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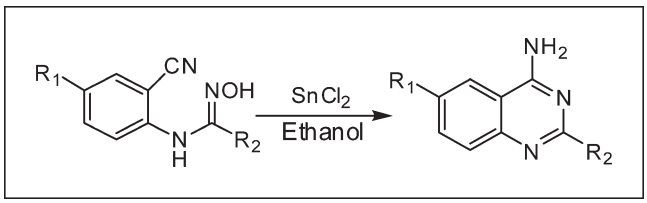

Preparation of 4-amino-2-aryl quinazolines by reduction of *N*-(2-cyanoaryl) amidoximes *via* amidines using $SnCl_2$ in ethanol.

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INTRODUCTION

Quinazoline, owing to its pharmacological properties, is an important heterocycle [1]. Quinazolines are well known to possess an array of physiological activities, e.g., anticancer [2], antifungal [3], antimalarial [4], anti-inflammatory [5], and antihypertensive [6]. Because of numerous applications in medicinal chemistry, the preparation of 4-aminoquinazoline derivatives has gained considerable attention. Many procedures are reported in literature, most of them start from cyanophenyltriazenes [7], *o*-azidobenzonitrile and nitrile [8], *o*-aminobenzonitrile, and benzonitrile [9], rearrangement of 3-amino-1-benzylindazone [10], reaction of *N*-arylbenzimidoyl chloride with cyanamide in the presence of $TiCl_4$ [11], and reaction of 2-aminobenzonitrile with orthoesters and ammonium acetate under solvent-free and microwave condition [12]. Recently in literature, copper-catalyzed reaction of 2-bromobenzonitrile and amidines has been reported [13]. However, these methodologies were not applicable for synthesis of halo-substituted quinazoline derivatives. All these above methods suffer from drawbacks such as harsh reaction condition (high temperature and pressure [9]) and use of toxic reagents. Anthranilonitrile undergoes self-condensation reaction forming 4-amino-2-(2-aminophenyl) quinazoline as a side product (entry **2k**). Therefore, this methodology is not useful for the preparation of cyano-substituted quinazoline derivatives.

RESULTS AND DISCUSSION

Herein, we wish to report a new general way for preparation of 4-amino-2-aryl quinazolines from *N*-(2-cyanoaryl) amidoximes which in turn are easily obtained from reaction of the corresponding anilines and *N*-hydroxyimidoyl chlorides [14] in Scheme 1.

The obtained amidoximes were then subjected to reduction using $SnCl_2$ in ethanol in which intermediate amidine was not isolated, but rather immediately converted *in situ* to 4-aminoquinazolines (Scheme 2). We anticipate that the reduction of amidoxime to amidine by $SnCl_2$ is analogy to the reported work [15].

To optimize the reaction conditions for the conversion of *N*-(2-cyanoaryl) amidoximes to 4-aminoquinazoline, the reactions were carried out with different molar ratios of $SnCl_2$ in appropriate solvents (Table 1). From these studies, ethanol was found to be the best solvent. Reaction also proceeded in DMF but required 48–50 h for completion. In the ethereal solution, the reaction was incomplete; this could be attributed to low solubility of the amidoximes in THF.

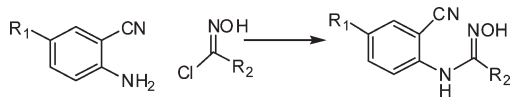
As reported in Table 2, a variety of aromatic and heteroaromatic substrates undergo facile reactions within 8–10 h in high yields. An obvious result was found for nitro-substituted derivative of amidoxime, which gave 4-amino-2-(2-aminophenyl) quinazoline (entry **2k**) using the optimized reaction condition. A noteworthy feature of this reaction is that the extra cyano-substituent of *N*-(2-cyanophenyl)-3-cyanobenzamidoxime remains unaffected (entry **2l** Table 2).

In conclusion, we have developed a rapid high-yielding procedure for conversion of *N*-arylamidoximes to 4-aminoqunazolines *via* amidine. This method provides a significant improvement over the existing methodology for the synthesis of 4-amino-2-aryl quinazolines.

EXPERIMENTAL

Chemicals were procured from Aldrich Chemical (Bangalore, India). Reactions were monitored, and purity of the products was checked by thin layer chromatography (TLC). TLC

Scheme 1. Synthesis of *N*-(2-cyanoaryl) amidoximes from 2-aminobenzonitriles.



was performed on Merck60 F-254 silica gel plates with visualization by UV light. Melting points were determined on a Buchi Melting Point B-545 apparatus. The IR spectra (in KBr pellets) were recorded on a Nicolet 6700 FTIR spectrometry. ^{1}H-NMR spectra were recorded on Bruker (200 MHz) spectrometer instruments, in and DMSO-d_6. Chemical shifts were recorded in parts per million downfield from tetramethylsilane. Mass spectra were recorded on MS-3200Q trap spectrometer. Elemental analysis was performed on a Carlo Erba Perkin-Elmer model 240 analyzer. Analysis results were within 0.4% of the calculated value. Column chromatography was performed on silica gel (230–400 mesh) supplied by Acme Chemical (Mumbai, India). The chemicals and solvents used were laboratory grade and were purified.

General experimental procedure for the synthesis 4-amino-2-phenyl-quinazoline. The procedure for reaction entry **2a** is illustrative. A 150-mL round-bottom flask equipped with stir bar and reflux condenser was dried under an atmosphere of nitrogen and charged with amidoxime (2.36 g, 1 mmol), followed by 20 mL of anhydrous ethanol. Stannous chloride (9.48 g, 5 mmol) was added to this solution, and the mixture was heated at 70°C under nitrogen atmosphere for 8 h. The mixture was distilled to half the volume and cooled at room temperature and rendered basic (pH 8) with 150 mL of 10% aqueous sodium bicarbonate. The mixture was extracted with 3 × 20 mL of ethylacetate. The organic phase was washed thoroughly with saturated NaCl (aq.), dried over sodium sulfate, and filtered through celite. The solvent was removed *in vacuo* to obtain the crude product. The product was purified by column chromatography on silica (20% ethylacetate in hexane), and the desired quinazoline was obtained in 90% yield as pale yellow solid.

General procedure for preparation of hydrochloride salt of 4-amino-2-phenyl-quinazoline. 4-Amino-2-phenyl-quinazoline base (1 g) was dissolved in methanol (5 mL), and dry HCl gas was passed till pH 3. Ethyl acetate (5 mL) was added dropwise to obtain the solid, which was filtered and washed with 1:1 mixture of methanol:ethylacetate.

4-Amino-2-phenyl-quinazoline: (2a). Pale yellow solid, m.p. 145–147°C (lit. [9a] 145–146°C), ^{1}H-NMR (DMSO-d_6, 200 MHz) δ 7.53–7.50 (m, 4H), 7.89–7.80 (m, 4H), 8.07 (d, 1H, J = 8.1 Hz, 1H), 8.54–8.51 (m, 2H), ^{13}C-NMR (DMSO-d_6, 50 MHz) δ 113.8, 124.1, 125.6, 128.2, 128.4, 128.7, 130.4, 133.5, 139.1, 150.9, 160.2, 162.6, IR (KBr) η_{max} (cm^{-1}): 3340, 1640, 1565. MS (EI): m/z = 221 (M + 1 = 222). Anal. Calcd. for $C_{14}H_{11}N_3$: C, 76.00; H, 5.01; N, 18.19. Found: C, 76.06; H, 5.04; N, 18.13.

4-Amino-6-chloro-2-phenyl-quinazoline: (2b). Pale yellow solid, m.p. 167–170°C, hydrochloride salt −233 to 235°C (lit [11] 230–231°C) ^{1}H-NMR (200 MHz, DMSO-d_6): 7.46–7.54 (m, 3H), 7.56 (bs, 2H), 7.76–7.78 (m, 2H), 8.0 (s, 1H), 8.42 (m, 2H), ^{13}C-NMR (50 MHz, DMSO-d_6): δ 117.6, 121.8, 128.4, 129.0, 129.4, 129.6, 130.5, 131.0, 136.3, 151.1, 160.3, 161.05. MS (EI): m/z = 255(M + 1 = 256.2, M + 2 = 258.1). Anal. Calcd. for $C_{14}H_{10}ClN_3$: C, 65.73; H, 3.94; N, 16.43. Found: C, 65.68; H, 3.90; N, 16.39.

4-Amino-2-(p-tolyl)-quinazoline: (2c). Pale yellow solid m.p. 134–136°C, ^{1}H-NMR (200 MHz, DMSO-d_6): δ 2.39 (s, 3H, —CH_3), 7.29 (d, 2H, J = 8.0 Hz), 7.42–7.50 (m, 3H), 7.80 (bs, 2H, NH_2), 8.23 (d, 1H), 8.40 (d, 2H, J = 8.0 Hz), ^{13}C-NMR (50 MHz, DMSO-d_6): δ 21.4, 112.9, 121.7, 125.4, 128.2, 128.4, 129.1, 133.1, 135.7, 140.1, 150.8, 160.9, 161.6. IR (KBr) η_{max} (cm^{-1}): 3317, 1645, 1566, 1436, MS (EI): m/z=235 (M + 1= 235.9), Anal. Calcd. for $C_{15}H_{13}N_3$: C, 76.57; H, 5.57; N, 17.86. Found: C, 76.52; H, 5.58; N, 17.88.

4-Amino-6-chloro-2-(p-tolyl) quinazoline: (2d). Pale yellow solid, m.p. 167–170°C, ^{1}H-NMR (200 MHz, DMSO-d_6): δ 2.38 (s, 3H, CH_3), 7.24 (d, 2H, J = 8.4 Hz), 7.75 (bs, 2H), 7.79–8.05 (m, 2H), 8.09 (s, 1H), 8.25 (d, 2H, J = 8.4 Hz), MS (EI): m/z=269 (M + 1=270.2, M+2=272.1). Anal. Calcd. for $C_{15}H_{12}ClN_3$ C, 66.79; H, 4.48; N, 15.58. Found: C, 66.72; H, 4.54; N, 15.54.

4-Amino-2-(4-flurophenyl)-quinazoline:(2e). Pale yellow solid, m.p. 170°C, ^{1}H-NMR (200 MHz, DMSO-d_6): δ 7.03 (m, 2H), 7.74 (m, 1H), 7.76 (m, 2H), 7.88 (bs, 2H), 8.24 (d, 1, H), 8.52 (m, 2H), ^{13}C-NMR (50 MHz, DMSO-d_6): δ 111.9, 113.2, 113.6, 122.1, 123.7, 126.2, 128.7, 128.9, 131.4, 133.6, 149.0, 157.9, 160.8, 164.9. Anal. Calcd. for $C_{14}H_{10}FN_3$ C, 70.28; H, 4.21; N, 17.56. Found: C, 70.21; H, 4.27; N, 17.53.

6-Methyl-2-phenyl-4-aminoquinazoline: (2f). White crystalline solid hydrochloride salt, m.p. 262–264°C (lit. [11] 260–260°C), ^{1}H-NMR (200 MHz DMSO-d_6) δ 2.48 (s, 3H), 7.47 (m, 3H), 7.71–7.60 (m, 4H), 8.05 (m, 1H), 8.46–8.43 (m, 2H), ^{13}C-NMR (50 MHz, DMSO-d_6) δ 21.7, 113.6, 123.1, 128.0, 128.2, 128.6, 130.2, 135.1, 135.1, 139.1, 149.6, 160.0, 162.2.

4-Amino-2-(4-pyridyl)-quinazoline: (2g). Pale yellow solid hydrochloride salt, m.p. 280–283°C (lit [6] 280°C), ^{1}H-NMR (200 MHz, DMSO-d_6): δ 6.50 (bs, 2H), 7.52–7.63 (m, 2H), 7.66–7.94 (m, 2H), 8.22 (d, 2H, J = 8.0 Hz), 8.66 (d, 2H, J = 8.0 Hz), ^{13}C-NMR (50 MHz, DMSO-d_6): 118.0, 121.1, 124.4, 128.4, 130.3, 132.9, 142.2, 149.5, 150.2, 160.4, 162.9. MS (EI): m/z = 222 (M + 1= 223).

4-Amino-2-(2-pyridyl)-quinazoline: (2h). Yellow solid as hydrochloride salt, m.p. 235–237°C (lit [6] 235°C), ^{1}H-NMR (200 MHz, DMSO-d_6) δ 7.59 (m, 1H), 7.72 (bs, NH_2, 2H) 7.78 (ddd, J = 8.2, 6.9, 1.2 Hz, 1H), 8.05 (m, 2H), 8.14 (m, 1H), 8.35 (d, J = 8.2 Hz, 1H), 8.58 (d, J = 7.7 Hz, 1H), 8.82 (1H, d, J = 4.3 Hz,1H); ^{13}C-NMR (50 MHz, DMSO-d_6) δ, 119.6 (C), 123.4, 124.3, 125.5, 126.2, 128.6, 129.3 (CH), 130.7 (C), 134.91 (CH), 149.9 (C), 150.1 (CH) 158.7, 169.7 (C).

Scheme 2. Synthesis 4-amino-2-phenyl substituted quinazolines from *N*-(2-cynoaryl) amidoximes *via* amidine using $SnCl_2$.

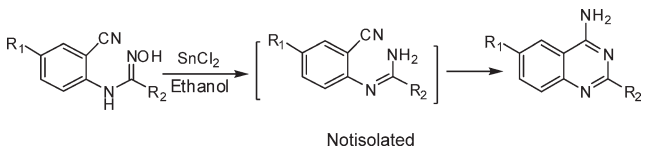
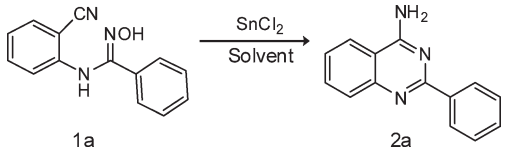


Table 1

Optimization of reagents and reaction condition.[a]



1a 2a

Entry	Reagent (mol eq)	Solvent	Time (h)	Yield[b] (%)
1	Amidoxime/$SnCl_2$ (1:1)	Ethanol	5	No reaction
2	Amidoxime/$SnCl_2$ (1:2)	Ethanol	5	No reaction
3	Amidoxime/$SnCl_2$ (1:3)	Ethanol	10	30
4	Amidoxime/$SnCl_2$ (1:5)	Ethanol	8	90
5	Amidoxime/$SnCl_2$ (1:5)	DMF	48	70
6	Amidoxime/$SnCl_2$ (1:5)	THF	10	No reaction

[a] Reactions were carried out on a 1mmol scale at reflux temperature.
[b] Yields obtained after column chromatography.

4-Amino-2-(4-methoxyphenyl)-quinazoline: (2i). Pale yellow solid hydrochloride, m.p. 255–257°C (lit [16] 256–259°C) ^{1}H-NMR (200 MHz, DMSO-d_6): δ 3.83 (s, OCH_3, 3H), 6.97 (d, J = 9.0 Hz, 2H), 7.50 (ddd, J = 8.2, 7.0, 1.2 Hz, 1H), 7.76 (bs, NH_2, 2H), 7.81 (m, 2H), 7.97(d, J = 8.3 Hz, 1H), 8.50 (d, J = 9.0 Hz, 2H).

4-Amino-6-bromo-2-phenyl-quinazoline: (2j). Pale yellow solid hydrochloride salt, m.p. 186–189°C (lit [11] 188–190°C) ^{1}H-NMR (200 MHz, DMSO-d_6) 6.58 (bs, NH_2, 2H), 7.50 (td, J = 7.7, 1.1 Hz, 1H), 7.56 (dt, J = 8.7, 1.9 Hz, 2H), 7.78 (td, J = 7.7, 1.4 Hz, 1H), 7.98 (d, J = 8.9 Hz, 1H), 8.01 (d, J = 8.9 Hz, 1H), 8.42 (dt, J = 8.7, 1.9 Hz, 2H); ^{13}C-NMR (50 MHz, DMSO-d_6): 122.0 (C), 124.0 (CH), 124.1 (C), 126.1, 128.2, 129.1, 130.7, 132.6 (CH), 136.2, 149.2, 158.1, 167.3 (C).

4-Amino-2-(2-aminophenyl)-quinazoline: (2k). Yellow solid hydrochloride, m.p. 286–289°C (lit [7] 287–288°C) ^{1}H-NMR (200 MHz, DMSO-d_6) δ 3.73 (bs, 2H), 6.69–7.12 (m, 3H), 6.83 (bs, 2H), 7.63–7.98 (m, 4H), 8.23(m, 1H).

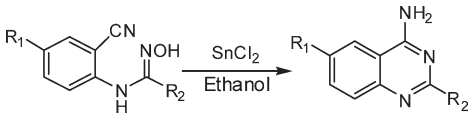
4-Amino-2-(3-cynophenyl)-quinazoline: (2l). Pale yellow solid, m.p. 225–227°C (lit [9b] 226–227°C) ^{1}H-NMR (200 MHz, DMSO-d_6): δ 7.62–7.97 (m, 5H), 8.20 (m, 1H), 8.39 (s, 1H), 8.59 (m, 1H), 9.75 (bs, 2H). ^{13}C-NMR (50 MHz, DMSO-d_6): δ 109.9, 113.6, 118.6, 118.7, 125.2, 127.0, 128.0, 129.1, 129.7, 132.3, 136.7, 139.4, 151.8, 160.3, 162.9.

4-Amino-2-(2-furanyl)-quinazoline: (2m). Pale yellow solid, m.p. 216–218°C (lit [6a] 219–221°C) ^{1}H-NMR (200 MHz, DMSO-d_6) 6.53 (dd, J = 3.4, 1.8 Hz, 1H), 7.38 (dd, J = 3.4, 0.8 Hz, 1H), 7.49 (ddd, J = 8.2, 7.0,1.2 Hz, 1H), 7.60 (m, 1H), 7.77 (ddd, J = 8.4, 6.9, 1.2 Hz, 1H), 7.99 (m, 2H) ^{13}C-NMR (50 MHz, DMSO-d_6): δ 111.0, 112.7 (CH), 121.6 (C), 124.2, 126.0, 128.1, 132.7, 144.1 (CH), 147.8, 149.2, 164.2, 167.7 (C).

4-Amino-2-(2-thiophenyl)-quinazoline:(2n). Pale yellow solid, m.p. 189–190°C (lit [9b]188–190°C) ^{1}H-NMR (200 MHz, DMSO-d_6): δ 7.59 (m, 1H), 7.69 (m, 3H), 7.85 (dd, J = 1.6 Hz), 8.22 (d, J = 4.4 Hz, 1H), 8.25 (ddd, J = 4.4 Hz, 1.6 Hz 1H), ^{13}C-NMR (50 MHz, DMSO-d_6): 112.0, 122.1, 123.6, 124.1,125.6, 126.1, 126.4, 131.5, 141.5, 149.1, 156.3, 160.9.

Table 2

Synthesis of substituted 4-aminoquinazolines.[a]



Entry	R_1	R_2	Time (h)	Yield[b] (%)
2a	H	C_6H_5	8	90
2b	Cl	C_6H_5	8	92
2c	H	p-CH_3-C_6H_4	6	91
2d	Cl	p-CH_3-C_6H_4	7	88
2e	H	p-F-C_6H_4	10	72
2f	CH_3	C_6H_5	10	68
2g	H	4-pyridyl	9	83
2h	H	2-pyridyl	10	73
2i	H	p-OCH_3-C_6H_4	8	98
2j	Br	C_6H_5	8	92
2k[c]	H	2-$NH_2C_6H_4$	12	59
2l	H	3-CN-C_6H_4	11	72
2m	H	2-furyl	6	87
2n	H	2-thienyl	8	83

[a]Reactions were carried out on a 1-mmol scale in ethanol at reflux temperature with $SnCl_2$ (1:5 equiv).
[b]Yields obtained after column chromatography.
[c] Obvious result for entry **2k** where in amidoxime **1** R_2 functionality is 2-NO_2-C_6H_4.

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