

Nickel-Catalyzed Suzuki–Miyaura Couplings in Green Solvents

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Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen using anhydrous solvents (either freshly distilled, purchased from a commercial source, or passed through activated alumina columns). DriSolv[®] anhydrous solvents were purchased from Acros Organics and used without further purification. Non-commercially available substrates were synthesized following protocols specified in Section A of the supporting information. Unless otherwise stated, commercially obtained reagents were used as received. Halogenated substrates were obtained from Combi-Blocks, Sigma-Aldrich, and Oakwood Products, Inc. NiCl₂(PCy₃)₂ was obtained from Strem Chemicals and Sigma-Aldrich. Finely powdered anhydrous K₃PO₄ was obtained from Acros Organics. Boronic acids were obtained from Oakwood Products, Inc., Frontier Scientific, Inc. and Combi-Blocks. 2-Methyltetrahydrofuran (2-Me-THF), anhydrous, was obtained from Acros Organics (the water content was measured to be 0.035–0.13% using the Karl Fischer titration; water content of unopened bottle is reported to be 0.005%). *Tert*-amyl alcohol (*t*-amyl alcohol) was obtained from Sigma-Aldrich and used as received (the water content was measured to be 0.0053–0.25% using the Karl Fischer titration). Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (rt, approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm) and visualized using a combination of UV, anisaldehyde, ceric ammonium molybdate, iodine, vanillin, and potassium permanganate staining techniques. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ¹H NMR spectra were recorded on Bruker spectrometers (at 300, 400, 500, 600 MHz) and are reported relative to deuterated solvent signals. Data for ¹H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ¹³C NMR spectra were recorded on Bruker Spectrometers (at 125 MHz). Data for ¹³C NMR spectra are reported in terms of chemical shift. IR spectra were recorded on a Perkin-Elmer 100 spectrometer and are reported in terms of frequency of absorption (cm⁻¹). High resolution mass spectra were obtained from the UC Irvine Mass Spectrometry Facility.

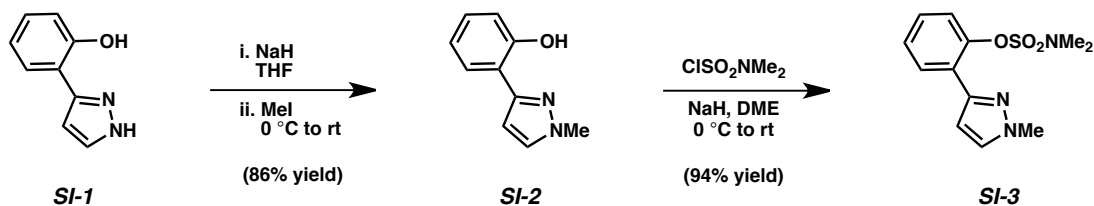
For more information about the ACS Green Chemistry Institute Pharmaceutical Roundtable Solvent Selection Guide, see:

<http://portal.acs.org/portal/PublicWebSite/greenchemistry/industriainnovation/roundtable/index.htm>

Experimental Procedures

A. Synthesis of Aryl Carbamate, Sulfamate, and Mesylate Substrates

Note: Supporting information for the synthesis of the aryl sulfamates, carbamates, and mesylates shown in Tables 1–2 and Figures 2–5 have previously been reported,¹ with the exception of pyrazole **SI-3**.

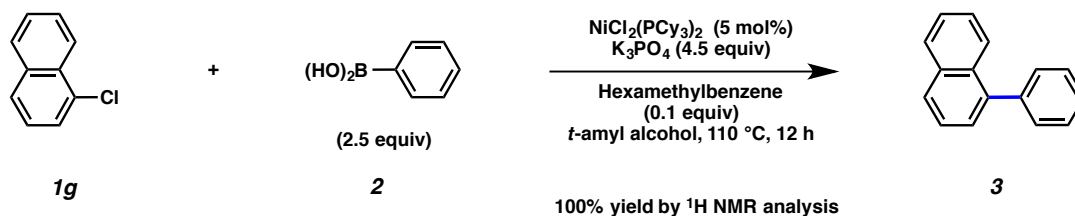


Pyrazole SI-2. To a round bottom flask charged with NaH (0.15 g, 3.74 mmol, 2.00 equiv, 60% dispersion in oil) was added a solution of pyrazole **SI-1** (0.30 g, 1.88 mmol, 1.00 equiv) in THF (11.1 mL) dropwise via cannula over several minutes at 0 °C. After stirring for 2 h, methyl iodide (0.12 mL, 1.88 mmol, 1.00 equiv) in THF (7.2 mL) was added dropwise over 1 min and the resulting mixture was stirred for 12 h while warming to rt. The volatiles were removed under reduced pressure and then, H₂O (10 mL) and Et₂O (10 mL) were added. The layers were separated, and the aqueous layer was extracted with Et₂O (10 mL). The combined organic layers were washed successively with 1 M aqueous NaHCO₃ (10 mL) and brine (10 mL), dried over MgSO₄, and concentrated under reduced pressure. The crude residue was purified by flash chromatography (3:1 Hexanes:EtOAc) to give pyrazole **SI-2** (0.22 g, 86% yield) as a yellow solid. *R*_f 0.36 (3:1 Hexanes:EtOAc). Spectral data match those previously reported.²

Sulfamate SI-3. To a round bottom flask charged with NaH (0.78 g, 1.94 mmol, 1.20 equiv, 60% dispersion in oil) was added a solution of pyrazole **SI-2** (0.28 g, 1.62 mmol, 1.00 equiv) in DME (6.5 mL) dropwise via cannula over several minutes at 0 °C. Dimethylsulfamoyl chloride (0.17 mL, 1.56 mmol, 0.95 equiv) was added dropwise to the reaction vessel. The reaction was allowed to stir for 12 h while warming to rt. The reaction was quenched with H₂O (0.3 mL) and the volatiles were removed under reduced pressure. Et₂O (45 mL) and H₂O (15 mL) were added

and the layers were separated. The organic layer was washed with 1 M aqueous KOH (2 x 15 mL). The combined aqueous layers were extracted with Et₂O (3 x 30 mL). The combined organic layers were then washed with brine (30 mL), dried over MgSO₄, and concentrated under reduced pressure. The crude residue was purified by flash chromatography (3:1 Hexanes:Acetone) to yield sulfamate **SI-3** as a yellow oil (0.42 g, 96% yield). *R_f* 0.12 (1:1 Hexanes: EtOAc); ¹H NMR (600 MHz, CDCl₃): δ 7.85 (dd, *J* = 7.2, 2.0, 1H), 7.54 (dd, *J* = 8.1, 1.5, 1H), 7.35 (d, *J* = 2.1, 1H), 7.32-7.27 (m, 2H), 6.69 (d, *J* = 2.2, 1H), 3.93 (s, 3H), 2.68 (s, 6H); ¹³C NMR (150 MHz, CDCl₃): δ 147.6, 147.0, 130.9, 129.7, 128.6, 127.1, 126.7, 122.5, 107.2, 39.1, 38.4; IR (film): 2941, 1460, 1366, 1191, 1156, 973, 740 cm⁻¹; HRMS-ESI (*m/z*) [M+H]⁺ calcd for C₁₂H₁₅N₃O₃S, 282.0912; found 282.0912.

B. Screening of Green Solvents



Representative Procedure (coupling of 1g is used as an example). 3 (Table 2). A 1-dram vial was charged with anhydrous powdered K₃PO₄ (419.3 mg, 1.98 mmol, 4.50 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N₂. Hexamethylbenzene (7.1 mg, 0.044 mmol, 0.10 equiv), boronic acid **2** (134.1 mg, 1.1 mmol, 2.50 equiv), NiCl₂(PCy₃)₂ (15.2 mg, 0.022 mmol, 5 mol%), and substrate **1g** (71.3 mg, 0.44 mmol, 1.00 equiv) were added. The vial was then evacuated and backfilled with N₂. To the vial, *t*-amyl alcohol (1.5 mL) was added and then the vial was sealed with a Teflon-lined screw cap. The mixture was allowed to stir rapidly at 23 °C for 1 h, and then heated to 110 °C for 12 h. After cooling the reaction vessel to 23 °C, the reaction was transferred to a test tube containing 1 M aqueous HCl (2 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were then washed with brine (2 mL), dried over MgSO₄, and passed through a SiO₂ plug. The pad was washed with EtOAc (10 mL)

and the organic layers were concentrated under reduced pressure. The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.^{1c}

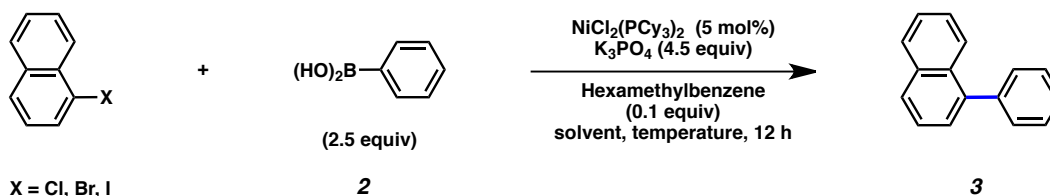
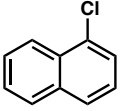
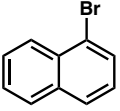
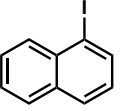
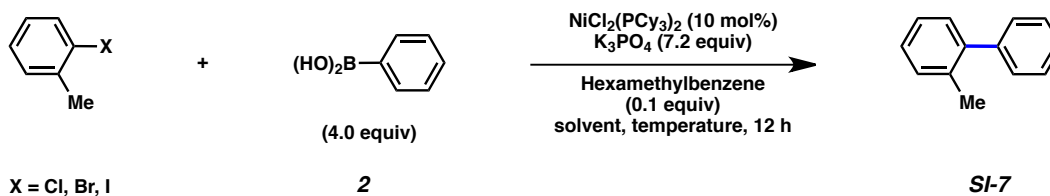


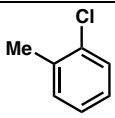
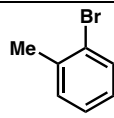
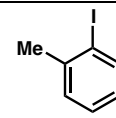
Table 1. Cross-coupling reactions of naphthyl halides in various solvents^a

Solvent Name (temperature (°C))	 1g	 1h	 1i
	^1H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard		
<i>t</i> -butanol (83)	88 ^b	100 ^b , 87 ^c	90 ^b , 82 ^c
<i>t</i> -amyl alcohol (110)	100 ^b	97 ^b	100 ^b
2-Me-THF (66)	94 ^d	92 ^d	97 ^d
CPME (106)	100 ^d	90 ^d	100 ^d
dimethyl isosorbide (110)	40 ^c	42 ^c	44 ^c
<i>i</i> -PrOAc (89)	90 ^c	90 ^c	95 ^c
DMSO (110)	100 ^b	91 ^b	86 ^b
DMF (106)	100 ^b	90 ^b	97 ^b
DMA (110)	0 ^b	0 ^b	0 ^b
NMP (110)	100 ^b	69 ^b	54 ^b
sulfolane (110)	100 ^b	98 ^b	96 ^b

^a Reaction conditions, unless otherwise noted, the reaction was carried out with $\text{NiCl}_2(\text{PCy}_3)_2$ complex (5 mol %), substrate **1g–i** (1.00 equiv), **2** (2.50 equiv), K_3PO_4 (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b Distilled from desiccant. ^c Used without purification.

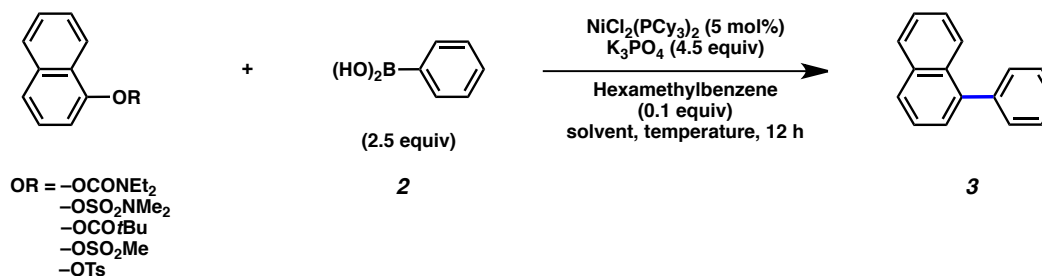
^d Drysol[®].


Table 2. Cross-coupling reactions of tolyl halides in selected solvents^a

Solvent Name (temperature (°C))	 SI-4	 SI-5	 SI-6
	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard		
<i>t</i> -butanol (83)	98 ^b , 92 ^c	99 ^b , 100 ^c	97 ^b , 95 ^c
2-Me-THF (80)	93 ^d	99 ^d	99 ^d
CPME (106)	96 ^d	96 ^d	100 ^d
<i>i</i> -PrOAc (89)	90 ^c	91 ^c	85 ^c

^a Reaction conditions, unless otherwise noted, the reaction was carried out with $\text{NiCl}_2(\text{PCy}_3)_2$ complex (10 mol %), substrate **SI-4–6** (1.00 equiv), **2** (4.00 equiv), K_3PO_4 (7.20 equiv), hexamethylbenzene (0.1 equiv), 12 h. ^b Distilled from desiccant. ^c Used without purification.

^d Drysol[®].


Table 3. Cross-coupling reactions of naphthol derivatives in various solvents^a

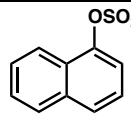
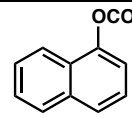
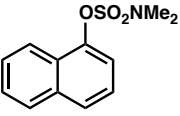
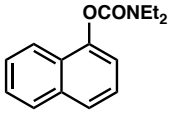
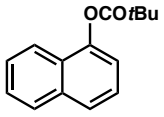
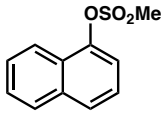
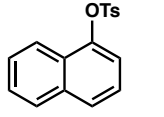
Solvent Name (temperature (°C))	 1a	 1b
	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
<i>t</i> -butanol (100)	100 ^{c,d}	100 ^{b,c,d}
<i>n</i> -butanol (110)	0 ^{c,d}	0 ^{b,c,d}

Table 3. Cross-coupling reactions of naphthol derivatives in various solvents (continued)^a

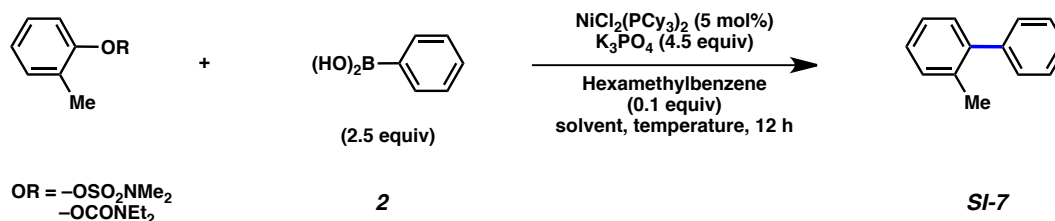
Solvent Name (temperature (°C))	 1a	 1b
	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
2-butanol (110)	0 ^{c,d}	0 ^{b,c,d,f}
<i>i</i> -butanol (110)	3 ^c , 2 ^d	0 ^{b,c,d,g}
<i>t</i> -amyl alcohol (100)	100 ^c	88 ^{b,c}
toluene (110)	100 ^b	100 ^b
xylenes (110)	100 ^{c,d}	100 ^{b,c,d,g}
triethylamine (110)	99 ^c , 100 ^d	91 ^{b,c} , 76 ^{b,d}
pyridine (110)	70 ^c , 67 ^d	0 ^{b,c,g} , 0 ^{b,d,g}
2-Me-THF (100)	100 ^e	97 ^{b,e}
CPME (110)	100 ^e	100 ^{b,e,g}
MTBE (80)	100 ^e	100 ^{b,e}
1,2-dimethoxyethane (110)	100 ^b	92 ^b
1,4-dioxane (110)	100 ^{c,d}	58 ^{b,c} , 75 ^{b,d}
diglyme	100 ^c , 54 ^d	93 ^{b,c} , 16 ^{b,d}
EtOAc (100)	100 ^{c,d}	100 ^{b,c,d}
<i>n</i> -butyl acetate (110)	100 ^d	97 ^{b,d}
<i>i</i> -PrOAc (110)	100 ^d	99 ^{b,d}
<i>n</i> -amyl acetate (110)	0 ^d	0 ^{b,d}
acetone (75)	96 ^c , 99 ^d	68 ^{b,c} , 54 ^{b,d}
2-butanone (79)	99 ^c	69 ^{b,c}
methyl <i>i</i> -butyl ketone (110)	97 ^{c,d}	97 ^{b,c} , 71 ^{b,d}
cyclohexanone (110)	100 ^c	100 ^{b,c}
acetonitrile (100)	99 ^b	0 ^b
DMSO (110)	0 ^c	0 ^{b,c}
DMA (110)	0 ^c	0 ^{b,c}
DMF (106)	97 ^b	86 ^b
NMP (110)	100 ^c	43 ^{b,c}
sulfolane (110)	100 ^c	64 ^{b,c}
<i>i</i> -octane (95)	86 ^c , 95 ^d	69 ^{b,c} , 73 ^{b,d}
<i>n</i> -heptane (98)	100 ^{c,d}	76 ^{b,c} , 77 ^{b,d}
cyclohexane (80)	96 ^c , 90 ^d	78 ^{b,c} , 50 ^{b,d}
1,2-dichloroethane (100)	0 ^c	0 ^{b,c}

^a Reaction conditions, unless otherwise noted, the reaction was carried out with NiCl₂(PCy₃)₂ complex (5 mol %), substrate **1a–b** (1.00 equiv), **2** (2.50 equiv), K₃PO₄ (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b The reaction was carried out with NiCl₂(PCy₃)₂ complex (10 mol %), substrate **1a–b** (1.00 equiv), **2** (4.00 equiv), K₃PO₄ (7.20 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^c Distilled from desiccant. ^d Used without purification. ^e Drysol[®]. ^f The reaction was performed at 120 °C. ^g The reaction was performed at 130 °C.

Table 4. Cross-coupling reactions of naphthyl mesylate, tosylate, and pivalates in selected green solvents^a

Solvent Name (temperature (°C))	 1c	 1d	 1e
	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard		
<i>t</i> -butanol (82)	100 ^{b,c} , 53 ^{b,d}	90 ^c , 90 ^d	100 ^c , 95 ^d
2-Me-THF (66)	99 ^{b,e}	95 ^e	96 ^e
CPME (106)	100 ^{b,e}	100 ^e	84 ^e
<i>i</i> -PrOAc (89)	92 ^{b,d}	95 ^d	100 ^d

^a Reaction conditions, unless otherwise noted, the reaction was carried out with NiCl₂(PCy₃)₂ complex (5 mol %), substrate **1c–e** (1.00 equiv), **2** (2.50 equiv), K₃PO₄ (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b The reaction was carried out with NiCl₂(PCy₃)₂ complex (10 mol %), substrate **1c–e** (1.00 equiv), **2** (4.00 equiv), K₃PO₄ (7.20 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^c Distilled from desiccant. ^d Used without purification. ^e Drysol[®].

**Table 5.** Cross-coupling reactions of *o*-methylphenyl sulfamate and carbamate in selected solvents^a

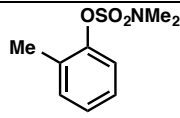
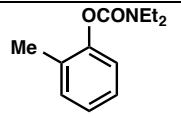
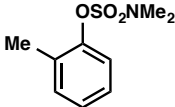
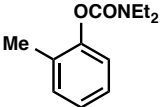
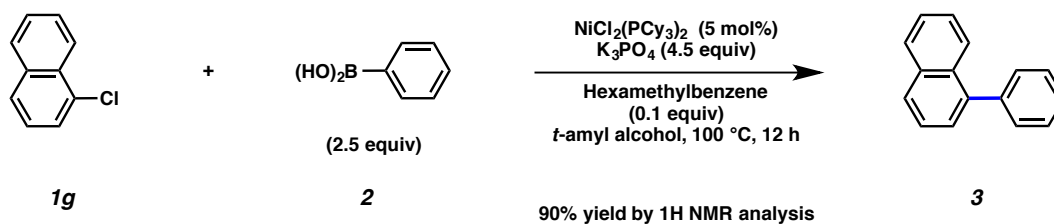
Solvent Name (temperature (°C))	 SI-8	 SI-9
	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
<i>t</i> -butanol (100)	78 ^c , 68 ^d	45 ^{b,c} , 50 ^{b,d}
<i>t</i> -amyl alcohol (120)	100 ^d	45 ^{b,d}
toluene (110)	92 ^b	61 ^b
xylenes (110)	100 ^c	69 ^{b,c,f}

Table 5. Cross-coupling reactions of *o*-methylphenyl sulfamate and carbamate in selected solvents (continued)^a

Solvent Name (temperature (°C))	 SI-8	 SI-9
	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
triethylamine (110)	52 ^c	29 ^{b,c}
2-Me-THF (100)	97 ^e	88 ^{b,e}
CPME (110)	96 ^e	58 ^{b,e,f}
MTBE (80)	99 ^e	60 ^{b,e}
1,2-dimethoxyethane (110)	99 ^b	65 ^b
1,4-dioxane (110)	99 ^c , 90 ^d	57 ^{b,c} , 34 ^{b,d}
EtOAc (100)	33 ^c , 26 ^d	17 ^{b,c} , 16 ^{b,d}
<i>n</i> -butyl acetate (110)	20 ^d	15 ^{b,d,f}
<i>i</i> -PrOAc (110)	78 ^d	70 ^{b,d}
acetone (75)	98 ^c	5 ^{b,c}

^a Reaction conditions, unless otherwise noted, the reaction was carried out with NiCl₂(PCy₃)₂ complex (5 mol %), substrate **SI-8-9** (1.00 equiv), **2** (2.50 equiv), K₃PO₄ (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b The reaction was carried out with NiCl₂(PCy₃)₂ complex (10 mol %), substrate **SI-8-9** (1 equiv), **2** (4.00 equiv), K₃PO₄ (7.20 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^c Distilled from desiccant. ^d Used without purification. ^e Drysol[®]. ^f The reaction was performed at 130 °C.

C. Screening of Arylboron-Based Nucleophiles



Representative Procedure (coupling of **1g is used as an example). **3** (Table 2).** A 1-dram vial was charged with anhydrous powdered K₃PO₄ (419.3 mg, 1.98 mmol, 4.50 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N₂. Hexamethylbenzene (7.1 mg, 0.044 mmol, 0.10 equiv), boronic acid **2** (134.1 mg, 1.1 mmol, 2.50 equiv), NiCl₂(PCy₃)₂ (15.2 mg, 0.022 mmol, 5 mol%), and substrate **1g** (71.3

mg, 0.44 mmol, 1.00 equiv) were added. The vial was then evacuated and backfilled with N₂. To the vial, *t*-amyl alcohol (1.5 mL) was added and then the vial was sealed with a Teflon-lined screw cap. The mixture was allowed to stir rapidly at 23 °C for 1 h, and then heated to 100 °C for 12 h. The reaction vessel was cooled to 23 °C, then transferred to a test tube containing 1 M aqueous HCl (2 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were then washed with brine (2 mL), dried over MgSO₄, and passed through a SiO₂ plug. The pad was washed with EtOAc (10 mL) and the organic layers were concentrated under reduced pressure. The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.^{1e}

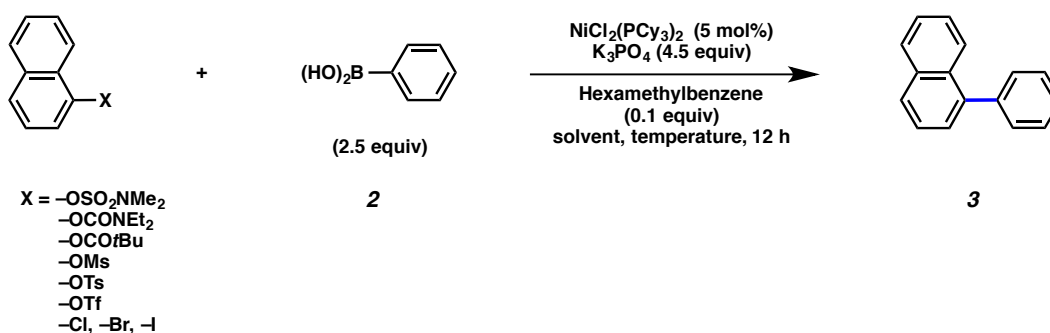
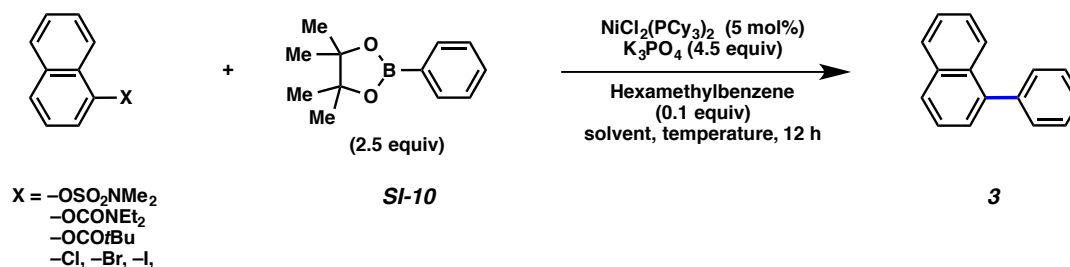


Table 6. Arylboron-Based Nucleophile Phenylboronic Acid^a

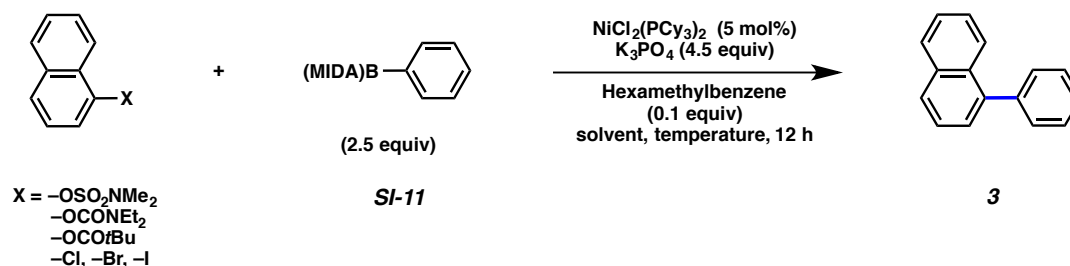
X	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
	<i>t</i> -amyl alcohol ^b (100 °C)	2-Me-THF ^c (66 °C)
OSO ₂ NMe ₂ (1a)	100	100
OCONEt ₂ (1b)	57	50
OCOtBu (1c)	94	100
OMs (1d)	97	95
OTs (1e)	100	98
OTf (1f)	100	100
Cl (1g)	100	94
Br (1h)	97	92
I (1i)	100	97

^a Reaction conditions, unless otherwise noted, the reaction was carried out with NiCl₂(PCy₃)₂ complex (5 mol %), substrate **1a–i** (1.00 equiv), **2** (2.50 equiv), K₃PO₄ (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b Used without purification. ^c Drysolv[®].


Table 8. Arylboron-Based Nucleophile Pinacol Ester^a

X	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
	<i>t</i> -amyl alcohol ^b (120 °C)	2-Me-THF ^c (100 °C)
OSO ₂ NMe ₂ (1a)	100	0
OCONEt ₂ (1b)	97	0
OCOtBu (1c)	99	75
Cl (1g)	96	0
Br (1h)	98	0
I (1i)	93	0

^a Reaction conditions, unless otherwise noted, the reaction was carried out with NiCl₂(PCy₃)₂ complex (5 mol %), substrate **1a–c**, **1g–i** (1.00 equiv), **2** (2.50 equiv), K₃PO₄ (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b Used without purification. ^c Drysol[®].


Table 9. Arylboron-Based Nucleophile MIDA (N-Methyliminodiacetic Acid)^a

X	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
	<i>t</i> -amyl alcohol ^b (100 °C)	2-Me-THF ^c (66 °C)
OSO ₂ NMe ₂ (1a)	0	0
OCONEt ₂ (1b)	0	0
OCOtBu (1c)	0	0
Cl (1g)	0	0
Br (1h)	0	0
I (1i)	0	0

^a Reaction conditions, unless otherwise noted, the reaction was carried out with NiCl₂(PCy₃)₂ complex (5 mol %), substrate **1a–c**, **1g–i** (1.00 equiv), **2** (2.50 equiv), K₃PO₄ (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b Used without purification. ^c Drysolv[®].

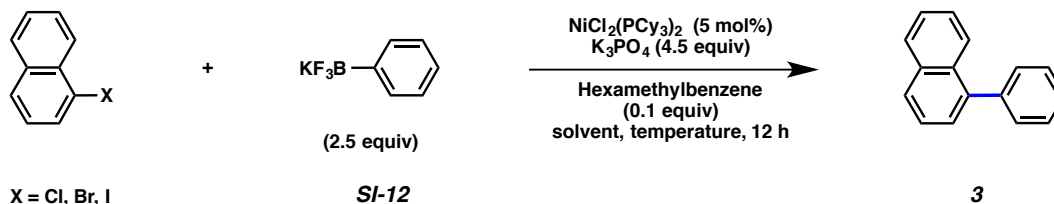
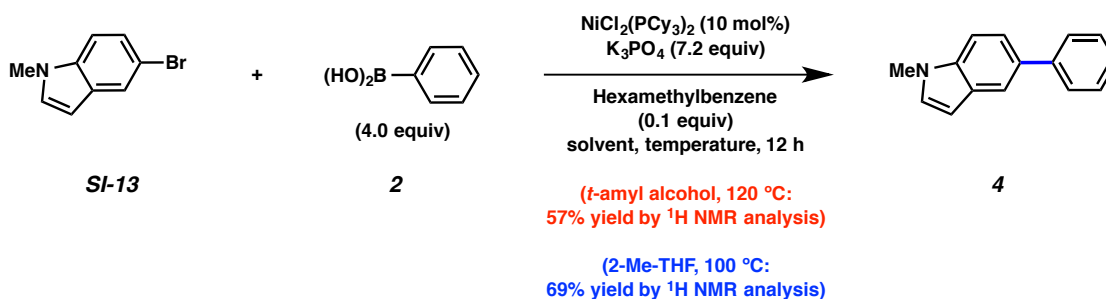


Table 10. Arylboron-Based Nucleophile Potassium Aryl Trifluoroborate^a

X	¹ H NMR yield of desired product (%) determined by hexamethylbenzene as an internal standard	
	<i>t</i> -amyl alcohol ^b (100 °C)	2-Me-THF ^c (66 °C)
Cl (1g)	0	0
Br (1h)	0	0
I (1i)	0	0

^a Reaction conditions, unless otherwise noted, the reaction was carried out with NiCl₂(PCy₃)₂ complex (5 mol %), substrate **1g–i** (1.00 equiv), **2** (2.50 equiv), K₃PO₄ (4.50 equiv), hexamethylbenzene (0.10 equiv), 12 h. ^b Used without purification. ^c Drysolv[®].

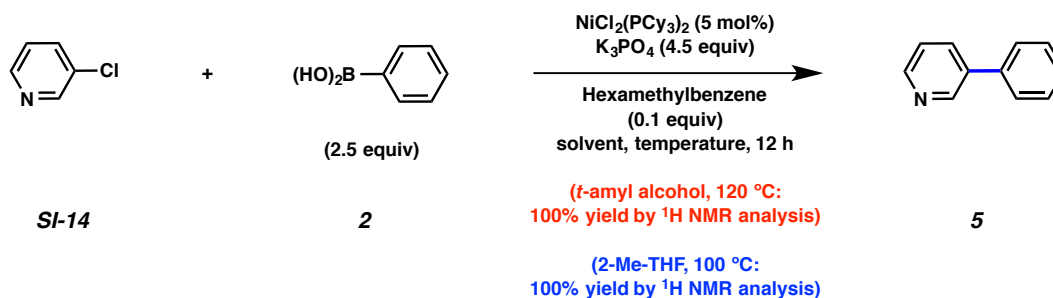
D. Cross-coupling Reactions of Aryl Halides



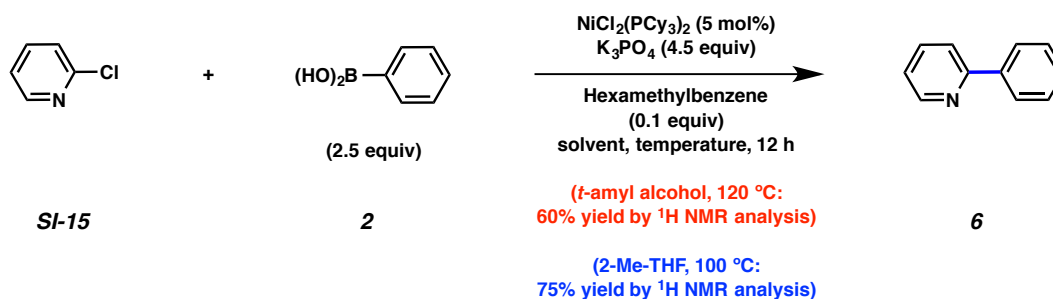
Representative Procedure (coupling of SI-13 is used as an example). 4 (Figure 2). A 1-dram vial was charged with anhydrous powdered K₃PO₄ (831.4 mg, 7.20 mmol, 7.20 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N₂. Hexamethylbenzene (8.8 mg, 0.054 mmol, 0.10 equiv), boronic acid **2** (265.3 mg, 2.20 mmol, 4.0 equiv), NiCl₂(PCy₃)₂ (37.5 mg, 0.054 mmol, 10 mol%), and indole **SI-13** (112.8 mg, 0.54 mmol, 1.00 equiv) were added. The vial was then evacuated and backfilled with

N₂. To the vial, solvent (1.5 mL) was added and then the vial was sealed with a Teflon-lined screw cap. The mixture was allowed to stir rapidly at 23 °C for 1 h, and then heated to 100 °C (2-Me-THF) or 120 °C (*t*-amyl alcohol) for 12 h. After cooling the reaction to 23 °C, the reaction was transferred to a test tube containing 1 M aqueous HCl (2 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were then washed with brine (2 mL), dried over MgSO₄, passed through a SiO₂ plug. The pad was washed with EtOAc (10 mL) and the organic layers were concentrated under reduced pressure. The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.^{1b}

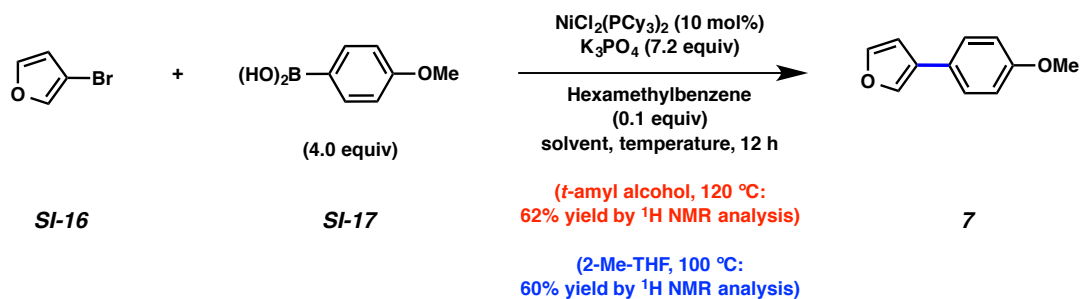
Any modifications of the conditions shown in this representative procedure are specified in the following schemes, which depict all of the results shown in Figure 2.



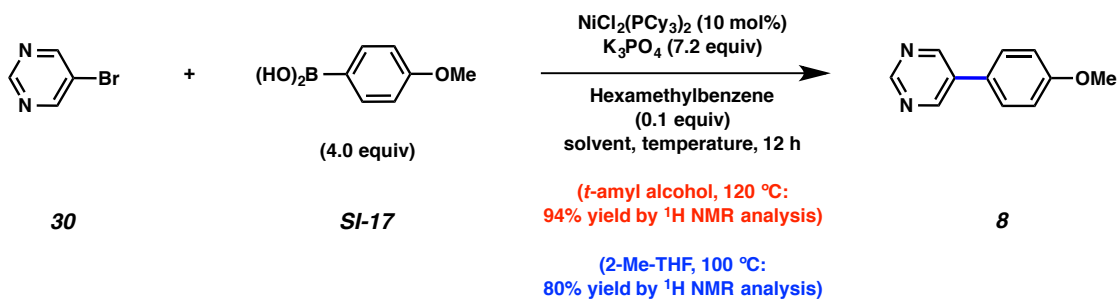
5 (Figure 2). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.³



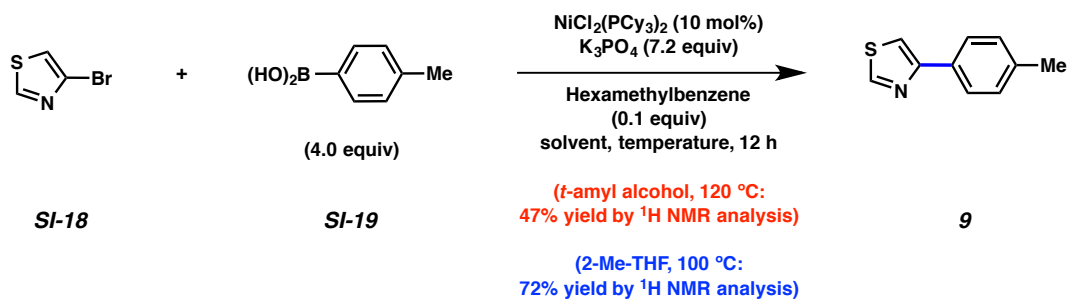
6 (Figure 2). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁴



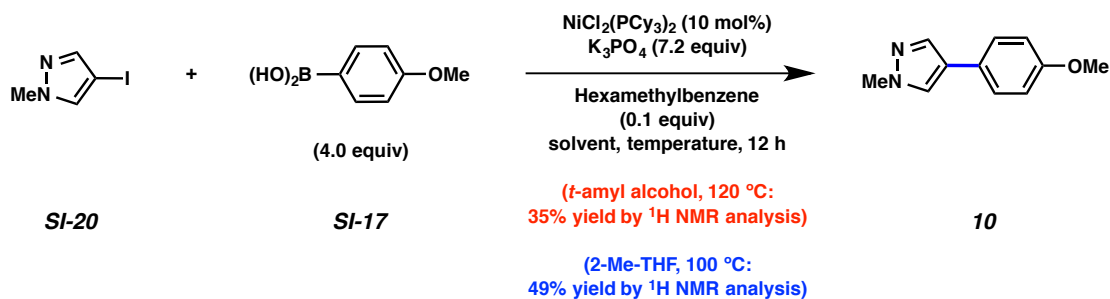
7 (Figure 2). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁵



8 (Figure 2). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁵

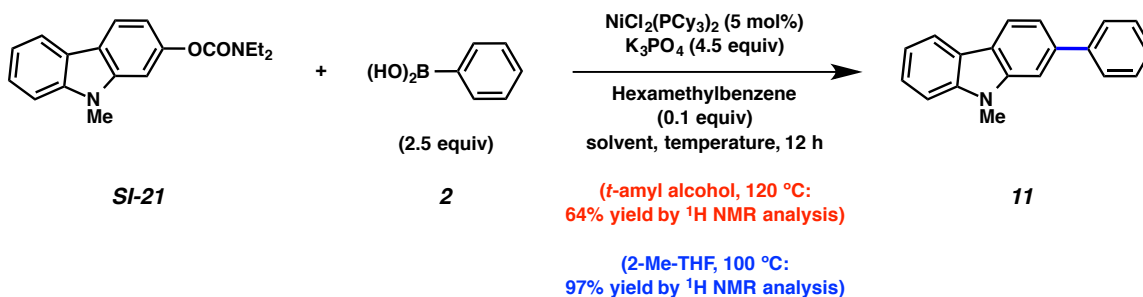


9 (Figure 2). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁶



10 (Figure 2). The yield was determined by $^1\text{H NMR}$ analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁷

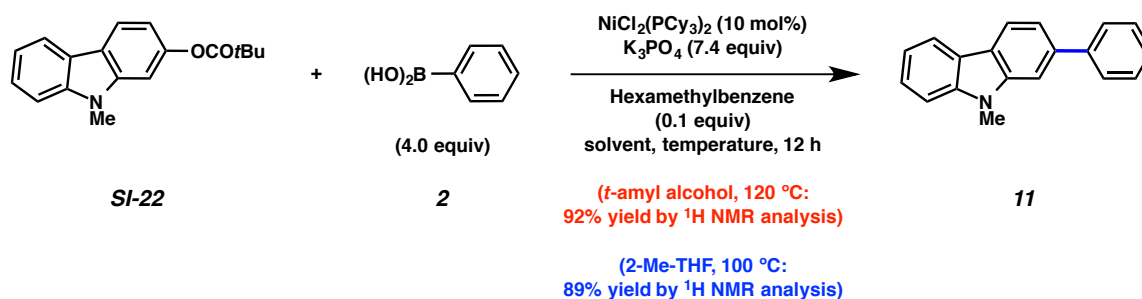
E. Cross-coupling Reactions of Aryl Carbamates, Sulfamates, and Mesylates



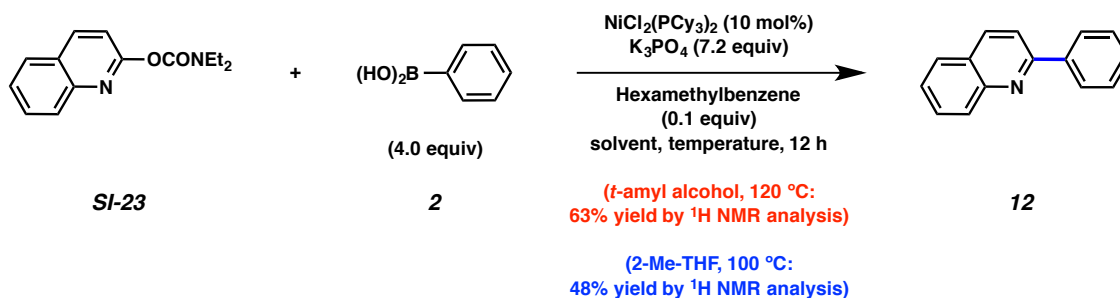
Representative Procedure (coupling of SI-21 is used as an example). 11 (Figure 3). A 1-dram vial was charged with anhydrous powdered K_3PO_4 (429.8 mg, 2.03 mmol, 4.50 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N_2 . Hexamethylbenzene (7.3 mg, 0.045 mmol, 0.10 equiv), boronic acid **2** (171.0 mg, 1.125 mmol, 2.50 equiv), $\text{NiCl}_2(\text{PCy}_3)_2$ (15.5 mg, 0.023 mmol, 5 mol%), and substrate **SI-21** (104.9 mg, 0.45 mmol, 1.00 equiv) were added. The vial was then evacuated and backfilled with N_2 . To the vial, solvent (1.5 mL) was added and then the vial was sealed with a Teflon-lined screw cap. The mixture was allowed to stir rapidly at 23 °C for 1 h, and then heated to 100 °C (2-Me-THF) or 120 °C (*t*-amyl alcohol) for 12 h. After cooling the reaction vessel to 23 °C, the reaction was transferred to a test tube containing 1 M aqueous HCl (4 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were then washed with brine (2 mL), dried over Na_2SO_4 , and passed through a SiO_2 plug. The pad was washed with EtOAc (10 mL) and the organic layers were concentrated under

reduced pressure. The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.^{1a}

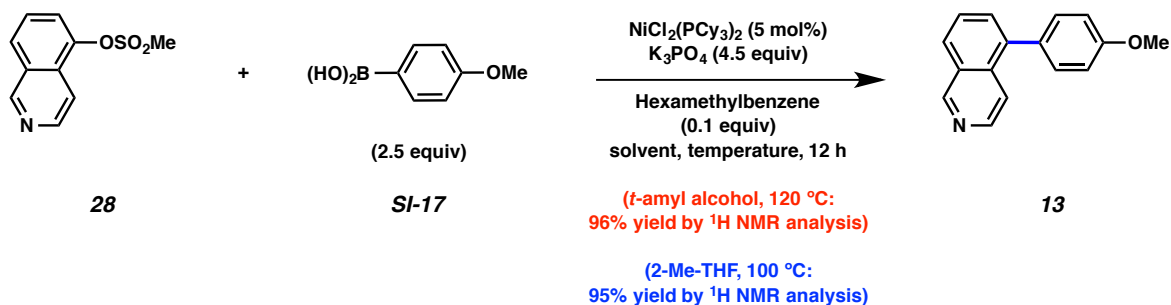
Any modifications of the conditions shown in this representative procedure are specified in the following schemes, which depict all of the results shown in Figure 3.



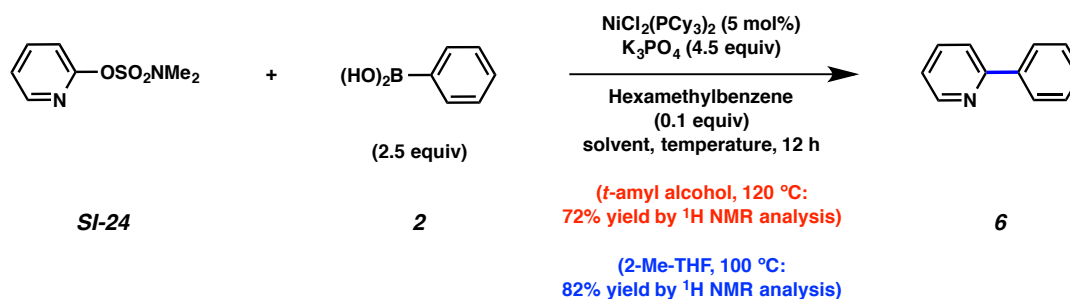
11 (Figure 3). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.^{1a}



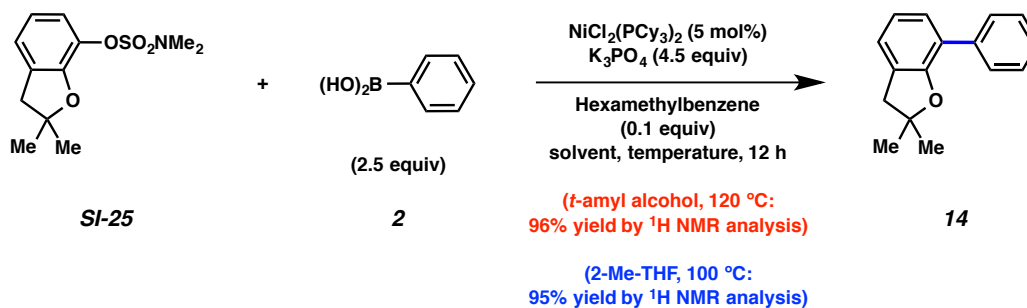
12 (Figure 3). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁸



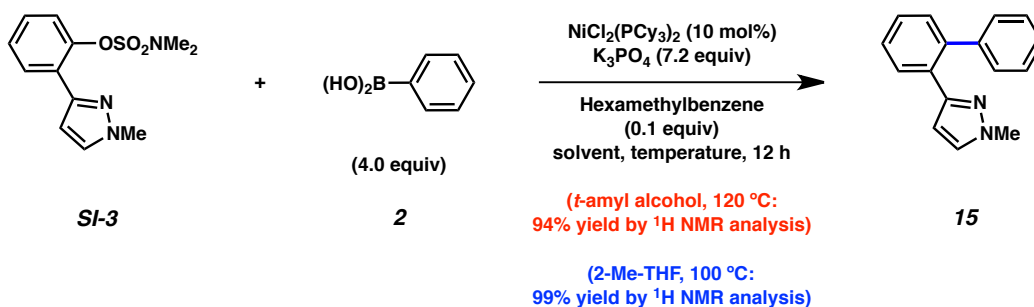
13 (Figure 3). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁸



6 (Figure 3). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁸

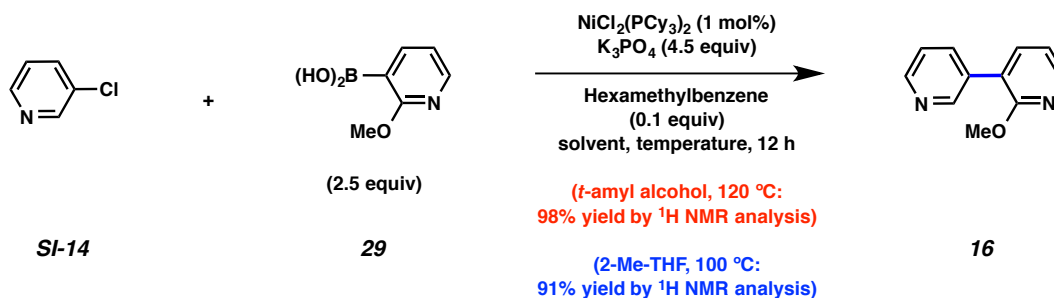


14 (Figure 3). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.^{1e}



15 (Figure 3). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. R_f 0.24 (3:1 Hexanes:EtOAc); ^1H NMR: (600 MHz, CDCl_3): δ 7.82 (dd, $J = 7.6, 1.4, 1\text{H}$), 7.41–7.35 (m, 2H), 7.33–7.26 (m, 6H), 7.08 (d, $J = 2.1, 1\text{H}$), 5.45 (d, $J = 2.1, 1\text{H}$), 3.89 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 151.3, 142.2, 140.7, 132.5, 130.5, 130.1, 129.7, 129.3, 128.0, 127.7, 127.6, 126.8, 106.7, 39.0; IR (film): 3055, 2934, 1597, 1501, 1461, 1392, 1221, 751, 699 cm^{-1} ; HRMS-ESI (m/z) [$\text{M}+\text{H}$] $^+$ calcd for $\text{C}_{16}\text{H}_{14}\text{N}_2$, 235.1235; found 235.1236.

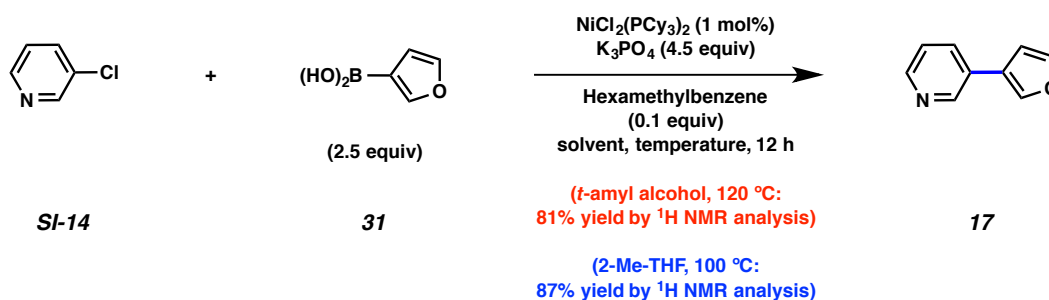
F. Heterocycle-Heterocycle Cross-Couplings of Aryl Halides



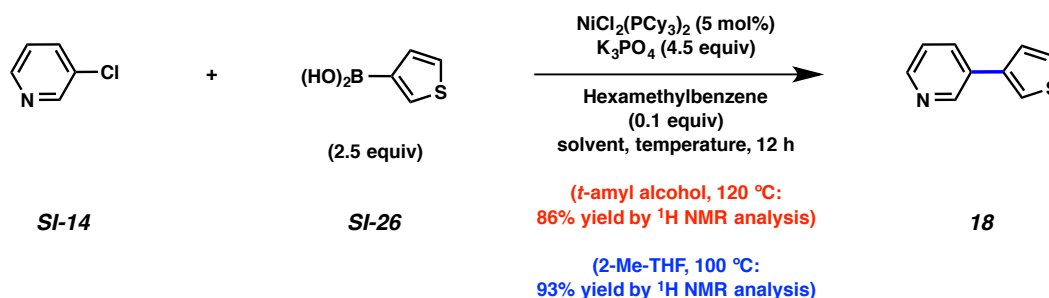
Representative Procedure (coupling of SI-14 is used as an example). 16 (Figure 4). A 1-dram vial was charged with anhydrous powdered K_3PO_4 (859.7 mg, 4.1 mmol, 4.50 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N_2 . Hexamethylbenzene (14.6 mg, 0.09 mmol, 0.10 equiv), boronic acid **29** (344.1 mg, 2.25 mmol, 2.5 equiv), $\text{NiCl}_2(\text{PCy}_3)_2$ (6.2 mg, 0.009 mmol, 1 mol%), and substrate **SI-14** (102.2 mg, 0.9 mmol, 1.00 equiv) were added. The vial was then evacuated and backfilled with N_2 . To the vial, solvent (1.5 mL) was added and then the vial was sealed with a Teflon-lined screw cap. The mixture was allowed to stir rapidly at 23 °C for 1 h, and then heated to 100 °C (2-Me-THF) or 120 °C (*t*-amyl alcohol) for 12 h. The reaction vessel was cooled to 23 °C and then

transferred to a test tube containing 1 M aqueous HCl (2 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were then washed with brine (2 mL), dried over MgSO₄, and passed through a SiO₂ plug. The pad was washed with EtOAc (10 mL) and the organic layers were concentrated under reduced pressure. The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.⁹

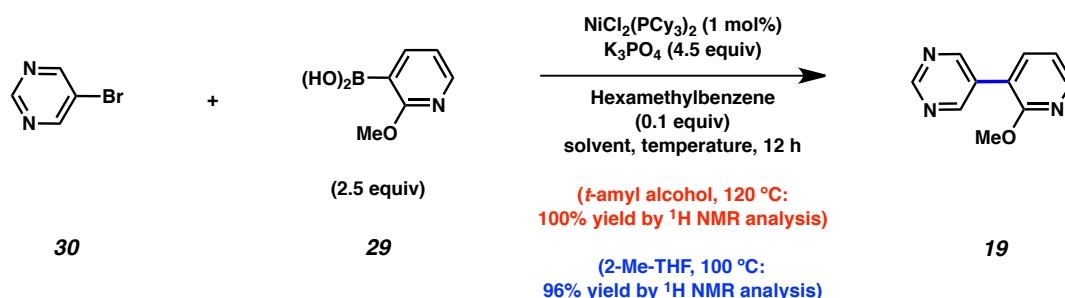
Any modifications of the conditions shown in this representative procedure are specified in the following schemes, which depict all of the results shown in Figure 4.



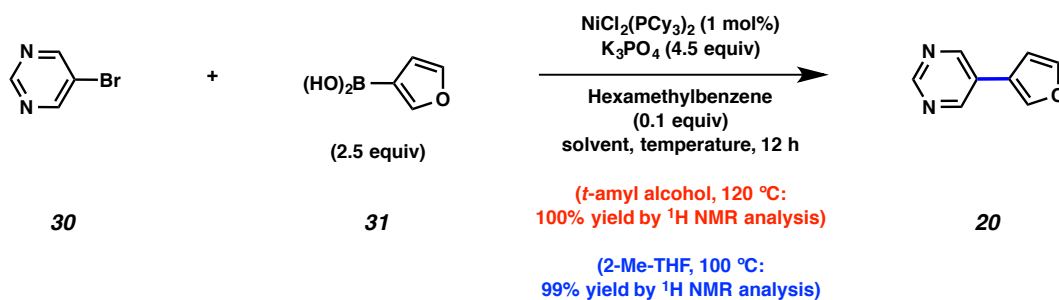
17 (Figure 4). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.¹⁰



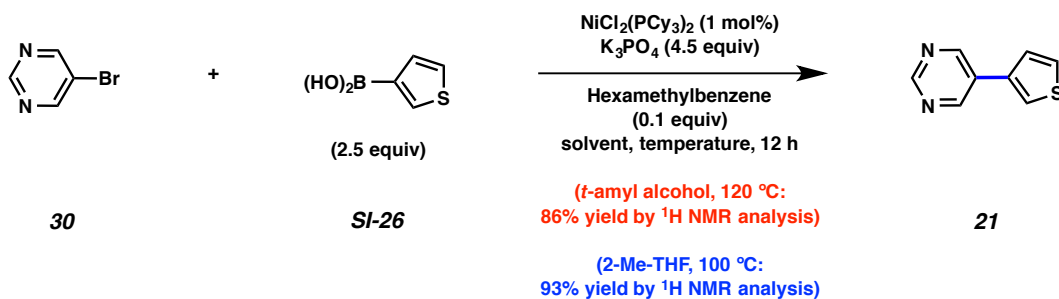
18 (Figure 4). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.^{1f}



19 (Figure 4). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. R_f 0.24 (1:1 Hexanes:EtOAc); ^1H NMR: (500 MHz, CDCl_3): δ 9.15 (s, 1H), 8.92 (s, 2H), 8.23 (dd, $J = 4.9, 1.8, 1\text{H}$), 7.63 (dd, $J = 7.3, 1.8, 1\text{H}$), 7.02 (dd, $J = 7.3, 4.9, 1\text{H}$), 3.97 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 160.8, 157.4, 156.5, 147.7, 138.2, 130.6, 117.6, 117.3, 53.7; IR (film): 3020, 2956, 1578, 1551, 1460, 1395, 1015, 798, 726 cm^{-1} ; HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{10}\text{H}_9\text{N}_3\text{O}$, 188.0824; found 188.0831.

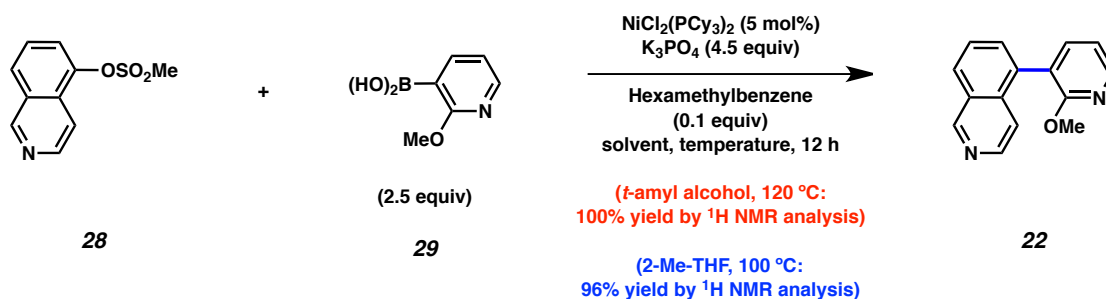


20 (Figure 4). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.¹¹



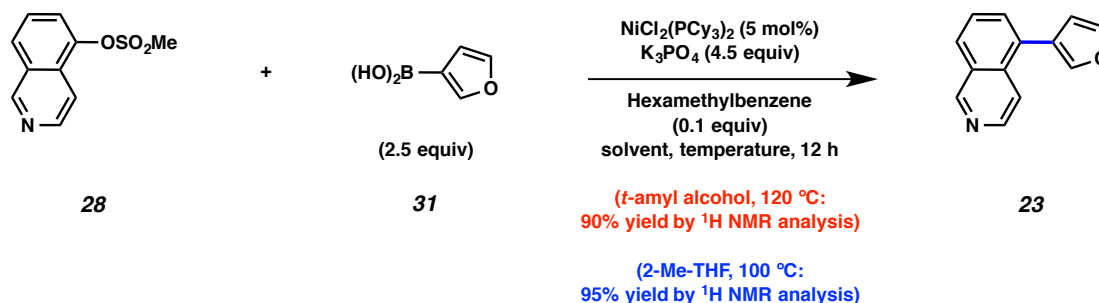
21 (Figure 4). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.¹²

G. Heterocycle-Heterocycle Cross-Couplings of Aryl Carbamates, Sulfamates, and Mesylates

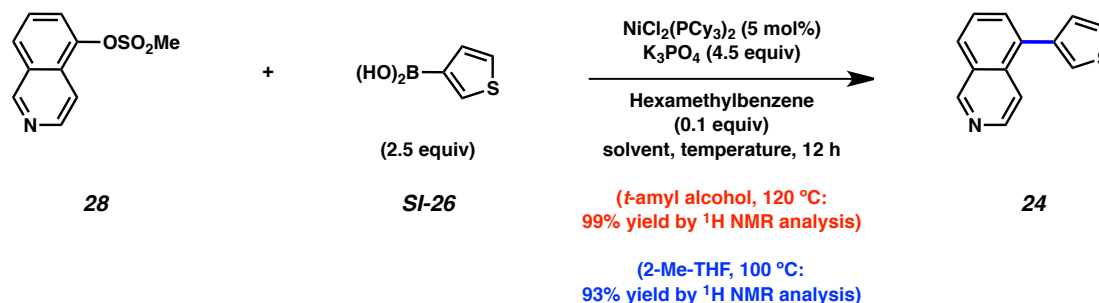


Representative Procedure (coupling of 28 is used as an example). 22 (Figure 4). A 1-dram vial was charged with anhydrous powdered K_3PO_4 (429.9 mg, 2.03 mmol, 4.50 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N_2 . Hexamethylbenzene (7.3 mg, 0.045 mmol, 0.10 equiv), boronic acid **29** (125.9 mg, 1.125 mmol, 2.50 equiv), $\text{NiCl}_2(\text{PCy}_3)_2$ (15.5 mg, 0.023 mmol, 5 mol%), and substrate **28** (104.9 mg, 0.45 mmol, 1.00 equiv) were added. The vial was then evacuated and backfilled with N_2 . To the vial, solvent (1.5 mL) was added and then the vial was sealed with a Teflon-lined screw cap. The mixture was allowed to stir rapidly at 23 °C for 1 h, and then heated to 100 °C (2-Me-THF) or 120 °C (*t*-amyl alcohol) for 12 h. The reaction vessel was cooled to 23 °C and then transferred to a test tube containing 1 M aqueous HCl (4 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were then washed with brine (2 mL), dried over Na_2SO_4 , and passed through a SiO_2 plug. The pad was washed with EtOAc (10 mL) and the organic layers were concentrated under reduced pressure. The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. R_f 0.40 (1:1 Hexanes:Acetone); ^1H NMR: (600 MHz, CDCl_3): δ 9.30 (d, $J = 0.9$, 1H), 8.46 (d, $J = 6.0$, 1H), 8.31 (dd, $J = 5.0$, 1.9, 1H), 8.01 (dt, $J = 8.0$, 0.9, 1H), 7.67 (dd, $J = 7.0$, 0.9, 1H), 7.63 (dd, $J = 7.0$, 1.3, 1H), 7.58 (dd, $J = 7.2$, 1.9, 1H), 7.35 (dt, $J = 5.9$, 0.9, 1H), 7.05 (dd, $J = 7.2$, 5.0, 1H), 3.87 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): 161.5, 153.0, 147.0, 143.3, 140.2, 134.5, 134.2, 131.7, 128.8, 127.8, 126.9, 122.0, 118.6, 116.9, 53.6; IR (film): 3404, 2931, 1618, 1574, 1459, 1400, 1015, 776 cm^{-1} ; HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{HC}_{15}\text{H}_{12}\text{N}_2\text{O}$, 237.1025; found 237.1028.

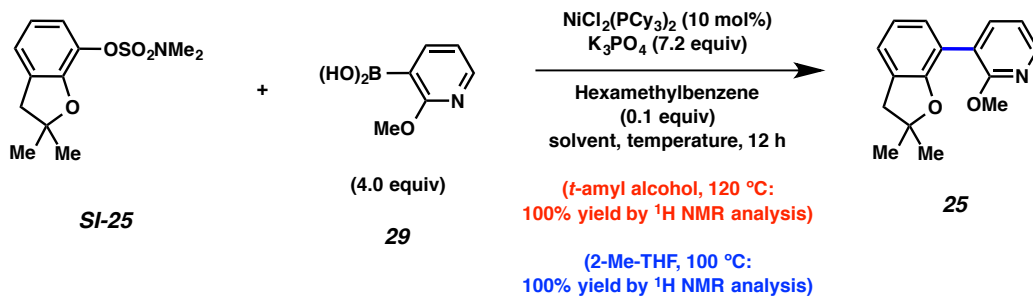
Any modifications of the conditions shown in this representative procedure are specified in the following schemes, which depict all of the results shown in Figure 4.



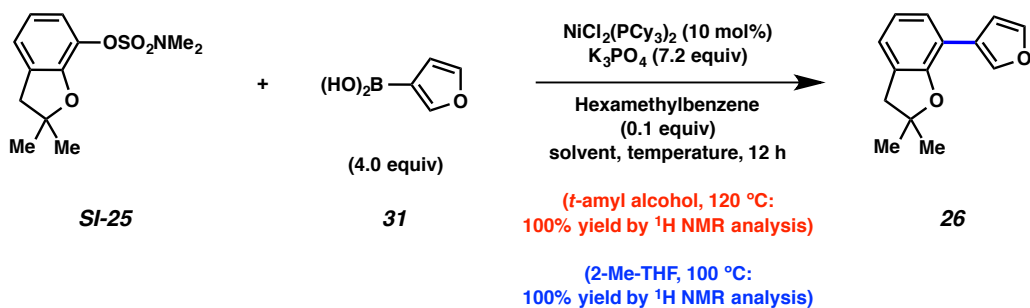
23 (Figure 4). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. Spectral data match those previously reported.¹⁰



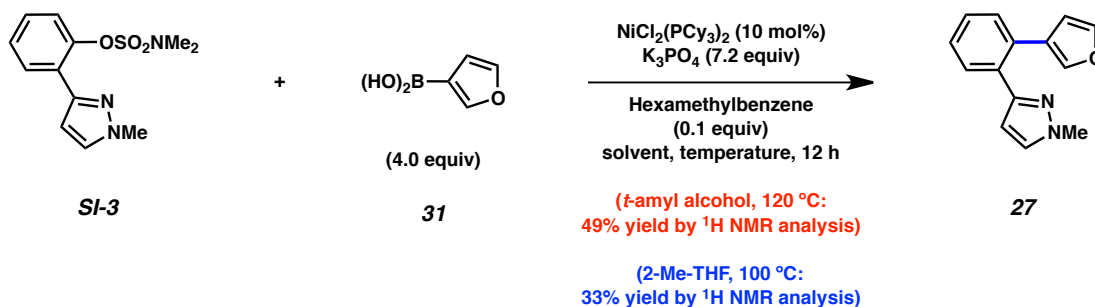
24 (Figure 4). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. R_f 0.43(1:1 Hexanes:EtOAc); ^1H NMR: (600 MHz, CDCl_3): δ 9.29 (s, 1H), 8.51 (d, $J = 6.0$, 1H), 7.96 (d, $J = 8.2$, 1H), 7.85 (d, $J = 6.0$, 1H), 7.71 (dd, $J = 7.2$, 1.2, 1H), 7.63 (t, $J = 7.2$, 1H), 7.50 (dd, $J = 4.9$, 3.0, 1H), 7.42 (dd, $J = 3.0$, 1.2, 1H), 7.30 (dd, $J = 4.9$, 1.2, 1H); ^{13}C NMR (150 MHz, CDCl_3): δ 151.9, 148.6, 142.5, 138.6, 133.4, 133.1, 129.9, 128.2, 128.1, 126.3, 125.9, 125.1, 123.0, 117.5; IR (film): 3072, 1938, 1721, 1615, 1585, 1370, 829, 787, 663 cm^{-1} ; HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_9\text{NS}$, 212.0534; found 212.0530.



25 (Figure 4). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. *R_f* 0.59 (3:1 Hexanes:EtOAc); ¹H NMR: (400 MHz, CDCl₃): δ 8.13 (dd, *J* = 4.9, 1.9, 1H), 7.70 (dd, *J* = 7.4, 1.9, 1H), 7.23 (d, *J* = 7.7, 1H), 7.12 (d, *J* = 7.2, 1H), 6.94 (dd, *J* = 7.2, 4.9, 1H), 6.87 (t, *J* = 7.4, 1H), 3.95 (s, 3H), 3.05 (s, 2H), 1.46 (s, 6H); ¹³C NMR (125 MHz, CDCl₃): δ 161.2, 156.7, 145.7, 145.6, 139.5, 129.4, 127.7, 124.7, 120.9, 119.7, 118.8, 116.7, 86.6, 53.5, 43.1, 28.3; IR (film): 2927, 1577, 1439, 1397, 1297, 1257 cm⁻¹; HRMS-ESI (*m/z*) [M+H]⁺ calcd for C₁₆H₁₇NO₂, 256.1338; found 256.1335.

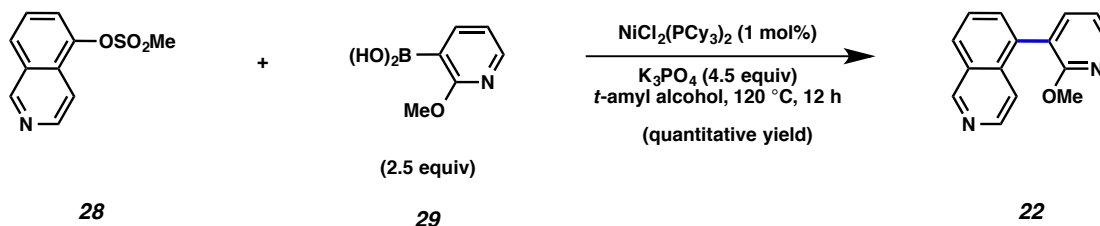


26 (Figure 4). The yield was determined by ¹H NMR analysis with hexamethylbenzene as an internal standard. *R_f* 0.38 (4:1 Hexanes:Benzene); ¹H NMR: (600 MHz, CDCl₃): δ 8.06 (s, 1H), 7.46 (t, *J* = 1.5, 1H), 7.30 (d, *J* = 7.5, 1H), 7.04 (dd, *J* = 7.2, 0.8, 1H), 6.86 (t, *J* = 7.5, 1H), 6.83 (d, *J* = 1.3, 1H), 3.06 (s, 2H), 1.53 (s, 6H); ¹³C NMR (125 MHz, CDCl₃): 155.8, 142.7, 141.3, 127.6, 125.1, 123.3, 121.7, 120.2, 114.9, 108.5, 86.9, 42.9, 28.6; IR (film): 2972, 1599, 1510, 1449, 1295, 1246, 1160, 1024 cm⁻¹; HRMS-ESI (*m/z*) [M+H]⁺ calcd for C₁₄H₁₄O₂, 215.1072; found 215.1065.



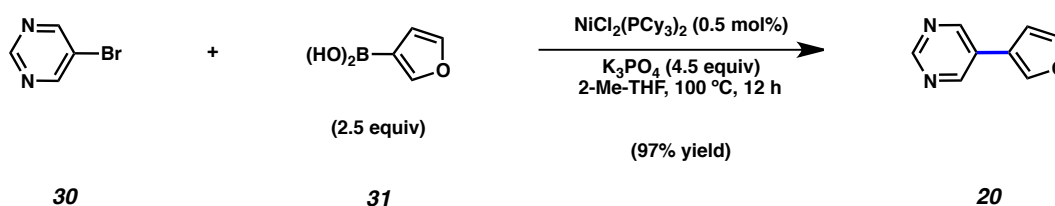
27 (Figure 4). The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. R_f 0.54 (1:1 Hexanes:EtOAc); ^1H NMR: (600 MHz, CDCl_3): δ 7.64–7.62 (m, 1H), 7.39–7.37 (m, 1H), 7.35–7.33 (m, 4H), 7.26–7.25 (m, 1H), 6.28 (d, $J = 0.9$, 1H), 5.99 (d, $J = 2.2$, 1H), 3.93 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 151.5, 142.2, 140.1, 133.0, 131.6, 130.3, 130.1, 129.9, 127.9, 127.4, 125.9, 111.9, 106.6, 39.0; IR (film): 2930, 1460, 1368, 1161, 850, 742 cm^{-1} .

H. Isolation Experiments



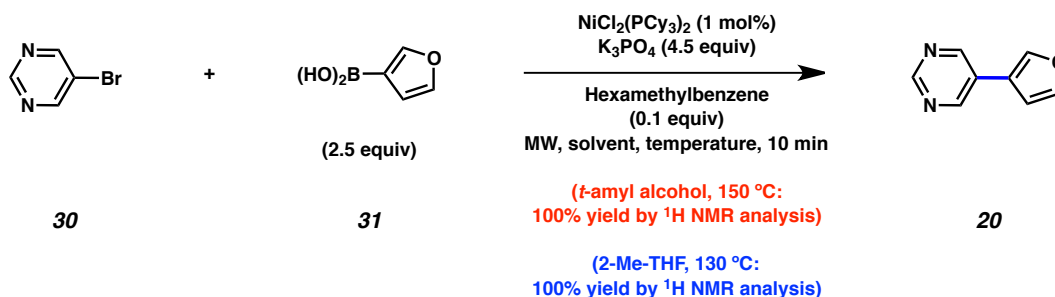
A 100 mL round bottom flask was charged with anhydrous powdered K_3PO_4 (4.28 g, 20.16 mmol, 4.50 equiv) and a magnetic stir bar. The flask and contents were flame-dried under reduced pressure, then allowed to cool under N_2 . Boronic acid **29** (1.71 g, 11.20 mmol, 2.50 equiv), $\text{NiCl}_2(\text{PCy}_3)_2$ (31.1 mg, 0.045 mmol, 1 mol%), and isoquinoline **28** (1.00 g, 4.48 mmol, 1.00 equiv) were added. The flask was then evacuated and backfilled with N_2 . *Tert*-amyl alcohol (14.93 mL) was added and the flask was equipped with a water-cooled condenser. The heterogeneous mixture was allowed to stir at 23 °C for 1 h, then heated to 120 °C for 12 h. The reaction vessel was cooled to 23 °C and then transferred to a round bottom flask containing CH_2Cl_2 (45 mL), EtOAc (15 mL), and MeOH (3 mL). Silica gel (24 mL) was added and the solvent was removed under reduced pressure to afford a free-flowing powder. This powder was

then dry-loaded onto a silica gel column (7 cm x 15 cm) and purified by flash chromatography (6:1 Hexanes:Acetone) to yield hetero-biaryl product **22** (1.06 g, 100% yield) as a white solid. R_f 0.40 (1:1 Hexanes:Acetone); $^1\text{H NMR}$: (600 MHz, CDCl_3): δ 9.30 (d, $J = 0.9$, 1H), 8.46 (d, $J = 6.0$, 1H), 8.31 (dd, $J = 5.0, 1.9$, 1H), 8.01 (dt, $J = 8.0, 0.9$, 1H), 7.67 (dd, $J = 7.0, 0.9$, 1H), 7.63 (dd, $J = 7.0, 1.3$, 1H), 7.58 (dd, $J = 7.2, 1.9$, 1H), 7.35 (dt, $J = 5.9, 0.9$, 1H), 7.05 (dd, $J = 7.2, 5.0$, 1H), 3.87 (s, 3H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): 161.5, 153.0, 147.0, 143.3, 140.2, 134.5, 134.2, 131.7, 128.8, 127.8, 126.9, 122.0, 118.6, 116.9, 53.6; IR (film): 3404, 2931, 1618, 1574, 1459, 1400, 1015, 776 cm^{-1} ; HRMS-ESI (m/z) $[\text{M}+\text{H}]^+$ calcd for $\text{HC}_{15}\text{H}_{12}\text{N}_2\text{O}$, 237.1025; found 237.1028.



A 250 mL round-bottom flask was charged with anhydrous powdered K_3PO_4 (30.04 g, 141.53 mmol, 4.50 equiv) and a magnetic stir bar. The flask and contents were flame-dried under reduced pressure, then allowed to cool under N_2 . Boronic acid **31** (8.80 g, 78.63 mmol, 2.50 equiv), $\text{NiCl}_2(\text{PCy}_3)_2$ (110.47 mg, 0.16 mmol, 0.5 mol%), and pyrimidine **30** (5.00 g, 31.45 mmol, 1.00 equiv) were added. The flask was then evacuated and backfilled with N_2 . 2-Me-THF (104.83 mL) was added and the flask was equipped with a water-cooled condenser. The heterogeneous mixture was allowed to stir at 23 $^\circ\text{C}$ for 1 h, then heated to 100 $^\circ\text{C}$ for 12 h. The reaction vessel was cooled to 23 $^\circ\text{C}$ and was quenched with 1 M HCl (20 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 100 mL). The combined organic layers were then washed with brine (50 mL), dried over MgSO_4 , and concentrated under reduced pressure. The crude residue was purified by flash chromatography (3:1 Hexanes:EtOAc) to yield bis(heterocyclic) compound **20** as a yellow solid (4.46 g, 97% yield). R_f 0.25 (3:1 Hexanes:EtOAc). Spectral data match those previously reported.¹¹

I. Cross-coupling Reactions Utilizing Microwave Heating



A 10 mL microwave vial was charged with anhydrous powdered K_3PO_4 (620.9 mg, 2.93 mmol, 4.50 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N_2 . Boronic acid **31** (181.8 mg, 1.63 mmol, 2.50 equiv), $\text{NiCl}_2(\text{PCy}_3)_2$ (4.5 mg, 0.033 mmol, 1 mol%), and pyrimidine **30** (103.3 mg, 0.65 mmol, 1.00 equiv) were added. The flask was then evacuated and backfilled with N_2 . The solvent (2.2 mL) was added and the heterogeneous mixture was subsequently placed in the (CEM Discover) microwave cavity and irradiated at the temperature indicated for 10 min (hold time). The reaction vessel was cooled to 23 °C and was quenched with 1 M HCl (2 mL). The layers were separated; the aqueous layer was extracted with EtOAc (3 x 2 mL). The combined organic layers were then washed with brine (2 mL), dried over MgSO_4 , and passed through a SiO_2 plug. The pad was washed with EtOAc (10 mL) and the organic layers were concentrated under reduced pressure. The yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard. R_f 0.25 (3:1 Hexanes:EtOAc). Spectral data match those previously reported.¹²

¹ (a) Quasdorf, K. W.; Tian, X.; Garg, N. K. *J. Am. Chem. Soc.* **2008**, *130*, 14422–14423. (b) Mesganaw, T.; Fine Nathel, N. F.; Garg, N. K. *Org. Lett.* **2012**, *14*, 2918–2921. (c) Antoft-Finch, A.; Blackburn, T.; Snickus, V. *J. Am. Chem. Soc.* **2009**, *131*, 17750–17752. (d) Mesganaw, T.; Silberstein, A. L.; Ramgren, S. D.; Fine Nathel, N. F.; Hong, X.; Liu, P.; Garg, N. K. *Chem. Sci.* **2011**, *2*, 1766–1771. (e) Quasdorf, K. W.; Antoft-Finch, A.; Liu, P.; Silberstein, A. L.; Komaromi, A.; Blackburn, T.; Ramgren, S. D.; Houk, K. N.; Snieckus, V.; Garg, N. K. *J. Am. Chem. Soc.* **2011**, *133*, 6352–6363. (f) Leowanawat, P.; Zhang, N.; Resmerita, A.-M.; Rosen, B. M.; Percec, V. *J. Org. Chem.* **2011**, *76*, 9946–9955. (g) Silberstein, A. L.; Ramgren, S. D.; Garg, N. K. *Org. Lett.* **2012**, *14*, 3796–3799.

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⁹ Pena, M. A.; Sestelo, J. P.; Sarandeses, L. A. *J. Org. Chem.* **2007**, *72*, 1271–1275.

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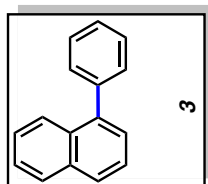
¹¹ Biolatto, B.; Molander, G. A. *J. Org. Chem.* **2003**, *68*, 4302–4314.

¹² Oberli, M. A.; Buchwald, S. L. *Org. Lett.* **2012**, *14*, 4606–4609.

¹H NMR Spectra

LH-2-239-2.1

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7.903
7.888
7.860
7.863
7.539
7.515
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7.476
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7.415
7.410
7.260

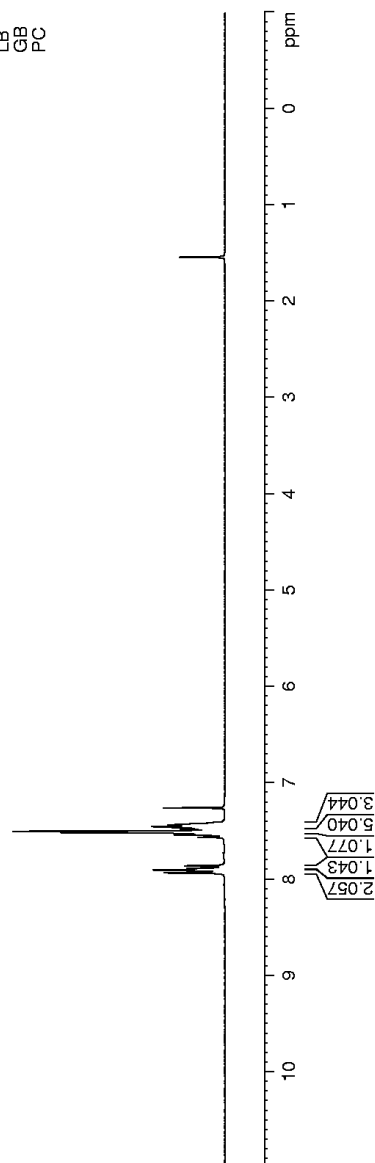


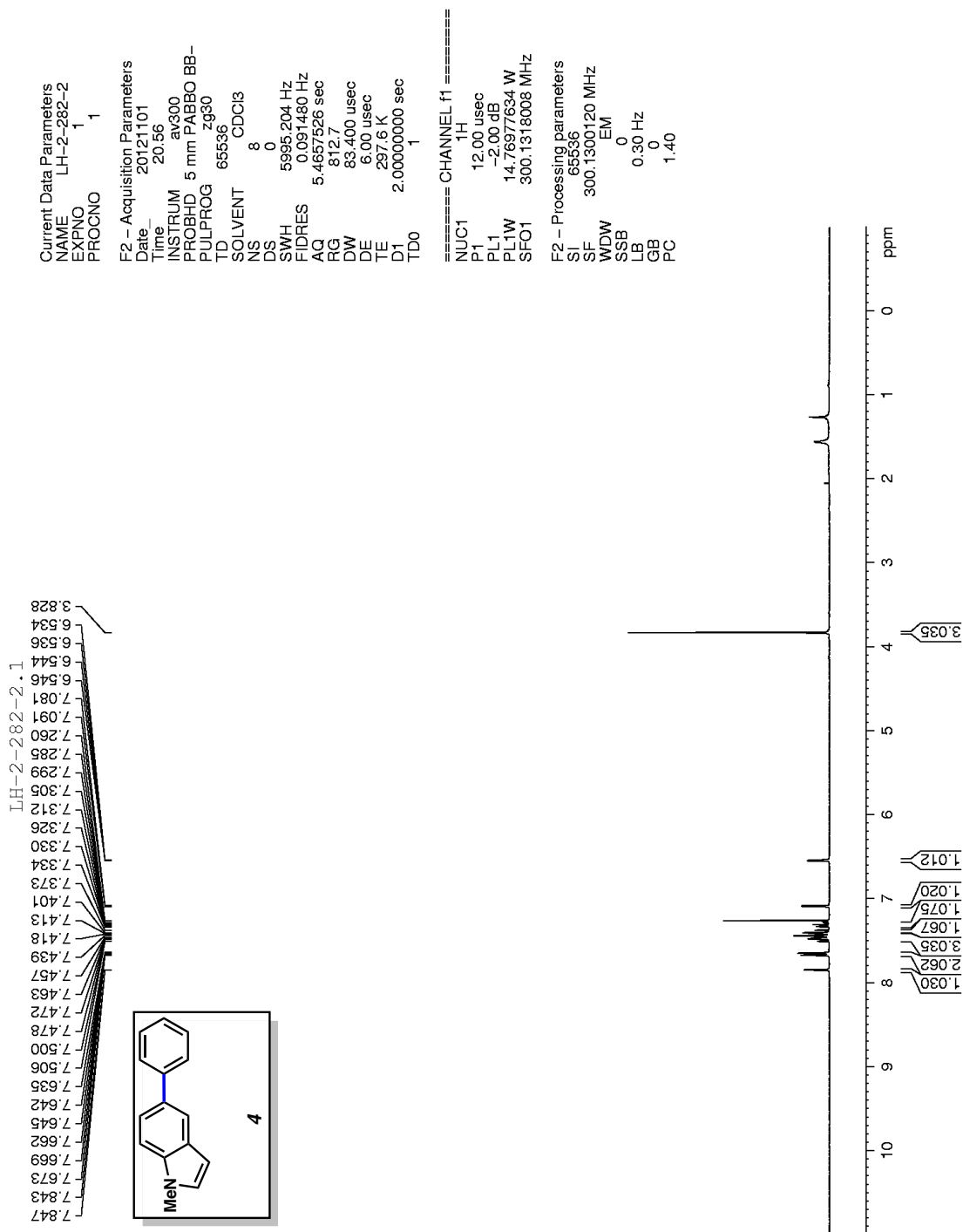
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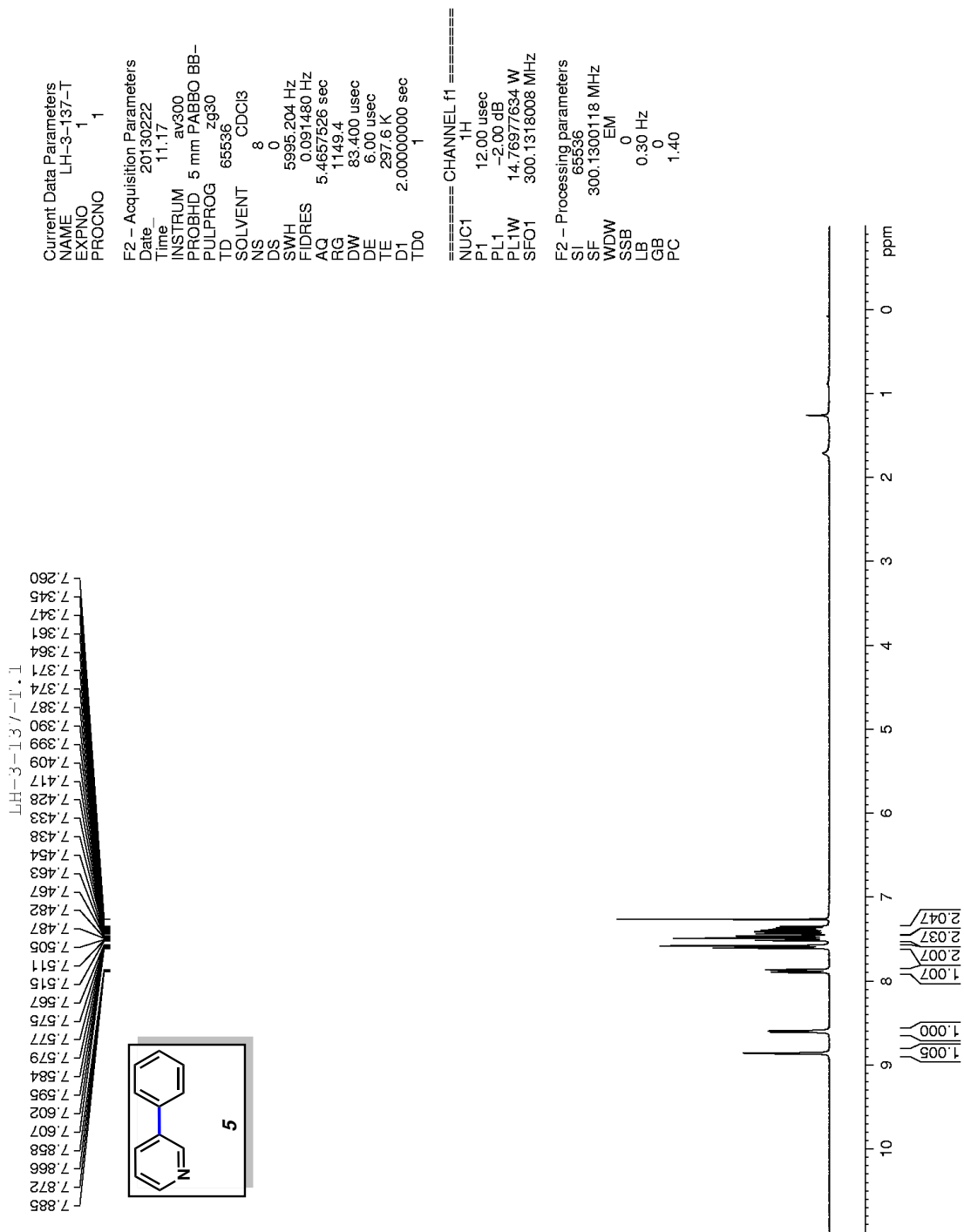
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 TD0 1

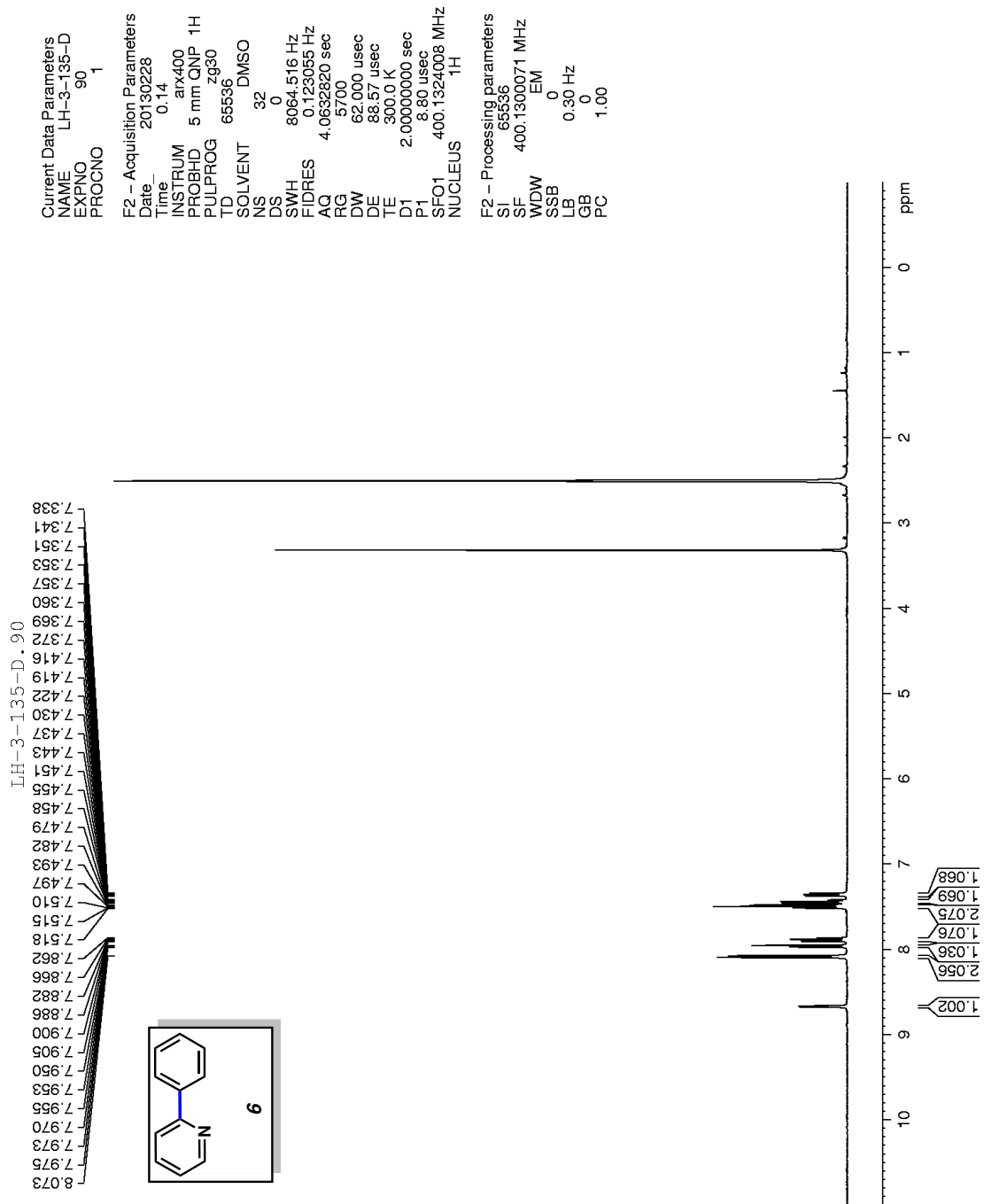
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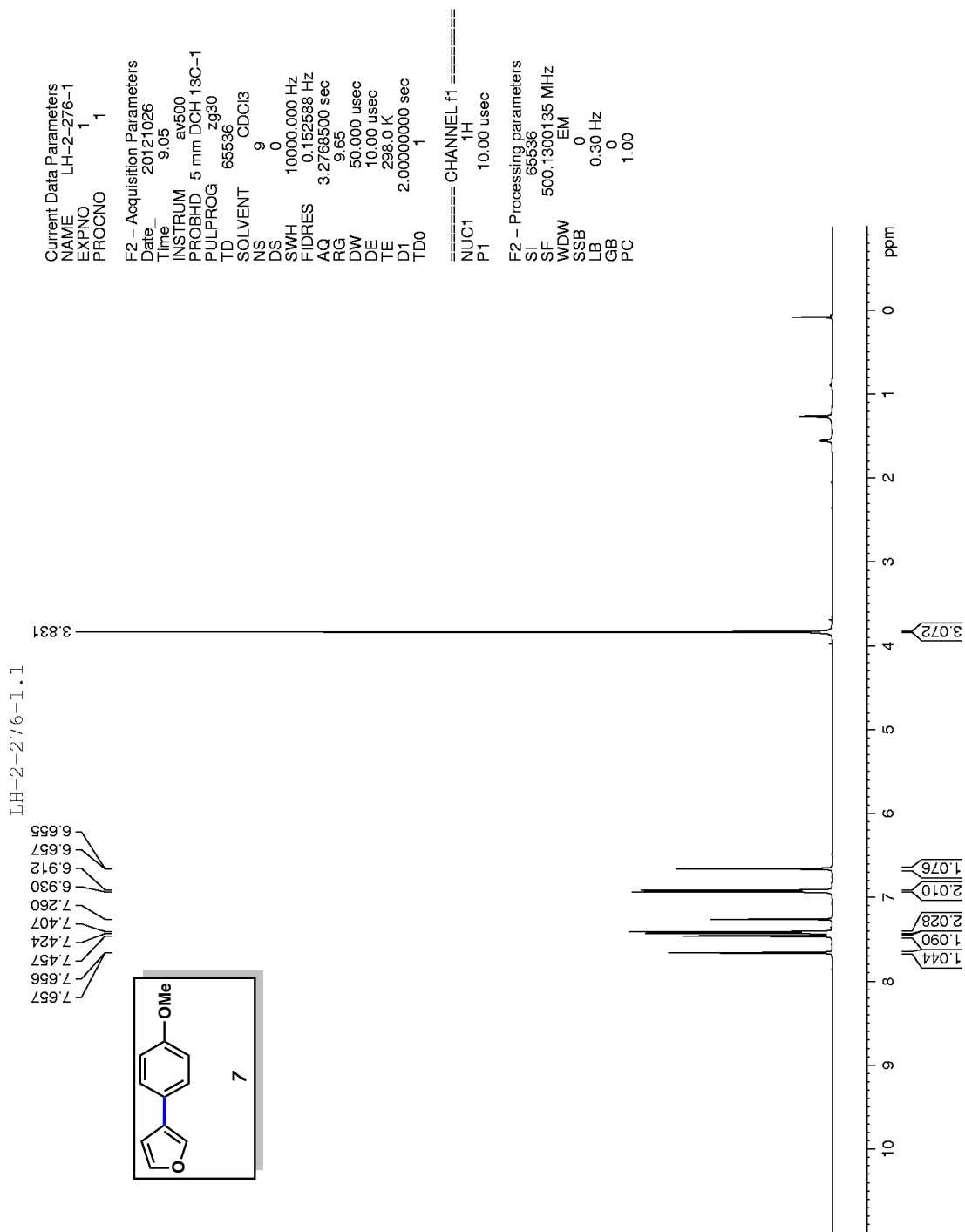
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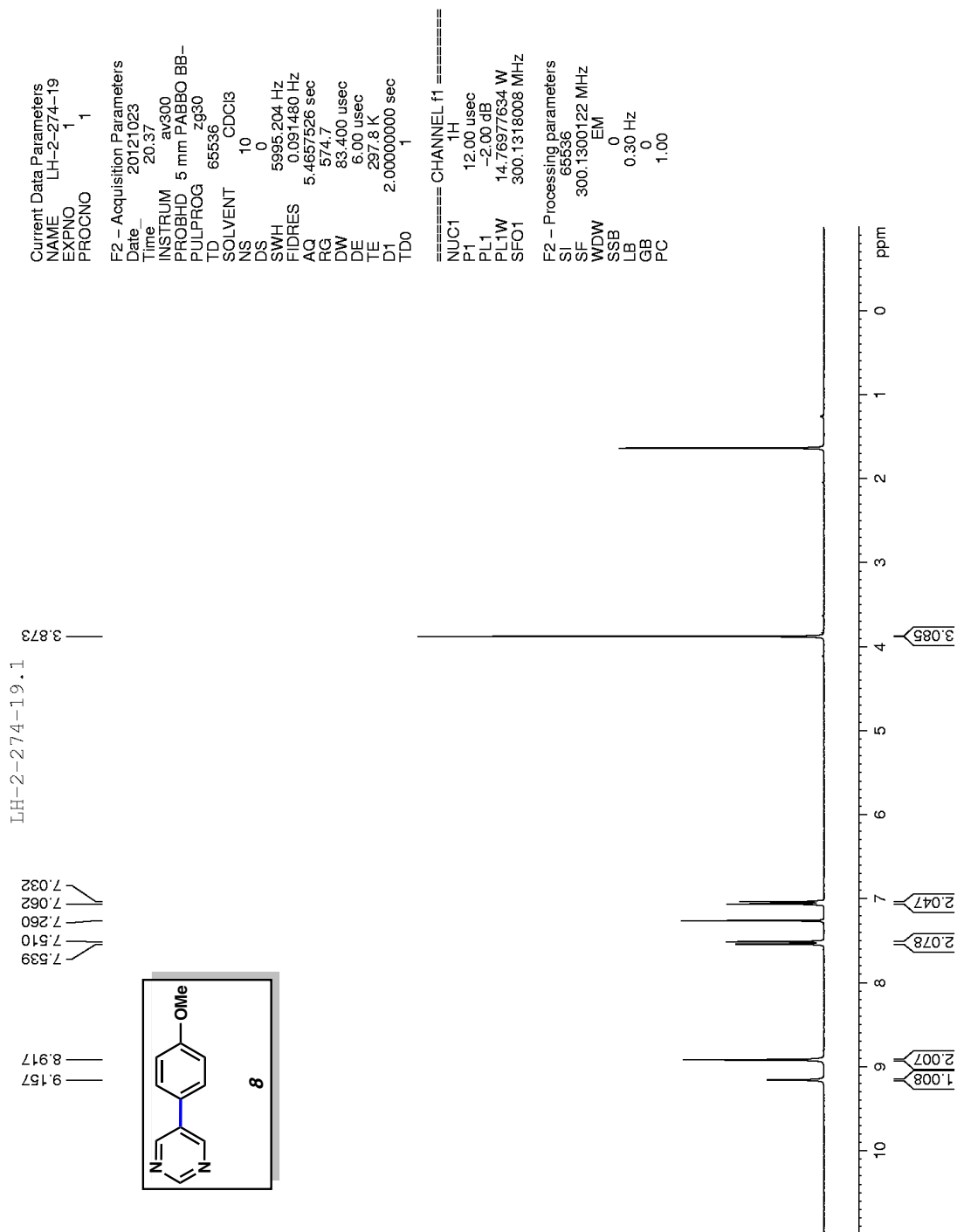


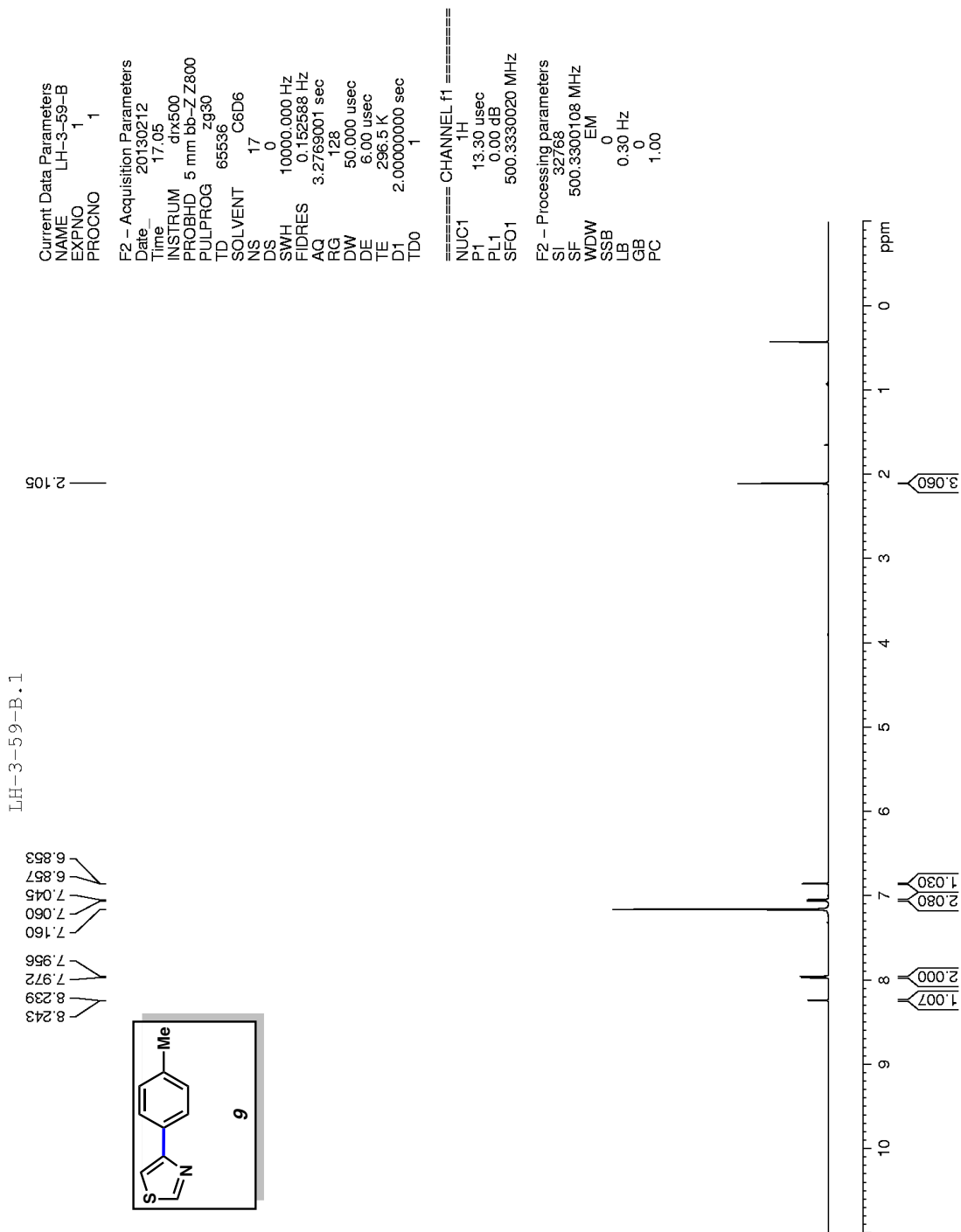


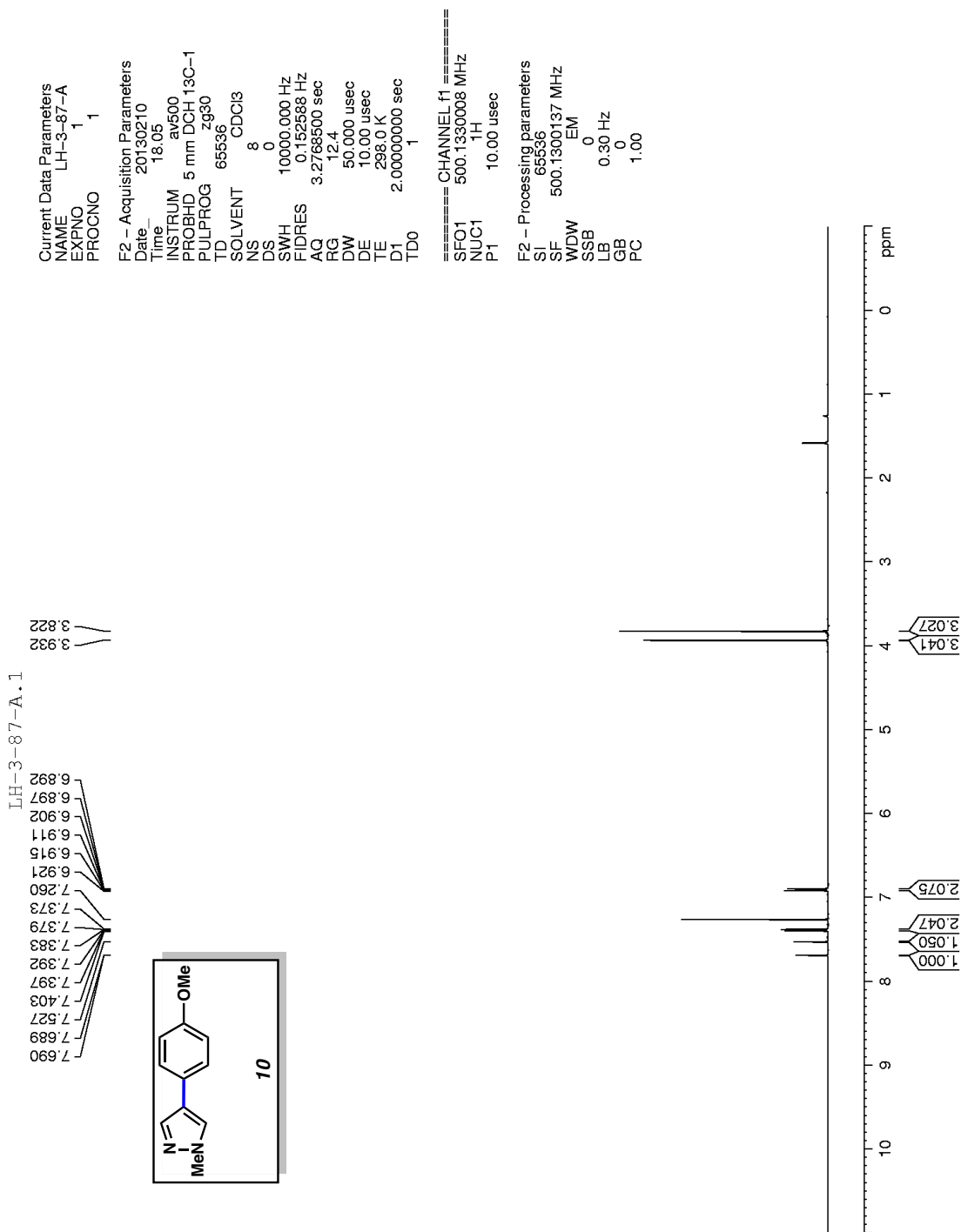


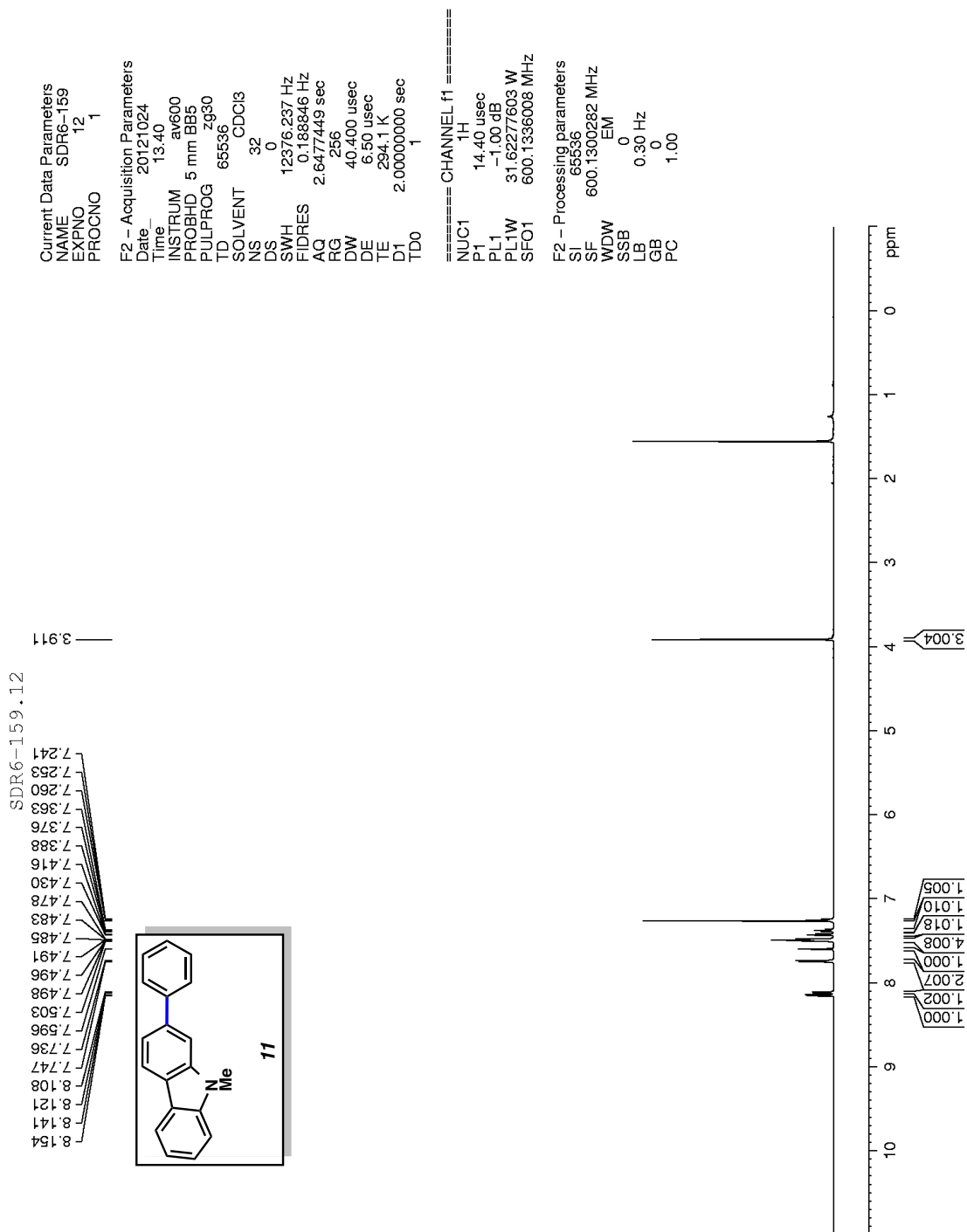






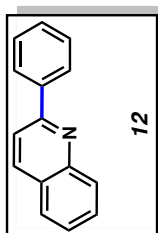






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7.260

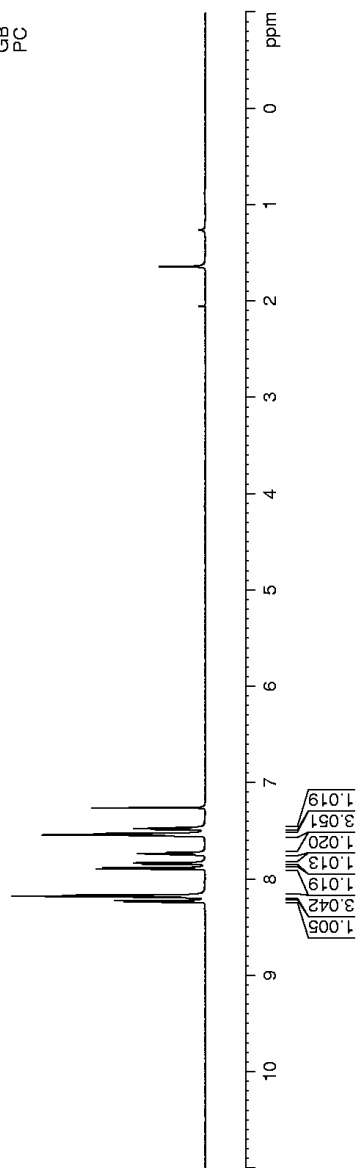


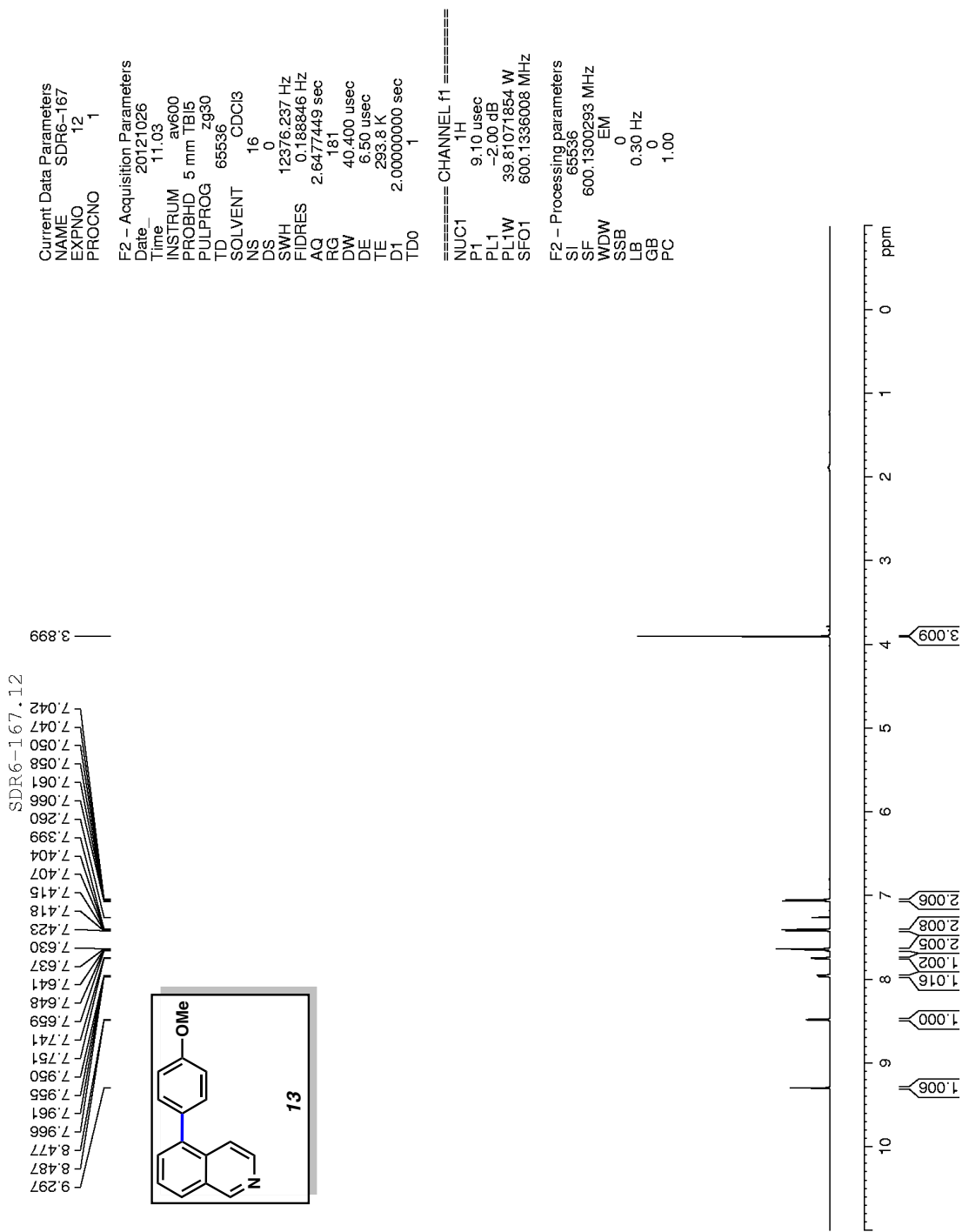
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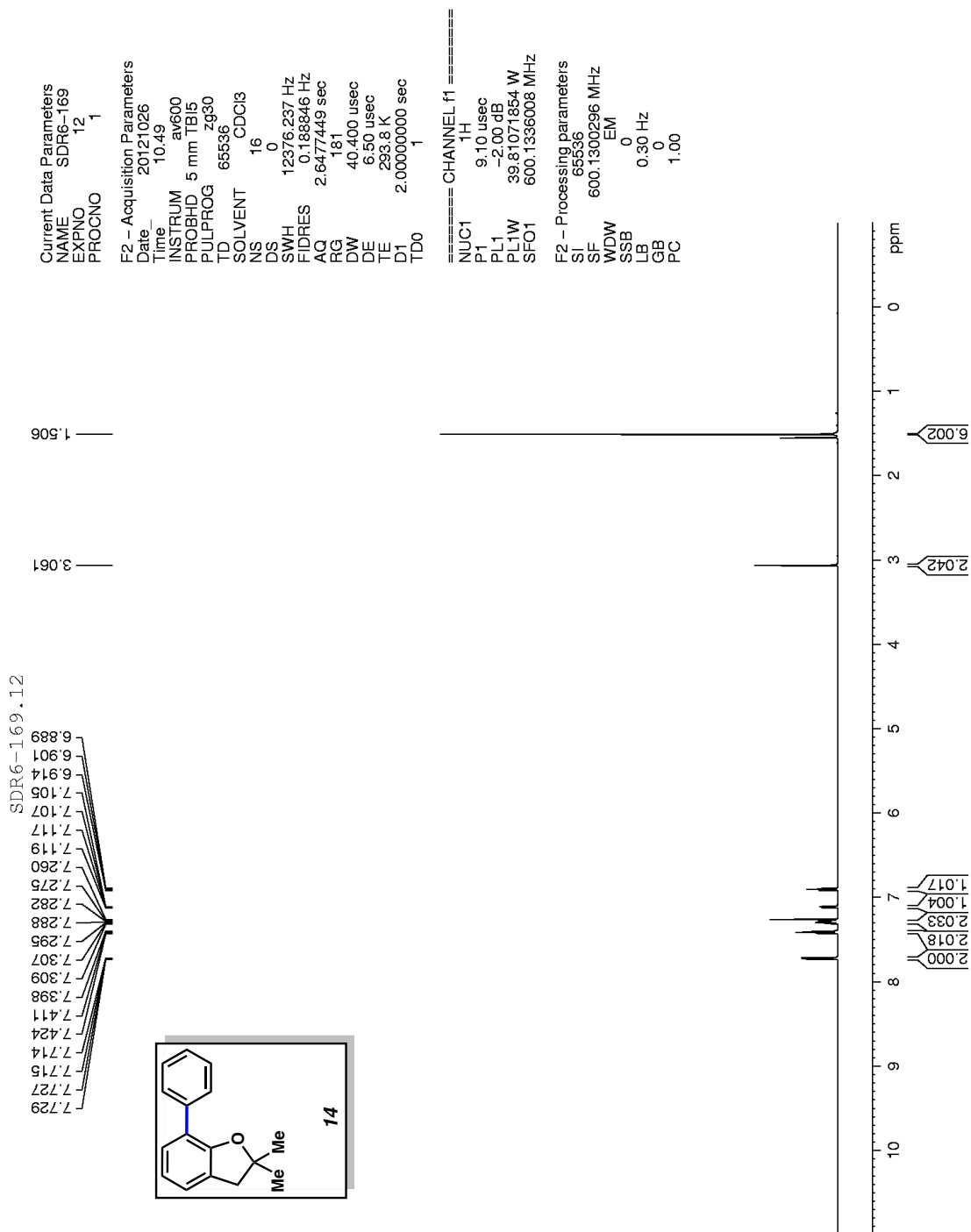
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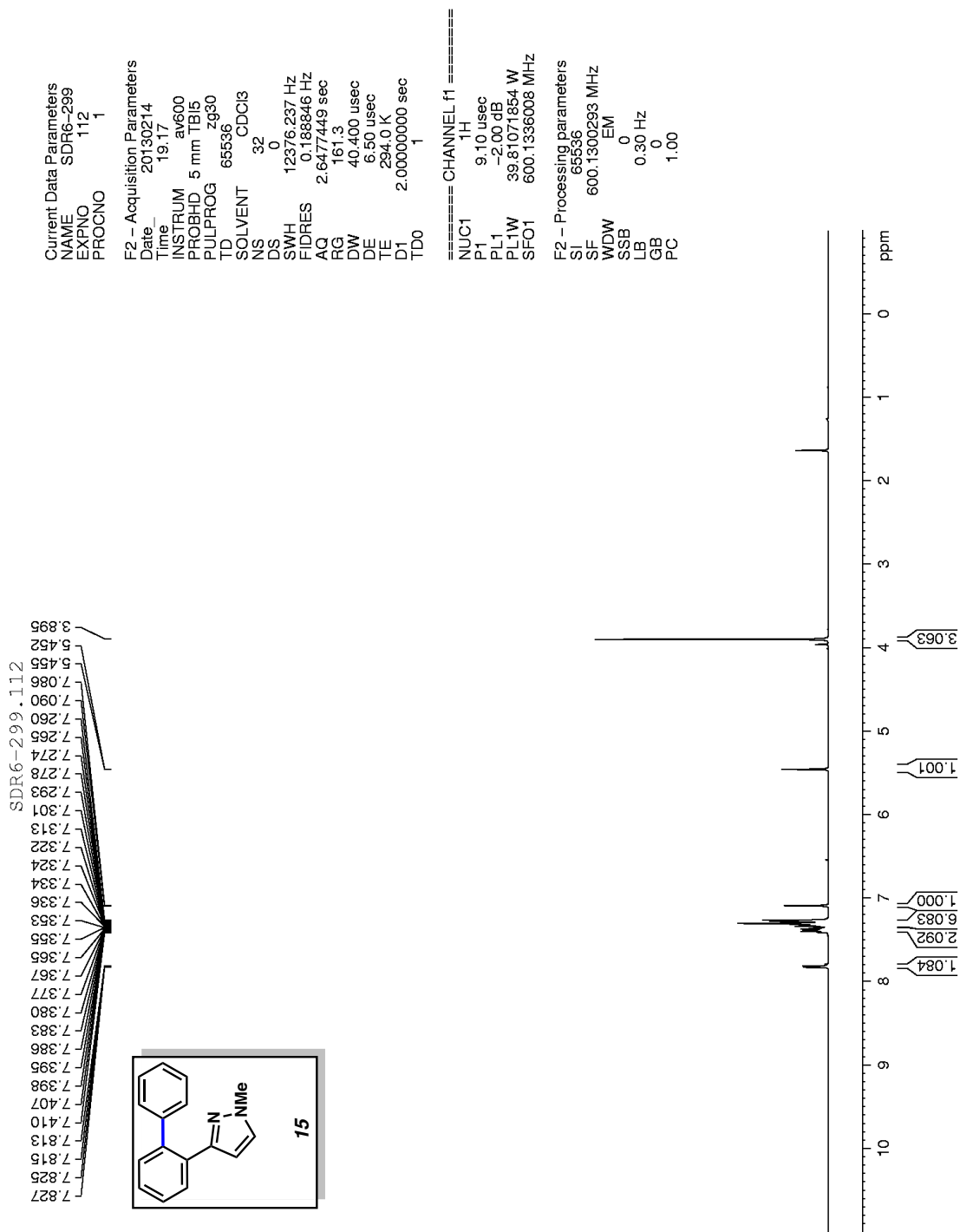
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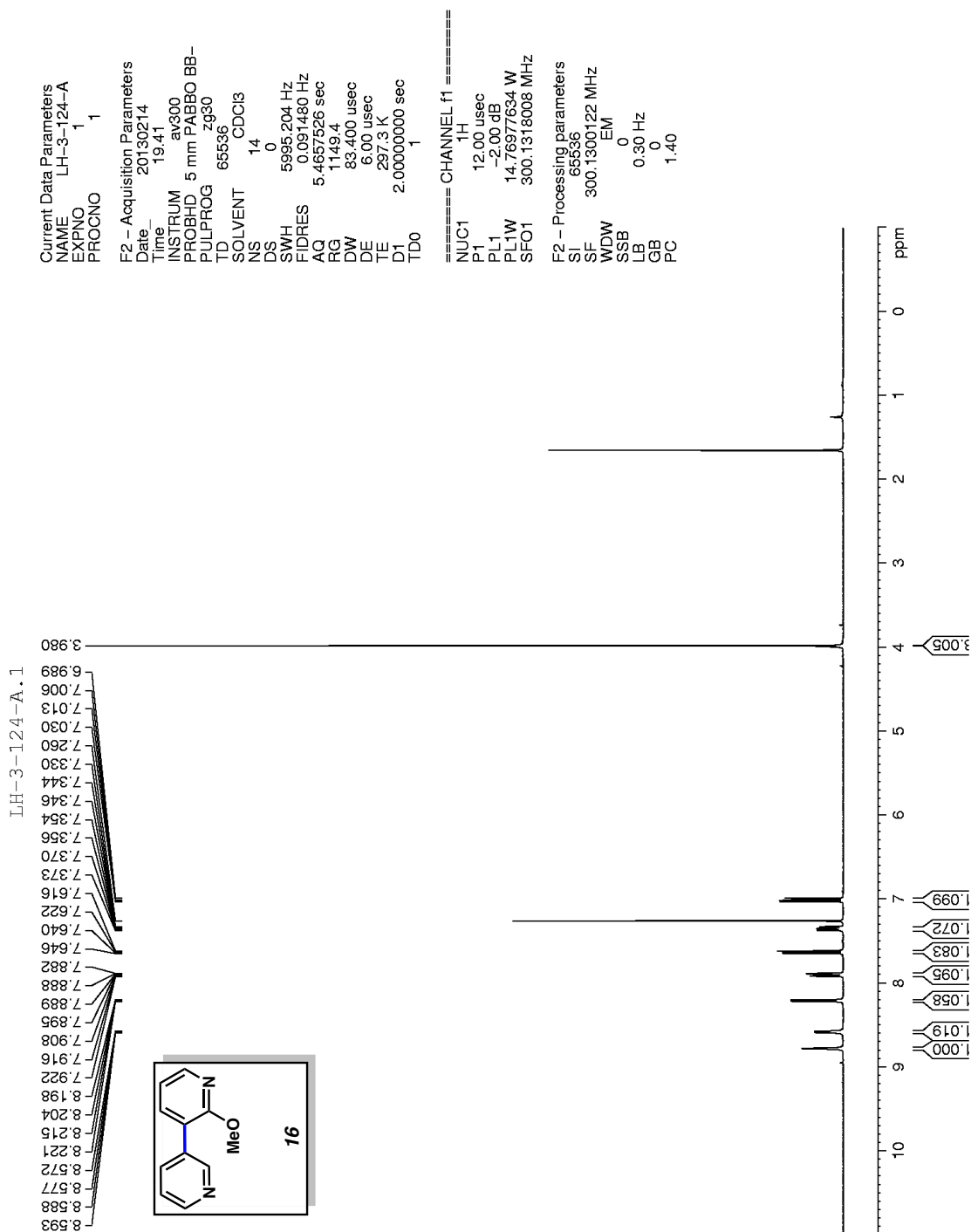
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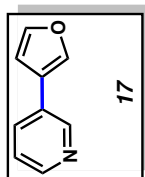






LH-3-97-A.180

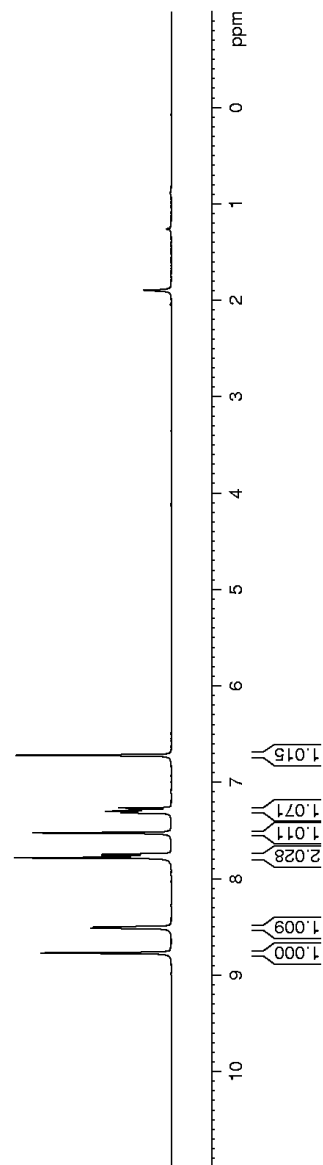
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7.260
6.716

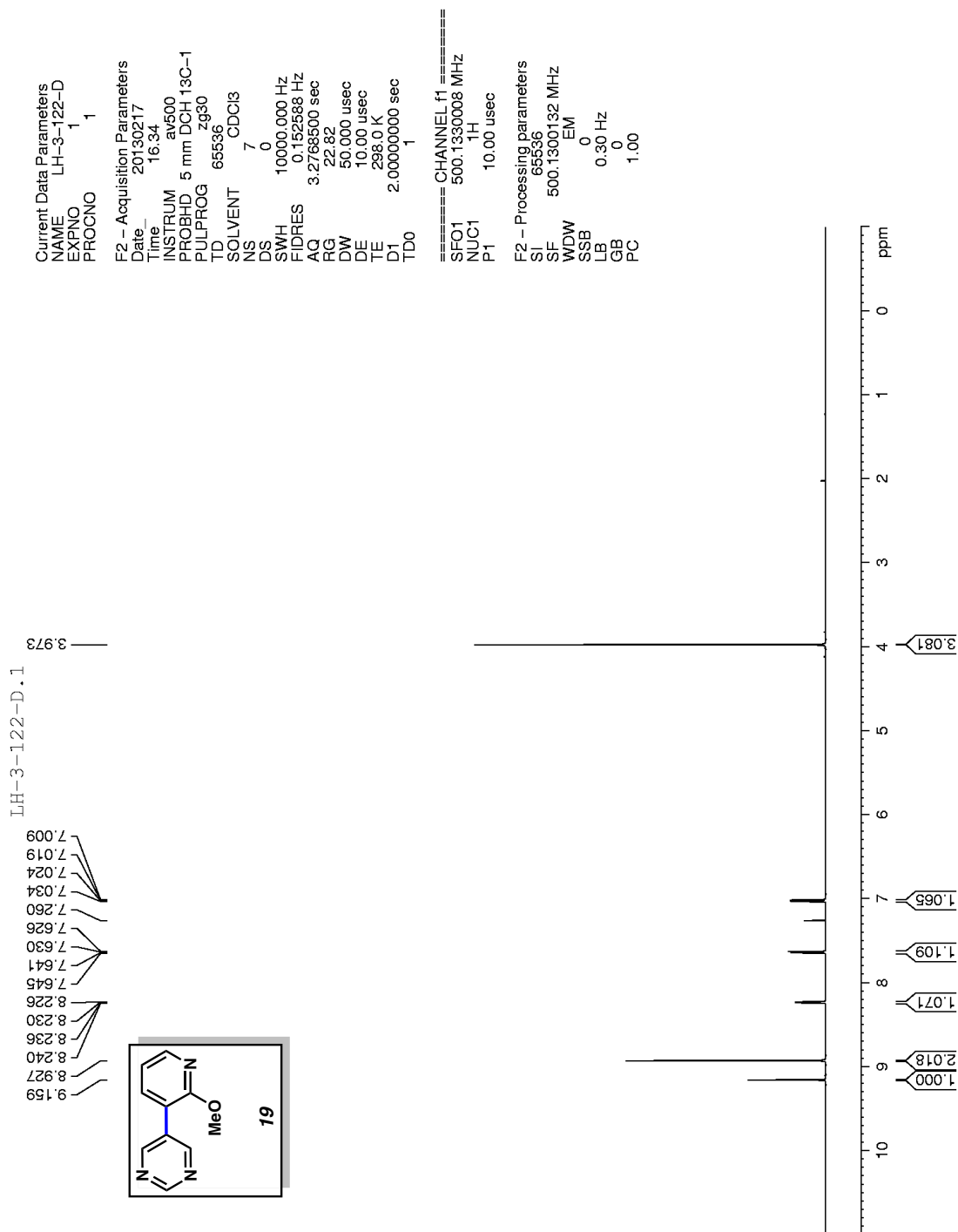


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 NUCLEUS 1H

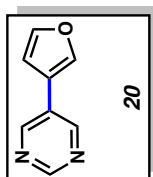
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LH-3-99-A.1

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6.734

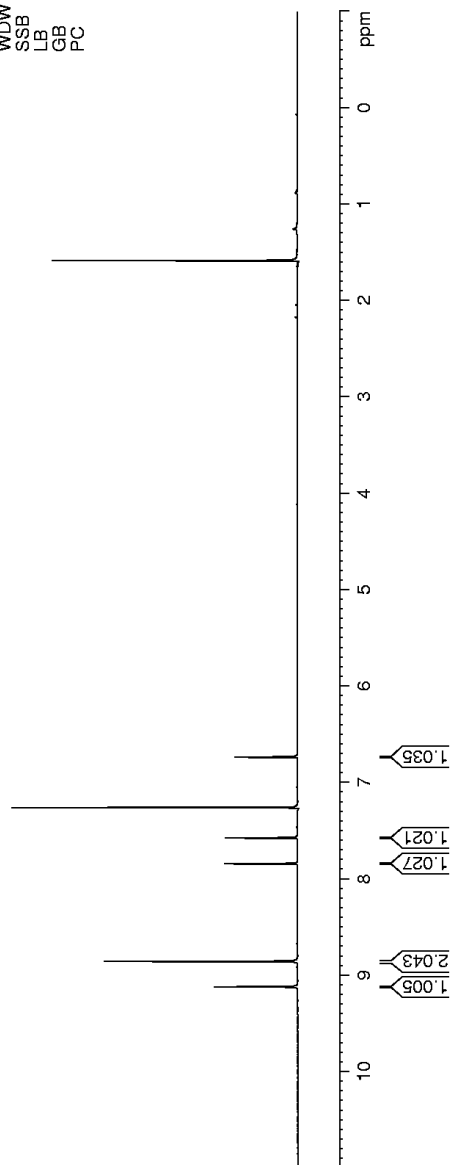


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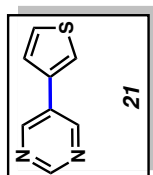
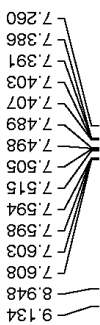
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DE 10.00 usec
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TD0 1

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NUC1 1H
P1 10.00 usec

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PC 1.00



LH-3-102-B.1

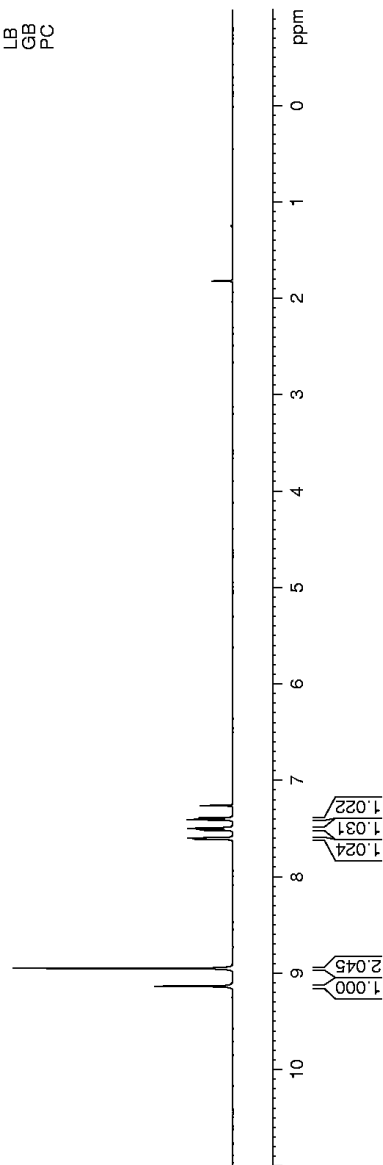


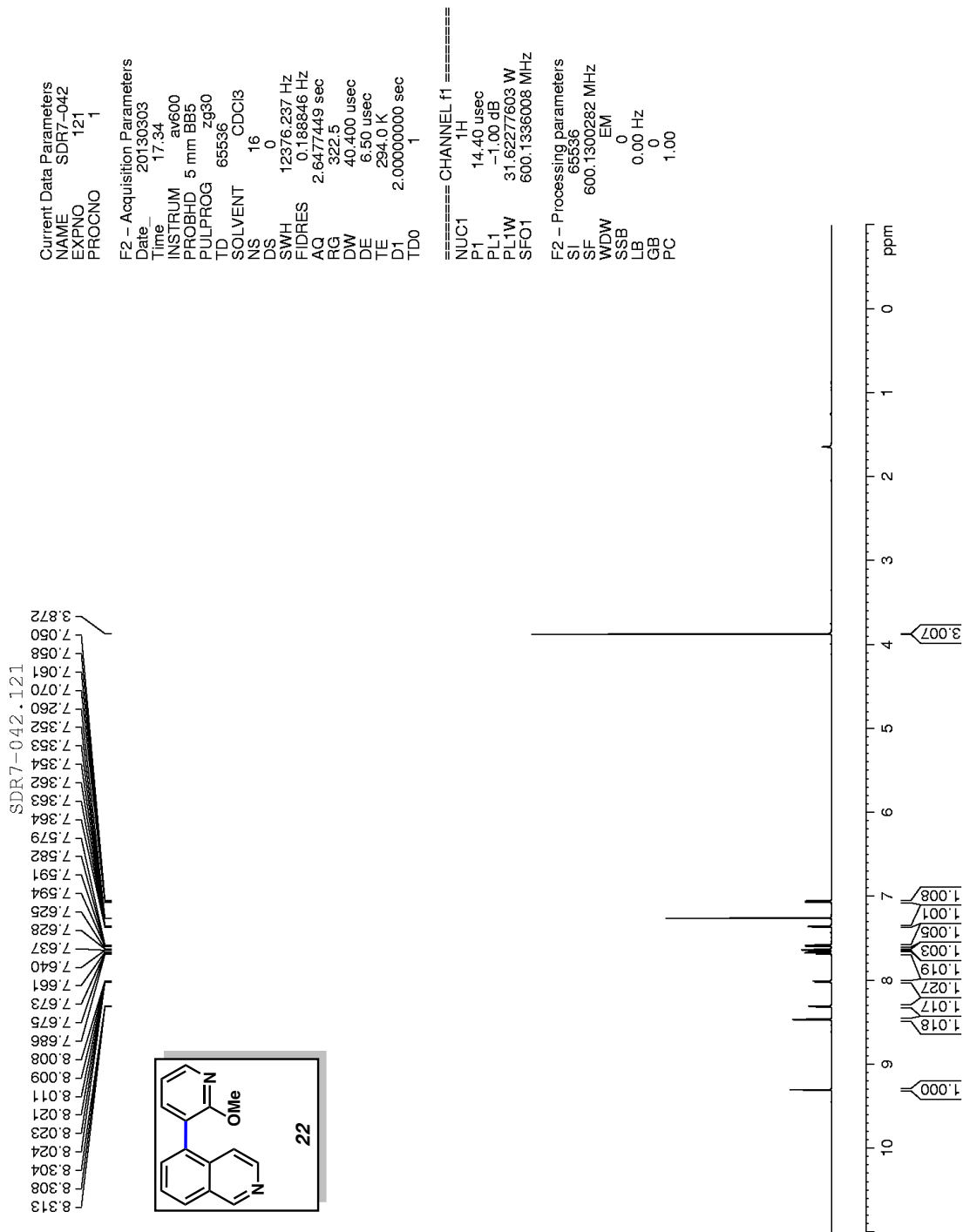
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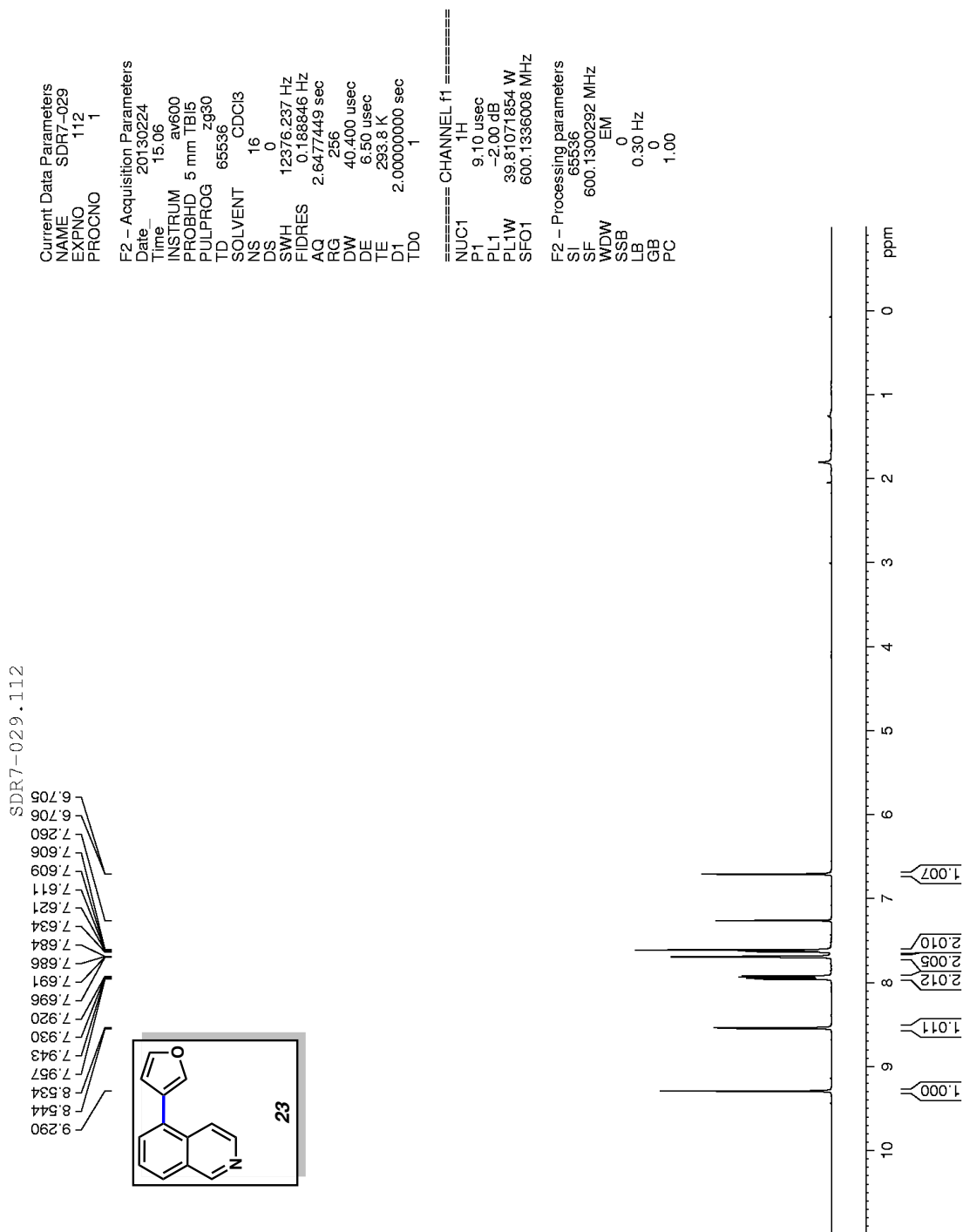
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 FIDRES 0.091480 Hz
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 RG 812.7
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 DE 6.00 usec
 TE 297.7 K
 D1 2.00000000 sec
 TD0 1

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 PL1 -2.00 dB
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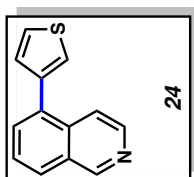






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7.260

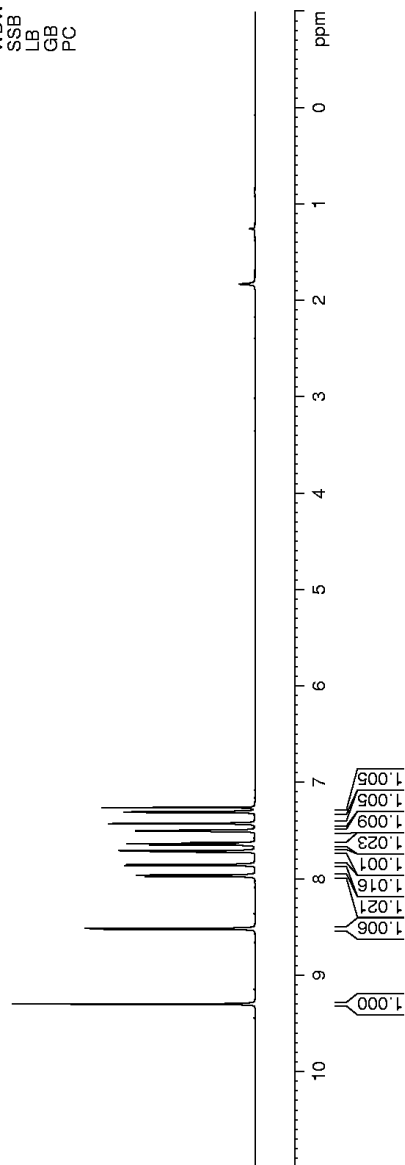


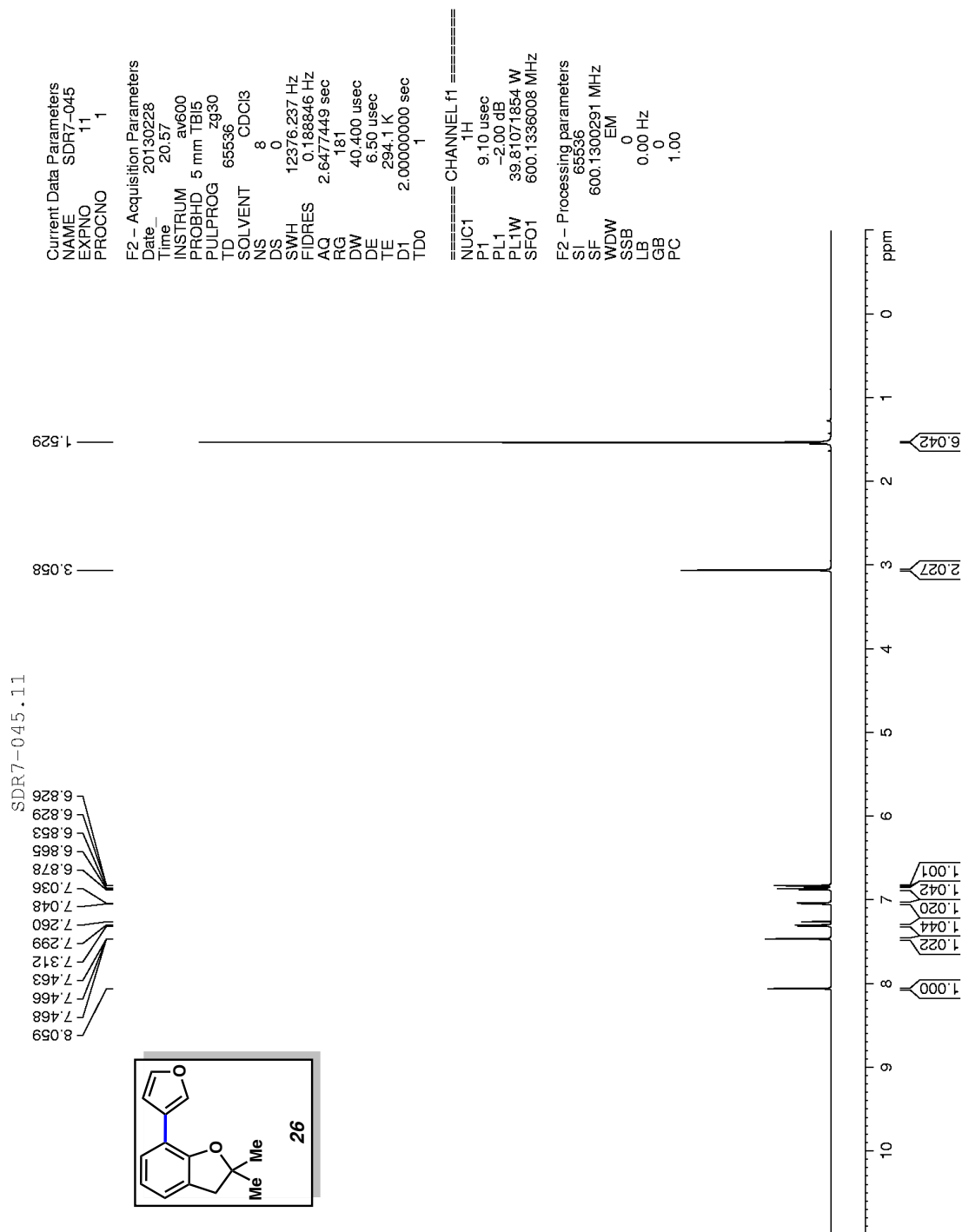
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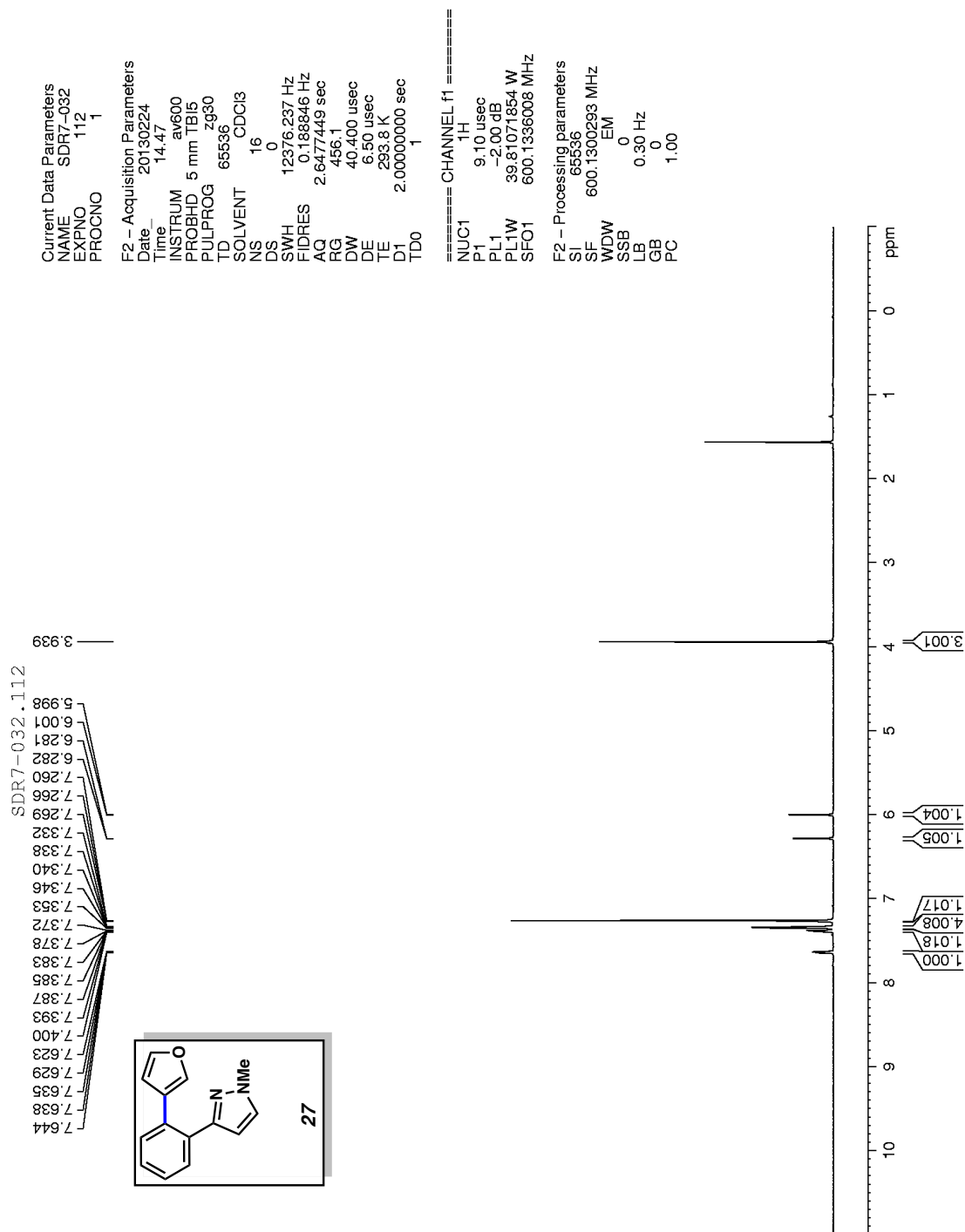
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AQ 2.6477449 sec
RG 228.1
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DE 6.50 usec
TE 293.9 K
D1 2.00000000 sec
TD0 1

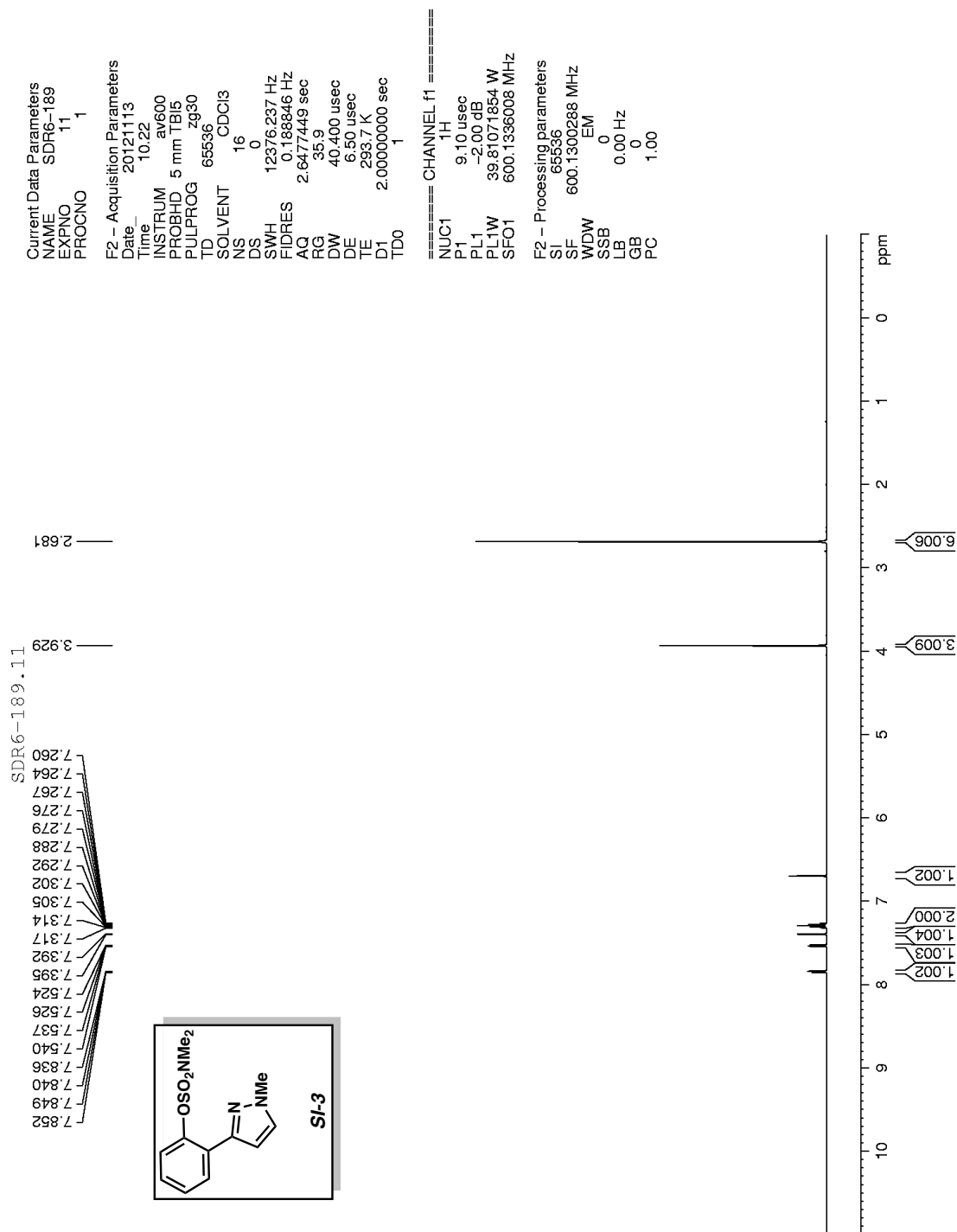
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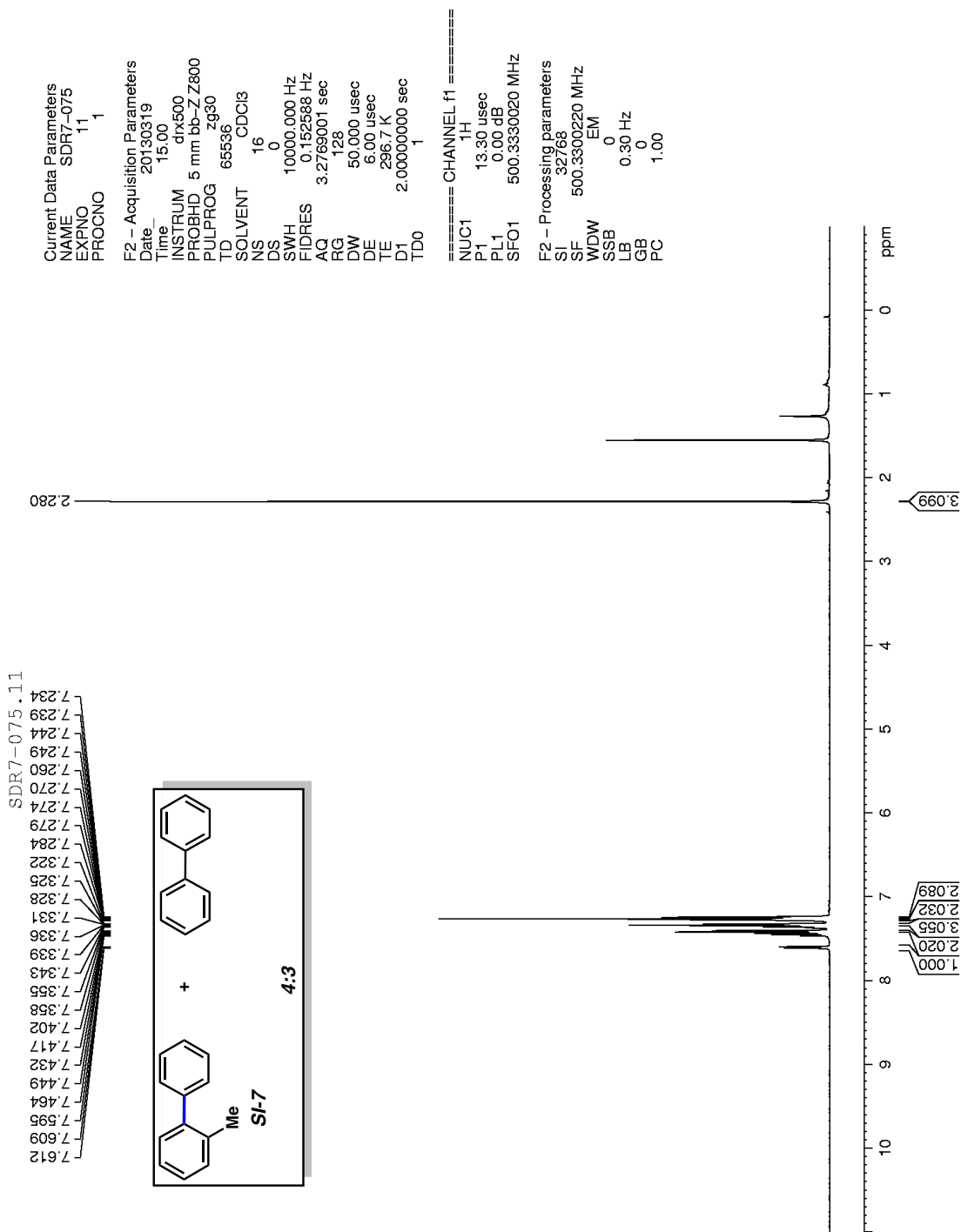
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SSB 0
LB 0.30 Hz
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^{13}C NMR Spectra

