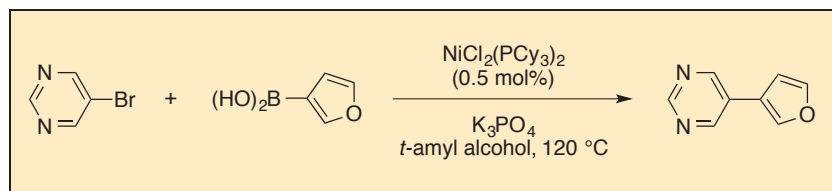# Nickel-Catalyzed Suzuki–Miyaura Coupling in *t*-Amyl Alcohol for the Preparation of 5-(Furan-3-yl)pyrimidine

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Checked by Kristen Flynn, Alyssa Antropow, and Mohammad Movassaghi

Procedure

5-(Furan-3-yl)pyrimidine. A 1000-mL round-bottomed flask is charged with a Teflon-coated, octagon-shaped magnetic stir bar (4.0 x 0.5 cm) and K_3PO_4 (60.0 g, 283 mmol, 4.50 equiv), and then attached to a gas inlet adapter (90°). The flask and its contents are flame-dried (Note 1) under oil pump vacuum (1.0 mmHg) for ca. 5 min (Note 2). After cooling to 23 °C under nitrogen, the gas inlet adapter is removed and the flask is charged with 5-bromopyrimidine (10.0 g, 63.0 mmol, 1 equiv), 3-furanylboronic acid (17.6 g, 158 mmol, 2.50 equiv), and $NiCl_2(PCy_3)_2$ (0.220 g, 0.315 mmol, 0.500 mol%) (Note 3). The flask is equipped with a 24/40 oven-dried reflux condenser and a septum with a needle, and then evacuated and backfilled with nitrogen (3x). *tert*-Amyl alcohol (210 mL, 0.3 M) is added via cannula from an oven-dried, argon purged graduated cylinder through the septum under a flow of argon. The resulting reddish heterogeneous mixture is stirred at 23 °C for 1 h under positive nitrogen pressure (Figure 1) (Note 4).

**Figure 1. Reddish heterogeneous mixture formed
after mixing the reagents (Photo provided by
Submitters)**

The reaction vessel containing a brown heterogeneous mixture and
equipped with a reflux condenser is placed into a pre-heated oil bath set at a
temperature of 120 °C (Figure 2) (Note 5).

**Figure 2. Brown heterogeneous mixture resulted after
stirring at 120 °C for 1 h (Photo provided by Submitters)**

After stirring for 1 h, the reaction vessel containing a brown greenish
heterogeneous mixture is removed from the oil bath and cooled to 23 °C.
Thin layer chromatography is performed to check the progress of the
reaction (hexanes:EtOAC = 3:1, R_f = 0.31) (Figure 3). Into the flask is added
deionized water (100 mL) and Et_2O (200 mL), and the resulting mixture is
stirred for 15 min (Figure 4).

Figure 3. Thin layer chromatography of the crude reaction mixture (left lane = 5-bromopyrimidine, middle lane = cross spots of 5-bromopyrimidine and the reaction mixture, right lane = reaction mixture) (Photo provided by Submitters)

Figure 4. Brown homogenous mixture was formed upon stirring with water and diethyl ether (Photo provided by Submitters)

The combined mixture is then poured into a 1000-mL separatory funnel. The reaction flask is rinsed once with H_2O (100 mL) and Et_2O (100 mL), and both washes are transferred to the separatory funnel (Figure 5a). The organic and aqueous layers are separated. The organic layer is washed

sequentially with 1.0 M NaOH (2 x 200 mL) (Figure 5b) and saturated sodium chloride solution (200 mL) (Figure 5c).
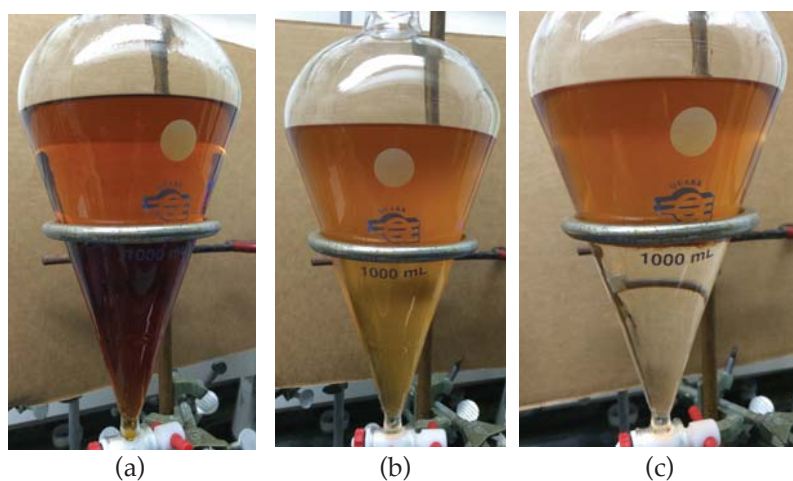

Figure 5. (a) The reaction mixture with water and diethyl ether in a separatory funnel. (b) The organic layer and 1.0 M NaOH. (c) The organic layer and saturated sodium chloride (Photo provided by Submitters)

The organic layer is then dried over Na_2SO_4 (100 g) for 15 min and filtered over a 150-mL glass-fritted filter packed with celite (30 g). The drying agent is rinsed with Et_2O (100 mL) and the combined organics are concentrated by rotary evaporation (275 mmHg, 30 °C to remove Et_2O and 30 mmHg, 50 °C to remove *tert*-amyl alcohol, The temperature was controlled by a water bath). In a 500-mL round-bottomed flask fitted with a 24/40 reflux condenser, the tan solid is dissolved in 200 mL of hexanes, which is brought to 80 °C by heating in an oil bath (Notes 6 and 7). Upon complete dissolution, the filtrate is allowed to cool to room temperature and then cooled at –20 °C for 12 h. The light tan crystals that form are collected by filtration on a 150 mL fritted funnel and washed with 100 mL of ice-cold hexane (0 °C). The resulting crystals are transferred to a 100 mL round-bottomed flask and then dried under oil pump vacuum (1.0 mmHg, 23 °C) for 8 h. The mother liquor is concentrated and a second recrystallization is performed to afford additional 5-(furan-3-yl)pyrimidine (total yield of both crops is 7.60 g, 83% yield) as a light tan crystalline solid (Notes 8 and 9).

Figure 6. Product collected as light tan crystalline solid (Photo provided by Submitters)

Notes

1. Heat resistant gloves should be worn to prevent burns.
2. Submitters observed bubbling upon flame-drying likely due to water content. Checkers used anhydrous base and did not observe bubbling.
3. Potassium phosphate tribasic (anhydrous) was purchased from Alfa-Aesar. 5-Bromopyrimidine (98%) and 3-furanylboronic acid (96%) were purchased from Combi-Blocks, Inc. and used as received. Bis(tricyclohexylphosphine)nickel(II) chloride (97%) and *tert*-amyl alcohol (ReagentPlus, 99%) were purchased from Sigma-Aldrich and used as received.
4. The reaction mixture was stirred at 900 rpm.
5. Temperature was monitored by a thermocouple placed directly in the oil bath.
6. Submitters recommend an extra quantity of EtOAc can be added to aid dissolution, if necessary. Checkers did not use any EtOAc during recrystallizations.
7. Hot filtration using a 150 mL fritted funnel can be performed to remove any brown residue, if needed. Checkers performed recrystallization with and without hot filtration, and no difference in purity was observed. Procedure for hot filtration: Glassware had been heated in the

oven at 200 °C for ca. 30 min and was used within five min of removal from the oven. The hot hexanes solution (80 °C) is filtered through a glass-fritted funnel into an Erlenmeyer flask. If room-temperature glassware is used for this procedure, the product precipitates.

8. A second run was performed at half scale, which provided 3.75 g (82%) of the product.

9. This compound exhibits the following physiochemical properties: mp = 80–81 °C; TLC (hexanes:EtOAc = 3:1), R_f = 0.31; ^{1}H NMR (400 MHz, CDCl$_3$) δ: 6.73 (dd, J = 1.9, 0.9 Hz, 1H), 7.57 (t, J = 1.7 Hz, 1H), 7.84 (dd, J = 1.5, 0.9 Hz, 1H), 8.85 (s, 2H), 9.12 (s, 1H); ^{13}C NMR (125 MHz, CDCl$_3$) δ: 108.1, 120.0, 126.7, 139.5, 144.7, 153.7, 157.2; IR (film): 3136, 3112, 3041, 1969, 1957, 1880, 1607, 1575, 1549, 1513, 1438, 1414, 1340, 1187, 1159, 1091, 1061, 1012, 924, 871, 790, 732, 721, 645, 634; HRMS-ESI (m/z) cacld for C$_8$H$_7$N$_2$O [M+H]$^+$: 147.0553, found: 147.0547; Anal. Calcd for C$_8$H$_6$N$_2$O: C 65.75, H 4.14, N 19.17, found: C 65.65, H 4.25, N 19.01.

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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

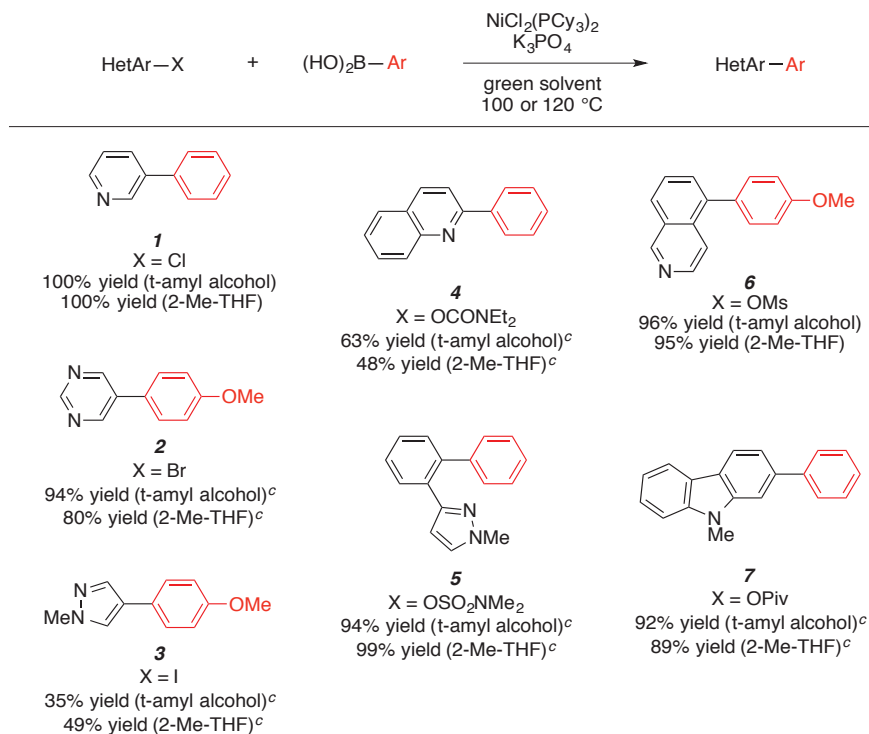and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

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.

Discussion

Transition metal-catalyzed cross-couplings are versatile methods for carbon–carbon (C–C) and carbon–heteroatom bond formation.[2] Traditionally, palladium catalysts have been most commonly utilized for this type of transformation; however, there has been a significant interest in the development of cross-coupling reactions that rely on non-precious metal catalysts. In particular, nickel has emerged as an alternative to palladium due to several factors. For example, nickel is significantly more abundant, much less expensive, and also possesses a much lower CO_2 footprint.[3] Moreover, nickel catalysts possess the unique ability to activate a wide range of electrophilic coupling partners, well beyond the scope of traditional cross-couplings that use palladium catalysis.[3]

Our research groups and many others have developed new protocols for aryl C–C and C–X bond formations using nickel catalysts.[4,5,6,7] In our efforts to render our methodologies more industrially friendly, we have developed the nickel-catalyzed Suzuki–Miyaura coupling in 'green' solvents,[8,9] such as 2-Me-THF and *tert*-amyl alcohol.[10] The methodology proceeds under mild conditions and has a broad scope with respect to the boronic acids, the electrophiles (halides and phenolic derivatives), and heterocycle constituents.[4a]

A variety of heterocyclic halides (i.e., chlorides, bromides, iodides) and phenol-derived electrophiles (i.e., carbamates, sulfamates, mesylates, and pivalates) were coupled with aryl boronic acids to give coupled products (**1–7**, Table 1). These results also demonstrate that heterocycles such as pyridine, pyrimidine, pyrazole, quinolone, isoquinoline, and carbazole are tolerated under these coupling conditions.
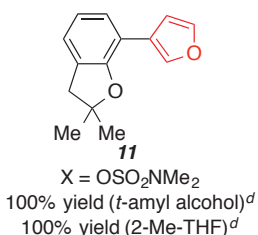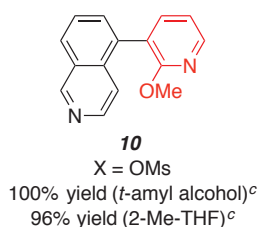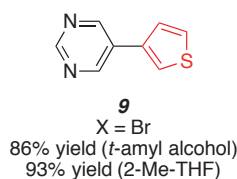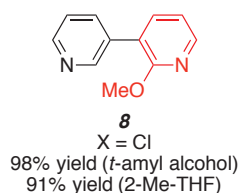
Table 1. Coupling of heterocyclic substrates with aryl boronic acids.[a,b]

$$\text{HetAr—X} \quad + \quad \text{(HO)}_2\text{B—Ar} \quad \xrightarrow[\substack{\text{green solvent}\\100 \text{ or } 120\ °\text{C}}]{\substack{\text{NiCl}_2(\text{PCy}_3)_2\\\text{K}_3\text{PO}_4}} \quad \text{HetAr—Ar}$$

1
X = Cl
100% yield (t-amyl alcohol)
100% yield (2-Me-THF)

4
X = OCONEt$_2$
63% yield (t-amyl alcohol)[c]
48% yield (2-Me-THF)[c]

6
X = OMs
96% yield (t-amyl alcohol)
95% yield (2-Me-THF)

2
X = Br
94% yield (t-amyl alcohol)[c]
80% yield (2-Me-THF)[c]

5
X = OSO$_2$NMe$_2$
94% yield (t-amyl alcohol)[c]
99% yield (2-Me-THF)[c]

7
X = OPiv
92% yield (t-amyl alcohol)[c]
89% yield (2-Me-THF)[c]

3
X = I
35% yield (t-amyl alcohol)[c]
49% yield (2-Me-THF)[c]

[a] Conditions: NiCl$_2$(PCy$_3$)$_2$ complex (5 mol %), halide substrate (1.00 equiv), aryl boronic acid (2.50 equiv), K$_3$PO$_4$ (4.50 equiv), hexamethylbenzene (0.10 equiv), 100 or 120 °C, 12 h. [b] Yield of product determined by ^{1}H NMR analysis of the crude reaction mixtures using hexamethylbenzene as an internal standard. [c] Conditions: NiCl$_2$(PCy$_3$)$_2$ complex (10 mol %), halide substrate (1.00 equiv), aryl boronic acid (4.00 equiv), K$_3$PO$_4$ (7.20 equiv), hexamethylbenzene (0.10 equiv), 100 or 120 °C, 12 h.

The methodology could also be applied to access bis(heterocyclic) frameworks, which are prevalent motifs in numerous bioactive molecules, but are sometimes difficult to access via palladium-catalyzed methods.[11] Pyridine- and pyrimidine-containing substrates were coupled with heterocyclic boronic acids to give bis(heterocycles) **8** and **9** in good to excellent yields with 1 mol% of the Ni precatalyst (Table 2). Isoquinoline- and dihydrobenzofuran-containing substrates were coupled with heterocyclic boronic acids to forge bis(heterocycles) **10** and **11** in excellent yields using 5 mol% Ni precatalyst. It should be noted that the couplings

shown in Tables 1 and 2 could be achieved in the presence of 2-Me-THF or *tert*-amyl alcohol as the reaction medium.

Table 2. Couplings of heterocyclic boronic acids
with heterocyclic substrates. [a,b]

$$\text{HetAr}-\text{X} \quad + \quad (\text{HO})_2\text{B}-\text{Ar} \quad \xrightarrow[\substack{\text{green solvent} \\ \text{100 or 120 °C}}]{\substack{\text{NiCl}_2(\text{PCy}_3)_2 \\ \text{K}_3\text{PO}_4}} \quad \text{HetAr}-\text{Ar}$$

8
X = Cl
98% yield (*t*-amyl alcohol)
91% yield (2-Me-THF)

9
X = Br
86% yield (*t*-amyl alcohol)
93% yield (2-Me-THF)

10
X = OMs
100% yield (*t*-amyl alcohol)[c]
96% yield (2-Me-THF)[c]

11
X = OSO$_2$NMe$_2$
100% yield (*t*-amyl alcohol)[d]
100% yield (2-Me-THF)[d]
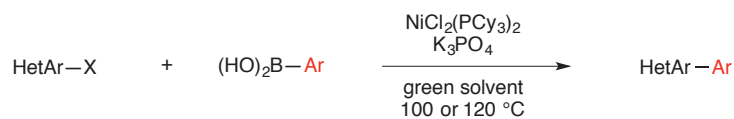
[a] Conditions: NiCl$_2$(PCy$_3$)$_2$ complex (1 mol %), halide substrate (1.00 equiv), aryl boronic acid (2.50 equiv), K$_3$PO$_4$ (4.50 equiv), hexamethylbenzene (0.10 equiv), 100 or 120 °C, 12 h. [b] Yield of product determined by ^{1}H NMR analysis of the crude reaction mixtures using hexamethylbenzene as an internal standard. [c] Conditions: NiCl$_2$(PCy$_3$)$_2$ complex (5 mol %), halide substrate (1.00 equiv), aryl boronic acid (4.00 equiv), K$_3$PO$_4$ (7.20 equiv), hexamethylbenzene (0.10 equiv), 100 or 120 °C, 12 h. [d] Conditions: NiCl$_2$(PCy$_3$)$_2$ complex (10 mol %), halide substrate (1.00 equiv), aryl boronic acid (4.00 equiv), K$_3$PO$_4$ (7.20 equiv), hexamethylbenzene (0.10 equiv), 100 or 120 °C, 12 h.

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   http://www.acs.org/content/dam/acsorg/greenchemistry/industriai nnovation/roundtable/acs-gci-pr-solvent-selection-guide.pdf

10. *tert*-Amyl alcohol and 2-Me-THF were selected in consultation with the ACS Green Chemistry Institute. *tert*-Amyl alcohol is attractive due to its safety profile, low freezing point (in comparison to *tert*-butanol), and ability to solubilize polar compounds. 2-Me-THF is advantageous because it is obtained from renewable feedstocks and possesses many process chemistry-related over THF. For a discussion of 2-Me-THF, see: Aycock, D. F. *Org. Process Res. Dev*. **2007**, *11*, 156–159.

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Appendix
Chemical Abstracts Nomenclature (Registry Number)

5-Bromopyrimidine; (4595-59-9)
3-Furanylboronic acid; (55552-70-0)
Potassium phosphate tribasic; (7778-53-2)
Bis(tricyclohexylphosphine)nickel(II) chloride; (19999-87-2)
tert-Amyl alcohol; (75-85-4)

Liana Hie was born and raised in Jakarta, Indonesia. She received her B. S. in chemistry from the University of California, Davis (UCD) in 2011. At UCD, she performed research in the laboratory of Prof. Xi Chen. She began her doctoral studies at the University of California, Los Angeles in 2011 under the supervision of Prof. Neil K. Garg. Her research focuses on the development of nickel-catalyzed cross-coupling reactions.
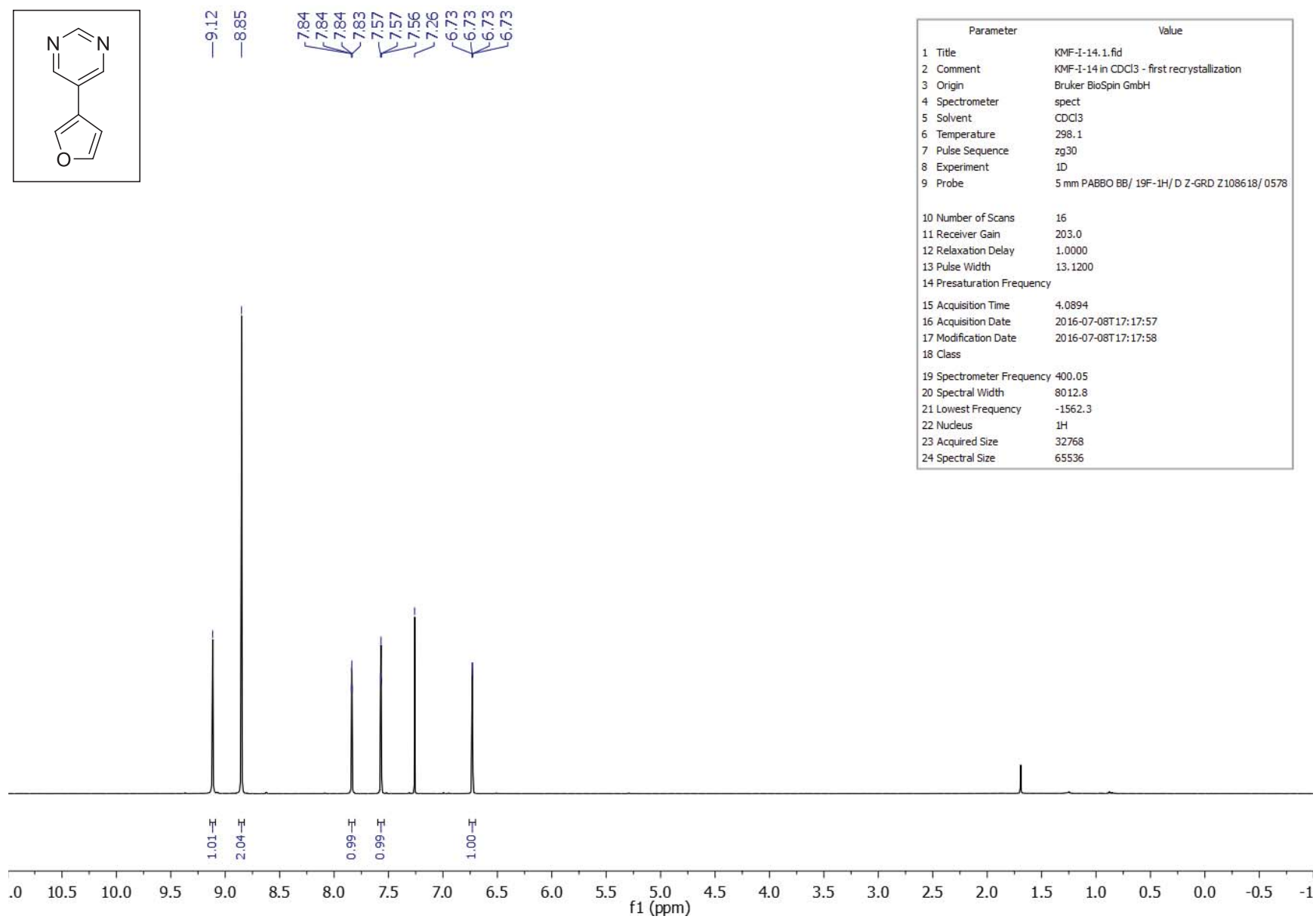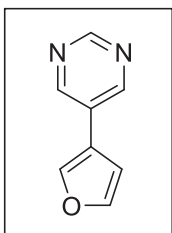
Neil Garg is a Professor of Chemistry at the University of California, Los Angeles. His laboratory develops novel synthetic strategies and methodologies to enable the total synthesis of complex bioactive molecules.

Kristen Flynn was born and raised in New Jersey. She obtained her B.S. in chemistry from Monmouth University in 2016. During her time there, she performed research under the guidance of Prof. Massimiliano Lamberto. She has a research appointment in the Movassaghi group at MIT and will begin her Ph.D. studies at in the fall.

Alyssa Antropow completed her B.S./M.S. in chemistry at Stevens Institute of Technology in 2013. As an undergraduate, she conducted research in the laboratory of Prof. A.K. Ganguly. In 2013, Alyssa began her graduate studies at MIT and joined the laboratory of Prof. Mohammad Movassaghi. Her research is focused on the total synthesis of alkaloid natural products.

Peaks: 9.12, 8.85, 7.84, 7.84, 7.84, 7.83, 7.57, 7.57, 7.56, 7.26, 6.73, 6.73, 6.73, 6.73

Parameter	Value
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8 Experiment	1D
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17 Modification Date	2016-07-08T17:17:58
18 Class	
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20 Spectral Width	8012.8
21 Lowest Frequency	-1562.3
22 Nucleus	1H
23 Acquired Size	32768
24 Spectral Size	65536

Integrations: 1.01, 2.04, 0.99, 0.99, 1.00

f1 (ppm)

— 157.2
— 153.7
— 144.7
— 139.5
— 126.7
— 120.0
— 108.1
— 77.4
— 77.2
— 76.9

Parameter	Value
1 Title	recrystallized-13C
2 Comment	STANDARD CARBON PARAMETERS
3 Origin	Varian
4 Solvent	CDCl3
5 Temperature	20.0
6 Pulse Sequence	s2pul
7 Experiment	1D
8 Probe	sw
9 Number of Scans	256
10 Receiver Gain	30
11 Relaxation Delay	1.0000
12 Pulse Width	4.5000
13 Presaturation Frequency	
14 Acquisition Time	1.3005
15 Acquisition Date	2016-07-08T11:36:53
16 Modification Date	2016-07-08T11:36:25
17 Class	
18 Spectrometer Frequency	125.80
19 Spectral Width	31446.5
20 Lowest Frequency	-1934.1
21 Nucleus	13C
22 Acquired Size	40896
23 Spectral Size	131072

f1 (ppm)