Synthesis of New 1,2,3-Triazolo[1,5-a]quinazolinones

Nazariy T. Pokhodylo* and Vasyl S. Matiychuk

Department of Organic Chemistry, Ivan Franko National University of Lviv, Lviv 79005, Ukraine
*E-mail: pokhodylo@gmail.com

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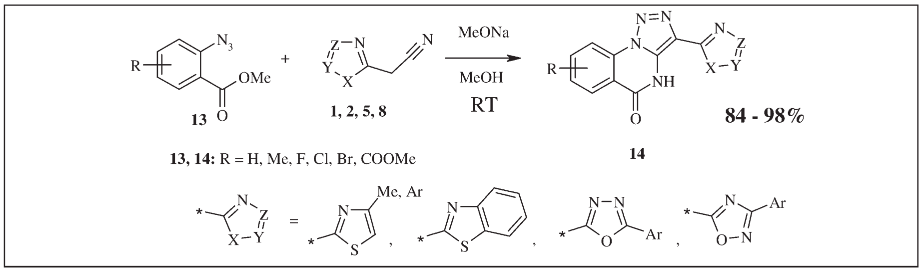

Synthesis of novel 3-substituted-1,2,3-triazolo[1,5-a]quinazolinones in high yields was performed *via* anionic hetero-domino reaction of appropriate substituted 2-azidobenzoates prepared from isatines and acetonitriles activated by 1,3-thiazole, 1,3-benzothiazole, 1,3,4-oxadiazole, and 1,2,4-oxadiazole rings. It was shown that acetonitriles exhibited high reactivity and were convenient methylenic compounds for such reactions providing rapid structural variation.

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INTRODUCTION

1,2,3-Triazolo[1,5-a]quinazolines are an important class of heterocycles, which have been the subject of great interest because of their biological activities. For instance, in the works of Jones and coworkers [1] the synthesis and evaluation of the biological affinity of a large number of C-5 substituted 1,2,3-triazolo[1,5-a]quinazolines are reported. It was shown that compounds of the current class were new ligands for $GABA_A$ receptors and potentially pharmaceutically acceptable for treatment of Alzheimer's disease. Moreover, Biagi et al [2]. found the activity of some 3-ethoxycarbonyl- or 3-phenyl-substituted 1,2,3-triazolo[1,5-a]quinazolines towards benzodiazepine, A_1 and A_{2A} adenosine receptors. The promising biological activity and the unique structure of these families make them attractive synthetic targets. Furthermore, in the article [1b,c] it was underlined that 1,2,3-triazolo[1,5-a]quinazolines were first synthesized in 1966 [3], however, this class of compounds has subsequently received little attention [1,4]. Their syntheses, using readily available raw materials with short and facile routes, continue to be challenging endeavors.

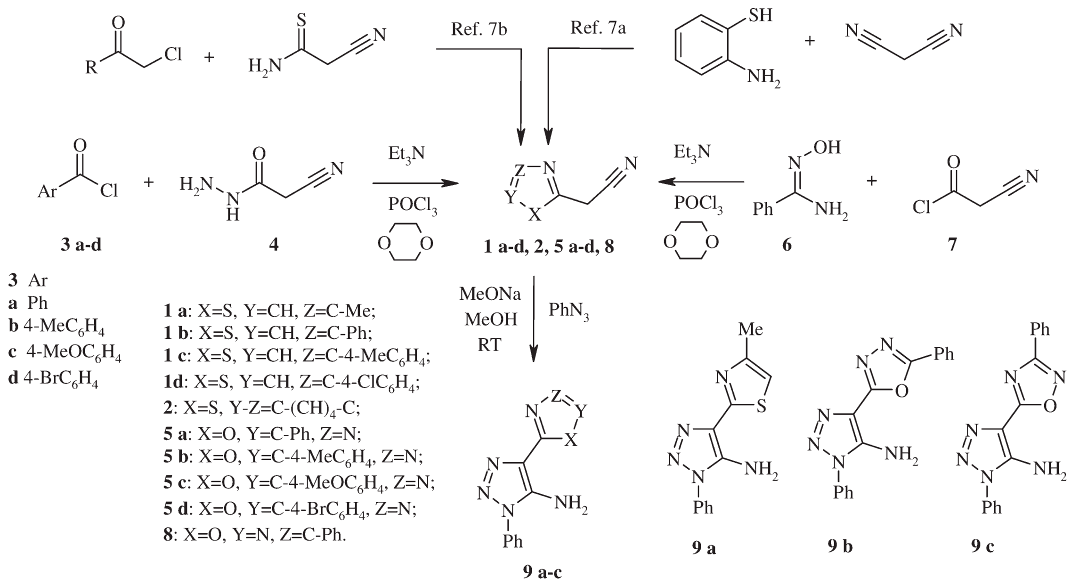
It is noteworthy that more attention was drawn to synthetic strategies, which have been developed to allow easy variation of the C-5 position of triazoloquinazolinone [1]. On the contrary, diversification of other positions has been discussed insufficiently. 2-Azidobenzoic acid and cyanoacetic acid derivatives were mostly used for construction of triazoloquinazolinone framework [4]. At the same time, the reaction of azides with acetonitriles activated by (het)aryl substituents is a perspective synthetic approach. The exclusive examples of the reaction of arylazides with (5-methylisoxazol-3-yl)acetonitrile [1] and phenylacetonitrile [5] have been described. In addition, introduction of new heterocyclic fragments to the C-3 position may extend the spectrum of biological activity of such compounds.

Recently, we have reported [6] the synthesis of 1*H*-1,2,3-triazole derivatives by cyclization of arylazides with (1,3-benzothiazol-2-yl)/(4-aryl-1,3-thiazol-2-yl)acetonitriles. It was shown that (1,3-benzothiazol-2-yl)acetonitrile undergoes an anionic domino reaction with methyl 2-azidobenzoate or 2-azidobenzonitrile to give [1,2,3]triazolo[1,5-a]quinazoline derivatives [6a]. Furthermore, it was found that acetonitrile possesses high reactivity, as an active methylenic compound for such reactions, for construction of [1,2,3]triazolo[1,5-a]pyrimidines system [6b] with heterocyclic substituents.

RESULTS AND DISCUSSION

In the current work we present the results of a study of hetarylacetonitriles in the reaction with substituted methyl 2-azidobenzoates. Starting hetarylacetonitriles were obtained by several synthetic procedures. First the required precursors **1a–d**, **2** were synthesized in moderate to good yields according to the literature procedure [7]. Then we developed a synthetic protocol for preparation of acetonitriles activated by 1,3,4-oxadiazole and 1,2,4-oxadiazole. Compounds containing the oxadiazole

Scheme 1



ring have been widely studied recently since they exhibit biological activity [8] and possess some attractive photophysical properties [9]. Initial (5-aryl-1,3,4-oxadiazol-2-yl)acetonitriles **5a–d** were synthesized by one-pot cyclization of diacylhydrazines prepared *in situ* by acylation of cyanoacetic acid hydrazide **4** with the corresponding substituted benzoyl chlorides **3a–d**, *via* modification of known procedures [10]. (5-Phenyl-1,2,4-oxadiazol-2-yl)acetonitrile **8** was prepared by heating phenylamide oxime **6** with cyanoacetyl chloride **7** in anhydrous DMF in the presence of pyridine (Scheme 1).

The obtained hetarylacetonitriles **1a, 5a, 8** were examined in the reaction with phenylazide in sodium methoxide solution at room temperature. These conditions allowed to avoid possible Dimroth rearrangement [11] occurring when 5-aminotriazoles were heated in a strong base medium. As a result, new triazole derivatives **9a–c** were isolated in high yields and no by-products were formed. The corresponding 1,2,3-triazoles **9a–c** precipitated in good yields from the reaction medium.

In an effort to diversify the azido component, the method of 2-azidobenzoate synthesis from isatines, prepared from the corresponding anilines **10** by the Sandmeyer method [12], was used. By the reaction of anilines **10a,b** with chloral hydrate and hydroxylamine hydrochloride in aqueous sodium sulfate, isonitrosoacetanilides were formed. The obtained isonitrosoacetanilides were treated with concentrated sulfuric acid and yielded isatines **11b,c** [12]. Compounds **11d,e** were prepared from isatine **11a** by the reaction with brome [13] or chlorine generated *in situ* from HCl and hydrogen peroxide [14] correspondingly. Isatines **11a–e** were oxidated with alkaline hydrogen peroxide to form substituted 2-aminobenzoic acids [15], which were converted into esters **12a–e** by the following reaction with methanol in the presence of sulphuric acid [16]. It is of note that compound **12d** can be prepared from commercially available methyl anthranilate **12a** *via* chlorization by the use of calcium hypochlorite [17]. Finally, compound **12f** was prepared by subsequent nitration and reduction of dimethyl benzene-1,4-dicarboxylate. Anthranilates **12a–f** were readily converted into 2-azidobenzoic acid esters **13a–f** by diazotisation and displacement with sodium azide (Scheme 2).

To obtain [1,2,3]triazolo[1,5-*a*]quinazolines, substituted methyl 2-azidobenzoates were allowed to react with acetonitriles **1, 2, 5, 8**. The reaction was carried out using 1 equiv. of sodium methoxylate at room temperature. It was found that the reaction exhibited an appreciable exothermal effect and was completed within 1–2 min. In general, the reaction product was formed immediately after mixing the reagents and precipitated from the reaction medium. The polycyclic compounds **14a–o** were isolated in excellent yields (84–98%) (Table 1). It is noteworthy that esters **13** used in such a reaction are more convenient instead of 2-azidobenzoic acids. In the reaction of 2-azidobenzoic acids, yields were lower and the reaction required more time. Moreover, in some cases the reaction mixture must be heated. For instance, in the work [1a] triazoloquinazolinone framework was prepared by treatment of an equimolar mixture of 2-azidobenzoic acid with isoxazole acetonitrile in the presence of sodium ethoxide at 80°C for 5 h. Bertelli et al. [2b] reported that 2-carboxy-4/5-chloro-phenylazide reacted with ethyl cyanoacetate in absolute ethanol in the presence of two equivalents of sodium ethoxide at

Scheme 2

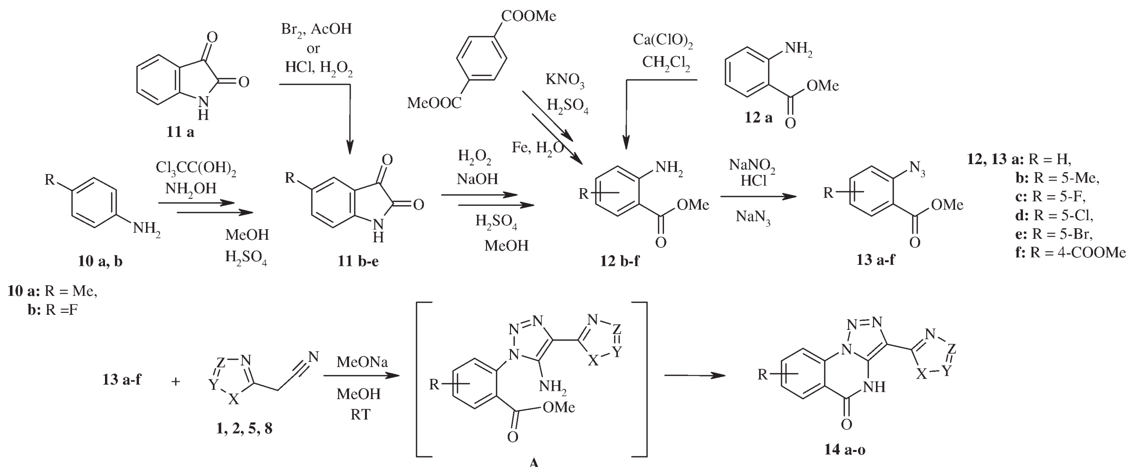


Table 1

Physical and analytical data of compounds **14a–o**.

Compound	*R*	C-3 substituent	Mp (°C)	Yield, %[a]	IR, ν_{max} (cm^{-1})[b]	MS (CI): [M+H$^+$]	Molecular formula	Analysis % Calcd./Found C	H	N
14a	H	4-methyl-1,3-thiazol-2-yl	>300	90	1679	284	$C_{13}H_9N_5OS$	55.11 55.28	3.20 3.02	24.72 24.93
14b	7-Br	–	>300	92	1683	362, 364	$C_{13}H_8BrN_5OS$	43.11 43.28	2.23 2.01	19.34 19.17
14c	H	4-phenyl-1,3-thiazol-2-yl	>300	96	1677	346	$C_{18}H_{11}N_5OS$	62.60 62.44	3.21 3.34	20.28 20.06
14d	–	4-(4-methylphenyl)-1,3-thiazol-2-yl	>300	95	1680	360	$C_{19}H_{13}N_5OS$	63.49 63.36	3.65 3.37	19.49 19.66
14e	–	4-(4-chlorophenyl)-1,3-thiazol-2-yl	>300	98	1671	380	$C_{18}H_{10}ClN_5OS$	56.92 57.08	2.65 2.41	18.44 18.38
14f	7-Me	1,3-benzothiazol-2-yl	>300	98	1674	334	$C_{17}H_{11}N_5OS$	61.25 61.15	3.33 3.19	21.01 21.77
14g	7-F	–	>300	94	1690	338	$C_{16}H_8FN_5OS$	56.97 56.84	2.39 2.30	20.76 20.52
14h	7-Cl	–	>300	97	1678	354	$C_{16}H_8ClN_5OS$	54.32 54.26	2.28 2.46	19.80 19.99
14i	8-CO$_2$Me	–	>300	94	1724, 1680, 1276	378	$C_{18}H_{11}N_5O_3S$	57.29 57.19	2.94 2.78	18.56 18.44
14j	H	5-phenyl-1,3,4-oxadiazol-2-yl	>300	87	1688	330	$C_{17}H_{10}N_6O_2$	61.82 61.73	3.05 2.92	25.44 25.35
14k	–	5-(4-methylphenyl)-1,3,4-oxadiazol-2-yl	>300	84	1672	345	$C_{18}H_{12}N_6O_2$	62.79 62.87	3.51 3.39	24.41 24.28
14l	–	5-(4-methoxyphenyl)-1,3,4-oxadiazol-2-yl	>300	93	1684	361	$C_{18}H_{12}N_6O_3$	60.00 60.15	3.36 3.15	23.32 23.41
14m	–	5-(4-bromophenyl)-1,3,4-oxadiazol-2-yl	>300	93	1677	409, 411	$C_{17}H_9BrN_6O_2$	49.90 49.77	2.22 2.13	20.54 20.31
14n		3-phenyl-1,2,4-oxadiazol-5-yl	>300	90	1680	331	$C_{17}H_{10}N_6O_2$	61.82 61.97	3.05 2.93	25.44 25.36
14o	7-Cl	–	>300	97	1686	365	$C_{17}H_9ClN_6O_2$	55.98 55.87	2.49 2.40	23.04 23.19

[a] Isolated yields.

[b] All compounds 14 showed a similar stretching peaks of associated NH (OH) groups with ν_{max} approximately 3400 cm^{-1}.

Table 2
^{1}H NMR spectra of products **14a–o** (J, Hz) 500 MHz.

		[1,2,3]triazolo[1,5-*a*]quinazoline protons				
Compound	Me	H-6	H-9	H-7	H-8	(Het)Aryl fragment
14a	2.42	8.18 (t, J 9.2)		7.78 (t, J 7.1)	7.52 (t, J 7.1)	7.08 (s, 1H)
14b	2.41	8.24 (s)	8.15 (d, J 7.0)	–	7.94 (d, J 7.0)	7.09 (s, 1H)
14c[a]		8.22 (t, J 7.6)		7.75 (t, J 7.6)	7.49 (t, J 7.6)	7.29 (t, J 7.4, 1H), 7.42 (t, J 7.6, 2H), 7.70 (s, 1H), 8.04 (d, J 7.2, 2H)
14d	2.39	8.20–8.25 (m)		7.74 (t, J 7.4)	7.51 (t, J 7.6)	7.22 (d, J 8.0, 2H, H-3,5), 7.70 (s, 1H, H_{Tz}), 7.86 (d, J 8.0, 2H, H-2,6)
14e		8.24 (t, J 8.0)		7.77 (t, J 7.6)	7.50 (t, J 7.6)	7.43 (d, J 8.8, 2H, H-3,5), 7.86 (s, 1H, H_{Tz}), 8.02 (d, J 8.8, 2H, H-2,6)
14f	2.42	7.89 (d, J 2.0)	7.87 (d, J 8.4)	–	7.62 (dd, J 8.4, 2.0),	7.32 (t, J 7.0, 1H, H-6), 7.44 (t, J 7.4, 1H, H-5), 7.99 (d, J 7.8, 1H, H-7), 8.05 (d, J 7.4, 1H, H-8)
14g		8.29 (dd, J 8.4, 3.9)	7.85 (dd, J 8.9, 1.7),	–	7.70 (dt, J 8.6, 2.5)	7.34 (t, J 7.0, 1H, H-6), 7.47 (t, J 7.4, 1H, H-5), 7.98 (d, J 8.0, 1H, H-7), 8.06 (d, J 7.4, 1H, H-8)
14h		8.12 (s)	8.25 (d, J 8.4)	–	7.85 (d, J 8.7)	7.31-7.38 (m, 1H, H-6), 7.47 (t, J 7.1, 1H, H-5), 7.97 (d, J 7.2, 1H, H-7), 8.07 (d, J 7.0, 1H, H-8)
14i	3.97	8.30 (d, J 7.6)	8.73 (s)	8.08 (d, J 7.6)	–	7.35 (t, J 7.1, 1H, H-6), 7.48 (t, J 6.7, 1H, H-5), 7.98 (d, J 7.4, 1H, H-7), 8.08 (d, J 7.3, 1H, H-8)
14ja		8.35 (d, J 8.0)	8.26 (d, J 7.9)	7.94 (t, J 7.1)	7.65 (t, J 7.6)	7.57-7.62 (m, 3H), 8.19 (m, 2H)
14k	2.47	8.34 (d, J 8.00)	8.28 (d, J 7.7)	7.94 (t, J 7.9)	7.67 (t, J 7.7)	7.40 (d, J 7.9, 2H), 8.04 (d, J 7.9, 2H)
14l	3.88	8.37 (d, J 8.0)	8.25 (d, J 8.0)	7.97 (t, J 7.9)	7.70 (t, J 7.9)	7.12 (d, J 8.2, 2H), 8.09 (d, J 8.2, 2H)
14m		8.22 (d, J 8.1)	8.09 (d, J 8.0)	7.80 (t, J 7.6)	7.56 (t, J 7.6)	7.86 (d, J 7.9, 2H), 8.03 (d, J 8.1, 2H)
14n		8.34 (d, J 8.0)	8.27 (d, J 7.9)	7.97 (t, J 7.9)	7.64 (t, J 8.0)	7.53-7.59 (m, 3H), 8.17 (m, 2H)
14o		8.14 (s)	8.27 (d, J 8.7)	–	7.83 (d, J 8.7)	7.55-7.62 (m, 3H), 8.19 (m, 2H)

[a] Recorded at 400 MHz.

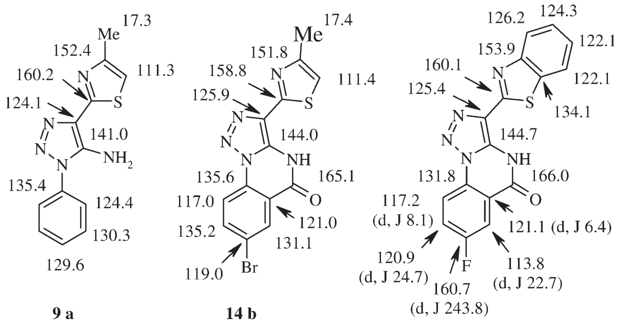
room temperature, affording 1-(4- or 5-chloro-2-carboxy-phenyl)-4-ethoxycarbonyl-5-amino-1H-1,2,3-triazole intermediates, and the corresponding triazoloquinazolinones were formed after acidification of the alkaline solution. The reaction of such azides with phenylacetonitrile required refluxing of the reaction mixture but it proceeded in the same manner [2b]. Obviously, replacement of the carboxylic group into the carboxylate one increased the rate of the reaction of the amino group in the intermediate triazole **A**, which was formed at the first stage of the reaction. The carboxylate function provided the formation of the pyrimidine ring without any additional procedures.

The structures of all new compounds were confirmed by analytical and spectroscopic data (Tables 1 and 2, Fig. 1). In the ^{1}H NMR spectra of compounds **14a–o** there were characteristic shift values for the H6, H7, H8, and H9 protons typical for the 1,2,3-triazolo[1,5-a]quinazoline system (Table 2). Furthermore, aryloxadiazolyl fragments were easily identified *via* low-field shifted aromatic protons *ortho* to the oxadiazole rings, usually found at 8.03–8.19 ppm.

In conclusion, in the current work we described efficient and economic routes to 1,2,3-triazolo[1,5-a]quinazolines from cheap abundant starting materials and with a possibility of substituent variation in triazoloquinazoline framework. Moreover, new active methylenic compounds were used for anionic domino reactions leading to formation of triazoloquinazolines.

EXPERIMENTAL

All melting points were determined in capillary tubes in a Thiele apparatus and are uncorrected. ^{1}H NMR spectra were recorded on a Varian Mercury 400 instrument (400 MHz for ^{1}H) and Bruker 500 (500 MHz for ^{1}H, 125 MHz for ^{13}C) with



9 a **14 b** **14 g**

Figure 1. ^{13}C NMR chemical shifts of compounds **9a**, **14b**, **14g**.

TMS or deuterated solvent as an internal reference. Mass spectra were run using Agilent 1100 series LC/MSD with an API-ES/APCI ionization mode. IR spectra were recorded on a Specord 80M spectrophotometer in KBr pellets. The evolution of the reactions and purity of the synthesized compounds were monitored chromatographically on Silufol UV-254 plates. ^{13}C NMR spectra of product **9b**,**c** and **14a**, **c–f**, **h–o** are not reported due to their very low solubility.

(5-Aryl-1,3,4-oxadiazol-2-yl)acetonitriles 5a–d (general procedure). The solution of 21 mmol of substituted benzoyl chloride **3** in 10 mL of anhydrous dioxane was added under continuous stirring to the solution of 2.1 g (21 mmol) of 2-cyanoacetohydrazide and 2.9 mL (21 mmol) of triethylamine in 30 mL of anhydrous dioxane. The mixture was stirred for 0.5 h at room temperature and 10 mL of $POCl_3$ was added. The mixture was heated for 3 h at 80°C, cooled to room temperature and poured onto ice. The precipitate was filtered off and dried in air. The crude product was purified by flash chromatography followed by recrystallization from ethanol.

(3-Phenyl-1,2,4-oxadiazol-5-yl)acetonitrile 8. Cyanoacetyl chloride **6** (0.52 g, 5 mmol) was added to the solution of 5 mmol of *N*'-hydroxybenzenecarboximidamide **7** in 2 mL of pyridine. The mixture was kept for 0.5 h and 5 mL of DMF was added. The mixture was heated for 3 h at 80°C, cooled to room temperature and mixed with 30 mL of water. The precipitate was filtered off, washed with water on a filter, recrystallized from alcohol and dried in air.

Azides 13a–f preparation. The solution of sodium nitrite (7.1 g, 0.1 mol) in water (30 mL) was added dropwise to a stirred solution of substituted anthranilic esters (0.1 mol) in 40 mL of concentrated HCl and 20 mL of water keeping the temperature below 5°C. After that the mixture was stirred for 10 min and rapidly filtered. To the obtained solution NaN_3 (6.5 g, 0.1 mol) in water (125 mL) was added dropwise. The mixture was stirred for 15 min at 0°C and then for 30 min at room temperature. Azides **13a,b** were extracted by diethyl ether (3 × 10 mL). Ether was evaporated *in vacuo*. Azides **13c–f** were filtered and washed with water twice. Azides were used without subsequent cleaning: **methyl 2-azidobenzoate 13a**, yield 11.5 g, 65%; dark red oil; MS: (CI) *m*/*z* (%) = 178 (100%) [M+H$^+$]; **methyl 2-azido-5-methylbenzoate 13b**, yield 13.7 g, 72%; dark red oil; MS: (CI) *m*/*z* (%)= 192 (100%) [M+H$^+$]; **methyl 2-azido-5-fluorobenzoate 13c**, yield 13.8 g, 71%; pink solid, mp: 28–29°C; MS: (CI) *m*/*z* (%)= 196 (100%) [M+H$^+$]; **methyl 2-azido-5-chlorobenzoate 13d**, yield 16.7 g, 79%; white pink solid, mp: 50–52°C; MS: (CI) *m*/*z* (%) = 212(100%) [M+H$^+$]; **methyl 2-azido-5-bromobenzoate 13e**, yield 20.8 g, 81%; white solid, mp: 71–72°C; MS: (CI) *m*/*z* (%) = 256 (100%) [M+H$^+$], 258 (100%) [M+H$^+$]; **dimethyl 2-azidobenzene-1,4-dicarboxylate 13f**, yield 17.7 g, 75%; white solid, mp: 83–85°C; MS: (CI) *m*/*z* (%) = 236 (100%) [M+H$^+$]. (Caution! All azides are potentially explosive and should not be heated).

General procedure for the synthesis of 1*H*-1,2,3-triazol-5-amines 9a–c and [1,2,3]triazolo[1,5-*a*]quinazolin-5(4*H*)-one 14a–o. To the solution of sodium methoxide (540 mg, 10.0 mmol) in dry methanol (20 mL) an appropriate substituted acetonitrile **1, 2, 5** or **8** (10.0 mmol) was added. To this solution substituted methyl 2-azidobenzoate **2** (10.0 mmol) in dry methanol (2 mL) was added dropwise and the solid started to precipitate. The mixture was stirred for 1 h. The resulting suspension was filtered and the solid product was washed with water and methanol to give the corresponding 1*H*-1,2,3-triazol-5-amines **9a–c** and [1,2,3]triazolo[1,5-*a*]quinazolin-5(4*H*)-ones **14a–o**.

4-(4-Methyl-1,3-thiazol-2-yl)-1-phenyl-1*H*-1,2,3-triazol-5-amine (9a). Yield: 2.2 g (87%); as white crystals; mp 168–169°C (DMF–EtOH); ^{1}H NMR (DMSO-d_6 400MHz): δ 2.45 (s, 3H, Me), 6.47 (s, 2H, NH_2), 6.95 (s, 1H, thiazole), 7.52 (t, ^3J = 7.1 Hz, 1H, H_{Ph}-4), 7.61 (t, ^3J = 7.2 Hz, 2H, H_{Ph}-3,5), 7.66 (d, ^3J = 7.2 Hz, 2H, H_{Ph}-2,6); MS: (CI) *m*/*z* (%) = 258 (100%) [M+H$^+$]. Anal. Calcd. for $C_{12}H_{11}N_5S$ (257.31): C 56.01; H 4.31; N 27.22. Found: C 56.19; H 4.24; N 27.07.

1-Phenyl-4-(5-phenyl-1,3,4-oxadiazol-2-yl)-1*H*-1,2,3-triazol-5-amine (9b). Yield: 2.8 g (93%); as white crystals; mp 214–215°C (DMF–EtOH); ^{1}H NMR (DMSO-d_6 400MHz): δ 6.58 (s, 2H, NH_2), 7.50–7.72(m, 8H, arom.), 8.11–8.16 (m, 2H, H_{Ph}-2,6); MS: (CI) *m*/*z* (%) = 305 (100%) [M+H$^+$]. Anal. Calcd. for $C_{16}H_{12}N_6O$ (304.31): C 63.15; H 3.97; N 27.62. Found: C 63.01; H 3.74; N 27.48.

1-Phenyl-4-(3-phenyl-1,2,4-oxadiazol-5-yl)-1*H*-1,2,3-triazol-5-amine (9c). Yield: 2.7 g (90%); as white crystals; mp 230–231°C (DMF–EtOH); ^{1}H NMR (DMSO-d_6 400MHz): δ 6.80 (s, 2H, NH_2), 7.52–7.59 (m, 5H, arom.), 7.63–7.66 (m, 3H, arom.), 8.16–8.18 (m, 2H, H_{Ph}-2,6); MS: (CI) *m*/*z* (%) = 305 (100%) [M+H$^+$]. Anal. Calcd. for $C_{16}H_{12}N_6O$ (304.31): C 63.15; H 3.97; N 27.62. Found: C 63.06; H 3.88; N 27.64.

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