One-Pot Synthesis of Simple Alkaloids: 2,3-Polymethylene-4(3*H*)-quinazolinones, Luotonin A, Tryptanthrin, and Rutaecarpine

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One-pot synthesis of various 2,3-polymethylene-4(3*H*)-quinazolinones from anthranilic acid, corresponding lactam and $SOCl_2$ is described, which can be applicable to the synthesis of simple 4(3*H*)-quinazolinone-derived alkaloids, such as luotonin A, tryptanthrin, and rutaecarpine.

Key words deoxyvasicinone; mackinazolinone; luotonin A; tryptanthrin; rutaecarpine; anthranilic acid

2,3-Trimethylene-4(3*H*)-quinazolinone (deoxyvasicinone, **1aa**)[1] and 2,3-tetramethylene-4(3*H*)-quinazolinone (mackinazolinone, **1ba**)[2,3] are not only the alkaloids isolated from *Peganum* and *Mackinlaya* species, respectively, but are also prepared chemically before the isolation from natural sources.[4—6] The establishment of a simple and efficient synthetic method for these alkaloids has long been a challenging subject.[7—20] In addition, these substances are a part of the family of intriguing alkaloids that include the bronchodilator vasicinone (**2a**) from *Adhatoda vasica*,[21] antiendotoxic isaindigotone (**2b**) from *Isatis indigotica*,[22] topoisomerase I-dependent cytotoxic luotonin A (**3**) from *Peganum nigellastrum*,[23] antibiotic tryptanthrin (**4**) from yeast (*Candida lipolytica*)[24,25] and later from cannon ball tree (*Cououpita guianensis*),[26] anti-inflammatory rutaecarpine (**5**)[27] from *Evodia rutaecarpa*, and related alkaloids (Fig. 1).[28]

Since Niementowski's preparation of 4(3*H*)-quinazolinone by fusing anthranilic acid with formamide,[29] several methods attempting the synthesis of modified quinazolinones have been reported.[1,30—33] Although most of these are applicable to acyclic amides, methods for cyclic amides, especially without an aryl group, are somewhat limited.[34—37] These limitations have led to the continuous development of new procedures for cyclic amides, such as cyclocondensation of isatoic anhydride with lactams,[12] metal catalyzed reductive *N*-heterocyclization of *N*-(2-nitrobenzoyl)lactams in the presence of CO,[13,14] reaction of 2-aminobenzamide with succinic anhydride,[15] intramolecular aza-Wittig reaction of *N*-(2-azidobenzoyl)lactams,[9,16] condensation of anthranilic acid with *O*-alkyllactims[6,18—20] or *S*-alkyllactims,[8] reaction of anthranilic acid with $SOCl_2$ followed by cyclization with lactams,[17] cyclization of iminochlorides generated from lactam and methyl anthranilate,[11] solid-phase synthesis,[7,9] and microwave assisted synthesis.[10,12] Nevertheless, the biological importance of 4(3*H*)-quinazolinone and related compounds still spurred the development of a new and simple synthetic method for theses compounds.[38—43]

As a part of our research on biologically important natural products, we herein described a simple one-pot procedure for the preparation of **1** as well as related alkaloids **3**, **4** and **5** in multi-gram quantity.

Although an earlier study revealed that iminoketene (**8**), generated from the reaction of anthranilic acid (**6**) and $SOCl_2$, underwent cycloaddition to cyclic iminols, which are tautomers of the corresponding lactams (**9**), to yield **1** in 70—82% yield,[19] we sometimes have been unable to repeat the reactions.[44] Whenever we failed to generate the desired products, imine compounds (**10**) were isolated as major products (Chart 1). Although we could convert these imines (**10**) to the desired 2,3-tri- and 2,3-tetramethylene-4(3*H*)-quinazolinones by employing polyphosphoric acid-catalyzed dehydration (see Experimental section in details), we examined and adjusted the reaction condition to find that such reaction could be carried out in a single step. One-pot reaction of anthranilic acid, lactams (**9**) and $SOCl_2$ in refluxing benzene yielded **1aa** and **1ba** in 87% and 78% yield, respectively.

The generality of the process was examined with a variety of substituted anthranilic acids (**6**) and various lactams (**9**) to provide the corresponding 2,3-polymethylene-4(3*H*)-quinazolinone derivatives (**1**) in 78—89% yields (Table 1).

The electronic properties of the substituent on anthranilic acid did not seem to greatly affect the reaction (compounds **1ab**, **1ac**, **1ad**, **1ag** *vs.* **1ae**, **1af**). It is worthy to note that the

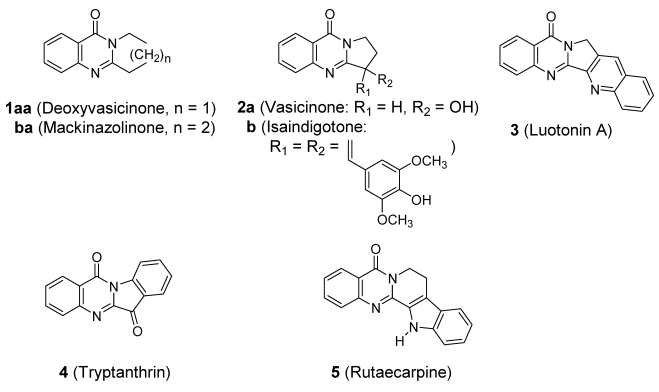

Fig. 1. Simple Alkaloids Derived from 4(3*H*)-Quinazolinone

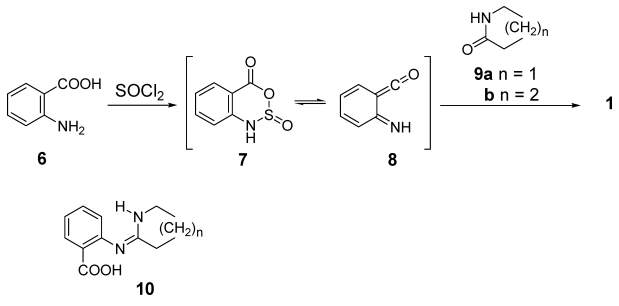

Chart 1

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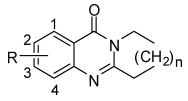
Table 1. Synthesis of 2,3-polymethylene-4(3*H*)-quinazolinones



Compd.	R	*n*	Yield (%)	mp (°C)	Compd.	R	*n*	Yield (%)	mp (°C)
1aa	H	1	87	109 [104[1)]]	**1bb**	2-Cl	2	87	107 [106[47)]]
1ab	2-Cl	1	85	177 [178—179[45)]]	**1bc**	2-Br	2	87	163—164
1ac	2-Br	1	86	158 [157—158[45)]]	**1bd**	2-I	2	84	286—287
1ad	2-I	1	87	150 [148—150[45)]]	**1be**	2-OH	2	85	257—259
1ae	2-OH	1	86	289—290	**1bf**	2-CH_3	2	88	90 [204—206[a)46)]]
1af	2-CH_3	1	89	110 [174—175[a)46)]]	**1bg**	2-NO_2	2	88	172 [171—172[46)]]
1ag	2-NO_2	1	85	187 [187—188[45)]]	**1bh**	3-Cl	2	82	133 [132—133[18)]]
1ah	3-Cl	1	86	188 [186—188[18)]]	**1ca**	H	3	78	97 [95—97[6)]]
1ba	H	2	78	99 [98.5—99.5[2)]]	**1da**	H	4	82	112 [109—110[16)]]

a) Melting point of corresponding HCl salt.

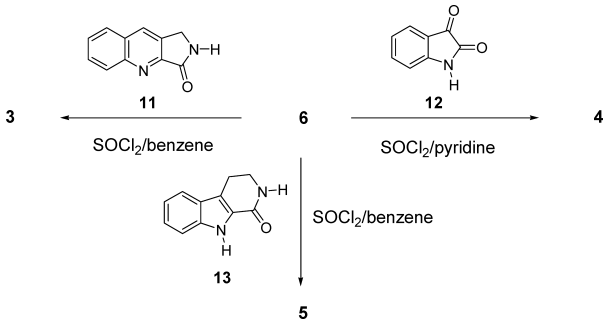


Chart 2

present method is applicable to compounds with an OH group. Although the compound with an OH group, 2-hydroxy-7,8-dihydro-6*H*,11*H*-pyrrolo[2,1-*b*]quinazolin-11-one (**1ae**), was previously prepared in two steps: condensation of corresponding 5-methoxyanthranilic acid with *O*-ethylbutyrolactim followed by acid catalyzed demethylation,[47)] the compounds with an OH as a substituent were prepared in 85—86% yields (compounds **1ae**, **1be**).

Following the reactions shown above, known lactams, 3-oxo-1*H*-pyrrolo[3,4-*b*]quinoline (**11**),[48)] isatin (**12**), and 1-oxo-1,2,3,4-tetrahydropyrido[3,4-*b*]indole (**13**),[49)] were converted to the corresponding alkaloids luotonin A (**3**), tryptanthrin (**4**), and rutaecarpine (**5**), respectively, in 78—85% yields (Chart 2).

It should be noted that the reactions of anthranilic acid or its esters with lactams in refluxing $POCl_3$ have already been attempted, but the yields were generally low.[6,45,50)] This has limited the utility for the alkaloids derived from 4(3*H*)-quinazolinone.

In conclusion, a one-pot procedure for the preparation of various 2,3-polymethylene-4(3*H*)-quinazolinones has been established by a reaction of anthranilic acid, lactam, and $SOCl_2$. The described procedure was applied to the preparation of three simple 4(3*H*)-quinazolinone alkaloids, luotonin A, tryptanthrin, and rutaecarpine.

Experimental

Melting points were determined using a Fischer–Jones melting points apparatus and are not corrected. IR spectra were obtained using a Perkin-Elmer 1330 spectrophotometer. NMR spectra were obtained using a Bruker-250 spectrometer 250 MHz for ^{1}H-NMR and 62.5 MHz for ^{13}C-NMR and are reported as parts per million (ppm) from the internal standard tetramethylsilane. Chemicals and solvents were commercial reagent grade and used without further purification. The compounds **11**[49)] and **13**[50)] were prepared by employing previously reported methods. Electrospray ionization (ESI) mass spectrometry (MS) experiments were performed on a LCQ advantage-trap mass spectrometer (Thermo Finnigan, San Jose, CA, U.S.A.). Elemental analyses were performed with a Hewlett-Packard Model 185B elemental analyzer.

General Procedure of 2,3-Polymethylene-4(3*H*)-quinazolinones A solution of anthranilic acid (1.37 g, 0.01 mol), lactam (0.12 mol) and $SOCl_2$ (2 ml, *ca.* 2 eq) in dry benzene or pyridine (20 ml) was refluxed for 4 h. The reaction mixture was poured to ice-water and made basic with NH_4OH (100 ml), and the precipitate was collected as a crude product.

2-Bromo-7,8-dihydro-6*H*,11*H*-pyrrolo[2,1-*b*]quinazolin-11-one (1ac) Yellow needles: mp 158 °C. Unreported spectral data are as follows: ^{1}H-NMR (DMSO-d_6, 250 MHz) δ: 8.20 (1H, d, J=1.8 Hz, H1), 7.99 (1H, dd, J=8.5, 1.8 Hz, H3), 7.65 (1H, d, J=8.5 Hz, H4), 4.08 (2H, t, J=7.3 Hz), 3.17 (2H, d, J=7.3 Hz), 2.20 (2H, quintet, J=7.3 Hz). ESI-MS *m*/*z*: 266 [Calcd for $C_{11}H_9BrN_2O$: 266 (M+1)]. *Anal.* Calcd for $C_{11}H_9BrN_2O$: C, 49.84; H, 3.42; N, 10.57. Found: C, 49.81; H, 3.43; N, 10.58.

2-Iodo-7,8-dihydro-6*H*,11*H*-pyrrolo[2,1-*b*]quinazolin-11-one (1ad) Yellow needles: mp 150 °C. Unreported spectral data are as follows: ^{1}H-NMR (DMSO-d_6, 250 MHz) δ: 8.37 (1H, s, H1), 8.11 (1H, dd, J=8.5, 2.0 Hz, H3), 7.46 (1H, d, J=8.5 Hz, H4), 4.07 (2H, t, J=7.3 Hz), 3.14 (2H, d, J=7.3 Hz), 2.18 (2H, quintet, J=7.3 Hz). ESI-MS *m*/*z*: 313 [Calcd for $C_{11}H_9IN_2O$: 313 (M+H)]. *Anal.* Calcd for $C_{11}H_9IN_2O$: C, 42.33; H, 2.91; N, 8.98. Found: C, 42.27; H, 3.03; N, 8.98.

2-Hydroxy-7,8-dihydro-6*H*,11*H*-pyrrolo[2,1-*b*]quinazolin-11-one (1ae) White needles: mp 289—290 °C. ^{1}H-NMR (DMSO-d_6, 250 MHz) δ: 10.50 (br s, OH), 7.65 (1H, d, J=8.8 Hz, H4), 7.45 (1H, d, J=2.5 Hz, H1), 7.38 (1H, dd, J=8.5, 2.5 Hz, H3), 4.09 (2H, t, J=7.0 Hz), 3.26 (2H, d, J=7.6 Hz), 2.23 (2H, quintet, J=7.6 Hz). ESI-MS *m*/*z*: 203 [Calcd for $C_{11}H_{10}N_2O_2$: 203 (M+H)]. *Anal.* Calcd for $C_{11}H_{10}N_2O_2$: C, 65.34; H, 4.98; N, 13.85. Found: C, 65.35; H, 5.01; N, 13.84.

2-Nitro-7,8-dihydro-6*H*,11*H*-pyrrolo[2,1-*b*]quinazolin-11-one (1ag) Yellow needles: mp 187 °C. Unreported spectral data are as follows: ^{1}H-NMR (DMSO-d_6, 250 MHz) δ: 8.78 (1H, d, J=2.0 Hz, H1), 8.52 (1H, dd, J=8.5, 2.0 Hz, H3), 7.79 (1H, d, J=8.5 Hz, H4), 4.09 (2H, t, J=7.3 Hz), 3.16 (2H, d, J=7.3 Hz), 2.20 (2H, quintet, J=7.3 Hz). ESI-MS *m*/*z*: 232 [Calcd for $C_{11}H_9N_3O_3$: 232 (M+H)]. *Anal.* Calcd for $C_{11}H_9N_3O_3$: C, 57.14; H, 3.92; N, 18.17. Found: C, 57.19; H, 3.93; N, 18.08.

2-Bromo-6,7,8,9-tetrahydro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (1bc) White needles: mp 163—164 °C. ^{1}H-NMR (DMSO-d_6, 250 MHz) δ: 8.22 (1H, d, J=2.3 Hz, H1), 8.04 (1H, dd, J=8.8, 2.3 Hz, H3), 7.71 (1H, d, J=8.8 Hz, H4), 3.93 (2H, t, J=6.0 Hz), 3.06 (2H, d, J=6.0 Hz), 1.98—1.82 (4H, m). ESI-MS *m*/*z*: 279 [Calcd for $C_{12}H_{11}BrN_2O$: 279 (M+H)]. *Anal.* Calcd for $C_{12}H_{11}BrN_2O$: C, 51.63; H, 3.97; N, 10.04. Found: C, 51.65; H, 3.95; N, 10.04.

2-Iodo-6,7,8,9-tetrahydro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (1bd) Yellow needles: mp 286—287 °C. ^{1}H-NMR (DMSO-d_6, 250 MHz) δ: 8.41 (1H, d, J=2.0 Hz, H1), 8.20 (1H, dd, J=8.8, 2.3 Hz, H3), 7.58 (1H, d, J=8.8 Hz, H4), 3.93 (2H, t, J=6.0 Hz), 3.08 (2H, d, J=6.0 Hz), 1.97—1.82

(4H, m). ESI-MS *m/z*: 327 [Calcd for $C_{12}H_{11}IN_2O$: 327 (M+H)]. *Anal.* Calcd for $C_{12}H_{11}IN_2O$: C, 44.19; H, 3.40; N, 8.59. Found: C, 44.21; H, 3.42; N, 8.61.

2-Hydroxy-6,7,8,9-tetrahydro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (1be) White needles: mp 257—259 °C. ^{1}H-NMR (DMSO-d_6, 250 MHz) δ: 10.67 (br s, OH), 7.69 (d, 1H, J=8.3 Hz, H4), 7.45 (d, 1H, J=0.8 Hz, H1), 7.43 (dd, 1H, J=8.3, 0.8 Hz, H3), 3.95 (t, 2H, J=5.6 Hz), 3.11 (d, 2H, J=5.6 Hz), 1.92 (quintet, 2H, J=5.6 Hz), 1.87 (quintet, 2H, J=5.6 Hz). ESI-MS *m/z*: 217 [Calcd for $C_{12}H_{12}N_2O_2$: 217 (M+H)]. *Anal.* Calcd for $C_{12}H_{12}N_2O_2$: C, 66.65; H, 5.59; N, 12.96. Found: C, 66.62; H, 5.61; N, 12.97.

Luotonin A (3) The crude product was recrystallized from $CHCl_3$ to yield pale yellow needles (78%): mp 281—283 °C (dec.) (lit.[23] mp 252 °C (dec.), lit.[51] mp 281—283 °C). Spectral data were identical to those reported previously.[23]

Tryptanthrin (4) The crude product was recrystallized from EtOH to yield **4** (85%) as yellow needles: mp 266—267 °C (lit.[26] mp 267—268 °C). Spectral data were identical to those reported previously.[11]

Rutaecarpine (5) The crude product was recrystallized from CH_3OH to yield **5** (82%) as white needles: mp 259—260 °C (lit.[27] mp 258 °C). Spectral data were identical to those reported previously.[52]

10b: A solution of anthranilic acid (13.72 g, 0.10 mol) and $SOCl_2$ (50 g) in dry benzene (500 ml) was refluxed for 2 h. The solvent was then evaporated under reduced pressure at room temperature to afford iminoketene as an oily liquid, to which a solution of 2-pyrrolidinone (7.20 g, 0.085 mol) in dry benzene (100 ml) was added. After setting overnight at room temperature, the precipitate formed was collected. Recrystallization from EtOH gave 20.0 g (91%) of white needles: mp 180 °C. IR (KBr) cm^{-1}: 3200—2500, 1702, 1644. ^{1}H-NMR (250 MHz, $CDCl_3$) δ: 10.92 (br s, D_2O exchangeable, OH), 8.22 (1H, dd, J=7.5, 1.3 Hz, H6), 8.19 (1H, d, J=7.8 Hz, H3), 7.78 (1H, td, J=8.5, 1.3 Hz, H5), 7.54 (1H, td, J=8.5, 1.3 Hz, H4), 4.15 (2H, t, J=6.2 Hz), 3.63 (2H, t, J=6.2 Hz), 2.18 (2H, quintet, J=6.2 Hz), 2.05 (2H, quintet, J=6.2 Hz). ^{13}C-NMR (62.5 MHz, $CDCl_3$) δ: 160.55, 158.51, 136.87, 136.55, 128.98, 127.49, 119.99, 117.85, 43.62, 27.82, 21.08, 17.92, 17.16. ESI-MS *m/z*: 219 [Calcd for $C_{12}H_{14}N_2O_2$ (M+H): 219]. MS *m/z*: 218 (M^+, 12) 200 (M^+-H_2O, 100), 173 (M^+-COOH, 4), 160 (11). *Anal.* Calcd for $C_{12}H_{14}N_2O_2$: C, 66.04; H, 6.47; N, 12.84. Found: C, 66.08; H, 6.43; N, 12.89.

2,3-Tetramethylene-4(3*H*)-quinazolinone (6,7,8,9-Tetrahydro-11*H*-pyrido[2,1-*b*]quinazolin-11-one, 1ba) A mixture of **10b** (4.36 g, 0.02 mol) in polyphosphoric acid (40 ml) was heated at 100—120 °C for 1 h. The reaction mixture was poured to ice (400 g) and the resulting mixture was made basic with 1 N NaOH. The precipitate formed was collected and recrystallized from EtOH to afford white needles (3.48 g, 87%): mp 98—99 °C (lit.[2] mp 98.5—99.5 °C). ^{1}H-NMR (250 MHz, $CDCl_3$) δ: 8.23 (1H, dd, J=8.0 Hz, H1), 7.68 (1H, td, J=8.3, 1.3 Hz, H2), 7.55 (1H, d, J=8.0 Hz, H4), 7.39 (1H, td, J=8.0, 1.0 Hz, H3), 4.05 (2H, t, J=6.2 Hz), 2.97 (2H, t, J=6.5 Hz), 2.01—1.87 (4H, m).

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