Monatshefte für Chemie - Chemical Monthly (2019) 150:747–756
https://doi.org/10.1007/s00706-018-2342-4

ORIGINAL PAPER

Synthesis of new 4-(1,2,3-triazolo)quinolin-2(1*H*)-ones via Cu-catalyzed [3 + 2] cycloaddition

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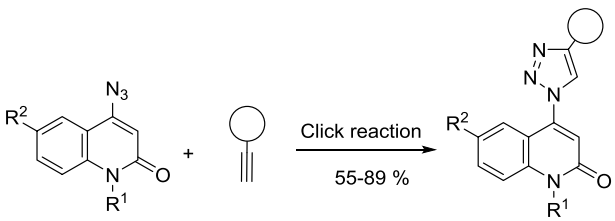
Received: 26 October 2018 / Accepted: 26 November 2018 / Published online: 9 March 2019

Abstract
In this investigation, new classes of 1,2,3-triazoles derived by 2-quinolone have been synthesized, via Cu-catalyzed [3 + 2] cycloadditions (Meldal-Sharpless 'click' reactions) of 4-azidoquinolin-2(1*H*)-ones with some alkynes. The structures of the products have been confirmed by different spectroscopic analyses.

Graphical abstract



Keywords 4-Azidoquinolin-2(1*H*)-ones · Terminal alkynes · 4-(1,2,3-triazolyl)quinolin-2(1*H*)-ones · Click reaction · Cycloaddition

Introduction

Quinoline molecules are present commonly in living organisms as important secondary metabolites [1], whereas 1,2,3-triazoles, chemically inert compounds, have not been detected in naturally occurring products [2]. The attachment of quinoline to 1,2,3-triazole skeletons in diverse and numerous ways, would yield valuable biomolecules for drug development. Moreover, 1,2,3-triazole can act as pharmacophores and linkers between quinoline and other pharmacophoric molecules that of interest in molecular hybridization approaches [3, 4].

Due to the large number of diseases, scientists tend to synthesize homogeneous and heterogeneous ring compounds that treated many of these diseases. One class of these compounds is 2-quinolones [5], which have received considerable attention in recent years because of their pharmacological [6] importance and various biological activities [7]. They possess antimicrobial [8], antifungal [9], anticancer [10], anti-HIV [11], anti-oxidant [12], enzyme inhibitory [13], and cytotoxic activities [14]. A second type of these compounds is 1,2,3-triazoles, which have assorted biological activities such as antibacterial, anti-tubercular, anticancer, antifungal, anti-tubercular, and which like 2-quinolones have anti-HIV properties [15, 16]. After development of the copper-catalyzed [3 + 2] cycloaddition of organic azides at terminal alkynes under mild conditions, the importance and wide application of 1,2,3-triazole compounds have increased significantly [17, 18]. Regioselective formation of 1,2,3-triazoles has proved to be the best example of click chemistry [19] and has found extensive applications in manifold domains of

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chemistry [20, 21]. Cu-catalyzed [3 + 2] cycloadditions of azides and alkynes (Meldal-Sharpless 'click' reaction) to give 1,2,3-triazoles have been used extensively together with a variety of functions to biomolecules [22–24]. Previously, it was reported on the selective synthesis of 1,2,3-triazole systems via non-catalyzed azide/acetylene [3 + 2] cycloadditions that are possible in the case of electrophilic activate acetylene derivatives [25]. Aly et al. reported the synthesis of ethyl pyrano[3,2-*c*]quinoline-4-carboxylates [26] and spiro(indoline-3,4′-pyrano[3,2-*c*]-quinoline)-3′-carbonitriles [27]. Besides that we have reported on one-pot synthesis of 2,3-bis(4-hydroxy-2-oxo-1,2-dihydroquinolin-3-yl)succinates and arylmethylene-bis(3,3′-quinoline-2-ones) [28]. Moreover, two series of *N*-2,3-bis(6-substituted-4-hydroxy-2-oxo-1,2-dihydroquinolin-3-yl)-naphthalene-1,4-diones and substituted *N*-(methyl/ethyl)bis-quinolinone triethylammonium salts were synthesized. The synthesized compounds were targeted as candidates to extracellular signal-regulated kinases 1/2 (ERK1/2) with considerable antineoplastic activity [29]. In a very recent approach, design and synthesis of novel series of fused naphthofuro[3,2-*c*]quinoline-6,7,12-triones and pyrano[3,2-*c*]quinoline-6,7,8,13-tetraones as potential ERK inhibitors were reported [30]. The new inhibitors were synthesized and identified by different spectroscopic techniques and X-ray crystallography. They were evaluated for their ability to inhibit ERK1/2 in an in vitro radioactive kinase assay [30]. In this paper, we design to synthesize products that combine the qualities of each of them together in one molecule by applying click-chemistry techniques between 4-azidoquinolin-2(1*H*)-ones and different terminal alkynes. We hope that the aforementioned stuff shows prospective biological activities.

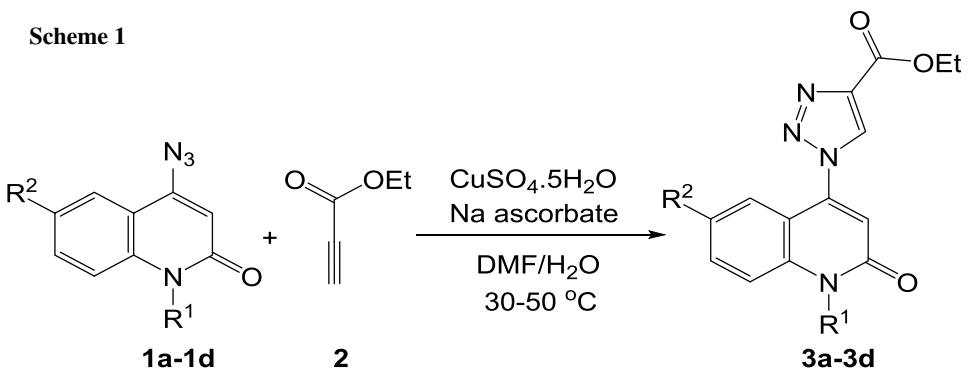
Results and discussion

Herein, we report the cycloaddition of 4-azidoquinolin-2(1*H*)-ones **1a**–**1d** with ethyl propiolate (**2**) to give, in good to excellent yields, the corresponding ethyl 1-(2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazole-4-carboxylates **3a**–**3d** (Scheme 1), under the reaction conditions $CuSO_4$/sodium ascorbate/DMF. The mixture of alkyne **2** with sodium ascorbate, $CuSO_4$, and 4-azido compounds **1a**–**1d** was gently heated for 12 h.

The solids of 1,2,3-triazoles **3a**–**3d** were appeared as colorless products. Their structures were confirmed by different spectroscopic methods such as elemental analyses, IR, and NMR (^{1}H, ^{13}C, HMBC, HSQC, and ^{15}N) and in addition to mass spectrometry were in good agreement with the assigned product structures. The elemental analyses and the mass spectra showed that compounds **3a**–**3d** are formed from one molecule of 4-azidoquinolin-2(1*H*)-one **1a**–**1d** and another of ethyl propiolate (**2**). Compounds **3a**–**3c** exhibited NH stretching in IR spectra at $\bar{\nu} = 3139$–3127 cm^{-1}, but compound **3d** did not. Other major features of the IR spectra of **3a**–**3d** were two carbonyl bands at $\bar{\nu} = 1740$–1717 cm^{-1} and 1677–1665 cm^{-1} for quinolinone-C-2 and ester carbonyls, respectively, which were further confirmed by ^{13}C NMR spectrum data which exhibited signals at $\delta_C = 159.10$–160.84 ppm for C-2 and 158.82–159.94 ppm for C-4a′.

Compound **3a** which was assigned as ethyl 1-(6-methyl-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazole-4-carboxylate (Fig. 1) has spectral data as shown in Table 1. The ^{1}H NMR of compound **3a** as an example showed methyl protons H-4c′ as a triplet signal at $\delta_H = 1.35$ ppm with coupling constant $J = 7.1$ Hz, which was confirmed from ^{13}C NMR at $\delta_C = 14.16$ ppm. H-4c′ gives COSY correlation, and C-4c′ gives HMBC correlation to the methylene protons H-4b′ as quartet at $\delta_H = 4.39$ ppm with coupling constant $J = 7.1$ Hz; the attached carbon appears at

Scheme 1



1a-1d **2** **3a-3d**

1,3: a, $R^1 = R^2 = H$; **b**, $R^1 = H$, $R^2 = Me$; **c**, $R^1 = H$, $R^2 = OMe$; **d**, $R^1 = Me$, $R^2 = H$

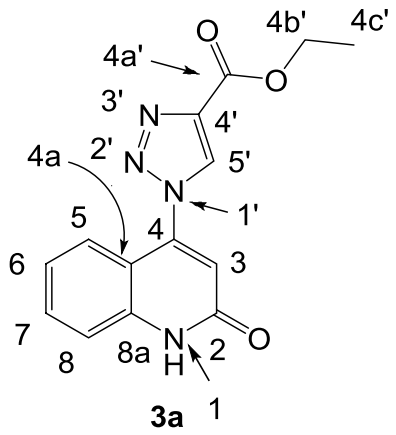


Fig. 1 Distinctive carbons of ethyl 1-(6-methyl-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazole-4-carboxylate (**3a**)

$\delta_C = 60.88$ ppm. Also, H-4b′ gives HMBC correlation with carbonyl carbon C-4a′ at $\delta_C = 159.91$ ppm. Furthermore, the ^{1}H NMR of compound **3a** showed a broad singlet at $\delta_H = 12.33$ ppm, due to quinoline-NH. On the other hand, the ^{13}C NMR spectrum of compound **3a** showed signals at $\delta_C = 160.84$, 131.11, 114.47, and 60.88 ppm, which were assigned as C-2, C-5′, C-4a, and C-4b′, respectively.

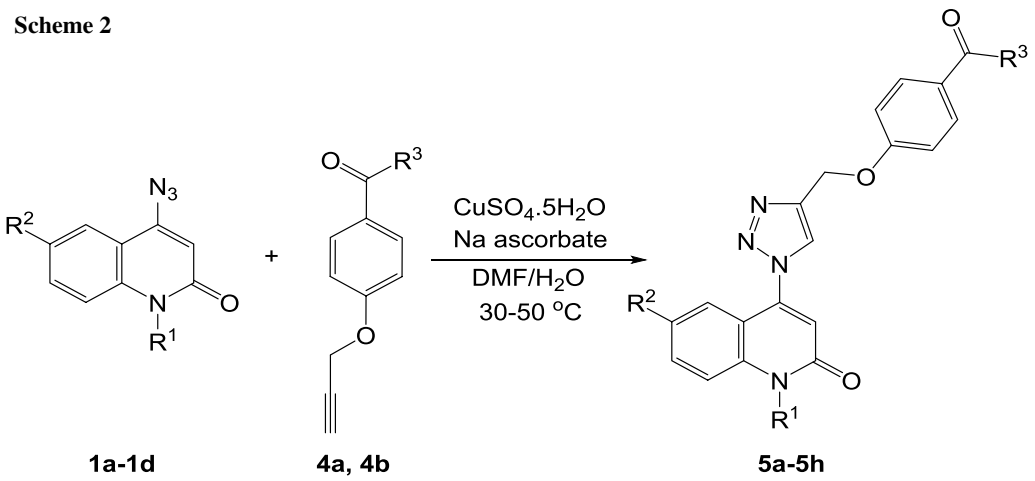
Based on these results, we applied similar methodology to other terminal alkynes, by allowing 4-(prop-2-yn-1-yloxy)-benzaldehyde (**4a**) or 1-[4-(prop-2-yn-1-yloxy)phenyl]ethanone (**4b**) to react with 4-azidoquinolin-2(1*H*)-ones **1a**–**1d** (Scheme 2). To illustrate our results, NMR (^{1}H, ^{13}C, ^{1}H-^{1}H COSY, HMBC, HSQC, and ^{15}N) was performed for all the obtained products. As an example of the NMR, spectroscopic data of compound **5a** (Fig. 2) are illustrated in Table 2.

We choose compound **5a** as an example to confirm the structures. Elemental analysis and mass spectrometry show that compound **5a** has gross formula $C_{19}H_{14}N_4O_3$ which

Table 1 Spectroscopic data of compound **3a**

^{1}H NMR		^{1}H-^{1}H COSY	Assig.
12.33 (s, 1H)		6.95	NH
9.39 (s, 1H)			H-5′
7.66 (“t”, J = 7.3 Hz, 1H)		7.48, 7.25	H-7
7.48 (d, J = 8.2 Hz, 1H)		7.66, 7.38, 7.25	H-8
7.38 (d, J = 8.0 Hz, 1H)		7.48, 7.25	H-5
7.25 (“t”, J = 7.6 Hz, 1H)		7.66, 7.48, 7.38	H-6
6.95 (s, 1H)		12.33	H-3
4.39 (q, J = 7.1 Hz, 2H)		1.35	H-4b′
1.35 (t, J = 7.1 Hz, 3H)		4.39	H-4c′
^{15}N NMR	HSQC	HMBC	Assig.
367.4		9.39	N-2′/3′
359.7		9.39	N-3′/2′
250.0		9.39, 6.95	N-1′
152.4	12.33		N-1
^{13}C NMR	HSQC	HMBC	Assig.
160.84			C-2
159.91		4.39	C-4a′
143.16		7.38, 6.95	C-8a
139.30, 139.22		9.39, 7.66, 7.38, 7.25	C-4,4′
132.00	7.66	7.38, 7.25	C-7
131.11	9.39		C-5′
123.90	7.38	7.66	C-5
122.68	7.25	7.48	C-6
118.76	6.95		C-3
115.86	7.48	7.66, 7.48, 7.25, 6.95	C-8
114.47		6.95	C-4a
60.88	4.39	1.35	C-4b′
14.16	1.35	4.39	C-4c′

Scheme 2



1a-1d **4a, 4b** **5a-5h**

1: **a**, $R^1 = R^2 = H$; **b**, $R^1 = H$, $R^2 = Me$; **c**, $R^1 = H$, $R^2 = OMe$; **d**, $R^1 = Me$, $R^2 = H$
4: **a**, $R^3 = H$; **b**, $R^3 = Me$
5: **a**, $R^1 = R^2 = R^3 = H$; **b**, $R^1 = R^3 = H$, $R^2 = Me$; **c**, $R^1 = R^3 = H$, $R^2 = OMe$;
d, $R^1 = Me$, $R^2 = R^3 = H$; **e**, $R^1 = R^2 = H$, $R^3 = Me$; **f**, $R^1 = H$, $R^2 = R^3 = Me$;
g, $R^1 = H$, $R^2 = OMe$, $R^3 = Me$; **h**, $R^1 = R^3 = Me$, $R^2 = H$

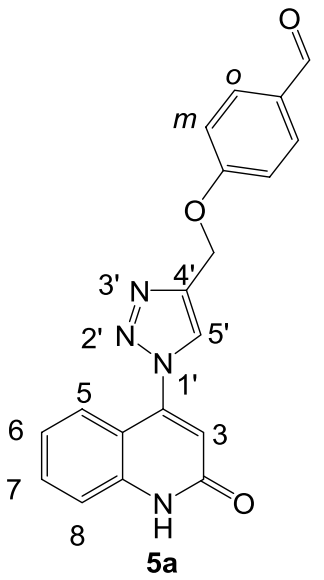


Fig. 2 Distinctive carbons of 4-[[(1-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl]methoxy]benzaldehyde (**5a**)

comes from the reaction of one mole of 4-azidquinoline-2(1*H*)-one (**1a**) with one mole of 4-(prop-2-yn-1-yloxy)-benzaldehyde (**4a**) (Fig. 2). The IR spectra of compound **5a** showed signals at $\bar{\nu} = 3124$ cm^{-1}, due to NH stretching, 2829 cm^{-1} for aliph.-CH, 1691, 1669 cm^{-1} due to two carbonyl groups, in addition to the aromatic absorption bands (Fig. 3). Further, the ^1H NMR spectrum (DMSO-d_6) (Table 2), for compound **5a**, showed signals at $\delta_H = 12.29$, 9.91, 8.92 ppm, due to quinoline-NH, CH=O, and triazole-H-5′, respectively. Two signals appeared as singlet at $\delta_H = 6.87$ and 5.44 ppm, which were assigned as quinoline-H-3 and methyl group H-4a′, respectively. ^{13}C NMR spectrum for compound **5a** showed two downfield signals at $\delta_C = 191.33$ and 162.84 ppm which were assigned as CH=O and C-4, respectively. Furthermore, other signals at $\delta_C = 126.85$, 117.79, 114.42, and 61.20 ppm were assigned as C-5′, C-3, C-4a, and C-4a′, respectively, in addition to aromatic signals.

Similarly, the compound **5h** comes from the reaction of equimolar amounts of 4-azido-1-methylquinolin-2(1*H*)-one (**1d**) and 1-[4-(prop-2-yn-1-yloxy)phenyl]ethanone (**4b**) (Fig. 3).

To confirm the structures of all obtained products, we chose focused the network coupling of compound **5h** (Table 3). The C–CH_3 singlet is distinctive at $\delta_H = 2.54$ ppm and is assigned as H-b; the attached carbon appears at $\delta_C = 26.41$ ppm. H-b gives HMBC correlation with a carbon at $\delta_C = 196.30$ ppm, assigned as C-a, and a carbon at $\delta_C = 130.28$ ppm, assigned as C-*i*. C-a gives HMBC correlation with a 2H doublet at $\delta_H = 7.98$ ppm, assigned as H-*o*; this is a three-bond coupling. The attached carbon appears at $\delta_C = 130.48$ ppm. H-*o* gives COSY correlation and C-*i* gives HMBC correlation, with the other 2H doublet at $\delta_H = 7.23$ ppm, assigned as H-*m*; the attached carbon appears at $\delta_C = 114.61$ ppm. The correlation between C-*i* and H-*m* is another three-bond correlation. Both H-*o* and H-*m* give HMBC correlation with a carbon at $\delta_C = 161.75$ ppm, assigned as C-*p*. Further, C-*p* gives HMBC correlation with

Table 2 Spectroscopic data of compound **5a**

^{1}H NMR	^{1}H-^{1}H COSY	Assig.
12.29 (s, 1H)		NH
9.91 (s, 1H)		CH = O
8.92 (s, 1H)		H-5′
7.92 (d, J = 8.5 Hz, 2H)	7.32	H–*o*
7.66 (“t”, J = 7.6 Hz, 1H)	7.48, 7.25	H-7
7.48 (d, J = 8.9 Hz, 1H)	7.66	H-8
7.46 (d, J = 9.0 Hz, 1H)	7.25	H-5
7.32 (d, J = 8.5 Hz, 2H)	7.92	H-*m*
7.25 (“t”, J = 7.6 Hz, 1H)	7.66, 7.46	H-6
6.87 (s, 1H)		H-3
5.44 (s, 2H)		H-4a′

^{15}N NMR	HSQC	HMBC	Assig.
247.7		8.92, 6.87	N-1′

^{13}C NMR	HSQC	HMBC	Assig.
191.33	9.91	7.92	CH = O
162.84			C-4
160.95		7.92, 5.44	C-*p*
143.57		6.87	C-2
142.61		8.92, 5.44	C-4′
139.42		7.66, 7.48, 7.46	C-8a
131.91	7.66	7.48, 7.46	C-7
131.79	7.92	9.91, 7.92	C-*o*
130.00		9.91, 7.32	C-*i*
126.85	8.92	5.44	C-5′
123.93	7.46		C-5
122.60	7.25	7.48, 7.46	C-6
117.79	6.87		C-3
115.93	7.48		C-8
115.27	7.32	7.32	C-*m*
114.42		7.48, 7.46, 7.25, 6.87	C-4a
61.20	5.44		C-4a′

a 2H singlet at δ_H = 5.41 ppm, assigned as H-4a′; the attached carbon appears at δ_C = 61.07 ppm. H-4a′ gives HMBC correlation with a protonated carbon at δ_C = 127.01 ppm, assigned as C-5′; the attached proton appears at δ_H = 8.90 ppm. H-4a′ also gives HMBC correlation with one of the two non-protonated carbons at δ_C = 142.76 and 142.57 ppm, assigned as C-4′, and with the sp^2 nitrogen at δ_N = 358.10 ppm, assigned as *N*-3′.

The other sp^2 nitrogen, *N*-2′, is not observed. H-5′ gives HMBC correlation with a nitrogen at δ_N = 246.8 ppm, assigned as *N*-1′. *N*-1′ gives HMBC correlation with the 1H singlet at δ_H = 7.00 ppm, assigned as H-3; the attached carbon appears at δ_C = 117.27 ppm. Also, H-3 gives HMBC correlation with the other of the two carbons at δ_C = 142.76 and 142.57 ppm, assigned as C-4; and with the non-protonated one of the two carbons at δ_C = 115.60 and 115.50 ppm, presumably the smaller line at δ_C = 115.50 ppm, which is assigned as C-4a. H-3 also gives HMBC correlation with the sp^3 nitrogen at δ_N = 151.3 ppm, assigned as *N*-1. *N*-1 gives HMBC correlation with the methyl singlet at δ_H = 3.73 ppm, assigned as H-1a; the attached carbon appears at δ_C = 29.59 ppm. H-1a gives HMBC correlation with carbons at δ_C = 160.27 ppm, assigned as C-2, and δ_C = 140.12 ppm, assigned as C-8a; both of these are three-bond correlations. C-8a gives HMBC correlation with a 1H doublet at δ_H = 7.45 ppm, assigned as H-5, and a 1H “triplet” at δ_H = 7.79 ppm, assigned as H-7; both of these are also three-bond correlations. The attached carbons appear at δ_C = 124.41 (C-5) and 132.35 ppm (C-7). The remaining protons of the four-spin benzene system are a 1H doublet at δ_H = 7.74 ppm, assigned as H-8, and a 1H “triplet” at δ_H = 7.35 ppm, assigned as H-6; the attached carbons appear

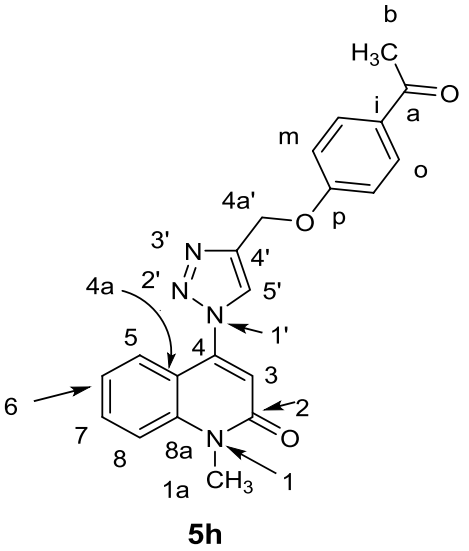


Fig. 3 Distinctive carbons of 4-[4-[(4-acetylphenoxy)methyl]-1*H*-1,2,3-triazol-1-yl]-1-methylquinolin-2(1*H*)-one (**5h**)

at $\delta_C = 115.60$ (C-8) and 122.75 ppm (C-6). H-6 and H-8 give HMBC correlation with C-4a; both of these are three-bond correlations.

The formation of products **3** and **5** via click reaction can be rationalized as shown in Scheme 3. First stage, Cu(II), was reduced by sodium ascorbate into Cu(I) which then replaced the alkyne-H proton to form Cu-salt (**A**). Secondly, nucleophilic addition of (**A**) to the aromatic azides **1a**–**1d** to form the intermediate (**B**) was accompanied by the loss of catalyzed Cu(I) that was followed by inter-nucleophilic cyclization to give adduct (**C**). Finally, reduction of adduct (**C**) with hydrogen proton leads to the formation of **3a**–**3d** and **5a**–**5h**. The mechanism was totally supported via literature [31].

Conclusion

The reactions of 4-azidoquinolin-2(1*H*)-ones with some terminal alkynes to obtain new quinolin-2-one-linked 1,2,3-triazoles are useful examples of Cu-catalyzed [3 + 2]cycloaddition of azides and alkynes (click reaction); all structures were established by spectroscopic analysis.

Experimental

Melting points were determined using open glass capillaries on a Gallenkamp melting point apparatus (Weiss–Gallenkamp, Loughborough, UK). The IR spectra were recorded from potassium bromide disks with an FT device, Faculty of Science Minia University. NMR spectra were measured in DMSO-d_6 on a Bruker AV-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, and 40.55 MHz for ^{15}N); chemical shifts are expressed in δ (ppm) versus internal tetramethylsilane (TMS) = 0 ppm for ^{1}H and ^{13}C, and external liquid ammonia = 0 ppm for ^{15}N. Coupling constants are stated in Hz. Correlations were established using ^{1}H-^{1}H COSY, and ^{1}H-^{13}C, and ^{1}H-^{15}N HSQC and HMBC experiments. Mass spectra were recorded on a Finnigan Fab 70 eV, Institute of Organic Chemistry, Karlsruhe University, Karlsruhe, Germany. TLC was performed on analytical Merck 9385 silica aluminum sheets (Kieselgel 60) with Pf_{254} indicator; TLCs were viewed at $\lambda_{max} = 254$ nm. Elemental analyses were carried out on Perkin device at the Microanalytical Institute of Organic Chemistry, Karlsruhe University, Karlsruhe, Germany.

4-Azidoquinoline-2(1*H*)-ones **1a**–**1d** were prepared according to the literature [32, 33]. Ethyl propiolate (**2**) was used as received (Aldrich), and 4-(prop-2-yn-1-yloxy)benzaldehyde (**4a**) and 1-[4-(prop-2-yn-1-yloxy)phenyl]ethanone (**4b**) were prepared according to the literature [34].

General procedure for the formation of compounds 3a–3d and 5a–5h

A mixture of terminal alkynes **2** or **4a**, **4b** (1.0 mmol) in 20 cm^3 dimethyl formamide (DMF), 5 cm^3 H_2O, sodium ascorbate (0.4 mmol), and $CuSO_4{\cdot}5H_2O$ (0.2 mmol) was stirred for 5 min at room temperature. Then, 4-azido compounds **1a**–**1d** (1.0 mmol) were added to the mixture. The reaction mixture was allowed to stir at 30–50 °C for 12 h, and another portion of sodium ascorbate (0.4 mmol) was added and the reaction was monitored with TLC. After completion, the mixture was concentrated, diluted with H_2O, and extracted with CH_2Cl_2 (3 × 15 cm^3). The combined organic layers were dried (Na_2SO_4), filtered, and concentrated. The products were recrystallized from absolute ethanol.

Ethyl 1-(2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazole-4-carboxylate (3a, $C_{14}H_{12}N_4O_3$) Colorless crystals; yield: 0.220 g (77%); m.p.: 252–254 °C; IR: $\bar{\nu} = 3137$ (NH), 3075 (Ar–H), 2966–2850 (Ali-H), 1717, 1677 (CO), 1610 (C=C) cm^{-1}; ^{1}H NMR: $\delta = 12.33$ (s, 1H, NH), 9.39 (s, 1H, H-5′), 7.66 (t, 1H, $J = 7.3$ Hz, H-7), 7.48 (d, 1H, $J = 8.2$ Hz, H-8), 7.38 (d, 1H, $J = 8.0$ Hz, H-5), 7.25 (t, 1H, $J = 7.6$ Hz, H-6), 6.95 (s, 1H, H-3), 4.39 (q, 2H, $J = 7.1$ Hz, H-4b′), 1.35 (t, 3H, $J = 7.1$ Hz, H-4c′) ppm; ^{13}C NMR: $\delta = 160.84$ (C-2), 195.91 (C-4a′), 143.16 (C-8a′), 139.30, 139.22 (C-4,4′), 132.00 (C-7), 131.11 (C-5′), 123.90 (C-5), 122.68 (C-6), 118.76 (C-3), 115.86 (C-8), 114.47 (C-4a), 60.88 (C-4b′), 14.16 (C-4c′) ppm; ^{15}N NMR: $\delta = 367.4$ (N-2′/3′), 359.7 (N-3′/N-2′), 250.0 (N-1′), 152.4 (N-1) ppm; MS (FAB): *m*/*z* (%) = 284 (M^+, 100).

Table 3 Spectroscopic data of compound **5h**

^{1}H NMR	^{1}H-^{1}H COSY	Assig.
8.90 (s, 1H)	7.00, 5.41	H-5′
7.98 (d, *J* = 8.6 Hz, 2H)	7.23	H-*o*
7.79 (“t”, *J* = 7.6 Hz, 1H)	7.74, 7.45, 7.35	H-7
7.74 (d, *J* = 8.4 Hz, 1H)	7.79	H-8
7.45 (d, *J* = 8.0 Hz, 1H)	7.79, 7.35	H-5
7.35 (“t”, *J* = 7.4 Hz, 1H)	7.79, 7.45	H-6
7.23 (d, *J* = 8.6 Hz, 2H)	7.98	H-*m*
7.00 (s, 1H)	8.90	H-3
5.41 (s, 2H)	8.90	H-4a′
3.73 (s, 3H)		H-1a
2.54 (s, 3H)		H-b

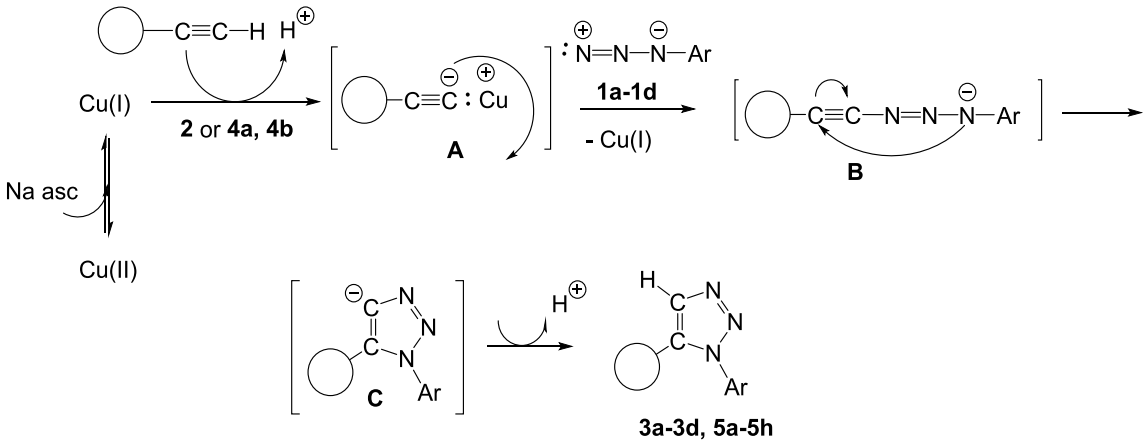
^{15}N NMR	HSQC	HMBC	Assig.
358.1		5.41	N-3′
246.8		8.90, 7.00	N-1′
148.0		7.00, 3.73	N-1

`^{13}C NMR	HSQC	HMBC	Assig.
196.30		7.98, 2.54	C-a
161.75		7.98, 7.23, 5.41	C-*p*
160.27		3.73	C-2
142.76, 142.57		8.90, 7.45, 7.00, 5.41	C-4,4′
140.12		7.79, 7.45, 3.73	C-8a
132.35	7.79	7.45	C-7
130.48	7.98	7.98	C-*o*
130.28		7.23, 2.54	C-*i*
127.01	8.90	5.41	C-5′
124.41	7.45	7.79	C-5
122.75	7.35	7.74	C-6
117.27	7.00		C-3
115.60	7.74		C-8
115.50		7.74, 7.35, 7.00	C-4a
114.61	7.23	7.23	C-*m*
61.07	5.41		C-4a′
29.59	3.73		C-1a
26.41	2.54		C-b

Ethyl 1-(6-methyl-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazole-4-carboxylate (3b, $C_{15}H_{14}N_4O_3$) Colorless crystals; yield: 0.240 (80%); m.p.: 251–253 °C; IR (KBr): $\bar{\nu}$ = 3127 (NH), 3077 (Ar–H), 2983-2865 (Ali-H), 1719, 1675 (CO), 1609 (C=C) cm^{-1}; ^{1}H NMR: δ = 12.26 (s, 1H, NH), 9.36 (s, 1H, H-5′), 7.49 (d, *J* = 8.2 Hz, 1H, H-7), 7.38 (d, *J* = 8.3 Hz, 1H, H-8), 7.14 (s, 1H, H-5), 6.90 (s, 1H, H-3), 4.39 (q, *J* = 7.0 Hz, 2H, H-4b′), 2.30 (s, 3H, H-6a), 1.35 (t, *J* = 7.1 Hz, 3H, H-4c′) ppm; ^{13}C NMR: δ = 160.69 (C-2), 159.94 (C-4a′), 143.00 (C-4), 139.22 (C-4′), 137.38 (C-8a), 133.31 (C-7), 131.91 (C-6), 131.09 (C-5′), 123.05 (C-5), 118.84 (C-3), 115.82 (C-8), 114.48 (C-4a), 60.86 (C-4b′), 20.44 (C-6a), 14.16 (C-4c′) ppm; ^{15}N NMR: δ = 249.6 (N-1′), 247.9, 246.9 (N-2′,3′), 152.4 (N-1) ppm.

Ethyl 1-(6-methoxy-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazole-4-carboxylate (3c, $C_{15}H_{14}N_4O_4$) Colorless crystals; yield: 0.230 g (73%); m.p.: 271–273 °C; IR (KBr): $\bar{\nu}$ = 3139 (NH), 3075 (Ar–H), 2918–2853 (Ali-H), 1719, 1676 (CO), 1628 (C=N) cm^{-1}; MS (FAB): *m*/*z* (%) = 314 (M^+, 100); ^{1}H NMR: δ = 12.23 (s, 1H, NH), 9.39 (s, 1H, H-5′), 7.43 (d, *J* = 9.0 Hz, 1H, H-8), 7.35 (dd, *J* = 9.0 Hz, 2.7 Hz, 1H, H-7), 6.93 (s, 1H, H-3), 6.86 (d, *J* = 2.6 Hz, 1H, H-5), 4.39 (q, *J* = 7.1 Hz, 2H, H-4b′), 3.71 (s, 3H, H-6a), 1.35 (t, *J* = 7.1 Hz, 3H, H-4c′)

Scheme 3



ppm; ^{13}C NMR: $\delta = 160.35$ (C-2), 159.93 (C-4a′), 154.56 (C-6), 142.63 (C-4), 139.27 (C-4′), 133.98 (C-8a), 131.05 (C-5′), 121.19 (C-7), 119.04 (C-3), 117.38 (C-8), 114.90 (C-4a), 105.50 (C-5), 60.87 (C-4b′), 55.48 (C-6a), 14.15 (C-4c′) ppm; ^{15}N NMR: $\delta = 367.0$, 360.2 (N-2′,3′), 250.3 (N-1′), 151.5 (N-1) ppm.

Ethyl 1-(1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazole-4-carboxylate (3d, $C_{15}H_{14}N_4O_3$) Colorless crystals; yield: 0.195 g (69%); m.p.: 269–271 °C; IR (KBr): $\bar{\nu} = 3081$ (Ar–H), 2984 (Ali-H), 1740, 1665 (CO), 1595 (C=C) cm^{-1}; ^{1}H NMR: $\delta = 9.41$ (s, 1H, H-5′), 7.65 ("t", $J = 7.2$ Hz, 1H, H-7), 7.49 (d, $J = 8.1$ Hz, 1H, H-8), 7.34 (d, $J = 8.0$ Hz, 1H, H-5), 7.28 ("t", $J = 7.4$ Hz, 1H, H-6), 6.95 (s, 1H, H-3), 4.37 (q, $J = 7.1$ Hz, 2H, H-4b′), 2.50 (s, 3H, H-1a), 1.36 (t, $J = 7.1$ Hz, 3H, H-4c′) ppm; ^{13}C NMR: $\delta = 159.10$ (C-2), 158.82 (C-4a′), 141.12 (C-8a), 139.49, 139.45 (C-4,4′), 132.23 (C-7), 126.94 (C-5′), 122.22, 122.12 (C-5,6), 118.70 (C-3), 115.32 (C-8), 114.06 (C-4a), 61.58 (C-4b′), 29.40 (C-1a), 13.63 (C-4c′) ppm.

4-[[[1-(2-Oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl]-oxy]methyl]benzaldehyde (5a, $C_{19}H_{14}N_4O_3$) Colorless crystals; yield 0.260 (71%); m.p.: 225–227 °C; IR (KBr): $\bar{\nu} = 3124$ (NH), 2859 (Ali-H), 1691, 1669 (CO), 1598 (C=C) cm^{-1}; ^{1}H NMR: $\delta = 12.29$ (s, 1H, NH), 9.91 (s, 1H, CHO), 8.92 (s, 1H, H-5′), 7.92 (d, 2H, $J = 8.5$ Hz, H-*o*), 7.66 (t, 1H, $J = 7.6$ Hz, H-7), 7.48 (d, 1H, $J = 8.9$ Hz, H-8), 7.46 (d, 1H, $J = 9.0$ Hz, H-5), 7.32 (d, 2H, $J = 8.5$ Hz, H-*m*), 7.25 (t, 1H, $J = 7.6$ Hz, H-6), 6.87 (s, 1H, H-3), 5.44 (s, 2H, H-4a′) ppm; ^{13}C NMR: $\delta = 191.33$ (CHO), 162.84 (C-4), 160.95 (C-*p*), 143.57 (C-2), 142.61 (C-4′), 139.42 (C-8a), 131.91 (C-7), 131.79 (2C-*o*), 130.00 (C-*i*), 126.85 (C-5′), 123.93 (C-5), 122.60 (C-6), 117.79 (C-3), 115.27 (2C-*m*), 114.42 (C-4a), 61.20 (C-4a′) ppm; ^{15}N NMR: $\delta = 247.7$ (N-1′) ppm.

4-[[[1-(6-Methyl-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl]oxy]methyl]benzaldehyde (5b, $C_{20}H_{16}N_4O_3$) Colorless crystals; yield: 0.3 g (83%); m.p.: 220–222 °C; IR (KBr): $\bar{\nu} = 3135$ (NH), 2950 (Ali-H), 1690, 1669 (CO), 1595 (C=C) cm^{-1}; ^{1}H NMR: $\delta = 12.22$ (s, 1H, NH), 9.91 (s, 1H, CH=O), 8.90 (s, 1H, H-5′), 7.92 (**AA′**XX′, $J_{AA'} = 3.6$ Hz, $J_{AX} = 10.0$ Hz, $J_{AX'} = 1.2$ Hz, 2H, H-*m*), 7.49 (dd, $J = 8.5$ Hz, 1.6 Hz, 1H, H-7), 7.39 (d, $J = 8.4$ Hz, 1H, H-8), 7.32 (AA′**XX′**, $J_{AX} = 10.0$ Hz, $J_{AX'} = 1.2$ Hz, $J_{XX'} = 0.9$ Hz, 2H, H-*o*), 7.21 (bs, 1H, H-5), 6.82 (s, 1H, H-3), 5.44 (s, 2H, H-4a′), 2.30 (s, 3H, H-6a) ppm; ^{13}C NMR: $\delta = 191.33$ (CH=O), 162.81 (C-p), 160.80 (C-2), 143.36, 142.57 (C-4,4′), 137.51 (C-8a), 133.21 (C-7), 131.79 (C-*m*), 131.76 (C-6), 130.00 (C-*i*), 126.85 (C-5′), 123.09 (C-5), 117.82 (C-3), 115.91 (C-8), 115.28 (C-*o*), 114.36 (C-4a), 61.20 (C-4a′), 20.55 (C-6a) ppm; ^{15}N NMR: $\delta = 364.6$ (N-2′), 358.1 (N-3′), 247.7 (N-1′), 152.1 (N-1) ppm.

4-[[[1-(6-Methoxy-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl]oxy]methyl]benzaldehyde (5c, $C_{20}H_{16}N_4O_4$) Colorless crystals; yield: 0.220 g (58%); m.p.: 245–247 °C; IR (KBr): $\bar{\nu} = 3144$ (NH), 3067 (Ar–H), 2994, 2851 (Ali-H), 1674(CO), 1625 (C=N), 1592 (C=C) cm^{-1}; ^{1}H NMR: $\delta = 12.20$ (s, 1H, NH), 9.90 (s, 1H, CH=O), 8.94 (s, 1H, H-5′), 7.91 (d, $J = 8.6$ Hz, 2H, H-*o*), 7.44 (d, $J = 9.0$ Hz, 1H, H-8), 7.34 (dd, $J = 9.0$ Hz, 2.3 Hz, 1H, H-7), 7.31 (d, $J = 8.6$ Hz, 2H, H-*m*), 6.90 (d, $J = 2.0$ Hz, 1H, H-5), 6.86 (s, 1H, H-3), 5.45 (s, 2H, H-4a′), 3.68 (s, 3H, H-6a) ppm; ^{13}C NMR: $\delta = 191.33$ (CH=O), 162.79 (C-*p*), 160.47 (C-2), 154.50 (C-6), 143.03, 142.68 (C-4,4′), 134.06 (C-8a), 131.79 (C-*o*), 129.99 (C-*i*), 126.75 (C-5′), 121.01 (C-7), 118.10 (C-3), 117.44 (C-8), 115.27 (C-*m*), 114.83 (C-4a), 105.53 (C-5), 61.19 (C-4a′), 55.38 (C-6a) ppm; ^{15}N NMR: $\delta = 248.0$ (N-1′), 151.3 (N-1) ppm.

4-[[[1-(1-Methyl-2-oxo-1,2-dihydroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl]oxy]methyl]benzaldehyde (5d, $C_{15}H_{14}N_4O_3$) Colorless crystals; yield: 0.2 g (55%), m.p.: 268–270 °C; IR (KBr): $\bar{\nu}$ = 2990, 2871 (Ali-H), 1687, 1665 (CO), 1585 (C=C) cm^{-1}; MS (FAB): *m/z* (%) = 360 (M^+, 100); ^{1}H NMR: δ = 9.91 (s, 1H, CH=O), 8.91 (s, 1H, H-5′), 7.92 (**AA′**XX′, J_{AX} = 8.8 Hz, $J_{AA'}$ = 3.7 Hz, $J_{XX'}$ = 0.9 Hz, 2H, H-*o*), 7.80 (d"t", J_d = 1.4 Hz, $J_{"t"}$ = 7.8 Hz, 1H, H-7), 7.75 (d, *J* = 8.0 Hz, 1H, H-8), 7.45 (dd, *J* = 8.1 Hz, 1.2 Hz, 1H, H-5), 7.35 (d"t", J_d = 1.1 Hz, $J_{"t"}$ = 7.0 Hz, 1H, H-6), 7.32 (d, *J* = 8.7 Hz, 2H, H-*m*), 7.01 (s, 1H, H-3), 5.44 (s, 2H, H-4a′), 3.73 (s, 3H, H-1a) ppm; ^{13}C NMR: δ = 191.34 (CH=O), 162.84 (C-*p*), 160.27 (C-2), 142.62, 142.57 (C-4′,8a), 140.13 (C-4), 132.36 (C-7), 131.80 (C-*o*), 130.01 (C-*i*), 127.08 (C-5′), 124.40 (C-5), 122.76 (C-6), 117.30 (C-3), 115.61, 115.51 (C-4a,8), 115.27 (C-*m*), 61.20 (C-4a′), 29.59 (C-1a) ppm; ^{15}N NMR: δ = 146.9 (N-1) ppm.

4-[4-[(4-Acetylphenoxy)methyl]-1*H*-1,2,3-triazol-1-yl]-quinolin-2(1*H*)-one (5e, $C_{20}H_{16}N_4O_3$) Colorless crystals; yield: 0.280 g (78%); m.p.: 252–254 °C; IR (KBr): $\bar{\nu}$ = 3166 (NH), 2944 (Ali-H), 1691, 1669 (CO), 1595 (C=C) cm^{-1}; ^{1}H NMR: δ = 12.29 (s, 1H, NH), 8.91 (s, 1H, H-5′), 7.97 (d, *J* = 8.7 Hz, 2H, H-*o*), 7.66 ("t", *J* = 7.6 Hz, 1H, H-7), 7.48 (d, *J* = 8.6 Hz, 1H, H-8), 7.46 (d, *J* = 8.6 Hz, 1H, H-5), 7.25 ("t", *J* = 7.5 Hz, 1H, H-6), 7.22 (d, *J* = 8.6 Hz, 2H, H-*m*), 6.86 (s, 1H, H-3), 5.41 (s, 2H, H-4a′), 2.54 (s, 3H, H-b) ppm; ^{13}C NMR: δ = 196.29 (C-a), 161.74, 160.95 (C-*p*,2), 143.57, 142.75 (C-4′,4), 139.42 (C-8a), 131.90 (C-7), 130.47 (C-*o*), 130.28 (C-*i*), 126.78 (C-5′), 123.94 (C-5), 122.59 (C-6), 117.76 (C-3), 115.93 (C-8), 114.61 (C-*m*), 114.42 (C-4a), 61.07 (C-4a′), 26.40 (C-b) ppm; ^{15}N NMR: δ = 364.4 (N-2′), 358.2 (N-3′), 247.3 (N-1′), 152.5 (N-1) ppm.

4-[4-[(4-Acetylphenoxy)methyl]-1*H*-1,2,3-triazol-1-yl]-6-methylquinolin-2(1*H*)-one (5f, $C_{21}H_{18}N_4O_3$) Colorless crystals; yield: 0.360 g (89%); m.p.: 263-265 °C; IR (KBr): $\bar{\nu}$ = 3165 (NH), 2950 (Ali-H), 1687, 1670 (CO), 1598 (C=C) cm^{-1}; MS (FAB): *m/z* (%) = 374 (M^+, 100); ^{1}H NMR: δ = 12.22 (bs, 1H, NH), 8.89 (s, 1H, H-5′), 7.98 (d, *J* = 8.6 Hz, 2H, H–*o*), 7.49 (bd, *J* = 8.2 Hz, 1H, H-7), 7.39 (d, *J* = 8.5 Hz, 1H, H-8), 7.22 (d, *J* = 8.5 Hz, 2H, H-*m*), 7.21 (bs, 1H, H-5), 6.81 (s, 1H, H-3), 5.41 (s, 2H, H-4a′), 2.54 (s, 3H, H-b), 2.30 (s, 3H, H-6a) ppm; ^{13}C NMR: δ = 196.28 (C-a), 161.71 (C-*p*), 160.80 (C-2), 143.36, 142.71 (C-4′,4), 137.51 (C-8a), 133.20 (C-7), 131.75 (C-6), 130.47 (C-*o*), 130.27 (C-*i*), 126.77 (C-5′), 123.09 (C-5), 117.79 (C-3), 115.91 (C-8), 114.61 (C-*m*), 114.35 (C-4a), 61.06 (C-4a′), 26.40 (C-b), 20.53 (C-6a) ppm; ^{15}N NMR: δ = 364.1 (N-2′), 357.1 (N-3′), 247.3 (N-1′), 151.8 (N-1) ppm.

4-[4-[(4-Acetylphenoxy)methyl]-1*H*-1,2,3-triazol-1-yl]-6-methoxyquinolin-2(1*H*)-one (5g, $C_{21}H_{18}N_4O_4$) Colorless crystals; yield 0.270 g (69%); m.p.: 253-255 °C; ^{1}H NMR: δ = 12.20 (bs, 1H, NH), 8.93 (s, 1H, H-5′), 7.97 (d, *J* = 8.8 Hz, 2H, H–*o*), 7.43 (d, *J* = 9.0 Hz, 1H, H-8), 7.34 (dd, *J* = 9.0 Hz, 2.5 Hz, 1H, H-7), 7.22 (d, *J* = 8.8 Hz, 2H, H-*m*), 6.90 (d, *J* = 2.5 Hz, 1H, H-5), 6.85 (s, 1H, H-3), 5.41 (s, 2H, H-4a′), 3.68 (s, 3H, H-6a), 2.54 (s, 3H, H-b) ppm; ^{13}C NMR: δ = 196.28 (C-a), 161.69 (C-*p*), 160.47 (C-2), 154.50 (C-6), 143.03, 142.83 (C-4′,4), 134.07 (C-8a), 130.46 (C-*o*), 130.27 (C-*i*), 126.67 (C-5′), 121.00 (C-5′), 118.06 (C-7), 117.44 (C-3), 114.83 (C-8), 114.61 (C-*m*), 105.53 (C-5), 61.06 (C-4a′), 55.37 (C-6a), 26.39 (C-b) ppm; ^{15}N NMR: δ = 364.3 (N-2′), 358.1 (N-3′), 247.9 (N-1′), 151.3 (N-1) ppm.

4-[4-[(4-Acetylphenoxy)methyl]-1*H*-1,2,3-triazol-1-yl]-1-methylquinolin-2(1*H*)-one (5h, $C_{21}H_{18}N_4O_3$) Colorless crystals; yield: 0.250 g (67%); m.p.: 218-220 °C; IR (KBr): $\bar{\nu}$ = 2895 (Ali-H), 1679, 1667 (CO), 1598 (C=C) cm^{-1}; ^{1}H NMR: δ = 8.90 (s, 1H, H-5′), 7.98 (d, 2H, *J* = 8.6 Hz, H-*o*), 7.79 (t, 1H, *J* = 7.6 Hz, H-7), 7.74 (d, 1H, *J* = 8.4 Hz, H-8), 7.45 (d, 1H, *J* = 8.0 Hz, H-5), 7.35 (t, 1H, *J* = 7.4 Hz, H-6), 7.23 (d, 2H, *J* = 8.6 Hz, H-*m*), 7.00 (s, 1H, H-3), 5.41 (s, 2H, -4a′), 3.73 (s, 3H, H-1a), 2.54 (s, 3H, H-b) ppm; ^{13}C NMR: δ = 196.30 (C-a), 161.75 (C-*p*), 160.27 (C-2), 142.76, 142.57 (C-4,4′), 140.12 (C-8a), 132.35 (C-7), 130.48 (2C-*o*), 130.28 (C-*i*), 127.01 (C-5′), 124.41 (C-5), 122.75 (C-6), 117.27 (C-3), 115.60 (C-8), 115.50 (C-4a), 114.61 (2C-*m*), 61.07 (C-4a′), 29.59 (C-1a), 26.41 (C-b) ppm; ^{15}N NMR: δ = 358.1 (N-3′), 246.8 (N-1′), 148.0 (N-1) ppm; MS (FAB): *m/z* (%) = 374 (M^+, 100).

Acknowledgements The NMR spectrometer at Florida Institute of Technology was purchased with assistance from the US National Science Foundation (CHE 03-42251).

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