
1.236
1.196
1.156
1.114
1.098
1.070
1.030
0.989
0.853
0.844
0.812
0.773
0.764
0.741
0.705
0.670
0.633
0.622
0.440
0.323
0.242
0.198
NBn2
TMS
2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8 0.7 0.6 0.5 0.4 0.3 0.2 ppm
1.00
0.99
4.11
3.10
1.98
8.52

Jason Rech 05-10-06 1 (AV 300) org syn 2nd step



Jason Rech 05-10-06 1 (AV 300) org syn 2nd step

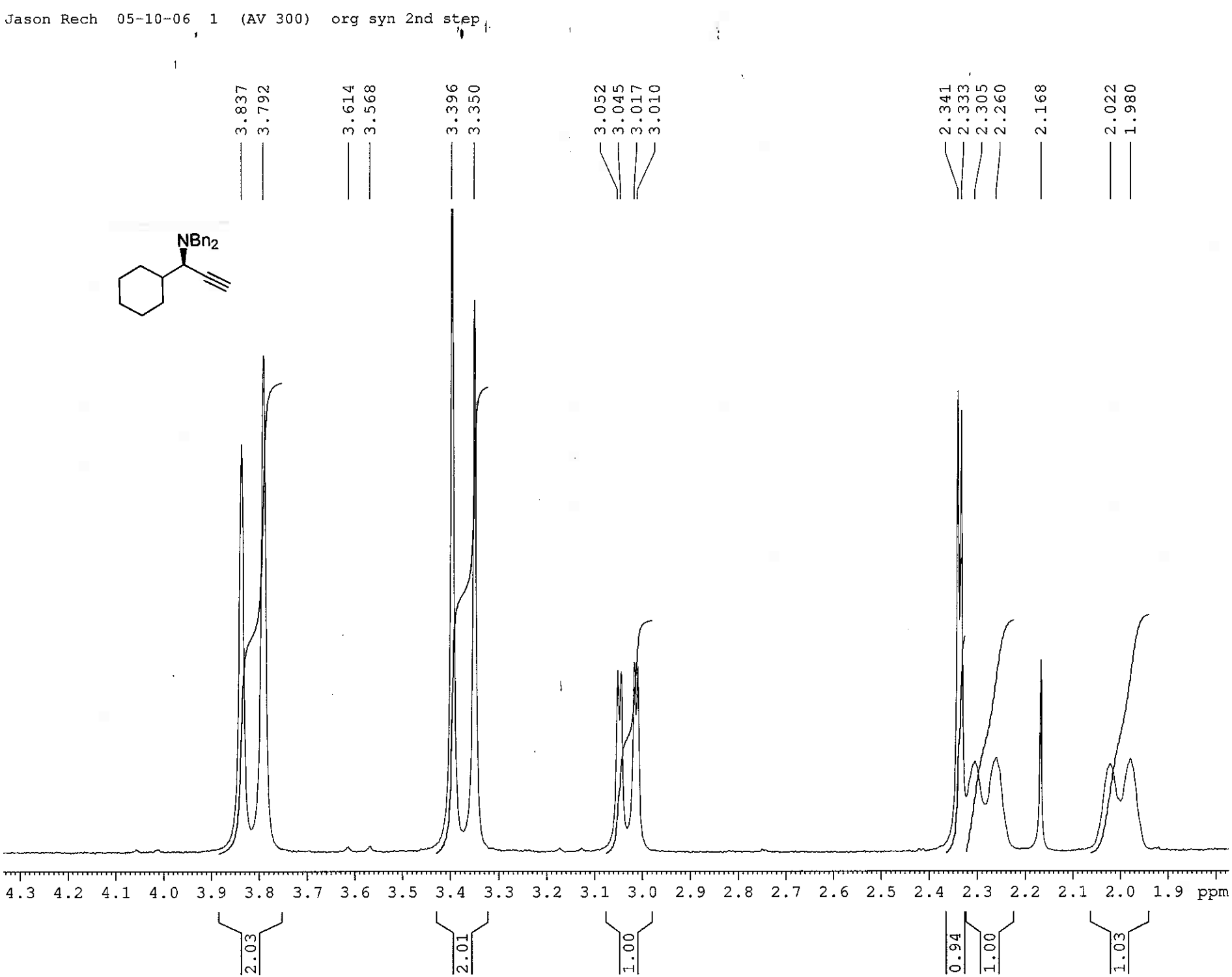


7.411
7.387
7.331
7.308
7.283
7.248
7.224
7.200
7.042

7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 ppm

4.04 4.06 2.35

Jason Rech 05-10-06 1 (AV 300) org syn 2nd step



Jason Rech 05-10-06 1 (AV 300) org syn 2nd step