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Sequential three-component reaction of homophthalonitrile, salicylaldehydes and nitromethane

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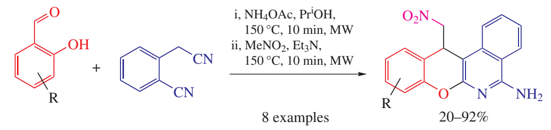
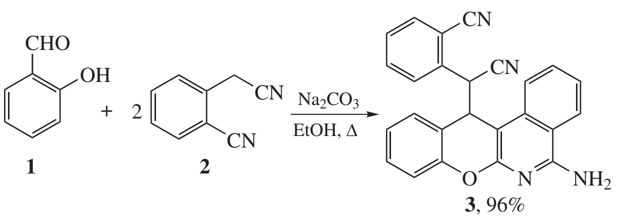
An effective preparative multi-component synthesis of 12-nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amines comprised sequential reactions of salicylaldehydes with homophthalonitrile and then with nitromethane under MW irradiation.



2-Aminochromene belongs to a privileged chemotype whose representatives exhibit a variety of biological activities[1–3] and are valuable platforms for drug discovery. For example, crolibulin (EPC2407) exerts antitumor activity and is undergoing Phase II clinical trials.[4] The properties of chromenes annulated to pyridine rings are of interest. For example, pranoprofen is an anti-inflammatory drug used in ophthalmology.[5] Chromenotacrines, *e.g.*, CT6, were recommended as potential agents for treating Alzheimer disease.[6]

The three-component reaction of malonodinitrile, *o*-hydroxybenzaldehyde, and nitromethane formed 2-aminochromenes bearing 4-positioned nitromethyl substituent.[7–10] The use of 2-aminoprop-1-ene-1,1,3-tricarbonitrile in the multicomponent reaction with *o*-hydroxybenzaldehyde and 3-phenylisoxazol-5(4*H*)-one has been recently reported.[11] The aim of this work was to verify the feasibility of using homophthalonitrile as a dinitrile in multicomponent reactions for preparing chromene derivatives.

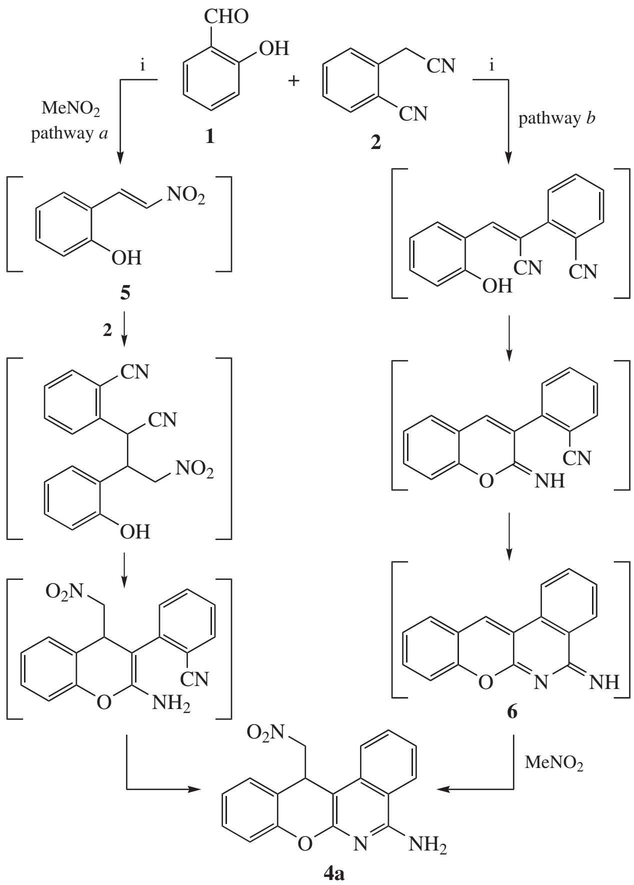
We found that refluxing salicylaldehyde **1** with homophthalonitrile **2** in EtOH in the presence of sodium carbonate induced a pseudo-three-component reaction leading to chromenoisoquinolinamine **3** in excellent yield (Scheme 1).



Scheme 1

We hypothesized that adding a different CH-acid as the third reaction component would bring about an analogous chromenoisoquinoline with a different substituent on the chromene ring. In fact, refluxing salicylaldehyde **1** and homophthalonitrile **2** with nitromethane in the presence of NH_4OAc in EtOH–H_2O (3 : 1) afforded the three-component reaction product **4a** in 18% yield (Scheme 2). This reaction could occur either through initial formation of nitrovinyl derivative **5** to which homophthalonitrile added, followed by two successive nucleophilic cyclizations and tautomerizations occurred (pathway *a*). Otherwise, initial condensation between **1** and **2** gives after two cyclizations intermediate **6** which finally adds nitromethane (pathway *b*).

Some insight into the reaction mechanism was gained on testing pure compound **5** which was reacted with **2** under various conditions (Table 1). As it turned out, the target compound **4a** was not formed in this case. The major reaction product was chromene **3**, which resulted from a pseudo-three-component reaction. Probably, nitrovinyl compound **5** in the presence of H_2O and base was reverted into starting salicylaldehyde and



Scheme 2 *Reagents and conditions*: i, $MeNO_2$, NH_4OAc, alcohol as a solvent, Δ.

Table 1 Reaction of nitrovinyl derivative **5** with homophthalonitrile **2**.

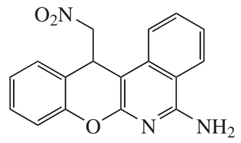
Entry	Reactant	Solvent	*T*/°C	*t*/min	Yield of **3** (%)
1	DBU (1 equiv.)	EtOH	reflux	480	5
2	NaOAc (1 equiv.)	EtOH	reflux	480	27
3	K_2CO_3 (1 equiv.)	EtOH–H_2O	reflux	60	–
4	K_2CO_3 (0.5 equiv.)	EtOH–H_2O	reflux	60	–
5	K_2CO_3 (1 equiv.)	THF	60 (MW)	10	–
6	Et_3N (1 equiv.)	Pr^iOH	150 (MW)	10	30

nitromethane, with the former reacting with **2** to give **3**. These results argue in favor of the formation of chromenoisoquinoline **4a** through pathway *b*.

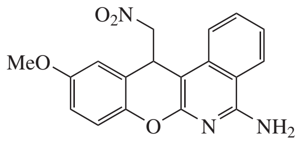
Taking into account that nitrovinyl derivatives **5** were formed as side products, we decided to perform the reaction sequentially, *i.e.*, first reacting **1** with **2** in the presence of a promoter in order to increase the concentration of intermediate **6** (Table 2, step *1*) followed by addition of nitromethane after some time (Table 2, step *2*). Such an experiment gave the target **4a** in 25% yield

Table 2 Optimization of multi-component reaction conditions for preparing chromenoisoquinolines **4**.

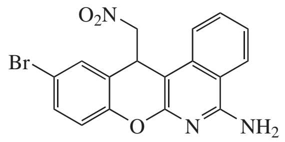
Entry	Ratio **1**:**2**: $MeNO_2$	Solvent	Conditions: Step *1*	Conditions: Step *2*	Yield of **4** (%)
1	1:1:3	EtOH–H_2O	NH_4OAc (1 equiv.), reflux, 70 min	NH_4OAc (1 equiv.), reflux, 5 h	25
2	3:1:3.5	EtOH–H_2O	NH_4OAc (1 equiv.), reflux, 30 min	reflux, 1 h	36
3	3:1:5.5	MeOH	NH_4OAc (2 equiv.), reflux, 30 min	NH_4OAc (2 equiv.), reflux, 2 h	40
4	3:1:10	MeOH	NH_4OAc (2 equiv.), reflux, 30 min	NH_4OAc (2 equiv.), reflux, 2 h	46
5	3:1:5.5	CF_3CH_2OH	NH_4OAc (2 equiv.), reflux, 30 min	NH_4OAc (2 equiv.), reflux, 2 h	9
6	3:1:5.5	Pr^iOH	NH_4OAc (2 equiv.), reflux, 30 min	NH_4OAc (2 equiv.), reflux, 2 h	52
7	3:1:5.5	MeOH	Et_3N (1 equiv.), reflux, 1 h	reflux, 1 h	–
8	3:1:5.5	Pr^iOH	NH_4OAc (2 equiv.), reflux, 1 h	Et_3N (1 equiv.), reflux, 2 h	52
9	3:1:5.5	MeOH	NH_4OAc (2 equiv.), reflux, 1 h	Et_3N (1 equiv.), reflux, 2 h	43
10	3:1:5.5	Pr^iOH	piperidine (1 equiv.), reflux, 30 min	reflux, 5 h	33
11	3:1:5.5	Pr^iOH	piperidine (1 equiv.), AcOH (10 mol%), reflux, 30 min	reflux, 5 h	25
12	3:1:5.5	MeOH	meglumine (2 equiv.), reflux, 30 min	reflux, 2 h	16
13	3:1:10	Pr^iOH	NH_4OAc (2 equiv.), MW, 150 °C, 10 min	DIPEA (1 equiv.), MW, 150 °C, 10 min	68
14	3:1:10	Pr^iOH	NH_4OAc (2 equiv.), MW, 150 °C, 10 min	Et_3N (1 equiv.), MW, 150 °C, 10 min	71
15	3:1:10	EtOH	NH_4OAc (2 equiv.), MW, 150 °C, 10 min	Et_3N (1 equiv.), MW, 150 °C, 10 min	41



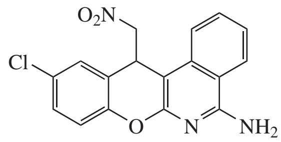
4a, 71%



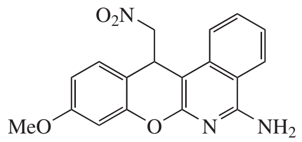
4b, 76%



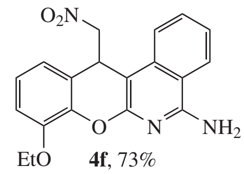
4c, 94%



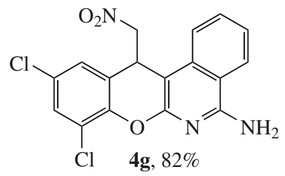
4d, 72%



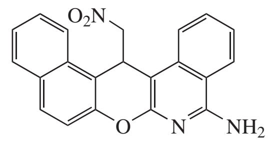
4e, 46%



4f, 73%



4g, 82%



4h, 20%

(Table 2, entry 1), however, compound **3** was also isolated in 27% yield. The use of excess aldehyde suppressed the side reaction and increased the yield of **4a** to 36% (entry 2). The products crystallized from the reaction mixture when H_2O–EtOH was used as a solvent. Apparently, the presence of H_2O could promote hydrolysis of intermediate **6** and the occurrence of other side reactions. Changing the solvent to MeOH and raising the amount of nitromethane from 5.5 to 10 equiv. increased the reaction yields to 40 and 46%, respectively (entries 3 and 4). The use of trifluoroethanol as a solvent produced **4a** in 9% yield (entry 5). The optimal solvent was found to be Pr^iOH, which provided yield of 52% (entry 6). The use of promoters such as Et_3N, piperidine, piperidine + HOAc, and meglumine in the first step was less effective than the use of NH_4OAc (entries 7–9, 11, 12). Carrying out the experiment under microwave (MW) irradiation reduced the reaction time and increased the yield of target compound to 71%. The second step was observed to proceed best with Et_3N (entries 13–15).

Therefore, the reaction was carried out under the optimal conditions by heating a Pr^iOH solution of salicylaldehyde, homophthalonitrile, and NH_4OAc in a sealed container to 150 °C in a microwave reactor, adding nitromethane and Et_3N, and heating again for 10 min at 150 °C.† The target compound was isolated by chromatography or simply with extraction. The developed method was used to study the scope of reaction. It turned out that a wide array of *o*-hydroxybenzaldehydes could be used to achieve excellent yields of variously substituted

† *Synthesis of chromenoisoquinolines* **4a**–**g** *(general procedure).* To a solution of homophthalonitrile **2** (129 mg, 0.82 mmol) in Pr^iOH (4 ml), salicylaldehyde **1** (2.46 mmol) and NH_4OAc (143 mg, 1.64 mmol) were added. The mixture was placed into a microwave reactor and heated at 150 °C for 10 min. The vessel was opened, $MeNO_2$ (0.439 ml, 8.2 mmol) and Et_3N (0.114 ml, 0.82 mmol) were added, the mixture was heated again in the microwave reactor at 150 °C for 10 min. The solvent was evaporated under reduced pressure, the residue was chromatographed on silica gel (EtOAc–hexane, 1:5–1:1) (method A) or was dissolved in a small volume of Et_2O or CH_2Cl_2 and extracted with HCl aqueous solution (0.6 M, 3×30 ml). The aqueous layer was neutralized with Na_2CO_3. The precipitate was filtered off and washed with H_2O several times and once with a small amount (1.5 ml) of Pr^iOH and dried in air (method B).

chromenoisoquinolinamines **4a–f**. Compound **4e** was formed in lower yield (46%), which could be due to lower reactivity of 4-methoxy-2-hydroxybenzaldehyde in the Knoevenagel condensation. Pentacyclic product **4h** was obtained in the lowest yield (20%), which may resulted from steric hindrance created by the additional benzene ring.

In conclusion, we have developed an effective preparative multicomponent synthesis of chromeno[2,3-*c*]isoquinolin-5-amines **4a–h**.

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Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.1016/j.mencom.2017.09.006.

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*12-Nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amine* **4a**. Yield 71% (A), 70% (B), yellow powder, mp 202 °C. IR (KBr, ν/cm^{-1}): 3480 (NH_2), 3381 (NH_2), 1536 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 4.68 (dd, 1H, *J* 11.9, 7.0 Hz), 4.83 (dd, 1H, *J* 11.9, 3.9 Hz), 5.33 (dd, 1H, *J* 7.0, 3.9 Hz), 7.16–7.18 (m, 2H), 7.23 (s, 2H), 7.34–7.37 (2H, m), 7.43 (t, 1H, *J* 7.4 Hz), 7.75 (t, 1H, *J* 7.4 Hz), 7.9 (d, 1H, *J* 8.2 Hz), 8.26 (d, 1H, *J* 8.2 Hz). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 34.3, 80.8, 90.3, 115.8, 116.4, 120.7, 120.9, 123.5, 123.7, 125.0, 128.4, 128.7, 131.0, 136.3, 151.6, 154.2, 157.2. MS (ESI) *m/z*: 308 [M+H]$^+$, 349 [M+H+MeCN]$^+$. Found (%): C, 66.59; H, 4.19; N, 13.59. Calc. for $C_{17}H_{13}N_3O_3$ (%): C, 66.44; H, 4.26; N, 13.67.

*10-Methoxy-12-nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amine* **4b**. Yield 76% (A), 53% (B), yellow powder, mp 199–200 °C. IR (KBr, ν/cm^{-1}): 3507 (NH_2), 3394 (NH_2), 1537 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 3.76 (s, 3H), 4.70 (dd, 1H, *J* 11.9, 6.7 Hz), 4.86 (dd, 1H, *J* 11.9, 4.0 Hz), 5.29 (dd, 1H, *J* 6.7, 4.0 Hz), 6.92–6.94 (m, 2H), 7.10 (d, 1H, *J* 8.5 Hz), 7.20 (s, 2H), 7.41 (t, 1H, *J* 7.6 Hz), 7.73 (d, 1H, *J* 7.6 Hz), 7.87 (d, 1H, *J* 8.4 Hz), 8.24 (d, 1H, *J* 8.4 Hz). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 34.6, 55.4, 80.6, 89.8, 112.8, 114.4, 115.7, 117.2, 120.8, 121.4, 123.3, 125.0, 131.0, 136.4, 145.5, 154.5, 155.2, 157.1. MS (ESI), *m/z*: 338 [M+H]$^+$, 379 [M+H+MeCN]$^+$. Found (%): C, 64.22; H, 4.36; N, 12.38. Calc. for $C_{18}H_{15}N_3O_4$ (%): C, 64.09; H, 4.48; N, 12.46.

*10-Bromo-12-nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amine* **4c**. Yield 94% (A), 42% (B), gray powder, mp 219 °C. IR (KBr, ν/cm^{-1}): 3507 (NH_2), 3400 (NH_2), 1536 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 4.77 (dd, 1H, *J* 12.1, 6.2 Hz), 4.89 (dd, 1H, *J* 12.1, 3.7 Hz), 5.35 (dd, 1H, *J* 6.2, 3.7 Hz), 7.14 (d, 1H, *J* 8.7 Hz), 7.26 (s, 2H), 7.43 (t, 1H, *J* 7.5 Hz), 7.51 (dd, 1H, *J* 8.7, 2.2 Hz), 7.62 (d, 1H, *J* 2.2 Hz), 7.75 (t, 1H, *J* 7.5 Hz), 7.86 (d, 1H, *J* 8.3 Hz), 8.26 (d, 1H, *J* 8.3 Hz). ^{13}C NMR (150 MHz, DMSO-d_6) δ: 33.9, 80.6, 89.8, 114.9, 116.0, 118.7, 121.1, 123.2, 123.7, 125.0, 131.0, 131.2, 131.5, 136.2, 151.1, 154.0, 157.3. MS (ESI), *m/z*: 386 [M+H]$^+$, 427 [M+H+MeCN]$^+$. Found (%): C, 52.99; H, 3.06; N, 10.75. Calc. for $C_{17}H_{12}BrN_3O_3$ (%): C, 52.87; H, 3.13; N, 10.88.

*10-Chloro-12-nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amine* **4d**. Yield 72% (A), 56% (B), gray powder, mp 225 °C. IR (KBr, ν/cm^{-1}): 3506 (NH_2), 3395 (NH_2), 1537 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 4.77 (dd, 1H, *J* 12.1, 6.3 Hz), 4.90 (dd, 1H, *J* 12.1, 3.8 Hz), 5.35 (dd, 1H, *J* 6.3, 3.8 Hz), 7.20 (d, 1H, *J* 8.7 Hz), 7.27 (s, 2H), 7.39 (dd, 1H, *J* 8.7, 2.5 Hz), 7.43 (t, 1H, *J* 7.6 Hz), 7.49 (d, 1H, *J* 2.5 Hz), 7.75 (t, 1H, *J* 7.6 Hz), 7.86 (d, 1H, *J* 8.3 Hz), 8.26 (d, 1H, *J* 8.3 Hz). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 34.0, 80.5, 89.7, 115.9, 118.3, 121.0, 122.7, 123.7, 125.0, 127.0, 128.1, 128.6, 131.1, 131.2, 150.6, 154.0, 157.3. MS (ESI), *m/z*: 342 [M+H]$^+$, 383 [M+H+MeCN]$^+$. Found (%): C, 59.87; H, 3.49; N, 12.21. Calc. for $C_{17}H_{12}ClN_3O_3$ (%): C, 59.75; H, 3.54; N, 12.30.

*9-Methoxy-12-nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amine* **4e**. Yield 46% (A), 25% (B), yellow powder, mp 196 °C. IR (KBr, ν/cm^{-1}): 3511 (NH_2), 3393 (NH_2), 1561–1542 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 3.78 (s, 3H), 4.63 (dd, 1H, *J* 11.6, 7.0 Hz), 4.80 (dd, 1H, *J* 11.6, 3.9 Hz), 5.24 (dd, 1H, *J* 7.0, 3.9 Hz), 6.73–6.77 (m, 2H), 7.22 (s, 2H), 7.25 (d, 1H, *J* 8.3 Hz), 7.42 (t, 1H, *J* 7.4 Hz), 7.74 (d, 1H, *J* 7.6 Hz), 7.88 (d, 1H, *J* 8.3 Hz), 8.26 (d, 1H, *J* 8.3 Hz). ^{13}C NMR (150 MHz, DMSO-d_6) δ: 33.8, 55.3, 80.9, 90.7, 101.5, 110.3, 112.6, 115.8, 121.0, 123.5, 125.0, 129.1, 131.1, 136.3, 152.5, 154.2, 157.2, 159.6. MS (ESI), *m/z*: 338 [M+H]$^+$, 379 [M+H+MeCN]$^+$. Found (%): C, 64.38; H, 4.35; N, 12.34. Calc. for $C_{18}H_{15}N_3O_4$ (%): C, 64.09; H, 4.48; N, 12.46.

*8-Ethoxy-12-nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amine* **4f**. Yield 73% (A), 71% (B), yellow powder, mp 207 °C. IR (KBr, ν/cm^{-1}): 3499 (NH_2), 3393 (NH_2), 1541 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 1.40 (t, 3H, *J* 7.0 Hz), 4.09 (q, 2H, *J* 7.0 Hz), 4.66 (dd, 1H, *J* 12.0, 7.0 Hz), 4.80 (dd, 1H, *J* 12.0, 3.9 Hz), 5.30 (dd, 1H, *J* 7.0, 3.9 Hz), 6.89 (d, 1H, *J* 7.6 Hz), 7.0 (d, 1H, *J* 8.0 Hz), 7.07 (t, 1H, *J* 7.9 Hz), 7.27 (s, 2H), 7.42 (t, 1H, *J* 7.5 Hz), 7.74 (t, 1H, *J* 7.5 Hz), 7.90 (d, 1H, *J* 8.5 Hz), 8.25 (d, 1H, *J* 8.3 Hz). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 14.7, 34.5, 63.9, 80.7, 90.2, 112.3, 115.9, 119.5, 120.9, 121.4, 123.42, 123.43, 125.0, 131.0, 136.3, 141.1, 146.8, 154.3, 157.2. MS (ESI), *m/z*: 291 [M+H−$MeNO_2$]$^+$, 352 [M+H]$^+$, 393 [M+H+MeCN]$^+$. Found (%): C, 65.10; H, 4.80; N, 11.88. Calc. for $C_{19}H_{17}N_3O_4$ (%): C, 64.95; H, 4.88; N, 11.96.

*8,10-Dichloro-12-nitromethyl-12*H*-chromeno[2,3-*c*]isoquinolin-5-amine* **4g**. Yield 82% (A), 26% (B), yellow powder, mp 260 °C. IR (KBr, ν/cm^{-1}): 3504 (NH_2), 3390 (NH_2), 1543 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 4.84 (dd, 1H, *J* 12.4, 6.0 Hz), 4.93 (d, 1H, *J* 12.4, 3.8 Hz), 5.39 (dd, 1H, *J* 6.0, 3.8 Hz), 7.40 (s, 2H), 7.45 (t, 1H, *J* 7.6 Hz), 7.49 (d, 1H, *J* 2.3 Hz), 7.65 (d, 1H, *J* 2.3 Hz), 7.75 (t, 1H, *J* 7.7 Hz), 7.85 (d, 1H, *J* 8.4 Hz), 8.26 (d, 1H, *J* 8.3 Hz). ^{13}C NMR (150 MHz, DMSO-d_6) δ: 34.3, 80.4, 89.6, 116.2, 121.2, 121.5, 124.0, 124.3, 125.0, 127.0, 127.2, 128.6, 131.3, 136.0, 146.8, 153.6, 157.5. MS (ESI), *m/z*: 376 [M+H]$^+$, 417 [M+H+MeCN]$^+$. Found (%): C, 54.40; H, 2.85; N, 11.08. Calc. for $C_{17}H_{11}Cl_2N_3O_3$ (%): C, 54.28; H, 2.95; N, 11.17.

*14-Nitromethyl-14*H*-benzo[5,6]chromeno[2,3-*c*]isoquinolin-5-amine* **4h**. Yield 20% (A), light-orange powder, mp 203 °C. IR (KBr, ν/cm^{-1}): 3504 (NH_2), 3394 (NH_2), 1548 (NO_2). ^{1}H NMR (600 MHz, DMSO-d_6) δ: 4.92 (d, 2H, *J* 3.6 Hz), 6.01 (m, 1H), 7.24 (s, 2H), 7.37 (d, 1H, *J* 8.8 Hz), 7.46 (t, 1H, *J* 7.5 Hz), 7.54 (t, 1H, *J* 7.5 Hz), 7.70 (t, 1H, *J* 7.5 Hz), 7.78 (t, 1H, *J* 7.5 Hz), 7.96 (d, 1H, *J* 8.8 Hz), 8.00 (d, 1H, *J* 8.0 Hz), 8.29 (d, 1H, *J* 8.0 Hz), 8.32 (d, 1H, *J* 8.3 Hz), 8.48 (d, 1H, *J* 8.3 Hz). ^{13}C NMR (150 MHz, DMSO-d_6) δ: 31.5, 79.4, 90.6, 112.1, 116.1, 117.6, 121.8, 122.6, 123.6, 124.6, 124.9, 127.3, 128.8, 129.5, 130.4, 130.6, 130.9, 136.6, 150.1, 154.1, 157.2. MS (ESI), *m/z*: 297 [M+H−$MeNO_2$]$^+$, 358 [M+H]$^+$, 399 [M+H+MeCN]$^+$. Found (%): C, 70.77; H, 4.29; N, 11.68. Calc. for $C_{21}H_{15}N_3O_3$ (%): C, 70.58; H, 4.23; N, 11.76.