A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses

Indium-Catalyzed Heteroaryl–Heteroaryl Bond Formation through Nucleophilic Aromatic Substitution: Preparation of 2-Methyl-3-(thien-2-yl)-1*H*-indole

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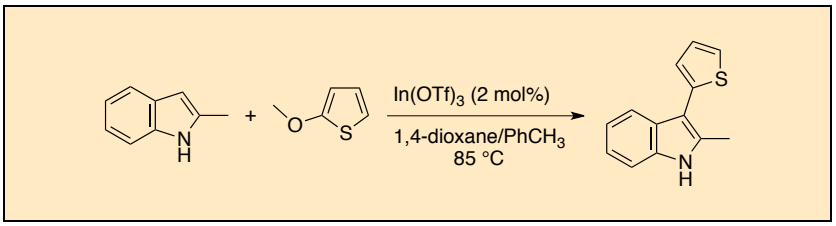

Procedure

2-Methyl-3-(thien-2-yl)-1H-indole. A 100-mL Schlenk tube equipped with a Teflon-coated, egg-shaped magnetic stir bar (9.5 x 19 mm) (Note 1) is charged with indium(III) trifluoromethanesulfonate (360 mg, 0.64 mmol, 2 mol %) (Note 2). The tube fitted with a glass stopper is slowly heated to 150 ºC over a period of 1 h by an oil bath with stirring under vacuum (0.50 mmHg) (Note 3). After heating at 150 °C for an additional 1 h, the tube is cooled down to room temperature and filled with nitrogen (Note 4). The glass stopper is replaced with a rubber septum under a flow of nitrogen, and the tube is evacuated and refilled with nitrogen three times. Dry 1,4-dioxane (16 mL) (Note 5) and dry toluene (1.3 mL) (Note 6) are added to the tube by syringes, and the resulting mixture is stirred at room temperature for approximately 10 min until the indium salt is dissolved. 2-Methyl-1*H*-indole (4.20 g, 32.0 mmol, 1.0 equiv) (Note 7) is added under a flow of

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nitrogen by temporarily removing the septum. 2-Methoxythiophene (4.02 g, 35.2 mmol, 1.1 equiv) (Note 7) is added by syringe, and the septum is replaced with the glass stopper. The reaction mixture is stirred at an oil bath temperature of 85 ºC for 5 h (Note 8). After cooling to room temperature, a saturated aqueous solution of $NaHCO_3$ (20 mL) is added to the mixture, which is then stirred for 5 min to form a white solid. The resulting mixture is filtered through a glass funnel with a cotton plug to remove the solid, which is then washed with EtOAc (10 mL). The filtrate is transferred to a 250-mL separatory funnel. The organic layer is retained, and the separated aqueous layer is extracted with EtOAc (3 x 30 mL). The combined organic layers are washed with brine (20 mL) and dried over anhydrous sodium sulfate. After filtration through a glass funnel with a cotton plug to remove the drying agent that is washed with EtOAc (10 mL), the solvent is removed by rotary evaporation (50 mmHg, 40 ºC water bath) and then under oil pump vacuum (0.50 mmHg, room temperature) for 20 min (Note 9). The crude product is re-dissolved in chloroform (30 mL) (Note 10), and silica gel (16 g) (Note 11) is added to the solution. Chloroform is removed by rotary evaporation (50 mmHg, 40 ºC water bath), and the mixture is further dried under oil pump vacuum (0.50 mmHg, room temperature) for 15 min (Note 12) to obtain a free-flowing powder, which is placed at the top of silica gel (310 g) pre-eluted with 600 mL of 25% Et_2O in hexanes in a column (7.5 cm diameter). After covering the surface of the silica gel with sand (Note 13), elution with 2.4 L of 25% Et_2O in hexanes gave fractions containing the desired product, which can be visualized on thin-layer chromatography (TLC) (Note 14). The fractions are combined, concentrated by rotary evaporation (50 mmHg, 40 ºC water bath), and then dried under oil pump vacuum (0.50 mmHg, room temperature) (Note 15) for 2 h to afford 2-methyl-3-(thien-2-yl)-1*H*-indole (5.66–5.80 g, 83–85% yield) as a white crystalline solid (Note 16).

Notes

1. The submitters used octagon-shaped magnetic stir bar (1.5 cm x 6.5 mm).
2. Indium(III) trifluoromethanesulfonate [$In(OTf)_3$, Tf = SO_2CF_3] was purchased from Strem Chemical, Inc., and should be stored in a

desiccator due to its highly hygroscopic nature. The submitters purchased indium (III) trifluoromethanesulfonate from Sigma Aldrich.

3. The submitters heated indium (III) trifluoromethanesulfonate at 150 °C under vacuum (0.083 mmHg).
4. The submitters conducted the reaction under the atmosphere of argon.
5. 1,4-Dioxane (CHROMASOLV® Plus, for HPLC, ≥99.5%) was purchased from Sigma Aldrich and was used as received without further purification.
6. Toluene was dried using a solvent purification system manufactured by SG Water U.S.A., LLC. The submitters purchased toluene (99.5+%) from Wako Pure Chemical Industries, Ltd. and distilled under argon from calcium chloride just prior to use.
7. 2-Methyl-1*H*-indole (>99.0%) and 2-methoxythiophene (>98.0%) were purchased from Tokyo Chemical Industry Co., Ltd. and used as received.
8. The progress of the reaction can be monitored by thin-layer chromatography (TLC) using Silicycle glass-backed extra hard layer, 60 Å plates (indicator F-254, 250 μm). A solution of 25% Et_2O in hexanes was used as the development solvent and *p*-anisaldehyde stain was used to visualized the spots of both reactants (2-methyl-1*H*-indole: R_f = 0.35; 2-methoxythiophene: R_f = 0.70) and products (R_f = 0.30). The submitters monitored the reaction progress by gas chromatography (GC). Complete conversion of 2-methoxythiophene was observed at the reaction time of 5 h. GC analyses were performed on a Shimadzu model GC-2014 instrument equipped with a capillary column of InertCap 5 (5% phenyl polysilphenylene-siloxane, 30 m x 0.25 mm x 0.25 μm) using nitrogen as carrier gas (1.78 mL/min) and measured under the following conditions: injector temperature: 280 °C; detector temperature: 300 °C; initial temperature: 50 °C for 2 min; rate of temperature rise: 30 °C/min; final temperature: 280 °C for 21 min. The retention times of the reagents and the product were as follows: 2.06 min for 2-methoxythiophene; 7.66 min for 2-methyl-1*H*-indole; 11.17 min for 2-methyl-3-(thien-2-yl)-1*H*-indole.
9. According to submitter's procedure, solvent was removed by rotary evaporator (1.5 mmHg, 30 °C) and then under oil pump (0.38 mmHg, room temperature) for 2 h.
10. Chloroform (Approx. 0.75% EtOH, HPLC) was purchased from Fisher and was used as received. The submitters purchased chloroform (>99.0%) from Junsei Chemical Co., Ltd. which was used as received.

11. Column Chromatography was performed with the indicated solvents using Silicycle SiliaFlash® P60 (230–400 mesh) silica gel. The submitters purchased Silica gel (Silica gel 60N, spherical neutral, particle size 40–50 μm) from Kanto Chemical Co., Inc. and used as received.
12. According to the submitter's procedure, chloroform was removed by rotary evaporator (1.5 mmHg, 30 °C) and then under oil pump (0.38 mmHg, room temperature) for 1 h.
13. The submitters covered the silica gel with anhydrous sodium sulfate.
14. TLC plates were eluted with 25% Et_2O in hexanes, and a spot of the desired product (R_f = 0.30) was visualized with 254 nm UV light and by staining with *p*-anisaldehyde stain. Fractions were collected in 25 x 200 mm Pyrex glass test tubes. According to submitter's procedure, TLC (TLC silica gel 60G F254) was purchased from Merck KGaA. A phosphomolybdic acid/ethanol solution was used as the TLC stain and desired product spot has an R_f value of 0.32. The desired product was obtained in fractions 20–39 (1.3 L).
15. According to submitter's procedure, combined fractions were concentrated by rotary evaporator (1.5 mmHg, 30 °C) and then dried under oil pump (0.38 mmHg, room temperature) for 2 h.
16. 2-Methyl-3-(thien-2-yl)-1*H*-indole has the following physical and spectroscopic properties: mp 80–81.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.59 (s, 3 H), 7.15–7.21 (m, 4 H), 7.31–7.33 (m, 2 H), 7.84 (d, *J* = 10.0 Hz, 1 H), 7.96 (bs, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 13.2, 108.0, 110.4, 119.2, 120.4, 122.0, 123.5, 124.6, 127.4, 127.9, 132.5, 135.1, 137.4; IR (neat): 3395, 3051, 2923, 2853, 1725, 1561, 1455, 1446, 743 cm^{-1}; HRMS (ESI) Calcd for $C_{13}H_{10}NS$: [M-H], 212.0534. Found: *m*/*z* 212.0557; Anal. Calcd. for $C_{13}H_{11}NS$: C, 73.20; H, 5.20; N, 6.57; S, 15.03. Found: C, 73.10; H, 5.25; N, 6.52; S, 15.09.

Handling and Disposal of Hazardous Chemicals

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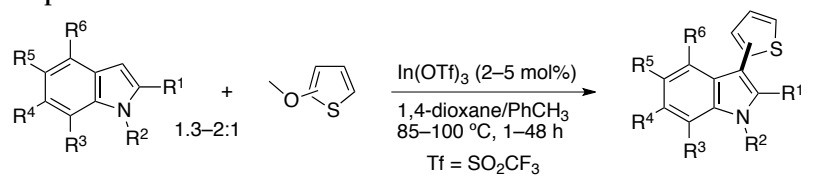
Discussion

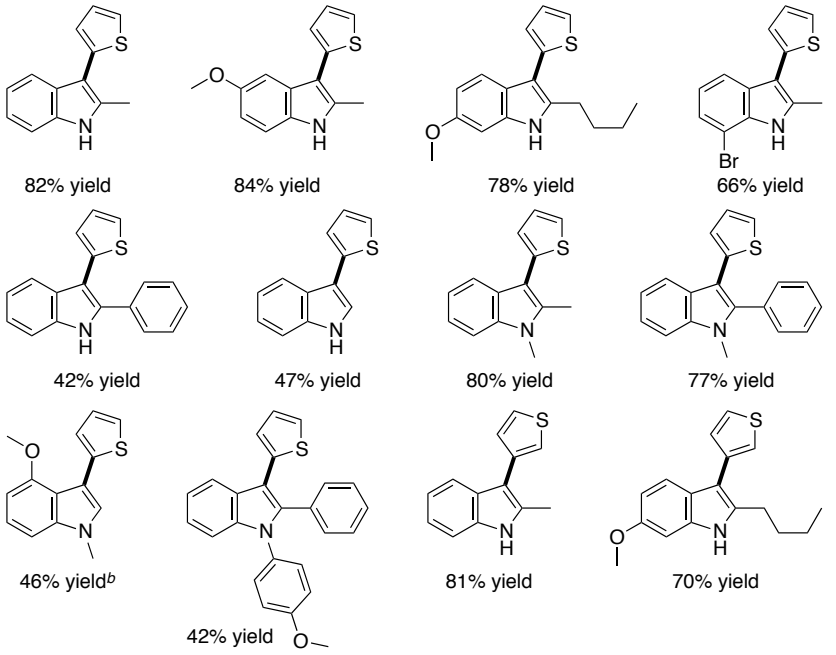
A heteroaryl–heteroaryl unit constitutes a core structure in many important organic molecules such as optoelectronic materials,[2] liquid crystals,[3] biologically active compounds,[4] and ligands for asymmetric catalysis.[5] Over the past 35 years, transition metal-catalyzed cross-coupling reactions have contributed mainly to constructing (hetero)aryl–(hetero)aryl bonds.[6] On the other hand, nucleophilic aromatic substitution (S_NAr) has actually been studied to make such biaryl units since the 1940s.[7] However, in order to synthesize biaryls by the S_NAr reaction, two aryl substrates with entirely opposite electronic demands must be arranged. Thus, electron-rich aryl nucleophiles with highly electropositive metals (e.g. Li^+, Mg^{2+}, Zn^{2+}) and/or electron-poor aryl electrophiles with one or more strong electron-withdrawing groups (EWGs; e.g. CF_3, NO_2, CN, CO_2R) are necessary for each substrate.[8] In addition, more than a stoichiometric amount of promoter is often required.[8c,p,q,r] In sharp contrast to such conventional approaches, we have achieved, for the first time, S_NAr-based catalytic heteroaryl–heteroaryl bond formation without using both the heteroarylmetal nucleophile and EWGs-substituted heteroaryl electrophile.[9]

Synthesis of 2-methyl-3-(thien-2-yl)-1*H*-indole on a 0.25 mmol scale[9] was carried out with 2-methyl-1*H*-indole and 2-methoxythiophene in a 1.3:1 molar ratio. The desired product could be isolated as a pure form by column chromatography on silica gel, but not from a reaction mixture performed on a large scale over 30 mmol, where the desired product contaminated with 2-methyl-1*H*-indole was obtained. In order to obtain the pure product in such a large-scale synthesis, the use of a slight excess molar amount of not 2-methyl-1*H*-indole but 2-methoxythiophene is important.

The indium-catalyzed S_NAr reaction is applicable to a range of indoles with OMe, alkyl, Br, Ph, and/or *p*-$MeOC_6H_4$ groups as shown in Table 1,

Table 1. Indium-catalyzed S_NAr reaction of indoles with 2- or 3-methoxythiophene[a]





[a] Yields of isolated products are shown here. Futher details on reaction conditions for each reaction are provided in Supporting Information of reference 9. [b] $In(ONf)_3$ (Nf = $SO_2C_4F_9$; 10 mol%) instead of $In(OTf)_3$ was used.

which shows results of reactions performed on a 0.25 or 0.40 mmol scale. It should be noted that the OMe groups on the indole substrates does not participate in the substitution reaction, thus indicating remarkable chemoselectivities. 3-Methoxythiophene also reacted with indoles to give the corresponding thienylindoles. Results of reactions using other substrates,[9] for instance, pyrroles as nucleophiles and 2,5-dimethoxythiophene, 5,5′-dimethoxy-2,2′-bithiophene, 2-pyridyl triflate and 3-acetoxyindole as electrophiles are presented. Possible reaction mechanisms are also discussed.[9]

As disclosed above, transition-metal-catalyzed cross-coupling reaction is, in general, an effective tool to connect two heteroaryl molecules. However, the cross-coupling by using indolylmetals seems to be impractical because pre-synthesis of indolylmetals requires multi-steps.[6a,10] Accordingly, our strategy will be helpful to prepare such type of heteroaryl compounds.

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1. Department of Applied Chemistry, School of Science and Technology, Meiji University, Higashimita, Tama-ku, Kawasaki 214-8571, Japan. E-mail: tsuchimo@isc.meiji.ac.jp. Financial support by a Grant-in-Aid for Scientific Research (No. 19750083) from the Ministry of Education, Culture, Sports, Science and Technology is highly acknowledged. Y.N. thanks the JSPS Research Fellowship for Young Scientist.
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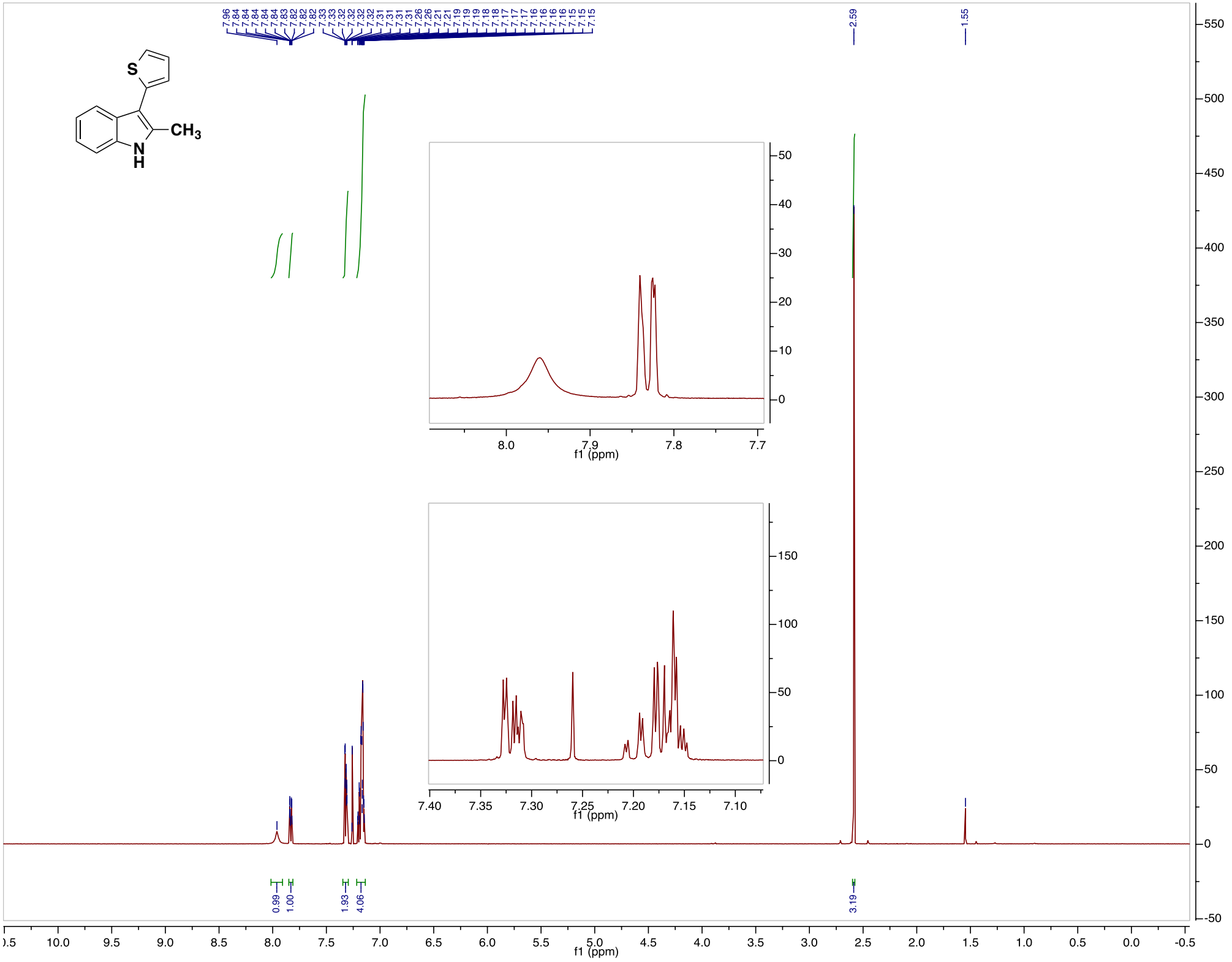
Appendix
Chemical Abstracts Nomenclature (Registry Number)

2-Methyl-1*H*-indole; (95-20-5)
2-Methoxythiophene; (16839-97-7)
Indium(III) trifluoromethanesulfonate; (128008-30-0)
1,4-Dioxane; (123-91-1)
2-Methyl-3-(thien-2-yl)-1*H*-indole; (1159415-23-2)

Yuta Nagase was born in 1985 in Ishikawa, Japan. He received his B.Eng. degree in 2008 and his M.Eng. degree in 2010 from Meiji University. Since 2010, he has started his Ph.D. study under supervision of Associate Professor Teruhisa Tsuchimoto. He has been a JSPS research fellow since 2012. His research interests focus on development of new Lewis acid-catalyzed reaction with indoles as nucleophiles.

Teruhisa Tsuchimoto was born in 1970 in Gifu, Japan. He received his Ph.D. degree in 1997 from Tokyo Institute of Technology under the supervision of Professor Tamejiro Hiyama. After working with Professor Peter Wipf at University of Pittsburgh as a postdoctoral research fellow, he returned to Japan in 1998 to join Professor Shirakawa's group at JAIST as an Assistant Professor. In 2006, he moved to Meiji University as an Associate Professor. He is a recipient of the Incentive Award in Synthetic Organic Chemistry, Japan in 2009. His research interests cover development of new synthetic methodologies based on Lewis acid-catalyzed activation of hydrocarbon functional groups, and their application to synthesis of novel optoelectronic materials.

Joyce Leung was born and raised in Hong Kong. She received her B.S. in Chemical Biology from University of California, Berkeley in 2007. Then she worked as a Research Associate at Nanosyn, Inc. for a year, and began her doctoral studies at University of Texas at Austin in 2008. She obtained her Ph.D. in 2013 under the supervision of Professor Michael J. Krische. Her doctoral research focused on transition metal catalyzed carbon-carbon bond formation method development. She is currently conducting postdoctoral research on natural product synthesis under the supervision of Professor John L. Wood at Baylor University.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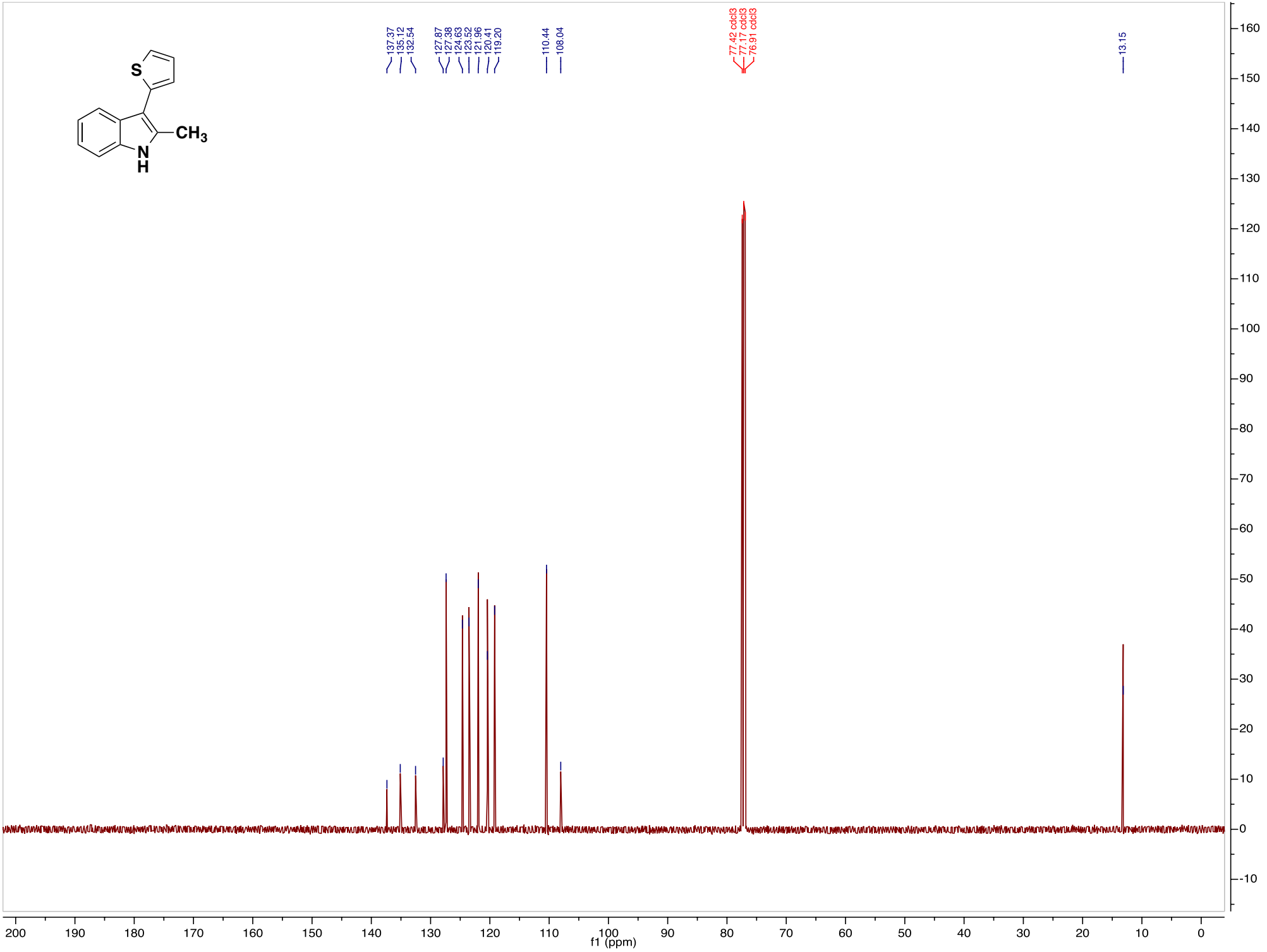
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