Practical synthesis of 6-amino-1-hydroxy-2,1benzoxaborolane: a key intermediate of DNDI-6148

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GENERAL METHODS

Reagents and solvents were obtained from commercial suppliers and used as received unless otherwise indicated. Reactions were carried out in oven-dried (120 °C) glassware, that was assembled while hot, and cooled to ambient temperature under an inert atmosphere. All reactions were carried out under inert atmosphere (N_2) unless otherwise noted. Reactions were monitored by TLC (precoated silica gel 60 F254 plates, EMD Chemicals), HPLC or LC/MS using various methods. TLC was visualized with UV light or by treatment with Phosphomolybdic acid (PMA), ninhydrin, and/or KMnO₄. Flash chromatography was performed on a Teledyne ISCO Combi-Flash NEXTGEN 300+ and/or a Biotage Isolera using solvents as indicated. HRMS was recorded using Perkin Elmer Axion 2 ToF MS, ionization mode: positive with scan range: 100 - 1000 m/z, flight tube voltage: 8 kV, spray voltage: 3.5 kV, solvent: methanol.¹HNMR and ¹³CNMR spectra were routinely recorded on Bruker Avance III HD Ascend 600 MHz spectrometer. The NMR solvents used were CDCl₃, CD₃OD or DMSO-d₆ as indicated. Tetramethylsilane (TMS) was used as an internal standard. Coupling constants J are reported in hertz (Hz). The following abbreviations were used to designate signal multiplicity: s, singlet; d, doublet; t, triplet; q, quartet, p, pentet; dd, doublet of doublets; dd, doublet of doublet of doublets; dt, double of triplets; ddt, doublet of doublet of triplets; m, multiplet; br, broad. 1,3,5-trimethoxybenzene and/or triphenylmethane, were used as internal standards for quantitative ¹H-NMR.

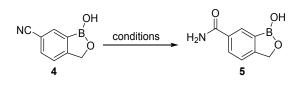
HPLC/LCMS Method

LC with UV detection was used for the analysis of reaction mixtures and isolated product and intermediates. An Agilent 1100 series LC equipped with a diode array detector was used with the parameters shown below.

Column	Phenomenex Kinetex Phenyl Hexyl (150 x 4.6 mm; 5 µm)
Flow Rate	2.0 mL/min
Column Temperature	40 °C
Injection Volume	1 μL
Mobile Phase A	0.1% H ₃ PO ₄ in water*
Mobile Phase B	Methanol

Gradient Table	Time (min)	%A	%В
	0.0	80%	20%
	9.0	25%	75%
	12.0	25%	75%
Post-Run Equilibration	2 min		
Detection Wavelength	210 nm		

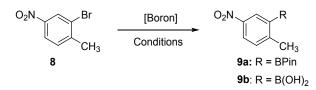
Table S1. Synthesis of amide 5 by hydrolysis of nitrile 4.



Entry	Conditions	Isolated yield of 5
1 (4 : 1 g)	NaOH, iPrOH, 90 °C, 16h	~18 A%
2 (4 : 1 g)	LiOH, iPrOH, 90 °C, 16h	NR
3 (4 : 1 g)	KOH, iPrOH, 90 °C, 16h	~60 A% (failed to isolate)
4 (4 : 1 g)	H ₂ SO ₄ , 90 °C, 1h	49%
5 (4 : 10 g)	H ₂ SO ₄ , 90 °C, 1h	53%
6 (4 : 1 g)	MsOH, 90 °C, 16h	72%
7 (4 : 10 g)	MsOH, 90 °C, 16h	78%
8 (4 : 1 g)	TFA, 90 °C, 16h	31%
9 (4 : 1 g)	TfOH, 90 °C, 16h	5%ª
10 (4 : 1 g)	H ₂ SO ₄ , 90 °C, 16h	O ^a
11 (4 : 1 g)	HCl, 90 °C, 16h	12 ^b
11 (4 : 1 g)	NaHSO ₄ , 90 °C, 16h	NR
11 (4 : 1 g)	NaHSO ₃ , 90 °C, 16h	NR

^{*a*}SM was consumed. Decomposition occurred. ^{*b*}76 A% carboxylic acid was formed.

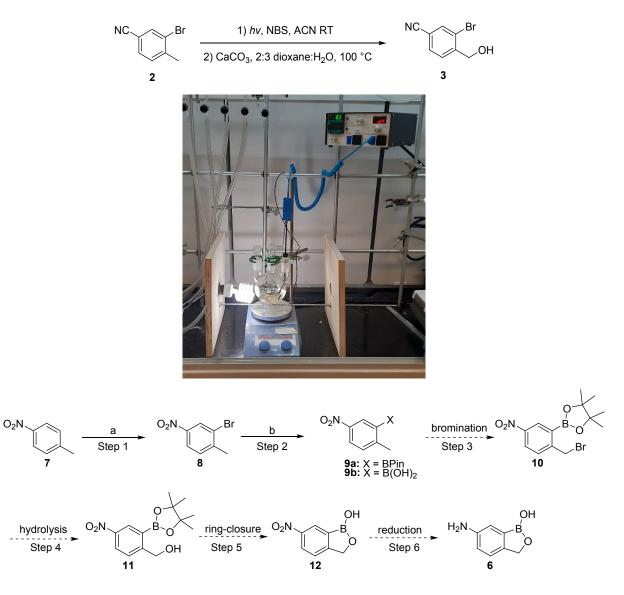




Entry ^a	[Boron]	Conditions	Solvent	Temp /ºC	A% ^{<i>b</i>}
1	B(OMe) ₃	iPrMgCl-LiCl	THF	-78	ND
2	B(OiPr) ₃	iPrMgCl-LiCl	THF	-78	ND
3	B(OiPr) ₃	n-BuLi	THF	-78	ND
4	iPrO-BPin	n-BuLi	THF	-78	ND
5	BH ₃ -NH(iPr) ₂	Mg, PhLi	THF	70	ND
6	B(OiPr) ₃	Mg	THF	-78	NR
7	B ₂ Pin ₂	5 mol% Pd(PPh ₃)Cl ₂ , PPh ₃ , KOAc	1,4-Dioxane	100	20
8	B ₂ Pin ₂	5 mol% Pd(OAc) ₂ , PPh ₃ , KOAc	1,4-Dioxane	100	ND
9	B ₂ Pin ₂	5 mol% Pd(OAc) ₂ , Xphos, KOAc	EtOH	80	10
10	B ₂ Pin ₂	5 mol% Pd(dppf)Cl ₂ , KOAc	1,4-Dioxane	100	>90
11	B ₂ Pin ₂	1% Pd(dppf)Cl2, KOAc	1,4-Dioxane	100	~30
12	B ₂ Pin ₂	1% Pd(OAc)2, dppf, KOAc	1,4-Dioxane	100	~56
13	B ₂ Pin ₂	0.1% Pd(dppf)Cl2, KOAc	1,4-Dioxane	100	~7
14	B ₂ Pin ₂	10 mol% Pd(OAc)2	1,4-Dioxane	100	ND
15	B ₂ Pin ₂	10 mol% PdCl2	1,4-Dioxane	100	ND
16	B ₂ Pin ₂	10 mol% Pd/C	1,4-Dioxane	100	ND
17	B ₂ (OH) ₄	5 mol% Pd(PPh3)Cl ₂ , PPh ₃ , KOAc	EtOH	80	ND
18	B ₂ (OH) ₄	5 mol/% Pd(OAc)2, Xphos,, KOAc	EtOH	80	20
19	B ₂ (OH) ₄	5 mol% Pd(dppf)Cl2, KOAc	EtOH	80	ND
20	B ₂ (OH) ₄	5 mol% Xphos-Pd-G2, KOAc	EtOH	80	ND

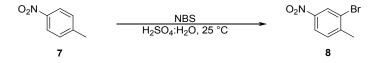
^aReaction conditions: For metal-halo exchange borylation: organometal (1.5 eq) and/or Mg (2 eq), 2bromo-1-methyl-4-nitrobenzene (1.0 eq.), [Boron] (2 eq.), Solvent (10V); For Pd-borylation: Pd-Catalyst (X mol%), 2-bromo-1-methyl-4-nitrobenzene (1.0 eq.), [Boron] (1.1 eq.), potassium acetate (2.0 eq.), Solvent (10V). ^bYields were calculated using HPLC A% at 210 nm.

Setup for synthesis of 3-bromo-4-(hydroxymethyl)benzonitrile (3) from 3-bromo-4methylbenzonitrile (2)



Reagents and conditions: (a) NBS, H₂SO₄, rt, 92%; (b) B₂Pin₂, 5 mol% Pd(dppf)Cl₂, KOAc, toluene, 100 °C, 90%.

Synthesis of 2-Bromo-4-nitrotoluene (8)¹



To a round bottom flask equipped with a magnetic stir bar, a mixture of p-nitrotoluene 7 (10.0 g, 1.0 eq. 72.9 mmol) and aqueous sulfuric acid (80 mL, 1:1 ratio by volume of conc. H_2SO_4 and water) were added. The flask was wrapped with aluminum foil so that darkness might prevent competitive radical reactions. The mixture was stirred for 10 minutes, then N-bromosuccinimide (15.5 g, 1.2 eq. 87.5 mmol)

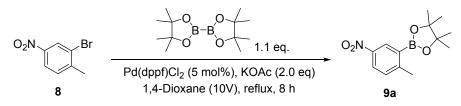
was added slowly (portion wise, over 20 minutes). The mixture was stirred at room temperature for 24 h. After completion (monitored by HPLC), the product was extracted by EtOAc (50 mL x 3). The combined organic phase was washed with brine and dried over anhydrous sodium sulfate. Solvent was removed to afford 2-Bromo-4-nitrotoluene **8** (14.42 g, 92% yield) as a yellow solid.

¹**H NMR** (600 MHz, CDCl₃) δ/ppm: 8.37 (s, 1H), 8.05 (d, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 8.2 Hz, 1H), 2.49 (s, 3H).

¹³C NMR (150 MHz, CDCl₃) δ/ppm: 146.7, 146.0, 131.2, 127.5, 125.1, 122.3, 23.4.

Melting Point 74-76 °C.

Synthesis of 4,4,5,5-tetramethyl-2-(2-methyl-5-nitrophenyl)-1,3,2-dioxaborolane (9a):



A mixture of Pd(dppf)Cl₂ (0.34 g, 5 mol%, 462.9 μ mol), 2-bromo-1-methyl-4-nitrobenzene **8** (2.0 g, 1.0 eq, 9.2 mmol), bis(pinacolato)diboron (2.58 g, 1.1 eq, 10.2 mmol), and potassium acetate (1.82 g, 2.0 eq, 18.5 mmol) in 1,4-dioxane (20 mL) was degassed with N₂ at rt for 5 min, then the mixture was refluxed under nitrogen for 8 h. After completion (monitored by HPLC), the mixture was extracted with EtOAc (20 mL x 3). The organic layers were combined and washed sequentially with water and brine. The organic ¹phase was separated and dried over anhydrous Na₂SO₄, and evaporated to dryness by rotary evaporation. The residue was stirred in hexanes (20 mL) for 30 min at rt, then the solid was collected by filtration and washed with hexanes (10 mL x 3) to afford a pure compound **9a**, 2.1 g, 86% yield.

¹**H NMR** (600 MHz, CDCl₃) δ/ppm: 8.59 (d, *J* = 2.6 Hz, 1H), 8.12 (dd, *J* = 8.4, 2.6 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 1H), 2.62 (s, 3H), 1.35 (s, 12H).

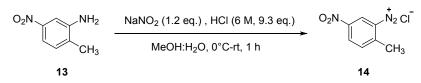
¹³C NMR (150 MHz, CDCl₃) δ/ppm: 152.9, 145.8, 130.8, 125.5, 84.4, 25.0, 22.6.

MS (m/z) (M+H): calc. for C₁₃H₁₉BNO₄ 264, found 264.

Melting Point 89-92 °C.

¹Wagner, P. J.; Wang, L. Electronic Effects of Ring Substituents on Triplet Benzylic Biradicals. *Org. Lett.* **2006**, *8*, 645–647.

General procedure for the synthesis of diazonium salt (14) for DSC/TGA studies



To an ice-cold solution of 2-methyl-5-nitroaniline **13** (1.0 g, 1.0 eq, 6.8 mmol) in methanol (20 mL) was added aq. HCl (10.2 mL, 6.0 M, 9.3 eq, 61.1 mmol). To this mixture was added a solution of sodium nitrite (0.59 g, 1.3 eq, 8.5 mmol) in water (10 mL) dropwise using addition funnel. The resulting mixture was stirred at 0 °C for 1h (Monitored by TLC, HPLC). After completion, the solid was collected by filtration and washed with methanol to get the pure diazonium salt as a brown solid **14** (1.29g, 98%).

¹**H NMR** (600 MHz, CD₃OD) δ/ppm: 9.64 (d, *J* = 2.3 Hz, 1H), 8.93 (dd, *J* = 8.7, 2.3 Hz, 1H), 8.15 (d, *J* = 8.7 Hz, 1H), 2.98 (s, 3H).

¹³**C NMR** (150 MHz, CD₃OD) δ/ppm: 141.9, 138.1, 126.1, 125.9, 119.4, 109.3, 9.6.

HRMS (ESI) m/z: [M]⁺ Calcd for C₇H₆N₃O₂⁺ 164.0455; Found 164.0456.

Melting Point 94-96 °C.

DSC and TGA studies of Diazonium salt 14:

Instrument: TA SDT 650 Sample holder: TA Alumina 90 uL sample cup Scan temperature range: 30 °C to 250 °C Scan rate: 10 °C/min Purge: Nitrogen, 100 mL/min

For the DSC-TGA analysis, samples were analyzed as received in alumina 90 μ L sample cups. Approximately 5 mg of sample was added to the sample cup after tearing. The thermogram ran from 30-250 °C at a ramp of 10 °C/min.

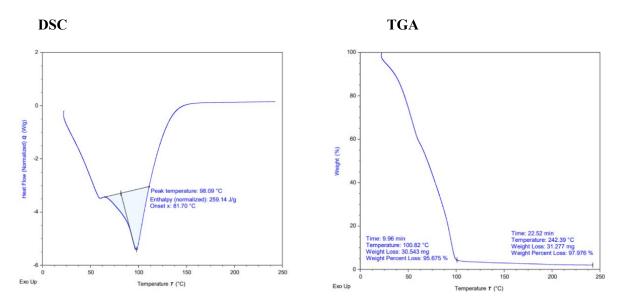


Figure S1. DSC (left) and TGA (right) results of the diazonium salt collected directly from a reaction mixture.

DSC

TGA

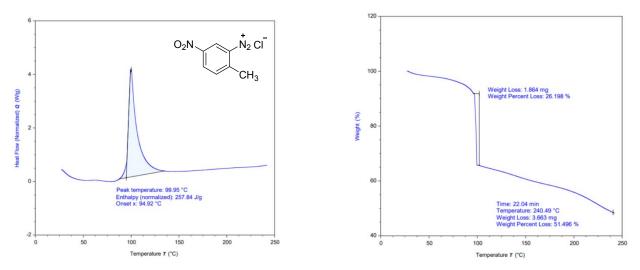
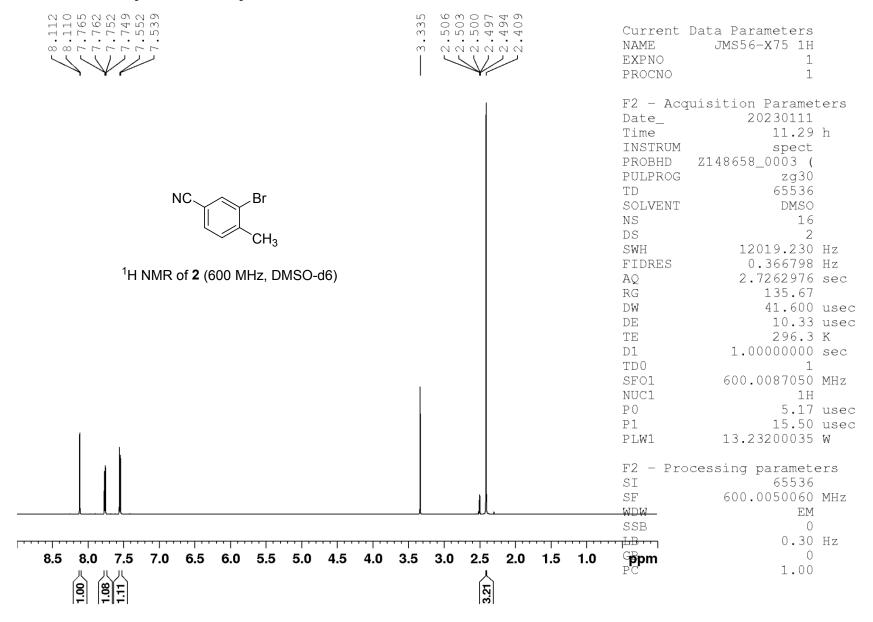
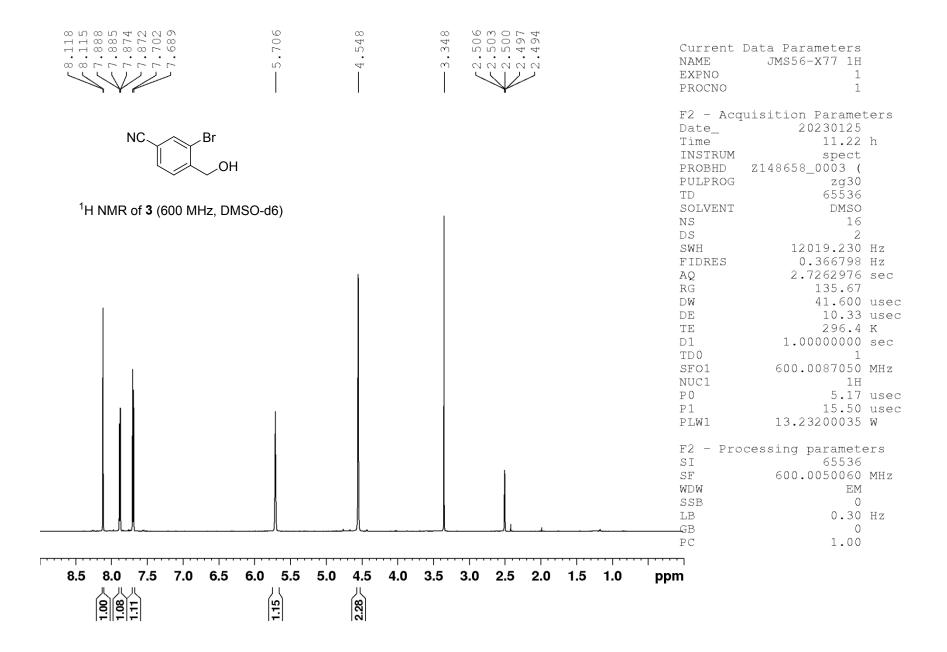


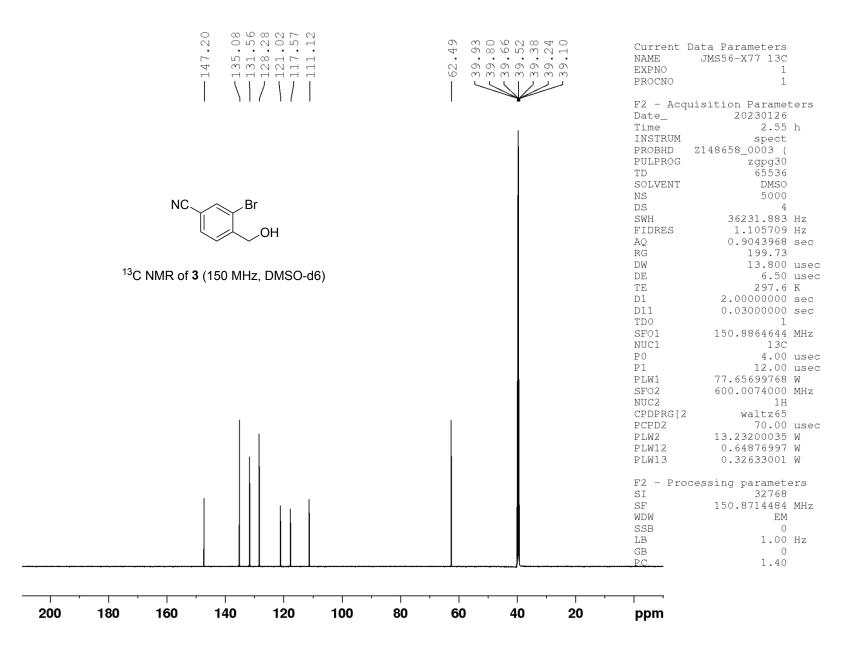
Figure S2. DSC (left) and TGA (right) results of the purified diazonium salt 14.

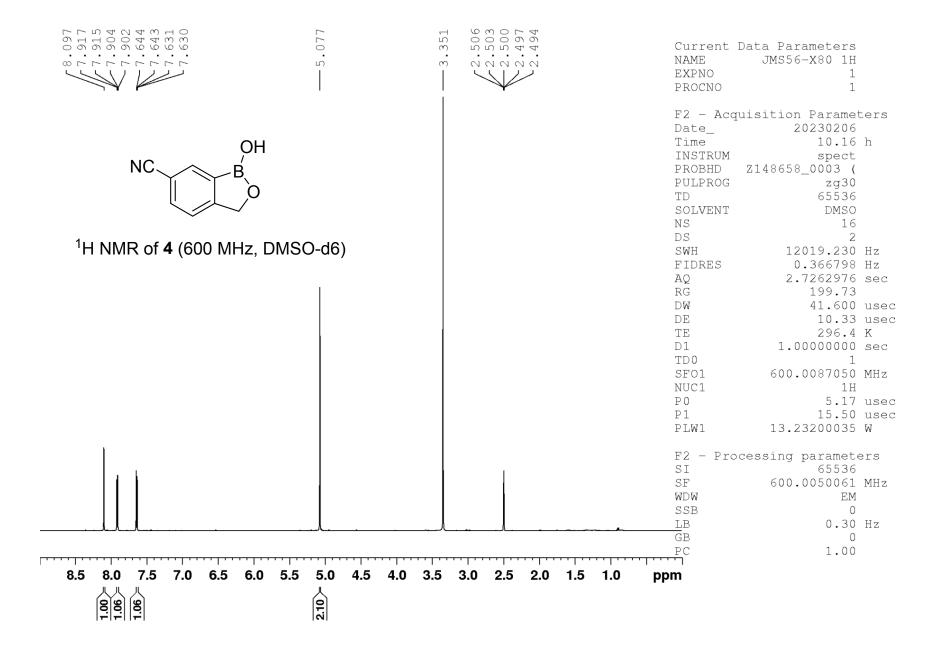
¹H and ¹³C NMR Spectra of all compounds

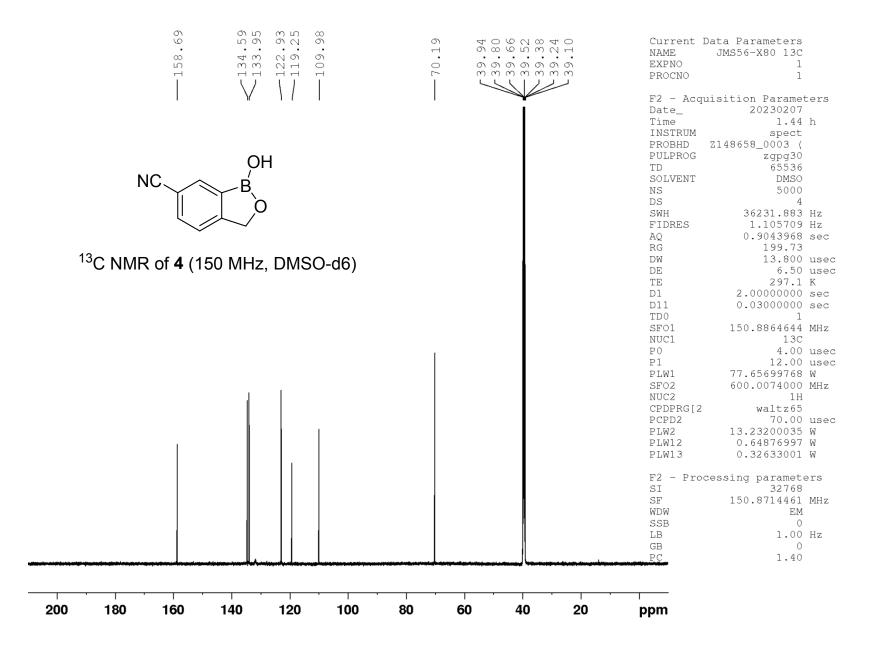


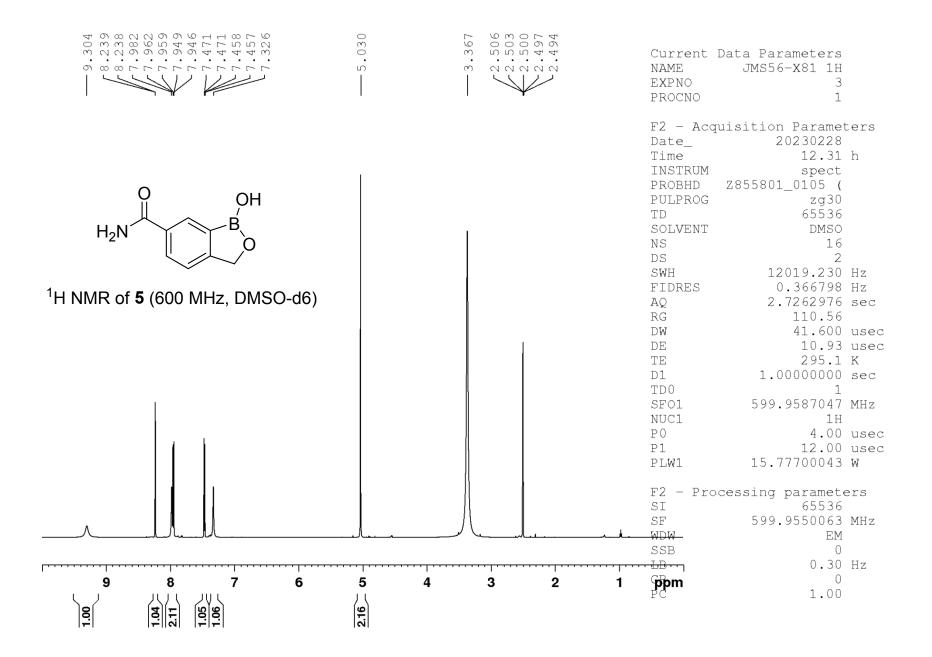
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		TD	65536
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		FIDRES	1.105709 Hz
		AQ	0.9043968 sec
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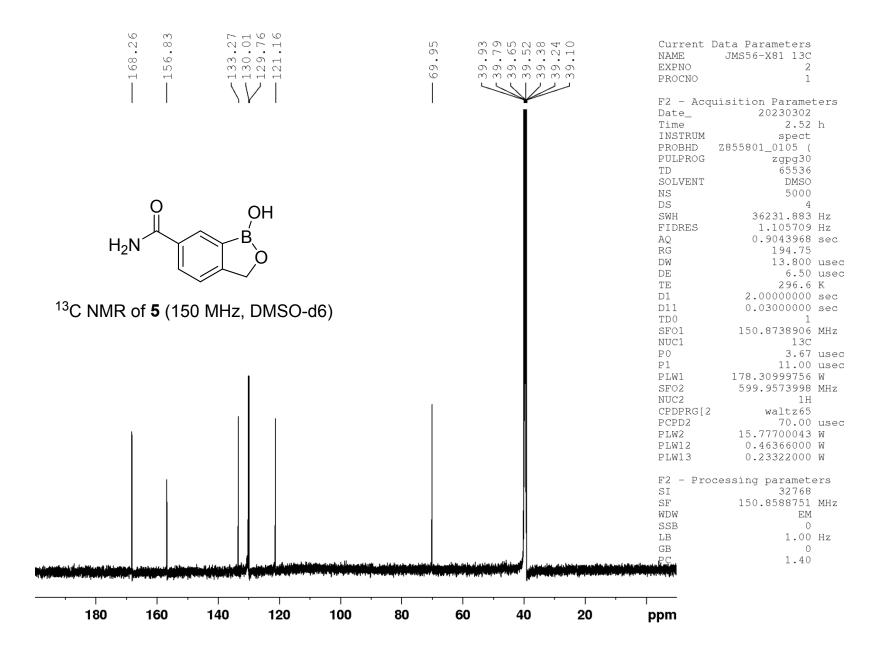


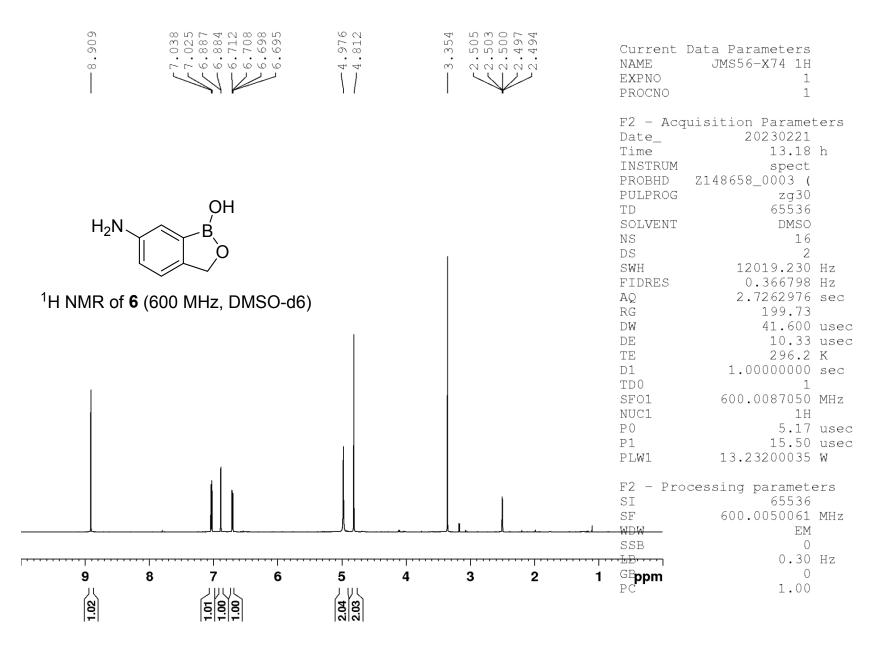




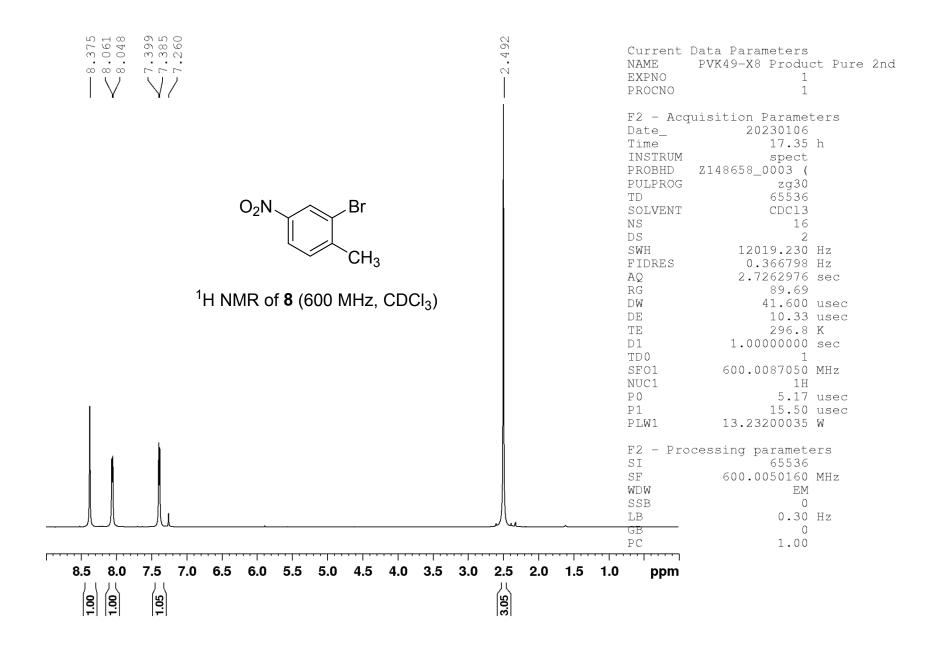








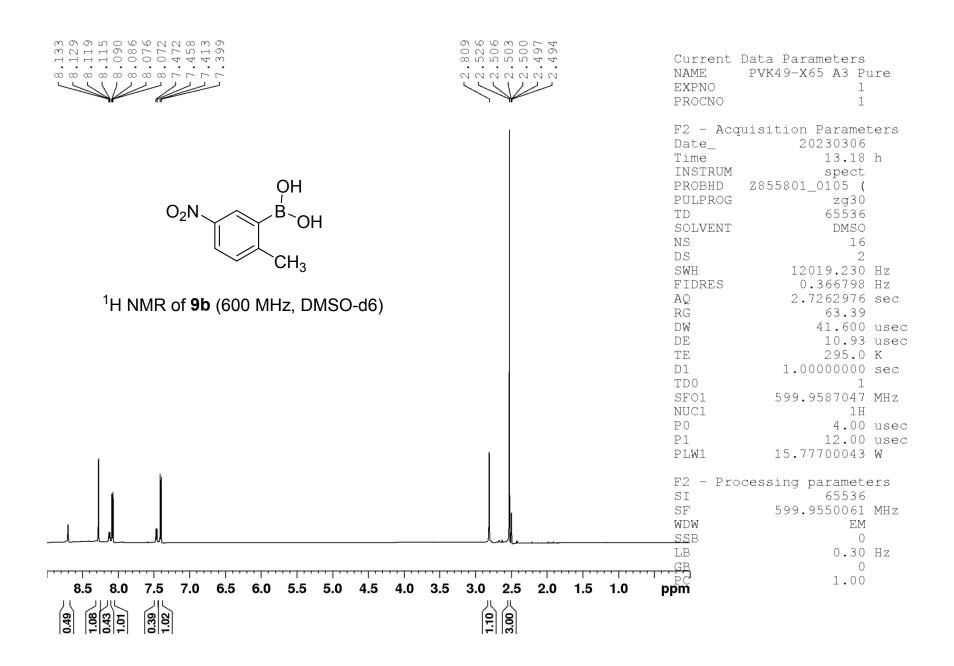
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				SF02	599.9573998 MHz
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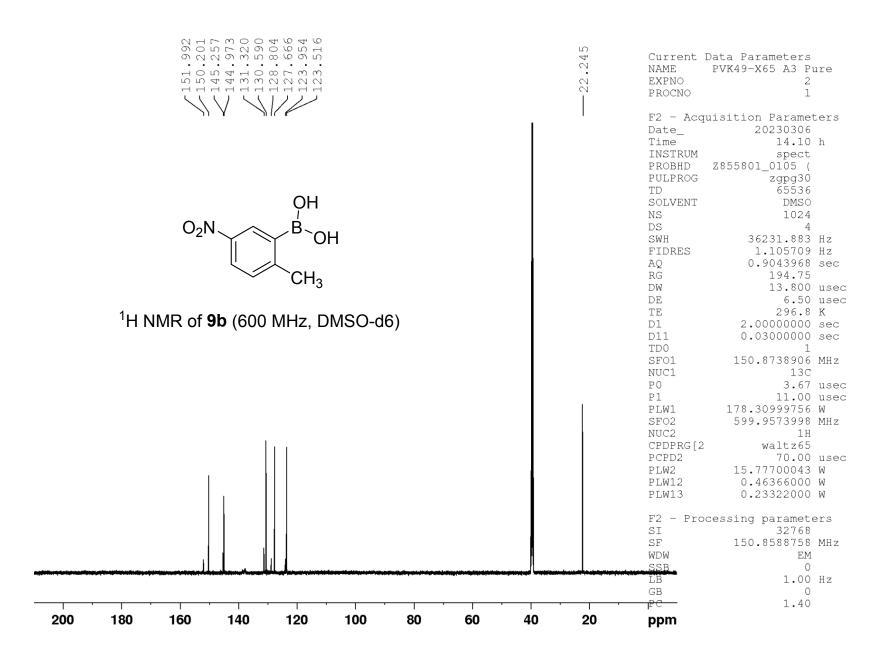


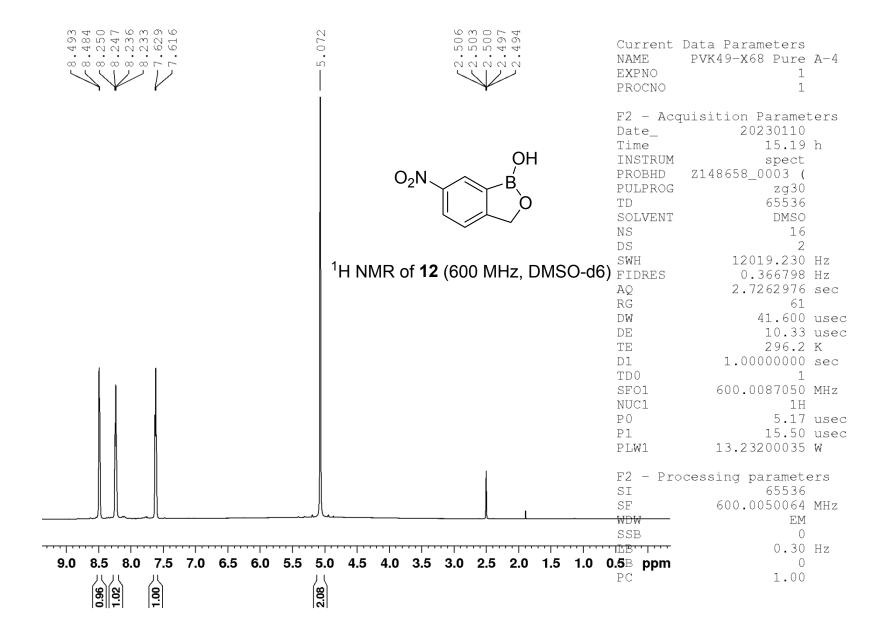
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AQ 0.9043968 sec RG 199.73 DW 13.800 usec DE 6.50 usec TE 298.2 K D1 2.0000000 sec D11 0.0300000 sec TD0 1 SF01 150.8864644 MHz NUC1 13C P0 4.00 usec P1 12.00 usec P1 12.00 usec P1 12.00 usec P1W1 77.65699768 W SF02 600.0074000 MHz NUC2 1H CPDPRG[2 waltz65 PCPD2 70.00 usec PLW13 0.32633001 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0			C NIVIN OF 0 (150 IVITIZ, CDCI3)		
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DE 6.50 usec TE 299.2 K D1 2.0000000 sec D11 0.0300000 sec TD0 1 SF01 150.8864644 MHz NUC1 13C P0 4.00 usec P1 12.00 usec P1 12.00 usec PLW1 77.65699768 W SF02 600.0074000 MHz NUC2 1H CPDPRG[2 waltz65 PCCPD2 70.00 usec PLW2 13.2320035 W PLW12 0.64876997 W PLW12 0.64876997 W PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
TE 298.2 K D1 2.0000000 sec D1 0.0300000 sec TD0 1 SF01 150.8864644 MHz NUC1 13C P0 4.00 usec P1 12.00 usec P1 0.000000 MHz NUC2 14 NUC2 14 NUC2 14 NUC2 10.00 MHz NUC2 13.23200035 W PLW12 0.6487697 W PLW13 0.32633001 W F2 Processing parameters S1 32768 SF 150.6713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
D1 2.0000000 sec D11 0.0300000 sec TD0 1 SF01 150.8864644 MHz NUC1 13C P0 4.00 usec P1 12.00 usec P1 12.00 usec P1W1 77.65699768 W SF02 600.0074000 MHz NUC2 1H CPDPRG[2 waltz65 PCPD2 70.00 usec PLW2 13.23200035 W PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					298 2 K
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TD0 1 SF01 150.8864644 MHz NUC1 13C P0 4.00 usec P1 12.00 usec P1 77.656997680 SF02 600.0074000 MHz NUC2 1H CPDPRG[2 waltz65 PLW2 13.23200035 W PLW12 0.64876997 W PLW13 0.32633001 W F2 Processing parameters SI 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
NUC1 13C P0 4.00 usec P1 12.00 usec P1 77.65699768 W SF02 600.0074000 MHz NUC2 11 CPDPRG[2 waltz65 PCPD2 70.00 usec PLW2 13.23200035 W PLW12 0.64876997 W PLW13 0.326801 W SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
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P1 12.00 usec PLW1 77.65699768 W SF02 600.0074000 MHz NUC2 1H CPDPRG[2 waltz65 PCPD2 70.00 usec PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0				NUC1	13C
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SF02 600.0074000 MHz NUC2 1H CPDPRG[2 waltz65 PCPD2 70.00 usec PLW2 13.23200035 W PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM WDW EM SSB 0 LB 1.00 Hz GB 0					
NUC2 1H CPDPRG[2 waltz65 PCPD2 70.00 U2 13.23200035 W PLW12 0.64876997 W PLW13 0.32633001 W F2 Processing parameters SI 32768 SF 150.8713585 MDW EM SSB 0 LB 1.00 Hz GB O 0					
CPDPRG[2 waltz65 PCPD2 70.00 usec PLW2 13.23200035 W PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
PCPD2 70.00 usec PLW2 13.23200035 W PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
PLW2 13.23200035 W PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0	1				
PLW12 0.64876997 W PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
PLW13 0.32633001 W F2 - Processing parameters SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					0 64876997 W
SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0					
SI 32768 SF 150.8713585 MHz WDW EM SSB 0 LB 1.00 Hz GB 0				F2 - Proc	essing parameters
WDW EM SSB 0 LB 1.00 Hz GB 0					32768
WDW EM SSB 0 LB 1.00 Hz GB 0				SF	150.8713585 MHz
LB 1.00 Hz GB 0					EM
GB 0					
1.40				GB	
				EC	1.4U

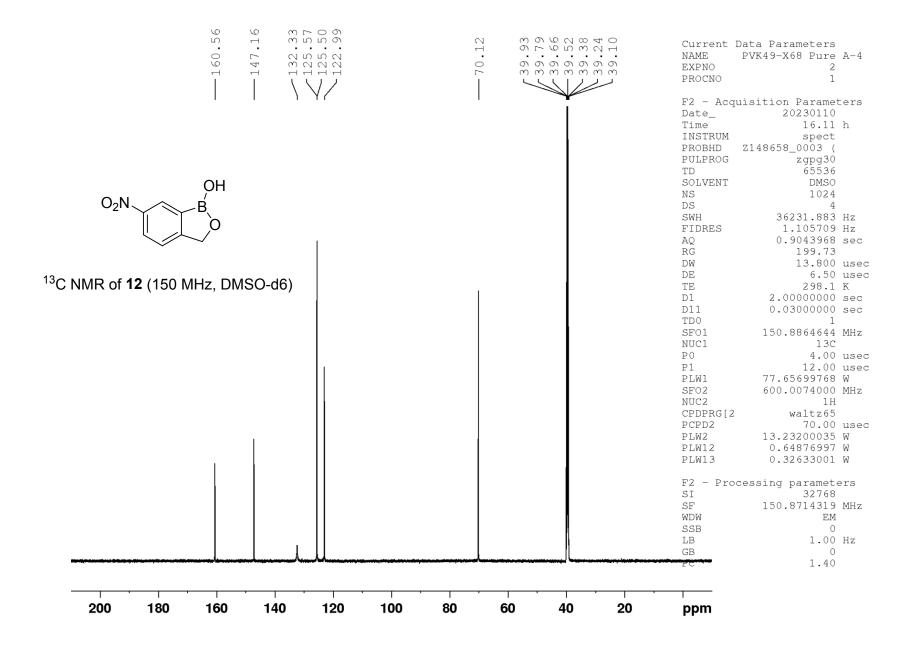
$ \begin{array}{c} & & & & & & & & & & & & & & & & & & &$	2.624	1.360	Current I NAME EXPNO PROCNO	Data Parameters PVK49-X42 A-3' Pure 1 1
$\begin{array}{c} \downarrow \\ O_2 N \\ \downarrow \\ \downarrow \\ CH_3 \end{array}$ ¹ H NMR of 9a (600 MHz, CDCl ₃)			Date_ Time INSTRUM PROBHD PULPROG TD SOLVENT NS DS SWH FIDRES AQ RG DW DE TE D1 TD0 SF01 NUC1 P0 P1 PLW1	disition Parameters 20230109 15.25 h spect Z148658_0003 (zg30 65536 CDC13 16 2 12019.230 Hz 0.366798 Hz 2.7262976 sec 78.64 41.600 usec 10.33 usec 296.3 K 1.00000000 sec 1 600.0087050 MHz 1H 5.17 usec 15.50 usec 13.23200035 W
	l		SI SF WDW	65536 - 600.0050155 MHz EM
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.8	5 3.0 2.5 2.0	1.5 1.5	<u>958</u> 1.0 ppn GB PC PC	ים 0 ח 0.30 Hz 0 1.00

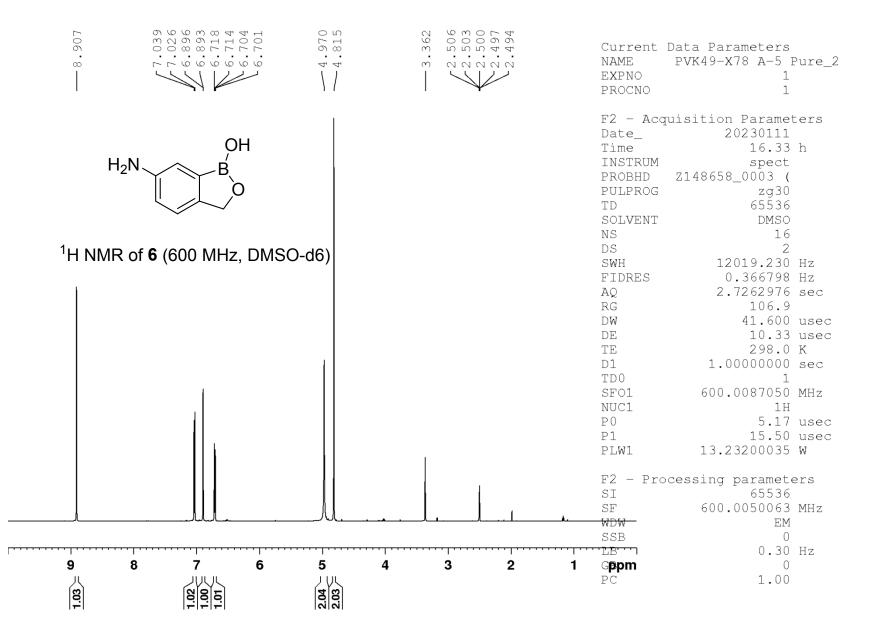
		$\langle \rangle$	PROCNO	2 1
O ₂ N	O B O CH_3 O MHz, CDCl ₃)		Date_ Time INSTRUM	isition Parameters 20230109 16.17 h spect 2148658_0003 (zgpg30 65536 CDC13 1024 4 36231.883 Hz 1.105709 Hz 0.9043968 sec 199.73 13.800 usec 6.50 usec 298.2 K 2.0000000 sec
			D11 TD0 SF01 NUC1 P0 P1 PLW1 SF02 NUC2 CPDPRG[2 PCPD2 PLW2 PLW12 PLW13 F2 - Proce SI SF WDW SSB LB GB PQ	0.03000000 sec 1 150.8864644 MHz 13C 4.00 usec 12.00 usec 77.65699768 W 600.0074000 MHz 1H waltz65 70.00 usec 13.23200035 W 0.64876997 W 0.32633001 W essing parameters 32768 150.8713540 MHz EM 0 1.00 Hz 0 1.40



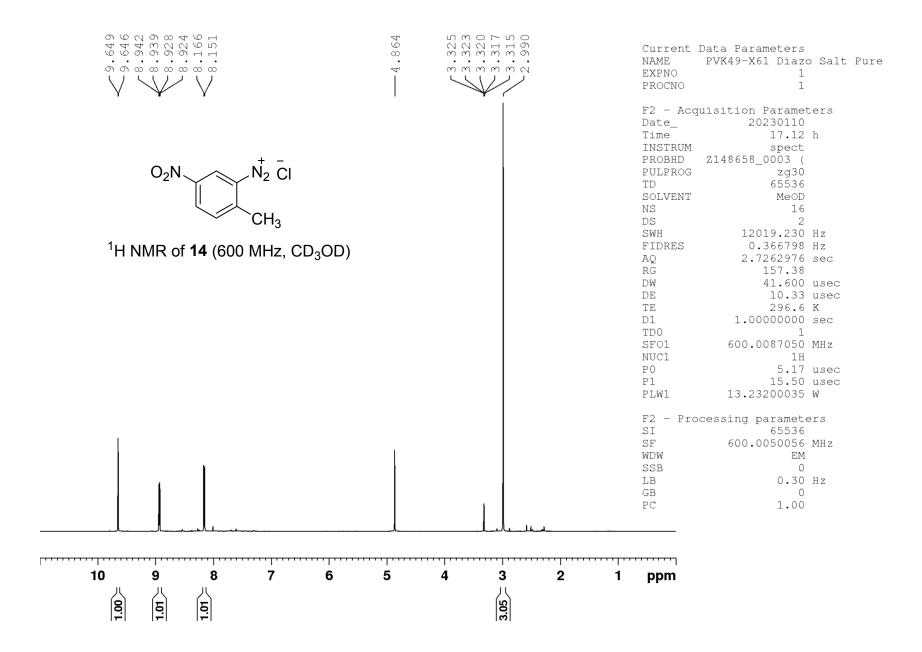








	69.63 39.93 39.56 39.52 39.38 39.10	Current Data Parameters NAME PVK49-X78 A-5 Pure EXPNO 2 PROCNO 1
	i i	F2 - Acquisition Parameters Date_ 20230111
ОН		Time 17.25 h
H_2N		INSTRUM spect
		PROBHD Z148658_0003 (
		PULPROG zgpg30
		TD 65536 SOLVENT DMSO
		NS 1024
		DS 4
¹³ C NMR of 6 (150 MHz, DMSO-d6)		SWH 36231.883 Hz
		FIDRES 1.105709 Hz
		AQ 0.9043968 sec
		RG 199.73 DW 13.800 usec
		DE 15.000 disec
		TE 298.7 K
		D1 2.00000000 sec
		D11 0.03000000 sec
		TDO 1
		SFO1 150.8864644 MHz
		NUC1 13C P0 4.00 usec
		P1 12.00 usec
		PLW1 77.65699768 W
		SF02 600.0074000 MHz
		NUC2 1H
		CPDPRG[2 waltz65
		PCPD2 70.00 usec
		PLW2 13.23200035 W PLW12 0.64876997 W
		PLW12 0.32633001 W
		F2 - Processing parameters
		SI 32768
		SF 150.8714473 MHz
		WDW EM SSB 0
		LB 1.00 Hz
		GB 0
wenter the processive building and the processive strengtheners the complexity of the processive the processive constitution and	unany terminany and any first and an experimentary and any second by an and the second and a second to re-	1.40
		· · · · · · · · · · · · · · · · · · ·
200 180 160 140 120 100	80 60 40 20	ppm
		FF····



-141.95 -138.13 -125.07 -119.37 -109.29	Current Data Parameters Current Data Parameters Current Data Parameters Current Data Parameters NAME PVK49-X61 Diazo Salt Pure Current Data Parameters NAME PVK49-X61 Diazo Salt Pure Current Data Parameters Current Data Parameters Current Data Parameters NAME PVK49-X61 Diazo Salt Pure PROCNO 1
	F2 - Acquisition Parameters Date_ 20230110 Time 18.04 h INSTRUM spect PROBHD Z148658_0003 (PULPROG zgpg30
	TD 65536 SOLVENT MeOD NS 1024 DS 4
[∼] CH ₃	SWH 36231.883 Hz FIDRES 1.105709 Hz
	AQ 0.9043968 sec
¹³ C NMR of 14 (150 MHz, CD ₃ OD)	RG 199.73
	DW 13.800 usec
	DE 6.50 usec TE 298.0 K
	D1 2.00000000 sec
	D11 0.03000000 sec
	TDO 1
	SFO1 150.8864644 MHz
	NUC1 13C P0 4.00 usec
	P0 4.00 usec P1 12.00 usec
	PLW1 77.65699768 W
	SF02 600.0074000 MHz
	NUC2 1H
	CPDPRG[2 waltz65
	PCPD2 70.00 usec
	PLW2 13.23200035 W
	PLW12 0.64876997 W PLW13 0.32633001 W
	F2 - Processing parameters
	SI 32768 SF 150.8726596 MHz
	WDW EM
	SSB 0
	LB 1.00 Hz
	GB 0
une and a species and and a second and a second and a second second second and a second second second second s	an and the part of the second se
00 180 160 140 120 100 80	60 40 20 ppm

$\bigwedge_{7.260}^{8.088}$	2.445	Current Data Parameters NAME PVK49-X8 S.M Pure EXPNO 1 PROCNO 1
O ₂ N CH ₃ ¹ H NMR of SP1 (600 MHz, CDCl ₃)		F2 - Acquisition Parameters Date_ 20230106 Time 15.42 h INSTRUM spect PROBHD Z148658_0003 (PULPROG zg30 TD 65536 SOLVENT CDC13 NS 16
		DS 2 SWH 12019.230 Hz FIDRES 0.366798 Hz AQ 2.7262976 sec RG 55.05 DW 41.600 usec DE 10.33 usec TE 296.3 K D1 1.0000000 sec TD0 1 SF01 600.0087050 MHz NUC1 1H P0 5.17 usec P1 15.50 usec
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	P1 15.50 usec PLW1 13.23200035 W F2 - Processing parameters 65536 SF 600.0050153 MHz WDW EM SSB 0 TLB 0.30 Hz PC 1.00

			77.41 77.19 77.19	•	21.67	NAME EXPNO PROCNO	Data Parameters PVK49-X8 S.M Pure 2 1 disition Parameters 20230106 16.34 h spect Z148658_0003 (zgpg30
			O ₂ N	CH3		TD SOLVENT NS DS SWH FIDRES AQ RG	65536 CDC13 1024 4 36231.883 Hz 1.105709 Hz 0.9043968 sec 199.73
			¹³ C NMR of SP1 (150 MHz, CDCl ₃)		DW DE TE D1 D11 TD0 SF01 NUC1 P0 P1 PLW1 SF02 NUC2 CPDPRG[2 PLW2 PLW2 PLW2 PLW12 PLW13 F2 - Proc SI SF WDW SSB LB GB PC	13.800 usec 6.50 usec 298.3 K 2.0000000 sec 0.03000000 sec 1 150.8864644 MHz 13C 4.00 usec 12.00 usec 77.65699768 W 600.0074000 MHz 1H waltz65 70.00 usec 13.23200035 W 0.64876997 W 0.32633001 W sessing parameters 32768 150.8713629 MHz EM 0 1.00 Hz 0 1.40
180 1	60 14	40 120	100 80	60 40	20	ppm	