Electronic supplementary information

A novel magnetic nanoparticle as an efficient and recyclable heterogeneous catalyst for suzuki cross-coupling reaction

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1. General Remarks

All the solvents used in this experiment are commercially available for analysis. In the absence of special instructions, it means that there is no need for any treatment or purification before use. Sodium dichromate, nitric acid, sulfuric acid and tetraethyl orthosilicate (TEOS) were all purchased from National Pharmaceutical Group Chemical Reagent Co., Ltd.,acenaphthene, diglycolamine, cuprous iodide and propyl triethoxysilane isocyanate were all purchased from Shanghai Aladdin Biochemical Technology Co., Ltd., N-bromosuccinamide (NBS), tetrphenylphosphine palladium, 2-aminomethylthiophene, cetyltrimethylammonium bromide (CTAB), palladium chloride, inorganic base. Organic base and silica gel used for column chromatography (300-400 mesh) were purchased from Shanghai Titan Technology Co., Ltd. Halogenated aromatic hydrocarbons and their derivatives, phenylboronic acid and their derivatives used in Suzuki-Miyaura coupling reaction were purchased from Shanghai Bide Pharmaceutical Technology Co., Ltd.

The nuclear magnetic resonance spectra (1H-NMR and 13C-NMR) for the structural analysis of the compounds were collected by Bruker- AVANCE III 400 MHz nuclear magnetic resonance (TMS internal standard), the high-resolution mass spectrometry was provided by mass spectrometer Wates XEVO G2 TOF (ESI source, and the mass spectra of Suzuki-Miyaura coupling products were collected from gas chromatography-mass spectrometry (GC-MS). The infrared spectrum was measured by Thermo Fisher-Nicolet 6700. The palladium content was measured by plasma emission spectrometer Agilent ICP-OES 725. The elemental analysis of the sample was obtained by German Elementar Vario EL III element analyzer, the morphology images were obtained by transmission electron microscope JEOL JEM 2100 F, the element valence on the surface of the sample was characterized by X-ray photoelectron spectroscopy Thermo Fisher 6700 Xi, and the crystal structure of the sample was measured by X-ray diffractometer Bruker D8 Advance. The hysteresis loop of the sample is obtained by vibrating sample magnetometer Lake Shore 7404.

2. Synthesis of Fe3O4@FSM@Pd

Synthesis and characterization of Palladium Ion fluorescence probe FP1

The synthesis route of palladium ion fluorescence probe is obtained from raw material acenaphthene FP8 through seven-step reactions. The key intermediate N-(2-(2-hydroxyethoxy) ethyl)-4-bromo-5-nitro-1-naphthalimide FP4 was prepared by bromination, nitration, oxidation of sodium dichromate and
reaction with diglycolamine. Then it was modified with 2-aminomethylthiophene and p-alkynyl anisole to prepare the palladium ion fluorescence probe FP2. Finally, FP2 was modified by propyl triethoxysilane isocyanate to prepare FP1, which bind to the silane on the surface of magnetic nanomaterials. The intermediates and products were characterized by $^1$H-NMR, $^{13}$C-NMR and some of them were characterized by mass spectrometry.

Scheme S1. The synthesis route of palladium ion fluorescence probe FP1.

**Synthesis of Fe$_3$O$_4$@SiO$_2$**

To a stirred solution of FeCl$_3$·6H$_2$O (2 g, 7.4 mmol) in 35mL ethylene glycol was added anhydrous sodium acetate (2 g, 24.4 mmol). The mixture was stirred at room temperature for 0.5 h and 200 °C for 8 h. The supernatant was removed by centrifugation, washed with ethanol for three times, and dried in an infrared oven to obtain nanometer ferric oxide (nano Fe$_3$O$_4$). Next, to a stirred solution of cetyltrimethyl ammonium bromide (CTAB, 1.5 g, 3.8 mmol) in 10 mL water was added the above nano Fe$_3$O$_4$ (200 mg). The mixture was mechanically stirred at 60 °C for 0.5 h. To a mechanically stirred mixture were slowly added 2M NaOH aqueous solution (60 mL) and tetraethyl orthosilicate (TEOS, 2
mL) at 70 °C for 3 h. The solid crude product was magnetically separated, and was washed with ethanol for three times, and then dried in an infrared oven to give magnetic nanomaterials (Fe₃O₄@ SiO₂).

**Synthesis of Fe₃O₄@FSM**

A mixture of Silanization probe FP1 (10 mg, 0.01 mmol) and dried and activated Fe₃O₄@SiO₂ (particle size about 100 nm) in anhydrous ethanol (20 mL) was stirred at and refluxed for 72 h under argon. After stopping the reaction, the solid crude product was magnetically separated, and washed with ethanol until the supernatant was no fluorescence, and then dried in an infrared oven to give the black solid (Fe₃O₄@FSM).

**Synthesis of Fe₃O₄@FSM@Pd**

To a stirred aqueous solution of 10 M palladium chloride (PdCl₂, 100 mL) was added Fe₃O₄@FSM (2 g). The mixture was stirred at room temperature for 3 h. The solid crude product was magnetically separated, and washed with water for three times, and then drying in infrared oven to give the black solid (Fe₃O₄@FSM@Pd).

3. **Characterization of Fe₃O₄@FSM@Pd**

**FT-IR characterization**

![FT-IR spectra](image)

**Fig. S1.** The FT-IR spectra of FP1, Fe₃O₄@SiO₂ and Fe₃O₄@FSM.
TEM characterization

**Fig. S2.** The TEM images of Fe₃O₄@FSM.

DLS characterization

**Fig. S3.** The DLS image of Fe₃O₄@FSM.

4. General Procedure for Suzuki-Miyaura Cross-Coupling Catalyzed by Fe₃O₄@FSM@Pd

Halogenated aromatic hydrocarbons (1.0 mmol), phenylboronic acid or their derivatives (1.5 mmol), catalyst Fe₃O₄@FSM@Pd (5 mg, 0.1 mol%), N, N-diisopropylamine ([(i-Pr)₂NH, 0.35 mL, 2.5mmol) were mixed in pure water (2 mL). The mixture was stirred at 100 °C for 30 min under air. The catalyst was separated and recovered from the reaction solution by external magnetic field. The crude product was diluted with dichloromethane, washed with water, dried (anhydrous Na₂SO₄), filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1, v/v) to give products as the white powdery solid. The products were confirmed by ¹H-NMR and ¹³C-NMR.
5. Study on the conditions of catalytic activity of different heterocyclic substrates

Halogenated aromatic hydrocarbons (1.0 mmol), Heteroarylboronic acid (1.5 mmol), catalyst Fe₃O₄@FSM@Pd (5 mg, 0.1 mol%), N, N-diisopropylamine ((i-Pr)₂NH, 0.35 mL, 2.5 mmol) were mixed in pure water (2 mL). The mixture was stirred at 100 °C for 30 min under air. The catalyst was separated and recovered from the reaction solution by external magnetic field. The crude product was diluted with dichloromethane, washed with water, dried (anhydrous Na₂SO₄), filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1, v/v) to give products as the white powdery solid. The products were confirmed by ¹H-NMR and ¹³C-NMR.

6. Catalyst Recycling and Reuse

**Fig. S4.** Recyclability of Fe₃O₄@FSM@Pd.

**Fig. S5.** The TEM images of the recycled Fe₃O₄@FSM@Pd.

**Table S1** Elemental analysis results of unused Fe₃O₄@FSM@Pd and recycled Fe₃O₄@FSM@Pd.
4-bromoanisole (125 μL, 1.0 mmol), phenylboronic acid (183 mg, 1.5 mmol), catalyst Fe₃O₄@FSM@Pd (5 mg, 0.1 mol%), N, N-diisopropylamine ((i-Pr)₂NH, 0.35 mL, 2.5 mmol) were mixed in pure water (2 mL). The mixture was stirred at 100 °C for 30 min under air. The catalyst was separated and recovered from the reaction solution by external magnetic field. The catalyst was separated and recovered from the reaction solution by external magnetic field. The crude product was diluted with dichloromethane, washed with water, dried (anhydrous Na₂SO₄), filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1, v/v) to give products as the white powdery solid. Wash the catalyst with water and ethanol at the end of each reaction, and then use it for the next time after drying in the infrared oven. The catalyst can be reused for 5 times without a significant loss of activity. The product was confirmed by ¹H-NMR, ¹³C-NMR and gas chromatography-mass spectrometry (GC-MS).

7. Maitlis’ filtration test

Firstly, 4-bromoanisole (125 μL, 1.0 mmol), phenylboronic acid (183 mg, 1.5 mmol), catalyst Fe₃O₄@FSM@Pd (5 mg, 0.1 mol%), N, N-diisopropylamine ((i-Pr)₂NH, 0.35 mL, 2.5 mmol) were mixed in pure water (2 mL). The mixture was stirred at 100 °C for 10 min under air. The catalyst was separated from the reaction solution by an external magnetic field, and then the reaction solution was stirred at 100 °C for 1 h under air. The crude product was diluted with dichloromethane, washed with water, dried (anhydrous Na₂SO₄), filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1, v/v) to give products as the white powdery solid.

Secondly, the catalyst Fe₃O₄@FSM@Pd (5 mg, 0.1 mol%) and N, N-diisopropylamine ((i-Pr)₂NH, 0.35 mL, 2.5 mmol) were mixed in pure water (2 mL). The mixture was stirred at 100 °C for 30 min under air. The catalyst was separated from the reaction solution by an external magnetic field, and then 4-bromoanisole (125 μL, 1.0 mmol) and phenylboronic acid (183 mg, 1.5 mmol) were added to the filtrate. The mixture was stirred at 100 °C for 30 min under air. The crude product was diluted with

<table>
<thead>
<tr>
<th>Sample</th>
<th>N (%)</th>
<th>C (%)</th>
<th>H (%)</th>
<th>S (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe₃O₄@FSM@Pd</td>
<td>0.78</td>
<td>3.80</td>
<td>1.35</td>
<td>0.16</td>
</tr>
<tr>
<td>Fe₃O₄@FSM@Pd b</td>
<td>0.62</td>
<td>3.42</td>
<td>1.34</td>
<td>0.17</td>
</tr>
</tbody>
</table>

*a unused Fe₃O₄@FSM@Pd. b The catalyst Fe₃O₄@FSM@Pd was reused 5 time.
dichloromethane, washed with water, dried (anhydrous Na₂SO₄), filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1, v/v) to give products as the white powdery solid.

8. Kinetic experiment of catalytic reaction

The three mixtures of 4-bromoanisole (125 μL, 1.0 mmol), phenylboronic acid (183 mg, 1.5 mmol), N,N-diisopropylamine ((i-Pr)₂NH, 0.35 mL, 2.5 mmol) in pure water (2 mL) were added with different amounts of catalyst Fe₃O₄@FSM@Pd (5 mg, 0.1 mol%; 10 mg, 0.2 mol%, 25 mg, 0.5 mol%, respectively). The three mixtures were stirred at 100 °C for 5 min, 10 min, 15 min, 20 min, 25 min and 30 min respectively under air. The catalyst was separated and recovered from the reaction solution by external magnetic field. The crude product was diluted with dichloromethane, washed with water, dried (anhydrous Na₂SO₄), filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1, v/v) to give products as the white powdery solid. The product was confirmed by ¹H-NMR and ¹³C-NMR.

9. Phase trajectory test

Phenylboronic acid (366 mg, 3.0 mmol), catalyst Fe₃O₄@FSM@Pd (10 mg, 0.2 mol%) or palladium acetate (Pd(OAc)₂, 0.2 mg, 0.2 mol%) and N,N-diisopropylamine ((i-Pr)₂NH, 0.7 mL, 5.0 mmol) were mixed in pure water (4 mL). To a stirred of mixtures were added bromobenzene (0.15 mL, 1.0 mmol) and 4-bromoacetophenone (199 mg, 1.0 mmol) at 100 °C for different times under air. The catalyst was separated and recovered from the reaction solution by an external magnetic field. The crude product was diluted with dichloromethane, washed with water, dried (anhydrous Na₂SO₄), filtered, and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1, v/v) to give products as the white powdery solid. The phase trajectory curves of the two catalysts were compared with the isolated yields of the two products.
Fig. S6. The proposed mechanism for Fe₃O₄@FSM@Pd in Suzuki reaction.

10. Spectroscopic data of fluorescence probe

FP7:

Light yellow solid. ¹H NMR (400 MHz, CDCl₃): δ 7.74 (d, J = 8.4 Hz, 1H), δ 7.61 (d, J = 7.2 Hz, 1H), 7.52 (t, J = 8.4 Hz, 1H), 7.50 (t, J = 8.4 Hz, 1H), 7.28 (t, J = 6.8 Hz, 1H), 7.08 (t, J = 7.6 Hz, 1H), 3.38-3.34 (m, 2H), 3.29-3.26 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 146.27, 145.98, 140.29, 130.90, 129.08, 121.77, 120.20, 120.07, 116.80, 30.65, 29.94.

FP6:

Yellow solid. ¹H NMR (400 MHz, CDCl₃): δ 7.82 (d, J = 7.6 Hz, 1H), 7.71 (t, J = 7.6 Hz, 1H), 7.29 (t, J = 7.2 Hz, 1H), 7.25 (t, J = 7.2 Hz, 1H), 3.46-3.37 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): δ 151.41, 146.49, 144.74, 141.22, 136.25, 125.36, 122.23, 121.39, 118.83, 111.58, 30.61, 30.16.
FP5:

Reddish brown solid. $^1$H NMR (400 MHz, DMSO-$d_6$): δ 8.68 (d, $J = 7.6$ Hz, 1H), 8.48-8.8.42 (m, 3H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 160.23, 159.62, 160.98, 136.91, 133.65, 132.92, 132.64, 125.11, 123.92, 123.88, 120.57, 120.43

FP4:

Brown solid. $^1$H NMR (400 MHz, DMSO-$d_6$): δ 8.63 (d, $J = 8.0$ Hz, 1H), 8.44-8.36 (m, 3H), 4.53 (m, 1H), 4.23 (t, $J = 6.4$ Hz, 2H), 3.67 (t, $J = 6.4$ Hz, 2H), 3.47 (s, 4H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 162.98, 162.27, 150.61, 136.66, 132.38, 131.53, 130.39, 126.14, 124.87, 122.96, 120.24, 72.60, 67.11, 60.64.

FP3:
Brown solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 8.22 (d, $J = 8.4$ Hz, 1H), 8.15 (d, $J = 8.0$ Hz, 1H), 8.05 (t, $J = 5.6$ Hz, 1H), 8.90 (d, $J = 8.0$ Hz, 1H), 7.47 (d, $J = 5.2$ Hz, 1H), 7.23 (d, $J = 4.4$ Hz, 1H), 7.04 (t, $J = 4.0$ Hz, 1H), 6.94 (d, $J = 8.8$ Hz, 1H), 4.85 (d, $J = 5.6$ Hz, 2H), 4.58 (s, 1H), 4.16 (t, $J = 6.4$ Hz, 2H), 3.61 (t, $J = 6.4$ Hz, 2H), 3.46 (s, 4H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 163.58, 162.95, 149.99, 141.08, 134.45, 132.54, 132.01, 131.42, 127.57, 126.74, 126.04, 125.07, 122.02, 117.81, 109.63, 107.41, 72.55, 67.37, 60.65, 42.66, 39.02.

FP2:

Red solid. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.39-8.35 (m, 2H), 8.32 (d, $J = 8.8$ Hz, 1H), 7.88 (d, $J = 7.6$ Hz, 1H), 7.52 (d, $J = 4.4$ Hz, 1H), 7.31 (d, $J = 2.8$ Hz, 1H), 7.16 (d, $J = 8.8$ Hz, 2H), 7.08-7.06 (m, 1H), 6.99 (d, $J = 8.8$ Hz, 1H), 6.94 (d, $J = 8.8$ Hz, 2H), 4.86 (d, $J = 4.4$ Hz, 2H), 4.58 (t, $J = 6.0$ Hz, 1H), 4.19
(t, J = 6.4 Hz, 2H), 3.82 (s, 3H), 3.63 (t, J = 6.4 Hz, 2H), 3.47 (d, J = 2.4 Hz, 4H). $^{13}$C NMR (100 MHz, CDCl$_3$): δ 164.57, 164.23, 160.63, 150.33, 138.95, 134.94, 133.06, 131.96, 130.86, 130.25, 127.24, 126.06, 124.08, 122.06, 118.48, 114.24, 113.17, 110.17, 105.26, 101.19, 88.24, 72.28, 68.66, 61.91, 55.43, 43.11, 39.37.

FP1:

\[
\begin{align*}
&\text{EtO}_2\text{Si} \quad \text{EtO} \quad \text{O} \\
&\text{HN} \quad \text{O} \\
&\text{O} \quad \text{O} \\
&\text{N} \quad \text{N} \\
&\text{O} \quad \text{N} \\
&\text{O} \quad \text{N} \\
&\text{NH} \quad \text{≡} \\
&\text{OCH}_3
\end{align*}
\]

Red solid. $^1$H NMR (400 MHz, CDCl$_3$): δ 8.52-8.48 (m, 3H), 7.78 (d, J = 7.6 Hz, 1H), 7.24 (dd, J = 4.8, 1.2 Hz, 1H), 7.17 (d, J = 3.6 Hz, 1H), 7.02 (d, J = 8.8 Hz, 2H), 6.98 (dd, J = 5.2, 3.2 Hz, 1H), 6.81-6.78 (m, 3H), 5.02 (s, 1H), 4.75 (d, J = 4.4 Hz, 2H), 4.42 (t, J = 6.4 Hz, 2H), 4.18 (t, J = 4.4 Hz, 2H), 3.85-3.80 (m, 11H), 3.74-3.72 (t, J = 4.4 Hz, 2H), 3.14 (q, J = 6.4 Hz, 2H), 1.25-1.19 (m, 9 H), 0.87 (q, J = 6.4 Hz, 2H), 0.62 (q, J = 8.0 Hz, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$): δ 163.31, 162.96, 161.54, 159.61, 155.39, 149.30, 138.00, 133.80, 132.03, 130.98, 129.91, 129.16, 126.20, 125.00, 123.01, 121.22, 117.61, 113.22, 112.17, 109.31, 104.22, 100.09, 87.19, 68.13, 66.96, 63.13, 57.44, 57.42, 54.39, 54.37, 42.44, 42.10, 37.82, 35.47, 30.41, 28.68, 22.27, 17.41, 17.27, 6.57. MS (HR-ESI) m/z, calcd. For [C$_{40}$H$_{47}$N$_3$O$_9$SSi + Na$^+$] 796.2694, found 796.2691.
11. $^1$H NMR and $^{13}$C NMR spectra of fluorescence probe

**Fig. S7.** The $^1$H NMR spectrum of FP7
Fig. S8. The $^{13}$C NMR spectrum of FP7
Fig. S9. The $^1$H NMR spectrum of FP6
Fig. S10. The $^{13}$C NMR spectrum of FP6
Fig. S11. The $^1$H NMR spectrum of FP5
Fig. S12. The $^{13}$C NMR spectrum of FP5
Fig. S13. The $^1$H NMR spectrum of FP4
Fig. S14. The $^{13}$C NMR spectrum of FP4.

Solvent: DMSO-d$_6$
Formula: C$_{nd}$H$_{10}$BrN$_2$O$_k$
Exact mass: 408.00
Fig. S15. The $^1$H NMR spectrum of FP3
Fig. S16. The $^{13}$C NMR spectrum of FP3.
Fig. S17. The $^1$H NMR spectrum of FP2
Fig. S18. The $^{13}$C NMR spectrum of FP2.
Fig. S19. The $^1$H NMR spectrum of FP1
Fig. S20. The $^{13}$C NMR spectrum of FP1
**Fig. S21.** The HRMS spectrum of FP1
12. Spectroscopic data of coupling products

X1-3: Diphenyl

Light yellow solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.72 - 7.62 (m, 4H), 7.47 (t, $J = 7.6$ Hz, 4H), 7.37 (t, $J = 7.3$ Hz, 2H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 140.68, 129.41, 127.90, 127.17.

X4-6: 4-Phenyltoluene

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.63 (d, $J = 7.9$ Hz, 2H), 7.55 (d, $J = 8.0$ Hz, 2H), 7.44 (t, $J = 7.6$ Hz, 2H), 7.34 (t, $J = 7.3$ Hz, 1H), 7.25 (d, $J = 7.9$ Hz, 2H), 2.33 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 140.62, 137.81, 137.14, 129.98, 129.32, 127.55, 126.97, 126.90, 21.12.

X7-9: 4-Nitrobiphenyl

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 8.28 (d, $J = 8.1$ Hz, 2H), 7.94 (d, $J = 8.1$ Hz, 2H), 7.77 (d, $J = 6.9$ Hz, 2H), 7.50 (dd, $J = 15.4, 6.9$ Hz, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 147.30, 147.13, 147.09, 138.29, 133.31, 129.83, 129.70, 129.53, 128.32, 127.74, 125.83, 124.55.

X10-12, B1: 4-Methoxybiphenyl

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.69 - 7.53 (m, 4H), 7.43 (t, $J = 7.6$ Hz, 2H), 7.31 (t, $J = 7.3$ Hz, 1H), 7.03 (d, $J = 8.7$ Hz, 2H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 159.37, 140.32, 133.01, 129.33, 128.22, 127.17, 126.64, 114.83, 55.62.

X13: 4-Chlorobiphenyl
Light yellow solid. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 7.75 - 7.61 (m, 4H), 7.56 - 7.43 (m, 4H), 7.39 (t, \(J = 7.3\) Hz, 1H). \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)): \(\delta\) 139.46, 139.30, 132.81, 129.50, 129.40, 129.35, 128.93, 128.28, 127.17, 127.12.

**X14: 4-(Trifluoromethyl)-biphenyl**

White solid. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 7.85 (d, \(J = 8.2\) Hz, 2H), 7.77 (d, \(J = 8.2\) Hz, 2H), 7.70 (d, \(J = 7.4\) Hz, 2H), 7.50 (t, \(J = 7.4\) Hz, 2H), 7.43 (t, \(J = 7.2\) Hz, 1H). \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)): \(\delta\) 197.97, 145.00, 139.38, 136.12, 129.57, 129.38, 128.86, 127.47, 127.35, 27.24.

**X15: 4-Acetylbiphenyl**

White solid. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 8.06 - 8.01 (m, 2H), 7.71 - 7.67 (m, 2H), 7.63 (dt, \(J = 3.1, 1.9\) Hz, 2H), 7.50 - 7.45 (m, 2H), 7.43 - 7.38 (m, 1H), 2.64 (s, 3H). \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)): \(\delta\) 197.96, 145.00, 139.39, 136.12, 129.57, 129.38, 128.85, 127.47, 127.34.

**X16: 2-Cyanobiphenyl**

Yellow solid. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 7.96 (d, \(J = 7.8\) Hz, 1H), 7.80 (d, \(J = 7.8, 1.1\) Hz, 1H), 7.69 - 7.46 (m, 7H). \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)): \(\delta\) 145.02, 138.32, 134.31, 134.00, 130.60, 129.19, 128.67, 119.03, 110.67.

**X17: Methyl biphenyl-2-carboxylate**
B2-3: 4-Methoxy-4'-Methylbiphenyl

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.68 - 7.38 (m, 4H), 7.23 (m, 2H), 7.00 (m, 2H), 3.78 (s, 3H), 2.32 (s, 3H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 159.13, 137.45, 136.34, 134.95, 132.96, 129.92, 129.00, 127.94, 126.47, 114.78, 83.93, 55.62, 25.15, 21.09.

B4: 4-Methoxy-3'-Methylbiphenyl

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.62 - 7.55 (m, 2H), 7.44 - 7.37 (m, 2H), 7.31 (t, $J = 7.6$ Hz, 1H), 7.12 (d, $J = 7.4$ Hz, 1H), 7.05 - 6.98 (m, 2H), 3.79 (s, 3H), 2.36 (s, 3H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 159.29, 140.27, 138.41, 133.11, 129.22, 128.20, 127.82, 127.32, 123.77, 114.77, 55.62, 21.61.

B5: 4-Methoxy-[1,1'-biphenyl]-4'-carbonitrile

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.86 (q, $J = 8.5$ Hz, 4H), 7.72 (t, $J = 5.8$ Hz, 2H), 7.12 - 7.03 (m, 2H), 3.82 (s, 3H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 160.39, 144.73, 133.26, 130.88, 128.80, 127.33, 119.49, 115.09, 109.60, 55.76.

B6: 4-Fluoro-4'-methoxybiphenyl
White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.68 - 7.61 (m, 2H), 7.61 - 7.55 (m, 2H), 7.30 - 7.21 (m, 2H), 7.05 - 6.99 (m, 2H), 3.80 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 163.12, 160.70, 159.33, 136.83, 136.80, 132.01, 128.57, 128.49, 128.20, 116.15, 115.94, 114.84, 55.63.

B7: 4-Methoxy-4'-nitrobiphenyl

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 8.33 - 8.22 (m, 2H), 7.98 - 7.88 (m, 2H), 7.82 - 7.71 (m, 2H), 7.09 (d, $J$ = 8.8 Hz, 2H), 3.83 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 160.67, 146.76, 146.48, 130.41, 129.05, 127.48, 124.57, 115.17, 55.79.

B8: 1-methoxy-2-(4-methoxyphenyl)benzene

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.41 (d, $J$ = 8.4 Hz, 2H), 7.35 - 7.22 (m, 2H), 7.10 (t, $J$ = 10.9 Hz, 1H), 6.99 (dd, $J$ = 17.9, 7.8 Hz, 3H), 3.77 (d, $J$ = 12.8 Hz, 6H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 158.71, 156.55, 130.86, 130.80, 130.62, 129.96, 128.78, 121.21, 113.92, 112.16, 55.89, 55.54.

B9: 1-methoxy-4-[4-(trifluoromethyl)phenyl]benzene

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.84 (d, $J$ = 8.2 Hz, 2H), 7.76 (d, $J$ = 8.3 Hz, 2H), 7.69 (d, $J$ = 8.8 Hz, 2H), 7.07 (d, $J$ = 8.8 Hz, 2H), 3.81 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 160.15, 131.26, 128.68, 127.25, 126.17, 126.13, 115.02, 55.70.

A1: 2-(4-methylphenyl)quinoxaline
Yellow solid. $^1$H NMR (400 MHz, DMSO-$d_6$): δ 9.57 (s, 1H), 8.26 (d, $J = 8.2$ Hz, 2H), 8.17 - 8.07 (m, 2H), 7.86 (dd, $J = 14.8$, 6.9, 1.4 Hz, 2H), 7.42 (d, $J = 8.0$ Hz, 2H), 2.42 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 151.41, 144.10, 141.91, 141.44, 140.81, 133.75, 131.04, 130.22, 130.14, 129.59, 129.31, 127.83, 21.45.

A2: 1-methyl-5-[5-(trifluoromethyl)-2-pyridinyl]-indole

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): δ 8.99 (s, 1H), 8.43 (s, 1H), 8.19 (d, $J = 1.4$ Hz, 2H), 8.02 (dd, $J = 8.7$, 1.5 Hz, 1H), 7.57 (d, $J = 8.7$ Hz, 1H), 7.42 (d, $J = 3.1$ Hz, 1H), 6.57 (d, $J = 2.9$ Hz, 1H), 3.84 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 161.72, 146.50, 146.46, 138.04, 134.76, 134.72, 131.46, 128.85, 128.81, 125.99, 123.29, 122.98, 122.66, 120.88, 120.36, 120.01, 110.62, 102.08, 33.10.

A3: 5-[4-(trifluoromethoxy)phenyl]-2-thiophenecarboxaldehyde

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): δ 9.94 (s, 1H), 8.07 (d, $J = 3.9$ Hz, 1H), 7.94 (d, $J = 8.7$ Hz, 2H), 7.79 (d, $J = 3.9$ Hz, 1H), 7.49 (d, $J = 8.4$ Hz, 2H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ 184.65, 151.09, 149.34, 143.10, 139.62, 132.22, 128.72, 126.63, 122.31.

A4: 1-(2,4-dimethoxyphenyl)-2-naphthalenol
White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 9.18 (s, 1H), 7.80 - 7.75 (m, 1H), 7.73 (d, $J$ = 8.8 Hz, 1H), 7.29 - 7.19 (m, 3H), 7.16 (dd, $J$ = 7.9, 1.0 Hz, 1H), 7.01 (d, $J$ = 8.2 Hz, 1H), 6.70 (d, $J$ = 2.3 Hz, 1H), 6.63 (dd, $J$ = 8.3, 2.4 Hz, 1H), 3.84 (s, 3H), 3.60 (s, 3H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 160.51, 158.87, 152.78, 134.57, 133.04, 128.66, 128.27, 128.19, 126.23, 124.79, 122.64, 118.83, 118.36, 117.64, 105.37, 99.15, 55.67.

M1: 2,6-diphenylpyridine

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 8.28 - 8.18 (m, 4H), 8.02 - 7.91 (m, 3H), 7.55 (t, $J$ = 7.4 Hz, 4H), 7.51 - 7.43 (m, 2H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 156.11, 139.16, 138.82, 129.64, 129.27, 127.10, 119.32.

M2: 4,4''-bis(trifluoromethoxy)-1,1':4',1''-terphenyl

White solid. $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.86 (d, $J$ = 7.9 Hz, 4H), 7.81 (s, 4H), 7.48 (d, $J$ = 7.6 Hz, 4H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 148.43, 139.26, 138.59, 129.02, 127.92, 122.00.
13. $^1$H NMR and $^{13}$C NMR spectra of coupling products

Fig. S22. The $^1$H NMR spectrum of X1-3
Chemical Formula: C_{12}H_{10}
Exact Mass: 154.08
Solvent: DMSO-d_6

Fig. S23. The $^{13}$C NMR spectrum of X1-3
Fig. S24. The $^1$H NMR spectrum of X4-6
Fig. S25. The $^{13}$C NMR spectrum of X4-6
Fig. S26 The $^1$H NMR spectrum of X7-9
Fig. S27. The $^{13}$C NMR spectrum of X7-9
Fig. S28. The $^1$H NMR spectrum of X10-12; B1
Fig. S29. The $^{13}$C NMR spectrum of X10-12; B1
Fig. S30. The $^1$H NMR spectrum of X13
Fig. S31. The $^{13}$C NMR spectrum of X13
Fig. S32. The $^1$H NMR spectrum of X14
Fig. S33. The $^{13}$C NMR spectrum of X14

Chemical Formula: C$_{12}$H$_{10}$F$_{2}$

Exact Mass: 222.07

Solvent: DMSO-d$_{6}$
Fig. S34. The $^1$H NMR spectrum of X15

Chemical Formula: C$_4$H$_{12}$O
Exact Mass: 196.09
Solvent: CDCl$_3$
Fig. S35. The $^{13}$C NMR spectrum of X15

Chemical Formula: C$_8$H$_5$O
Exact Mass: 196.09
Solvant: DMSO-d$_6$
Fig. S36. The $^1$H NMR spectrum of X16
Fig. S37. The $^{13}$C NMR spectrum of X16
Fig. S38. The $^1$H NMR spectrum of X17

Chemical Formula: C$_{14}$H$_{12}$O$_2$
Exact Mass: 212.08
Solvent: DMSO-d$_6$
Fig. S39. The $^{13}$C NMR spectrum of X17

Chemical Formula: C_{12}H_{24}O_{2}
Exact Mass: 212.08
Solvent: DMSO-d$_6$
Fig. S40. The $^1$H NMR spectrum of B2-3
Fig. S41. The $^{13}$C NMR spectrum of B2-3
Fig. S42. The $^1$H NMR spectrum of B4
Fig. S43. The $^{13}$C NMR spectrum of B4

Chemical Formula: C$_{14}$H$_{14}$O
Exact Mass: 198.10
Solvent: DMSO-d$_6$
Fig. S44. The $^1$H NMR spectrum of B5
Fig. S45. The $^{13}$C NMR spectrum of B5

Chemical Formula: C$_8$H$_{15}$NO
Exact Mass: 209.08
Solvent: DMSO-d$_6$
Fig. S46. The $^1$H NMR spectrum of B6
Fig. S47. The $^{13}$C NMR spectrum of B6
Fig. S48. The $^1$H NMR spectrum of B7
Fig. S49. The $^{13}$C NMR spectrum of B7

Chemical Formula: C$_{13}$H$_{12}$NO$_3$

Exact Mass: 229.07

Solvent: DMSO-d$_6$
Fig. S50. The $^1$H NMR spectrum of B8
Fig. S51. The $^{13}$C NMR spectrum of B8
Fig. S52. The $^1$H NMR spectrum of B9
Fig S53. The $^{13}$C NMR spectrum of B9
Fig. S54. The $^1$H NMR spectrum of A1
Fig. S55. The $^{13}$C NMR spectrum of A1
Fig. S56. The $^1$H NMR spectrum of A2

Chemical Formula: C$_{15}$H$_{11}$F$_{3}$N$_{2}$
Exact Mass: 276.09
Solvent: DMSO-$d_6$
Fig. S57. The $^{13}$C NMR spectrum of A2
Fig. S58. The $^1$H NMR spectrum of A3

Chemical Formula: C$_{12}$H$_7$F$_3$O$_2$S
Exact Mass: 272.01
Solvent: DMSO-d$_6$
Chemical Formula: C_{13}H_{2}F_{3}O_{2}S
Exact Mass: 272.01
Solvent: DMSO-d$_6$

Fig. S59. The $^{13}$C NMR spectrum of A3
Fig. S60. The $^1$H NMR spectrum of A4

Chemical Formula: C$_{18}$H$_{16}$O$_3$
Exact Mass: 280.11
Solvent: DMSO-d$_6$
Fig. S61. The $^{13}$C NMR spectrum of A4
Fig. S62. The $^1$H NMR spectrum of M1.
Fig. S63. The $^{13}$C NMR spectrum of M1

Chemical Formula: C$_{13}$H$_{13}$N
Exact Mass: 231.10
Solvent: DMSO-$d_6$
Fig. S64. The $^1$H NMR spectrum of M2
Fig. S65. The $^{13}$C NMR spectrum of M2
14. Spectroscopic data of API intermediates

API intermediates 1: 4'-Methyl-2-cyanobiphenyl

White solid. $^1$H-NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.94 (dd, $J = 7.7$, 0.7 Hz, 1H), 7.78 (td, $J = 7.8$, 1.2 Hz, 1H), 7.57 (dd, $J = 9.6$, 8.6, 4.4 Hz, 2H), 7.48 (d, $J = 8.1$ Hz, 2H), 7.35 (d, $J = 7.9$ Hz, 2H), 3.35 (s, 2H), 2.39 (s, 3H). $^{13}$C-NMR (100 MHz, DMSO-$d_6$): $\delta$ 145.02, 138.73, 135.45, 134.28, 133.92, 130.45, 129.77, 129.01, 128.39, 119.12, 110.57, 21.23. MS (HR-ESI) $m/z$, calcd. For [C$_{14}$H$_{11}$N + Na$^+$] 216.0784, found 216.0790.

API intermediates 2: Methyl 2-methyl-4'-(trifluoromethoxy)[1,1'-biphenyl]-3-carboxylate

White solid. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.83 (dd, $J = 7.5$, 1.3 Hz, 1H), 7.36 - 7.30 (m, 2H), 7.27 (dd, $J = 7.4$, 2.5 Hz, 3H), 3.92 (s, 3H), 2.40 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 168.49, 148.07, 142.17, 140.50, 136.15, 135.69, 133.49, 132.05, 131.66, 129.51, 126.31, 121.33, 52.61, 18.45. MS (HR-ESI) $m/z$, calcd. For [C$_{16}$H$_{13}$F$_3$O$_3$ + H$^+$] 311.0890, found 311.0894.

API intermediates 3: 7-Bromo-2-(1-methyl-1H-pyrazol-4-yl)quinoxaline

White solid. $^1$H-NMR (400 MHz, DMSO-$d_6$): $\delta$ 9.32 (s, 1H), 8.63 (s, 1H), 8.29 (s, 1H), 8.17 (d, $J = 2.1$ Hz, 1H), 7.97 (d, $J = 8.8$ Hz, 1H), 7.86 (dd, $J = 8.8$, 2.2 Hz, 1H), 3.97 (s, 3H). $^{13}$C-NMR (101 MHz, DMSO-$d_6$): $\delta$ 148.44 (s), 144.91 (s), 142.89 (s), 139.64 (s), 138.73 (s), 132.17 (s), 131.86 (s), 131.23 (s), 130.75 (s), 123.79 (s), 120.18 (s), 25.42 (s), 25.07 (s). MS (HR-ESI) $m/z$, calcd. For [C$_{12}$H$_9$BrN$_4$ + H$^+$] 289.0083, found 289.0087.

API intermediates 4: 1-chloro-7-(1-methyl-1H-indol-5-yl)-isoquinoline

White solid. $^1$H-NMR (400 MHz, DMSO-$d_6$): $\delta$ 8.43 (s, 1H), 8.33 - 8.23 (m, 2H), 8.15 (d, $J = 8.6$ Hz, 1H), 8.04 (d, $J = 1.0$ Hz, 1H), 7.92 (d, $J = 5.5$ Hz, 1H), 7.63 (dt, $J = 16.8$, 5.1 Hz, 2H), 7.42 (d,
\[ J = 3.0 \text{ Hz, 1H}, 6.57 (d, J = 2.8 \text{ Hz, 1H}), 3.86 (s, 3H) \]. \(^{13}\text{C-NMR (100 MHz, DMSO-} \text{d}_6)\): \( \delta \) 150.57, 142.94, 141.47, 136.93, 136.56, 131.76, 131.28, 130.43, 129.25, 128.50, 127.05, 122.17, 121.60, 121.10, 119.78, 111.00, 101.61, 33.10. MS (HR-ESI) \( m/z \), calcd. For \([\text{C}_{18}\text{H}_{13}\text{ClN}_2 + \text{H}^+]\) 293.0840, found 293.0874.

**API intermediates 5: tert-Butyl 3-(3-methylpyridin-2-yl)benzoate**

White solid. \(^1\text{H-NMR (400 MHz, DMSO-} \text{d}_6): \delta 8.52 (dd, J = 4.6, 1.1 \text{ Hz, 1H}), 8.05 (t, J = 1.6 \text{ Hz, 1H}), 7.99 - 7.93 (m, 1H), 7.82 (dd, J = 6.5, 1.2 \text{ Hz, 1H}), 7.76 (d, J = 7.7 \text{ Hz, 1H}), 7.60 (t, J = 7.7 \text{ Hz, 1H}), 7.34 (dd, J = 7.7, 4.7 \text{ Hz, 1H}), 2.33 (s, 3H), 1.56 (s, 9H). \(^{13}\text{C-NMR (100 MHz, DMSO-} \text{d}_6): \delta 165.26, 157.13, 147.48, 141.02, 139.29, 133.72, 131.75, 131.09, 129.98, 128.90, 128.79, 123.19, 81.36, 28.23, 20.06. MS (HR-ESI) \( m/z \), calcd. For \([\text{C}_{17}\text{H}_{19}\text{NO}_2 + \text{H}^+]\) 270.1489, found 270.1495.
15. $^1$H NMR, $^{13}$C NMR and HRMS spectra of API intermediates

Fig. S66. The $^1$H NMR spectrum of intermediate 1
Fig. S67. The $^{13}$C NMR spectrum of intermediate 1

Chemical Formula: C$_4$H$_7$N
Exact Mass: 193.09
Solvent: DMSO-$d_6$
Fig. S68. The HRMS spectrum of intermediate 1.
Fig. S69. The $^1$H NMR spectrum of intermediate 2

Chemical Formula: C$_{14}$H$_{13}$F$_3$O$_3$
Exact Mass: 310.06
Solvent: CDCl$_3$
Fig. S70. The $^{13}$C NMR spectrum of intermediate 2
Fig. S71. The HRMS spectrum of intermediate 2
Fig. S72. The $^1$H NMR spectrum of intermediate 3
Fig. S73. The $^{13}$C NMR spectrum of intermediate 3
Fig. S74. The HRMS spectrum of intermediate 3
Fig. S75. The $^1$H NMR spectrum of intermediate 4

Chemical Formula: $C_{13}H_{12}ClN_2$

Exact Mass: 292.08

Solvent: DMSO-$_d$6
Fig. S76. The $^{13}$C NMR spectrum of intermediate 4

Chemical Formula: $C_{14}H_{13}ClN_2$
Exact Mass: 292.08
Solvent: DMSO-$d_6$
Fig. S77. The HRMS spectrum of intermediate 4
Fig. S78. The $^1$H NMR spectrum of intermediate 5
Fig. S79. The $^{13}$C NMR spectrum of intermediate 5
Fig. S80. The HRMS spectrum of intermediate 5
16. Comparison of the results obtained from Fe₃O₄@FSM@Pd and those reported with other Pd catalyst

Table S2. Performance comparison of different catalysts

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<th>Catalyst</th>
<th>Reaction temperature (°C)</th>
<th>Solvent</th>
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<th>Base usage</th>
<th>Reaction time (h)</th>
<th>Catalyst usage (mol %)</th>
<th>Yield (%)</th>
<th>TON</th>
<th>TOF</th>
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¹ The catalyst Fe₃O₄@FSM@Pd was exposed to air for 6 months.

17. References