A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

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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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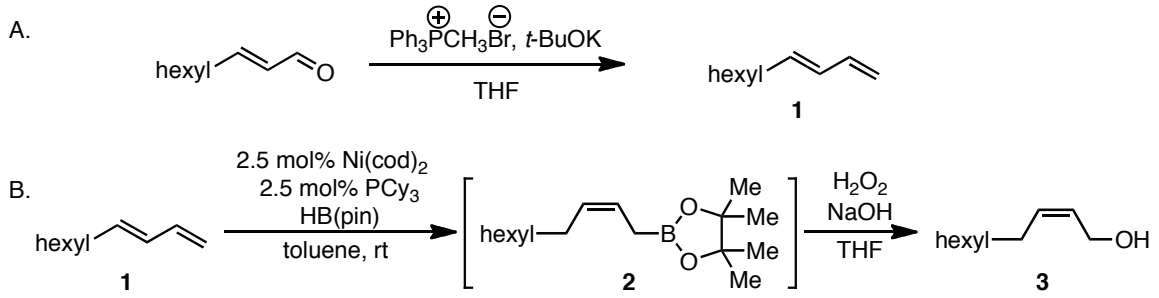
STEREOSELECTIVE NICKEL-CATALYZED 1,4-HYDROBORATION OF 1,3-DIENES

[*(Z)-Dec-2-en-1-ol*]



Submitted by Robert J. Ely and James P. Morken.[1]
Checked by Pamela M. Tadross and Brian M. Stoltz.

Caution! Reactions and subsequent operations involving peracids and peroxy compounds should be run behind a safety shield. Peroxy compounds should be added to the organic material, never the reverse. For relatively fast reactions, the rate of addition of the peroxy compound should be slow enough so that it reacts rapidly and no significant unreacted excess is allowed to build up. The reaction mixture should be stirred efficiently while the peroxy compound is being added, and cooling should generally be provided since many reactions of peroxy compounds are exothermic. New or unfamiliar reactions, particularly those run at elevated temperatures, should be run first on a small scale. Reaction products should never be recovered from the final reaction mixture by distillation until all residual active oxygen compounds (including unreacted peroxy compounds) have been destroyed. Decomposition of active oxygen compounds may be accomplished by the procedure described in Korach, M.; Nielsen, D. R.; Rideout, W. H. Org. Synth. 1962, 42, 50 (Org. Synth. 1973, Coll. Vol. 5, 414).

1. Procedure

A. *(E)-deca-1,3-diene* (**1**). In a glove box, a flame-dried one-necked 500-mL round-bottomed flask equipped with a magnetic stir bar (40 mm x 15 mm) is charged with methyltriphenylphosphonium bromide (18.97 g, 53.1 mmol, 1.1 equiv) (Note 1) and potassium *tert*-butoxide (5.96 g, 53.1 mmol, 1.1 equiv) (Note 2). The flask is sealed with a septum, removed from the glove box and transferred to a fume hood. A nitrogen inlet (Note 3) is then attached to the flask and the flask is cooled to 0-5 °C in an ice bath.

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Tetrahydrofuran (160 mL *via* syringe) (Note 4) is added and the solution turns yellow immediately. After 5 min, the ice bath is removed, and the suspension is allowed to warm to room temperature with stirring over 20–30 min. The suspension is then stirred at room temperature for 30 min. The suspension is then cooled to 0–5 °C in an ice bath, and *trans*-2-nonenal (8.0 mL, 48.3 mmol, 1.0 equiv) (Note 5) is added dropwise *via* syringe over 5 min. After 5 min, the ice bath is removed, and the suspension is allowed to warm to room temperature with stirring over 20–30 min. The suspension is then stirred at room temperature for 2 h. The reaction can be monitored by TLC analysis on SiO_2 (30:1 hexanes:EtOAc as the eluent; visualization with a $KMnO_4$ stain; the starting material aldehyde has a R_f = 0.16, and the product diene has a R_f = 0.72) (Note 6). The light brown heterogeneous solution is concentrated by rotary evaporation (10 mmHg, 25 °C) to approximately 1/3 of the original volume (Note 7), and Et_2O (50 mL) is added to form a white precipitate. The mixture is filtered over a pad of SiO_2 (8.5 cm diameter x 2.5 cm height, 52 g) in a sintered glass funnel (medium porosity). The flask is rinsed with Et_2O (2 x 50 mL), and the SiO_2 is washed with additional Et_2O (300 mL) (Note 8). The filtrate is concentrated to a slurry by rotary evaporation (10 mmHg, 25 °C), and pentane (50 mL) is added to form a white precipitate. The mixture is filtered over a pad of SiO_2 (8.5 cm diameter x 2.5 cm height, 52 g) in a sintered glass funnel (medium porosity). The flask is rinsed with pentane (2 x 50 mL), and the SiO_2 is washed with additional pentane (300 mL). The filtrate is concentrated by rotary evaporation (10 mmHg, 25 °C) to a yellow oil. The oil is transferred to a 50-mL round-bottomed flask equipped with a magnetic stir bar and short-path distillation head (Note 9). The residue is distilled under vacuum (bp 55–58 °C at 12 mmHg, receiver flask cooled in a dry ice/acetone bath throughout the distillation) (Note 10), which provides the desired diene **1** as a clear, colorless oil (5.3–5.8 g, 80–87% yield) (Note 11).

B. *(Z)-dec-2-en-1-ol* (**3**). In a glove box, a flame-dried one-necked 500-mL round-bottomed flask equipped with a magnetic stir bar (40 mm x 15 mm) is charged with $Ni(cod)_2$ (259 mg, 0.94 mmol, 0.025 equiv) (Note 12), tricyclohexylphosphine (264 mg, 0.94 mmol, 0.025 equiv) (Note 13), toluene (120 mL *via* syringe) (Note 14), 4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5.73 mL, 39.5 mmol, 1.05 equiv) (Note 15) *via* syringe, and *(E)-deca-1,3-diene* (**1**) (5.2 g, 37.6 mmol, 1.0 equiv) *via* syringe. The flask containing the diene is rinsed with toluene (30 mL), which is then transferred to the reaction flask. The resulting orange homogeneous solution

is capped with a septum, taken out of the glove box, and transferred to a fume hood. A nitrogen inlet is added, and the mixture is stirred at room temperature for 1 h. The reaction can be monitored by TLC analysis on SiO_2 (40:1 hexanes:EtOAc as the eluent; visualization with a $KMnO_4$ stain; the starting material diene has a $R_f = 0.76$, and the allylic boronate ester has a $R_f = 0.16$). The stir bar is removed with a magnetic wand and rinsed with CH_2Cl_2 (2 mL), which turns the solution black. The mixture is concentrated by rotary evaporation (10 mmHg, 25 °C) to a black oil. The intermediate allylic boronate ester (**2**) can be easily purified (Note 16), or oxidized in the same flask. A stir bar is added and the flask is cooled to 0–5 °C in an ice bath followed by the addition of THF (72 mL) (Note 17), 3 M NaOH (8.0 mL *via* syringe) (Note 18), and 30 wt. % chilled hydrogen peroxide in water (8.0 mL dropwise over 3 min *via* syringe; exothermic reaction) (Notes 19 and 20). Following addition, the ice bath is removed and the solution is allowed to warm to room temperature over 10–15 min. The cloudy solution is then stirred vigorously at room temperature for 4.5 h. The oxidation can be monitored by TLC analysis on SiO_2 (7:1 hexanes:EtOAc as the eluent; visualization with a $KMnO_4$ stain; the starting material allylboronate ester has a $R_f = 0.55$, and the product alcohol has a $R_f = 0.23$). The solution is cooled to 0–5 °C in an ice bath and the excess hydrogen peroxide is quenched with a saturated solution of sodium thiosulfate (8.0 mL *via* syringe, dropwise over 3 min) (Notes 21 and 22). The reaction is diluted with deionized water (100 mL), and transferred to a 500-mL separatory funnel, rinsing the flask with EtOAc (2 x 25 mL). The layers are separated, and the aqueous layer is extracted with EtOAc (2 x 80 mL). The combined organic layers are dried over Na_2SO_4 (65 g), filtered, and concentrated by rotary evaporation (10 mmHg, 25 °C) to afford a clear, colorless oil. The crude oil is purified by flash chromatography on SiO_2 (Note 23), eluting with 7:1 hexanes:EtOAc to afford **2** as a clear, colorless oil (5.58–5.61 g, 95–96% yield) (Note 24).

2. Notes

1. Methyltriphenylphosphonium bromide (98%) was purchased from Aldrich and dried under vacuum (1.70 mmHg, 110 °C) 12 h before use.

2. Potassium *tert*-butoxide (95%) was purchased from Aldrich and used as received.

3. All "nitrogen inlet" references are defined as inserting a 16-gauge needle attached to a positive flow of nitrogen through the top of a rubber septum.

4. The submitters used THF (HPLC grade) that was purchased from Fisher Scientific and purified using a Pure Solv MD-4 solvent purification system, from Innovative Technology, Inc., by passing the solvent through two activated alumina columns after being purged with argon. The checkers used THF (HPLC grade) that was purchased from Fisher Scientific and purified by passing the solvent through two activated alumina columns under argon.

5. *trans*-2-Nonenal (97%) was purchased from Aldrich and used as received.

6. The submitters performed thin layer chromatography using 0.25 mm silica gel glass backed plated from Silicycle, with visualization performed with either potassium permanganate ($KMnO_4$) or phosphomolybdic acid (PMA). The checkers performed thin layer chromatography using E. Merck silica gel 60 F254 precoated glass plates (0.25 mm), with visualization performed with potassium permanganate ($KMnO_4$).

7. Evaporation of all the solvent will cause the triphenylphosphine oxide to occlude the product, and the yield will be reduced.

8. The submitters reported that occasional stirring of the top layer of the SiO_2 is needed to keep the triphenylphosphine oxide from clogging the filter. The checkers found that this problem could be circumvented by using a filter with a larger diameter.

9. The short-path distillation head and receiver flask were both dried in an oven for 12 h before distillation of compound **1**.

10. The submitters reported distillation of compound **1** at 3.5 mmHg (bp 27–30 °C). The checkers found that somewhat higher yields could be obtained by increasing the pressure to 12 mmHg (bp = 55–58 °C) and cooling the receiver flask in a dry ice/acetone bath for the duration of the distillation.

11. Compound **1** has the following properties: ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.89 (t, J = 7.0 Hz, C$\mathbf{H_3}$, 3 H), 1.22–1.45 (m, (C$\mathbf{H_2}$)$_4$, 8 H), 2.08 (dt, J = 6.9, 6.9 Hz, C$\mathbf{H_2}$CH=CH, 2 H), 4.95 (ddd, J = 10.1, 1.1, 0.6 Hz, CH=C$\mathbf{H}_{cis}$H$_{trans}$, 1 H), 5.09 (ddd, J = 17.0, 1.2, 0.6 Hz, CH=CH$_{cis}$$\mathbf{H}_{trans}$, 1 H), 5.71 (dt, J = 15.0, 7.0 Hz, CH$_2$C**H**=CH, 1 H), 6.05 (dd, J = 15.2, 10.4 Hz, CH$_2$CH=C**H**, 1 H), 6.32 (dddd, J = 17.0, 10.3, 10.3, 2.0 Hz, C**H**=CH$_{cis}$H$_{trans}$,

1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 14.1, 22.6, 28.9, 29.2, 31.7, 32.6, 114.5, 130.8, 135.6, 137.4; IR (neat) 2958 (m), 2926 (s), 2856 (m), 1001 (s), 895 (m) cm^{-1}; HRMS (EI+) *m/z* calc'd for $C_{10}H_{18}$ $[M]^+$: 138.1408, found 138.1423; Anal. calcd. for $C_{10}H_{18}$: C, 86.88; H, 13.12. Found: C, 87.02; H, 12.78. The spectral data are in agreement with the reported values.[2]

12. $Ni(cod)_2$ (98%) was purchased from Strem and used as received.

13. Tricyclohexylphosphine (97%) was purchased from Strem and used as received.

14. The submitters used toluene (HPLC grade) that was purchased from Fisher Scientific and purified using a Pure Solv MD-4 solvent purification system, from Innovative Technology, Inc., by passing the solvent through two activated alumina columns after being purged with argon. The checkers used toluene (HPLC grade) that was purchased from Fisher Scientific and was purified by passing the solvent through two activated alumina columns under argon.

15. The submitters reported that 4,4,5,5-tetramethyl-1,3,2-dioxaborolane was generously donated by BASF containing 0.06% dimethyl sulfide, and was used as received. The submitters also noted that 4,4,5,5-tetramethyl-1,3,2-dioxaborolane (97%) purchased from Aldrich can also be used as received. The checkers purchased 4,4,5,5-tetramethyl-1,3,2-dioxaborolane (97%) from Aldrich and used it as received.

16. The checkers performed the hydroboration on a 1.45 mmol (200 mg) scale of diene: after the catalyzed reaction is complete, the stir bar (15 mm x 5 mm) is removed using a magnetic wand, and rinsed with Et_2O (2 mL; rinsing with CH_2Cl_2 causes the solution to turn black and reduces the yield for an unknown reason). The brown residue is then filtered through a short SiO_2 plug (8.2 g, 4.5 cm diameter x 1.0 cm height) eluting with 40:1 hexanes:EtOAc (80 mL hexanes, 2 mL EtOAc). The filtrate is then concentrated by rotary evaporation (10 mmHg, 25 °C) and then at 1.70 mmHg to provide the boronate as a clear, colorless oil (359 mg, 93% yield). Compound **2** has the following properties: ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.88 (t, J = 7.0 Hz, $CH_2C\mathbf{H_3}$, 3 H), 1.15–1.41 (m, $(C\mathbf{H_2})_5$ + $(C\mathbf{H_3})_4$, 22 H), 1.67 (d, J = 7.8 Hz, $C\mathbf{H_2}B$, 2 H), 2.01 (dt, J = 6.9 Hz, 6.9 Hz, $C\mathbf{H_2}CH{=}CH$, 2 H), 5.39 (dtd, J = 8.5 Hz, 7.0 Hz, 1.0 Hz, $CH{=}C\mathbf{H}CH_2B$, 1 H), 5.49 (dtd, J = 9.5 Hz, 8.0 Hz, 1.5 Hz, $C\mathbf{H}{=}CHCH_2B$, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 14.1, 22.7, 24.7, 27.1, 29.3, 29.3, 29.6, 31.9, 83.2, 123.9, 130.0; IR (neat) 2978 (m), 2957 (m), 2926 (s), 2855 (m), 1325 (s), 1145 (s), 968 (w), 848 (w) cm^{-1}; HRMS (EI+) *m/z* calc'd for $C_{16}H_{31}BO_2$ $[M]^+$: 266.2417, found

266.2419; Anal. calcd. for $C_{16}H_{31}BO_2$: C, 72.18; H, 11.74. Found: C, 72.51; H, 12.01.

17. THF (HPLC grade) for oxidation is purchased from Fisher Scientific and used as received.

18. Sodium hydroxide pellets (A.C.S. grade) are purchased from Fisher Scientific.

19. During the addition of the H_2O_2 solution, an internal thermometer was used to monitor the temperature of the reaction. Over the course of the addition, the checkers observed that the internal temperature of the reaction increased from 5 °C to 30 °C.

20. Hydrogen peroxide (30% wt. in H_2O) is purchased from Aldrich and is stored and used chilled (~4 °C).

21. The submitters reported that quenching of excess peroxide with sodium thiosulfate solution was exothermic. However, the checkers used an internal thermometer to monitor the temperature of the reaction and found no evidence of an exotherm during quenching.

22. Sodium thiosulfate pentahydrate (99.5%) is purchased from Fisher Scientific and is added to deionized water until saturated.

23. Flash chromatography was performed using Silia*Flash* P60 Academic Silica gel (SiO_2, particle size 0.040–0.063 mm) purchased from Silicycle (wet packed in 7:1 hexanes:ethyl acetate, 2.5 cm diameter x 25 cm height, 186 g), eluting with 7:1 hexanes:EtOAc (2.21 L hexanes, 0.29 L EtOAc; 300 mL was passed through, then 200 mL fractions were taken). Fractions 4–9 were collected, concentrated by rotary evaporation (10 mmHg, 25 °C) and then dried under vacuum at 1.70 mmHg to provide a clear, colorless oil.

24. Compound **3** has the following properties: ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.89 (t, J = 7.0 Hz, $CH_2C\mathbf{H_3}$, 3 H), 1.16-1.48 (m, $CH_3(C\mathbf{H_2})_4$, 8 H), 2.07 (dt, J = 7.3 Hz, 7.2 Hz, $C\mathbf{H_2}CH{=}CH$, 2 H), 4.22 (dd, J = 6.3 Hz, 0.7 Hz, $C\mathbf{H_2}OH$, 2 H), 4.74–5.42 (m, C**H**=C**H,** 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 14.1, 22.6, 27.4, 29.1, 29.2, 29.6, 31.8, 58.6, 128.3, 133.3; IR (neat): 3325 (m br), 2956 (m), 2922 (s), 2855 (m), 1465 (m), 1032 (s), 723 (m) cm^{-1}; HRMS (EI+) *m/z* calc'd for $C_{10}H_{20}O$ $[M]^+$: 156.1514, found 156.1549; Anal. calcd. for $C_{10}H_{20}O$: C, 76.86; H, 12.90. Found: C, 76.86; H, 13.28. The spectral data are in agreement with the reported values.[3]

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

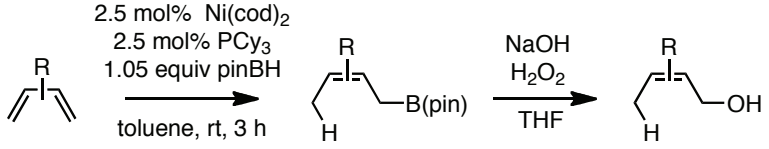
Allyboronates are synthetically useful intermediates, specifically (*Z*)-crotylboronate esters. They are commonly used for the allylation of carbonyl compounds to furnish *syn*-homoallylic alcohols, or directly oxidized to provide stereodefined (*Z*)-allylic alcohols.[4] Current methods to synthesize (*Z*)-crotylboronates include the trapping of allylic anions using Schlosser's method, but can only be applied to simple crotylboronates.[5] Transmetalation of allylmetal reagents or Matteson homologation of alkenylmetal precursors can provide substituted crotylboronates, but both require the use of a stoichiometric amount of transition metal, and the precursors can be difficult to synthesize.[4] One useful method that has been described in the literature is the 1,4-hydroboration of conjugated dienes. Suzuki and Miyaura found that 1,4-hydroboration can be accomplished with Pd and Rh catalysis.[6] This is an excellent reaction with 2-substituted and 2,3-disubstituted butadienes, but for terminally substituted dienes is limited to cyclic substrates (i.e., cyclohexadiene). Recently, Ritter described an iron catalyst that exhibits remarkable selectivity in the 1,4-hydroboration of 2-substituted dienes, but it is less general for terminally substituted diene substrates that lack substitution at the 2-position.[7] The procedure described here is the only method that is general for terminally substituted dienes.[8]

The Ni-catalyzed 1,4-hydroboration of 1,3-dienes allows for the operationally convenient synthesis of (*Z*)-crotylboronates and derived (*Z*)-allylic alcohols. The catalyst is composed of inexpensive and commercially available $Ni(cod)_2$ and PCy_3. The stoichiometric reagent, pinacolborane, is commercially available and bench-top stable. The hydroboration is completely regio- and stereoselective for terminal dienes (Table 1), and tolerates several synthetically common functional groups including silyl ethers, benzyl ethers, pthalimides, esters and unprotected alcohols (entries 7-11). Internal dienes are also competent substrates exhibiting high regioselectivity (Table 2).

The Ni-catalyzed 1,4-hydroboration of 1,3-dienes conveniently

provides (*Z*)-allylboronates that would be difficult to synthesize by other methods. The allyboronate can be used to synthesize homoallylic alcohols by allylation to carbonyl compounds, or directly oxidize to the stereodefined allylic alcohol.

Table 1.



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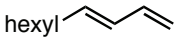
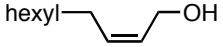
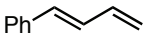
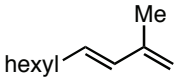
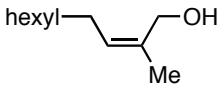
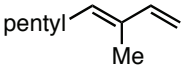
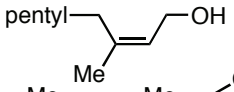
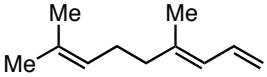
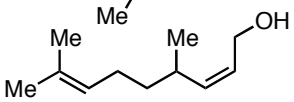
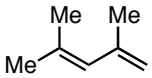
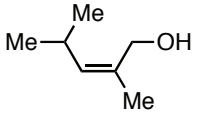
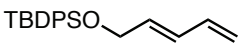
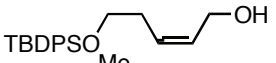
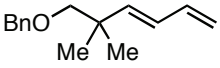
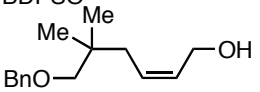
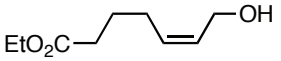
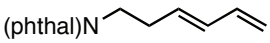
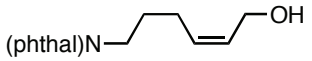
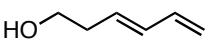
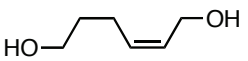
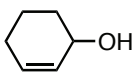
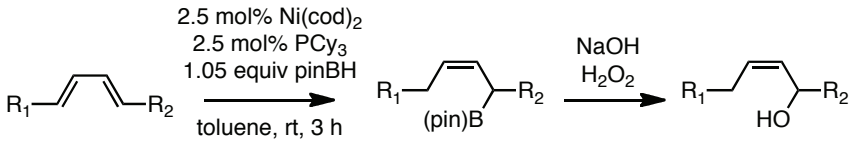
1			85
2			91[a]
3			93
4			81
5			29[b]
6			63[c]
7			56
8			89
9			81[d]
10			61[d]
11			72[e]
12			60

(a) 1 mol % catalyst $Ni(cod)_2$ and 1 mol % PCy_3 employed for this experiment. (b) Reaction 12 h at 60 °C. Product isolated with an equimolar quantity of 4,8-dimethyl-3,7-nonadien-2-ol. (c) Reaction for 12 h at 25 °C. (f) Oxidation with buffered (pH=7) H_2O_2. (d) 2.1 equiv pinBH employed.

Table 2.



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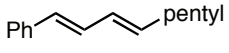
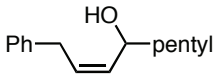
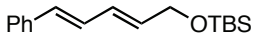
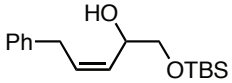
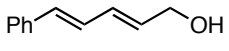
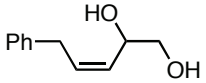
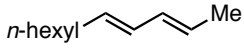
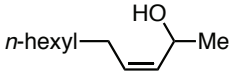
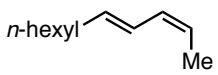
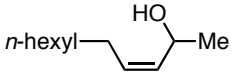
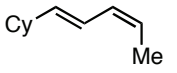
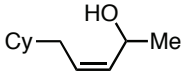
1			10:1	84[a]
2			>20:1	91[b]
3			>20:1	54[b,c]
4			5:1	61[a]
5	 10:1 *E,Z:E,E*		>20:1	82[a]
6	 4:1 *E,Z:E,E*		>20:1	83[a]

(a) Reaction for 12 h. (b) 1 mol % $Ni(cod)_2$ and 1 mol % PCy_3 employed for this experiment. (c) 2.1 equiv pinBH employed.

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8. Ely, R. J.; Morken, J. P. *J. Am. Chem. Soc.* **2010**, *132*, 2534–2535.

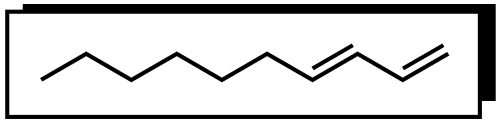
Appendix
Chemical Abstracts Nomenclature; (Registry Number)

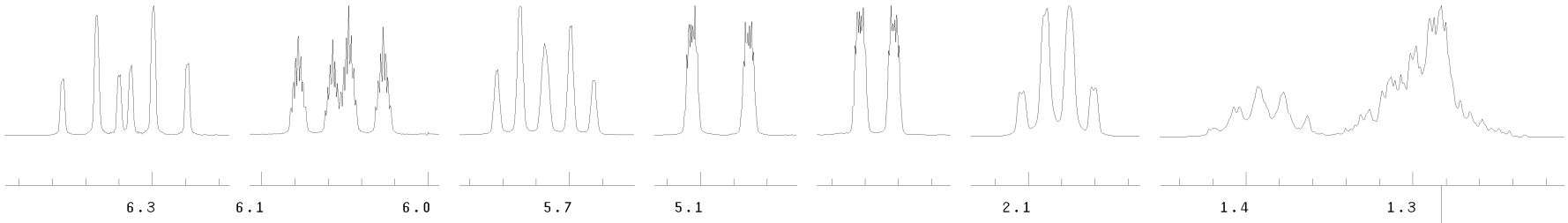
Methyltriphenylphosphonium bromide; (1779-49-3)
Potassium tert-butoxide: 2-Propanol, 2-methyl-, potassium salt (1:1); (865-47-4)
(*E*)-Deca-1,3-diene: 1,3-Decadiene, (3*E*)-; (58396-45-5)
(*Z*)-Dec-2-en-1-ol: 2-Decen-1-ol, (2*Z*)-; (4194-71-2)
Hydrogen peroxide; (7722-84-1)
trans-2-Nonenal: 2-Nonenal, (2*E*)-; (18829-56-6)
$Ni(cod)_2$Bis(cyclooctadiene)nickel(0); (1295-35-8)
Tricyclohexylphosphine; (2622-14-2)
4,4,5,5-Tetramethyl-1,3,2-dioxaborolane; (25015-63-8)

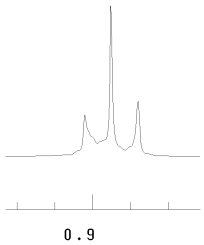
James P. Morken was born in Concord, CA in 1967. He obtained his B.S. in chemistry in 1989 from UC Santa Barbara working with Prof. Bruce Rickborn, and a Ph.D. from Boston College in 1995 with Prof. Amir Hoveyda. He was an NSF Postdoctoral Fellow with Stuart Schreiber at Harvard University and, in 1997, joined the University of North Carolina at Chapel Hill as an Assistant Professor. He was promoted to Associate Professor in 2002 and in 2006 joined the faculty of Boston College as a Professor of Chemistry. His research focuses on the development of transition-metal-catalyzed asymmetric processes and their use in complex molecule synthesis.

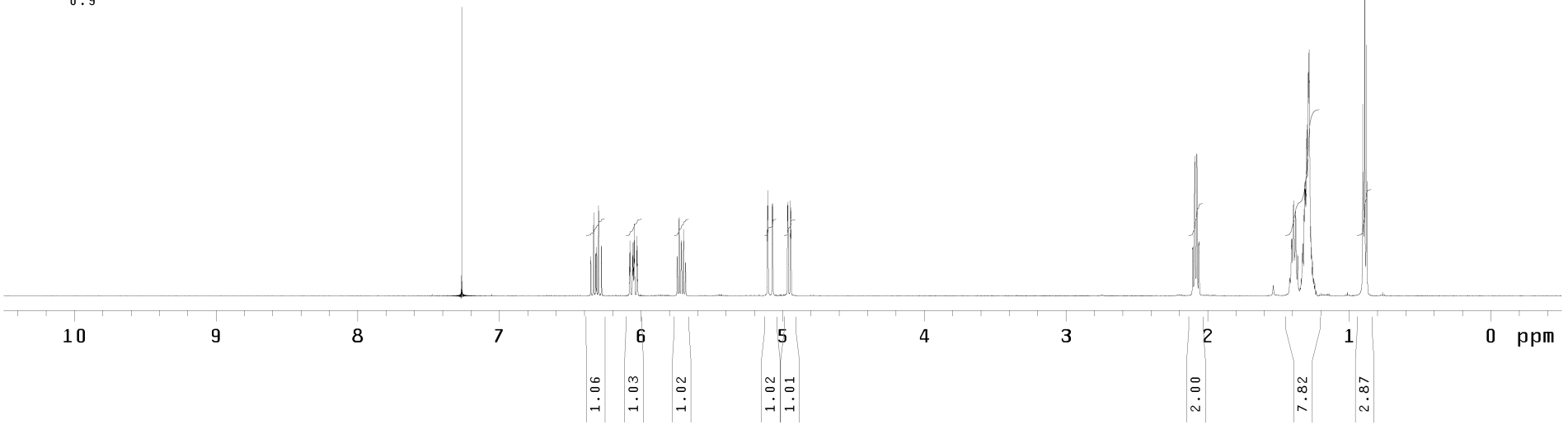
Robert J. Ely was born in 1984 in Grand Junction, CO. He earned a BA in chemistry and biochemistry from the University of Colorado, Boulder in 2007. While there, he interned as a medicinal chemist at Array BioPharma. After graduating, he joined the group of Prof. Morken in 2007. At Boston College he has conducted research involving the Ni-catalyzed hydroboration and diboration of 1,3-dienes.

Pamela M. Tadross was born in Brooklyn, NY in 1983 and received her B.S. degree in chemistry in 2005 from New York University where she conducted research with Professor Marc A. Walters. She subsequently joined the labs of Professor Brian M. Stoltz at the California Institute of Technology in 2005 where she has pursued her Ph.D. as a fellow of the California HIV/AIDS Research Program. Her research interests focus on the exploitation of aryne reactive intermediates in the synthesis of biologically active natural products.









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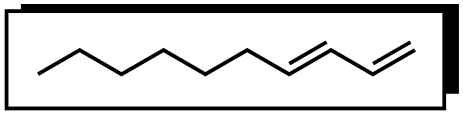

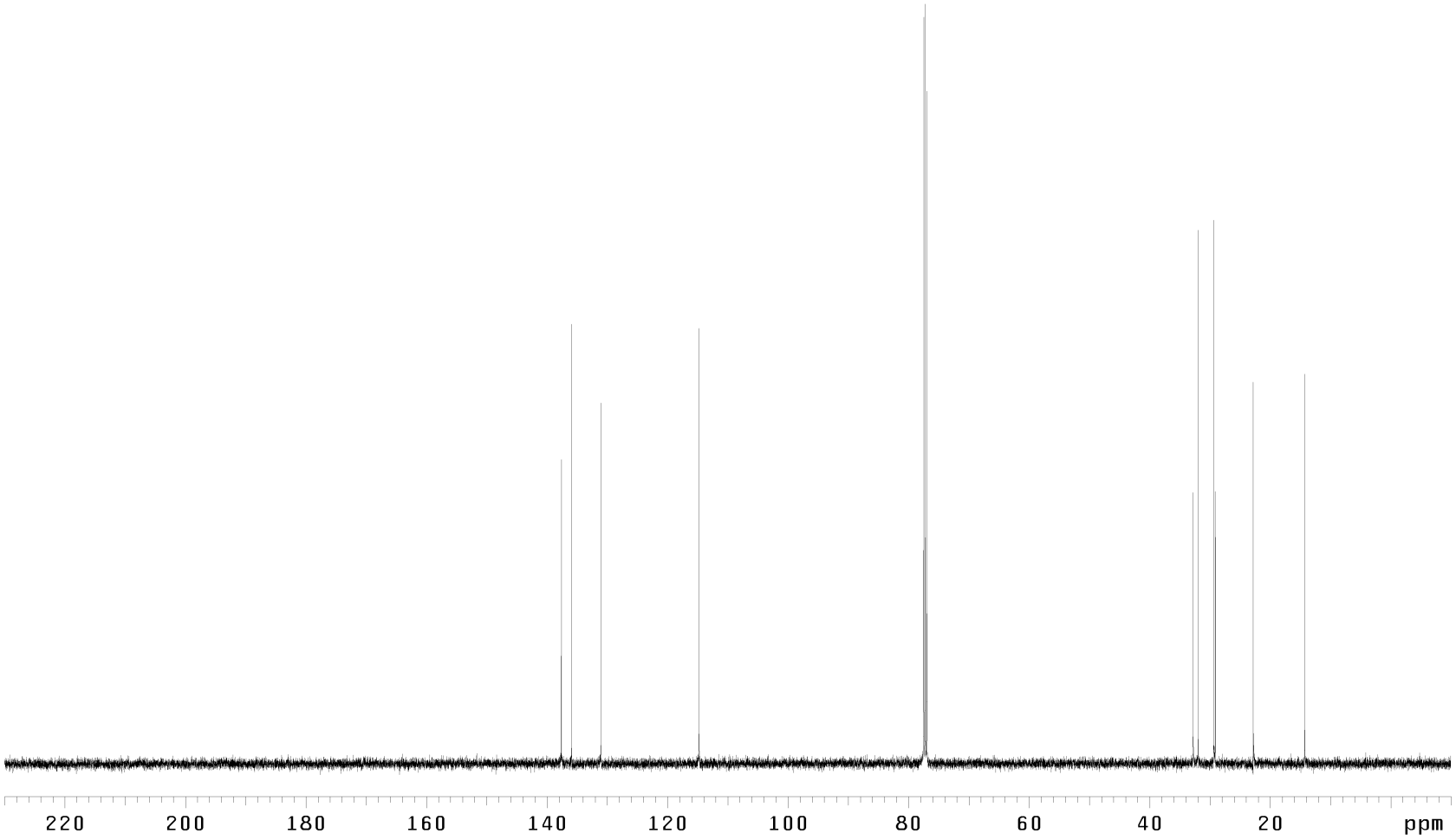
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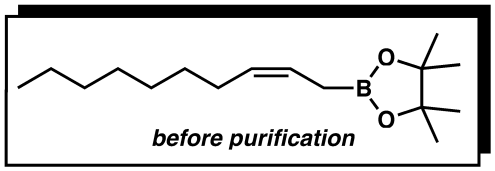
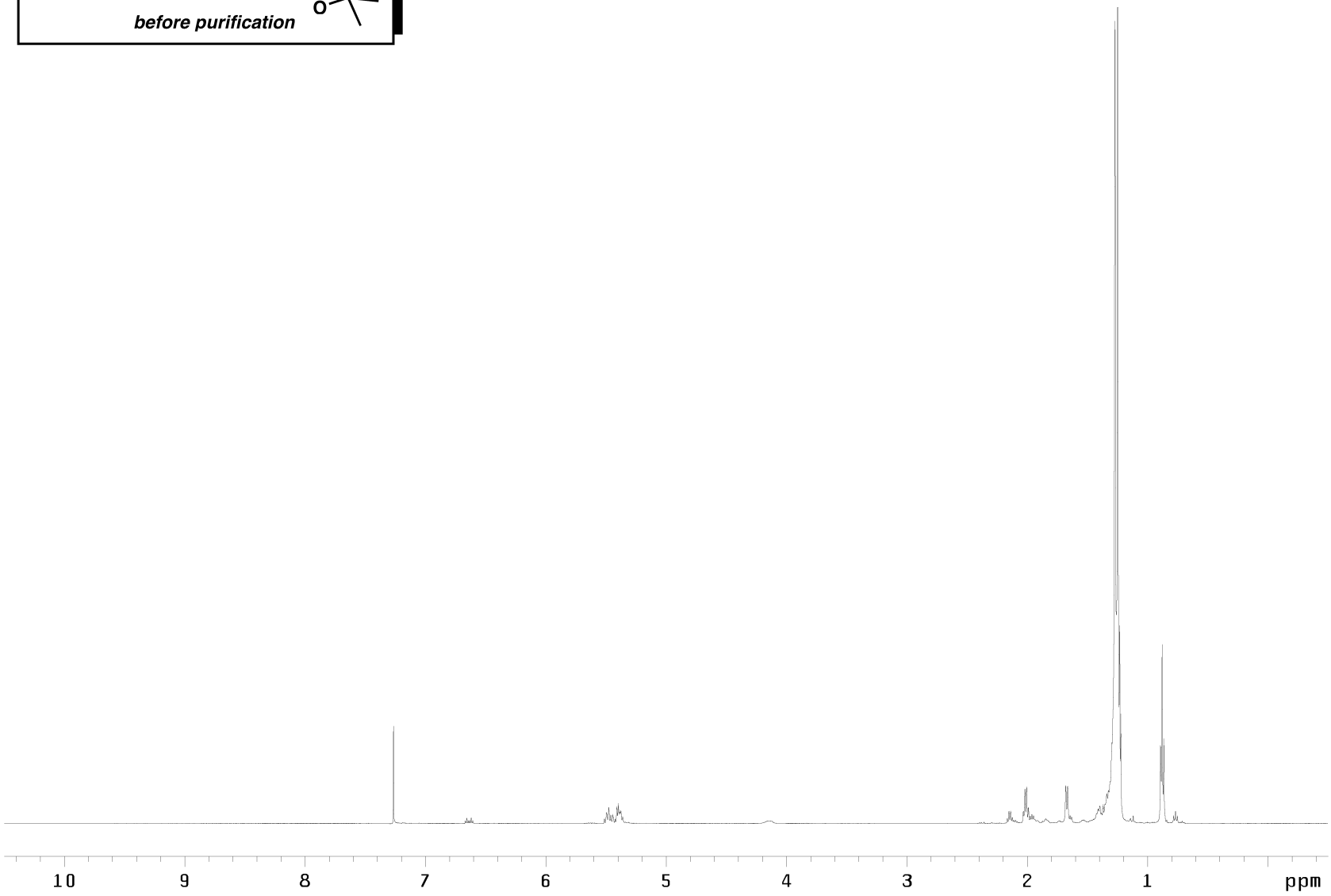
120
100
80
60
40
20
ppm


O
B
O
before purification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4
3
2
1
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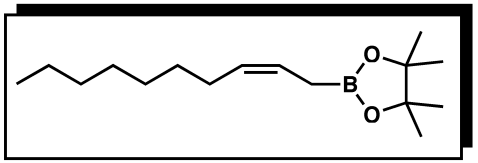

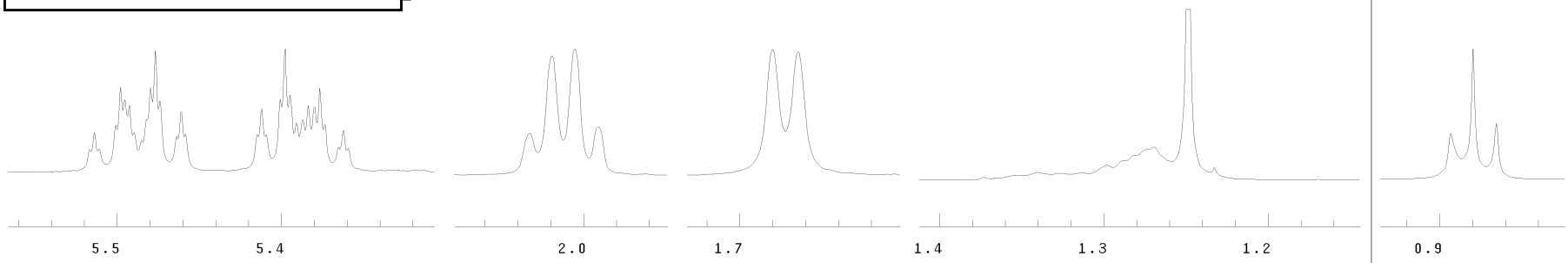

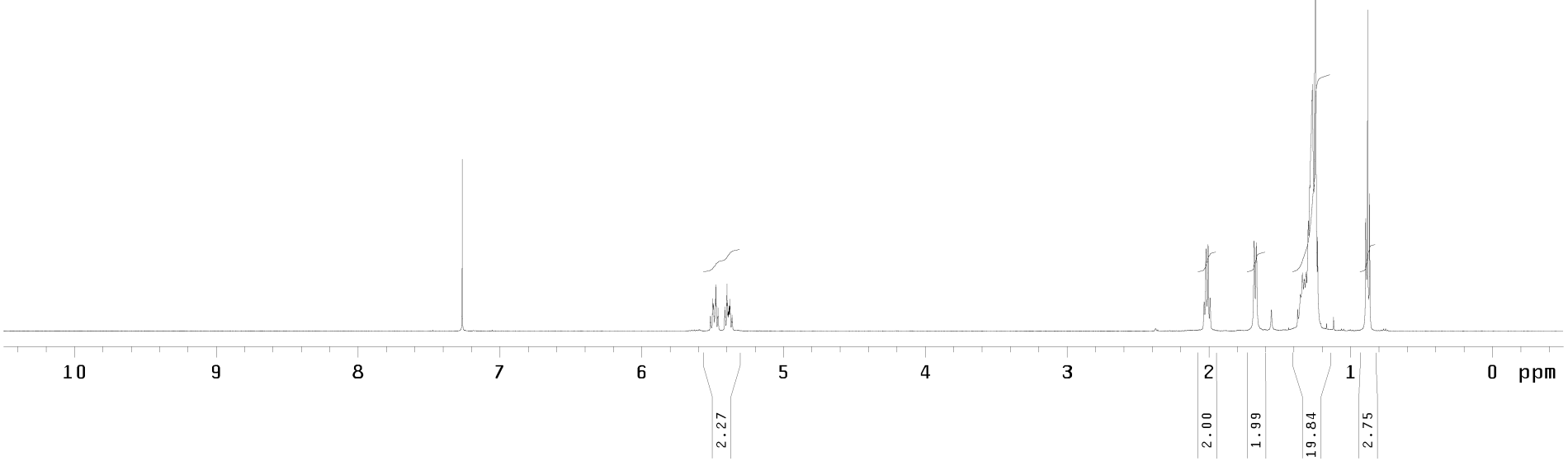

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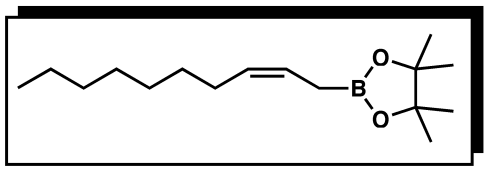
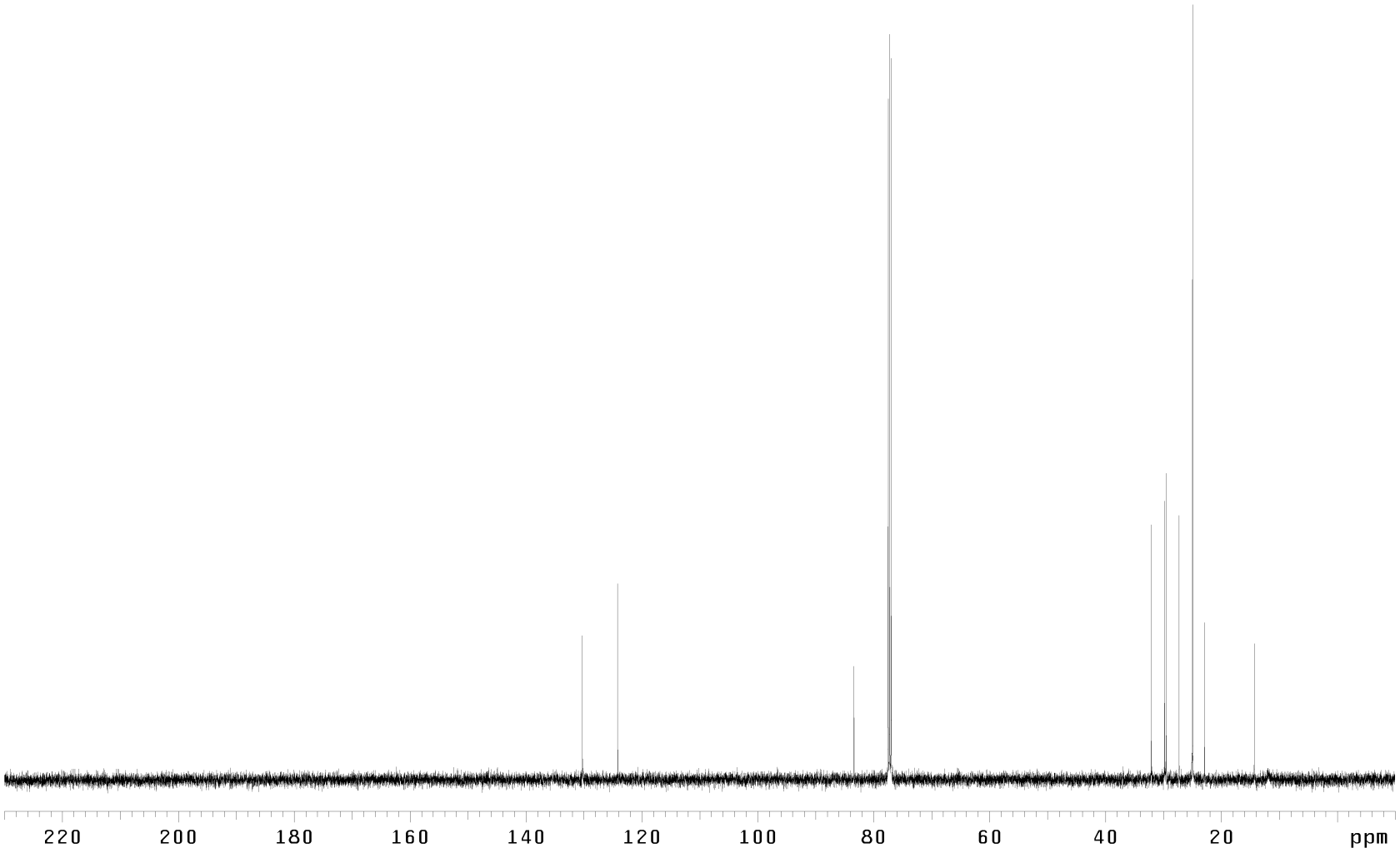

O
B
O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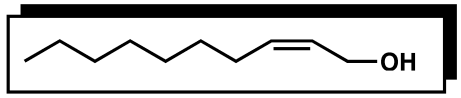
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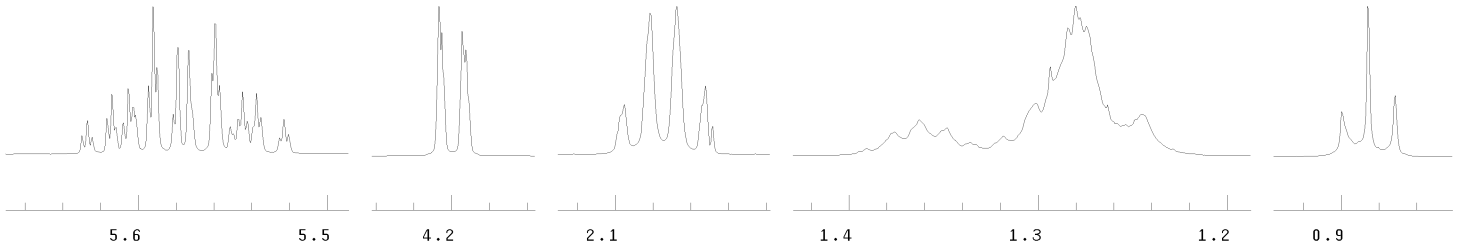
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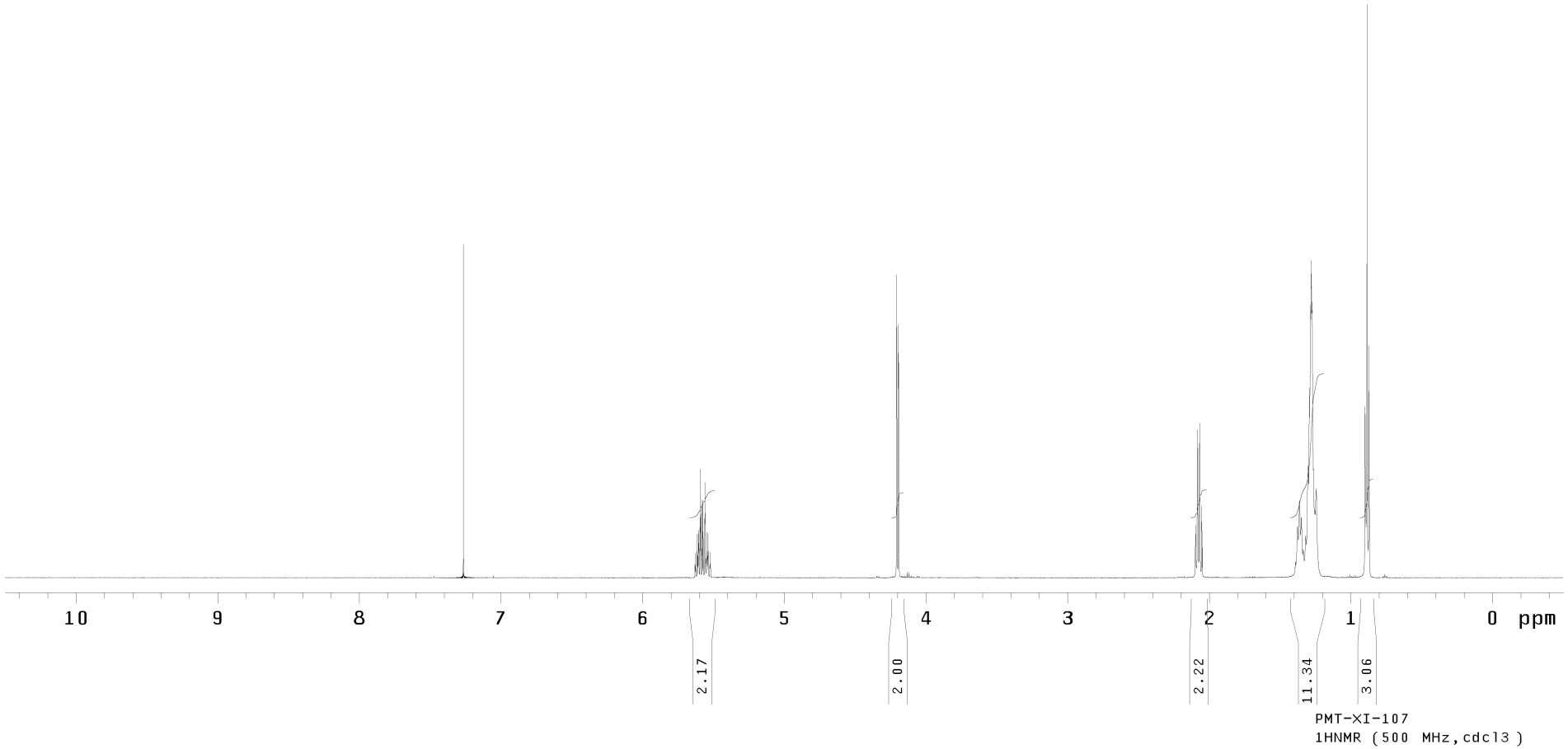
1.3
1.2
0.9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ppm
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2.22
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PMT-XI-107
1HNMR (500 MHz,cdcl3)