Regular Article

$ZnCl_2$-Promoted Intramolecular Hetero-Diels–Alder Reaction of *o*-Alkynylphenylcarbodiimides for Synthesis of Dihydrodibenzo[*b*,*g*][1,8]-naphthyridines

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The $ZnCl_2$-promoted intramolecular hetero-Diels–Alder reaction of *N*-(*ortho*-propargylphenyl)-*N*′-arylcarbodiimides, in which the aryl-N=C moiety functioned as a 2-azabuta-1,3-diene, 4π component, has been achieved. By this method, very rare 5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridines and fully aromatized dibenzo[*b*,*g*][1,8]naphthyridines were successfully synthesized.

Key words hetero-Diels–Alder; carbodiimide; naphthyridine; zinc chloride; heterocycle; one-pot

Indoloquinoline alkaloids are biologically attractive compounds because they have potential uses as antifungal,[1,2] antibacterial,[3] antimalarial,[4,5] anticancer,[6–8] anti-inflammatory,[9] and DNA intercalating agents,[10–15] as well as inhibitors of topoisomerase II.[16,17] The intramolecular hetero-Diels–Alder (HDA) reaction is a powerful synthetic method for such heterocyclic compounds. For example, the intramolecular aza-DA reactions of the carbon–carbon double or triple bond-conjugated diarylcarbodiimides under thermal conditions have been reported for the synthesis of indolo[2,3-*b*]quinoline derivatives **1** by the Saito and Motoki,[18,19] Molina and Alajarín,[20–22] Wang,[23–26] Pieters,[27] and Schmittel[28,29] groups (Chart 1, Eq. 1). Furthermore, Saito *et al.* previously reported the Lewis acid-controlled, highly periselective intramolecular aza-DA reactions of the *N*-arylcarbodiimides with an inner carbon–carbon double bond dienophile, depressing the formation of by-products 2-aminoquinolines **2** *via* 6π-electrocyclization.[19] Therefore, the Lewis acid-promoted reaction under mild conditions offers a more effective method for the synthesis of the intramolecular HDA products[30] than obtained with thermal reactions. Based on this background of research, we envisioned that the Lewis acid-promoted reaction of *N*-(*ortho*-propargylphenyl)-*N*′-arylcarbodiimides **3**[31–34] would enable a facile synthetic approach to the 5,12-dihydrodibenzo[*b,g*][1,8]-naphthyridine derivatives **4**[35–39] (Chart 1, Eq. 2). Herein, we describe the results in detail.

We initially screened various Lewis and Brønsted acids using **3a** and **d** as model substrates. The selected data are shown in Table 1. As a result, the reactions of **3a** and **d** in the presence of 1.0 eq of zinc chloride (1.0 M in diethyl ether) at room temperature gave the best results; 11-phenyl- and 11-propyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine **4a** and **m** were

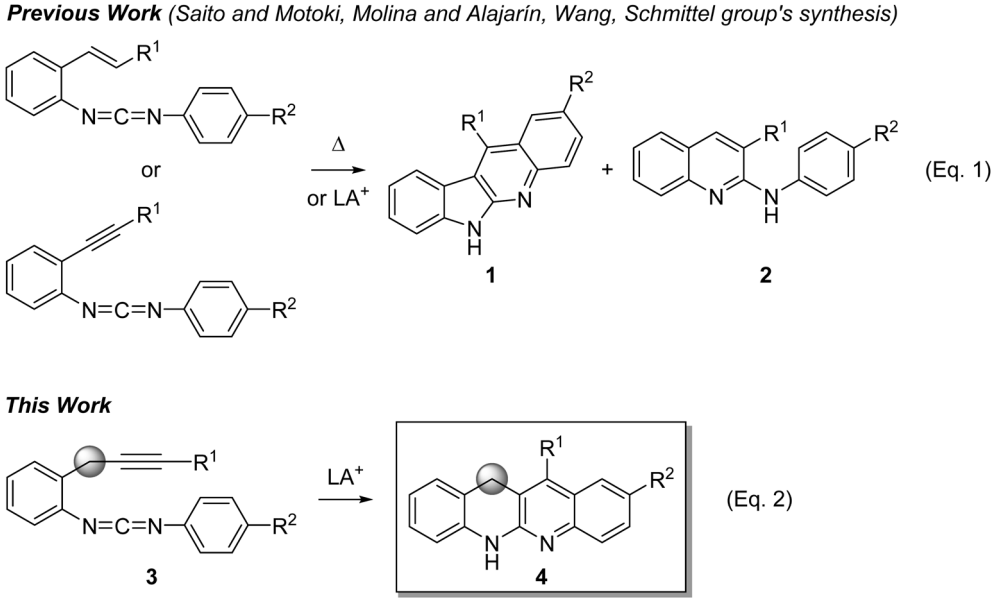

Chart 1. Intramolecular Ring-Forming Reactions of the Carbon–Carbon Double or Triple Bond Bearing Diarylcarbodiimides under Thermal or Lewis Acid Conditions

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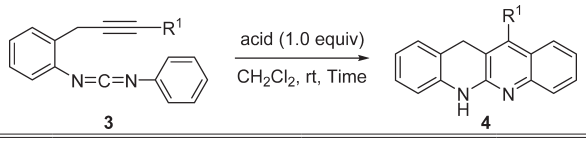
Table 1. Screening of Acids



3 4

Entry	R^1 (S.M.)	Acids	Time (h)	Product	Yield (%)[a]
1	Ph (**3a**)	$AlCl_3$	2.0	**4a**	30
2	Ph (**3a**)	$TiCl_4$	1.0	**4a**	35
3	Ph (**3a**)	AgOTf	6.0	**4a**	55
4	Ph (**3a**)	TfOH	1.0	**4a**	66
5	^{n}Pr (**3d**)	TfOH	3.0	**4m**	8
6	Ph (**3a**)	$BF_3 \cdot OEt_2$	2.5	**4a**	71
7	^{n}Pr (**3d**)	$BF_3 \cdot OEt_2$	3.0	**4m**	40
8	Ph (**3a**)	$ZnCl_2$	2.5	**4a**	73
9	^{n}Pr (**3d**)	$ZnCl_2$	48.0	**4m**	67
10	Ph (**3a**)	Δ[b]	4.0	**4a**	65

a) Yields of isolated **4**. *b*) Reflux in toluene.

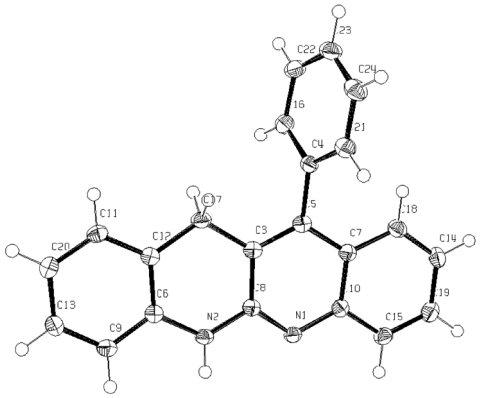


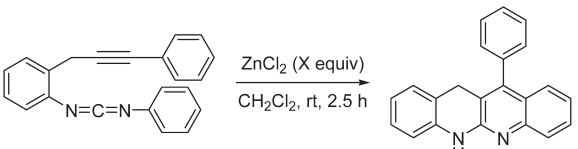
Fig. 1. X-Ray Structure of 11-Phenyl-5,12-dihydrodibenzo[*b*,*g*][1,8]-naphthyridine **4a**

satisfactorily obtained in 73 and 67% yields (Table 1, Entries 8, 9). The both reaction systems were relatively clear, though there was a difference in their reaction times. The thermal reaction was also performed for comparisons. However, the yield did not exceed 73% (Entry 8 *vs.* Entry 10). The chemical structures of **4a** and **m** were determined by using ^{1}H- and ^{13}C-NMR spectroscopic analysis and X-ray crystallographic analysis of **4a**[40] (Fig. 1).

Next, we examined appropriate amounts of zinc chloride in this HDA reaction of **3a** as shown in Table 2. When 0.2 eq of zinc chloride were used, the reaction proceeded very slowly at room temperature and 25% of the starting material **3a** remained even after 24 h (Table 2, Entry 1). Treatment of **3a** with 2.0 eq of zinc chloride gave the desired **4a** in higher yield (86%) than when only 1.0 eq zinc chloride was used (Entries 2, 3), whereas the use of 3.0 or 5.0 eq of zinc chloride had adverse effects on the yields of **4a** (Entries 4, 5).

In addition, we incorporated our one-pot methodology[41–43] into a series of these reactions to establish more efficient synthetic method of **4**. Specifically, we performed the aza-Wittig reaction[44–46] of iminophosphoranes **5** with aryl isocyanates and the following intramolecular HDA cyclization caused by subsequent addition of zinc chloride in one pot under the same reaction conditions. After confirming that the one-pot reaction proceeded without any difficulty, we next examined the

Table 2. Evaluation of Stoichiometry of $ZnCl_2$ Used



3a 4a

Entry	X (eq)	Yield (%)[a]
1[b]	0.2	48
2	1.0	73
3	2.0	86
4	3.0	72
5	5.0	58

a) Yields of isolated **4a**. *b*) The reaction time was 24 h and **3a** was recovered (25%).

generality of this one-pot reaction to give **4** using substrates **5** bearing a variety of substituents in R^1 and *para*-R^2-substituents on phenyl isocyanates (R^2=H, NO_2, OMe, Me) (Table 3). In all cases, the initial aza-Wittig reactions of the iminophosphoranes **5** with the isocyanates proceeded smoothly at room temperature for 1.0–4.0 h to afford the corresponding functionalized carbodiimides **3**. Subsequent intramolecular HDA reactions in one pot gave the desired 11-phenyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine derivatives **4a**–**l** in good-to-excellent yields (61–99%) from **5a**–**d**, when the substituted group R^1 was an aromatic ring (Entries 1–12). On the other hand, when R^1 was the ^{n}Pr group, the second intramolecular HDA reaction notably required slightly longer reaction times (Entries 13–16), resulting in lower yields of **4n**–**p** (Entries 14–16).

In addition, the aza-Wittig reaction of the iminophosphorane **5a** with 2-thienyl isocyanate or *trans*-styryl isocyanate and the following intramolecular HDA reaction in one pot also gave 4-phenyl-5,10-dihydrobenzo[*b*]thieno[3,2-*g*][1,8]naphthyridine **7** in 33% yield and 3,4-diphenyl-5,10-dihydrobenzo[*b*][1,8]-naphthyridine **9** in 90% yield, respectively (Chart 2). It is noteworthy that Saito *et al.*[18] and Molina *et al.*[21] separately reported that the HDA reaction of *N*-(*ortho*-alkynylphenyl)-*N*′-2-thienyl- and *N*′-*trans*-styryl-carbodiimides also proceeded under thermal reaction conditions to produce indolo[2,3-*b*]-pyridine derivatives.

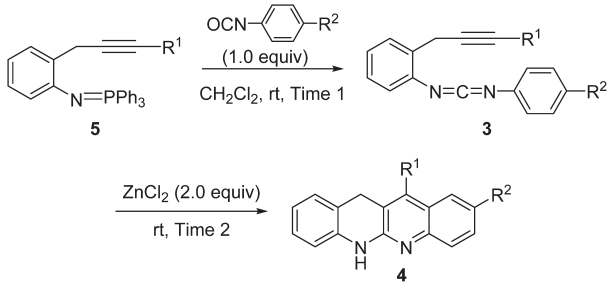
Moreover, since it was found that the dihydropyridine rings of products **4** were slowly aromatized in chloroform, we therefore carried out oxidation of **4a** with manganese dioxide in dichloromethane at an ambient temperature. Gratifyingly, 11-phenyl-dibenzo[*b*,*g*][1,8]naphthyridine **10**[36] was readily obtained in excellent yield (Chart 3). Such ring-fused aromatic *N*-containing molecules have the potential to intercalate into the DNA base stack as intercalator reagents by forming the corresponding complexes.[12,47]

In conclusion, we developed a one-pot aza-Wittig–$ZnCl_2$-promoted intramolecular HDA reaction of *N*-(*ortho*-propargylphenyl)-*N*′-arylcarbodiimides **3** for unique and effective syntheses of the novel 5,12-dihydrodibenzo[*b*,*g*][1,8]-naphthyridines **4** and the analogues **7** and **9**.

Experimental

General All melting points were determined on a Yanaco MP melting point (mp) apparatus and are uncorrected. Infra-

Table 3. Synthesis of 5,12-Dihydrodibenzo[*b*,*g*][1,8]naphthyridines **4** *via* the One-Pot Aza-Wittig/$ZnCl_2$-Promoted Intramolecular HDA Reaction



Entry	R^1	R^2	Time 1 (h)	Time 2 (h)	Product	Yield (%)[a)]
1	Ph (**5a**)	H	1.0	4.0	**4a**	99
2	Ph (**5a**)	NO_2	2.0	3.5	**4b**	91
3	Ph (**5a**)	OMe	4.0	5.0	**4c**	81
4	Ph (**5a**)	Me	2.5	2.5	**4d**	77
5	*p*-$CF_3C_6H_4$ (**5b**)	H	1.5	17.0	**4e**	79
6	*p*-$CF_3C_6H_4$ (**5b**)	NO_2	2.0	22.0	**4f**	61
7	*p*-$CF_3C_6H_4$ (**5b**)	OMe	1.0	11.0	**4g**	85
8	*p*-$CF_3C_6H_4$ (**5b**)	Me	1.5	44.0	**4h**	75
9	*p*-Tolyl (**5c**)	H	1.5	2.0	**4i**	74
10	*p*-Tolyl (**5c**)	NO_2	2.0	2.0	**4j**	70
11	*p*-Tolyl (**5c**)	OMe	1.5	2.0	**4k**	70
12	*p*-Tolyl (**5c**)	Me	2.5	2.5	**4l**	74
13	^{n}Pr (**5d**)	H	3.5	48.0	**4m**	73
14	^{n}Pr (**5d**)	NO_2	3.0	48.0	**4n**	52
15	^{n}Pr (**5d**)	OMe	3.0	48.0	**4o**	37
16	^{n}Pr (**5d**)	Me	2.0	48.0	**4p**	47

a) Yields of isolated **4** from **5** in a one-pot reaction.

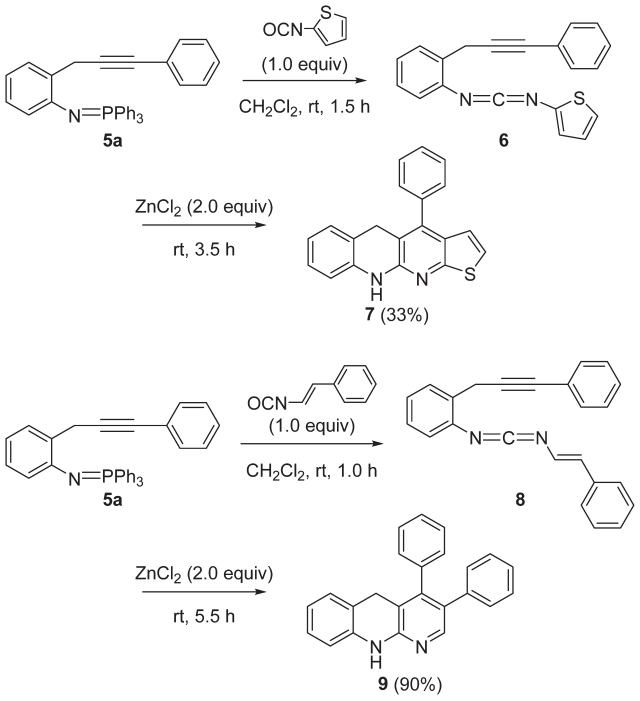


Chart 2. Synthesis of 5,10-Dihydrobenzonaphthyridines **7** and **9** *via* the One-Pot Aza-Wittig/$ZnCl_2$-Promoted Intramolecular HDA Reaction

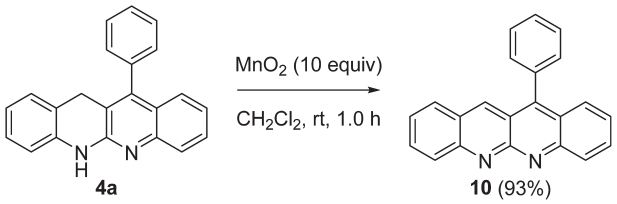


Chart 3. Oxidation of **4a** to the Aromatized Naphthyridine Derivative **10**

red spectra were recorded with a Horiba FT-710 or a JASCO FT/IR 4100 spectrophotometer. ^{1}H- and ^{13}C-NMR spectral data were obtained with JEOL JNM-ECS 400, JEOL JNM-LA 500, or JEOL JNM-AL 300 instruments. Chemical shifts are quoted in ppm using tetramethylsilane (δ=0 ppm) as the reference for ^{1}H-NMR spectroscopy, and $CDCl_3$ (δ=77.0 ppm) for ^{13}C-NMR spectroscopy. Mass spectra were measured with a Bruker Daltonics micrOTOF, a Hitachi double focusing M-80B, or a JEOL JMS-T100LP spectrometer. Column chromatography was carried out on silica gel (spherical, neutral, 40–60 μm, Kanto Chemical Co., Japan or Merck Co., Ltd., U.S.A.) or Aluminum oxide 90 active neutral (70–230 mesh ASTM, Merck). All reactions were performed under an argon atmosphere.

Typical Procedure for the $ZnCl_2$-Promoted HDA Reaction (the Diarylcarbodiimide 3a to the Dihydrodibenzo[*b*,*g*]-[1,8]naphthyridine 4a, Table 2, Entry 3) A mixture of **3a** (27.0 mg, 0.088 mmol) and zinc chloride (1.0 M in Et_2O, 0.18 mL, 0.18 mmol) in dichloromethane (2.0 mL) was stirred at room temperature for 2.5 h. The reaction was quenched with sat. aq $NaHCO_3$ (15 mL), and then mixture was extracted with chloroform (20 mL×3). The combined organic extracts were washed with brine (30 mL), dried ($MgSO_4$), and concentrated *in vacuo*. The residue was purified by silica gel column chro-

matography (hexane–ethyl acetate=10 : 1) to afford **4a** as yellow crystals (23.1 mg, 86%).

Typical Procedure for aza-Wittig Reaction/the $ZnCl_2$-Promoted HDA Reaction in One Pot (the Iminophosphorane 5a to the Dihydrodibenzo[*b*,*g*][1,8]naphthyridine 4a, Table 3, Entry 1) A mixture of **5a** (56.7 mg, 0.121 mmol) and phenyl isocyanate (13.2 μL, 0.121 mmol) in dichloromethane (1.2 mL) was stirred at room temperature for 1.0 h. After confirming the consumption of **5a** by TLC, zinc chloride (1.0 M in Et_2O, 0.24 mL, 0.24 mmol) was added to the reaction mixture at room temperature, and the mixture was stirred for 4 h. The reaction was quenched with sat. aq NaOH (1.0 M, 4.0 mL), and then mixture was extracted with chloroform (10 mL×3). The combined organic extracts were washed with brine (10 mL), dried ($MgSO_4$), and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (hexane–ethyl acetate=10 : 1) to afford **4a** as yellow crystals (37.3 mg, 99%).

11-Phenyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4a) Yellow crystals; mp 196.4–196.9°C; ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.95 (2H, s), 6.70 (1H, d, J=7.7 Hz), 6.81 (1H, dd, J=7.4, 7.4 Hz), 6.96 (1H, d, J=7.4 Hz), 7.04 (1H, dd, J=7.7, 7.4 Hz), 7.14 (1H, dd, J=8.0, 7.4 Hz), 7.24 (1H, d, J=8.0 Hz), 7.30 (2H, d, J=8.0 Hz), 7.51 (2H, dd, J=7.8, 7.4 Hz), 7.59 (2H, dd, J=7.8, 7.4 Hz), 7.77 (1H, d, J=7.7 Hz), 8.47 (1H, brs) ppm; ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 30.1, 114.1, 115.5, 119.5, 121.2, 122.9, 125.5, 126.0, 126.1, 127.3, 128.0, 128.4, 128.8 (×2), 129.0 (×2), 129.1, 136.5, 138.5, 146.2, 147.2, 151.94 ppm; IR (KBr) ν 3294, 1574, 756 cm^{-1}; high resolution (HR)-MS (electrospray ionization (ESI) *m*/*z*) Calcd for $C_{22}H_{17}N_2$ ($M+H^+$) 309.1386. Found 309.1382.

9-Nitro-11-phenyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4b) Red crystals; mp 228.1–229.0°C; ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.99 (2H, s), 6.74 (1H, d, J=8.2 Hz), 6.91 (1H, dd, J=7.7, 7.2 Hz), 7.00 (1H, d, J=7.7 Hz), 7.12 (1H, dd, J=8.2, 7.2 Hz), 7.30 (2H, d, J=6.7 Hz), 7.58–7.65 (3H, m), 7.73 (1H, d, J=9.0 Hz), 8.11 (1H, brs), 8.17 (1H, d, J=2.3 Hz), 8.29 (1H, dd, J=9.0, 2.3 Hz) ppm; ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 30.2, 114.7, 118.0, 119.7, 122.7, 123.3, 123.6, 124.7, 127.3, 128.0, 128.9, 128.9 (×2), 129.2, 129.6 (×2), 135.0, 137.3, 149.1, 148.9, 150.0, 154.1 ppm; IR (KBr) ν 3390, 3026, 1581, 1419 cm^{-1}; HR-MS (ESI *m*/*z*) Calcd for $C_{22}H_{16}N_3O_2$ ($M+H^+$) 354.1237. Found 354.1246.

9-Methoxy-11-phenyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4c) Brown crystals; mp 179.7–180.3°C; ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.66 (3H, s), 3.92 (2H, s), 6.58 (1H, d, J=2.3 Hz), 6.70 (1H, d, J=7.6 Hz), 6.81 (1H, dd, J=7.7, 7.4 Hz), 6.95 (1H, d, J=7.7 Hz), 7.06 (1H, dd, J=7.6, 7.4 Hz), 7.21 (1H, dd, J=9.2, 2.3 Hz), 7.30 (2H, d, J=7.3 Hz), 7.51 (1H, dd, J=7.3, 7.3 Hz), 7.57 (2H, dd, J=7.3, 7.3 Hz), 7.68 (1H, d, J=9.2 Hz), 7.98 (1H, brs) ppm; ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 30.2, 55.3, 105.6, 113.9, 115.7, 119.4, 120.3, 121.0, 126.0, 127.3, 127.5, 128.1, 128.4, 128.9 (×2), 128.9 (×2), 136.6, 138.8, 141.7, 146.2, 150.4, 155.3 ppm; IR (KBr) ν 3440, 1489, 1412, 748 cm^{-1}; HR-MS (ESI *m*/*z*) Calcd for $C_{23}H_{19}N_2O$ ($M+H^+$) 339.1492. Found 339.1492.

9-Methyl-11-phenyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4d) Yellow amorphous; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.32 (3H, s), 3.92 (2H, s), 6.70 (1H, d, J=7.8 Hz), 6.82 (1H, dd, J=7.5, 7.3 Hz), 6.95 (1H, d, J=7.3 Hz), 6.99 (1H, s), 7.06 (1H, dd, J=7.8, 7.5 Hz), 7.29 (2H, d, J=6.9 Hz), 7.36 (1H, d, J=8.7 Hz), 7.50–7.60 (3H, m), 7.66 (1H, dd, J=8.7, 3.4 Hz), 8.07 (1H, brs) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 21.4, 30.1, 114.0, 115.4, 119.5, 121.1, 125.1, 125.4, 125.9, 127.3, 128.0, 128.4, 128.8 (×2), 129.0 (×2), 131.2, 132.5, 136.6, 138.7, 144.6, 146.6, 151.2 ppm; IR (KBr) ν 3449, 1585, 751 cm^{-1}; HR-MS (ESI *m*/*z*) Calcd for $C_{23}H_{19}N_2$ ($M+H^+$) 323.1548. Found 323.1553.

11-[4-(Trifluoromethyl)phenyl]-5,12-dihydrodibenzo-[*b*,*g*][1,8]naphthyridine (4e) Yellow crystals; mp 216.2–217.3°C; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 3.91 (2H, s), 6.73 (1H, d, J=8.2 Hz), 6.86 (1H, dd, J=7.6, 7.2 Hz), 6.99 (1H, d, J=7.2 Hz), 7.08–7.20 (3H, m), 7.45 (2H, d, J=7.9 Hz), 7.55 (1H, ddd, J=8.2, 7.9, 2.5 Hz), 7.73 (1H, brs), 7.75 (1H, d, J=8.5 Hz), 7.86 (2H, d, J=7.9 Hz) ppm; ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 30.1, 114.1, 115.4, 119.1, 121.5, 123.1 (q, J=271.3 Hz), 123.3, 125.0, 125.6, 125.9 (×2, q, J=3.6 Hz), 126.3, 127.6, 128.4, 129.5 (×2), 130.5 (q, J=32.5 Hz), 137.7, 138.2, 140.3, 145.5, 146.3, 151.5 ppm; IR (KBr) ν 3419, 1425, 1331, 1068, 756 cm^{-1}; HR-MS (ESI *m*/*z*) Calcd for $C_{23}H_{16}F_3N_2$ ($M+H^+$) 377.1260. Found 377.1258.

9-Nitro-11-[4-(trifluoromethyl)phenyl]-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4f) Red crystals; mp 203.0–203.4°C; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 3.95 (2H, s), 6.79 (1H, d, J=8.2 Hz), 6.93 (1H, dd, J=7.3, 6.8 Hz), 7.01 (1H, d, J=6.8 Hz), 7.16 (1H, dd, J=8.2, 7.3 Hz), 7.46 (2H, d, J=8.0 Hz), 7.67 (1H, brs), 7.75 (1H, d, J=9.0 Hz), 7.92 (2H, d, J=8.0 Hz), 8.07 (1H, d, J=2.3 Hz), 8.32 (1H, dd, J=9.0, 2.3 Hz) ppm; ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 30.0, 114.6, 117.8, 118.9, 122.5, 122.7, 123.6, 123.8, 126.5 (×2, q, J=3.8 Hz), 126.9 (q, J=270.1 Hz), 127.2, 127.9, 128.6, 129.3 (×2), 130.9 (q, J=32.2 Hz), 136.8, 138.6, 142.9, 146.9, 150.0, 153.6 ppm; IR (KBr) ν 3433, 1581, 1419, 1327, 748 cm^{-1}; HR-MS (ESI *m*/*z*) Calcd for $C_{23}H_{15}F_3N_2O_2$ ($M+H^+$) 422.1111. Found 422.1112.

9-Methoxy-11-[4-(trifluoromethyl)phenyl]-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4g) Brown crystals; mp 218.5–219.7°C; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 3.68 (3H, s), 3.88 (2H, s), 6.47 (1H, d, J=2.7 Hz), 6.72 (1H, d, J=7.7 Hz), 6.85 (1H, dd, J=7.4, 7.1 Hz), 6.98 (1H, d, J=7.4 Hz), 7.10 (1H, dd, J=7.7, 7.1 Hz), 7.24 (1H, dd, J=9.2, 2.7 Hz), 7.46 (2H, d, J=8.0 Hz), 7.56 (1H, brs), 7.69 (1H, d, J=9.2 Hz), 7.86 (2H, d, J=8.0 Hz) ppm; ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 30.2, 55.4, 105.1, 113.9, 115.7, 119.0, 120.1, 121.2, 125.4, 126.0 (×2, q, J=3.8 Hz), 127.5, 127.8, 128.4, 129.5 (×2), 130.4 (q, J=30.7 Hz), 130.3 (q, J=267.7 Hz), 138.6, 140.5, 141.8, 144.5, 150.1, 155.6 ppm; IR (KBr) ν 3433, 3286, 1489, 1327 cm^{-1}; HR-MS (ESI *m*/*z*) Calcd for $C_{24}H_{18}F_3N_2O$ ($M+H^+$) 407.1366. Found 407.1366.

9-Methyl-11-[4-(trifluoromethyl)phenyl]-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4h) Yellow crystals; mp 212.2–214.0°C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.34 (3H, s), 3.88 (2H, s), 6.71 (1H, d, J=7.8 Hz), 6.84 (1H, dd, J=7.4, 7.3 Hz), 6.88 (1H, s), 6.98 (1H, d, J=7.3 Hz), 7.08 (1H, dd, J=7.8, 7.4 Hz), 7.38 (1H, d, J=8.2 Hz), 7.44 (2H, d, J=7.8 Hz), 7.66 (1H, d, J=8.7 Hz), 7.86 (2H, d, J=7.8 Hz), 7.90 (1H, brs) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 21.4, 30.1, 114.0, 115.4, 119.1, 121.3, 124.1 (q, J=272.2 Hz), 124.6, 124.8, 125.9 (×2, q, J=2.9 Hz), 126.1, 127.5, 128.4, 129.5 (×2), 130.3 (q, J=32.6 Hz), 131.5, 133.0, 138.4, 140.5, 144.6, 145.0, 151.1 ppm; IR (KBr) ν 3434, 2925, 1587, 749 cm^{-1}; HR-MS (ESI *m*/*z*) Calcd for $C_{24}H_{18}F_3N_2$ ($M+H^+$) 391.1422. Found 391.1418.

11-(*p*-Tolyl)-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine

(4i) Yellow amorphous; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.50 (3H, s), 3.96 (2H, s), 6.71 (1H, d, J=7.8 Hz), 6.83 (1H, dd, J=7.8, 7.5 Hz), 6.97 (1H, d, J=7.8 Hz), 7.07 (1H, dd, J=7.8, 7.3 Hz), 7.15 (1H, dd, J=8.2, 7.5 Hz), 7.19 (2H, d, J=7.8 Hz), 7.27 (1H, d, J=8.2 Hz), 7.38 (2H, d, J=7.8 Hz), 7.51 (1H, dd, J=8.2, 6.9 Hz), 7.74 (1H, d, J=8.2 Hz), 8.00 (1H, brs) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 21.4, 30.1, 114.0, 115.5, 119.6, 121.3, 122.9, 125.7, 126.09, 126.11, 127.3, 128.4, 128.8 (×2), 129.1, 129.5 (×2), 133.3, 137.8, 138.5, 146.3, 147.3, 151.8 ppm; IR (NaCl) ν 2922, 1585, 751 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{23}H_{19}N_2$ ($M+H^+$) 323.1548. Found 323.1544.

9-Nitro-11-(*p*-tolyl)-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4j) Yellow amorphous; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.53 (3H, s), 4.01 (2H, s), 6.74 (1H, d, J=7.8 Hz), 6.91 (1H, dd, J=7.3, 7.2 Hz), 7.00 (1H, d, J=7.2 Hz), 7.12 (1H, dd, J=7.8, 7.3 Hz), 7.18 (2H, d, J=7.8 Hz), 7.43 (2H, d, J=7.8 Hz), 7.74 (1H, d, J=9.2 Hz), 8.11 (1H, brs), 8.20 (1H, d, J=2.8 Hz), 8.29 (1H, dd, J=9.2, 2.8 Hz) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 21.4, 30.0, 114.4, 117.8, 119.5, 122.5, 123.20, 123.24, 124.6, 126.9, 127.7, 128.56 (×2), 128.60, 130.0 (×2), 131.6, 137.1, 138.8, 142.8, 148.9, 149.7, 153.8 ppm; IR (KBr) ν 3422, 2923, 1580, 748 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{23}H_{18}N_3O_2$ ($M+H^+$) 368.1399. Found 368.1394.

9-Methoxy-11-(*p*-tolyl)-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4k) Yellow crystals; mp 201.2–203.1°C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.50 (3H, s), 3.68 (3H, s), 3.92 (2H, s), 6.61 (1H, d, J=2.8 Hz), 6.69 (1H, d, J=7.6 Hz), 6.82 (1H, dd, J=7.4, 7.3 Hz), 6.97 (1H, d, J=7.3 Hz), 7.07 (1H, dd, J=7.6, 7.4 Hz), 7.18 (2H, d, J=7.9 Hz), 7.19 (1H, dd, J=8.9, 3.2 Hz), 7.37 (2H, d, J=7.9 Hz), 7.50 (1H, brs), 7.64 (1H, d, J=8.9 Hz) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 21.4, 30.2, 55.4, 105.6, 113.8, 115.7, 119.5, 120.3, 121.0, 126.2, 127.3, 127.4, 128.4, 128.8 (×2), 129.6 (×2), 133.4, 137.8, 138.8, 141.7, 146.3, 150.2, 155.3 ppm; IR (KBr) ν 3423, 2922, 1585, 752 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{24}H_{21}N_2O$ ($M+H^+$) 353.1654. Found 353.1652.

9-Methyl-11-(*p*-tolyl)-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4l) Yellow amorphous; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.33 (3H, s), 2.51 (3H, s), 3.92 (2H, s), 6.70 (1H, d, J=8.0 Hz), 6.82 (1H, dd, J=7.4, 7.3 Hz), 6.96 (1H, d, J=7.3 Hz), 7.02 (1H, s), 7.06 (1H, dd, J=8.0, 7.4 Hz), 7.18 (2H, d, J=7.8 Hz), 7.34 (1H, dd, J=8.2, 1.8 Hz), 7.38 (2H, d, J=7.8 Hz), 7.65 (1H, d, J=8.2 Hz), 7.86 (1H, brs) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 21.4 (×2), 30.1, 113.9, 115.4, 119.6, 121.1, 125.1, 125.6, 125.9, 127.3, 128.4, 128.8 (×2), 129.5 (×2), 131.1, 132.5, 133.5, 137.7, 138.7, 144.6, 146.7, 151.2 ppm; IR (KBr) ν 3423, 2919, 1586, 747 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{24}H_{21}N_2$ ($M+H^+$) 337.1705. Found 337.1712.

11-Propyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4m) Yellow crystals; mp 156.3–157.0°C; ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 1.14 (3H, t, J=7.4 Hz), 1.70 (2H, qt, J=8.0, 7.4 Hz), 3.00 (2H, t, J=8.0 Hz), 4.28 (2H, s), 6.70 (1H, d, J=7.8 Hz), 6.90 (1H, dd, J=7.5, 7.2 Hz), 7.11 (1H, dd, J=7.8, 7.2 Hz), 7.18 (1H, d, J=7.5 Hz), 7.29 (1H, dd, J=8.2, 7.5 Hz), 7.52 (1H, dd, J=8.1, 7.5 Hz), 7.52 (1H, brs), 7.68 (1H, d, J=8.1, Hz), 7.84 (1H, d, J=8.2 Hz) ppm; ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 14.6, 22.7, 28.9, 29.8, 113.9, 115.0, 119.3, 121.2, 122.9, 123.6, 124.8, 126.8, 127.5, 128.5, 128.9, 138.4, 146.2, 146.4, 151.3 ppm; IR (KBr) ν 3448, 2954, 1589, 1427, 748 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{19}H_{19}N_2$ ($M+H^+$) 275.1543. Found 275.1545.

9-Nitro-11-propyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4n) Yellow crystals; mp 269.9–271.2°C; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.19 (3H, t, J=7.3 Hz), 1.71 (2H, qt, J=8.2, 7.3 Hz), 3.06 (2H, t, J=8.2 Hz), 4.34 (2H, s), 6.77 (1H, d, J=7.7 Hz), 7.00 (1H, dd, J=7.8, 7.1 Hz), 7.19 (1H, dd, J=7.7, 7.1 Hz), 7.23 (1H, d, J=7.8 Hz), 7.47 (1H, brs), 7.69 (1H, d, J=9.4 Hz), 8.32 (1H, dd, J=9.4, 2.5 Hz), 8.80 (1H, d, J=2.5 Hz) ppm; ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 14.6, 22.9, 28.9, 29.9, 114.4, 117.1, 119.2, 120.8, 122.4, 123.0, 123.6, 127.7, 127.9, 128.7, 137.1, 142.8, 148.0, 150.2, 153.4 ppm; IR (KBr) ν 3439, 1652, 1332, 468 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{19}H_{18}N_2O_2$ ($M+H^+$) 320.1394. Found 320.1398.

9-Methoxy-11-propyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4o) Yellow crystals; mp 174.2–175.3°C; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.13 (3H, t, J=7.3 Hz), 1.71 (2H, qt, J=7.8, 7.3 Hz), 2.97 (2H, t, J=7.8 Hz), 3.91 (3H, s), 4.26 (2H, s), 6.67 (1H, d, J=8.0 Hz), 6.87 (1H, dd, J=7.5, 7.2 Hz), 7.09 (1H, dd, J=8.0, 7.2 Hz), 7.16 (1H, d, J=7.5 Hz), 7.17 (1H, d, J=2.6 Hz), 7.21 (1H, dd, J=8.6, 2.6 Hz), 7.63 (1H, d, J=8.6 Hz), 7.70 (1H, brs) ppm; ^{13}C-NMR (75 MHz, $CDCl_3$) δ: 14.4, 22.0, 28.8, 29.7, 55.2, 103.4, 113.6, 115.1, 118.9, 119.5, 120.6, 125.0, 127.2, 127.8, 128.2, 138.5, 141.4, 144.8, 149.8, 155.1 ppm; IR (KBr) ν 3432, 2954, 1589, 1496, 748 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{20}H_{21}N_2O$ ($M+H^+$) 305.1648. Found 305.1651.

9-Methyl-11-propyl-5,12-dihydrodibenzo[*b*,*g*][1,8]naphthyridine (4p) Yellow crystals; mp 226.6–228.0°C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 1.14 (3H, t, J=7.3 Hz), 1.64–1.74 (2H, m), 2.49 (3H, s), 2.98 (2H, t, J=8.2 Hz), 4.25 (2H, s), 6.67 (1H, d, J=7.8 Hz), 6.88 (1H, dd, J=7.4, 7.3 Hz), 7.09 (1H, dd, J=7.8, 7.4 Hz), 7.16 (1H, d, J=7.3 Hz), 7.35 (1H, dd, J=8.5, 1.4 Hz), 7.590 (1H, s), 7.592 (1H, d, J=8.5 Hz), 7.69 (1H, brs) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 14.6, 21.7, 22.6, 29.0, 29.7, 113.8, 114.9, 119.3, 120.9, 122.7, 124.6, 126.5, 127.4, 128.5, 130.8, 132.3, 138.7, 144.6, 145.6, 150.9 ppm; IR (KBr) ν 3448, 2958, 1591, 745 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{20}H_{21}N_2$ ($M+H^+$) 289.1705. Found 289.1700.

4-Phenyl-5,10-dihydrobenzo[*b*]thieno[3,2-*g*][1,8]naphthyridine (7) Brown amorphous; ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 3.99 (2H, s), 6.74 (1H, d, J=8.0 Hz), 6.78 (1H, d, J=6.0 Hz), 6.85 (1H, dd, J=7.4, 7.3 Hz), 6.98 (1H, d, J=7.3 Hz), 7.06 (1H, d, J=6.0 Hz), 7.09 (1H, brs), 7.10 (1H, dd, J=8.0, 7.4 Hz), 7.36 (2H, ddd, J=6.4, 2.3, 1.4 Hz), 7.41–7.57 (3H, m) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 29.4, 110.3, 113.9, 119.5, 120.9, 121.36, 121.40, 127.3, 127.7, 128.2, 128.4, 128.6 (×2), 128.7 (×2), 136.8, 138.8, 144.5, 151.0, 158.4 ppm; IR (NaCl) ν 3247, 2926, 1564 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{20}H_{15}N_2S$ ($M+H^+$) 315.0956. Found 315.0944.

3,4-Diphenyl-5,10-dihydrobenzo[*b*][1,8]naphthyridine (9) Yellow crystals; mp 230.4–231.0°C; ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.89 (2H, s), 6.74 (1H, d, J=7.8 Hz), 6.83 (1H, dd, J=7.5, 7.4 Hz), 6.94 (1H, d, J=7.4 Hz), 7.02 (2H, dd, J=7.3, 1.7 Hz), 7.07–7.11 (3H, m), 7.13–7.17 (3H, m), 7.25 (1H, brs), 7.28–7.33 (3H, m), 8.10 (1H, s) ppm; ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 29.8, 112.6, 113.8, 119.4, 121.3, 126.3, 127.3, 127.4, 127.8 (×2), 128.2 (×2), 128.5, 129.3 (×2), 129.7 (×2), 130.5, 137.2, 138.3, 138.9, 146.4, 148.2, 152.1 ppm; IR (KBr) ν 3410, 3248, 3024, 1412, 748 cm^{-1}; HR-MS (ESI m/z) Calcd for $C_{24}H_{19}N_2$ ($M+H^+$) 335.1548. Found 335.1540.

Synthesis of 10 A mixture of **4a** (22.7 mg, 0.0736 mmol) and manganese dioxide (90% active, 71.0 mg, 0.735 mmol) in dichloromethane (0.7 mL) was stirred at room temperature for

1 h, and filtered through a Celite pad. The filtrate was evaporated and purified by aluminum oxide column chromatography (hexane–ethyl acetate=2 : 1) to afford **10** (21.0 mg, 93%) as a red amorphous.

11-Phenyldibenzo[*b*,*g*][1,8]naphthyridine (10)[36] Red amorphous; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 7.38–7.47 (2H, m), 7.52–7.56 (2H, m), 7.66–7.71 (3H, m), 7.74 (1H, d, J=8.6 Hz), 7.80 (2H, ddd, J=8.9, 6.5, 1.4 Hz), 7.85 (1H, d, J=8.6 Hz), 8.34 (1H, d, J=8.9 Hz), 8.38 (1H, d, J=9.1 Hz), 8.76 (1H, s) ppm.

Conflict of Interest The authors declare no conflict of interest.

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