Organic Syntheses

A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

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.

SYNTHESIS OF SPIROBORATE ESTERS FROM 1,2-AMINOALCOHOLS, ETHYLENE GLYCOL AND TRIISOPROPYL BORATE: PREPARATION OF (*S*)-1-(1,3,2-DIOXABOROLAN-2-YLOXY)-3-METHYL-1,1-DIPHENYLBUTAN-2-AMINE.
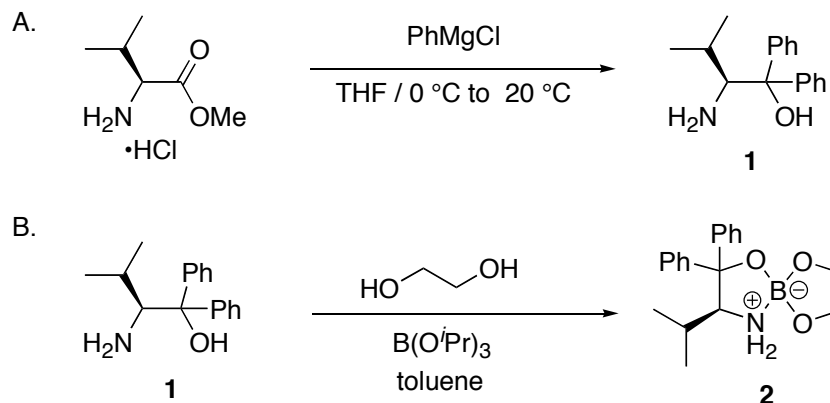
Submitted by Viatcheslav Stepanenko, Kun Huang and Margarita Ortiz-Marciales.[1]

Checked by David Hughes.[2]

CAUTION: Benzene is generated during the quench of Step A. All subsequent handling should be carried out in a well-ventilated hood.

1. Procedure

A.　*(2S)-2-Amino-3-methyl-1,1-diphenyl-butan-1-ol (1).*[3]　An oven-dried, 1-L, three-necked round-bottomed flask is fitted with rubber septa on two of the three necks and a hose adapter connected to a nitrogen line and bubbler on the third neck. A thermocouple temperature probe is inserted through one of the septa. Nitrogen is flowed through the flask while cooling to room temperature. To the flask is added via cannula, 2.0 M phenylmagnesium chloride in THF (300 g, 288 mL, 0.576 mol, 5.0 equiv) (Notes 1, 2, and 3). The mixture is stirred using a 2.5-cm oval Teflon-coated magnetic stir bar and cooled to –2 °C using an ice/acetone bath. *L*-Valine methyl ester hydrochloride (19.2 g, 114 mmol) is added portion-wise as a solid over 50 min while keeping the temperature below 5 °C. After addition, the cooling bath is removed. The solution is allowed to warm to room temperature over an hour and is stirred at room temperature for 2 h (Note 4). The mixture is cooled to 5 °C with an ice-bath, then carefully

hydrolyzed with half-saturated aqueous NH$_4$Cl (250 mL) (Note 5). Ethyl acetate (150 mL) and saturated NaCl (50 mL) are added to the flask and the mixture is stirred vigorously for 5 min, then the solids are allowed to settle. The aqueous and organic layers are decanted into a 1-L separatory funnel and the layers separated. The aqueous layer is returned to the reaction flask and extracted in a similar fashion with two 150-mL portions of EtOAc (Note 6). The organic layers are combined, washed with saturated NaCl (100 mL), and filtered through a bed of Na$_2$SO$_4$ (50 g) followed by an EtOAc rinse (100 mL) of the bed. The resulting clear solution is concentrated by rotary evaporation (20 mmHg, 40 °C bath) to afford a yellow solid (26.5 g), which is purified by column chromatography on silica gel (Note 7). The product is obtained as an off-white solid (18.1 g, 62% yield), which is used without further purification in the next step (Notes 8 and 9).

B.   *(-)-(S)-1-(1,3,2-Dioxaborolan-2-yloxy)-3-methyl-1,1-diphenylbutan-2-amine (2)*. An oven-dried, 3-necked 250-mL round-bottomed flask is fitted with a short reflux condenser (10 cm) on one outer neck, a stoppered 50-mL addition funnel on the middle neck, and a rubber septum on the other outer neck. A thermocouple temperature probe is inserted through the septum. A 2.5-cm oval Teflon-coated magnetic stirrer is added to the flask. A 24/40 hose adapter is attached to the top of the reflux condenser and connected to a nitrogen line and gas bubbler. The flask is charged with toluene (60 mL) followed by triisopropyl borate (3.88 g, 4.7 mL, 20.6 mmol) and ethylene glycol (1.26 g, 20.3 mmol), each added via syringe (Note 10). The mixture is stirred and heated with a heating mantle over 20 min to 80 °C, at which time the mixture becomes homogeneous. The solution is cooled to 60 °C, then a solution of (2*S*)-2-amino-3-methyl-1,1-diphenyl-butanol (5.20 g, 20.4 mmol) (**1**) in toluene (25 mL) is added via the addition funnel over 3 min, during which time the temperature decreases to 55 °C and white crystalline product is formed. The addition funnel is rinsed with toluene (5 mL). The resulting mixture is cooled to ambient temperature over 30 min. The mixture is transferred to a 500-mL round-bottomed flask and the mixture is concentrated to dryness by rotary evaporation (20 mmHg, 50 °C bath), and then dried for 15 h at 80 °C in a vacuum oven (20 mmHg) to yield 6.56 g (99%) of spiroborate ester **2** as a white solid (Notes 11 and 12).

2. Notes

1.   Reagents and solvents used in this preparation were sourced from Sigma-Aldrich and used without further purification, including 2.0 M phenylmagnesium chloride in THF, *L*-valine methyl ester hydrochloride (99%), ethyl acetate (ACS reagent grade, >99.5%), hexanes (ACS reagent grade, >98.5%), *n*-heptane (anhydrous, 99%), ethylene glycol (99.8%, anhydrous), toluene (ACS reagent grade, >99.5%, dried over pelleted 4 Å molecular sieves), triisopropyl borate (>98%, Sure/Seal^{TM}), and silica gel (200-400 mesh, 60 Å).

2.   The 1-L flask is marked prior to oven drying at a 280-mL fill to help estimate the amount added to the flask during cannulation. The amount of Grignard reagent added is determined by weighing the bottle before and after addition.

3.   This reaction can also be carried out with 4 equiv PhMgCl. A slightly lower yield is obtained (57% vs 62%).

4.   The reaction is followed by ^{1}H NMR as follows: a sample (0.2 mL) is quenched into saturated aqueous NH_4Cl (1 mL), extracted with $CDCl_3$, and the bottom organic layer is filtered through a cotton plug (to remove water) into an NMR tube. The set of 2 doublets of valine methyl ester at 0.9 ppm is diagnostic to assess complete reaction. All starting material is reacted (<1 %) after 1 h at room temperature.

5.   The initial addition of NH_4Cl is highly exothermic. The first 25 mL is added dropwise with the temperature controlled below 40 °C. Total addition time is 20 min.

6.   The solids (magnesium salts) cause poor layer separation, therefore it is important to decant the solution from the solids for improved separation.

7.   The chromatography is carried out using silica gel (150 g) and a 5-cm diameter flash column. The column is packed using 10:1 hexanes:EtOAc and topped with 0.5 cm of sea sand. The solid product from concentration is dissolved with sonication in CH_2Cl_2 (20 mL) and loaded onto the column. The product is eluted with hexane/ethyl acetate (8:1 v/v), collecting 75 mL fractions. TLC is used to monitor fraction collection, using 1:3 EtOAc:hexanes, UV and iodine visualization, with the product having an R_f of 0.3. The product tails off the column and is collected in about 1.5 L of eluent. The column is then eluted with 1:1 EtOAc:hexanes (1 L) which affords a mixture of by-products (4.3 g), which are discarded. Fractions 6-

25 containing pure product by TLC are combined and concentrated by rotary evaporation at 100 mmHg and 50 °C. Due to the formation of solids during concentration, bumping occurs if a higher vacuum is used.

8.   An analytically pure sample is prepared by recrystallization from *n*-heptane, as follows.   The product (5.0 g) from the chromatography is added to *n*-heptane (50 mL) and warmed to 80 °C to dissolve the solids. The mixture is slowly cooled to ambient temperature over 1 h and held at ambient temperature for 2 h.   The resulting solids are filtered and washed with *n*-heptane (10 mL) to afford 4.45 g (89%) of product.

9.   *(-)-(2S)-2-Amino-3-methyl-1,1-diphenyl-butan-1-ol (1)*: mp 98–99 °C, Lit[3a] 94–95 °C; $[\alpha]^{20}_D$ −126 (*c* 2, CHCl$_3$), Lit[3a] $[\alpha]^{25}_D$ −127.5 (*c* 0.639, CHCl$_3$); ^{1}H NMR (400 MHz, CDCl$_3$) δ: 0.90 (d, 3 H, *J* = 6.8 Hz, CH$_3$), 0.95 (d, 3 H, *J* = 7.0 Hz, CH$_3$), 1.10 (br s, 2 H, NH$_2$), 1.77 (septet of doublets, 1H, *J* = 2.2, 6.9 Hz, CH) 3.85 (d, 1 H, *J* = 2.2 Hz, NCH), 4.40 (br s, 1 H, OH), 7.15–7.21 (m, 2 H, Ph), 7.27–7.34 (m, 4 H, Ph), 7.50–7.53 (m, 2 H, Ph), 7.61–7.65 (m, 2 H, Ph); ^{13}C NMR (100 MHz, CDCl$_3$) δ: 16.3, 23.2, 28.0, 60.4, 79.9, 125.7, 126.1, 126.5, 126.8, 128.2, 128.6, 145.1, 148.2; Anal. Cald. for C$_{17}$H$_{21}$NO: C, 79.96; H, 8.29; N, 5.50. Found: C, 79.69; H, 8.62, N, 5.50.

10.   The amount added is determined by weighing the syringe before and after addition.

11.   The spiro-borate ester product can be isolated by filtration instead of concentration to dryness.   A 65% isolated yield of analytically pure material is obtained when the reaction mixture is vacuum-filtered through a 60-mL sintered glass funnel at ambient temperature.   Catalyst isolated in this manner is purer by NMR than material concentrated to dryness, but both perform comparably in the oxime ether asymmetric reduction (see accompanying procedure).

12.   *(-)-(S)-1-(1,3,2-Dioxaborolan-2-yloxy)-3-methyl-1,1-diphenylbutan-2-amine (2)*:   mp 209–211 °C; $[\alpha]^{20}_D$ −112 (*c* 2.0, DMSO); ^{1}H NMR (400 MHz, d$_6$-DMSO) δ: 0.38 (d, 3 H, *J* = 6.8 Hz, CH$_3$), 0.98 (d, 3 H, *J* = 6.8 Hz, CH$_3$), 1.86 (septet of doublets, 1 H, *J* = 3.0, 6.9 Hz, CH), 3.67 (m, 4 H, 2 × OCH$_2$), 3.80 (dt, 1 H, *J* = 3.0, 7.0 Hz, NCH), 5.0 (br s, 1H, NH), 6.0 (br s, 1H, NH), 7.12–7.29 (m, 6 H, Ph), 7.41–7.46 (m, 4 H, Ph); ^{13}C NMR (100 MHz, d$_6$-DMSO) δ: 16.2, 21.8, 27.5, 63.2, 64.0, 83.1, 126.2, 126.3, 126.8, 127.2 (2 degenerate C), 127.5, 145.3, 148.1; ^{11}B NMR (d$_6$-DMSO) δ: 12.9 (s); the catalyst is not stable in CDCl$_3$, therefore, dry d$_6$-DMSO is selected as the solvent for NMR; HRMS *m/z* calc for

$[C_{19}H_{25}O_3NB]^+$ 326.1928 ($[M+H]^+$), found 326.1924; Anal. Cald. for $C_{19}H_{24}BNO_3$: C, 70.17; H, 7.44, N, 4.31. Found: C, 70.25; H, 7.79, N, 4.26. The compound showed no change by NMR or decrease in reaction performance when kept in a sealed bottle for a month at ambient temperature.

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

Chiral organoborane reagents, in particular [1,3,2]-oxazaborolidines, have been extensively studied and applied as efficient Lewis acid catalysts to a wide range of asymmetric transformations.[4] The B-H oxazaborolidine-borane complexes are frequently reported as convenient catalysts for enantioselective borane reduction of prochiral ketones, imines and oximes, since they are readily prepared from the corresponding aminoalcohols and either borane-THF or borane-DMS complexes.[4a,c-d,5] However, the extreme sensitivity of these reagents to atmospheric moisture makes them difficult to isolate and purify. Consequently, they are prepared *in situ* prior to use in asymmetric reductions. Moreover, B-H oxazaborolidines can form dimers and other species which can affect the nature of catalyst.[6] Impurities present in the catalyst may lead to irreproducible results.[7] On the other hand, B-substituted oxazaborolidines show excellent synthetic utility due to their highly reproducible enantioselectivity, but require careful purification procedures to eliminate traces of boronic acid and their esters. Moreover, the commercially available reagents are expensive, moisture sensitive and unstable during extended storage. Accordingly, the design of new stable, easily available and efficient catalysts is always a challenging task in synthetic organic chemistry.

Recently, we prepared new oxazaborolidine-like spiroborate esters that were successfully applied for asymmetric borane reduction of prochiral ketones[8] and oxime ethers.[9] The spiroborate esters **2-10** were obtained by a simple procedure from commercially available chiral 1,2-aminoalcohols, ethylene glycol and triisopropyl borate with good purity and in essentially quantitative yields (Scheme 1). White crystalline spiroborate complexes **2**

and **3** were found to be particularly stable, since no changes were observed after exposure to moist air for 24 h at 25 °C, as evidenced by their ^{11}B, ^{1}H and ^{13}C NMR spectra.

Scheme 1
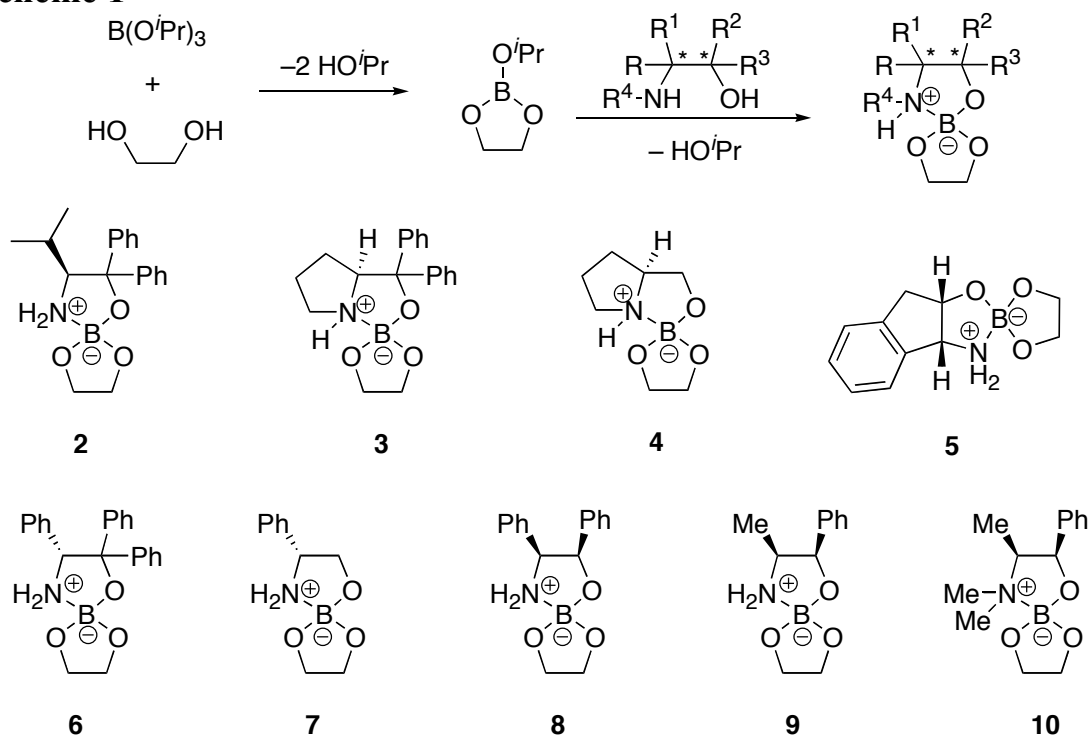
Catalysts **2** and **3** proved to be most valuable due to their convenience of handling and outstanding enantioselectivity in the asymmetric borane reduction of *O*-benzyl oximes ethers and ketones, respectively (Scheme 2). The application of spiroborate **2** for the synthesis of non-racemic secondary amines is presented in the accompanying procedure.

Scheme 2
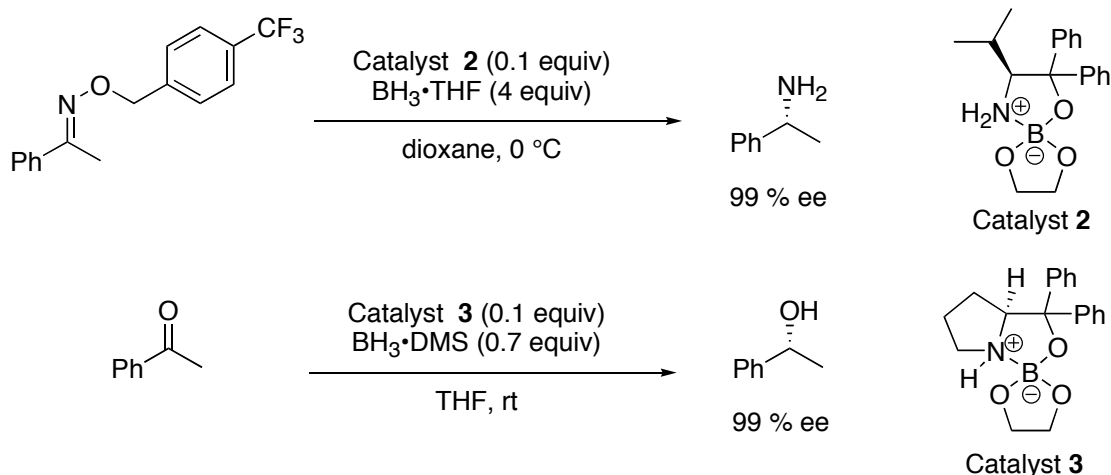
Table 1. Preparation of Chiral Alcohols via Borane Reduction Catalyzed by Spiroborate Ester **3**[a]

Entry	Cat. 3 (mol %)	Product	Yield[b] (%)	ee[c] (%)
1	10		84	99
2	10		99	>99
3	10		98	99
4	10		98	99
5	1		88	98
6	1		95	96
7	1		92	99
8[d]	10		97	>99
9[d]	1		96	98
10[d]	1		77	98
11[d]	1		92	99

[a] The reactions were carried out using 1 equiv of ketone (10 mmol), 0.1 or 0.01 equiv of catalyst **3** and 0.7 equiv of borane-DMS complex in THF at rt. [b] Isolated yield after chromatography or distillation. [c] Determined by GC of *O*-acetates on a chiral column (CP-Chirasil-Dex-CB). [d] 1.7 equiv of borane-DMS complex was used.

In addition, catalyst **3** demonstrated an excellent alternative for asymmetric reduction of a variety of ketones (Table 1), similar in enantioselectivity to those reported for the B-methyl CBS reagent. Moreover, the amount of catalyst load can be decreased to 1 mol % without significantly affecting the enantioselectivity.

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2. The checker would like to thank Dr. Peter Dormer for assistance with the NMR spectra.
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Appendix
Chemical Abstracts Nomenclature; (Registry Number)

(2*S*)-2-Amino-3-methyl-1,1-diphenyl-butanol; (78603-95-9)
Bromobenzene; (108-86-1)
L-Valine methyl ester hydrochloride; (6306-52-1)
Phenylmagnesium chloride, 2 M in THF; (100-59-4)
Ethylene glycol; (107-21-1)
Triisopropyl borate; (5419-55-6)
(–)-(*S*)-1-(1,3,2-Dioxaborolan-2-yloxy)-3-methyl-1,1-diphenylbutan-2-
    amine; (879981-94-9)

Margarita Ortiz-Marciales was born in 1943 in Bogotá, Colombia, and obtained her B.S. from Universidad Nacional de Colombia in 1968. She studied at Freiburg and Mainz universities, Germany, for two years with a DAAD fellowship. She received her M.S. from the University of Alabama in Huntsville under Prof. S. McManus supervision in 1973, and her Ph.D. in Organic Chemistry at the University of Alabama-Tuscaloosa in 1979 under the direction of Prof. McManus and Prof. R. Abramovitch. In 1980, she did postdoctoral studies in Prof. G. Larson's group at the University of Puerto Rico-Río Piedras. She joined the University of Puerto Rico-Humacao in 1981, where she is currently a professor. Her interests are in the development of new synthetic methodologies using boron and silicon compounds for the preparation of important organic intermediates and biologically-active amines.

Viatcheslav Stepanenko received his M.S. degree from Belarusian State University, Belarus in 1994. He obtained his Ph. D. in 2000 at Institute of Organic Chemistry, Polish Academy of Sciences, Poland, under Prof. J. Wicha supervision, working on the application of tandem Mukaiyama-Michael reaction for synthesis of Vitamin D. Then, he worked as a postdoctoral fellow at the University of Leeds, UK under the direction of Prof. P. J. Kocienski studying a copper-mediated rearrangement. In 2003 he moved to University of Puerto Rico where he was a postdoctoral research associate with Prof. Margarita Ortiz-Marciales until 2009, studying applications of new chiral organoborane reagents for asymmetric synthesis.

Kun Huang received his B.S. degree in 1996 from Sichuan University China. He obtained his Ph.D. in 2006 at Nanjing University, China, working on the asymmetric epoxidation and cyclopropanation of chiral sulfonium ylides. He worked as an advanced synthetic researcher in Wuxi Pharma Tech Co., Ltd, Shanghai, China until he moved to Puerto Rico University in Humacao for his postdoctoral studies with Professor Margarita Ortiz-Marciales on the asymmetric reduction of *O*-benzyl oximes and ketones catalyzed by spiroborate esters. Later, he had a postdoctoral research position at the chemistry department at Oregon State University where his research was focused on the total synthesis of natural products. Currently, he is a postdoctoral researcher at Peking University in China.

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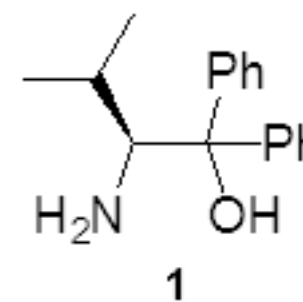
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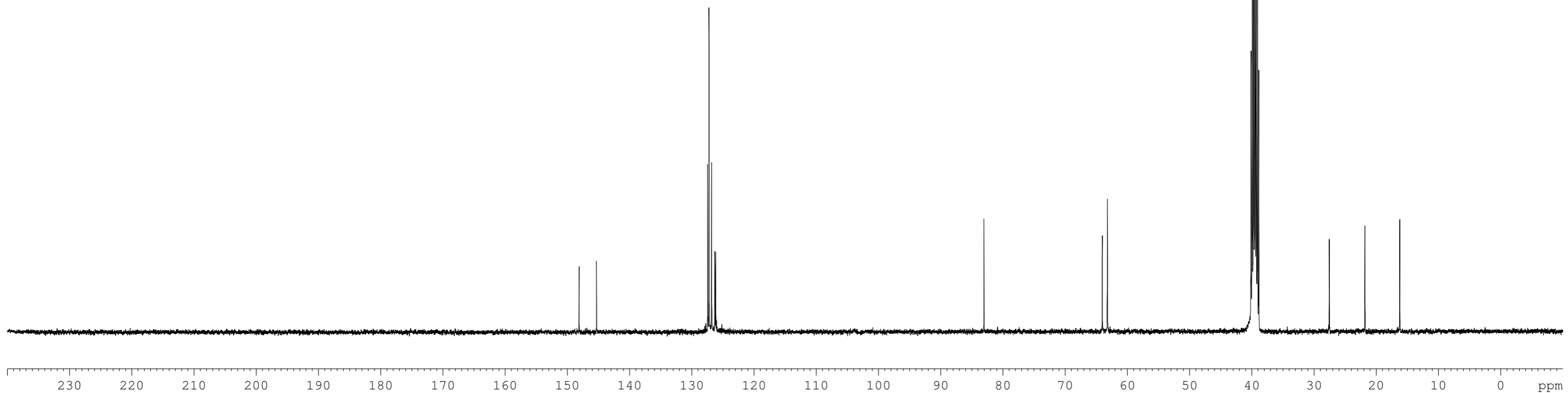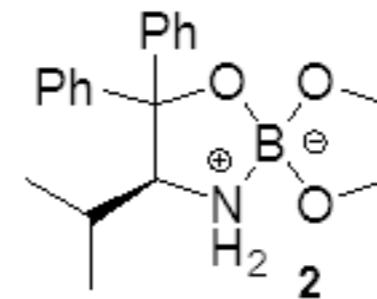GB   0
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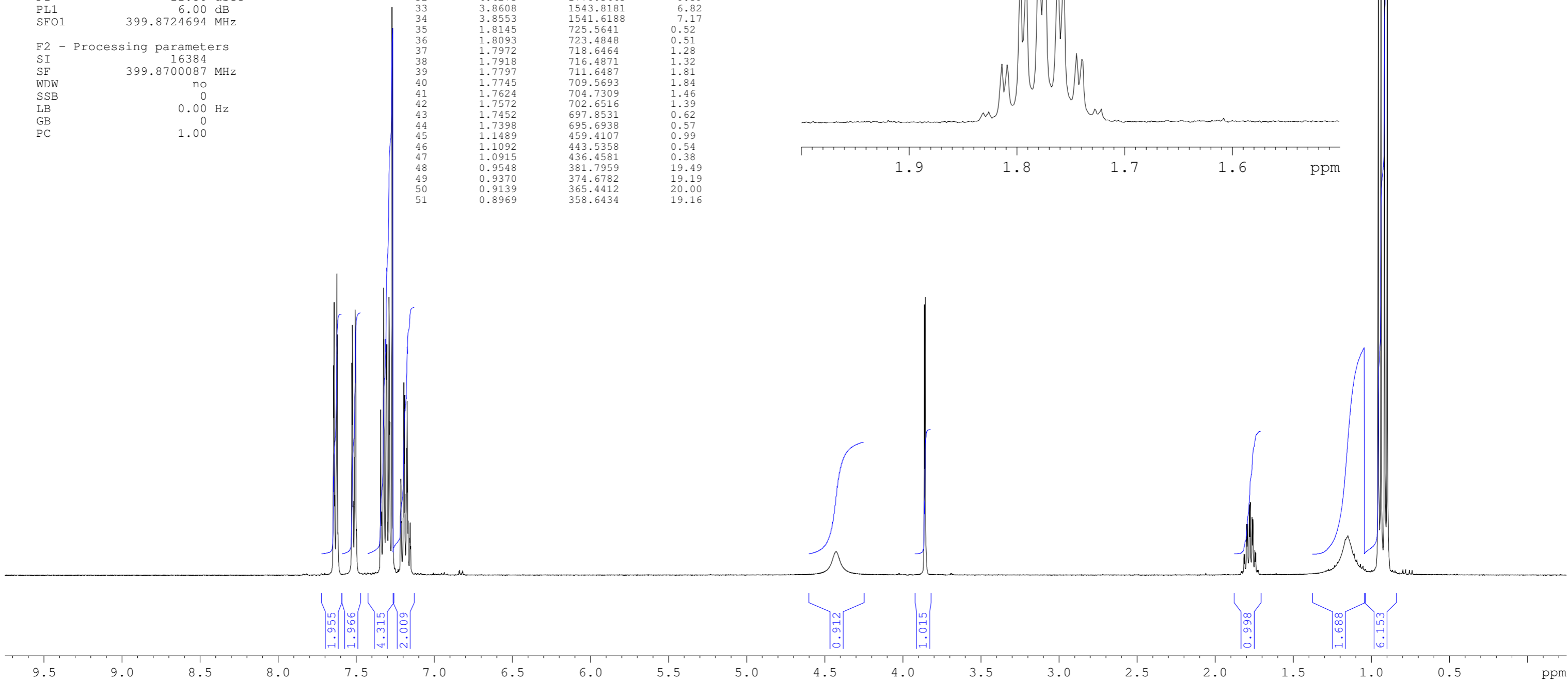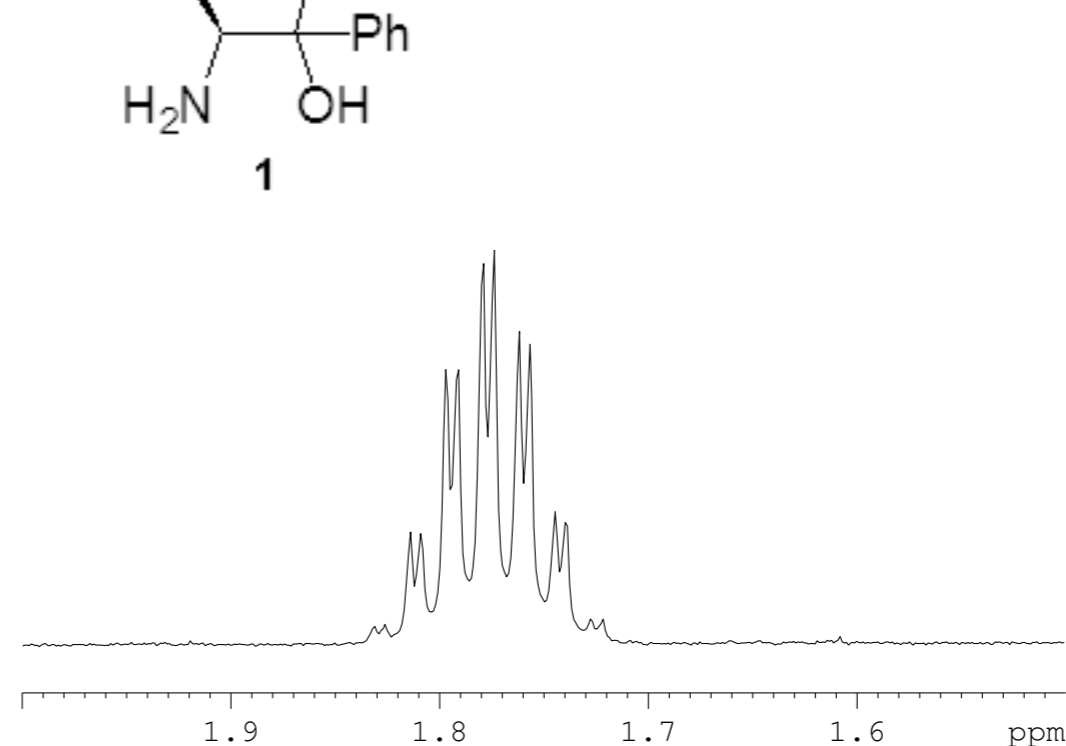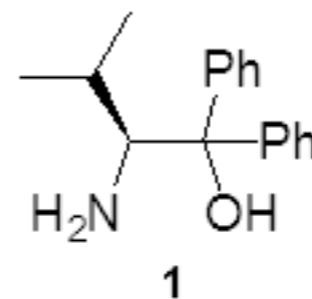
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NS   32
DS   2
SWH   6578.947 Hz
FIDRES   0.200774 Hz
AQ   2.4904180 sec
RG   287.4
DW   76.000 usec
DE   7.00 usec
TE   300.0 K
D1   0.10000000 sec
TD0   1

======== CHANNEL f1 ========
NUC1   1H
P1   11.50 usec
PL1   6.00 dB
SFO1   399.8724694 MHz

F2 - Processing parameters
SI   16384
SF   399.8700087 MHz
WDW   no
SSB   0
LB   0.00 Hz
GB   0
PC   1.00

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity
1	7.6448	3056.9262	5.24
2	7.6418	3055.7266	6.82
3	7.6370	3053.8073	2.03
4	7.6236	3048.4490	7.56
5	7.6211	3047.4493	6.31
6	7.6160	3045.4100	1.09
7	7.5272	3009.9015	5.32
8	7.5241	3008.6619	6.32
9	7.5193	3006.7426	1.85
10	7.5059	3001.3843	6.74
11	7.4985	2998.4253	1.17
12	7.3428	2936.1655	4.23
13	7.3377	2934.1262	1.61
14	7.3245	2928.8479	7.18
15	7.3229	2928.2081	6.13
16	7.3078	2922.1700	5.84
17	7.3043	2920.7705	5.77
18	7.2892	2914.7325	6.95
19	7.2856	2913.2929	3.93
20	7.2702	2907.1349	14.20
21	7.2137	2884.5423	2.44
22	7.2110	2883.4626	1.34
23	7.1950	2877.0647	4.82
24	7.1910	2875.4652	4.51
25	7.1881	2874.3056	2.05
26	7.1768	2869.7871	3.17
27	7.1731	2868.3076	4.42
28	7.1680	2866.2682	1.38
29	7.1575	2862.0696	0.94
30	7.1545	2860.8700	1.32
31	7.1511	2859.5104	0.76
32	4.4273	1770.3445	0.59
33	3.8608	1543.8181	6.82
34	3.8553	1541.6188	7.17
35	1.8145	725.5641	0.52
36	1.8093	723.4848	0.51
37	1.7972	718.6464	1.28
38	1.7918	716.4871	1.32
39	1.7797	711.6487	1.81
40	1.7745	709.5693	1.84
41	1.7624	704.7309	1.46
42	1.7572	702.6516	1.39
43	1.7452	697.8531	0.62
44	1.7398	695.6938	0.57
45	1.1489	459.4107	0.99
46	1.1092	443.5358	0.54
47	1.0915	436.4581	0.38
48	0.9548	381.7959	19.49
49	0.9370	374.6782	19.19
50	0.9139	365.4412	20.00
51	0.8969	358.6434	19.16

1

1.955 1.966 4.315 2.009 0.912 1.015 0.998 1.688 6.153

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity
4	7.4268	2969.7545	5.68
5	7.3035	2920.4505	2.94
6	7.2993	2918.7711	1.21
7	7.2851	2913.0929	6.17
8	7.2654	2905.2155	4.04
9	7.2539	2900.6170	3.07
10	7.2358	2893.3793	6.31
11	7.2159	2885.4219	4.76
12	7.2120	2883.8624	2.97
13	7.1936	2876.5048	3.35
14	7.1888	2874.5855	0.98
15	7.1755	2869.2672	3.46
16	7.1624	2864.0289	0.95
17	7.1574	2862.0295	3.32
18	7.1518	2859.7903	0.81
19	7.1393	2854.7919	1.04
20	3.8357	1533.7814	0.75
21	3.8282	1530.7823	0.86
22	3.8185	1526.9036	1.66
23	3.8107	1523.7846	1.67
24	3.8006	1519.7459	0.84
25	3.7931	1516.7469	0.71
26	3.7069	1482.2781	2.38
27	3.6738	1469.0424	2.41
28	3.3210	1327.9683	16.65
29	2.5255	1009.8717	6.44
30	2.5209	1008.0323	14.76
31	2.5164	1006.2329	20.00
32	2.5119	1004.4335	15.61
33	2.5073	1002.5941	7.49
34	2.3160	926.0989	0.42
35	1.9005	759.9529	0.75
36	1.8932	757.0339	0.78
37	1.8840	753.3551	1.05
38	1.8759	750.1161	1.05
39	1.8667	746.4373	0.84
40	1.8587	743.2384	0.80
41	1.0027	400.9496	9.91
42	0.9853	393.9919	9.49
43	0.4089	163.5068	10.36
44	0.3920	156.7490	10.24

Current Data Parameters
NAME   2009-047
EXPNO   1
PROCNO   1

F2 - Acquisition Parameters
Date_   20090607
Time   16.01
INSTRUM   spect
PROBHD   5 mm QNP 1H/1
PULPROG   zg30
TD   32768
SOLVENT   DMSO
NS   32
DS   2
SWH   6578.947 Hz
FIDRES   0.200774 Hz
AQ   2.4904180 sec
RG   812.7
DW   76.000 usec
DE   7.00 usec
TE   300.0 K
D1   0.10000000 sec
TD0   1

======== CHANNEL f1 ========
NUC1   1H
P1   11.50 usec
PL1   6.00 dB
SFO1   399.8724694 MHz

F2 - Processing parameters
SI   16384
SF   399.8700000 MHz
WDW   no
SSB   0
LB   0.00 Hz
GB   0
PC   1.00

2009-047
isolated boronate
nmr400b h-1