Supporting information

# 1. General Remarks

• JEOL JNM-ECA 500 or ECX 600 spectrometers were used for NMR measurement using CDCl<sub>3</sub> as solvents unless otherwise noted. Tetramethylsilane was used as an internal standard for <sup>1</sup>H NMR ( $\delta = 0$  ppm), and CDCl<sub>3</sub> was used for <sup>13</sup>C NMR ( $\delta = 77.00$  ppm). Structures of known compounds were confirmed by comparing them with the data shown in the literature.

• ICP-AES analysis was performed on a Shimadzu ICPS-7510 equipment.

• STEM/EDS images were obtained using a JEOL JEM-2100F instrument operated at 200kV. All STEM specimens were prepared by placing a catalyst directly on carbon-coated copper grids.

- Nitrogen adsorption/desorption isotherm was recorded on a BELSORP-mini Microtrac Bell.
- IR spectra were measured using JASCO FT/IR-610 spectrometer.
- Centrifugation was carried out using Kokusan H-36α.
- Preparative thin-layer chromatography was carried out using Wakogel B-5F.
- CARiACT-Q10 was purchased from Fuji Silysia Chemical Ltd.
- *N*-(3-triethoxysilylpropyl)-4,5-dihydroimidazole and heteropoly acids were purchased from Oakwood Products, Inc. and Wako Pure Chemical Company.
- Cp\*H, Cp<sup>4Me</sup>H, Cp\*'<sup>Bu</sup>H, and Ind\*H were prepared by following the literature. <sup>[1-5]</sup>
- [Cp\*RhCl<sub>2</sub>]<sub>2</sub>, [Cp<sup>4Me</sup>RhCl<sub>2</sub>]<sub>2</sub>, [Cp\*'<sup>Bu</sup>RhCl<sub>2</sub>]<sub>2</sub>, and [Ind\*RhCl<sub>2</sub>]<sub>2</sub> were prepared by following the literature.<sup>[6-8]</sup>
- 2-phenylpyridine was purchased from Thermo Fisher Scientific Inc. and purified by distillation.
- Other substrates including 2-phenylpyridine derivatives, 2-phenylquinoline, 1-phenylisoquinoline and ethyl benzimidate were prepared by following the literature. <sup>[9-12]</sup>
- 3-phenyl-1,4,2-dioxazolone and its derivatives were prepared by following the literature.<sup>[13]</sup>
- Ethyl acetate, butyl acetate and isoamyl acetate for reaction were purified by distillation.
- All commercially available reagents, unless otherwise stated, were used without further purification.
- All reactions, unless otherwise stated, were carried out under argon atmosphere.

• For apparatuses for flow systems, a HPLC pump (Shimadzu LC-20AB) and each size stainless column with column ends were used. For heating the column, a continuous-flow reaction equipment (EYELA FlowMaster CCR-1000G) was used.

## 2. Experimental procedure

# 2.1. Preparation of Cp\*<sup>CF3</sup>H and [Cp\*<sup>CF3</sup>RhCl<sub>2</sub>]<sub>2</sub>

(a) Preparation of Cp\*CF<sub>3</sub>H



Scheme S1. Preparation of Cp\*CF3H and [Cp\*CF3RhCl2]2

# 2.1. (a) Preparation of Cp\*<sup>CF3</sup>H (Scheme S1a)<sup>[14]</sup>

A 100 mL 2-necked round bottom flask equipped with a magnetic stirring bar, a reflux condenser and a dropping funnel was charged with lithium shot (40 mmol, 277.6 mg) and dry diethyl ether (2 mL). An initial portion of 2-bromo-2-butene (8.9 mmol, 1.203 g, mixture of isomers) was passed through short column with basic Al<sub>2</sub>O<sub>3</sub> and added to the stirred solution dropwise over the course of several minutes. Additional dry diethyl ether (15 mL) was added, and additional Al<sub>2</sub>O<sub>3</sub>-treated 2-bromo-2-butene (11.6 mmol, 1.564 g) was added slowly in dropwise manner. After the completion of the addition, the mixture was stirred for 1 h at room temperature. Then the mixture was cooled to 0 °C in an ice bath and ethyl trifluoroacetate (10 mmol, 1.421 g) diluted in dry diethyl ether was added dropwise. After the completion of the addition, the mixture was warmed to room temperature and stirred for another 1 h. Saturated aqueous NH<sub>4</sub>Cl (30 mL) was added to reaction mixture, and it was extracted with diethyl ether (20 mL\*5). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was removed to obtain yellow oil containing the alcohol. Under an inert atmosphere the yellow oil was added via syringe to a solution of trifluoromethanesulfonic acid (ca. 1.9 mL, 15 mmol) in diethyl ether (20 mL). The mixture was refluxed for 8 h, and then quenched saturated aqueous NaHCO<sub>3</sub> (30 mL). The organic layer was separated, and the aqueous layer was extracted with diethyl ether (30 mL\*4). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was removed. The crude products were purified by silica gel chromatography (hexane: $Et_3N = 200:1$ ) to afford  $Cp^{*CF3}H$  and its isomers as pale-yellow oil (532.3 mg, 28% overall yield). The product was used in the next step without further analysis.

## 2.1. (b) Preparation of [Cp\*<sup>CF3</sup>RhCl<sub>2</sub>]<sub>2</sub> (Scheme S1b)<sup>[14]</sup>

A 6 mL vial was charged with RhCl<sub>3</sub>•3H<sub>2</sub>O (0.19 mmol, 50.0 mg),  $Cp^{*CF3}H$  (0.57 mmol, 108.4 mg) and MeOH (1.0 mL). The vial was sealed with screw cap, and the mixture was heated at 75 °C for 64 h. Then the

mixture was cooled to room temperature and methanol was removed under reduced pressure to give crude mixture. The crude mixture was purified by silica gel chromatography (DCM:MeOH = 9:1) to afford  $[Cp^{*CF3}RhCl_2]_2$  as red solid (42.4 mg, 62% yield).

#### 2.2. Preparation of substrates

(a) Synthesis of 2-phenylpyridine derivatives





Scheme S2. Preparation of substrates.

## 2.2. (a) 2-phenylpyridine derivatives (Scheme S2a)<sup>[9]</sup>

To a 100 mL round bottom flask equipped with a reflux condenser and a magnetic stirring bar was added aryl boronic acid (7.8 mmol) and sodium carbonate (4.71 g, 44.4 mmol). Under inert atmosphere,  $Pd(PPh_3)_4$  (208.0 mg, 0.18 mmol) was subsequently added to the flask. Toluene (21 mL), ethanol (4.6 mL), water (21 mL) was mixed in another 100 mL flask, then bubbled with argon for 30 minutes to remove dissolving oxygen. The deoxygenated solvent was poured into the mixture of solid materials, followed by addition of 2-bromopyridine (948.0 mg, 6.0 mmol). Then, the mixture was heated at 110 °C for 24 h. After cooling to room temperature, saturated aqueous solution of ammonium chloride (40 mL) was poured to the mixture. The organic layer was separated, and the water layer was extracted with ethyl acetate (40 mL\*3). Combined organic layer was dried over anhydrous sodium sulfate, then filtered and evaporated. The crude material was purified by silica gel chromatography (hexane:ethyl acetate = 9:1).

## 2.2. (b) 2-phenylquinoline and 1-phenylisoquinoline (Scheme S2b) [9, 10, 11]

A 20 mL two-necked round bottom flask equipped with a magnetic stirring bar was charged with quinoline (1.29g, 10 mmol) and acetic acid (5.0 mL). 35% aqueous hydrogen peroxide was subsequently added to the flask, then heated at 70 °C. After 24 h, excess acetic acid was removed under vacuum. The resulting oily compound was transferred to a 200 mL round bottom flask, then diluted with water (100 mL). To this mixture was added mesyl chloride (2.05 g, 18 mmol). After 10 minutes, resulting solid was collected by filtration and washed with ethyl acetate (5 mL). The product, 2-quinolone, was dried and used in next step without further purification.

A 200 mL two-necked round bottom flask equipped with a magnetic stirring bar was dried by heating under vacuum, then charged with 2-quinolone (1.45 g, 10 mmol) and anhydrous toluene (100 mL). Phosphoryl chloride (9.32 mL, 100 mmol) was added to this mixture and heated at 125 °C. After 21 h, the mixture was cooled to room temperature and quenched carefully with water. Solid sodium bicarbonate was slowly added to neutralize acid (pH > 7). The organic layer was separated, and the water layer was extracted with ethyl acetate. Combined organic layer was dried over anhydrous sodium sulfate, then filtered and evaporated. The crude material was purified by silica gel chromatography (hexane:ethyl acetate = 15:1)

For coupling reaction, overall procedure was almost the same as preparation of 2-phenylpyridine derivatives as described above. The prepared 2-chloroquinoline was used instead of 2-bromopyridine.

To synthesize 1-phenylisoquinoline, isoquinoline was used as the starting material of this procedure instead of quinoline.

## 2.2. (c) Ethyl benzimidate (Scheme S2c) <sup>[12]</sup>

A 50 mL two-necked round bottom flask equipped with a magnetic stirring bar was dried by heating under vacuum, then charged with benzonitrile (1.25 mL, 12.1 mmol) and anhydrous ethanol (8.5 mL, 145.5 mmol). To this mixture, acetyl chloride (6.92 mL, 97 mmol) was added dropwise. After completion of the addition, the mixture was stirred at room temperature for overnight. To quench the reaction, the mixture was cooled to 0 °C, and then aqueous solution of sodium bicarbonate was added until generation of  $CO_2$  ceased. The mixture was extracted with diethyl ether. The organic layer was washed with brine, dried over anhydrous sodium sulfate, filtrated, and evaporated. The crude mixture was purified by distillation.

## 2.2. (d) 3-phenyl-1,4,2-dioxazolone and its derivatives (Scheme S2d) <sup>[13]</sup>

A 200 mL round bottom flask equipped with a magnetic stirring bar was charged with ethyl acetate (60 mL) and water (30 mL). Potassium carbonate (13.82 g, 100 mmol) and hydroxylamine hydrochloride (7.00 g, 100 mmol) were added to this solvent, and the mixture was cooled to 0 °C. Corresponding acid chloride (50 mmol) was added dropwise to this mixture. After the completion of the addition, the mixture was warmed to room temperature and stirred for 2 h. Excess water was poured to the mixture until inorganic salt dissolved, and then the organic layer was separated. The water layer was extracted with ethyl acetate (50 mL\*2), and combined organic layer was dried over anhydrous sodium sulfate. After evaporation of the solvent, the crude material was recrystallized from ethyl acetate or ethyl acetate/ethanol.

A 100 mL round bottom flask equipped with a magnetic stirring bar was charged with the obtained hydroxamic acid (20 mmol) and ethyl acetate (80 mL). To this mixture, CDI (4.22 g, 26 mmol) was added in one portion and stirred for 1 h. The reaction was quenched with 1M HCl (40 mL) and the organic layer was separated. The water layer was extracted with ethyl acetate (40 mL\*2), then combined organic layer was dried over anhydrous sodium sulfate. After evaporation of the solvent, the crude material was re-dissolved to

toluene (25 mL) and passed through silica pad. Evaporation of toluene from the collected solution afforded the products.

## 2.3. Catalyst preparation<sup>[15]</sup>



Scheme S3. Preparation of immobilized Cp\*Rh(III) catalysts

## 2.3. (a) A general procedure for the functionalization of SiO<sub>2</sub> (Scheme S3a)

CARiACT Q-10 was used as typical SiO<sub>2</sub>. To a 200 mL round bottom flask equipped with a magnetic stirring bar and reflux condenser was added SiO<sub>2</sub> (3.0 g). SiO<sub>2</sub> was dried under vacuum at 100 °C for 1 h. After cooling to room temperature, the flask was charged with Ar. Anhydrous toluene (100 mL) was added to the flask followed by the addition of freshly distilled *N*-(3-triethoxysilylpropyl)-4,5-dihydroimidazole (823.3 mg, 3.0 mmol). The mixture was refluxed for 18 h. After cooling to room temperature, the solid was collected by filtration and washed with toluene (20 mL\*3) and DCM (20 mL\*2). Finally, the solid was dried under vacuum at room temperature for 3 h to get SiO<sub>2</sub>-im as a white solid (3.31 g). Aminopropyl trimethoxysilane, *N*-butyl aminopropyl triethoxysilylpropyl)-4,5-dihydroimidazolefor the preparation of SiO<sub>2</sub>-NH<sub>2</sub>, SiO<sub>2</sub>-NH<sup>n</sup>Bu, SiO<sub>2</sub>-NEt<sub>2</sub>, respectively.

## 2.3. (b) A general procedure for the salt formation with HPA (Scheme S3b)

To a 100 mL round bottom flask equipped with a magnetic stirring bar was added SiO<sub>2</sub>-im (2.0 g). EtOH (30 mL) was added to the flask followed by the addition of the solution of STA (662.1 mg, 0.2 mmol) in EtOH (20 mL). The mixture was stirred for 18 h at room temperature. The solid was collected by filtration and washed with EtOH (20 mL\*3) and DCM (20 mL\*2). Finally, the solid was dried under vacuum at room temperature for 3 h to get STA/SiO<sub>2</sub>-im as a white solid (2.60 g). Supports with other heteropoly acids were prepared by following the same procedure described above.

# **2.3.** (c) A general procedure for the immobilization of Cp\*Rh(III) complex (Scheme S3c, for batch conditions)

To a test tube equipped with a magnetic stirring bar was added STA/SiO<sub>2</sub>-im (160 mg). The solid was dried under vacuum at room temperature for 1 h. Meanwhile, to another test tube was added [Cp\*RhCl<sub>2</sub>]<sub>2</sub> (0.002

mmol, 1.2 mg) and AgNTf<sub>2</sub> (0.008 mmol, 3.1 mg). EtOAc (1 mL) was added to the tube, and the solution was stirred at room temperature for 15 min to give a yellow solution of Cp\*Rh(III) complex and precipitation of AgCl. The tube was centrifuged at 3,000 rpm for 10 min to separate the precipitation. EtOAc (1 mL) was added to the tube with STA/SiO<sub>2</sub>-im and the yellow supernatant of Cp\*Rh(III) complex via cannula. The mixture was stirred for 30 min at room temperature. The tube was centrifuged at 3,000 rpm for 10 min. After completion of centrifugation, the colorless supernatant was removed by cannula. EtOAc (1 mL) was added to the tube and stirred for 10 min at room temperature. This washing process was repeated 2 times, and after removal of the supernatant, the Cp\*Rh(III)/STA/SiO<sub>2</sub>-im was obtained as yellow solid. The catalysts were used for the following C–H amidation reaction without further treatment. If necessary, the supernatant was collected and subjected to ICP analysis in the following procedure to determine immobilization efficiency.

# 2.4. A typical procedure of C–H amidation reaction of 2-phenylpyridine with 3-phenyl-1,4,2-dioxazolone under batch conditions (Table 1)

To the test tube with Cp\*Rh(III)/STA/SiO<sub>2</sub>-im (Rh: 0.004 mmol), the solution of 3-phenyl-1,4,2-dioxazolone (0.40 mmol, 65.2 mg) in EtOAc (1 mL) was added followed by the addition of 2-phenylpyridine (0.20 mmol, 31.0 mg). The reaction mixture was stirred for 18 h at 60 °C. After completion of the reaction monitored by TLC analysis, the solid material was removed by filtration and washed with EtOAc. The filtrate was collected, and the solvent was removed. The crude material was analyzed by <sup>1</sup>H NMR using durene as internal standard. Rh leaching was measured in the following procedure.

#### 2.5. A typical procedure of measuring Rh leaching

The sample for ICP analysis was dissolved to diethyl ether and transferred to a test tube. The solvent was removed by heating. Concentrated  $H_2SO_4$  (1 mL) was added to the tube and the tube was heated to 200 °C. Concentrated HNO<sub>3</sub> (3 drops) was added to the tube and the mixture was heated for 30 min. This degradation process was repeated until the generation of NO<sub>2</sub> ceased. After the completion of degradation, the mixture was cooled to room temperature, and diluted with deionized water (50 mL). The solution was analyzed by ICP analysis equipment.

# 2.6. A typical procedure of C–H amidation reaction of 2-phenylpyridine with 3-phenyl-1,4,2-dioxazolone under continuous-flow conditions (flow-immobilization method; Figure 1, 2; Scheme 1)

A SUS column ( $\Phi 10*100$  mm) equipped with column ends with filters was used as a fixed-bed reactor. STA/Q-10-im (2.40 g) and non-modified Q-10 (1.40 g) was mixed, then dried at 80 °C under vacuum for 2 h. After the completion of drying, the mixture was packed inside the column under Ar atmosphere. The bottom of the column end was connected to a HPLC pump by PTFE tubings and fittings. The top of the column end was connected to a back pressure regulator. A test tube equipped with a magnetic stirring bar was added [Cp\*RhCl<sub>2</sub>]<sub>2</sub> (0.02 mmol, 12.4 mg) and AgNTf<sub>2</sub> (0.08 mmol, 31.0 mg). EtOAc (4.5 mL) was added to the tube, and the solution was stirred at room temperature for 15 min to give a yellow solution of Cp\*Rh(III) complex and precipitation of AgCl. The tube was centrifuged at 3,000 rpm for 10 min to separate the precipitation. After the centrifugation, the solution was diluted by adding EtOAc (3 mL). Before the immobilization of Cp\*Rh(III), EtOAc was pumped into the column at 0.3 mL/min flow rate for 1 h. Then, the solution of Cp\*Rh(III) was pumped into the column at 0.1 mL/min flow rate. After all the solution pumped into the column, the test tube was washed with EtOAc(1.5 mL) and the washing was also pumped. To substitute EtOAc, butyl acetate was pumped into the column at 0.2 mL/min flow rate. The column was then heated to 80 °C and back pressure (ca. 0.3-0.6 MPa) was applied. Into the column, a solution of 2-phenylpyridine (1.8 mmol, 279.4 mg) and 3-phenyl-1,4,2-dioxazolone (2.16 mmol, 352.4 mg) in butyl acetate (80 mL) was pumped at 0.05 mL/min flow rate. The resulting outlet solution was collected in a reservoir to give a crude solution of desired product. The reservoir was changed at certain time intervals (e.g. 12 h) to investigate change in yields over time. To determine NMR yields, a 2.0 mL of outlet solution was collected, and solvent was removed. Resulting material was analyzed by <sup>1</sup>H NMR using DMSO-*d*<sub>6</sub> as solvent and trimethoxybenzene as internal standard. To determine isolated yields, a 3.0 mL of outlet solution from the sampling solution which showed the highest NMR yield was collected, and solvent was removed. The target product was separated by pTLC (hexane:acetone = 6:1), and the yield was determined based on the mass of obtained product. To determine the amount of Rh leaching, 5.0 mL of solution was collected, and the solvent was removed. The sample was treated with H<sub>2</sub>SO<sub>4</sub>/HNO<sub>3</sub> and the amount of Rh was measured as described above.

#### 3. Characterization of catalyst

#### 3.1. N<sub>2</sub> adsorption/desorption analysis

To confirm whether the surface modification of support proceeded as intended, N<sub>2</sub> adsorption/desorption isotherms were measured for each modification steps. From the results, surface area and total pore volume was calculated (Figure S1, S2, Table S1). Values for non-modified Q-10 was obtained from Fuji Silysia Chemical Ltd. The result showed gradual decrease in both surface area and total pore volume as the modification proceeded, which indicated that each modification proceeded on the surface of the silica support.



Figure S1. Surface area of the silica composites at each modification steps.





 Table S1. Surface area of the silica composites at each modification steps.

| Sample  | Surface area $(m^2/g)$ |
|---|------------------------|
| Q-10  | 300                    |
| Q-10-H <sub>2</sub> -imidazole                | 228                    |
| STA/Q-10-H <sub>2</sub> -imidazole            | 203                    |
| Cp*Rh(III)/STA/Q-10-H <sub>2</sub> -imidazole | 206                    |

 Table S2. Total pore volume of the silica composites at each modification steps.

| Sample  | Surface area $(m^2/g)$ |
|---|------------------------|
| Q-10  | 1.0                    |
| Q-10-H <sub>2</sub> -imidazole                | 0.877                  |
| STA/Q-10-H <sub>2</sub> -imidazole            | 0.646                  |
| Cp*Rh(III)/STA/Q-10-H <sub>2</sub> -imidazole | 0.572                  |

## **3.2. STEM/EDS analysis**

In addition to  $N_2$  adsorption/desorption analysis, scanning transmission electron microscopy (STEM) and energy dispersive X-ray spectroscopy (EDS) analysis were conducted to obtain detailed information about the catalyst (Figure S3). The results showed uniform distribution of H<sub>2</sub>-imidazole moiety and silicotungstic acid on silica support. Although distribution of Cp\*Rh(III) was not clearly confirmed due to its low concentration, no aggregation of Rh was observed.



Figure S3. STEM/EDS analysis of Cp\*Rh(III)/STA/Q-10-H<sub>2</sub>-imidazole.

# 4. Optimization of reaction conditions for continuous-flow

# 4.1. Optimization of temperature



Scheme S4. Effects of reaction temperature on yields.

# 4.2. Optimization of flow rate



Scheme S5. Effects of flow rate on yields.

## 4.3. Optimization of substrate concentrations



Scheme S6. Effects of substrate concentrations on yields.

## 5. Investigation on deactivation of the catalyst

#### 5.1. Treatment of the used catalyst

After the investigation on catalyst lifetime, the used catalyst was collected and dried under vacuum. On completion of drying, the catalyst was used in the following experiments without further treatment.

## 5.2. Reaction under batch condition with used catalyst



Scheme S7. Reaction using the used catalyst under batch condition.

To confirm the possibility of technical issue particular to continuous-flow conditions, the activity of used catalyst was examined using the method described in 2.3.(c) and 2.4. The result showed actual deactivation of the used catalyst.

## 5.3. Washing experiment

Another possible mechanism for deactivation is adsorption of side-products on the catalyst. A washing experiment was conducted to confirm whether it is possible to regenerate the activity by washing the used catalyst.



Scheme S7. Washing experiment.

The 1:9 mixture of <sup>*i*</sup>PrOH/ethyl acetate was chosen as washing solvent considering the appropriate polarity for washing. Although this process could remove organic compounds on the catalyst without Rh leaching (Figure S4), the catalyst was not regenerated.



**Figure S4.** <sup>1</sup>H NMR analysis of the concentrated washing solution. Some aromatic compounds were observed. Rh leaching in washing solution: 0.2% (determined by ICP analysis)

To find aggregation of Rh, STEM/EDS analysis was also conducted for the used catalyst. Although no aggregation of Rh was found, aggregation of Ag was observed.



Figure S5. STEM/EDS analysis of the used catalyst.

## 5.5. Investigation on the limit of TON



Table S3. Measurement of TON/TOF under batch conditions.

To find the limit of TON of the Cp\*Rh(III) catalyst, C–H amidation reaction using homogeneous catalyst was conducted with different substrate amount and reaction time. The reaction condition was based on the previous report<sup>[13]</sup> except reaction temperature and substrate/catalyst (S/C) ratio. The results in entry 1 and 2 can be regarded as initial TOF of this catalyst. If the limit of TON existed around 300, the decrease in TOF would be observed when S/C was set 500. However, the result in entry 3 showed comparable TOF, which indicates the limit of TON of the homogeneous catalyst is over 400.

## 6. Compound data

• *N*-[2-(pyridine-2-yl)phenyl]benzamide (3a) <sup>[16]</sup>



18.0 mg (97% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm):  $\delta$  13.31 (s, 1H), 8.80 (dd, J = 8.3, 1.1 Hz, 1H), 8.67 (dq, J = 4.9, 0.9 Hz, 1H), 8.04 (dt, J = 6.3, 1.7 Hz, 2H), 7.86-7.83 (m, 1H), 7.80 (d, J = 8.1 Hz, 1H), 7.73 (dd, J = 7.8, 1.5 Hz, 1H), 7.54-7.46 (m, 4H), 7.29 (ddd, J = 7.4, 4.9, 1.2 Hz, 1H), 7.20 (td, J = 7.6, 1.1 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl3):  $\delta$  (ppm): 165.5, 158.2, 147.2, 138.1, 137.8, 135.7, 131.5, 130.2, 128.7, 128.5, 127.3, 125.5,

123.5, 122.9, 121.9, 121.8.

• N-(5-methyl-2-(pyridin-2-yl)phenyl)benzamide (3b)<sup>[16]</sup>



13.0 mg (67% yield), white solid. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 13.41 (s, 1H), 8.66 (s, 1H), 8.64 (d, 1H, J = 4.95 Hz), 8.04 (d, 2H, J = 6.61 Hz), 7.81 (td, 1H, J = 7.84, 1.65 Hz), 7.77 (d, 1H, J = 8.26 Hz), 7.62 (d, 1H, J = 8.26 Hz), 7.53-7.50 (m, 3H), 7.24 (d, 1H, J = 5.78 Hz), 7.01 (d, 1H, J = 8.26 Hz), 2.44 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 165.5, 158.3, 147.1, 140.6, 138.0, 137.7, 135.8, 131.4, 128.5, 128.5, 127.3, 124.4, 122.7, 122.5, 122.2, 121.6, 21.7.

• N-(5-methoxy-2-(pyridin-2-yl)phenyl)benzamide (3c)<sup>[16]</sup>



17.4 mg (84% yield), white solid. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 13.80 (s, 1H), 8.62 (d, 1H, J = 4.95 Hz), 8.58 (d, 1H, J = 2.48 Hz), 8.06 (d, 2H, J = 7.43 Hz), 7.82-7.79 (m, 1H), 7.75 (d, 1H, J = 7.43 Hz), 7.68 (d, 1H, J = 8.26 Hz), 7.56-7.50 (m, 3H), 7.22 (dd, 1H, J = 7.02, 5.37 Hz), 6.75 (dd, 1H, J = 8.67, 2.89 Hz), 3.93 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 165.8, 161.1, 158.2, 147.0, 140.0, 137.7, 135.8, 131.5, 129.6, 128.6, 127.4, 122.0, 121.1, 117.6, 110.7, 105.5, 55.5.

• N-(2-(pyridin-2-yl)-5-(trifluoromethyl)phenyl)benzamide (3d)<sup>[16]</sup>



15.4 mg (67% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 13.47 (s, 1H), 9.18 (d, J = 1.0 Hz, 1H), 8.70 (dq, J = 4.9, 0.9 Hz, 1H), 8.02 (dt, J = 6.6, 1.6 Hz, 2H), 7.89 (td, J = 7.8, 1.9 Hz, 1H), 7.81 (dt, J = 8.2, 1.3 Hz, 2H), 7.57-7.49 (m, 3H), 7.40 (dd, J = 8.3, 1.4 Hz, 1H), 7.35 (ddd, J = 7.5, 4.9, 1.1 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 165.7, 156.9, 147.4, 138.6, 138.2, 135.1, 131.8 (q, J = 32.6 Hz), 129.0, 128.7, 127.9, 127.3, 127.1, 123.8 (q, J = 270.9 Hz), 123.3, 122.8, 119.8 (q, J = 3.6 Hz), 118.7 (q, J = 3.9 Hz); <sup>19</sup>F NMR (470 MHz,

CDCl<sub>3</sub>): δ (ppm): -62.7.

• N-(5-chloro-2-(pyridin-2-yl)phenyl)benzamide (3e)<sup>[16]</sup>



14.4 mg (69% yield), white solid. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 13.52 (s, 1H), 8.92 (d, 1H, J = 1.65 Hz), 8.66 (d, 1H, J = 4.13 Hz), 8.02 (dd, 2H, J = 6.61, 1.65 Hz), 7.85 (td, 1H, J = 7.84, 1.65 Hz), 7.76 (d, 1H, J = 8.26 Hz), 7.64 (d, 1H, J = 8.26 Hz), 7.56-7.54 (m, 1H), 7.52-7.49 (m, 2H), 7.30 (dd, 1H, J = 7.43, 4.95 Hz), 7.14 (dd, 1H, J = 8.26, 2.48 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 165.6, 157.3, 147.2, 139.2, 138.0, 136.0, 135.3, 131.7, 129.5, 128.6, 127.3, 123.5, 123.4, 122.7, 122.2, 121.5.

• N-(4-methyl-2-(pyridin-2-yl)phenyl)benzamide (3f)<sup>[17]</sup>



17.4 mg (89% yield), white solid. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 13.14 (s, 1H), 8.67-8.66 (m, 2H), 8.03-8.01 (m, 2H), 7.82 (td, 1H, J = 7.84, 1.65 Hz), 7.78 (d, 1H, J = 8.26 Hz), 7.53-7.47 (m, 4H), 7.29-7.26 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 165.3, 158.4, 147.3, 137.7, 135.8, 135.6, 132.9, 131.4, 130.8, 129.2, 128.5, 127.3, 125.5, 122.9, 121.9, 121.8, 21.0.

• *N*-(4-methoxy-2-(pyridin-2-yl)phenyl)benzamide (**3g**)<sup>[17]</sup>



9.1 mg (44% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 12.91 (s, 1H), 8.69-8.67 (m, 2H), 8.01 (dt, J = 6.3, 1.7 Hz, 2H), 7.84 (ddd, J = 8.8, 6.9, 1.2 Hz, 1H), 7.76 (d, J = 8.3 Hz, 1H), 7.52-7.47 (m, 3H), 7.29 (ddd, J = 7.4, 4.9, 1.1 Hz, 1H), 7.24 (d, J = 2.9 Hz, 1H), 7.03 (dd, J = 9.1, 2.9 Hz, 1H), 3.87 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 165.1, 157.9, 155.5, 147.4, 137.8, 135.7, 131.3, 128.7, 128.5, 127.2, 123.4, 123.0, 122.1, 119.3, 114.7, 114.7, 55.6.

• *N*-(2-(pyridin-2-yl)-4-(trifluoromethyl)phenyl)benzamide (**3h**)<sup>[17]</sup>



13.9 mg (60% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 13.58 (s, 1H), 8.97 (d, J = 8.6 Hz, 1H), 8.65 (dq, J = 4.9, 0.9 Hz, 1H), 8.01 (dt, J = 6.6, 1.6 Hz, 2H), 7.94 (d, J = 1.5 Hz, 1H), 7.85 (dt, J = 11.0, 3.9 Hz, 1H), 7.80 (d, J = 8.3 Hz, 1H), 7.67 (dd, J = 8.8, 2.1 Hz, 1H), 7.56-7.46 (m, 3H), 7.31 (ddd, J = 7.4, 4.8, 1.2 Hz, 1H);<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 165.5, 156.5, 147.0, 141.2, 138.0, 134.9, 131.7,

128.5, 127.2, 126.7 (q, J = 3.6 Hz), 125.3 (q, J = 3.9 Hz), 124.7 (q, J = 32.8 Hz), 124.5, 124.0 (q, J = 269.6 Hz), 122.6, 122.5, 121.4; <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>): δ (ppm): -61.8.

• N-(4-chloro-2-(pyridin-2-yl)phenyl)benzamide (3i)<sup>[17]</sup>



13.3 mg (64% yield), pale-yellow solid. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 13.27 (s, 1H), 8.79 (d, 1H, J = 9.08 Hz), 8.68 (d, 1H, J = 4.13 Hz), 8.01 (dt, 2H, J = 6.61, 1.65 Hz), 7.87 (td, 1H, J = 7.84, 1.65 Hz), 7.78 (d, 1H, J = 8.26 Hz), 7.70 (d, 1H, J = 2.48 Hz), 7.56-7.53 (m, 1H), 7.52-7.49 (m, 2H), 7.42 (dd, 1H, J = 9.08, 2.48 Hz), 7.32 (dd, 1H, J = 7.02, 5.37 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 165.4, 156.9, 147.3, 138.0, 136.7, 135.3,

131.6, 129.9, 128.6, 128.4, 128.3, 127.3, 126.7, 123.1, 122.9, 122.5.

• 4-methyl-*N*-(2-(pyridin-2-yl)phenyl)benzamide (**3j**)<sup>[16]</sup>



15.8 mg (81% yield), yellow solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 13.25 (s, 1H), 8.79 (dd, J = 8.3, 1.0 Hz, 1H), 8.65 (dt, J = 4.9, 0.9 Hz, 1H), 7.93 (d, J = 8.3 Hz, 2H), 7.83-7.80 (m, 1H), 7.76 (d, J = 8.1 Hz, 1H), 7.70 (dd, J = 7.9, 1.4 Hz, 1H), 7.47-7.44 (m, 1H), 7.30-7.25 (m, 3H), 7.19-7.16 (m, 1H), 2.41 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 165.5, 158.3, 147.2, 141.9, 138.2, 137.8, 132.9, 130.2, 129.2, 128.7,

127.3, 125.5, 123.4, 122.9, 121.9, 121.8, 21.4.

• N-(2-(pyridin-2-yl)phenyl)-4-(trifluoromethyl)benzamide (3k)<sup>[16]</sup>



22.2 mg (96% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 13.57 (s, 1H), 8.78 (dd, J = 8.3, 1.1 Hz, 1H), 8.65 (dq, J = 4.9, 0.9 Hz, 1H), 8.14 (d, J = 8.1 Hz, 2H), 7.86 (ddd, J = 8.6, 6.8, 1.4 Hz, 1H), 7.82 (dt, J = 8.2, 1.0 Hz, 1H), 7.77-7.74 (m, 3H), 7.49-7.46 (m, 1H), 7.30 (ddd, J = 7.2, 5.0, 1.4 Hz, 1H), 7.22 (td, J = 7.6, 1.1 Hz,

1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 164.1, 158.1, 147.1, 139.1, 138.0, 137.8, 133.1 (q, J = 32.5 Hz), 130.3, 128.7, 127.8, 125.6 (q, J = 3.6 Hz), 125.3, 123.9, 123.8 (q, J = 270.9 Hz), 122.9, 122.1, 121.8; <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>): δ (ppm): -62.7.

• 4-chloro-*N*-(2-(pyridin-2-yl)phenyl)benzamide (**31**)<sup>[16]</sup>



17.3 mg (83% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 13.39 (s, 1H), 8.76 (dd, J = 8.3, 1.1 Hz, 1H), 8.64 (dq, J = 4.9, 0.9 Hz, 1H), 7.97 (dt, J = 9.0, 2.2 Hz, 2H), 7.85 (ddd, J = 8.7, 6.8, 1.2 Hz, 1H), 7.80 (d, J = 8.1 Hz, 1H), 7.73 (dd, J = 7.9, 1.4 Hz, 1H), 7.48-7.45 (m, 3H), 7.29 (ddd, J = 7.4, 4.9, 1.2 Hz, 1H), 7.20 (td, J = 7.6, 1.1 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 164.4, 158.2, 147.1, 138.0, 137.9,

137.7, 134.2, 130.3, 128.8 (2C), 128.7, 125.3, 123.7, 123.0, 122.0, 121.8.

## • *N*-(4-methoxy-2-(pyridin-2-yl)phenyl)-4-(trifluoromethyl)benzamide (**3m**)



12.7 mg (51% yield), light-yellow solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 13.16 (s, 1H), 8.70-8.67 (m, 2H), 8.11 (d, J = 8.1 Hz, 2H), 7.88 (ddd, J = 8.8, 6.9, 1.2 Hz, 1H), 7.80 (d, J = 8.1 Hz, 1H), 7.76 (d, J = 8.3 Hz, 2H), 7.32 (ddd, J = 7.4, 4.9, 1.1 Hz, 1H), 7.27 (d, J = 2.9 Hz, 1H), 3.89 (s, 3H); <sup>13</sup>C NMR (125) MHz, CDCl<sub>3</sub>): δ (ppm): 163.7, 157.8, 155.8, 147.3, 139.1, 138.0, 132.9 (q, J =

32.4 Hz), 131.1, 127.6, 127.0, 125.6 (q, J = 3.5 Hz), 123.8 (q, J = 270.9 Hz), 123.3, 123.0, 122.7, 122.3, 114.8, 114.7, 55.6; <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>): δ (ppm): -62.7. Mass: calcd. for C<sub>20</sub>H<sub>15</sub>F<sub>3</sub>N<sub>2</sub>NaO<sub>2</sub>: 395.0978; [M+Na]<sup>+</sup>, found: 395.0961. FTIR (cm<sup>-1</sup>): 1657, 1590, 1514, 1414, 1331, 1293, 1214, 1197, 1160, 1105, 1068, 857, 814, 786, 764, 737, 694, 684. Melting point: 134-138 °C.

• N-(2-(pyridin-2-yl)thiophen-3-yl)benzamide (3n)<sup>[18]</sup>



13.7 mg (73% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 13.29 (s, 1H), 8.58 (dq, J = 4.9, 0.9 Hz, 1H), 8.38 (d, J = 5.3 Hz, 1H), 8.09 (dt, J = 6.4, 1.7 Hz, 2H), 7.71 (ddd, J = 8.7, 6.9, 1.2 Hz, 1H), 7.57-7.51 (m, 4H), 7.32 (d, J = 5.5 Hz, 1H), 7.13 (ddd, J = 7.5, 4.9, 1.0 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ (ppm): 164.3, 154.2, 147.6, 139.0, 137.3, 134.9, 131.6, 128.6, 127.5, 124.9, 123.8, 120.5, 120.4, 120.0.

• N-(2-(isoquinolin-1-yl)phenyl)benzamide (30)<sup>[17]</sup>



13.7 mg (63% yield), mixture of colorless oil and solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 10.89 (s, 1H), 8.65-8.62 (m, 2H), 8.12 (dd, J = 8.6, 0.9 Hz, 1H), 7.86 (d, J = 8.2 Hz, 1H), 7.78-7.76 (m, 2H), 7.70-7.67 (m, 2H), 7.56-7.52 (m, 3H), 7.47-7.42 (m, 1H), 7.41-7.37 (m, 2H), 7.26 (td, J = 7.6, 1.1 Hz, 1H);  $^{13}$ C NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 164.9, 158.6, 140.7, 137.6, 136.5, 134.9, 131.8, 131.5, 130.6, 129.8, 128.5, 127.6 (2C), 127.4, 127.3, 127.1, 126.9, 123.2, 122.7, 120.6.

• N-(2-(quinolin-2-yl)phenyl)benzamide (**3p**)<sup>[19]</sup>



11.0 mg (50% yield), white solid. <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>): δ (ppm): 13.62 (s, 1H), 8.88 (d, 1H, J = 9.08 Hz), 8.30 (d, 1H, J = 8.26 Hz), 8.08-8.07 (m, 3H), 7.93 (d, 1H, J = 9.91 Hz), 7.87 (t, 2H, J = 7.84 Hz), 7.77 (t, 1H, J = 7.84 Hz), 7.60-7.49 (m, 5H), 7.26 (td, 1H, J = 7.43, 1.65 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): 166.0, 158.2, 146.1, 138.5, 137.7, 136.0, 131.6, 130.5, 130.2, 129.5, 128.5, 128.1, 127.7, 127.6, 126.9, 126.5, 125.5, 123.5, 121.8, 120.9.

• 4-ethoxy-2-phenylquinazoline (**3q**)<sup>[20]</sup>



14.1 mg (from 5.0 mL of crude solution, 50% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 8.59-8.56 (m, 2H), 8.13 (dq, J = 8.2, 0.7 Hz, 1H), 7.96 (dt, J = 8.4, 0.9 Hz, 1H), 7.78-7.75 (m, 1H), 7.51-7.44 (m, 4H), 4.74 (q, J = 7.1 Hz, 2H), 1.54 (t, J = 7.1 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): 166.6, 160.0, 151.8, 138.2, 133.3, 130.4, 128.4, 128.3, 127.9, 5.3, 62.7, 14.4

126.2, 123.4, 115.3, 62.7, 14.4.

• N-(1-(pyridin-2-yl)-1H-indol-2-yl)benzamide (3r)<sup>[21]</sup>



10.6 mg (51% yield, hexane:acetone = 3:1), pale yellow solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ (ppm): 11.86 (s, 1H), 8.63 (ddd, J = 5.0, 1.9, 0.8 Hz, 1H), 7.95-7.91 (m, 3H), 7.78 (dt, J = 8.3, 0.8 Hz, 1H), 7.63-7.59 (m, 2H), 7.56-7.53 (m, 1H), 7.51-7.48 (m, 2H), 7.30-7.26 (m, 2H), 7.22-7.16 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): 163.6, 152.0, 148.2, 139.4, 134.8, 134.2, 131.9, 131.8, 129.6, 128.7, 127.0, 122.0, 121.7, 120.7 (2C), 117.9, 110.5, 93.6.

• N-(1-(pyrimidin-2-yl)-1H-indol-2-yl)benzamide (3s)<sup>[17]</sup>



14.6 mg (from 5.0 mL of crude solution, 41% yield), white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 13.05 (s, 1H), 8.74 (d, J = 4.8 Hz, 2H), 8.71-8.69 (m, 1H), 8.00-7.98 (m, 2H), 7.56-7.50 (m, 4H), 7.44 (s, 1H), 7.25-7.22 (m, 2H), 7.11 (t, J = 4.8 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): 163.6, 158.7, 157.4, 135.5, 134.5, 132.6, 131.8, 129.9, 128.7, 127.0, 123.1, 122.4, 119.8, 116.4, 116.0, 95.7.

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# 8. NMR Charts



N-(5-methyl-2-(pyridin-2-yl)phenyl)benzamide (3b)



*N*-(5-methoxy-2-(pyridin-2-yl)phenyl)benzamide (3c)



N-(2-(pyridin-2-yl)-5-(trifluoromethyl)phenyl)benzamide (3d)





N-(5-chloro-2-(pyridin-2-yl)phenyl)benzamide (3e)



N-(4-methyl-2-(pyridin-2-yl)phenyl)benzamide (3f)







N-(2-(pyridin-2-yl)-4-(trifluoromethyl)phenyl)benzamide (3h)





























N-(2-(pyridin-2-yl)thiophen-3-yl)benzamide (3n)











*N*-(1-(pyrimidin-2-yl)-1H-indol-2-yl)benzamide (3s)

