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Copper-catalyzed intramolecular domino synthesis of 6*H*-chromeno[4,3-*b*]quinolines in green condition

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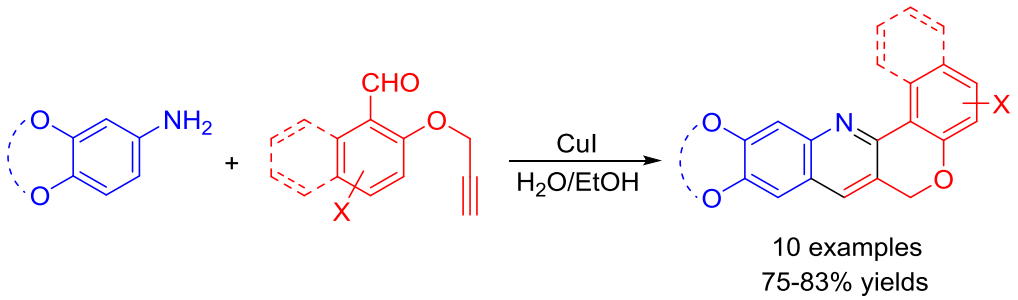
Abstract

A one-pot and efficient copper-catalyzed approach for synthesis of tetracyclic 6*H*-chromeno[4,3-*b*]quinolines through the intramolecular domino condensation-aza-Diels–Alder reaction of electron-rich anilines and *O*-propargylated salicylaldehydes under green conditions has been described.



Keywords: Aza-Diels–Alder, copper-catalyzed, 6*H*-chromeno[4,3-*b*]quinoline, domino reaction, green chemistry, electron-rich anilines, O-propargylated salicylaldehydes

Introduction

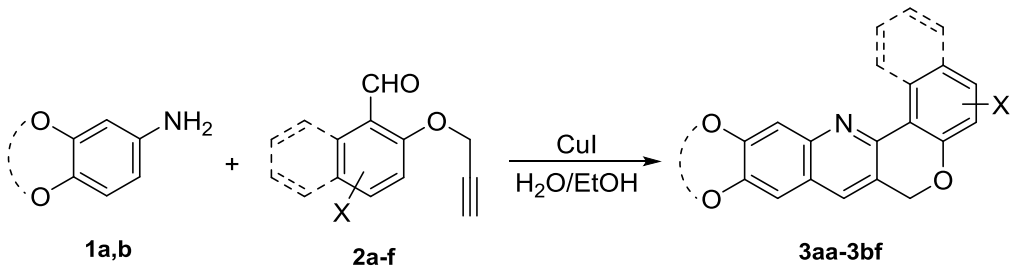
One of the fascinating fields of heterocyclic chemistry is domino reactions.[1,2] It has been proven that these reactions are a powerful and efficient tool for the construction of polyheterocyclic molecules. Among them, intramolecular domino hetero-Diels–Alder reactions are of great interest in synthetic chemistry for the preparation of fused heterocycles because of their great economical and stereocontrolled nature.[3,4] These reactions allow the formation of two or more rings at once, avoiding sequential chemical transformations.
The use of water as a green solvent in organic transformations instead of harmful organic solvents has received a great deal of attention in both academic and industrial research. Organic reactions in aqueous media are cost effective, safe, clean and eco-friendly, provide greater reactivity and selectivity, give cleaner products, and can minimize generation of waste and consuMption of energy by increasing reaction rates.[5,6] Owing to these unique properties, designing organic reactions in water is an iMportant subject in synthetic chemistry.[7,8]

Chromenoquinoline structural motifs are interesting classes of fused N-containing heterocyclic scaffolds due to their remarkable biological and pharmacological activities,[9,10] such as anti-inflammatory activities and serotonin and estrogen receptor functions.[11,12] Some chromenoquinoline derivatives have been used as drugs that modulate the transcriptional activity of the human progesterone receptor, which plays an iMportant role in medicine, and has been used therapeutically.[13] It was also reported that some of the chromenoquinoline-based molecules act as a fluorescense sensor.[14,15] Alkaloids containing the pyranoquinoline core are an iMportant class of quinoline alkaloids which exhibit a number of iMportant biological and pharmacological properties.[16] Also, some natural products such as dutadrupine, helietidine and geibalansine are containing the pyranoquinoline core structure.[17,18]

This broad range of applications has stimulated considerable interest in developing novel synthetic methods for the construction of polycyclic chromene-annulated quinoline derivatives. Therefore, a number of synthetic methods have been reported in literature for the preparation of chromenoquinolines.[19-22] All these reports involve the reaction of aniline derivatives with *O*-propargylated salicylaldehydes in the presence of metal or Lewis acid catalysts, in toxic organic solvents such as toluene, acetonitrile or DMF or in ionic liquids. So, we became interested in the development of a green and practical method to synthesize 6*H*-chromeno[4,3-*b*]quinolines which are of potential pharmacological and biological interest.

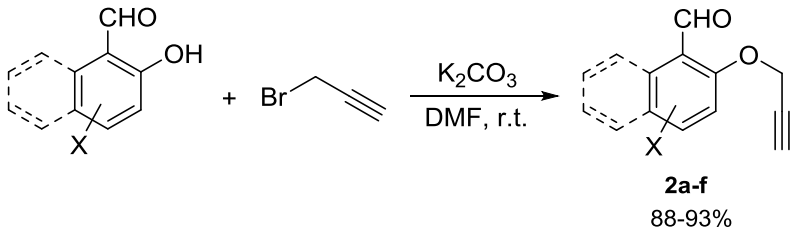
Results and Discussion

In continuation of our research to prepare N-containing heterocycles,[23-30] herein we wish to report a green and siMple intramolecular domino condensation-aza-Diels–Alder reaction between electron-rich anilines **1a,b** and O-propargylated salicylaldehydes **2a–f** in the presence of CuI as catalyst in H_2O-EtOH affording 6*H*-chromeno[4,3-*b*]quinolines **3aa–3bf** in 75**–** 83% yield (Scheme 1).



Scheme 1. Copper-catalyzed synthesis of the 6*H*-chromeno[4,3-*b*]quinolines.

The O-propargylated salicylaldehydes **2a–f** were prepared by the Williamson ether reaction of corresponding substituted salicylaldehydes with propargyl bromide in 88**–**93% yield (Scheme 2).

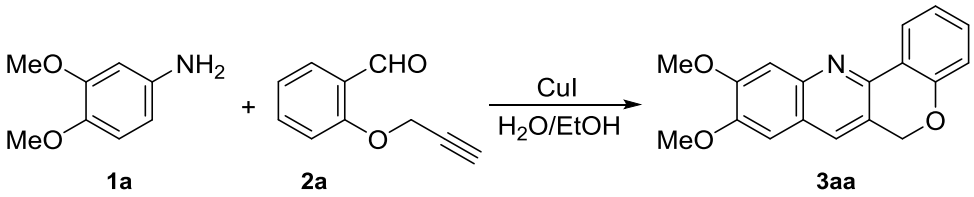


Scheme 2. Williamson ether synthesis of *O*-propargylated salicylaldehydes.

Initially, to develop optimized condition, the reaction of 3,4-dimethoxyaniline **1a** with aldehyde **2a** affording 9,10-dimethoxy-6*H*-chromeno[4,3-*b*]quinoline **3aa** was investigated as model reaction (Table 1). Heating the reaction mixture in water under reflux did not provide our goal (Entry 1). After this failure, we evaluated various copper salts, but only got satisfactory results with 20 mol% CuI (Entry 3). By variation of the CuI ratios and the solvent, good yields were obtained with 20 mol% CuI and H_2O/EtOH (50:50) as reaction medium under reflux for 6 hours. The results are summarized in Table 1.

To investigate the scope of this reaction, the reaction between various aniline derivatives **1a–f**, and *O*-propargylated salicylaldehyde **2a** were explored under these optimized conditions. As shown in Table 2, the best yield was only obtained using highly electron-rich aniline **1a** (Table 2, entry 1). Therefore, the reaction between 3,4-dimethoxyaniline **1a** and benzo[*d*][1,3]dioxol-5-amine **1b**, with various *O*-propargylated salicylaldehyde **2a–f**, carrying both electron-donating or electron-withdrawing substituent, were tested to afford the 6*H*-chromeno[4,3-*b*]quinolines **3aa–3bf** in 75**–**83% yields (Table 3).

Table 1. Effect of Catalyst and Solvent on the synthesis of (**3aa**)[a]

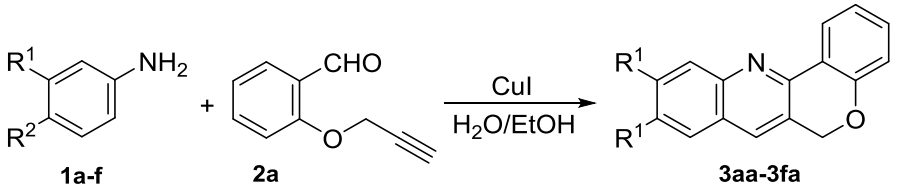


Entry	Catalyst (mol%)	Solvent	Yield (%)[b]
1		H_2O	NR[c]
2	CuI (20)	H_2O	48
3	**CuI (20)**	**H_2O/EtOH**	**78**

Entry	Catalyst (mol%)	Solvent	Yield (%)[b]
4	CuI (10)	H_2O/EtOH	35
5	CuI (30)	H_2O/EtOH	65
6	CuBr (20)	H_2O/EtOH	72
7	$CuBr_2$ (20)	H_2O/EtOH	64
8	CuCl (20)	H_2O/EtOH	66
9	$CuCl_2$ (20)	H_2O/EtOH	58
10	CuO (20)	H_2O/EtOH	54
11	$CuSO_4$ (20)	H_2O/EtOH	42

[a] Reaction condition: **1a** (1.0 mmol), **2a** (2.0 mmol), solvent (3 mL), reflux, 6 h; [b] Isolated yield; [c] NR = no reaction.

Table 2. Investigation of the reaction scope

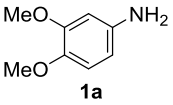
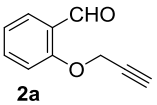
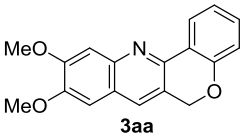
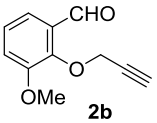
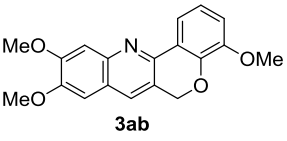
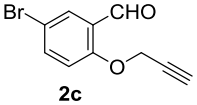
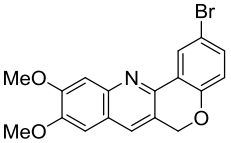


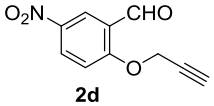
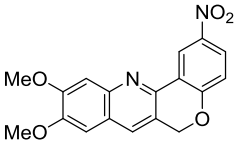
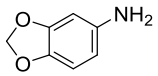
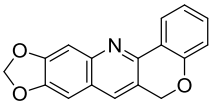
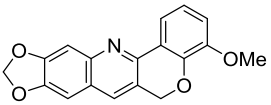
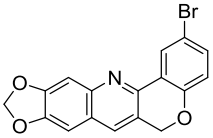
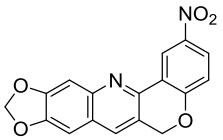
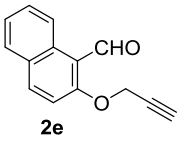
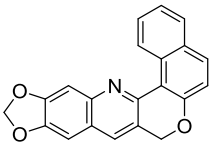
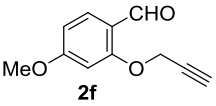
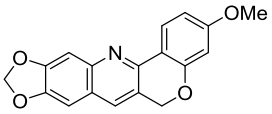
1a-f **2a** **3aa-3fa**

Entry	R^1	R^2	Product	Yield (%)[b]
1	OMe	OMe	**3aa**	78
2	H	OMe	**3ba**	37
3	H	Me	**3ca**	17
4	H	H	**3da**	10
5	Cl	H	**3ea**	NR[c]
6	H	NO_2	**3fa**	NR

[a] Reaction condition: **1** (1.0 mmol), **2** (2.0 mmol), CuI (20 mol%), H_2O/EtOH (50:50, 3 mL), reflux, 6 h; [b] Isolated yield; [c] NR = no reaction.

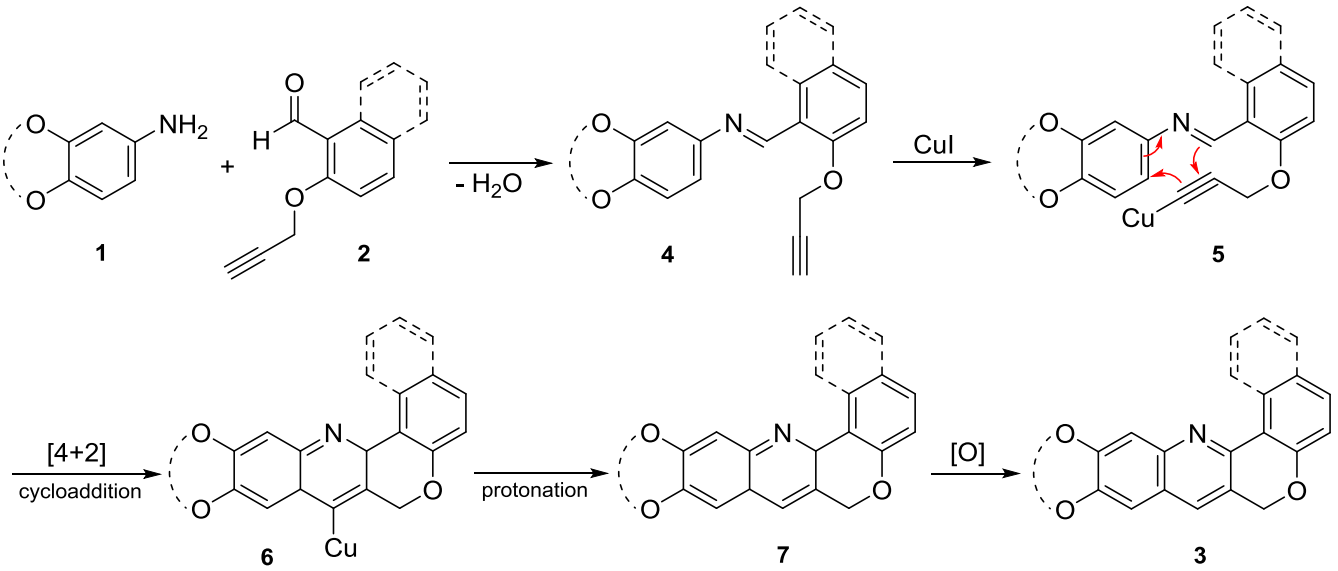
Table 3. Substrate scope for the synthesis of 6*H*-chromeno[4,3-*b*]quinoline (**3aa-3bf**)[a]

Entry	Amine	Aldehyde	Product	Yield (%)[b]
1	 **1a**	 **2a**	 **3aa**	78
2	**1a**	 **2b**	 **3ab**	81
3	**1a**	 **2c**	 **3ac**	76

Entry	Amine	Aldehyde	Product	Yield (%)[b]
4	**1a**	 **2d**	 **3ad**	75
5	 **1b**	**2a**	 **3ba**	83
6	**1b**	**2b**	 **3bb**	78
7	**1b**	**2c**	 **3bc**	80
8	**1b**	**2d**	 **3bd**	76
9	**1b**	 **2e**	 **3be**	80
10	**1b**	 **2f**	 **3bf**	82

[a] Reaction condition: **1** (1.0 mmol), **2** (2.0 mmol), CuI (20 mol%), H_2O/EtOH (50:50, 3 mL), reflux, 6 h; [b] Isolated yield.

A plausible mechanism for the copper-catalyzed intramolecular domino condensation and aza-Diels–Alder formation of 6*H*-chromeno[4,3-*b*]quinolines is proposed in Scheme 3. Initially, it is reasonable to assume that the copper-acetylide imine intermediate **5** was formed through the condensation of amine **1** with aldehyde **2**, followed by deprotonation in the presence of the copper catalyst. Next, the sequential intramolecular[4+2]cycloadditon, protonation and oxidation generate the desired product **3** (Scheme 3).



Scheme 3. Plausible mechanism for synthesis of the 6*H*-chromeno[4,3-*b*]quinolines.

Conclusions

In summary, a green and efficient approach was developed for the synthesis of 6*H*-chromeno[4,3-*b*]quinolines *via* the copper-catalyzed intramolecular domino condensation and aza-Diels–Alder reaction of electron-rich anilines with O-propargylated salicylaldehydes. The siMplicity of the starting materials, good yields of the products and the use of H_2O/EtOH as a green, cheap and nontoxic solvent are the main advantages of this method.

Experimental Section

General. All chemicals were purchased from Merck and Fluka coMpanies. All yields refer to isolated products. ^{1}H and ^{13}C NMR spectra were recorded on a Brucker, Rheinstetten, Germany (at 500 and 400 MHz) NMR spectrometer using tetramethylsilane (TMS) as internal standard. Melting points were determined in a capillary tube and are not corrected. The progress of reaction was followed with TLC using silica gel SILG/UV 254 and 365 plates. All products are known coMpounds and their structures were deduced by ^{1}Hm ^{13}C NMR spectroscopy and elemental analysis.

General procedure for the preparation of coMpounds 3aa–3bf, exeMplified with 3aa. A mixture of 3,4-dimethoxyaniline (1.0 mmol), 2-propargyl salicylaldehyde (1.0 mmol), CuI (0.2 mmol), in 3 mL H_2O-EtOH (50:50) was stirred in a sealed vessel for 6 hours at 100 °C. After reaction coMpletion (TLC), the reaction mixture was cooled to room teMperature, then, the aqueous phase was separated by suction and the semisolid residue was purified by column chromatography on silica gel (eluent: hexane–EtOAc) afforded **3aa** (78%).

9,10-Dimethoxy-6*H*-chromeno[4,3-b]quinoline (**3aa**). Mp 138–140 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.02 (s, 3H), 4.08 (s, 3H), 5.26 (s, 2H), 7.03 (s, 1H), 7.21 (d, *J* 9.0 Hz, 1H), 7.45 (td, *J* 7.0 , 1.5 Hz, 1H), 7.54 (s, 1H), 7.77 (td, *J* 7.0, 1.5 Hz, 1H), 7.82 (d, *J* 9.0 Hz, 1H), 9.90 (d, *J* 9.0 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 56.0,

56.2, 68.8, 104.9, 108.5, 118.2, 122.2, 124.3, 127.1, 127.6, 128.3, 129.2, 132.3, 144.9, 148.7, 149.8, 152,4, 157.1 ppm; Anal. Calcd for C, 73.71; H, 5.15; N, 4.78; O, 16.36 : C, 73.71; H, 5.15; N, 4.78. Found: C, 73.27; H, 5.01; N, 4.52.

4,9,10-Trimethoxy-6*H*-chromeno[4,3-*b*]quinoline (3ab). Mp 151–153 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.94 (s, 3H), 4.01 (s, 3H), 4.06 (s, 3H), 5.39 (s, 2H), 6.96 (d, *J* 8.0 Hz, 1H), 7.01 (s, 1H), 7.09 (t, *J* 8.0 Hz, 1H), 7.46 (s, 1H), 7.72 (s, 1H), 8.02 (d, *J* 7.5 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 56.0, 56.1, 56.2, 68.8, 105.0, 108.3, 113.2, 116.9, 121.9, 123.2, 124.4, 127.8, 129.3, 145.5, 148.9, 149.9, 152.7, 152.8, 157.2 ppm; Anal. Calcd for C, 70.58; H, 5.30; N, 4.33; O, 19.79: C, 70.58; H, 5.30; N, 4.33. Found: C, 70.09; H, 5.03; N, 4.12.

2-Bromo-9,10-dimethoxy-6*H*-chromeno[4,3-*b*]quinoline (3ac). Mp 144–146 °C; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 2.64 (s, 3H), 2.68 (s, 3H), 4.09 (s, 2H), 5.75-5.88 (m, 3H), 6.01-612 (m, 2H), 6.70 (s, 1H), 7.03 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6, 125 MHz): δ = 55.6, 55.7, 67.5, 105.5, 107.5, 117.0, 122.0, 122.8, 123.1, 124.4, 129.6, 131.1, 144.4, 145.5, 149.4, 152.2, 156.5 ppm; Anal. Calcd for C, 58.08; H, 3.79; Br, 21.47; N, 3.76; O, 12.89: C, 58.08; H, 3.79; N, 3.76. Found: C, 57.61; H, 3.32; N, 3.52.

9,10-Dimethoxy-2-nitro-6*H*-chromeno[4,3-*b*]quinoline (**3ad**). Mp 160–162 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 4.01 (s, 3H), 4.06 (s, 3H), 5.30 (s, 2H), 6.87 (d, *J* 8.5 Hz, 1H), 6.99 (s, 1H), 7.40 (dd, *J* 9.0, 2.0 Hz, 1H), 7.44 (s, 1H), 7.68 (s, 1H), 8.51 (d, *J* 2.5 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 56.0, 56.1, 68.4, 105.0, 108.1, 115.0, 119.0, 122.8, 123.4, 125.3, 127.6, 129.3, 133.7, 145.3, 145.4, 150.1, 152.7, 155.8 ppm ; Anal. Calcd for C, 63.90; H, 4.17; N, 8.28; O, 23.64: C, 63.90; H, 4.17; N, 8.28. Found: C, 63.18; H, 3.78; N, 8.01.

6*H*-Chromeno[4,3-b][1,3]dioxolo[4,5-g]quinoline (**3ba**). Mp 139–141 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 5.29 (s, 2H), 6.11 (s, 2H), 6.87 (dd, *J* 8.5, 1.5 Hz, 1H), 7.01 (s, 1H), 7.40 (m, 3H), 7.67 (s, 1H), 8.50 (s, 1H), ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 68.3, 101.2, 102.5, 106.3, 119.3, 123.8, 124.7, 126.1, 127.7, 130.0, 133.7, 142.8, 148.1, 148.6, 149.0, 156.8 ppm ; Anal. Calcd for C, 73.64; H, 4.00; N, 5.05; O, 17.31: C, 73.64; H, 4.00; N, 5.05. Found: C, 73.17; H, 3.56; N, 4.72.

4-Methoxy-6*H*-chromeno[4,3-*b*][1,3]dioxolo[4,5-*g*]quinoline (**3bb**). Mp 175–177 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.92 (s, 3H), 5.35 (s, 2H), 6.08 (s, 2H), 6.95 (d, *J* 8.0 Hz, 1H), 6.98 (s, 1H), 7.07 (t, *J* 8.0 Hz, 1H), 7.39 (s, 1H), 7.65 (s, 1H), 8.0 (d, *J* 8.0 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 56.1, 68.6, 101.6, 102.6, 106.0, 113.2, 116.9, 121.8, 123.1, 124.2, 124.5, 129.8, 146.3, 146.5, 146.7, 147.7, 148.7, 150.6 ppm; Anal. Calcd for C, 70.35; H, 4.26; N, 4.56; O, 20.82: C, 70.35; H, 4.26; N, 4.56. Found: C, 69.85; H, 4.01; N, 4.31.

2-Bromo-6*H*-chromeno[4,3-*b*][1,3]dioxolo[4,5-*g*]quinoline (**3bc**). Mp 160–162 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 5.27 (s, 2H), 6.09 (s, 2H), 6.85 (d, *J* 8.5 Hz, 1H), 6.98 (s, 1H), 7.37-7.39 (m, 2H), 7.62 (s, 1H), 8.48 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 68.3, 101.7, 102.5, 106.0, 115.0, 119.0, 122.8, 124,8, 125.1, 127.6, 129.6, 133.7, 145.4, 146.6, 148.0, 150.9, 155.8 ppm; Anal. Calcd for C, 57.33; H, 2.83; Br, 22.43; N, 3.93; O, 13.48: C, 57.33; H, 2.83; N, 3.93. Found: C, 56.90; H, 2.42; N, 3.52.

2-Nitro-6*H*-chromeno[4,3-*b*][1,3]dioxolo[4,5-*g*]quinoline (**3bd**). Mp 153–155 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 5.30 (s, 2H), 6.10 (s, 2H), 6.85 (d, *J* 8.0 Hz, 1H), 7.14 (t, *J* 8.0 Hz, 1H), 7.33 (t, *J* 8.0 Hz, 1H), 7.44 (s, 1H), 7.67 (s, 1H), 8.48 (d, *J* 8.0 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 68.2, 101.6, 102.7, 106.7, 108.8, 115.6, 117.1, 119.9, 122.6, 126.3, 130.2, 133.5, 145.6, 146.0, 149.0, 152.7, 160.2 ppm; Anal. Calcd for C, 63.36; H, 3.13; N, 8.69; O, 24.82: C, 63.36; H, 3.13; N, 8.69. Found: C, 63.08; H, 3.02; N, 8.42.

8*H*-Benzo[5,6]chromeno[4,3-*b*][1,3]dioxolo[4,5-*g*]quinoline (**3be**). Mp 166–168 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 5.23 (s, 2H), 6.09 (s, 2H), 7.03 (s, 1H), 7.20 (d, *J* 8.5 Hz, 1H), 7.44 (t, *J* 7.5 Hz, 1H), 7.51 (s, 1H), 7.64 (t, *J* 7.5 Hz, 1H),7.73 (s, 1H), 7.80-7.82 (m, 2H), 9.88 (d, *J* 7.5 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 68.7, 101.6, 102.5, 106.2, 116.3, 118.2, 123.5, 124.3, 124.8, 127.1, 127.6, 128.3, 129.8, 130.7, 131.2, 132.3, 146.1, 147.8, 148.7, 150.6, 157.1 ppm; Anal. Calcd for C, 77.05; H, 4.00; N, 4.28; O, 14.66: C, 77.05; H, 4.00; N, 4.28. Found: C, 76.72; H, 3.61; N, 4.06.

3-Methoxy-6*H*-chromeno[4,3-*b*][1,3]dioxolo[4,5-*g*]quinoline (**3bf**). Mp 170–172 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.84 (s, 3H), 5.27 (s, 2H), 6.08 (s, 2H), 6.52 (d, *J* 2.5 Hz, 1H), 6.70 (dd, *J* 8.0, 2.5 Hz, 1H), 6.99 (s, 1H), 7.38 (s, 1H), 7.62 (s, 1H), 8.29 (d, *J* 9.0 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 55.4, 68.6, 101.6, 101.8, 102.7, 105.9, 109.4, 113.5, 117.8, 121.4, 122.4, 123.8, 126.2, 129.7, 147.1, 148.7, 149.4, 157.7 ppm; Anal. Calcd for C, 70.35; H, 4.26; N, 4.56; O, 20.82: C, 70.35; H, 4.26; N, 4.56. Found: C, 70.11; H, 4.01; N, 4.32.

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Supplementary Material

General procedure for the preparation of compounds **3aa**–**3bf**, exemplified with 3aa; Characterization data for 6*H*-chromeno[4,3-*b*]quinolines (**3aa**-**3bf**) ; and Copies of NMR spectra.

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