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ORIGINAL PAPER

Synthesis of novel 5-phenylimidazo[1,2-*c*]quinazolin-3-amine derivatives via Groebke–Blackburn–Bienaymé multicomponent reaction

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Abstract We have developed an efficient and environmentally benign one-pot procedure for synthesis of novel *N*-alkyl-2-aryl-5-phenylimidazo[1,2-*c*]quinazolin-3-amine derivatives by use of the Groebke–Blackburn–Bienaymé multicomponent reaction. The title compounds were synthesized in good yields from 2-phenylquinazolin-4-amine, aromatic aldehydes, and isocyanides in the presence of ammonium chloride under solvent-free conditions.

Keywords Groebke–Blackburn–Bienaymé · Isocyanides · Multicomponent reactions · Phenylimidazo[1,2-*c*]quinazolines

Introduction

Fused imidazo[1,2-*c*]quinazoline heterocycles are of interest to medicinal chemists because of their wide variety of pharmacological and biological properties. In this regard, versatile strategies for obtaining new structures are of great interest to synthetic chemists [1]. Some imidazo[1,2-*c*]quinazolines synthesized by Bourguignon et al. [2] act as coronary smooth muscle relaxants, and Hansen et al. [3] have reported they are also useful for preparation of psychopharmaceuticals, for example anticonvulsants, anxiolytics, hypnotics, antipsychotics, and antiemetics, for improving cognitive function of the brains of mammals, or as benzodiazepine antagonists. Imidazo[1,2-*c*]quinazolines synthesized by Chern et al. [4] were useful as active ingredients for treatment of hypertension. Their antioxidant activity acts as a potent inhibitor of lipid peroxidation [5]. Literature survey shows that 6-arylbenzimidazo[1,2-*c*]quinazolines act as inhibitors of LPS-induced TNF-alpha secretion [6]. Therefore, new synthetic methods for synthesis of imidazo[1,2-*c*]quinazolines need active study.

In recent years there has been much interest in the use of solvent-free conditions. Because there is no reaction medium to collect, purify, and recycle, they result in reduced pollution, low cost, and simplicity in process and handling, which are very important in industry [7]. Sequential solvent-free reactions are possible in high-yielding systems [8]. In recent years, solvent-free conditions for synthesis of heterocyclic compounds have become increasingly significant and are regarded as important and useful synthetic methods [9].

To the best of our knowledge, there is no report of the synthesis of *N*-alkyl-2-aryl-5-phenylimidazo[1,2-*c*]quinazolines. Herein, in the context of our interest in designing new heterocyclic structures [10–12], we wish to report a green procedure for synthesis of novel imidazo[1,2-*c*]quinazolines **6** by use of the Groebke–Blackburn–Bienaymé multicomponent reaction under solvent-free conditions (Scheme 1).

Results and discussion

We have developed a method for synthesis of novel imidazo[1,2-*c*]quinazolines **6** based on the Groebke–

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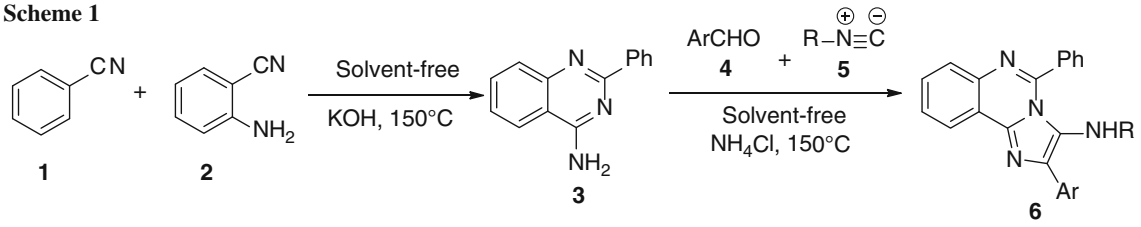
Scheme 1



Blackburn–Bienaymé three-component reaction of heteroaromatic amidines, aldehydes, and isocyanides for synthesis of fused 3-aminoimidazoles [13–15]. In their work, heteroaromatic amidines, for example 2-aminopyridine or pyrimidine, were reacted with isocyanides and aldehydes in the presence of a catalytic amount of protic acids. This led to reports of the use of different heteroamidines and catalysts [16–20].

During our investigations, we found that 2-phenylquinazolin-4-amine (**3**) [21] is a very good substrate with excellent performance in cyclization reactions. It is worth mentioning that **3** can easily be prepared by a green procedure, by reaction of benzonitrile (**1**) and 2-aminobenzonitrile (**2**) in the presence of KOH under solvent-free conditions at 150 °C (Scheme 1).

In a preliminary study, reaction of **3** (1 mmol), 2-fluorobenzaldehyde (1 mmol), and *tert*-butyl isocyanide (1 mmol) was selected as model reaction; the substrates were reacted in different solvents and in the absence of solvent. Because the solvent can be of crucial importance in organic reactions, such solvents as toluene, methanol, ethanol, and acetonitrile were used either at room temperature or under reflux. Some results are summarized in Table 1. However, examination of solvents revealed that they suppressed or did not improve the cyclization reaction, and formation of the desired product, *N*-*tert*-butyl-2-(2-fluorophenyl)-5-phenylimidazo[1,2-*c*]quinazolin-3-amine (Table 2, **6a**) did not occur. Surprisingly, when the model reaction was conducted under solvent-free conditions, the starting materials were consumed (according to TLC) in 5 h, and the expected product **6a** was obtained in 45 % yield.

Because the efficiency of ammonium chloride in isocyanide-based multicomponent reactions has been proved [10, 22–24], the model reaction was conducted in the presence of NH_4Cl. It was found that use of ammonium chloride (1 mmol) is essential to obtain the corresponding product **6a** in good yield and shorter reaction time (83 %, 2 h).

Compound **6a** furnished well-defined ^1H NMR and ^{13}C NMR spectra. The structure was also confirmed by analysis of the mass spectroscopy fragmentation pattern. The peak at $m/z = 410.19$ was in accordance with the calculated mass for $C_{26}H_{23}FN_4$. The mass spectrum contained a strong peak at $m/z = 354$ which is related to elimination of *tert*-butyl. Another peak was observed at $m/z = 334$, resulting from the loss of Ph. The strongest peak was observed at $m/z = 205$, related to formation of the 2-phenylquinazolinium ion. After confirmation of the structure of **6a**, and using the optimized conditions, we next set out to investigate the scope of the reaction. For this purpose,

Table 1 Optimization of the reaction conditions

Entry	Solvent	Conditions	Additive	Time/h	Yield/%[a]
1	$PhCH_3$	Reflux	–	24	Trace
2	$PhCH_3$	Reflux	NH_4Cl (1 mmol)	24	10
3	CH_3CN	Reflux	–	24	Trace
4	EtOH	Reflux	–	24	5
5	MeOH	Reflux	–	24	30
6	MeOH	Reflux	NH_4Cl (1 mmol)	24	42
7	Solvent-free	150 °C	–	5	45
8	Solvent-free	150 °C	NH_4Cl (1 mmol)	2	83

Conducted with 2-phenylquinazolin-4-amine (1 mmol), 2-fluorobenzaldehyde (1 mmol), and *tert*-butyl isocyanide (1 mmol)

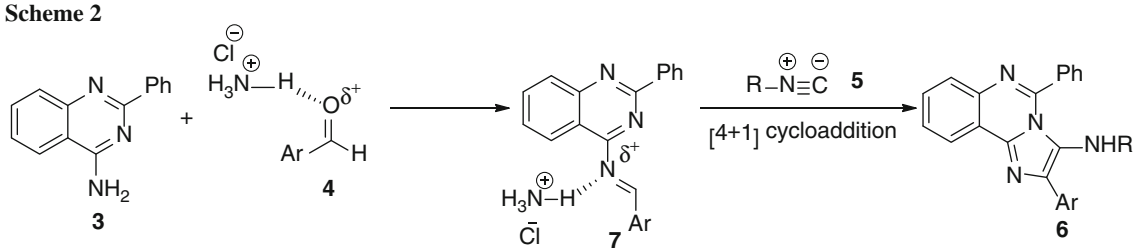
[a] Yield of isolated product

Table 2 Synthesis of 5-phenylimidazo[1,2-*c*]quinazolin-3-amines **6** (Scheme 1)

Entry	Ar	R	Product	Yield/%[a]
1	2-F-C_6H_4	*tert*-Butyl	**6a**	83
3	4-Cl-C_6H_4	Cyclohexyl	**6b**	75
4	4-Pyridinyl	Cyclohexyl	**6c**	82
6	4-NO_2-C_6H_4	Cyclohexyl	**6d**	58
5	4-F-C_6H_4	*tert*-Butyl	**6e**	88
6	4-NO_2-C_6H_4	*tert*-Butyl	**6f**	71
7	4-MeO-C_6H_4	*tert*-Butyl	**6g**	83
8	3,4,5-tri-MeO-C_6H_2	*tert*-Butyl	**6h**	77

[a] Yield of isolated product

Scheme 2



different aromatic aldehydes and isocyanides were reacted with 2-phenylquinazolin-4-amine **3** in the presence of ammonium chloride; the corresponding compounds **6** were obtained in good yields (Table 2). The results showed that the reaction is compatible with different benzaldehydes including those with electron-donating and electron-withdrawing substituents at different positions.

The sequence of reactions used for preparation of imidazo[1,2-*c*]quinazolines **6** is depicted in Scheme 2. The reaction begins by formation of iminium ion **7** by condensation of the 2-phenylquinazolin-4-amine **3** and aromatic aldehydes **4**, probably catalyzed by NH_4Cl. Nonconcerted [4 + 1] cycloaddition between **7** and isocyanide **5** then leads to formation of the product **6** [13–15].

Conclusion

We report a convenient, green method for preparation of *N*-alkyl-2-aryl-5-phenylimidazo[1,2-*c*]quinazoline-3-amine derivatives by three-component reaction of 2-phenylquinazolin-4-amine, aromatic aldehydes, and isocyanides in the presence of ammonium chloride under solvent-free conditions. The main advantages of the procedure include preparation of new compounds in the absence of expensive catalyst and solvent, good yields, and product diversity. It is worth mentioning that better yields of products and shorter reaction times were obtained under solvent-free conditions than when a variety of different solvents were used. Use of solvent-free conditions is vital for environmental reasons, among other advantages.

Experimental

All chemicals were purchased from Merck and Sigma–Aldrich and used without further purification. Melting points were taken on a Kofler hot-stage apparatus. ^1H and ^{13}C NMR spectra were recorded on a Bruker FT-500, with TMS as internal standard. IR spectra were obtained on a Nicolet Magna FTIR 550 spectrophotometer in KBr. Mass spectra were determined on an Agilent Technology (HP) mass spectrometer operating at an ionization potential of 70 eV. Elemental analysis was performed with an Elementar Analysensystem VarioEL in CHNS mode.

General procedure for synthesis of 5-phenylimidazo[1,2-c]quinazolin-3-amine derivatives ***6***

A mixture of 0.26 g 2-phenylquinazolin-4-amine **3** (1 mmol), aromatic aldehyde **4** (1 mmol), isocyanide **5** (1.2 mmol), and ammonium chloride (1 mmol) was stirred at 150 °C for 2–3 h. After completion of the reaction (checked by TLC) the mixture was cooled to room temperature and the residue was purified by column chromatography using silicagel and petroleum ether–ethyl acetate (5:1) as eluent.

N-tert-Butyl-2-(2-fluorophenyl)-5-phenylimidazo[1,2-c]-quinazolin-3-amine (**6a**, $C_{26}H_{23}FN_4$)
Yellow crystals; m.p.: 164–166 °C; ^1H NMR (500 MHz, $CDCl_3$): $\delta = 8.66$ (d, $J = 6.8$ Hz, 1H, H_7), 8.00 (d, $J = 7.4$ Hz, 2H, H_{10}, $H_{6'}$), 7.81–7.79 (m, 2H, H_8, H_9), 7.70–7.53 (m, 5H, Ph), 7.37–7.30 (m, 2H, $H_{3'}$, $H_{5'}$), 7.18 (t, $J = 6.8$ Hz, 1H, $H_{4'}$), 2.75 (d, $J = 6.0$ Hz, 1H, NH), 0.60 (s, 9H, *tert*-butyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 159.3$ (d, $J_{C–F} = 244.1$ Hz), 147.2, 141.3, 140.3, 134.4, 133.9, 131.5, 129.5, 129.4, 129.3, 129.2, 127.8, 127.6, 127.5, 127.0, 124.2, 122.2 (d, $J_{C–F} = 31.0$ Hz), 121.8, 118.0, 115.2 (d, $J_{C–F} = 22.7$ Hz), 55.2, 28.5 ppm; IR (KBr): $\bar{\nu} = 3{,}394$, 3,237, 2,968, 2,863, 1,614, 1,595, 1,553 cm^{-1}; MS (70 eV): $m/z = 410.19$ (M^+).

2-(4-Chlorophenyl)-N-cyclohexyl-5-phenylimidazo[1,2-c]-quinazolin-3-amine (**6b**, $C_{28}H_{25}ClN_4$)
Yellow crystals; m.p.: 259–261 °C; ^1H NMR (500 MHz, $CDCl_3$): $\delta = 8.66$ (d, $J = 7.6$ Hz, 1H, H_7), 8.28 (d, $J = 8.5$ Hz, 2H, $H_{2'}$, $H_{6'}$), 7.97 (d, $J = 8.3$ Hz, 1H, H_{10}), 7.77–7.76 (m, 2H, H_8, H_9), 7.70–7.63 (m, 5H, Ph), 7.41 (d, $J = 8.5$ Hz, 2H, $H_{3'}$, $H_{5'}$), 2.42 (s, 1H, NH), 2.30–2.26 (m, 1H, NCH), 1.39–0.62 (m, 10H, cyclohexyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 145.1$, 141.2, 140.3, 134.9, 130.6, 130.1, 129.5, 129.3, 129.2, 128.6, 128.5, 128.3, 128.2, 127.8, 127.5, 125.4, 125.3, 122.0, 56.3, 31.9, 29.0, 28.2 ppm; IR (KBr): $\bar{\nu} = 3{,}415$, 3,231, 3,131, 2,924,

1,614, 1,565 cm^{-1}; MS (70 eV): $m/z = 454.17$ ($M^+ + 2$), 452.18 (M^+).

N-Cyclohexyl-5-phenyl-2-(pyridine-4-yl)imidazo[1,2-c]-quinazolin-3-amine (**6c**, $C_{27}H_{25}N_5$)
Yellow crystals; m.p.: 204–206 °C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 8.68$–8.63 (m, 3H, $H_{3'}$, $H_{5'}$, H_7), 8.28–8.25 (m, 2H, $H_{2'}$, $H_{6'}$), 7.96 (d, $J = 7.6$ Hz, 1H, H_{10}), 7.77–7.74 (m, 2H, H_8, H_9), 7.70–7.65 (m, 5H, Ph), 2.53 (d, $J = 4.1$ Hz, 1H, NH), 2.35–2.30 (m, 1H, NCH), 1.44–0.61 (m, 10H, cyclohexyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 148.5$, 145.7, 141.9, 141.0, 140.2, 134.0, 131.9, 130.3, 129.7, 128.8, 128.5, 128.0, 127.9, 127.6, 121.9, 121.1, 118.3, 57.6, 32.2, 24.9, 24.1 ppm; IR (KBr): $\bar{\nu} = 3{,}379$, 3,128, 2,928, 2,852, 1,712, 1,598, 1,572 cm^{-1}; MS (70 eV): $m/z = 419.21$.

N-Cyclohexyl-2-(4-nitrophenyl)-5-phenylimidazo[1,2-c]-quinazolin-3-amine (**6d**, $C_{28}H_{25}N_5O_2$)
Yellow crystals; m.p.: 260–261 °C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 8.66$ (dd, $J = 8.3$, 1.7 Hz, 1H, H_7), 8.54 (d, $J = 9.0$ Hz, 2H, $H_{3'}$, $H_{5'}$), 8.29 (d, $J = 9.0$ Hz, 2H, $H_{2'}$, $H_{6'}$), 7.98 (dd, $J = 8.3$, 1.4 Hz, 1H, H_{10}), 7.78–7.76 (m, 2H, H_8, H_9), 7.73–7.66 (m, 5H, Ph), 2.53 (d, $J = 5.1$ Hz, 1H, NH), 2.34–2.33 (m, 1H, NCH), 1.60–0.88 (m, 10 H, cyclohexyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 145.9$, 145.6, 141.0, 140.3, 140.2, 134.0, 130.4, 130.3, 129.8, 128.6, 128.3, 128.1, 127.9, 127.7, 127.3, 123.1, 122.0, 118.2, 57.6, 32.2, 24.9, 24.1 ppm; IR (KBr): $\bar{\nu} = 3{,}376$, 3,119, 2,926, 2,853, 1,596, 1,569, 1,336 cm^{-1}; MS (70 eV): $m/z = 463.20$.

N-tert-Butyl-2-(4-fluorophenyl)-5-phenylimidazo[1,2-c]-quinazolin-3-amine (**6e**, $C_{26}H_{23}FN_4$)
Yellow crystals; m.p.: 165–167 °C; ^{1}H NMR (500 MHz, DMSO-d_6): $\delta = 8.49$ (d, $J = 6.6$ Hz, 1H, H_7), 7.95–7.71 (m, 6H, ArH), 7.58–7.33 (m, 6H, ArH), 3.12 (bs, 1H, NH), 0.34 (s, 9H, *tert*-butyl) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 159.5$ (d, $J_{C-F} = 245.5$ Hz), 147.4, 140.3 (d, $J_{C-F} = 45.0$ Hz), 134.5, 132.3, 130.0, 129.7, 128.4, 128.2 (d, $J_{C-F} = 23.5$ Hz), 127.9, 127.5, 127.3, 124.7, 124.5, 122.7 (d, $J_{C-F} = 13.4$ Hz), 121.8, 118.1, 115.6 (d, $J_{C-F} = 21.5$ Hz), 54.8, 28.7 ppm; IR (KBr): $\bar{\nu} = 3{,}421$, 2,975, 1,654, 1,597, 1,557, 1,488 cm^{-1}; MS (70 eV): $m/z = 410.19$.

N-tert-Butyl-2-(4-nitrophenyl)-5-phenylimidazo[1,2-c]-quinazolin-3-amine (**6f**, $C_{26}H_{23}N_5O_2$)
Yellow crystals; m.p.: 264–265 °C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 8.67$ (d, $J = 6.3$ Hz, 1H, H_7), 8.54 (d, $J = 7.4$ Hz, 2H, $H_{3'}$, $H_{5'}$), 8.27 (d, $J = 7.4$ Hz, 2H, $H_{2'}$, $H_{6'}$), 7.98 (d, $J = 7.4$ Hz, 1H, H_{10}), 7.78–7.76 (m, 2H, H_8, H_9), 7.72–7.66 (m, 5H, Ph), 2.52 (s, 1H, NH), 0.53 (s, 9H, *tert*-butyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 146.2$, 146.1, 141.9, 141.0, 140.6, 136.0, 134.7, 130.3, 129.9, 128.7, 128.4, 128.1, 128.0, 127.6, 127.1, 122.9, 122.0, 118.0, 56.8, 28.9 ppm; IR (KBr): $\bar{\nu} = 3{,}390$, 2,967, 1,599, 1,516, 1,466, 1,336 cm^{-1}; MS (70 eV): $m/z = 437.19$ (M^+).

N-tert-Butyl-2-(4-methoxyphenyl)-5-phenylimidazo[1,2-c]-quinazolin-3-amine (**6g**, $C_{27}H_{26}N_4O$)
Yellow crystals; m.p.: 163–165 °C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 8.71$ (d, $J = 8.0$ Hz, 1H, H_7), 8.18–7.95 (m, 3H, H_8, H_9, H_{10}), 7.78 (d, $J = 7.0$, 2H, $H_{2'}$, $H_{6'}$), 7.68–7.61 (m, 5H, Ph), 6.95 (d, $J = 7.0$ Hz, 2H, $H_{3'}$, $H_{5'}$), 3.84 (s, 3H, OCH_3), 2.41 (s, 1H, NH), 0.53 (s, 9H, *tert*-butyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 158.7$, 146.4, 143.6, 143.5, 141.1, 141.0, 140.5, 135.0, 129.9, 129.3, 128.4, 128.3, 127.6, 127.4, 126.7, 124.6, 122.0, 113.0, 55.9, 54.7, 28.9 ppm; IR (KBr): $\bar{\nu} = 3{,}380$, 2,960, 2,837, 1,613, 1,567, 1,506 cm^{-1}; MS (70 eV): $m/z = 422.21$ (M^+).

N-tert-Butyl-5-phenyl-2-(3,4,5-trimethoxyphenyl)-imidazo[1,2-c]quinazolin-3-amine (**6h**, $C_{29}H_{30}N_4O_3$)
Yellow crystals; m.p.: 208–210 °C; ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 8.74$ (d, $J = 7.5$ Hz, 1H, H_7), 7.97 (d, $J = 7.5$ Hz, 1H, H_{10}), 7.80–7.78 (m, 2H, H_8, H_9), 7.67–7.60 (m, 7H, ArH), 3.99 (s, 6H, OCH_3), 3.91 (s, 3H, OCH_3), 2.42 (s, 1H, NH), 0.59 (s, 9H, *tert*-butyl) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 152.9$, 152.0, 146.6, 141.4, 141.1, 139.2, 137.9, 135.4, 130.5, 130.1, 129.0, 128.7, 128.3, 128.0, 127.2, 125.5, 122.7, 105.9, 61.0, 56.6, 56.3, 29.4 ppm; IR (KBr): $\bar{\nu} = 3{,}357$, 2,964, 1,605, 1,589, 1,500, 1,460 cm^{-1}; MS (70 eV): $m/z = 482.23$ (M^+).

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