

Na₂S₂O₈ 介导的咪唑并[1,2-*α*]吡啶与杂芳胺在无金属条件下的 C—H 胺化反应

孙一平^a 陈德茂^b 何玲^{*,c} 王祖利^{*,b}

(^a 中国科学院兰州化学物理研究所 固体润滑国家重点实验室 兰州 730000)

(^b 青岛农业大学化学与药学院 青岛 266109)

(^c 重庆大学药学院创新药物研究中心 天然产物合成与药物研究重庆重点实验室 重庆 401331)

摘要 发展了一种高效实用的氨化咪唑[1,2-*α*]吡啶类化合物的方法。值得注意的是,该反应无需任何金属催化剂参与。在中等至高收率下,成功得到了带有多样取代基的产物。

关键词 咪唑[1,2-*α*]吡啶; 过硫酸盐; 杂芳胺; 绿色有机合成

Na₂S₂O₈ Mediated C—H Amination of Imidazo[1,2-*α*]pyridines with Heteroaromatic Amines under Metal-Free Conditions

Sun, Yiping^a Chen, Demao^b He, Ling^{*,c} Wang, Zuli^{*,b}

(^a State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000)

(^b College of Chemistry and Pharmaceutical Sciences, Qingdao Agricultural University, Qingdao 266109)

(^c Chongqing Key Laboratory of Natural Product Synthesis and Drug Research, Innovative Drug Research Center, School of Pharmaceutical Sciences, Chongqing University, Chongqing 401331)

Abstract A highly efficient and practical amination protocol for imidazo[1,2-*α*]pyridines using heteroaromatic amines was developed. Notably, the transformation proceeds without the need for any metal catalyst. Isolation of products bearing diverse substituents was achieved in moderate to high yields.

Keywords imidazo[1,2-*α*]pyridines; persulfates; heteroaromatic amines; green organic synthesis

1 Introduction

Imidazo[1,2-*α*]pyridines as the core structural skeletal framework of many natural products and pharmaceuticals,^[1] have attracted considerable interest due to their important biologically active properties. Therefore, developing efficient and facile method for the synthesis of diverse imidazo[1,2-*α*]pyridine derivatives has become an attractive research topic. At present, the construction of C—C,^[2] C—N,^[3] C—O,^[4] C—F,^[5] C—X^[6] and C—S^[7] bonds at the C-3 position of the imidazopyridine ring has been realized. In 2017, the visible light-induced regioselective sulfonamidation of imidazo[1,2-*α*]pyridines was developed by Sun's group (Scheme 1a).^[8] In 2020, Lei's group^[3a] realized an electrochemical intermolecular oxida-

tive C—H/N—H cross-coupling of imidazo[1,2-*α*]pyridines with *N*-nucleophiles (Scheme 1b). The PhI(OAc)₂-mediated oxidative C—H sulfoximation of imidazopyridines under mild conditions was presented by Wu's group (Scheme 1c).^[9] In these reactions, metal catalyst was sometimes needed to promote animation, which was not eco-friendly. As a consequence, developing an alternative synthetic method for construction of C-3 aminated imidazo[1,2-*α*]pyridines is still desirable.

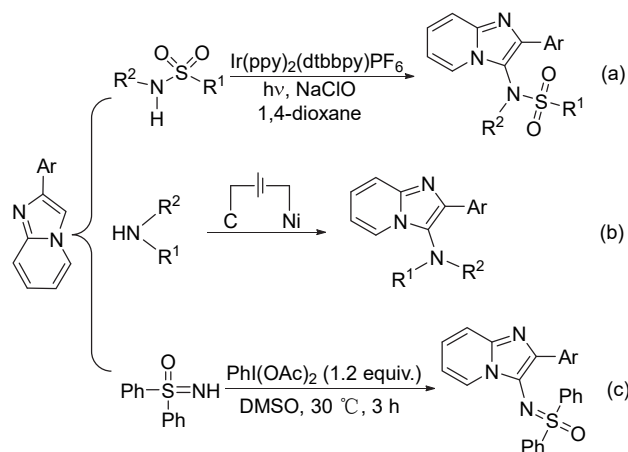
Because of its readily available, environmental friendliness and easy operation, persulfates have been widely used in the field of organic synthesis.^[10] Persulfate is relatively stable, but it can be easily decomposed into a more active sulfate radical negative ion by mean of light, heating or transition metal reduction. At present, free radical cycliza-

* Corresponding authors. E-mail: wangzulichem@163.com, heling2015@cqu.edu.cn

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 Scheme 1 Amination of imidazo[1,2-*a*]pyridines

tion,^[11] C—H bond functionalization,^[12] and other reactions^[13] have been presented in the presence of persulfates. However, compared with other types of free radical reactions,^[14] reactions promoted by persulfate have fewer reaction types and lower reaction efficiency. With our continuous efforts in radical reaction and C—H bond functionalization^[15] and radical chemistry,^[16] herein, we demonstrated the Na₂S₂O₈ promoted amination reaction of imidazo[1,2-*a*]pyridines with triazoles.

2 Results and discussion

At the outset of our investigation, a model reaction of 2-phenylimidazo[1,2-*a*]pyridine and 1*H*-benzo[*d*][1,2,3]-triazole was chosen to examine the reaction conditions. Solvent screening showed that dichloromethane (DCM)/H₂O was superior to other solvents, delivering the corresponding product in 91% yield (Table 1, Entry 7). Products with moderate yields of 42%~76% were isolated when other solvents such as MeCN/H₂O, CHCl₃/H₂O, 1,2-dichloroethane (DCE)/H₂O, acetone/H₂O and *o*-DCB/H₂O were used (Table 1, Entries 1~6). We then turn our attention to investigate the effect of oxidant on this reaction. Slight lower yields of the product were obtained when K₂S₂O₈ and (NH₄)₂S₂O₈ were used (Table 1, Entries 8~9). When dibenzoyl peroxide (BPO) was used as oxidant, only 42% yield of the product was isolated (Table 1, Entry 16). Other oxidants were not suitable for this reaction (Table 1, Entries 10~15). The reaction was no significant change on the yield when the reaction temperature was elevated to 120 °C (Table 1, Entry 19). But lowering the temperature to 80 °C resulted in a slightly lower yield than 85% (Table 1, Entry 18).

After completion of the search for the optimized reaction conditions, we set out to examine the scope and limitations of this system. Triazole substituted with methyl group at different positions proceeded smoothly to give the desired products with moderate to high yields (C1~C3). When pyridine ring of imidazopyridines was substituted with CH₃ or Cl group at different position, the reaction could also react efficiently (C4~C10). The effect of substituents on the phenyl group moiety of the imida-

 Table 1 Screening the optimized reaction conditions^a

Entry	Oxidant	Solvent	Yield ^b /%
1	Na ₂ S ₂ O ₈	DMF/H ₂ O	42
2	Na ₂ S ₂ O ₈	MeCN/H ₂ O	76
3	Na ₂ S ₂ O ₈	CHCl ₃ /H ₂ O	65
4	Na ₂ S ₂ O ₈	DCE/H ₂ O	61
5	Na ₂ S ₂ O ₈	Acetone/H ₂ O	49
6	Na ₂ S ₂ O ₈	<i>o</i> -DCB/H ₂ O	59
7	Na ₂ S ₂ O ₈	DCM/H ₂ O	91
8	K ₂ S ₂ O ₈	DCM/H ₂ O	83
9	(NH ₄) ₂ S ₂ O ₈	DCM/H ₂ O	78
10	PIDA	DCM/H ₂ O	N.R.
11	PIFA	DCM/H ₂ O	N.R.
12	TBHP	DCM/H ₂ O	Trace
13	DTBP	DCM/H ₂ O	N.R.
14	DCP	DCM/H ₂ O	N.R.
15	TBPB	DCM/H ₂ O	N.R.
16	BPO	DCM/H ₂ O	42
18 ^c	Na ₂ S ₂ O ₈	DCM/H ₂ O	78
19 ^d	Na ₂ S ₂ O ₈	DCM/H ₂ O	85

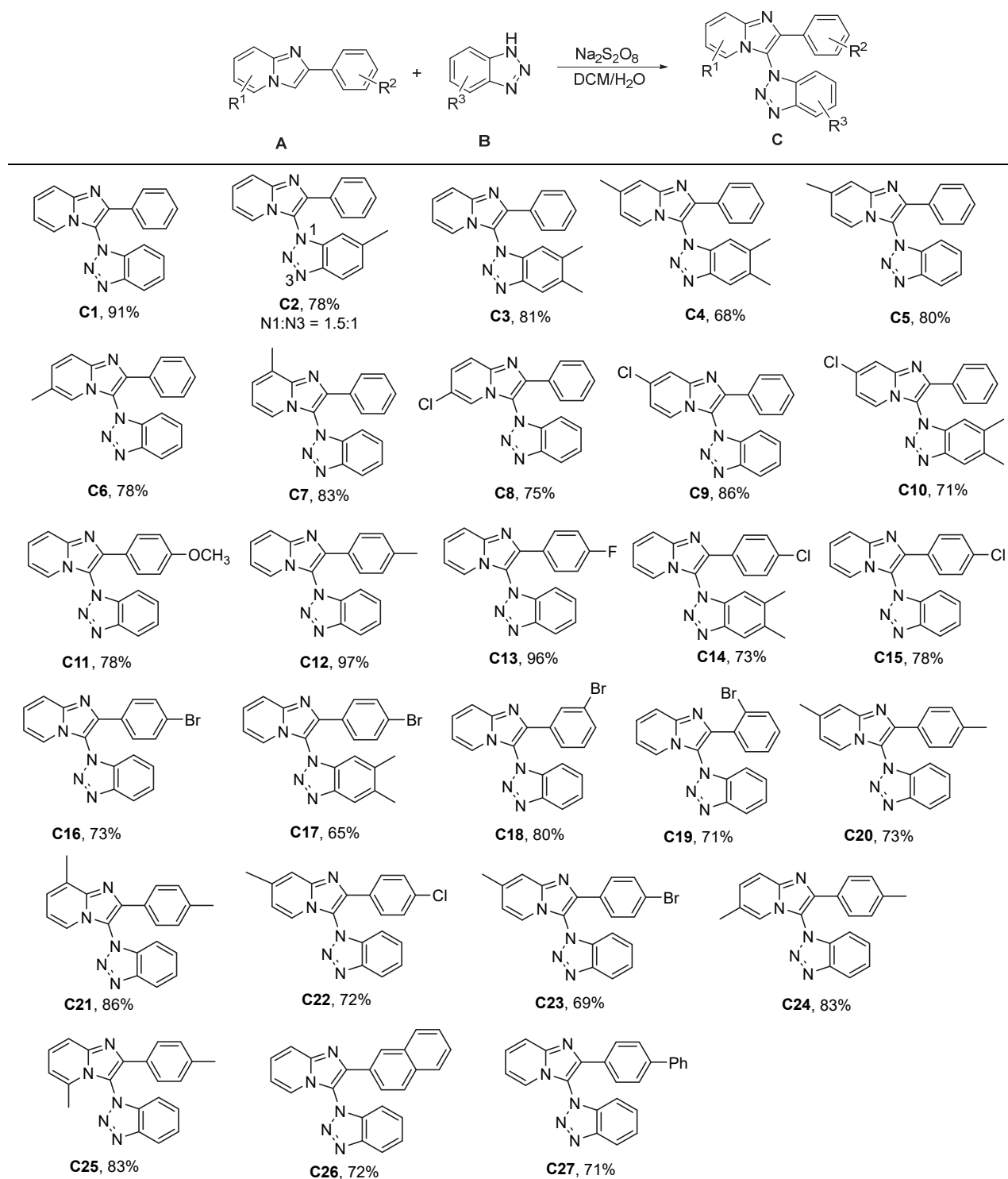
^a Reaction conditions: **A1** (0.2 mmol), **B1** (0.4 mmol), oxidant (3 equiv.), solvent (*V*: *V* = 1 : 1, 4 mL) at 100 °C for 15 h. ^b Isolated yield. ^c At 80 °C. ^d At 120 °C.

zopyridines was also investigated. It was found that electron-withdrawing or electron-donating groups, such as bromo, fluoro, chloro and methyl, on the phenyl ring at different position were well tolerated in this system (C11~C19). To our delight, when there are substituents on both pyridine ring and phenyl ring of imidazopyridines, triazole could react smoothly with imidazopyridines to afford products with 69%~86% isolated yields (C20~C25). It is worth noting that polyphenyl-substituted substrates were also proved to be good substrates, which could deliver the corresponding products with moderate yields (C26~C27).

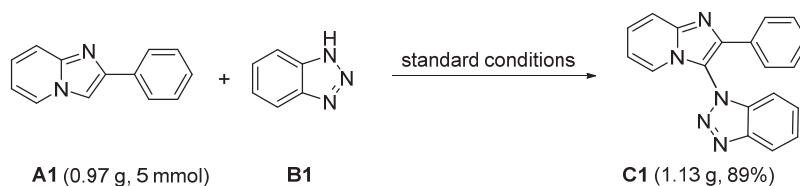
In order to prove the practicability of the reaction, a large-scale synthesis of C1 was performed. As illustrated in Scheme 2, a reaction of 2-phenylimidazo[1,2-*a*]pyridine and 1*H*-benzo[*d*][1,2,3]triazole was conducted on a 5 mmol scale under the standard conditions, and the corresponding product was isolated in 84% yield (Scheme 2). There was no significant decrease on the yield.

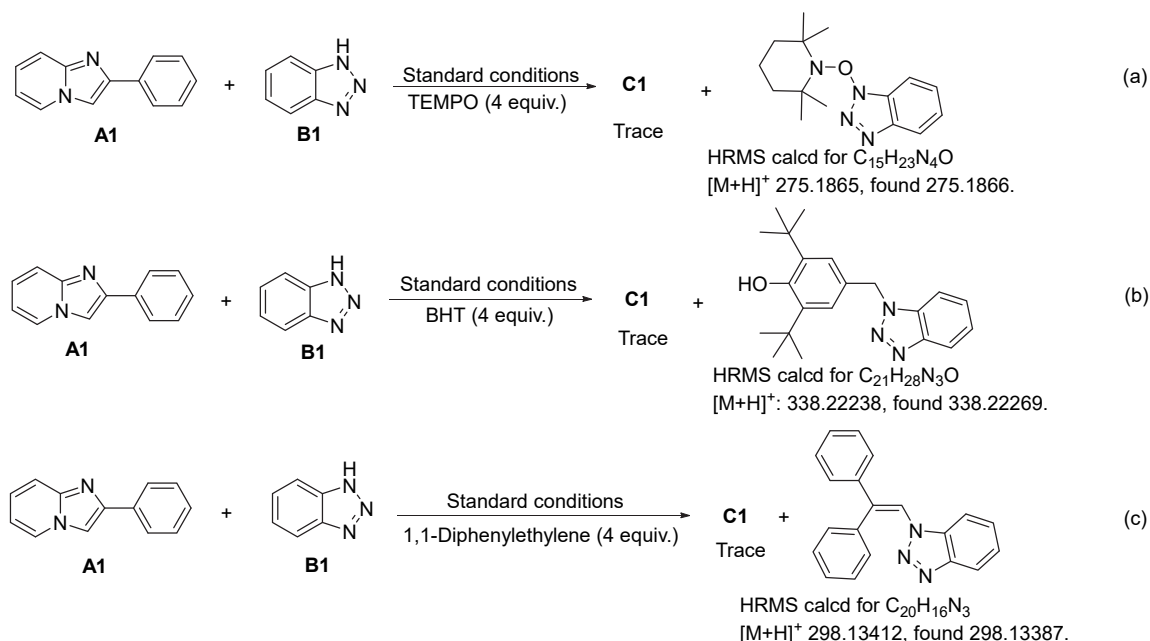
Several control experiments were conducted to examine the reaction mechanism. When 2,2,6,6-tetramethylpiperidinoxy (TEMPO), 2,6-di-*t*-butyl-*p*-cresol (butylated hydroxytoluene) (BHT) or 1,1-diphenylethylene was added into the reaction system, the current reaction was completely inhibited (Scheme 3). These results indicated that radical pathway was possible for this system.

On the basis of the above-mentioned observations and

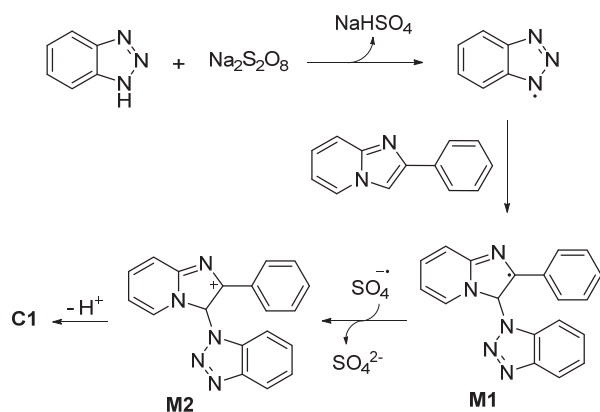
Table 2 Substrates scope of C—H amination of imidazo[1,2-*a*]pyridines

^a Reaction conditions: **A** (0.2 mmol), **B** (0.4 mmol), Na₂S₂O₈ (3 equiv.), DCM/H₂O (*V* : *V* = 1 : 1, 4 mL) at 100 °C for 15 h. ^b Isolated yield.

Scheme 2 Large-scale synthesis of **C1**



Scheme 3 Preliminary mechanistic studies

Scheme 4 Possible mechanism of C—H amination of imidazo[1,2-*a*]pyridines

related references,^[10a,10e,17-19] a probable reaction mechanism is outlined in Scheme 4. At the beginning, nitrogen radical was generated from the oxidation reaction of 1*H*-benzo[*d*][1,2,3]triazole with Na₂S₂O₈. Then the addition reaction of nitrogen radical to 2-phenylimidazo[1,2-*a*]pyridine occurred to provide alkyl radical intermediate **M1**, which was converted to **M2** via oxidation. Finally, the desired product was obtained via deportation reaction of **M2**.

3 Conclusions

In conclusion, an efficient and practical amination of imidazo[1,2-*a*]pyridines with heteroaromatic amines was established. No metal was needed for this transformation. Products with various substituents were isolated in moderate to high yields. Further synthetic application and detailed mechanistic studies of this methodology are currently ongoing in our laboratory.

4 Experimental section

4.1 Instruments and reagents

NMR spectra were recorded on a BRUKER AVANCE III HD 500 MHz spectrometers, operating at 500 MHz for ¹H NMR and at 126 MHz for ¹³C NMR acquisitions. High-resolution mass spectrometry (HRMS) measurements were performed on an Orbitrap Fusion Lumos. All major chemicals and solvents were obtained from commercial sources and used without further purification.

4.2 General procedure for the synthesis of **C1**

A sealable reaction tube equipped with a magnetic stirrer bar was charged with 2-phenylimidazo[1,2-*a*]pyridine (0.2 mmol), 1*H*-benzo[*d*][1,2,3]triazole (2 equiv.), Na₂S₂O₈ (3 equiv.), DCM/H₂O (*V*:*V*=1:1, 4 mL). The mixture reacted under air atmosphere at 100 °C for 15 h. After completion, it was diluted with ethyl acetate. After the solvent was removed under reduced pressure, the residue was purified by column chromatography on silica gel to afford the corresponding product.

4.3 Characterization of the compounds

1-(2-Phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C1**): The pure product was obtained as a yellow solid (33.2 mg, 53%). ¹H NMR (500 MHz, CDCl₃) δ: 8.26 (d, *J*=8.5 Hz, 1H), 7.80 (d, *J*=9.1 Hz, 1H), 7.53~7.43 (m, 5H), 7.41~7.36 (m, 1H), 7.25 (d, *J*=6.7 Hz, 3H), 7.14 (d, *J*=7.0 Hz, 1H), 6.86 (t, *J*=6.7 Hz, 1H); ¹³C NMR (126 MHz, DMSO) δ: 145.7, 143.9, 140.8, 134.5, 132.0, 130.3, 129.3, 128.0, 126.7, 125.8, 123.9, 120.7, 117.8, 114.7, 112.0, 110.5. HRMS (ESI) calcd for C₁₉H₁₄N₅ [*M*+*H*]⁺ 312.1244, found 312.1246.

6-Methyl-1-(2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C2**)/5-methyl-1-(2-phenylimid-

azo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C2'**): Isomer $n(\mathbf{C2'}) : n(\mathbf{C2'}) = 1.4 : 1$ (based on NMR). The pure product was obtained as a white solid (31.5 mg, 48%). ¹H NMR (500 MHz, CDCl₃) δ : 8.11 (d, $J=8.5$ Hz, 1H), 8.00 (s, 1H), 7.79 (d, $J=9.1$ Hz, 2H), 7.56~7.47 (m, 5H), 7.44 (d, $J=6.8$ Hz, 1H), 7.40~7.34 (m, 2H), 7.30 (dd, $J=8.3, 4.8$ Hz, 2H), 7.26~7.22 (m, 5H), 7.02 (d, $J=8.4$ Hz, 1H), 6.91 (s, 1H), 6.85 (t, $J=6.7$ Hz, 2H), 2.55 (s, 2H), 2.40 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 146.4, 144.4, 144.0, 141.5, 140.4, 135.3, 134.6, 132.5, 131.7, 131.3, 128.7, 127.3, 127.1, 126.7, 122.4, 120.6, 120.2, 119.8, 118.0, 113.6, 112.3, 109.2, 108.9, 21.9, 21.5. HRMS (ESI) calcd for C₂₀H₁₆N₅ [M+H]⁺ 326.1399, found 326.1400.

5,6-Dimethyl-1-(2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C3**): The pure product was obtained as a white solid (30.6 mg, 45%). ¹H NMR (500 MHz, CDCl₃) δ : 7.97 (s, 1H), 7.78 (d, $J=9.1$ Hz, 1H), 7.56~7.49 (m, 2H), 7.42 (d, $J=6.8$ Hz, 1H), 7.38~7.32 (m, 1H), 7.25 (dd, $J=7.3, 5.6$ Hz, 3H), 6.91 (s, 1H), 6.82 (t, $J=6.8$ Hz, 1H), 2.44 (s, 3H), 2.30 (d, $J=10.6$ Hz, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.0, 144.0, 141.4, 140.0, 134.9, 133.2, 131.7, 128.7, 127.1, 126.5, 122.4, 119.7, 118.0, 113.5, 112.5, 109.1, 20.8, 20.4. HRMS (ESI) calcd for C₂₁H₁₈N₅ [M+H]⁺ 340.1555, found 340.1556.

5,6-Dimethyl-1-(7-methyl-2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C4**): The pure product was obtained as a white solid (22.6 mg, 32%). ¹H NMR (500 MHz, CDCl₃) δ : 7.96 (s, 1H), 7.55~7.47 (m, 3H), 7.29 (d, $J=6.9$ Hz, 1H), 7.25~7.20 (m, 3H), 6.90 (s, 1H), 6.65 (d, $J=6.9$ Hz, 1H), 2.45 (s, 3H), 2.44 (s, 3H), 2.28 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.0, 144.4, 141.1, 139.9, 137.7, 134.9, 133.2, 131.8, 128.6, 127.0, 121.6, 119.7, 116.4, 116.1, 112.0, 109.1, 21.4, 20.8, 20.4. HRMS (ESI) calcd for C₂₂H₂₀N₅ [M+H]⁺ 354.1711, found 354.1713.

1-(7-Methyl-2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C5**): The pure product was obtained as a white solid (26.1 mg, 40%). ¹H NMR (500 MHz, CDCl₃) δ : 8.24 (d, $J=1.7$ Hz, 1H), 7.54 (s, 1H), 7.48 (d, $J=2.1$ Hz, 4H), 7.37 (d, $J=3.3$ Hz, 1H), 7.23 (s, 3H), 7.13 (s, 1H), 6.68 (s, 1H), 2.45 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.5, 141.1, 138.0, 134.1, 131.7, 129.2, 128.7, 127.0, 125.0, 121.6, 120.6, 116.3, 111.7, 109.8, 21.4. HRMS (ESI) calcd for C₂₀H₁₆N₅ [M+H]⁺ 326.1398, found 326.1400.

1-(6-Methyl-2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C6**): The pure product was obtained as a white solid (24.4 mg, 38%). ¹H NMR (500 MHz, CDCl₃) δ : 8.25 (d, $J=7.0$ Hz, 1H), 7.70 (d, $J=9.1$ Hz, 1H), 7.51~7.42 (m, 4H), 7.28 (s, 1H), 7.22 (d, $J=3.0$ Hz, 4H), 7.14 (dd, $J=6.1, 1.5$ Hz, 1H), 2.23 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 143.1, 141.1, 134.1, 131.6, 130.67, 130.2, 129.9, 128.9, 126.9, 125.0, 123.7, 120.6, 120.0, 117.3, 111.8, 109.8, 18.2. HRMS (ESI) calcd for C₂₀H₁₆N₅ [M+H]⁺ 326.1398, found 326.1400.

1-(8-Methyl-2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C7**): The pure product was ob-

tained as a white solid (31.3 mg, 48%). ¹H NMR (500 MHz, CDCl₃) δ : 8.23 (d, $J=6.3$ Hz, 1H), 7.53 (s, 2H), 7.49~7.39 (m, 2H), 7.33 (d, $J=5.3$ Hz, 1H), 7.28~7.19 (m, 3H), 7.17~7.07 (m, 2H), 6.79~6.67 (m, 1H), 2.76 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.4, 140.9, 134.1, 131.8, 129.2, 128.7, 128.7, 128.1, 127.2, 125.3, 125.1, 120.6, 120.4, 113.6, 112.5, 109.8, 16.7. HRMS (ESI) calcd for C₂₀H₁₆N₅ [M+H]⁺ 326.1398, found 326.1400.

1-(6-Chloro-2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C8**): The pure product was obtained as a white solid (31.2 mg, 45%). ¹H NMR (500 MHz, CDCl₃) δ : 8.26 (d, $J=7.5$ Hz, 1H), 7.73 (d, $J=9.6$ Hz, 1H), 7.57 (s, 1H), 7.48 (ddd, $J=18.9, 12.9, 7.4$ Hz, 4H), 7.34 (dd, $J=9.5, 0.9$ Hz, 1H), 7.24 (dd, $J=16.9, 9.4$ Hz, 3H), 7.13 (d, $J=7.5$ Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 142.4, 142.1, 133.8, 131.1, 130.2, 129.9, 129.5, 128.9, 128.2, 127.0, 125.2, 122.1, 121.4, 120.9, 120.6, 118.5, 112.6, 109.7. HRMS (ESI) calcd for C₁₉H₁₃N₅Cl [M+H]⁺ 346.0852, found 346.0854.

1-(7-Chloro-2-phenylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C9**): The pure product was obtained as a white solid (31.4 mg, 46%). ¹H NMR (500 MHz, CDCl₃) δ : 8.24 (d, $J=7.3$ Hz, 1H), 7.78 (s, 1H), 7.46 (d, $J=7.3$ Hz, 5H), 7.23 (d, $J=7.2$ Hz, 3H), 7.12 (d, $J=7.5$ Hz, 1H), 6.84 (d, $J=7.1$ Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 143.8, 142.0, 133.9, 133.4, 131.1, 130.2, 129.7, 129.5, 128.9, 127.1, 125.2, 122.9, 120.7, 116.9, 115.3, 112.5, 109.7. HRMS (ESI) calcd for C₁₉H₁₃N₅Cl [M+H]⁺ 346.0851, found 346.0854.

1-(7-Chloro-2-phenylimidazo[1,2-*a*]pyridin-3-yl)-5,6-dimethyl-1*H*-benzo[*d*][1,2,3]triazole (**C10**): The pure product was obtained as a yellow solid (23.2 mg, 31%). ¹H NMR (500 MHz, CDCl₃) δ : 7.97 (s, 1H), 7.78 (d, $J=1.1$ Hz, 1H), 7.49 (dd, $J=7.4, 1.8$ Hz, 2H), 7.37 (d, $J=7.2$ Hz, 1H), 7.26 (d, $J=11.4$ Hz, 3H), 6.89 (s, 1H), 6.82 (dd, $J=7.2, 1.8$ Hz, 1H), 2.44 (s, 3H), 2.29 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.0, 143.7, 142.1, 140.2, 135.1, 133.3, 133.0, 131.2, 129.0, 128.7, 127.1, 122.8, 119.8, 116.9, 115.2, 112.8, 109.0, 20.9, 20.4. HRMS (ESI) calcd for C₂₁H₁₇N₅Cl [M+H]⁺ 374.1165, found 374.1167.

1-(2-(4-Methoxyphenyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C11**): The pure product was obtained as a white solid (25.8 mg, 38%). ¹H NMR (500 MHz, CDCl₃) δ : 8.25 (d, $J=7.3$ Hz, 1H), 7.76 (d, $J=9.0$ Hz, 1H), 7.60~7.40 (m, 5H), 7.38~7.33 (m, 1H), 7.15 (d, $J=5.5$ Hz, 1H), 6.89~6.72 (m, 3H), 3.74 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 160.0, 145.8, 144.0, 141.4, 134.0, 129.3, 128.4, 126.5, 125.0, 124.0, 122.3, 120.6, 117.8, 114.2, 113.4, 111.4, 109.8, 55.2. HRMS (ESI) calcd for C₂₀H₁₆ON₅ [M+H]⁺ 342.1347, found 342.1349.

1-(2-(*p*-Tolyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C12**): The pure product was obtained as a white solid (37.2 mg, 57%). ¹H NMR (500 MHz, CDCl₃) δ : 8.25 (d, $J=7.2$ Hz, 1H), 7.79 (d, $J=9.0$ Hz, 1H), 7.47 (s, 3H), 7.43~7.34 (m, 3H), 7.14 (d, $J=7.4$ Hz, 1H), 7.04 (d, $J=7.5$ Hz, 2H), 6.89~6.80 (m, 1H), 2.27 (s, 3H); ¹³C

NMR (126 MHz, CDCl₃) δ : 145.8, 144.0, 141.5, 138.9, 134.0, 130.2, 129.7, 129.4, 128.6, 127.0, 126.8, 125.0, 122.4, 120.6, 117.9, 113.6, 111.8, 109.8, 21.2. HRMS (ESI) calcd for C₂₀H₁₆N₅ [M + H]⁺ 326.1398, found 326.1400.

1-(2-(4-Fluorophenyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C13**): The pure product was obtained as a white solid (44.9 mg, 56%). ¹H NMR (500 MHz, CDCl₃) δ : 8.32~8.19 (m, 1H), 7.78 (d, *J*=9.1 Hz, 1H), 7.57~7.44 (m, 5H), 7.38 (ddd, *J*=9.1, 6.8, 1.2 Hz, 1H), 7.19~7.09 (m, 1H), 6.99~6.89 (m, 2H), 6.86 (td, *J*=6.8, 1.0 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ : 164.0, 162.0, 145.8, 144.0, 140.6, 133.9, 129.4, 128.9, 127.7, 126.8, 125.1, 122.5, 120.7, 118.0, 115.9, 115.7, 113.7, 111.9, 109.6. HRMS (ESI) calcd for C₁₉H₁₃N₅F [M + H]⁺ 330.1149, found 330.1149.

1-(2-(4-Chlorophenyl)imidazo[1,2-*a*]pyridin-3-yl)-5,6-dimethyl-1*H*-benzo[*d*][1,2,3]triazole (**C14**): The pure product was obtained as a white solid (24.3 mg, 33%). ¹H NMR (500 MHz, CDCl₃) δ : 7.98 (s, 1H), 7.77 (d, *J*=9.1 Hz, 1H), 7.48~7.40 (m, 3H), 7.40~7.35 (m, 1H), 7.22 (d, *J*=8.6 Hz, 2H), 6.89 (s, 1H), 6.84 (t, *J*=6.8 Hz, 1H), 2.45 (s, 3H), 2.31 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.0, 144.0, 140.3, 135.1, 134.7, 133.0, 130.2, 129.0, 128.3, 126.8, 122.5, 119.8, 118.0, 113.7, 112.5, 108.9, 20.9, 20.4. HRMS (ESI) calcd for C₂₁H₁₇N₅Cl [M + H]⁺ 374.1164, found 374.1167.

1-(2-(4-Chlorophenyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C15**): The pure product was obtained as a white solid (26.0 mg, 38%). ¹H NMR (500 MHz, CDCl₃) δ : 8.26 (d, *J*=7.5 Hz, 1H), 7.79 (d, *J*=9.1 Hz, 1H), 7.50 (d, *J*=7.3 Hz, 3H), 7.46~7.38 (m, 3H), 7.21 (d, *J*=7.9 Hz, 2H), 7.13 (d, *J*=5.4 Hz, 1H), 6.90~6.84 (m, 1H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.1, 140.3, 134.8, 133.9, 130.6, 130.0, 130.0, 129.2, 128.4, 126.9, 125.2, 122.5, 120.8, 118.1, 113.8, 109.6. HRMS (ESI) calcd for C₁₉H₁₃N₅Cl [M + H]⁺ 346.0852, found 346.0854.

1-(2-(4-Bromophenyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C16**): The pure product was obtained as a white solid (25.3 mg, 33%). ¹H NMR (500 MHz, CDCl₃) δ : 8.26 (d, *J*=8.3 Hz, 1H), 7.79 (d, *J*=9.1 Hz, 1H), 7.60~7.47 (m, 4H), 7.43~7.33 (m, 4H), 7.13 (d, *J*=4.9 Hz, 1H), 6.92~6.83 (m, 1H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.1, 137.9, 133.9, 131.9, 129.5, 128.6, 127.0, 125.2, 123.2, 122.5, 120.8, 118.1, 113.8, 109.6. HRMS (ESI) calcd for C₁₉H₁₃N₅Br [M + H]⁺ 390.0346, found 390.0348.

1-(2-(4-Bromophenyl)imidazo[1,2-*a*]pyridin-3-yl)-5,6-dimethyl-1*H*-benzo[*d*][1,2,3]triazole (**C17**): The pure product was obtained as a white solid (20.6 mg, 25%). ¹H NMR (500 MHz, CDCl₃) δ : 7.98 (s, 1H), 7.77 (d, *J*=9.1 Hz, 1H), 7.45~7.33 (m, 6H), 6.88 (d, *J*=11.8 Hz, 1H), 6.84 (t, *J*=6.8 Hz, 1H), 2.45 (s, 3H), 2.31 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.0, 144.0, 140.3, 137.8, 135.2, 133.0, 131.9, 130.6, 128.6, 126.8, 123.0, 122.5, 119.8, 118.0, 113.7, 112.6, 108.9, 20.9, 20.4. HRMS (ESI)

calcd for C₂₁H₁₇N₅Br [M + H]⁺ 418.0658, found 418.0661.

1-(2-(3-Bromophenyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C18**): The pure product was obtained as a yellow solid (31.0 mg, 40%). ¹H NMR (500 MHz, CDCl₃) δ : 8.31~8.20 (m, 1H), 7.93 (s, 1H), 7.79 (d, *J*=9.1 Hz, 1H), 7.58~7.46 (m, 3H), 7.39 (dd, *J*=16.6, 8.3 Hz, 2H), 7.13 (d, *J*=7.2 Hz, 2H), 7.02 (t, *J*=7.9 Hz, 1H), 6.87 (t, *J*=6.7 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.1, 139.8, 133.9, 133.7, 131.7, 130.2, 129.5, 127.0, 125.2, 123.1, 122.7, 120.8, 118.1, 113.9, 112.5, 109.6. HRMS (ESI) calcd for C₁₉H₁₃N₅Br [M + H]⁺ 390.0346, found 390.0348.

1-(2-(2-Bromophenyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C19**): The pure product was obtained as a yellow solid (24.0 mg, 31%). ¹H NMR (500 MHz, CDCl₃) δ : 8.25 (d, *J*=6.7 Hz, 1H), 7.80 (d, *J*=8.4 Hz, 1H), 7.49 (s, 4H), 7.39 (d, *J*=6.9 Hz, 1H), 7.25 (s, 3H), 7.14 (d, *J*=6.6 Hz, 1H), 6.86 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.1, 141.4, 134.0, 131.5, 129.3, 128.8, 127.1, 126.7, 125.0, 122.4, 120.7, 118.1, 113.6, 112.2, 109.8. HRMS (ESI) calcd for C₁₉H₁₂N₅Br [M] 389.0280, found 389.0270.

1-(7-Methyl-2-(*p*-tolyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C20**): The pure product was obtained as a white solid (29.1 mg, 43%). ¹H NMR (500 MHz, CDCl₃) δ : 8.24 (d, *J*=7.2 Hz, 1H), 7.57~7.44 (m, 3H), 7.36 (d, *J*=6.8 Hz, 3H), 7.13 (d, *J*=7.5 Hz, 1H), 7.03 (d, *J*=7.5 Hz, 2H), 6.66 (d, *J*=6.7 Hz, 1H), 2.45 (s, 3H), 2.27 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.5, 138.6, 137.8, 134.1, 130.0, 129.8, 129.20, 126.9, 125.0, 121.6, 120.6, 117.3, 116.6, 116.2, 109.8, 21.5, 21.2. HRMS (ESI) calcd for C₂₁H₁₈N₅ [M + H]⁺ 340.1555, found 340.1556.

1-(8-Methyl-2-(*p*-tolyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C21**): The pure product was obtained as a white solid (31.4 mg, 46%). ¹H NMR (500 MHz, CDCl₃) δ : 8.23 (d, *J*=7.9 Hz, 1H), 7.50~7.37 (m, 4H), 7.32 (d, *J*=6.7 Hz, 1H), 7.13 (t, *J*=7.2 Hz, 2H), 7.03 (d, *J*=7.8 Hz, 2H), 6.73 (t, *J*=6.8 Hz, 1H), 2.75 (s, 3H), 2.26 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.4, 141.1, 138.5, 134.1, 129.4, 129.2, 128.9, 128.1, 127.0, 125.2, 124.9, 120.6, 120.1, 113.5, 112.1, 109.8, 21.2, 16.7. HRMS (ESI) calcd for C₂₁H₁₈N₅ [M + H]⁺ 340.1554, found 340.1556.

1-(2-(4-Chlorophenyl)-7-methylimidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C22**): The pure product was obtained as a yellow solid (23.1 mg, 32%). ¹H NMR (500 MHz, CDCl₃) δ : 8.25 (dd, *J*=5.5, 3.7 Hz, 1H), 7.50 (dd, *J*=13.0, 9.2 Hz, 3H), 7.39 (dd, *J*=17.0, 7.8 Hz, 3H), 7.20 (d, *J*=8.5 Hz, 2H), 7.12 (dd, *J*=5.1, 3.4 Hz, 1H), 6.69 (d, *J*=6.9 Hz, 1H), 2.47 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ : 145.8, 144.5, 140.0, 138.2, 134.7, 134.0, 130.2, 129.4, 129.0, 128.2, 125.1, 121.6, 120.7, 116.4, 111.8, 109.6, 21.5. HRMS (ESI) calcd for C₂₀H₁₅N₅Cl [M + H]⁺ 360.1008, found 360.1010.

1-(2-(4-Bromophenyl)-7-methylimidazo[1,2-*a*]pyridin-

3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C23**): The pure product was obtained as a yellow solid (27.3 mg, 29%). ¹H NMR (500 MHz, CDCl₃) δ: 8.41~8.20 (m, 1H), 7.69~7.46 (m, 4H), 7.46~7.31 (m, 4H), 7.22~7.09 (m, 1H), 6.69 (d, *J*=6.1 Hz, 1H), 2.46 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ: 145.8, 144.5, 140.0, 138.3, 137.8, 134.0, 131.9, 130.7, 129.4, 128.5, 125.1, 123.0, 121.6, 120.7, 116.4, 111.8, 109.6, 21.5. HRMS (ESI) calcd for C₂₀H₁₅N₅Br [M+H]⁺ 404.0499, found 404.0505.

1-(6-Methyl-2-(*p*-tolyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C24**): The pure product was obtained as a white solid (29.0 mg, 43%). ¹H NMR (500 MHz, CDCl₃) δ: 8.18 (d, *J*=6.9 Hz, 1H), 7.63 (d, *J*=9.1 Hz, 1H), 7.49~7.37 (m, 2H), 7.32 (dd, *J*=13.1, 10.3 Hz, 2H), 7.18 (d, *J*=6.8 Hz, 1H), 7.14 (d, *J*=9.2 Hz, 1H), 7.11~7.04 (m, 1H), 6.95 (d, *J*=7.9 Hz, 2H), 2.18 (d, *J*=11.8 Hz, 6H); ¹³C NMR (126 MHz, CDCl₃) δ: 145.8, 143.1, 141.3, 138.7, 134.1, 129.9, 129.4, 128.7, 126.8, 125.0, 123.7, 120.6, 120.0, 117.2, 111.5, 109.9, 21.2, 18.2. HRMS (ESI) calcd for C₂₁H₁₈N₅ [M+H]⁺ 340.1554, found 340.1556.

1-(5-Methyl-2-(*p*-tolyl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C25**): The pure product was obtained as a white solid (29.4 mg, 43%). ¹H NMR (500 MHz, CDCl₃) δ: 8.21 (d, *J*=8.1 Hz, 1H), 7.69 (d, *J*=9.0 Hz, 1H), 7.48 (dt, *J*=14.9, 7.2 Hz, 2H), 7.28 (d, *J*=12.6 Hz, 3H), 7.20 (d, *J*=7.9 Hz, 1H), 6.99 (d, *J*=7.7 Hz, 2H), 6.57 (d, *J*=6.8 Hz, 1H), 2.24 (s, 3H), 1.74 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ: 145.7, 145.2, 143.5, 138.7, 137.3, 135.4, 129.5, 129.4, 128.8, 126.8, 124.9, 120.6, 116.1, 114.6, 111.8, 110.0, 21.2, 17.1. HRMS (ESI) calcd for C₂₁H₁₈N₅ [M+H]⁺ 340.1554, found 340.1556.

1-(2-(Naphthalen-2-yl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C26**): The pure product was obtained as a yellow solid (25.6 mg, 32%). ¹H NMR (500 MHz, CDCl₃) δ: 8.27 (d, *J*=8.2 Hz, 1H), 8.13 (s, 1H), 7.83 (d, *J*=9.1 Hz, 1H), 7.73 (dd, *J*=7.6, 5.4 Hz, 1H), 7.71~7.63 (m, 2H), 7.55 (d, *J*=6.8 Hz, 1H), 7.50~7.37 (m, 6H), 7.14 (d, *J*=8.1 Hz, 1H), 6.87 (td, *J*=6.8, 0.8 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ: 145.9, 144.2, 141.4, 134.1, 133.3, 129.4, 128.9, 128.5, 127.6, 126.8, 126.6, 126.3, 125.1, 124.2, 122.5, 120.7, 118.1, 113.7, 112.5, 109.8. HRMS (ESI) calcd for C₂₃H₁₆N₅ [M+H]⁺ 362.1399, found 362.1400.

1-(2-([1,1'-Biphenyl]-4-yl)imidazo[1,2-*a*]pyridin-3-yl)-1*H*-benzo[*d*][1,2,3]triazole (**C27**): The pure product was obtained as a yellow solid (24.9 mg, 31%). ¹H NMR (500 MHz, CDCl₃) δ: 8.31~8.22 (m, 1H), 7.81 (d, *J*=9.1 Hz, 1H), 7.52 (ddd, *J*=18.7, 13.9, 8.5 Hz, 9H), 7.39 (t, *J*=7.5 Hz, 3H), 7.31 (t, *J*=7.2 Hz, 1H), 7.22~7.15 (m, 1H), 6.86 (t, *J*=6.8 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ: 145.9, 141.9, 141.4, 141.3, 140.2, 134.0, 130.4, 129.4, 128.7, 128.1, 127.3, 127.3, 127.1, 126.8, 125.1, 122.4, 120.7, 118.1, 113.6, 109.8. HRMS (ESI) calcd for C₂₅H₁₈N₅ [M+H]⁺ 388.1556, found 388.1556.

Supporting Information ¹H NMR and ¹³C NMR spectra of compounds. The Supporting Information is available free of charge via the Internet at <http://sioc-journal.cn>.

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